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FINAL
Sampling and Analysis Plan
(Field Sampling Plan and Quality Assurance Project Plan)

Pre-Design Investigation For Soil At Lower Subase

Volume 2 - Appendices B through G

Naval Submarine Base New London

Groton, Connecticut

Naval Facilities Engineering Command Mid-Atlantic

Contract Number N62470-08-D-1001

Contract Task Orders WE24 and WE57

August 2010

APPENDIX B

KRIGING AND UNCERTAINTY TECHNICAL MEMORANDUMS AND FIGURES

B-1 TECHNICAL MEMORANDUM ON KRIGING OF SOIL DATA FOR LOWER SUBASE SOIL PDI SAP

B-2 TECHNICAL MEMORANDUM ON UNCERTAINTY MODELING IN SUPPORT OF SAMPLING DESIGN FOR LOWER SUBASE SOIL PDI SAP

B-3 KRIGING AND UNCERTAINTY FIGURES (FIGURES B-1 THROUGH B-143)

B-1 TECHNICAL MEMORANDUM ON KRIGING OF SOIL DATA FOR LOWER SUBASE SOIL PDI SAP

TECHNICAL MEMORANDUM
KRIGING OF SOIL DATA FOR LOWER SUBASE SOIL PDI SAP
NSB-NLON, GROTON, CONNECTICUT

By: S. Paxton

Date: July 15, 2010

INTRODUCTION

In support of the Lower Subase Soil Pre-Design Investigation (PDI) Sampling and Analysis Plan (SAP) and Lower Subase Feasibility Study, three-dimensional modeling was performed using C Tech's Mining Visualization System (MVS) software, Version 9.22. MVS is a state-of-the-art visualization software package with a robust series of modules to assist in the creation of three-dimensional volumetric models for environmental analysis. This software was used to create estimated concentration distribution maps for polynuclear aromatic hydrocarbons (PAHs), total petroleum hydrocarbons (TPH), and lead in soil at Zones 1 through 4 and Zone 7 at the Lower Subase.

EXTENT OF CONTAMINATION

Kriging is a group of statistical techniques for estimating the value of an attribute at any unsampled location using a number of available data near the unsampled location. The process essentially provides weights for the available data in the local neighborhood and uses linear regression estimators to estimate the data values at unsampled locations. During the interpolation process, the estimation or error variance is minimized. MVS conducts the kriging process automatically. MVS uses the ordinary kriging method for interpolation. Ordinary kriging is a specific type of kriging technique which allows one to account for variations of local means of data.

When conducting kriging using MVS, input data (i.e., analytical results with x-, y-, z- coordinates) need to be provided and kriging parameters need to be specified. This input information is discussed below in detail.

Input Data

For this project, the soil data from Zones 1 through 4 were assessed together and the data from Zone 7 were assessed separately. Zones 1 through 4 were analyzed together due to the proximity of the zones to each other and the spatial correlation among the data. Zone 7 was assessed separately due to its spatial detachment from Zones 1 through 4. Zones 5 and 6 did not have good data coverage spatially

and the spatial correlation of the data could not be quantified due to a limited number of data, so it was decided that modeling in the two zones would not be beneficial.

The input data provided to MVS for kriging include the following:

- X and Y coordinates for each sample location
- Elevation in feet for each sample location
- Sample result in mg/kg
- Ground Surface Elevation in feet for each sample location

All available data were provided to MVS. For locations where there were duplicate results, the software first calculates an average and then only uses the average as the data point. After the averaging, all data points were assumed to be equally weighted for kriging.

Kriging Parameters

Grid

Based on the data density, it was agreed that the grid spacing is five-foot in both x- and y- directions horizontally and two-foot vertically. Provided below are the number of nodes of the kriging domains for the two established zones (i.e., Zone 1 through 4 and Zone 7):

- Zones 1 through 4
  x = 201
  y = 364
  z = 10

- Zone 7
  x = 94
  y = 159
  z = 10

Examples of the calculations to determine the number of nodes in the kriging domains are provided in Attachment A to this memorandum. An example figure (e.g., Figure 17-22) is also included in Attachment A that shows the grid spacing used for kriging Zone 7 data.

The same grid was used for Uncertainty Analysis (discussed in separate memorandum). After kriging, the kriging results were exported into a text format file and imported into the Geostatistical Software Library (GSLIB) for the Uncertainty Analysis. Because GSLIB uses cell data for analysis while MVS uses nodal data, the kriging domain is slightly smaller than the domain for uncertainty analysis; however, these differences were noticed and accounted for in the uncertainty analysis.

Searching Mechanism

When kriging, it is important to specify reasonable Reach and Point values. The Reach and Point values together define a searching window that controls how many sample data will be used in kriging at a specific location. The Reach indicates a radial distance from any model node and essentially controls the size of the searching window. Samples outside of the searching window will not be used in interpolation at the specific location. The Point parameter indicates the maximum number of closest sample data that will be used within the searching window during analysis

The Reach value for kriging was generated automatically by the MVS software. By setting zero (default value) for the Reach in MVS, the software automatically calculates a Reach value that is approximately two-thirds of the longest distance between any two data points in the data set. In this way, the Reach value for each zone and parameter is unique. Examination of data values and data density and preliminary contouring suggested that local data variation (i.e., from a contaminated zone to a clean zone) was relatively significant; therefore, the Point value was set to 6 and kept uniform for each model, which means the 6 nearest data points were used for kriging at a specific location within the radius of the Reach.

Semivariogram

The MVS software contains an algorithm that can assess the user's dataset and automatically determine the semivariogram based on a best fit of the data. A semivariogram is essentially the quantification of spatial correlation of data, where the quantified spatial correlation will then be used in kriging. A semivariogram model is typically determined as follows: 1) the dataset is evaluated to calculate the differences between all data pairs separated by a series of distances; 2) a graph is then generated to show the calculated variances with distances; 3) a mathematical model (i.e., semivariogram model) is then fit to the graph and usually the model is determined by the best fit with professional judgment. Parameters describing the mathematical model include range, sill, and nugget. The range is the distance at which spatial correlation disappears. The sill is the variance plateau (highest variance) that the semivariogram model reaches at the range. The nugget accounts for the variance among data collected from the same location (if sampling repeats at the same location), and is usually used to represent

possible measurement errors or other sources of variation. For this project, MVS automatically determined the semivariogram model based on the input data and no constraints were placed on its semivariogram modeling algorithm. It is important to note that MVS uses a nugget value of 0 which cannot be changed. CTech explains that "this restriction of the nugget [to only be a value of 0] provides much more representative results than allowing the nugget to be greater than zero, and thus allowing estimated data points to be different than the measured data points when they coincide."

Based on the input data and parameters set, the MVS conducts ordinary kriging automatically. The kriging results of the three-dimensional model were then processed into two-foot slices for presentation purposes. In order to achieve this, the Krig 3D Geology module in MVS was used. This module is generally used to create models of the lithology of a site. Lithology data was not available, but the module still proved useful for this analysis. A geology file was created for each parameter and each modeled area using the coordinates for the sample locations that were used for kriging the analytical data. For each location, two-foot elevation data was entered into the geology file to create the two-foot elevation slices from ground surface to the lowest sampled elevation. Once this data was processed, the three-dimensional model could be subset into the desired two-foot elevation slices. The ultimate goal was to show the resulting slices in plan view figures; therefore, the geology file was designed to display the middle elevation of the each two-foot elevation interval. For example, an elevation interval of 8 to 10 feet would show the data for elevation 9 feet.

The focus of the SAP was to identify the extent of contaminated soil. Both Residential and Industrial criteria were considered to determine the extent. It was deemed more valuable to subset the data based on a color scale as opposed to physically subsetting the three-dimensional model based on the different criterion. As a result, the slices were color coded based on the different criteria to assist engineering staff in their analysis of the data and decisions for future sampling locations.

The two-foot slices were exported as georeferenced image files. These image files were then imported into ArcGIS 9.3 software and displayed along with the New London base map spatial data for the generation of report figures. The resulting figures showing the kriging results for PAHs, TPH, and lead are provided in Appendix B of the Lower Subase Soil PDI SAP.

CROSS-SECTIONS

Cross-sections were also generated using C Tech's MVS software to help depict the vertical distribution of soil contamination and determine appropriate sample locations. This software package has modules that allow users to easily create fence diagrams from their three-dimensional models. For this project, fence lines were drawn through all of the sample locations that yielded the highest concentrations. This

provided a good illustration of the vertical extent of the contamination near these locations. A text file was generated and imported into the model to accurately visualize the groundwater table in relation to the estimated concentrations where groundwater level data was available. All cross-section figures displayed sample locations that were used in creating the cross-sections and sample locations that may have potentially influenced the estimated concentration. The two sets of locations were symbolized differently in the resulting cross-section figures and the offset distance was indicated for the potentially influential locations. Images of these cross-sections with the posted sample locations were exported as image files. These images were imported into ArcGIS 9.3 where additional textual information was added to the figures. The resulting cross-section figures are provided and discussed in WS #17 of the Lower Subase Soil PDI SAP.

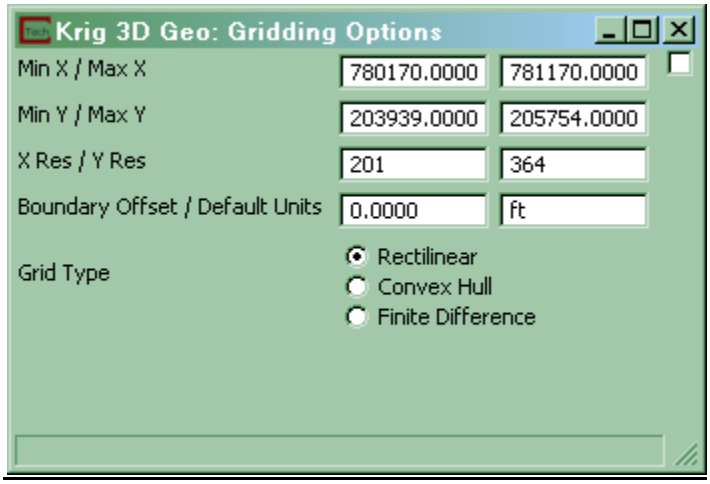
VOLUMES

In addition to supporting the preparation of the SAP, the same MVS model was used to support the Feasibility Study for the Lower Subase. The model was used for calculating the volume and mass of soil contaminated by the various constituents. MVS contains a module called Volumetrics that allows users to export these values concerning the modeled data. This module takes into account numerous criteria including the type of media modeled, whether the data were log processed, data units, geologic data, scale of the model, and subsetting based on regulatory criteria values. The goal was to obtain mass and volume values for each zone modeled. This process was streamlined for Zone 7 because this zone was not modeled with other zones. The Volumetrics module was connected to the three-dimensional kriging model. Once connected, the volume and mass data were calculated in the Volumetrics module interface and a text file was exported containing these values. For Zones 1 through 4, the three-dimensional model was cut into individual zones using a module called Area Cut. Once the data was clipped to a singular zone, the Volumetrics module could be connected to the model. At this point a text file, containing only the information for the zone displayed in the model, was generated and exported,. This process was repeated for all four zones to obtain the volume and mass values. This approach will be used to process the new data collected as part of the Soil PDI to develop new volume estimates of the contaminated soil present in the Lower Subase zones.

ATTACHMENT A

NODE CALCULATIONS AND EXAMPLE ZONE 7 GRID SYSTEM

Zone 1-4 Extents



Number of X nodes for Kriging

(781170 – 780170)/5 = 200

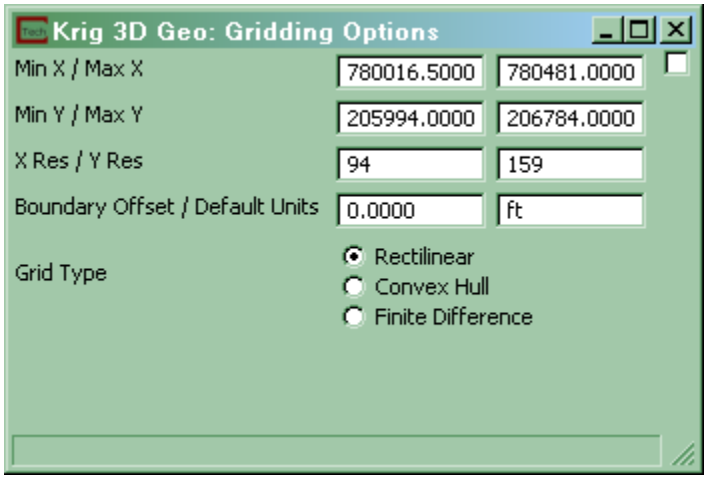
200 + 1 = **201**

Number of Y nodes for Kriging

(205754 – 203939)/5 = 363

363 + 1 = **364**

Zone 7 Extents



Number of X nodes for Kriging

(780481 – 780016.5)/5 = 93.1 (we rounded to 93)

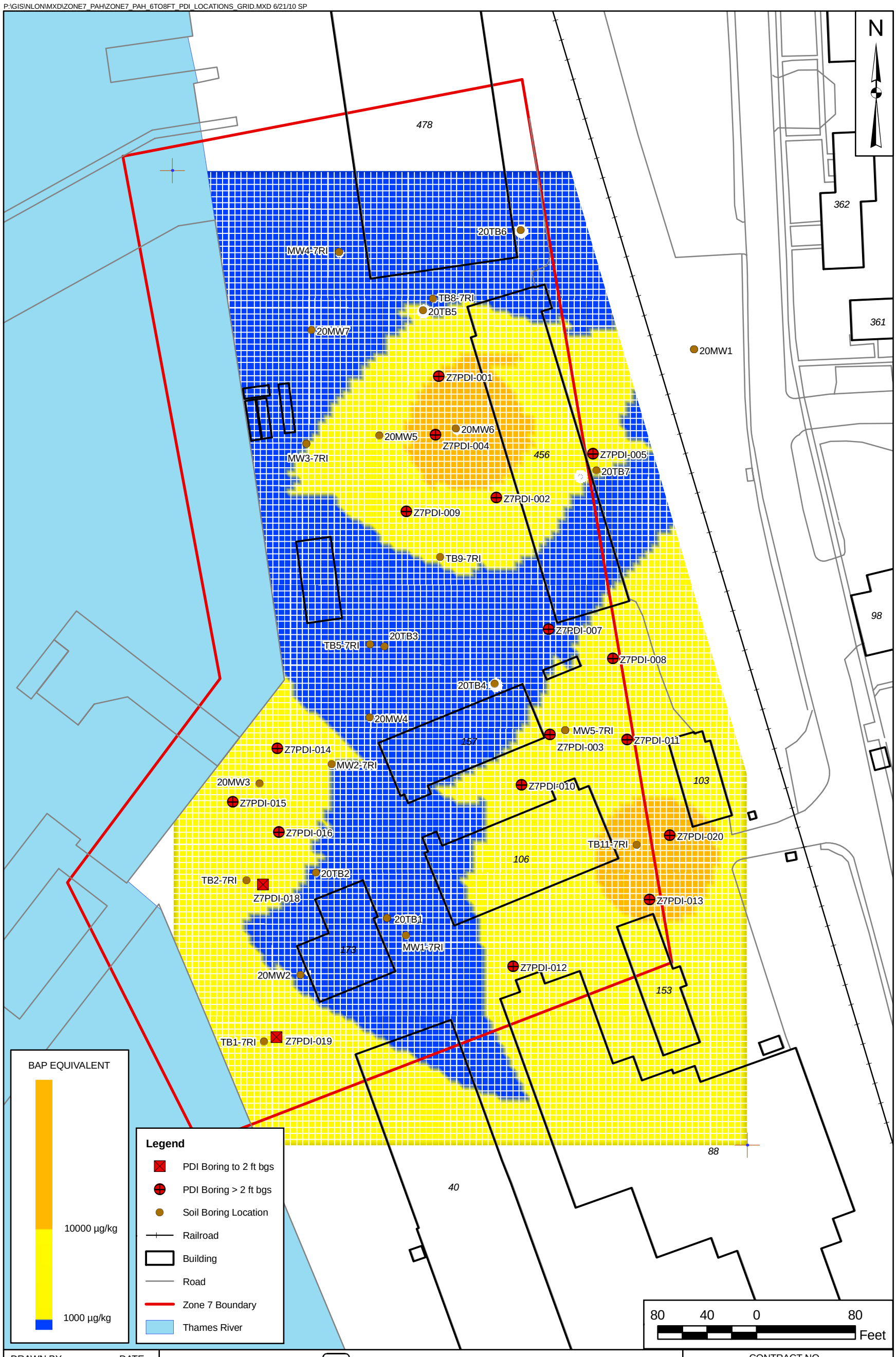
93 + 1 = **94**

Number of Y nodes for Kriging

(206784 – 205994)/5 = 158

158 + 1 = **159**

P:\GIS\NLON\MXD\ZONE7_PAH\ZONE7_PAH_6TO8FT_PDI_LOCATIONS_GRID.MXD 6/21/10 SP



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COST/SCHED-AREA			APPROVED BY -- DATE --
SCALE AS NOTED			FIGURE NO. 17 - 22 REV.

B-2 TECHNICAL MEMORANDUM ON UNCERTAINTY MODELING IN SUPPORT OF SAMPLING DESIGN FOR LOWER SUBASE SOIL PDI SAP

Technical Memorandum

Uncertainty Modeling in Support of Sampling Design for Lower Subase Soil PDI SAP

NSB-NLON, Groton, Connecticut

By: B. Li

Date: February 4, 2010

INTRODUCTION

Upon review of the Draft Lower Subase Feasibility Study (Tetra Tech, 2008), EPA recommended that a Pre-Design Investigation (PDI) be performed prior to selecting the final soil remedies and performing the remedial designs for each zone. The purposes of the PDI for soil at the Lower Subase are 1) to sample and analyze soil for primary contaminants of concern (COCs) at horizontal and vertical locations of greatest uncertainty of complying with the Industrial/Commercial (I/C) regulatory criteria to determine a more accurate estimate of the volume of soil requiring remediation in each zone, and 2) to sample and analyze soil for primary COCs at horizontal and vertical locations of greatest uncertainty of complying with the Residential regulatory criteria to determine a more accurate estimate of the area of soil requiring Land Use Controls (LUCs) as long as the soil presents risks .

In support of development of the PDI Sampling and Analysis Plan (SAP), three-dimensional (3D) uncertainty modeling was conducted using Geostatistical Software Library (GSLIB) to process the soil data of primary COCs, including polycyclic aromatic hydrocarbons (PAHs), total petroleum hydrocarbons (TPH), and lead from Zones 1 through 4 and Zone 7 at the Lower Subase. Due to the requirement of data quantity, only Zones 1 through 4 and Zone 7 data were analyzed. The data from Zones 1 through 4 were analyzed together because the four zones are physically connected and spatial continuity in data was observed.

The results of the uncertainty modeling are 3D maps that show the uncertainty scale at horizontal and vertical locations for individual contaminants. On each map, the uncertainty scale at each location is determined by a specific uncertainty model that considers several factors, including spatial distribution of data (data sparsity), level of contamination, and contaminant concentrations in relation to regulatory criteria. For presentation purposes, the 3D uncertainty maps are presented in a series of two dimensional (2D) maps representing two-foot intervals in the vertical direction. The 2D uncertainty maps are provided in Appendix B of the Lower Subase Soil PDI SAP.

METHODOLOGY

The uncertainty modeling methodology used for the Lower Subase was developed and tested by researchers in the University of Michigan in 2001. The methodology was built on the concepts and

theories of geostatistical uncertainty modeling. The state-of-the-art technology was published in Environmental Science & Technology (Barabas et al., 2001), where the technology was tested and demonstrated using 3D dioxin data from sediments in an estuarine river. As stated, this uncertainty modeling technology differs from the others (e.g. kriging variance-based technologies, such as the one used in Environmental Visualization System) in that it considers both where existing samples are located and the concentration levels of existing samples. Kriging-variance based technologies only consider sample locations, which may lead to unnecessary sampling in relatively clean areas. Moreover, the technology used herein was designed to consider regulatory criteria, which makes it specifically suitable for criteria-based zone delineation problems. An example of this technology using Zone 7 lead data was presented to the Partnering Team on May 14, 2009 (see Appendix A of the Lower Subase Soil PDI SAP) and the team subsequently provided approval of the method for use in developing the Lower Subase Soil PDI SAP.

The methodology consists of the followingsteps:

1) Local Uncertainty Modeling

A 3D grid is first established to bound the two study areas (i.e., Zones 1 through 4 and Zone 7). Based on the density of existing sample data (i.e., the sparsity level of data), a 5-foot by 5-foot grid was established. The sample data were then analyzed for spatial features (e.g., semi-variograms) and the features were mathematically modeled. The sample data and the modeled spatial features were then used to derive a conditional cumulative distribution function (ccdf) $F(\mathbf{u}; z|(n))$ for each location (represented by $\mathbf{u}$) within the 3D grid to provide a measure of local uncertainty. The ccdf is conditioned to the n existing sample data in the neighborhood of $\mathbf{u}$, represented by $z|(n)$. Several approaches exist to derive the ccdf from existing sample data. For the Lower Subase study, a non-parametric indicator approach was used because it does not make assumptions about the multiple-point distribution function of the data.

2) Uncertainty Scale Computation

Once a ccdf is derived at each location of the 3D grid, a statistic is computed at each location to quantify its uncertainty of complying with a certain regulatory criterion (i.e., the uncertainty in zone delineation in relation to this criterion). This statistic is a unitless, non-negative, real number that represents the scale of uncertainty. A value of zero represents the least uncertainty. A large value represents high uncertainty. There is no maximum on the monotonic uncertainty scale. The statistic is computed as

$$r(\mathbf{u}) = \frac{s(\mathbf{u})}{\left|z_E^*(\mathbf{u}) - z_c\right|} \qquad with \quad z_E^*(\mathbf{u}) \neq z_c \tag{1}$$

Here, $s(\mathbf{u})$ is the standard deviation of the ccdf at location $\mathbf{u}$, and can be calculated from the ccdf as follows:

$$s^2(\mathbf{u}) = \int_{-\infty}^{+\infty} [z - z_E^*(\mathbf{u})]^2 \cdot f(\mathbf{u}; z \mid (n)) dz \qquad (2)$$

In Equations (1) and (2), $f(\mathbf{u};z|(n))$ is the probability density function of the random variable $Z(\mathbf{u})$ (e.g. contaminant concentration), $z^*_E(\mathbf{u})$ is the expectation of the ccdf, and z_c is the regulatory criterion. For the Lower Subase study, the kriging estimates (see Technical Memorandum on Kriging in Appendix B of the Lower Subase Soil PDI SAP) were used for $z^*_E(\mathbf{u})$ to ensure consistency between the kriging maps and the uncertainty maps.

As stated, one of the benefits provided by this technology is its incorporation of regulatory criteria in defining a zone. As reflected in Equation (1), locations with the largest $r(\mathbf{u})$ values are the ones with the highest uncertainty in delineating a contaminated zone defined by the regulatory criterion z_c. The $r(\mathbf{u})$ value will be large (i.e., high uncertainty) in two cases: 1) when there is great uncertainty in estimating the contaminant concentration at a location (e.g. because of sparse data or contrasting data), reflected by a large value of the standard deviation of the ccdf ($s(\mathbf{u})$); and 2) when the estimated contaminant concentration ($z^*_E(\mathbf{u})$) is close to the regulatory criterion z_c, indicating high uncertainty about whether the criterion is exceeded (i.e. the uncertainty in delineating the boundary of the contaminated zone). As a result, if sampling occurs according to the uncertainty scale, priority is placed on the boundary of the contaminated zone defined by the specified regulatory criterion, not the areas with very high concentrations (sure exceedances) or the areas with very low concentrations (sure non-exceedances).

Execution of the described methodology was through the use of GSLIB. GSLIB is a collection of geostatistical programs developed at Stanford University (Deutsch and Journel, 1992).

RESULTS

For presentation purposes, the 3D uncertainty scale maps were processed into a series of 2D plan view maps at 2-foot elevation intervals in the vertical direction of the 3D domain. Each 2D map shows the plan view uncertainty at the mid-elevation of the 2-foot interval. The 2D maps are provided in Appendix B of the Lower Subase Soil PDI SAP. For each chemical, both the I/C regulatory criterion and the Residential criterion were considered in the uncertainty modeling and two sets of maps were created. The uncertainty maps for the I/C criterion can be used to determine the locations with greatest uncertainty in support of the delineation of the zones that need soil remediation. The maps for the Residential criterion can be used to determine the locations with greatest uncertainy for the delineation of the zones that require LUCs.

For illustration purposes, the maximum of the uncertainty scale was set to 10 for all maps. A color was assigned to each grid location of the map according to its uncertainty scale. The rainbow color scale was set so that high uncertainty is presented by red and low uncertainty is represented by blue. Examination of the uncertainty maps in combination of the kriging maps suggest that high uncertainty is typically observed near the boundary of criteria-defined zones as expected.

References

Barabas, N, Gooaverts, P, and Adriaens, P, 2001, *Geostatistical Assessment and Validation of Uncertainty for Three-Dimentional Dioxin Data from Sediments in an Estuarine River*, Environmental Science & Technology, 35, 3294-3301

Deutsch, C. V. and Journel, A. G., 1992, *GSLIB: Geostatistical Software Library and User's Guide*, Oxford University Press, New York

Tetra Tech (Tetra Tech NUS, Inc.), 2008. Draft Lower Subase Feasibility Study Report, Naval Submarine Base – New London, Groton, Connecticut. King of Prussia, Pennsylvania, March.

B-3 KRIGING AND UNCERTAINTY FIGURES (FIGURES B-1 THROUGH B-143)

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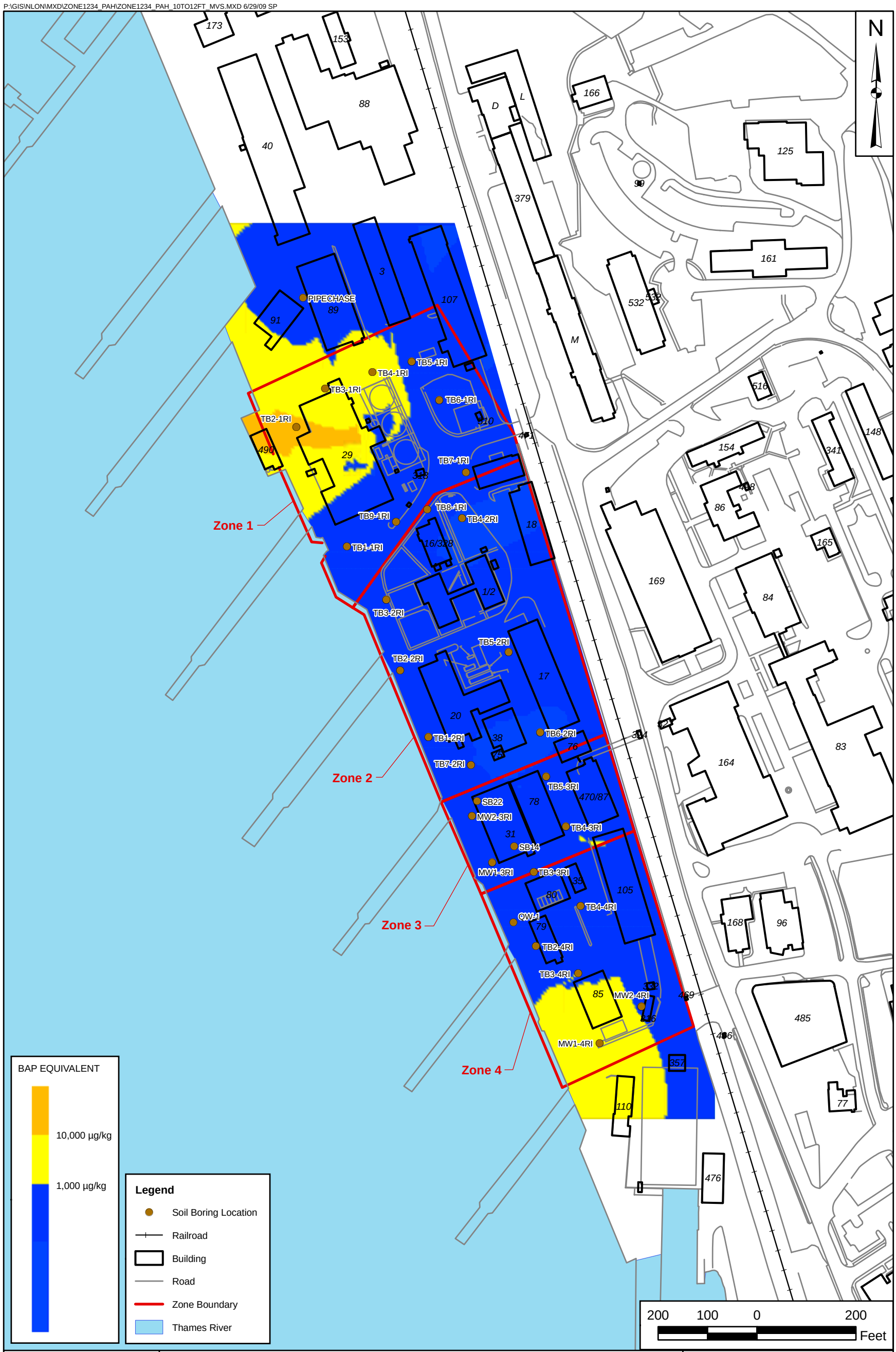

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COST/SCHED-AREA		ZONES 1, 2, 3, AND 4	APPROVED BY / DATE
		NAVAL SUBMARINE BASE - NEW LONDON	-- / --
SCALE		GROTON, CONNECTICUT	FIGURE NO. / REV.
AS NOTED			FIGURE B-1

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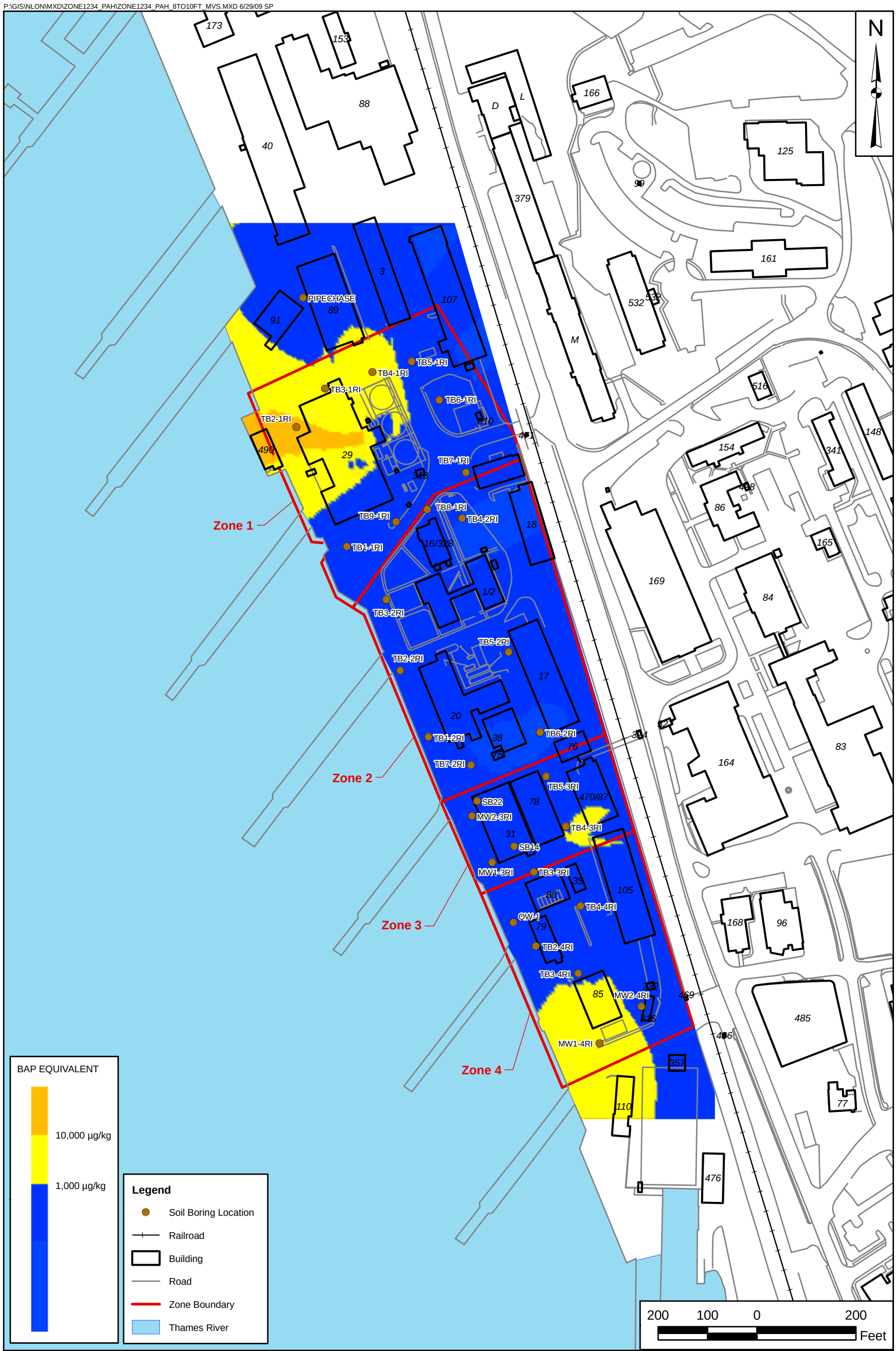

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COST/SCHED-AREA			APPROVED BY -- DATE --
SCALE AS NOTED			FIGURE NO. FIGURE B-2 REV.

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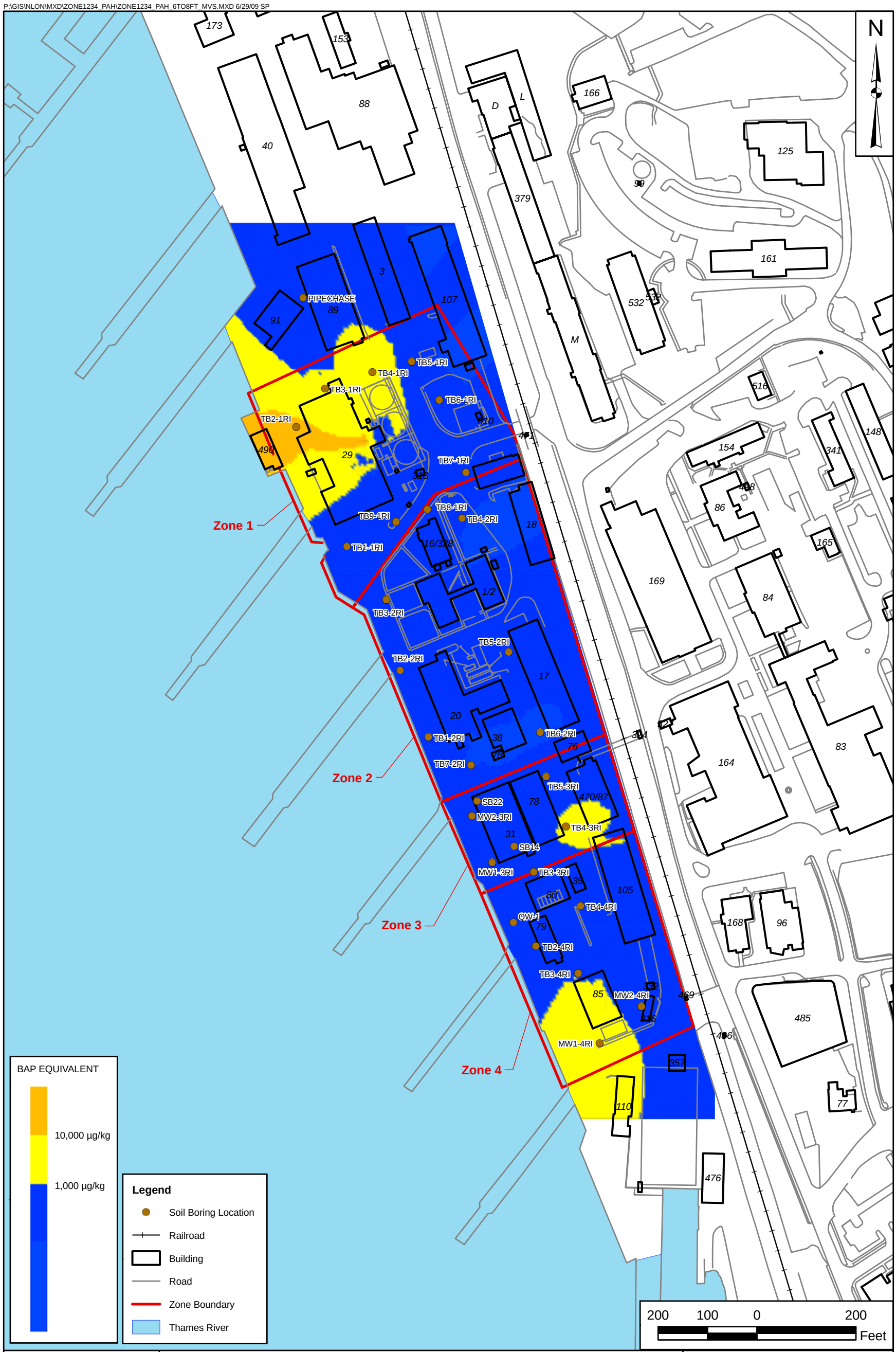

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COST/SCHED-AREA			APPROVED BY -- / DATE --
SCALE AS NOTED			FIGURE NO. FIGURE B-3 / REV.

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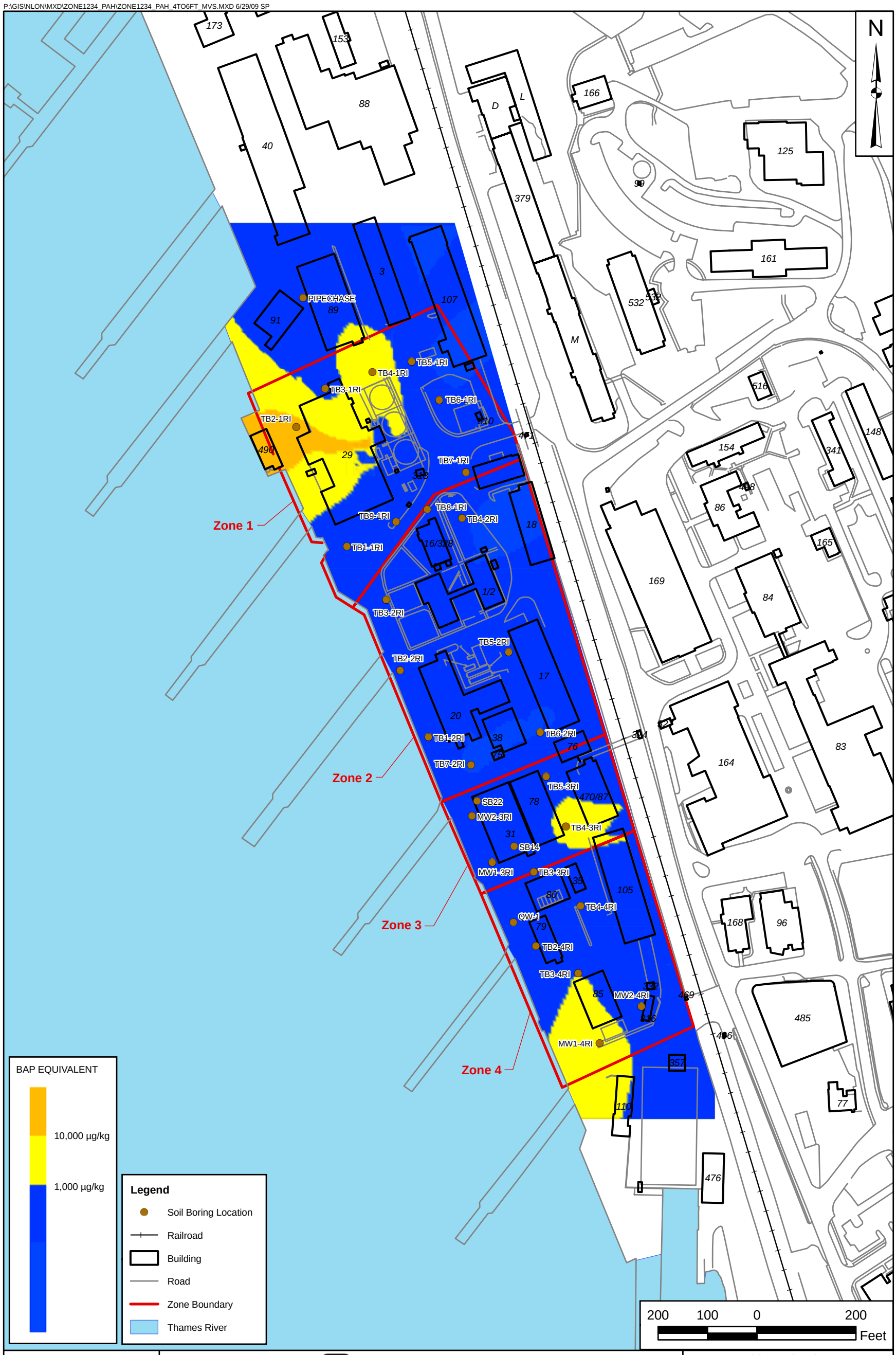

DRAWN BY S. PAXTON	DATE 4/02/09	Tetra Tech NUS, Inc.	CONTRACT NO. 0811
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COST/SCHED-AREA			APPROVED BY -- / DATE --
SCALE AS NOTED			FIGURE NO. FIGURE B-4 / REV.

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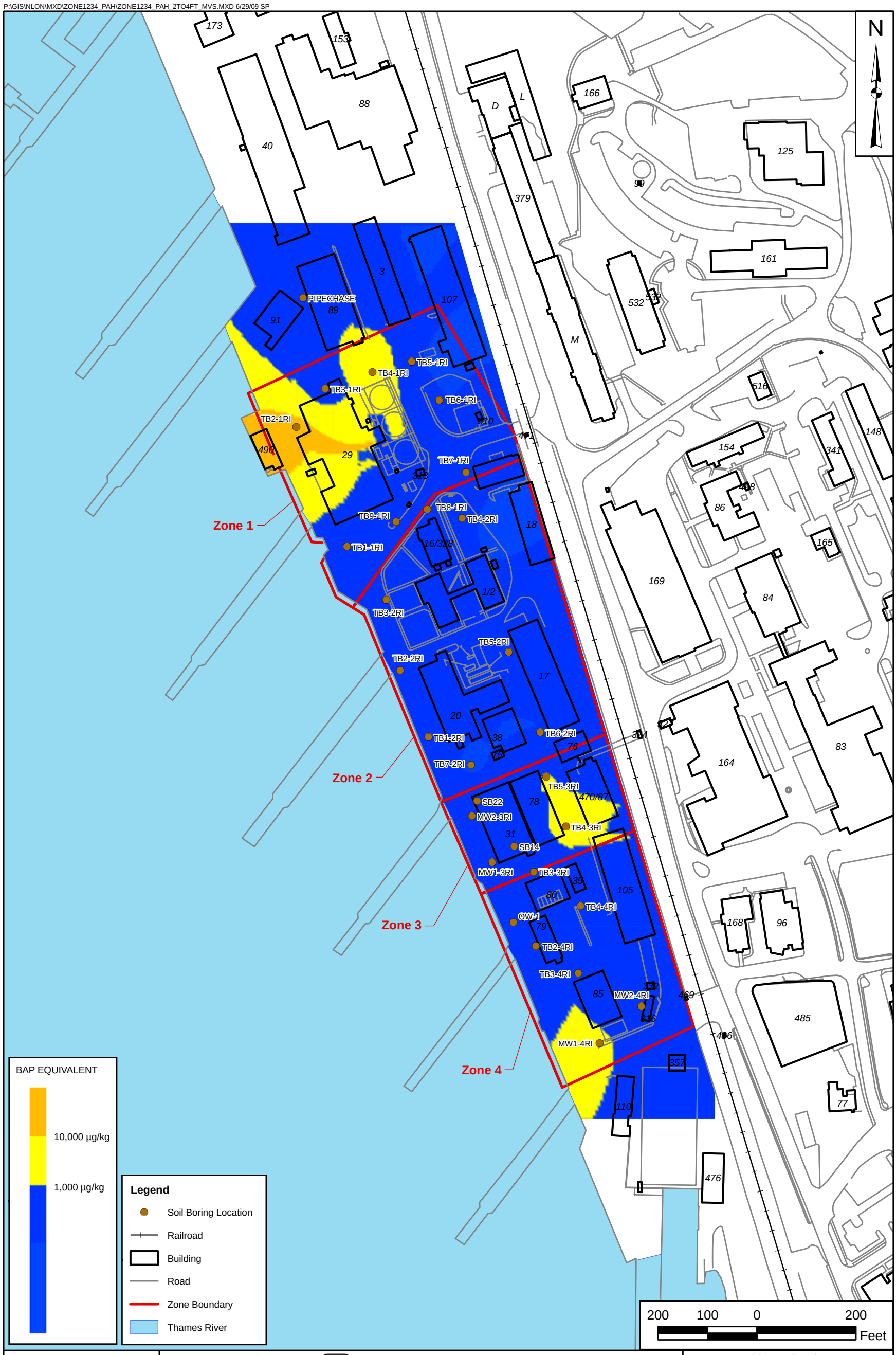

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COST/SCHED-AREA		ZONES 1, 2, 3, AND 4	APPROVED BY / DATE
		NAVAL SUBMARINE BASE - NEW LONDON	-- / --
SCALE		GROTON, CONNECTICUT	FIGURE NO. / REV.
AS NOTED			FIGURE B-5

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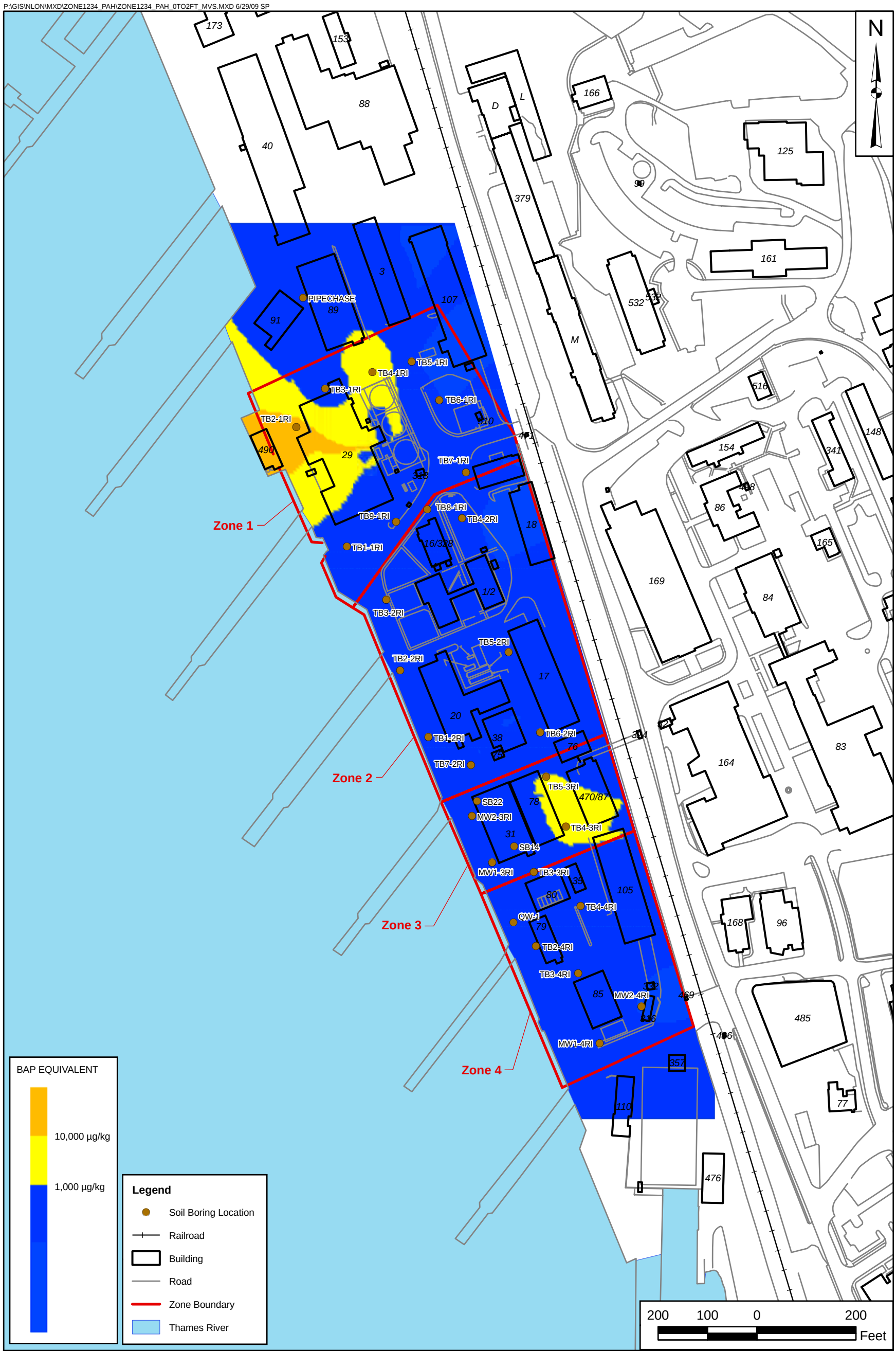

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N. BALSAMO	6/29/09	ELEVATION: 0 TO 2 FEET	_ / --
COST/SCHED-AREA		ZONES 1, 2, 3, AND 4	APPROVED BY / DATE
		NAVAL SUBMARINE BASE - NEW LONDON	-- / --
SCALE		GROTON, CONNECTICUT	FIGURE NO. / REV.
AS NOTED			FIGURE B-6

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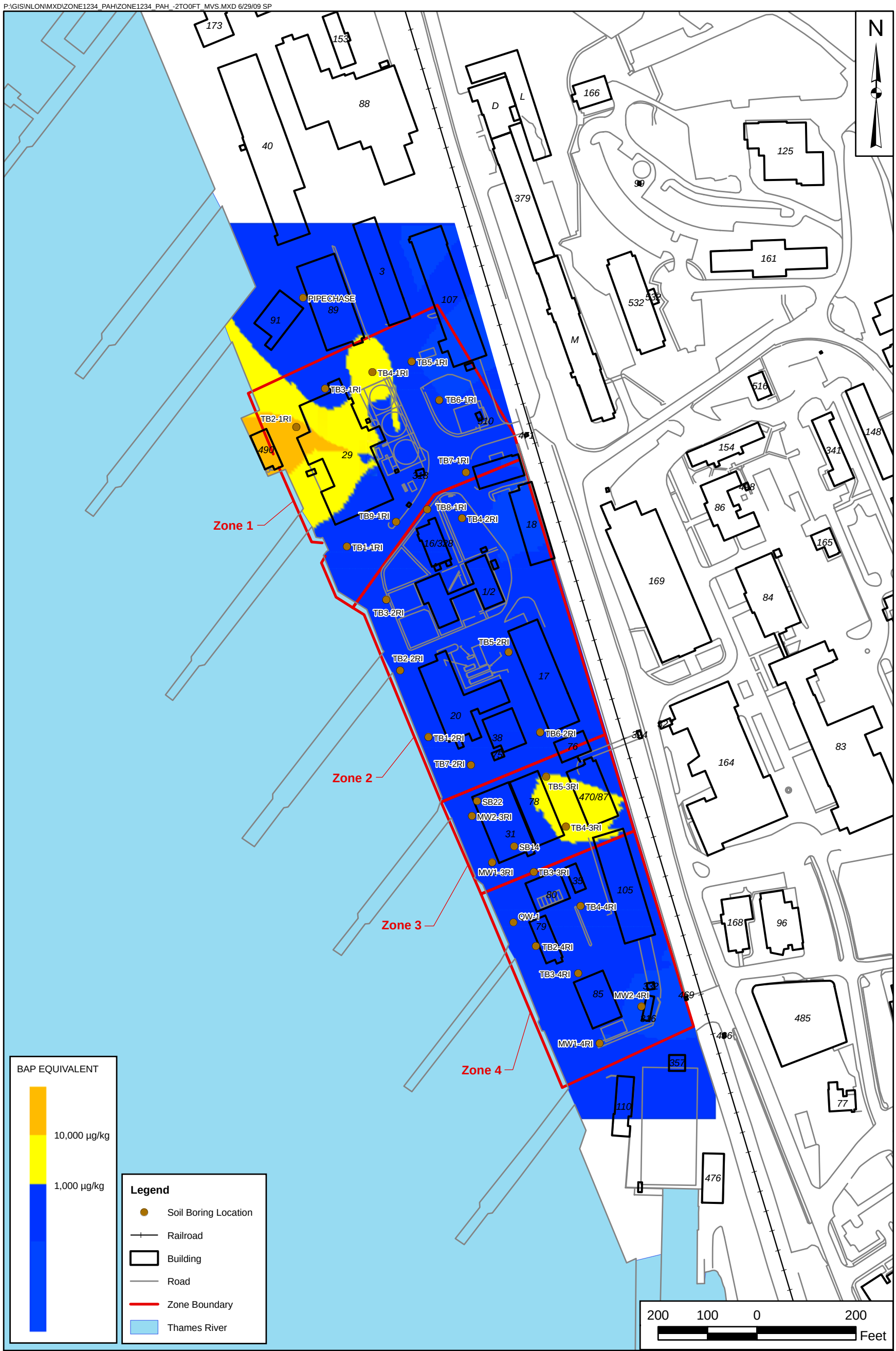
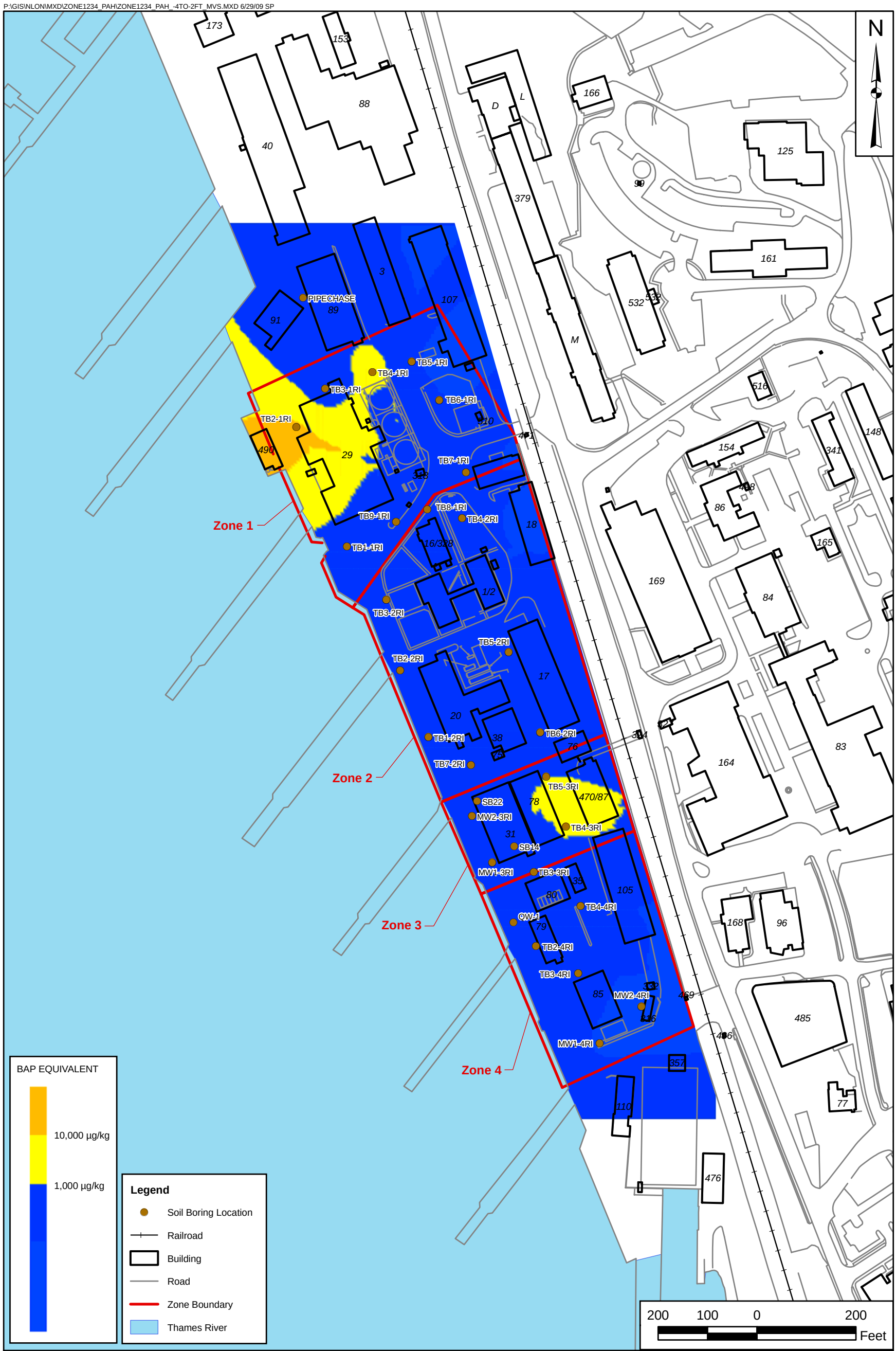


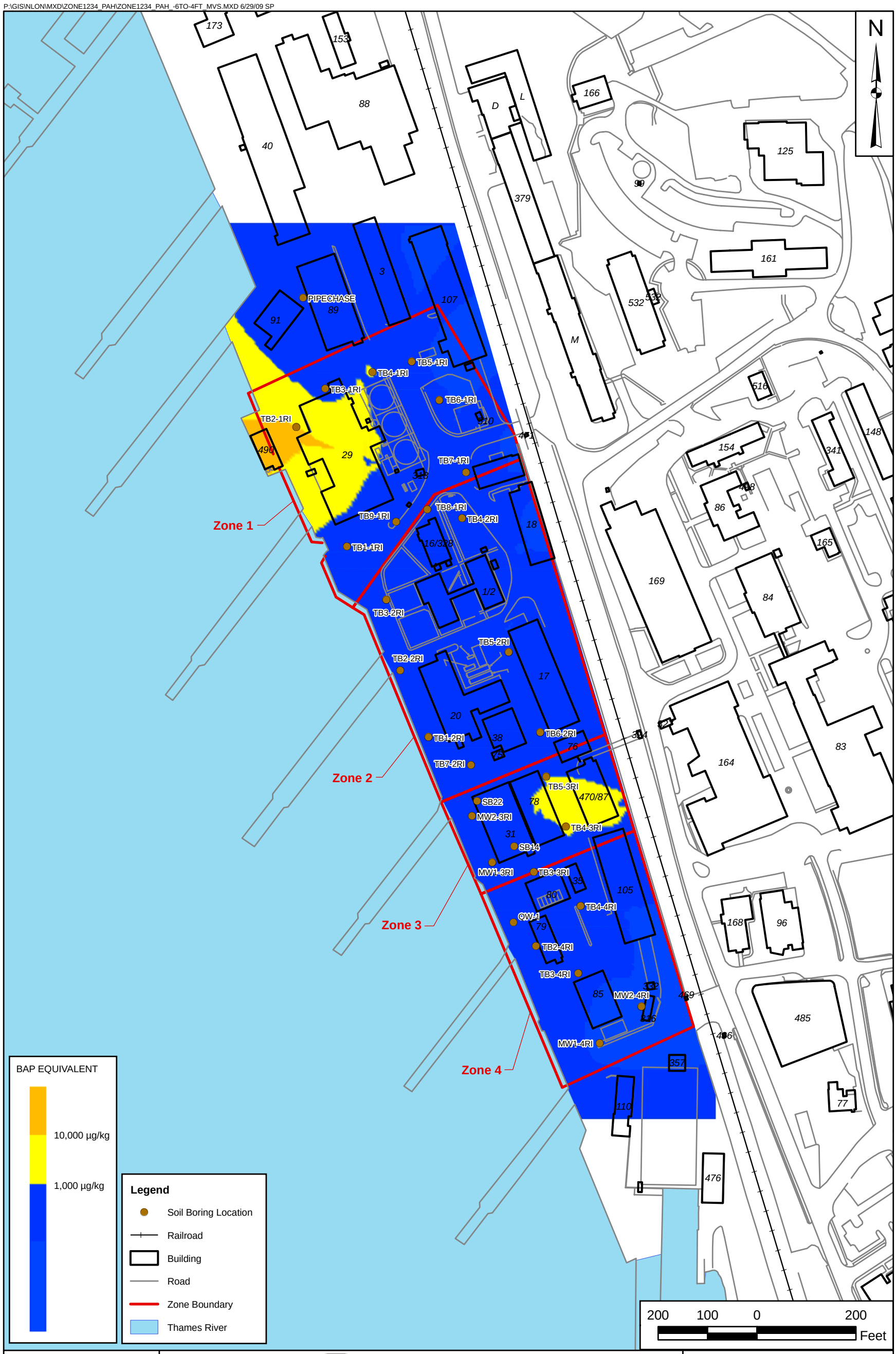
DRAWN BY S. PAXTON — DATE 4/02/09	Tetra Tech NUS, Inc.		CONTRACT NO. 0811
CHECKED BY N. BALSAMO — DATE 6/29/09	ESTIMATED BAP EQUIVALENT CONCENTRATIONS (KRIGING RESULTS) ELEVATION: -2 TO 0 FEET ZONES 1, 2, 3, AND 4 NAVAL SUBMARINE BASE - NEW LONDON GROTON, CONNECTICUT		APPROVED BY _ — DATE --
COST/SCHED-AREA			APPROVED BY -- — DATE --
SCALE AS NOTED			FIGURE NO. FIGURE B-7 — REV.

P:\GIS\NLON\MXD\ZONE1234_PAH\ZONE1234_PAH_-4TO-2FT_MVS.MXD 6/29/09 SP



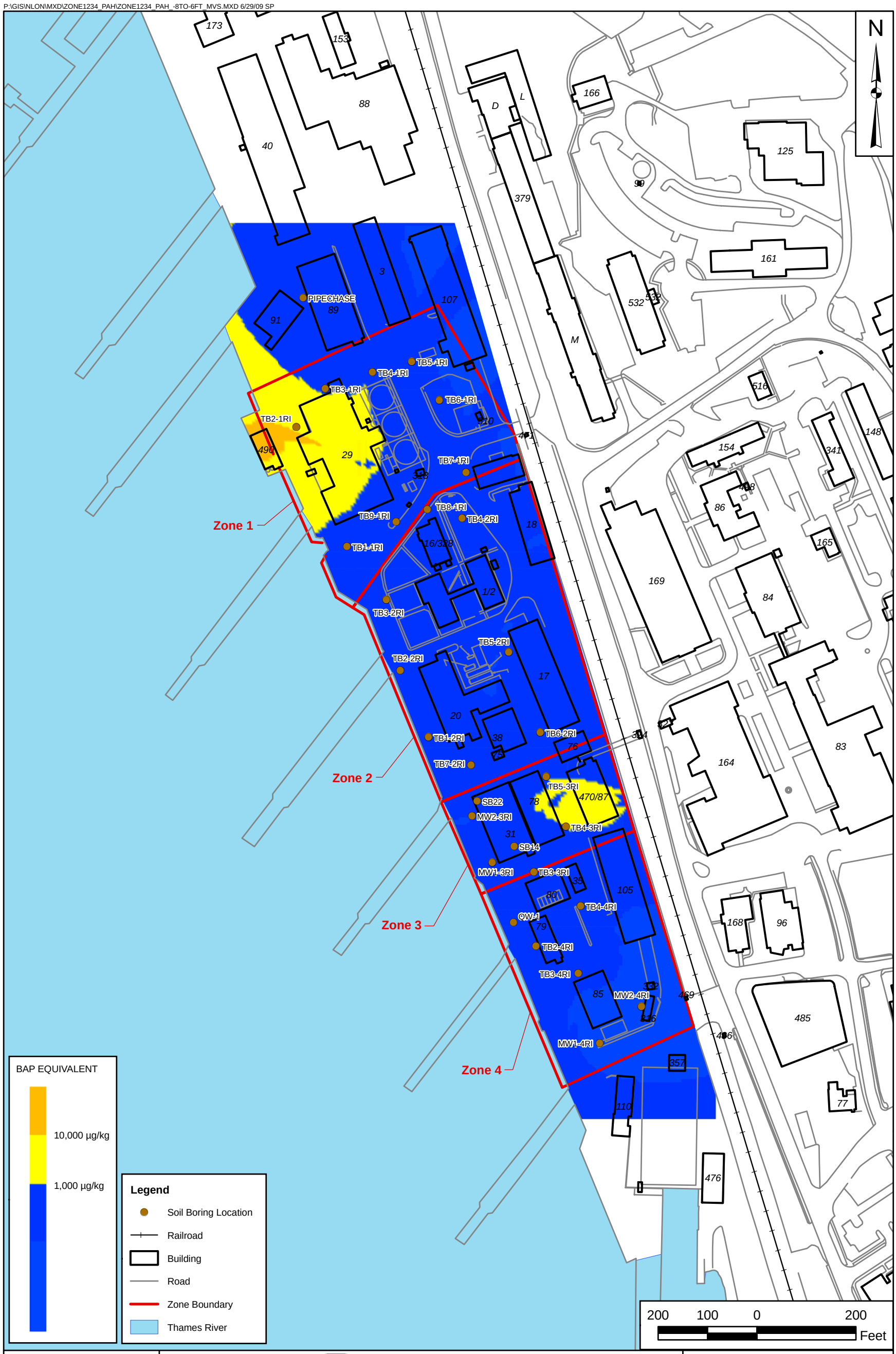
DRAWN BY	DATE	Tetra Tech NUS, Inc.	CONTRACT NO.
S. PAXTON	4/02/09		0811
CHECKED BY	DATE	ESTIMATED BAP EQUIVALENT CONCENTRATIONS (KRIGING RESULTS)	APPROVED BY / DATE
N. BALSAMO	6/29/09	ELEVATION: -4 TO -2 FEET	_ / --
COST/SCHED-AREA		ZONES 1, 2, 3, AND 4	APPROVED BY / DATE
		NAVAL SUBMARINE BASE - NEW LONDON	-- / --
SCALE		GROTON, CONNECTICUT	FIGURE NO. / REV.
AS NOTED			FIGURE B-8

P:\GIS\NLON\MXD\ZONE1234_PAH\ZONE1234_PAH_-6TO-4FT_MVS.MXD 6/29/09 SP



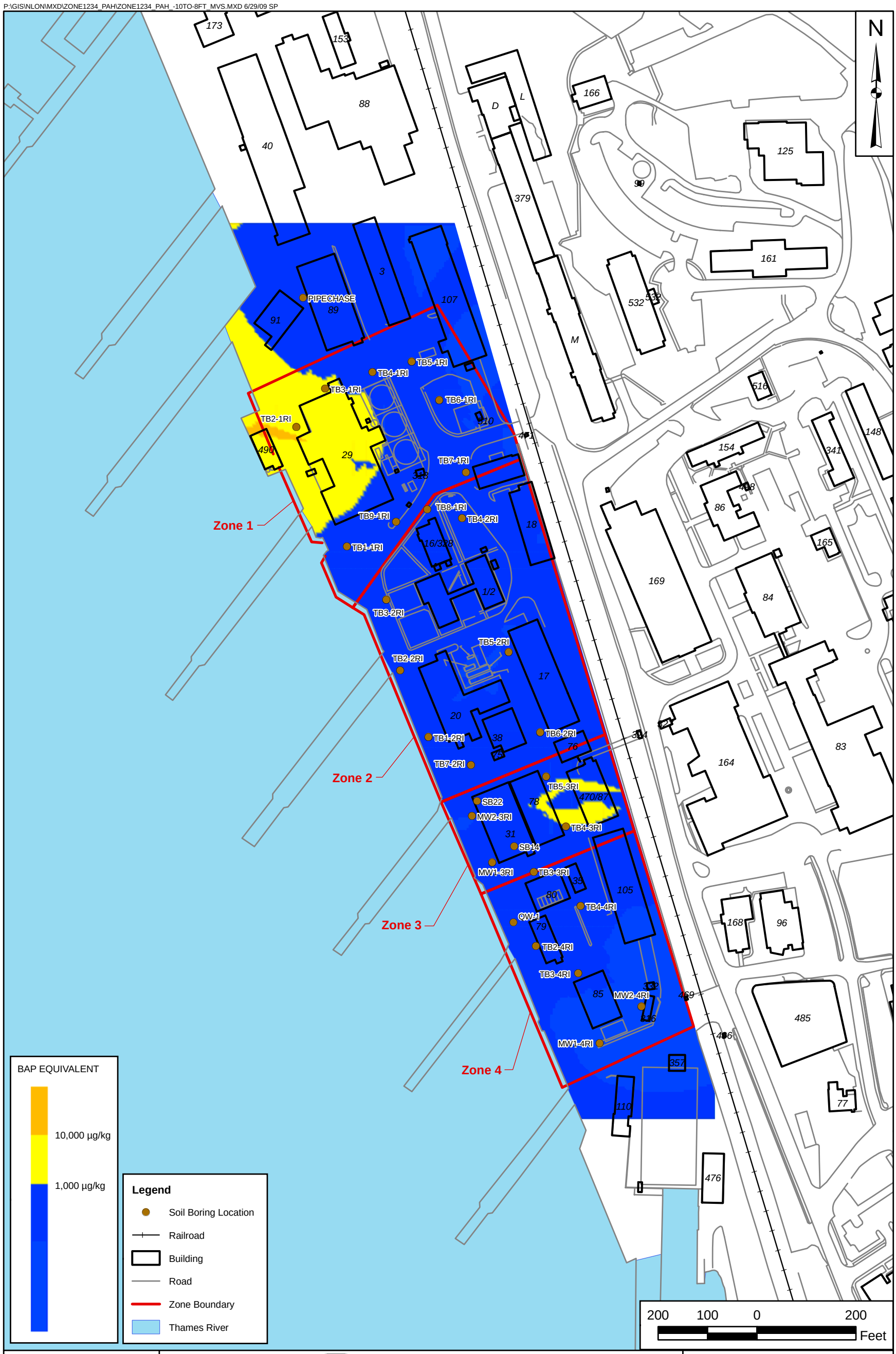
DRAWN BY S. PAXTON	DATE 4/02/09	Tetra Tech NUS, Inc.	CONTRACT NO. 0811
CHECKED BY N. BALSAMO	DATE 6/29/09	ESTIMATED BAP EQUIVALENT CONCENTRATIONS (KRIGING RESULTS) ELEVATION: -6 TO -4 FEET ZONES 1, 2, 3, AND 4 NAVAL SUBMARINE BASE - NEW LONDON GROTON, CONNECTICUT	APPROVED BY _ / DATE --
COST/SCHED-AREA			APPROVED BY -- / DATE --
SCALE AS NOTED			FIGURE NO. FIGURE B-9 / REV.

P:\GIS\NLON\MXD\ZONE1234_PAH\ZONE1234_PAH_-8TO-6FT_MVS.MXD 6/29/09 SP



DRAWN BY S. PAXTON	DATE 4/02/09	Tetra Tech NUS, Inc.	CONTRACT NO. 0811
CHECKED BY N. BALSAMO	DATE 6/29/09	ESTIMATED BAP EQUIVALENT CONCENTRATIONS (KRIGING RESULTS) ELEVATION: -8 TO -6 FEET ZONES 1, 2, 3, AND 4 NAVAL SUBMARINE BASE - NEW LONDON GROTON, CONNECTICUT	APPROVED BY _ / DATE --
COST/SCHED-AREA			APPROVED BY -- / DATE --
SCALE AS NOTED			FIGURE NO. FIGURE B-10 / REV.

P:\GIS\NLON\MXD\ZONE1234_PAH\ZONE1234_PAH_-10TO-8FT_MVS.MXD 6/29/09 SP



DRAWN BY S. PAXTON	DATE 4/02/09	Tetra Tech NUS, Inc.	CONTRACT NO. 0811
CHECKED BY N. BALSAMO	DATE 6/29/09	ESTIMATED BAP EQUIVALENT CONCENTRATIONS (KRIGING RESULTS) ELEVATION: -10 TO -8 FEET ZONES 1, 2, 3, AND 4 NAVAL SUBMARINE BASE - NEW LONDON GROTON, CONNECTICUT	APPROVED BY _ / DATE --
COST/SCHED-AREA			APPROVED BY -- / DATE --
SCALE AS NOTED			FIGURE NO. FIGURE B-11 / REV.

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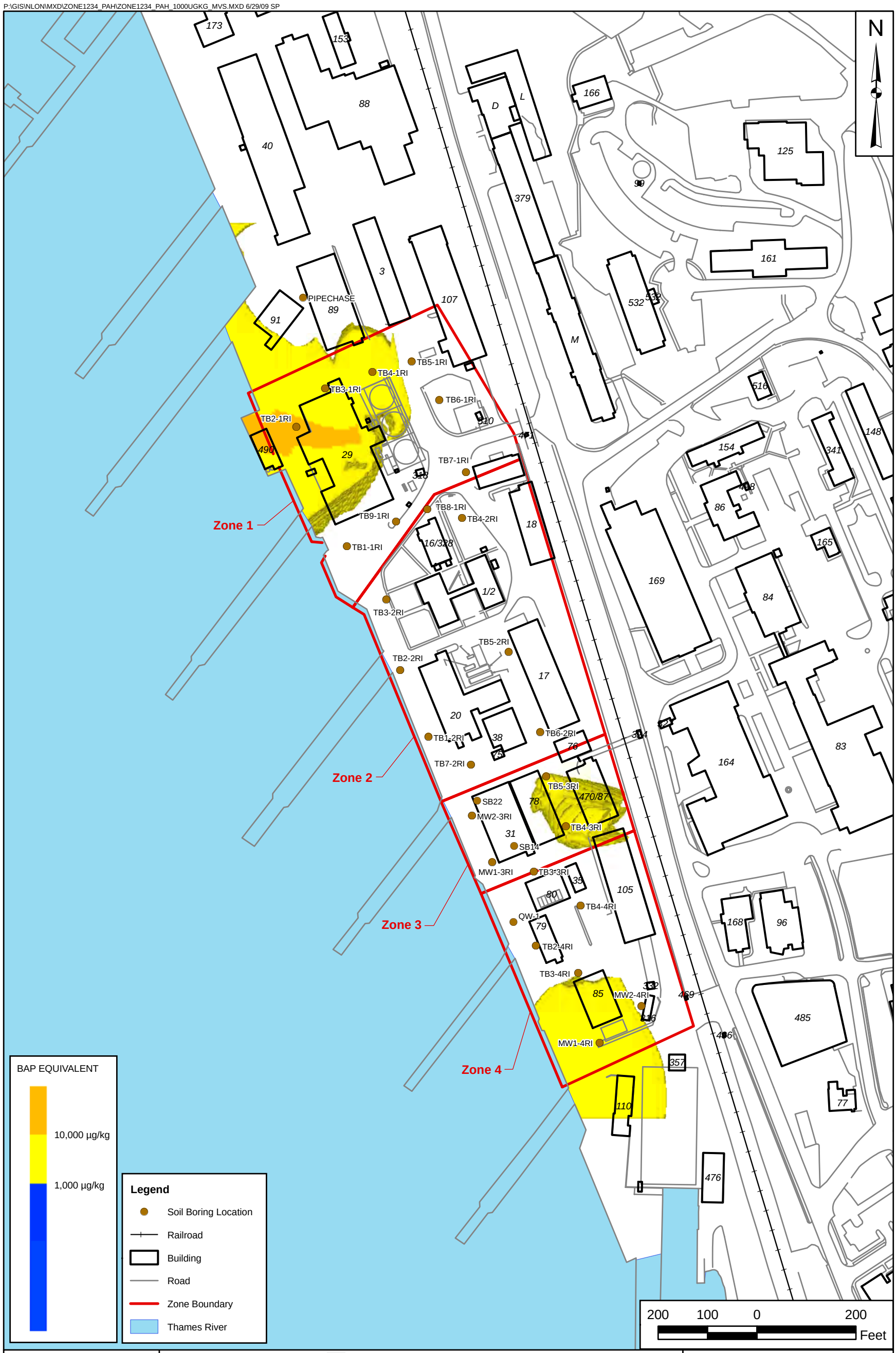
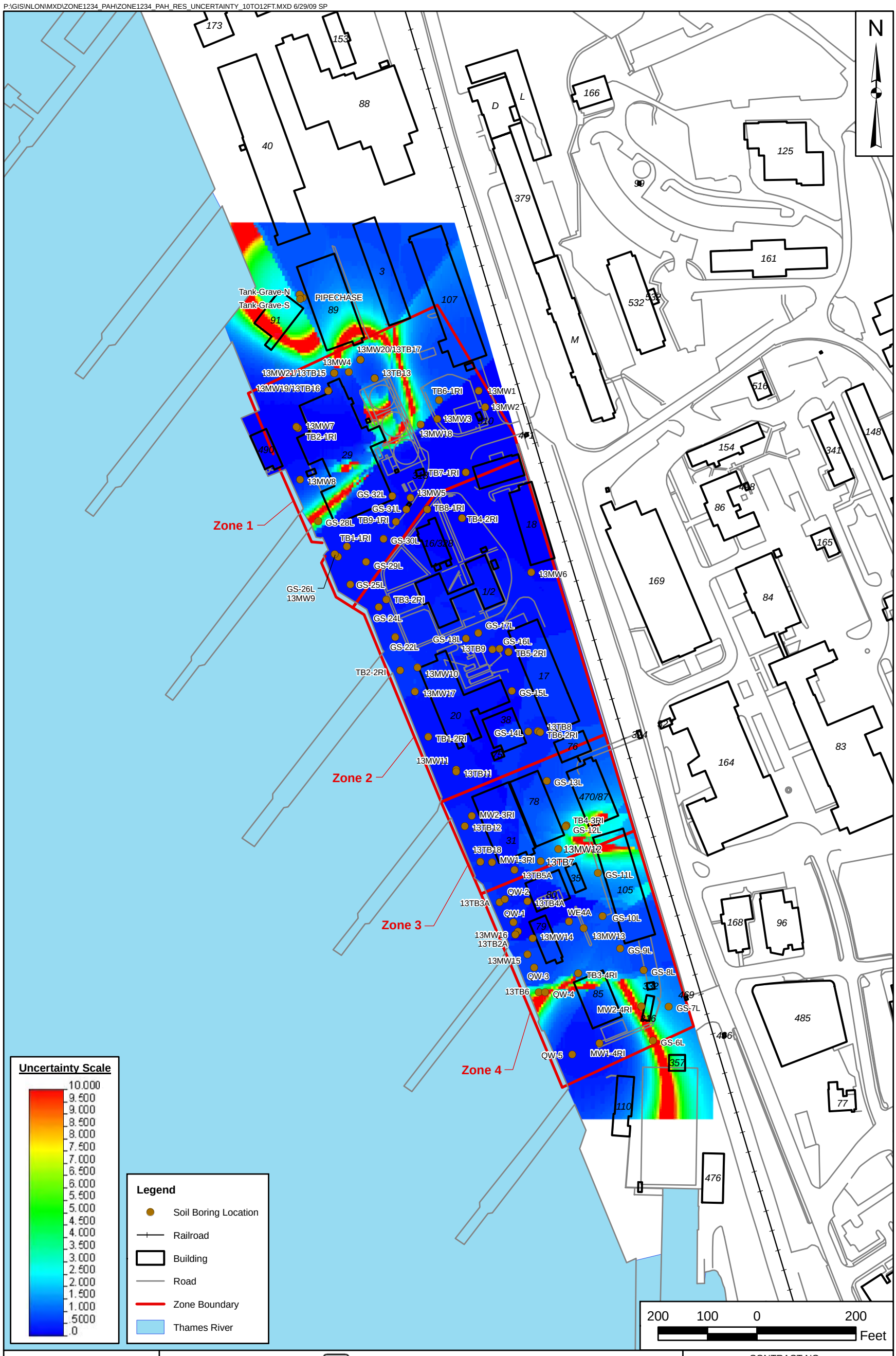


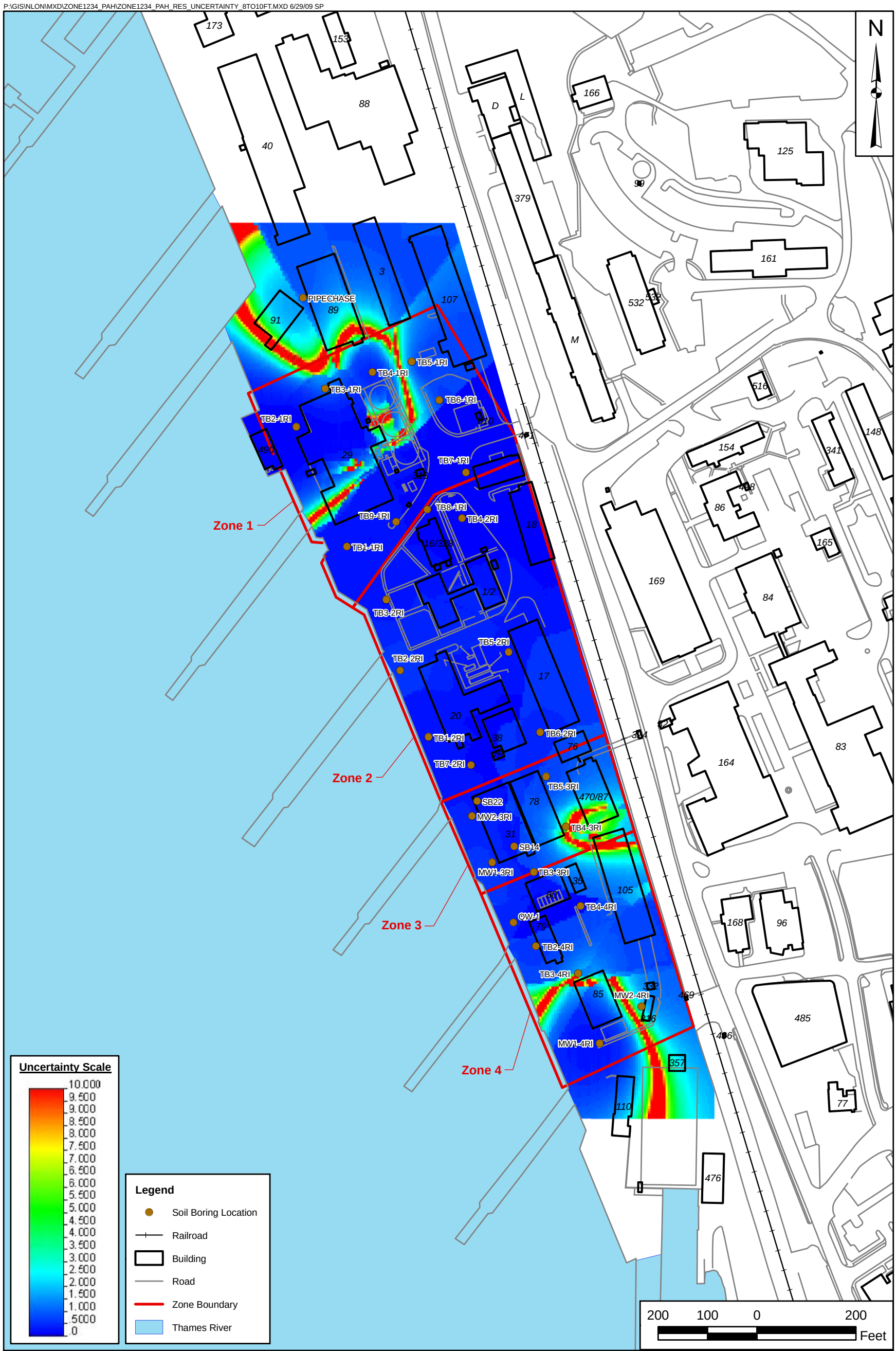
DRAWN BY	DATE	Tetra Tech NUS, Inc.	CONTRACT NO.
S. PAXTON	4/02/09		0811
CHECKED BY	DATE	ESTIMATED BAP EQUIVALENT CONCENTRATIONS (KRIGING RESULTS)	APPROVED BY / DATE
N. BALSAMO	6/29/09	ELEVATION: 10 TO -10 FEET	_ / --
COST/SCHED-AREA		ZONES 1, 2, 3, AND 4	APPROVED BY / DATE
		NAVAL SUBMARINE BASE - NEW LONDON	-- / --
SCALE		GROTON, CONNECTICUT	FIGURE NO. / REV.
AS NOTED			FIGURE B-12

P:\GIS\NLON\MXD\ZONE1234_PAH\ZONE1234_PAH_RES_UNCERTAINTY_10TO12FT.MXD 6/29/09 SP



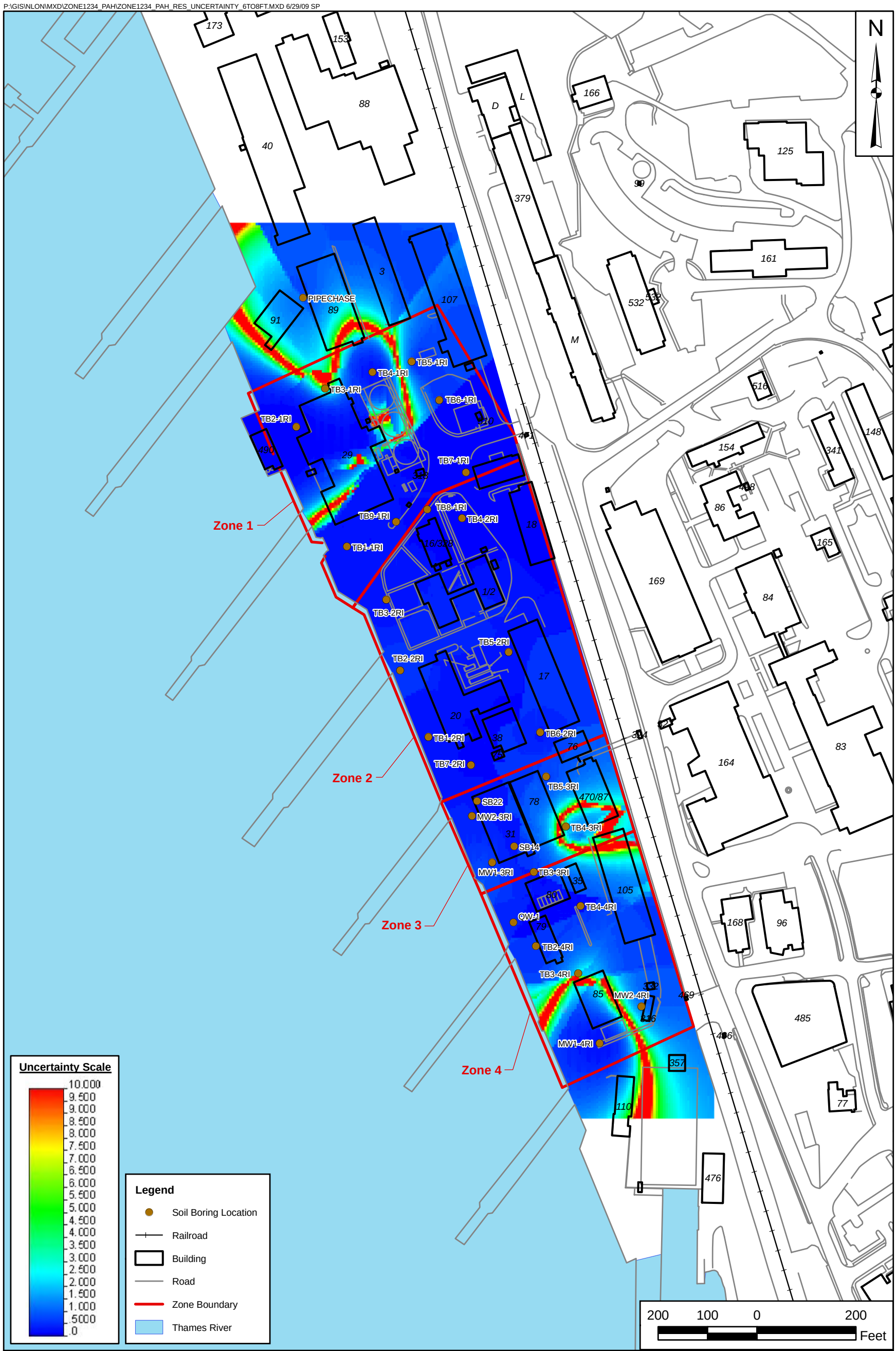
DRAWN BY S. PAXTON	DATE 4/02/09	Tetra Tech NUS, Inc.	CONTRACT NO. 0811
CHECKED BY N. BALSAMO	DATE 6/29/09	BAP EQUIVALENT UNCERTAINTY FOR 1000 UG/KG ELEVATION: 10 TO 12 FEET ZONES 1, 2, 3, AND 4 NAVAL SUBMARINE BASE - NEW LONDON GROTON, CONNECTICUT	APPROVED BY _ / DATE --
COST/SCHED-AREA			APPROVED BY -- / DATE --
SCALE AS NOTED			FIGURE NO. FIGURE B-13 / REV.

P:\GIS\NLON\MXD\ZONE1234_PAH\ZONE1234_PAH_RES_UNCERTAINTY_8TO10FT.MXD 6/29/09 SP



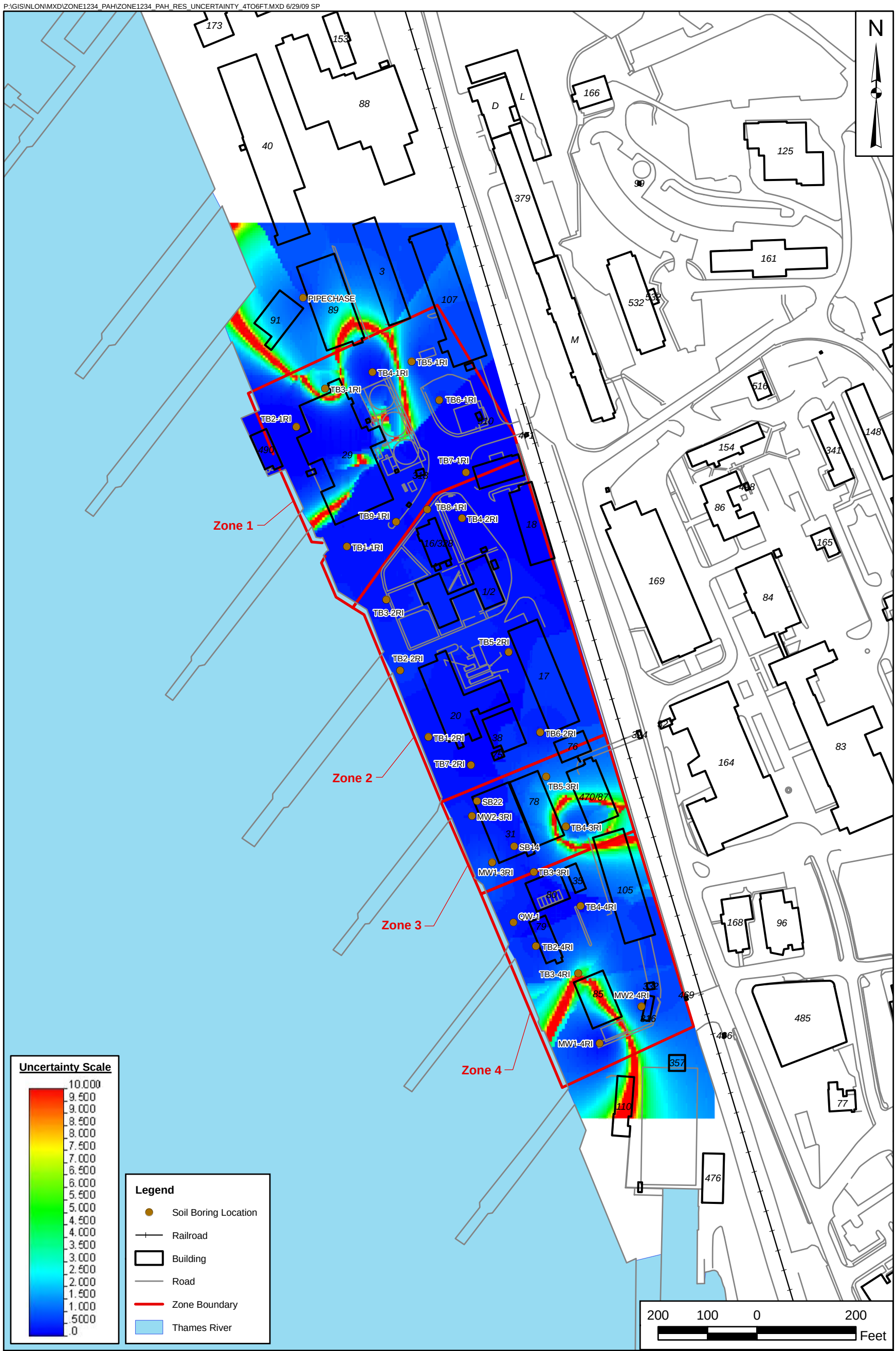
DRAWN BY	DATE	Tetra Tech NUS, Inc.	CONTRACT NO.
S. PAXTON	4/02/09		0811
CHECKED BY	DATE	BAP EQUIVALENT UNCERTAINTY FOR 1000 UG/KG	APPROVED BY / DATE
N. BALSAMO	6/29/09	ELEVATION: 8 TO 10 FEET	_ / --
COST/SCHED-AREA		ZONES 1, 2, 3, AND 4	APPROVED BY / DATE
		NAVAL SUBMARINE BASE - NEW LONDON	-- / --
SCALE		GROTON, CONNECTICUT	FIGURE NO. / REV.
AS NOTED			FIGURE B-14

P:\GIS\NLON\MXD\ZONE1234_PAH\ZONE1234_PAH_RES_UNCERTAINTY_6TO8FT.MXD 6/29/09 SP



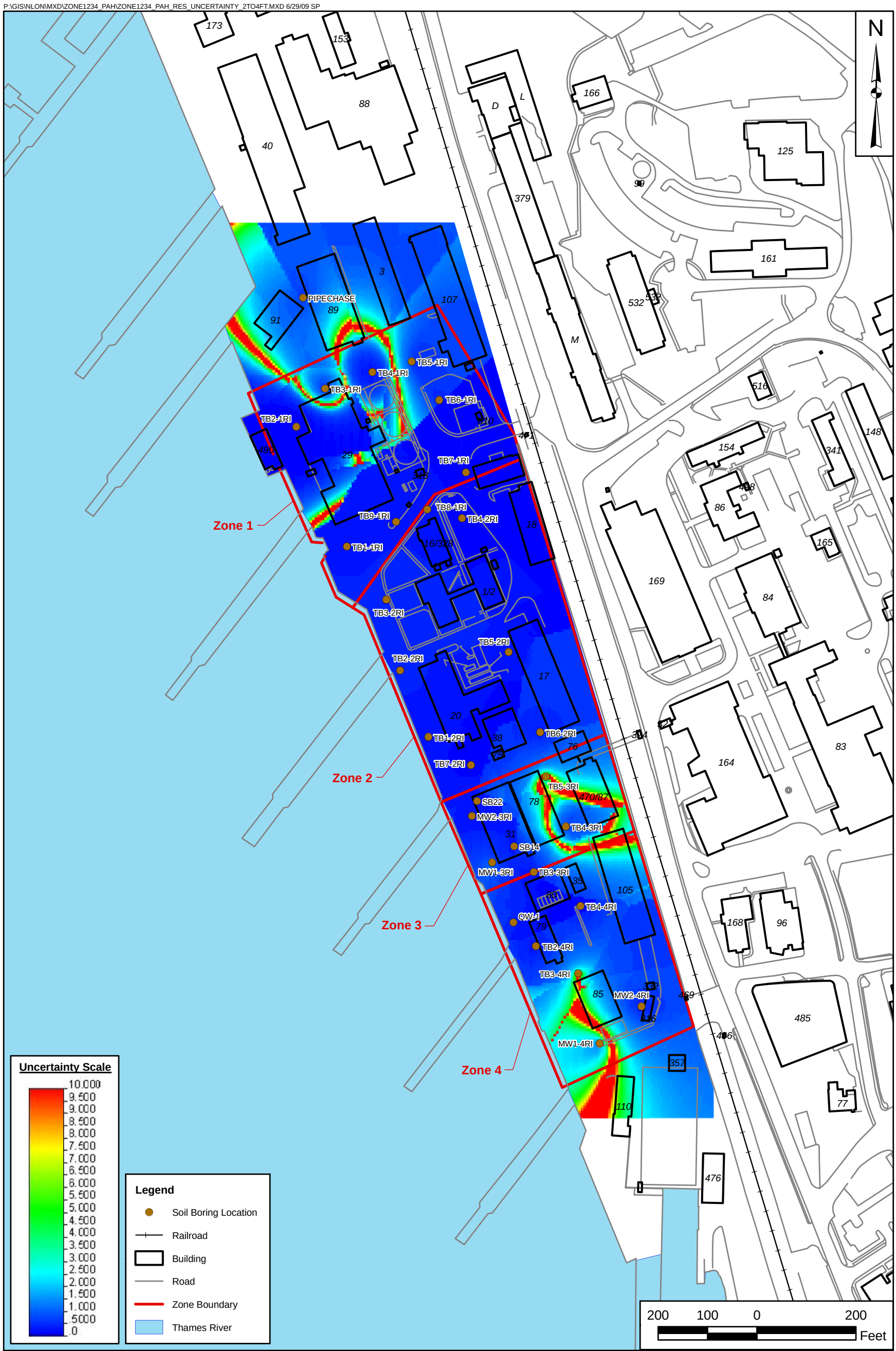
DRAWN BY S. PAXTON	DATE 4/02/09	Tetra Tech NUS, Inc.	CONTRACT NO. 0811
CHECKED BY N. BALSAMO	DATE 6/29/09	BAP EQUIVALENT UNCERTAINTY FOR 1000 UG/KG ELEVATION: 6 TO 8 FEET ZONES 1, 2, 3, AND 4 NAVAL SUBMARINE BASE - NEW LONDON GROTON, CONNECTICUT	APPROVED BY _ / DATE --
COST/SCHED-AREA			APPROVED BY -- / DATE --
SCALE AS NOTED			FIGURE NO. FIGURE B-15 / REV.

P:\GIS\NLON\MXD\ZONE1234_PAH\ZONE1234_PAH_RES_UNCERTAINTY_4TO6FT.MXD 6/29/09 SP



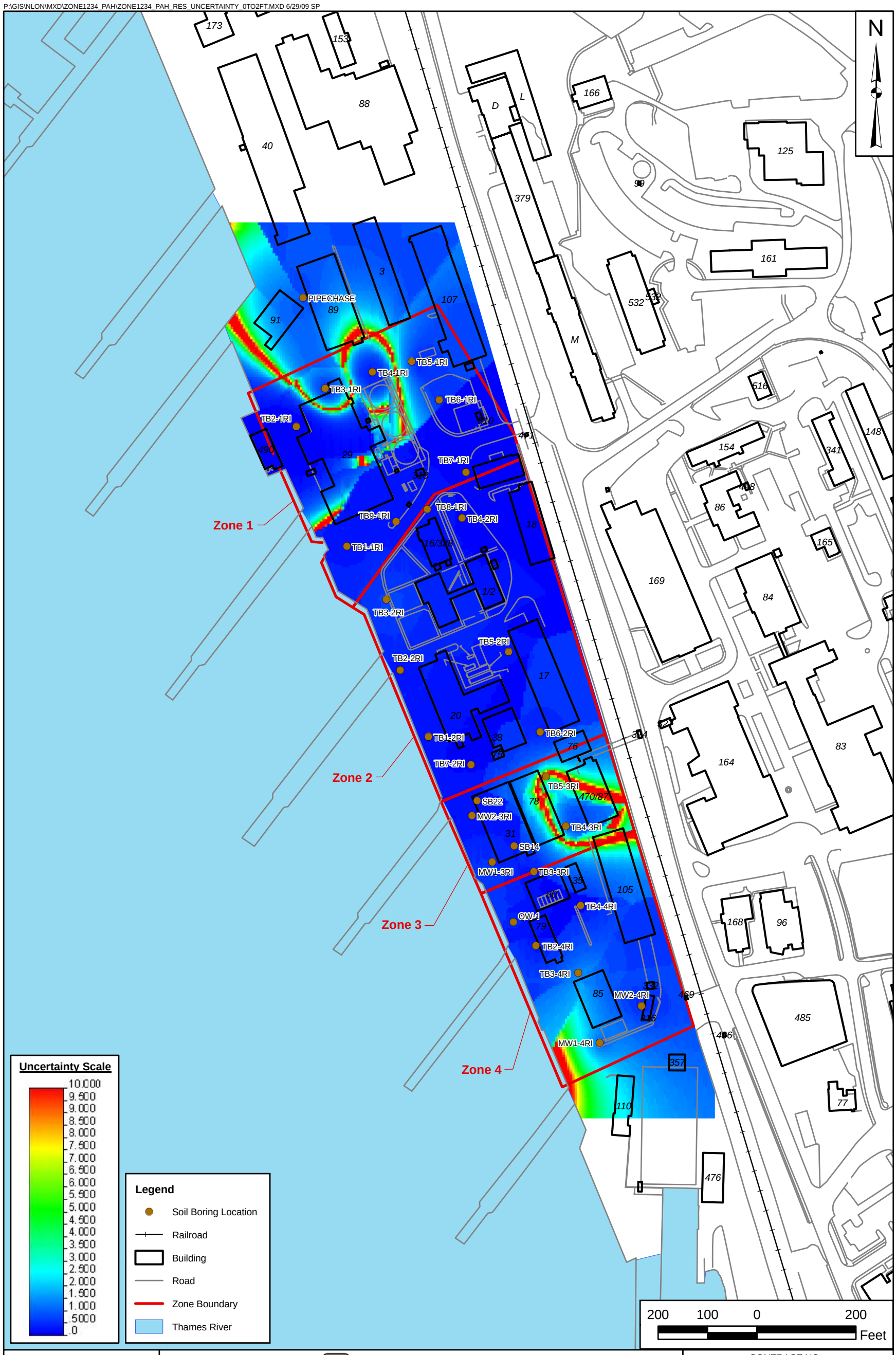
DRAWN BY S. PAXTON	DATE 4/02/09	Tetra Tech NUS, Inc.	CONTRACT NO. 0811
CHECKED BY N. BALSAMO	DATE 6/29/09	BAP EQUIVALENT UNCERTAINTY FOR 1000 UG/KG ELEVATION: 4 TO 6 FEET ZONES 1, 2, 3, AND 4 NAVAL SUBMARINE BASE - NEW LONDON GROTON, CONNECTICUT	APPROVED BY _ / DATE --
COST/SCHED-AREA			APPROVED BY -- / DATE --
SCALE AS NOTED			FIGURE NO. FIGURE B-16 / REV.

P:\GIS\NLON\MXD\ZONE1234_PAH\ZONE1234_PAH_RES_UNCERTAINTY_2TO4FT.MXD 6/29/09 SP



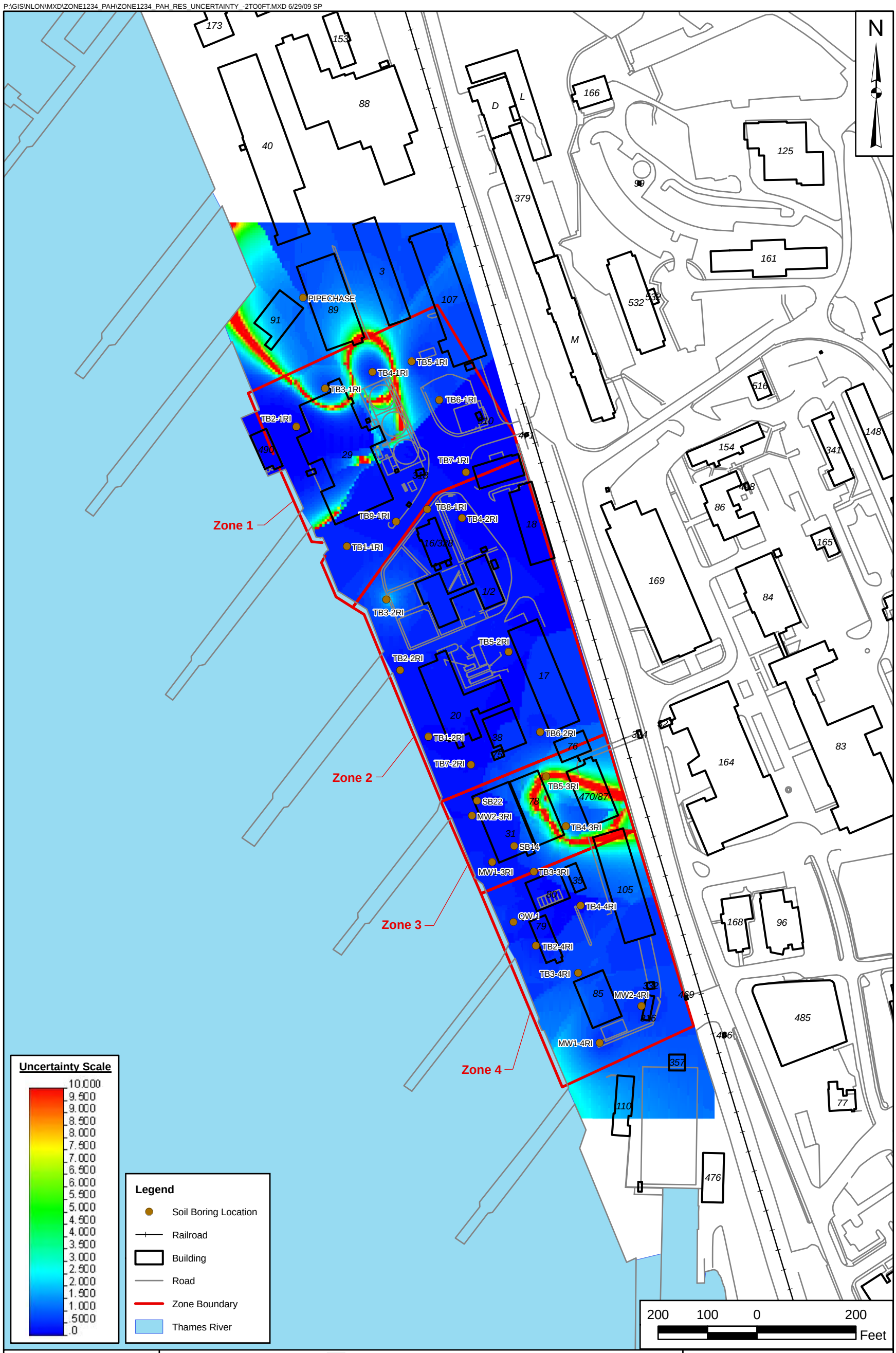
DRAWN BY S. PAXTON	DATE 4/02/09	Tetra Tech NUS, Inc.	CONTRACT NO. 0811
CHECKED BY N. BALSAMO	DATE 6/29/09	BAP EQUIVALENT UNCERTAINTY FOR 1000 UG/KG ELEVATION: 2 TO 4 FEET ZONES 1, 2, 3, AND 4 NAVAL SUBMARINE BASE - NEW LONDON GROTON, CONNECTICUT	APPROVED BY _ / DATE --
COST/SCHED-AREA			APPROVED BY -- / DATE --
SCALE AS NOTED			FIGURE NO. FIGURE B-17 / REV.

P:\GIS\NLON\MXD\ZONE1234_PAH\ZONE1234_PAH_RES_UNCERTAINTY_0TO2FT.MXD 6/29/09 SP



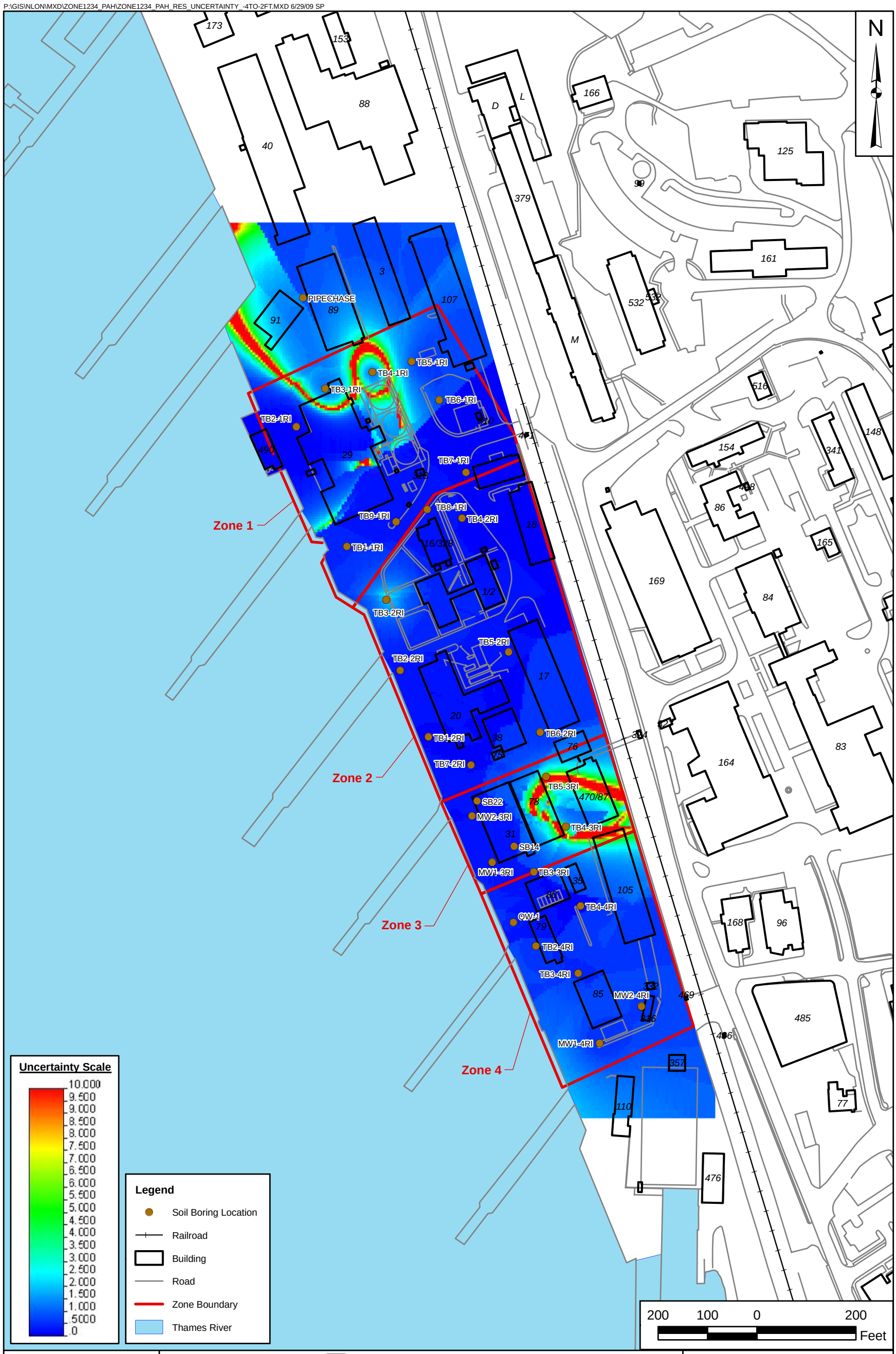
DRAWN BY S. PAXTON	DATE 4/02/09	Tetra Tech NUS, Inc.	CONTRACT NO. 0811
CHECKED BY N. BALSAMO	DATE 6/29/09	BAP EQUIVALENT UNCERTAINTY FOR 1000 UG/KG ELEVATION: 0 TO 2 FEET ZONES 1, 2, 3, AND 4 NAVAL SUBMARINE BASE - NEW LONDON GROTON, CONNECTICUT	APPROVED BY _ / DATE --
COST/SCHED-AREA			APPROVED BY -- / DATE --
SCALE AS NOTED			FIGURE NO. FIGURE B-18 / REV.

P:\GIS\NLON\MXD\ZONE1234_PAH\ZONE1234_PAH_RES_UNCERTAINTY_-2TO0FT.MXD 6/29/09 SP



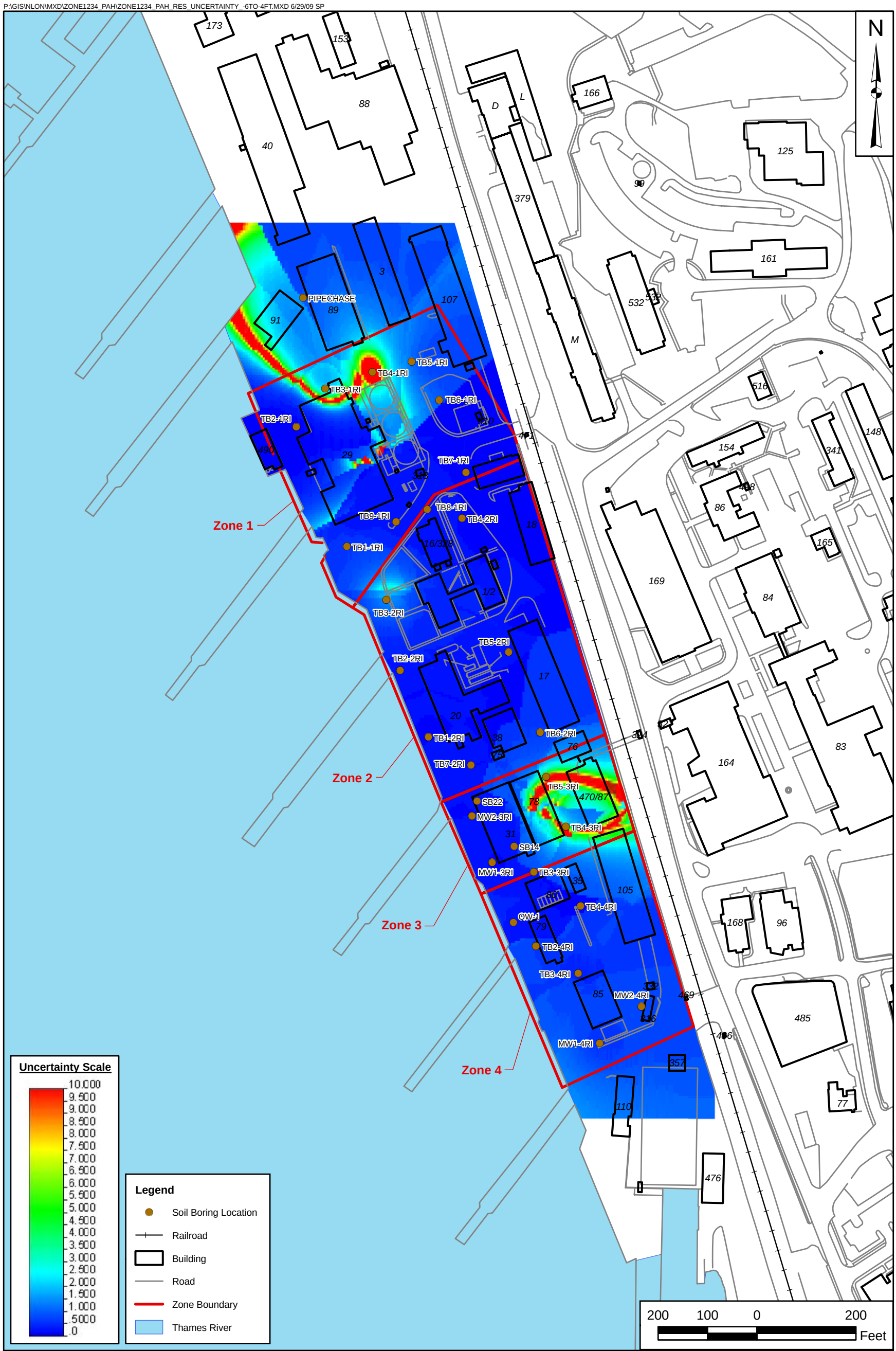
DRAWN BY S. PAXTON	DATE 4/02/09	Tetra Tech NUS, Inc.	CONTRACT NO. 0811
CHECKED BY N. BALSAMO	DATE 6/29/09	BAP EQUIVALENT UNCERTAINTY FOR 1000 UG/KG ELEVATION: -2 TO 0 FEET ZONES 1, 2, 3, AND 4 NAVAL SUBMARINE BASE - NEW LONDON GROTON, CONNECTICUT	APPROVED BY _ / DATE --
COST/SCHED-AREA			APPROVED BY -- / DATE --
SCALE AS NOTED			FIGURE NO. FIGURE B-19 / REV.

P:\GIS\NLON\MXD\ZONE1234_PAH\ZONE1234_PAH_RES_UNCERTAINTY_-4TO-2FT.MXD 6/29/09 SP



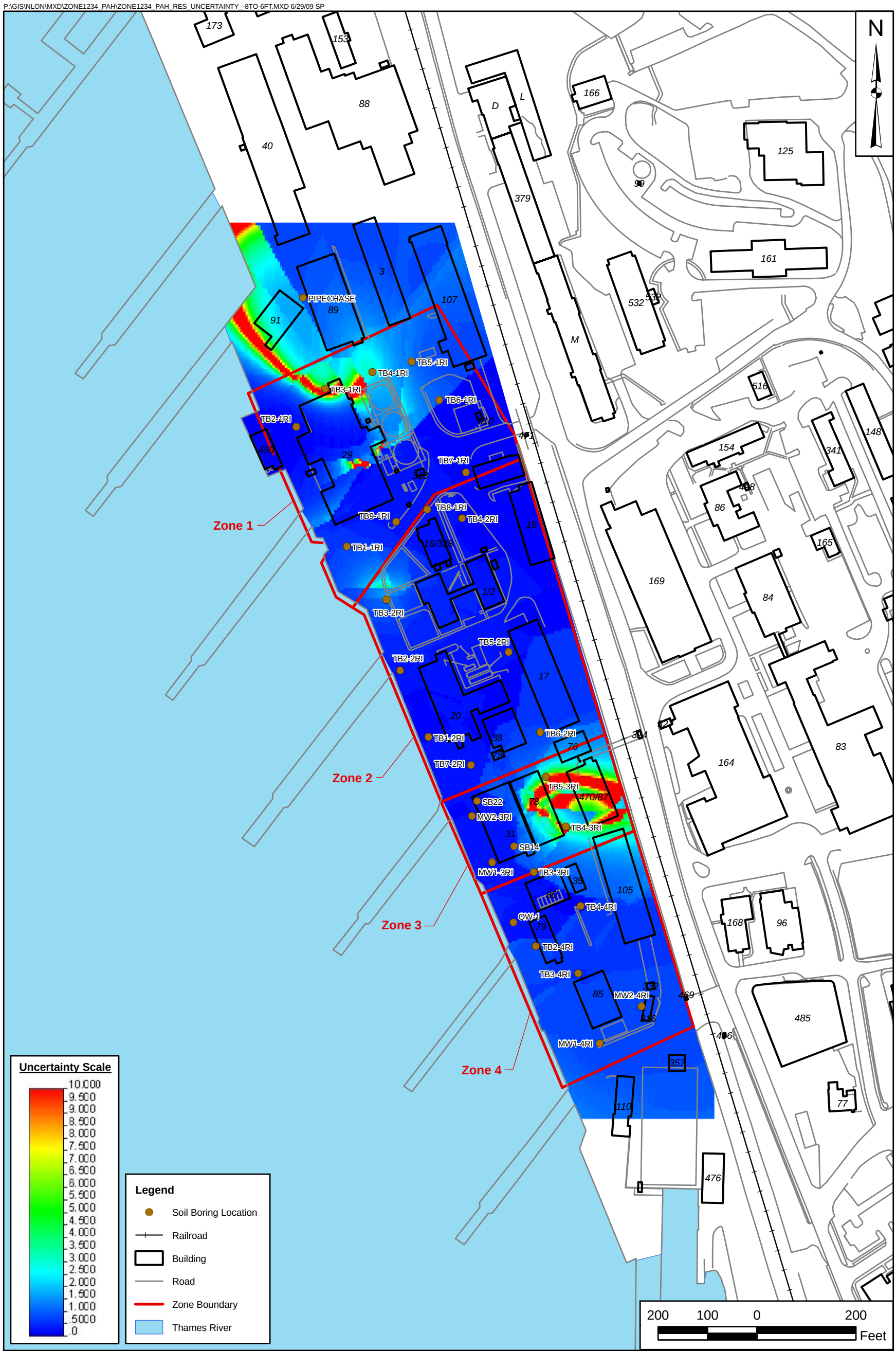
DRAWN BY S. PAXTON	DATE 4/02/09	Tetra Tech NUS, Inc.	CONTRACT NO. 0811
CHECKED BY N. BALSAMO	DATE 6/29/09	BAP EQUIVALENT UNCERTAINTY FOR 1000 UG/KG ELEVATION: -4 TO -2 FEET ZONES 1, 2, 3, AND 4 NAVAL SUBMARINE BASE - NEW LONDON GROTON, CONNECTICUT	APPROVED BY _ / DATE --
COST/SCHED-AREA			APPROVED BY -- / DATE --
SCALE AS NOTED			FIGURE NO. FIGURE B-20 / REV.

P:\GIS\NLON\MXD\ZONE1234_PAH\ZONE1234_PAH_RES_UNCERTAINTY_-6TO-4FT.MXD 6/29/09 SP



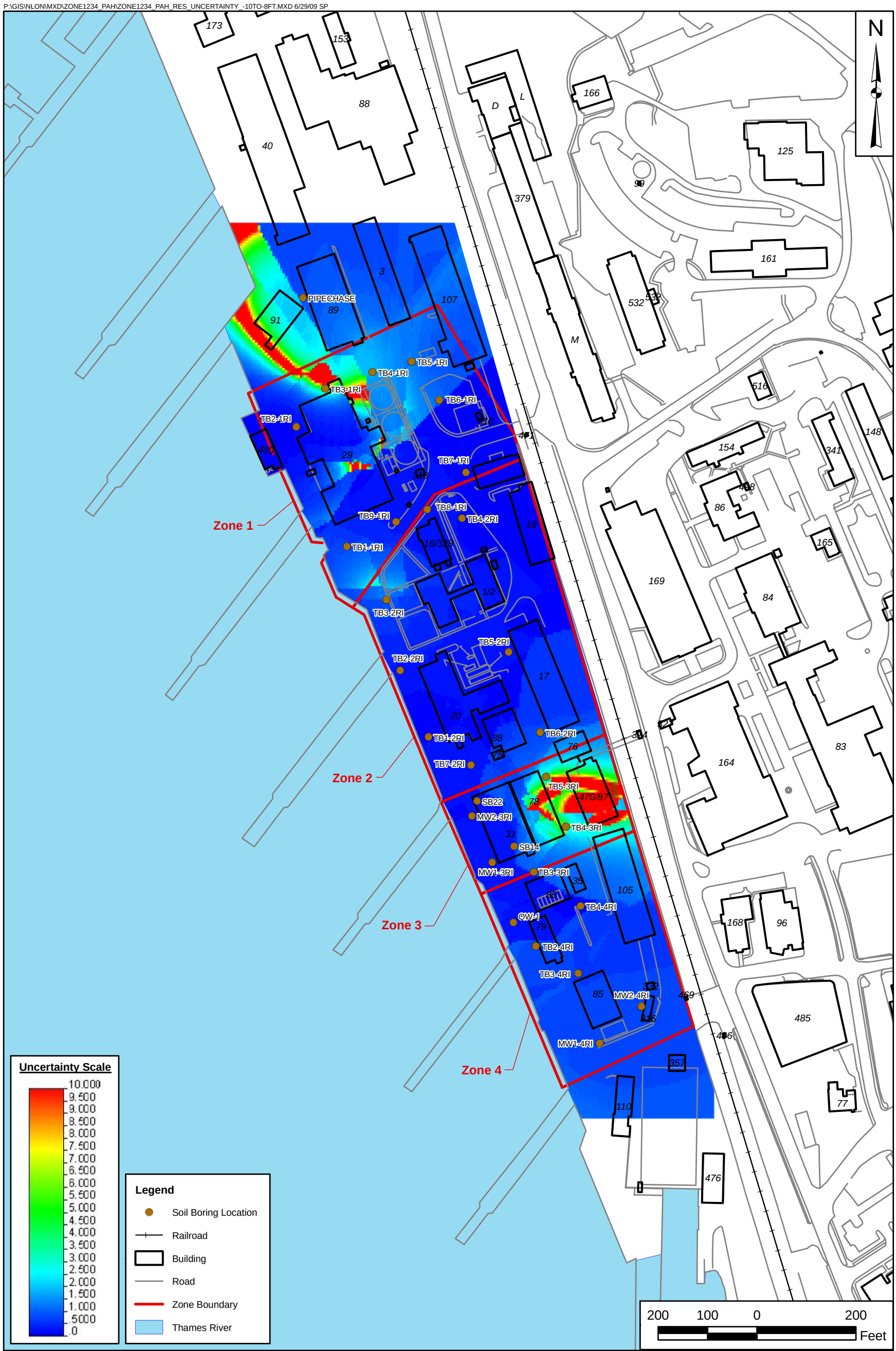
DRAWN BY S. PAXTON	DATE 4/02/09	Tetra Tech NUS, Inc.	CONTRACT NO. 0811
CHECKED BY N. BALSAMO	DATE 6/29/09	BAP EQUIVALENT UNCERTAINTY FOR 1000 UG/KG ELEVATION: -6 TO -4 FEET ZONES 1, 2, 3, AND 4 NAVAL SUBMARINE BASE - NEW LONDON GROTON, CONNECTICUT	APPROVED BY _ / DATE --
COST/SCHED-AREA			APPROVED BY -- / DATE --
SCALE AS NOTED			FIGURE NO. FIGURE B-21 / REV.

P:\GIS\NLON\MXD\ZONE1234_PAH\ZONE1234_PAH_RES_UNCERTAINTY_-8TO-6FT.MXD 6/29/09 SP



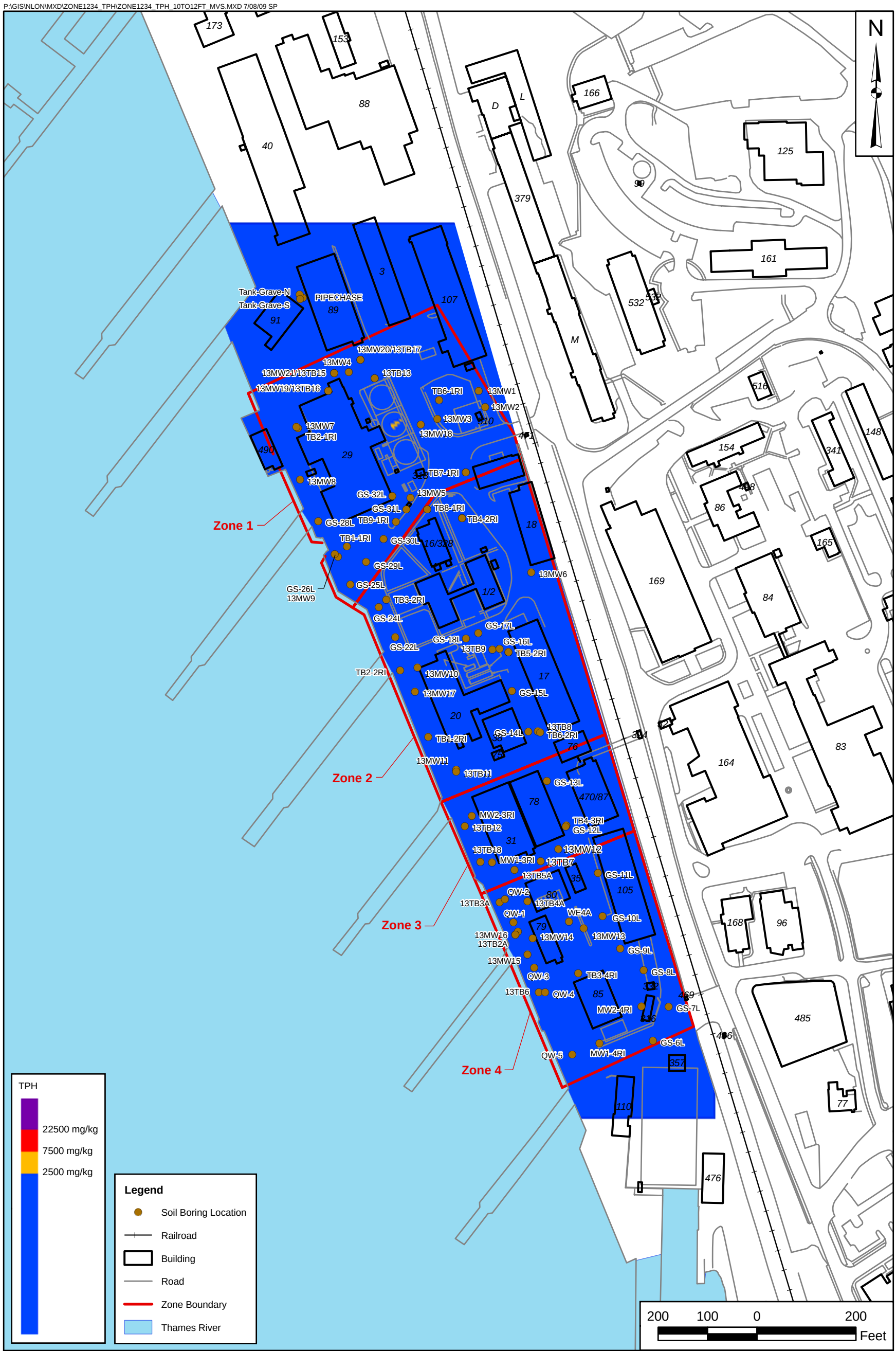
DRAWN BY	DATE	Tetra Tech NUS, Inc.	CONTRACT NO.
S. PAXTON	4/02/09		0811
CHECKED BY	DATE	BAP EQUIVALENT UNCERTAINTY FOR 1000 UG/KG	APPROVED BY / DATE
N. BALSAMO	6/29/09	ELEVATION: -8 TO -6 FEET	_ / --
COST/SCHED-AREA		ZONES 1, 2, 3, AND 4	APPROVED BY / DATE
		NAVAL SUBMARINE BASE - NEW LONDON	-- / --
SCALE		GROTON, CONNECTICUT	FIGURE NO. / REV.
AS NOTED			FIGURE B-22

P:\GIS\NLON\MXD\ZONE1234_PAH\ZONE1234_PAH_RES_UNCERTAINTY_-10TO-8FT.MXD 6/29/09 SP



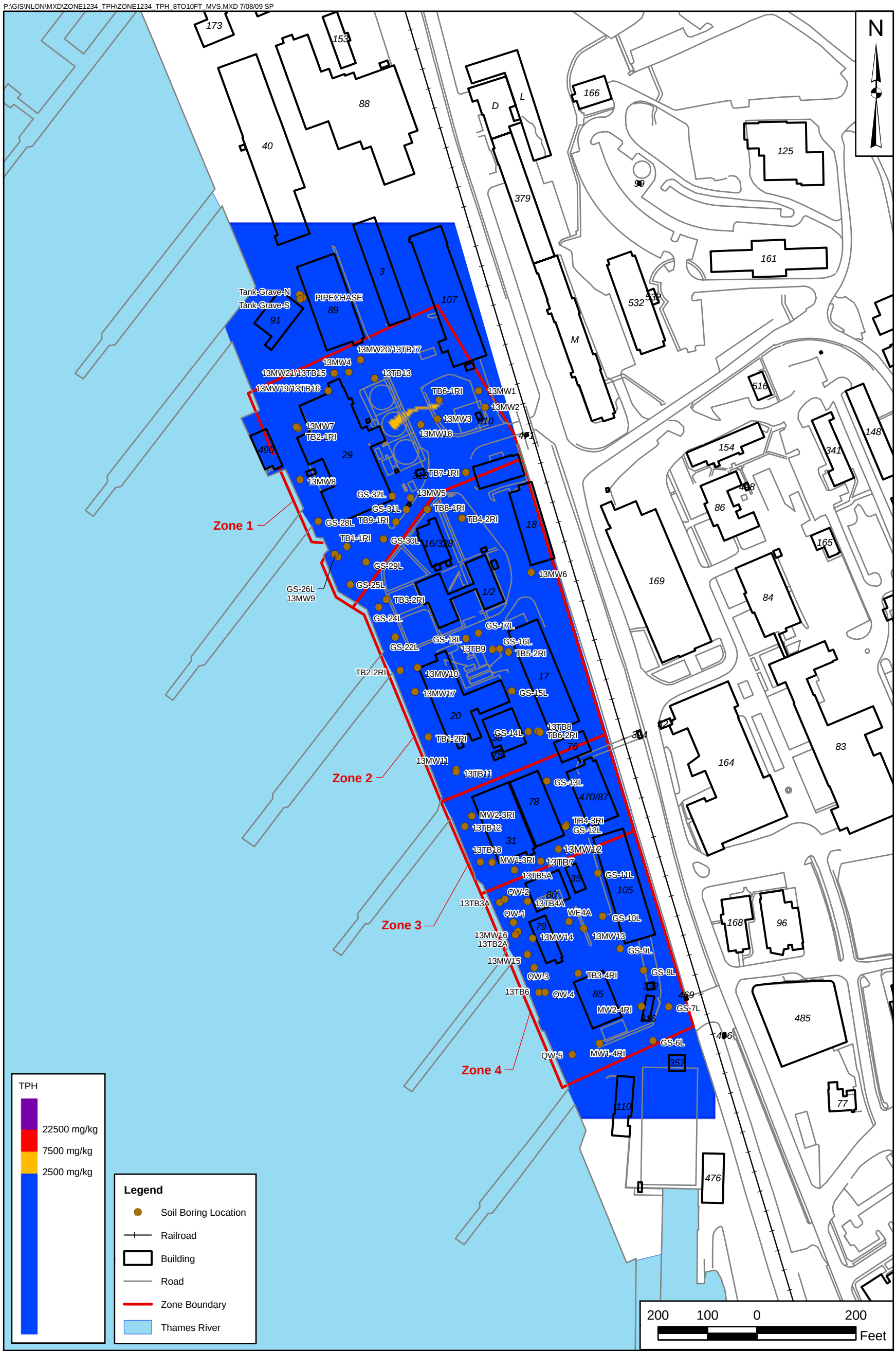
DRAWN BY S. PAXTON	DATE 4/02/09	Tetra Tech NUS, Inc.	CONTRACT NO. 0811
CHECKED BY N. BALSAMO	DATE 6/29/09	BAP EQUIVALENT UNCERTAINTY FOR 1000 UG/KG ELEVATION: -10 TO -8 FEET ZONES 1, 2, 3, AND 4 NAVAL SUBMARINE BASE - NEW LONDON GROTON, CONNECTICUT	APPROVED BY _ / DATE --
COST/SCHED-AREA			APPROVED BY -- / DATE --
SCALE AS NOTED			FIGURE NO. FIGURE B-23 / REV.

P:\GIS\NLON\MXD\ZONE1234_TPH\ZONE1234_TPH_10TO12FT_MVS.MXD 7/08/09 SP



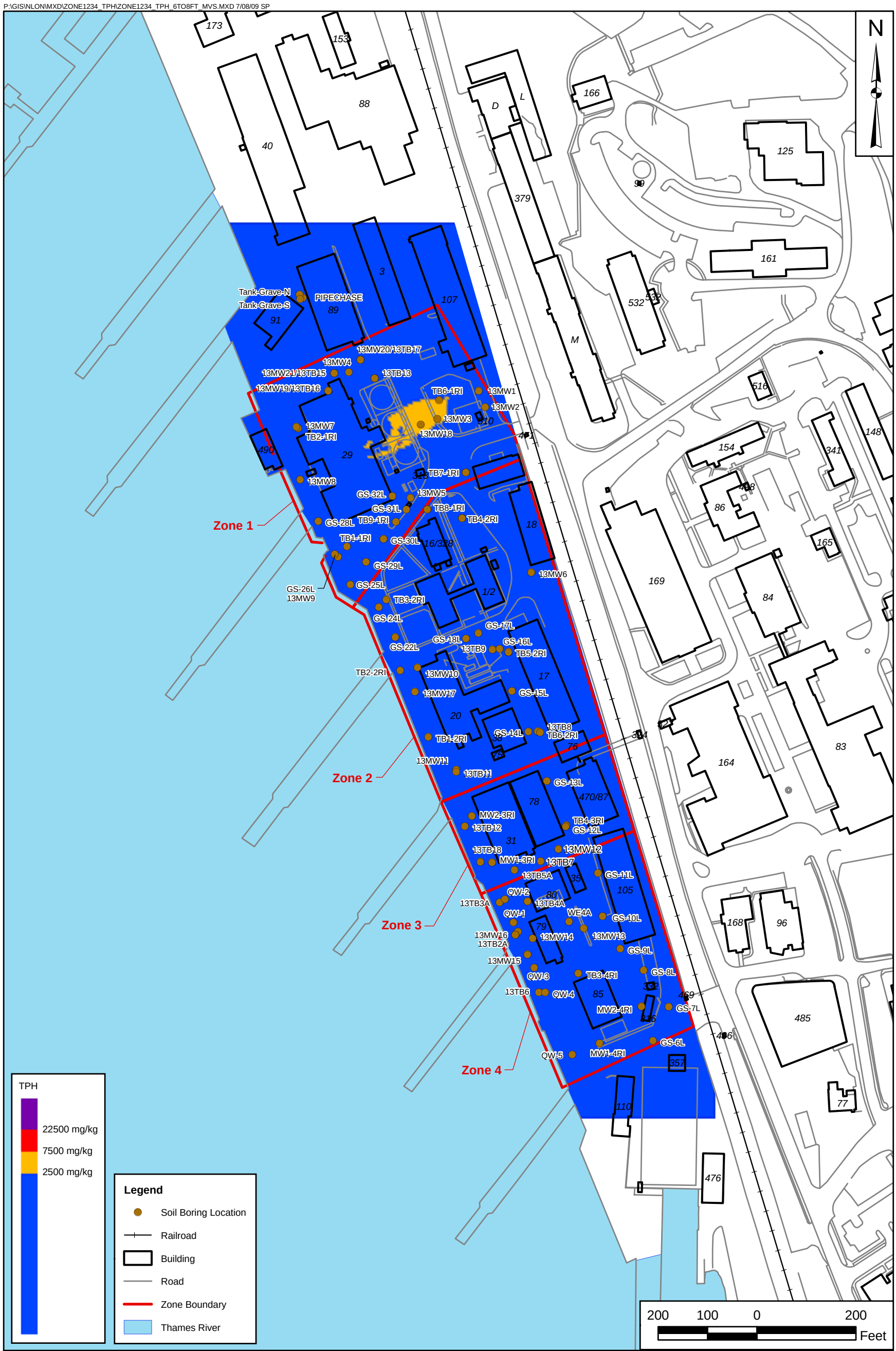
DRAWN BY S. PAXTON	DATE 4/02/09	Tetra Tech NUS, Inc.	CONTRACT NO. 0811
CHECKED BY N. BALSAMO	DATE 7/08/09	ESTIMATED TPH CONCENTRATIONS (KRIGING RESULTS) ELEVATION: 10 TO 12 FEET ZONES 1, 2, 3, AND 4 NAVAL SUBMARINE BASE - NEW LONDON GROTON, CONNECTICUT	APPROVED BY _ / DATE --
COST/SCHED-AREA			APPROVED BY -- / DATE --
SCALE AS NOTED			FIGURE NO. FIGURE B-24 / REV.

P:\GIS\NLON\MXD\ZONE1234_TPH\ZONE1234_TPH_8TO10FT_MVS.MXD 7/08/09 SP



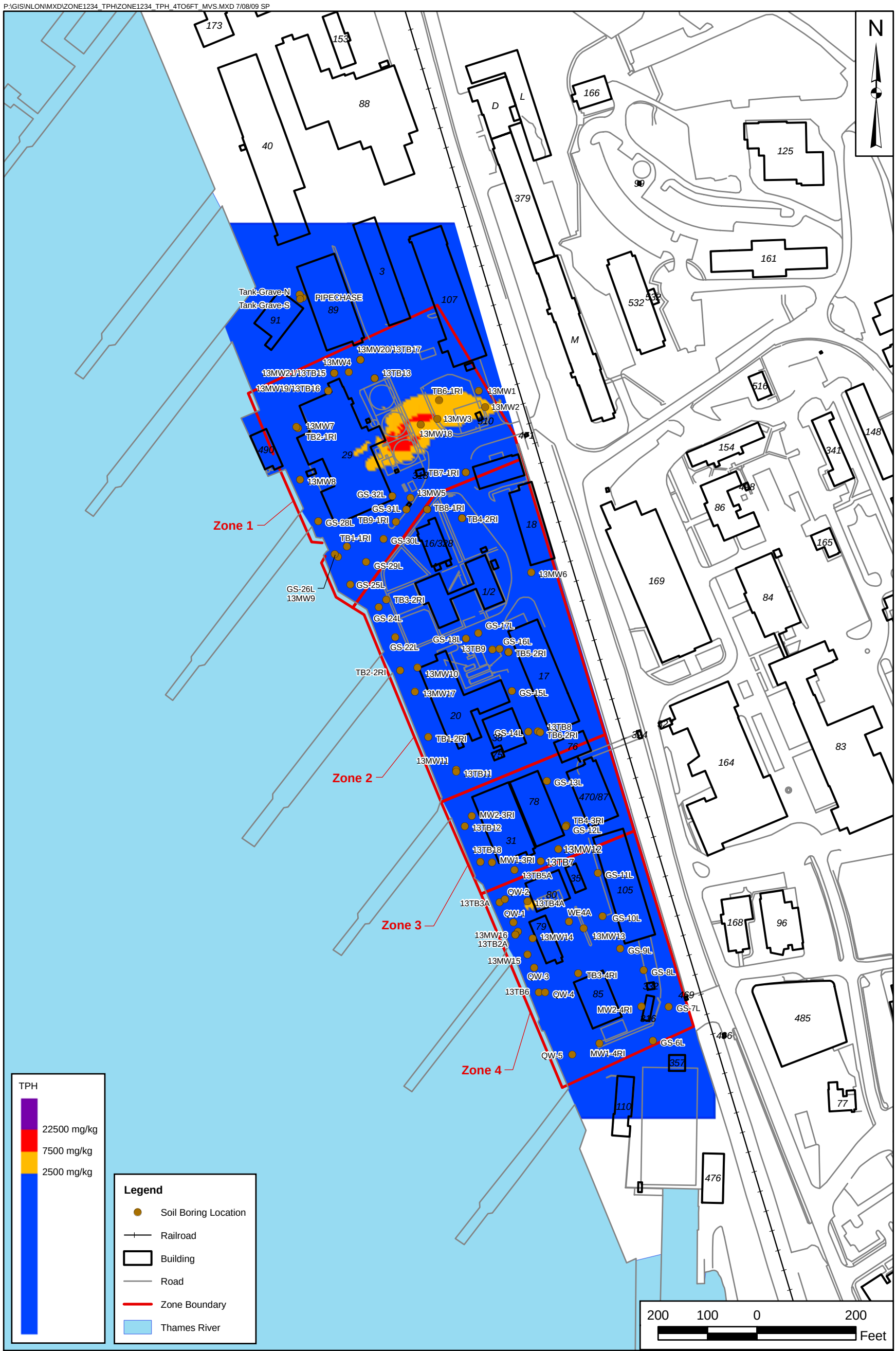
DRAWN BY S. PAXTON	DATE 4/02/09	Tetra Tech NUS, Inc.	CONTRACT NO. 0811
CHECKED BY N. BALSAMO	DATE 7/08/09	ESTIMATED TPH CONCENTRATIONS (KRIGING RESULTS) ELEVATION: 8 TO 10 FEET ZONES 1, 2, 3, AND 4 NAVAL SUBMARINE BASE - NEW LONDON GROTON, CONNECTICUT	APPROVED BY _ / DATE --
COST/SCHED-AREA			APPROVED BY -- / DATE --
SCALE AS NOTED			FIGURE NO. FIGURE B-25 / REV.

P:\GIS\NLON\MXD\ZONE1234_TPH\ZONE1234_TPH_6TO8FT_MVS.MXD 7/08/09 SP



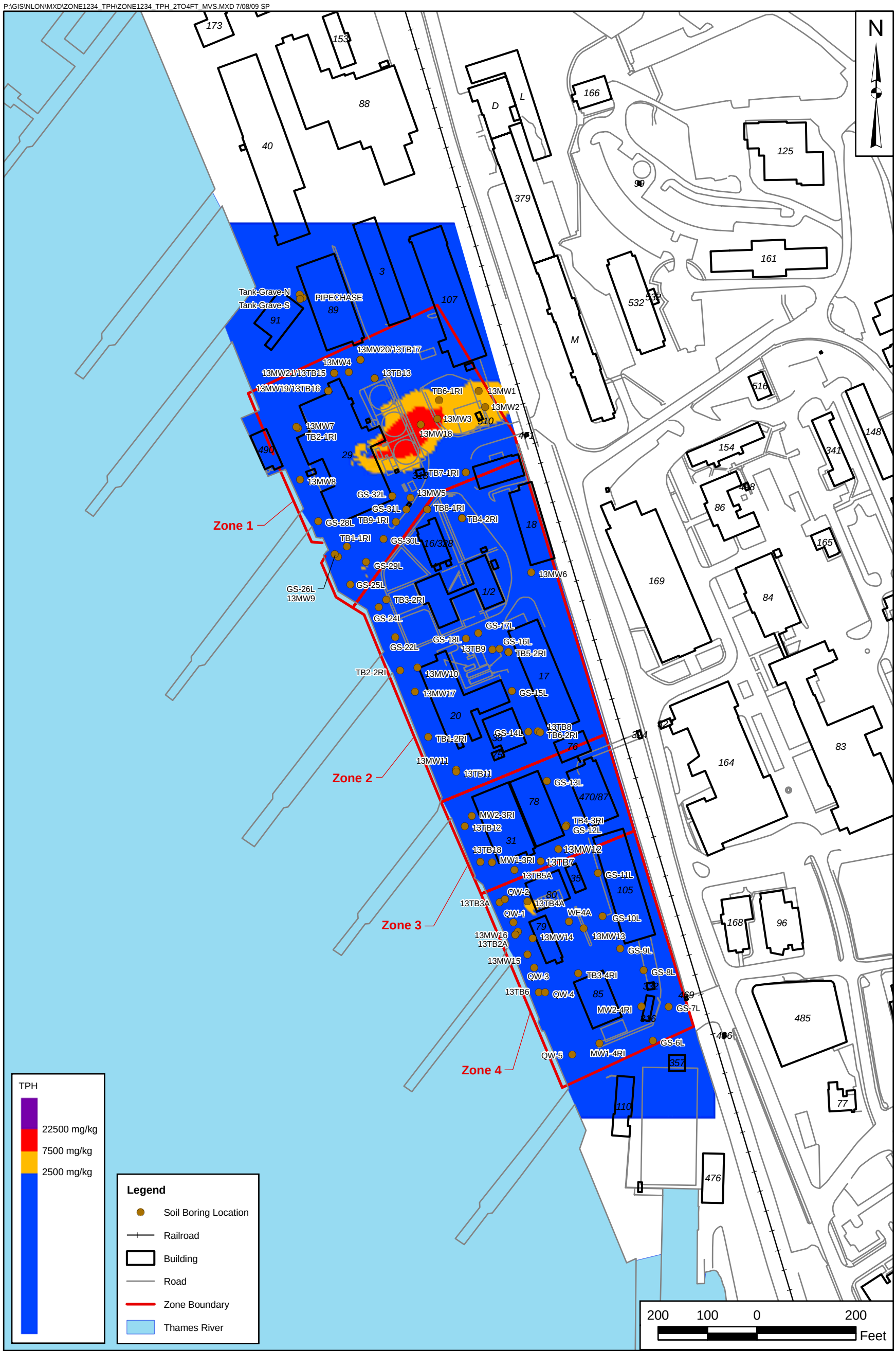
DRAWN BY	DATE	Tetra Tech NUS, Inc.	CONTRACT NO.
S. PAXTON	4/02/09		0811
CHECKED BY	DATE	ESTIMATED TPH CONCENTRATIONS (KRIGING RESULTS)	APPROVED BY / DATE
N. BALSAMO	7/08/09	ELEVATION: 6 TO 8 FEET	_ / --
COST/SCHED-AREA		ZONES 1, 2, 3, AND 4	APPROVED BY / DATE
		NAVAL SUBMARINE BASE - NEW LONDON	-- / --
SCALE		GROTON, CONNECTICUT	FIGURE NO. / REV.
AS NOTED			FIGURE B-26

P:\GIS\NLON\MXD\ZONE1234_TPH\ZONE1234_TPH_4TO6FT_MVS.MXD 7/08/09 SP



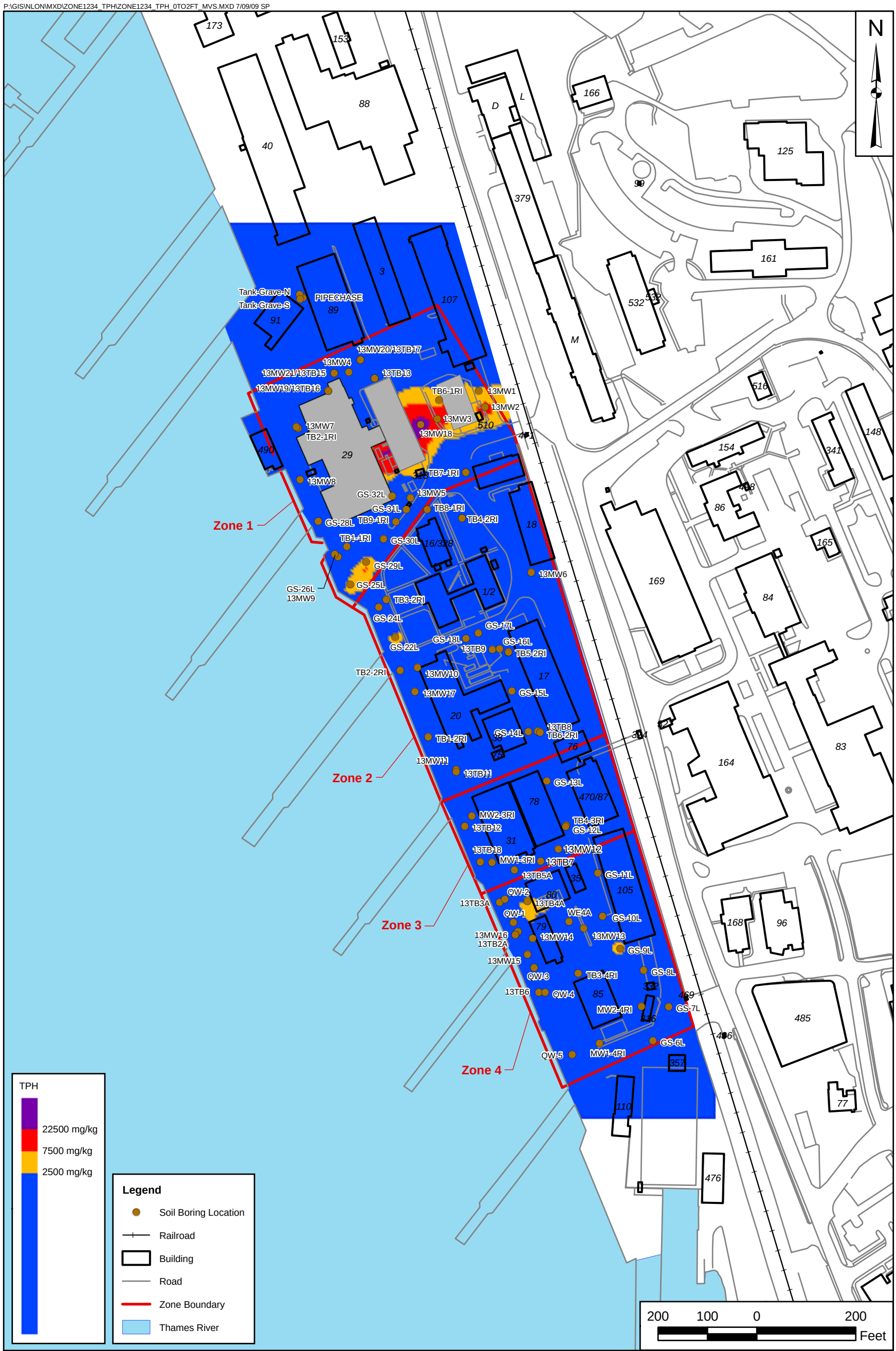
DRAWN BY	DATE	Tetra Tech NUS, Inc.	CONTRACT NO.
S. PAXTON	4/02/09		0811
CHECKED BY	DATE	ESTIMATED TPH CONCENTRATIONS (KRIGING RESULTS)	APPROVED BY / DATE
N. BALSAMO	7/08/09	ELEVATION: 4 TO 6 FEET	_ / --
COST/SCHED-AREA		ZONES 1, 2, 3, AND 4	APPROVED BY / DATE
		NAVAL SUBMARINE BASE - NEW LONDON	-- / --
SCALE		GROTON, CONNECTICUT	FIGURE NO. / REV.
AS NOTED			FIGURE B-27

P:\GIS\NLON\MXD\ZONE1234_TPH\ZONE1234_TPH_2TO4FT_MVS.MXD 7/08/09 SP



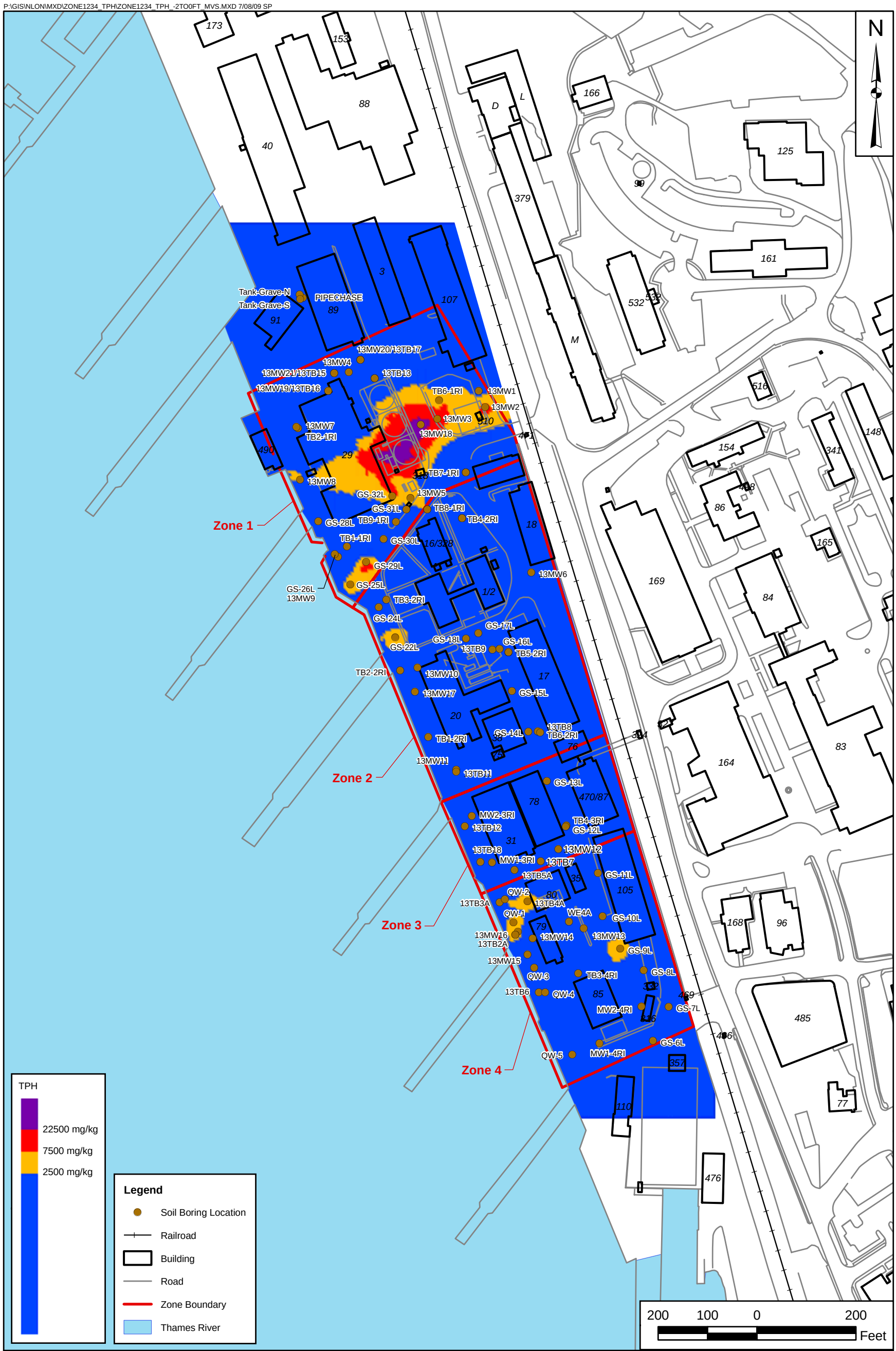
DRAWN BY S. PAXTON	DATE 4/02/09	Tetra Tech NUS, Inc.	CONTRACT NO. 0811
CHECKED BY N. BALSAMO	DATE 7/08/09	ESTIMATED TPH CONCENTRATIONS (KRIGING RESULTS) ELEVATION: 2 TO 4 FEET ZONES 1, 2, 3, AND 4 NAVAL SUBMARINE BASE - NEW LONDON GROTON, CONNECTICUT	APPROVED BY _ / DATE --
COST/SCHED-AREA			APPROVED BY -- / DATE --
SCALE AS NOTED			FIGURE NO. FIGURE B-28 / REV.

P:\GIS\NLON\MXD\ZONE1234_TPH\ZONE1234_TPH_0TO2FT_MVS.MXD 7/09/09 SP



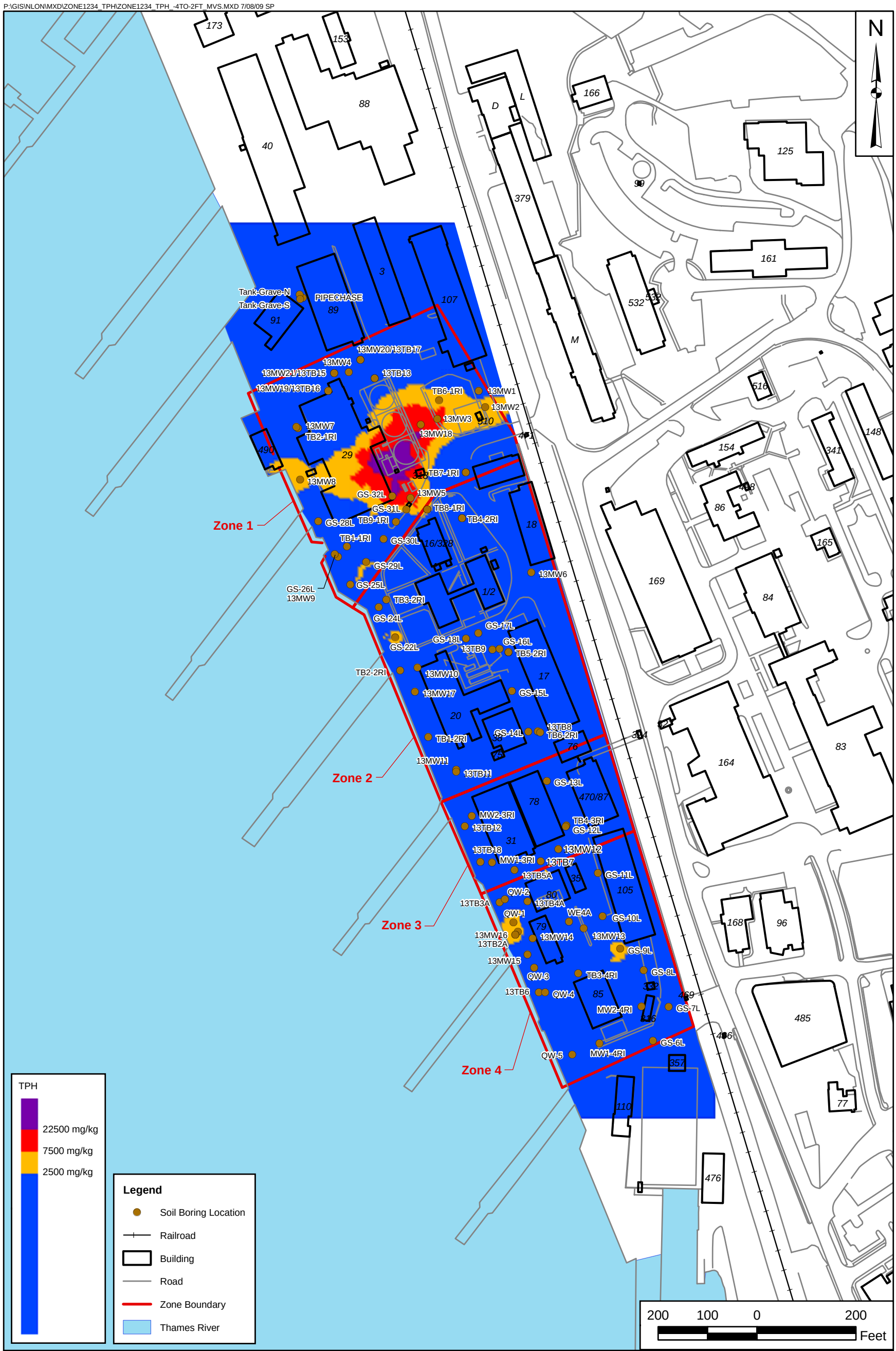
DRAWN BY S. PAXTON	DATE 4/02/09	Tetra Tech NUS, Inc.	CONTRACT NO. 0811
CHECKED BY N. BALSAMO	DATE 7/09/09	ESTIMATED TPH CONCENTRATIONS (KRIGING RESULTS) ELEVATION: 0 TO 2 FEET ZONES 1, 2, 3, AND 4 NAVAL SUBMARINE BASE - NEW LONDON GROTON, CONNECTICUT	APPROVED BY _ / DATE --
COST/SCHED-AREA			APPROVED BY -- / DATE --
SCALE AS NOTED			FIGURE NO. FIGURE B-29 / REV.

P:\GIS\NLON\MXD\ZONE1234_TPH\ZONE1234_TPH_-2TO0FT_MVS.MXD 7/08/09 SP



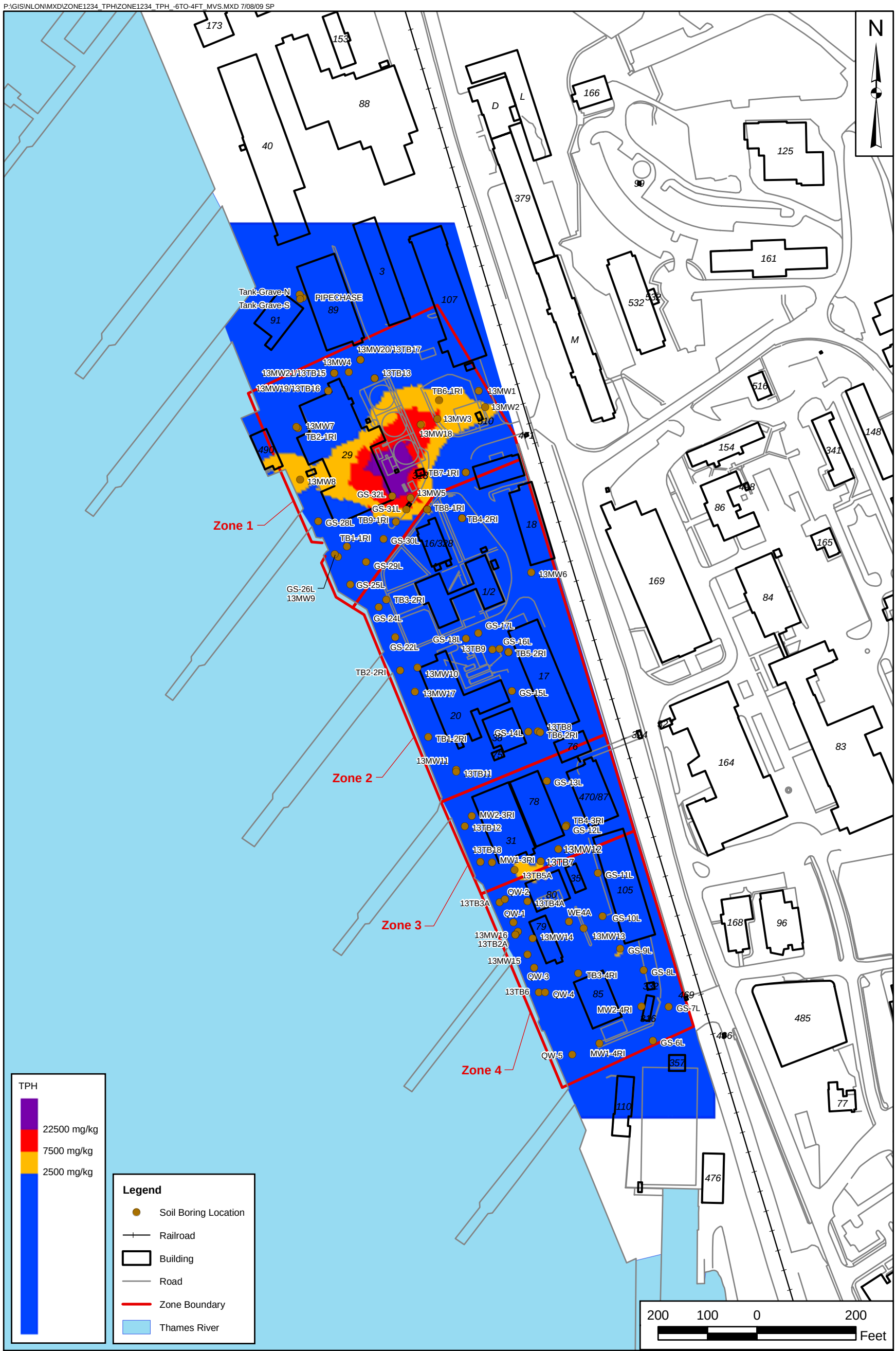
DRAWN BY S. PAXTON	DATE 4/02/09	Tetra Tech NUS, Inc.	CONTRACT NO. 0811
CHECKED BY N. BALSAMO	DATE 7/08/09	ESTIMATED TPH CONCENTRATIONS (KRIGING RESULTS) ELEVATION: -2 TO 0 FEET ZONES 1, 2, 3, AND4 NAVAL SUBMARINE BASE - NEW LONDON GROTON, CONNECTICUT	APPROVED BY _ / DATE --
COST/SCHED-AREA			APPROVED BY -- / DATE --
SCALE AS NOTED			FIGURE NO. FIGURE B-30 / REV.

P:\GIS\NLON\MXD\ZONE1234_TPH\ZONE1234_TPH_-4TO-2FT_MVS.MXD 7/08/09 SP



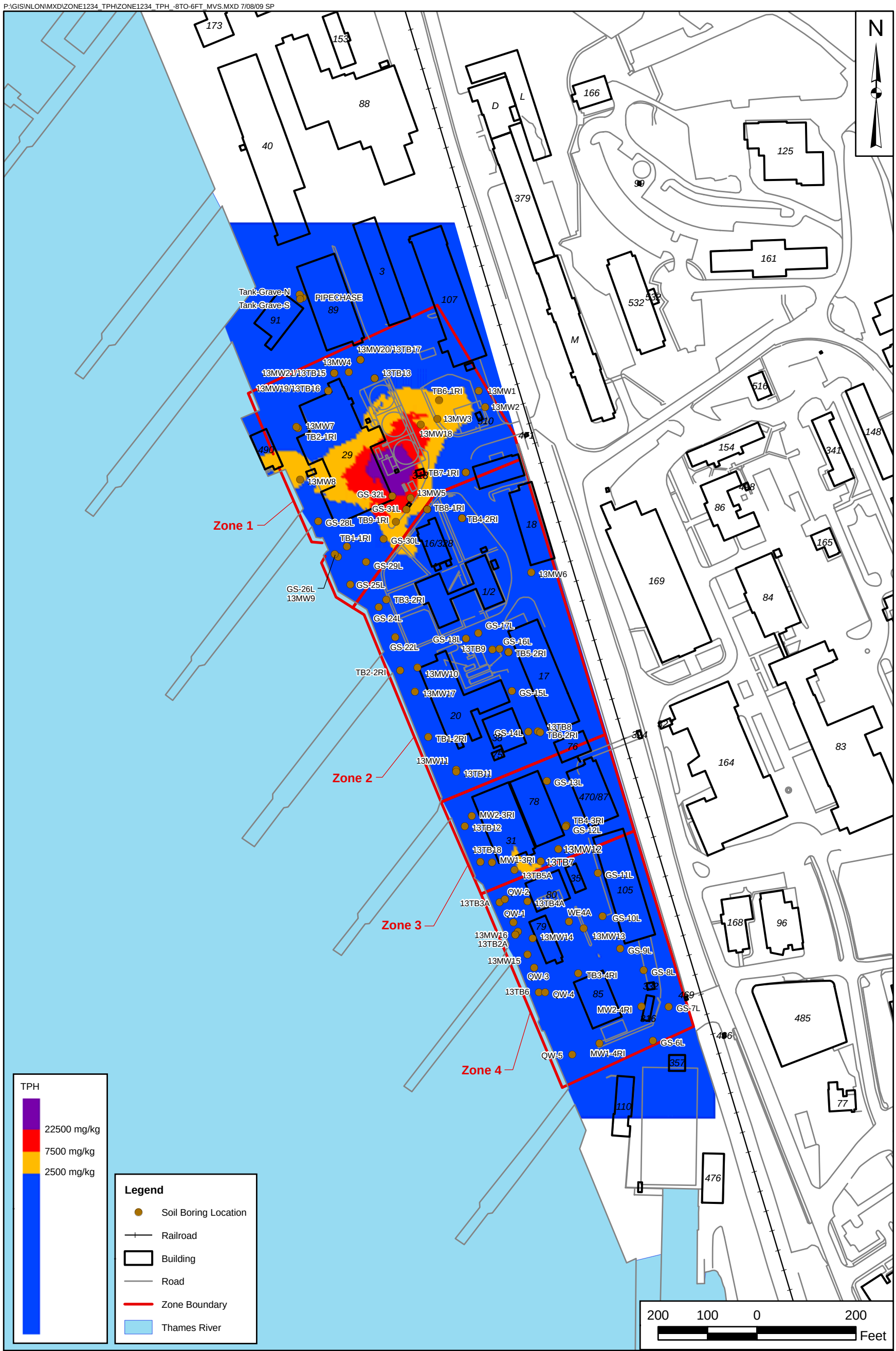
DRAWN BY	DATE	Tetra Tech NUS, Inc.	CONTRACT NO.
S. PAXTON	4/02/09		0811
CHECKED BY	DATE	ESTIMATED TPH CONCENTRATIONS (KRIGING RESULTS)	APPROVED BY / DATE
N. BALSAMO	7/08/09	ELEVATION: -4 TO -2 FEET	_ / --
COST/SCHED-AREA		ZONES 1, 2, 3, AND 4	APPROVED BY / DATE
		NAVAL SUBMARINE BASE - NEW LONDON	-- / --
SCALE		GROTON, CONNECTICUT	FIGURE NO. / REV.
AS NOTED			FIGURE B-31

P:\GIS\NLON\MXD\ZONE1234_TPH\ZONE1234_TPH_-6TO-4FT_MVS.MXD 7/08/09 SP



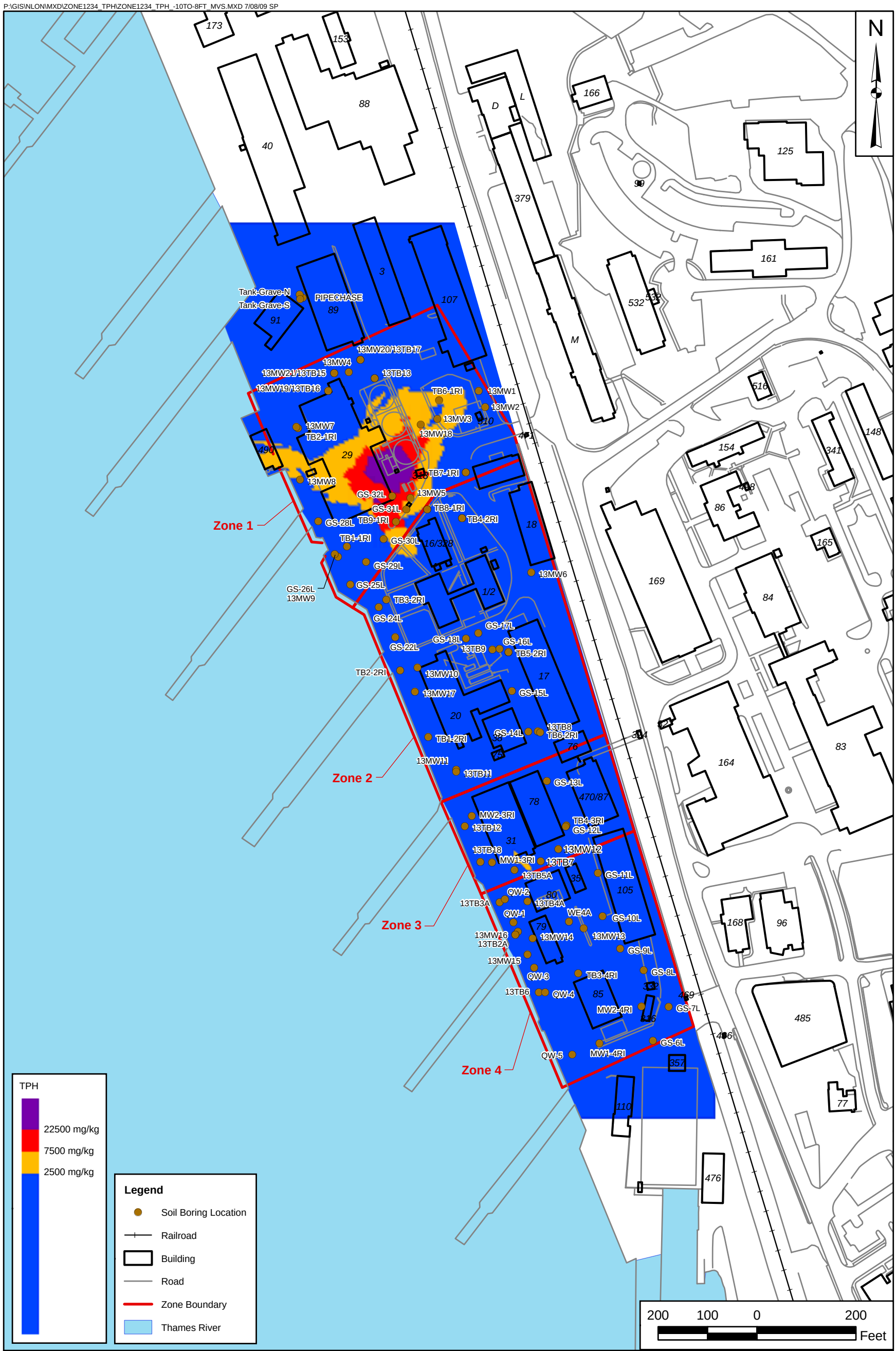
DRAWN BY S. PAXTON	DATE 4/02/09	Tetra Tech NUS, Inc.	CONTRACT NO. 0811
CHECKED BY N. BALSAMO	DATE 7/08/09	ESTIMATED TPH CONCENTRATIONS (KRIGING RESULTS) ELEVATION: -6 TO -4 FEET ZONES 1, 2, 3, AND 4 NAVAL SUBMARINE BASE - NEW LONDON GROTON, CONNECTICUT	APPROVED BY _ / DATE --
COST/SCHED-AREA			APPROVED BY -- / DATE --
SCALE AS NOTED			FIGURE NO. FIGURE B-32 / REV.

P:\GIS\NLON\MXD\ZONE1234_TPH\ZONE1234_TPH_-8TO-6FT_MVS.MXD 7/08/09 SP



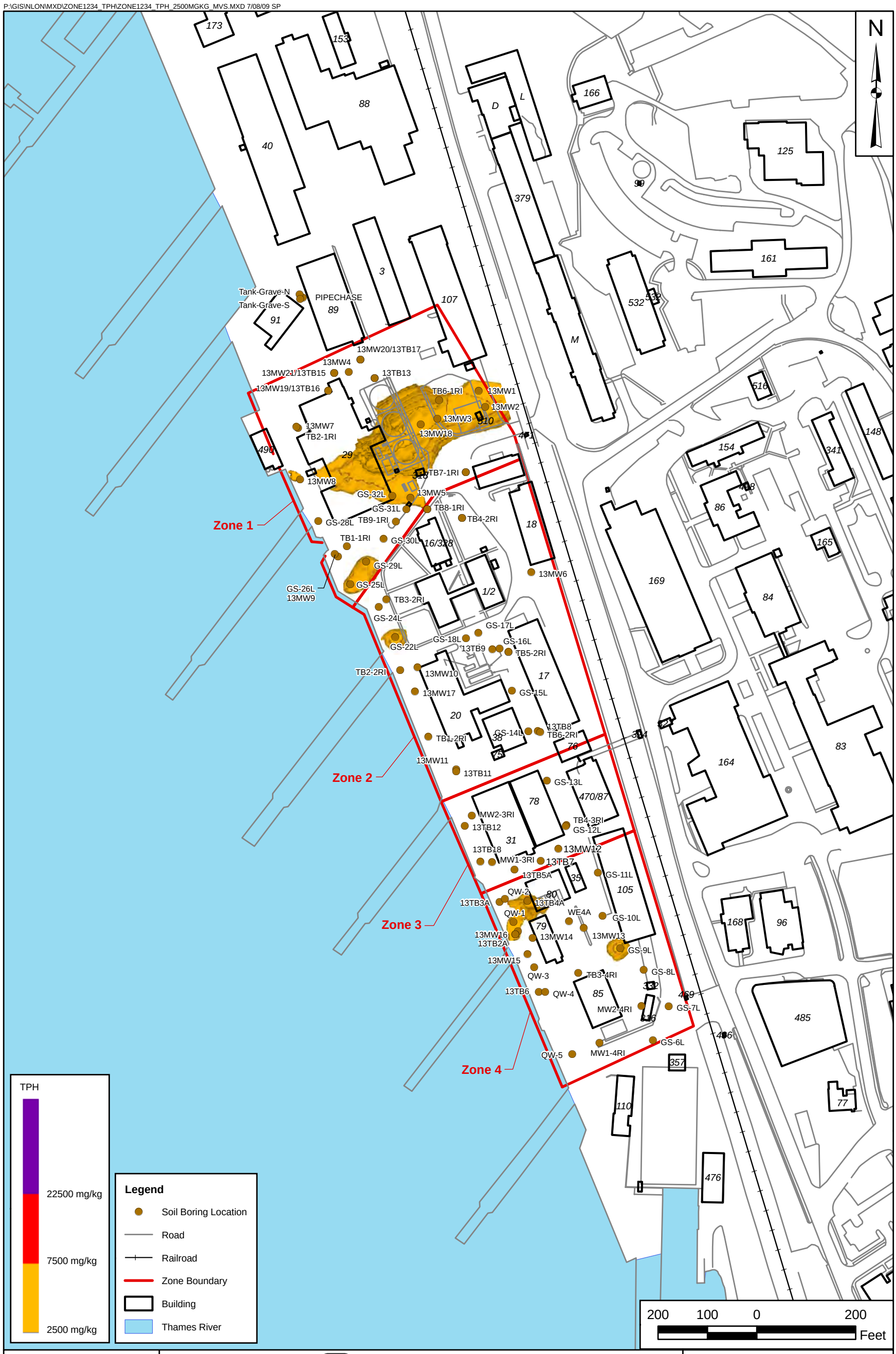
DRAWN BY S. PAXTON	DATE 4/02/09	Tetra Tech NUS, Inc.	CONTRACT NO. 0811
CHECKED BY N. BALSAMO	DATE 7/08/09	ESTIMATED TPH CONCENTRATIONS (KRIGING RESULTS) ELEVATION: -8 TO -6 FEET ZONES 1, 2, 3, AND 4 NAVAL SUBMARINE BASE - NEW LONDON GROTON, CONNECTICUT	APPROVED BY _ / DATE --
COST/SCHED-AREA			APPROVED BY -- / DATE --
SCALE AS NOTED			FIGURE NO. FIGURE B-33 / REV.

P:\GIS\NLON\MXD\ZONE1234_TPH\ZONE1234_TPH_-10TO-8FT_MVS.MXD 7/08/09 SP



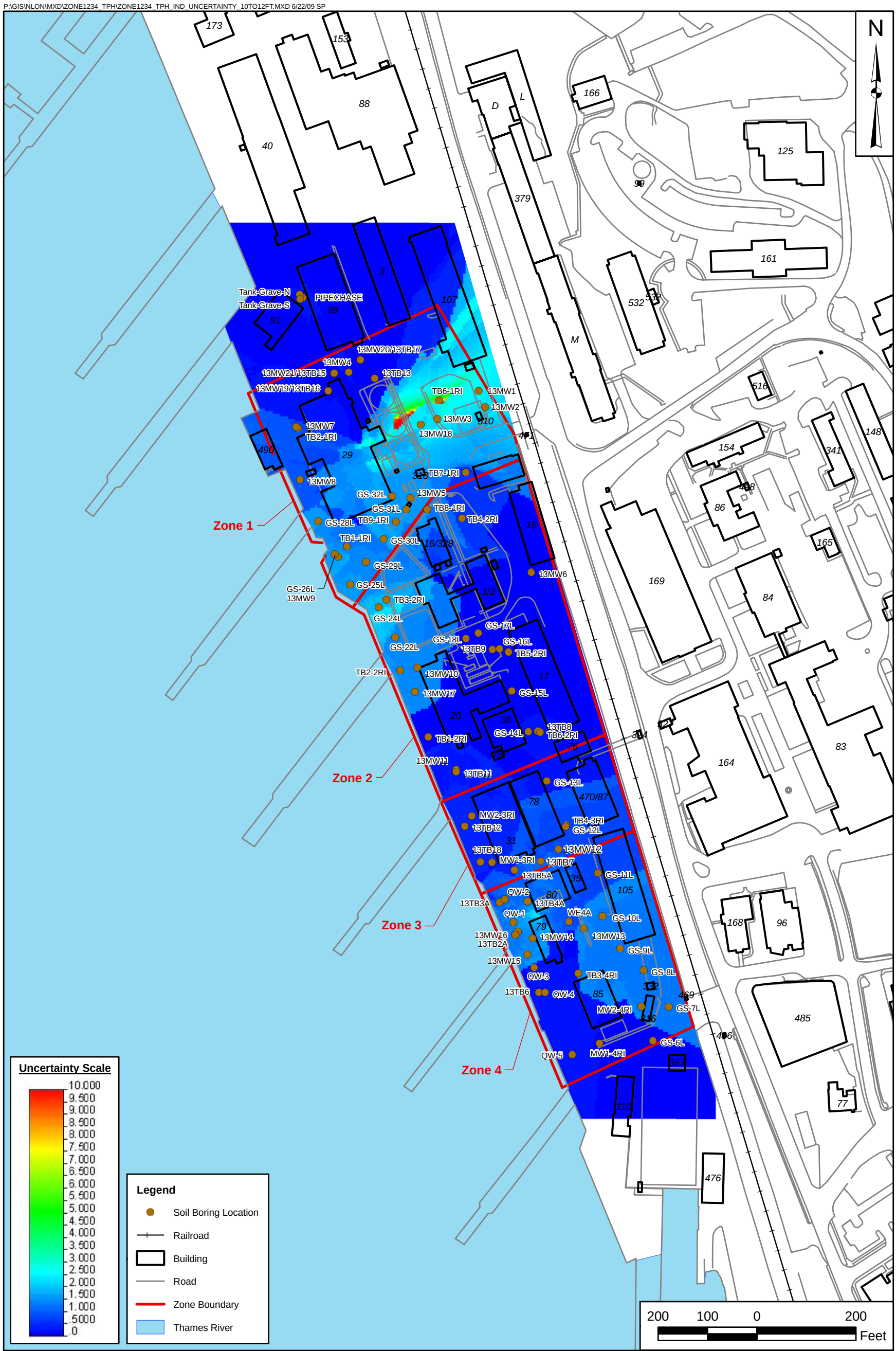
DRAWN BY S. PAXTON	DATE 4/02/09	Tetra Tech NUS, Inc.	CONTRACT NO. 0811
CHECKED BY N. BALSAMO	DATE 7/08/09	ESTIMATED TPH CONCENTRATIONS (KRIGING RESULTS) ELEVATION: -10 TO -8 FEET ZONES 1, 2, 3, AND 4 NAVAL SUBMARINE BASE - NEW LONDON GROTON, CONNECTICUT	APPROVED BY _ / DATE --
COST/SCHED-AREA			APPROVED BY -- / DATE --
SCALE AS NOTED			FIGURE NO. FIGURE B-34 / REV.

P:\GIS\NLON\MXD\ZONE1234_TPH\ZONE1234_TPH_2500MGKG_MVS.MXD 7/08/09 SP



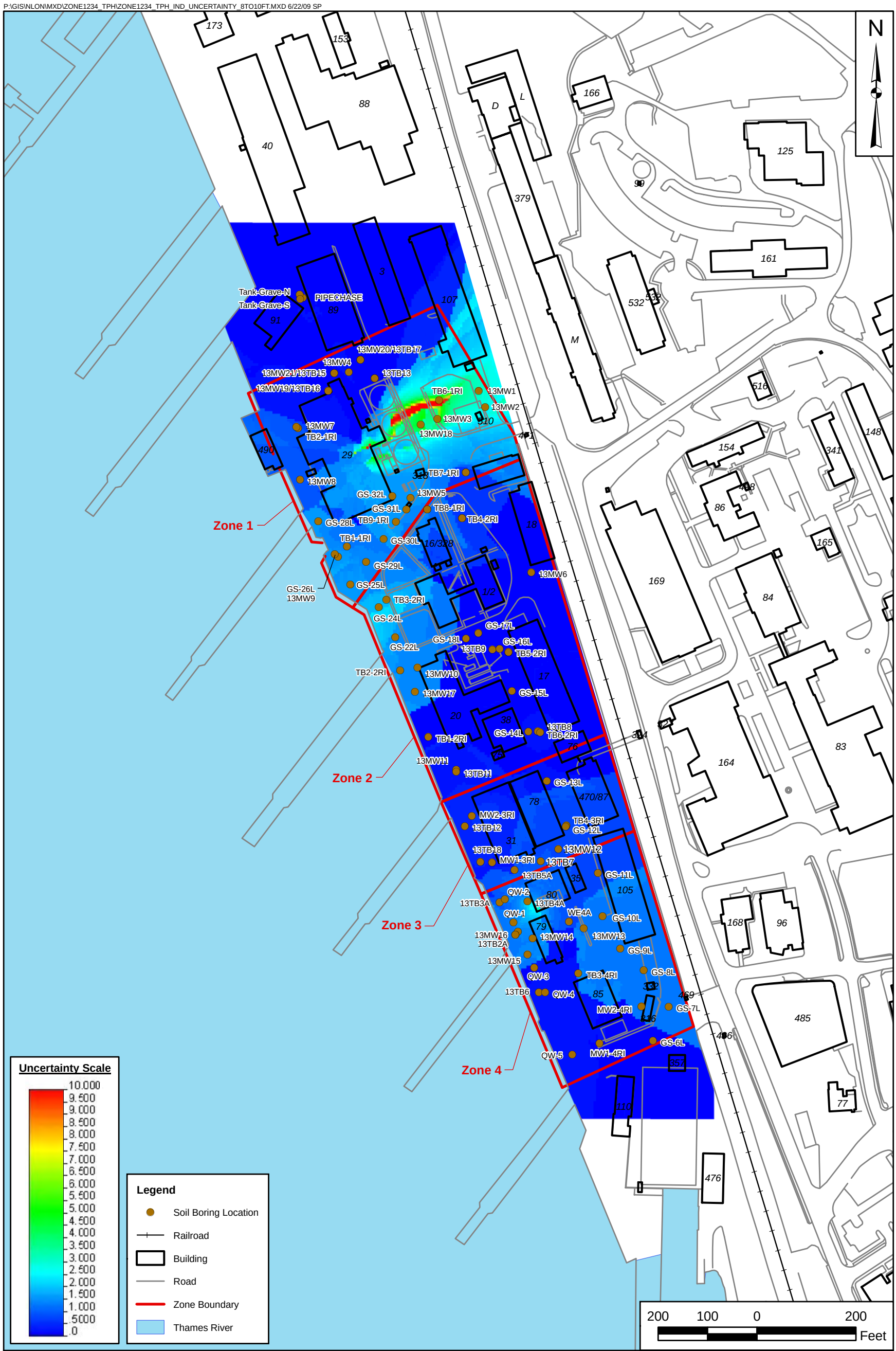
DRAWN BY	DATE	Tetra Tech NUS, Inc.	CONTRACT NO.
S. PAXTON	4/02/09		0811
CHECKED BY	DATE	ESTIMATED TPH CONCENTRATIONS (KRIGING RESULTS)	APPROVED BY / DATE
N. BALSAMO	7/08/09	ELEVATION: 10 TO 0 FEET	_ / --
COST/SCHED-AREA		ZONES 1, 2, 3, AND 4	APPROVED BY / DATE
		NAVAL SUBMARINE BASE - NEW LONDON	-- / --
SCALE		GROTON, CONNECTICUT	FIGURE NO. / REV.
AS NOTED			FIGURE B-35

P:\GIS\NLON\MXD\ZONE1234_TPH\ZONE1234_TPH_IND_UNCERTAINTY_10TO12FT.MXD 6/22/09 SP



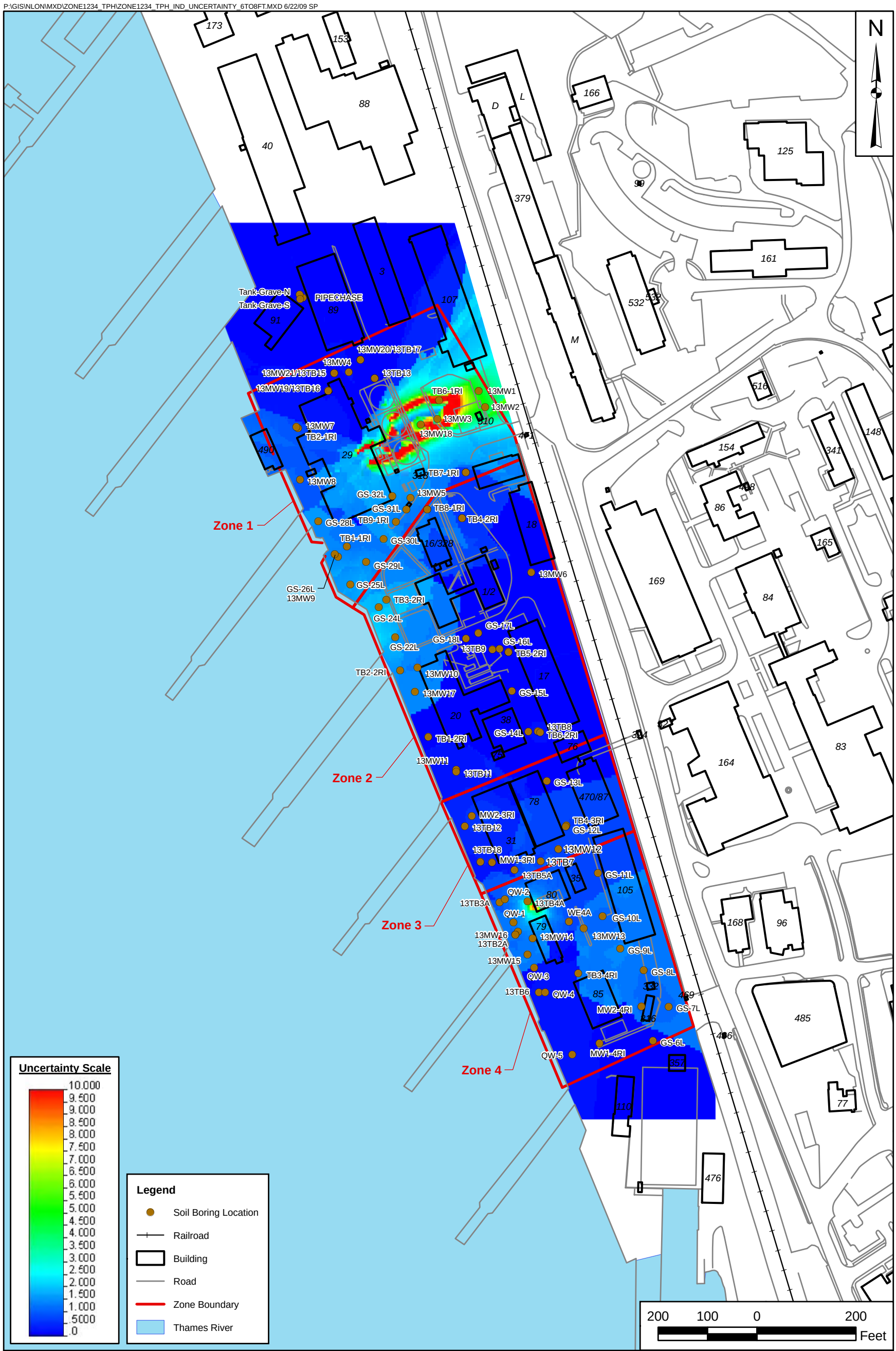
DRAWN BY	DATE	Tetra Tech NUS, Inc.	CONTRACT NO.
S. PAXTON	4/02/09		0811
CHECKED BY	DATE	TPH UNCERTAINTY FOR INDUSTRIAL CRITERIA	APPROVED BY / DATE
N. BALSAMO	6/22/09	ELEVATION: 10 TO 12 FEET	_ / --
COST/SCHED-AREA		ZONES 1, 2, 3, AND 4	APPROVED BY / DATE
		NAVAL SUBMARINE BASE - NEW LONDON	-- / --
SCALE		GROTON, CONNECTICUT	FIGURE NO. / REV.
AS NOTED			FIGURE B-36

P:\GIS\NLON\MXD\ZONE1234_TPH\ZONE1234_TPH_IND_UNCERTAINTY_8TO10FT.MXD 6/22/09 SP



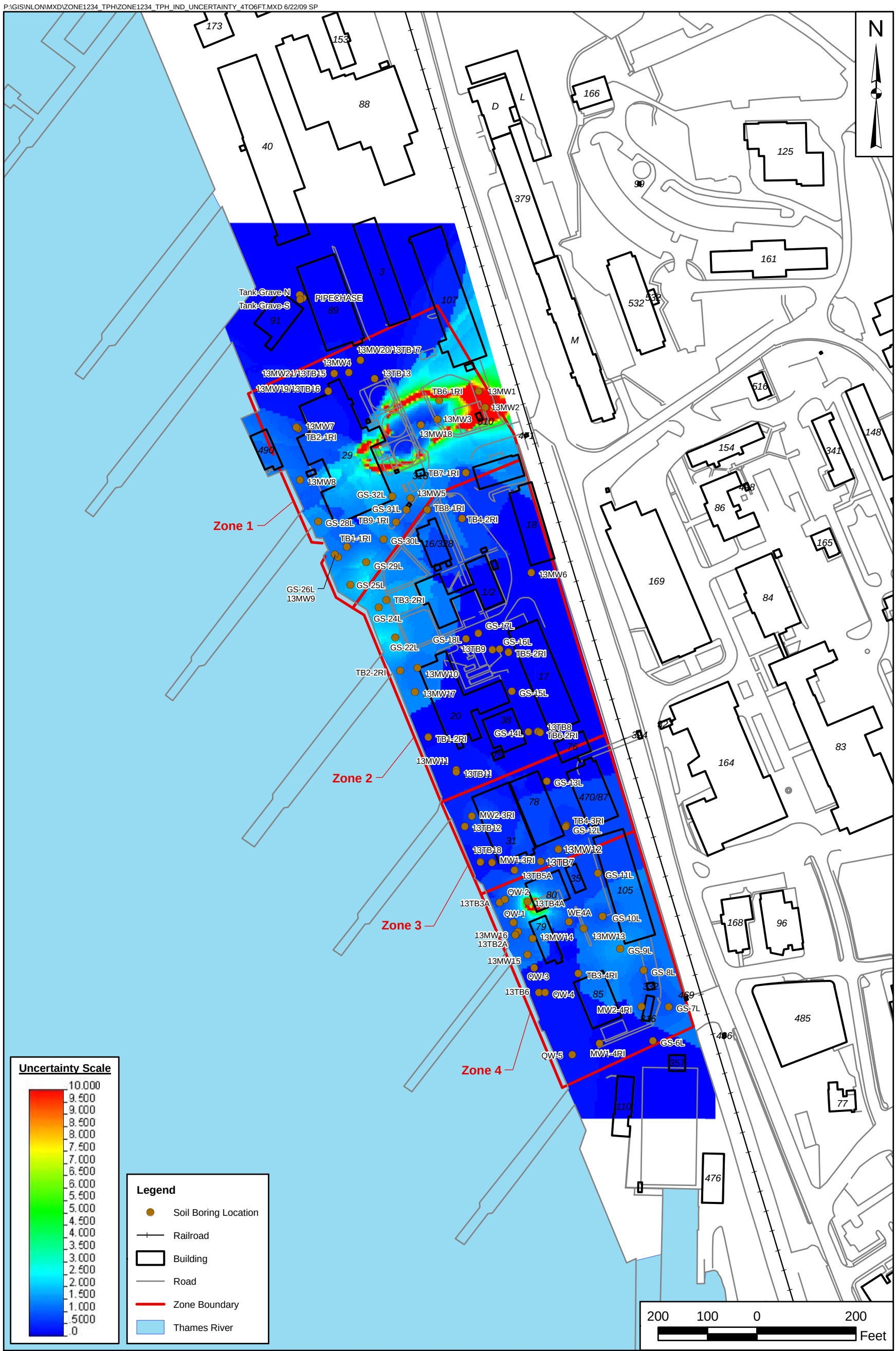
DRAWN BY	DATE	Tetra Tech NUS, Inc.	CONTRACT NO.
S. PAXTON	4/02/09		0811
CHECKED BY	DATE	TPH UNCERTAINTY FOR INDUSTRIAL CRITERIA	APPROVED BY / DATE
N. BALSAMO	6/22/09	ELEVATION: 8 TO 10 FEET	_ / --
COST/SCHED-AREA		ZONES 1, 2, 3, AND 4	APPROVED BY / DATE
		NAVAL SUBMARINE BASE - NEW LONDON	-- / --
SCALE		GROTON, CONNECTICUT	FIGURE NO. / REV.
AS NOTED			FIGURE B-37

P:\GIS\NLON\MXD\ZONE1234_TPH\ZONE1234_TPH_IND_UNCERTAINTY_6TO8FT.MXD 6/22/09 SP



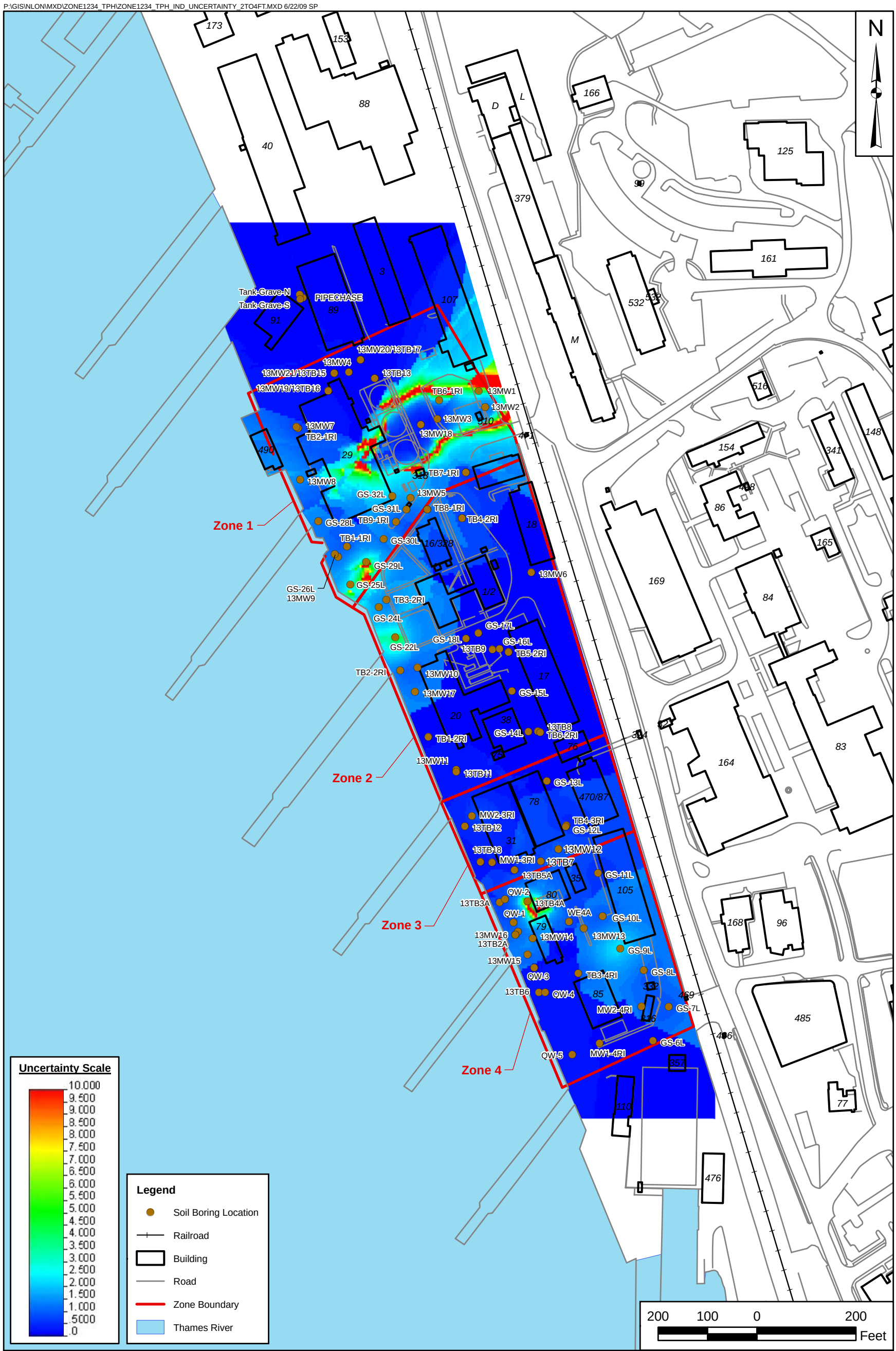
DRAWN BY	DATE	Tetra Tech NUS, Inc.	CONTRACT NO.
S. PAXTON	4/02/09		0811
CHECKED BY	DATE	TPH UNCERTAINTY FOR INDUSTRIAL CRITERIA	APPROVED BY / DATE
N. BALSAMO	6/22/09	ELEVATION: 6 TO 8 FEET	_ / --
COST/SCHED-AREA		ZONES 1, 2, 3, AND 4	APPROVED BY / DATE
		NAVAL SUBMARINE BASE - NEW LONDON	-- / --
SCALE		GROTON, CONNECTICUT	FIGURE NO. / REV.
AS NOTED			FIGURE B-38

P:\GIS\NLON\MXD\ZONE1234_TPH\ZONE1234_TPH_IND_UNCERTAINTY_4TO6FT.MXD 6/22/09 SP



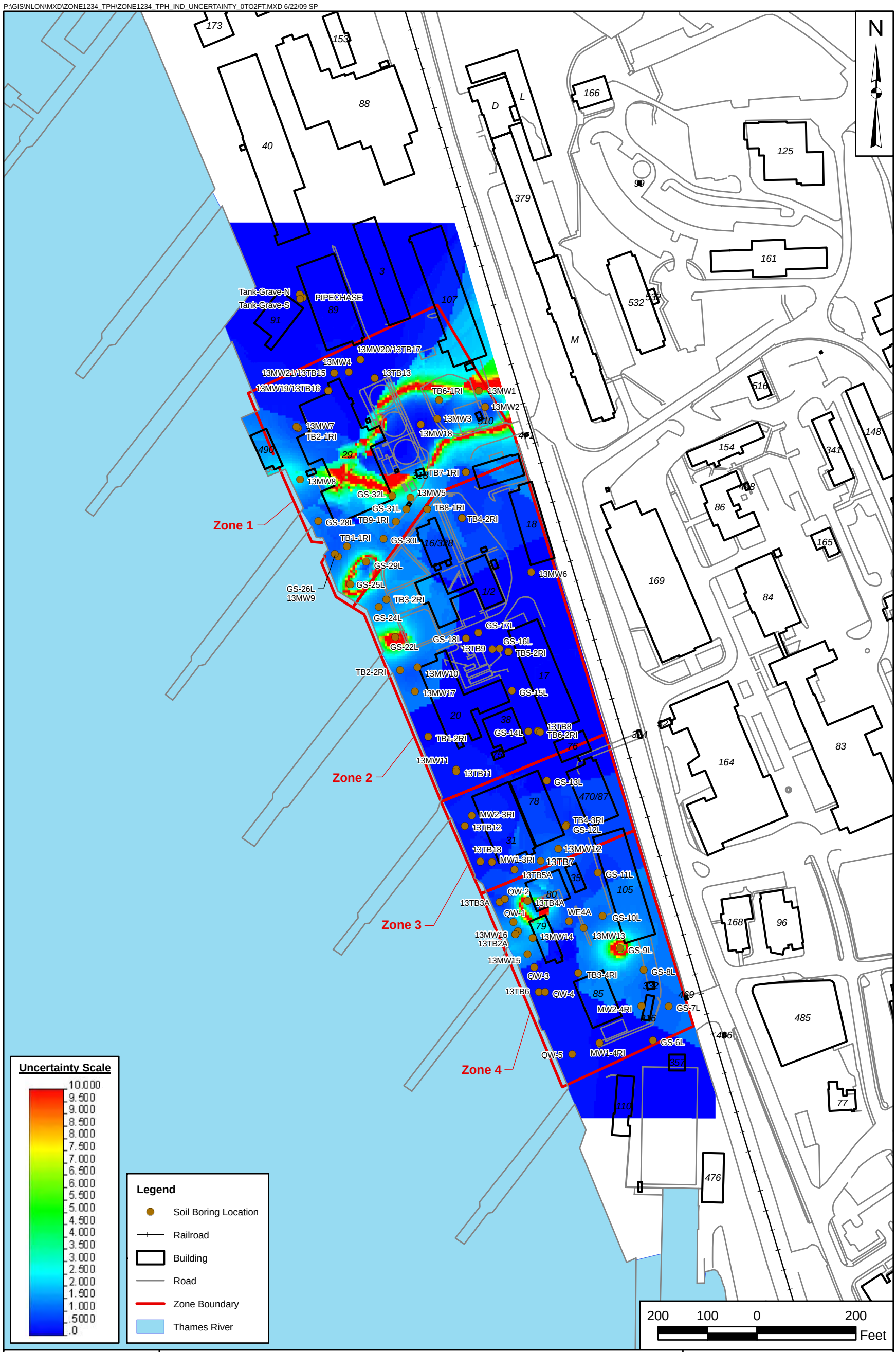
DRAWN BY	DATE	Tetra Tech NUS, Inc.	CONTRACT NO.
S. PAXTON	4/02/09		0811
CHECKED BY	DATE	TPH UNCERTAINTY FOR INDUSTRIAL CRITERIA	APPROVED BY / DATE
N. BALSAMO	6/22/09	ELEVATION: 4 TO 6 FEET	_ / --
COST/SCHED-AREA		ZONES 1, 2, 3, AND 4	APPROVED BY / DATE
		NAVAL SUBMARINE BASE - NEW LONDON	-- / --
SCALE		GROTON, CONNECTICUT	FIGURE NO. / REV.
AS NOTED			FIGURE B-39

P:\GIS\NLON\MXD\ZONE1234_TPH\ZONE1234_TPH_IND_UNCERTAINTY_2TO4FT.MXD 6/22/09 SP



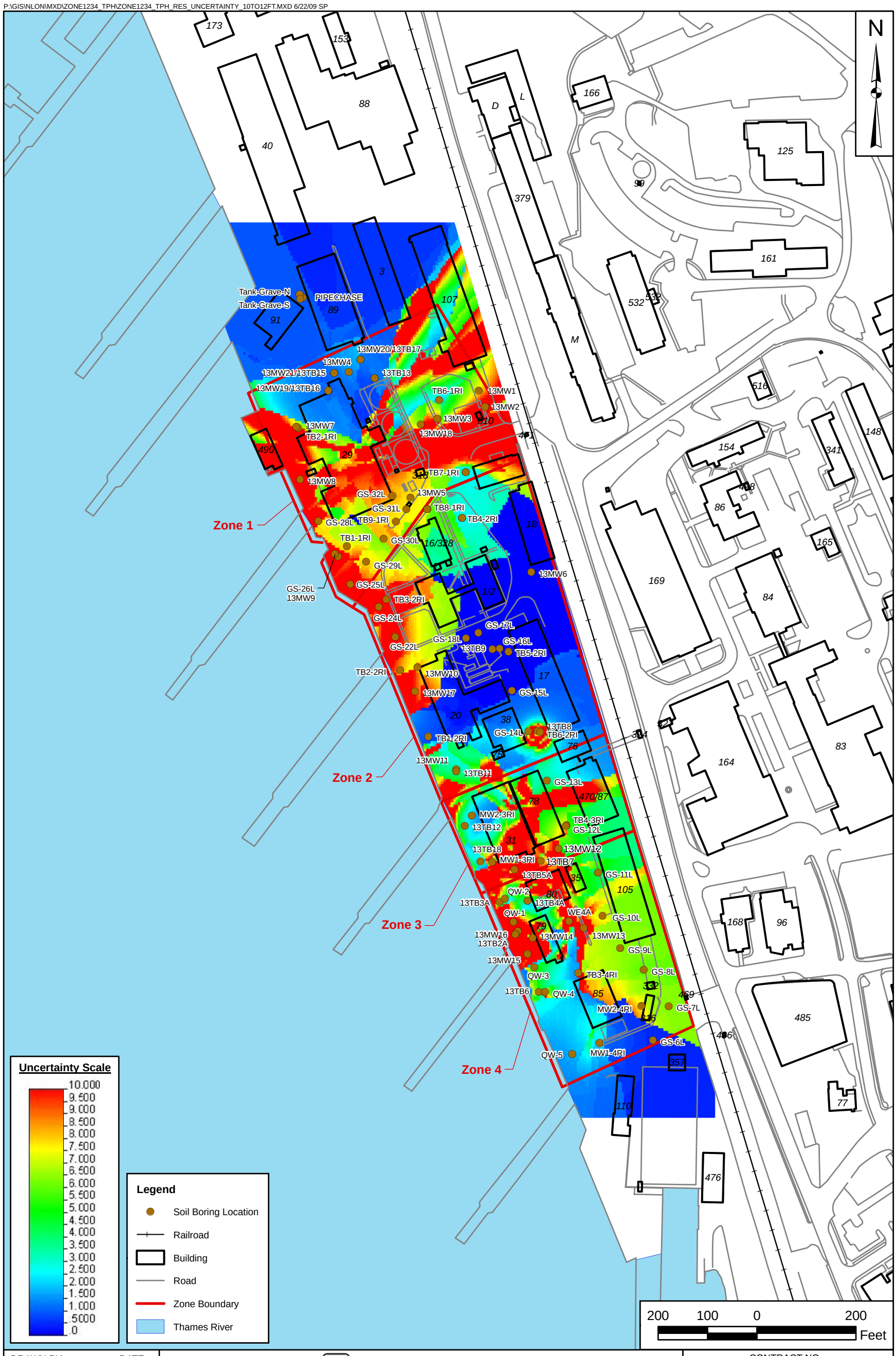
DRAWN BY	DATE	Tetra Tech NUS, Inc.	CONTRACT NO.
S. PAXTON	4/02/09		0811
CHECKED BY	DATE	TPH UNCERTAINTY FOR INDUSTRIAL CRITERIA	APPROVED BY / DATE
N. BALSAMO	6/22/09	ELEVATION: 2 TO 4 FEET	_ / --
COST/SCHED-AREA		ZONES 1, 2, 3, AND 4	APPROVED BY / DATE
		NAVAL SUBMARINE BASE - NEW LONDON	-- / --
SCALE		GROTON, CONNECTICUT	FIGURE NO. / REV.
AS NOTED			FIGURE B-40

P:\GIS\NLON\MXD\ZONE1234_TPH\ZONE1234_TPH_IND_UNCERTAINTY_0TO2FT.MXD 6/22/09 SP



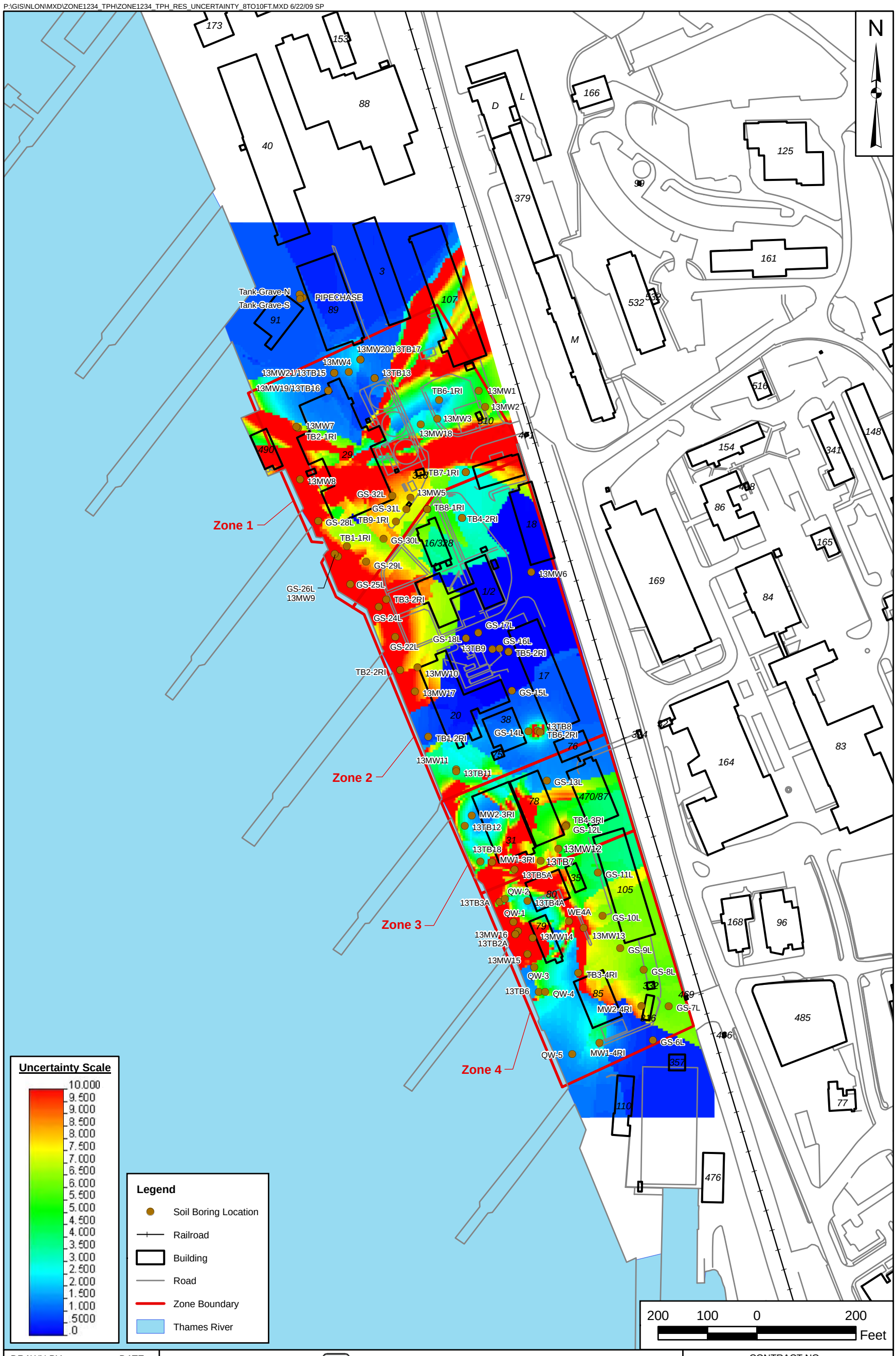
DRAWN BY	DATE	Tetra Tech NUS, Inc.	CONTRACT NO.
S. PAXTON	4/02/09		0811
CHECKED BY	DATE	TPH UNCERTAINTY FOR INDUSTRIAL CRITERIA	APPROVED BY / DATE
N. BALSAMO	6/22/09	ELEVATION: 0 TO 2 FEET	_ / --
COST/SCHED-AREA		ZONES 1, 2, 3, AND 4	APPROVED BY / DATE
		NAVAL SUBMARINE BASE - NEW LONDON	-- / --
SCALE		GROTON, CONNECTICUT	FIGURE NO. / REV.
AS NOTED			FIGURE B-41

P:\GIS\NLON\MXD\ZONE1234_TPH\ZONE1234_TPH_RES_UNCERTAINTY_10TO12FT.MXD 6/22/09 SP



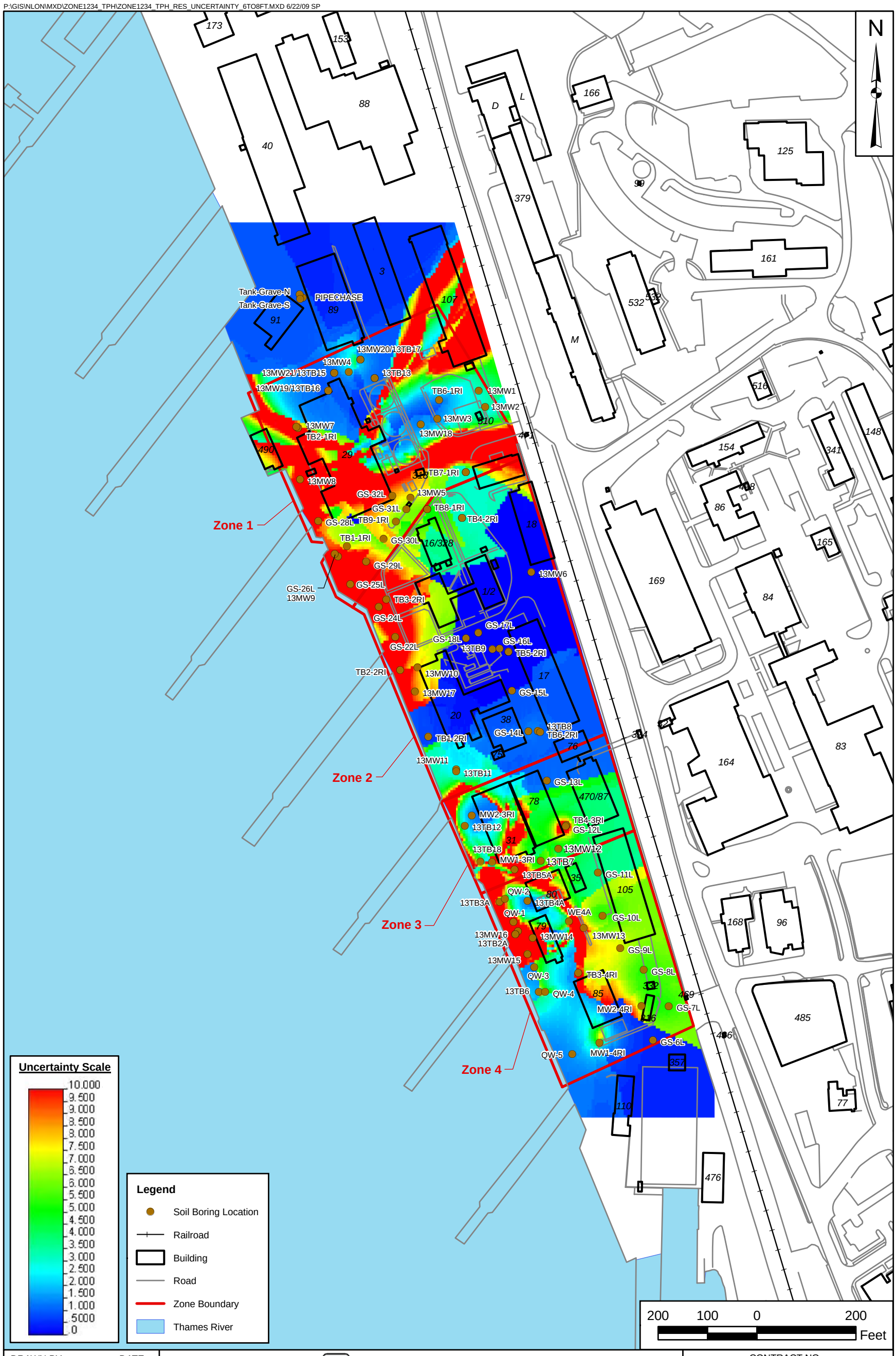
DRAWN BY S. PAXTON	DATE 4/02/09	Tetra Tech NUS, Inc.	CONTRACT NO. 0811
CHECKED BY N. BALSAMO	DATE 6/22/09	TPH UNCERTAINTY FOR RESIDENTIAL CRITERIA ELEVATION: 10 TO 12 FEET ZONES 1, 2, 3, AND 4 NAVAL SUBMARINE BASE - NEW LONDON GROTON, CONNECTICUT	APPROVED BY _ / DATE --
COST/SCHED-AREA			APPROVED BY -- / DATE --
SCALE AS NOTED			FIGURE NO. FIGURE B-42 / REV.

P:\GIS\NLON\MXD\ZONE1234_TPH\ZONE1234_TPH_RES_UNCERTAINTY_8TO10FT.MXD 6/22/09 SP



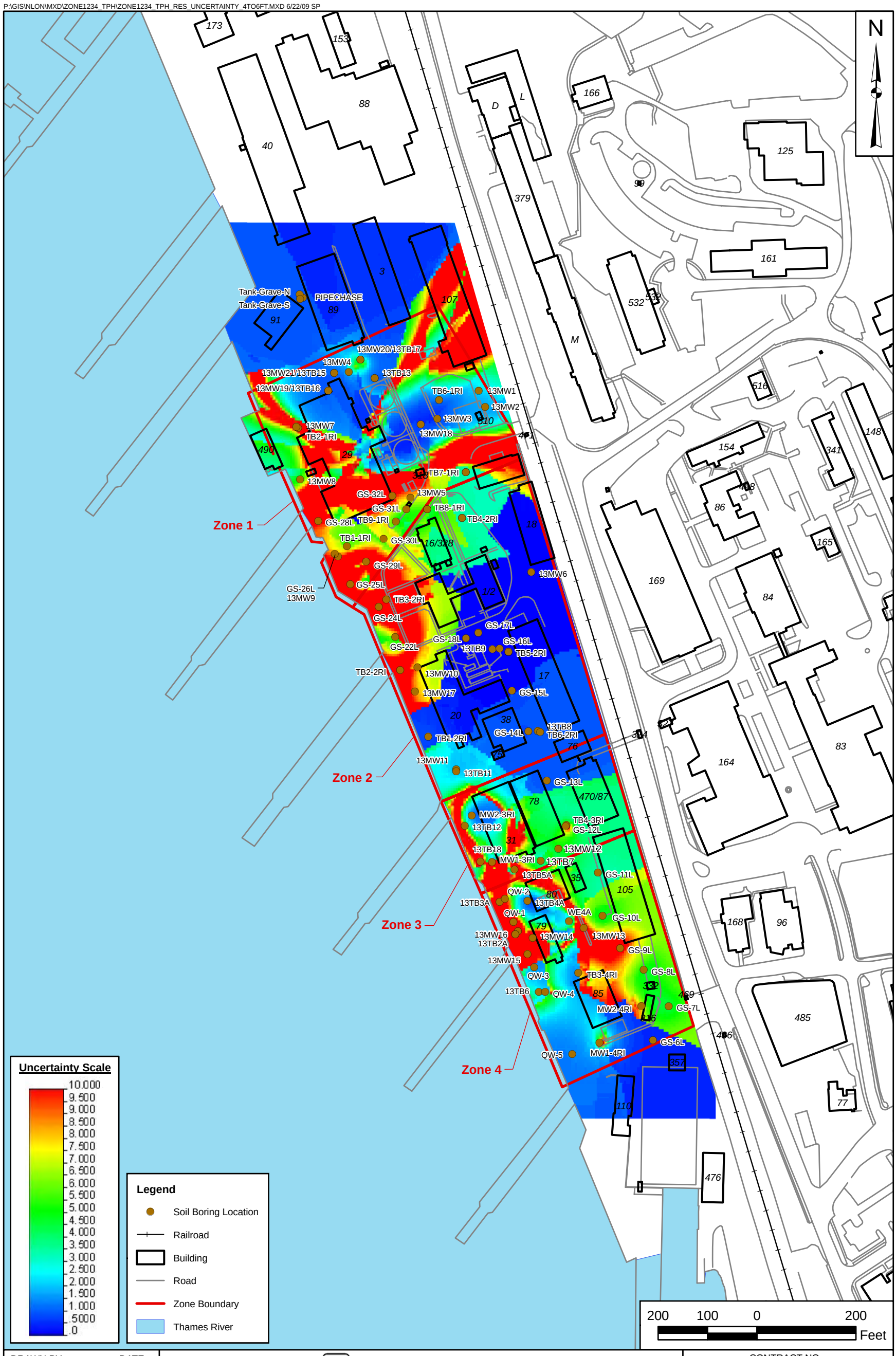
DRAWN BY	DATE	Tetra Tech NUS, Inc.	CONTRACT NO.
S. PAXTON	4/02/09		0811
CHECKED BY	DATE	TPH UNCERTAINTY FOR RESIDENTIAL CRITERIA ELEVATION: 8 TO 10 FEET ZONES 1, 2, 3, AND 4 NAVAL SUBMARINE BASE - NEW LONDON GROTON, CONNECTICUT	APPROVED BY / DATE
N. BALSAMO	6/22/09		_ / --
COST/SCHED-AREA			APPROVED BY / DATE
			-- / --
SCALE			FIGURE NO. / REV.
AS NOTED			FIGURE B-43

P:\GIS\NLON\MXD\ZONE1234_TPH\ZONE1234_TPH_RES_UNCERTAINTY_6TO8FT.MXD 6/22/09 SP



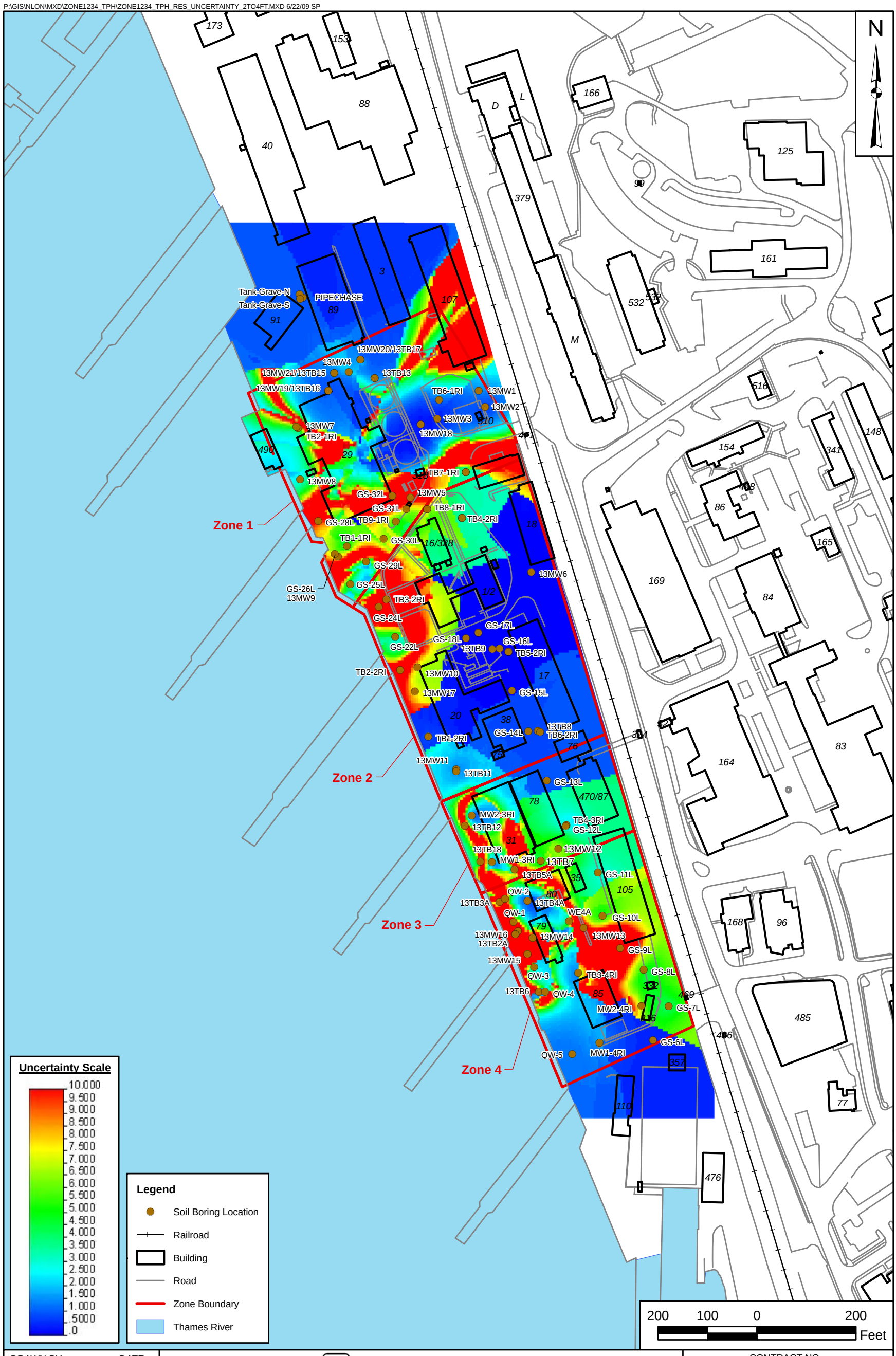
DRAWN BY S. PAXTON	DATE 4/02/09	Tetra Tech NUS, Inc.	CONTRACT NO. 0811
CHECKED BY N. BALSAMO	DATE 6/22/09	TPH UNCERTAINTY FOR RESIDENTIAL CRITERIA ELEVATION: 6 TO 8 FEET ZONES 1, 2, 3, AND 4 NAVAL SUBMARINE BASE - NEW LONDON GROTON, CONNECTICUT	APPROVED BY _ / DATE --
COST/SCHED-AREA			APPROVED BY -- / DATE --
SCALE AS NOTED			FIGURE NO. FIGURE B-44 / REV.

P:\GIS\NLON\MXD\ZONE1234_TPH\ZONE1234_TPH_RES_UNCERTAINTY_4TO6FT.MXD 6/22/09 SP



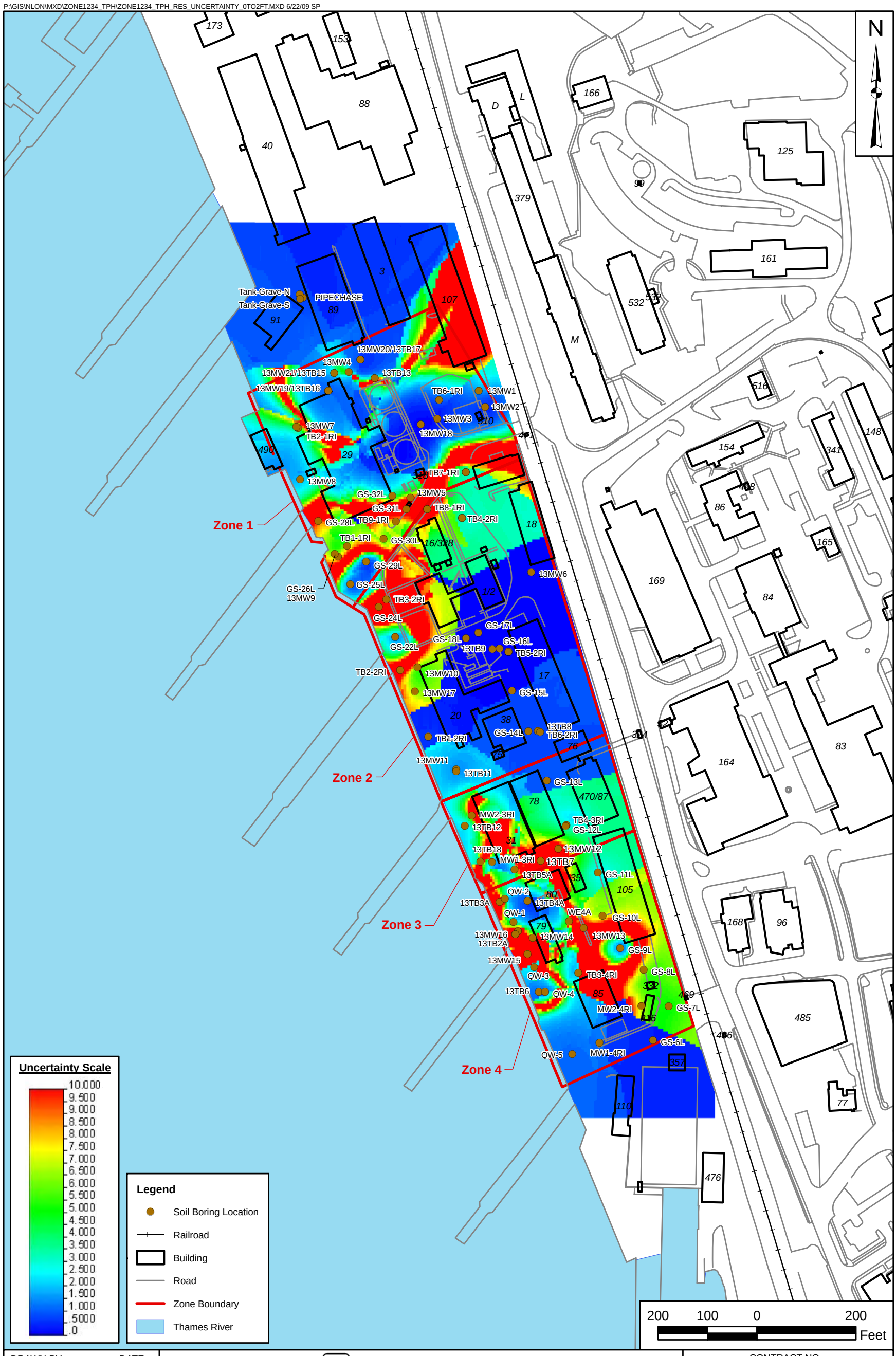
DRAWN BY S. PAXTON	DATE 4/02/09	Tetra Tech NUS, Inc.	CONTRACT NO. 0811
CHECKED BY N. BALSAMO	DATE 6/22/09	TPH UNCERTAINTY FOR RESIDENTIAL CRITERIA ELEVATION: 4 TO 6 FEET ZONES 1, 2, 3, AND 4 NAVAL SUBMARINE BASE - NEW LONDON GROTON, CONNECTICUT	APPROVED BY _ / DATE --
COST/SCHED-AREA			APPROVED BY -- / DATE --
SCALE AS NOTED			FIGURE NO. FIGURE B-45 / REV.

P:\GIS\NLON\MXD\ZONE1234_TPH\ZONE1234_TPH_RES_UNCERTAINTY_2TO4FT.MXD 6/22/09 SP



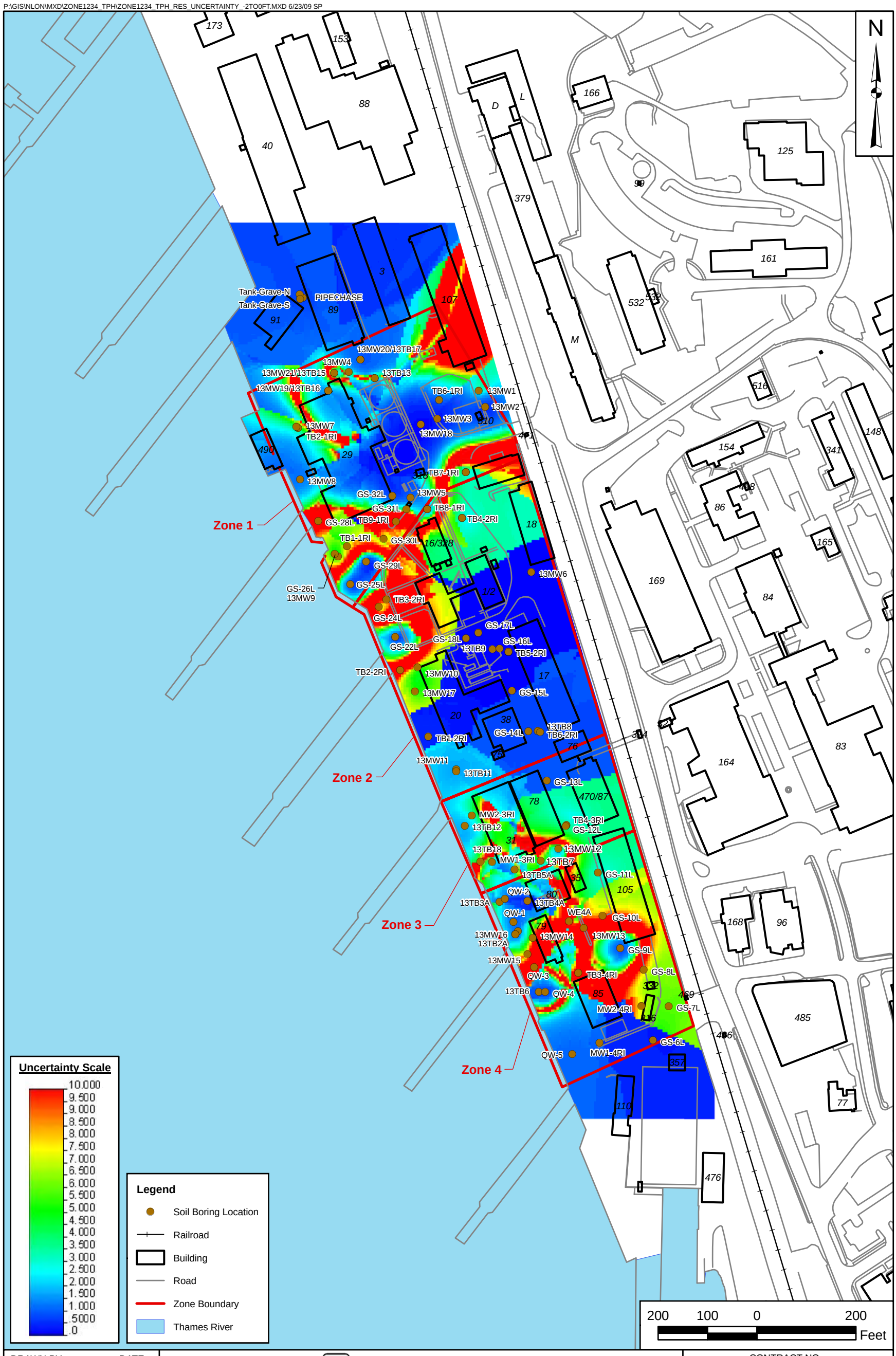
DRAWN BY S. PAXTON	DATE 4/02/09	Tetra Tech NUS, Inc.	CONTRACT NO. 0811
CHECKED BY N. BALSAMO	DATE 6/22/09	TPH UNCERTAINTY FOR RESIDENTIAL CRITERIA ELEVATION: 2 TO 4 FEET ZONES 1, 2, 3, AND 4 NAVAL SUBMARINE BASE - NEW LONDON GROTON, CONNECTICUT	APPROVED BY _ / DATE --
COST/SCHED-AREA			APPROVED BY -- / DATE --
SCALE AS NOTED			FIGURE NO. FIGURE B-46 / REV.

P:\GIS\NLON\MXD\ZONE1234_TPH\ZONE1234_TPH_RES_UNCERTAINTY_0TO2FT.MXD 6/22/09 SP



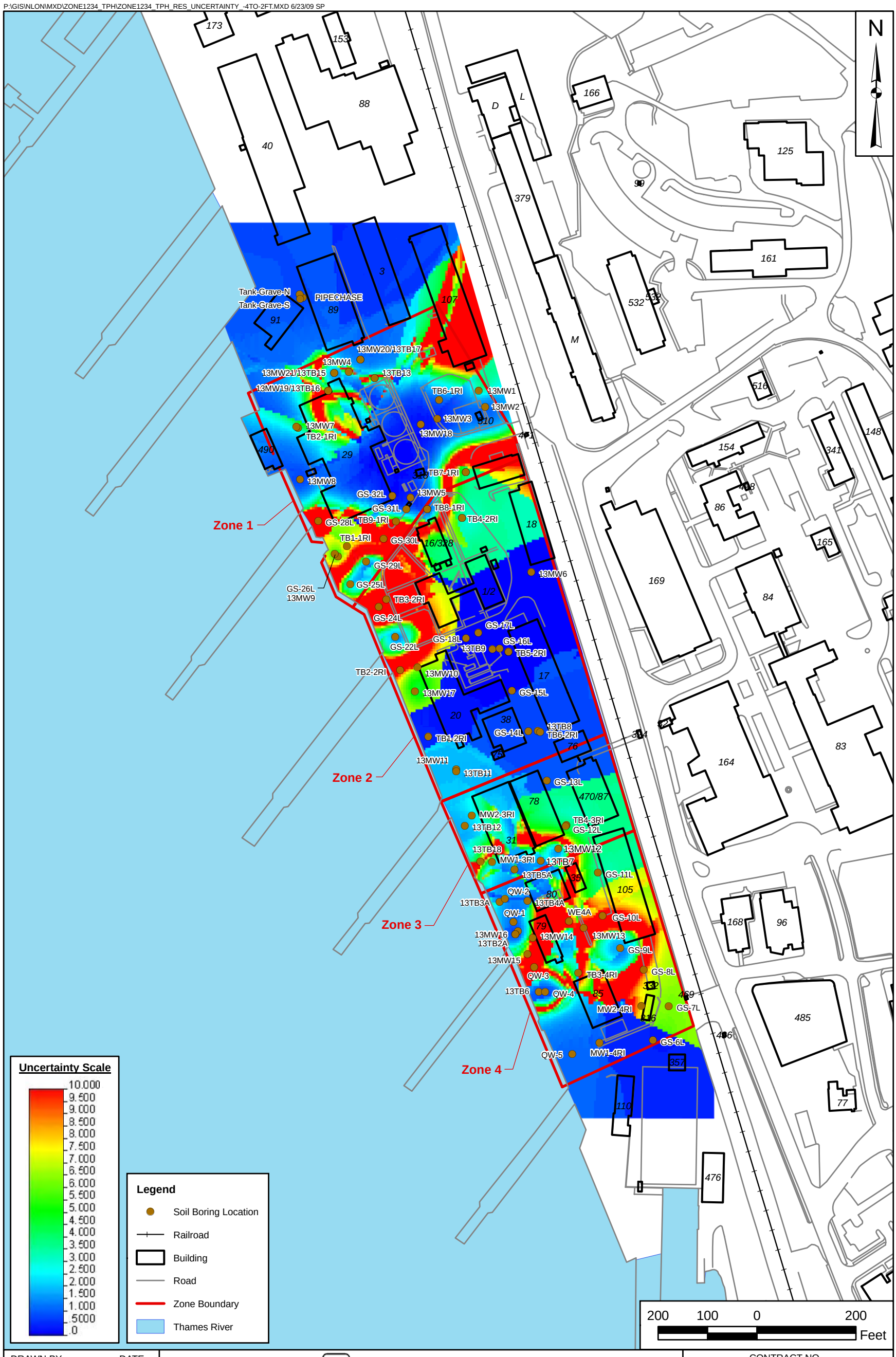
DRAWN BY S. PAXTON	DATE 4/02/09	Tetra Tech NUS, Inc.	CONTRACT NO. 0811
CHECKED BY N. BALSAMO	DATE 6/22/09	TPH UNCERTAINTY FOR RESIDENTIAL CRITERIA ELEVATION: 0 TO 2 FEET ZONES 1, 2, 3, AND 4 NAVAL SUBMARINE BASE - NEW LONDON GROTON, CONNECTICUT	APPROVED BY _ / DATE --
COST/SCHED-AREA			APPROVED BY -- / DATE --
SCALE AS NOTED			FIGURE NO. FIGURE B-47 / REV.

P:\GIS\NLON\MXD\ZONE1234_TPH\ZONE1234_TPH_RES_UNCERTAINTY_-2TO0FT.MXD 6/23/09 SP



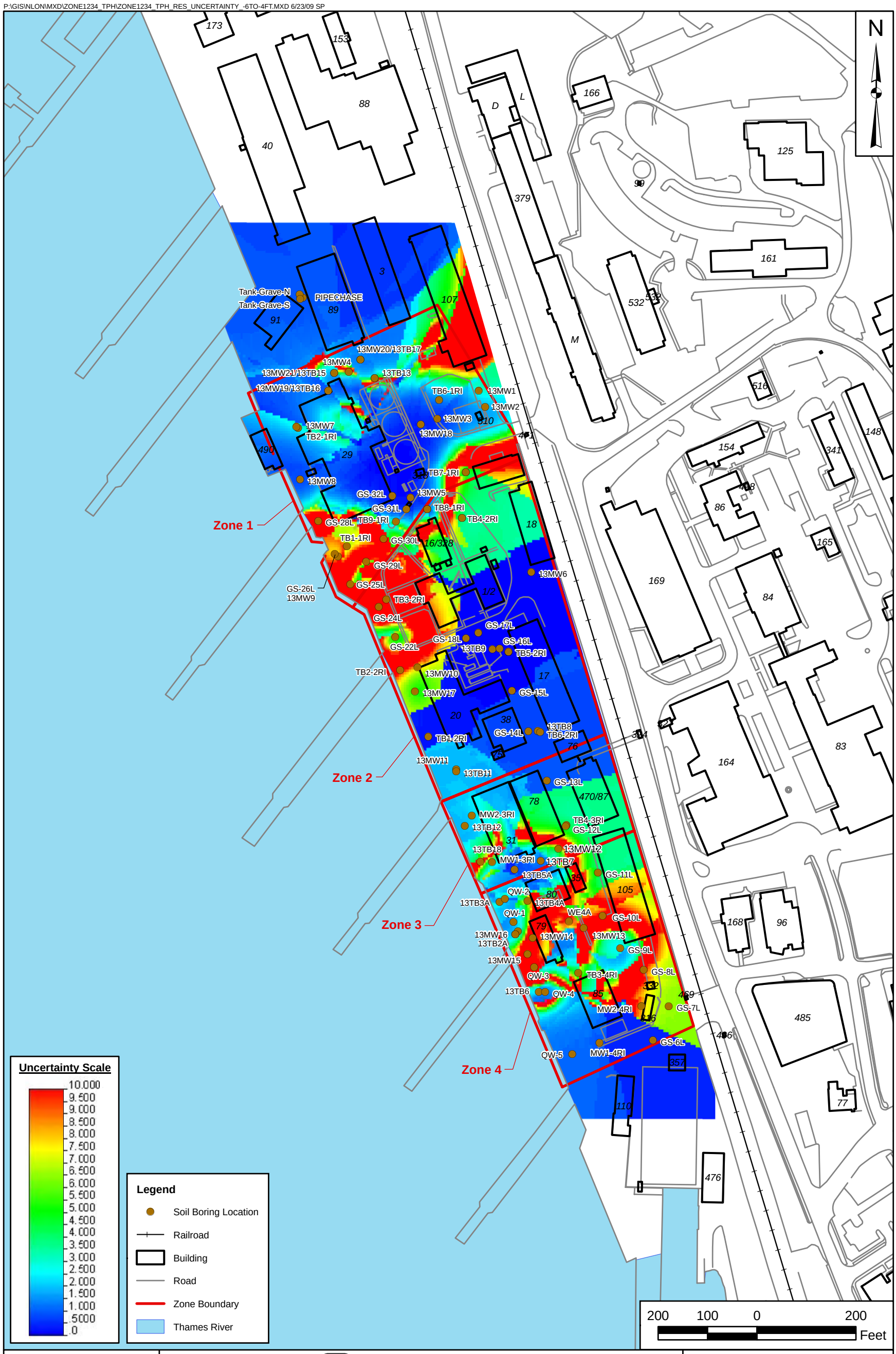
DRAWN BY S. PAXTON	DATE 4/02/09	Tetra Tech NUS, Inc.	CONTRACT NO. 0811
CHECKED BY N. BALSAMO	DATE 6/23/09	TPH UNCERTAINTY FOR RESIDENTIAL CRITERIA ELEVATION: -2 TO 0 FEET ZONES 1, 2, 3, AND 4 NAVAL SUBMARINE BASE - NEW LONDON GROTON, CONNECTICUT	APPROVED BY _ / DATE --
COST/SCHED-AREA			APPROVED BY -- / DATE --
SCALE AS NOTED			FIGURE NO. FIGURE B-48 / REV.

P:\GIS\NLON\MXD\ZONE1234_TPH\ZONE1234_TPH_RES_UNCERTAINTY_-4TO-2FT.MXD 6/23/09 SP



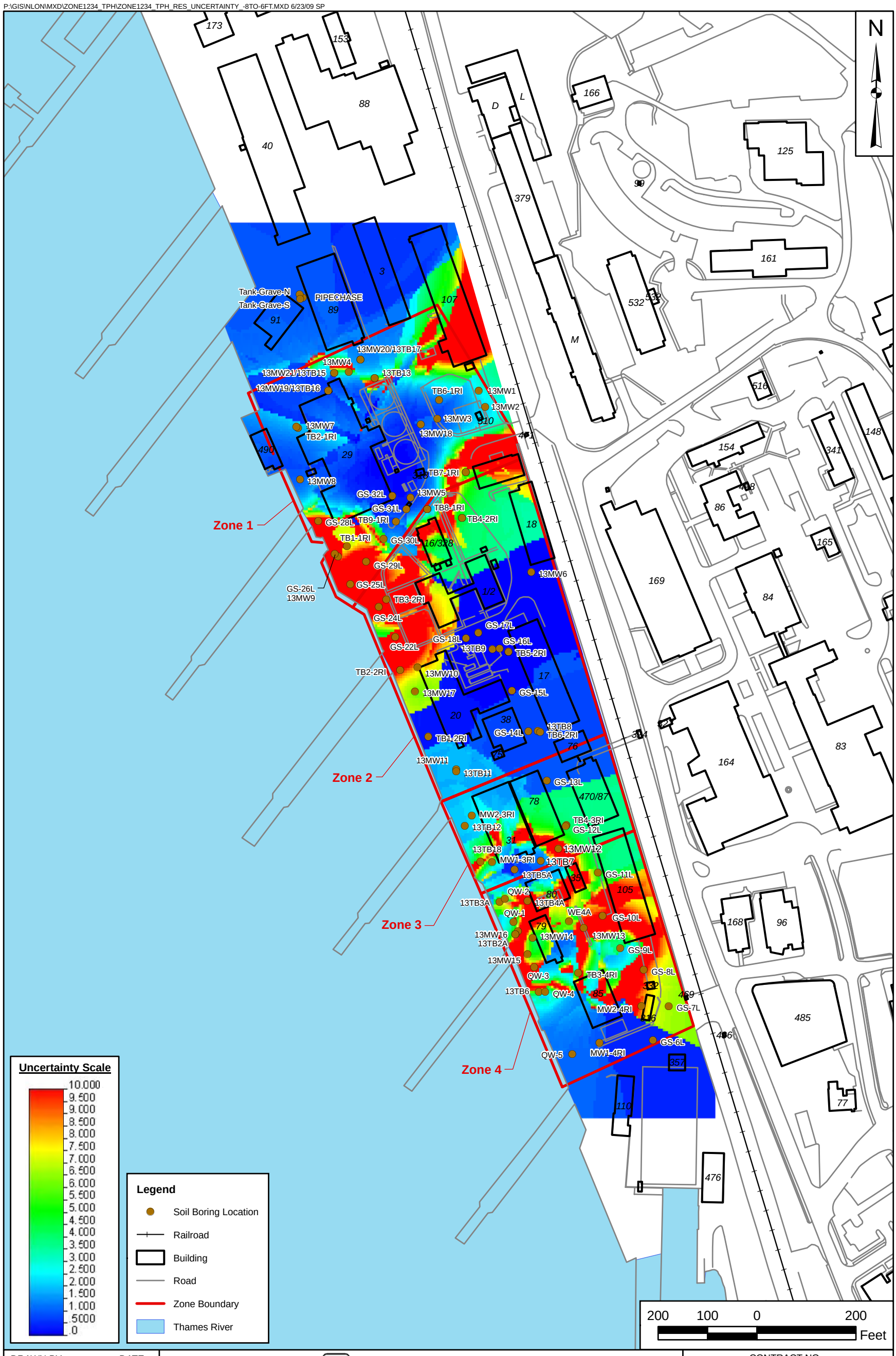
DRAWN BY	DATE	Tetra Tech NUS, Inc.	CONTRACT NO.
S. PAXTON	4/02/09		0811
CHECKED BY	DATE	TPH UNCERTAINTY FOR RESIDENTIAL CRITERIA ELEVATION: -4 TO -2 FEET ZONES 1, 2, 3, AND 4 NAVAL SUBMARINE BASE - NEW LONDON GROTON, CONNECTICUT	APPROVED BY / DATE
N. BALSAMO	6/23/09		_ / --
COST/SCHED-AREA			APPROVED BY / DATE
			-- / --
SCALE			FIGURE NO. / REV.
AS NOTED			FIGURE B-49

P:\GIS\NLON\MXD\ZONE1234_TPH\ZONE1234_TPH_RES_UNCERTAINTY_-6TO-4FT.MXD 6/23/09 SP



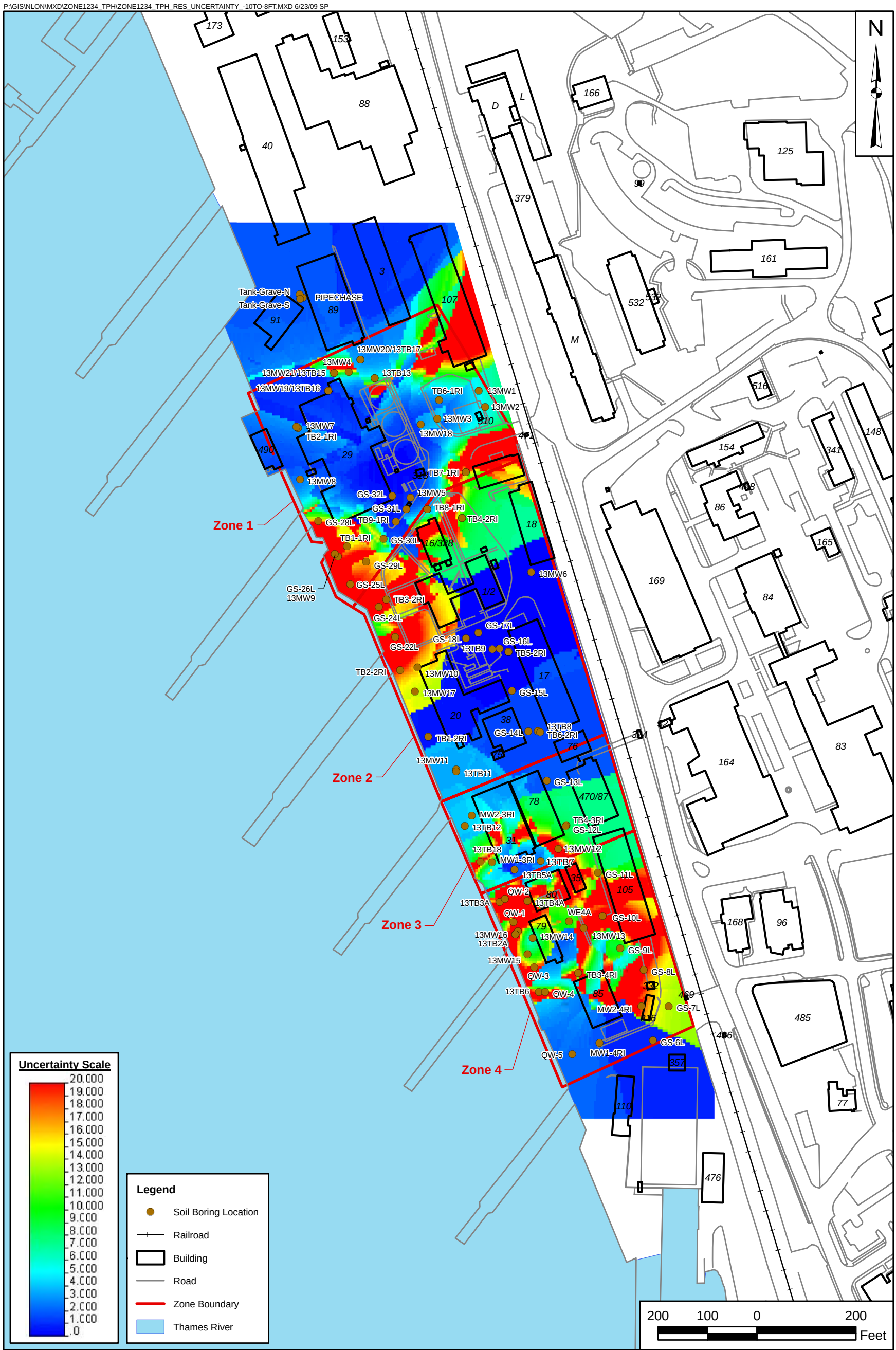
DRAWN BY	DATE	Tetra Tech NUS, Inc.	CONTRACT NO.
S. PAXTON	4/02/09		0811
CHECKED BY	DATE	TPH UNCERTAINTY FOR RESIDENTIAL CRITERIA ELEVATION: -6 TO -4 FEET ZONES 1, 2, 3, AND 4 NAVAL SUBMARINE BASE - NEW LONDON GROTON, CONNECTICUT	APPROVED BY / DATE
N. BALSAMO	6/23/09		_ / --
COST/SCHED-AREA			APPROVED BY / DATE
			-- / --
SCALE			FIGURE NO. / REV.
AS NOTED			FIGURE B-50

P:\GIS\NLON\MXD\ZONE1234_TPH\ZONE1234_TPH_RES_UNCERTAINTY_-8TO-6FT.MXD 6/23/09 SP



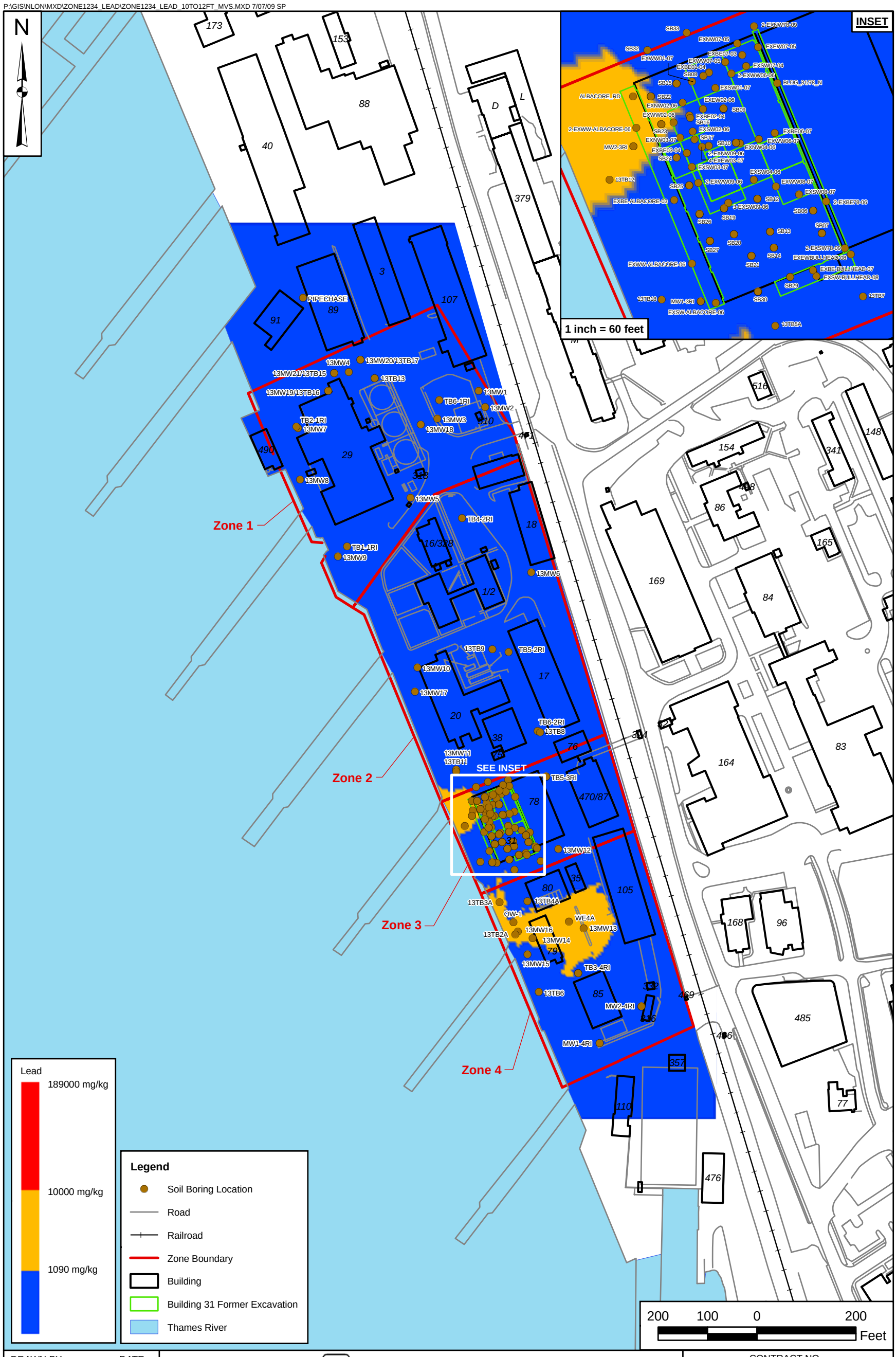
DRAWN BY S. PAXTON	DATE 4/02/09	Tetra Tech NUS, Inc.	CONTRACT NO. 0811
CHECKED BY N. BALSAMO	DATE 6/23/09	TPH UNCERTAINTY FOR RESIDENTIAL CRITERIA ELEVATION: -8 TO -6 FEET ZONES 1, 2, 3, AND 4 NAVAL SUBMARINE BASE - NEW LONDON GROTON, CONNECTICUT	APPROVED BY _ / DATE --
COST/SCHED-AREA			APPROVED BY -- / DATE --
SCALE AS NOTED			FIGURE NO. FIGURE B-51 / REV.

P:\GIS\NLON\MXD\ZONE1234_TPH\ZONE1234_TPH_RES_UNCERTAINTY_-10TO-8FT.MXD 6/23/09 SP



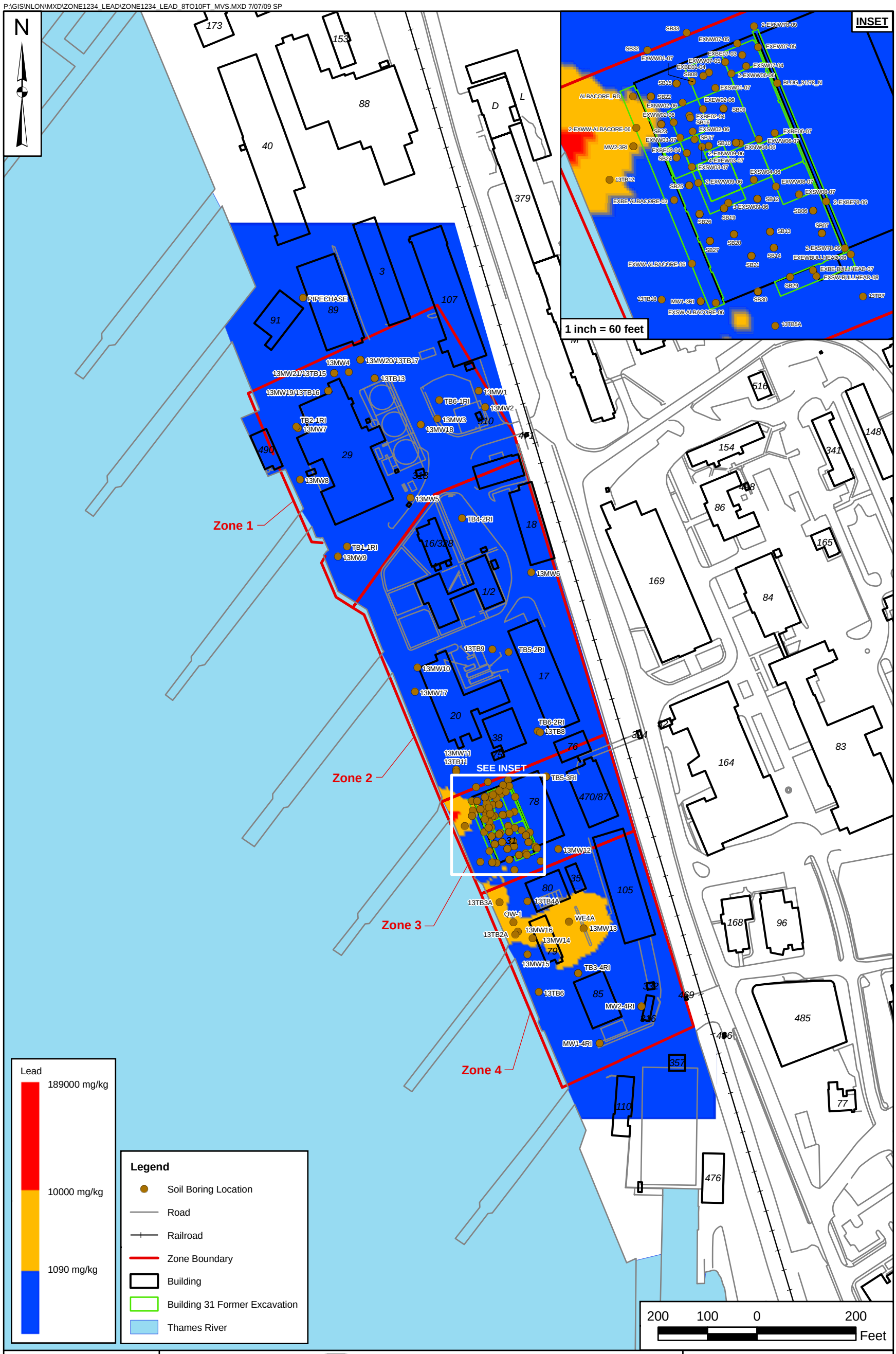
DRAWN BY S. PAXTON	DATE 4/02/09	Tetra Tech NUS, Inc.	CONTRACT NO. 0811
CHECKED BY N. BALSAMO	DATE 6/23/09	TPH UNCERTAINTY FOR RESIDENTIAL CRITERIA ELEVATION: -10 TO -8 FEET ZONES 1, 2, 3, AND 4 NAVAL SUBMARINE BASE - NEW LONDON GROTON, CONNECTICUT	APPROVED BY _ / DATE --
COST/SCHED-AREA			APPROVED BY -- / DATE --
SCALE AS NOTED			FIGURE NO. FIGURE B-52 / REV.

P:\GIS\NLON\MXD\ZONE1234_LEAD\ZONE1234_LEAD_10TO12FT_MVS.MXD 7/07/09 SP



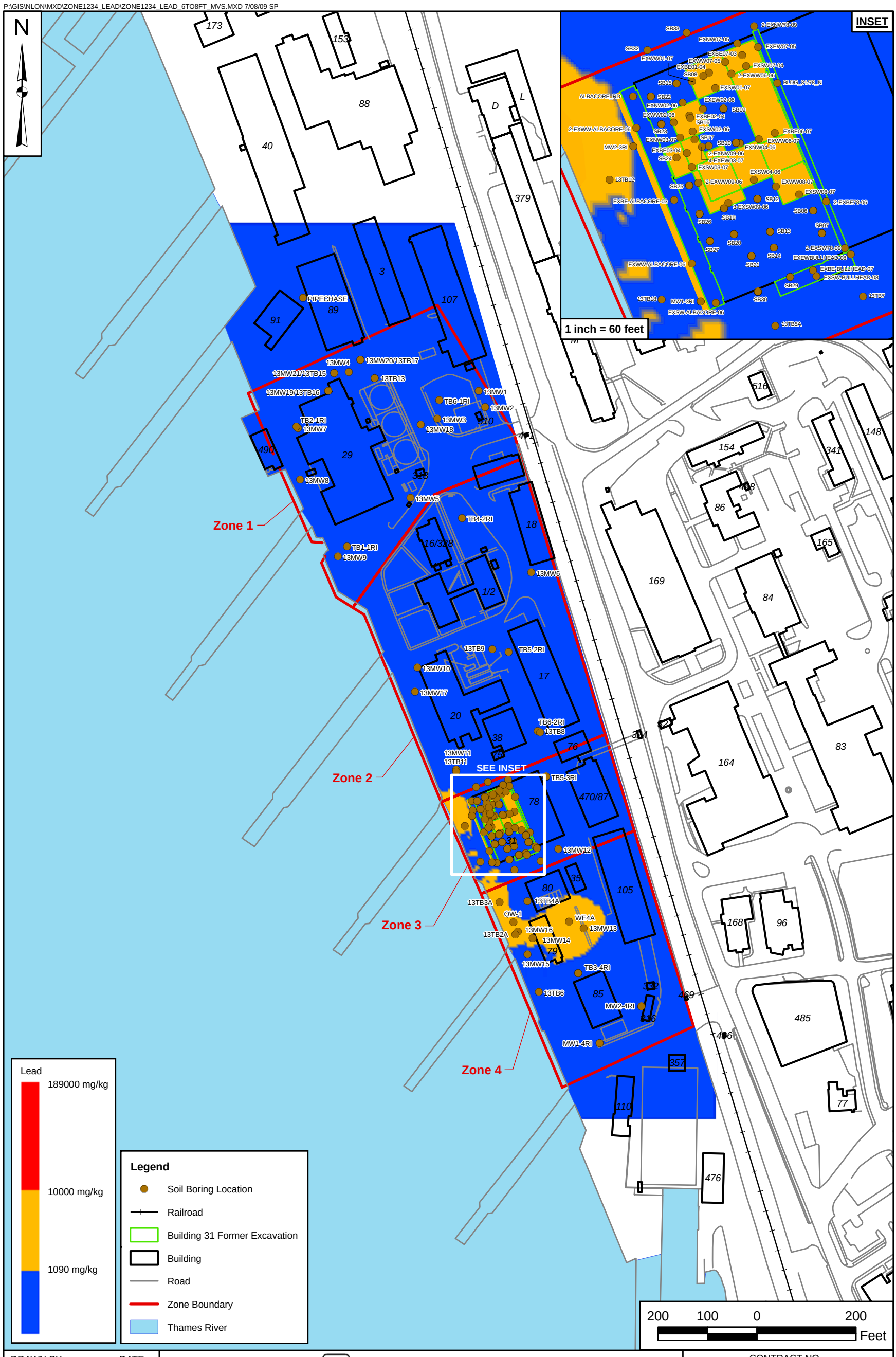
DRAWN BY	DATE	Tetra Tech NUS, Inc.	CONTRACT NO.
S. PAXTON	4/02/09		0811
CHECKED BY	DATE	ESTIMATED LEAD CONCENTRATIONS (KRIGING RESULTS)	APPROVED BY / DATE
N. BALSAMO	7/07/09	ELEVATION: 10 TO 12 FEET	_ / --
COST/SCHED-AREA		ZONES 1, 2, 3, AND 4	APPROVED BY / DATE
		NAVAL SUBMARINE BASE - NEW LONDON	-- / --
SCALE		GROTON, CONNECTICUT	FIGURE NO. / REV.
AS NOTED			FIGURE B-53

P:\GIS\NLON\MXD\ZONE1234_LEAD\ZONE1234_LEAD_8TO10FT_MVS.MXD 7/07/09 SP



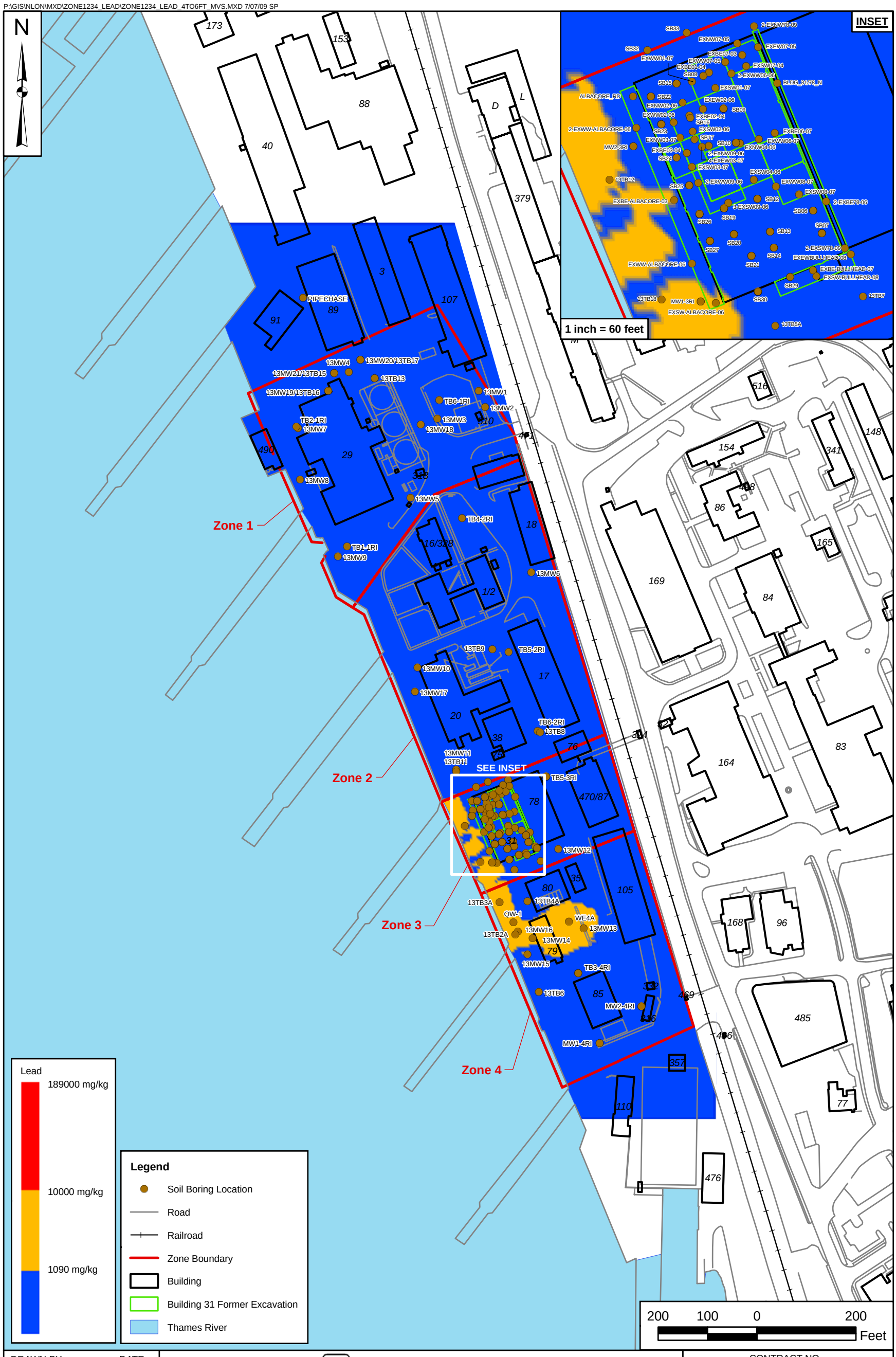
DRAWN BY	DATE	Tetra Tech NUS, Inc.	CONTRACT NO.
S. PAXTON	4/02/09		0811
CHECKED BY	DATE	ESTIMATED LEAD CONCENTRATIONS (KRIGING RESULTS)	APPROVED BY / DATE
N. BALSAMO	7/07/09	ELEVATION: 8 TO 10 FEET	_ / --
COST/SCHED-AREA		ZONES 1, 2, 3, AND 4	APPROVED BY / DATE
		NAVAL SUBMARINE BASE - NEW LONDON	-- / --
SCALE		GROTON, CONNECTICUT	FIGURE NO. / REV.
AS NOTED			FIGURE B-54

P:\GIS\NLON\MXD\ZONE1234_LEAD\ZONE1234_LEAD_6TO8FT_MVS.MXD 7/08/09 SP



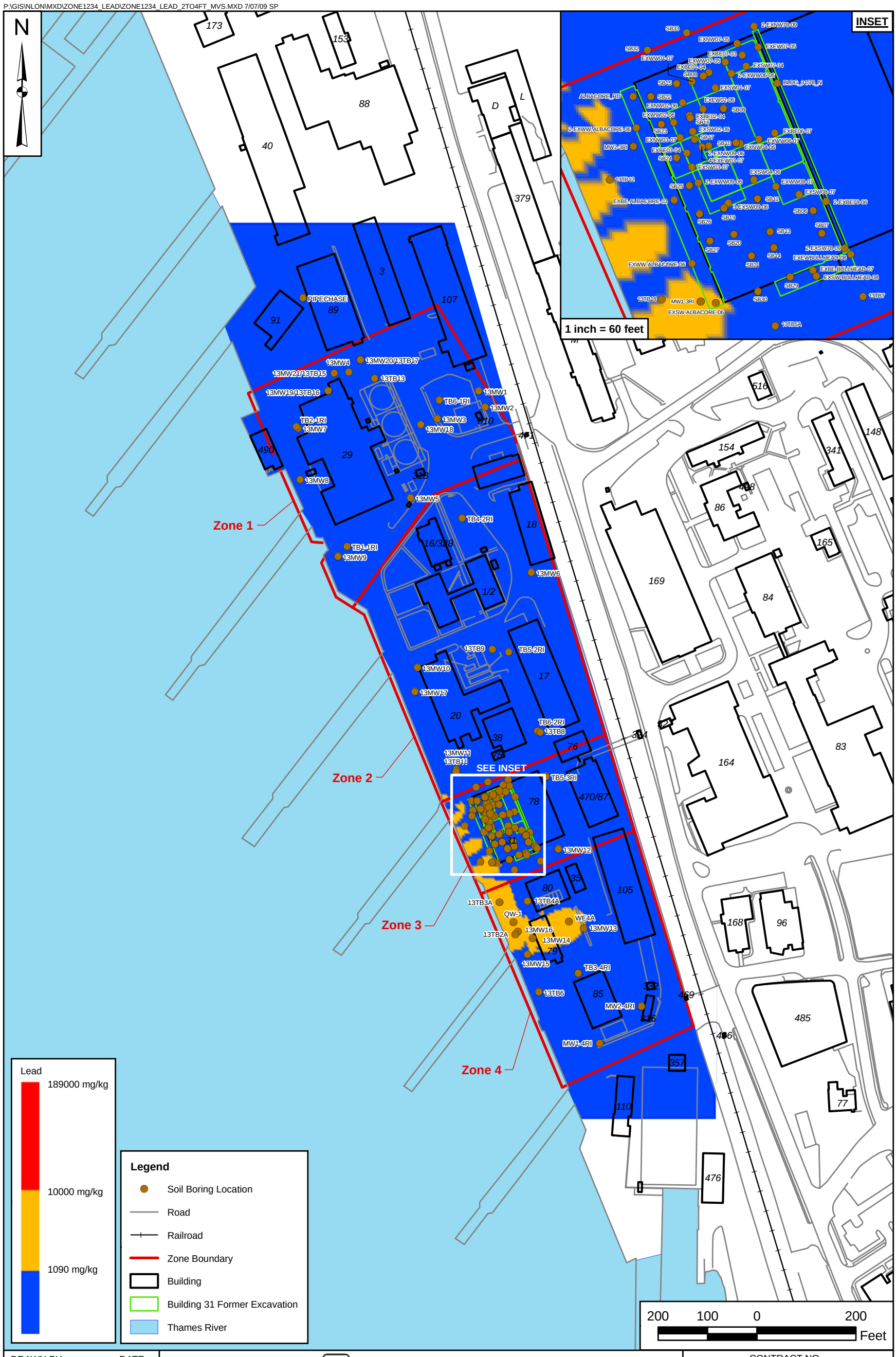
DRAWN BY	DATE	Tetra Tech NUS, Inc.	CONTRACT NO.
S. PAXTON	4/02/09		0811
CHECKED BY	DATE	ESTIMATED LEAD CONCENTRATIONS (KRIGING RESULTS)	APPROVED BY / DATE
N. BALSAMO	7/08/09	ELEVATION: 6 TO 8 FEET	_ / --
COST/SCHED-AREA		ZONES 1, 2, 3, AND 4	APPROVED BY / DATE
		NAVAL SUBMARINE BASE - NEW LONDON	-- / --
SCALE		GROTON, CONNECTICUT	FIGURE NO. / REV.
AS NOTED			FIGURE B-55

P:\GIS\NLON\MXD\ZONE1234_LEAD\ZONE1234_LEAD_4TO6FT_MVS.MXD 7/07/09 SP



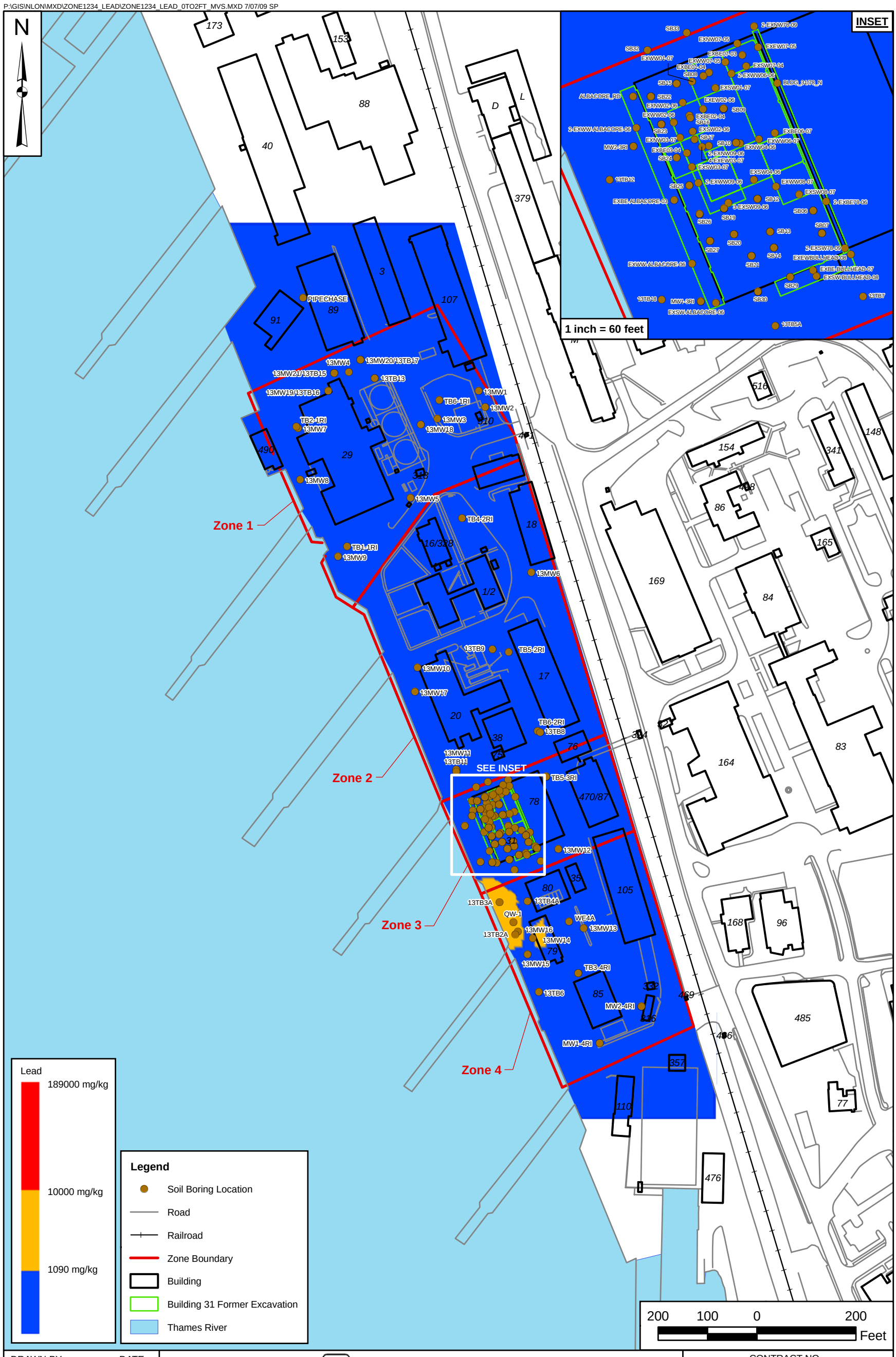
DRAWN BY	DATE	Tetra Tech NUS, Inc.	CONTRACT NO.
S. PAXTON	4/02/09		0811
CHECKED BY	DATE	ESTIMATED LEAD CONCENTRATIONS (KRIGING RESULTS)	APPROVED BY / DATE
N. BALSAMO	7/07/09	ELEVATION: 4 TO 6 FEET	_ / --
COST/SCHED-AREA		ZONES 1, 2, 3, AND 4	APPROVED BY / DATE
		NAVAL SUBMARINE BASE - NEW LONDON	-- / --
SCALE		GROTON, CONNECTICUT	FIGURE NO. / REV.
AS NOTED			FIGURE B-56

P:\GIS\NLON\MXD\ZONE1234_LEAD\ZONE1234_LEAD_2TO4FT_MVS.MXD 7/07/09 SP



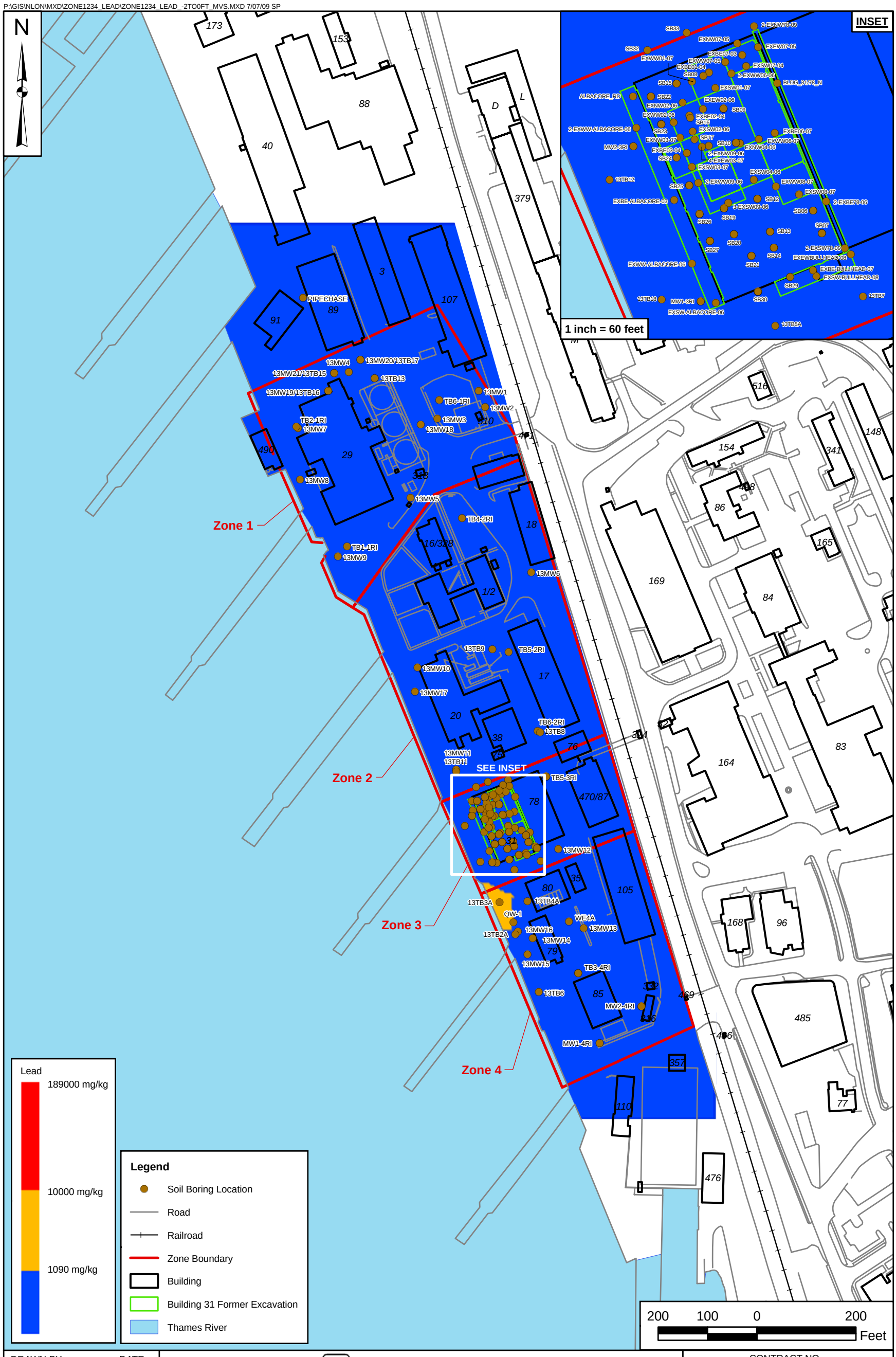
DRAWN BY S. PAXTON	DATE 4/02/09	Tetra Tech NUS, Inc.	CONTRACT NO. 0811
CHECKED BY N. BALSAMO	DATE 7/07/09	ESTIMATED LEAD CONCENTRATIONS (KRIGING RESULTS) ELEVATION: 2 TO 4 FEET ZONES 1, 2, 3, AND 4 NAVAL SUBMARINE BASE - NEW LONDON GROTON, CONNECTICUT	APPROVED BY _ / DATE --
COST/SCHED-AREA			APPROVED BY -- / DATE --
SCALE AS NOTED			FIGURE NO. FIGURE B-57 / REV.

P:\GIS\NLON\MXD\ZONE1234_LEAD\ZONE1234_LEAD_0TO2FT_MVS.MXD 7/07/09 SP



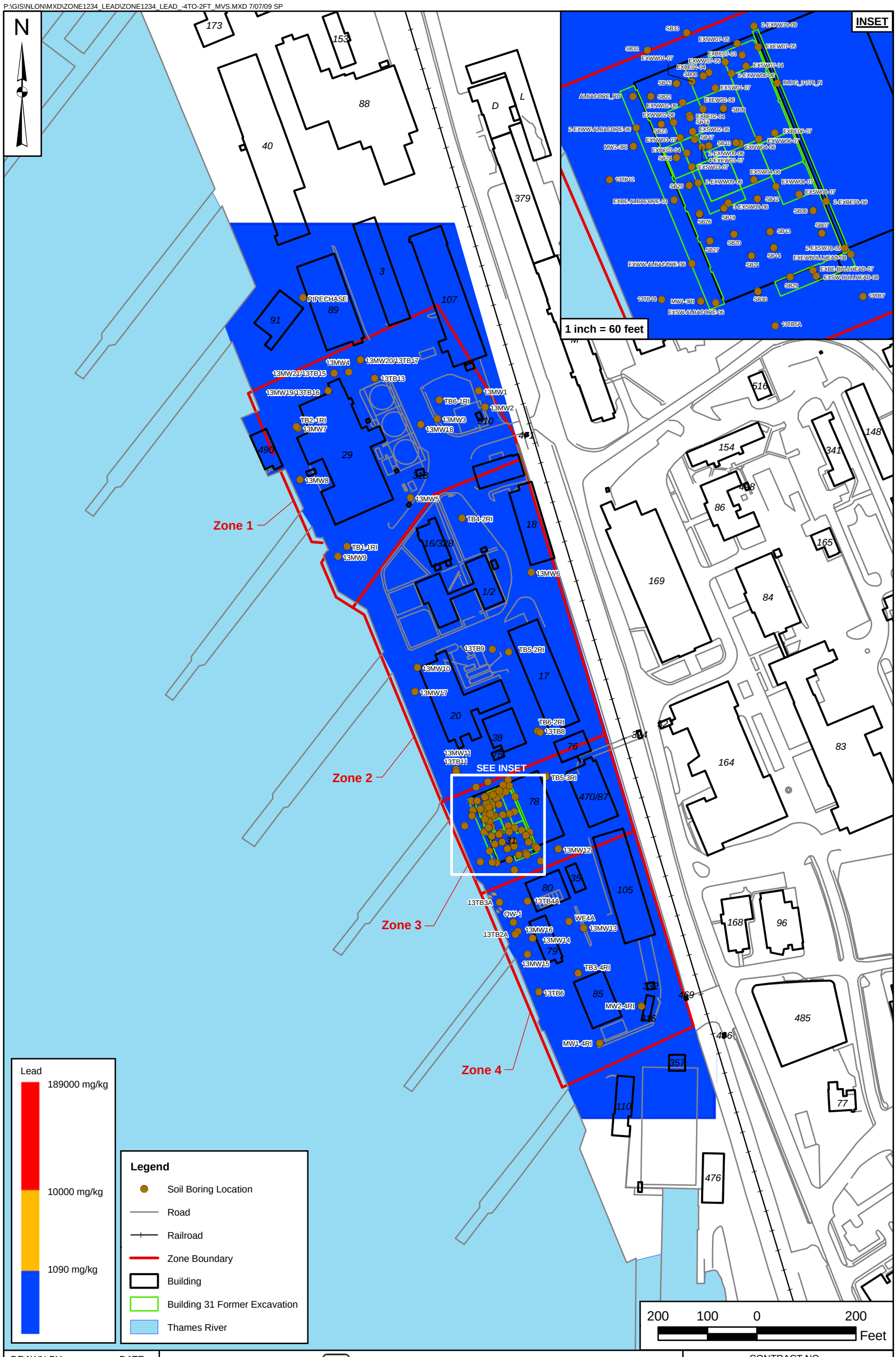
DRAWN BY	DATE	Tetra Tech NUS, Inc.	CONTRACT NO.
S. PAXTON	4/02/09		0811
CHECKED BY	DATE	ESTIMATED LEAD CONCENTRATIONS (KRIGING RESULTS)	APPROVED BY / DATE
N. BALSAMO	7/07/09	ELEVATION: 0 TO 2 FEET	_ / --
COST/SCHED-AREA		ZONES 1, 2, 3, AND 4	APPROVED BY / DATE
		NAVAL SUBMARINE BASE - NEW LONDON	-- / --
SCALE		GROTON, CONNECTICUT	FIGURE NO. / REV.
AS NOTED			FIGURE B-58

P:\GIS\NLON\MXD\ZONE1234_LEAD\ZONE1234_LEAD_-2TO0FT_MVS.MXD 7/07/09 SP



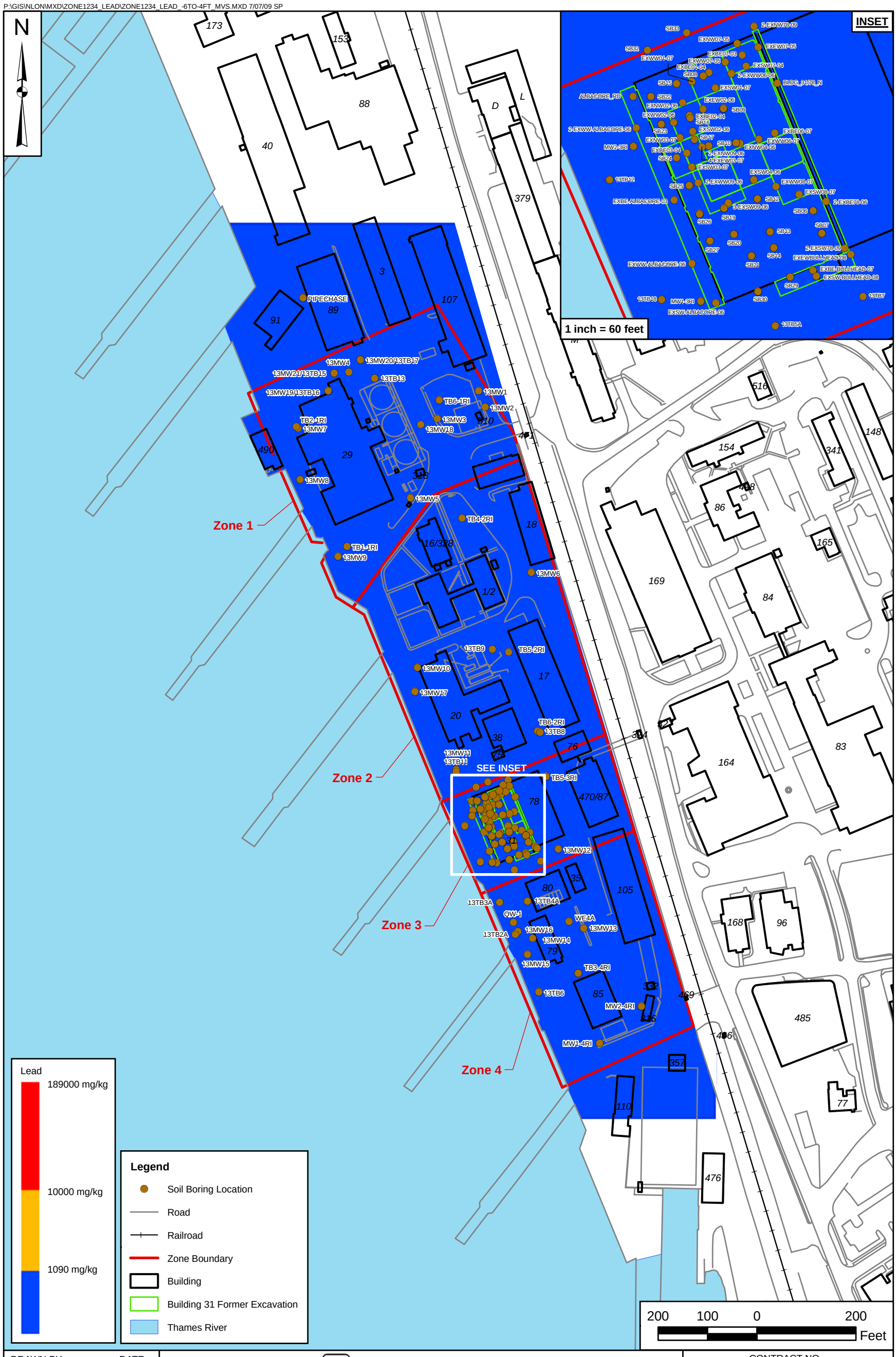
DRAWN BY	DATE	Tetra Tech NUS, Inc.	CONTRACT NO.
S. PAXTON	4/02/09		0811
CHECKED BY	DATE	ESTIMATED LEAD CONCENTRATIONS (KRIGING RESULTS)	APPROVED BY / DATE
N. BALSAMO	7/07/09	ELEVATION: -2 TO 0 FEET	_ / --
COST/SCHED-AREA		ZONES 1, 2, 3, AND 4	APPROVED BY / DATE
		NAVAL SUBMARINE BASE - NEW LONDON	-- / --
SCALE		GROTON, CONNECTICUT	FIGURE NO. / REV.
AS NOTED			FIGURE B-59

P:\GIS\NLON\MXD\ZONE1234_LEAD\ZONE1234_LEAD_-4TO-2FT_MVS.MXD 7/07/09 SP



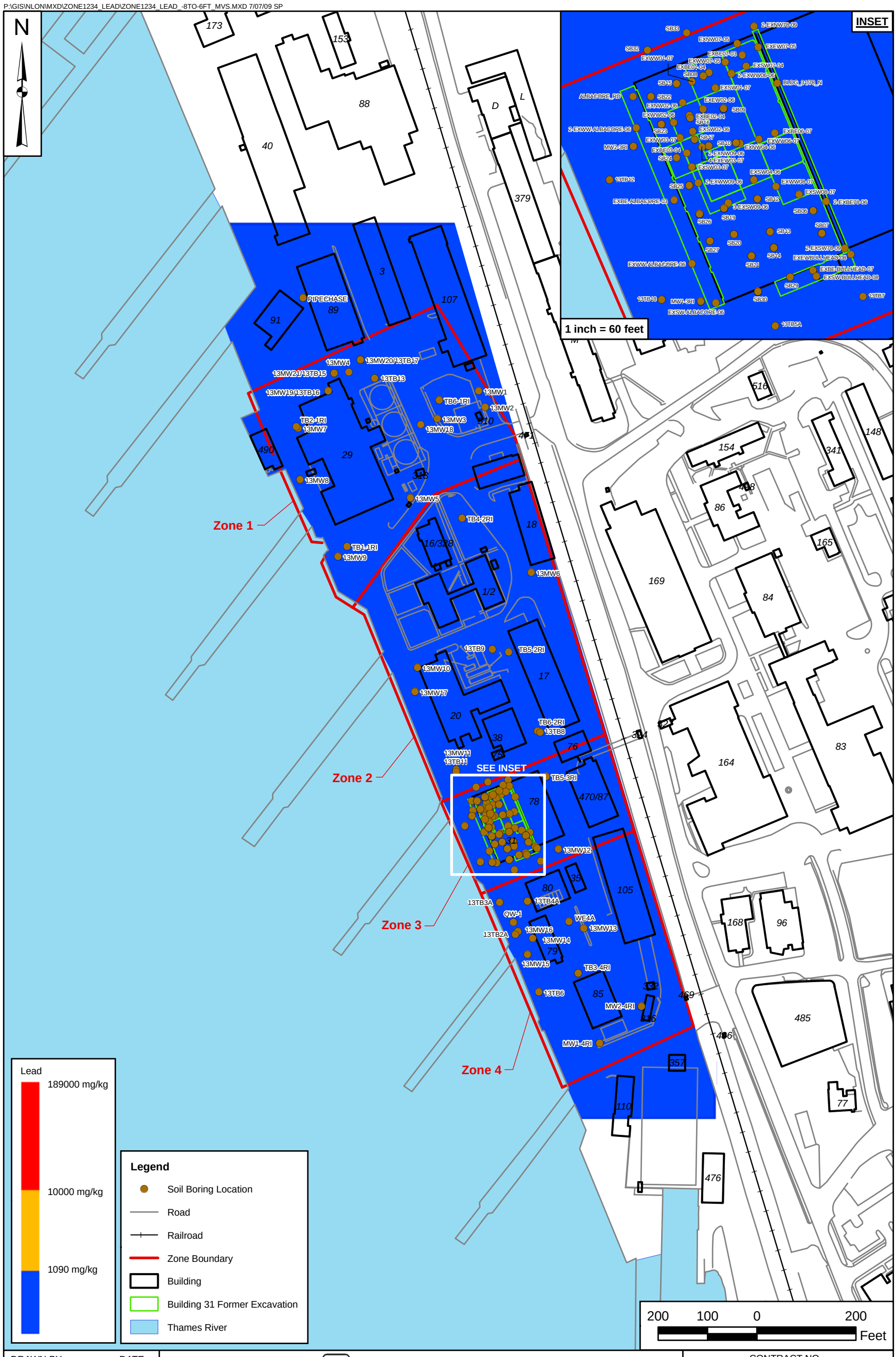
DRAWN BY	DATE	Tetra Tech NUS, Inc.	CONTRACT NO.
S. PAXTON	4/02/09		0811
CHECKED BY	DATE	ESTIMATED LEAD CONCENTRATIONS (KRIGING RESULTS)	APPROVED BY / DATE
N. BALSAMO	7/07/09	ELEVATION: -4 TO -2 FEET	_ / --
COST/SCHED-AREA		ZONES 1, 2, 3, AND 4	APPROVED BY / DATE
		NAVAL SUBMARINE BASE - NEW LONDON	-- / --
SCALE		GROTON, CONNECTICUT	FIGURE NO. / REV.
AS NOTED			FIGURE B-60

P:\GIS\NLON\MXD\ZONE1234_LEAD\ZONE1234_LEAD_-6TO-4FT_MVS.MXD 7/07/09 SP



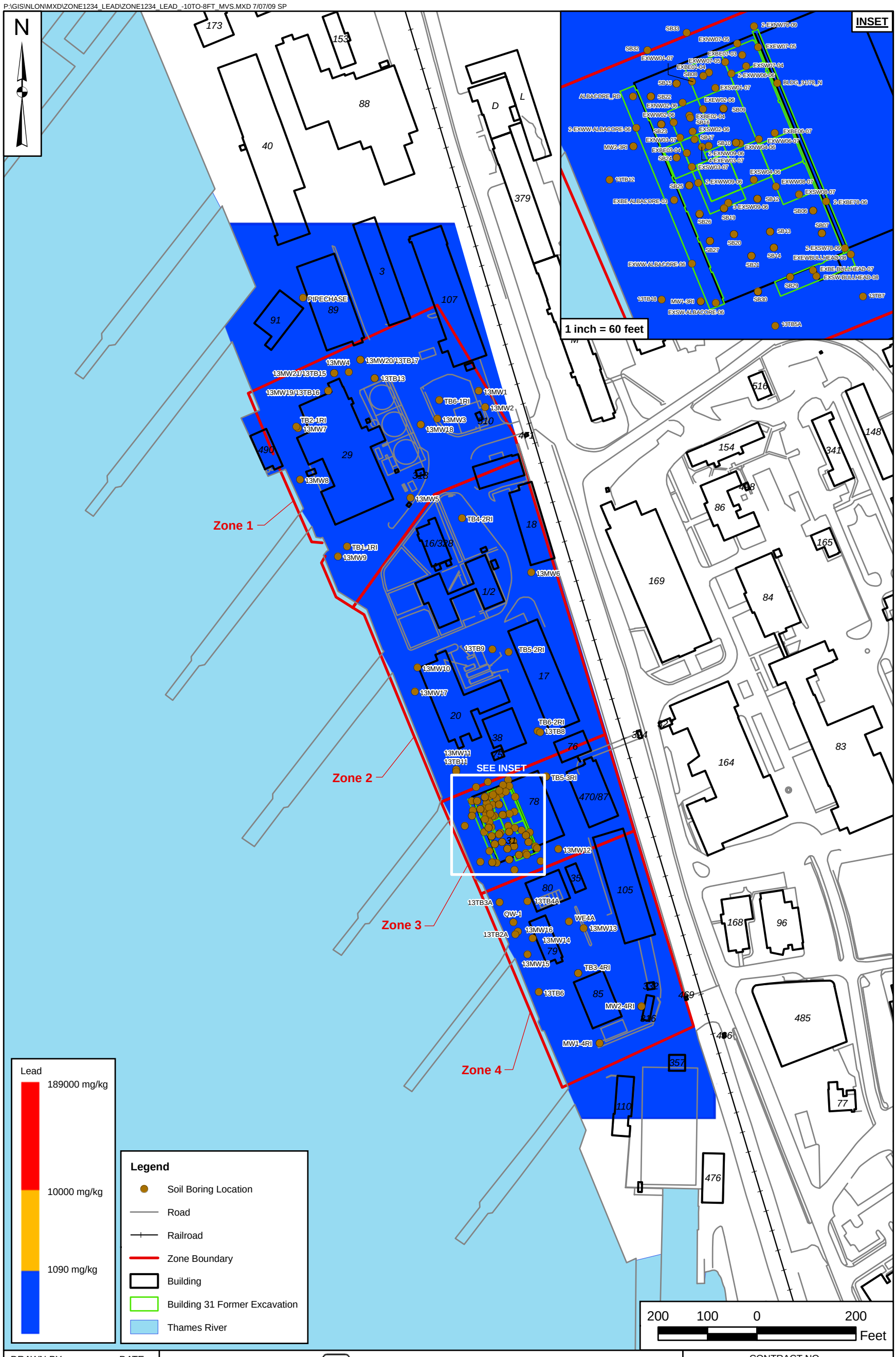
DRAWN BY	DATE	Tetra Tech NUS, Inc.	CONTRACT NO.
S. PAXTON	4/02/09		0811
CHECKED BY	DATE	ESTIMATED LEAD CONCENTRATIONS (KRIGING RESULTS)	APPROVED BY / DATE
N. BALSAMO	7/07/09	ELEVATION: -6 TO -4 FEET	_ / --
COST/SCHED-AREA		ZONES 1, 2, 3, AND 4	APPROVED BY / DATE
		NAVAL SUBMARINE BASE - NEW LONDON	-- / --
SCALE		GROTON, CONNECTICUT	FIGURE NO. / REV.
AS NOTED			FIGURE B-61

P:\GIS\NLON\MXD\ZONE1234_LEAD\ZONE1234_LEAD_-8TO-6FT_MVS.MXD 7/07/09 SP



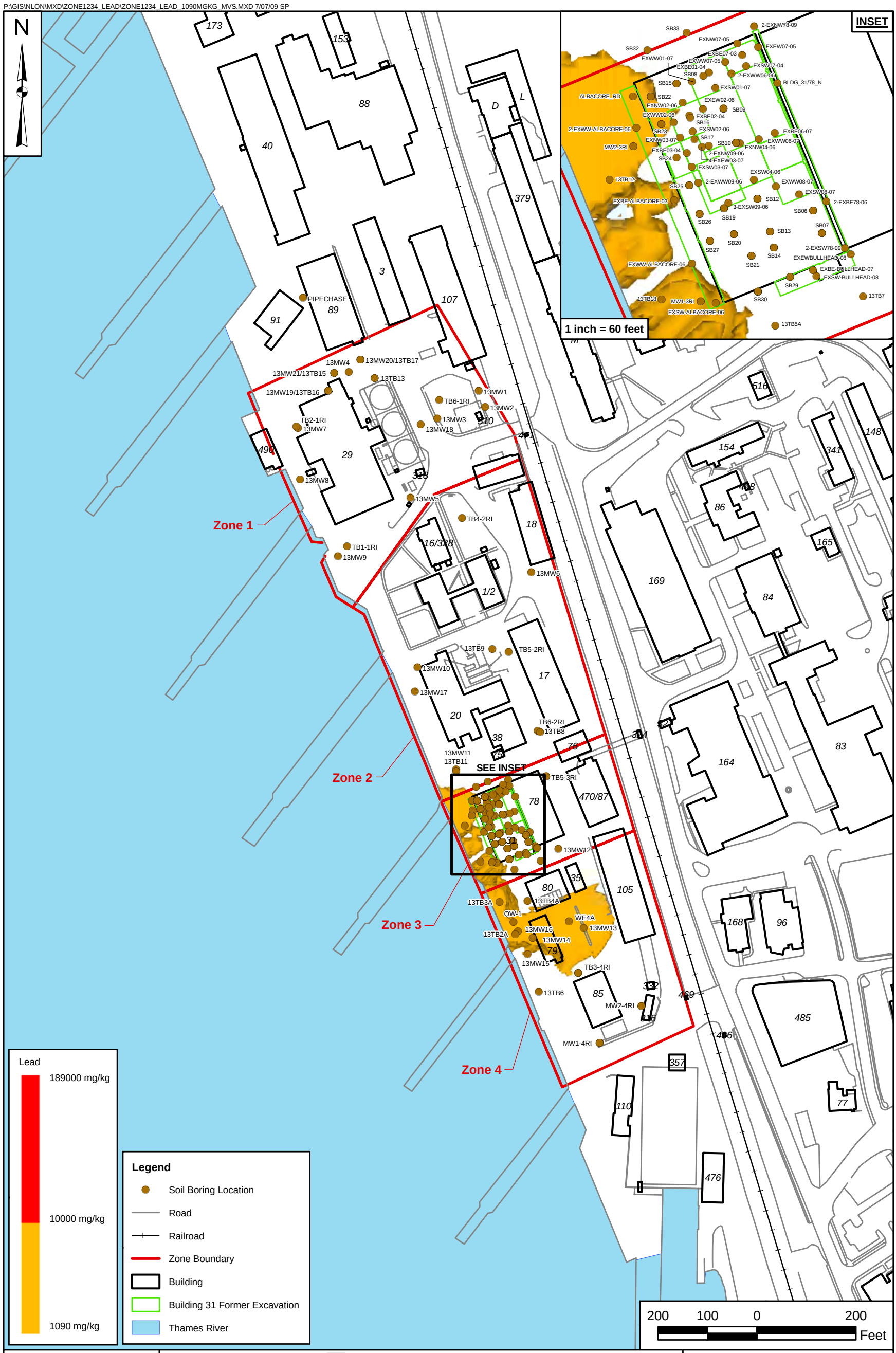
DRAWN BY	DATE	Tetra Tech NUS, Inc.	CONTRACT NO.
S. PAXTON	4/02/09		0811
CHECKED BY	DATE	ESTIMATED LEAD CONCENTRATIONS (KRIGING RESULTS)	APPROVED BY / DATE
N. BALSAMO	7/07/09	ELEVATION: -8 TO -6 FEET	_ / --
COST/SCHED-AREA		ZONES 1, 2, 3, AND 4	APPROVED BY / DATE
		NAVAL SUBMARINE BASE - NEW LONDON	-- / --
SCALE		GROTON, CONNECTICUT	FIGURE NO. / REV.
AS NOTED			FIGURE B-62

P:\GIS\NLON\MXD\ZONE1234_LEAD\ZONE1234_LEAD_-10TO-8FT_MVS.MXD 7/07/09 SP



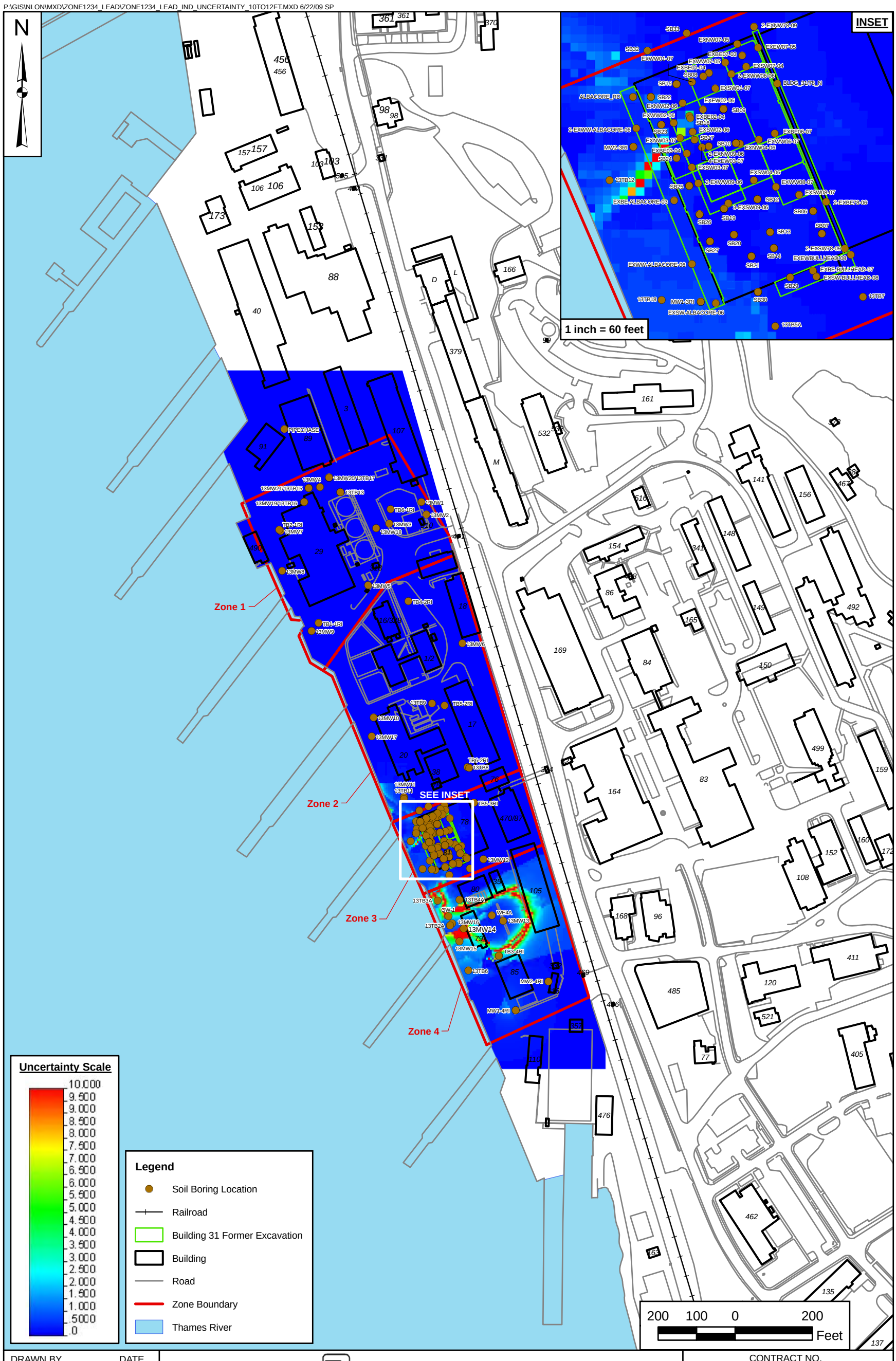
DRAWN BY	DATE	Tetra Tech NUS, Inc.	CONTRACT NO.
S. PAXTON	4/02/09		0811
CHECKED BY	DATE	ESTIMATED LEAD CONCENTRATIONS (KRIGING RESULTS)	APPROVED BY / DATE
N. BALSAMO	7/07/09	ELEVATION: -10 TO -8 FEET	_ / --
COST/SCHED-AREA		ZONES 1, 2, 3, AND 4	APPROVED BY / DATE
		NAVAL SUBMARINE BASE - NEW LONDON	-- / --
SCALE		GROTON, CONNECTICUT	FIGURE NO. / REV.
AS NOTED			FIGURE B-63

P:\GIS\NLON\MXD\ZONE1234_LEAD\ZONE1234_LEAD_1090MGKG_MVS.MXD 7/07/09 SP



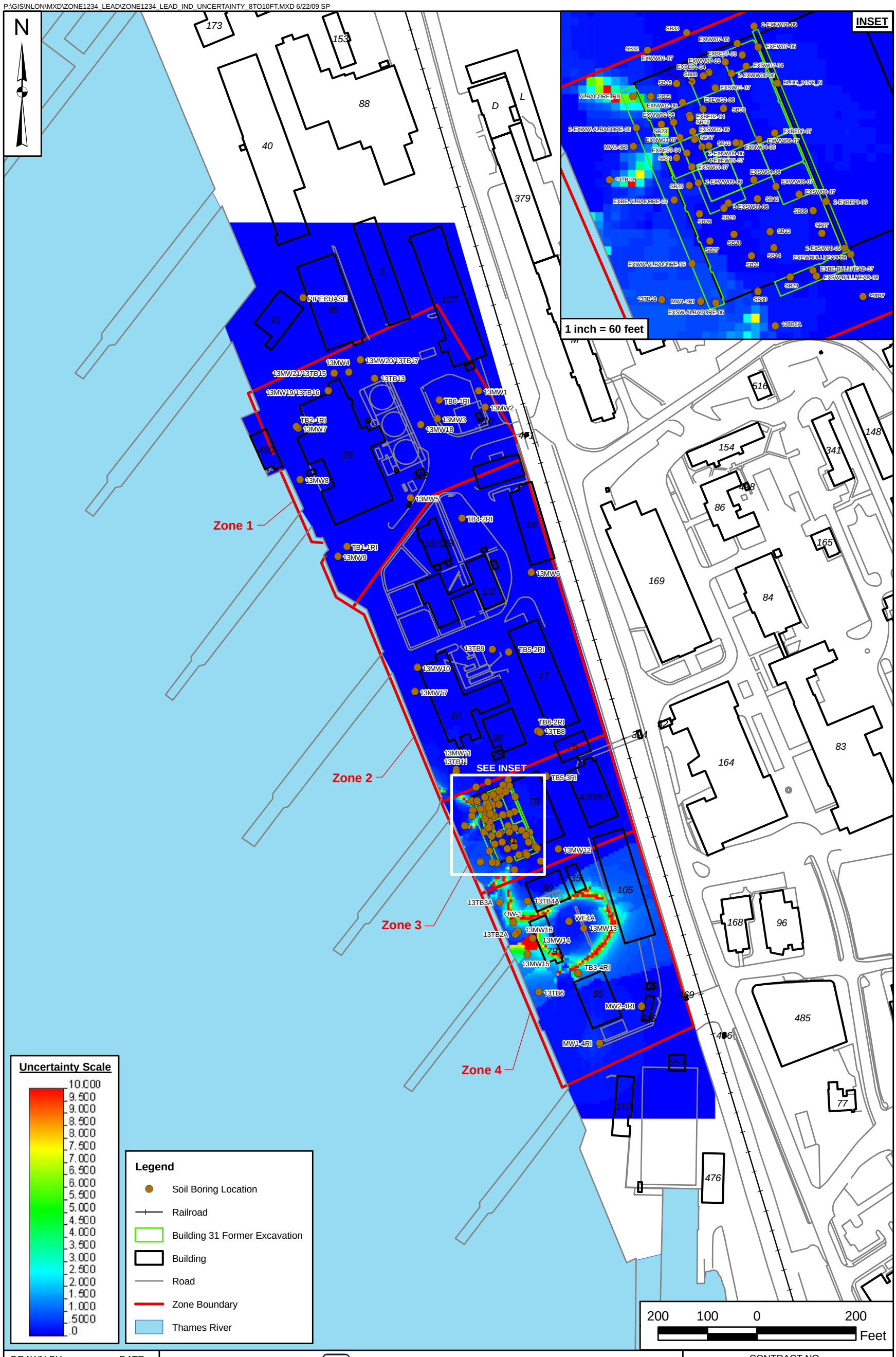
DRAWN BY	DATE	Tetra Tech NUS, Inc.	CONTRACT NO.
S. PAXTON	4/02/09		0811
CHECKED BY	DATE	ESTIMATED LEAD CONCENTRATIONS (KRIGING RESULTS)	APPROVED BY / DATE
N. BALSAMO	7/07/09	ELEVATION: 10 TO 0 FEET	_ / --
COST/SCHED-AREA		ZONES 1, 2, 3, AND 4	APPROVED BY / DATE
		NAVAL SUBMARINE BASE - NEW LONDON	-- / --
SCALE		GROTON, CONNECTICUT	FIGURE NO. / REV.
AS NOTED			FIGURE B-64

P:\GIS\NLON\MXD\ZONE1234_LEAD\ZONE1234_LEAD_IND_UNCERTAINTY_10TO12FT.MXD 6/22/09 SP



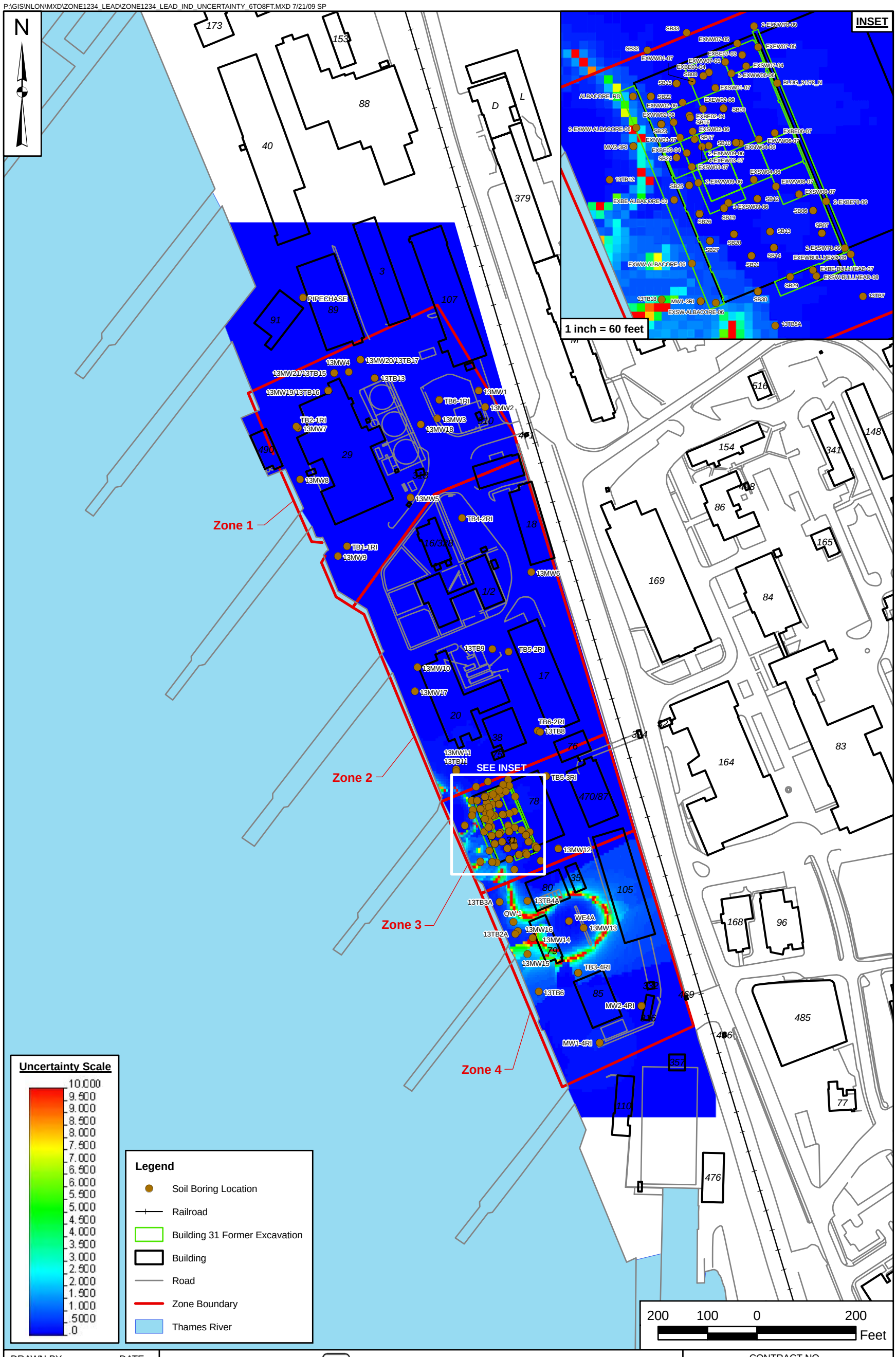
DRAWN BY S. PAXTON	DATE 4/02/09	Tetra Tech NUS, Inc.	CONTRACT NO. 0811
CHECKED BY N. BALSAMO	DATE 6/22/09	LEAD UNCERTAINTY FOR INDUSTRIAL CRITERIA ELEVATION: 10 TO 12 FEET ZONES 1, 2, 3, AND 4 NAVAL SUBMARINE BASE - NEW LONDON GROTON, CONNECTICUT	APPROVED BY _ / DATE --
COST/SCHED-AREA			APPROVED BY -- / DATE --
SCALE AS NOTED			FIGURE NO. FIGURE B-65 / REV.

P:\GIS\NLON\MXD\ZONE1234_LEAD\ZONE1234_LEAD_IND_UNCERTAINTY_8TO10FT.MXD 6/22/09 SP



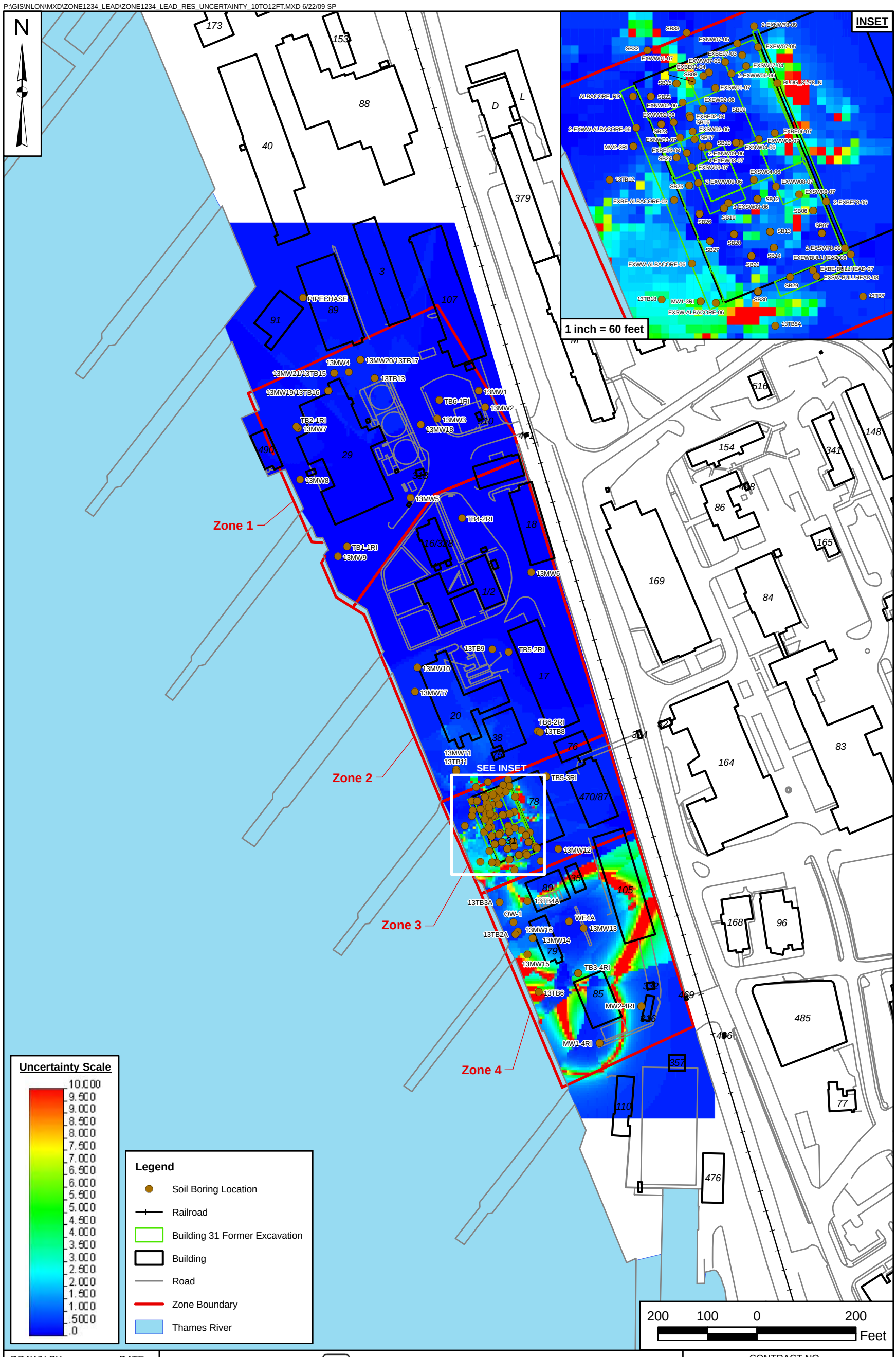
DRAWN BY	DATE	Tetra Tech NUS, Inc.	CONTRACT NO.
S. PAXTON	4/02/09		0811
CHECKED BY	DATE	LEAD UNCERTAINTY FOR INDUSTRIAL CRITERIA ELEVATION: 8 TO 10 FEET ZONES 1, 2, 3, AND 4 NAVAL SUBMARINE BASE - NEW LONDON GROTON, CONNECTICUT	APPROVED BY / DATE
N. BALSAMO	6/22/09		_ / --
COST/SCHED-AREA			APPROVED BY / DATE
			-- / --
SCALE			FIGURE NO. / REV.
AS NOTED			FIGURE B-66

P:\GIS\NLON\MXD\ZONE1234_LEAD\ZONE1234_LEAD_IND_UNCERTAINTY_6TO8FT.MXD 7/21/09 SP



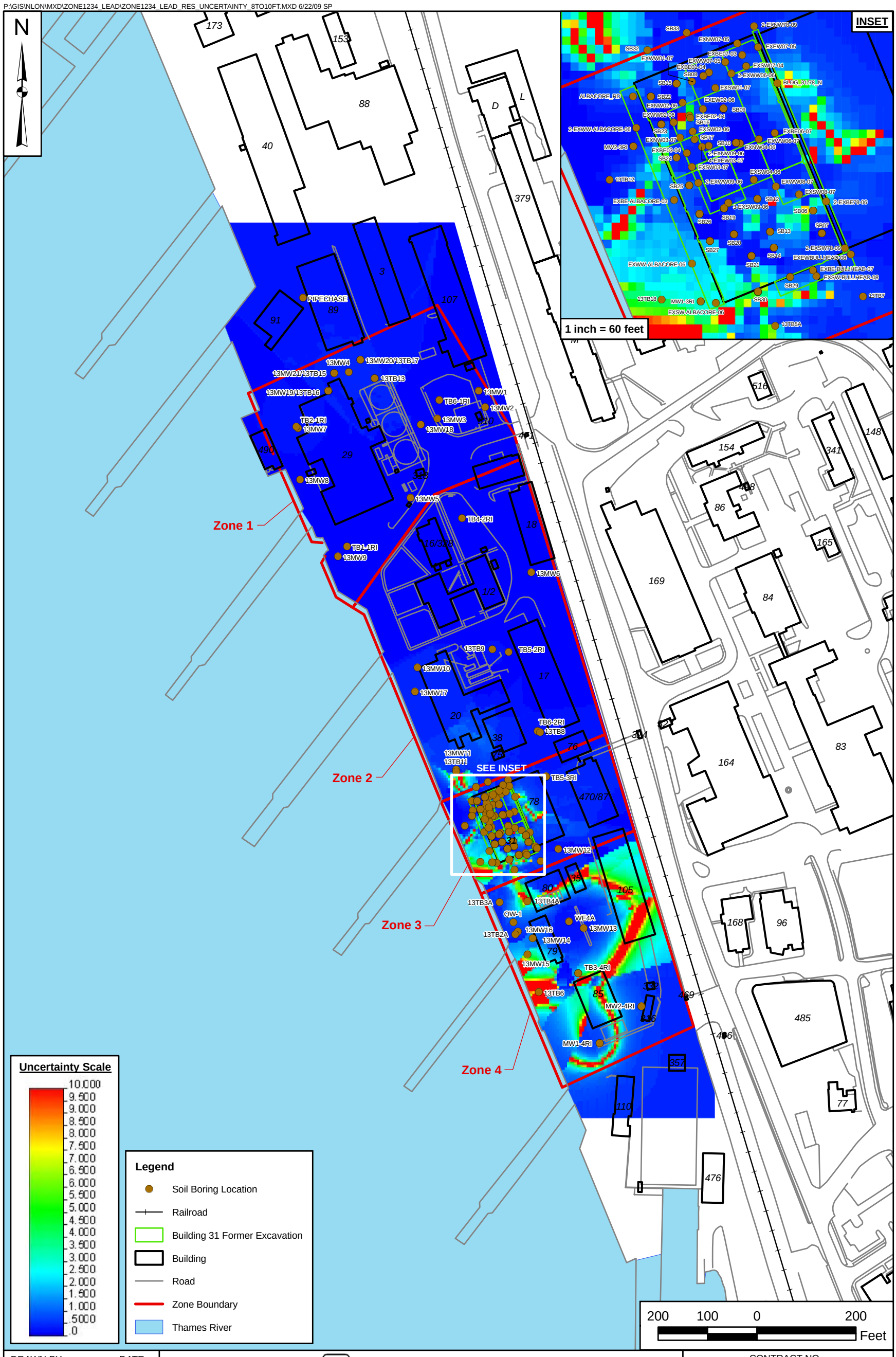
DRAWN BY	DATE	Tetra Tech NUS, Inc.	CONTRACT NO.
S. PAXTON	4/02/09		0811
CHECKED BY	DATE	LEAD UNCERTAINTY FOR INDUSTRIAL CRITERIA ELEVATION: 6 TO 8 FEET ZONES 1, 2, 3, AND 4 NAVAL SUBMARINE BASE - NEW LONDON GROTON, CONNECTICUT	APPROVED BY / DATE
N. BALSAMO	7/21/09		_ / --
COST/SCHED-AREA			APPROVED BY / DATE
			-- / --
SCALE			FIGURE NO. / REV.
AS NOTED			FIGURE B-67

P:\GIS\NLON\MXD\ZONE1234_LEAD\ZONE1234_LEAD_RES_UNCERTAINTY_10TO12FT.MXD 6/22/09 SP



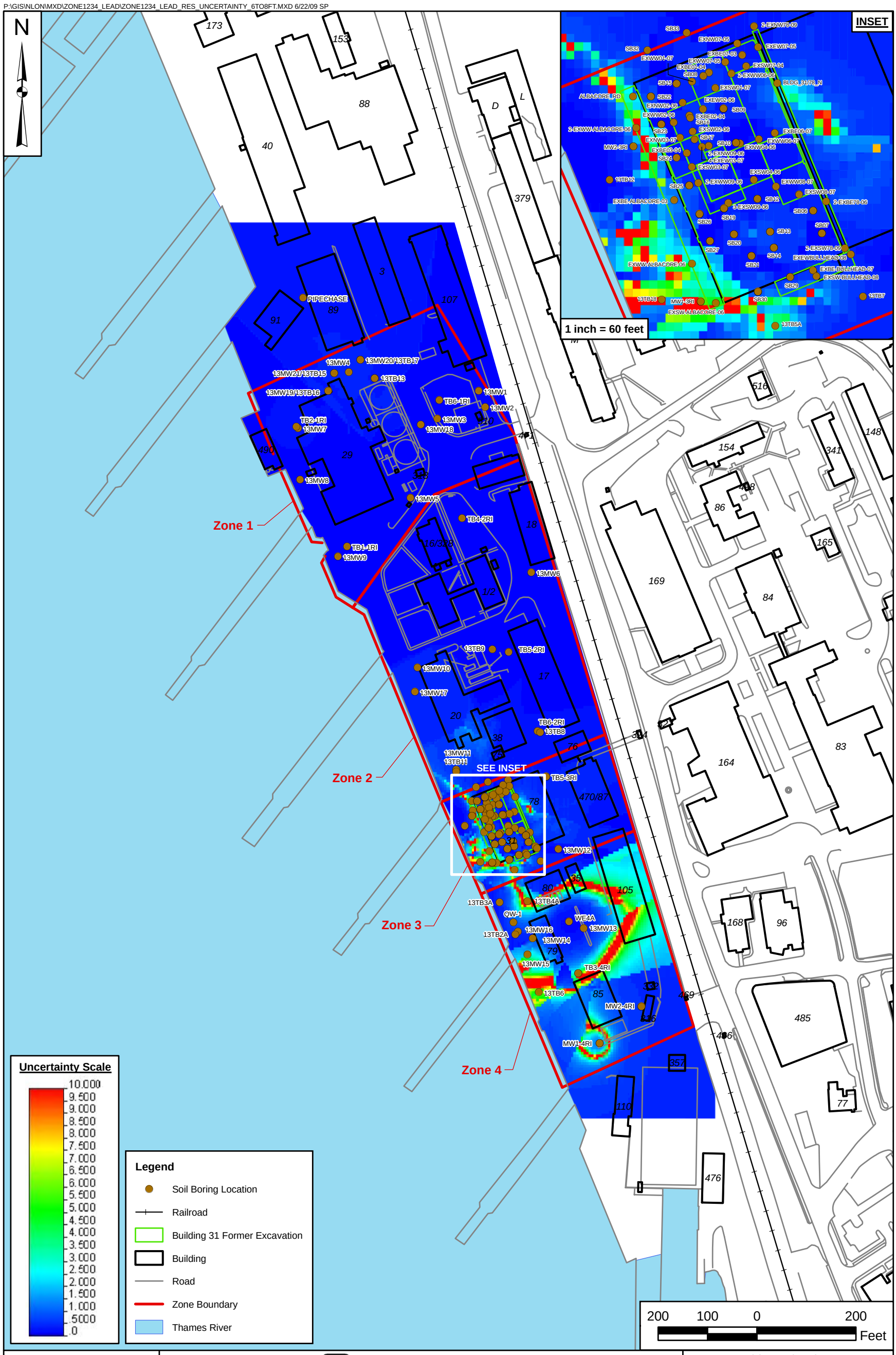
DRAWN BY S. PAXTON	DATE 4/02/09	Tetra Tech NUS, Inc.	CONTRACT NO. 0811
CHECKED BY N. BALSAMO	DATE 6/22/09	LEAD UNCERTAINTY FOR RESIDENTIAL CRITERIA ELEVATION: 10 TO 12 FEET ZONES 1, 2, 3, AND 4 NAVAL SUBMARINE BASE - NEW LONDON GROTON, CONNECTICUT	APPROVED BY _ / DATE --
COST/SCHED-AREA			APPROVED BY -- / DATE --
SCALE AS NOTED			FIGURE NO. FIGURE B-68 / REV.

P:\GIS\NLON\MXD\ZONE1234_LEAD\ZONE1234_LEAD_RES_UNCERTAINTY_8TO10FT.MXD 6/22/09 SP



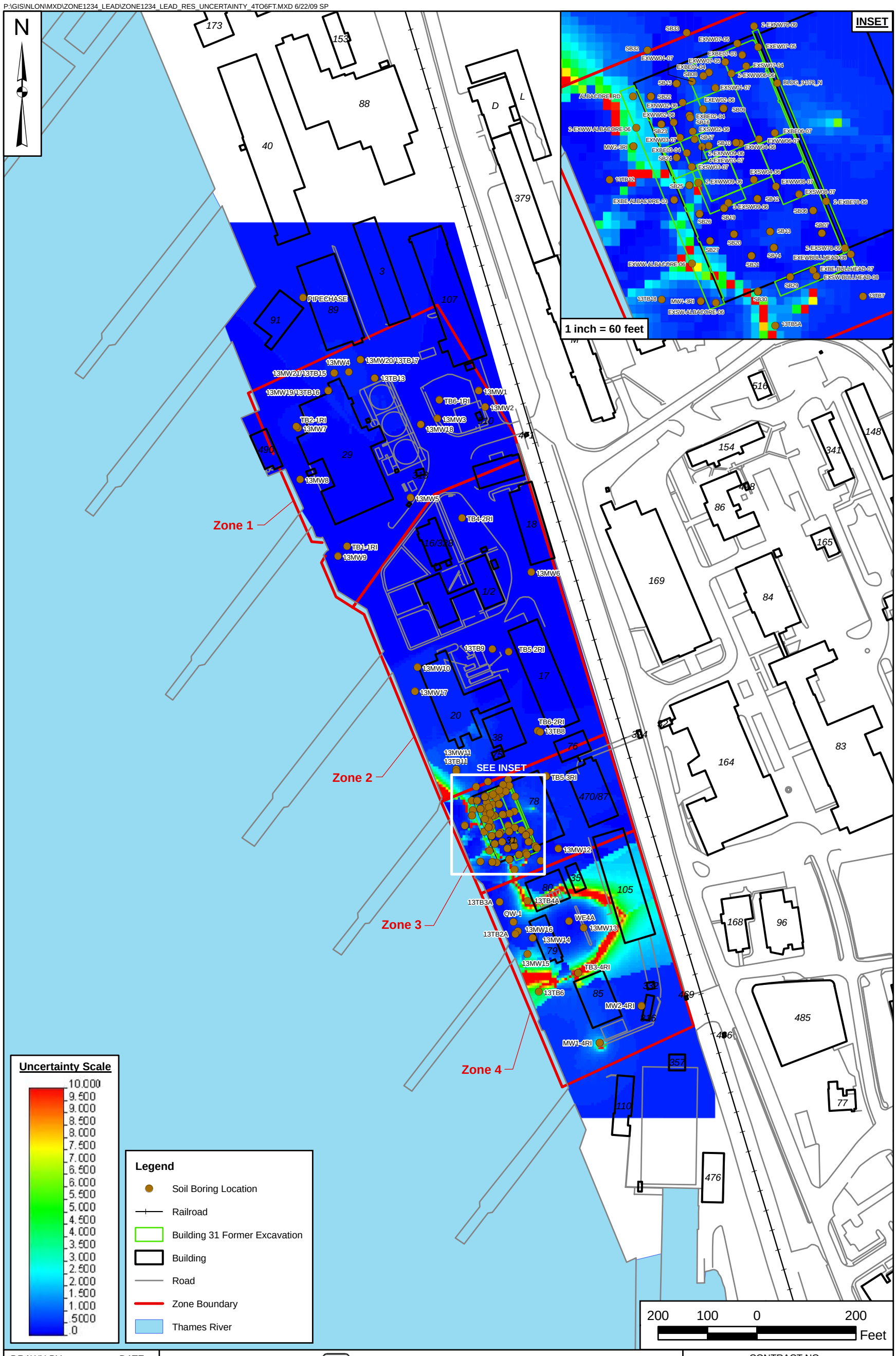
DRAWN BY	DATE	Tetra Tech NUS, Inc.	CONTRACT NO.
S. PAXTON	4/02/09		0811
CHECKED BY	DATE	LEAD UNCERTAINTY FOR RESIDENTIAL CRITERIA ELEVATION: 8 TO 10 FEET ZONES 1, 2, 3, AND 4 NAVAL SUBMARINE BASE - NEW LONDON GROTON, CONNECTICUT	APPROVED BY / DATE
N. BALSAMO	6/22/09		_ / --
COST/SCHED-AREA			APPROVED BY / DATE
			-- / --
SCALE			FIGURE NO. / REV.
AS NOTED			FIGURE B-69

P:\GIS\NLON\MXD\ZONE1234_LEAD\ZONE1234_LEAD_RES_UNCERTAINTY_6TO8FT.MXD 6/22/09 SP



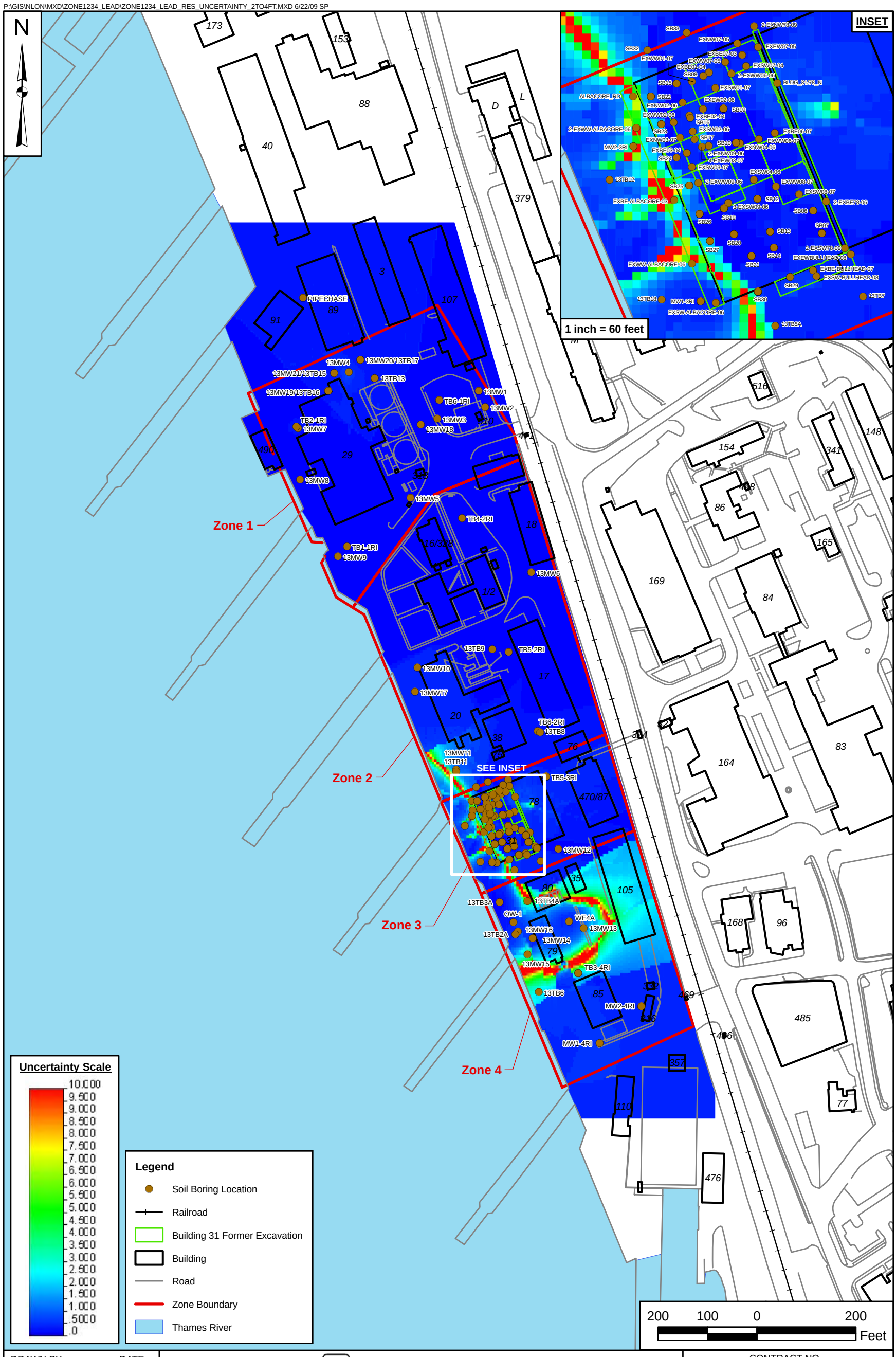
DRAWN BY	DATE	Tetra Tech NUS, Inc.	CONTRACT NO.
S. PAXTON	4/02/09		0811
CHECKED BY	DATE	LEAD UNCERTAINTY FOR RESIDENTIAL CRITERIA ELEVATION: 6 TO 8 FEET ZONES 1, 2, 3, AND 4 NAVAL SUBMARINE BASE - NEW LONDON GROTON, CONNECTICUT	APPROVED BY / DATE
N. BALSAMO	6/22/09		_ / --
COST/SCHED-AREA			APPROVED BY / DATE
			-- / --
SCALE			FIGURE NO. / REV.
AS NOTED			FIGURE B-70

P:\GIS\NLON\MXD\ZONE1234_LEAD\ZONE1234_LEAD_RES_UNCERTAINTY_4TO6FT.MXD 6/22/09 SP



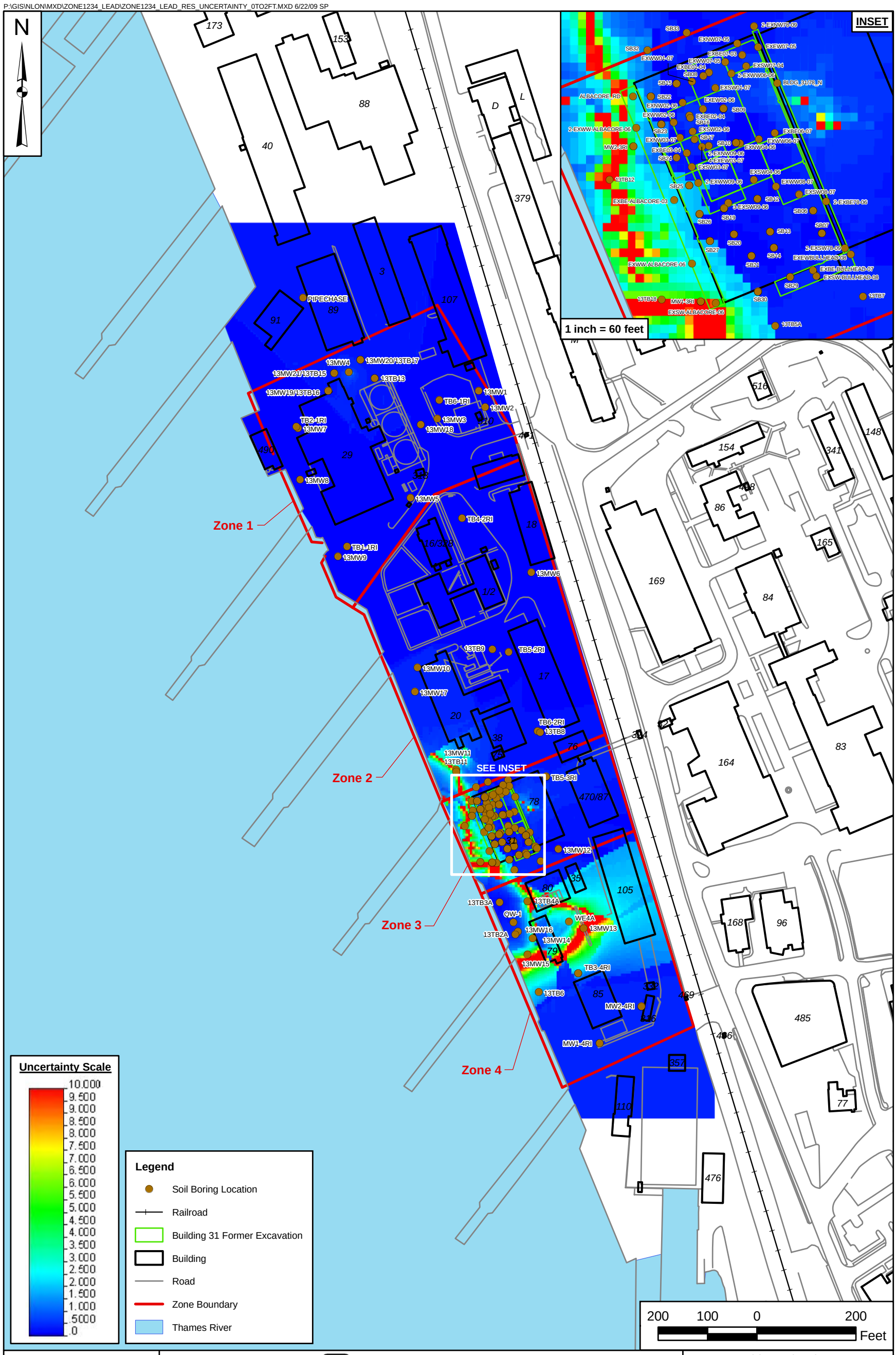
DRAWN BY	DATE	Tetra Tech NUS, Inc.	CONTRACT NO.
S. PAXTON	4/02/09		0811
CHECKED BY	DATE	LEAD UNCERTAINTY FOR RESIDENTIAL CRITERIA ELEVATION: 4 TO 6 FEET ZONES 1, 2, 3, AND 4 NAVAL SUBMARINE BASE - NEW LONDON GROTON, CONNECTICUT	APPROVED BY / DATE
N. BALSAMO	6/22/09		_ / --
COST/SCHED-AREA			APPROVED BY / DATE
			-- / --
SCALE			FIGURE NO. / REV.
AS NOTED			FIGURE B-71

P:\GIS\NLON\MXD\ZONE1234_LEAD\ZONE1234_LEAD_RES_UNCERTAINTY_2TO4FT.MXD 6/22/09 SP



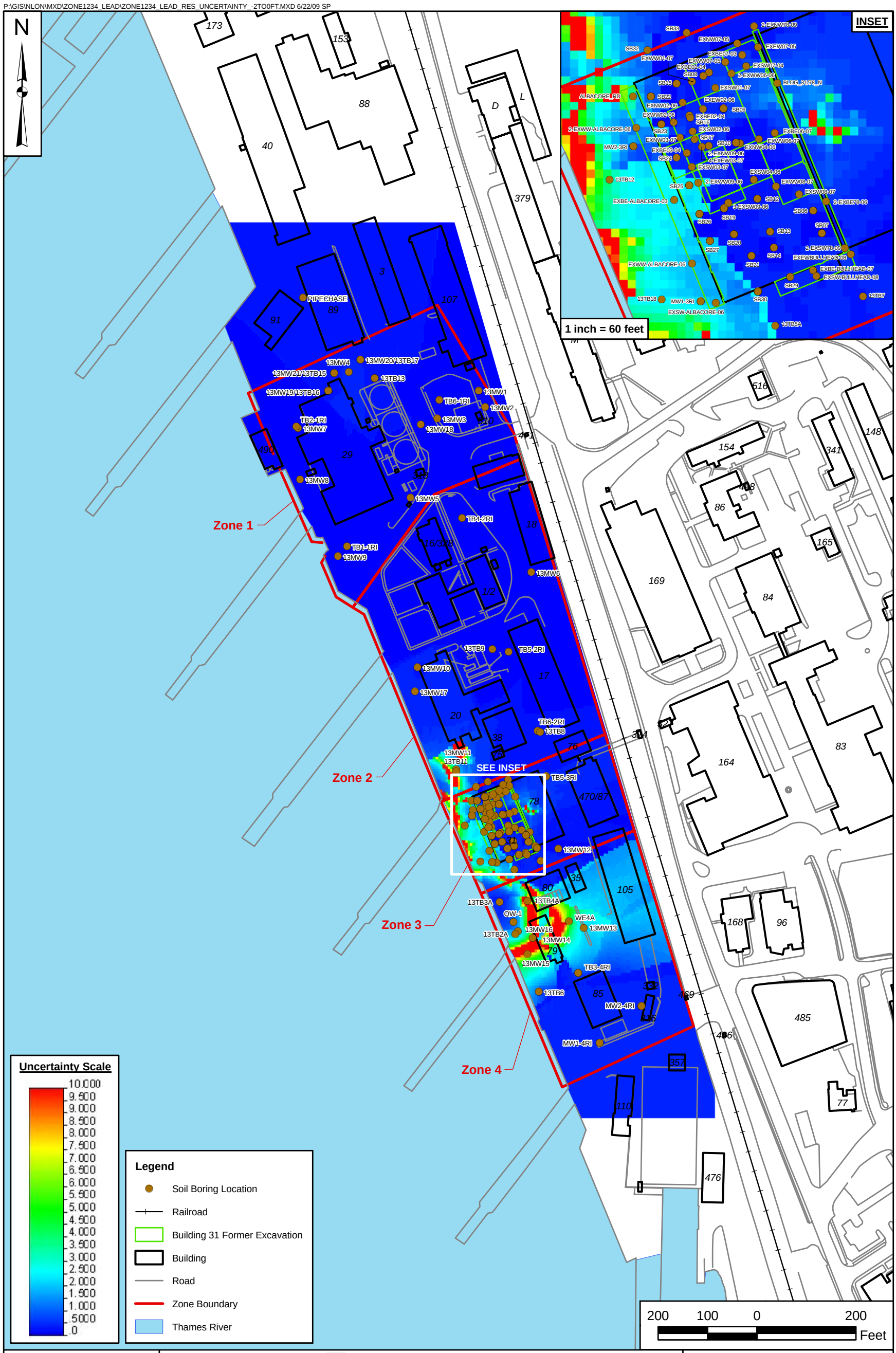
DRAWN BY	DATE	Tetra Tech NUS, Inc.	CONTRACT NO.
S. PAXTON	4/02/09		0811
CHECKED BY	DATE	LEAD UNCERTAINTY FOR RESIDENTIAL CRITERIA ELEVATION: 2 TO 4 FEET ZONES 1, 2, 3, AND 4 NAVAL SUBMARINE BASE - NEW LONDON GROTON, CONNECTICUT	APPROVED BY / DATE
N. BALSAMO	6/22/09		_ / --
COST/SCHED-AREA			APPROVED BY / DATE
			-- / --
SCALE			FIGURE NO. / REV.
AS NOTED			FIGURE B-72

P:\GIS\NLON\MXD\ZONE1234_LEAD\ZONE1234_LEAD_RES_UNCERTAINTY_0TO2FT.MXD 6/22/09 SP



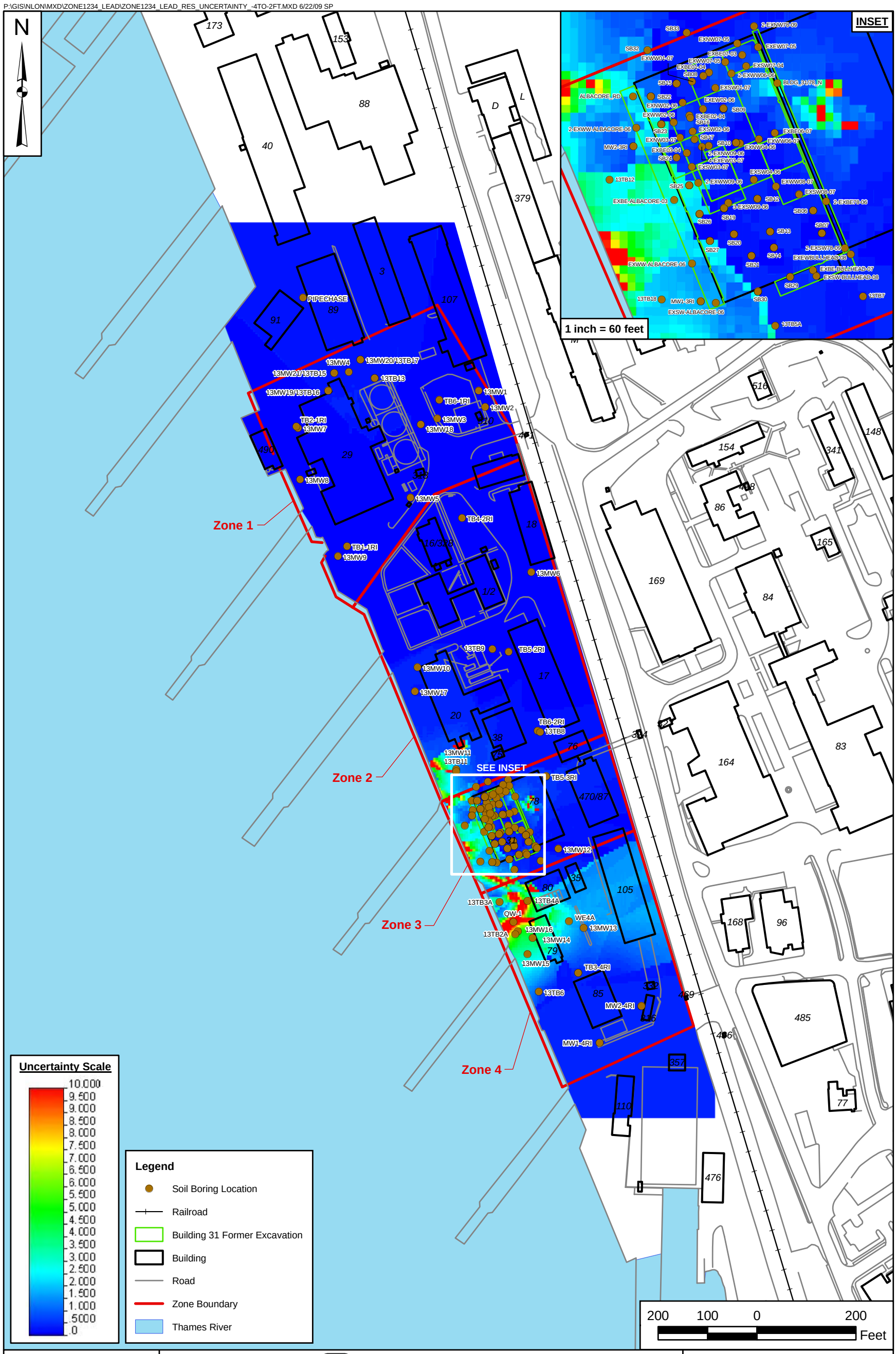
DRAWN BY S. PAXTON	DATE 4/02/09	Tetra Tech NUS, Inc.	CONTRACT NO. 0811
CHECKED BY N. BALSAMO	DATE 6/22/09	LEAD UNCERTAINTY FOR RESIDENTIAL CRITERIA ELEVATION: 0 TO 2 FEET ZONES 1, 2, 3, AND 4 NAVAL SUBMARINE BASE - NEW LONDON GROTON, CONNECTICUT	APPROVED BY – DATE --
COST/SCHED-AREA			APPROVED BY -- DATE --
SCALE AS NOTED			FIGURE NO. FIGURE B-73 REV.

P:\GIS\NLON\MXD\ZONE1234_LEAD\ZONE1234_LEAD_RES_UNCERTAINTY_-2TO0FT.MXD 6/22/09 SP



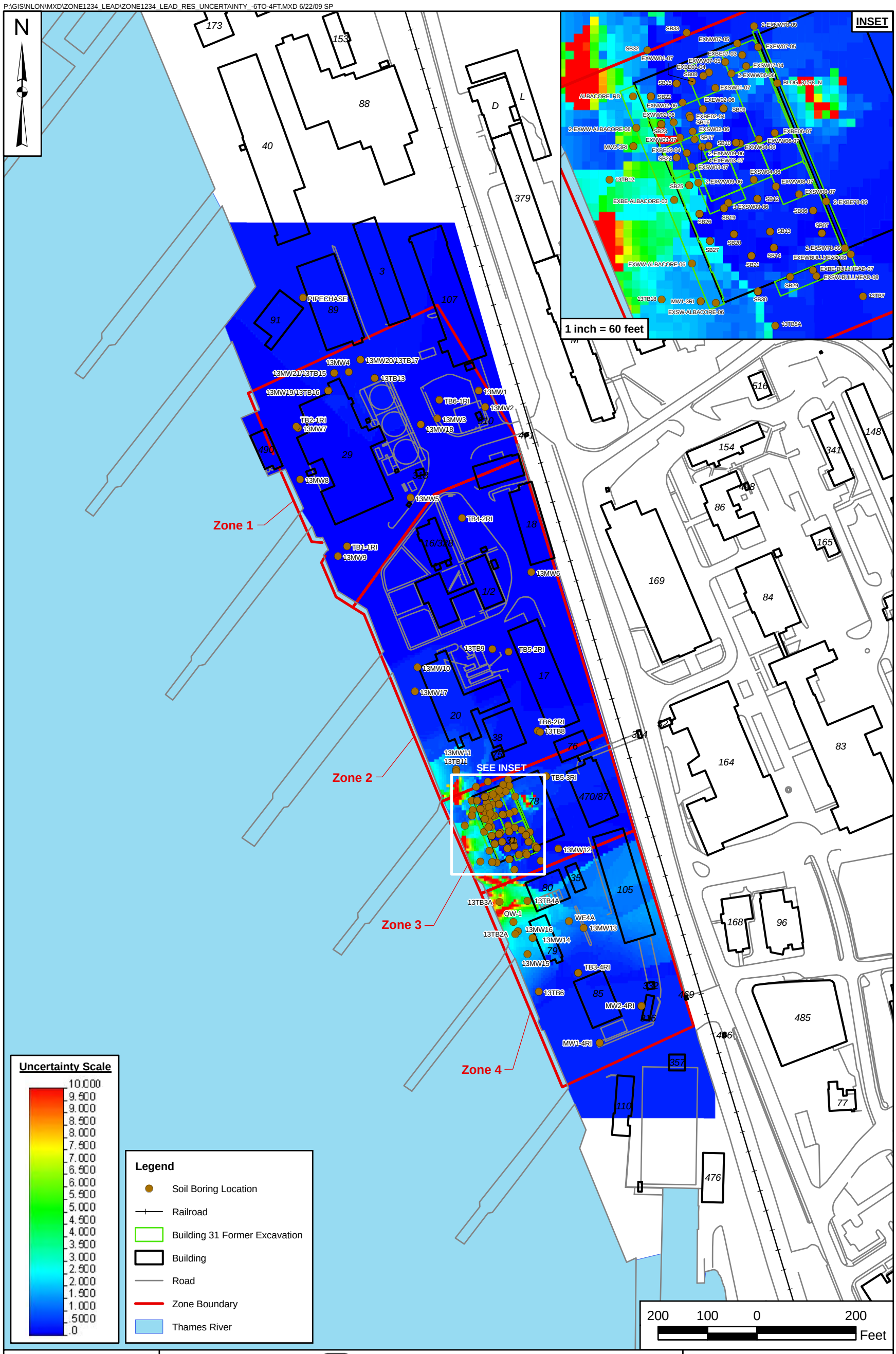
DRAWN BY S. PAXTON	DATE 4/02/09	Tetra Tech NUS, Inc.	CONTRACT NO. 0811
CHECKED BY N. BALSAMO	DATE 6/22/09	LEAD UNCERTAINTY FOR RESIDENTIAL CRITERIA ELEVATION: -2 TO 0 FEET ZONES 1, 2, 3, AND 4 NAVAL SUBMARINE BASE - NEW LONDON GROTON, CONNECTICUT	APPROVED BY _ DATE --
COST/SCHED-AREA			APPROVED BY -- DATE --
SCALE AS NOTED			FIGURE NO. FIGURE B-74 REV.

P:\GIS\NLON\MXD\ZONE1234_LEAD\ZONE1234_LEAD_RES_UNCERTAINTY_-4TO-2FT.MXD 6/22/09 SP



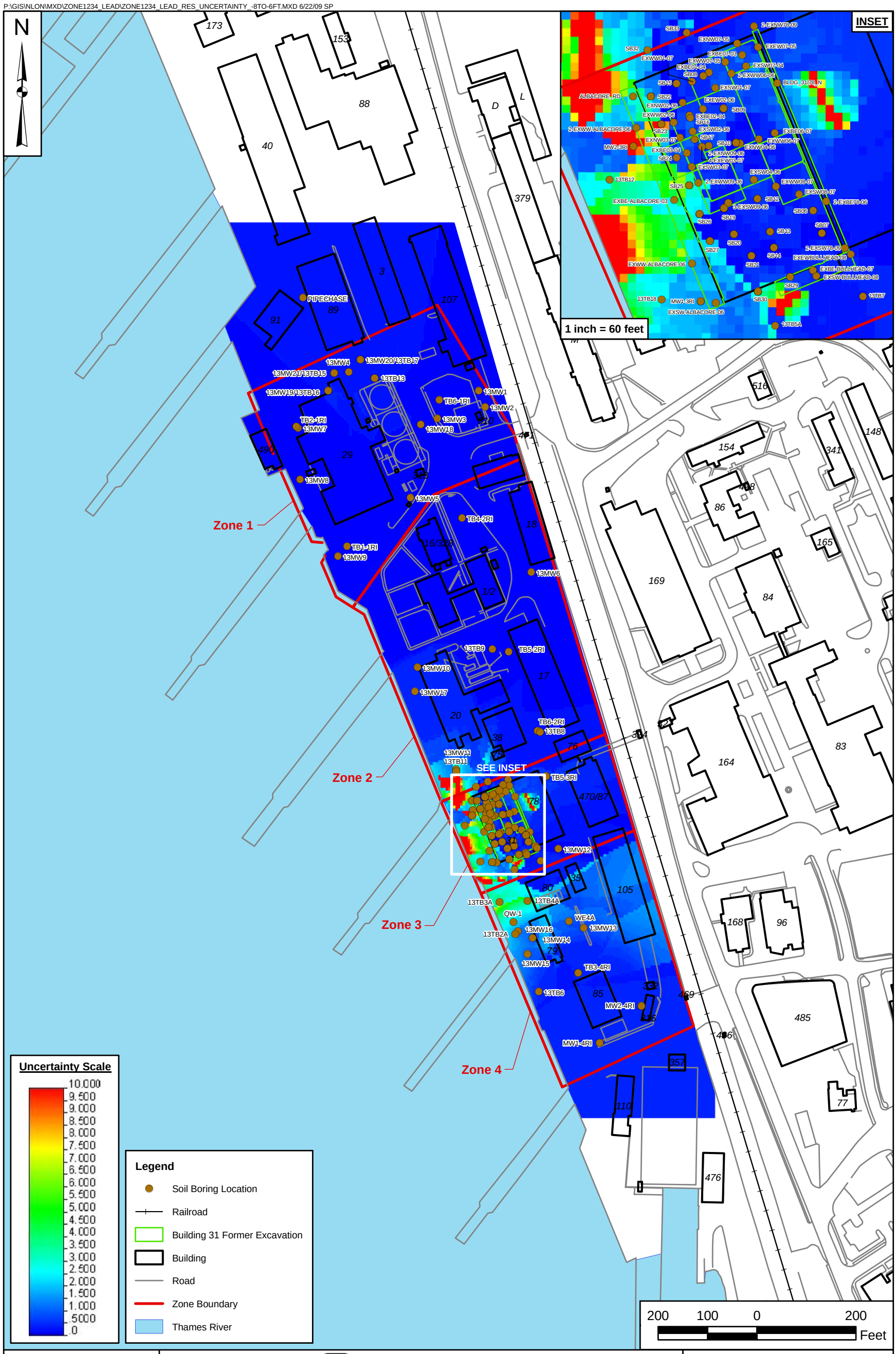
DRAWN BY	DATE	Tetra Tech NUS, Inc.	CONTRACT NO.
S. PAXTON	4/02/09		0811
CHECKED BY	DATE	LEAD UNCERTAINTY FOR RESIDENTIAL CRITERIA ELEVATION: -4 TO -2 FEET ZONES 1, 2, 3, AND 4 NAVAL SUBMARINE BASE - NEW LONDON GROTON, CONNECTICUT	APPROVED BY / DATE
N. BALSAMO	6/22/09		_ / --
COST/SCHED-AREA			APPROVED BY / DATE
			-- / --
SCALE			FIGURE NO. / REV.
AS NOTED			FIGURE B-75

P:\GIS\NLON\MXD\ZONE1234_LEAD\ZONE1234_LEAD_RES_UNCERTAINTY_-6TO-4FT.MXD 6/22/09 SP



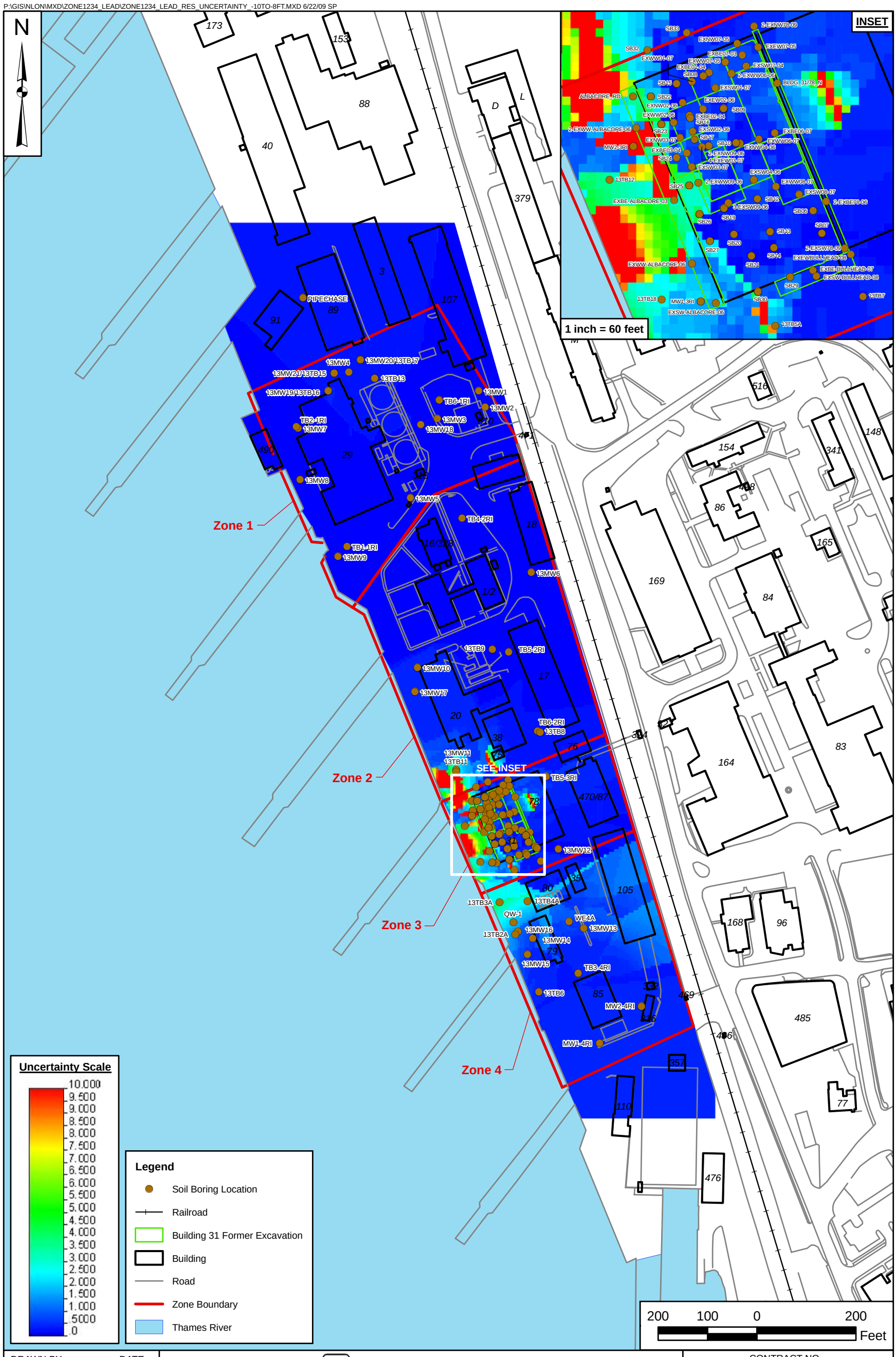
DRAWN BY S. PAXTON	DATE 4/02/09	Tetra Tech NUS, Inc.	CONTRACT NO. 0811
CHECKED BY N. BALSAMO	DATE 6/22/09	LEAD UNCERTAINTY FOR RESIDENTIAL CRITERIA ELEVATION: -6 TO -4 FEET ZONES 1, 2, 3, AND 4 NAVAL SUBMARINE BASE - NEW LONDON GROTON, CONNECTICUT	APPROVED BY _ / DATE --
COST/SCHED-AREA			APPROVED BY -- / DATE --
SCALE AS NOTED			FIGURE NO. FIGURE B-76 / REV.

P:\GIS\NLON\MXD\ZONE1234_LEAD\ZONE1234_LEAD_RES_UNCERTAINTY_-8TO-6FT.MXD 6/22/09 SP



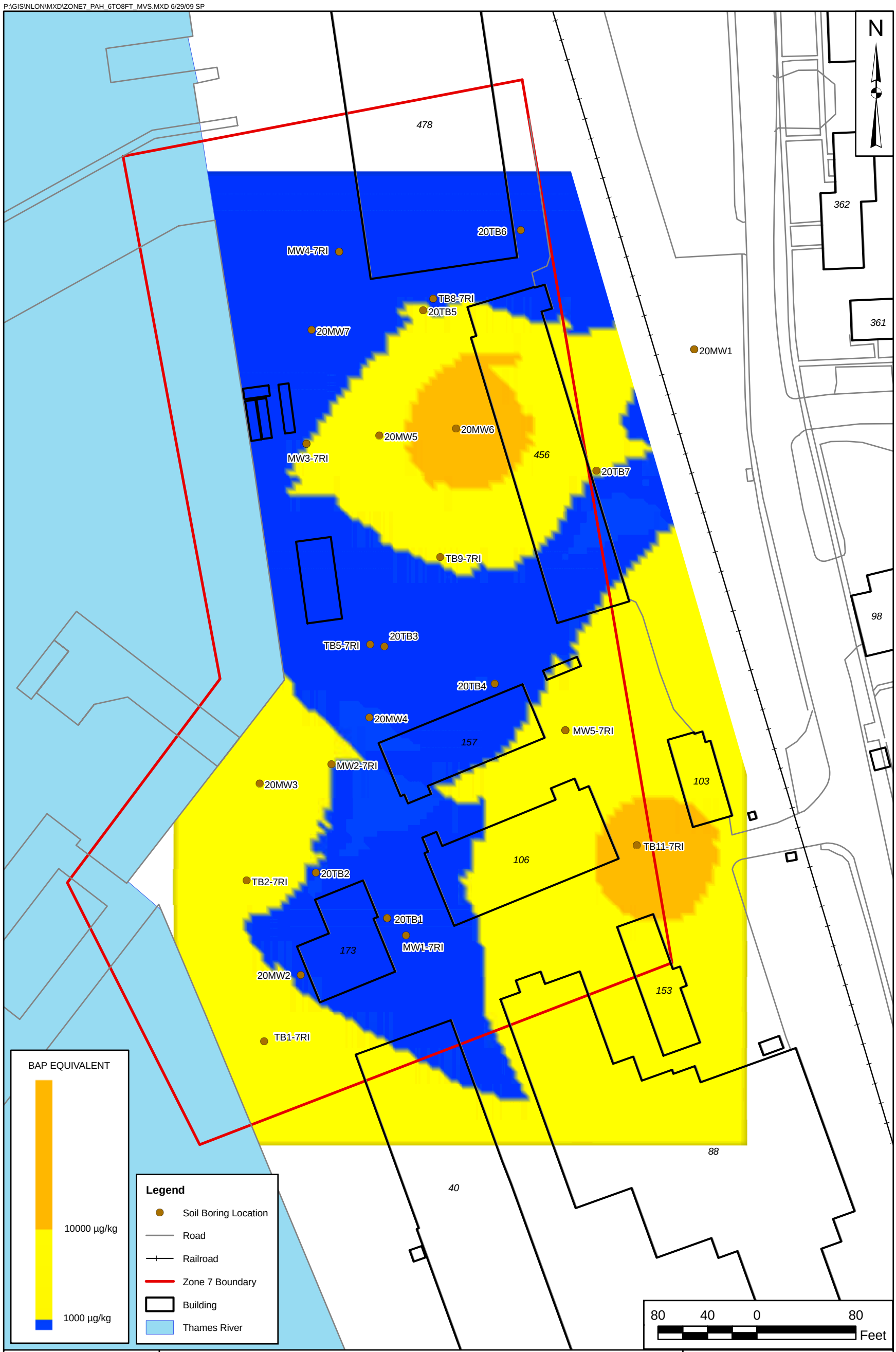
DRAWN BY S. PAXTON	DATE 4/02/09	Tetra Tech NUS, Inc.	CONTRACT NO. 0811
CHECKED BY N. BALSAMO	DATE 6/22/09	LEAD UNCERTAINTY FOR RESIDENTIAL CRITERIA ELEVATION: -8 TO -6 FEET ZONES 1, 2, 3, AND 4 NAVAL SUBMARINE BASE - NEW LONDON GROTON, CONNECTICUT	APPROVED BY _ / DATE --
COST/SCHED-AREA			APPROVED BY -- / DATE --
SCALE AS NOTED			FIGURE NO. FIGURE B-77 / REV.

P:\GIS\NLON\MXD\ZONE1234_LEAD\ZONE1234_LEAD_RES_UNCERTAINTY_-10TO-8FT.MXD 6/22/09 SP



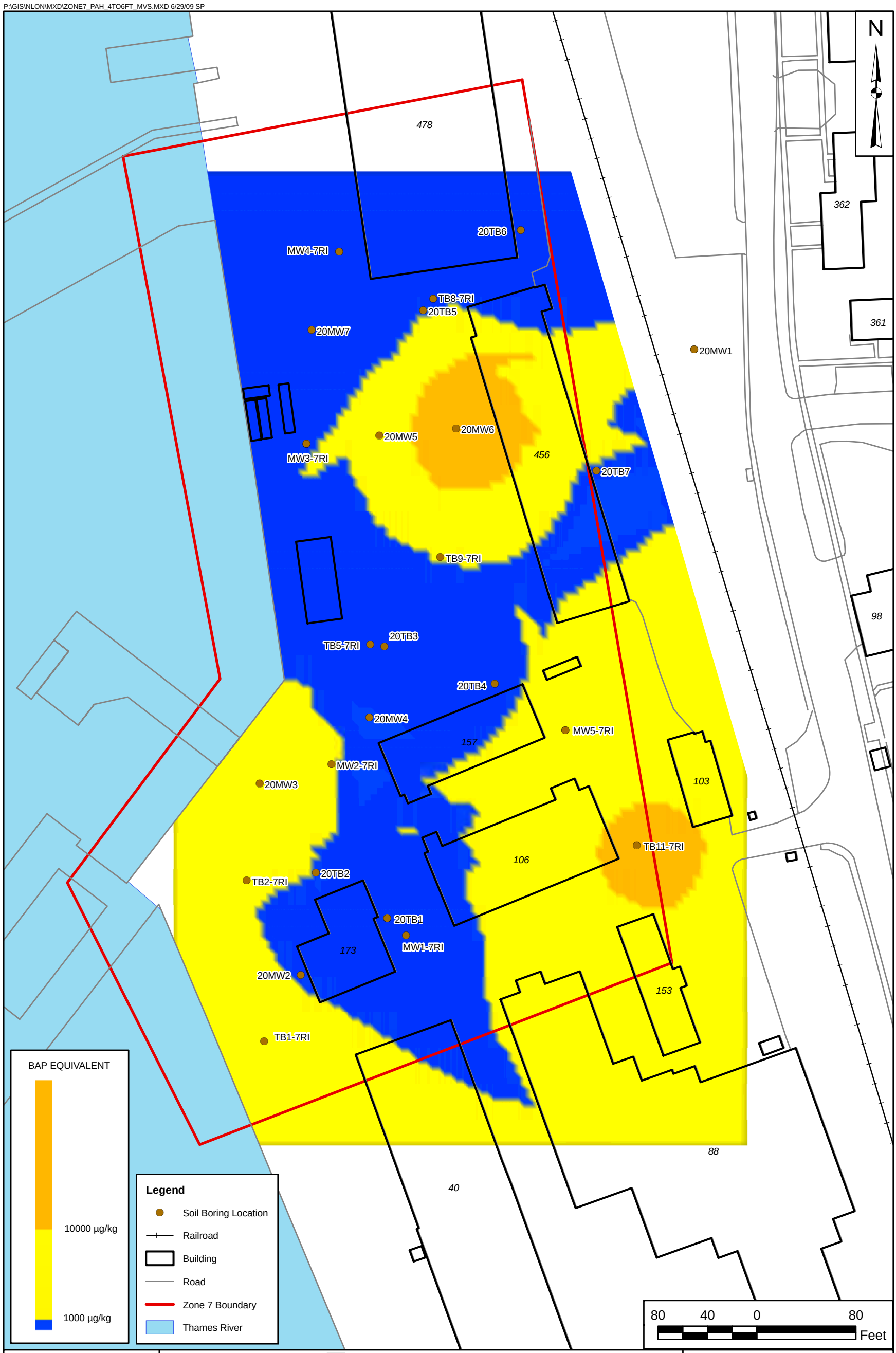
DRAWN BY	DATE	Tetra Tech NUS, Inc.	CONTRACT NO.
S. PAXTON	4/02/09		0811
CHECKED BY	DATE	LEAD UNCERTAINTY FOR RESIDENTIAL CRITERIA ELEVATION: -10 TO -8 FEET ZONES 1, 2, 3, AND 4 NAVAL SUBMARINE BASE - NEW LONDON GROTON, CONNECTICUT	APPROVED BY / DATE
N. BALSAMO	6/22/09		_ / --
COST/SCHED-AREA			APPROVED BY / DATE
			-- / --
SCALE			FIGURE NO. / REV.
AS NOTED			FIGURE B-78

P:\GIS\NLON\MXD\ZONE7_PAH_6TO8FT_MVS.MXD 6/29/09 SP



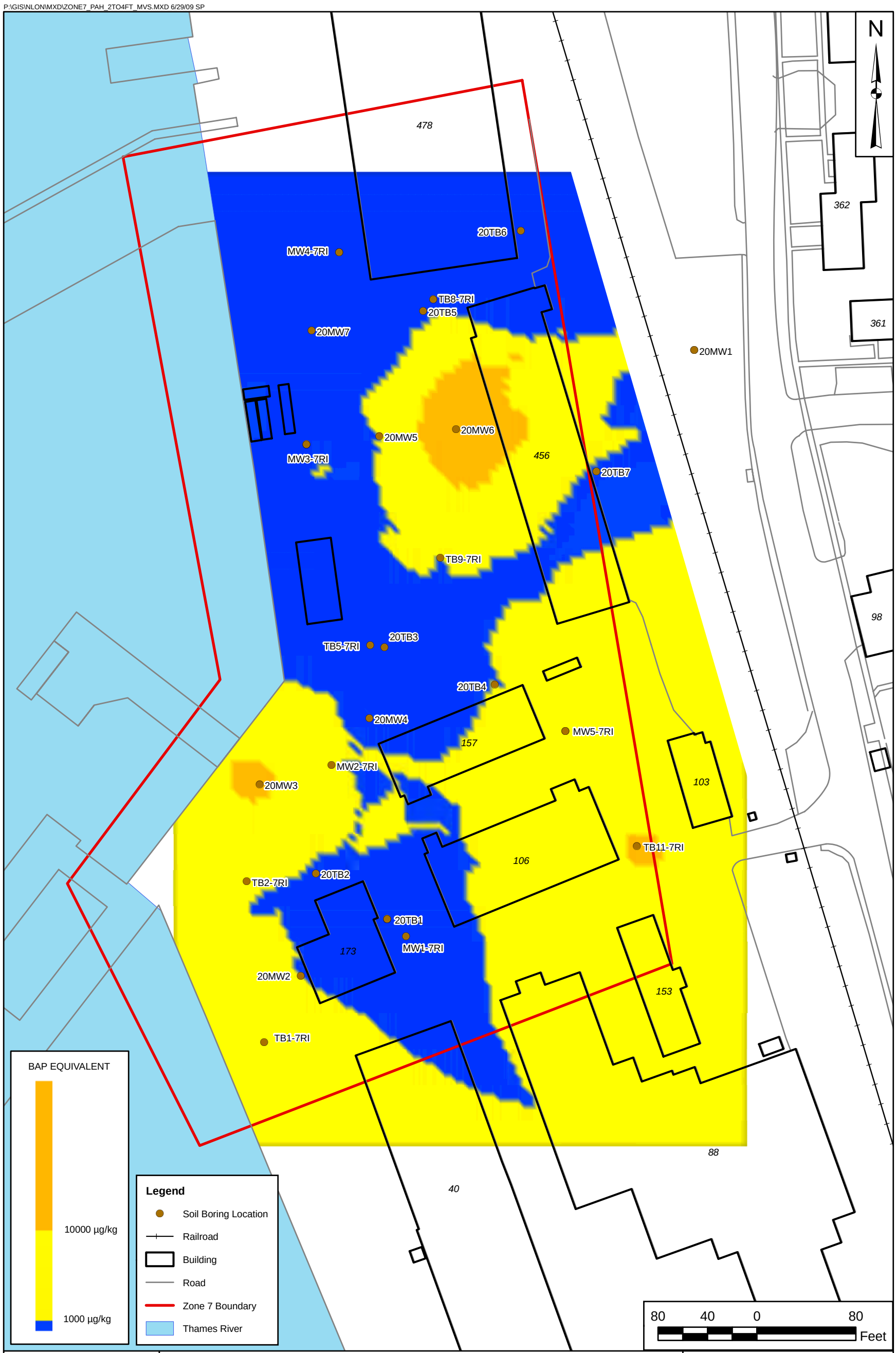
DRAWN BY S. PAXTON	DATE 4/02/09	Tetra Tech NUS, Inc.	CONTRACT NO. 0811
CHECKED BY N. BALSAMO	DATE 6/29/09	ESTIMATED BAP EQUIVALENT CONCENTRATIONS (KRIGING RESULTS) ELEVATION: 6 TO 8 FEET ZONE 7 NAVAL SUBMARINE BASE - NEW LONDON GROTON, CONNECTICUT	APPROVED BY _ / DATE --
COST/SCHED-AREA			APPROVED BY -- / DATE --
SCALE AS NOTED			FIGURE NO. FIGURE B-79 / REV.

P:\GIS\NLON\MXD\ZONE7_PAH_4TO6FT_MVS.MXD 6/29/09 SP



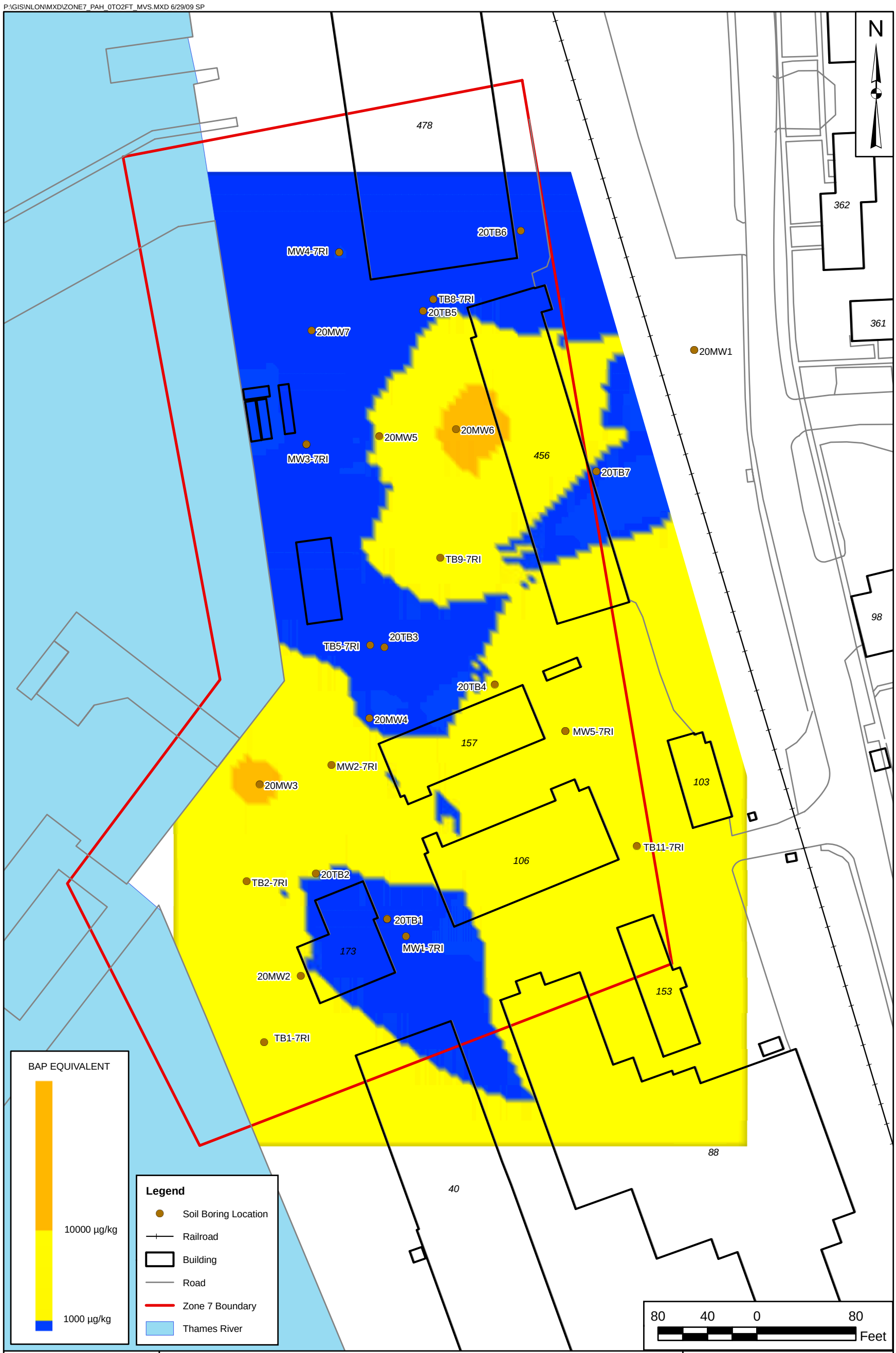
DRAWN BY S. PAXTON	DATE 4/02/09	Tetra Tech NUS, Inc.	CONTRACT NO. 0811
CHECKED BY N. BALSAMO	DATE 6/29/09	ESTIMATED BAP EQUIVALENT CONCENTRATIONS (KRIGING RESULTS) ELEVATION: 4 TO 6 FEET ZONE 7 NAVAL SUBMARINE BASE - NEW LONDON GROTON, CONNECTICUT	APPROVED BY _ / DATE --
COST/SCHED-AREA			APPROVED BY -- / DATE --
SCALE AS NOTED			FIGURE NO. FIGURE B-80 / REV.

P:\GIS\NLON\MXD\ZONE7_PAH_2TO4FT_MVS.MXD 6/29/09 SP



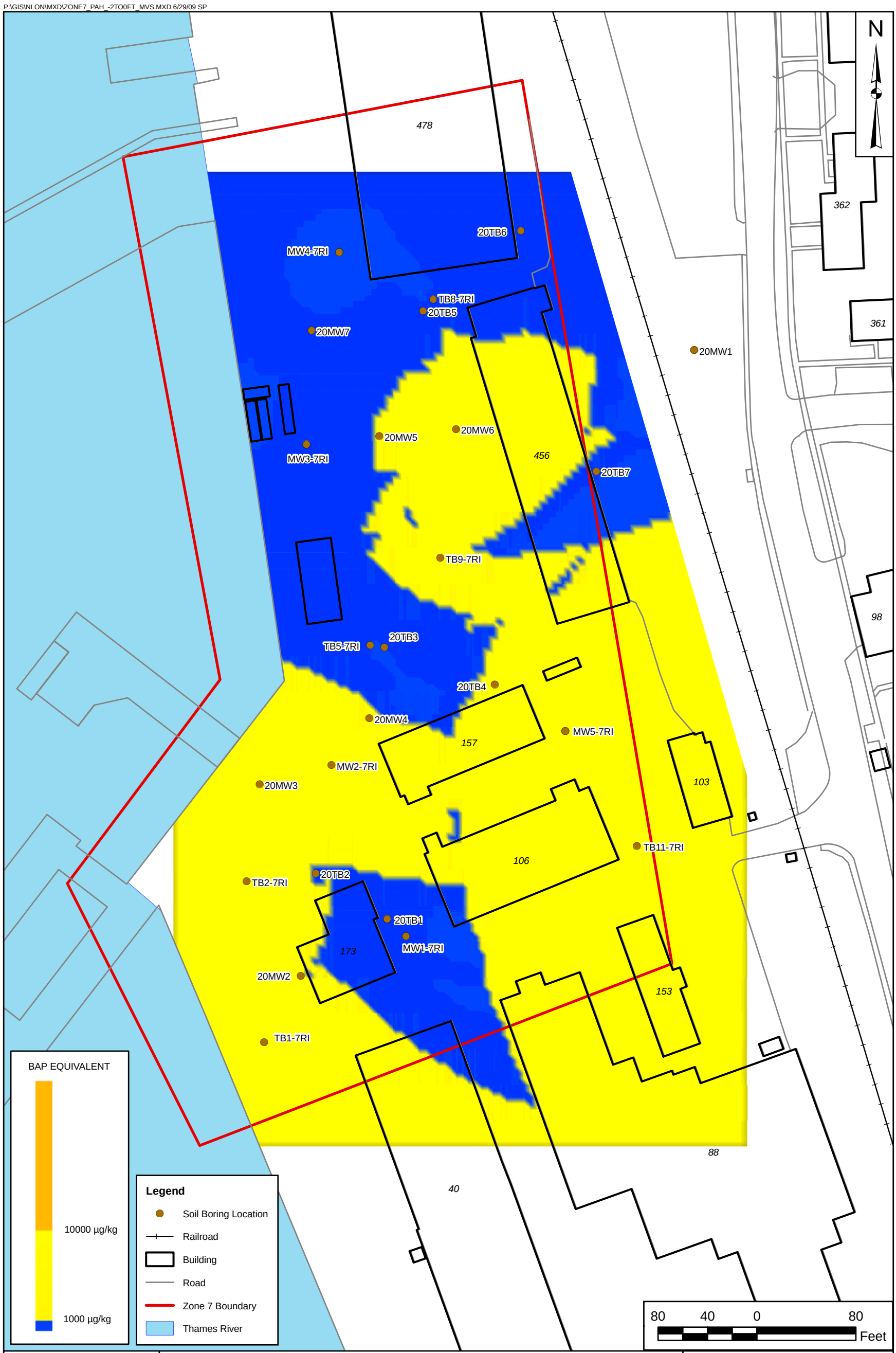
DRAWN BY S. PAXTON	DATE 4/02/09	Tetra Tech NUS, Inc.	CONTRACT NO. 0811
CHECKED BY N. BALSAMO	DATE 6/29/09	ESTIMATED BAP EQUIVALENT CONCENTRATIONS (KRIGING RESULTS) ELEVATION: 2 TO 4 FEET ZONE 7 NAVAL SUBMARINE BASE - NEW LONDON GROTON, CONNECTICUT	APPROVED BY _ / DATE --
COST/SCHED-AREA			APPROVED BY -- / DATE --
SCALE AS NOTED			FIGURE NO. FIGURE B-81 / REV.

P:\GIS\NLON\MXD\ZONE7_PAH_0TO2FT_MVS.MXD 6/29/09 SP



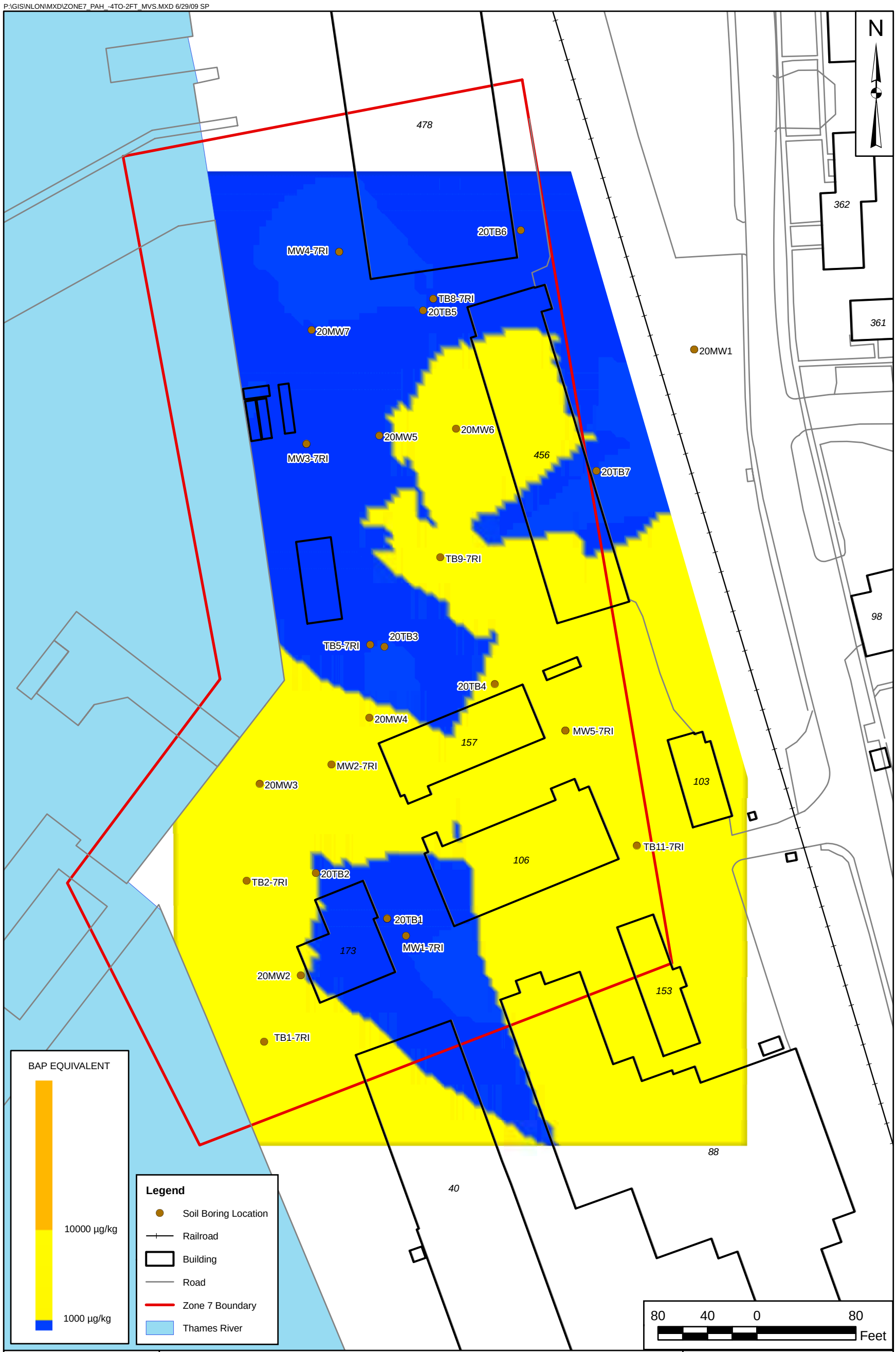
DRAWN BY S. PAXTON	DATE 4/02/09	Tetra Tech NUS, Inc.	CONTRACT NO. 0811
CHECKED BY N. BALSAMO	DATE 6/29/09	ESTIMATED BAP EQUIVALENT CONCENTRATIONS (KRIGING RESULTS) ELEVATION: 0 TO 2 FEET ZONE 7 NAVAL SUBMARINE BASE - NEW LONDON GROTON, CONNECTICUT	APPROVED BY _ / DATE --
COST/SCHED-AREA			APPROVED BY -- / DATE --
SCALE AS NOTED			FIGURE NO. FIGURE B-82 / REV.

P:\GIS\NLON\MXD\ZONE7_PAH_-2TO0FT_MVS.MXD 6/29/09 SP



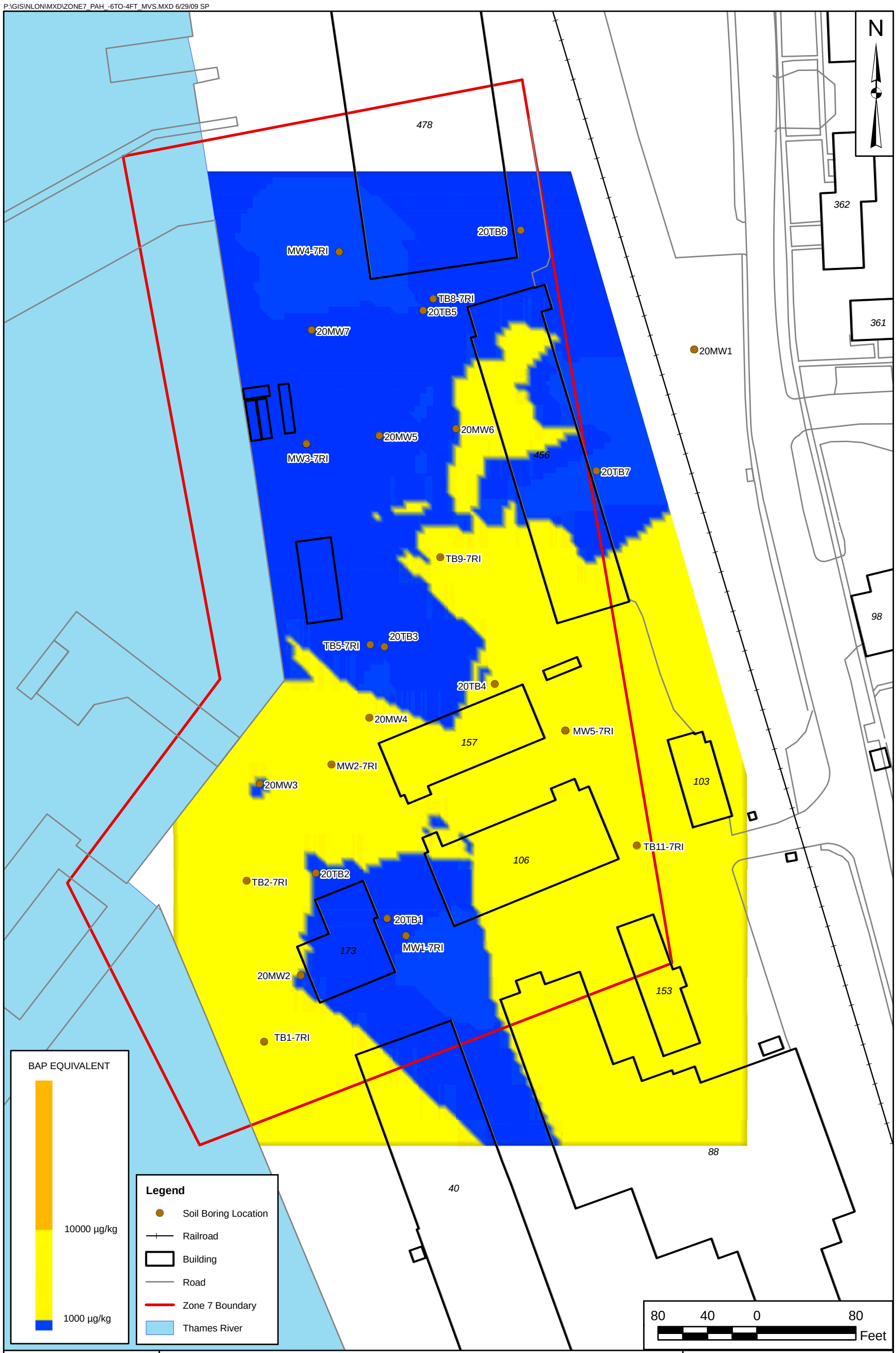
DRAWN BY S. PAXTON	DATE 4/02/09	Tetra Tech NUS, Inc.	CONTRACT NO. 0811
CHECKED BY N. BALSAMO	DATE 6/29/09	ESTIMATED BAP EQUIVALENT CONCENTRATIONS (KRIGING RESULTS) ELEVATION: -2 TO 0 FEET ZONE 7 NAVAL SUBMARINE BASE - NEW LONDON GROTON, CONNECTICUT	APPROVED BY _ / DATE --
COST/SCHED-AREA			APPROVED BY -- / DATE --
SCALE AS NOTED			FIGURE NO. FIGURE B-83 / REV.

P:\GIS\NLON\MXD\ZONE7_PAH_-4TO-2FT_MVS.MXD 6/29/09 SP



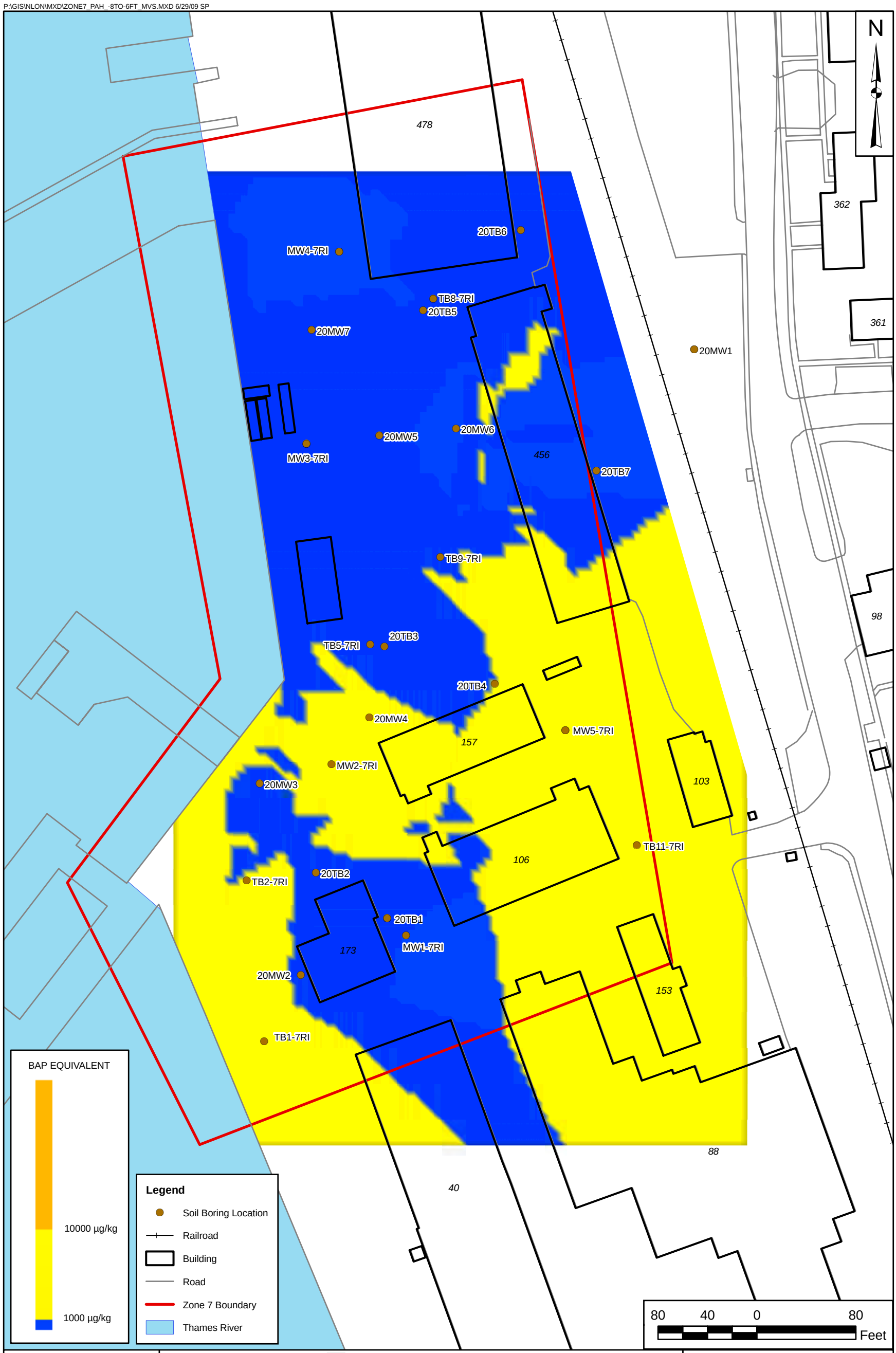
DRAWN BY S. PAXTON	DATE 4/02/09	Tetra Tech NUS, Inc.	CONTRACT NO. 0811
CHECKED BY N. BALSAMO	DATE 6/29/09	ESTIMATED BAP EQUIVALENT CONCENTRATIONS (KRIGING RESULTS) ELEVATION: -4 TO -2 FEET ZONE 7 NAVAL SUBMARINE BASE - NEW LONDON GROTON, CONNECTICUT	APPROVED BY _ / DATE --
COST/SCHED-AREA			APPROVED BY -- / DATE --
SCALE AS NOTED			FIGURE NO. FIGURE B-84 / REV.

P:\GIS\NLON\MXD\ZONE7_PAH_-6TO-4FT_MVS.MXD 6/29/09 SP



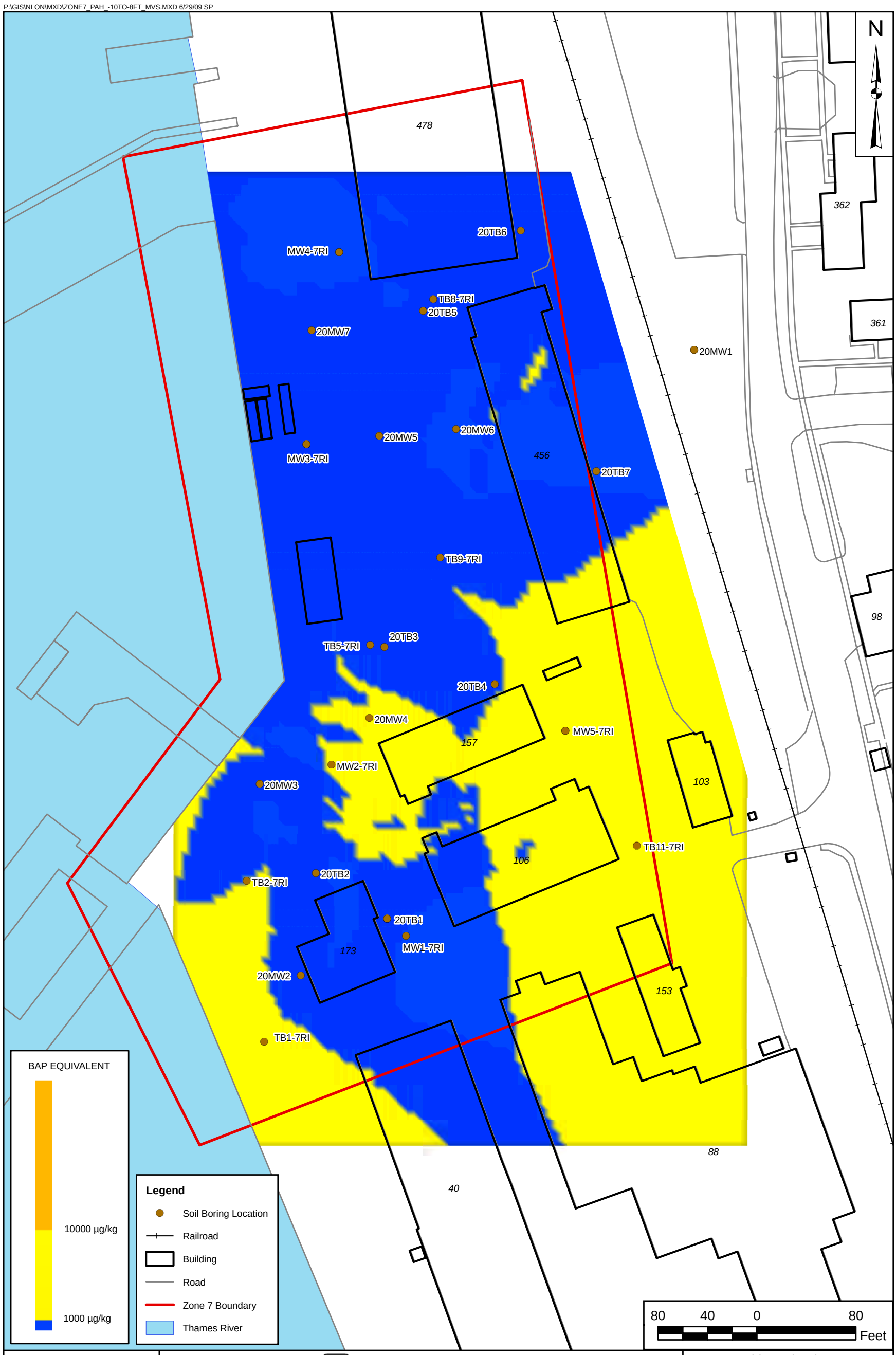
DRAWN BY S. PAXTON	DATE 4/02/09	Tetra Tech NUS, Inc.	CONTRACT NO. 0811
CHECKED BY N. BALSAMO	DATE 6/29/09	ESTIMATED BAP EQUIVALENT CONCENTRATIONS (KRIGING RESULTS) ELEVATION: -6 TO -4 FEET ZONE 7 NAVAL SUBMARINE BASE - NEW LONDON GROTON, CONNECTICUT	APPROVED BY _ / DATE --
COST/SCHED-AREA			APPROVED BY -- / DATE --
SCALE AS NOTED			FIGURE NO. FIGURE B-85 / REV.

P:\GIS\NLON\MXD\ZONE7_PAH_-8TO-6FT_MVS.MXD 6/29/09 SP



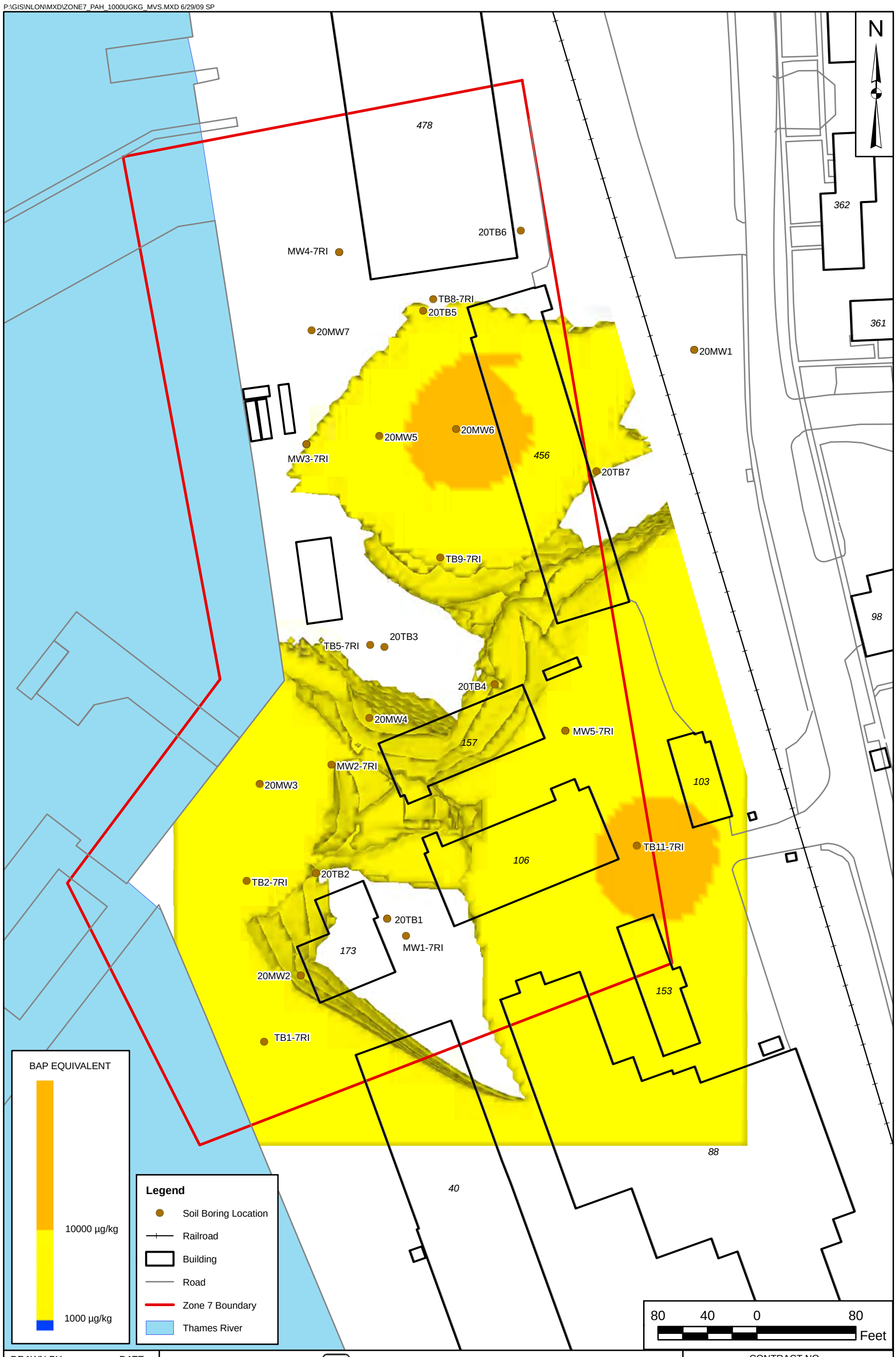
DRAWN BY S. PAXTON	DATE 4/02/09	Tetra Tech NUS, Inc.	CONTRACT NO. 0811
CHECKED BY N. BALSAMO	DATE 6/29/09	ESTIMATED BAP EQUIVALENT CONCENTRATIONS (KRIGING RESULTS) ELEVATION: -8 TO -6 FEET ZONE 7 NAVAL SUBMARINE BASE - NEW LONDON GROTON, CONNECTICUT	APPROVED BY _ / DATE --
COST/SCHED-AREA			APPROVED BY -- / DATE --
SCALE AS NOTED			FIGURE NO. FIGURE B-86 / REV.

P:\GIS\NLON\MXD\ZONE7_PAH_-10TO-8FT_MVS.MXD 6/29/09 SP



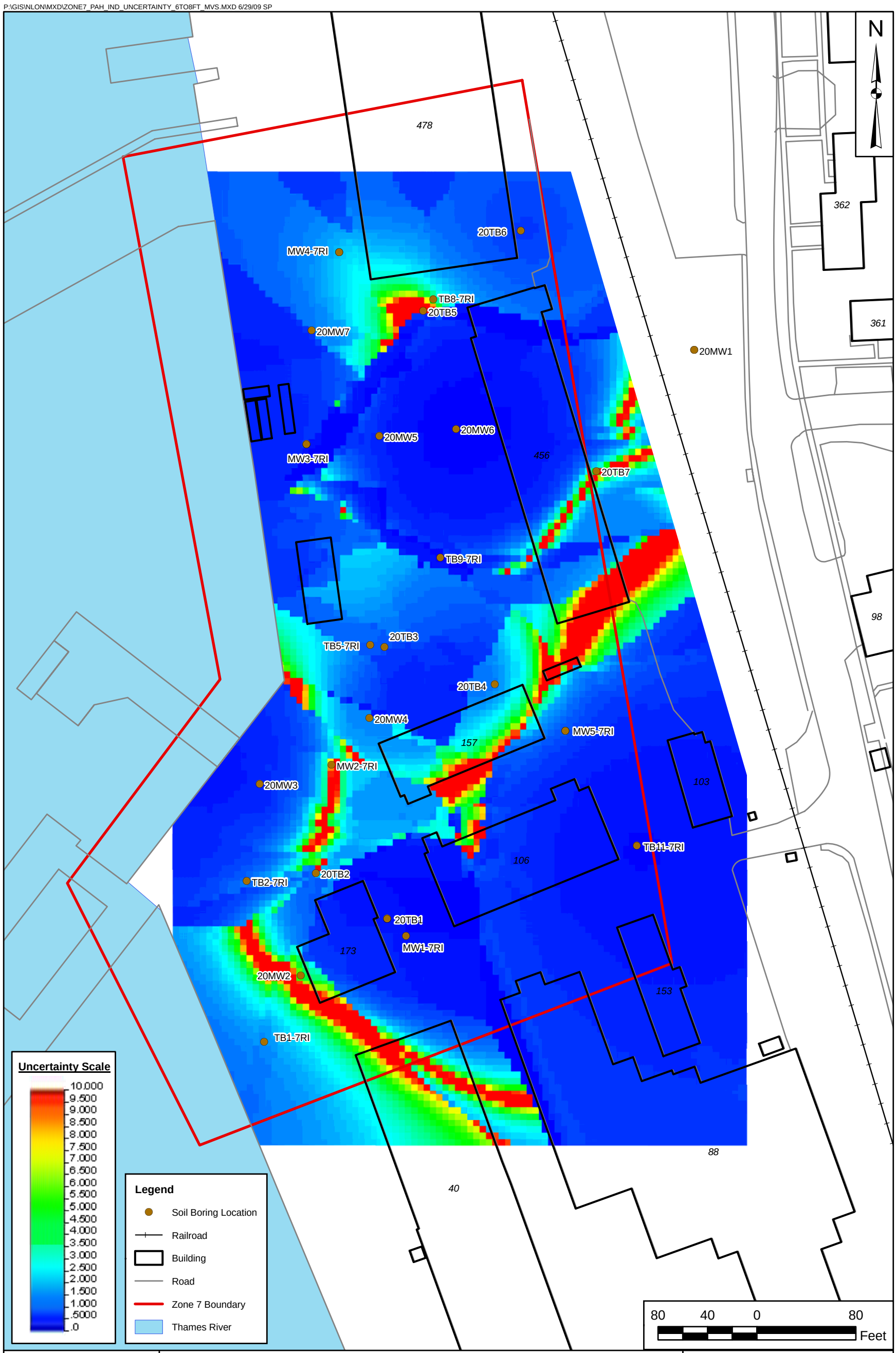
DRAWN BY S. PAXTON	DATE 4/02/09	Tetra Tech NUS, Inc.	CONTRACT NO. 0811
CHECKED BY N. BALSAMO	DATE 6/29/09	ESTIMATED BAP EQUIVALENT CONCENTRATIONS (KRIGING RESULTS) ELEVATION: -10 TO -8 FEET ZONE 7 NAVAL SUBMARINE BASE - NEW LONDON GROTON, CONNECTICUT	APPROVED BY _ / DATE --
COST/SCHED-AREA			APPROVED BY -- / DATE --
SCALE AS NOTED			FIGURE NO. FIGURE B-87 / REV.

P:\GIS\NLON\MXD\ZONE7_PAH_1000UGKG_MVS.MXD 6/29/09 SP



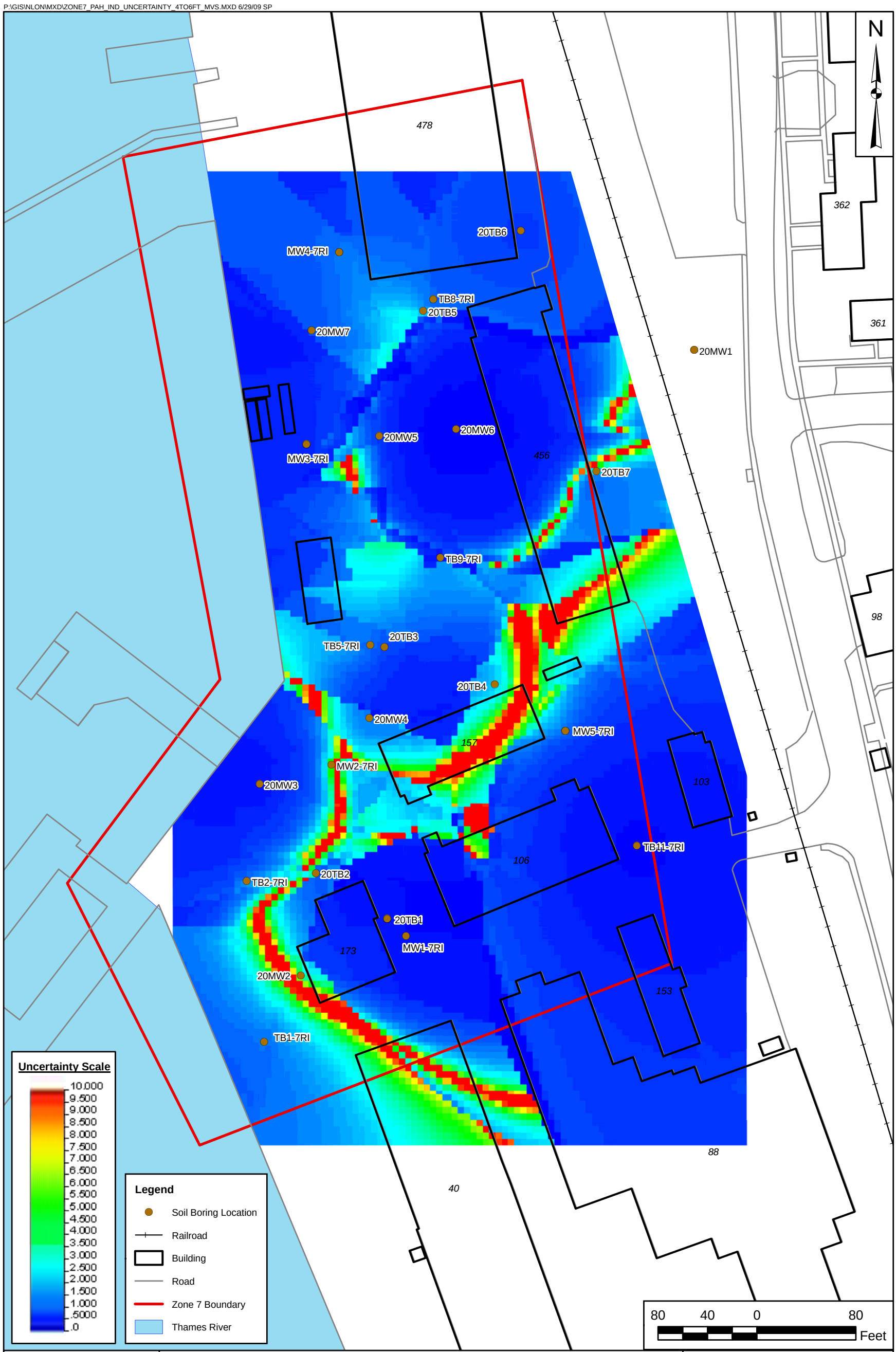
DRAWN BY S. PAXTON	DATE 4/02/09	Tetra Tech NUS, Inc.	CONTRACT NO. 0811
CHECKED BY N. BALSAMO	DATE 6/29/09	ESTIMATED BAP EQUIVALENT CONCENTRATIONS (KRIGING RESULTS) ELEVATION: 8 TO -10 FEET ZONE 7 NAVAL SUBMARINE BASE - NEW LONDON GROTON, CONNECTICUT	APPROVED BY _ / DATE --
COST/SCHED-AREA			APPROVED BY -- / DATE --
SCALE AS NOTED			FIGURE NO. FIGURE B-88 / REV.

P:\GIS\NLON\MXD\ZONE7_PAH_IND_UNCERTAINTY_6TO8FT_MVS.MXD 6/29/09 SP



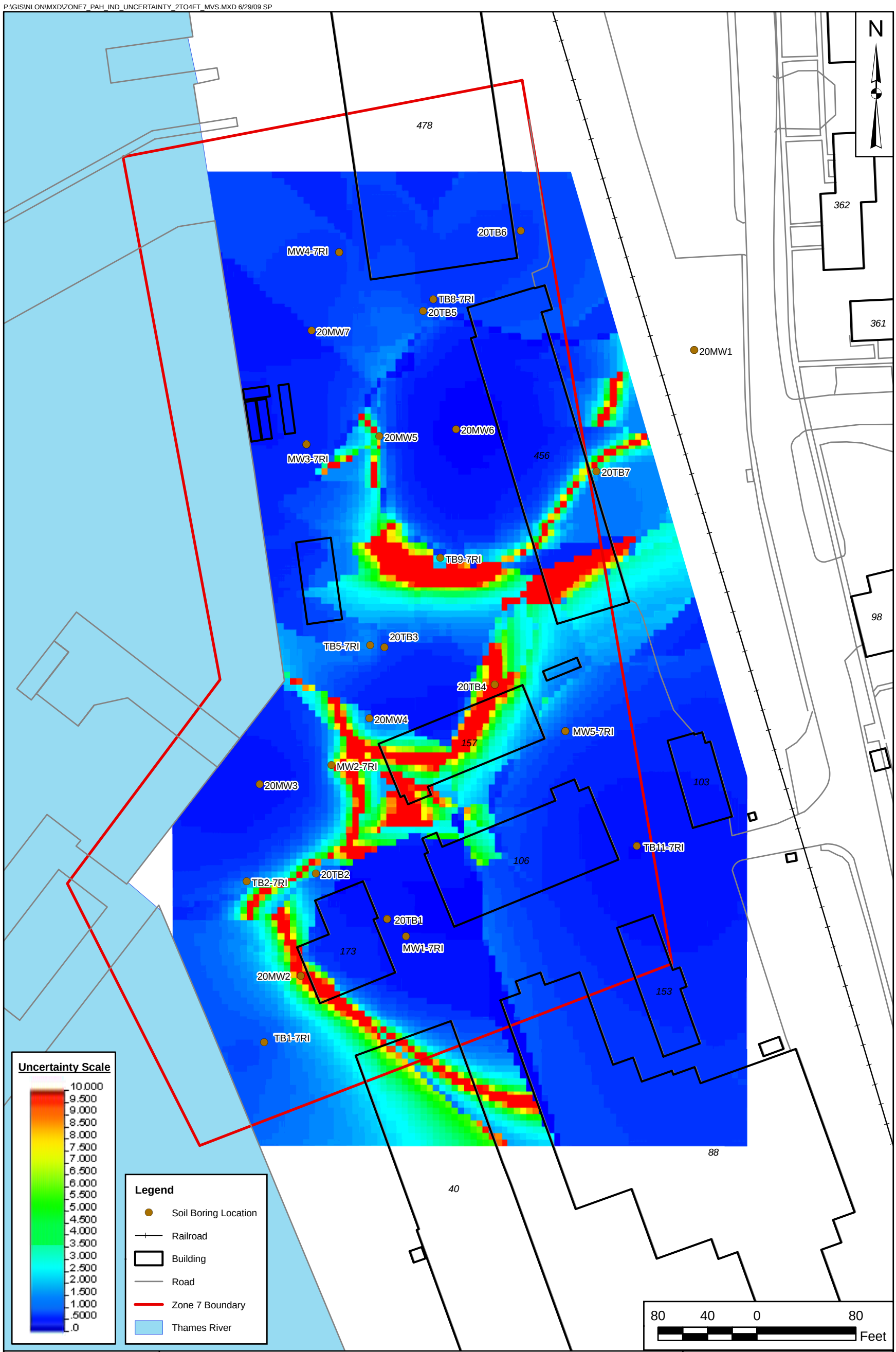
DRAWN BY S. PAXTON	DATE 4/02/09	Tetra Tech NUS, Inc.	CONTRACT NO. 0811
CHECKED BY N. BALSAMO	DATE 6/29/09	BAP EQUIVALENT UNCERTAINTY FOR 1000 UG/KG ELEVATION: 6 TO 8 FEET ZONE 7 NAVAL SUBMARINE BASE - NEW LONDON GROTON, CONNECTICUT	APPROVED BY _ / DATE --
COST/SCHED-AREA			APPROVED BY -- / DATE --
SCALE AS NOTED			FIGURE NO. FIGURE B-89 / REV.

P:\GIS\NLON\MXD\ZONE7_PAH_IND_UNCERTAINTY_4TO6FT_MVS.MXD 6/29/09 SP



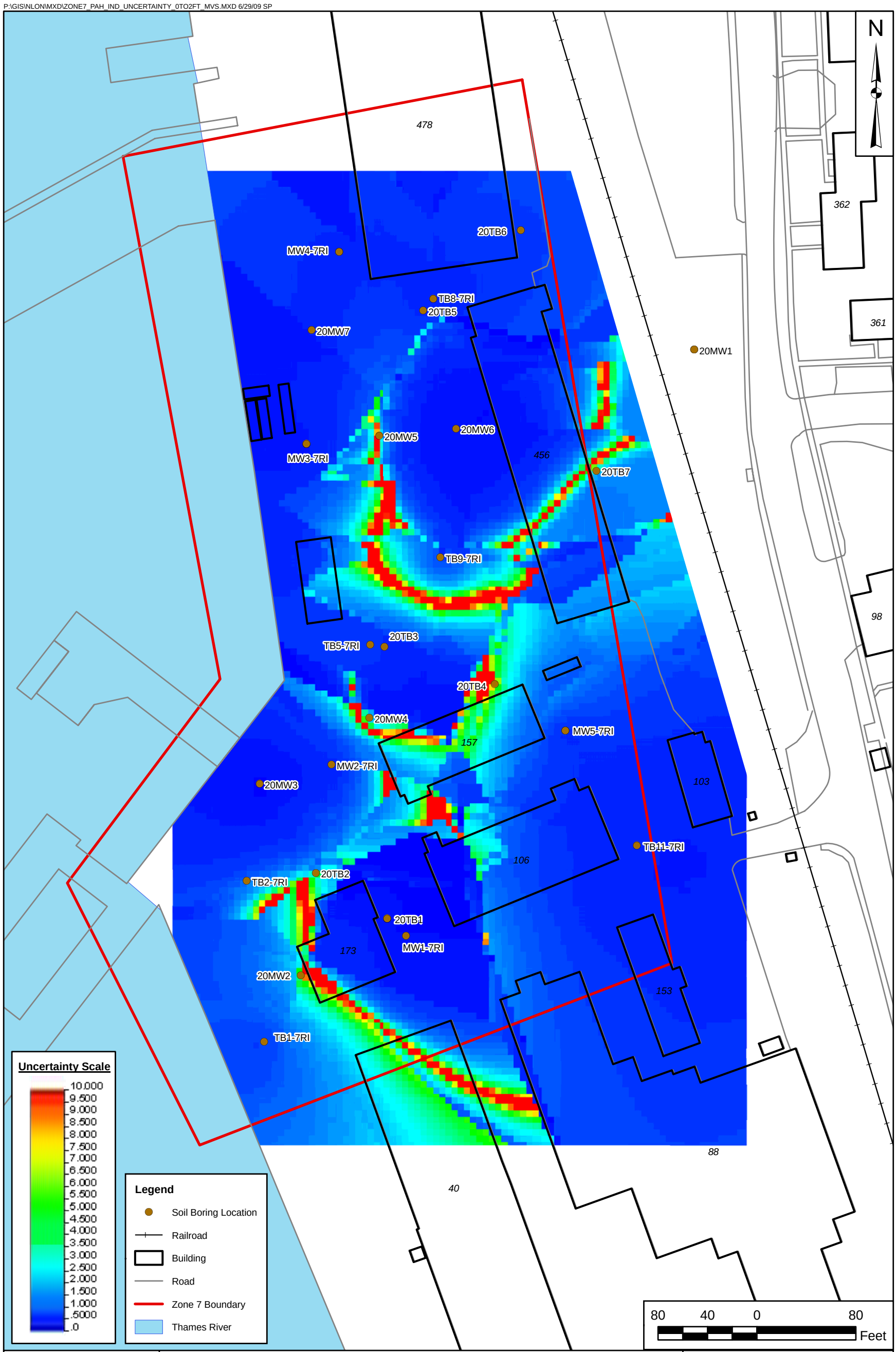
DRAWN BY S. PAXTON	DATE 4/02/09	Tetra Tech NUS, Inc.	CONTRACT NO. 0811
CHECKED BY N. BALSAMO	DATE 6/29/09	BAP EQUIVALENT UNCERTAINTY FOR 1000 UG/KG ELEVATION: 4 TO 6 FEET ZONE 7 NAVAL SUBMARINE BASE - NEW LONDON GROTON, CONNECTICUT	APPROVED BY _ / DATE --
COST/SCHED-AREA			APPROVED BY -- / DATE --
SCALE AS NOTED			FIGURE NO. FIGURE B-90 / REV.

P:\GIS\NLON\MXD\ZONE7_PAH_IND_UNCERTAINTY_2TO4FT_MVS.MXD 6/29/09 SP



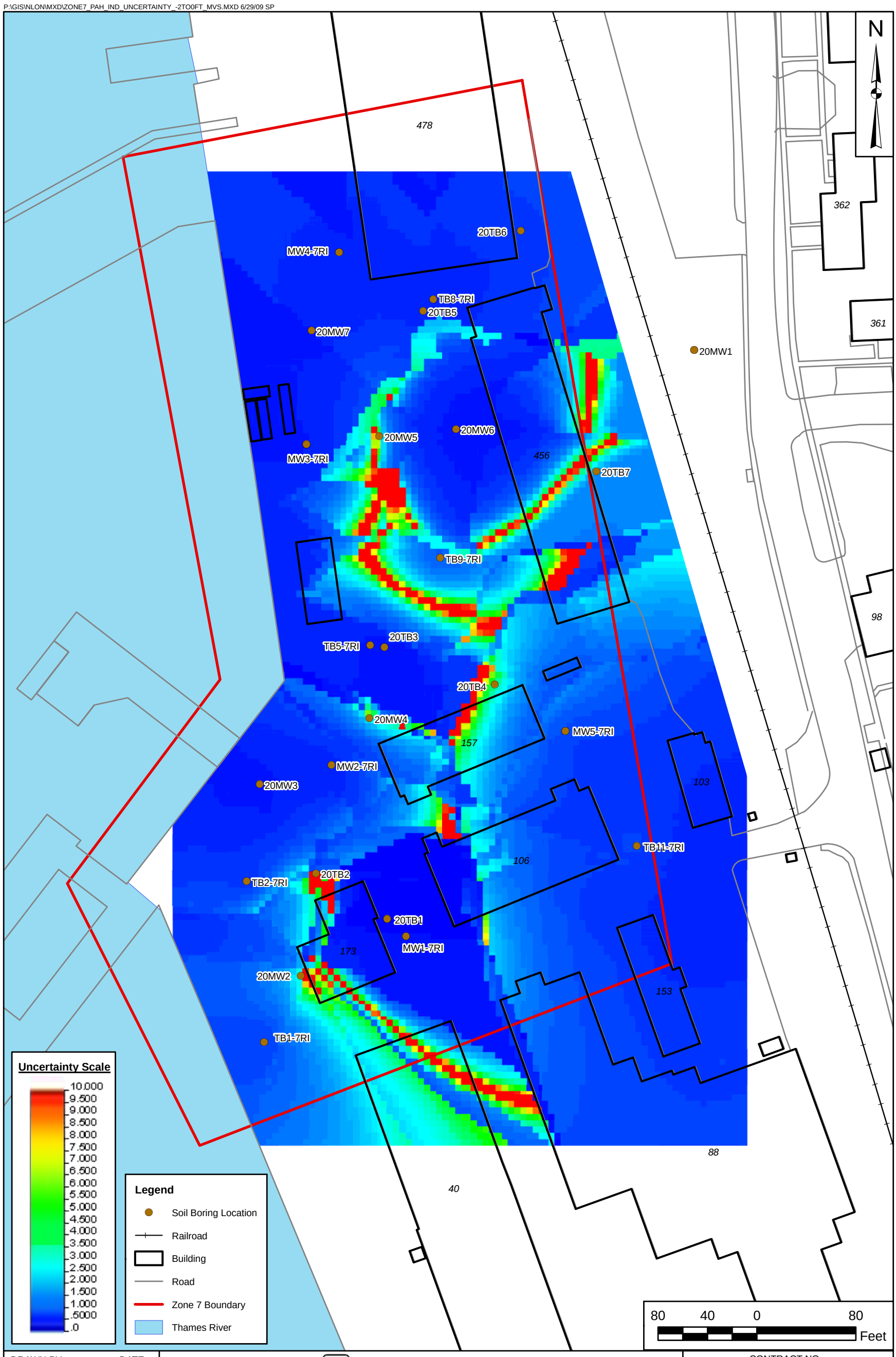
DRAWN BY S. PAXTON	DATE 4/02/09	Tetra Tech NUS, Inc.	CONTRACT NO. 0811
CHECKED BY N. BALSAMO	DATE 6/29/09	BAP EQUIVALENT UNCERTAINTY FOR 1000 UG/KG ELEVATION: 2 TO 4 FEET ZONE 7 NAVAL SUBMARINE BASE - NEW LONDON GROTON, CONNECTICUT	APPROVED BY _ / DATE --
COST/SCHED-AREA			APPROVED BY -- / DATE --
SCALE AS NOTED			FIGURE NO. FIGURE B-91 / REV.

P:\GIS\NLON\MXD\ZONE7_PAH_IND_UNCERTAINTY_0TO2FT_MVS.MXD 6/29/09 SP



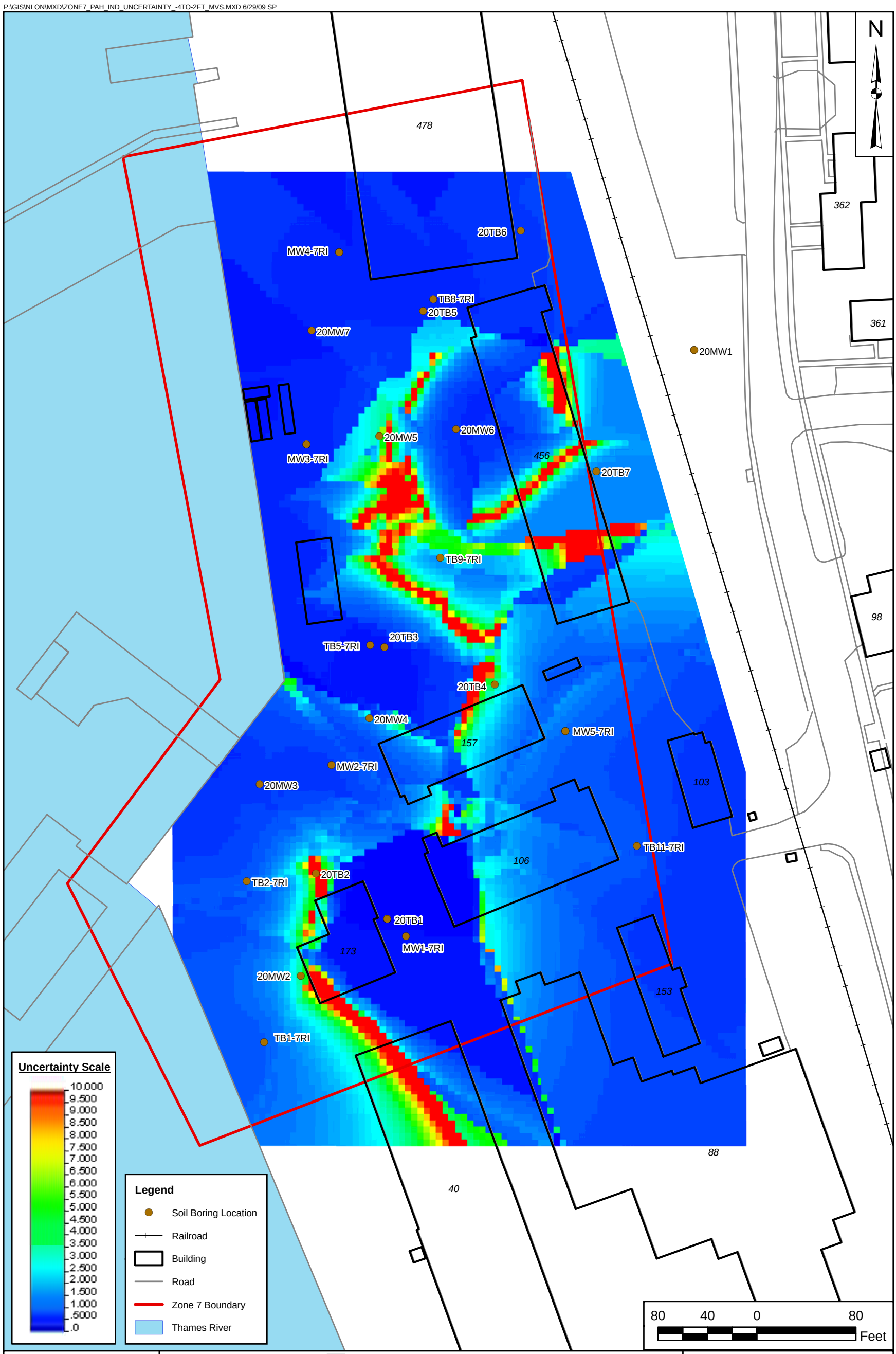
DRAWN BY S. PAXTON	DATE 4/02/09	Tetra Tech NUS, Inc.	CONTRACT NO. 0811
CHECKED BY N. BALSAMO	DATE 6/29/09	BAP EQUIVALENT UNCERTAINTY FOR 1000 UG/KG ELEVATION: 0 TO 2 FEET ZONE 7 NAVAL SUBMARINE BASE - NEW LONDON GROTON, CONNECTICUT	APPROVED BY _ / DATE --
COST/SCHED-AREA			APPROVED BY -- / DATE --
SCALE AS NOTED			FIGURE NO. FIGURE B-92 / REV.

P:\GIS\NLON\MXD\ZONE7_PAH_IND_UNCERTAINTY_-2TO0FT_MVS.MXD 6/29/09 SP



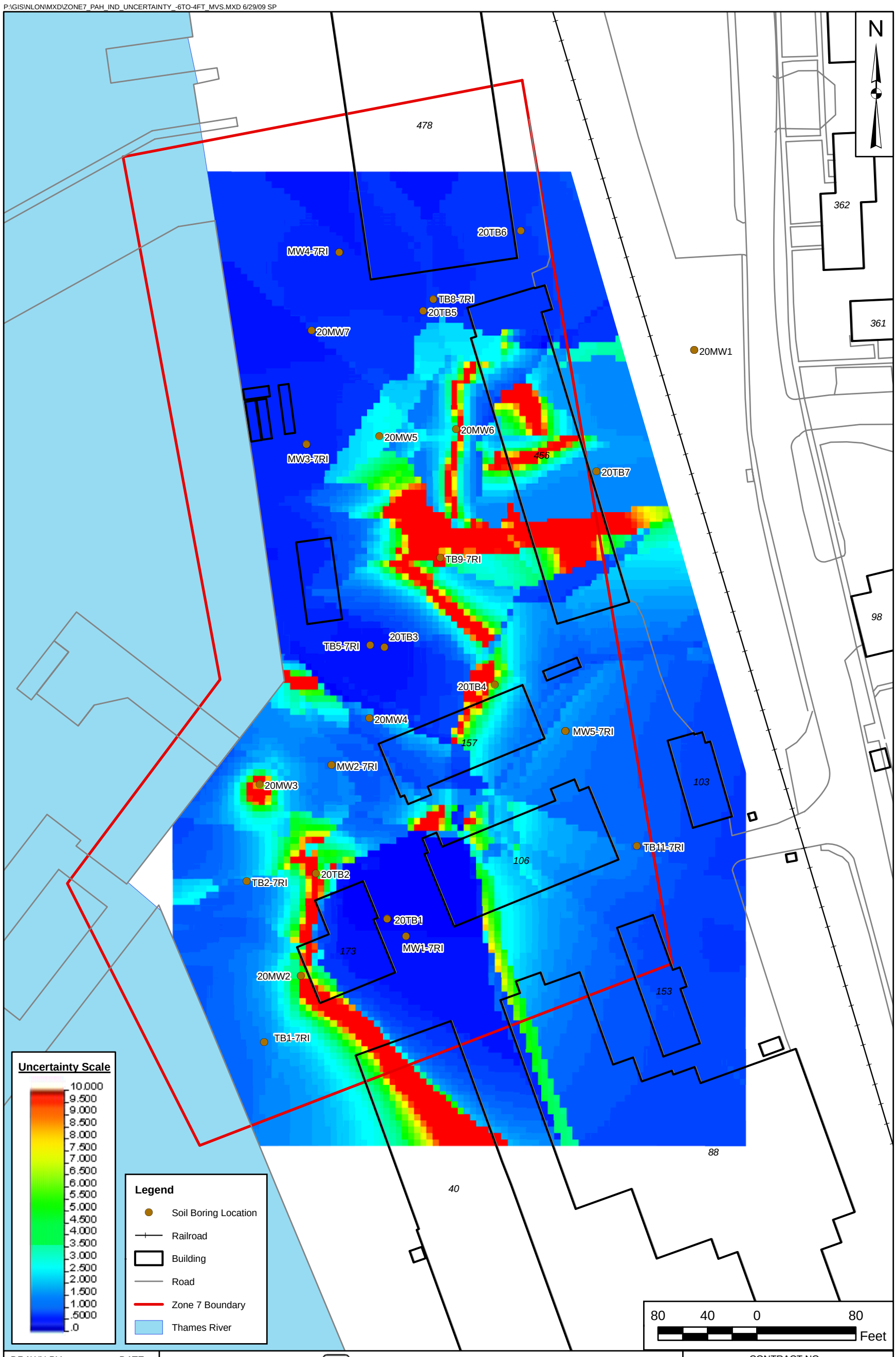
DRAWN BY S. PAXTON	DATE 4/02/09	Tetra Tech NUS, Inc.	CONTRACT NO. 0811
CHECKED BY N. BALSAMO	DATE 6/29/09	BAP EQUIVALENT UNCERTAINTY FOR 1000 UG/KG ELEVATION: -2 TO 0 FEET ZONE 7 NAVAL SUBMARINE BASE - NEW LONDON GROTON, CONNECTICUT	APPROVED BY _ / DATE --
COST/SCHED-AREA			APPROVED BY -- / DATE --
SCALE AS NOTED			FIGURE NO. FIGURE B-93 / REV.

P:\GIS\NLON\MXD\ZONE7_PAH_IND_UNCERTAINTY_-4TO-2FT_MVS.MXD 6/29/09 SP



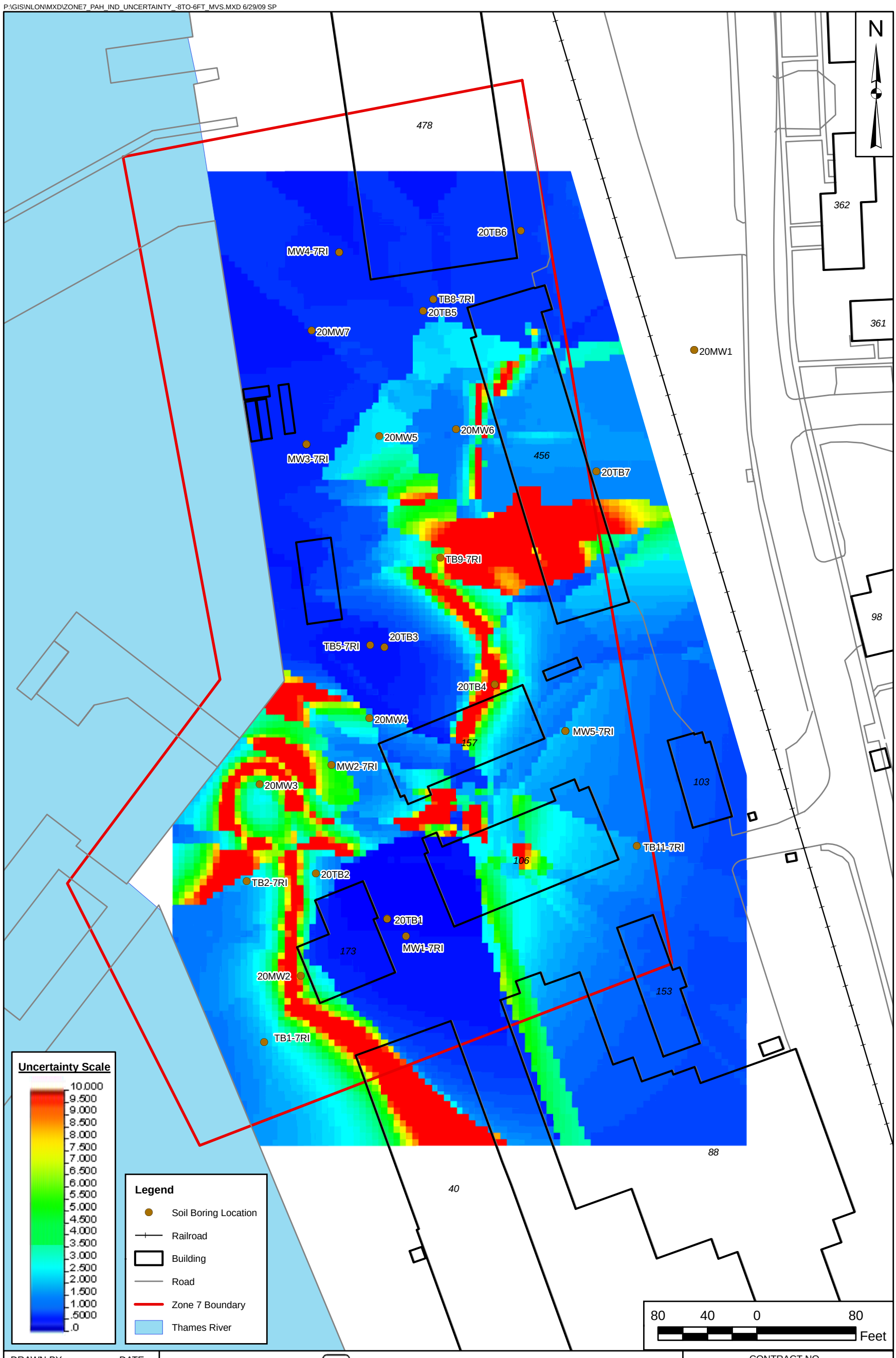
DRAWN BY	DATE	Tetra Tech NUS, Inc.	CONTRACT NO.
S. PAXTON	4/02/09		0811
CHECKED BY	DATE	BAP EQUIVALENT UNCERTAINTY FOR 1000 UG/KG ELEVATION: -4 TO -2 FEET ZONE 7 NAVAL SUBMARINE BASE - NEW LONDON GROTON, CONNECTICUT	APPROVED BY / DATE
N. BALSAMO	6/29/09		_ / --
COST/SCHED-AREA			APPROVED BY / DATE
			-- / --
SCALE			FIGURE NO. / REV.
AS NOTED			FIGURE B-94

P:\GIS\NLON\MXD\ZONE7_PAH_IND_UNCERTAINTY_-6TO-4FT_MVS.MXD 6/29/09 SP



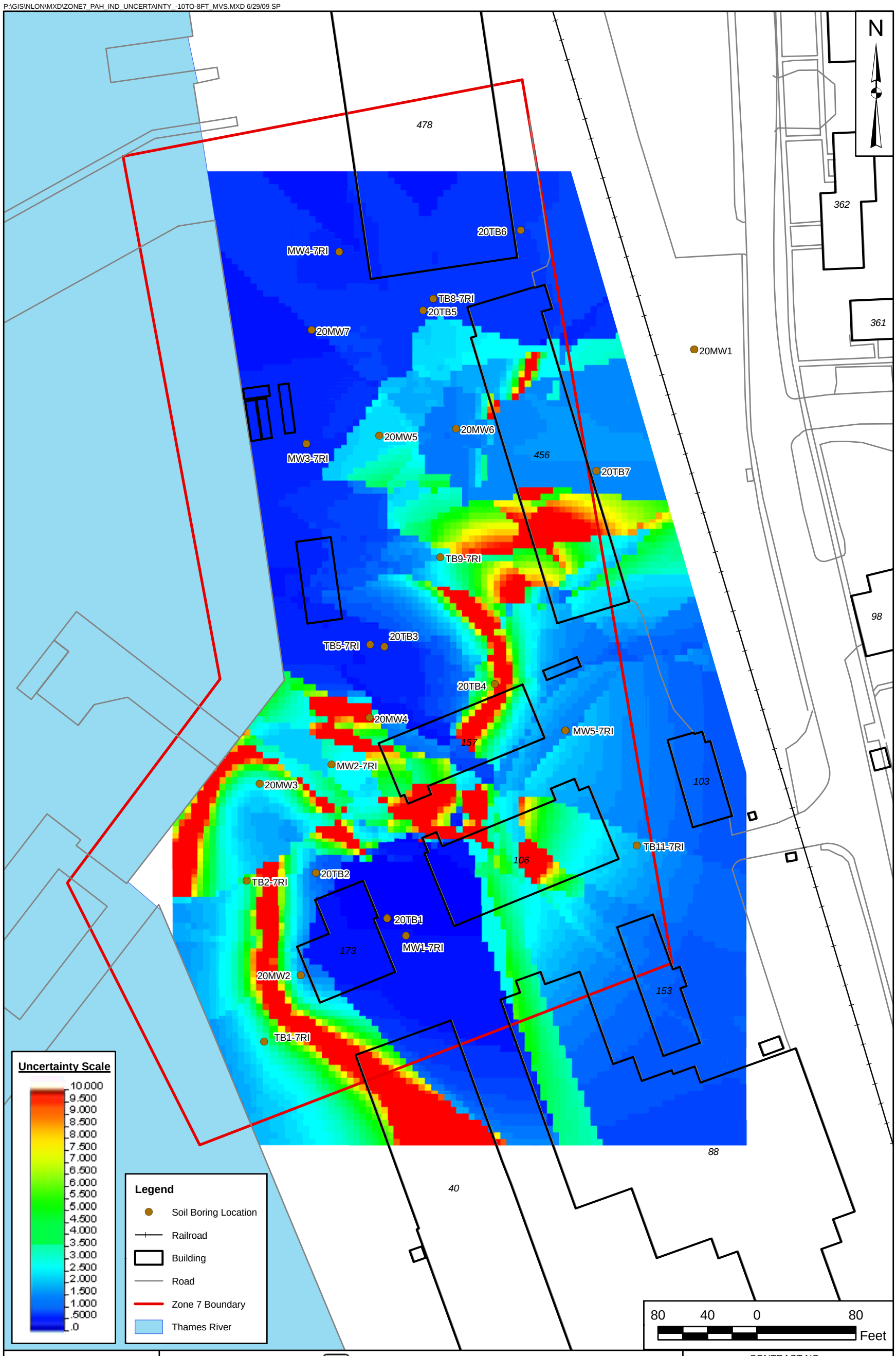
DRAWN BY	DATE	Tetra Tech NUS, Inc.	CONTRACT NO.
S. PAXTON	4/02/09		0811
CHECKED BY	DATE	BAP EQUIVALENT UNCERTAINTY FOR 1000 UG/KG ELEVATION: -6 TO -4 FEET ZONE 7 NAVAL SUBMARINE BASE - NEW LONDON GROTON, CONNECTICUT	APPROVED BY / DATE
N. BALSAMO	6/29/09		_ / --
COST/SCHED-AREA			APPROVED BY / DATE
			-- / --
SCALE			FIGURE NO. / REV.
AS NOTED			FIGURE B-95

P:\GIS\NLON\MXD\ZONE7_PAH_IND_UNCERTAINTY_-8TO-6FT_MVS.MXD 6/29/09 SP



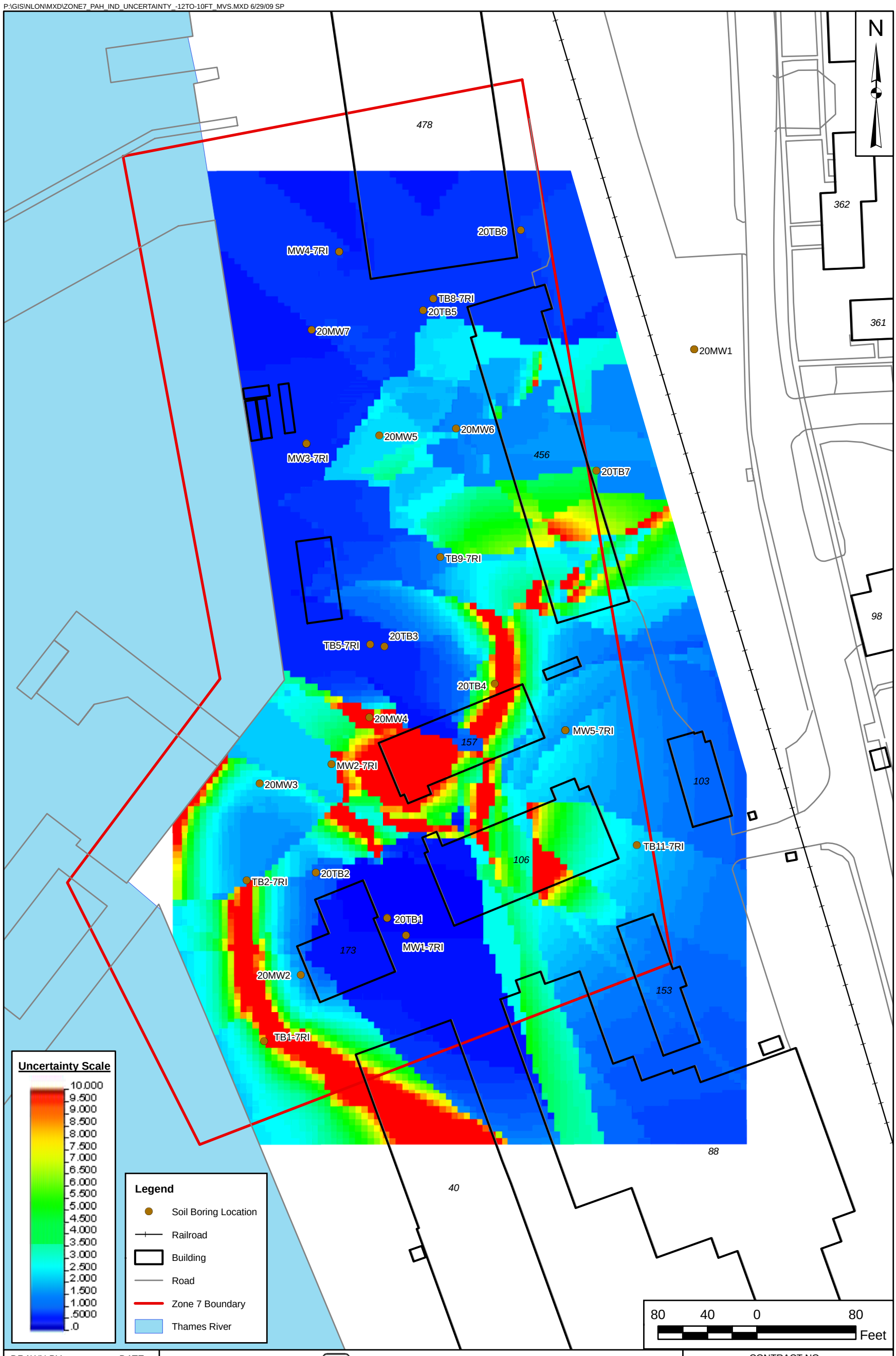
DRAWN BY S. PAXTON	DATE 4/02/09	Tetra Tech NUS, Inc.	CONTRACT NO. 0811
CHECKED BY N. BALSAMO	DATE 6/29/09	BAP EQUIVALENT UNCERTAINTY FOR 1000 UG/KG ELEVATION: -8 TO -6 FEET ZONE 7 NAVAL SUBMARINE BASE - NEW LONDON GROTON, CONNECTICUT	APPROVED BY _ / DATE --
COST/SCHED-AREA			APPROVED BY -- / DATE --
SCALE AS NOTED			FIGURE NO. FIGURE B-96 / REV.

P:\GIS\NLON\MXD\ZONE7_PAH_IND_UNCERTAINTY_-10TO-8FT_MVS.MXD 6/29/09 SP



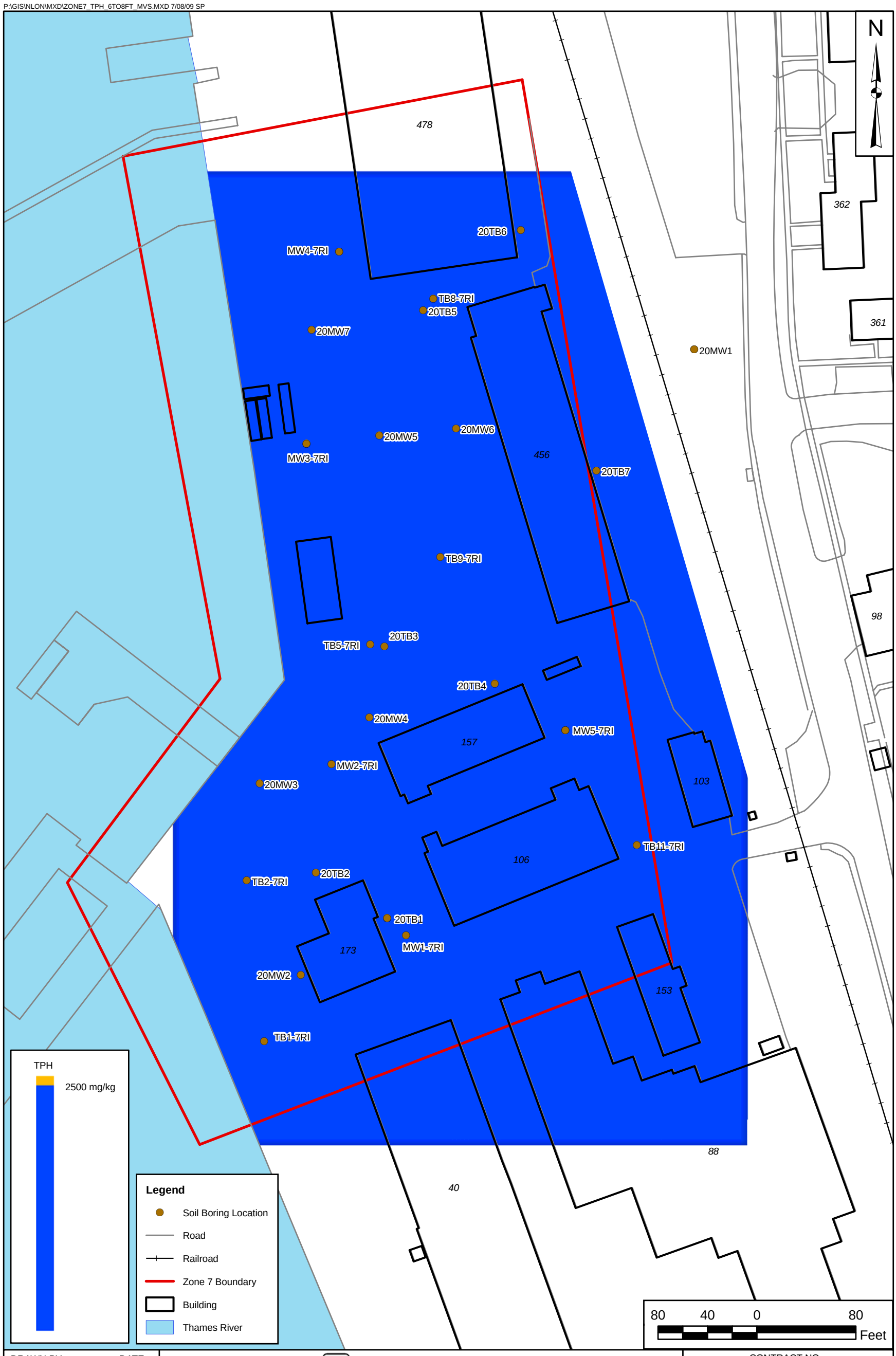
DRAWN BY S. PAXTON	DATE 4/02/09	Tetra Tech NUS, Inc.	CONTRACT NO. 0811
CHECKED BY N. BALSAMO	DATE 6/29/09	BAP EQUIVALENT UNCERTAINTY FOR 1000 UG/KG ELEVATION: -10 TO -8 FEET ZONE 7 NAVAL SUBMARINE BASE - NEW LONDON GROTON, CONNECTICUT	APPROVED BY _ / DATE --
COST/SCHED-AREA			APPROVED BY -- / DATE --
SCALE AS NOTED			FIGURE NO. FIGURE B-97 / REV.

P:\GIS\NLON\MXD\ZONE7_PAH_IND_UNCERTAINTY_-12TO-10FT_MVS.MXD 6/29/09 SP



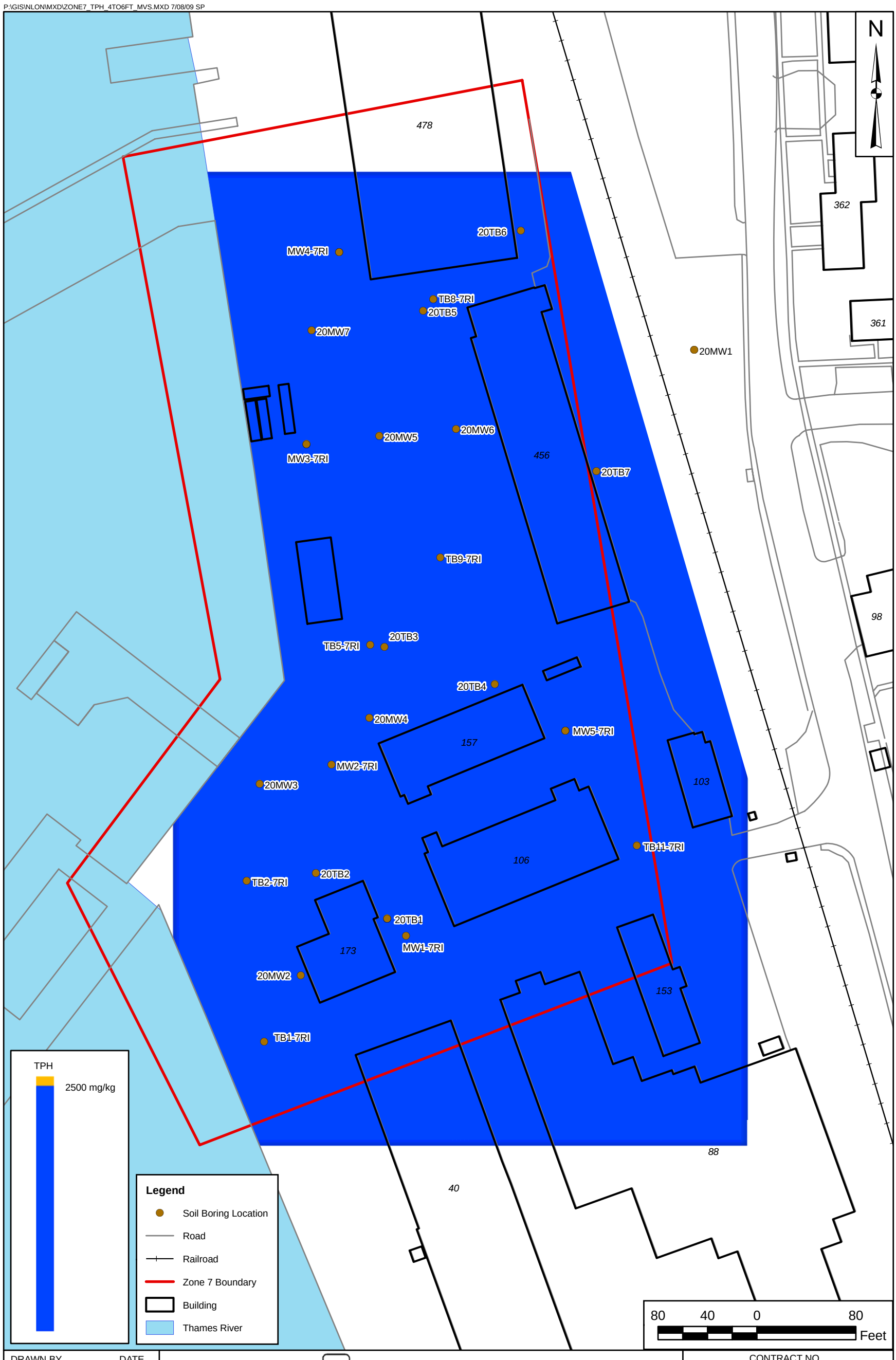
DRAWN BY S. PAXTON	DATE 4/02/09	Tetra Tech NUS, Inc.	CONTRACT NO. 0811
CHECKED BY N. BALSAMO	DATE 6/29/09	BAP EQUIVALENT UNCERTAINTY FOR 1000 UG/KG ELEVATION: -12 TO -10 FEET ZONE 7 NAVAL SUBMARINE BASE - NEW LONDON GROTON, CONNECTICUT	APPROVED BY _ / DATE --
COST/SCHED-AREA			APPROVED BY -- / DATE --
SCALE AS NOTED			FIGURE NO. FIGURE B-98 / REV.

P:\GIS\NLON\MXD\ZONE7_TPH_6TO8FT_MVS.MXD 7/08/09 SP



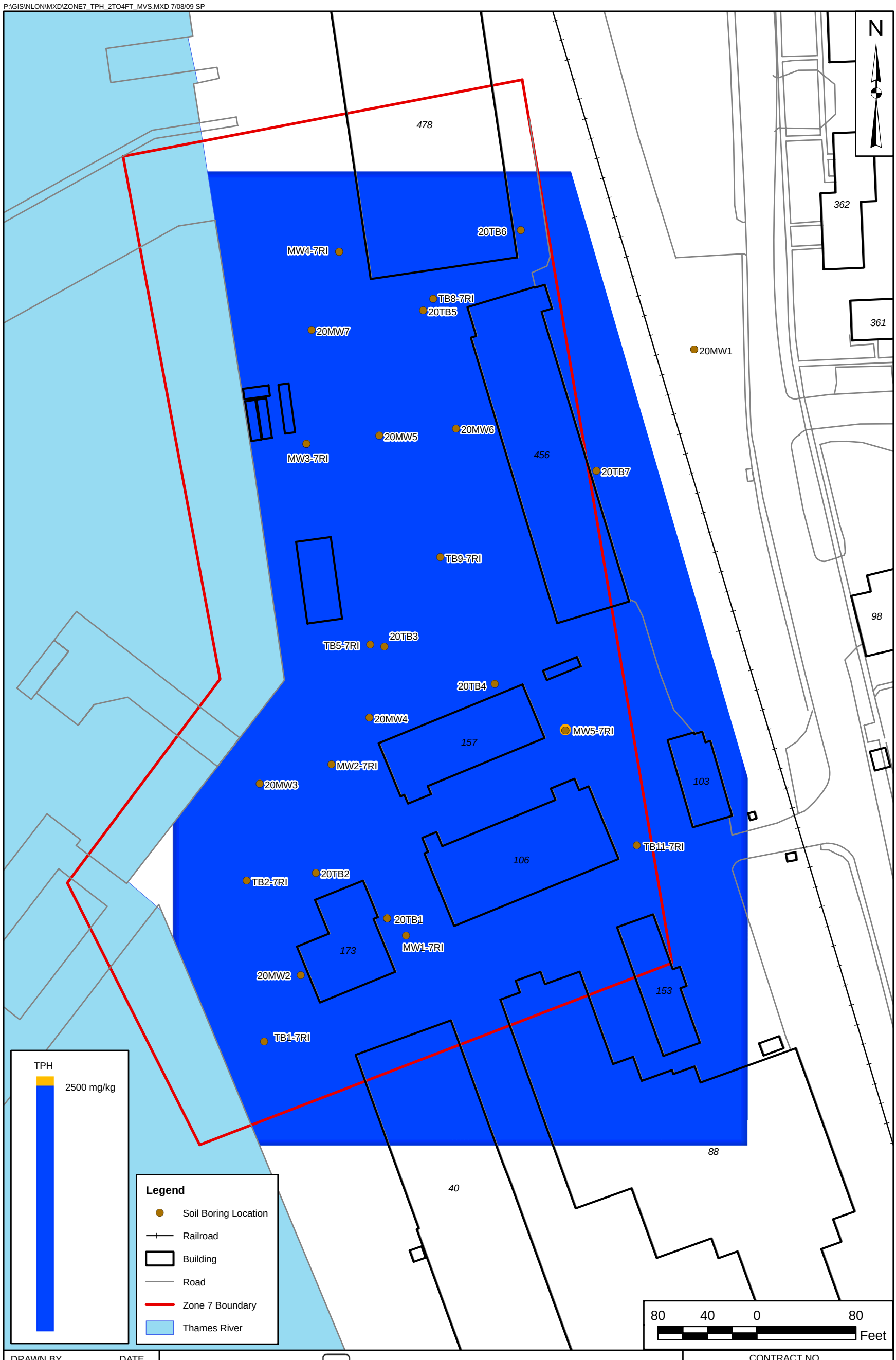
DRAWN BY S. PAXTON	DATE 4/02/09	Tetra Tech NUS, Inc.	CONTRACT NO. 0811
CHECKED BY N. BALSAMO	DATE 7/08/09	ESTIMATED TPH CONCENTRATIONS (KRIGING RESULTS) ELEVATION: 6 TO 8 FEET ZONE 7 NAVAL SUBMARINE BASE - NEW LONDON GROTON, CONNECTICUT	APPROVED BY _ / DATE --
COST/SCHED-AREA			APPROVED BY -- / DATE --
SCALE AS NOTED			FIGURE NO. FIGURE B-99 / REV.

P:\GIS\NLON\MXD\ZONE7_TPH_4TO6FT_MVS.MXD 7/08/09 SP



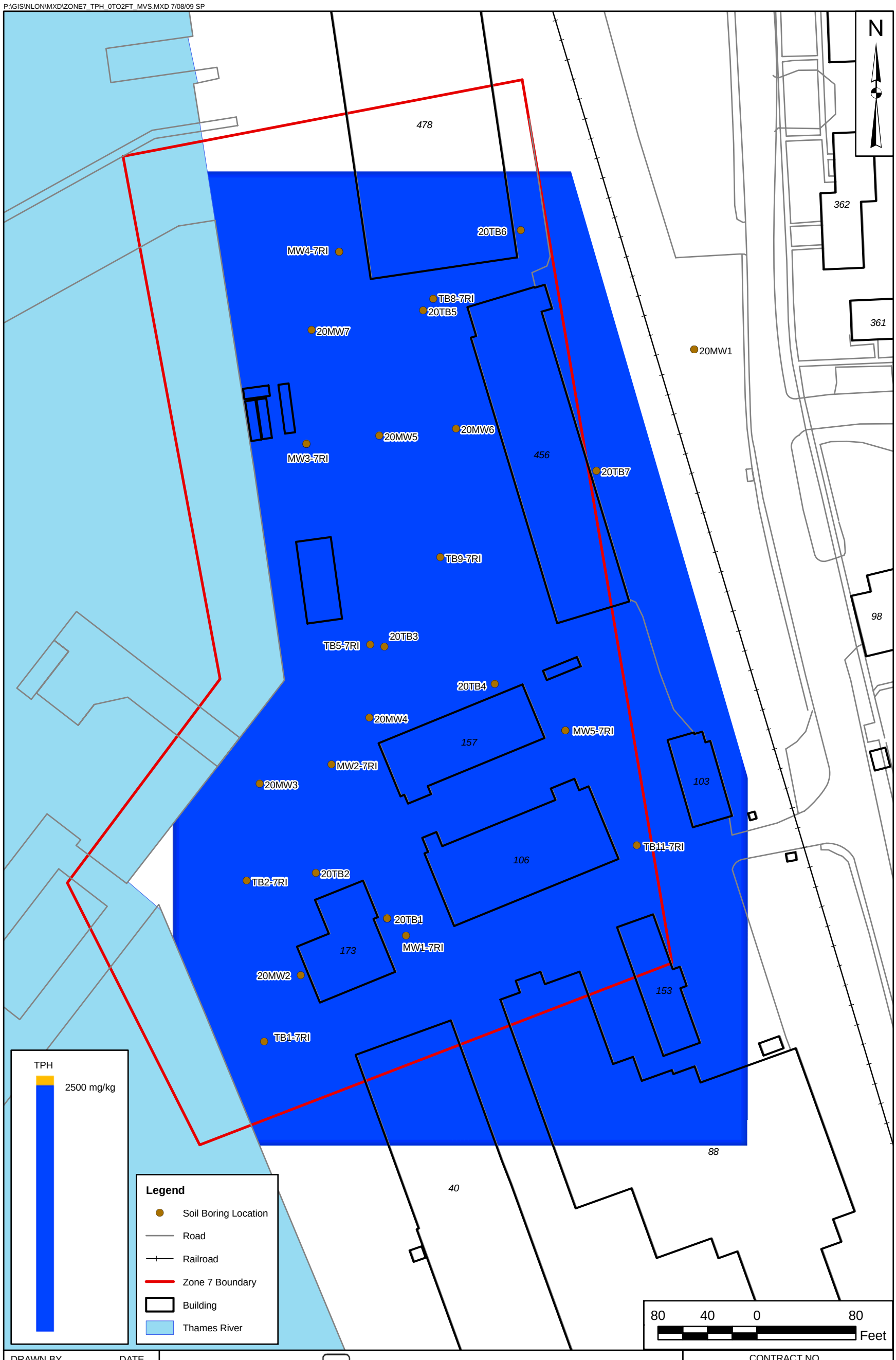
DRAWN BY S. PAXTON	DATE 4/02/09	Tetra Tech NUS, Inc.	CONTRACT NO. 0811
CHECKED BY N. BALSAMO	DATE 7/08/09	ESTIMATED TPH CONCENTRATIONS (KRIGING RESULTS) ELEVATION: 4 TO 6 FEET ZONE 7 NAVAL SUBMARINE BASE - NEW LONDON GROTON, CONNECTICUT	APPROVED BY _ / DATE --
COST/SCHED-AREA			APPROVED BY -- / DATE --
SCALE AS NOTED			FIGURE NO. FIGURE B-100 / REV.

P:\GIS\NLON\MXD\ZONE7_TPH_2TO4FT_MVS.MXD 7/08/09 SP



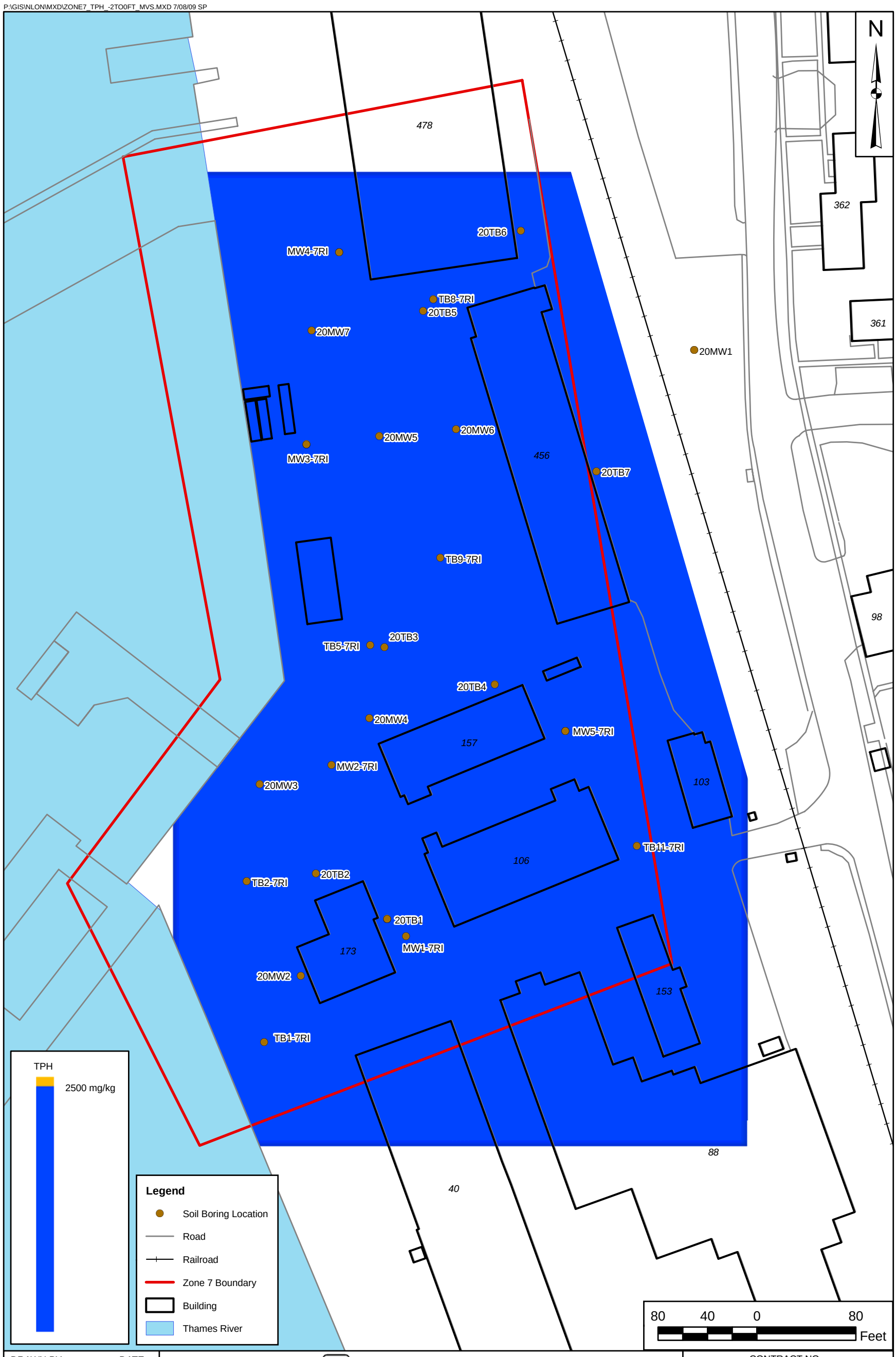
DRAWN BY S. PAXTON	DATE 4/02/09	Tetra Tech NUS, Inc.	CONTRACT NO. 0811
CHECKED BY N. BALSAMO	DATE 7/08/09	ESTIMATED TPH CONCENTRATIONS (KRIGING RESULTS) ELEVATION: 2 TO 4 FEET ZONE 7 NAVAL SUBMARINE BASE - NEW LONDON GROTON, CONNECTICUT	APPROVED BY _ / DATE --
COST/SCHED-AREA			APPROVED BY -- / DATE --
SCALE AS NOTED			FIGURE NO. FIGURE B-101 / REV.

P:\GIS\NLON\MXD\ZONE7_TPH_0TO2FT_MVS.MXD 7/08/09 SP



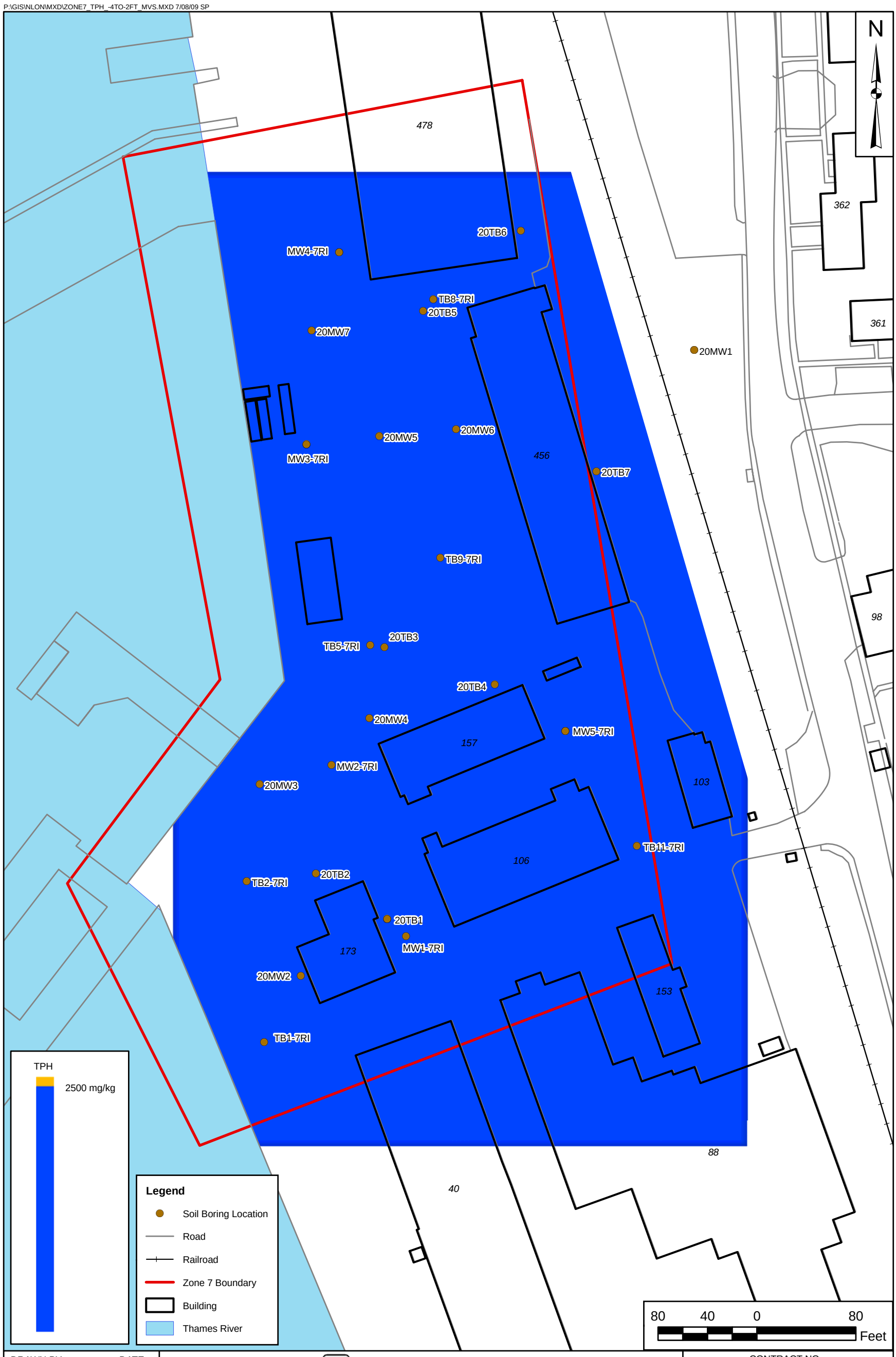
DRAWN BY S. PAXTON	DATE 4/02/09	Tetra Tech NUS, Inc.	CONTRACT NO. 0811
CHECKED BY N. BALSAMO	DATE 7/08/09	ESTIMATED TPH CONCENTRATIONS (KRIGING RESULTS) ELEVATION: 0 TO 2 FEET ZONE 7 NAVAL SUBMARINE BASE - NEW LONDON GROTON, CONNECTICUT	APPROVED BY _ / DATE --
COST/SCHED-AREA			APPROVED BY -- / DATE --
SCALE AS NOTED			FIGURE NO. FIGURE B-102 / REV.

P:\GIS\NLON\MXD\ZONE7_TPH_-2TO0FT_MVS.MXD 7/08/09 SP



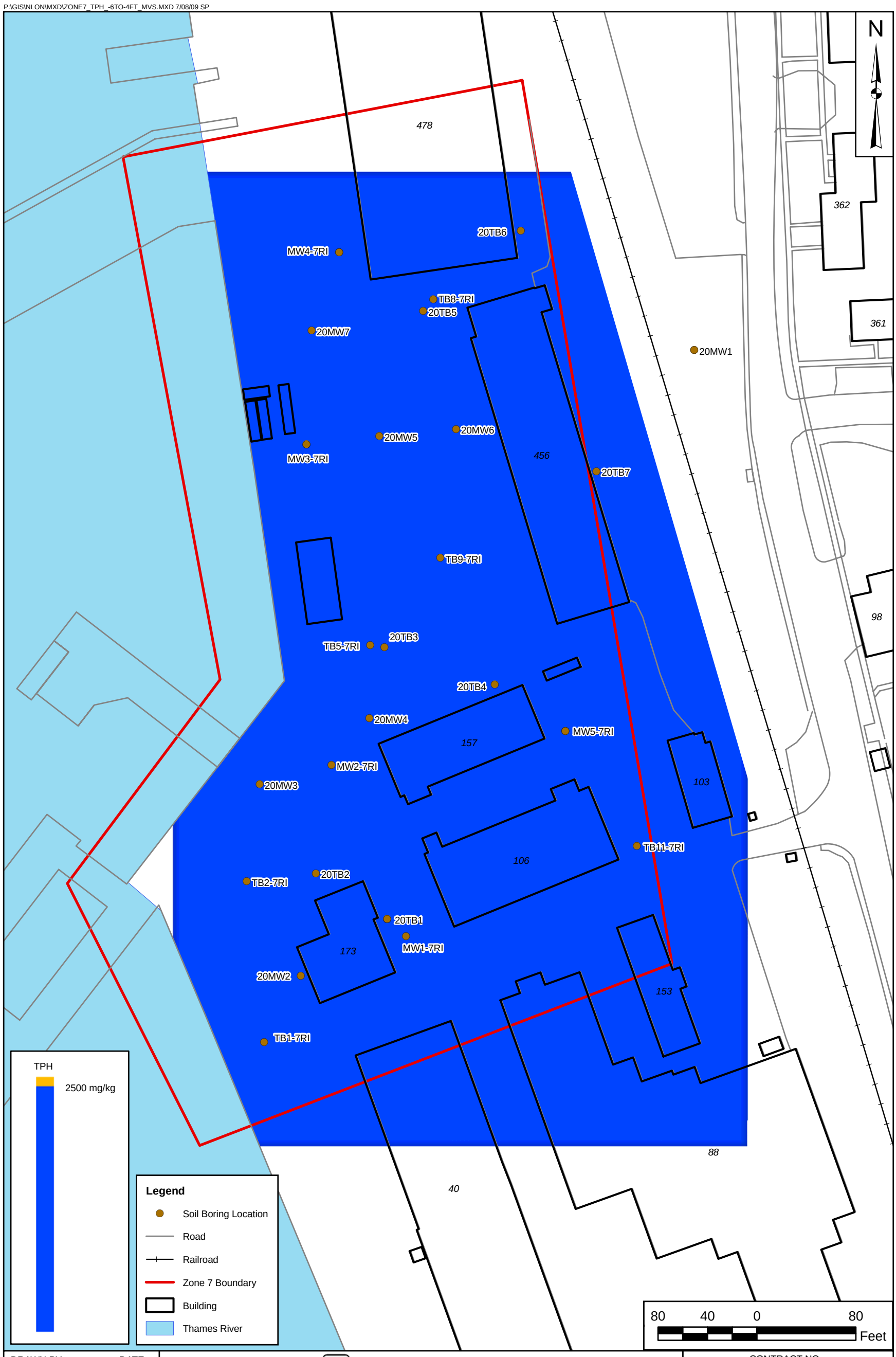
DRAWN BY S. PAXTON	DATE 4/02/09	Tetra Tech NUS, Inc.	CONTRACT NO. 0811
CHECKED BY N. BALSAMO	DATE 7/08/09	ESTIMATED TPH CONCENTRATIONS (KRIGING RESULTS) ELEVATION: -2 TO 0 FEET ZONE 7 NAVAL SUBMARINE BASE - NEW LONDON GROTON, CONNECTICUT	APPROVED BY _ / DATE --
COST/SCHED-AREA			APPROVED BY -- / DATE --
SCALE AS NOTED			FIGURE NO. FIGURE B-103 / REV.

P:\GIS\NLON\MXD\ZONE7_TPH_-4TO-2FT_MVS.MXD 7/08/09 SP



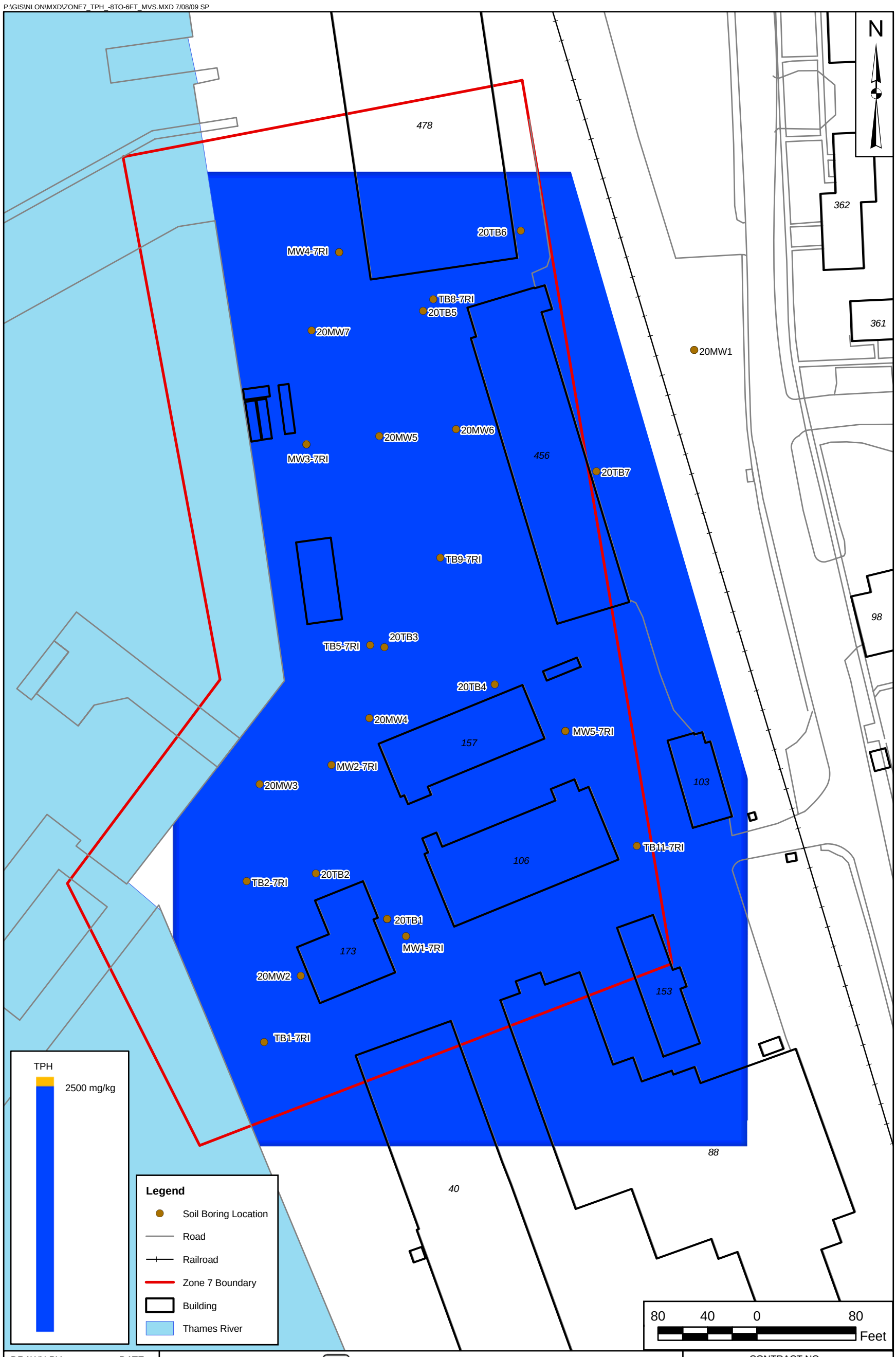
DRAWN BY S. PAXTON	DATE 4/02/09	Tetra Tech NUS, Inc.	CONTRACT NO. 0811
CHECKED BY N. BALSAMO	DATE 7/08/09	ESTIMATED TPH CONCENTRATIONS (KRIGING RESULTS) ELEVATION: -4 TO -2 FEET ZONE 7 NAVAL SUBMARINE BASE - NEW LONDON GROTON, CONNECTICUT	APPROVED BY – / DATE --
COST/SCHED-AREA			APPROVED BY -- / DATE --
SCALE AS NOTED			FIGURE NO. FIGURE B-104 / REV.

P:\GIS\NLON\MXD\ZONE7_TPH_-6TO-4FT_MVS.MXD 7/08/09 SP



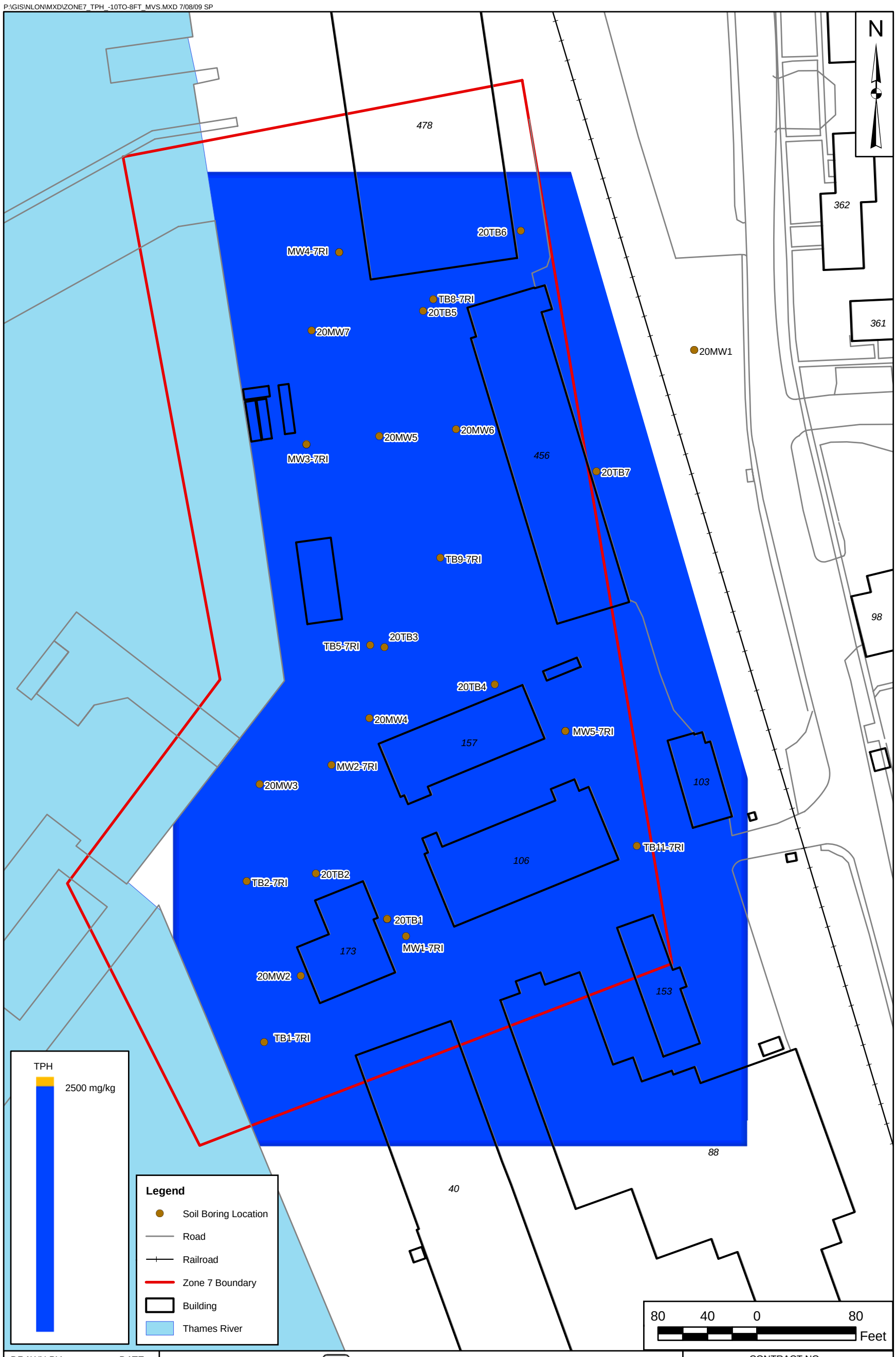
DRAWN BY S. PAXTON	DATE 4/02/09	Tetra Tech NUS, Inc.	CONTRACT NO. 0811
CHECKED BY N. BALSAMO	DATE 7/08/09	ESTIMATED TPH CONCENTRATIONS (KRIGING RESULTS) ELEVATION: -6 TO -4 FEET ZONE 7 NAVAL SUBMARINE BASE - NEW LONDON GROTON, CONNECTICUT	APPROVED BY – / DATE --
COST/SCHED-AREA			APPROVED BY -- / DATE --
SCALE AS NOTED			FIGURE NO. FIGURE B-105 / REV.

P:\GIS\NLON\MXD\ZONE7_TPH_-8TO-6FT_MVS.MXD 7/08/09 SP



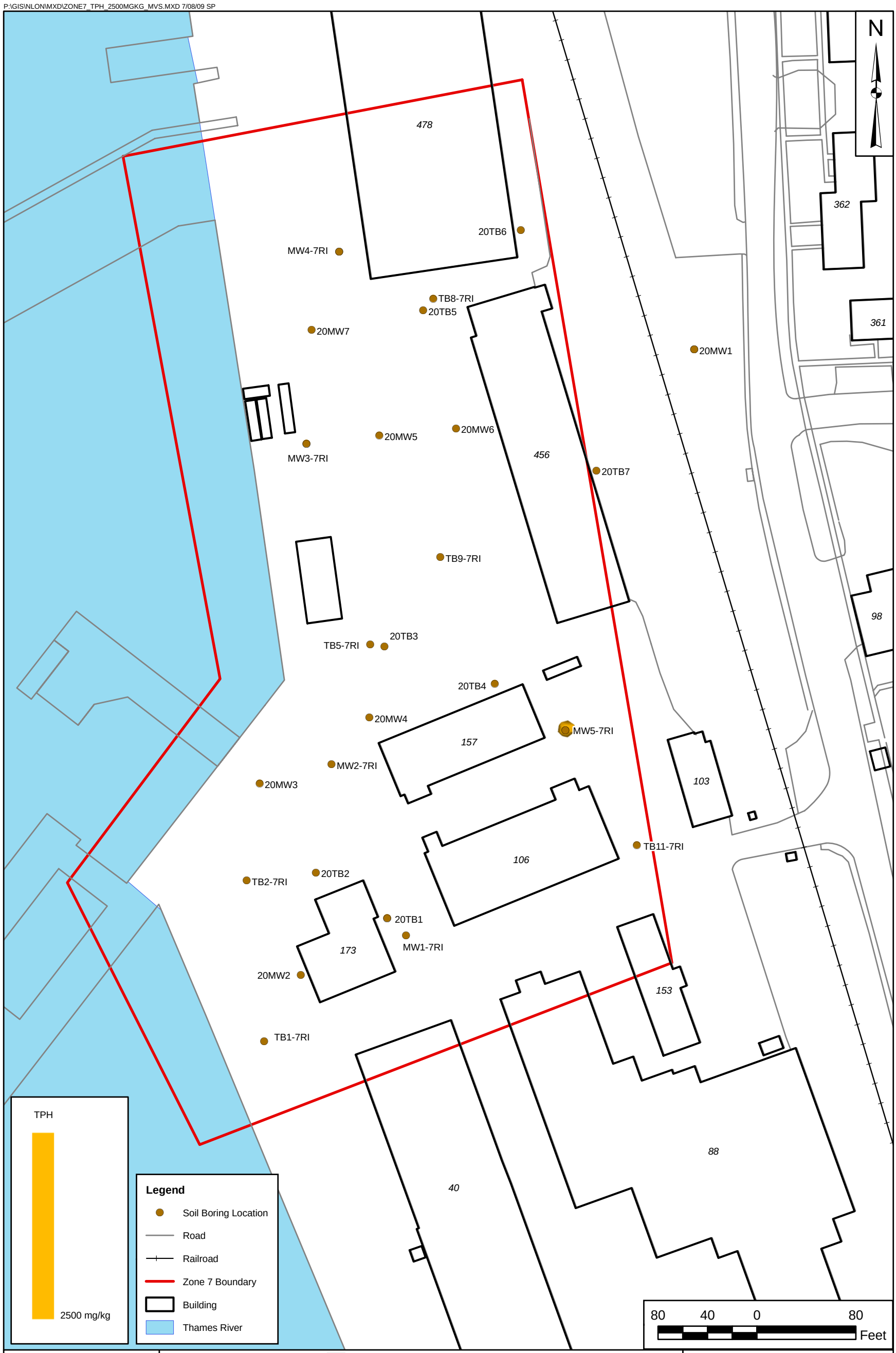
DRAWN BY S. PAXTON	DATE 4/02/09	Tetra Tech NUS, Inc.	CONTRACT NO. 0811
CHECKED BY N. BALSAMO	DATE 7/08/09	ESTIMATED TPH CONCENTRATIONS (KRIGING RESULTS) ELEVATION: -8 TO -6 FEET ZONE 7 NAVAL SUBMARINE BASE - NEW LONDON GROTON, CONNECTICUT	APPROVED BY – / DATE --
COST/SCHED-AREA			APPROVED BY -- / DATE --
SCALE AS NOTED			FIGURE NO. FIGURE B-106 / REV.

P:\GIS\NLON\MXD\ZONE7_TPH_-10TO-8FT_MVS.MXD 7/08/09 SP



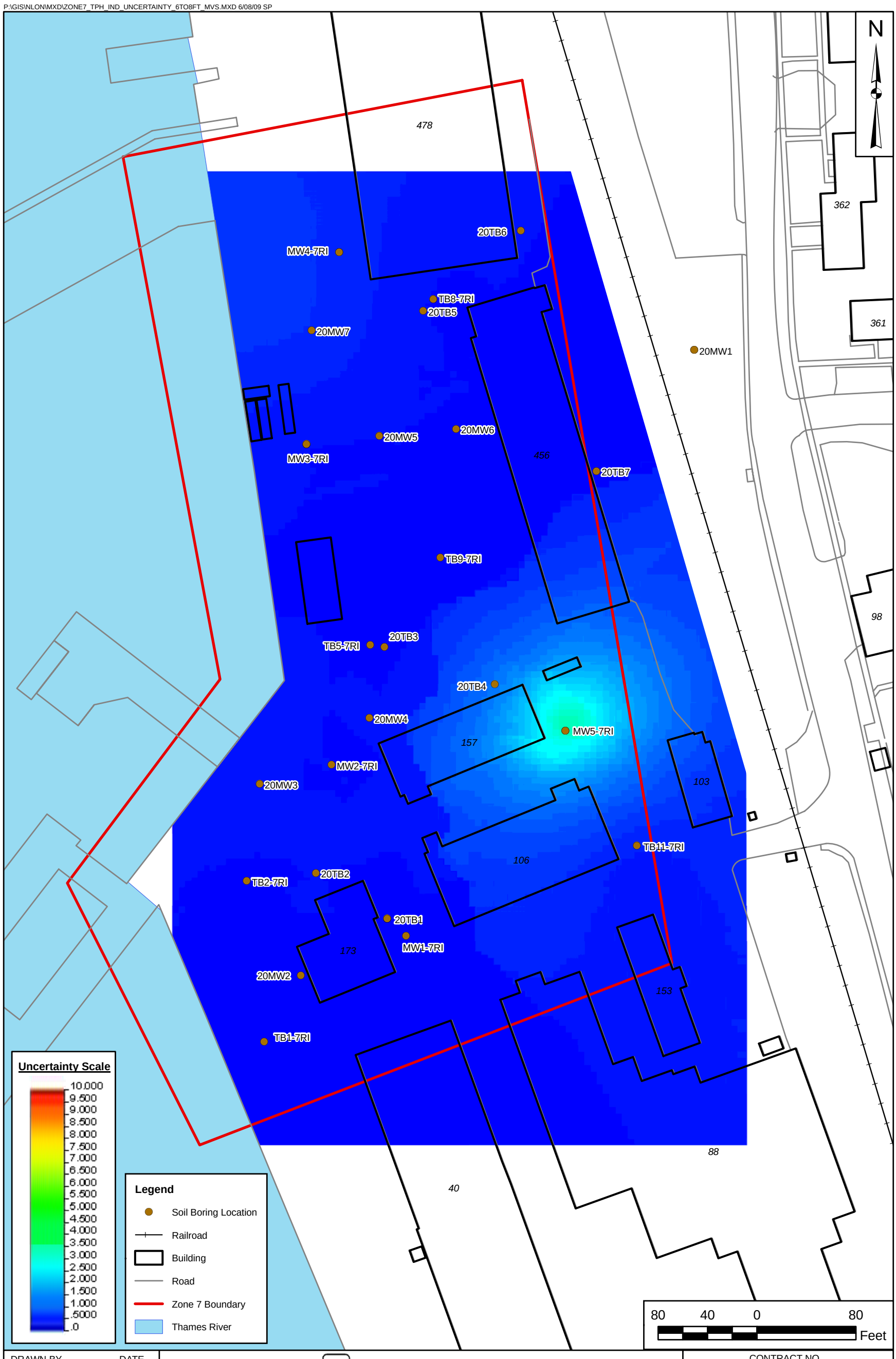
DRAWN BY S. PAXTON	DATE 4/02/09	Tetra Tech NUS, Inc.	CONTRACT NO. 0811
CHECKED BY N. BALSAMO	DATE 7/08/09	ESTIMATED TPH CONCENTRATIONS (KRIGING RESULTS) ELEVATION: -10 TO -8 FEET ZONE 7 NAVAL SUBMARINE BASE - NEW LONDON GROTON, CONNECTICUT	APPROVED BY _ / DATE --
COST/SCHED-AREA			APPROVED BY -- / DATE --
SCALE AS NOTED			FIGURE NO. FIGURE B-107 / REV.

P:\GIS\NLON\MXD\ZONE7_TPH_2500MGKG_MVS.MXD 7/08/09 SP



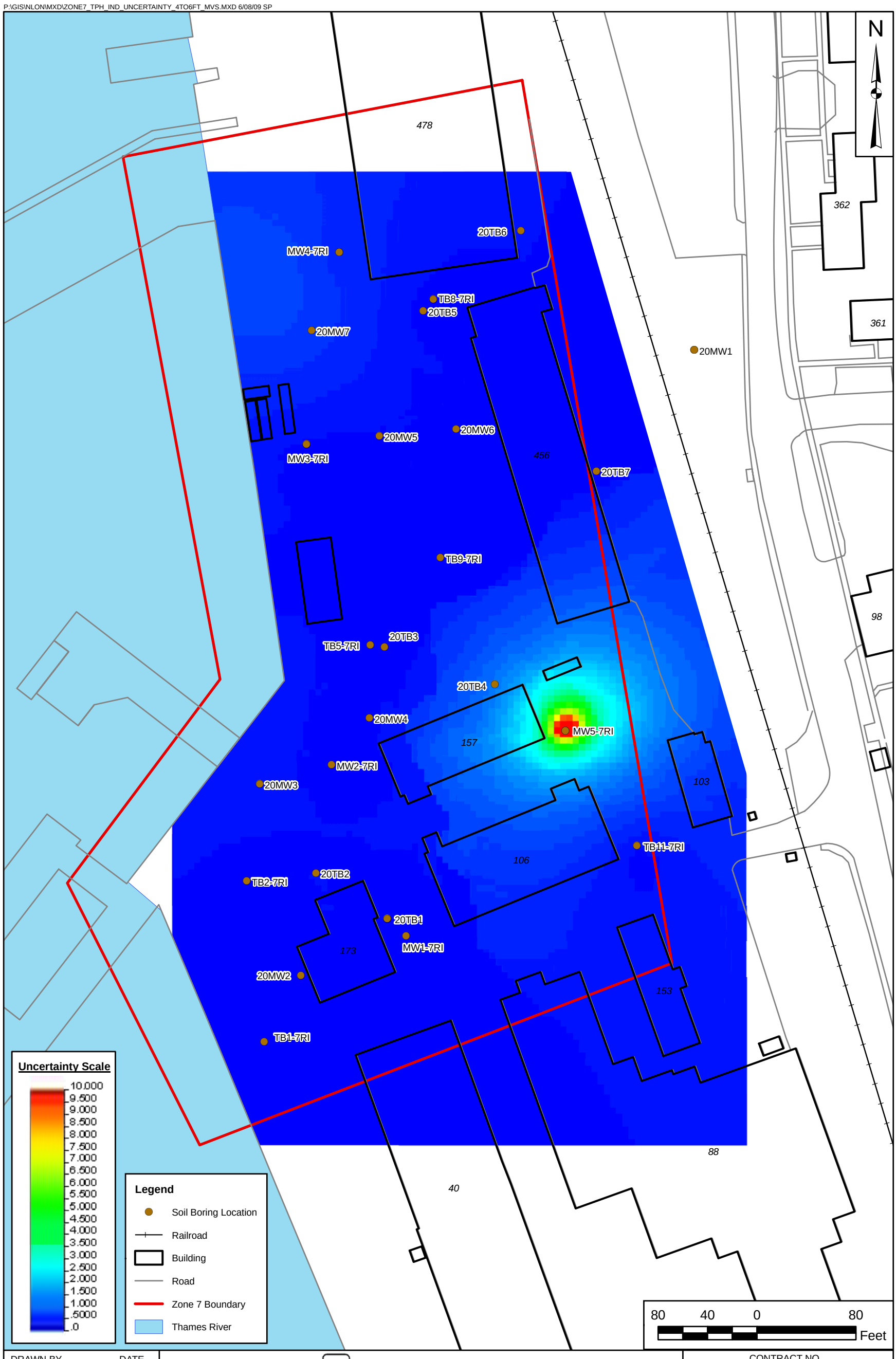
DRAWN BY S. PAXTON	DATE 4/02/09	Tetra Tech NUS, Inc.	CONTRACT NO. 0811
CHECKED BY N. BALSAMO	DATE 7/08/09	ESTIMATED TPH CONCENTRATIONS (KRIGING RESULTS) ELEVATION: 8 TO 0 FEET ZONE 7 NAVAL SUBMARINE BASE - NEW LONDON GROTON, CONNECTICUT	APPROVED BY _ / DATE --
COST/SCHED-AREA			APPROVED BY -- / DATE --
SCALE AS NOTED			FIGURE NO. FIGURE B-108 / REV.

P:\GIS\NLON\MXD\ZONE7_TPH_IND_UNCERTAINTY_6TO8FT_MVS.MXD 6/08/09 SP



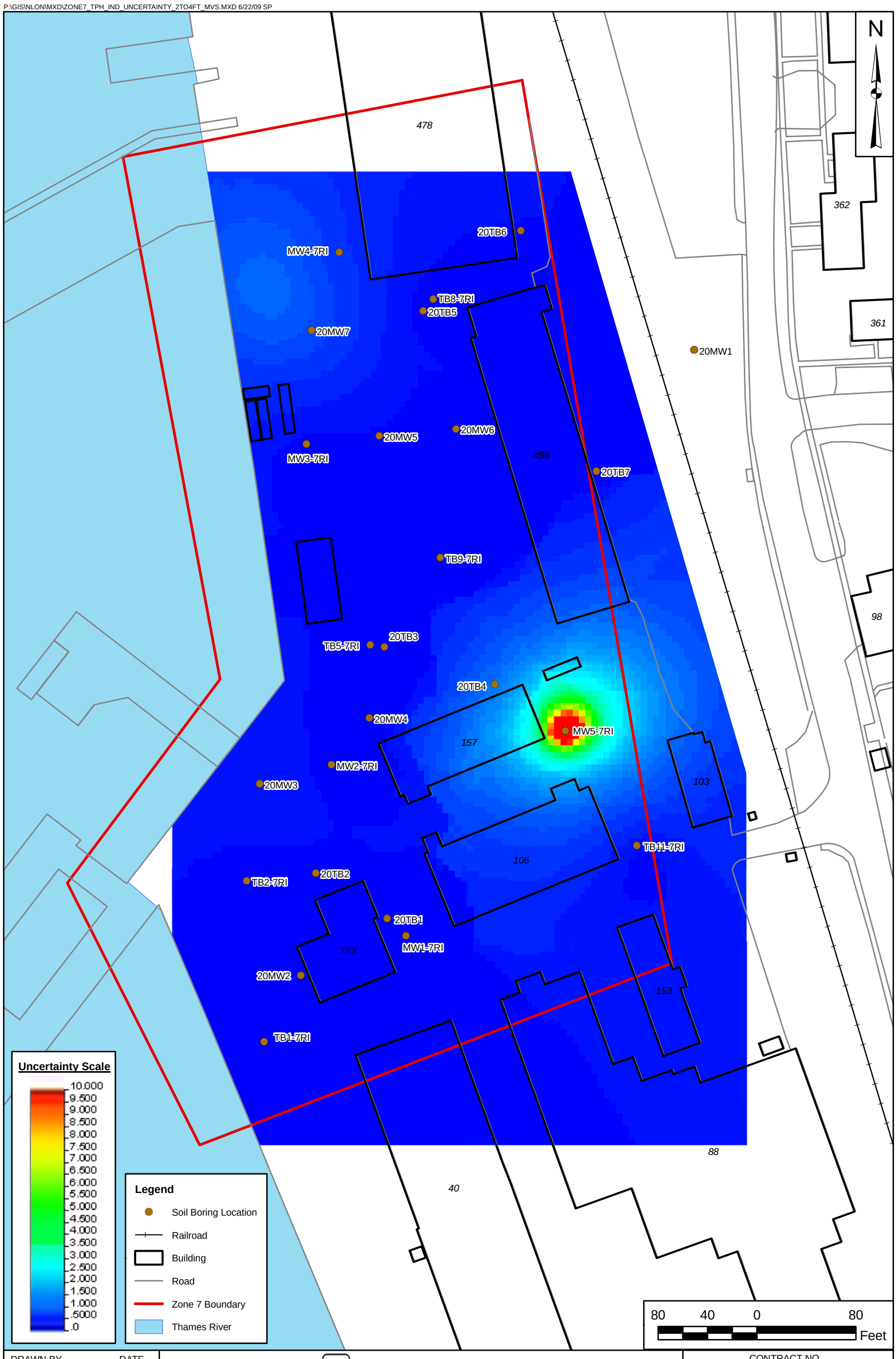
DRAWN BY S. PAXTON	DATE 4/02/09	Tetra Tech NUS, Inc.	CONTRACT NO. 0811
CHECKED BY N. BALSAMO	DATE 6/09/09	TPH UNCERTAINTY FOR INDUSTRIAL CRITERIA ELEVATION: 6 TO 8 FEET ZONE 7 NAVAL SUBMARINE BASE - NEW LONDON GROTON, CONNECTICUT	APPROVED BY _ / DATE --
COST/SCHED-AREA			APPROVED BY -- / DATE --
SCALE AS NOTED			FIGURE NO. FIGURE B-109 / REV.

P:\GIS\NLON\MXD\ZONE7_TPH_IND_UNCERTAINTY_4TO6FT_MVS.MXD 6/08/09 SP



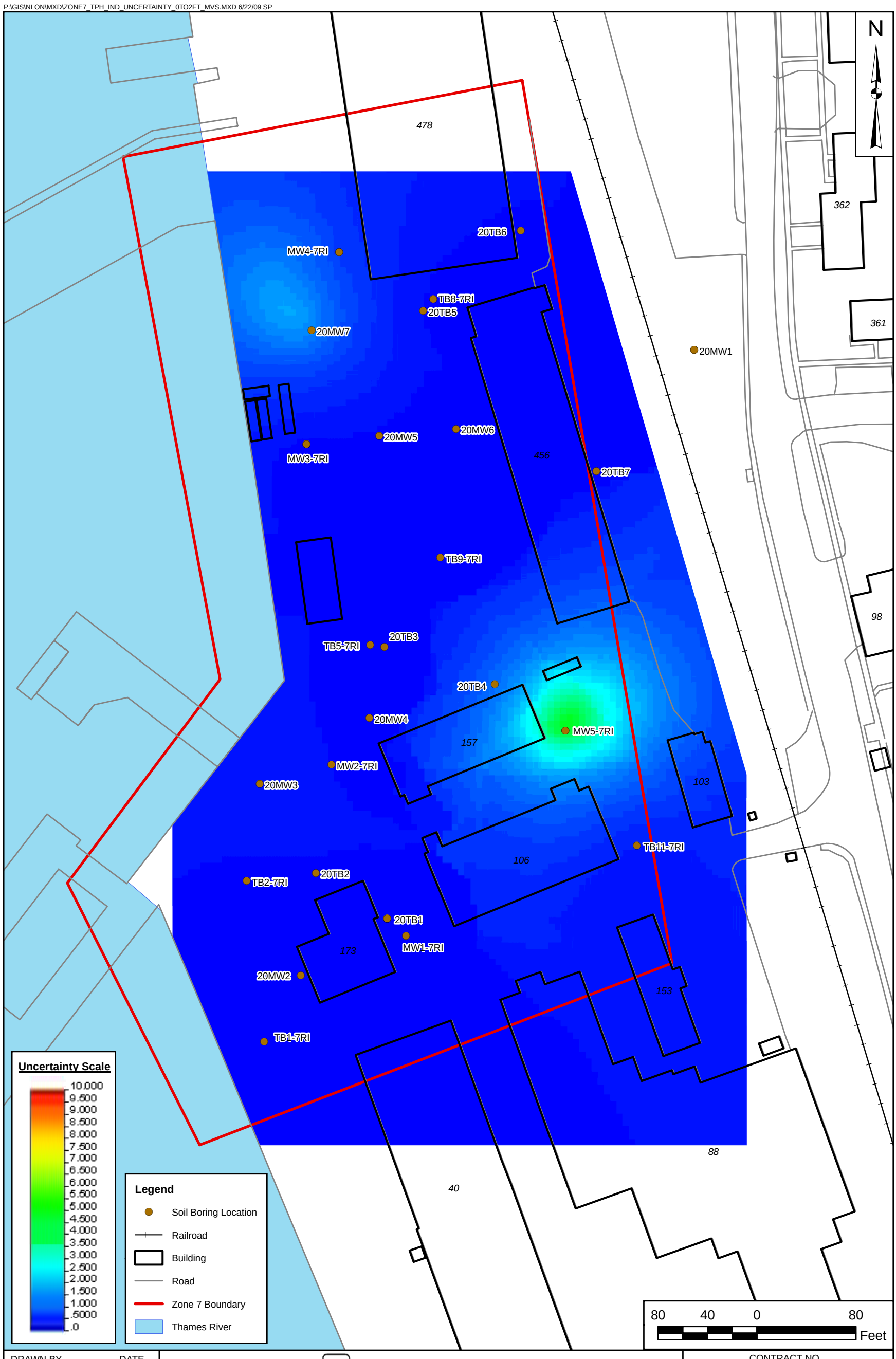
DRAWN BY S. PAXTON	DATE 4/02/09	Tetra Tech NUS, Inc.	CONTRACT NO. 0811
CHECKED BY N. BALSAMO	DATE 6/09/09	TPH UNCERTAINTY FOR INDUSTRIAL CRITERIA ELEVATION: 4 TO 6 FEET ZONE 7 NAVAL SUBMARINE BASE - NEW LONDON GROTON, CONNECTICUT	APPROVED BY _ / DATE --
COST/SCHED-AREA			APPROVED BY -- / DATE --
SCALE AS NOTED			FIGURE NO. FIGURE B-110 / REV.

P:\GIS\NLON\MXD\ZONE7_TPH_IND_UNCERTAINTY_2TO4FT_MVS.MXD 6/22/09 SP



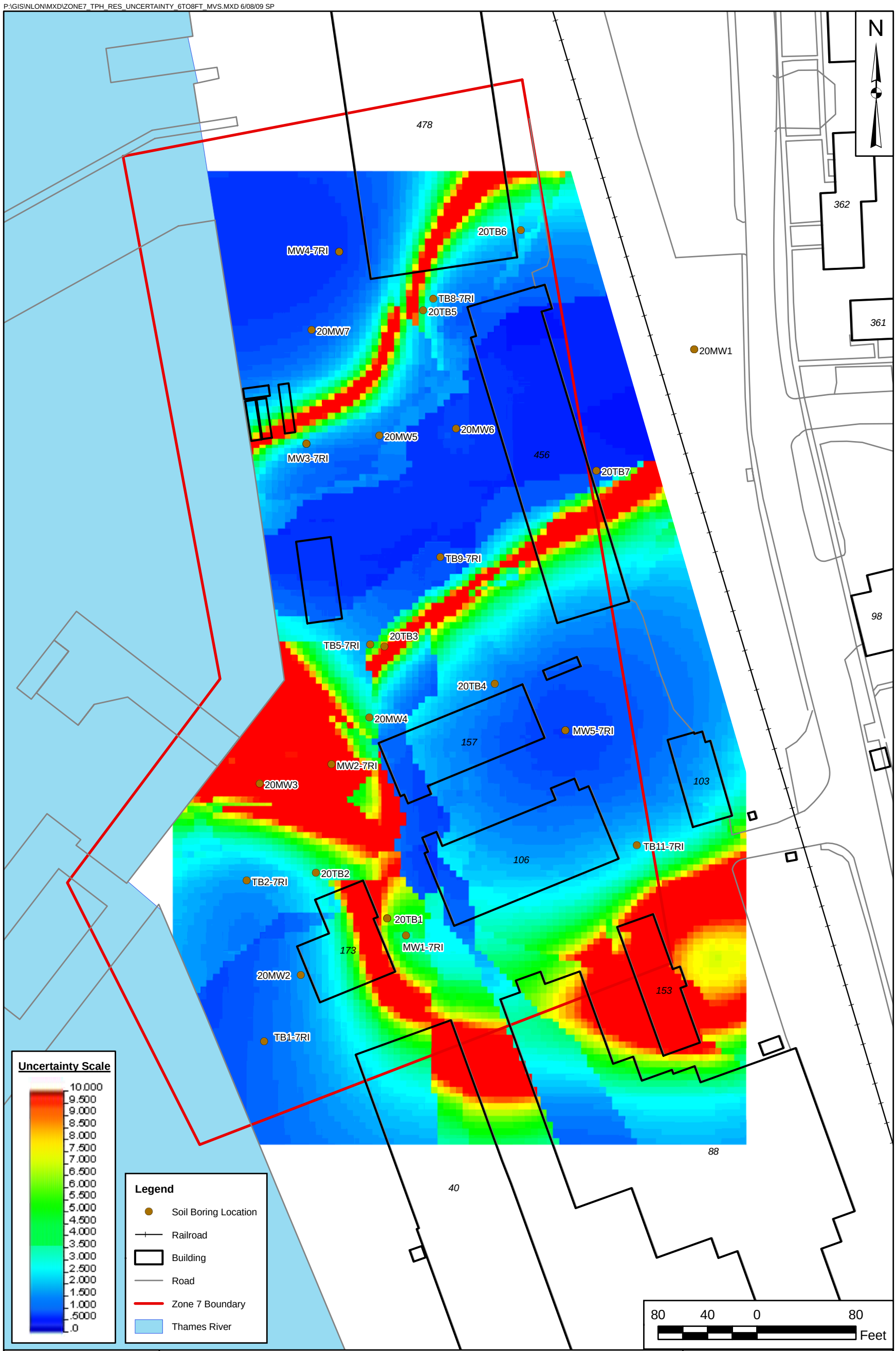
DRAWN BY S. PAXTON	DATE 4/02/09	Tetra Tech NUS, Inc.	CONTRACT NO. 0811
CHECKED BY N. BALSAMO	DATE 6/22/09	TPH UNCERTAINTY FOR INDUSTRIAL CRITERIA ELEVATION: 2 TO 4 FEET ZONE 7 NAVAL SUBMARINE BASE - NEW LONDON GROTON, CONNECTICUT	APPROVED BY _ / DATE --
COST/SCHED-AREA			APPROVED BY -- / DATE --
SCALE AS NOTED			FIGURE NO. FIGURE B-111 / REV.

P:\GIS\NLON\MXD\ZONE7_TPH_IND_UNCERTAINTY_0TO2FT_MVS.MXD 6/22/09 SP



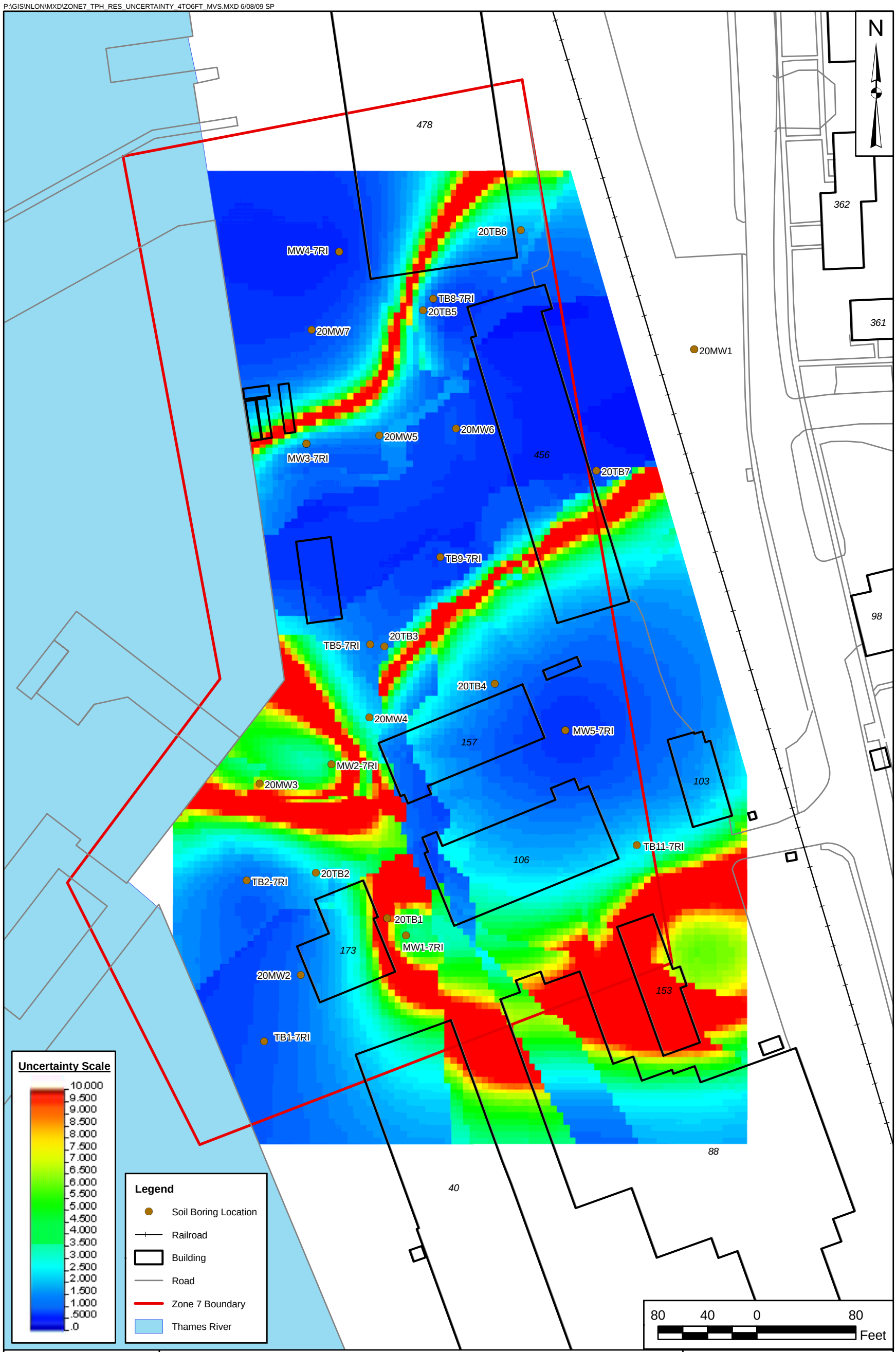
DRAWN BY S. PAXTON	DATE 4/02/09	Tetra Tech NUS, Inc.	CONTRACT NO. 0811
CHECKED BY N. BALSAMO	DATE 6/22/09	TPH UNCERTAINTY FOR INDUSTRIAL CRITERIA ELEVATION: 0 TO 2 FEET ZONE 7 NAVAL SUBMARINE BASE - NEW LONDON GROTON, CONNECTICUT	APPROVED BY _ / DATE --
COST/SCHED-AREA			APPROVED BY -- / DATE --
SCALE AS NOTED			FIGURE NO. FIGURE B-112 / REV.

P:\GIS\NLON\MXD\ZONE7_TPH_RES_UNCERTAINTY_6TO8FT_MVS.MXD 6/08/09 SP



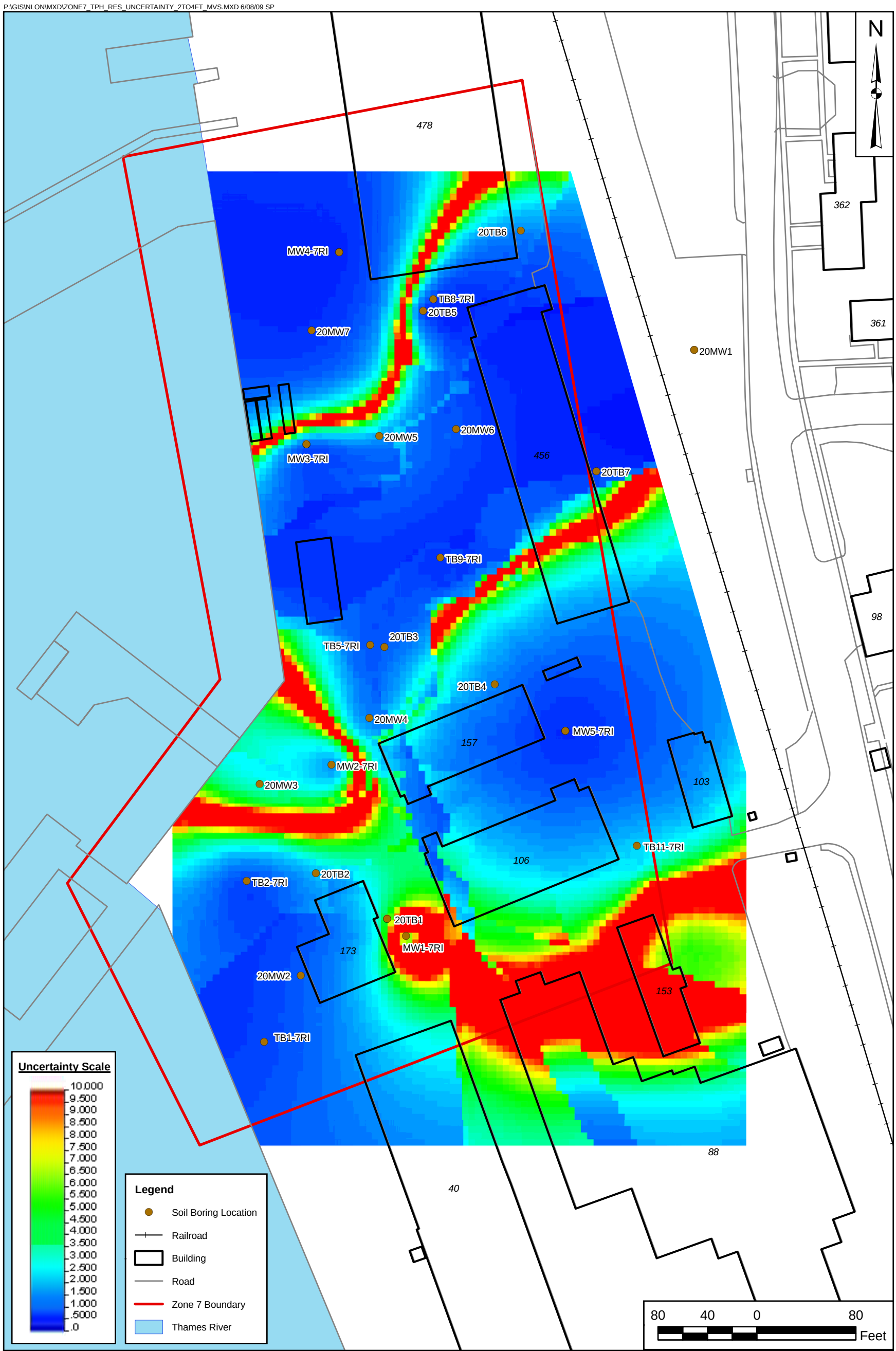
DRAWN BY S. PAXTON	DATE 4/02/09	Tetra Tech NUS, Inc.	CONTRACT NO. 0811
CHECKED BY N. BALSAMO	DATE 6/09/09	TPH UNCERTAINTY FOR RESIDENTIAL CRITERIA ELEVATION: 6 TO 8 FEET ZONE 7 NAVAL SUBMARINE BASE - NEW LONDON GROTON, CONNECTICUT	APPROVED BY _ / DATE --
COST/SCHED-AREA			APPROVED BY -- / DATE --
SCALE AS NOTED			FIGURE NO. FIGURE B-113 / REV.

P:\GIS\NLON\MXD\ZONE7_TPH_RES_UNCERTAINTY_4TO6FT_MVS.MXD 6/08/09 SP



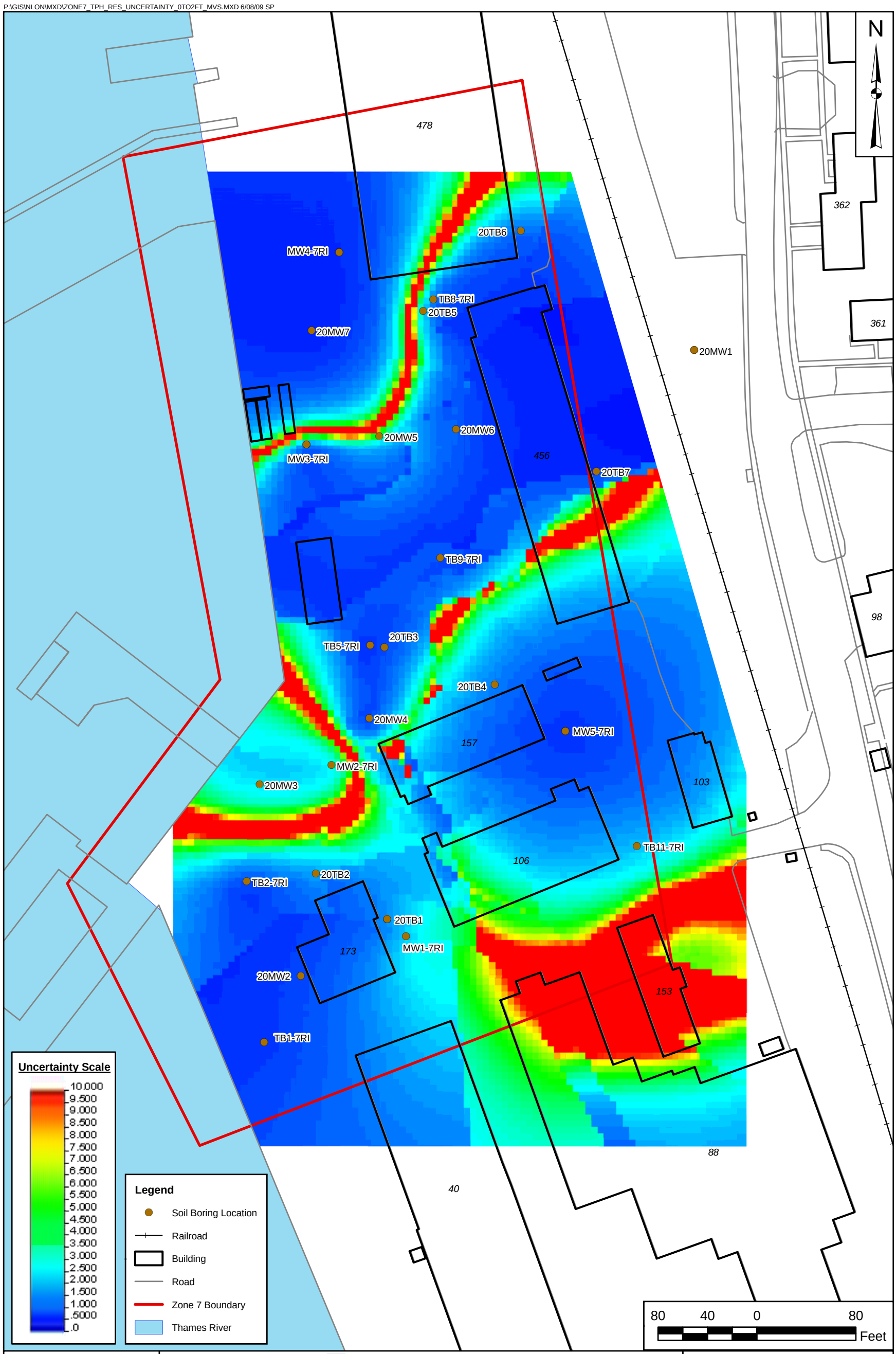
DRAWN BY S. PAXTON	DATE 4/02/09	Tetra Tech NUS, Inc.	CONTRACT NO. 0811
CHECKED BY N. BALSAMO	DATE 6/09/09	TPH UNCERTAINTY FOR RESIDENTIAL CRITERIA ELEVATION: 4 TO 6 FEET ZONE 7 NAVAL SUBMARINE BASE - NEW LONDON GROTON, CONNECTICUT	APPROVED BY _ / DATE --
COST/SCHED-AREA			APPROVED BY -- / DATE --
SCALE AS NOTED			FIGURE NO. FIGURE B-114 / REV.

P:\GIS\NLON\MXD\ZONE7_TPH_RES_UNCERTAINTY_2TO4FT_MVS.MXD 6/08/09 SP



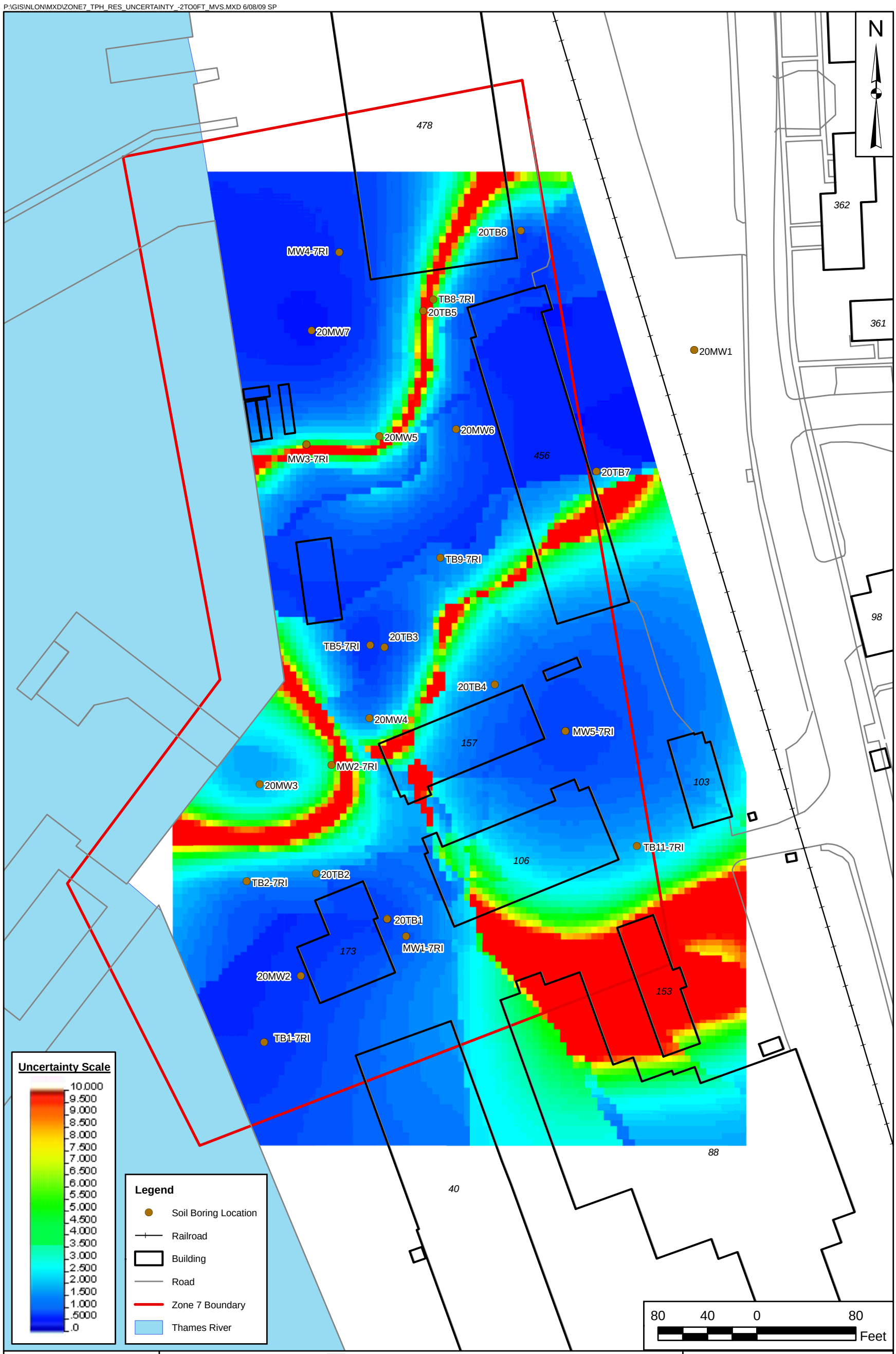
DRAWN BY S. PAXTON	DATE 4/02/09	Tetra Tech NUS, Inc.	CONTRACT NO. 0811
CHECKED BY N. BALSAMO	DATE 6/09/09	TPH UNCERTAINTY FOR RESIDENTIAL CRITERIA ELEVATION: 2 TO 4 FEET ZONE 7 NAVAL SUBMARINE BASE - NEW LONDON GROTON, CONNECTICUT	APPROVED BY _ / DATE --
COST/SCHED-AREA			APPROVED BY -- / DATE --
SCALE AS NOTED			FIGURE NO. FIGURE B-115 / REV.

P:\GIS\NLON\MXD\ZONE7_TPH_RES_UNCERTAINTY_0TO2FT_MVS.MXD 6/08/09 SP



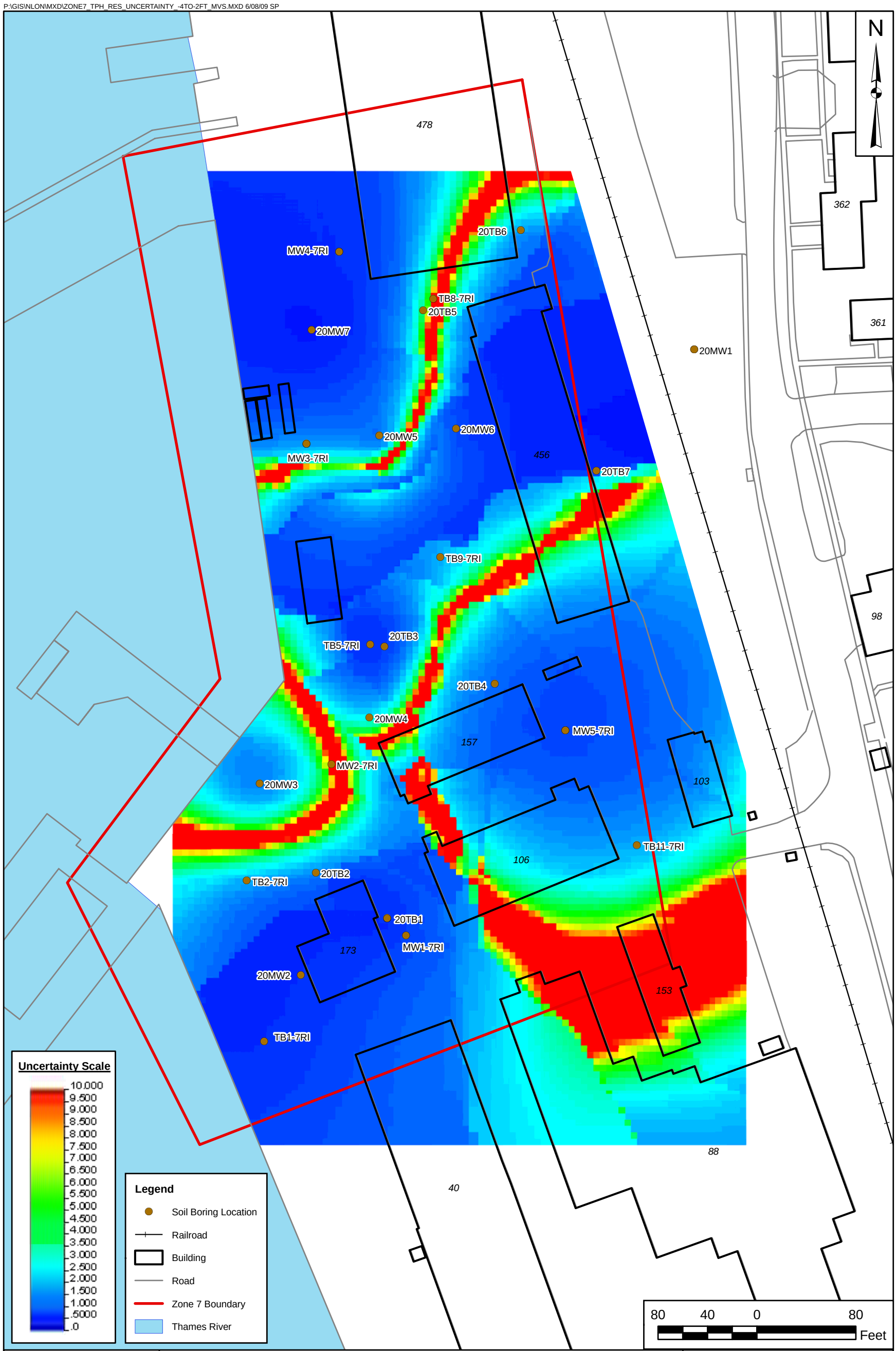
DRAWN BY S. PAXTON	DATE 4/02/09	Tetra Tech NUS, Inc.	CONTRACT NO. 0811
CHECKED BY N. BALSAMO	DATE 6/09/09	TPH UNCERTAINTY FOR RESIDENTIAL CRITERIA ELEVATION: 0 TO 2 FEET ZONE 7 NAVAL SUBMARINE BASE - NEW LONDON GROTON, CONNECTICUT	APPROVED BY _ / DATE --
COST/SCHED-AREA			APPROVED BY -- / DATE --
SCALE AS NOTED			FIGURE NO. FIGURE B-116 / REV.

P:\GIS\NLON\MXD\ZONE7_TPH_RES_UNCERTAINTY_-2TO0FT_MVS.MXD 6/08/09 SP



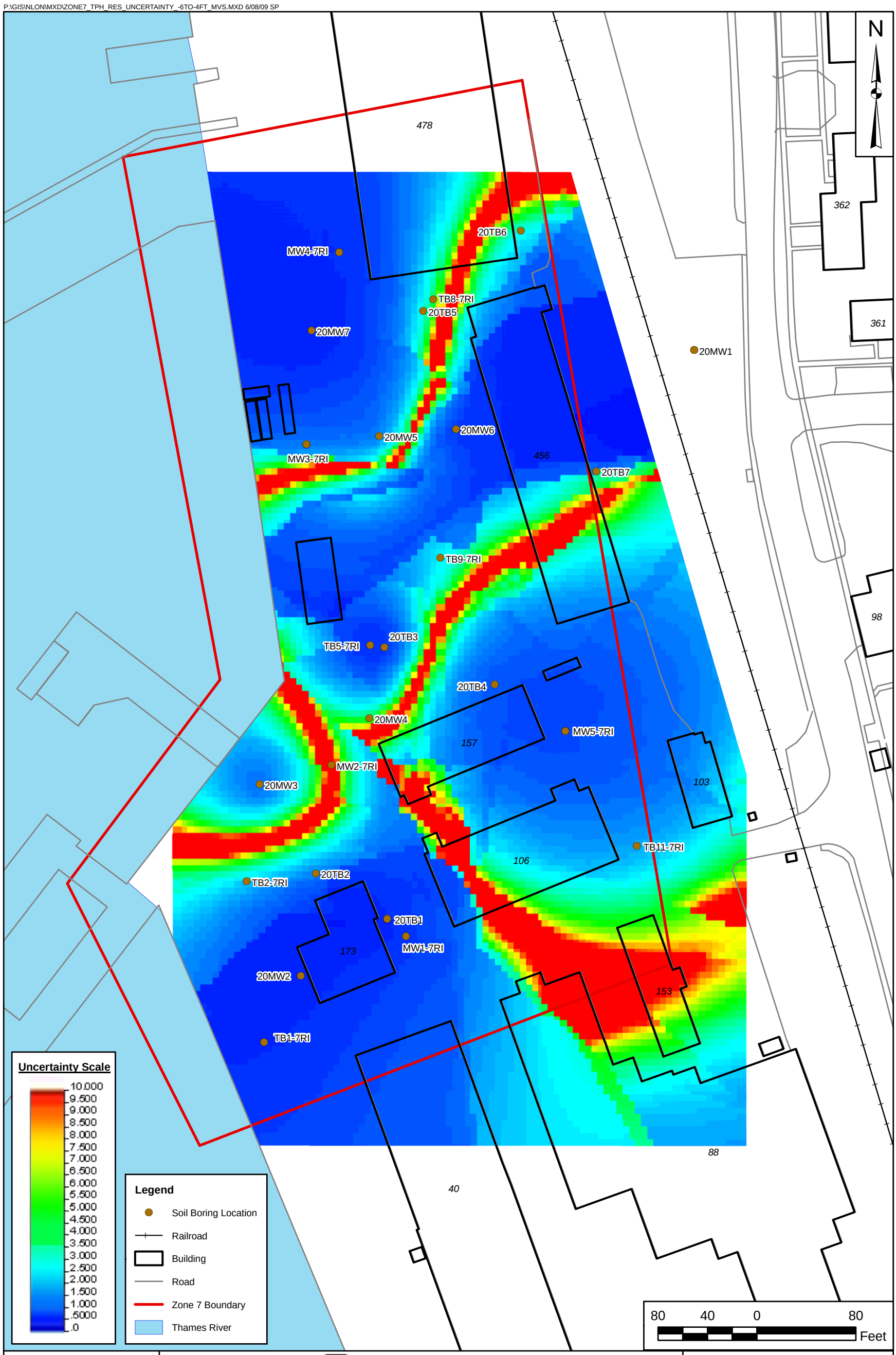
DRAWN BY	DATE	Tetra Tech NUS, Inc.	CONTRACT NO.
S. PAXTON	4/02/09		0811
CHECKED BY	DATE	TPH UNCERTAINTY FOR RESIDENTIAL CRITERIA ELEVATION: -2 TO 0 FEET ZONE 7 NAVAL SUBMARINE BASE - NEW LONDON GROTON, CONNECTICUT	APPROVED BY / DATE
N. BALSAMO	6/09/09		_ / --
COST/SCHED-AREA			APPROVED BY / DATE
			-- / --
SCALE			FIGURE NO. / REV.
AS NOTED			FIGURE B-117

P:\GIS\NLON\MXD\ZONE7_TPH_RES_UNCERTAINTY_-4TO-2FT_MVS.MXD 6/08/09 SP



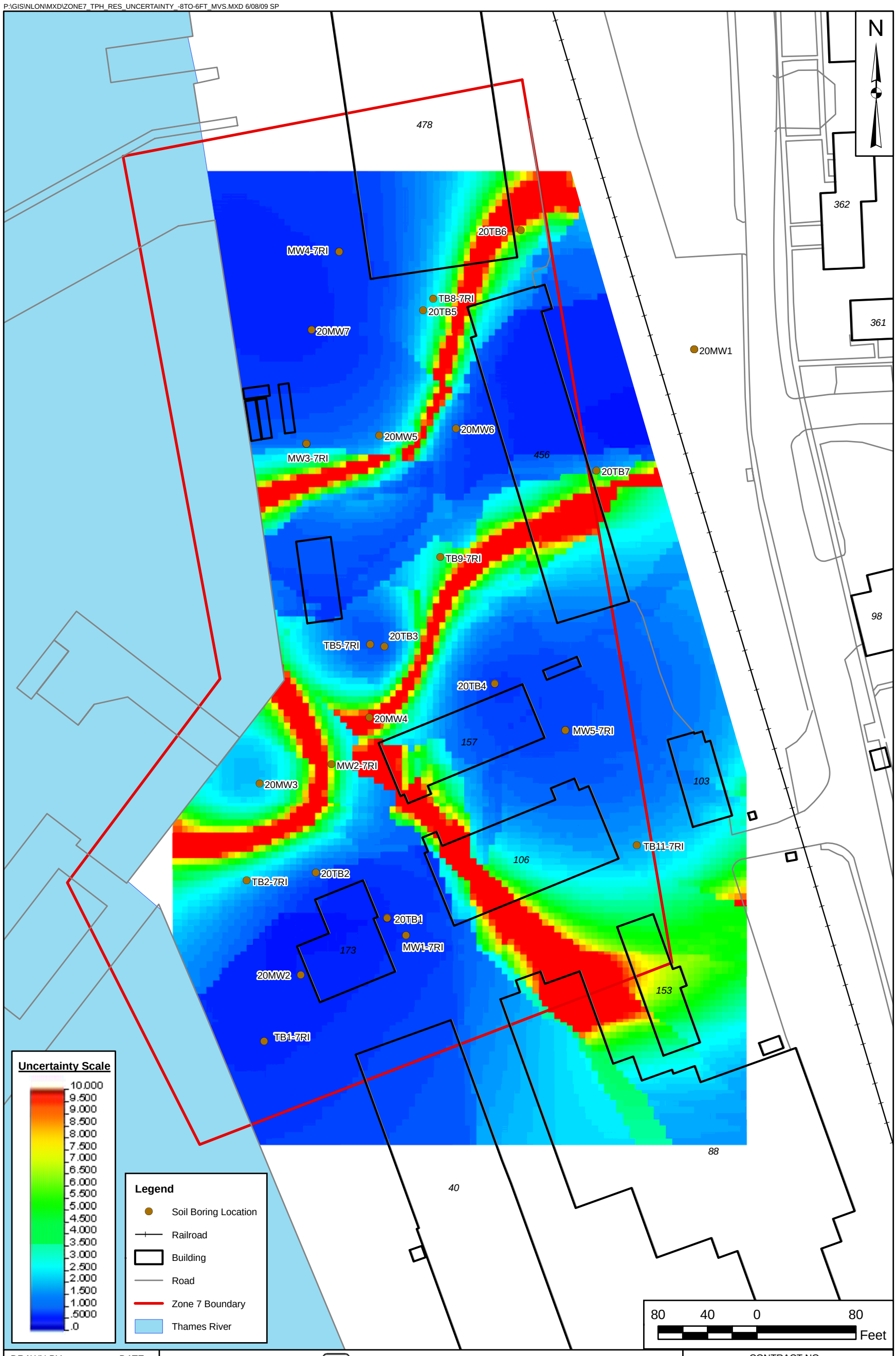
DRAWN BY S. PAXTON	DATE 4/02/09	Tetra Tech NUS, Inc.	CONTRACT NO. 0811
CHECKED BY N. BALSAMO	DATE 6/09/09	TPH UNCERTAINTY FOR RESIDENTIAL CRITERIA ELEVATION: -4 TO -2 FEET ZONE 7 NAVAL SUBMARINE BASE - NEW LONDON GROTON, CONNECTICUT	APPROVED BY _ / DATE --
COST/SCHED-AREA			APPROVED BY -- / DATE --
SCALE AS NOTED			FIGURE NO. FIGURE B-118 / REV.

P:\GIS\NLON\MXD\ZONE7_TPH_RES_UNCERTAINTY_-6TO-4FT_MVS.MXD 6/08/09 SP



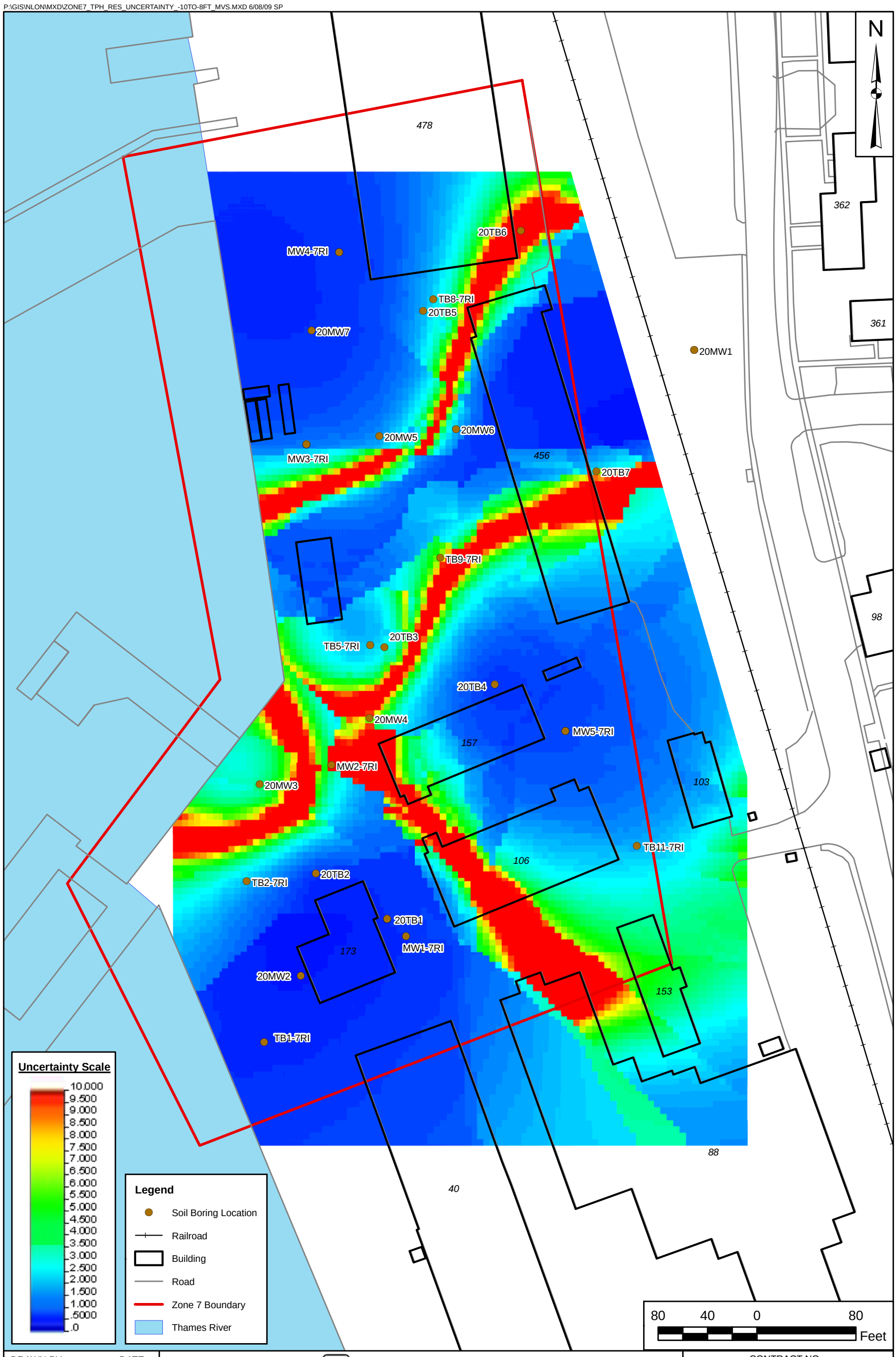
DRAWN BY S. PAXTON	DATE 4/02/09	Tetra Tech NUS, Inc.	CONTRACT NO. 0811
CHECKED BY N. BALSAMO	DATE 6/09/09	TPH UNCERTAINTY FOR RESIDENTIAL CRITERIA ELEVATION: -6 TO -4 FEET ZONE 7 NAVAL SUBMARINE BASE - NEW LONDON GROTON, CONNECTICUT	APPROVED BY _ / DATE --
COST/SCHED-AREA			APPROVED BY -- / DATE --
SCALE AS NOTED			FIGURE NO. FIGURE B-119 / REV.

P:\GIS\NLON\MXD\ZONE7_TPH_RES_UNCERTAINTY_-8TO-6FT_MVS.MXD 6/08/09 SP



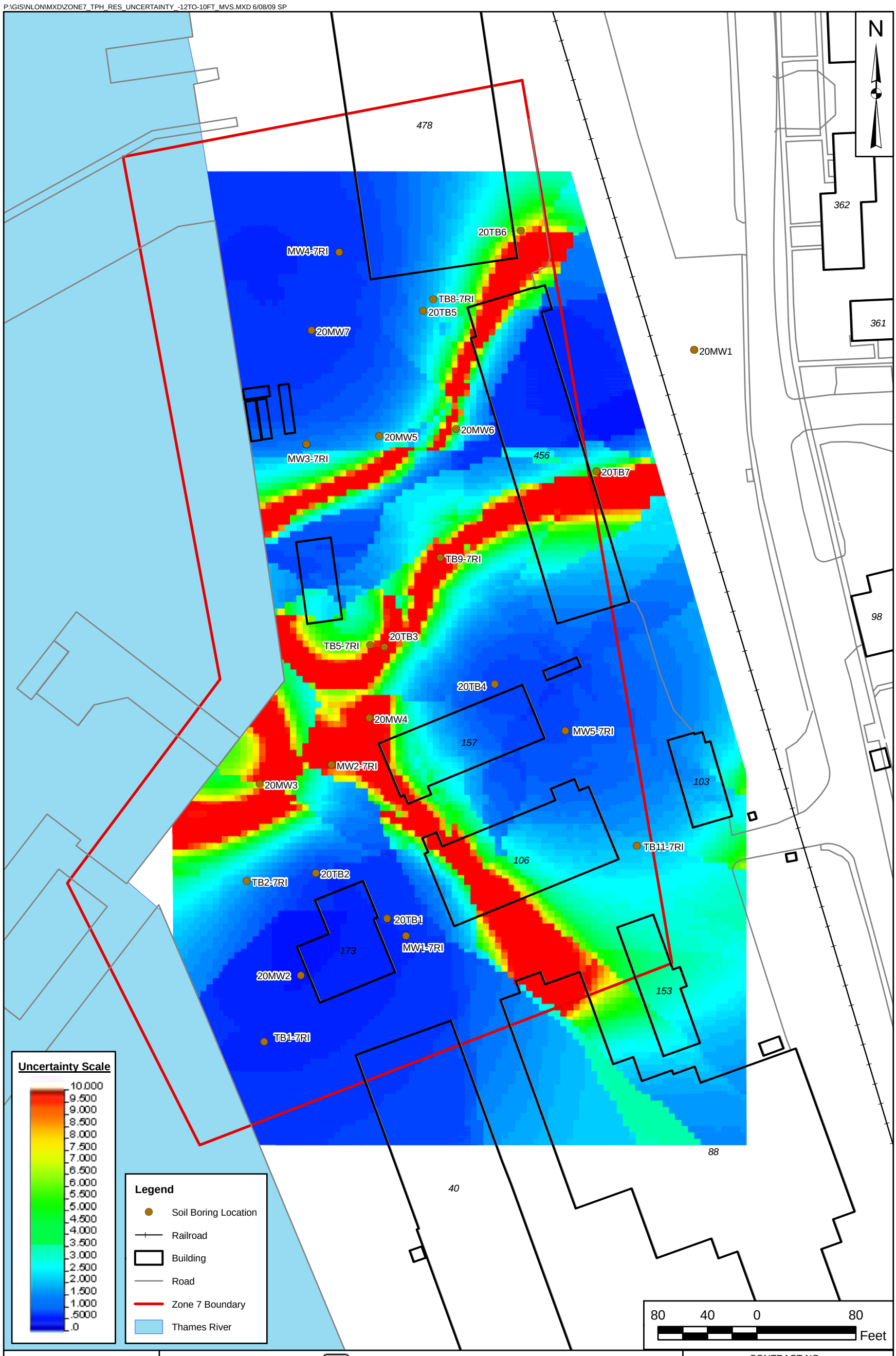
DRAWN BY S. PAXTON	DATE 4/02/09	Tetra Tech NUS, Inc.	CONTRACT NO. 0811
CHECKED BY N. BALSAMO	DATE 6/09/09	TPH UNCERTAINTY FOR RESIDENTIAL CRITERIA ELEVATION: -8 TO -6 FEET ZONE 7 NAVAL SUBMARINE BASE - NEW LONDON GROTON, CONNECTICUT	APPROVED BY _ / DATE --
COST/SCHED-AREA			APPROVED BY -- / DATE --
SCALE AS NOTED			FIGURE NO. FIGURE B-120 / REV.

P:\GIS\NLON\MXD\ZONE7_TPH_RES_UNCERTAINTY_-10TO-8FT_MVS.MXD 6/08/09 SP



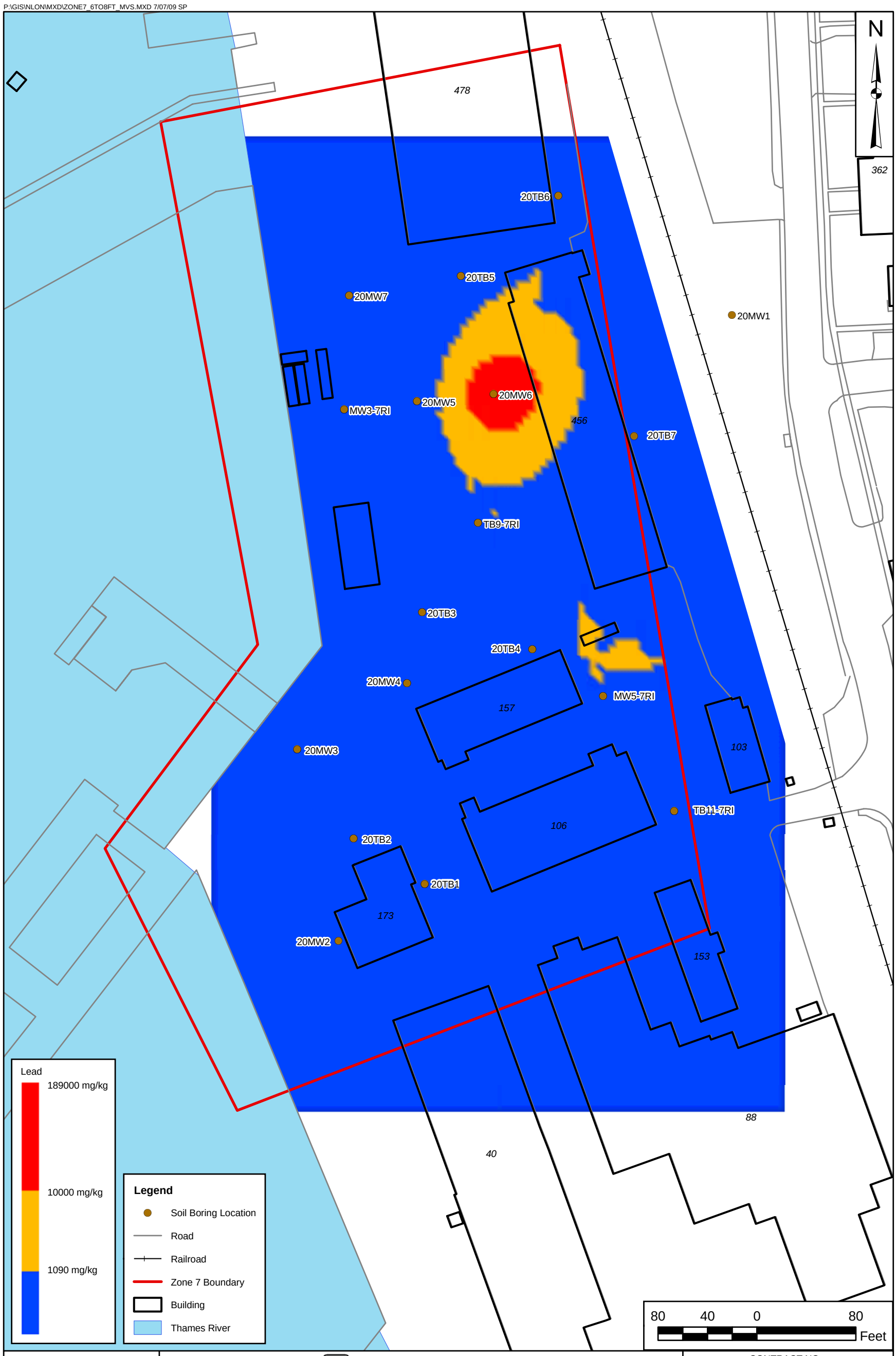
DRAWN BY S. PAXTON	DATE 4/02/09	Tetra Tech NUS, Inc.	CONTRACT NO. 0811
CHECKED BY N. BALSAMO	DATE 6/09/09	TPH UNCERTAINTY FOR RESIDENTIAL CRITERIA ELEVATION: -10 TO -8 FEET ZONE 7 NAVAL SUBMARINE BASE - NEW LONDON GROTON, CONNECTICUT	APPROVED BY _ / DATE --
COST/SCHED-AREA			APPROVED BY -- / DATE --
SCALE AS NOTED			FIGURE NO. FIGURE B-121 / REV.

P:\GIS\NLON\MXD\ZONE7_TPH_RES_UNCERTAINTY_-12TO-10FT_MVS.MXD 6/08/09 SP



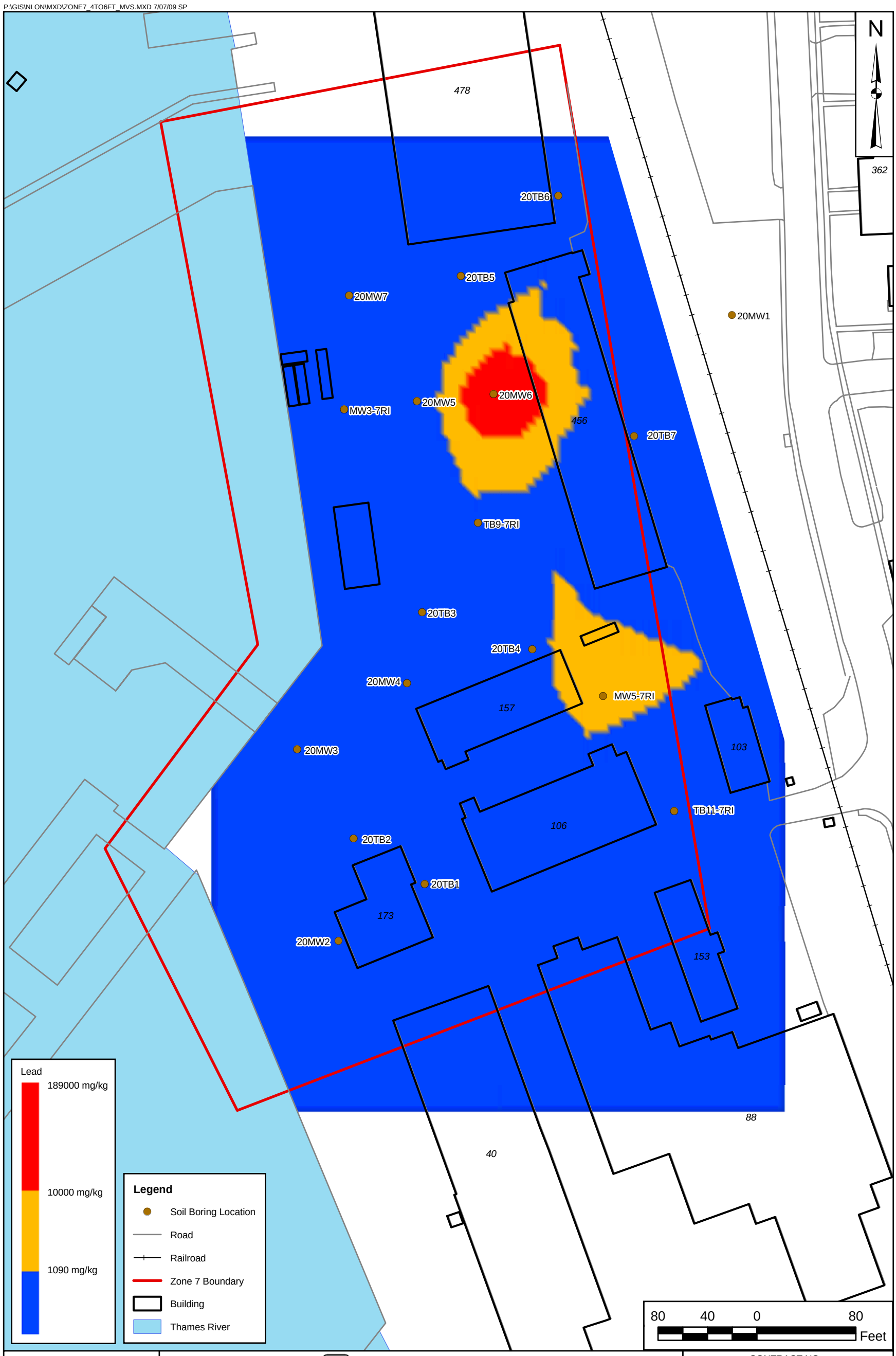
DRAWN BY S. PAXTON	DATE 4/02/09	Tetra Tech NUS, Inc.	CONTRACT NO. 0811
CHECKED BY N. BALSAMO	DATE 6/09/09	TPH UNCERTAINTY FOR RESIDENTIAL CRITERIA ELEVATION: -12 TO -10 FEET ZONE 7 NAVAL SUBMARINE BASE - NEW LONDON GROTON, CONNECTICUT	APPROVED BY _ / DATE --
COST/SCHED-AREA			APPROVED BY -- / DATE --
SCALE AS NOTED			FIGURE NO. FIGURE B-122 / REV.

P:\GIS\NLON\MXD\ZONE7_6TO8FT_MVS.MXD 7/07/09 SP



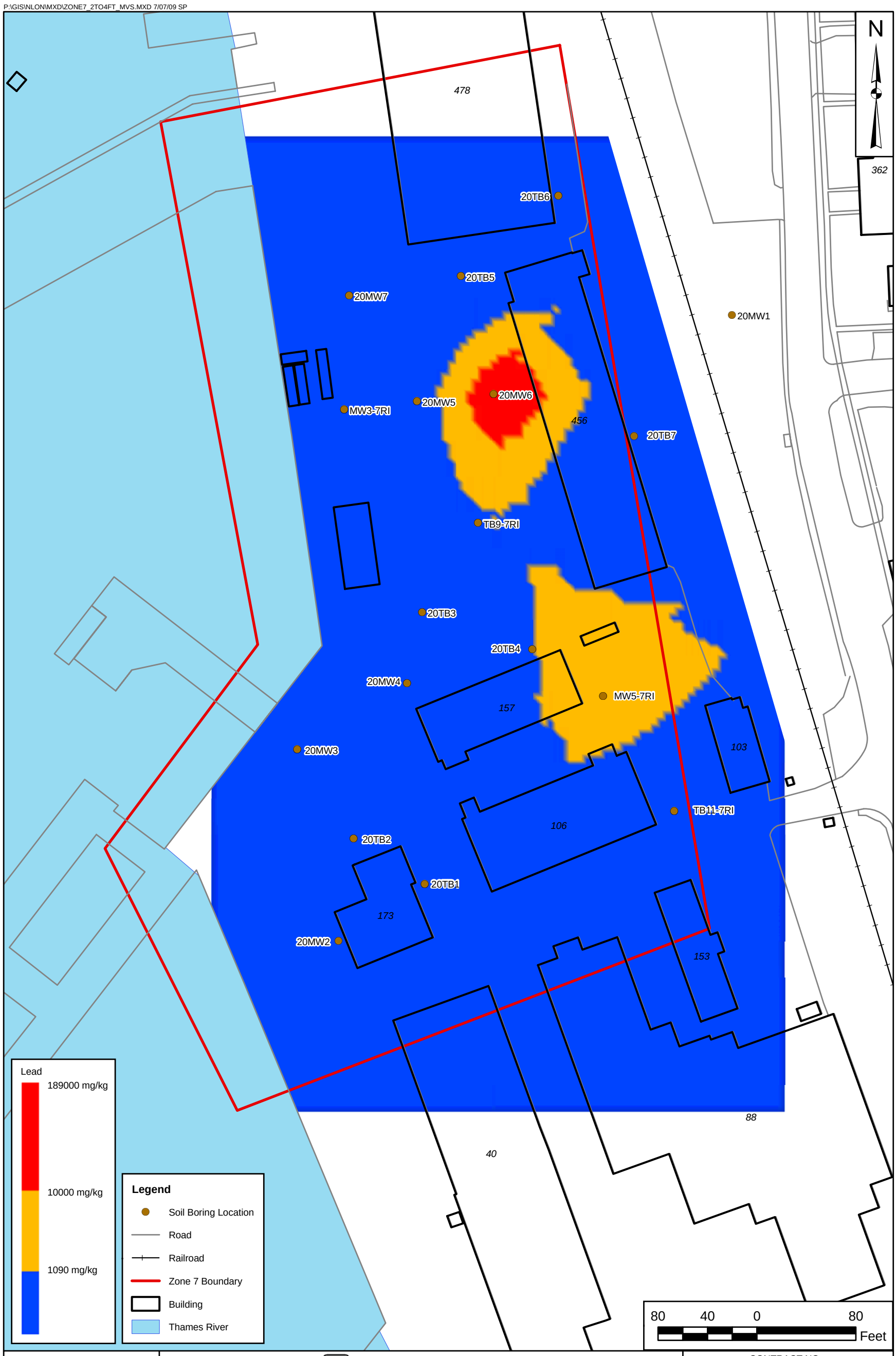
DRAWN BY S. PAXTON	DATE 4/02/09	Tetra Tech NUS, Inc.	CONTRACT NO. 0811
CHECKED BY N. BALSAMO	DATE 7/07/09	ESTIMATED LEAD CONCENTRATIONS (KRIGING RESULTS) ELEVATION: 6 TO 8 FEET ZONE 7 NAVAL SUBMARINE BASE - NEW LONDON GROTON, CONNECTICUT	APPROVED BY _ / DATE --
COST/SCHED-AREA			APPROVED BY -- / DATE --
SCALE AS NOTED			FIGURE NO. FIGURE B-123 / REV.

P:\GIS\NLON\MXD\ZONE7_4TO6FT_MVS.MXD 7/07/09 SP



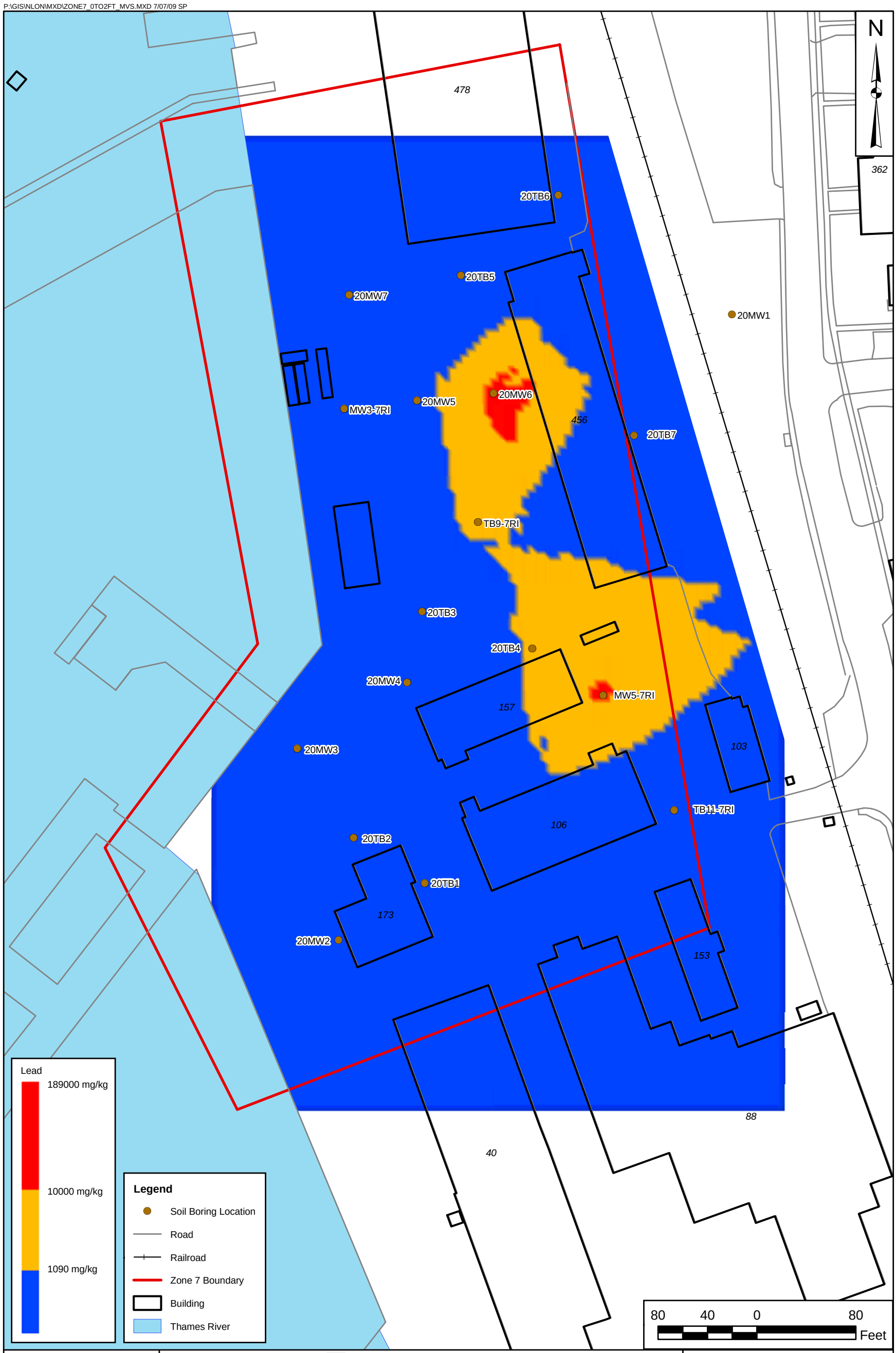
DRAWN BY S. PAXTON	DATE 4/02/09	Tetra Tech NUS, Inc.	CONTRACT NO. 0811
CHECKED BY N. BALSAMO	DATE 7/07/09	ESTIMATED LEAD CONCENTRATIONS (KRIGING RESULTS) ELEVATION: 4 TO 6 FEET ZONE 7 NAVAL SUBMARINE BASE - NEW LONDON GROTON, CONNECTICUT	APPROVED BY _ / DATE --
COST/SCHED-AREA			APPROVED BY -- / DATE --
SCALE AS NOTED			FIGURE NO. FIGURE B-124 / REV.

P:\GIS\NLON\MXD\ZONE7_2TO4FT_MVS.MXD 7/07/09 SP



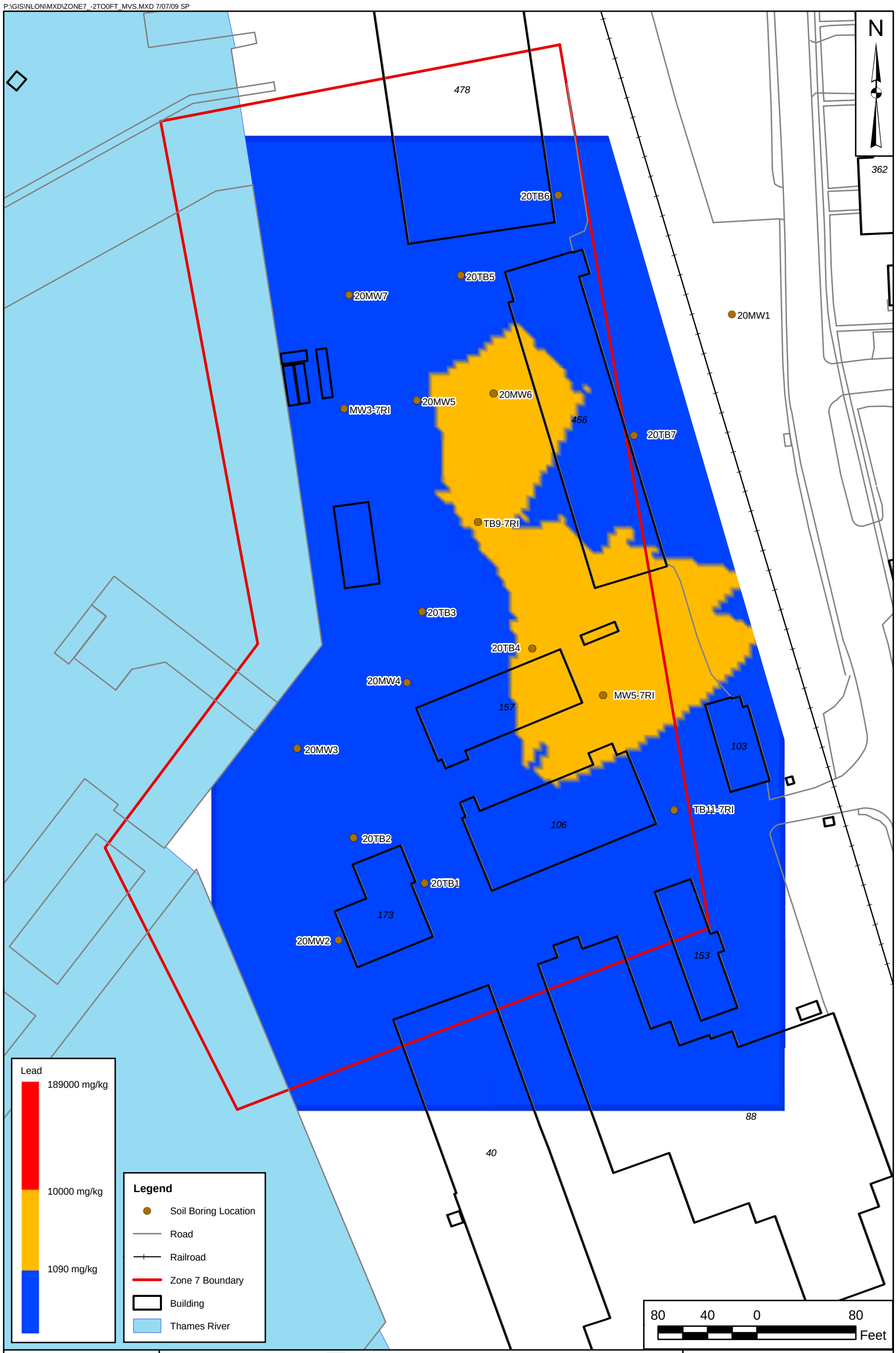
DRAWN BY S. PAXTON	DATE 4/02/09	Tetra Tech NUS, Inc.	CONTRACT NO. 0811
CHECKED BY N. BALSAMO	DATE 7/07/09	ESTIMATED LEAD CONCENTRATIONS (KRIGING RESULTS) ELEVATION: 2 TO 4 FEET ZONE 7 NAVAL SUBMARINE BASE - NEW LONDON GROTON, CONNECTICUT	APPROVED BY _ / DATE --
COST/SCHED-AREA			APPROVED BY -- / DATE --
SCALE AS NOTED			FIGURE NO. FIGURE B-125 / REV.

P:\GIS\NLON\MXD\ZONE7_0TO2FT_MVS.MXD 7/07/09 SP



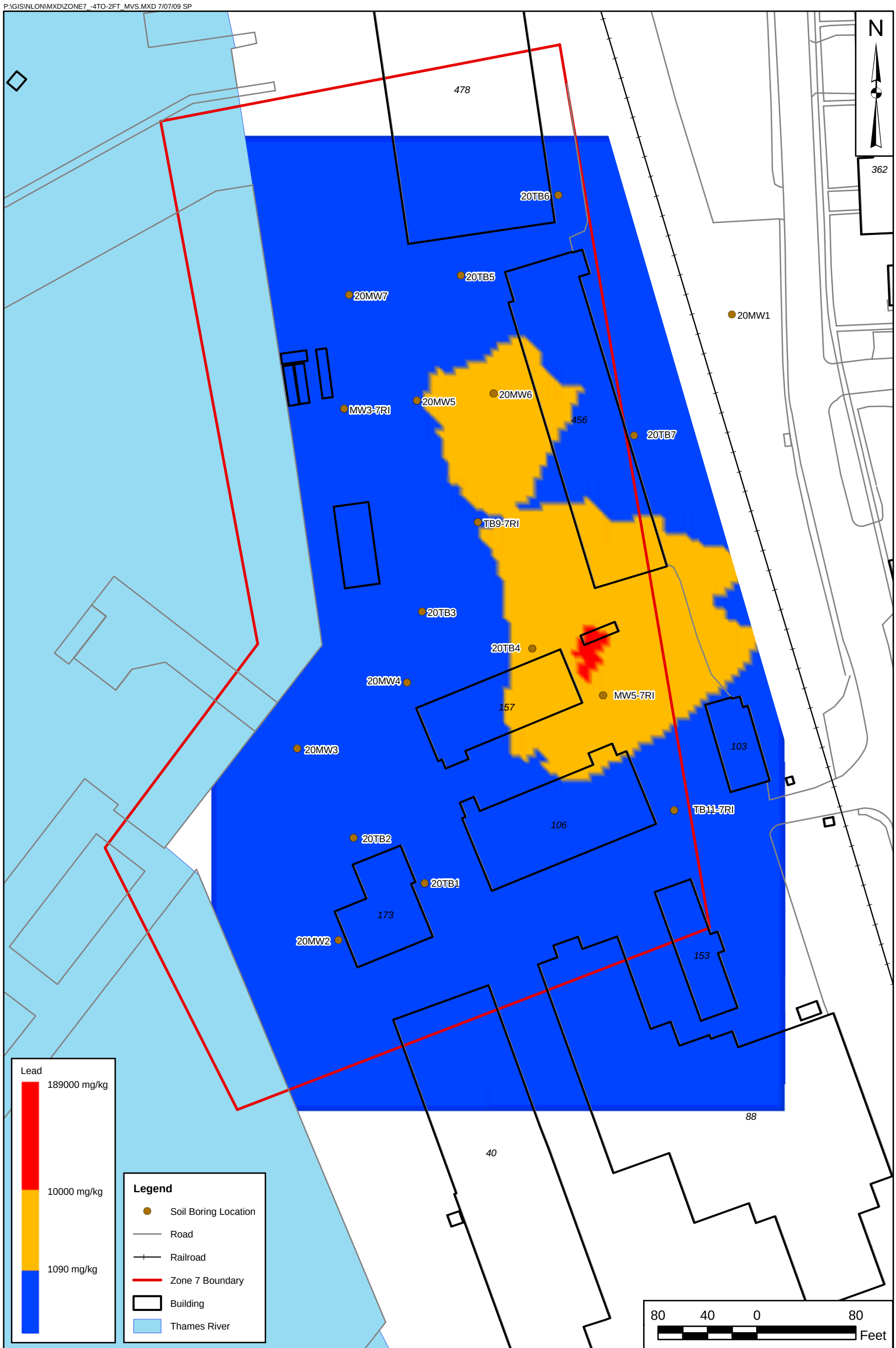
DRAWN BY	DATE	Tetra Tech NUS, Inc.	CONTRACT NO.
S. PAXTON	4/02/09		0811
CHECKED BY	DATE	ESTIMATED LEAD CONCENTRATIONS (KRIGING RESULTS) ELEVATION: 0 TO 2 FEET ZONE 7 NAVAL SUBMARINE BASE - NEW LONDON GROTON, CONNECTICUT	APPROVED BY / DATE
N. BALSAMO	7/07/09		_ / --
COST/SCHED-AREA			APPROVED BY / DATE
			-- / --
SCALE			FIGURE NO. / REV.
AS NOTED			FIGURE B-126

P:\GIS\NLON\MXD\ZONE7_-2TO0FT_MVS.MXD 7/07/09 SP



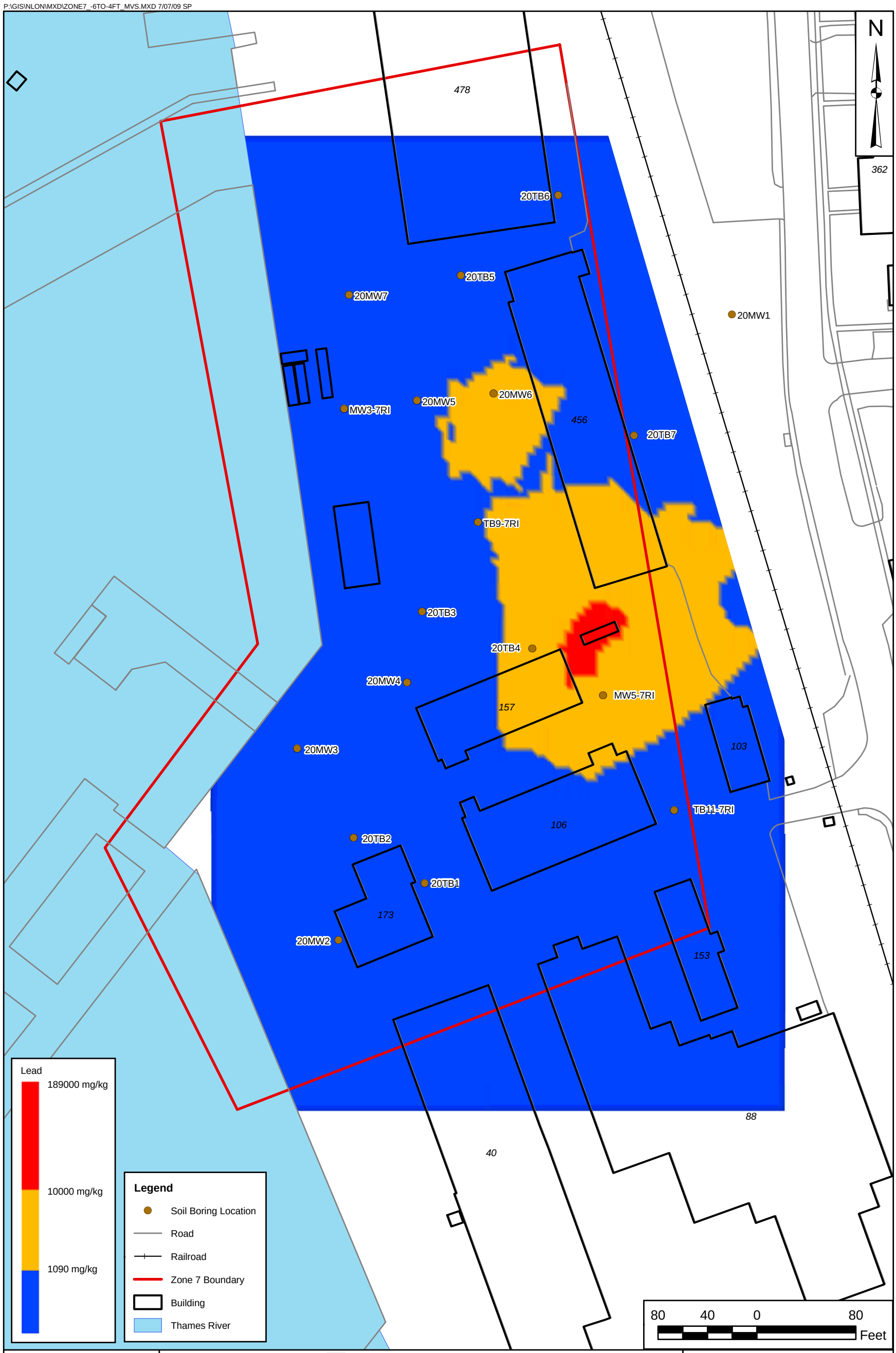
DRAWN BY	DATE	Tetra Tech NUS, Inc.	CONTRACT NO.
S. PAXTON	4/02/09		0811
CHECKED BY	DATE	ESTIMATED LEAD CONCENTRATIONS (KRIGING RESULTS) ELEVATION: -2 TO 0 FEET ZONE 7 NAVAL SUBMARINE BASE - NEW LONDON GROTON, CONNECTICUT	APPROVED BY / DATE
N. BALSAMO	7/07/09		_ / --
COST/SCHED-AREA			APPROVED BY / DATE
			-- / --
SCALE			FIGURE NO. / REV.
AS NOTED			FIGURE B-127

P:\GIS\NLON\MXD\ZONE7_-4TO-2FT_MVS.MXD 7/07/09 SP



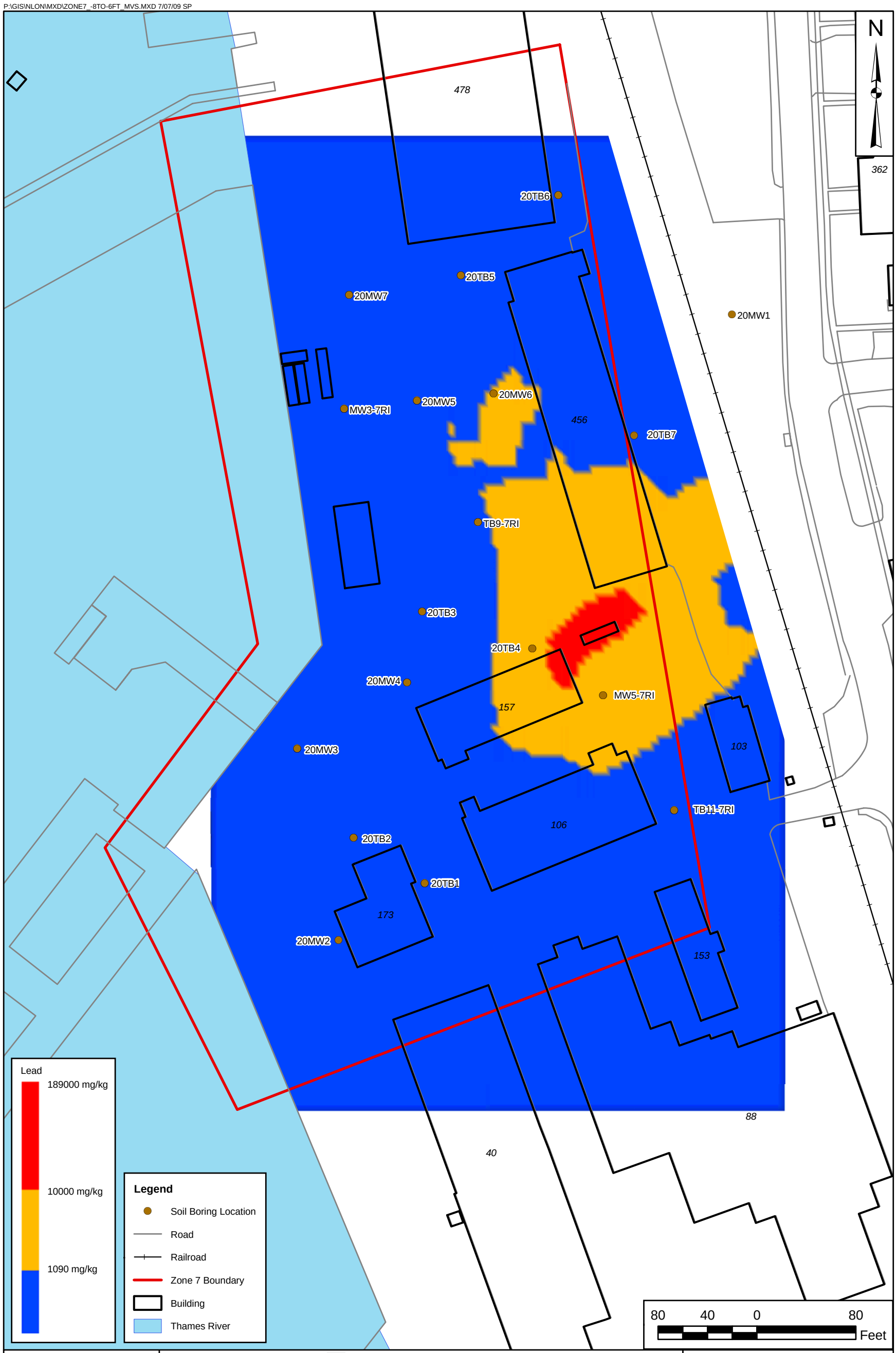
DRAWN BY S. PAXTON	DATE 4/02/09	Tetra Tech NUS, Inc.	CONTRACT NO. 0811
CHECKED BY N. BALSAMO	DATE 7/07/09	ESTIMATED LEAD CONCENTRATIONS (KRIGING RESULTS) ELEVATION: -4 TO -2 FEET ZONE 7 NAVAL SUBMARINE BASE - NEW LONDON GROTON, CONNECTICUT	APPROVED BY _ / DATE --
COST/SCHED-AREA			APPROVED BY -- / DATE --
SCALE AS NOTED			FIGURE NO. FIGURE B-128 / REV.

P:\GIS\NLON\MXD\ZONE7_-6TO-4FT_MVS.MXD 7/07/09 SP



DRAWN BY	DATE	Tetra Tech NUS, Inc.	CONTRACT NO.
S. PAXTON	4/02/09		0811
CHECKED BY	DATE	ESTIMATED LEAD CONCENTRATIONS (KRIGING RESULTS) ELEVATION: -6 TO -4 FEET ZONE 7 NAVAL SUBMARINE BASE - NEW LONDON GROTON, CONNECTICUT	APPROVED BY / DATE
N. BALSAMO	7/07/09		_ / --
COST/SCHED-AREA			APPROVED BY / DATE
			-- / --
SCALE			FIGURE NO. / REV.
AS NOTED			FIGURE B-129

P:\GIS\NLON\MXD\ZONE7_-8TO-6FT_MVS.MXD 7/07/09 SP



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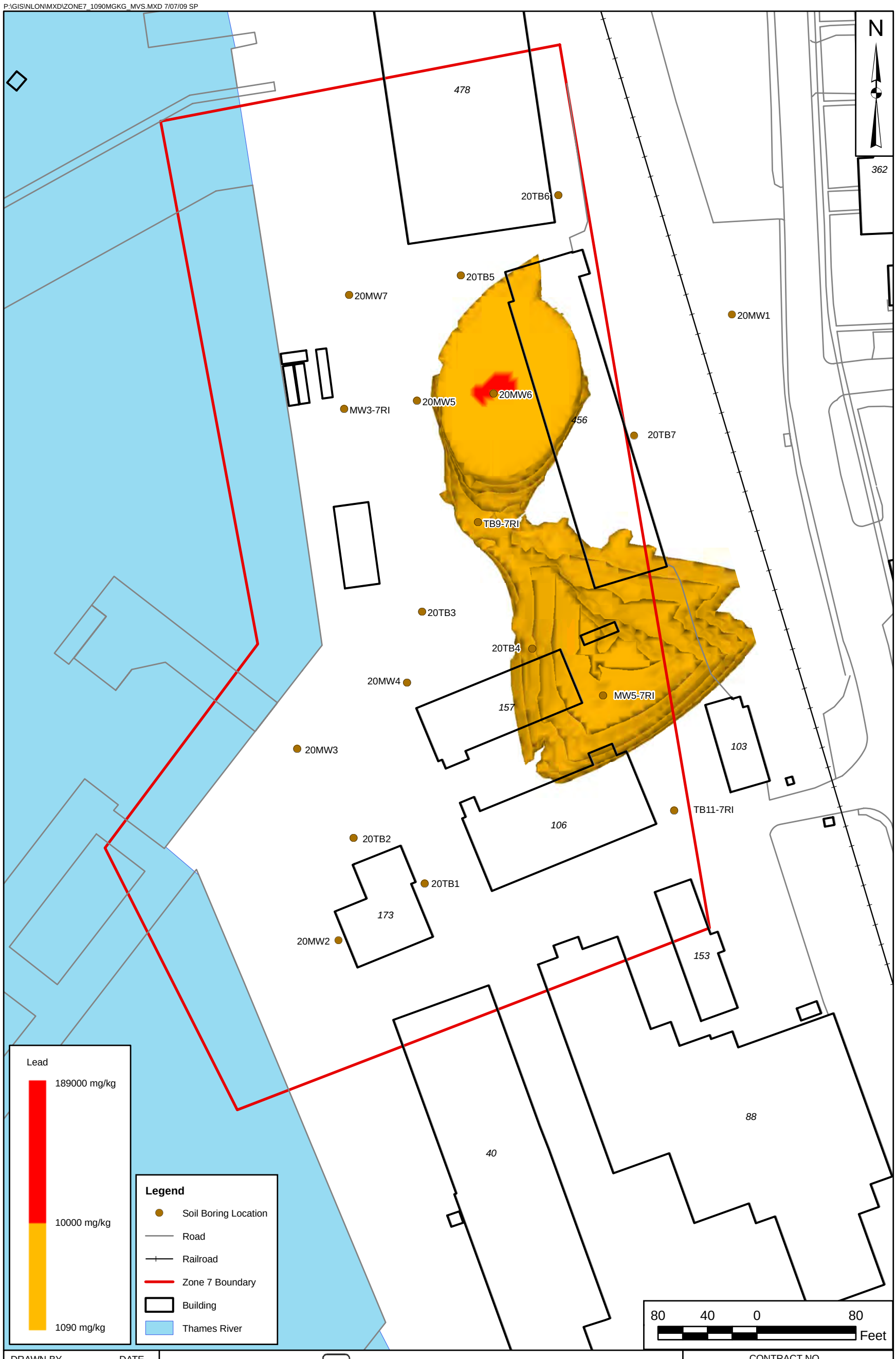
N. BALSAMO	7/07/09		_ / --
COST/SCHED-AREA			APPROVED BY / DATE
			-- / --
SCALE			FIGURE NO. / REV.
AS NOTED			FIGURE B-130

P:\GIS\NLON\MXD\ZONE7_1090MGKG_MVS.MXD 7/07/09 SP



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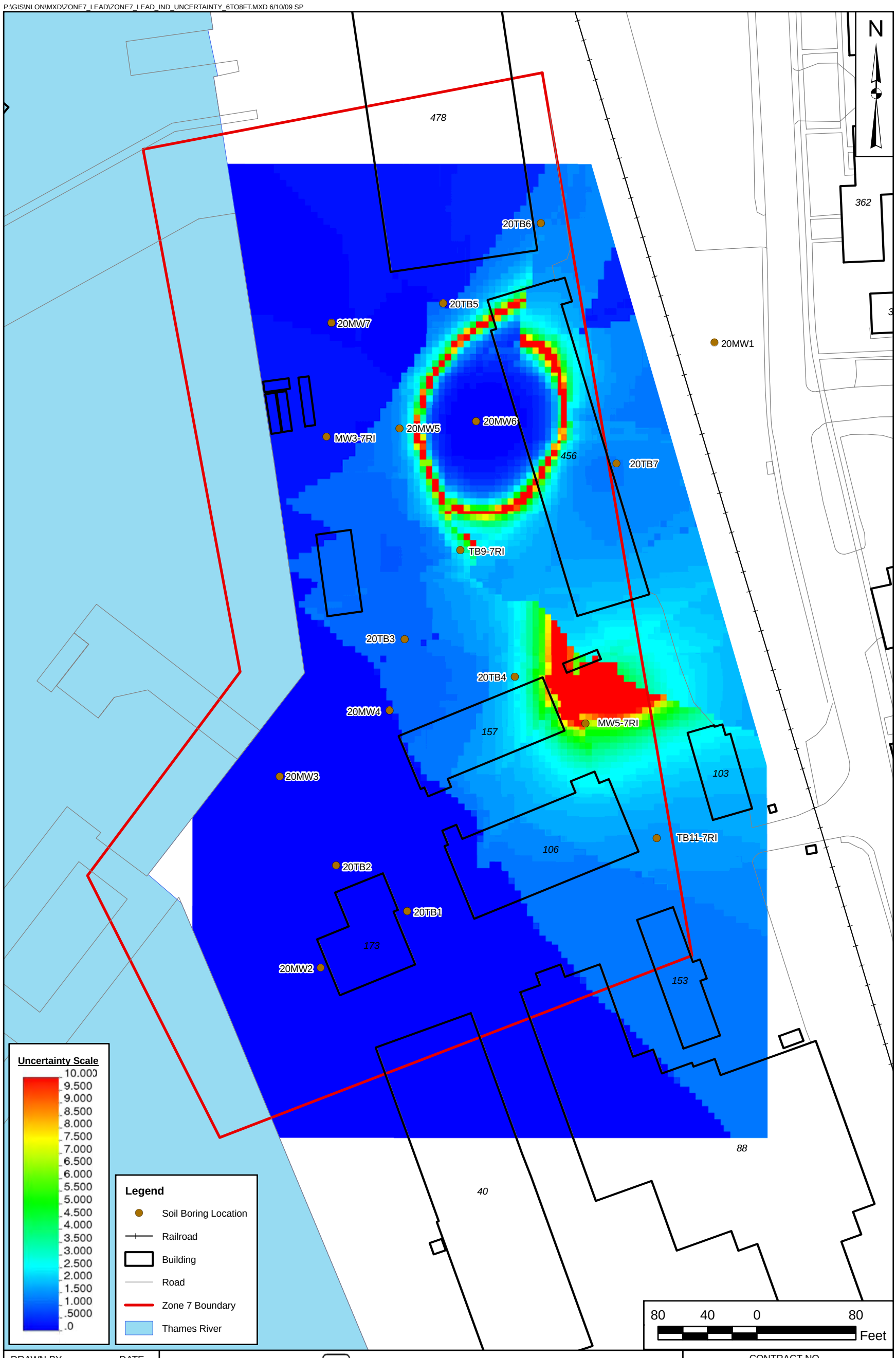
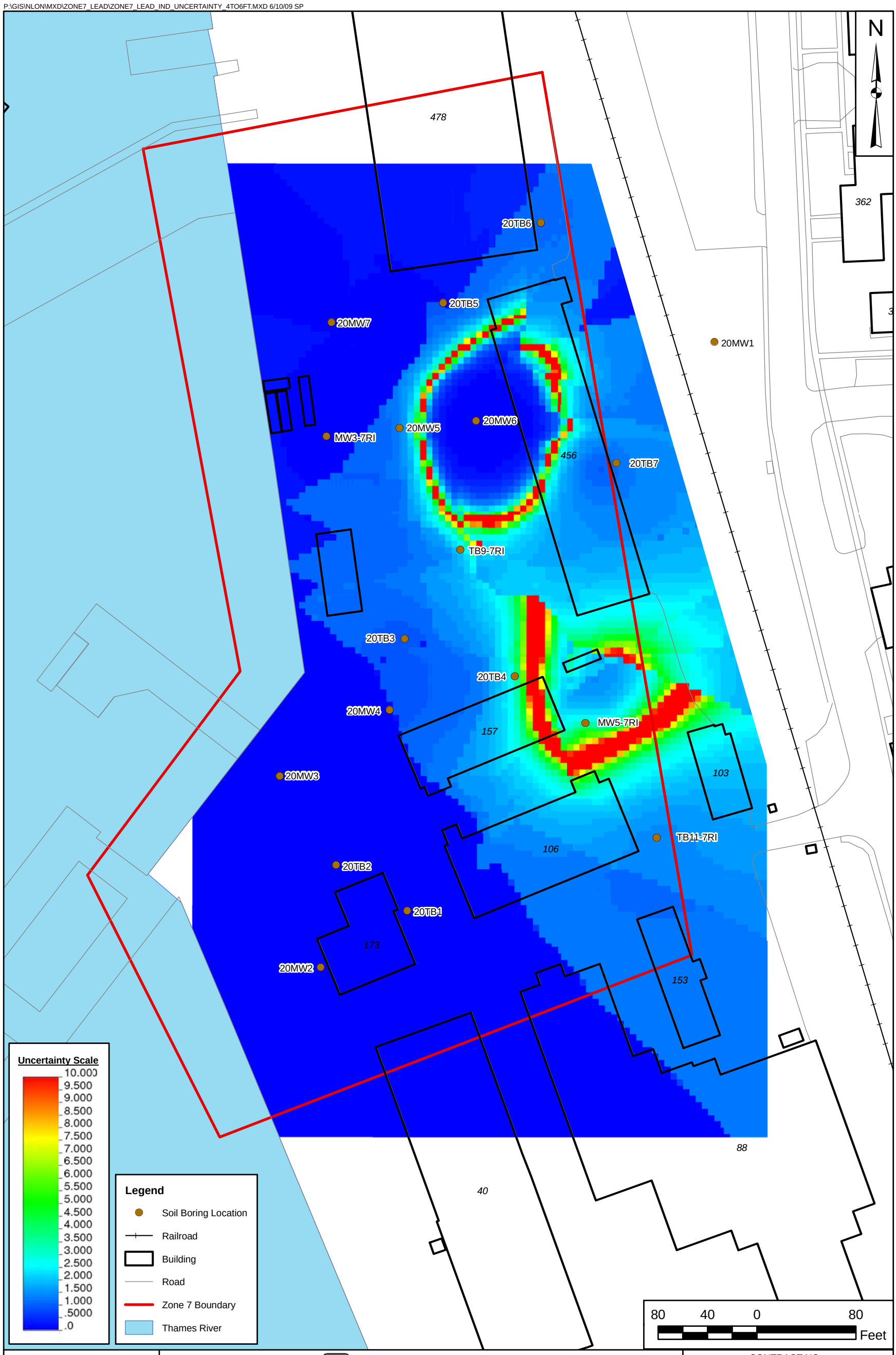
S. PAXTON	4/02/09		0811
CHECKED BY	DATE	ESTIMATED LEAD CONCENTRATIONS (KRIGING RESULTS) ELEVATION: 10 TO 0 FEET ZONE 7 NAVAL SUBMARINE BASE - NEW LONDON GROTON, CONNECTICUT	APPROVED BY / DATE
N. BALSAMO	7/07/09		_ / --
COST/SCHED-AREA			APPROVED BY / DATE
			-- / --
SCALE			FIGURE NO. / REV.
AS NOTED			FIGURE B-131

P:\GIS\NLON\MXD\ZONE7_LEAD\ZONE7_LEAD_IND_UNCERTAINTY_6TO8FT.MXD 6/10/09 SP



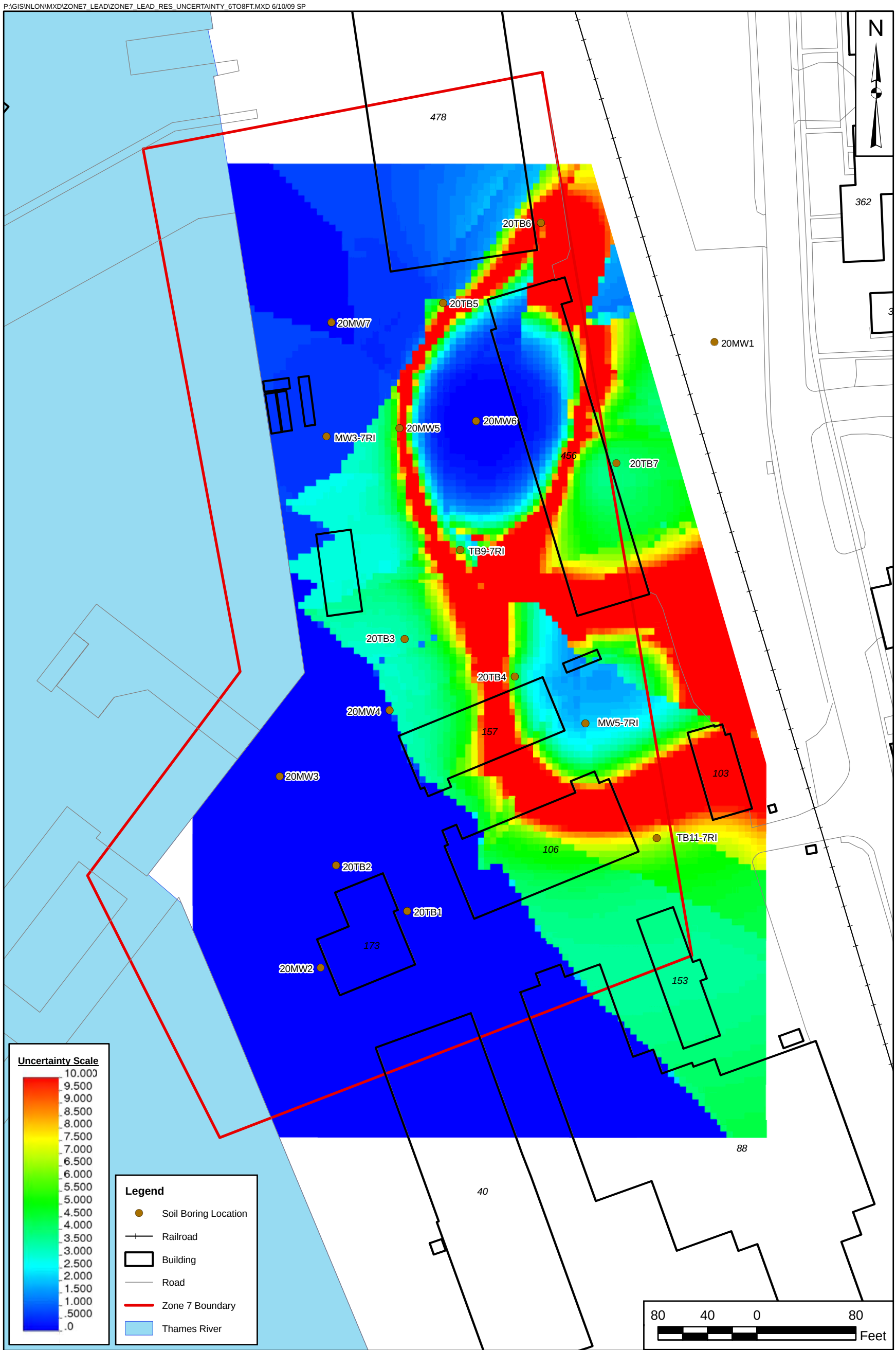
DRAWN BY	DATE	Tetra Tech NUS, Inc.	CONTRACT NO.
S. PAXTON	4/02/09		0811
CHECKED BY	DATE	LEAD UNCERTAINTY FOR INDUSTRIAL CRITERIA ELEVATION: 6 TO 8 FEET ZONE 7 NAVAL SUBMARINE BASE - NEW LONDON GROTON, CONNECTICUT	APPROVED BY / DATE
N. BALSAMO	6/10/09		_ / --
COST/SCHED-AREA			APPROVED BY / DATE
			-- / --
SCALE			FIGURE NO. / REV.
AS NOTED			FIGURE B-132

P:\GIS\NLON\MXD\ZONE7_LEAD\ZONE7_LEAD_IND_UNCERTAINTY_4TO6FT.MXD 6/10/09 SP



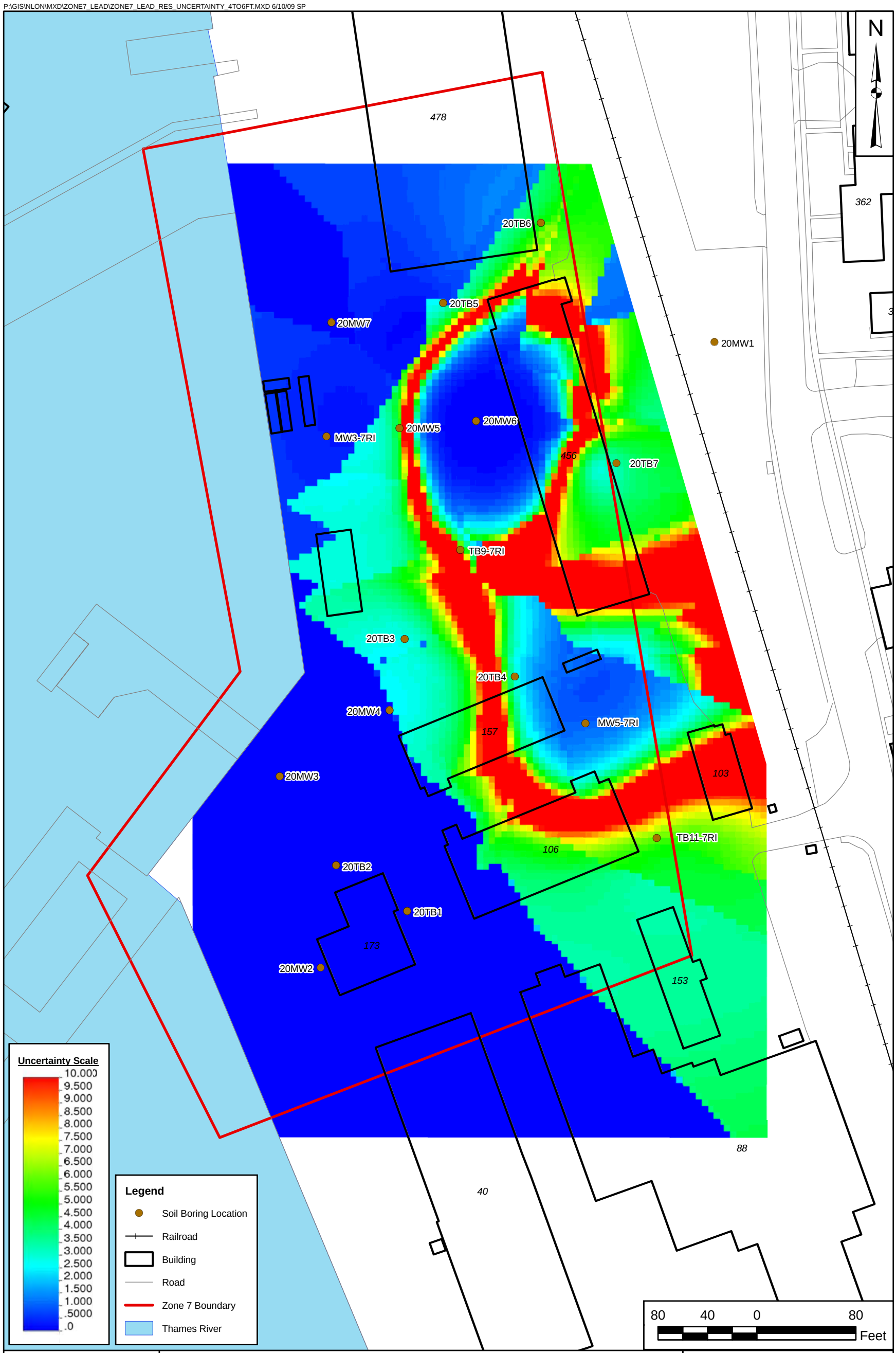
DRAWN BY S. PAXTON	DATE 4/02/09	Tetra Tech NUS, Inc.	CONTRACT NO. 0811
CHECKED BY N. BALSAMO	DATE 6/10/09	LEAD UNCERTAINTY FOR INDUSTRIAL CRITERIA ELEVATION: 4 TO 6 FEET ZONE 7 NAVAL SUBMARINE BASE - NEW LONDON GROTON, CONNECTICUT	APPROVED BY _ / DATE --
COST/SCHED-AREA			APPROVED BY -- / DATE --
SCALE AS NOTED			FIGURE NO. FIGURE B-133 / REV.

P:\GIS\NLON\MXD\ZONE7_LEAD\ZONE7_LEAD_RES_UNCERTAINTY_6TO8FT.MXD 6/10/09 SP



DRAWN BY S. PAXTON	DATE 4/02/09	Tetra Tech NUS, Inc.	CONTRACT NO. 0811
CHECKED BY N. BALSAMO	DATE 6/10/09	LEAD UNCERTAINTY FOR RESIDENTIAL CRITERIA ELEVATION: 6 TO 8 FEET ZONE 7 NAVAL SUBMARINE BASE - NEW LONDON GROTON, CONNECTICUT	APPROVED BY _ / DATE --
COST/SCHED-AREA			APPROVED BY -- / DATE --
SCALE AS NOTED			FIGURE NO. FIGURE B-134 / REV.

P:\GIS\NLON\MXD\ZONE7_LEAD\ZONE7_LEAD_RES_UNCERTAINTY_4TO6FT.MXD 6/10/09 SP



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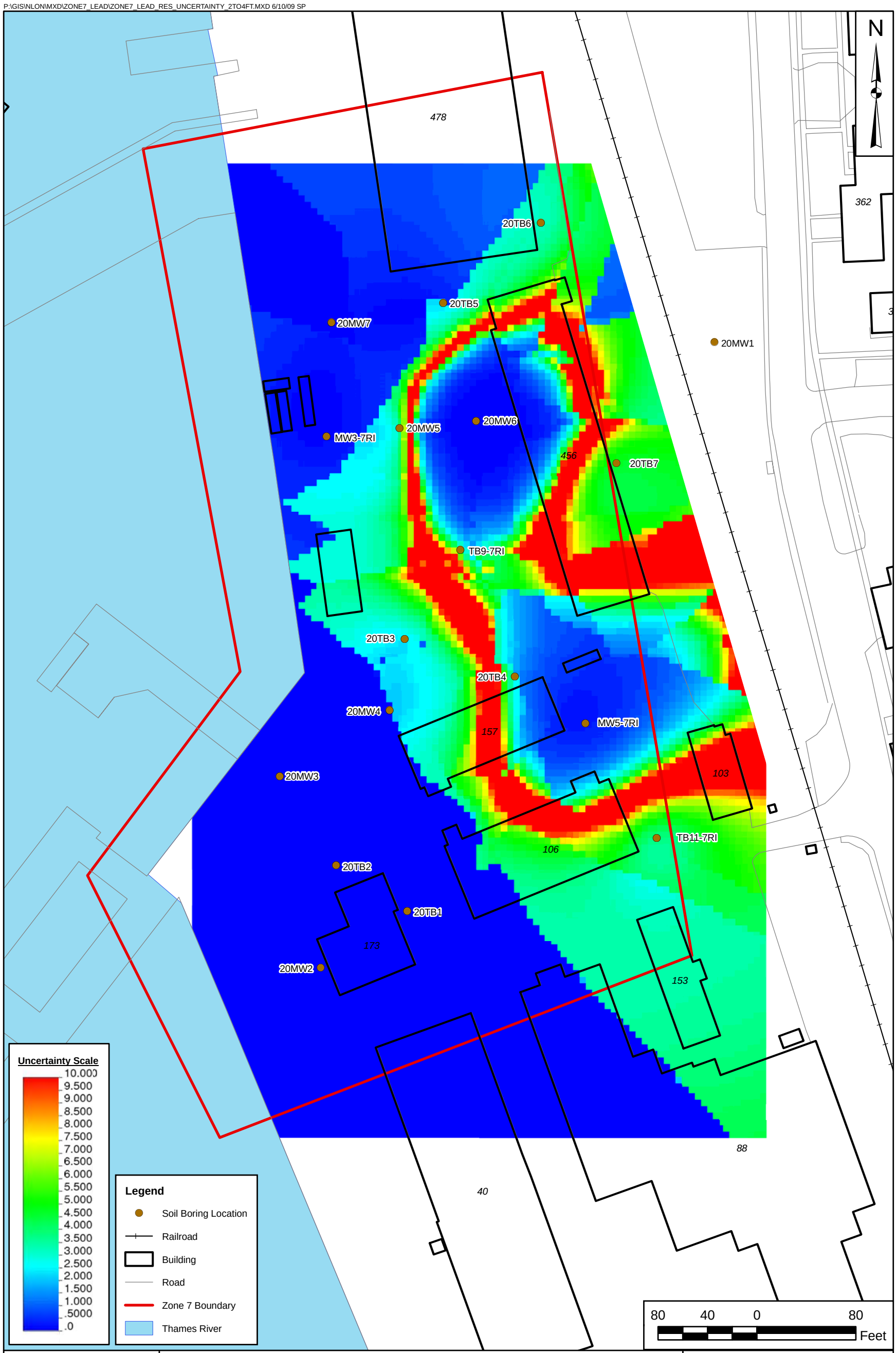
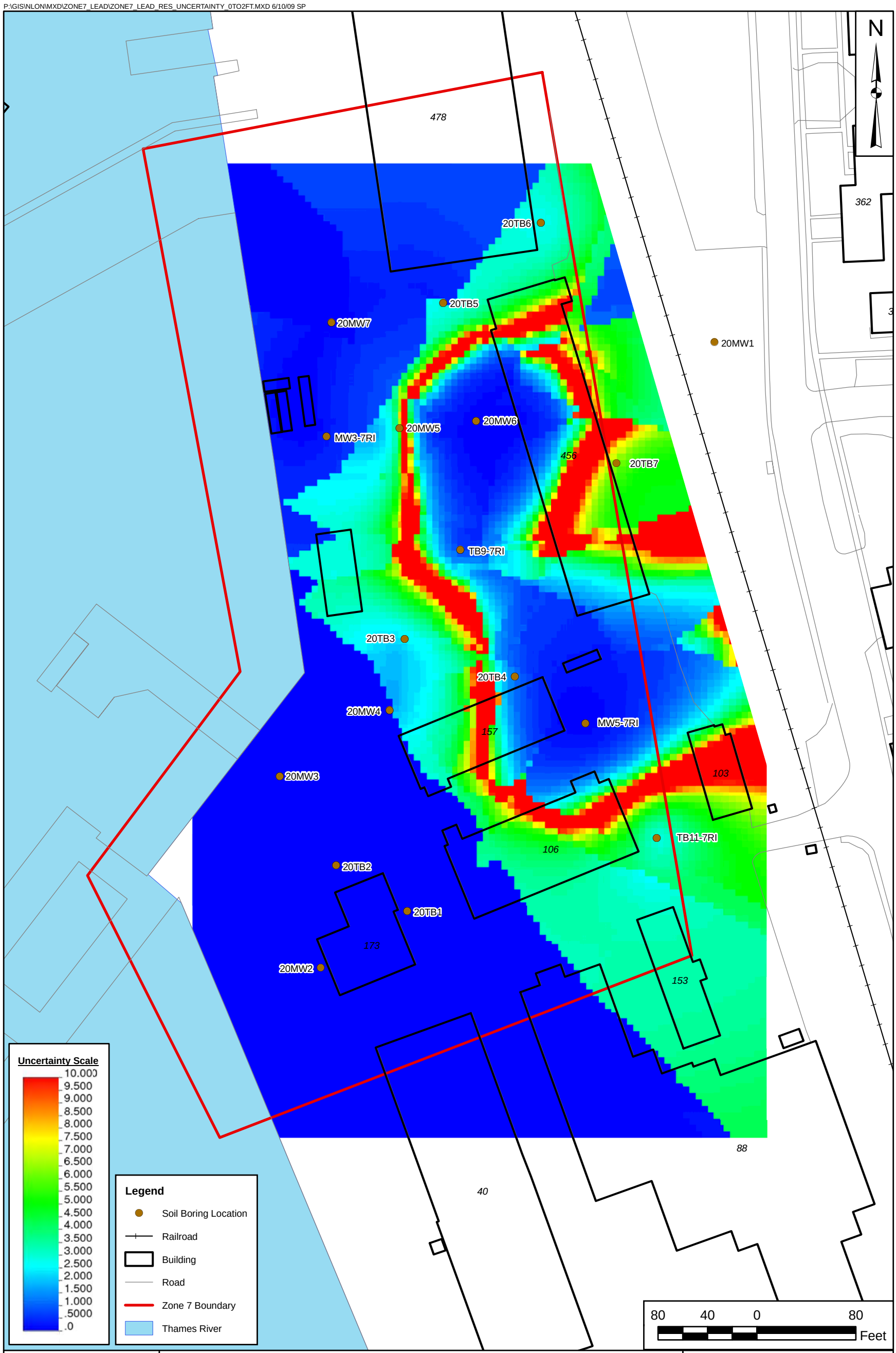
CHECKED BY N. BALSAMO	DATE 6/10/09	LEAD UNCERTAINTY FOR RESIDENTIAL CRITERIA ELEVATION: 4 TO 6 FEET ZONE 7 NAVAL SUBMARINE BASE - NEW LONDON GROTON, CONNECTICUT	APPROVED BY _ / DATE --
COST/SCHED-AREA			APPROVED BY -- / DATE --
SCALE AS NOTED			FIGURE NO. FIGURE B-135 / REV.

P:\GIS\NLON\MXD\ZONE7_LEAD\ZONE7_LEAD_RES_UNCERTAINTY_2TO4FT.MXD 6/10/09 SP



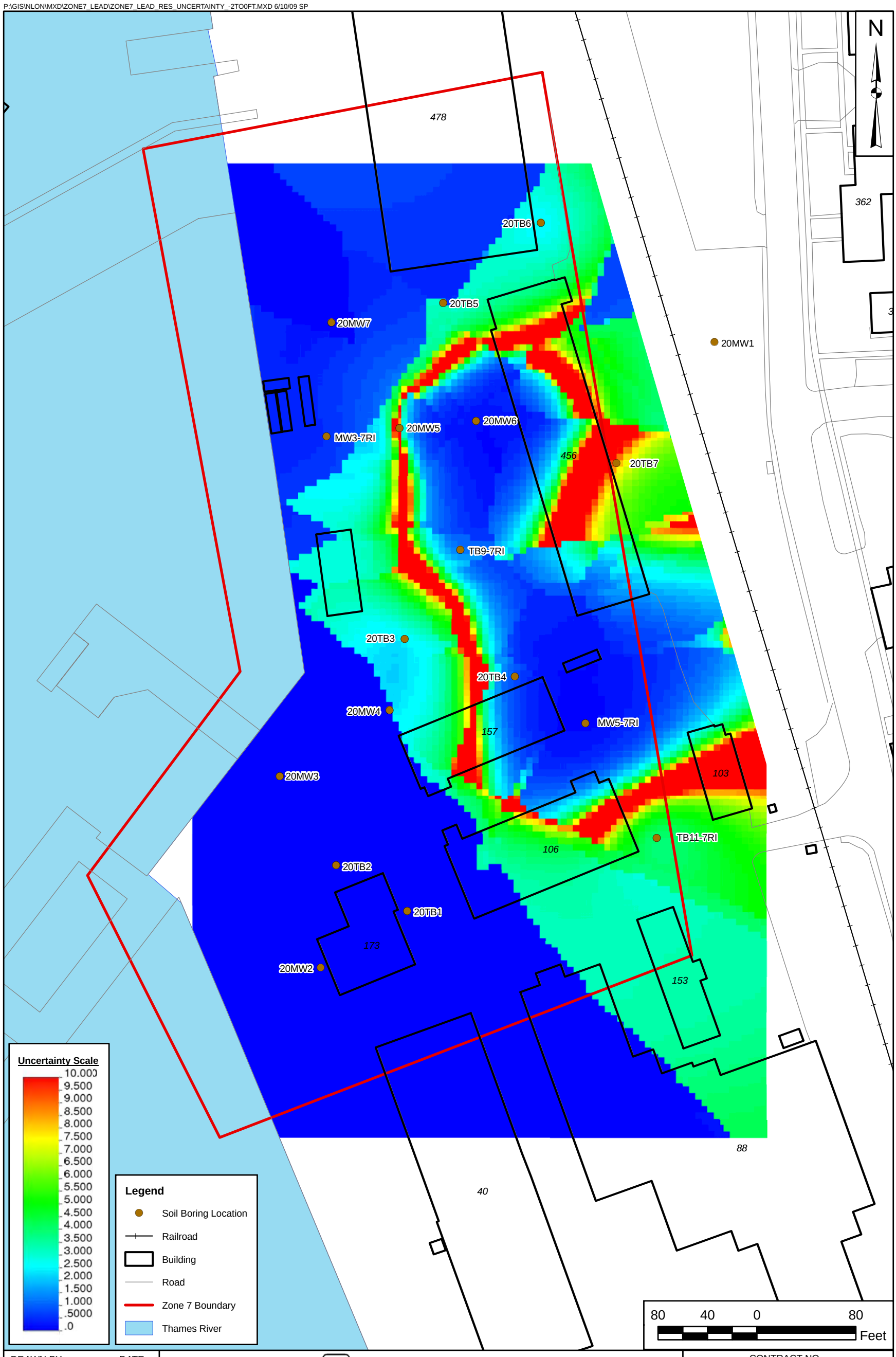
DRAWN BY S. PAXTON	DATE 4/02/09	Tetra Tech NUS, Inc.	CONTRACT NO. 0811
CHECKED BY N. BALSAMO	DATE 6/10/09	LEAD UNCERTAINTY FOR RESIDENTIAL CRITERIA ELEVATION: 2 TO 4 FEET ZONE 7 NAVAL SUBMARINE BASE - NEW LONDON GROTON, CONNECTICUT	APPROVED BY _ / DATE --
COST/SCHED-AREA			APPROVED BY -- / DATE --
SCALE AS NOTED			FIGURE NO. FIGURE B-136 / REV.

P:\GIS\NLON\MXD\ZONE7_LEAD\ZONE7_LEAD_RES_UNCERTAINTY_0TO2FT.MXD 6/10/09 SP



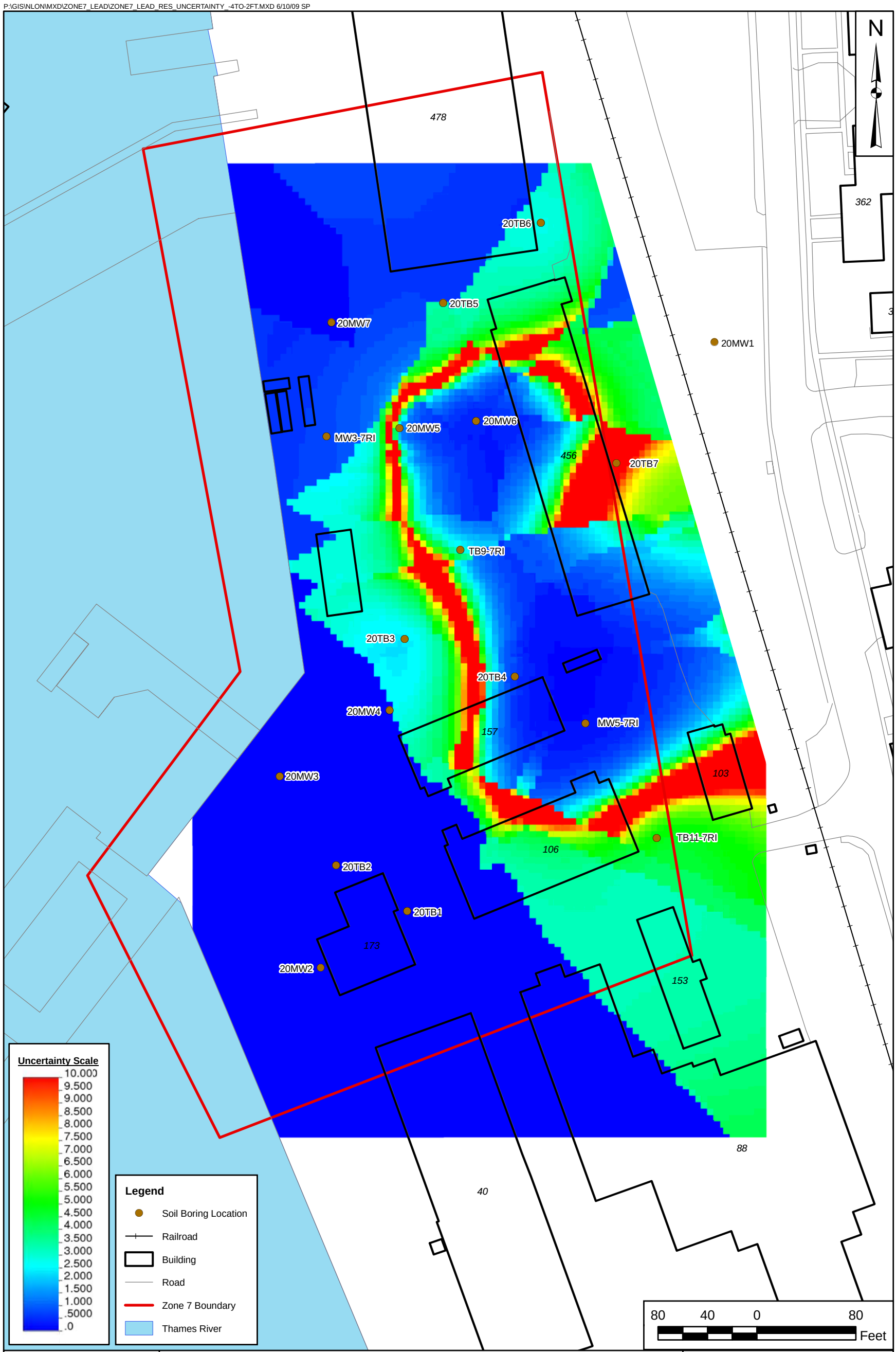
DRAWN BY S. PAXTON	DATE 4/02/09	Tetra Tech NUS, Inc.	CONTRACT NO. 0811
CHECKED BY N. BALSAMO	DATE 6/10/09	LEAD UNCERTAINTY FOR RESIDENTIAL CRITERIA ELEVATION: 0 TO 2 FEET ZONE 7 NAVAL SUBMARINE BASE - NEW LONDON GROTON, CONNECTICUT	APPROVED BY _ / DATE --
COST/SCHED-AREA			APPROVED BY -- / DATE --
SCALE AS NOTED			FIGURE NO. FIGURE B-137 / REV.

P:\GIS\NLON\MXD\ZONE7_LEAD\ZONE7_LEAD_RES_UNCERTAINTY_-2TO0FT.MXD 6/10/09 SP



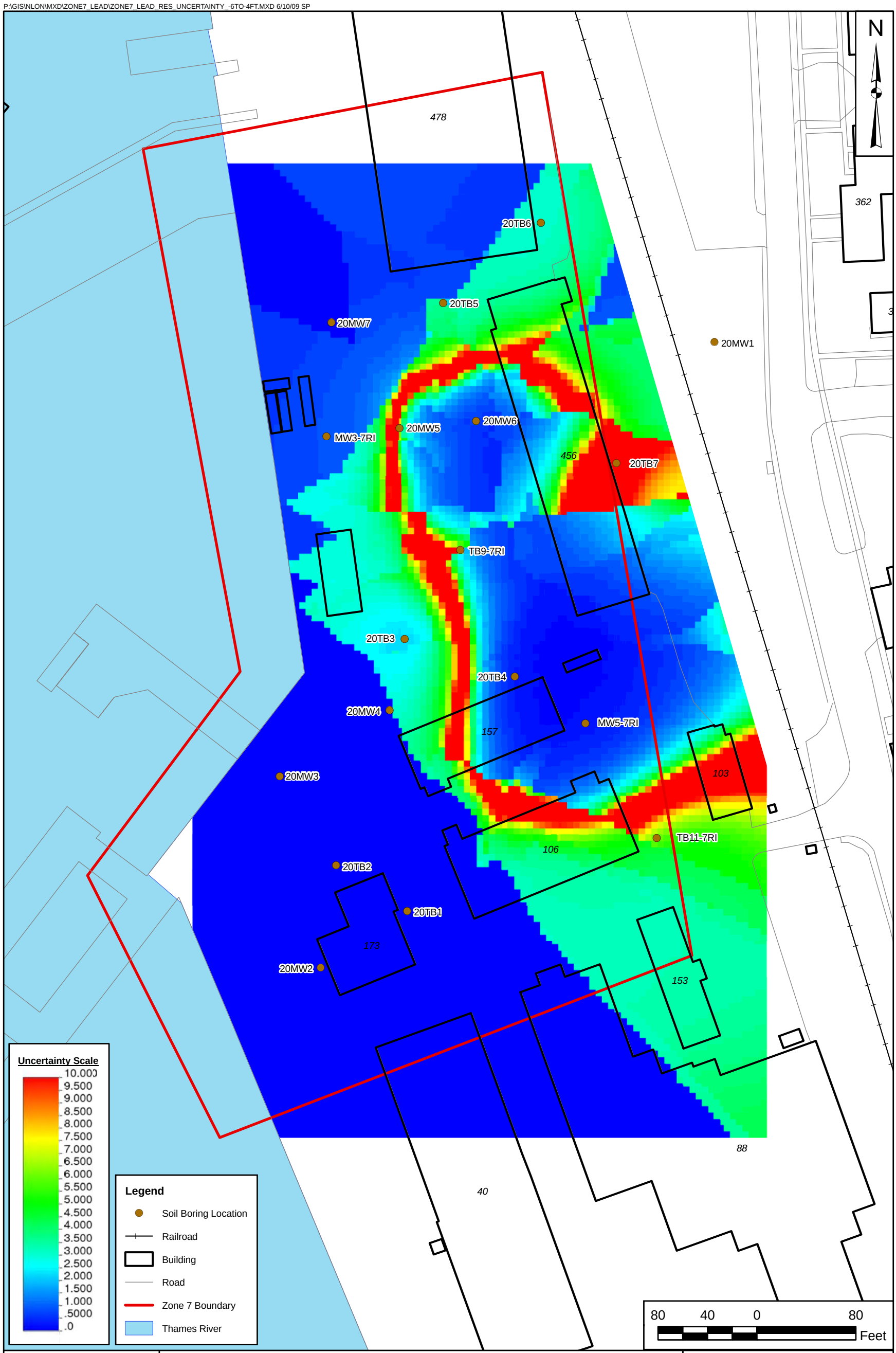
DRAWN BY S. PAXTON	DATE 4/02/09	Tetra Tech NUS, Inc.	CONTRACT NO. 0811
CHECKED BY N. BALSAMO	DATE 6/10/09	LEAD UNCERTAINTY FOR RESIDENTIAL CRITERIA ELEVATION: -2 TO 0 FEET ZONE 7 NAVAL SUBMARINE BASE - NEW LONDON GROTON, CONNECTICUT	APPROVED BY _ / DATE --
COST/SCHED-AREA			APPROVED BY -- / DATE --
SCALE AS NOTED			FIGURE NO. FIGURE B-138 / REV.

P:\GIS\NLON\MXD\ZONE7_LEAD\ZONE7_LEAD_RES_UNCERTAINTY_-4TO-2FT.MXD 6/10/09 SP



DRAWN BY S. PAXTON	DATE 4/02/09	Tetra Tech NUS, Inc.	CONTRACT NO. 0811
CHECKED BY N. BALSAMO	DATE 6/10/09	LEAD UNCERTAINTY FOR RESIDENTIAL CRITERIA ELEVATION: -4 TO -2 FEET ZONE 7 NAVAL SUBMARINE BASE - NEW LONDON GROTON, CONNECTICUT	APPROVED BY _ / DATE --
COST/SCHED-AREA			APPROVED BY -- / DATE --
SCALE AS NOTED			FIGURE NO. FIGURE B-139 / REV.

P:\GIS\NLON\MXD\ZONE7_LEAD\ZONE7_LEAD_RES_UNCERTAINTY_-6TO-4FT.MXD 6/10/09 SP



DRAWN BY S. PAXTON	DATE 4/02/09	Tetra Tech NUS, Inc.	CONTRACT NO. 0811
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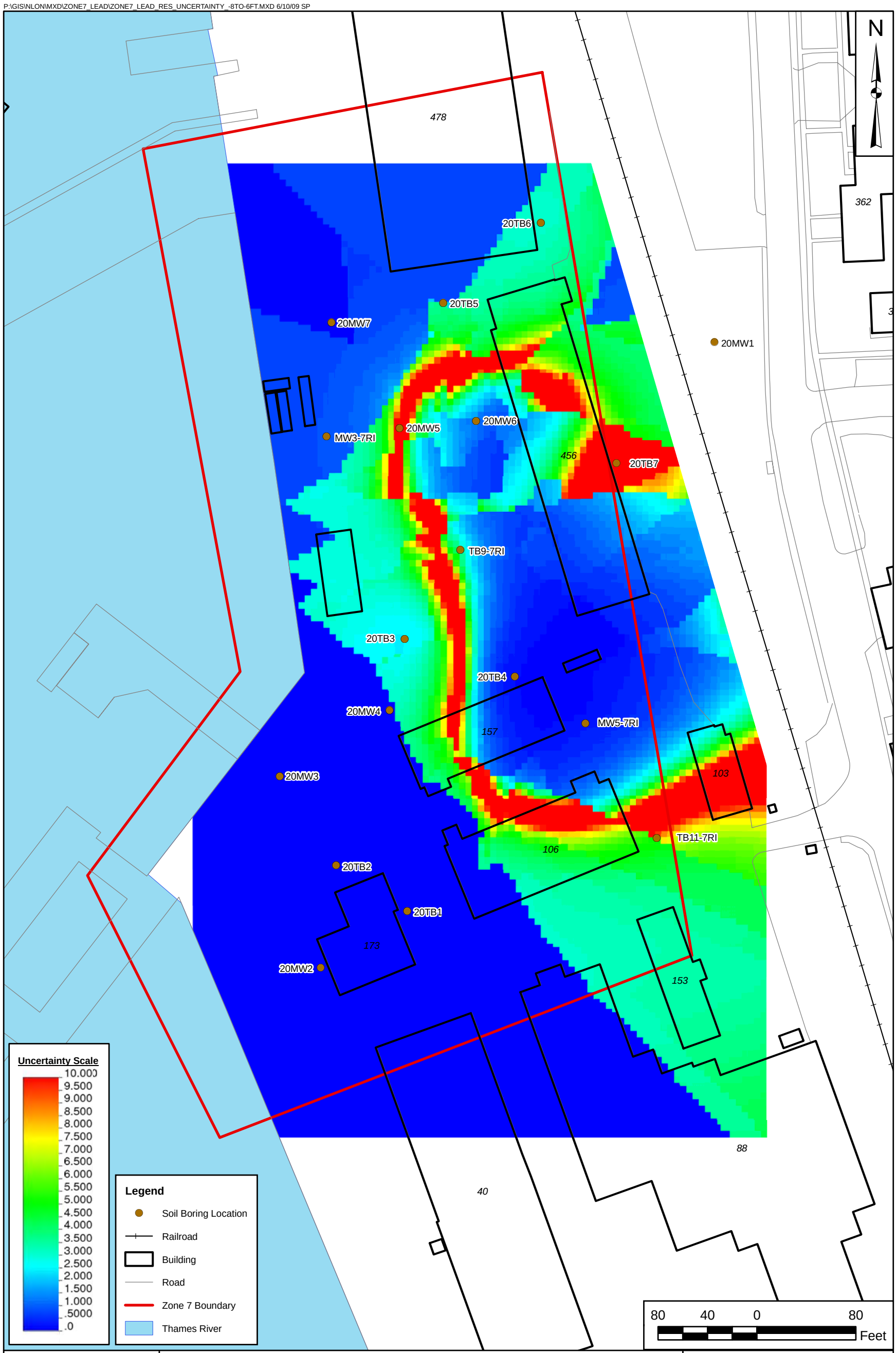
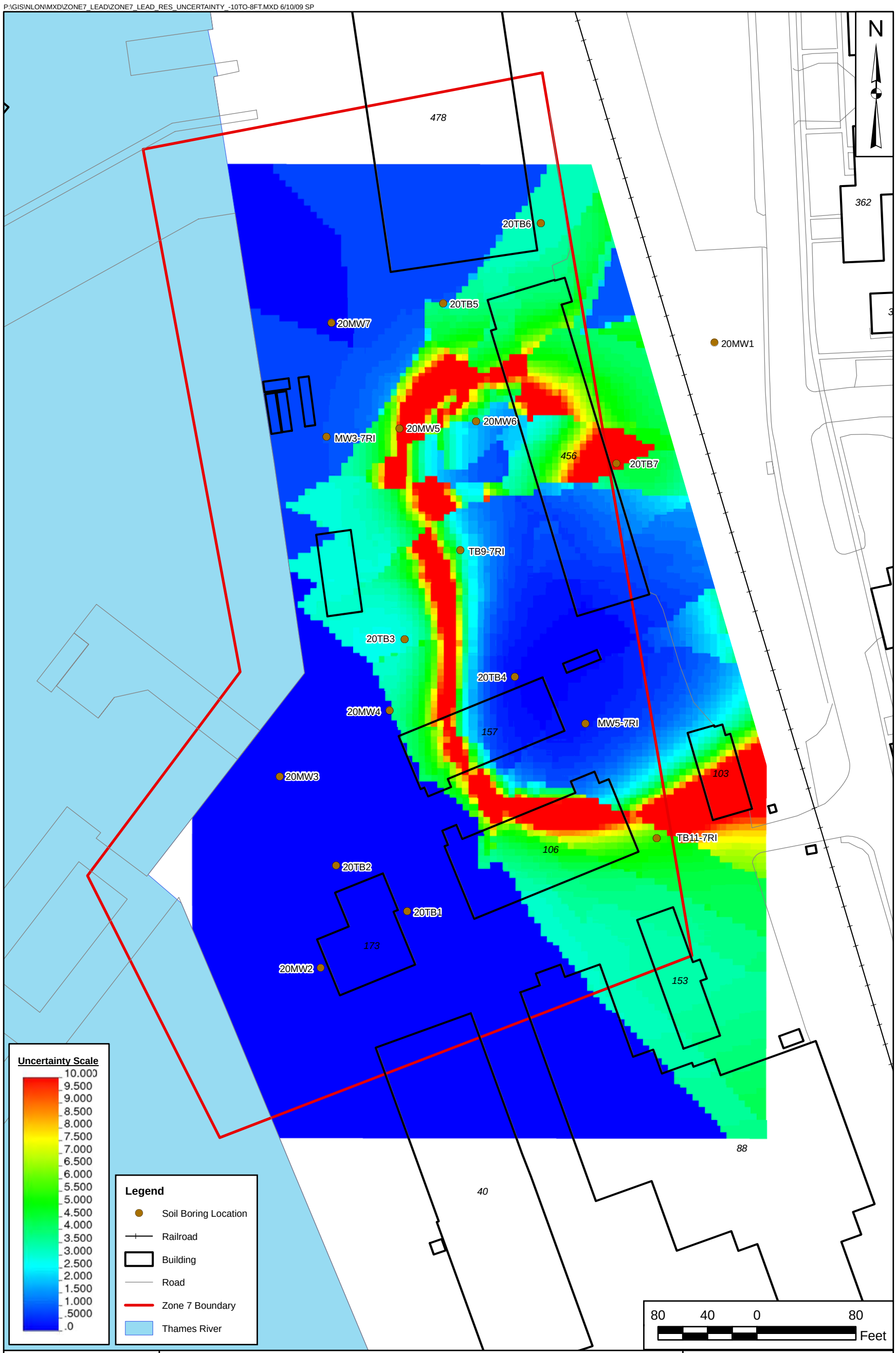
COST/SCHED-AREA			APPROVED BY -- / DATE --
SCALE AS NOTED			FIGURE NO. FIGURE B-140 / REV.

P:\GIS\NLON\MXD\ZONE7_LEAD\ZONE7_LEAD_RES_UNCERTAINTY_-8TO-6FT.MXD 6/10/09 SP



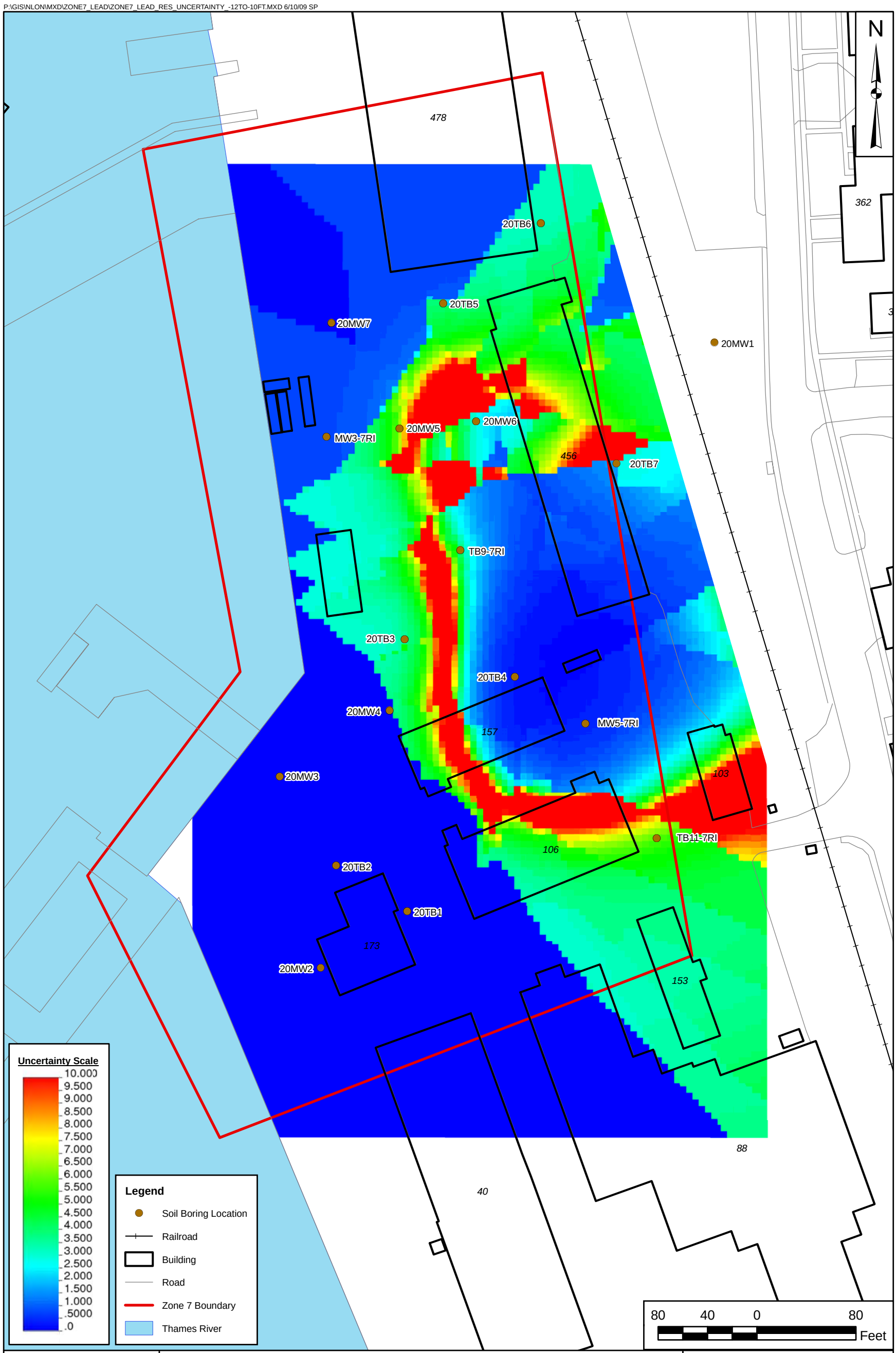
DRAWN BY S. PAXTON	DATE 4/02/09	Tetra Tech NUS, Inc.	CONTRACT NO. 0811
CHECKED BY N. BALSAMO	DATE 6/10/09	LEAD UNCERTAINTY FOR RESIDENTIAL CRITERIA ELEVATION: -8 TO -6 FEET ZONE 7 NAVAL SUBMARINE BASE - NEW LONDON GROTON, CONNECTICUT	APPROVED BY _ / DATE --
COST/SCHED-AREA			APPROVED BY -- / DATE --
SCALE AS NOTED			FIGURE NO. FIGURE B-141 / REV.

P:\GIS\NLON\MXD\ZONE7_LEAD\ZONE7_LEAD_RES_UNCERTAINTY_-10TO-8FT.MXD 6/10/09 SP



DRAWN BY S. PAXTON	DATE 4/02/09	Tetra Tech NUS, Inc.	CONTRACT NO. 0811
CHECKED BY N. BALSAMO	DATE 6/10/09	LEAD UNCERTAINTY FOR RESIDENTIAL CRITERIA ELEVATION: -10 TO -8 FEET ZONE 7 NAVAL SUBMARINE BASE - NEW LONDON GROTON, CONNECTICUT	APPROVED BY _ / DATE --
COST/SCHED-AREA			APPROVED BY -- / DATE --
SCALE AS NOTED			FIGURE NO. FIGURE B-142 / REV.

P:\GIS\NLON\MXD\ZONE7_LEAD\ZONE7_LEAD_RES_UNCERTAINTY_-12TO-10FT.MXD 6/10/09 SP



DRAWN BY S. PAXTON	DATE 4/02/09	Tetra Tech NUS, Inc.	CONTRACT NO. 0811
CHECKED BY N. BALSAMO	DATE 6/10/09	LEAD UNCERTAINTY FOR RESIDENTIAL CRITERIA ELEVATION: -12 TO -10 FEET ZONE 7 NAVAL SUBMARINE BASE - NEW LONDON GROTON, CONNECTICUT	APPROVED BY _ / DATE --
COST/SCHED-AREA			APPROVED BY -- / DATE --
SCALE AS NOTED			FIGURE NO. FIGURE B-143 / REV.

APPENDIX C

FIELD STANDARD OPERATING PROCEDURES

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6.0 DEVIATIONS **7**

1.0 PURPOSE

The purpose of this Standard Operating Procedure (SOP) is to specify a consistent sample nomenclature system that will facilitate subsequent data management in a cost-effective manner. The sample nomenclature system has been devised such that the following objectives can be attained:

- Sorting of data by matrix
- Sorting of data by depth
- Maintenance of consistency (field, laboratory, and database sample numbers)
- Accommodation of all project-specific requirements
- Accommodation of laboratory sample number length constraints (maximum of 20 characters)

2.0 SCOPE

The methods described in this SOP shall be used consistently for all projects requiring electronic data. Other contract- or project-specific sample nomenclature requirements may also be applicable.

3.0 GLOSSARY

None.

4.0 RESPONSIBILITIES AND PERSONNEL QUALIFICATIONS

Program Manager - It shall be the responsibility of the Project Manager (or designee) to inform contract-specific Project Managers (PMs) of the existence and requirements of this SOP.

Project Manager - It shall be the responsibility of the PM to determine the applicability of this SOP based on: (1) program-specific requirements and (2) project size and objectives. It shall be the responsibility of the PM (or designee) to ensure that sample nomenclature requirements are thoroughly specified in the relevant project planning document (e.g., sampling and analysis plan) and are consistent with this SOP if relevant. It shall be the responsibility of the PM to ensure that the FOL is familiar with the sample nomenclature system.

Field Operations Leader (FOL) - It shall be the responsibility of the FOL to ensure that all field technicians or sampling personnel are thoroughly familiar with this SOP and the project-specific sample nomenclature system. It shall be the responsibility of the FOL to ensure that the sample nomenclature system is used during all project-specific sampling efforts.

General personnel qualifications for sample nomenclature activities in the field include the following:

- Occupational Safety and Health Administration (OSHA) 40-hour and applicable refresher training.
- Capability of performing field work under the expected physical and environmental (i.e., weather) conditions.
- Familiarity with appropriate procedures for field documentation, handling, packaging, and shipping.

5.0 PROCEDURES

5.1 INTRODUCTION

The sample identification (ID) system can consist of as few as eight but not more than 20 distinct alpha-numeric characters. The sample ID will be provided to the laboratory on the sample labels and chain-of-custody forms. The basic sample ID provided to the laboratory has three segments and shall be as follows, where "A" indicates "alpha," and "N" indicates "numeric":

A or N 3 or 4 Characters	AAA 2 or 3 Characters	A or N 3 to 6 Characters
Site Identifier	Sample Type	Sample Location

Additional segments may be added as needed. For example:

(1) Soil and sediment sample ID

A or N 3 or 4 Characters	AAA 2 or 3 Characters	A or N 3 to 6 Characters	NNNN 4 Characters
Site identifier	Sample type	Sample location	Sample depth

(2) Aqueous (groundwater or surface water) sample ID

A or N 3 or 4 Characters	AAA 2 or 3 Characters	A or N 3 to 6 Characters	NN 2 Characters	-A 1 Character
Site identifier	Sample type	Sample location	Round number	Filtered sample only

(3) Biota sample ID

A or N 3 or 4 Characters	AAA 2 or 3 Characters	A or N 3 to 6 Characters	AA 2 Characters	NNN 3 Characters
Site identifier	Sample type	Sample location	Species identifier	Sample group number

5.2 SAMPLE IDENTIFICATION FIELD REQUIREMENTS

The various fields in the sample ID include but are not limited to the following:

- Site identifier
- Sample type
- Sample location
- Sample depth
- Sampling round number
- Filtered
- Species identifier
- Sample group number

The site identifier must be a three- or four-character field (numeric characters, alpha characters, or a mixture of alpha and numeric characters may be used). A site number is necessary because many facilities/sites have multiple individual sites, Solid Waste Management Units (SWMUs), Operable Units (OUs), etc. Several examples are presented in Section 5.3 of this SOP.

The sample type must be a two- or three-character alpha field. Suggested codes are provided in Section 5.3 of this SOP.

The sample location must be at least a three-character field but may have up to six characters (alpha, numeric, or a mixture). The six characters may be useful in identifying a monitoring well to be sampled or describing a grid location.

The sample depth field is used to note the depth below ground surface (bgs) at which a soil or sediment sample is collected. The first two numbers of the four-number code specify the top interval, and the third and fourth specify the bottom interval in feet bgs of the sample. If the sample depth is equal to or greater than 100, then only the top interval would be represented and the sampling depth would be truncated to three characters. The depths will be noted in whole numbers only; further detail, if needed, will be recorded on the sample log sheet or boring log, in the logbook, etc.

A two-digit round number will be used to track the number of aqueous samples collected from a particular aqueous sample location. The first sample collected from a location will be assigned the round identifier 01, the second 02, etc. This applies to both existing and proposed monitoring wells and surface water locations.

Aqueous samples that are field filtered (dissolved analysis) will be identified with an "-F" in the last field segment. No entry in this segment signifies an unfiltered (total) sample.

The species identifier must be a two-character alpha field. Several suggested codes are provided in Section 5.3 of this SOP.

The three-digit sample group number will be used to track the number of biota sample groups (a particular group size may be determined by sample technique, media type, the number of individual caught, weight issues, time, etc.) by species and location. The first sample group of a particular species collected from a given location will be assigned the sample group number 001, and the second sample group of the same species collected from the same location will be assigned the sample group number 002.

5.3 EXAMPLE SAMPLE FIELD DESIGNATIONS

Examples of each of the fields are as follows:

Site identifier - Examples of site numbers/designations are as follows:

A01 - Area of Concern (AOC) 1
125 - SWMU 125
000 - Base- or facility-wide sample (e.g., upgradient well)
BBG - Base background

The examples cited are only suggestions. Each PM (or designee) must designate appropriate (and consistent) site designations for their individual project.

Sample type - Examples of sample types are as follows:

AH - Ash Sample

AS - Air Sample
BM - Building Material Sample
BSB - Biota Sample Full Body
BSF - Biota Sample Fillet
CP - Composite Sample
CS - Chip Sample
DS - Drum Sample
DU - Dust Sample
FP - Free Product
IDW - Investigation-Derived Waste Sample
LT - Leachate Sample
MW - Monitoring Well Groundwater Sample
OF - Outfall Sample
RW - Residential Well Sample
SB - Soil Boring Sample
SD - Sediment Sample
SC - Scrape Sample
SG - Soil Gas Sample
SL - Sludge Sample
SP - Seep Sample
SS - Surface Soil Sample
ST - Storm Sewer Water Sample
SW - Surface Water Sample
TP - Test Pit Sample
TW - Temporary Well Sample
WC - Well Construction Material Sample
WP - Wipe Sample
WS - Waste/Solid Sample
WW - Wastewater Sample

Sample location - Examples of the location field are as follows:

001 - Monitoring well 1
N32E92 - Grid location 32 North and 92 East
D096 - Investigation-derived waste drum number 96

Species identifier - Examples of species identifier are as follows:

BC - Blue Crab
GB - Blue Gill
CO - Corn
SB - Soybean

5.4 EXAMPLES OF SAMPLE NOMENCLATURE

The first round monitoring well groundwater sample collected from existing monitoring well 001 at SWMU 16 for a filtered sample would be designated as 016MW00101-F.

The second round monitoring well groundwater sample collected from existing monitoring well C20P2 at Site 23 for an unfiltered sample would be designated as 023MWC20P202.

The second surface water sample collected from point 01 at SWMU 130 for an unfiltered sample would be designated as 130SW00102.

A surface soil sample collected from grid location 32 North and 92 East at Site 32 at the 0- to 2-foot interval would be designated as 032SSN32E920002.

A subsurface soil sample from soil boring 03 at SWMU 32 at an interval of 4 to 5 feet bgs would be designated as 032SB0030405.

A sediment sample collected at SWMU 19 from 0 to 6 inches at location 14 would be designated as 019SD0140001. The sample data sheet would reflect the precise depth at which this sample was collected.

During biota sampling for full-body analysis, the first time a minnow trap was checked at grid location A25 of SWMU 1415, three small blue gills were captured, collected, and designated with the sample ID of 1415BSBA25BG001. The second time blue gill were collected at the same location (grid location A25 at SWMU 1415), the sample ID would be 1415BSBA25BG002.

Note: No dash (-) or spacing is used between the segments with the exception of the filtered segment. The "F" used for a filtered aqueous sample is preceded by a dash (-F).

5.5 FIELD QA/QC SAMPLE NOMENCLATURE

Field Quality Assurance (QA)/Quality Control (QC) samples are designated using a different coding system. The QC code will consist of a three- to four-segment alpha-numeric code that identifies the sample QC type, the date the sample was collected, and the number of this type of QC sample collected on that date.

AA	NNNNNN	NN	-F
QC type	Date	Sequence number (per day)	Filtered (aqueous only, if needed)

The QC types are identified as:

TB = Trip Blank
RB = Rinsate Blank (Equipment Blank)
FD = Field Duplicate
AB = Ambient Conditions Blank
WB = Source Water Blank

The sampling time recorded on the chain-of-custody form, labels, and tags for duplicate samples will be 0000 so that the samples are "blind" to the laboratory. Notes detailing the sample number, time, date, and type will be recorded on the routine sample log sheets and will document the location of the duplicate sample (sample log sheets are not provided to the laboratory). Documentation for all other QC types (TB, RB, AB, and WB) will be recorded on the QC Sample Log Sheet (see SOP SA-6.3, Field Documentation).

5.6 EXAMPLES OF FIELD QA/QC SAMPLE NOMENCLATURE

The first duplicate of the day for a filtered groundwater sample collected on June 3, 2000, would be designated as FD06030001-F.

The third duplicate of the day taken of a subsurface soil sample collected on November 17, 2003, would be designated as FD11170303.

The first trip blank associated with samples collected on October 12, 2000, would be designated as TB10120001.

The only rinsate blank collected on November 17, 2001, would be designated as RB11170101.

6.0 DEVIATIONS

Any deviation from this SOP must be addressed in detail in the site-specific planning documents.

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1.0 PURPOSE

The purpose of this document is to specify a consistent procedure for the quality assurance review of electronic and hard copy databases. This SOP outlines the requirements for establishment of a Database Record File, Quality Assurance review procedures, and documentation of the Quality Assurance Review Process.

2.0 SCOPE

The methods described in this Standard Operating Procedure (SOP) shall be used consistently for all projects managed by Tetra Tech NUS (TtNUS).

3.0 GLOSSARY

Chain-of-Custody Form - A Chain-of-Custody Form is a printed form that accompanies a sample or a group of samples from the time of sample collection to the laboratory. The Chain-of-Custody Form is retained with the samples during transfer of samples from one custodian to another. The Chain-of-Custody Form is a controlled document that becomes part of the permanent project file. Chain-of-Custody and field documentation requirements are addressed in SOP SA-6.1.

Electronic Database - A database provided on a compact laser disk (CD). Such electronic databases will generally be prepared using public domain software such as DBase, RBase, Oracle, Visual FoxPro, Microsoft Access, Paradox, etc.

Hardcopy Database - A printed copy of a database prepared using the software discussed under the definition of an electronic database.

Form I - A printed copy of the analytical results for each sample.

Sample Tracking Summary - A printed record of sample information including the date the samples were collected, the number of samples collected, the sample matrix, the laboratory to which the samples were shipped, the associated analytical requirements for the samples, the date the analytical data were received from the laboratory, and the date that validation of the sample data was completed.

4.0 RESPONSIBILITIES

Database Records Custodian - It shall be the responsibility of the Database Records Custodian to update and file the Sample Tracking Summaries for all active projects on a weekly basis. It shall be the responsibility of the Database Records Custodian to ensure that the most recent copies of the Sample Tracking Summaries are placed in the Database Records file. It shall be the responsibility of the Database Records Custodian to ensure that a copy of all validation deliverables is provided to the Project Manager (for placement in the project file). It shall be the responsibility of the Database Records Custodian to ensure that photocopies of all validation deliverables and historical data and reports (as applicable) are placed in the Database Records file.

Data Validation Coordinator - It shall be the responsibility of the Data Validation Coordinator (or designee) to ensure that the Sample Tracking Summaries are maintained by the Database Records Custodian. It shall be the responsibility of the Data Validation Coordinator (or designee) to ensure that photocopies of all data validation deliverables are placed in the applicable Database Records file by the Database Records Custodian.

Earth Sciences Department Manager - It shall be the responsibility of the Earth Sciences Department Manager (or equivalent) to ensure that all field personnel are familiar with the requirements of this Standard Operating Procedure (specifically Section 5.5).

FOL - It shall be the responsibility of the FOL (FOL) of each project to ensure that all field technicians or sampling personnel are thoroughly familiar with this SOP, specifically regarding provision of the Chain-of-Custody Forms to the Database Records Custodian. Other responsibilities of the FOL are described in Sections 5.4 and 5.5.

Management Information Systems (MIS) Manager - It shall be the responsibility of the MIS Manager to ensure that copies of original electronic deliverables (CDs) are placed in both the project files and the Database Records File. It shall be the responsibility of the MIS Manager (or designee) to verify the completeness of the database (presence of all samples) in both electronic and hardcopy form in the Database Records File. It shall be the responsibility of the MIS Manager to ensure that Quality Assurance Reviews are completed and are attested to by Quality Assurance Reviewers. It shall be the responsibility of the MIS Manager to ensure that records of the Quality Assurance review process are placed in the Database Records File. It shall be the responsibility of the MIS Manager to ensure that both electronic and hardcopy forms of the final database are placed in both the project and the Database Record File. It shall be the responsibility of the MIS Manager to ensure that data validation qualifiers are entered in the database.

Furthermore, it shall be the responsibility of the MIS Manager to participate in project planning at the request of the Project Manager, specifically with respect to the generation of level of effort and schedule estimates. To support the project planning effort, the MIS Manager shall provide a copy of the MIS Request From included as Attachment A to the project manager. It shall be the responsibility of the MIS Manager to generate level of effort and budget estimates at the time database support is requested if a budget does not exist at the time of the request. The MIS Request Form shall be provided to the Project Manager at the time of any such requests. It shall be the responsibility of the MIS Manager to notify the Project Manager of any anticipated level of effort overruns or schedule noncompliances as soon as such problems arise along with full justification for any deviations from the budget estimates (provided they were generated by the MIS Manager). It shall be the responsibility of the MIS Manager to document any changes to the scope of work dictated by the Project Manager, along with an estimate of the impact of the change on the level of effort and the schedule.

Program/Department Managers - It shall be the responsibility of the Department and/or Program Managers (or designees) to inform their respective department's Project Managers of the existence and requirements of this SOP.

Project Manager - It shall be the responsibility of each Project Manager to determine the applicability of this SOP based on: (1) program-specific requirements, and (2) project size and objectives. It shall be the responsibility of the Project Manager (or designee) to ensure that the FOL is familiar with the requirements regarding Chain-of-Custody Form provision to the Database Records Custodian. It shall be the responsibility of the Project Manager (or designee) to determine which, if any, historical data are relevant and to ensure that such data (including all relevant information such as originating entity, sample locations, sampling dates, etc.) are provided to the Database Records Custodian for inclusion in the Database Records File. It shall be the responsibility of the Project Manager to obtain project planning input regarding the level of effort and schedule from the MIS Manager. It shall be the responsibility of the Project Manager to complete the database checklist (Attachment A) to support the level of effort and schedule estimate and to facilitate database preparation and subroutine execution.

Risk Assessment Department Manager - It shall be the responsibility of the Risk Assessment Department Manager to monitor compliance with this Standard Operating Procedure, to modify this SOP as necessary, and to take corrective action if necessary. Monitoring of the process shall be completed on a quarterly basis.

Quality Assurance Reviewers - It shall be the responsibility of the Quality Assurance Reviewers to verify the completeness of the sample results via review of the Chain-of-Custody Forms and Sample Tracking Summaries. It shall be the responsibility of the Quality Assurance Reviewers to ensure the correctness of the database via direct comparison of the hardcopy printout of the database and the hardcopy summaries of the original analytical data (e.g., Form Is provided in data validation deliverables). Correctness includes the presence of all relevant sample information (all sample information fields), agreement of the laboratory and database analytical results, and the presence of data validation qualifiers.

Quality Manager - It shall be the responsibility of the Quality Manager to monitor compliance with this Standard Operating Procedure via routine audits.

5.0 PROCEDURES

5.1 Introduction

Verification of the accuracy and completeness of an electronic database can only be accomplished via comparison of a hardcopy of the database with hardcopy of all relevant sample information. The primary purposes of this SOP are to ensure that 1) all necessary hardcopy information is readily available to Quality Assurance Reviewers; 2) ensure that the Quality Assurance review is completed in a consistent and comprehensive manner, and; 3) ensure that documentation of the Quality Assurance review process is maintained in the project file.

5.2 File Establishment

A Database Record file shall be established for a specific project at the discretion of the Project Manager. Initiation of the filing procedure will commence upon receipt of the first set of Chain-of-Custody documents from a FOL or sampling technician. The Database Record Custodian shall establish a project-specific file for placement in the Database Record File. Each file in the Database Record File shall consist of standard components placed in the file as the project progresses. Each file shall be clearly labeled with the project number, which shall be placed on the front of the file drawer and on each and every hanging file folder relevant to the project. The following constitute the minimum components of a completed file:

- Electronic Deliverables
- Sample Tracking Forms
- Chain-of-Custody Forms
- Data Validation Letters
- Quality Assurance Records

5.3 Electronic Deliverables

The format of electronic deliverables shall be specified in the laboratory procurement specification and shall be provided by the laboratory. The integrity of all original electronic data deliverables shall be maintained. This shall be accomplished via the generation of copies of each electronic deliverable provided by the laboratory. The original electronic deliverable shall be provided to the project manager for inclusion in the project file. A copy of the original electronic deliverable shall be placed in the Database Record File. The second copy shall be maintained by the MIS Manager (or designee) to be used as a working copy.

5.4 Sample Tracking Forms

Updated versions of the sample tracking form for each relevant project shall be maintained by the Database Record Custodian. The Sample Tracking Forms shall be updated any time additional Chain-of-Custody Forms are received from a FOL or sampling technician, or at any time that data are received from a laboratory, or at any time that validation of a given data package (sample delivery group) is completed. The Data Validation Coordinator shall inform the Database Record Custodian of the receipt of any data packages from the laboratory and of completion of validation of a given data package to facilitate updating of the Sample Tracking Form. The Database Record Custodian shall place a revised copy of the Sample Tracking Form in the Database Record File anytime it has been updated. Copies of the updated Sample Tracking Form shall also be provided to the project manager to apprise the project manager of sample package receipt, completion of validation, etc.

5.5 Chain-of-Custody Forms

The Chain-of-Custody Forms for all sampling efforts will be used as the basis for (1) updating the Sample Tracking Form, and (2) confirming that all required samples and associated analyses have been completed. It shall be the responsibility of the FOL (or sample technician) to provide a photocopy of all Chain-of-Custody Forms to the Database Record Custodian immediately upon completion of a sampling effort. The Database Record Custodian shall then place the copies of the Chain-of-Custody Form(s) in the Database Record File. Upon receipt of a sample data package from an analytical laboratory, the Data Validation Coordinator shall provide a copy of the laboratory Chain-of-Custody Form to the Database Record Custodian. The Database Record Custodian shall use this copy to update the Sample Tracking Summary and shall place the copy of the laboratory-provided Chain-of-Custody Form in the Database Record File. The photocopy of the laboratory-provided Chain-of Custody Form shall be stapled to the previously filed field copy. Upon receipt of all analytical data, two copies of the Chain-of-Custody will therefore be in the file. Review of the Chain-of-Custody Forms will therefore be a simple mechanism to determine if all data have been received. Chain-of-Custody is addressed in SOP SA-6.1.

5.6 Data Validation Letters

All data validation deliverables (or raw data summaries if validation is not conducted) shall be provided for inclusion in both the Database Record File and the project file. If USEPA regional- or client-specific requirements are such that Form Is (or similar analytical results) need not be provided with the validation deliverable, copies of such results must be appended to the deliverable. It is preferable, although not essential that the validation qualifiers be hand-written directly on the data summary forms. The data validation deliverables (and attendant analytical summaries) will provide the basis for direct comparison of the database printout and the raw data and qualifiers.

5.7 Historical Data

At the direction of the Project Manager, historical data may also be included in a project-specific analytical database. In the event that historical data are germane to the project, hardcopy of the historical data must be included in the Database Record File. Historical data may be maintained in the form of final reports or as raw data. The information contained in the historical data file must be sufficient to identify its origin, its collection date, the sample location, the matrix, and any and all other pertinent information. All available analytical data, Chain-of-Custody Forms, boring logs, well construction logs, sample location maps, shall be photocopied by the Project Manager (or designee) and placed in one or more 3-ring binders. All information shall be organized chronologically by matrix. It shall be the responsibility of the Project Manager (or designee) to ensure that all inconsistencies between analytical data, Chain-of-Custody Forms, boring logs, sample log sheets, and field logbooks are identified and corrected. The Project Manager (or designee) shall decide which nomenclature is appropriate and edit, initial and date all relevant forms. Data entry may only be performed on information that has undergone the aforementioned

editing process, thereby having a direct correlation between hardcopy information and what will become the electronic database.

6.0 RECORDS

Records regarding database preparation and quality assurance review include all those identified in the previous section. Upon completion of the database task, records from the file will be forwarded to the Project Manager for inclusion in the project file, or will be placed in bankers boxes (or equivalent) for storage. The final records for storage shall include the following minimum information on placards placed on both the top and end of the storage box:

Database Record File
PROJECT NUMBER: ____
SITE NAME: _____
DATE FILED: __/__/__
SUMMARY OF CONTENTS ENCLOSED
BOX _ OF _

Project- or program-specific record keeping requirements shall take precedence over the record keeping requirements of this SOP.

ATTACHMENT A

MIS REQUEST FORM

Tetra Tech NUS, Inc.

Project Name:
CTO:
Project Manager:
Requestor:
Program/Client:
State/EPA Region:

Request Date:
Date Data Available for Production:
Request in Support of:
Database Lead:
GIS Lead:
Statistics Lead:
Risk Lead:

Site Name(s) (Area, OU, etc.):
Sampling Date(s):
Matrix: ☐ GW ☐ SO ☐ SD ☐ SW ☐ Other:

Labels: ☐ Labels needed for an upcoming sampling event _____ Total # of Samples
_____ Estimated Hours
_____ Due Date
_____ Complete ETS Charge No.
_____ FOL
Additional Instructions:

Data Entry:
☐ Chemical data needs to be entered from hardcopy _____ Estimated # of Samples
☐ Chemical data needs to be formated electronically
☐ Field analytical data needs to be entered from hardcopy
☐ Geologic data needs to be entered from hardcopy
☐ Hydrology data needs to be entered from hardcopy
_____ Estimated Hours
_____ Due Date
_____ Complete ETS Charge No.
Additional Instructions:

Tables:
☐ Full Data Printout
☐ Summary of Positive Hits
☐ Occurance and Distribution ☐ with criteria
☐ Sampling Analytical Summary
☐ Other:
_____ Estimated Hours
_____ Due Date
_____ Complete ETS Charge No.
Additional Instructions:

GIS:
☐ General Facility Location
☐ Site Location
☐ Potentiometric Contours/Groundwater Flow
☐ Sample Location Proposed
☐ Sample Location Existing
☐ Tag Map Single Round
☐ Tag Map Multiple Round
☐ Isoconcentrations
☐ Chart Map
☐ 3D Visualization
☐ EGIS CD
☐ Other:
_____ Estimated Hours
_____ Due Date
_____ Complete ETS Charge No.
Additional Instructions:

Statistics: ☐ Yes
_____ Estimated Hours
_____ Due Date
_____ Complete ETS Charge No.
Additional Instructions:

Geostatistics: ☐ Yes
_____ Estimated Hours
_____ Due Date
_____ Complete ETS Charge No.
Additional Instructions:

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1.0 PURPOSE

The purpose of this document is to establish standard procedures and technical guidance on borehole and sample logging.

2.0 SCOPE

These procedures provide descriptions of the standard techniques for borehole and sample logging. These techniques shall be used for each boring logged to provide consistent descriptions of subsurface lithology. While experience is the only method to develop confidence and accuracy in the description of soil and rock, the field geologist/engineer can do a good job of classification by careful, thoughtful observation and by being consistent throughout the classification procedure.

3.0 GLOSSARY

None.

4.0 RESPONSIBILITIES

Site Geologist. Responsible for supervising all boring activities and assuring that each borehole is completely logged. If more than one rig is being used on site, the Site Geologist must make sure that each field geologist is properly trained in logging procedures. A brief review or training session may be necessary prior to the start up of the field program and/or upon completion of the first boring.

5.0 PROCEDURES

The classification of soil and rocks is one of the most important jobs of the field geologist/engineer. To maintain a consistent flow of information, it is imperative that the field geologist/engineer understand and accurately use the field classification system described in this SOP. This identification is based on visual examination and manual tests.

5.1 Materials Needed

When logging soil and rock samples, the geologist or engineer may be equipped with the following:

- Rock hammer
- Knife
- Camera
- Dilute hydrochloric acid (HCl)
- Ruler (marked in tenths and hundredths of feet)
- Hand Lens

5.2 Classification of Soils

All data shall be written directly on the boring log (Figure 1) or in a field notebook if more space is needed. Details on filling out the boring log are discussed in Section 5.5.

FIGURE 1
BORING LOG (EXAMPLE)

BORING LOG

Page ___ of ___

PROJECT NAME: _____ BORING NUMBER: _____
PROJECT NUMBER: _____ DATE: _____
DRILLING COMPANY: _____ GEOLOGIST: _____
DRILLING RIG: _____ DRILLER: _____

Sample No. and Type or RQD	Depth (Ft.) or Run No.	Blows / 6" or RQD (%)	Sample Recovery / Sample Length	Lithology Change (Depth/Ft.) or Screened Interval	MATERIAL DESCRIPTION: Soil Density/ Consistency or Rock Hardness	MATERIAL DESCRIPTION: Color	MATERIAL DESCRIPTION: Material Classification	USCS *	Remarks	PID/FID Reading (ppm): Sample	PID/FID Reading (ppm): Sampler BZ	PID/FID Reading (ppm): Borehole**	PID/FID Reading (ppm): Driller BZ**

* When rock coring, enter rock brokeness.

** Include monitor reading in 6 foot intervals @ borehole. Increase reading frequency if elevated response-read.

Remarks: _____

Drilling Area Background (ppm): _____

Converted to Well: Yes _____ No _____ Well I.D. #: _____

FIGURE 1 (CONTINUED)

SOIL TERMS

UNIFIED SOIL CLASSIFICATION (USCS)

COARSE-GRAINED SOILS
More Than Half of Material is LARGER Than No. 200 Sieve Size

FIELD IDENTIFICATION PROCEDURES (Excluding Particles Larger Than 3 Inches and Basing Fractions on Estimated Weights)			GROUP SYMBOL	TYPICAL NAMES
GRAVELS (50% or more of coarse fraction is larger than No. 4 sieve size)	CLEAN GRAVELS (Less than 5% Fines)	Wide range in grain size and substantial amounts of all intermediate particle sizes.	GW	Well graded gravels, gravel-sand mixtures, little or no fines.
		Predominantly one size or a range of sizes with some intermediate sizes missing.	GP	Poorly graded gravels, gravel-sand mixtures, little or no fines.
	GRAVELS WITH FINES (More than 12% Fines)	Non-plastic fines (for identification procedures, see ML)	GM	Silty gravels, poorly graded gravel-sand-silt mixtures.
		Plastic fines (for identification procedures, see CL)	GC	Clayey gravels, poorly graded gravel-sand-clay mixtures.
SANDS (50% or more of coarse fraction is smaller than No. 4 sieve size)	CLEAN SANDS (Less than 5% Fines)	Wide range in grain size and substantial amounts of all intermediate particle sizes.	SW	Well graded sand, gravelly sands, little or no fines.
		Predominantly one size or a range of sizes with some intermediate sizes missing.	SP	Poorly graded sands, gravelly sands, little or no fines.
	SANDS WITH FINES (More than 12% Fines)	Non-plastic fines (for identification procedures, see ML)	SM	Silty sands, poorly graded sand-silt mixtures.
		Plastic fines (for identification procedures, see CL)	SC	Clayey sands, poorly graded sand-clay mixtures.

FINE-GRAINED SOILS
More Than Half of Material is SMALLER Than No. 200 Sieve Size

FIELD IDENTIFICATION PROCEDURES (Excluding Particles Larger Than 3 Inches and Basing Fractions on Estimated Weights)					GROUP SYMBOL	TYPICAL NAMES
Identification Procedures on Fraction Smaller Than No. 40 Sieve Size						
	DILATANCY (Reaction to Shaking)	TOUGHNESS (Consistency Near Plastic Limit)				
SILTS AND CLAYS Liquid Limit < 50	None to Slight	Quick to Slow	None		ML	Inorganic silts and very fine sands, rock flour, silty or clayey fine sands with slight plasticity.
	Medium to High	None to Very Slow	Medium		CL	Inorganic clays of low to medium plasticity, gravelly clays, sandy clays, silty clays, lean clays.
	Slight to Medium	Slow	Slight		OL	Organic silts and organic silt-clays of low plasticity.
SILTS AND CLAYS Liquid Limit > 50	Slight to Medium	Slow to None	Slight to Medium		MH	Inorganic silts, micaceous or diatomaceous fine sandy or silty soils, elastic silts.
	High to Very High	None	High		CH	Inorganic clays of high plasticity, fat clays.
	Medium to High	None to Very Slow	Slight to Medium		OH	Organic clays of medium to high plasticity.
HIGHLY ORGANIC SOILS	Readily identified by color, odor, spongy feel and frequently by fibrous texture.				Pt	Peat and other organic soils

Boundary classifications: Soils possessing characteristics of two groups are designated by combining group symbols. For example, GW-GC, well graded gravel-sand mixture with clay binder.
All sieve sizes on this chart are U.S. Standard.

DENSITY OF GRANULAR SOILS

DESIGNATION	STANDARD PENETRATION RESISTANCE - BLOWS/FOOT
Very Loose	0-4
Loose	5-10
Medium Loose	11-30
Dense	31-50
Very Dense	Over 50

CONSISTENCY OF COHESIVE SOILS

CONSISTENCY	UNCONFINED STRENGTH (TONS/SQ. FT.)	STANDARD PENETRATION RESISTANCE - BLOWS/FOOT	FIELD IDENTIFICATION METHODS
Very Soft	Less than 0.25	0 to 1	Easily penetrated several inches by fist
Soft	0.25 to 0.50	1 to 4	Easily penetrated several inches by thumb.
Medium Stiff	0.50 to 1.0	4 to 8	Can be penetrated several inches by thumb.
Stiff	1.0 to 2.0	8 to 15	Readily indented by thumb.
Very Stiff	2.0 to 4.0	15 to 30	Readily indented by thumbnail.
Hard	More than 4.0	Over 30	Indented with difficulty by thumbnail.

ROCK TERMS

ROCK HARDNESS (FROM CORE SAMPLES)

Descriptive Terms	Scratches or Knife Blade	Hammer Blows
Soft	Easily Gouged	Crushes when pressed with hammer
Medium Soft	Can be Gouged	Breaks (one blow); crumbly edges
Medium Hard	Can be scratched	Breaks (one blow); sharp edges
Hard	Cannot be scratched	Breaks (one to many blows); sharp edges

ROCK BROKENNESS

Descriptive Terms	Abbreviation	Spacing
Very Broken	(VB)	< 0.1'
Broken	(Br)	0.1'-0.5'
Blocky	(Bl)	0.5'-1'
Massive	(M)	> 1'

LEGEND :

SOIL SAMPLER - TYPES

S-1" Split Barrel Sample

ST-3" O.D. Undisturbed Sample

O - Other Samples, Specify in Remarks

ROCK SAMPLER - TYPES

X-NQ (Conventional) Core (0.3-1.8" O.D.)

Q/9

Q-NQ (Extreme) Core (0.5-1.8" O.D.)

Z - Other Core Bits, Specify in Remarks

Q/9

WATER LEVELS

W.L.' NW Level w/Date + Depth

W.S.' Stabilized Level w/Date + Depth

5.2.1 USCS Classification

Soils are to be classified according to the Unified Soil Classification System (USCS). This method of classification is detailed in Figure 1 (Continued).
This method of classification identifies soil types on the basis of grain size and cohesiveness.

Fine-grained soils, or fines, are smaller than the No. 200 sieve and are of two types: silt (M) and clay (C). Some classification systems define size ranges for these soil particles, but for field classification purposes, they are identified by their respective behaviors. Organic material (O) is a common component of soil but has no size range; it is recognized by its composition. The careful study of the USCS will aid in developing the competence and consistency necessary for the classification of soils.

Coarse-grained soils shall be divided into rock fragments, sand, or gravel. The terms sand and gravel not only refer to the size of the soil particles but also to their depositional history. To insure accuracy in description, the term rock fragments shall be used to indicate angular granular materials resulting from the breakup of rock. The sharp edges typically observed indicate little or no transport from their source area, and therefore the term provides additional information in reconstructing the depositional environment of the soils encountered. When the term "rock fragments" is used it shall be followed by a size designation such as "(1/4 inchΦ-1/2 inchΦ)" or "coarse-sand size" either immediately after the entry or in the remarks column. The USCS classification would not be affected by this variation in terms.

5.2.2 Color

Soil colors shall be described utilizing a single color descriptor preceded, when necessary, by a modifier to denote variations in shade or color mixtures. A soil could therefore be referred to as "gray" or "light gray" or "blue-gray." Since color can be utilized in correlating units between sampling locations, it is important for color descriptions to be consistent from one boring to another.

Colors must be described while the sample is still moist. Soil samples shall be broken or split vertically to describe colors. Samplers tend to smear the sample surface creating color variations between the sample interior and exterior.

The term "mottled" shall be used to indicate soils irregularly marked with spots of different colors. Mottling in soils usually indicates poor aeration and lack of good drainage.

Soil Color Charts shall not be used unless specified by the project manager.

5.2.3 Relative Density and Consistency

To classify the relative density and/or consistency of a soil, the geologist is to first identify the soil type. Granular soils contain predominantly sands and gravels. They are noncohesive (particles do not adhere well when compressed). Finer-grained soils (silts and clays) are cohesive (particles will adhere together when compressed).

The density of noncohesive, granular soils is classified according to standard penetration resistances obtained from split-barrel sampling performed according to the methods detailed in Standard Operating Procedures GH-1.3 and SA-1.3. Those designations are:

Designation	Standard Penetration Resistance (Blows per Foot)
Very loose	0 to 4
Loose	5 to 10
Medium dense	11 to 30
Dense	31 to 50
Very dense	Over 50

Standard penetration resistance is the number of blows required to drive a split-barrel sampler with a 2-inch outside diameter 12 inches into the material using a 140-pound hammer falling freely through 30 inches. The sampler is driven through an 18-inch sample interval, and the number of blows is recorded for each 6-inch increment. The density designation of granular soils is obtained by adding the number of blows required to penetrate the last 12 inches of each sample interval. It is important to note that if gravel or rock fragments are broken by the sampler or if rock fragments are lodged in the tip, the resulting blow count will be erroneously high, reflecting a higher density than actually exists. This shall be noted on the log and referenced to the sample number. Granular soils are given the USCS classifications GW, GP, GM, SW, SP, SM, GC, or SC (see Figure 1).

The consistency of cohesive soils is determined by performing field tests and identifying the consistency as shown in Figure 2.

Cohesive soils are given the USCS classifications ML, MH, CL, CH, OL, or OH (see Figure 1).

The consistency of cohesive soils is determined either by blow counts, a pocket penetrometer (values listed in the table as Unconfined Compressive Strength), or by hand by determining the resistance to penetration by the thumb. The pocket penetrometer and thumb determination methods are conducted on a selected sample of the soil, preferably the lowest 0.5 foot of the sample in the split-barrel sampler. The sample shall be broken in half and the thumb or penetrometer pushed into the end of the sample to determine the consistency. Do not determine consistency by attempting to penetrate a rock fragment. If the sample is decomposed rock, it is classified as a soft decomposed rock rather than a hard soil. Consistency shall not be determined solely by blow counts. One of the other methods shall be used in conjunction with it. The designations used to describe the consistency of cohesive soils are shown in Figure 2.

5.2.4 Weight Percentages

In nature, soils are comprised of particles of varying size and shape, and are combinations of the various grain types. The following terms are useful in the description of soil:

Terms of Identifying Proportion of the Component	Defining Range of Percentages by Weight
Trace	0 - 10 percent
Some	11 - 30 percent
Adjective form of the soil type (e.g., "sandy")	31 - 50 percent

FIGURE 2

CONSISTENCY FOR COHESIVE SOILS

Consistency	Standard Penetration Resistance (Blows per Foot)	Unconfined Compressive Strength (Tons/Sq. Foot by pocket penetration)	Field Identification
Very soft	0 to 2	Less than 0.25	Easily penetrated several inches by fist
Soft	2 to 4	0.25 to 0.50	Easily penetrated several inches by thumb
Medium stiff	4 to 8	0.50 to 1.0	Can be penetrated several inches by thumb with moderate effort
Stiff	8 to 15	1.0 to 2.0	Readily indented by thumb but penetrated only with great effort
Very stiff	15 to 30	2.0 to 4.0	Readily indented by thumbnail
Hard	Over 30	More than 4.0	Indented with difficulty by thumbnail

Examples:

- Silty fine sand: 50 to 69 percent fine sand, 31 to 50 percent silt.
- Medium to coarse sand, some silt: 70 to 80 percent medium to coarse sand, 11 to 30 percent silt.
- Fine sandy silt, trace clay: 50 to 68 percent silt, 31 to 49 percent fine sand, 1 to 10 percent clay.
- Clayey silt, some coarse sand: 70 to 89 percent clayey silt, 11 to 30 percent coarse sand.

5.2.5 Moisture

Moisture content is estimated in the field according to four categories: dry, moist, wet, and saturated. In dry soil, there appears to be little or no water. Saturated samples obviously have all the water they can hold. Moist and wet classifications are somewhat subjective and often are determined by the individual's judgment. A suggested parameter for this would be calling a soil wet if rolling it in the hand or on a porous surface liberates water, i.e., dirties or muddies the surface. Whatever method is adopted for describing moisture, it is important that the method used by an individual remains consistent throughout an entire drilling job.

Laboratory tests for water content shall be performed if the natural water content is important.

5.2.6 Stratification

Stratification can only be determined after the sample barrel is opened. The stratification or bedding thickness for soil and rock is depending on grain size and composition. The classification to be used for stratification description is shown in Figure 3.

5.2.7 Texture/Fabric/Bedding

The texture/fabric/bedding of the soil shall be described. Texture is described as the relative angularity of the particles: rounded, subrounded, subangular, and angular. Fabric shall be noted as to whether the particles are flat or bulky and whether there is a particular relation between particles (i.e., all the flat particles are parallel or there is some cementation). The bedding or structure shall also be noted (e.g., stratified, lensed, nonstratified, heterogeneous varved).

5.2.8 Summary of Soil Classification

In summary, soils shall be classified in a similar manner by each geologist/engineer at a project site. The hierarchy of classification is as follows:

- Density and/or consistency
- Color
- Plasticity (Optional)
- Soil types
- Moisture content
- Stratification
- Texture, fabric, bedding
- Other distinguishing features

FIGURE 3

BEDDING THICKNESS CLASSIFICATION

Thickness (metric)	Thickness (Approximate English Equivalent)	Classification
> 1.0 meter	> 3.3'	Massive
30 cm - 1 meter	1.0' - 3.3'	Thick Bedded
10 cm - 30 cm	4" - 1.0'	Medium Bedded
3 cm - 10 cm	1" - 4"	Thin Bedded
1 cm - 3 cm	2/5" - 1"	Very Thin Bedded
3 mm - 1 cm	1/8" - 2/5"	Laminated
1 mm - 3 mm	1/32" - 1/8"	Thinly Laminated
< 1 mm	<1/32"	Micro Laminated

(Weir, 1973 and Ingram, 1954)

5.3 Classification of Rocks

Rocks are grouped into three main divisions: sedimentary, igneous and metamorphic. Sedimentary rocks are by far the predominant type exposed at the earth's surface. The following basic names are applied to the types of rocks found in sedimentary sequences:

- Sandstone - Made up predominantly of granular materials ranging between 1/16 to 2 mm in diameter.
- Siltstone - Made up of granular materials less than 1/16 to 1/256 mm in diameter. Fractures irregularly. Medium thick to thick bedded.
- Claystone - Very fine-grained rock made up of clay and silt-size materials. Fractures irregularly. Very smooth to touch. Generally has irregularly spaced pitting on surface of drilled cores.
- Shale - A fissile very fine-grained rock. Fractures along bedding planes.
- Limestone - Rock made up predominantly of calcite ($CaCO_3$). Effervesces strongly upon the application of dilute hydrochloric acid.
- Coal - Rock consisting mainly of organic remains.
- Others - Numerous other sedimentary rock types are present in lesser amounts in the stratigraphic record. The local abundance of any of these rock types is dependent upon the depositional history of the area. Conglomerate, halite, gypsum, dolomite, anhydrite, lignite, etc. are some of the rock types found in lesser amounts.

In classifying a sedimentary rock the following hierarchy shall be noted:

- Rock type
- Color
- Bedding thickness
- Hardness
- Fracturing
- Weathering
- Other characteristics

5.3.1 Rock Type

As described above, there are numerous types of sedimentary rocks. In most cases, a rock will be a combination of several grain types, therefore, a modifier such as a sandy siltstone, or a silty sandstone can be used. The modifier indicates that a significant portion of the rock type is composed of the modifier. Other modifiers can include carbonaceous, calcareous, siliceous, etc.

Grain size is the basis for the classification of clastic sedimentary rocks. Figure 4 is the Udden-Wentworth classification that will be assigned to sedimentary rocks. The individual boundaries are slightly different than the USCS subdivision for soil classification. For field determination of grain sizes, a scale can be used for the coarse grained rocks. For example, the division between siltstone and claystone may not be measurable in the field. The boundary shall be determined by use of a hand lens. If the grains cannot be seen with the naked eye but are distinguishable with a hand lens, the rock is a siltstone. If the grains are not distinguishable with a hand lens, the rock is a claystone.

FIGURE 4

GRAIN SIZE CLASSIFICATION FOR ROCKS

Particle Name	Grain Size Diameter
Cobbles	> 64 mm
Pebbles	4 - 64 mm
Granules	2 - 4 mm
Very Coarse Sand	1 - 2 mm
Coarse Sand	0.5 - 1 mm
Medium Sand	0.25 - 0.5 mm
Fine Sand	0.125 - 0.25 mm
Very Fine Sand	0.0625 - 0.125 mm
Silt	0.0039 - 0.0625 mm

After Wentworth, 1922

5.3.2 Color

The color of a rock can be determined in a similar manner as for soil samples. Rock core samples shall be classified while wet, when possible, and air cored samples shall be scraped clean of cuttings prior to color classifications.

Rock color charts shall not be used unless specified by the Project Manager.

5.3.3 Bedding Thickness

The bedding thickness designations applied to soil classification (see Figure 3) will also be used for rock classification.

5.3.4 Hardness

The hardness of a rock is a function of the compaction, cementation, and mineralogical composition of the rock. A relative scale for sedimentary rock hardness is as follows:

- Soft - Weathered, considerable erosion of core, easily gouged by screwdriver, scratched by fingernail. Soft rock crushes or deforms under pressure of a pressed hammer. This term is always used for the hardness of the saprolite (decomposed rock which occupies the zone between the lowest soil horizon and firm bedrock).
- Medium soft - Slight erosion of core, slightly gouged by screwdriver, or breaks with crumbly edges from single hammer blow.
- Medium hard - No core erosion, easily scratched by screwdriver, or breaks with sharp edges from single hammer blow.
- Hard - Requires several hammer blows to break and has sharp conchoidal breaks. Cannot be scratched with screwdriver.

Note the difference in usage here of the works "scratch" and "gouge." A scratch shall be considered a slight depression in the rock (do not mistake the scraping off of rock flour from drilling with a scratch in the rock itself), while a gouge is much deeper.

5.3.5 Fracturing

The degree of fracturing or brokenness of a rock is described by measuring the fractures or joint spacing. After eliminating drilling breaks, the average spacing is calculated and the fracturing is described by the following terms:

- Very broken (V. BR.) - Less than 2-inch spacing between fractures
- Broken (BR.) - 2-inch to 1-foot spacing between fractures
- Blocky (BL.) - 1- to 3-foot spacing between fractures
- Massive (M.) - 3 to 10-foot spacing between fractures

The structural integrity of the rock can be approximated by calculating the Rock Quality Designation (RQD) of cores recovered. The RQD is determined by adding the total lengths of all pieces exceeding 4 inches and dividing by the total length of the coring run, to obtain a percentage.

Method of Calculating RQD
(After Deere, 1964)

$$RQD\ \% = r/l \times 100$$

r = Total length of all pieces of the lithologic unit being measured, which are greater than 4 inches length, and have resulted from natural breaks. Natural breaks include slickensides, joints, compaction slicks, bedding plane partings (not caused by drilling), friable zones, etc.

l = Total length of the coring run.

5.3.6 Weathering

The degree of weathering is a significant parameter that is important in determining weathering profiles and is also useful in engineering designs. The following terms can be applied to distinguish the degree of weathering:

- Fresh - Rock shows little or no weathering effect. Fractures or joints have little or no staining and rock has a bright appearance.
- Slight - Rock has some staining which may penetrate several centimeters into the rock. Clay filling of joints may occur. Feldspar grains may show some alteration.
- Moderate - Most of the rock, with exception of quartz grains, is stained. Rock is weakened due to weathering and can be easily broken with hammer.
- Severe - All rock including quartz grains is stained. Some of the rock is weathered to the extent of becoming a soil. Rock is very weak.

5.3.7 Other Characteristics

The following items shall be included in the rock description:

- Description of contact between two rock units. These can be sharp or gradational.
- Stratification (parallel, cross stratified).
- Description of any filled cavities or vugs.
- Cementation (calcareous, siliceous, hematitic).
- Description of any joints or open fractures.
- Observation of the presence of fossils.
- Notation of joints with depth, approximate angle to horizontal, any mineral filling or coating, and degree of weathering.

All information shown on the boring logs shall be neat to the point where it can be reproduced on a copy machine for report presentation. The data shall be kept current to provide control of the drilling program and to indicate various areas requiring special consideration and sampling.

5.3.8 Additional Terms Used in the Description of Rock

The following terms are used to further identify rocks:

- Seam - Thin (12 inches or less), probably continuous layer.
- Some - Indicates significant (15 to 40 percent) amounts of the accessory material. For example, rock composed of seams of sandstone (70 percent) and shale (30 percent) would be "sandstone -- some shale seams."
- Few - Indicates insignificant (0 to 15 percent) amounts of the accessory material. For example, rock composed of seam of sandstone (90 percent) and shale (10 percent) would be "sandstone -- few shale seams."
- Interbedded - Used to indicate thin or very thin alternating seams of material occurring in approximately equal amounts. For example, rock composed of thin alternating seams of sandstone (50 percent) and shale (50 percent) would be "interbedded sandstone and shale."
- Interlayered - Used to indicate thick alternating seams of material occurring in approximately equal amounts.

The preceding sections describe the classification of sedimentary rocks. The following are some basic names that are applied to igneous rocks:

- Basalt - A fine-grained extrusive rock composed primarily of calcic plagioclase and pyroxene.
- Rhyolite - A fine-grained volcanic rock containing abundant quartz and orthoclase. The fine-grained equivalent of a granite.
- Granite - A coarse-grained plutonic rock consisting essentially of alkali feldspar and quartz.
- Diorite - A coarse-grained plutonic rock consisting essentially of sodic plagioclase and hornblende.
- Gabbro - A coarse-grained plutonic rock consisting of calcic plagioclase and clinopyroxene. Loosely used for any coarse-grained dark igneous rock.

The following are some basic names that are applied to metamorphic rocks:

- Slate - A very fine-grained foliated rock possessing a well developed slaty cleavage. Contains predominantly chlorite, mica, quartz, and sericite.
- Phyllite - A fine-grained foliated rock that splits into thin flaky sheets with a silky sheen on cleavage surface.
- Schist - A medium to coarse-grained foliated rock with subparallel arrangement of the micaceous minerals which dominate its composition.
- Gneiss - A coarse-grained foliated rock with bands rich in granular and platy minerals.
- Quartzite - A fine- to coarse-grained nonfoliated rock breaking across grains, consisting essentially of quartz sand with silica cement.

5.4 Abbreviations

Abbreviations may be used in the description of a rock or soil. However, they shall be kept at a minimum. Following are some of the abbreviations that may be used:

C	-	Coarse	Lt	-	Light	Yl	-	Yellow
Med	-	Medium	BR	-	Broken	Or	-	Orange
F	-	Fine	BL	-	Blocky	SS	-	Sandstone
V	-	Very	M	-	Massive	Sh	-	Shale
Sl	-	Slight	Br	-	Brown	LS	-	Limestone
Occ	-	Occasional	Bl	-	Black	Fgr	-	Fine-grained
Tr	-	Trace						

5.5 Boring Logs and Documentation

This section describes in more detail the procedures to be used in completing boring logs in the field. Information obtained from the preceding sections shall be used to complete the logs. A sample boring log has been provided as Figure 5.

The field geologist/engineer shall use this example as a guide in completing each boring log. Each boring log shall be fully described by the geologist/engineer as the boring is being drilled. Every sheet contains space for 25 feet of log. Information regarding classification details is provided either on the back of the boring log or on a separate sheet, for field use.

5.5.1 Soil Classification

- Identify site name, boring number, job number, etc. Elevations and water level data to be entered when surveyed data is available.

- Enter sample number (from SPT) under appropriate column. Enter depth sample was taken from (1 block = 1 foot). Fractional footages, i.e., change of lithology at 13.7 feet, shall be lined off at the proportional location between the 13- and 14-foot marks. Enter blow counts (Standard Penetration Resistance) diagonally (as shown). Standard penetration resistance is covered in Section 5.2.3.

- Determine sample recovery/sample length as shown. Measure the total length of sample recovered from the split-spoon sampler, including material in the drive shoe. Do not include cuttings or wash material that may be in the upper portion of the sample tube.

- Indicate any change in lithology by drawing a line at the appropriate depth. For example, if clayey silt was encountered from 0 to 5.5 feet and shale from 5.5 to 6.0 feet, a line shall be drawn at this increment. This information is helpful in the construction of cross-sections. As an alternative, symbols may be used to identify each change in lithology.

- The density of granular soils is obtained by adding the number of blows for the last two increments. Refer to Density of Granular Soils Chart on back of log sheet. For consistency of cohesive soils refer also to the back of log sheet - Consistency of Cohesive Soils. Enter this information under the appropriate column. Refer to Section 5.2.3.

FIGURE 5
COMPLETED BORING LOG (EXAMPLE)

BORING LOG

Page 1 of 1

PROJECT NAME: NSB- SITE
PROJECT NUMBER: 9594
DRILLING COMPANY: SOILTEST CO.
DRILLING RIG: CME-55

BORING NUMBER: SB/MW 1
DATE: 3/8/96
GEOLOGIST: SJ CONTI
DRILLER: R. ROCK

Sample No. and Type or RQD	Depth (Ft.) or Run No.	Blows / 6" or RQD (%)	Sample Recovery / Sample Length	Lithology Change (Depth/Ft.) or Screened Interval	Soil Density/ Consistency or Rock Hardness	Color	Material Classification	USCS*	Remarks	PID/FID Reading (ppm) Sample	Sampler BZ	Borehole**	Driller BZ**
S-1 @ 0800	0.0	7/6	1.5/2.0		M DENSE	BRN TO BLK	SILTY SAND - SOME	SM	MOIST SL. ORG. ODOR	5	0	0	0
	2.0	9/10					ROCK FR.: TR BRICKS						
							(FILL)		FILL TO 4'±				
	4.0			4.0									
S-2 @ 0810		5/7	2.0/2.0		M DENSE	BRN	SILTY SAND - TR FINE	SM	MOIST - W ODOR	10	0	-	-
	6.0	9/8					GRAVEL		NAT. MATL. TOOK SAMPLE				
	•			7' ∇					SB01-0406 FOR ANALYSIS				
	8.0			8.0									
S-3 @ 0820		6/8	1.9/2.0		DENSE	TAN BRN	FINE TO COARSE SAND	SW	WET	0	0	0	0
	10.0	17/16					TR. F. GRAVEL		HIT WATER ≈ 7'±				
	12.0			12.0									
S-4 @ 0830		7/6	1.6/2.0		STIFF	GRAY	SILTY CLAY	CL	MOIST → WET	0	.5	-	-
	14.0	5/8											
	15.0			15.0					AUGER REF @ 15'				
				11 HE HE 16	M HARD	BRN	SILTSTONE	VER	WEATHERED				
									LD X JNTS @ 15.5				
9/5	①		4.0/5.0						WATER STAINS @ 16.5, 17.1, 17.5	0	0	0	0
				19'					LOSING SOME				
	20.0				HARD	GRAY	SANDSTONE - SOME	BR	DRILL H2O @ 17'±				
							SILTSTONE		SET TEMP 6" CAS TO 15.5				
4.9/5.0	②		5.0/5.0						SET 2" Φ PVC SCREEN 16-25	0	0	0	0
									SAND 14-25				
	25.0			25					PELLETS 12-14				

* When rock coring, enter rock brokeness.

** Include monitor reading in 6 foot intervals @ borehole. Increase reading frequency if elevated response read.

• 1-20Z
1-80Z

Drilling Area Background (ppm): 0

Remarks: CME-55 R16, 4 1/4" ID HSA - 9" OD ±
2" SPLIT SPOONS - 140 LB HAMMER - 30" DROP
NX CORE IN BEDROCK RUN ① = 25 min. RUN ② = 15 min

Converted to Well: Yes ✓ No _____ Well I.D. #: MW-1

- Enter color of the material in the appropriate column.

- Describe material using the USCS. Limit this column for sample description only. The predominant material is described last. If the primary soil is silt but has fines (clay) - use clayey silt. Limit soil descriptors to the following:

  - Trace: 0 - 10 percent
  - Some: 11 - 30 percent
  - And/Or: 31 - 50 percent

- Also indicate under Material Classification if the material is fill or natural soils. Indicate roots, organic material, etc.

- Enter USCS symbol - use chart on back of boring log as a guide. If the soils fall into one of two basic groups, a borderline symbol may be used with the two symbols separated by a slash. For example ML/CL or SM/SP.

- The following information shall be entered under the "Remarks" column and shall include, but is not limited by, the following:

  - Moisture - estimate moisture content using the following terms - dry, moist, wet and saturated. These terms are determined by the individual. Whatever method is used to determine moisture, be consistent throughout the log.

  - Angularity - describe angularity of coarse grained particles using the terms angular, subangular, subrounded, or rounded. Refer to ASTM D 2488 or Earth Manual for criteria for these terms.

  - Particle shape - flat, elongated, or flat and elongated.

  - Maximum particle size or dimension.

  - Water level observations.

  - Reaction with HCl - none, weak, or strong.

- Additional comments:

  - Indicate presence of mica, caving of hole, when water was encountered, difficulty in drilling, loss or gain of water.

  - Indicate odor and Photoionization Detector (PID) or Flame Ionization Detector (FID) reading if applicable.

  - Indicate any change in lithology by drawing a line through the lithology change column and indicate the depth. This will help when cross-sections are subsequently constructed.

  - At the bottom of the page indicate type of rig, drilling method, hammer size and drop, and any other useful information (i.e., borehole size, casing set, changes in drilling method).

- Vertical lines shall be drawn (as shown in Figure 5) in columns 6 to 8 from the bottom of each sample to the top of the next sample to indicate consistency of material from sample to sample, if the material is consistent. Horizontal lines shall be drawn if there is a change in lithology, then vertical lines drawn to that point.

- Indicate screened interval of well, as needed, in the lithology column. Show top and bottom of screen. Other details of well construction are provided on the well construction forms.

5.5.2 Rock Classification

- Indicate depth at which coring began by drawing a line at the appropriate depth. Indicate core run depths by drawing coring run lines (as shown) under the first and fourth columns on the log sheet. Indicate RQD, core run number, RQD percent, and core recovery under the appropriate columns.

- Indicate lithology change by drawing a line at the appropriate depth as explained in Section 5.5.1.

- Rock hardness is entered under designated column using terms as described on the back of the log or as explained earlier in this section.

- Enter color as determined while the core sample is wet; if the sample is cored by air, the core shall be scraped clean prior to describing color.

- Enter rock type based on sedimentary, igneous or metamorphic. For sedimentary rocks use terms as described in Section 5.3. Again, be consistent in classification. Use modifiers and additional terms as needed. For igneous and metamorphic rock types use terms as described in Sections 5.3.8.

- Enter brokenness of rock or degree of fracturing under the appropriate column using symbols VBR, BR, BL, or M as explained in Section 5.3.5 and as noted on the back of the Boring Log.

- The following information shall be entered under the remarks column. Items shall include but are not limited to the following:

  - Indicate depths of joints, fractures and breaks and also approximate to horizontal angle (such as high, low), i.e., 70° angle from horizontal, high angle.
  - Indicate calcareous zones, description of any cavities or vugs.
  - Indicate any loss or gain of drill water.
  - Indicate drop of drill tools or change in color of drill water.

- Remarks at the bottom of Boring Log shall include:

  - Type and size of core obtained.
  - Depth casing was set.
  - Type of rig used.

- As a final check the boring log shall include the following:

  - Vertical lines shall be drawn as explained for soil classification to indicate consistency of bedrock material.

  - If applicable, indicate screened interval in the lithology column. Show top and bottom of screen. Other details of well construction are provided on the well construction forms.

5.5.3 Classification of Soil and Rock from Drill Cuttings

The previous sections describe procedures for classifying soil and rock samples when cores are obtained. However, some drilling methods (air/mud rotary) may require classification and borehole logging based on identifying drill cuttings removed from the borehole. Such cuttings provide only general information on subsurface lithology. Some procedures that shall be followed when logging cuttings are:

- Obtain cutting samples at approximately 5-foot intervals, sieve the cuttings (if mud rotary drilling) to obtain a cleaner sample, place the sample into a small sample bottle or "zip lock" bag for future reference, and label the jar or bag (i.e. hole number, depth, date, etc.). Cuttings shall be closely examined to determine general lithology.
- Note any change in color of drilling fluid or cuttings, to estimate changes in lithology.
- Note drop or chattering of drilling tools or a change in the rate of drilling, to determine fracture locations or lithologic changes.
- Observe loss or gain of drilling fluids or air (if air rotary methods are used), to identify potential fracture zones.
- Record this and any other useful information onto the boring log as provided in Figure 1.

This logging provides a general description of subsurface lithology and adequate information can be obtained through careful observation of the drilling process. It is recommended that split-barrel and rock core sampling methods be used at selected boring locations during the field investigation to provide detailed information to supplement the less detailed data generated through borings drilled using air/mud rotary methods.

5.6 Review

Upon completion of the borings logs, copies shall be made and reviewed. Items to be reviewed include:

- Checking for consistency of all logs.
- Checking for conformance to the guideline.
- Checking to see that all information is entered in their respective columns and spaces.

6.0 REFERENCES

Unified Soil Classification System (USCS).

ASTM D2488, 1985.

Earth Manual, U.S. Department of the Interior, 1974.

7.0 RECORDS

Originals of the boring logs shall be retained in the project files.

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1.0 PURPOSE

Utilities such as electric service lines, natural or propane gas lines, water and sewage lines, telecommunications, and steam lines are very often in the immediate vicinity of work locations. Contact with underground or overhead utilities can have serious consequences including employee injury/fatality, property and equipment damage, substantial financial impacts, and loss of utility service to users.

The purpose of this procedure is to provide minimum requirements and technical guidelines regarding the appropriate procedures to be followed when performing subsurface and overhead utility locating services. It is the policy of Tetra Tech NUS, Inc. (TtNUS) to provide a safe and healthful work environment for the protection of our employees. The purpose of this Standard Operating Procedure (SOP) is to aid in achieving the objectives of this policy, to present the acceptable procedures pertaining to utility locating and excavation clearance activities, and to present requirements and restrictions relevant to these types of activities. This SOP must be reviewed by any employee potentially involved with underground or overhead utility locating and avoidance activities.

2.0 SCOPE

This procedure applies to all TtNUS field activities where there may be potential contact with underground or overhead utilities. This procedure provides a description of the principles of operation, instrumentation, applicability, and implementability of typical methods used to determine the presence and avoidance of contact with utility services. This procedure is intended to assist with work planning and scheduling, resource planning, field implementation, and subcontractor procurement. Utility locating and excavation clearance requires site-specific information prior to the initiation of any such activities on a specific project. This SOP is not intended to provide a detailed description of methodology and instrument operation. Specialized expertise during both planning and execution of several of the methods presented may also be required.

3.0 GLOSSARY

Electromagnetic Induction (EMI) Survey - A geophysical exploration method whereby electromagnetic fields are induced in the ground and the resultant secondary electromagnetic fields are detected as a measure of ground conductivity.

Magnetometer – A device used for precise and sensitive measurements of magnetic fields.

Magnetic Survey – A geophysical survey method that depends on detection of magnetic anomalies caused by the presence of buried ferromagnetic objects.

Metal Detection – A geophysical survey method that is based on electromagnetic coupling caused by underground conductive objects.

Vertical Gradiometer – A magnetometer equipped with two sensors that are vertically separated by a fixed distance. It is best suited to map near surface features and is less susceptible to deep geologic features.

Ground Penetrating Radar – Ground Penetrating Radar (GPR) involves specialized radar equipment whereby a signal is sent into the ground via a transmitter. Some portion of the signal will be reflected from the subsurface material, which is then recorded with a receiver and electronically converted into a graphic picture.

4.0 RESPONSIBILITIES

Project Manager (PM)/Task Order Manager (TOM) - Responsible for ensuring that all field activities are conducted in accordance with this procedure.

Site Manager (SM)/Field Operations Leader (FOL) - Responsible for the onsite verification that all field activities are performed in compliance with approved SOPs or as otherwise directed by the approved project plan(s).

Site Health & Safety Officer (SHSO) – Responsible to provide technical assistance and verify full compliance with this SOP. The SHSO is also responsible for reporting any deficiencies to the Corporate Health and Safety Manager (HSM) and to the PM/TOM.

Health & Safety Manager (HSM) – Responsible for preparing, implementing, and modifying corporate health and safety policy and this SOP.

Site Personnel – Responsible for performing their work activities in accordance with this SOP and the TtNUS Health and Safety Policy.

5.0 PROCEDURES

This procedure addresses the requirements and technical procedures that must be performed to minimize the potential for contact with underground and overhead utility services. These procedures are addressed individually from a buried and overhead standpoint.

5.1 Buried Utilities

Buried utilities present a heightened concern because their location is not typically obvious by visual observation, and it is common that their presence and/or location is unknown or incorrectly known on client properties. This procedure must be followed prior to beginning any subsurface probing or excavation that might potentially be in the vicinity of underground utility services. In addition, the Utility Clearance Form (Attachment 3) must be completed for every location or cluster of locations where intrusive activities will occur.

Where the positive identification and de-energizing of underground utilities cannot be obtained and confirmed using the following steps, the PM/TOM is responsible for arranging for the procurement of a qualified, experienced, utility locating subcontractor who will accomplish the utility location and demarcation duties specified herein.

1. A comprehensive review must be made of any available property maps, blue lines, or as-builts prior to site activities. Interviews with local personnel familiar with the area should be performed to provide additional information concerning the location of potential underground utilities. Information regarding utility locations shall be added to project maps upon completion of this exercise.

2., A visual site inspection must be performed to compare the site plan information to actual field conditions. Any findings must be documented and the site plan/maps revised. The area(s) of proposed excavation or other subsurface activities must be marked at the site in white paint or pin flags to identify those locations of the proposed intrusive activities. The site inspection should focus on locating surface indications of potential underground utilities. Items of interest include the presence of nearby area lights, telephone service, drainage grates, fire hydrants, electrical service vaults/panels, asphalt/concrete scares and patches, and topographical depressions. Note the location of any emergency shut off switches. Any additional information regarding utility

locations shall be added to project maps upon completion of this exercise and returned to the PM/TOM.

3. If the planned work is to be conducted on private property (e.g., military installations, manufacturing facilities, etc.) the FOL must identify and contact appropriate facility personnel (e.g., public works or facility engineering) before any intrusive work begins to inquire about (and comply with) property owner requirements. It is important to note that private property owners may require several days to several weeks advance notice prior to locating utilities.

4. If the work location is on public property, the state agency that performs utility clearances must be notified (see Attachment 1). State "one-call" services must be notified prior to commencing fieldwork per their requirements. Most one-call services require, by law, 48- to 72-hour advance notice prior to beginning any excavation. Such services typically assign a "ticket" number to the particular site. This ticket number must be recorded for future reference and is valid for a specific period of time, but may be extended by contacting the service again. The utility service will notify utility representatives who then mark their respective lines within the specified time frame. It should be noted that most military installations own their own utilities but may lease service and maintenance from area providers. Given this situation, "one call" systems may still be required to provide location services on military installations.

5. Utilities must be identified and their locations plainly marked using pin flags, spray paint, or other accepted means. The location of all utilities must be noted on a field sketch for future inclusion on project maps. Utility locations are to be identified using the following industry-standard color code scheme, unless the property owner or utility locator service uses a different color code:

white	excavation/subsurface investigation location
red	electrical
yellow	gas, oil, steam
orange	telephone, communications
blue	water, irrigation, slurry
green	sewer, drain

6. Where utility locations are not confirmed with a high degree of confidence through drawings, schematics, location services, etc., the work area must be thoroughly investigated prior to beginning the excavation. In these situations, utilities must be identified using safe and effective methods such as passive and intrusive surveys, or the use of non-conductive hand tools. Also, in situations where such hand tools are used, they should always be used in conjunction with suitable detection equipment, such as the items described in Section 6.0 of this SOP. Each method has advantages and disadvantages including complexity, applicability, and price. It also should be noted that in some states, initial excavation is required by hand to a specified depth.

7. At each location where trenching or excavating will occur using a backhoe or other heavy equipment, and where utility identifications and locations cannot be confirmed prior to groundbreaking, the soil must be probed using a device such as a tile probe which is made of non-conductive material such as fiberglass. If these efforts are not successful in clearing the excavation area of suspect utilities, hand shoveling must be performed for the perimeter of the intended excavation.

8. All utilities uncovered or undermined during excavation must be structurally supported to prevent potential damage. Unless necessary as an emergency corrective measure, TtNUS shall not make any repairs or modifications to existing utility lines without prior permission of the utility owner, property owner, and Corporate HSM. All repairs require that the line be locked-out/tagged-out prior to work.

5.2 Overhead Power Lines

If it is necessary to work within the minimum clearance distance of an overhead power line, the overhead line must be de-energized and grounded, or re-routed by the utility company or a registered electrician. If protective measures such as guarding, isolating, or insulating are provided, these precautions must be adequate to prevent employees from contacting such lines directly with any part of their body or indirectly though conductive materials, tools, or equipment.

The following table provides the required minimum clearances for working in proximity to overhead power lines.

Nominal Voltage	Minimum Clearance
0 -50 kV	10 feet, or one mast length; whichever is greater
50+ kV	10 feet plus 4 inches for every 10 kV over 50 kV or 1.5 mast lengths; whichever is greater

6.0 UNDERGROUND LOCATING TECHNIQUES

A variety of supplemental utility locating approaches are available and can be applied when additional assurance is needed. The selection of the appropriate method(s) to employ is site-specific and should be tailored to the anticipated conditions, site and project constraints, and personnel capabilities.

6.1 Geophysical Methods

Geophysical methods include electromagnetic induction, magnetics, and ground penetrating radar. Additional details concerning the design and implementation of electromagnetic induction, magnetics, and ground penetrating radar surveys can be found in one or more of the TtNUS SOPs included in the References (Section 8.0).

Electromagnetic Induction

Electromagnetic Induction (EMI) line locators operate either by locating a background signal or by locating a signal introduced into the utility line using a transmitter. A utility line acts like a radio antenna, producing electrons, which can be picked up with a radiofrequency receiver. Electrical current carrying conductors have a 60HZ signal associated with them. This signal occurs in all power lines regardless of voltage. Utilities in close proximity to power lines or used as grounds may also have a 60HZ signal, which can be picked up with an EM receiver. A typical example of this type of geophysical equipment is an EM-61.

EMI locators specifically designed for utility locating use a special signal that is either indirectly induced onto a utility line by placing the transmitter above the line or directly induced using an induction clamp. The clamp induces a signal on the specific utility and is the preferred method of tracing since there is little chance of the resulting signals being interfered with. A good example of this type of equipment is the Schonstedt® MAC-51B locator. The MAC-51B performs inductively traced surveys, simple magnetic locating, and traced nonmetallic surveys.

When access can be gained inside a conduit to be traced, a flexible insulated trace wire can be used. This is very useful for non-metallic conduits but is limited by the availability of gaining access inside the pipe.

Magnetics

Magnetic locators operate by detecting the relative amounts of buried ferrous metal. They are incapable of locating or identifying nonferrous utility lines but can be very useful for locating underground storage tanks (UST's), steel utility lines, and buried electrical lines. A typical example of this type of equipment is the Schonstedt® GA-52Cx locator. The GA-52Cx is capable of locating 4-inch steel pipe up to 8 feet deep.

Non-ferrous lines are often located by using a typical plumbing tool (snake) fed through the line. A signal is then introduced to the snake that is then traced.

Ground Penetrating Radar

Ground Penetrating Radar (GPR) involves specialized radar equipment whereby a signal is sent into the ground via a transmitter. Some portion of the signal will be reflected from the subsurface material, which is then recorded with a receiver and electronically converted into a graphic picture. In general, an object which is harder than the surrounding soil will reflect a stronger signal. Utilities, tunnels, UST's, and footings will reflect a stronger signal than the surrounding soil. Although this surface detection method may determine the location of a utility, this method does not specifically identify utilities (i.e., water vs. gas, electrical vs. telephone); hence, verification may be necessary using other methods. This method is somewhat limited when used in areas with clay soil types or with a high water table.

6.2 Passive Detection Surveys

Acoustic Surveys

Acoustic location methods are generally most applicable to waterlines or gas lines. A highly sensitive Acoustic Receiver listens for background sounds of water flowing (at joints, leaks, etc.) or to sounds introduced into the water main using a transducer. Acoustics may also be applicable to determine the location of plastic gas lines.

Thermal Imaging

Thermal (i.e., infrared) imaging is a passive method for detecting the heat emitted by an object. Electronics in the infrared camera convert subtle heat differentials into a visual image on the viewfinder or a monitor. The operator does not look for an exact temperature; rather they look for heat anomalies (either elevated or suppressed temperatures) characteristic of a potential utility line.

The thermal fingerprint of underground utilities results from differences in temperature between the atmosphere and the fluid present in a pipe or the heat generated by electrical resistance. In addition, infrared scanners may be capable of detecting differences in the compaction, temperature and moisture content of underground utility trenches. High-performance thermal imagery can detect temperature differences to hundredths of a degree.

6.3 Intrusive Detection Surveys

Vacuum Excavation

Vacuum excavation is used to physically expose utility services. The process involves removing the surface material over approximately a 1' x 1' area at the site location. The air-vacuum process proceeds with the simultaneous action of compressed air-jets to loosen soil and vacuum extraction of the resulting

debris. This process ensures the integrity of the utility line during the excavation process, as no hammers, blades, or heavy mechanical equipment comes into contact with the utility line, eliminating the risk of damage to utilities. The process continues until the utility is uncovered. Vacuum excavation can be used at the proposed site location to excavate below the "utility window" which is usually 8 feet.

Hand Excavation

When the identification and location of underground utilities cannot be positively confirmed through document reviews and/or other methods, borings and excavations may be cleared via the use of non-conductive hand tools. This should always be done in conjunction with the use of detection equipment. This would be required for all locations where there is a potential to impact buried utilities. The minimum hand-excavation depth that must be reached is to be determined considering the geographical location of the work site. This approach recognizes that the placement of buried utilities is influenced by frost line depths that vary by geographical region. Attachment 2 presents frost line depths for the regions of the contiguous United States. At a minimum, hand excavation depths must be at least to the frost line depth (see Attachment 2) plus two (2) feet, but never less than 4 feet below ground surface (bgs). For hand excavation, the hole created must be reamed large enough to be at least the diameter of the drill rig auger or bit prior to drilling. For soil gas surveys, the survey probe shall be placed as close as possible to the cleared hand excavation. It is important to note that a post-hole digger must not be used in this type of hand excavation activity.

Tile Probe Surveys

For some soil types, site conditions, and excavation requirements, non-conductive tile probes may be used. A tile probe is a "T"-handled rod of varying lengths that can be pushed into the soil to determine if any obstructions exist at that location. Tile probes constructed of fiberglass or other nonconductive material are readily-available from numerous vendors. Tile probes must be performed to the same depth requirements as previously specified. As with other types of hand excavating activities, the use of a non-conductive tile probe, should always be in conjunction with suitable utility locating detection equipment.

7.0 INTRUSIVE ACTIVITIES SUMMARY

The following list summarizes the activities that must be performed prior to beginning subsurface activities:

1. Map and mark all subsurface locations and excavation boundaries using white paint or markers specified by the client or property owner.

2. Notify the property owner and/or client that the locations are marked. At this point, drawings of locations or excavation boundaries shall be provided to the property owner and/or client so they may initiate (if applicable) utility clearance.

   Note: Drawings with confirmed locations should be provided to the property owner and/or client as soon as possible to reduce potential time delays.

3. Notify "One Call" service. If possible, arrange for an appointment to show the One Call representative the surface locations or excavation boundaries in person. This will provide a better location designation to the utilities they represent. You should have additional drawings should you need to provide plot plans to the One Call service.

4. Implement supplemental utility detection techniques as necessary and appropriate to conform utility locations or the absence thereof.

5. Complete Attachment 3, Utility Clearance Form. This form should be completed for each excavation location. In situations where multiple subsurface locations exist within the close proximity of one another, one form may be used for multiple locations provided those locations are noted on the Utility Clearance Form. Upon completion, the Utility Clearance Form and revised/annotated utility location map becomes part of the project file.

8.0 REFERENCES

OSHA Letter of Interpretation, Mr. Joseph Caldwell, Attachment 4
OSHA 29 CFR 1926(b)(2)
OSHA 29 CFR 1926(b)(3)
TtNUS Utility Locating and Clearance Policy
TtNUS SOP GH-3.1; Resistivity and Electromagnetic Induction
TtNUS SOP GH-3.2; Magnetic and Metal Detection Surveys
TtNUS SOP GH-3.4; Ground-penetrating Radar Surveys

ATTACHMENT 1
LISTING OF UNDERGROUND UTILITY CLEARANCE RESOURCES

American Public Works Association
2345 Grand Boulevard, Suite 500, Kansas City, MO 64108-2625
Phone (816) 472-6100 • Fax (816) 472-1610
Web www.apwa.net • E-mail apwa@apwa.net

ONE-CALL SYSTEMS INTERNATIONAL CONDENSED DIRECTORY

Alabama
Alabama One-Call
1-800-292-8525

Alaska
Locate Call Center of Alaska, Inc.
1-800-478-3121

Arizona
Arizona Blue Stake
1-800-782-5348

Arkansas
Arkansas One Call System, Inc.
1-800-482-8998

California
Underground Service Alert North
1-800-227-2600
Underground Service Alert of Southern California
1-800-227-2600

Colorado
Utility Notification Center of Colorado
1-800-922-1987

Connecticut
Call Before You Dig
1-800-922-4455

Delaware
Miss Utility of Delmarva
1-800-282-8555

Florida
Sunshine State One-Call of Florida, Inc.
1-800-432-4770

Georgia
Underground Protection Center, Inc.
1-800-282-7411

Hawaii
Underground Service Alert North
1-800-227-2600

Idaho
Dig Line Inc.
1-800-342-1585
Kootenai County One-Call
1-800-428-4950
Shoshone - Benewah One-Call
1-800-398-3285

Illinois
JULIE, Inc.
1-800-892-0123
Digger (Chicago Utility Alert Network)
312-744-7000

Indiana
Indiana Underground Plant Protection Service
1-800-382-5544

Iowa
Iowa One-Call
1-800-292-8989

Kansas
Kansas One-Call System, Inc.
1-800-344-7233

Kentucky
Kentucky Underground Protection Inc.
1-800-752-6007

Louisiana
Louisiana One Call System, Inc.
1-800-272-3020

Maine
Dig Safe System, Inc.
1-888-344-7233

Maryland
Miss Utility
1-800-257-7777
Miss Utility of Delmarva
1-800-282-8555

Massachusetts
Dig Safe System, Inc.
1-888-344-7233

Michigan
Miss Dig System, Inc.
1-800-482-7171

Minnesota
Gopher State One Call
1-800-252-1166

Mississippi
Mississippi One-Call System, Inc
1-800-227-6477

Missouri
Missouri One-Call System, Inc.
1-800-344-7483

Montana
Utilities Underground Protection Center
1-800-424-5555
Montana One Call Center
1-800-551-8344

Nebraska
Diggers Hotline of Nebraska
1-800-331-5666

Nevada
Underground Service Alert North
1-800-227-2600

New Hampshire
Dig Safe System, Inc.
1-888-344-7233

New Jersey
New Jersey One Call
1-800-272-1000

New Mexico
New Mexico One Call System, Inc.
1-800-321-2537
Las Cruces- Dona Ana Blue Stakes
1-888-526-0400

New York
Dig Safely New York
1-800-962-7962
New York City- Long Island One Call Center
1-800-272-4480

North Carolina
The North Carolina One-Call Center, Inc.
1-800-632-4949

North Dakota
North Dakota One-Call
1-800-795-0555

Ohio
Ohio Utilities Protection Service
1-800-362-2764
Oil & Gas Producers Underground Protect'n Svc
1-800-925-0988

Oklahoma
Call Okie
1-800-522-6543

Oregon
Oregon Utility Notification Center/One Call Concepts
1-800-332-2344

Pennsylvania
Pennsylvania One Call System, Inc.
1-800-242-1776

Rhode Island
Dig Safe System, Inc.
1-888-344-7233

South Carolina
Palmetto Utility Protection Service Inc.
1-888-721-7877

South Dakota
South Dakota One Call
1-800-781-7474

Tennessee
Tennessee One-Call System, Inc.
1-800-351-1111

ATTACHMENT 1 (Continued)

Texas
Texas One Call System
1-800-245-4545
Texas Excavation Safety System, Inc.
1-800-344-8377
Lone Star Notification Center
1-800-669-8344

Utah
Blue Stakes of Utah
1-800-662-4111

Vermont
Dig Safe System, Inc.
1-888-344-7233

Virginia
Miss Utility of Virginia
1-800-552-7001
Miss Utility (Northern Virginia)
1-800-257-7777

Washington
Utilities Underground Location Center
1-800-424-5555
Northwest Utility Notification Center
1-800-553-4344
Inland Empire Utility Coordinating Council
509-456-8000

West Virginia
Miss Utility of West Virginia, Inc.
1-800-245-4848

Wisconsin
Diggers Hotline, Inc.
1-800-242-8511

Wyoming
Wyoming One-Call System, Inc.
1-800-348-1030
Call Before You Dig of Wyoming
1-800-849-2476

District of Columbia
Miss Utility
1-800-257-7777

Alberta
Alberta One-Call Corporation
1-800-242-3447

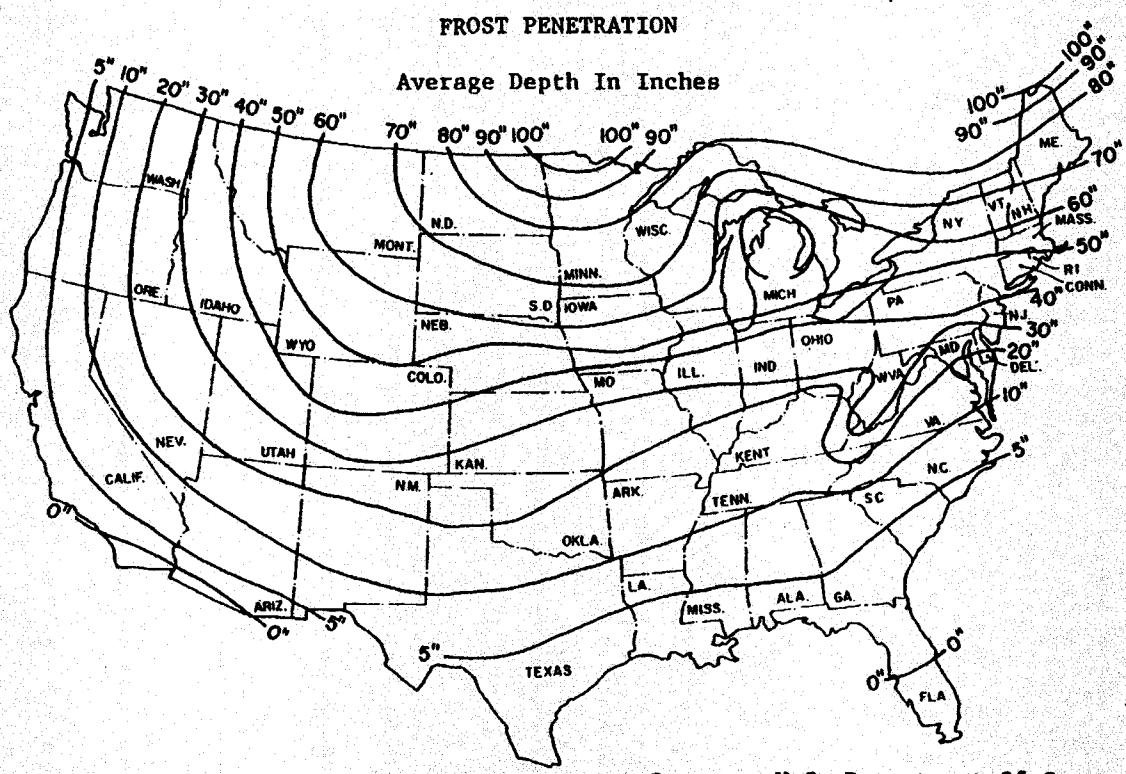
British Columbia
BC One Call
1-800-474-6886

Ontario
Ontario One-Call System
1-800-400-2255

Quebec
Info-Excavation
1-800-663-9228

ATTACHMENT 2

FROST LINE PENETRATION DEPTHS BY GEOGRAPHIC LOCATION



Courtesy U.S. Department Of Commerce

ATTACHMENT 3
UTILITY CLEARANCE FORM

Client: _____ Project Name: _____

Project No.: _____ Completed By: _____

Location Name: _____ Work Date: _____

Excavation Method/Overhead Equipment: _____

1. Underground Utilities — Circle One

a)	Review of existing maps?	yes no N/A
b)	Interview local personnel?	yes no N/A
c)	Site visit and inspection?	yes no N/A
d)	Excavation areas marked in the field?	yes no N/A
e)	Utilities located in the field?	yes no N/A
f)	Located utilities marked/added to site maps?	yes no N/A
g)	Client contact notified Name _____ Telephone: _____ Date: _____	yes no N/A
g)	State One-Call agency called? Caller: _____ Ticket Number: _____ Date: _____	yes no N/A
h)	Geophysical survey performed? Survey performed by: _____ Method: _____ Date: _____	yes no N/A
i)	Hand excavation performed (with concurrent use of utility detection device)? Completed by: _____ Total depth: _____ feet Date: _____	yes no N/A
j)	Trench/excavation probed? Probing completed by: _____ Depth/frequency: _____ Date: _____	yes no N/A

2. Overhead Utilities — Present Absent

a)	Determination of nominal voltage	yes no N/A
b)	Marked on site maps	yes no N/A
c)	Necessary to lockout/insulate/re-route	yes no N/A
d)	Document procedures used to lockout/insulate/re-route	yes no N/A
e)	Minimum acceptable clearance (SOP Section 5.2): _____	

3. Notes:

_____

_____

_____

Approval:

_____ _____

Site Manager/Field Operations Leader Date

c: PM/Project File
Program File

ATTACHMENT 4
OSHA LETTER OF INTERPRETATION

Mr. Joseph Caldwell
Consultant
Governmental Liaison
Pipeline Safety Regulations
211 Wilson Boulevard
Suite 700
Arlington, Virginia 22201

Re: Use of hydro-vacuum or non-conductive hand tools to locate underground utilities.

Dear Mr. Caldwell:

In a letter dated July 7, 2003, we responded to your inquiry of September 18, 2002, regarding the use of hydro-vacuum equipment to locate underground utilities by excavation. After our letter to you was posted on the OSHA website, we received numerous inquiries that make it apparent that aspects of our July 7 letter are being misunderstood. In addition, a number of industry stakeholders, including the National Utility Contractors Association (NUCA), have provided new information regarding equipment that is available for this work.

To clarify these issues, we are withdrawing our July 7 letter and issuing this replacement response to your inquiry.

Question: *Section 1926.651 contains several requirements that relate to the safety of employees engaged in excavation work. Specifically, paragraphs (b)(2) and (b)(3) relate in part to the safety of the means used to locate underground utility installations that, if damaged during an uncovering operation, could pose serious hazards to employees.*

Under these provisions, what constitutes an acceptable method of uncovering underground utility lines, and further, would the use of hydro-vacuum excavation be acceptable under the standard?

Answer

Background

Two sections of 29 CFR 1926 Subpart P (Excavations), 1926.651(Specific excavation requirements), govern methods for uncovering underground utility installations. Specifically, paragraph (b)(2) states:

> When utility companies or owners cannot respond to a request to locate underground utility installations within 24 hours * * * or cannot establish the exact location of these installations, the employer may proceed, provided the employer does so with caution, and provided detection equipment or other acceptable means to locate utility installations are used. (emphasis added).

Paragraph (b)(3) provides:

ATTACHMENT 4 (Continued)

> When excavation operations approach the estimated location of underground installations, the exact location of the installations shall be determined by safe and acceptable means. (emphasis added).

Therefore, "acceptable means" must be used where the location of the underground utilities have not been identified by the utility companies and detection equipment is not used.

Subpart P does not contain a definition of either "other acceptable means" or "safe and acceptable means." The preambles to both the proposed rule and the final rule discussed the rationale behind the wording at issue. For example, the preamble to the proposed rule, 52 Fed. Reg. 12301 (April 15, 1987), noted that a 1972 version of this standard contained language that specified "careful probing or hand digging" as the means to uncover utilities. The preamble then noted that an amendment to the 1972 standard later deleted that language "to allow other, *equally effective means* of locating such installations." The preamble continued that in the 1987 proposed rule, OSHA again proposed using language in section (b)(3) that would provide another example of an acceptable method of uncovering utilities that could be used *where the utilities have not been marked and detection equipment is not being used* – "probing with hand-held tools." This method was rejected in the final version of 29 CFR 1926. As OSHA explained in the preamble to the final rule, 54 Fed. Reg. 45916 (October 31, 1989):

> OSHA received two comments * * * and input from ACCSH [OSHA's Advisory Committee on Construction Safety and Health] * * * on this provision. All commenters recommended dropping 'such as probing with hand-held tools' from the proposed provision, because this could create a hazard to employees by damaging the installation or its insulation.

In other words, the commenters objected to the use of hand tools being used unless detection equipment was used in conjunction with them. OSHA then concluded its discussion relative to this provision by agreeing with the commentators and ultimately not including any examples of "acceptable means" in the final provision.

Non-conductive hand tools are permitted

This raises the question of whether the standard permits the use of hand tools alone -- without also using detection equipment. NUCA and other industry stakeholders have recently informed us that non-conductive hand tools that are appropriate to be used to locate underground utilities are now commonly available.

Such tools, such as a "shooter" (which has a non-conductive handle and a snub nose) and non-conductive or insulated probes were not discussed in the rulemaking. Since they were not considered at that time, they were not part of the class of equipment that was thought to be unsafe for this purpose. Therefore, we conclude that the use of these types of hand tools, when used with appropriate caution, is an "acceptable means" for locating underground utilities.

ATTACHMENT 4 (Continued)

Hydro-vacuum excavation

It is our understanding that some hydro-vacuum excavation equipment can be adjusted to use a minimum amount of water and suction pressure. When appropriately adjusted so that the equipment will not damage underground utilities (especially utilities that are particularly vulnerable to damage, such as electrical lines), use of such equipment would be considered a "acceptable means" of locating underground utilities. However, if the equipment cannot be sufficiently adjusted, then this method would not be acceptable under the standard.

Other technologies

We are not suggesting that these are the only devices that would be "acceptable means" under the standard. Industry stakeholders have informed us that there are other types of special excavation equipment designed for safely locating utilities as well.

We apologize for any confusion our July 7 letter may have caused. If you have further concerns or questions, please feel free to contact us again by fax at: U.S. Department of Labor, OSHA, Directorate of Construction, Office of Construction Standards and Compliance Assistance, fax # 202-693-1689. You can also contact us by mail at the above office, Room N3468, 200 Constitution Avenue, N.W., Washington, D.C. 20210, although there will be a delay in our receiving correspondence by mail.

Sincerely,

Russell B. Swanson, Director
Directorate of Construction

NOTE: OSHA requirements are set by statute, standards and regulations. Our interpretation letters explain these requirements and how they apply to particular circumstances, but they cannot create additional employer obligations. This letter constitutes OSHA=s interpretation of the requirements discussed. Note that our enforcement guidance may be affected by changes to OSHA rules. Also, from time to time we update our guidance in response to new information. To keep apprised of such developments, you can consult OSHA's website at http://www.osha.gov.

TETRA TECH	STANDARD OPERATING PROCEDURES	Number SA-1.3	Page 1 of 31
		Effective Date 04/072008	Revision 9
		Applicability Tetra Tech NUS, Inc.	
		Prepared Earth Sciences Department	
Subject SOIL SAMPLING		Approved Tom Johnston T E Johnston	

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1.0 PURPOSE

This Standard Operating Procedure (SOP) describes the procedures to be used to collect surface, near-surface, and subsurface soil samples. Additionally, it describes the methods for sampling of test pits and trenches to determine subsurface soil and rock conditions and for recovery of small-volume or bulk samples from pits.

2.0 SCOPE

This document applies to the collection of surface, near-surface, and subsurface soil samples exposed through hand digging, hand augering, drilling, or machine excavating at hazardous substance sites for laboratory testing, onsite visual examination, and onsite testing.

3.0 GLOSSARY

Composite Sample - A composite sample is a combination of more than one grab sample from various locations and/or depths and times that is homogenized and treated as one sample. This type of sample is usually collected when determination of an average waste concentration for a specific area is required. Composite samples shall not be collected for volatile organics analysis.

Confined Space - As stipulated in 29 Code of Federal Regulations (CFR) 1910.146, a confined space means a space that: (1) is large enough and so configured that an employee can bodily enter and perform assigned work; (2) has limited or restricted means for entry or exit (e.g., tanks, vessels, silos, storage bins, hoppers, vaults, pits, and excavations); and (3) is not designed for continuous employee occupancy. TtNUS considers all confined space as permit-required confined spaces.

Grab Sample - One sample collected at one location and at one specific time.

Hand Auger - A sampling device used to extract soil from the ground.

Representativeness – A qualitative description of the degree to which an individual sample accurately reflects population characteristics or parameter variations at a sampling point. It is therefore an important characteristic not only of assessment and quantification of environmental threats posed by the site, but also for providing information for engineering design and construction. Proper sample location selection and proper sample collection methods are important to ensure that a truly representative sample has been collected.

Sample for Non-Volatile Analyses - Includes all chemical parameters other than volatile organics (e.g., semivolatiles, pesticides/PCBs, metals, etc.) and those engineering parameters that do not require undisturbed soil for their analysis.

Split-Barrel Sampler - A steel tube, split in half lengthwise, with the halves held together by threaded collars at either end of the tube. Also called a split-spoon sampler, this device can be driven into resistant materials using a drive weight mounted in the drilling string. A standard split-barrel sampler is typically available in two common lengths, providing either 20-inch or 26-inch longitudinal clearance for obtaining 18-inch or 24-inch-long samples, respectively. These split-barrel samplers commonly range in size from 2 to 3.5 inches OD. The larger sizes are commonly used when a larger volume of sample material is required (see Attachment B).

Test Pit and Trench - Open, shallow excavations, typically rectangular (if a test pit) or longitudinal (if a trench), excavated to determine shallow subsurface conditions for engineering, geological, and soil chemistry exploration and/or sampling purposes. These pits are excavated manually or by machine (e.g., backhoe, clamshell, trencher, excavator, or bulldozer).

Thin-Walled Tube Sampler - A thin-walled metal tube (also called a Shelby tube) used to recover relatively undisturbed soil samples. These tubes are available in various sizes, ranging from 2 to 5 inches outside diameter (OD) and from 18 to 54 inches in length.

4.0 RESPONSIBILITIES AND PERSONNEL QUALIFICATIONS

Project Manager - The Project Manager is responsible for determining the sampling objectives, selecting proposed sampling locations, and selecting field procedures used in the collection of soil samples. Additionally, in consultation with other project personnel (geologist, hydrogeologist, etc.), the Project Manager establishes the need for test pits or trenches and determines their approximate locations and dimensions.

Site Safety Officer (SSO) - The SSO (or a qualified designee) is responsible for providing the technical support necessary to implement the project Health and Safety Plan. This will include (but not be limited to) performing air quality monitoring during sampling, boring, and excavation activities and to ensure that workers and offsite (downwind) individuals are not exposed to hazardous levels of airborne contaminants. The SSO/designee may also be required to advise the FOL on other safety-related matters regarding boring, excavation, and sampling, such as mitigative measures to address potential hazards from unstable trench walls, puncturing of drums or other hazardous objects, etc.

Field Operations Leader (FOL) - This individual is primarily responsible for the execution of the planning document containing the Sampling and Analysis Plan (SAP). This is accomplished through management of a field sampling team for the proper acquisition of samples. He or she is responsible for the supervision of onsite analyses; ensuring proper instrument calibration, care, and maintenance; sample collection and handling; the completion and accuracy of all field documentation; and making sure that custody of all samples obtained is maintained according to proper procedures. When appropriate and as directed by the FOL, such responsibilities may be performed by other qualified personnel (e.g., field technicians) where credentials and time permit. The FOL is responsible for finalizing the locations for collection of surface, near-surface, and subsurface (hand and machine borings, test pits/trenches) soil samples. He/she is ultimately responsible for the sampling and backfilling of boreholes, test pits, and trenches and for adherence to Occupational Safety and Health Administration (OSHA) regulations during these operations through self acquisition or through the management of a field team of samplers.

Project Geologist/Sampler - The project geologist/sampler is responsible for the proper acquisition of samples in accordance with this SOP and/or other project-specific documents. In addition, this individual is responsible for the completion of all required paperwork (e.g., sample log sheets, field notebook, boring logs, test pit logs, container labels, custody seals, and chain-of-custody forms) associated with the collection of those samples.

Competent Person - A Competent Person, as defined in 29 CFR 1929.650 of Subpart P - Excavations, means one who is capable of identifying existing and predictable hazards in the surroundings, or working conditions that are unsanitary, hazardous, or dangerous to employees, and who has authorization to take prompt corrective measures to eliminate them.

General personnel qualifications for groundwater sample collection and onsite water quality testing include the following:

- Occupational Safety and Health Administration (OSHA) 40-hour and applicable refresher training.
- Capability of performing field work under the expected physical and environmental (i.e., weather) conditions.

- Familiarity with appropriate procedures for sample documentation, handling, packaging, and shipping.

5.0 HEALTH AND SAFETY

Health and safety precautions are identified for individual sample collection procedures throughout this SOP. In addition to those precautions, the following general hazards may be incurred during sampling activities:

- Knee injuries from kneeling on hard or uneven surfaces
- Slips, trips, and falls
- Cuts and lacerations
- Traffic hazards associated with sampling in parking areas, along roadways and highways.

Methods of avoiding these hazards are provided below.

Knee injuries – If kneeling is required during soil sampling, this could result in knee injuries from stones/foreign objects and general damage due to stress on the joints. To minimize this hazard:

- Clear any foreign objects from the work area.
- Wear hard-sided knee pads.
- Stretch ligaments, tendons and muscles before, during and after. Take breaks as frequently as necessary.
- Report pre-existing conditions to the SSO if you feel this activity will aggravate an existing condition.

Slips, Trips, and Falls – These hazards exist while traversing varying terrains carrying equipment to sample locations. To minimize these hazards:

- Pre-survey sampling locations. Eliminate, barricade, or otherwise mark physical hazards leading to the locations.
- Carry small loads that do not restrict the field of vision.
- Travel the safest and clearest route (not necessarily the shortest).

Cuts and Lacerations - To prevent cuts and lacerations associated with soil sampling, the following provisions are required:

- Always cut away from yourself and others when cutting tubing or rope. This will prevent injury to yourself and others if the knife slips.
- Do not place items to be cut in your hand or on your knee.
- Change blades as necessary to maintain a sharp cutting edge. Many accidents result from struggling with dull cutting attachments.

- Whenever practical, wear cut-resistant gloves (e.g., leather or heavy cotton work gloves) at least on the hand not using the knife.
- Keep cutting surfaces clean and smooth.
- Secure items to be cut – do not hold them against the opposing hand, a leg, or other body part.
- When transporting glassware, keep it in a hard-sided container such as a cooler so that if there is a fall, you will be less likely to get cut by broken glass.
- DO NOT throw broken sample jars or glass ampoules into garbage bags. Place broken glass and glass ampoules in hard-sided containers such as a cardboard box or directly into a dumpster. DO NOT reach into garbage bags to retrieve any item accidentally thrown away. Empty the contents onto a flat surface to avoid punctures and lacerations from reaching where you cannot see.

Vehicular and Foot Traffic Hazards – When sampling along the roadway or near traffic patterns, follow the following precautions:

- Motorists may be distracted by onsite activities – ASSUME THEY DO NOT SEE YOU OR MEMBERS OF YOUR FIELD CREW.
- DO NOT place obstructions (such as vehicles) along the sides of the road that may cause site personnel to move into the flow of traffic to avoid your activities or equipment or that will create a blind spot.
- **Provide a required free space of travel**. Maintain at least 6 feet of space between you and moving traffic. Where this is not possible, use flaggers and/or signs to warn oncoming traffic of activities near or within the travel lanes.
- Face Traffic. Whenever feasible, if you must move within the 6 feet of the required free space or into traffic, attempt to face moving traffic at all times. Always leave yourself an escape route.
- Wear high-visibility vests to increase visual recognition by motorists.
- Do not rely on the vehicle operator's visibility, judgment, or ability. Make eye contact with the driver. Carefully and deliberately use hand signals so they will not startle or confuse motorists or be mistaken for a flagger's direction before moving into traffic.
- Your movements may startle a motorist and cause an accident, so move deliberately. Do not make sudden movements that might confuse a motorist.

6.0 PROCEDURES

The following procedures address surface and subsurface sampling.

CAUTION

Each situation must be evaluated individually to determine the applicability and necessity for obtaining a utility clearance ticket/dig permit. Common sense dictates, prior to digging or boring with power equipment, no matter what the depth, or digging by hand in a manner that could damage unprotected underground utilities, that a dig permit is required. See SOP HS-1.0, Utility Locating and Excavation Clearance, for additional clarification. If you do not know or are unsure as to whether a ticket is necessary – **Get the Ticket**.

6.1 Overview

Soil sampling is an important adjunct to groundwater monitoring. Sampling of the soil horizons above the groundwater table can detect contaminants before they migrate to the water table, and can establish the amount of contamination absorbed or adsorbed on aquifer solids that have the potential of contributing to groundwater contamination.

Soil types can vary considerably on a hazardous waste site. These variations, along with vegetation, can affect the rate of contaminant migration through the soil. It is important, therefore, that a detailed record be maintained during sampling operations, particularly noting sampling locations, depths, and such characteristics as grain size, color, and odor. Subsurface conditions are often stable on a daily basis and may demonstrate only slight seasonal variation especially with respect to temperature, available oxygen and light penetration. Changes in any of these conditions can radically alter the rate of chemical reactions or the associated microbiological community, thus further altering specific site conditions. Certain vegetation species can create degradation products that can alter contaminant concentrations in soil. This is why vegetation types and extent of degradation of this foliage must be recorded. To prevent degradation, samples must be kept at their at-depth temperature or lower, protected from direct light, sealed tightly in approved glass containers, and be analyzed as soon as possible after collection. In addition, to the extent possible, vegetation should be removed from the sample.

The physical properties of the soil, its grain size, cohesiveness, associated moisture, and such factors as depth to bedrock and water table, will limit the depth from which samples can be collected and the method required to collect them. It is the intent of this document to present the most commonly employed soil sampling methods used at hazardous waste sites.

6.2 Soil Sample Collection

6.2.1 Procedure for Preserving and Collecting Soil Samples for Volatile Organic Compound Analysis

Samples collected using traditional methods such as collection in a jar with no preservation have been known to yield non-representative samples due to loss of volatile organic compounds (VOCs). To prevent such losses, preservation of samples with methanol or sodium bisulfate may be used to minimize volatilization and biodegradation. This preservation may be performed either in the field or laboratory, depending on the sampling methodology employed. Because of the large number of sampling methods and associated equipment required, careful coordination between field and laboratory personnel is needed.

Soil samples to be preserved by the laboratory are currently being collected using Method SW-846, 5035. For samples preserved in the field, laboratories are currently performing low-level analyses (sodium bisulfate preservation) and high- to medium-level analyses (methanol preservation) depending on the needs of the end user.

The following procedures outline the necessary steps for collecting soil samples to be preserved at the laboratory, and for collecting soil samples to be preserved in the field with methanol or sodium bisulfate.

6.2.1.1 Soil Samples to be Preserved at the Laboratory

Soil samples collected for volatile organic analysis that are to be preserved at the laboratory shall be obtained using a hermetically sealed sample vial such as an EnCore™ sampler. Each sample shall be

obtained using a reusable sampling handle (T-handle) that can be provided with the EnCore™ sampler when requested and purchased. Collect the sample in the following manner for each EnCore™ sampler:

1. Scene Safety - Evaluate the area where sampling will occur. Ensure that the area is safe from physical, chemical, and natural hazards. Clear or barricade those hazards that have been identified.

2. Wear the appropriate personal protective equipment (PPE). This will include, at a minimum, safety glasses and nitrile surgeon's gloves. If you must kneel on the ground or place equipment on the surface being sampled, cover the ground surface with plastic to minimize surface contamination of your equipment and clothing. Wear knee pads to protect your knees from kneeling on hard or uneven surfaces.

3. Load the Encore™ sampler into the T-handle with the plunger fully depressed.

4. Expose the area to be sampled using a hand trowel or similar device to remove surface debris.

5. Press the T-handle against the freshly exposed soil surface, forcing soil into the sampler. The plunger will be forced upward as the cavity fills with soil.

6. When the sampler is full, rotate the plunger and lock it into place. If the plunger does not lock, the sampler is not full. This method ensures there is no headspace. Soft soil may require several plunges or forcing soil against a hard surface such as a sample trowel to ensure that headspace is eliminated.

7. Use a paper towel to remove soil from the side of the sampler so a tight seal can be made between the sample cap and the rubber O-ring.

8. With soil slightly piled above the rim of the sampler, force the cap on until the catches hook the side of the sampler.

9. Remove any surface soil from the outside of the sampler and place in the foil bag provided with the sampler. Good work hygiene practices and diligent decontamination procedures prevents the spread of contamination even on the outside of the containers.

10. Label the bag with appropriate information in accordance with SOP SA-6.3.

11. Place the full sampler inside a lined cooler with ice and cool to 4 °C ± 2 °C. Make sure any required trip blanks and temperature blanks are also in the cooler. Secure custody of the cooler in accordance with SOP SA-6.3.

12. Typically, collect three Encore™ samplers at each location. Consult the SAP or laboratory to determine the required number of Encore™ samplers to be collected.

13. The T-handle shall be decontaminated before moving to the next interval or location using a soap and water wash and rinse, and where applicable, the selected solvent as defined in the project planning documents.

Using this type of sampling device eliminates the need for field preservation and the shipping restrictions associated with preservatives. A complete set of instructions is included with each Encore™ sampler.

After the Encore™samples are collected, they should be placed on ice immediately and delivered to the laboratory within 48 hours (following the chain-of-custody and documentation procedures outlined in SOP SA-6.1). Samples must be preserved by the laboratory within 48 hours of sample collection.

6.2.1.2 Soil Samples to be Preserved in the Field

Soil samples preserved in the field may be prepared for analyses using both the low-level (sodium bisulfate preservation) and high- to medium-level (methanol preservation) methods.

Safety Reminder

When using chemicals in the field to preserve samples, the FOL and/or SSO must ensure that Materials Safety Data Sheets (MSDSs) have been provided with the chemicals to be used. They also must ensure that these chemicals have been added to the Chemical Inventory List contained within Section 5.0, Hazard Communication, of your Health and Safety Guidance Manual (HSGM). Lastly, but most importantly, the FOL and/or SSO must review the hazards with personnel using these chemicals and ensure that provisions are available for recommended PPE and emergency measures (e.g., eyewash, etc.).

Methanol Preservation (High to Medium Level):

Bottles may be pre-spiked with methanol in the laboratory or prepared in the field. Soil samples to be preserved in the field with methanol shall utilize 40 to 60 mL glass vials with septum-lined lids. Each sample bottle shall be filled with 25 mL of demonstrated analyte-free purge-and-trap grade methanol. The preferred method for adding methanol to the sample bottle is by removing the lid and using a pipette or scaled syringe to add the methanol directly to the bottle.

CAUTION

NEVER attempt to pipette by mouth

In situations where personnel are required to spike the septum using a hypodermic needle, the following provisions for handling sharps must be in place:

- Training of personnel regarding methods for handling of sharps
- Hard-sided containers for the disposal of sharps
- Provisions for treatment in cases where persons have received a puncture wound

Soil shall be collected with the use of a decontaminated (or disposable), small-diameter coring device such as a disposable tube/plunger-type syringe with the tip cut off. The outside diameter of the coring device must be smaller than the inside diameter of the sample bottle neck.

A small electronic balance or manual scale will be necessary for measuring the volume of soil to be added to the methanol-preserved sample bottle. Calibration of the scale shall be performed prior to use and intermittently throughout the day according to the manufacturer's requirements.

The sample should be collected as follows:

1. Weigh the unused syringe and plunger to the nearest 0.01 gram.
2. Pull the plunger back and insert the syringe into the soil to be sampled.
3. Collect 8 to 12 grams of soil by pushing the syringe barrel into the soil.
4. Weigh the sample and adjust until obtaining the required amount of sample.

5. Record the sample weight to the nearest 0.01 gram in the field logbook and/or on the sample log sheet.

6. Extrude the weighed soil sample into the methanol-preserved sample bottle taking care not to contact the sample container with the syringe.

7. If dirty, wipe soil particles from the threads of the bottle and cap. Cap the bottle tightly.

8. After capping the bottle, swirl the sample (do not shake) in the methanol and break up the soil such that all of the soil is covered with methanol.

9. Place the sample on ice immediately and prepare for shipment to the laboratory as described in SOP SA-6.1.

Sodium Bisulfate Preservation (Low Level):

> ***CAUTION***
> Care should be taken when adding the soil to the sodium bisulfate solution. A chemical reaction of soil containing carbonates (limestone) may cause the sample to effervesce or the vial to possibly explode. To avoid this hazard or hazards of this type, a small sample aliquot should be subjected to the sample preservative. If it effervesces in an open air environment, utilize an alternative method such as Encore™ or 2-ounce jar.

Bottles may be prepared in the laboratory or in the field with sodium bisulfate solution. Samples to be preserved in the field using the sodium bisulfate method are to be prepared and collected as follows:

1. Add 1 gram of sodium bisulfate to 5 mL of laboratory-grade deionized water in a 40 to 60 mL glass vial with septum-lined lid.

2. Collect the soil sample and record the sample weight to the nearest 0.01 gram in the field logbook or on the sample log sheet as described for methanol preservation

3. Add the weighed sample to the sample vial.

4. Collect duplicate samples using the methanol preservation method on a one-for-one sample basis because it is necessary for the laboratory to perform both low-level and medium-level analyses.

5. Place the samples on ice immediately and prepare for shipment to the laboratory as described in SOP SA-6.1.

> ***NOTE***
> If lower detection limits are necessary, an option to field preserving with sodium bisulfate may be to collect EnCore™ samplers at a given sample location. Consult the planning documents to determine whether this is required. If it is, collect samples in accordance with the Encore™ sampling procedure above and then send all samplers to the laboratory to perform the required preservation and analyses.

6.2.2 Procedure for Collecting Soil Samples for Non-Volatile Analyses

Samples collected for non-volatile analyses may be collected as either grab or composite samples as follows:

1. With a stainless steel trowel or other approved tool, transfer a portion of soil to be sampled to a stainless steel bowl or disposable inert plastic tray.

2. Remove roots, vegetation, sticks, and stones larger than the size of a green pea.

3. Thoroughly mix the soil in the bowl or tray to obtain as uniform a texture and color as practicable. The soil type, moisture content, amount of vegetation, and other factors may affect the amount of time required to obtain a properly mixed sample. In some cases, it may be impossible to obtain a uniform sample appearance. Use the field logbook to describe any significant difficulties encountered in obtaining a uniform mixture.

4. Transfer the mixed soil to the appropriate sample containers and close the containers.

5. Label the sample containers in accordance with SOP SA-6.3.

6. Place the containers in a cooler of ice as soon after collection as possible.

7. Prepare the sample shipment and ship the samples in accordance with SOP SA-6.1.

> ***NOTE***
> Cooling may not be required for some samples depending on the scheduled analyses. Consult the planning documents if in doubt regarding correct sample preservation conditions. When in doubt – Cool to 4°C.

> ***NOTE***
> Head space is permitted in soil sample containers for non-volatile analyses to allow for sample expansion.

6.2.3 Procedure for Collecting Undisturbed Soil Samples

> ***NOTE***
> Use of thin-walled undisturbed tube samplers is restricted by the consistency of the soil to be sampled. Often, very loose and/or wet samples cannot be retrieved by the samplers, and soil with a consistency in excess of very stiff cannot be penetrated by the sampler. Devices such as Dennison or Pitcher core samplers can be used to obtain undisturbed samples of stiff soil. Using these devices normally increases sampling costs, and therefore their use should be weighed against the need for acquiring an undisturbed sample. These devices are not discussed in this SOP because they are not commonly used.

When it is necessary to acquire undisturbed samples of soil for purposes of engineering parameter analysis (e.g., permeability), a thin-walled, seamless tube sampler (Shelby tube) shall be employed using the following collection procedure:

1. In preparation for sampling utilizing a drill rig, field personnel must complete the following activities:

   - Ensure that all subsurface drilling activities are preceded by a utility clearance for the area to be investigated. This includes activities described in SOP HS-1.0, Utility Location and Excavation Clearance, as well as any location-specific procedures that may apply.

REMEMBER

If you are digging near a marked utility (within the diameter of an underground utility that has been marked plus 18 inches), you must first locate the utility through vacuum extraction or hand digging to ensure that your activities will not damage the utility.

- Complete an Equipment Inspection Checklist for the drill rig or direct-push technology (DPT) rig. This checklist will be provided in the HASP.
- Review the Safe Work Permit prior to conducting the activity.
- Review the activity to be conducted.

2. Remove all surface debris (e.g., vegetation, roots, twigs, etc.) from the specific sampling location and drill and/or clean out the borehole to the desired sampling depth. Be careful to minimize potential disturbance of the material to be sampled. In saturated material, withdraw the drill bit slowly to prevent loosening of the soil around the borehole and to maintain the water level in the hole at or above groundwater level.

CAUTION

The use of bottom-discharge bits or jetting through an open-tube sampler to clean out the borehole shall not be allowed. Only the use of side-discharge bits is permitted.

3. Determine whether a stationary piston-type sampler is required to limit sample disturbance and aid in retaining the sample. Either the hydraulically operated or control rod activated-type of stationary piston sampler may be used.

4. Prior to inserting the tube sampler into the borehole, check to ensure that the sampler head contains a check valve. The check valve is necessary to keep water in the rods from pushing the sample out the tube sampler during sample withdrawal. In addition, the check valve maintains a positive suction within the tube to help retain the sample.

5. A stainless steel tube sampler is typically used to minimize chemical reaction between the sample and the sampling tube.

6. With the sampling tube resting on the bottom of the hole and the water level in the boring at groundwater level or above, push the tube into the soil with a continuous and rapid motion, without impacting or twisting. If the soil is too hard to penetrate by pushing alone, careful hammering may be used by minimizing drop distance (tapping) of the hammer. Before pulling the tube, turn it at least one revolution to shear the sample off at the bottom. In no case shall the tube be pushed farther than the length provided for the soil sample. Allow about 3 inches in the tube for cuttings and sludge.

7. Upon removal of the sampling tube from the hole, measure the length of sample in the tube and also the length penetrated.

8. Remove disturbed material in the upper end of the tube and measure the length of sample again.

9. After removing at least 1 inch of soil from the lower end, place enough packing material (clean inert material such as paper or cloth) tightly in each end of the Shelby tube and then pour melted wax into each end to make at least a ½-inch wax plug and then add more packing material to fill the voids at both ends.

10. Place plastic caps on the ends, tape the caps in place, and dip the ends in wax to prevent loss of soil.

11. Affix label(s) to the tube as required and record sample number, depth, penetration, and recovery length on the label.

12. Mark the "up" direction on the side and upper end of the tube with indelible ink.

13. Complete a chain-of-custody form (see SOP SA-6.3) and other required forms (including Attachment A of this SOP).

14. Ship samples protected with suitable resilient packing material to reduce shock, vibration, and disturbance.

CAUTION

To preserve sample integrity do not allow tubes to freeze, and store the samples vertically with the same orientation they had in the ground, (i.e., top of sample is up) in a cool place out of the sun at all times.

CAUTION

A primary concern in the preparation of the wax plugs is the potential for the heat source and melted wax to cause a fire and/or burns. Follow the directions below to prevent injury or fire.

Electrical Heating

Using hot plates to melt the wax is acceptable. In an outdoor setting, make sure a Ground Fault Circuit Interrupter (GFCI) is employed within the electrical circuit. If a portable generator is used, ensure that the generator is an adequate distance from the sampling operation (at least 50 feet). Ensure that the extension cord is rated for the intended load and for outdoor use and is free from recognizable damage. Ensure flammable preservatives are not employed or stored near the hot plate. Although a Hot Work Permit is not required, scene safety evaluation by site personnel of the above elements is. As always, if a fire potential exists, the provisions for extinguishing must be immediately accessible as well as any provisions for first aid measures.

Open Flame

If an open flame is used, the following provisions are necessary:

- Complete a Hot Work Permit and any local permit required for elevated temperature applications. The Hot Work Permit, provided in your HASP, will aid the FOL and/or the SSO in ensuring that fire protection provisions (extinguishers, fire watches, etc.) are in place as well as ensuring that local requirements have been addressed.

- Ensure that water is available to address any wax splashes or contact. If possible, immerse the contacted area. Where this is not possible, run water over the area and apply cold compresses. The need for medical attention or first aid shall be determined on site under the direction of the SSO.

6.3 Surface Soil Sampling

The simplest, most direct method of collecting surface soil samples for subsequent analysis is by use of a stainless steel shovel, hand auger, soil corer, or stainless steel or disposable plastic trowel.

> ***NOTE***
>
> Multiple depth intervals are used to describe surface soil. Sometimes surface soil is defined as soil from 0 to 2 inches below ground surface (bgs), and sometimes it is defined as soil from other depths such as 0 to 2 feet bgs. Ensure that the definition of surface soil depth is clear before collecting surface soil samples.
>
> For the purposes of instruction, the terms "surface soil" and "near-surface soil" are used in this SOP as follows:
>
> - Surface soil - 0 to 6 inches bgs
> - Near-surface soil - 6 to 18 inches bgs
>
> If these intervals are defined differently in the planning documents, substitute the appropriate depth ranges.

In general, the following equipment is necessary for obtaining surface soil samples:

- Stainless steel or pre-cleaned disposable trowel.
- Stainless steel hand auger, soil corer, or shovel.
- Real-time air monitoring instrument (e.g., PID, FID) as directed in project planning document.
- Required PPE.
  - Nitrile surgeon's or latex gloves may be used, layered as necessary.
  - Safety glasses
  - Other – Items identified on the Safe Work Permit may be required based on location-specific requirements such as hearing protection, steel-toed work boots, and a hard hat when working near a drill rig. These provisions will be listed in the HASP or directed by the FOL and/or SSO.

> ***Safety Reminder***
>
> The use of latex products may elicit an allergic reaction in some people. Should this occur, remove the latex gloves, treat for an allergic reaction, and seek medical attention as necessary.

- Required paperwork (see SOP SA-6.3 and Attachment A of this SOP)
- Required decontamination equipment
- Required sample container(s)
- Wooden stakes or pin flags

- Sealable polyethylene bags (e.g., Ziploc® baggies)
- Heavy duty cooler
- Ice
- Chain-of-custody records and custody seals

When acquiring surface soil samples, use the following procedure:

1. Place padding or use knee pads when kneeling near the sample location. If necessary, place plastic sheeting to provide a clean surface for sample equipment to avoid possible cross- contamination.
2. Carefully remove vegetation, roots, twigs, litter, etc. to expose an adequate soil surface area to accommodate sample volume requirements.
3. Using a precleaned syringe or EnCore™ samplers, follow the procedure in Section 6.2.1 for collecting surface soil samples for volatile analysis. Surface soil samples for volatile organic analysis should be collected deeper than 6 inches bgs because shallower material has usually lost most of the volatiles through evaporation. Ensure that the appropriate surface soil depth is being analyzed in accordance with the planning document.
4. Using decontaminated sampling tools, thoroughly mix in place a sufficient amount of soil to fill the remaining sample containers. See Section 6.5 of this procedure for hand auger instruction, as needed.
5. Transfer the sample into those containers utilizing a stainless steel trowel.
6. Cap and securely tighten all sample containers.
7. Affix a sample label to each container. Be sure to fill out each label carefully and clearly, addressing all the categories described in SOP SA-6.3.
8. Proceed with the handling and processing of each sample container as described in SOP SA-6.2.
9. Site restoration – Whenever removing sample materials, always restore the surface. It is our intent to leave the area better than we found it. Do NOT create trip hazards in areas when pedestrian traffic may exist.

6.4 Near-Surface Soil Sampling

Collection of samples from near the surface (depth of 6 to 18 inches) can be accomplished with tools such as shovels, hand auger, soil corers, and stainless steel or pre-cleaned disposable trowels and the equipment listed under Section 6.5 of this procedure.

To obtain near-surface soil samples, the following protocol shall be used:

1. With a clean shovel, make a series of vertical cuts in the soil to the depth required to form a square approximately 1 foot by 1 foot.
2. Lever out the formed plug and scrape the bottom of the freshly dug hole with a decontaminated stainless steel or pre-cleaned disposable trowel to remove any loose soil.

3. Follow steps 1 through 9 of Section 6.3.

6.5 Subsurface Soil Sampling With a Hand Auger

A hand augering system generally consists of a variety of stainless steel bucket bits (approximately 6.5 inches long and 2, 2.75, 3.25, and 4 inches in diameter), series of extension rods (available in 2-, 3-, 4- and 5-inch lengths), and a T-handle connected to extension rods and to the auger bucket. A larger-diameter bucket bit is commonly used to bore a hole to the desired sampling depth and then it is withdrawn. The larger-diameter bit is then replaced with a smaller-diameter bit, lowered down the hole, and slowly turned into the soil to the completion depth (approximately 6 inches). The apparatus is then withdrawn and the soil sample collected.

The hand auger can be used in a wide variety of soil conditions. It can be used to sample soil either from the surface, or to depths in excess of 12 feet. However, the presence of subsurface rocks and landfill material and collapse of the borehole normally limit sampling depth.

To accomplish soil sampling using a hand augering system, the following equipment is required:

- Complete hand auger assembly (variety of bucket bit sizes)
- Stainless steel mixing bowls
- The equipment listed in Section 6.3
- Miscellaneous hand tools as required to assemble and disassemble the hand auger units

CAUTION

Potential hazards associated with hand augering include:

- Muscle strain and sprain due to over twisting and/or over compromising yourself.
- Equipment failure due to excessive stress on the T-handle or rods through twisting. Failure of any of these components will result in a sudden release and potential injury due to that failure.

As in all situations, any intrusive activities that could damage underground utilities shall be proceeded by a Dig/Excavation permit/ticket. Call the Utility Locating service in the area or your Project Health and Safety Officer for more information. When in doubt – **Get the Ticket!**

To obtain soil samples using a hand auger, use the following procedure:

1. Wearing designated PPE, attach a properly decontaminated bucket bit to a clean extension rod and attach the T-handle to the extension rod.
2. Clear the area to be sampled of any surface debris (vegetation, twigs, rocks, litter, etc.).
3. Twist the bucket into the ground while pushing vertically downward on the auger. The cutting shoes fill the bucket as it is advanced into the ground.
4. As the auger bucket fills with soil, periodically remove any unneeded soil.

5. Add rod extensions as necessary to extend the reach of the auger. Also, note (in a field notebook, boring log, and/or on a standardized data sheet) any changes in the color, texture or odor of the soil as a function of depth. The project-specific planning document (SAP, HASP, etc.) describe requirements for scanning the soil with a real-time air monitoring instrument (e.g., PID, FID, etc.) and recording the measurements.

6. After reaching the desired depth (e.g., the top of the interval to be sampled), slowly and carefully withdraw the apparatus from the borehole to prevent or minimize movement of soil from shallower intervals to the bottom of the hole.

7. Remove the soiled bucket bit from the rod extension and replace it with another properly decontaminated bucket bit. The bucket bit used for sampling is to be smaller in diameter than the bucket bit employed to initiate the borehole.

8. Carefully lower the apparatus down the borehole. Care must be taken to avoid scraping the borehole sides.

9. Slowly turn the apparatus until the bucket bit is advanced approximately 6 inches.

10. Discard the top of the core (approximately 1 inch), which represents any loose material collected by the bucket bit before penetrating the sample material.

11. Using a precleaned syringe or EnCore™ samplers, follow the procedure in Section 6.2.1 for collecting a soil sample for volatile compound analysis directly from the bucket bit.
12. Utilizing a properly decontaminated stainless steel trowel or dedicated disposable trowel, remove the remaining sample material from the bucket bit and place into a properly decontaminated stainless steel mixing bowl.

13. Homogenize the sample material as thoroughly as practicable then fill the remaining sample containers. Refer to Section 6.2.2.

14. Follow steps 4 through 7 listed in Section 6.3.

6.5.1 Sampling Using Stainless Steel Soil Corers

A soil corer is a stainless steel tube equipped with a cutting shoe and sample window in the side. The soil corer is advanced into the soil by applying downward pressure (body weight). The soil is unloaded by then forcing a ram towards the cutting shoe, which results in the discharge of the soil core through a window in the sleeve.

Use, application, and sample protocol is the same as for hand augering provided above, but without necessarily rotating the corer while advancing it.

SAFETY REMINDER

Hand augering and soil corer sampling can be physically demanding based on the type of geology and subsurface encumbrances encountered. Soil coring has some added hazards such the corer collapsing under your weight. To reduce the potential for muscle strain and damage, the following measures will be incorporated:

- Stretch and limber your muscles before heavy exertion. This hazard becomes more predominant in the early morning hours (prior to muscles becoming limber) and later in the day (as a result of fatigue).

- Job rotation – Share the duties so that repetitive actions do not result in fatigue and injury.

- Increase break frequencies as needed, especially as ambient conditions of heat and/or cold stress may dictate.

- Do not force the hand tools or use cheater pipes or similar devices to bypass an obstruction. Move to another location near the sampling point. Exerting additional forces on the sampling devices can result in damage and/or failure that could potentially injure someone in the immediate vicinity.

- Do not over compromise yourself when applying force to the soil corer or hand auger. If there is a sudden release, it could result in a fall or muscle injury due to strain.

6.6 Subsurface Soil Sampling with a Split-Barrel Sampler

A split-barrel (split-spoon) sampler consists of a heavy carbon steel or stainless steel sampling tube that can be split into two equal halves to reveal the soil sample (see Attachment B). A drive head is attached to the upper end of the tube and serves as a point of attachment for the drill rod. A removable tapered nosepiece/drive shoe attaches to the lower end of the tube and facilitates cutting. A basket-like sample retainer can be fitted to the lower end of the split tube to hold loose, dry soil samples in the tube when the sampler is removed from the drill hole. This split-barrel sampler is made to be attached to a drill rod and forced into the ground by means of a 140-pound or larger casing driver.

Safety Reminder

It is intended through the Equipment Inspection for Drill Rigs form provided in the HASP that the hammer and hemp rope, where applicable, associated with this activity will be inspected (no physical damage is obvious), properly attached to the hammer (suitable knots or sufficient mechanical devices), and is in overall good condition.

Split-barrel samplers are used to collect soil samples from a wide variety of soil types and from depths greater than those attainable with other soil sampling equipment.

The following equipment is used for obtaining split-barrel samples:

- Drilling equipment (provided by subcontractor).
- Split-barrel samplers (2-inch OD, 1-3/8-inch ID, either 20 inches or 26 inches long); Larger OD samplers are available if a larger volume of sample is needed.
- Drive weight assembly, 140-pound weight, driving head, and guide permitting free fall of 30 inches.
- Stainless steel mixing bowls.
- Equipment listed in Section 6.3.

The following steps shall be followed to obtain split-barrel samples (Steps 1 through 4 are typically performed by the drilling subcontractor):

1. Attach the split-barrel sampler to the sampling rods.

2. Lower the sampler into the borehole inside the hollow stem auger bits.

3. Advance the split-barrel sampler by hammering the length (typically 18 or 24 inches) of the split-barrel sampler into the soil using 140-pound or larger hammer.

4. When the desired depth is achieved, extract the drill rods and sampler from the augers and/or borehole.

5. Detach the sampler from the drill rods.

6. Place the sampler securely in a vise so it can be opened using pipe wrenches.

> ***CAUTION***
> Pipe wrenches are used to separate the split spoon into several components. The driller's helper should not apply excessive force through the use of cheater pipes or push or pull in the direction where, if the wrench slips, hands or fingers will be trapped against an immovable object.

7. Remove the drive head and nosepiece with the wrenches, and open the sampler to reveal the soil sample.

8. Immediately scan the sample core with a real-time air monitoring instrument (e.g., FID, PID, etc.) (as project-specific planning documents dictate). Carefully separate (or cut) the soil core, with a decontaminated stainless steel knife or trowel, at about 6-inch intervals while scanning the center of the core for elevated readings. Also scan stained soil, soil lenses, and anomalies (if present), and record readings.

9. If elevated vapor readings were observed, collect the sample scheduled for volatile analysis from the center of the core where elevated readings occurred. If no elevated readings where encountered, the sample material should be collected from the core's center (this area represents the least disturbed area with minimal atmospheric contact) (refer to Section 6.2.1).

10. Using the same trowel, remove remaining sample material from the split-barrel sampler (except for the small portion of disturbed soil usually found at the top of the core sample) and place the soil into a decontaminated stainless steel mixing bowl.

11. Homogenize the sample material as thoroughly as practicable then fill the remaining sample containers (refer to Section 6.2.2).

12. Follow steps 4 through 7 in Section 6.3.

6.7 Subsurface Soil Sampling Using Direct-Push Technology

Subsurface soil samples can be collected to depths of 40+ feet using DPT. DPT equipment, responsibilities, and procedures are described in SOP SA-2.5.

6.8 Excavation and Sampling of Test Pits and Trenches

6.8.1 Applicability

This subsection presents routine test pit or trench excavation techniques and specialized techniques that are applicable under certain conditions.

> ***CAUTION***
>
> During the excavation of trenches or pits at hazardous waste sites, several health and safety concerns arise from the method of excavation. No personnel shall enter any test pit or excavation over 4 feet deep except as a last resort, and then only under direct supervision of a Competent Person (as defined in 29 CFR 1929.650 of Subpart P - Excavations). Whenever possible, all required chemical and lithological samples should be collected using the excavator bucket or other remote sampling apparatus. If entrance is required, all test pits or excavations must be stabilized by bracing the pit sides using specifically designed wooden, steel, or aluminum support structures or through sloping and benching. Personnel entering the excavation may be exposed to toxic or explosive gases and oxygen-deficient environments; therefore, monitoring will be conducted by the Competent Person to determine if it is safe to enter. Any entry into a trench greater than 4 feet deep will constitute a Confined Space Entry and must be conducted in conformance with OSHA standard 29 CFR 1910.146. In all cases involving entry, substantial air monitoring, before entry, appropriate respiratory gear and protective clothing determination, and rescue provisions are mandatory. There must be at least three people present at the immediate site before entry by one of the field team members. This minimum number of people will increase based on the potential hazards or complexity of the work to be performed. The reader shall refer to OSHA regulations 29 CFR 1926.650, 29 CFR 1910.120, 29 CFR 1910.134, and 29 CFR 1910.146. High-hazard entries such as this will be supported by members of the Health Sciences Group professionally trained in these activities.

Excavations are generally not practical where a depth of more than about 15 to 20-feet is desired, and they are usually limited to a few feet below the water table. In some cases, a pumping system may be required to control water levels within the pit, providing that pumped water can be adequately stored or disposed. If soil data at depths greater than 15-feet are required, the data are usually obtained through test borings instead of test pits.

In addition, hazardous wastes may be brought to the surface by excavation equipment. This material, whether removed from the site or returned to the subsurface, must be properly handled according to any and all applicable federal, state, and local regulations.

6.8.2 Test Pit and Trench Excavation

Test pits or trench excavations are constructed with the intent that they will provide an open view of subsurface lithology and/or disposal conditions that a boring will not provide. These procedures describe the methods for excavating and logging test pits and trenches installed to determine subsurface soil and rock conditions. Test pit operations shall be logged and documented (see Attachment C).

Test pits and trenches may be excavated by hand or power equipment to permit detailed descriptions of the nature and contamination of the in-situ materials. The size of the excavation will depend primarily on the following:

- The purpose and extent of the exploration

- The space required for efficient excavation
- The chemicals of concern
- The economics and efficiency of available equipment

Test pits normally have a cross section that is 4 to 10 feet square; test trenches are usually 3 to 6 feet wide and may be extended for any length required to reveal conditions along a specific line. The following table provides guidelines for design consideration based on equipment efficiencies.

Equipment	Typical Widths, in Feet
Trenching machine	0.25 to 1.0
Backhoe/Track Hoe	2 to 6

The lateral limits of excavation of trenches and the position of test pits shall be carefully marked on area base maps. If precise positioning is required to indicate the location of highly hazardous materials, nearby utilities, or dangerous conditions, the limits of the excavation shall be surveyed. Also, if precise determination of the depth of buried materials is needed for design or environmental assessment purposes, the elevation of the ground surface at the test pit or trench location shall also be determined by survey. If the test pit/trench will not be surveyed immediately, it shall be backfilled and its position identified with stakes placed in the ground at the margin of the excavation for later surveying.

The construction of test pits and trenches shall be planned and designed in advance as much as possible. However, the following field conditions may necessitate revisions to the initial plans:

- Subsurface utilities
- Surface and subsurface encumbrances
- Vehicle and pedestrian traffic patterns
- Purpose for excavation (e.g., the excavation of potential ordnance items)

The final depth and construction method shall be collectively determined by the FOL and designated Competent Person. The actual layout of each test pit, temporary staging area, and spoils pile may further be predicated based on site conditions and wind direction at the time the test pit is excavated. Prior to excavation, the area may be surveyed by magnetometer or metal detector or other passive methods specified in SOP HS1.0, Utility Location and Excavation Clearance, to identify the presence of underground utilities or drums. Where possible, the excavator should be positioned upwind and preferably within an enclosed cab.

No personnel shall enter any test pit or excavation except as a last resort, and then only under direct supervision of a Competent Person. If entrance is required, OSHA requirements must be met (e.g., walls must be braced with wooden or steel braces, ladders must be placed for every 25 feet of lateral travel and extended 3 feet above ground surface). A temporary guard rail or vehicle stop must be placed along the surface of the hole before entry in situations where the excavation may be approached by traffic. Spoils will be stockpiled no closer than 2 feet from the sidewall of the excavation. The excavation equipment operator shall be careful not to undercut sidewalls and will, where necessary, bench back to increase stability. The top cover, when considered clean, will be placed separately from the subsurface materials to permit clean cover. It is emphasized that the project data needs should be structured such that required samples can be collected without requiring entrance into the excavation. For example,

samples of leachate, groundwater, or sidewall soil can be collected with telescoping poles or similar equipment.

Dewatering and watering may be required to ensure the stability of the side walls, to prevent the bottom of the pit from heaving, and to keep the excavation stable. This is an important consideration for excavations in cohesionless material below the groundwater table and for excavations left open greater than a day. Liquids removed as a result of dewatering operations must be handled as potentially contaminated materials. Procedures for the collection and disposal of such materials should be discussed in the site-specific project plans.

Where possible excavations and test pits shall be opened and closed within the same working day. Where this is not possible, the following engineering controls shall be put in place to control access:

- Trench covers/street plates
- Fences encompassing the entire excavation intended to control access
- Warning signs warning personnel of the hazards
- Amber flashing lights to demarcate boundaries of the excavation at night

Excavations left open will have emergency means to exit should someone accidentally enter.

6.8.3 Sampling in Test Pits and Trenches

6.8.3.1 General

Log test pits and trenches as they are excavated in accordance with the Test Pit Log presented in Attachment C. These records include plan and profile sketches of the test pit/trench showing materials encountered, their depth and distribution in the pit/trench, and sample locations. These records also include safety and sample screening information.

Entry of test pits by personnel is extremely dangerous, shall be avoided unless absolutely necessary, and can occur only after all applicable health and safety and OSHA requirements have been met as stated above. These provisions will be reiterated as appropriate in the project-specific HASP.

The final depth and type of samples obtained from each test pit will be determined at the time the test pit is excavated. Sufficient samples are usually obtained and analyzed to quantify contaminant distribution as a function of depth for each test pit. Additional samples of each waste phase and any fluids encountered in each test pit may also be collected.

In some cases, samples of soil may be extracted from the test pit for reasons other than waste sampling and chemical analysis, for instance, to obtain geotechnical information. Such information includes soil types, stratigraphy, strength, etc., and could therefore entail the collection of disturbed (grab or bulk) or relatively undisturbed (hand-carved or pushed/driven) samples that can be tested for geotechnical properties. The purposes of such explorations are very similar to those of shallow exploratory or test borings, but often test pits offer a faster, more cost-effective method of sampling than installing borings.

6.8.3.2 Sampling Equipment

The following equipment is needed for obtaining samples for chemical or geotechnical analysis from test pits and trenches:

- Backhoe or other excavating machinery.
- Shovels, picks, hand augers, and stainless steel trowels/disposable trowels.
- Sample container - bucket with locking lid for large samples; appropriate bottle ware for chemical or geotechnical analysis samples.
- Polyethylene bags for enclosing sample containers; buckets.
- Remote sampler consisting of 10-foot sections of steel conduit (1-inch-diameter), hose clamps, and right angle adapter for conduit (see Attachment D).

6.8.3.3 Sampling Methods

The methods discussed in this section refer to test pit sampling from grade level. If test pit entry is required, see Section 6.8.3.4.

- Excavate the trench or pit in several 0.5- to 1.0-foot depth increments. Where soil types support the use of a sand bar cutting plate, use of this device is recommended to avoid potentially snagging utilities with the excavator teeth. It is recommended that soil probes or similar devices be employed where buried items or utilities may be encountered. This permits the trench floor to be probed prior to the next cut.
- After each increment:
  - the operator shall wait while the sampler inspects the test pit from grade level
  - the sampler shall probe the next interval where this is considered necessary. Practical depth increments for lithological evaluations may range from 2 to 4 feet i or where lithological changes are noted.
- The backhoe operator, who will have the best view of the test pit, shall immediately cease digging if:
  - Any fluid phase, including groundwater seepage, is encountered in the test pit
  - Any drums, other potential waste containers, obstructions, or utility lines are encountered
  - Distinct changes of material being excavated are encountered

This action is necessary to permit proper sampling of the test pit and to prevent a breach of safety protocol. Depending on the conditions encountered, it may be required to excavate more slowly and carefully with the backhoe.

For obtaining test pit samples from grade level, the following procedure shall be followed:

- Use the backhoe to remove loose material from the excavation walls and floor to the greatest extent possible.
- Secure the walls of the pit, if necessary. (There is seldom any need to enter a pit or trench that would justify the expense of shoring the walls. All observations and samples should be taken from the ground surface.)

- Samples of the test pit material are to be obtained either directly from the backhoe bucket or from the material after it has been deposited on the ground, as follows:

  a. The sampler or FOL shall direct the backhoe operator to remove material from the selected depth or location within the test pit/trench.

  b. The backhoe operator shall bring the bucket over to a designated location on the sidewall a sufficient distance from the pit (at least 5 feet) to allow the sampler to work around the bucket.

  c. After the bucket has been set on the ground, the backhoe operator shall either disengage the controls or shut the machine down.

  d. When signaled by the operator that it is safe to do, the sampler will approach the bucket.

  e. The soil shall be monitored with a photoionization or flame ionization detector (PID or FID) as directed in the project -specific planning documents.

  f. The sampler shall collect the sample from the center of the bucket or pile in accordance with surface soil sampling procedures of Section 6.3 or 6.4, as applicable. Collecting samples from the center of a pile or bucket eliminates cross-contamination from the bucket or other depth intervals.

- If a composite sample is desired, several depths or locations within the pit/trench will be selected, and the bucket will be filled from each area. It is preferable to send individual sample bottles filled from each bucket to the laboratory for compositing under the more controlled laboratory conditions. However, if compositing in the field is required, each sample container shall be filled from materials that have been transferred into a mixing bucket and homogenized. Note that homogenization/compositing is not applicable for samples to be subjected to volatile organic analysis.

> ***CAUTION***
>
> Care must be exercised when using the remote sampler described in the next step because of potential instability of trench walls. In situations where someone must move closer than 2 feet to the excavation edge, a board or platform should be used to displace the sampler's weight to minimize the chance of collapse of the excavation edge. Fall protection should also be employed when working near the edges or trenches greater than 6 feet deep. An immediate means to extract people who have fallen into the trench will be immediately available. These means may include ladders or rope anchor points.

- Using the remote sampler shown in Attachment D, samples can be taken at the desired depth from the sidewall or bottom of the pit as follows:

  a. Scrape the face of the pit/trench using a long-handled shovel or hoe to remove the smeared zone that has contacted the backhoe bucket.

  b. Collect the sample directly into the sample jar, by scraping with the jar edge, eliminating the need for sample handling equipment and minimizing the likelihood of cross-contamination.

  c. Cap the sample jar, remove it from the remote sampler assembly, and package the sample for shipment in accordance with SOP SA-6.3.

- Complete documentation as described in SOP SA-6.3 and Attachment C of this SOP.

6.8.3.4 In-Pit Sampling

Under rare conditions, personnel may be required to enter the test pit/trench. This is necessary only when soil conditions preclude obtaining suitable samples from the backhoe bucket (e.g., excessive mixing of soil or wastes within the test pit/trench) or when samples from relatively small discrete zones within the test pit are required. This approach may also be necessary to sample any seepage occurring at discrete levels or zones in the test pit that are not accessible with remote samplers.

In general, personnel shall sample and log pits and trenches from the ground surface, except as provided for by the following criteria:

- There are no practical alternative means of obtaining such data.
- The SSO and Competent Person determine that such action can be accomplished without breaching site safety protocol. This determination will be based on actual monitoring of the pit/trench after it is dug (including, at a minimum, measurements of oxygen concentration, flammable gases, and toxic compounds, in that order). Action levels will be provided in project-specific planning documents.
- A company-designated Competent Person determines that the pit/trench is stable trough soil classification evaluation/inspections or is made stable (by cutting/grading the sidewalls or using shoring) prior to entrance of any personnel. OSHA requirements shall be strictly observed.

If these conditions are satisfied, only one person may enter the pit/trench. On potentially hazardous waste sites, this individual shall be dressed in selected PPE as required by the conditions in the pit. He/she shall be affixed to a harness and lifeline and continuously monitored while in the pit.

A second and possible third individual shall be fully dressed in protective clothing including a self-contained breathing device and on standby during all pit entry operations to support self rescue or assisted self rescue. The individual entering the pit shall remain therein for as brief a period as practical, commensurate with performance of his/her work. After removing the smeared zone, samples shall be obtained with a decontaminated trowel or spoon.

6.8.3.5 Geotechnical Sampling

In addition to the equipment described in Section 6.8.3.2, the following equipment is needed for geotechnical sampling:

- Soil sampling equipment, similar to that used in shallow drilled boring (i.e., thin-walled tube samplers), that can be pushed or driven into the floor of the test pit.
- Suitable driving (e.g., sledge hammer) or pushing (e.g., backhoe bucket) equipment used to advance the sampler into the soil.
- Knives, spatulas, and other suitable devices for trimming hand-carved samples.
- Suitable containers (bags, jars, tubes, boxes, etc.), labels, wax, etc. for holding and safely transporting collected soil samples.
- Geotechnical equipment (pocket penetrometer, torvane, etc.) for field testing collected soil samples for classification and strength properties.

Disturbed grab or bulk geotechnical soil samples may be collected for most soil in the same manner as comparable soil samples for chemical analysis. These collected samples may be stored in jars or plastic-lined sacks (larger samples), which will preserve their moisture content. Smaller samples of this type are usually tested for their index properties to aid in soil identification and classification: larger bulk samples are usually required to perform compaction tests.

Relatively undisturbed samples are usually extracted in cohesive soil using thin-walled tube samplers, and such samples are then tested in a geotechnical laboratory for their strength, permeability, and/or compressibility. The techniques for extracting and preserving such samples are similar to those used in performing Shelby tube sampling in borings, except that the sampler is advanced by hand or backhoe, rather than by a drill rig. Also, the sampler may be extracted from the test pit by excavation around the tube when it is difficult to pull it out of the ground. If this excavation requires entry of the test pit, the requirements described in Section 6.8.3.4 shall be followed. The thin-walled tube sampler shall be pushed or driven vertically into the floor or steps excavated in the test pit at the desired sampling elevations. Extracting tube samples horizontally from the walls of the test pit is not appropriate because the sample will not have the correct orientation.

A sledge hammer or backhoe may be used to drive or push the tube into the ground. Place a piece of wood over the top of the sampler or sampling tube to prevent damage during driving/pushing of the sample. Pushing the sampler with a constant thrust is always preferable to driving it with repeated blows, thus minimizing disturbance to the sample. When using a sledge hammer, it is recommended that the sampler be stabilized using a rope/strap wrench or pipe wrench to remove the person's hands holding the sampler from the strike zone. If the sample cannot be extracted by rotating it at least two revolutions (to shear off the sample at the bottom), hook the sampler to the excavator or backhoe and extract. This means an alternative head will be used as a connection point or that multiple choke hitches will be applied to extract the sampler. If this fails and the excavator can dig deeper without potentially impacting subsurface utilities, excavate the sampler. If this fails or if the excavator cannot be used due to subsurface utilities, hand-excavate to remove the soil from around the sides of the sampler. If hand-excavation requires entry into the test pit, the requirements in Section 6.8.3.4 must be followed. Prepare the sample as described in Steps 9 through 13 in Section 6.2.3, and label, pack and transport the sample in the required manner, as described in SOPs SA-6.3 and SA-6.1.

6.8.4 Backfilling of Trenches and Test Pits

All test pits and excavations must be either backfilled, covered, or otherwise protected at the end of each day. No excavations shall remain open during non-working hours unless adequately covered or otherwise protected.

Before backfilling, the onsite crew may photograph, if required by the project-specific work plan, all significant features exposed by the test pit and trench and shall include in the photograph a scale to show dimensions. Photographs of test pits shall be marked to include site number, test pit number, depth, description of feature, and date of photograph. In addition, a geologic description of each photograph shall be entered in the site logbook. All photographs shall be indexed and maintained as part of the project file for future reference.

After inspection, backfill material shall be returned to the pit under the direction of the FOL. Backfill should be returned to the trench or test pit in 6-inch to 1-foot lifts and compacted with the bucket. Remote controlled tampers or rollers may be lowered into the trench and operated from top side. This procedure will continue to the grade surface. It is recommended that the trench be tracked or rolled in. During excavation, clean soil from the top 2 feet may have been separated to be used to cover the last segments. Where these materials are not clean, it is recommended that clean fill be used for the top cover.

~~If a low-permeability layer is penetrated (resulting in groundwater flow from an upper contaminated flow zone into a lower uncontaminated flow zone), backfill material must represent original conditions or be impermeable. Backfill could consist of a soil-bentonite mix prepared in a proportion specified by the FOL (representing a permeability equal to or less than original conditions). Backfill can be covered by "clean" soil and graded to the original land contour. Revegetation of the disturbed area may also be required.~~

6.9 Records

The appropriate sample log sheet (see Attachment A of this SOP) must be completed by the site geologist/sampler for all samples collected. All soil sampling locations should be documented by tying in the location of two or more nearby permanent landmarks (building, telephone pole, fence, etc.) or obtaining GPS coordinates; and shall be noted on the appropriate sample log sheet, site map, or field notebook. Surveying may also be necessary, depending on the project requirements.

Test pit logs (see Attachment C of this SOP) shall contain a sketch of pit conditions. If the project-specific work plan requires photographs, at least one photograph with a scale for comparison shall be taken of each pit. Included in the photograph shall be a card showing the test pit number. Boreholes, test pits, and trenches shall be logged by the field geologist in accordance with SOP GH-1.5.

Other data to be recorded in the field logbook include the following:

- Name and location of job
- Date of boring and excavation
- Approximate surface elevation
- Total depth of boring and excavation
- Dimensions of pit
- Method of sample acquisition
- Type and size of samples
- Soil and rock descriptions
- Photographs if required
- Groundwater levels
- PID/FID/LEL/O_2 meter readings
- Other pertinent information, such as waste material encountered

In addition, site-specific documentation to be maintained by the SSO and/or Competent Person will be required including:

- Calibration logs
- Excavation inspection checklists

- Soil type classification

7.0 REFERENCES

American Society for Testing and Materials, 1987. ASTM Standards D1587-83 and D1586-84. ASTM Annual Book of Standards. ASTM. Philadelphia, Pennsylvania. Volume 4.08.

NUS Corporation, 1986. Hazardous Material Handling Training Manual.

NUS Corporation and CH2M Hill, August, 1987. Compendium of Field Operation Methods. Prepared for the U.S. EPA.

OSHA, Excavation, Trenching and Shoring 29 CFR 1926.650-653.

OSHA, Confined Space Entry 29 CFR 1910.146.

USEPA, November 2001. Environmental Investigations Standard Operating Procedures and Quality Assurance Manual.

ATTACHMENT A
SOIL & SEDIMENT SAMPLE LOG SHEET

Tetra Tech NUS, Inc.

SOIL & SEDIMENT SAMPLE LOG SHEET

Page___ of ___

Project Site Name: _____
Project No.: _____

[] Surface Soil
[] Subsurface Soil
[] Sediment
[] Other: _____
[] QA Sample Type: _____

Sample ID No.: _____
Sample Location: _____
Sampled By: _____
C.O.C. No.: _____

Type of Sample:
[] Low Concentration
[] High Concentration

GRAB SAMPLE DATA:

Date:	Depth	Color	Description (Sand, Silt, Clay, Moisture, etc.)
Time:			
Method:			
Monitor Reading (ppm):			

COMPOSITE SAMPLE DATA:

Date:	Time	Depth	Color	Description (Sand, Silt, Clay, Moisture, etc.)
Method:				
Monitor Readings (Range in ppm):				

SAMPLE COLLECTION INFORMATION:

Analysis	Container Requirements	Collected	Other

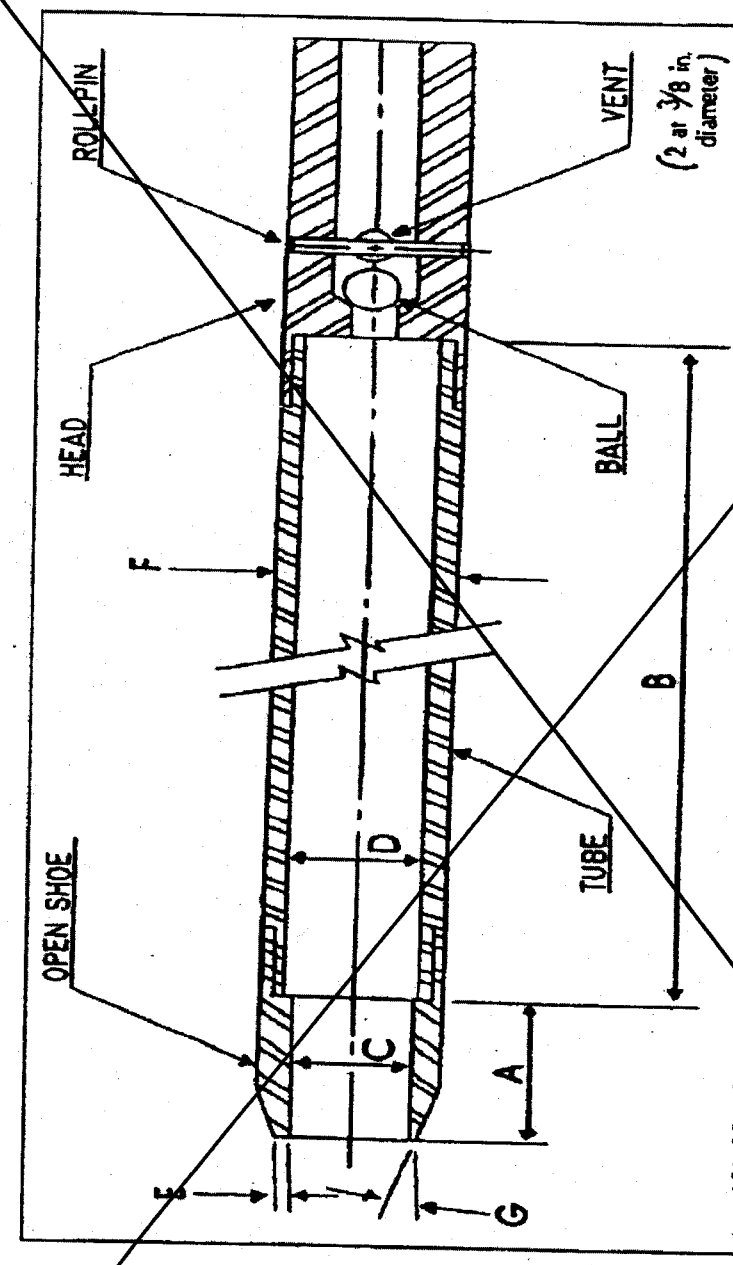
OBSERVATIONS / NOTES:

MAP:

Circle if Applicable:

MS/MSD	Duplicate ID No.:	Signature(s):

ATTACHMENT B
SPLIT-SPOON SAMPLER



A = 1.0 to 2.0 in. (25 to 50 mm)
B = 18.0 to 30.0 in. (0.457 to 0.762 m)
C = 1.375 ± 0.005 in. (34.93 ± 0.13 mm)
D = 1.50 ± 0.05 – 0.00 in. (38.1 ± 1.3 – 0.0 mm)
E = 0.10 ± 0.02 in. (2.54 ± 0.25 mm)
F = 2.00 ± 0.05 – 0.00 in. (50.8 ± 1.3 – 0.0 mm)
G = 18.0° to 23.0°

The 1½ in. (38 mm) inside diameter split barrel may be used with a 16-gage wall thickness split liner. The penetrating end of the drive shoe may be slightly rounded. Metal or plastic retainers may be used to retain soil samples.

ATTACHMENT C
TEST PIT LOG

Tetra Tech NUS, Inc.

TEST PIT LOG

Page ___ of ___

PROJECT NAME: _____ TEST PIT No.: _____
PROJECT NUMBER: _____ DATE: _____
LOCATION: _____ GEOLOGIST: _____

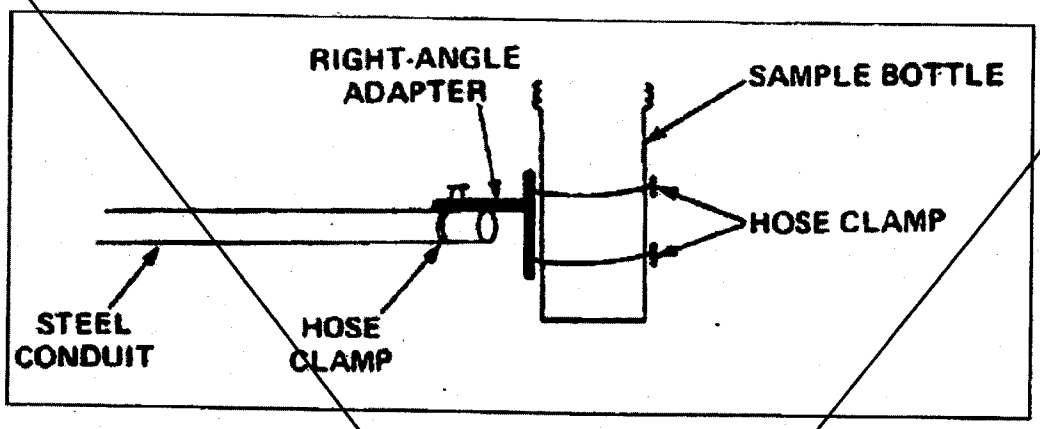
Depth (Ft.)	Lithology Change (Depth/Ft.)	MATERIAL DESCRIPTION Soil/Waste Characteristics (lithology, density, color, etc.)	USCS	Remarks	PID/FID READING Source (ppm)	BZ (ppm)

TEST PIT CROSS SECTION AND / OR PLAN VIEW

REMARKS: _____

PHOTO LOG: _____

ATTACHMENT D
REMOTE SAMPLE HOLDER FOR TEST PIT/TRENCH SAMPLING



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1.0 PURPOSE

The purpose of this procedure is to provide general reference information on Direct Push Technology (DPT). DPT is designed to collect soil, groundwater, and soil gas samples without using conventional drilling techniques. The advantage of using DPT over conventional drilling includes the generation of little or no drill cuttings, sampling in locations with difficult accessibility, reduced overhead clearance requirements, no fluid introduction during probing, and typical lower costs per sample than with conventional techniques. Disadvantages include a maximum penetration depth of approximately 15 to 40 feet in dense soils (although it may be as much as 60 to 80 feet in certain types of geological environments), reduced capability of obtaining accurate water-level measurements, and the inability to install permanent groundwater monitoring wells. The methods and equipment described herein are for collection of surface and subsurface soil samples and groundwater samples. Soil gas sampling is discussed in SOP SA-2.4.

2.0 SCOPE

This procedure provides information on proper sampling equipment and techniques for DPT. Review of the information contained herein will facilitate planning of the field sampling effort by describing standard sampling techniques. The techniques described shall be followed whenever applicable, noting that site-specific conditions or project-specific plans may require adjustments in methodology.

3.0 GLOSSARY

Direct Push Technology (DPT) - DPT refers to sampling tools and sensors that are driven directly into the ground without the use of conventional drilling equipment. DPT typically utilizes hydraulic pressure and/or percussion hammers to advance the sampling tools. A primary advantage of DPT over conventional drilling techniques is that DPT results in the generation of little or no investigation derived waste.

Geoprobe® - Geoprobe® is a manufacturer of a hydraulically-powered, percussion/probing machines utilizing DPT to collect subsurface environmental samples. Geoprobe® relies on a relatively small amount of static weight (vehicle) combined with percussion as the energy for advancement of a tool string. The Geoprobe® equipment can be mounted in a multitude of vehicles for access to all types of environmental sites.

HydroPunch™ - HydroPunch™ is a manufacturer of stainless steel and Teflon® sampling tools that are capable of collecting representative groundwater and/or soil samples without requiring the installation of a groundwater monitoring well or conventional soil boring. HydroPunch™ is an example of DPT sampling equipment.

Flame Ionization Detector (FID) - A portable instrument for the measurement of many combustible organic compounds and a few inorganic compounds in air at parts-per million levels. The basis for the detection is the ionization of gaseous species utilizing a flame as the energizing source.

Photo Ionization Detector (PID) - A portable instrument for the measurement of many combustible organic compounds and a few inorganic compounds in air at parts-per million levels. The basis for the detection is the ionization of gaseous species utilizing ultraviolet radiation as the energizing source.

4.0 RESPONSIBILITIES

Project Manager - The Project Manager is responsible for selecting and/or reviewing the appropriate DPT drilling procedure required to support the project objectives.

Field Operations Leader (FOL)- The FOL is primarily responsible for performing the DPT in accordance with the project-specific plan.

5.0 SOIL SAMPLING PROCEDURES

5.1 General

The common methodology for the investigation of the vadose zone is soil boring drilling and soil sampling. However, drilling soil borings can be very expensive. Generally the advantage of DPT for subsurface soil sampling is the reduced cost of disposal of drilling cuttings and shorter sampling times.

5.2 Sampling Equipment

Equipment needed for conducting DPT drilling for subsurface soil sampling includes, but is not limited to, the following:

- Geoprobe® Sampling Kit
- Cut-resistant gloves
- 4-foot x 1.5-inch diameter macrocore sampler
- Probe sampling adapters
- Roto-hammer with 1.5-inch bit
- Disposable acetate liners for soil macrocore sampler
- Cast aluminum or steel drive points
- Geoprobe® AT-660 Series Large Bore Soil Sampler, or equivalent
- Standard decontamination equipment and solutions

For health and safety equipment and procedures, follow the direction provided in the Safe Work Permit in Attachment 1, or the more detailed directions provided in the project's Health and Safety Plan.

5.3 DPT Sampling Methodology

There are several methods for the collection of soil samples using DPT drilling. The most common method is discussed in the following section. Variations of the following method may be conducted upon approval of the Project Manager in accordance with the project-specific plan.

- Macrocore samplers fitted with detachable aluminum or steel drive points are driven into the ground using hydraulic pressure. If there is concrete or pavement over a sampling location, a Roto-hammer is used to drill a minimum 1.5-inch diameter hole through the surface material. A Roto-hammer may also be used if very dense soils are encountered.

- The sampler is advanced continuously in 4-foot intervals or less if desired. No soil cuttings are generated because the soil which is not collected in the sampler is displaced within the formation.

- The sampler is retracted from the hole, and the 4-foot continuous sample is removed from the outer coring tube. The sample is contained within an inner acetate liner.

- Attach the metal trough from the Geoprobe® Sampling Kit firmly to the tail gate of a vehicle. If a vehicle with a tail gate is not available, secure the trough on another suitable surface.

- Place the acetate liner containing the soils in the trough.

- While wearing cut-resistant gloves (constructed of leather or other suitable material), cut the acetate liner through its entire length using the double-bladed knife that accompanies the Geoprobe® Sampling Kit. Then remove the strip of acetate from the trough to gain access to the collected soils. Do not attempt to cut the acetate liner while holding it in your hand.

- Field screen the sample with an FID or PID, and observe/examine the sample (according to SOP GH-1.3). If appropriate, transfer the sample to sample bottles for laboratory analysis. If additional volume is required, push an additional boring adjacent to the first and composite/mix the same interval. Field compositing is usually not acceptable for sample requiring volatile organics analysis.

- Once sampling has been completed, the hole is backfilled with bentonite chips or bentonite cement grout, depending upon project requirements. Asphalt or concrete patch is used to cap holes through paved or concrete areas. All holes should be finished smooth to existing grade.

- In the event the direct push van/truck cannot be driven to a remote location or a sampling location with difficult accessibility, sampling probes may be advanced and sampled manually or with air/electric operated equipment (e.g., jack hammer).

- Sampling equipment is decontaminated prior to collecting the next sample.

6.0 GROUNDWATER SAMPLING PROCEDURES

6.1 General

The most common methodology for the investigation of groundwater is the installation and sampling of permanent monitoring wells. If only groundwater screening is required, the installation and sampling of temporary well points may be performed. The advantage of temporary well point installation using DPT is reduced cost due to no or minimal disposal of drilling cuttings and well construction materials, and shorter installation/times sampling.

Two disadvantages of DPT drilling for well point installation are:

- In aquifers with low yields, well points may have to be sampled without purging or development.
- If volume requirements are high, this method can be time consuming for low yield aquifers.

6.2 Sampling Equipment

Equipment needed for temporary well installation and sampling using DPT includes, but is not limited, to the following:

- 2-foot x 1-inch diameter mill-slotted (0.005 to 0.02-inch) well point
- Connecting rods
- Roto-hammer with 1.5-inch bit
- Mechanical jack
- 1/4-inch OD polyethylene tubing
- 3/8-inch OD polyethylene tubing
- Peristaltic pump
- Standard decontamination equipment and solutions

6.3 DPT Temporary Well Point Installation and Sampling Methodology

There are several methods for the installation and sampling of temporary well points using DPT. The most common methodology is discussed below. Variations of the following method may be conducted upon approval of the Project Manager in accordance with the project specific plan.

- A 2-foot x 1-inch diameter mill-slotted (0.005 to 0.02-inch) well point attached to connecting rods is driven into the ground to the desired depth using a rotary electric hammer or other direct push drill rig. If there is concrete or pavement over a sampling location, a Roto-hammer or electric coring machine is used to drill a hole through the surface material.

- The well point will be allowed to equilibrate for at least 15 minutes, after which a measurement of the static water level will be taken. The initial measurement of the water level will be used to assess the amount of water which is present in the well point and to determine the amount of silt and sand infiltration that may have occurred.

- The well point will be developed using a peristaltic pump and polyethylene tubing to remove silt and sand which may have entered the well point. The well point is developed by inserting polyethylene tubing to the bottom of the well point and lifting and lowering the tubing slightly while the pump is operating. The pump will be operated at a maximum rate of approximately 2 liters per minute. After removal of sediment from the bottom of the well point, the well point will be vigorously pumped at maximum capacity until discharge water is visibly clear and no further sediments are being generated. Measurements of pH, specific conductance, temperature, and turbidity shall be recorded every 5 to 10 minutes during the purging process. After two consistent readings of pH, specific conductance, temperature and turbidity (±10 percent), the well may be sampled.

- A sample will be collected using the peristaltic pump set at the same or reduced speed as during well development. Samples (with the exception of the samples to be analyzed for volatile organic compounds, VOCs) will be collected directly from the pump discharge. Sample containers for VOCs will be filled by (first shutting off the pump) crimping the discharge end of the sample tubing when filled, removing the inlet end of the sample tubing from the well, suspending the inlet tubing above the vial, and allowing water to fill each vial by gravity flow.

- Once the groundwater sample has been collected, the connecting rods and well point will be removed from the hole with the direct push rig hydraulics. The hole will be backfilled with bentonite chips or bentonite cement grout, depending upon project requirements. Asphalt or concrete patch will be used to cap holes through paved or concrete areas. All holes will be finished smooth to existing grade.

- In the event the direct push van/truck cannot be driven to a remote location or sampling location with difficult accessibility, sampling probes may be advanced and sampled manually or with air/electric-operated equipment (e.g., jack hammer).

- Decontaminate the equipment before moving to the next location.

7.0 RECORDS

A record of all field procedures, tests, and observations must be recorded in the field logbook, boring logs, and sample log sheets, as needed. Entries should include all pertinent data regarding the investigation. The use of sketches and field landmarks will help to supplement the investigation and evaluation.

ATTACHMENT 1
SAFE WORK PERMIT FOR DPT OPERATIONS

Permit No. _____ Date: _____ Time: From _____ to _____

SECTION I: General Job Scope

I. Work limited to the following (description, area, equipment used): **Monitoring well drilling and installation through direct push technology**

II. Required Monitoring Instruments: _____

III. Field Crew: _____

IV. On-site Inspection conducted ☐ Yes ☐ No Initials of Inspector _____ _____ TtNUS

SECTION II: General Safety Requirements (To be filled in by permit issuer)

V. Protective equipment required

Level D ☒ Level B ☐
Level C ☐ Level A ☐
Detailed on Reverse

Respiratory equipment required

Full face APR ☐
Half face APR ☐
SKA-PAC SAR ☐
Skid Rig ☐

Escape Pack ☐
SCBA ☐
Bottle Trailer ☐
None ☒

Level D Minimum Requirements: Sleeved shirt and long pants, safety footwear, and work gloves. Safety glasses, hard hats, and hearing protection will be worn when working near or sampling in the vicinity of the DPT rig.

Modifications/Exceptions.

VI. Chemicals of Concern | Action Level(s) | Response Measures

VII. Additional Safety Equipment/Procedures

Hard-hat	☒ Yes	☐ No	Hearing Protection (Plugs/Muffs)	☒ Yes	☐ No
Safety Glasses	☒ Yes	☐ No	Safety belt/harness	☐ Yes	☒ No
Chemical/splash goggles	☐ Yes	☒ No	Radio	☐ Yes	☒ No
Splash Shield	☐ Yes	☒ No	Barricades	☒ Yes	☐ No
Splash suits/coveralls	☐ Yes	☐ No	Gloves (Type - _____)	☐ Yes	☐ No
Steel toe Work shoes or boots	☒ Yes	☐ No	Work/warming regimen	☐ Yes	☐ No

Modifications/Exceptions: Reflective vests for high traffic areas.

VIII. Procedure review with permit acceptors

	Yes	NA		Yes	NA
Safety shower/eyewash (Location & Use)	☐	☒	Emergency alarms	☐	☐
Daily tail gate meetings	☒	☐	Evacuation routes	☐	☐
Contractor tools/equipment/PPE inspected	☐	☐	Assembly points	☐	☐

IX. Site Preparation

Utility Clearances obtained for areas of subsurface investigation	☐ Yes	☐ No
Physical hazards removed or blockaded	☐ Yes	☐ No
Site control boundaries demarcated/signage	☐ Yes	☐ No

X. Equipment Preparation

	Yes	NA
Equipment drained/depressurized	☐	☒
Equipment purged/cleaned	☐	☒
Isolation checklist completed	☐	☒
Electrical lockout required/field switch tested	☐	☒
Blinds/misalignments/blocks & bleeds in place	☐	☒
Hazardous materials on walls/behind liners considered	☐	☒

XI. Additional Permits required (Hot work, confined space entry). ☐ Yes ☐ No

If yes, complete permit required or contact Health Sciences, Pittsburgh Office

XII. Special instructions, precautions:

_____

Permit Issued by: _____ Permit Accepted by: _____

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1.0 PURPOSE

The purpose of this Standard Operating Procedure (SOP) is to provide information on sample preservation, packaging, and shipping procedures to be used in handling environmental samples submitted for chemical constituent, biological, or geotechnical analysis. Sample chain-of-custody procedures and other aspects of field documentation are addressed in SOP SA-6.3. Sample identification is addressed in SOP CT-04.

2.0 SCOPE

This procedure describes the appropriate containers to be used for samples depending on the analyses to be performed, and the steps necessary to preserve the samples when shipped off site for chemical analysis.

3.0 GLOSSARY

Hazardous Material - A substance or material which has been determined by the Secretary of Transportation to be capable of posing an unreasonable risk to health, safety, and property when transported in commerce, and which has been so designated. Under 49 CFR, the term includes hazardous substances, hazardous wastes, marine pollutants, and elevated temperature materials, as well as materials designated as hazardous under the provisions of §172.101 and §172.102 and materials that meet the defining criteria for hazard classes and divisions in Part 173. With slight modifications, IATA has adopted DOT "hazardous materials" as IATA "Dangerous Goods."

Hazardous Waste - Any substance listed in 40 CFR, Subpart D (~~y~~261.30 et seq.), or otherwise characterized as ignitable, corrosive, reactive, or toxic (as defined by Toxicity Characteristic Leaching Procedure, TCLP, analysis) as specified under 40 CFR, Subpart C (~~y~~261.20 et seq.), that would be subject to manifest requirements specified in 40 CFR 262. Such substances are defined and regulated by EPA.

Marking - A descriptive name, identification number, instructions, cautions, weight, specification or UN marks, or combination thereof required on outer packaging of hazardous materials.

n.o.i - Not otherwise indicated (may be used interchangeably with n.o.s.).

n.o.s. - Not otherwise specified.

Packaging - A receptacle and any other components or materials necessary for compliance with the minimum packaging requirements of 49 CFR 174, including containers (other than freight containers or overpacks), portable tanks, cargo tanks, tank cars, and multi-unit tank-car tanks to perform a containment function in conformance with the minimum packaging requirements of 49 CFR 173.24(a) & (b).

Placard - Color-coded, pictorial sign which depicts the hazard class symbol and name and which is placed on the side of a vehicle transporting certain hazardous materials.

Common Preservatives:

- Hydrochloric Acid - HCl
- Sulfuric Acid - H_2SO_4
- Nitric Acid - HNO_3
- Sodium Hydroxide - NaOH

Other Preservatives

- Zinc Acetate
- Sodium Thiosulfate - $Na_2S_2O_3$

Normality (N) - Concentration of a solution expressed as equivalent per liter, an equivalent being the amount of a substance containing 1 gram-atom of replaceable hydrogen or its equivalent.

Reportable Quantity (RQ) - For the purposes of this SOP, means the quantity specified in column 3 of the Appendix to DOT 49 CFR §172.101 for any material identified in column 1 of the appendix. A spill greater than the amount specified must be reported to the National Response Center.

Sample - A sample is physical evidence collected from a facility or the environment, which is representative of conditions at the location and time of collection.

4.0 RESPONSIBILITIES

Field Operations Leader - Directly responsible for the bottling, preservation, labeling, packaging, shipping, and custody of samples up to and including release to the shipper.

Field Samplers - Responsible for initiating the Chain-of-Custody Record (per SOP SA-6.3), implementing the packaging and shipping requirements, and maintaining custody of samples until they are relinquished to another custodian or to the shipper.

5.0 PROCEDURES

Sample identification, labeling, documentation, and chain-of-custody are addressed by SOP SA-6.3.

5.1 Sample Containers

Different types of chemicals react differently with sample containers made of various materials. For example, trace metals adsorb more strongly to glass than to plastic, whereas many organic chemicals may dissolve various types of plastic containers. Attachments A and B show proper containers (as well as other information) per 40 CFR 136. In general, the sample container shall allow approximately 5-10 percent air space ("ullage") to allow for expansion/vaporization if the sample warms during transport. However, for collection of volatile organic compounds, head space shall be omitted. The analytical laboratory will generally provide certified-clean containers for samples to be analyzed for chemical constituents. Shelby tubes or other sample containers are generally provided by the driller for samples requiring geotechnical analysis. Sufficient lead time shall be allowed for a delivery of sample container orders. Therefore, it is critical to use the correct container to maintain the integrity of the sample prior to analysis.

Once opened, the container must be used at once for storage of a particular sample. Unused but opened containers are to be considered contaminated and must be discarded. Because of the potential for introduction of contamination, they cannot be reclosed and saved for later use. Likewise, any unused containers which appear contaminated upon receipt, or which are found to have loose caps or a missing Teflon liner (if required for the container), shall be discarded.

5.2 Sample Preservation

Many water and soil samples are unstable and therefore require preservation to prevent changes in either the concentration or the physical condition of the constituent(s) requiring analysis. Although complete and irreversible preservation of samples is not possible, preservation does retard the chemical and biological

changes that inevitably take place after the sample is collected. Preservation techniques are usually limited to pH control, chemical addition(s), and refrigeration/ freezing (certain biological samples only).

5.2.1 Overview

The preservation techniques to be used for various analytes are listed in Attachments A and B. Reagents required for sample preservation will either be added to the sample containers by the laboratory prior to their shipment to the field or be added in the field (in a clean environment). Only high purity reagents shall be used for preservation. In general, aqueous samples of low-concentration organics (or soil samples of low- or medium-concentration organics) are cooled to 4°C. Medium-concentration aqueous samples, high-hazard organic samples, and some gas samples are typically not preserved. Low-concentration aqueous samples for metals are acidified with HNO_3, whereas medium-concentration and high-hazard aqueous metal samples are not preserved. Low- or medium-concentration soil samples for metals are cooled to 4°C, whereas high-hazard samples are not cooled.

The following subsections describe the procedures for preparing and adding chemical preservatives. Attachments A and B indicate the specific analytes which require these preservatives.

The FOL is responsible for ensuring that an accurate Chemical Inventory is created and maintained for all hazardous chemicals brought to the work site (see Section 5 of the TtNUS Health and Safety Guidance Manual). Furthermore, the FOL must ensure that a corresponding Material Safety Data Sheet (MSDS) is collected for every substance entered on the site Chemical Inventory, and that all persons using/handling/ disposing of these substances review the appropriate MSDS for substances they will work with. The Chemical Inventory and the MSDSs must be maintained at each work site in a location and manner where they are readily-accessible to all personnel.

5.2.2 Preparation and Addition of Reagents

Addition of the following acids or bases may be specified for sample preservation; these reagents shall be analytical reagent (AR) grade or purer and shall be diluted to the required concentration with deionized water before field sampling commences. To avoid uncontrolled reactions, be sure to Add Acid to water (not vice versa). A dilutions guide is provided below.

Acid/Base	Dilution	Concentration	Estimated Amount Required for Preservation
Hydrochloric Acid (HCl)	1 part concentrated HCl: 1 part double-distilled, deionized water	6N	5-10 mL
Sulfuric Acid (H_2SO_4)	1 part concentrated H_2SO_4: 1 part double-distilled, deionized water	18N	2 - 5 mL
Nitric Acid (HNO_3)	Undiluted concentrated HNO_3	16N	2 - 5 mL
Sodium Hydroxide (NaOH)	400 grams solid NaOH dissolved in 870 mL double-distilled, deionized water; yields 1 liter of solution	10N	2 mL

The amounts required for preservation shown in the above table assumes proper preparation of the preservative and addition of the preservative to one liter of aqueous sample. This assumes that the sample is initially at pH 7, is poorly buffered, and does not contain particulate matter; as these conditions vary, more preservative may be required. Consequently, the final sample pH must be checked using narrow-range pH paper, as described in the generalized procedure detailed below:

- Pour off 5-10 mL of sample into a dedicated, clean container. Use some of this sample to check the initial sample pH using wide range (0-14) pH paper. Never dip the pH paper into the sample; always apply a drop of sample to the pH paper using a clean stirring rod or pipette.

- Add about one-half of the estimated preservative required to the original sample bottle. Cap and invert gently several times to mix. Check pH (as described above) using medium range pH paper (pH 0-6 or pH 7.5-14, as applicable).

- Cap sample bottle and seal securely.

Additional considerations are discussed below:

- To test if ascorbic acid must be used to remove oxidizing agents present in the sample before it can be properly preserved, place a drop of sample on KI-starch paper. A blue color indicates the need for ascorbic acid addition.

  If required, add a few crystals of ascorbic acid to the sample and retest with the KI-starch paper. Repeat until a drop of sample produces no color on the KI-starch paper. Then add an additional 0.6 grams of ascorbic acid per each liter of sample volume.

  Continue with proper base preservation of the sample as described above.

- Samples for sulfide analysis must be treated by the addition of 4 drops (0.2 mL) of 2N zinc acetate solution per 100 ml of sample.

  The 2N zinc acetate solution is made by dissolving 220 grams of zinc acetate in 870 mL of double-distilled, deionized water to make 1 liter of solution.

  The sample pH is then raised to 9 using the NaOH preservative.

- Sodium thiosulfate must be added to remove residual chlorine from a sample. To test the sample for residual chlorine use a field test kit specially made for this purpose.

  If residual chlorine is present, add 0.08 grams of sodium thiosulfate per liter of sample to remove the residual chlorine.

  Continue with proper acidification of the sample as described above.

For biological samples, 10% buffered formalin or isopropanol may also be required for preservation. Questions regarding preservation requirements should be resolved through communication with the laboratory before sampling begins.

5.3 Field Filtration

At times, field-filtration may be required to provide for the analysis of dissolved chemical constituents. Field-filtration must be performed prior to the preservation of samples as described above. General procedures for field filtration are described below:

- The sample shall be filtered through a non-metallic, 0.45-micron membrane filter, immediately after collection. The filtration system shall consist of dedicated filter canister, dedicated tubing, and a peristaltic pump with pressure or vacuum pumping squeeze action (since the sample is filtered by mechanical peristalsis, the sample travels only through the tubing).

- To perform filtration, thread the tubing through the peristaltic pump head. Attach the filter canister to the discharge end of the silicon tubing (note flow direction arrow); attach the aqueous sample container to the intake end of the silicon tubing. Turn the peristaltic pump on and perform filtration. Run approximately 100 ml of sample through the filter and discard prior to sample collection.

- Continue by preserving the filtrate (contained in the filter canister), as applicable and generally described above.

5.4 Sample Packaging and Shipping

Only employees who have successfully completed the TtNUS "Shipping Hazardous Materials" training course are authorized to package and ship hazardous substances. These trained individuals are responsible for performing shipping duties in accordance with this training.

Samples collected for shipment from a site shall be classified as either environmental or hazardous material samples. Samples from drums containing materials other than Investigative Derived Waste (IDW) and samples obtained from waste piles or bulk storage tanks are generally shipped as hazardous materials. A distinction must be made between the two types of samples in order to:

- Determine appropriate procedures for transportation of samples (if there is any doubt, a sample shall be considered hazardous and shipped accordingly.)

- Protect the health and safety of transport and laboratory personnel receiving the samples (special precautions are used by the shipper and at laboratories when hazardous materials are received.)

Detailed procedures for packaging environmental samples are outlined in the remainder of this section.

5.4.1 Environmental Samples

Environmental samples are packaged as follows:

- Place properly identified sample container, with lid securely fastened, in a plastic bag (e.g. Ziploc baggie), and seal the bag.

- Place sample in a cooler constructed of sturdy material which has been lined with a large, plastic bag (e.g. "garbage" bag). Drain plugs on coolers must be taped shut.

- Pack with enough cushioning materials such as bubble wrap (shoulders of bottles must be iced if required) to minimize the possibility of the container breaking.

- If cooling is required (see Attachments A and B), place ice around sample container shoulders, and on top of packing material (minimum of 8 pounds of ice for a medium-size cooler).

- Seal (i.e., tape or tie top in knot) large liner bag.

- The original (top, signed copy) of the COC form shall be placed inside a large Ziploc-type bag and taped inside the lid of the shipping cooler. If multiple coolers are sent but are included on one COC form, the COC form should be sent with the cooler containing the vials for VOC analysis. The COC form should then state how many coolers are included with that shipment.

- Close and seal outside of cooler as described in SOP SA-6.3. Signed custody seals must be used.

Coolers must be marked as containing "Environmental Samples." The appropriate side of the container must be marked "This End Up" and arrows placed appropriately. No DOT marking or labeling is required; there are no DOT restrictions on mode of transportation.

6.0 REFERENCES

American Public Health Association, 1981. Standard Methods for the Examination of Water and Wastewater, 15th Edition. APHA, Washington, D.C.

International Air Transport Association (latest issue). Dangerous Goods Regulations, Montreal, Quebec, Canada.

U.S. Department of Transportation (latest issue). Hazardous Materials Regulations, 49 CFR 171-177.

U.S. EPA, 1984. "Guidelines Establishing Test Procedures for the Analysis of Pollutants under Clean Water Act." Federal Register, Volume 49 (209), October 26, 1984, p. 43234.

U.S. EPA, 1979. Methods for Chemical Analysis of Water and Wastes. EPA-600/4-79-020, U.S. EPA-EMSL, Cincinnati, Ohio.

ATTACHMENT A

GENERAL SAMPLE CONTAINER AND PRESERVATION REQUIREMENTS

Sample Type and Concentration			Container[1]	Sample Size	Preservation[2]	Holding Time[2]
WATER						
Organics (GC&GC/MS)	VOC	Low	Borosilicate glass	2 x 40 mL	Cool to 4°C HCl to ≤ 2	14 days[9]
	Extractables SVOCs and pesticide/PCBs)	(Low	Amber glass	2x2 L or 4x1 L	Cool to 4°C	7 days to extraction; 40 days after extraction
	Extractables SVOCs and pesticide/PCBs)	(Medium	Amber glass	2x2 L or 4x1 L	None	7 days to extraction; 40 days after extraction
Inorganics	Metals	Low	High-density polyethylene	1 L	HNO_3 to pH ≤2	6 months (Hg-28 days)
		Medium	Wide-mouth glass	16 oz.	None	6 months
	Cyanide	Low	High-density polyethylene	1 L	NaOH to pH>12	14 days
	Cyanide	Medium	Wide-mouth glass	16 oz.	None	14 days
Organic/ Inorganic	High Hazard		Wide-mouth glass	8 oz.	None	14 days
SOIL						
Organics (GC&GC/MS)	VOC		EnCore Sampler	(3) 5 g Samplers	Cool to 4°C	48 hours to lab preservation
	Extractables SVOCs and pesticides/PCBs)	(Low	Wide-mouth glass	8 oz.	Cool to 4°C	14 days to extraction; 40 days after extraction
	Extractables SVOCs and pesticides/PCBs)	(Medium	Wide-mouth glass	8 oz.	Cool to 4°C	14 days to extraction; 40 days after extraction
Inorganics	Low/Medium		Wide-mouth glass	8 oz.	Cool to 4°C	6 months (Hg - 28 days) Cyanide (14 days)
Organic/Inorganic	High Hazard		Wide-mouth glass	8 oz.	None	NA
Dioxin/Furan	All		Wide-mouth glass	4 oz.	None	35 days until extraction; 40 days after extraction
TCLP	All		Wide-mouth glass	8 oz.	None	7 days until preparation; analysis as per fraction
AIR						
Volatile Organics	Low/Medium		Charcoal tube -- 7 cm long, 6 mm OD, 4 mm ID	100 L air	Cool to 4°C	5 days recommended

1 All glass containers should have Teflon cap liners or septa.
2 See Attachment E. Preservation and maximum holding time allowances per 40 CFR 136.

ATTACHMENT B

ADDITIONAL REQUIRED CONTAINERS, PRESERVATION TECHNIQUES, AND HOLDING TIMES

Parameter Number/Name	Container[1]	Preservation[2][3]	Maximum Holding Time[4]
INORGANIC TESTS:			
Acidity	P, G	Cool, 4°C	14 days
Alkalinity	P, G	Cool, 4°C	14 days
Ammonia - Nitrogen	P, G	Cool, 4°C; H_2SO_4 to pH 2	28 days
Biochemical Oxygen Demand (BOD)	P, G	Cool, 4°C	48 hours
Bromide	P, G	None required	28 days
Chemical Oxygen Demand (COD)	P, G	Cool, 4°C; H_2SO_4 to pH 2	28 days
Chloride	P, G	None required	28 days
Chlorine, Total Residual	P, G	None required	Analyze immediately
Color	P, G	Cool, 4°C	48 hours
Cyanide, Total and Amenable to Chlorination	P, G	Cool, 4°C; NaOH to pH 12; 0.6 g ascorbic acid[5]	14 days[6]
Fluoride	P	None required	28 days
Hardness	P, G	HNO_3 to pH 2; H_2SO_4 to pH 2	6 months
Total Kjeldahl and Organic Nitrogen	P, G	Cool, 4°C; H_2SO_4 to pH 2	28 days
Nitrate - Nitrogen	P, G	None required	48 hours
Nitrate-Nitrite - Nitrogen	P, G	Cool, 4°C; H_2SO_4 to pH 2	28 days
Nitrite - Nitrogen	P, G	Cool, 4°C	48 hours
Oil & Grease	G	Cool, 4°C; H_2SO_4 to pH 2	28 days
Total Organic Carbon (TOC)	P, G	Cool, 4°C; HCl or H_2SO_4 to pH 2	28 days
Orthophosphate	P, G	Filter immediately; Cool, 4°C	48 hours
Oxygen, Dissolved-Probe	G Bottle & top	None required	Analyze immediately
Oxygen, Dissolved-Winkler	G Bottle & top	Fix on site and store in dark	8 hours
Phenols	G	Cool, 4°C; H_2SO_4 to pH 2	28 days
Phosphorus, Total	P, G	Cool, 4°C; H_2SO_4 to pH 2	28 days
Residue, Total	P, G	Cool, 4°C	7 days
Residue, Filterable (TDS)	P, G	Cool, 4°C	7 days
Residue, Nonfilterable (TSS)	P, G	Cool, 4°C	7 days
Residue, Settleable	P, G	Cool, 4°C	48 hours
Residue, Volatile (Ash Content)	P, G	Cool, 4°C	7 days
Silica	P	Cool, 4°C	28 days
Specific Conductance	P, G	Cool, 4°C	28 days
Sulfate	P, G	Cool, 4°C	28 days

ATTACHMENT B
ADDITIONAL REQUIRED CONTAINERS, PRESERVATION TECHNIQUES, AND HOLDING TIMES
PAGE TWO

Parameter Number/Name	Container[1]	Preservation[2][3]	Maximum Holding Time[4]
INORGANIC TESTS (Cont'd):			
Sulfide	P, G	Cool, 4°C; add zinc acetate plus sodium hydroxide to pH 9	7 days
Sulfite	P, G	None required	Analyze immediately
Turbidity	P, G	Cool, 4°C	48 hours
METALS:[7]			
Chromium VI (Hexachrome)	P, G	Cool, 4°C	24 hours
Mercury (Hg)	P, G	HNO_3 to pH 2	28 days
Metals, except Chromium VI and Mercury	P, G	HNO_3 to pH 2	6 months
ORGANIC TESTS:[8]			
Purgeable Halocarbons	G, Teflon-lined septum	Cool, 4°C; 0.008% $Na_2S_2O_3$[5]	14 days
Purgeable Aromatic Hydrocarbons	G, Teflon-lined septum	Cool, 4°C; 0.008% $Na_2S_2O_3$[5] HCl to pH 2 [9]	14 days
Acrolein and Acrylonitrile	G, Teflon-lined septum	Cool, 4°C; 0.008% $Na_2S_2O_3$[5] adjust pH to 4-5 [10]	14 days
Phenols[11]	G, Teflon-lined cap	Cool, 4°C; 0.008% $Na_2S_2O_3$[5]	7 days until extraction; 40 days after extraction
Benzidines[11],[12]	G, Teflon-lined cap	Cool, 4°C; 0.008% $Na_2S_2O_3$[5]	7 days until extraction[13]
Phthalate esters[11]	G, Teflon-lined cap	Cool, 4°C	7 days until extraction; 40 days after extraction
Nitrosamines[11],[14]	G, Teflon-lined cap	Cool, 4°C; store in dark; 0.008% $Na_2S_2O_3$[5]	7 days until extraction; 40 days after extraction
PCBs[11]	G, Teflon-lined cap	Cool, 4°C	7 days until extraction; 40 days after extraction
Nitroaromatics & Isophorone[11]	G, Teflon-lined cap	Cool, 4°C; 0.008% $Na_2S_2O_3$[5]; store in dark	7 days until extraction; 40 days after extraction
Polynuclear Aromatic Hydrocarbons (PAHs)[11],[14]	G, Teflon-lined cap	Cool, 4°C; 0.008% $Na_2S_2O_3$[5]; store in dark	7 days until extraction; 40 days after extraction
Haloethers[11]	G, Teflon-lined cap	Cool, 4°C; 0.008% $Na_2S_2O_3$[5]	7 days until extraction; 40 days after extraction
Dioxin/Furan (TCDD/TCDF)[11]	G, Teflon-lined cap	Cool, 4°C; 0.008% $Na_2S_2O_3$[5]	7 days until extraction; 40 days after extraction

ATTACHMENT B
ADDITIONAL REQUIRED CONTAINERS, PRESERVATION TECHNIQUES, AND HOLDING TIMES
PAGE THREE

(1) Polyethylene (P): generally 500 ml or Glass (G): generally 1L.
(2) Sample preservation should be performed immediately upon sample collection. For composite chemical samples each aliquot should be preserved at the time of collection. When use of an automated sampler makes it impossible to preserve each aliquot, then chemical samples may be preserved by maintaining at 4°C until compositing and sample splitting is completed.
(3) When any sample is to be shipped by common carrier or sent through the United States Mail, it must comply with the Department of Transportation Hazardous Materials Regulations (49 CFR Part 172).
(4) Samples should be analyzed as soon as possible after collection. The times listed are the maximum times that samples may be held before analysis and still be considered valid. Samples may be held for longer periods only if the permittee, or monitoring laboratory, has data on file to show that the specific types of samples under study are stable for the longer periods, and has received a variance from the Regional Administrator.
(5) Should only be used in the presence of residual chlorine.
(6) Maximum holding time is 24 hours when sulfide is present. Optionally, all samples may be tested with lead acetate paper before pH adjustments are made to determine if sulfide is present. If sulfide is present, it can be removed by the addition of cadmium nitrate powder until a negative spot test is obtained. The sample is filtered and then NaOH is added to pH 12.
(7) Samples should be filtered immediately on site before adding preservative for dissolved metals.
(8) Guidance applies to samples to be analyzed by GC, LC, or GC/MS for specific compounds.
(9) Sample receiving no pH adjustment must be analyzed within 7 days of sampling.
(10) The pH adjustment is not required if acrolein will not be measured. Samples for acrolein receiving no pH adjustment must be analyzed within 3 days of sampling.
(11) When the extractable analytes of concern fall within a single chemical category, the specified preservative and maximum holding times should be observed for optimum safeguard of sample integrity. When the analytes of concern fall within two or more chemical categories, the sample may be preserved by cooling to 4°C, reducing residual chlorine with 0.008% sodium thiosulfate, storing in the dark, and adjusting the pH to 6-9; samples preserved in this manner may be held for 7 days before extraction and for 40 days after extraction. Exceptions to this optional preservation and holding time procedure are noted in footnote 5 (re: the requirement for thiosulfate reduction of residual chlorine) and footnotes 12, 13 (re: the analysis of benzidine).
(12) If 1,2-diphenylthydrazine is likely to be present, adjust the pH of the sample to 4.0±0.2 to prevent rearrangement to benzidine.
(13) Extracts may be stored up to 7 days before analysis if storage is conducted under an inert (oxidant-free) atmosphere.
(14) For the analysis of diphenylnitrosamine, add 0.008% $Na_2S_2O_3$ and adjust pH to 7-10 with NaOH within 24 hours of sampling.
(15) The pH adjustment may be performed upon receipt at the laboratory and may be omitted if the samples are extracted within 72 hours of collection. For the analysis of aldrin, add 0.008% $Na_2S_2O_3$.

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1.0 PURPOSE

The purpose of this Standard Operating Procedure (SOP) is to identify and designate the field data record forms, logs, and reports generally initiated and maintained for documenting Tetra Tech NUS, Inc. (TtNUS) field activities.

2.0 SCOPE

Documents presented within this SOP (or equivalents) shall be used for all TtNUS field activities, as applicable. Other or additional documents may be required by specific client contracts or project planning documents.

3.0 GLOSSARY

None.

4.0 RESPONSIBILITIES AND PERSONNEL QUALIFICATIONS

Project Manager (PM) - The PM is responsible for obtaining hardbound controlled-distribution logbooks (from the appropriate source), as needed. In addition, the Project Manager is responsible for placing all field documentation used in site activities (i.e., records, field reports, sample data sheets, field notebooks, and the site logbook) in the project's central file upon the completion of field work.

Field Operations Leader (FOL) - The FOL is responsible for ensuring that the site logbook, notebooks, and all appropriate and current forms and field reports included in this SOP (and any additional forms required by the contract) are correctly used, accurately filled out, and completed in the required time frame.

General personnel qualifications for field documentation activities include the following:

- Occupational Safety and Health Administration (OSHA) 40-hour and applicable refresher training.
- Capability of performing field work under the expected physical and environmental (i.e., weather) conditions.
- Familiarity with appropriate procedures for documentation, handling, packaging, and shipping.

5.0 PROCEDURES

5.1 SITE LOGBOOK

5.1.1 General

The site logbook is a hard-bound, paginated, controlled-distribution record book in which all major on-site activities are documented. At a minimum, record or reference the following activities/events (daily) in the site logbook:

- All field personnel present
- Arrival/departure times and names of site visitors
- Times and dates of health and safety training
- Arrival/departure times of equipment
- Times and dates of equipment calibration

- Start and/or completion of borehole, trench, monitoring well installation activities, etc.
- Daily on-site activities
- Sample pickup information
- Health and safety issues (level of protection, personal protective equipment [PPE], etc.)
- Weather conditions

Maintain a site logbook for each project and initiate it at the start of the first on-site activity (e.g., site visit or initial reconnaissance survey). Make entries every day that on-site activities take place involving TtNUS or subcontractor personnel. Upon completion of the fieldwork, provide the site logbook to the PM or designee for inclusion in the project's central file.

Record the following information on the cover of each site logbook:

- Project name
- TtNUS project number
- Sequential book number
- Start date
- End date

Information recorded daily in the site logbook need not be duplicated in other field notebooks (see Section 5.2) but must summarize the contents of these other notebooks and refer to specific page locations in these notebooks for detailed information (where applicable). An example of a typical site logbook entry is shown in Attachment A.

If measurements are made at any location, either record the measurements and equipment used in the site logbook or reference the field notebook in which the measurements are recorded (see Attachment A).

Make all logbook, notebook, and log sheet entries in indelible ink (black pen is preferred). No erasures are permitted. If an incorrect entry is made, cross out the entry with a single strike mark, initial, and date it. At the completion of entries by any individual, the logbook pages used must be signed and dated by the person making the entries. The site logbook must also be signed by the FOL at the end of each day.

5.1.2 Photographs

Sequentially number movies, slides, or photographs taken of a site or any monitoring location to correspond to logbook/notebook entries. Enter the name of the photographer, date, time, site location, site description, and weather conditions in the logbook/notebook as the photographs are taken. A series entry may be used for rapid-sequence photographs. The photographer is not required to record the aperture settings and shutter speeds for photographs taken within the normal automatic exposure range. However, special lenses, films, filters, and other image-enhancement techniques must be noted in the logbook/notebook. If possible, such techniques shall be avoided because they can adversely affect the accuracy of photographs. Chain-of-custody procedures depend on the subject matter, type of camera (digital or film), and the processing it requires. Follow chain-of-custody procedures for film used for aerial photography, confidential information, or criminal investigation. After processed, consecutively number the slides of photographic prints and label them according to the logbook/notebook descriptions. Docket the site photographs and associated negatives and/or digitally saved images to compact disks into the project's central file.

5.2 FIELD NOTEBOOKS

Key field team personnel may maintain a separate dedicated field notebook to document the pertinent field activities conducted directly under their supervision. For example, on large projects with multiple investigative sites and varying operating conditions, the Health and Safety Officer may elect to maintain a

separate field notebook. Where several drill rigs are in operation simultaneously, each site geologist assigned to oversee a rig must maintain a field notebook.

5.3 FIELD FORMS

All TtNUS field forms (see list in Section 6.0 of this SOP) can be found on the company's intranet site (http://intranet.ttnus.com) under Field Log Sheets. Forms may be altered or revised for project-specific needs, subject to client approval. Care must be taken to ensure that all essential information can be documented. Guidelines for completing these forms can be found in the related sampling SOPs.

5.3.1 Sample Collection, Labeling, Shipment, Request for Analysis, and Field Test Results

5.3.1.1 Sample Log Sheet

Sample log sheets are used to record specified types of data while sampling. The data recorded on these sheets are useful in describing the sample as well as pointing out any problems, difficulties, or irregularities encountered during sampling. Complete a sample log sheet for each sample obtained, including field quality control (QC) samples.

5.3.1.2 Sample Label

A typical sample label is illustrated in Attachment B. Complete the required information on the adhesive labels and apply them to every sample container. Obtain sample labels from the appropriate program/project source, request that they be electronically generated in house, or request them the laboratory subcontractor.

5.3.1.3 Chain-of-Custody Record

The chain-of-custody record is a multi-part form that is initiated as samples are acquired and accompanies a sample (or group of samples) as they are transferred from person to person. This form must be used as follows for any samples collected for chemical or geotechnical analysis whether the analyses are performed on site or off site:

- Retain one carbonless copy of the completed chain-of custody form in the field.
- Send one copy is sent to the PM (or designee)
- Send the original to the laboratory with the associated samples. Place the original (top, signed copy) of the chain-of custody form inside a large Ziploc®-type bag taped inside the lid of the shipping cooler. If multiple coolers are sent but are included on one chain-of custody form, send the form with the cooler containing vials for volatile organic compound (VOC) analysis or the cooler with the air bill attached. Indicate on the air bill how many coolers are included with that shipment.

An example of a chain-of-custody form is provided as Attachment C. After the samples are received at the laboratory, the sample cooler and contents are checked and any problems are noted on the enclosed chain-of custody form (any discrepancies between the sample labels and chain-of custody form and any other problems that are noted are resolved through communication between the laboratory point-of-contact and the TtNUS PM). The chain-of custody form is signed and copied. The laboratory will retain the copy, and the original becomes part of the samples' corresponding analytical data package.

5.3.1.4 Chain-of-Custody Seal

Attachment D is an example of a custody seal. The custody seal is an adhesive-backed label that is part of a chain-of-custody process and is used to prevent tampering with samples after they have been collected in the field and sealed in coolers for transport to the laboratory. Sign and date custody seals

and affix them across the lid and body of each cooler (front and back) containing environmental samples (see SOP SA-6.1). Obtain custody seals from the laboratory (if available) or purchase them from a supplier.

5.3.1.5 Geochemical Parameters Log Sheets

Complete Field Analytical Log Sheets to record geochemical and/or natural attenuation field test results.

5.3.2 Hydrogeological and Geotechnical Forms

5.3.2.1 Groundwater Level Measurement Sheet

Complete a Groundwater Level Measurement Sheet for each round of water level measurements made at a site.

5.3.2.2 Data Sheet for Pumping Test

During the performance of a pumping test (or an in situ hydraulic conductivity test), a large amount of data must be recorded, often within a short time period. Use a Pumping Test Data Sheet to facilitate this task by standardizing the data collection format for the pumping well and observation wells, and allowing the time interval for collection to be established in advance.

5.3.2.3 Packer Test Report Form

Complete a Packer Test Report Form for each well at which a packer test is conducted.

5.3.2.4 Boring Log

Complete a Summary Log of Boring, or Boring Log for each soil boring performed to document the materials encountered, operation and driving of casing, and locations/depths of samples collected. In addition, if volatile organics are monitored on cores, samples, cuttings from the borehole, or breathing zone, (using a photoionization detector [PID] or flame ionization detector [FID]), enter these readings on the boring log at the appropriate depth. When they become available, enter the laboratory sample number, concentrations of key contaminants, or other pertinent information in the "Remarks" column. This feature allows direct comparison of contaminant concentrations with soil characteristics.

5.3.2.5 Monitoring Well Construction Details Form

Complete a Monitoring Well Construction Details Form for every monitoring well, piezometer, or temporary well point installed. This form contains specific information on length and type of well riser pipe and screen, backfill, filter pack, annular seal and grout characteristics, and surface seal characteristics. This information is important in evaluating the performance of the monitoring well, particularly in areas where water levels show temporal variation or where there are multiple (immiscible) phases of contaminants. Depending on the type of monitoring well (in overburden or bedrock, stick-up or flush mount), different forms are used.

5.3.2.6 Test Pit Log

When a test pit or trench is constructed for investigative or sampling purposes, a Test Pit Log must be filled out by the responsible field geologist or sampling technician.

5.3.2.7 Miscellaneous Monitoring Well Forms

Miscellaneous monitoring well forms that may be required on a project-specific basis include the Monitoring Well Materials Certificate of Conformance and Monitoring Well Development Record. Use a Monitoring Well Materials Certificate of Conformance to document all materials utilized during each monitoring well installation. Use a Monitoring Well Development Record to document all well development activities.

5.3.2.8 Miscellaneous Field Forms – Quality Assurance and Checklists

Miscellaneous field forms/checklists forms that may be required on a project-specific basis include the following:

- Container Sample and Inspection Sheet – use this form when a container (drum, tank, etc.) is sampled and/or inspected.
- QA Sample Log Sheet – use this form when a QA sample such as an equipment rinsate blank, source blank, etc. is collected.
- Field Task Modification Request (FTMR) – use this form to document deviations from the project planning documents. The FOL is responsible for initiating the FTMRs. Maintain copies of all FTMRs with the on-site planning documents, and place originals in the final evidence file.
- Field Project Daily Activities Checklist and Field Project Pre-Mobilization Checklist – used these during both the planning and field effort to ensure that all necessary tasks are planned for and completed. These two forms are not requirements but are useful tools for most field work.

5.3.3 Equipment Calibration and Maintenance Form

The calibration or standardization of monitoring, measuring, or test equipment is necessary to ensure the proper operation and response of the equipment, to document the accuracy, precision, or sensitivity of the measurements, and determine if correction should be applied to the readings. Some items of equipment require frequent calibration, others infrequent. Some are calibrated by the manufacturer, others by the user.

Each instrument requiring calibration has its own Equipment Calibration Log, which documents that the manufacturer's instructions were followed for calibration of the equipment, including frequency and type of standard or calibration device. Maintain an Equipment Calibration Log for each electronic measuring device used in the field; make entries for each day the equipment is used or in accordance with manufacturer recommendations.

5.4 FIELD REPORTS

The primary means of recording on-site activities is the site logbook. Other field notebooks may also be maintained. These logbooks and notebooks (and supporting forms) contain detailed information required for data interpretation or documentation but are not easily used for tracking and reporting of progress. Furthermore, the field logbook/notebooks remain on site for extended periods of time and are thus not accessible for timely review by project management. Other reports useful for tracking and reporting the progress of field activities are described below.

5.4.1 Daily Activities Report

To provide timely oversight of on-site contractors, complete and submit Daily Activities Reports (DARs) as described below.

5.4.1.1 Description

The DAR documents the activities and progress for each day's field work. Complete this report on a daily basis whenever there are drilling, test pitting, well construction, or other related activities occurring that involve subcontractor personnel. These sheets summarize the work performed and form the basis of payment to subcontractors. The DAR form can be found on the TtNUS intranet site.

5.4.1.2 Responsibilities

It is the responsibility of the rig geologist to complete the DAR and obtain the driller's signature acknowledging that the times and quantities of material entered are correct.

5.4.1.3 Submittal and Approval

At the end of the shift, the rig geologist must submit the DAR to the FOL for review and filing. The Daily Activities Report is not a formal report and thus requires no further approval. The DARs are retained by the FOL for use in preparing the site logbook and in preparing weekly status reports for submission to the PM.

5.4.2 Weekly Status Reports

To facilitate timely review by project management, photocopies of logbook/notebook entries may be made for internal use.

In addition to those described herein, other summary reports may also be contractually required.

All TtNUS field forms can be found on the company's intranet site at http://intranet.ttnus.com under Field Log Sheets.

6.0 LISTING OF FIELD FORMS ON THE TtNUS INTRANET SITE

- Boring Log
- Container Sample and Inspection Sheet
- Daily Activities Checklist
- Daily Activities Record
- Equipment Calibration Log
- Field Task Modification Request
- Field Analytical Log sheet - Geochemical Parameters
- Groundwater Level Measurement Sheet
- Groundwater Sample Log Sheet
- Hydraulic Conductivity Test Data Sheet
- Low Flow Purge Data Sheet
- Bedrock Monitoring Well Construction (Stick Up)
- Bedrock Monitoring Well Construction Flush Mount
- Bedrock Monitoring Well Construction Open Hole
- Confining Layer Monitoring Well Construction
- Monitoring Well Development Record

- Monitoring Well Materials Certificate of Conformance
- Overburden Monitoring Well Construction Flush Mount
- Overburden Monitoring Well Construction Stick Up
- Packer Test Report Form
- Pumping Test Data Sheet
- QA Sample Log Sheet
- Soil/Sediment Sample Log Sheet
- Surface Water Sample Log Sheet
- Test Pit Log
- Field Project Pre-Mobilization Checklist

ATTACHMENT A
TYPICAL SITE LOGBOOK ENTRY

START TIME: _____ DATE: _____

SITE LEADER: _____
PERSONNEL:

TtNUS	DRILLER	SITE VISITORS
_____	_____	_____
_____	_____	_____
_____	_____	_____

WEATHER: Clear, 68°F, 2-5 mph wind from SE

ACTIVITIES:

1. Steam jenney and fire hoses were set up.
2. Drilling activities at well ____ resumes. Rig geologist was _____. See Geologist's Notebook, No. 1, page 29-30, for details of drilling activity. Sample No. 123-21-S4 collected; see sample logbook, page 42. Drilling activities completed at 11:50 and a 4-inch stainless steel well installed. See Geologist's Notebook, No. 1, page 31, and well construction details for well _____.
3. Drilling rig No. 2 steam-cleaned at decontamination pit. Then set up at location of well _____.
4. Well _____ drilled. Rig geologist was _____. See Geologist's Notebook, No. 2, page ____ for details of drilling activities. Sample numbers 123-22-S1, 123-22-S2, and 123-22-S3 collected; see sample logbook, pages 43, 44, and 45.
5. Well _____ was developed. Seven 55-gallon drums were filled in the flushing stage. The well was then pumped using the pitcher pump for 1 hour. At the end of the hour, water pumped from well was "sand free."
6. EPA remedial project manger arrives on site at 14:25 hours.
7. Large dump truck arrives at 14:45 and is steam-cleaned. Backhoe and dump truck set up over test pit _____.
8. Test pit _____ dug with cuttings placed in dump truck. Rig geologist was _____. See Geologist's Notebook, No. 1, page 32, for details of test pit activities. Test pit subsequently filled. No samples taken for chemical analysis. Due to shallow groundwater table, filling in of test pit ___ resulted in a very soft and wet area. A mound was developed and the area roped off.
9. Express carrier picked up samples (see Sample Logbook, pages 42 through 45) at 17:50 hours. Site activities terminated at 18:22 hours. All personnel off site, gate locked.

_____

Field Operations Leader

ATTACHMENT B
SAMPLE LABEL

Tetra Tech NUS, Inc. 661 Andersen Drive Pittsburgh, 15220 (412)921-7090	**Project:** **Site:** **Location:**	
Sample No:		**Matrix:**
Date:	**Time:**	**Preserve:**
Analysis:		
Sampled by:	**Laboratory:**	

ATTACHMENT C
CHAIN-OF-CUSTODY RECORD FORM

TETRA TECH NUS, INC. CHAIN OF CUSTODY | NUMBER 3413 | PAGE ___ OF _____

PROJECT NO:	FACILITY:	PROJECT MANAGER	PHONE NUMBER	LABORATORY NAME AND CONTACT:
SAMPLERS (SIGNATURE)		FIELD OPERATIONS LEADER	PHONE NUMBER	ADDRESS
		CARRIER/WAYBILL NUMBER		CITY, STATE

STANDARD TAT ☐
RUSH TAT ☐
☐ 24 hr. ☐ 48 hr. ☐ 72 hr. ☐ 7 day ☐ 14 day

CONTAINER TYPE PLASTIC (P) or GLASS (G)
PRESERVATIVE USED
TYPE OF ANALYSIS

DATE YEAR ___	TIME	SAMPLE ID	LOCATION ID	TOP DEPTH (FT)	BOTTOM DEPTH (FT)	MATRIX (GW, SO, SW, SD, QC, ETC.)	COLLECTION METHOD GRAP (G) COMP (C)	No. OF CONTAINERS									COMMENTS

1. RELINQUISHED BY	DATE	TIME	1. RECEIVED BY	DATE	TIME
2. RELINQUISHED BY	DATE	TIME	2. RECEIVED BY	DATE	TIME
3. RELINQUISHED BY	DATE	TIME	3. RECEIVED BY	DATE	TIME

COMMENTS

DISTRIBUTION: WHITE (ACCOMPANIES SAMPLE) YELLOW (FIELD COPY) PINK (FILE COPY)

4/02R
FORM NO. TtNUS-001

ATTACHMENT D
CHAIN-OF-CUSTODY SEAL

CUSTODY SEAL	**CUSTODY SEAL**
Date	**Date**
Signature	**Signature**

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ATTACHMENTS

A INVESTIGATION-DERIVED WASTE LABEL 15

1.0 PURPOSE

Decontamination is the process of removing and/or neutralizing site contaminants that have contacted and/or accumulated on equipment. The purpose of this Standard Operating Procedure (SOP) is to protect site personnel, the general public, and the environment while preserving or maintaining sample integrity. It is further intended through this procedure to describe the steps necessary for proper decontamination of drilling equipment, earth-moving equipment , chemical sampling equipment and field operation and analytical equipment.

2.0 SCOPE AND APPLICABILITY

This procedure applies to all equipment used to provide access to/acquire environmental samples that may have become contaminated through direct contact with contaminated media including air, water, and soil. This equipment includes drilling and heavy equipment and chemical sampling and field analytical equipment. Where technologically and economically feasible, single-use sealed disposable equipment will be employed to minimize the potential for cross-contamination. This SOP also provides general reference information on the control of contaminated materials.

Decontamination methods and equipment requirements may differ from one project to another. General equipment items are specified in Section 6.0, but project-specific equipment must be obtained to address the project-specific decontamination procedures presented in Section 7.0 and applicable subsections.

3.0 GLOSSARY

Alconox/Liquinox - A brand of phosphate-free laboratory-grade detergent.

Decontamination Solution - A solution selected/identified in the Health and Safety Plan or Project-Specific Quality Assurance Plan. The solution is selected and employed as directed by the project chemist/health and safety professional.

Deionized Water (DI) - Tap water that has been treated by passing through a standard deionizing resin column. This water may also pass through additional filtering media to attain various levels of analyte-free status. The DI water should meet College of American Pathologists (CAP) and National Committee for Clinical Laboratory Standards (NCCLS) specifications for reagent-grade Type I water.

Potable Water - Tap water from any municipal water treatment system. Use of an untreated potable water supply is not an acceptable substitute for tap water.

Pressure Washing - Process employing a high-pressure pump and nozzle configuration to create a high-pressure spray of potable water. High-pressure spray is employed to remove solids from equipment.

Solvent – A liquid in which solid chemicals or other liquids are dissolved. The solvent of choice is pesticide-grade isopropanol. Use of other solvents (methanol, acetone, or hexane) may be required for particular projects or for a particular purpose (e.g., removal of concentrated waste) and must be justified in the project planning documents. For example, it may be necessary to use hexane when analyzing for trace levels of pesticides, PCBs, or fuels. In addition, because many of these solvents are not miscible in water, the equipment should be air dried prior to use. Solvents should not be used on PVC equipment or well construction materials.

Steam Pressure Washing - A cleaning method employing a high-pressure spray of heated potable water to remove various organic/inorganic chemicals from equipment.

4.0 RESPONSIBILITIES AND PERSONNEL QUALIFICATIONS

Project Manager - Responsible for ensuring that all field activities are conducted in accordance with approved project plan(s) requirements.

Decontamination Personnel - Individuals assigned the task of decontamination. It is the responsibility of these individuals to understand the use and application of the decontamination process and solutions as well as the monitoring of that process to ensure that it is working properly. This is accomplished through visual evaluation, monitoring instrument scanning of decontaminated items, and/or through the collection of rinsate blanks to verify contaminant removal.

Field Operations Leader (FOL) - Responsible for the implementation of project-specific planning documents. This includes on-site verification that all field activities are performed in compliance with approved SOPs or as otherwise dictated by the approved project plan(s). The FOL is also responsible for the completion and accuracy of all field documentation.

Site Safety Officer (SSO) - Exercises shared responsibility with the FOL concerning decontamination effectiveness. All equipment arriving on site (as part of the equipment inspection), leaving the site, and moving between locations is required to go through a decontamination evaluation. This is accomplished through visual examination and/or instrument screening to determine the effectiveness of the decontamination process. Improper or incomplete decontamination is sufficient to restrict equipment from entering the site, exiting the site, or moving to a new location on the site until the objectives are successfully completed.

General personnel qualifications for decontamination activities include the following:

- Occupational Safety and Health Administration (OSHA) 40-hour and applicable refresher training.
- Capability of performing field work under the expected physical and environmental (i.e., weather) conditions.
- Familiarity with appropriate decontamination procedures.

5.0 HEALTH AND SAFETY

In addition to the health and safety issues and reminders specified in subsections of this SOP, the following considerations and requirements must be observed as SOPs for field equipment decontamination activities:

- If any solvents or hazardous chemicals (e.g., isopropyl alcohol) are to be used in equipment decontamination activities, the FOL must first obtain the manufacturer's/supplier's Material Safety Data Sheet (MSDS) and assure that it is reviewed by all users (prior to its use), added to the site Hazardous Chemical Inventory, and maintained on site as part of the project Hazard Communication Program.
- Review and observe specific health and safety requirements (e.g., personal protective equipment [PPE]) specified in the project-specific health and safety plan for this activity.

6.0 EQUIPMENT LIST

- Wood for decontamination pad construction, when applicable (see Section 7.1).

- Tools for constructing decontamination pad frame, when applicable (see Section 7.1).
- Visqueen sheeting or comparable material to cover decontamination pad frame, when applicable (see Section 7.1).
- Wash/drying racks for auger flights and drill/drive rods, when applicable (see Section 7.2).
- PPE as specified in the project health and safety plan.
- Soap and water for washing and rinsing.
- Deionized water for final rinsing.
- Solvents (e.g., pesticide-grade isopropanol) for rinsing (see applicable portions of Section 7.2).
- Tubs, buckets, etc. for containerizing rinse water (see applicable portions of Section 7.2).
- Sample bottles for collecting rinsate blanks (see Section 7.2).
- Calibrated photoionization detector (PID) or flame ionization detector (FID) to monitor decontaminated equipment for organic vapors generated through the existence of residual contamination or the presence of decontamination solvent remaining after the piece was rinsed.
- Aluminum foil or clear clean plastic bag for covering cleaned equipment (see applicable portions of Section 7.2).
- Paper towels or cloths for wiping.
- Brushes, scrapers, or other hand tools useful for removing solid materials from equipment.
- Clear plastic wrap for covering or wrapping large decontaminated equipment items (see Section 7.2.2).
- Drum-moving equipment for moving filled waste drums (optional) (see Section 7.3).
- Drum labels for waste drums (see Attachment A).

7.0 PROCEDURES

The process of decontamination is accomplished through the removal of contaminants, neutralization of contaminants, or isolation of contaminants. To accomplish this activity, preparation is required including site preparation, equipment selection, and evaluation of the decontamination requirements and processes. Site contaminant types, concentrations, and media types are primary drivers in the selection of the types of decontamination and where it will be conducted. For purposes of this SOP, discussion is limited to decontamination procedures for general environmental investigations.

Decontamination processes will be performed at the location(s) specified in project-specific planning documents. Typical decontamination locations include the following:

- Temporary decontamination pads/facilities
- Sample locations
- Centralized decontamination pad/facilities

- Combination of some or all of the above

The following discussion includes general considerations for the decontamination process. Specific construction and implementation procedures will be as specified in the project-specific planning documents and/or may be as dictated by site-specific conditions as long as the intent of the requirements in the planning documents is met. This intent is to contain any residual fluids and solids generated through the decontamination process.

7.1 Decontamination Pad Design/Construction Considerations

7.1.1 Temporary Decontamination Pads

Temporary decontamination pads may be constructed at satellite locations within the site area in support of temporary work areas. These structures are generally constructed to support the decontamination of heavy equipment such as drill rigs and earth-moving equipment but can be employed for smaller articles.

The purpose of the decontamination pad is to contain wash waters and potentially contaminated soil generated during decontamination procedures. Therefore, construction of these pads should take into account the following considerations:

- Site location – The decontamination site selected should be far enough from the work site to maximize decontamination effectiveness while minimizing travel distance. The location of the decontamination site shall be selected to provide, in the judgment of the FOL or FOL designee, compliance with as many of the following characteristics as practicable:
  - Well removed from pedestrian/vehicle thoroughfares.
  - Avoidance of areas where control/custody cannot be maintained.
  - Avoidance of areas where potential releases of contaminated media or decontamination fluids may be compounded through access to storm water transport systems, streams, or other potentially sensitive areas.
  - Avoidance of potentially contaminated areas.
  - Avoidance of areas too close to the ongoing operation, where cross-contamination may occur.

  The selected decontamination site should include the following, where possible:

  - Areas where potable water and electricity are provided.

Safety Reminder

When utilizing electrical power sources, either hard-wired or portable-generated sources, ensure that:

- All power is routed through a Ground Fault Circuit Interrupter (GFCI).
- All power cords are in good condition (no physical damage), rated for the intended energy load, and designated for outdoor use.

In situations where accomplishing these elements is not possible, it will be necessary to implement a site electrical grounding program.

- Areas where support activities such as removing decontamination waters soil and sediment are possible without entering an active exclusion zone.

- Areas that offer sufficient size to carry out the specific decontamination sequence.

- Decontamination pad (decon pad) – The decon pad shall be constructed to meet the following characteristics:

  - Size – The size of the pad should be sufficient to accept the equipment to be decontaminated as well as permitting free movement around the equipment by the personnel conducting the decontamination. The size should permit these movements utilizing pressure/steam washer wands and hoses and minimizing splash due to work in close quarters.

  - Slope – An adequate slope will be constructed to permit the collection of water and potentially contaminated soil within a trough or sump constructed at one end. The collection point for wash waters should be of adequate distance that the decontamination workers do not have to walk through the wash waters while completing their tasks. Because the pad will be sloped, place a light coating of sand over the plastic to minimize potential slips and falls. See the text about liners below.

  - Sidewalls – The sidewalls shall be at least 6 inches in height (or as high as possible if 6 inches is not achievable) to provide adequate containment for wash waters and soil. If splash represents a potential problem, splash guards should be constructed to control overspray. Sidewalls may be constructed of wood, inflatables, sand bags, etc. to permit containment. Splash guards are typically wood frames with Visqueen coverings to control overspray.

  - Liner – Depending on the types of equipment and decontamination method to be used, the liner should be of sufficient thickness to provide a puncture-resistant barrier between the decontamination operation and the unprotected environment. Care should be taken to examine the surface area prior to placing the liner to remove sharp articles (sticks, stones, debris) that could puncture the liner. Liners are intended to form an impermeable barrier. The thickness may vary from a minimum recommended thickness of 10 mil to 30 mil. The desired thickness may be achieved through layering materials of lighter construction. It should be noted that various materials (rubber, polyethylene sheeting) become slippery when wet. To minimize this potential hazard associated with a sloped liner, a light coating of sand shall be applied to provide traction as necessary.

  - Wash/drying racks – Auger flights, drill/drive rods, and similar equipment require racks positioned off of the ground to permit these articles to be washed, drained, and dried while secured from falling during this process.

For decontamination of direct-push technology (DPT) equipment, the pad may be as simple as a mortar tub containing buckets of soapy water for washing and an empty bucket to capture rinse waters. Decontamination may be conducted at the rear of the rig to permit rapid tool exchange.

- Maintenance – Maintain the decontamination area by:

  - Periodically clearing the work area of standing water, soil, and debris, and coiling hoses to aid in eliminating slip, trip, and fall hazards. In addition, these articles will reduce potential backsplash and cross-contamination.

- Regularly changing the decontamination fluids to ensure proper cleaning and prevent cross-contamination.

- PPE – Periodically evaluate the condition of, and maintain the decontamination equipment, including regular cleaning of face shields and safety glasses. This is critical to ensuring the safety of decontamination personnel and the integrity of the decontamination process, and it will ensure that equipment is functioning properly.

7.1.2 Decontamination Activities at Drill Rigs/DPT Units

During subsurface sampling activities including drilling and DPT activities, decontamination of drive rods, Macro Core Samplers, split spoons, etc. is typically conducted at an area adjacent to the operation. Decontamination is generally accomplished using a soap/water wash and rinse utilizing buckets and brushes. This area requires sufficient preparation to accomplish the decontamination objectives.

Buckets shall be placed within mortar tubs or similar secondary containment tubs to prevent splash and spills from reaching unprotected environmental media. Drying racks shall be employed as directed for temporary pads to permit parts to dry and be evaluated prior to use/reuse. Methodology regarding this activity is provided in Section 7.2.

7.1.3 Decontamination Activities at Remote Sample Locations

When sampling at remote locations, sampling equipment such as trowels and pumps/tubing should be evacuated of potentially contaminated media to the extent possible. This equipment should be wrapped in plastic for transport to the temporary/centralized decontamination location for final cleaning and disposition. Flushing and cleaning of single-use equipment such as disposable trowels, tubing, and surgeon's gloves may allow disposal of this equipment after visible soil and water remnants have been removed.

7.2 Equipment Decontamination Procedures

The following represents procedures to be employed for the decontamination of equipment that may have contacted and/or accumulated contamination through site investigation activities.

7.2.1 Monitoring Well Sampling Equipment

7.2.1.1 Groundwater sampling equipment – This includes pumps inserted into monitoring wells such as bladder pumps, Whale pumps, and Redi-Flo pumps and reusable bailers, etc.

1. Evacuate to the extent possible, any purge water within the pump/bailer.

2. Scrub using soap and water and/or steam clean the outside of the pump/bailer and, if applicable, the pump tubing.

3. Insert the pump and tubing/bailer into a clean container of soapy water. Pump/run a sufficient amount of soapy water through the pump/bailer to flush out any residual well water. After the pump is flushed, circulate soapy water through the pump to ensure that the internal components are thoroughly flushed.

4. Remove the pump and tubing/bailer from the container

5. Rinse external pump components using tap water.

6. Insert the pump and tubing/bailer into a clean container of tap water. Pump/run a sufficient amount of tap water through the pump/bailer to evacuate all of the soapy water (until clear).

CAUTION

Do not rinse PE, PVC, and associated tubing with solvents – Use the procedures defined in the project-specific planning documents. If they are not defined, contact the FOL for guidance. The solvent rinse described in Step 7 may be omitted if groundwater does not contain oil, grease, PAHs, PCBs, or other hard to remove organic materials.

7. If groundwater contains or is suspected to contain oil, grease, PAHs, PCBs, or other hard to remove organic materials, rinse the equipment to be cleaned with pesticide-grade isopropanol.

8. Pass deionized water through the hose to flush out the tap water and solvent residue as applicable.

9. Drain residual deionized water to the extent possible.

10. Allow components of the equipment to air dry.

11. For bladder pumps, disassemble the pump and wash the internal components with soap and water, then rinse with tap water, isopropanol, and deionized water and allow to dry. After the parts are dry, conduct a visual inspection and a monitoring instrument scan to ensure that potential contaminants and all decontamination solvent have been removed. Collect a rinsate blank in accordance with the project-specific planning documents to ensure that the decontamination process is functioning as intended. The typical frequency of collection for rinsate blanks is 1 per 20 field samples. In addition, wipe samples or field tests such as UV light may be used.

12. Wrap pump/bailer in aluminum foil or a clear clean plastic bag for storage.

SAFETY REMINDER

Remember when handling powered equipment to disconnect the power source and render the equipment to a zero energy state (both potential and kinetic) before opening valves, disconnecting lines, etc.

7.2.1.2 Electronic Water Level Indicators/Sounders/Tapes

During water level measurements, rinsing the extracted tape and probe with deionized water and wiping the surface of the extracted tape between locations is acceptable. However, periodic full decontamination should be conducted as follows:

1. Wash with soap and water
2. Rinse with tap water
3. Rinse with deionized water

NOTE

In situations where oil, grease, free product, other hard to remove materials are encountered, probes and exposed tapes should be washed in hot soapy water. If probes or tapes cannot be satisfactorily decontaminated (they are still stained, discolored, etc.), they should be removed from service.

7.2.1.3 Miscellaneous Equipment

Miscellaneous equipment including analytical equipment (water quality testing equipment) shall be cleaned per manufacturers' instructions. This generally includes wiping the sensor housing and rinsing with tap and deionized water.

Coolers/shipping containers employed to ship samples are received from the laboratory in a variety of conditions including marginal to extremely poor. Coolers shall be evaluated prior to use for the following:

- Structural integrity – Coolers missing handles or having breaks in the outer housing should be removed and not used. Notify the laboratory that the risk of shipping samples in the cooler(s) provided is too great and request a replacement unit.

- Cleanliness – As per protocol, only volatile organic samples are accompanied by a trip blank. If a cooler's cleanliness is in question (visibly dirty/stained) or if there are noticeable odors, the cooler should be decontaminated prior to use as follows:

  1. Wash with soap and water
  2. Rinse with tap water
  3. Dry

If these measures fail to clean the cooler to an acceptable level, remove the unit from use as a shipping container and ask the cooler provider (e.g., the analytical laboratory) to provide a replacement unit.

7.2.2 Downhole Drilling Equipment

This includes any portion of the drill rig that is over the borehole, including auger flights, drill stems, rods, and associated tooling that would extend over the borehole. The following procedure is to be employed prior to initiating the drilling/sampling activity, then between locations:

> ***CAUTION***
> Exercise care when using scrapers to remove soil and debris from downhole drilling equipment. Inadvertent slips of scrapers have resulted in cuts, scrapes, and injured knuckles, so use scrapers carefully when removing soil from these items.

1. Remove loose soil using shovels, scrapers, etc.

2. Through a combination of scrubbing using soap and water and/or steam cleaning or pressure washing, remove visible dirt/soil from the equipment being decontaminated.

> ***CAUTION***
> In Step 3, do not rinse PE, PVC, and associated tubing with solvents. The appropriate procedures should be defined within the project-specific planning documents. If they are not defined, contact the FOL for guidance. The solvent rinse described in Step 4 may be omitted if groundwater does not contain oil, grease, PAHs, PCBs, or other hard to remove organic materials.

3. Rinse the equipment with tap water, where applicable (steam cleaning and pressure washing incorporate rinsing as part of the process).

4. If the equipment has directly or indirectly contacted contaminated sample media and is known or suspected of being contaminated with oil, grease, PAHs, PCBs, or other hard to remove organic materials, rinse equipment with pesticide-grade isopropanol

5. To the extent possible, allow components to air dry.

6. If the decontaminated equipment is to be used immediately after decontamination, screen it with a calibrated photoionization detector (PID)/flame ionization detector (FID) to ensure that all contaminants and possible decontamination solvents (if they were used) have been adequately removed.

7. Wrap or cover equipment in clear plastic until it is time to be used.

SAFETY REMINDER

Even when equipment is disconnected from power sources, dangers such as the following may persist:

Falls - An auger flight standing on its end may fall and injure someone. Secure all loose articles to prevent heavy articles from falling onto people or equipment.

Burns - Steam cleaner water is heated to more than 212 °F and exhibits thermal energy that can cause burns. Prevent contact of skin with hot water or surfaces.

High water pressure - Pressure washer discharge can have 2,000 to 4,000 psi of water pressure. Water under this amount of pressure can rupture skin and other human tissues. Water at 4,000 psi exiting a 0° tip can be dangerous because of its relatively high cutting power. The exit velocity and cutting power of the water are reduced when exiting a 40° fan tip, but damage to soft tissues is still possible.

In general, follow the rules below to avoid injury, equipment damage, or incomplete decontamination:

1. Read the operating manual and follow the manufacturers' recommended safety practices before operating pressure washers and steam cleaners.

2. Never point the pressure washer or steam cleaner at another person or use to clean your boots or other parts of your body. Water lacerations and burns may appear to be minor at first but can be life threatening. Do not attempt to hold small parts in your hand while washing them with high-temperature or high-pressure water.

3. Always wear PPE as specified in the HASP such as:

   - Hard hat, safety glasses, splash shield, impermeable apron or splash suit, and hearing protection. Remember that excessive noise is a hazard when operating gas-powered engines and electrically driven pressure washers. PPE will be identified in your project specific planning documents.

4. Inspect each device before use. An inspection checklist will be provided in the project-specific planning documents. If it is a rented device, safety measures are typically provided by the vendor. In all cases, if you are not familiar with the operation of a pressure washer/steam cleaner, do not operate it until you obtain and thoroughly review operating instructions and recommended safety practices.

5. Do not modify equipment unless the manufacturer has approved the modifications.

7.2.3 Soil/Sediment Sampling Equipment

This section applies to soil sampling equipment including but not limited to hand augers, stainless steel trowels/spoons, bowls, dredges, scoops, split spoons, Macro Core samplers, etc.

1. Remove all loose soil from the equipment through manual means.
2. Through a combination of scrubbing using soap and water and/or steam cleaning or pressure washing, remove visible dirt/soil from the equipment.
3. Rinse the equipment with tap water.

> ***CAUTION***
> Do not rinse PE, PVC, and associated tubing with solvents. The appropriate procedures should be defined within the project-specific planning documents. If they are not defined, contact the FOL for guidance. The solvent rinse described in Step 4 may be omitted if groundwater does not contain oil, grease, PAHs, PCBs, or other hard to remove organic materials.

4. If the equipment is contaminated or suspected to be contaminated with oil, grease, PAHs, PCBs, or other hard to remove organic materials, rinse the equipment with pesticide-grade isopropanol.
5. Rinse the equipment with deionized water.
6. To the extent possible, allow components to air dry.
7. If the equipment is to be used immediately after decontamination, screen it with a calibrated PID/FID to ensure that all solvents (if they were used) and trace contaminants have been adequately removed.
8. After the equipment has dried, wrap it in aluminum foil for storage until use.

Dredges employed in sediment sampling are typically decontaminated as follows:

- Remove the sediment sample from the sampling device
- If sufficient associated surface water is available at the sampling site, place the dredge in the water and flush to remove visible sediment.
- Extract the dredge and wash it in soap and water per the project-specific planning documents.

> ***CAUTION***
> When handling dredges, the primary safety concern is trapping fingers or extremities in the larger dredge samplers within the jaws or pinch points of the mechanical jaws. Keep hands, fingers, and extremities away from these pinch and compression points. Either handle the device by the rope or preferably lock the jaws in place to control the potential for closing during maintenance and/or cleaning.

7.3 Contact Waste/Materials

During the course of field investigations, disposable/single-use equipment becomes contaminated. These items include tubing, trowels, PPE (gloves, overboots, splash suits, etc.), and broken sample containers.

With the exception of the broken glass, single-use articles should be cleaned (washed and rinsed) of visible materials and disposed as normal refuse. The exception to this rule is that extremely soiled materials that cannot be cleaned shall be containerized for disposal in accordance with applicable federal, state, and local regulations.

7.3.1 Investigation-Derived Wastes - Decontamination Wash Waters and Sediments

> ***NOTE***
> Requirements for waste storage may differ from one facility to the next. Facility-specific directions for waste storage areas will be provided in project-specific documents, or separate direction will be provided by the Project Manager.

1. Assume that all investigation-derived waste (IDW) generated from decontamination activities contains the hazardous chemicals associated with the site unless there are analytical or other data to the contrary. Waste solution volumes could vary from a few gallons to several hundred gallons in cases where large equipment required cleaning.

2. Where possible, use filtering systems to extend the use of water within a closed system wash unit to recycle water and to reduce possible waste amounts.

> ***NOTE***
> Containerized waste rinse solutions are best stored in 55-gallon drums (or equivalent containers) that can be sealed until ultimate disposal at an approved facility.

3. Label waste storage containers appropriately labeled (see Attachment A).

4. Ensure that the IDW storage area is configured to meet the following specifications to permit access to the containers and to conduct spill/leak monitoring, sampling, and extraction when the disposal route is determined:

   - Enclose areas accessible by the general public using construction fencing and signs.
   - Stored materials in 55-gallon drums on pallets with four (or fewer) drums per pallet.
   - Maintain the retaining bolt and label on the outside of storage containers where readily visible.
   - Provide at least 4 feet of room between each row of pallets to allow access to containers for sampling, drum removal, and spill response.
   - As directed in project-specific planning documents, maintain an IDW Inventory List and provide the list to the site Point of Contact at the termination of each shift.
   - Maintain spill response equipment at the IDW storage area in case it is required for immediate access.

- Where possible, use equipment for moving containers. Where not possible, obtain help to manipulate containers.

CAUTION

Each container of water can weigh up to 490 pounds. Each 55-gallon drum of wet soil can weigh more than 750 pounds. Fill drums and temporary containers to 80 percent capacity to minimize spill and handling difficulties. Use drum carts to move filled drums.

See safe lifting techniques provided in Section 4.4 of the Tetra Tech NUS, Inc. Health and Safety Guidance Manual.

When placing drums, keep your fingers out of pinch and smash points such as between the drums. In some cases such as well development and/or purge water, you can place the drums to be filled on the pallet and transport materials in smaller easier to handle containers.

7.4 Decontamination Evaluation

Upon decontamination of equipment, determine the effectiveness of the decontamination process in the following manner:

- Visual evaluation – A visual evaluation will be conducted to ensure the removal of particulate matter. This shall be done to ensure that the washing/rinsing process is working as intended.

- Instrument Screening – A properly calibrated PID/FID should be used to evaluate the presence of site contaminants and solvents used in the cleaning process. The air intake of the instrument shall be passed over the article to be evaluated. Avoid placing the instrument probe into residual waters. A PID/FID reading greater than the daily established background level requires a repeat of the decontamination process, followed by rescreening with the PID/FID. This sequence must be repeated until no instrument readings greater than the daily established background level are observed. It should be noted that the instrument scan is only viable if the contaminants are detectable within the instrument's capabilities.

NOTE

When required by project-specific planning documents, collection of rinsate blanks (see next step) shall be completed without exception unless approval to not collect these samples is obtained from the Project Manager.

- Collection of Rinsate Blanks – It is recommended that rinsate samples be collected to:

  - Evaluate the decontamination procedure representing different equipment applications (pumps versus drilling equipment) and different decontamination applications.

  - Single-use disposable equipment – The number of samples should represent different types of equipment as well as different lot numbers of single-use articles.

  - The collection and the frequency of collection of rinsate samples are as follows unless specified differently in the project-specific planning documents:

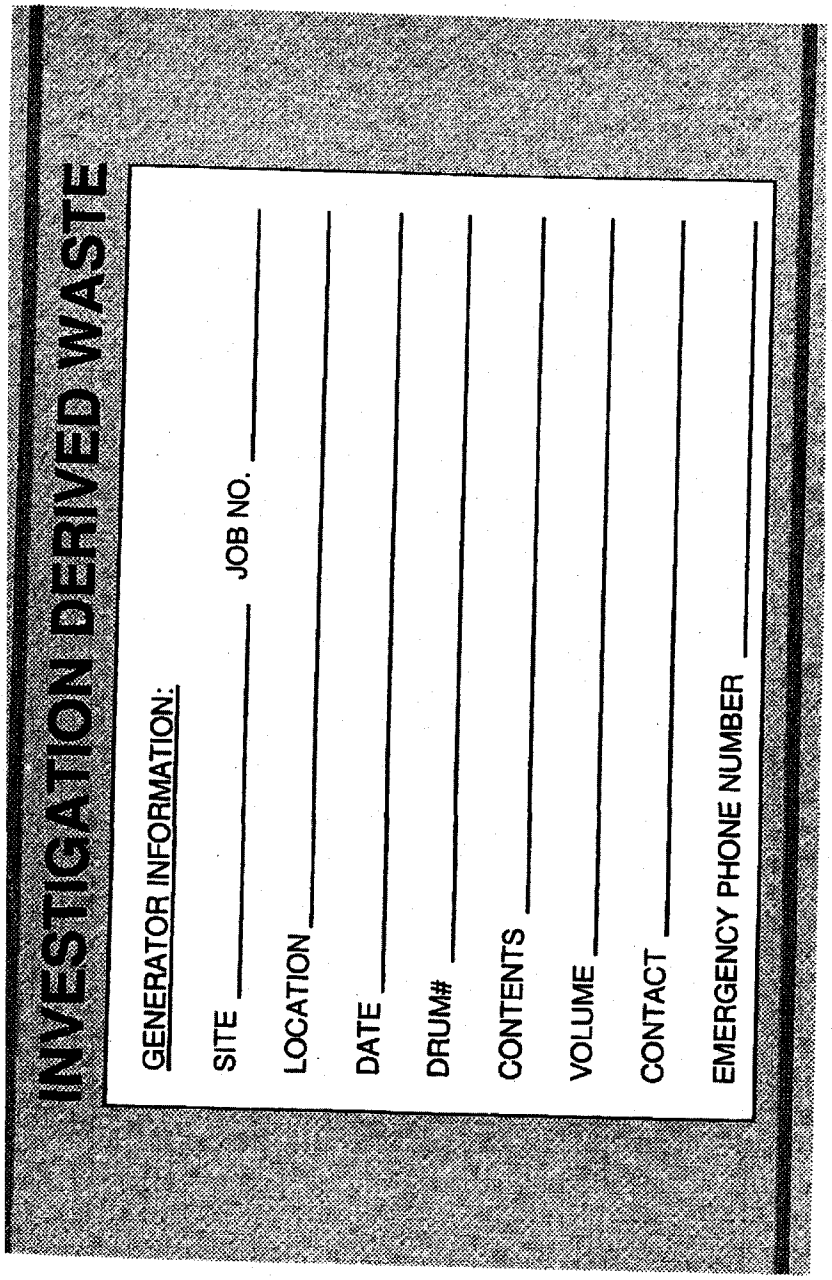
    - Per decontamination method
    - Per disposable article/batch number of disposable articles

NOTE

It is recommended that an initial rinsate sample be collected early in the project to ensure that the decontamination process is functioning properly and to avoid using a contaminated batch of single-use articles. It is recommended that a follow-up sample be collected later during the execution of the project to ensure that those conditions do not change.

Rinsate samples collection may be driven by types of and/or levels of contaminant. Difficult to remove contaminants, oils/greases, some PAHs/PCBs, etc. may also support the collection of additional rinsates due to the obvious challenges to the decontamination process. This is a field consideration to be determined by the FOL.

Attachment A
iDW Label



APPENDIX D

FIELD FORMS

Tetra Tech NUS, Inc.

PROJECT: _____ LOCATION: _____

JOB & CTO #: _____ MOBILIZATION DATE: _____

PROJECT MANAGER: _____ RETURN DATE: _____

FIELD PROJECT PRE-MOBILIZATION CHECKLIST

TRAVEL

___ Airline reservations
___ Hotel reservations/BOQs
___ Vehicle rental
___ Itinerary
___ Phone/pager number

DRILLING/DPT/SURVEY

Subcontractor

___ POC phone #/address
___ Drill Specification RFP
___ Contact (time & place to meet)
___ Confirm subcontract w/ TtNUS Procurement
___ Health and Safety documentation for all personnel on site
___ Copy of Drillers license
___ Well / boring permits

Utilities (2 weeks lead time)

___ Contact Site POC (Date:_____)
___ Contact Local "Call Before You Dig"
___ Utility Clearance Form

Forms

___ Boring logs / Test Pit logs
___ Well construction / development forms
___ Daily activity forms
___ IDW inventory
___ IDW drum labels
___ Chemical Inventory
___ MSDS's

EQUIPMENT MOBILIZATION

___ Equipment Requisition form completed / equipment ordered
___ 3rd Party rental / misc. equipment ordered
___ Equipment calibration forms
___ Span / calibration gas and regulator

SAMPLING

Forms

___ Sample log sheets
___ Low-flow purge data sheets
___ COC records
___ COC seals
___ Sample labels (from database group)

Laboratory

___ POC address/phone#
___ Order bottles / preservatives
___ Shipping address, also check Sat. address
___ Bottle & preservation req'ts from lab
___ _____

MISCELLANEOUS

Schedule

___ Plan field operations w/ Project manager

Documents for Field Program

___ Logbook(s)
___ Field Sampling plan
___ Health & Safety plan
___ Maps
___ H & S Guidance Manual

Authorization

___ Kick-off meeting held
___ Gov't rate letter
___ H&S/OSHA 40-hour certifcate
___ 8-Hour Refresher Training Certificate
___ Medical Clearance Letter
___ Supervisory Training Certificate
___ Health & Safety Clearance Letter
___ Full-size OSHA Poster

HYDROGEOLOGY EQUIPMENT

___ Slug test/pumping test forms
___ Groundwater elevation data sheets
___ Graph paper
___ Data Logger/transducer/data cable
___ Existing well construction & water level data
___ M-Scope, slug

SHIPPING

Forms

___ FedEx Airbills, local dropoff location & hours
___ FedEx Gov. Acct# (1771-8058-0)
___ Lab Shipping Labels
___ Warehouse Shipping Labels
___ Blank Labels

Supplies

___ Tape
___ Packing materials
___ Baggies, Large garbage bags

OTHER

___ Site POC name/phone #
___ Personnel information to POC
___ Mobilization schedule to POC
___ Site access authorizations
___ Field office / trailer arrangements made
___ Electric, phone hookups arranged
___ Steel-toed boots, safety glasses, & hard hat
___ First aid equipment
___ Insect repellent
___ _____
___ _____

Note - not all items listed apply to every job, and some additional requirements may apply on a job-specific basis.

Tetra Tech NUS, Inc.

PROJECT: _____ JOB #: _____
LOCATION: _____ DATE: _____
PROJECT MANAGER: _____ FOL: _____

DAILY ACTIVITIES CHECKLIST

Startup Checklist

Activity	Yes	No	N/A
Pertinent site activities/information entered into site logbook			
All onsite personnel listed in logbook			
Required medical information onsite for all workers (TtNUS and Subcontractors)			
Required MSDS's onsite			
Proper equipment calibrations performed (list equipment)			
1			
2			
3			
4			
Calibration logs filled out			
Tailgate H&S meeting held prior to beginning field activities			
Required work permits filled out/signed			
Required utility clearances obtained			
Required PPE onsite and in use			
Information required to be posted is in place			
(OSHA poster, hospital route, key phone numbers, etc.)			

Exit Checklist

Activity	Yes	No	N/A
Logbooks completely and comprehensively filled out			
Field forms complete and accounted for/properly filed			
Samples properly packaged/shipped			
COCs faxed to appropriate in-house personnel			
All equipment accounted for, on charge if needed, and properly secured			
All personnel accounted for			
Arrangements made for upcoming work (permits, clearances, equipment, etc.)			
Site properly secured			

Note - not all items listed apply to every job, and some additional requirements may apply on a job-specific basis.

Tetra Tech NUS, Inc.

EQUIPMENT CALIBRATION LOG

PROJECT NAME : _____ INSTRUMENT NAME/MODEL: _____

SITE NAME: _____ MANUFACTURER: _____

PROJECT No.: _____ SERIAL NUMBER: _____

Date of Calibration	Instrument I.D. Number	Person Performing Calibration	Instrument Settings		Instrument Readings		Calibration Standard (Lot No.)	Remarks and Comments
			Pre-calibration	Post-calibration	Pre-calibration	Post-calibration		

Tetra Tech NUS, Inc.

BORING LOG

Page ___ of ___

PROJECT NAME: _____ BORING No.: _____
PROJECT NUMBER: _____ DATE: _____
DRILLING COMPANY: _____ GEOLOGIST: _____
DRILLING RIG: _____ DRILLER: _____

Sample No. and Type or RQD	Depth (Ft.) or Run No.	Blows / 6" or RQD (%)	Sample Recovery / Sample Length	Lithology Change (Depth/Ft.) or Screened Interval	MATERIAL DESCRIPTION: Soil Density/ Consistency or Rock Hardness	Color	Material Classification	USCS *	Remarks	PID/FID Reading (ppm): Sample	Sampler BZ	Borehole**	Driller BZ**

* When rock coring, enter rock brokeness.

** Include monitor reading in 6 foot intervals @ borehole. Increase reading frequency if elevated reponse read.

Drilling Area Background (ppm): ____

Remarks: _____

Converted to Well: Yes _____ No _____ Well I.D. #: _____

Tetra Tech NUS, Inc.

SOIL & SEDIMENT SAMPLE LOG SHEET

Page___ of ___

Project Site Name: _____
Project No.: _____

[] Surface Soil
[] Subsurface Soil
[] Sediment
[] Other: _____
[] QA Sample Type: _____

Sample ID No.: _____
Sample Location: _____
Sampled By: _____
C.O.C. No.: _____

Type of Sample:
[] Low Concentration
[] High Concentration

GRAB SAMPLE DATA:

Date:	Depth Interval	Color	Description (Sand, Silt, Clay, Moisture, etc.)
Time:			
Method:			
Monitor Reading (ppm):			

COMPOSITE SAMPLE DATA:

Date:	Time	Depth Interval	Color	Description (Sand, Silt, Clay, Moisture, etc.)
Method:				
Monitor Readings (Range in ppm):				

SAMPLE COLLECTION INFORMATION:

Analysis	Container Requirements	Collected	Other

OBSERVATIONS / NOTES:

MAP:

Circle if Applicable:

MS/MSD	Duplicate ID No.:

Signature(s):

Tetra Tech NUS, Inc.

QA SAMPLE LOG SHEET

Page___ of ___

Project Site Name: _____ Sample ID Number: _____
Project Number: _____ Sampled By: _____
Sample Location: _____ C.O.C. Number: _____
QA Sample Type:

[] Trip Blank [] Rinsate Blank
[] Source Water Blank [] Other Blank _____

SAMPLING DATA:

Date: _____
Time: _____
Method: _____

WATER SOURCE:

[] Laboratory Prepared [] Tap
[] Purchased [] Fire Hydrant
[] Other _____

PURCHASED WATER INFORMATION (If Applicable as Source or Rinsate Water):

Product Name: _____
Supplier: _____
Manufacturer: _____
Order Number: _____
Lot Number: _____
Expiration Date: _____

RINSATE INFORMATION (If Applicable):

Media Type: _____
Equipment Used: _____
Equipment Type:
[] Dedicated
[] Reusable

SAMPLE COLLECTION INFORMATION:

Analysis	Preservative	Container Requirements	Collected
Volatiles	Cool 4°C & HCl		YES / NO
Semivolatiles	Cool 4°C		YES / NO
Pesticide / PCB	Cool 4°C		YES / NO
Metals	Cool 4°C & HNO_3		YES / NO
Cyanide	Cool 4°C & NaOH		YES / NO

OBSERVATIONS / NOTES:

Signature(s):

Tetra Tech NUS, Inc.

CONTAINER SAMPLE & INSPECTION SHEET

Page_____ of _____

Project Site Name: _____
Project Number: _____
Site Identification: _____
Container Number(s): _____
Sample Type: [] Grab
[] Composite

Sample ID No. _____
Sampled By: _____
C.O.C. No.: _____
Concentration: [] High
[] Medium
[] Low

CONTAINER SOURCE

DRUM:
[] Bung Top
[] Lever Lock
[] Bolted Ring
[] Other _____

TANK:
[] Plastic
[] Metal
[] Other _____

OTHER: _____

CONTAINER DESCRIPTION

COLOR: _____

CONDITION: _____

MARKINGS: _____

VOL. OF CONTENTS: _____

OTHER: _____

CONTAINER DISPOSITION

SAMPLED: _____

OPENED BUT NOT SAMPLED:
Reason _____
_____

NOT OPENED:
Reason _____
_____

CONTENTS DESCRIPTION

SINGLE PHASED: _____
_____

MULTIPHASE :

	Layer 1	Layer 2	Layer 3
Phase (Sol. or Liq.)	_____	_____	_____
Color	_____	_____	_____
Viscosity	L, M or H	L, M or H	L, M orH
% of Total Volume	_____	_____	_____

MONITOR READING:

SAMPLE and /or INSPECTION DATE & TIME:
_____HRS.

METHOD:_____

SAMPLER(S) and / or INSPECTOR(S) SIGNATURE:

ANALYSIS:

TETRA TECH NUS
FIELD TASK MODIFICATION REQUEST FORM

Project/Installation Name _____ CTO & Project Number _____ Task Mod. Number _____

Modification To (e.g. Work Plan) _____ Site/Sample Location _____ Date _____

Activity Description: _____

Reason for Change: _____

Recommended Disposition: _____

Field Operations Leader (Signature) _____ Date _____

Approved Disposition:

Project/Task Order Manager (Signature) _____ Date _____

Distribution:

Program/Project File –
Project/Task Order Manager –
Field Operations Leader –

Other: _____

APPENDIX E

LABORATORY TECHNICAL SPECIFICATION AND CERTIFICATION LETTER

ATTACHMENT NO. 2

STATEMENT OF WORK/PRICE TABLES

TECHNICAL SPECIFICATION FOR LABORATORY SERVICES
NAVAL SUBMARINE BASE (NSB) NEW LONDON, CONNECTICUT

COMPREHENSIVE LONG-TERM ENVIRONMENTAL ACTION - NAVY (CLEAN)
CONTRACT *N62470-08-D-1001,* CONTRACT TASK ORDER (CTO) NO. WE57

PRE-DESIGN INVESTIGATION
CHEMICAL ANALYSES

1.0 INTRODUCTION

Tetra Tech NUS, Inc. (TtNUS) under CLEAN Contract ***N62470-08-D-1001***, is procuring laboratory analytical services for NSB New London, CT in support of a soil Pre-Design Investigation. Requested analyses include Polycyclic Aromatic Hydrocarbons (PAHs), Extractable Total Petroleum Hydrocarbons (ETPH), Metals (lead and antimony) and Synthetic Precipitation Leaching Procedure (SPLP) Leachate Metals (lead and antimony)**.**

The laboratory performing these analyses must be certified in the State of Connecticut. Additionally the laboratory must provide a current copy of their NFESC or DOD ELAP approval letter, in which all methods requested must be addressed.

The responding laboratory must submit Method Detection Limits (MDLs); Limit of Detection (LOD); and Limits of Quantitation (LOQs) for all analyses and matrices requested by filling out the last two columns of Attachment A and including the completed attachment with the bid response. After award, the laboratory will be required to submit Standard Operating Procedures (SOPs) and relevant precision and accuracy limits for all preparation and analytical methods required under this scope of work. The laboratory will also be asked to complete Uniform Federal Policy (UFP) Sampling and Analysis Plan (SAP) Worksheets 19, and 23 through 28 for inclusion in the SAP. The SAP will be prepared according to the UFP for quality assurance project plans (March 2005) and utilize the 37 worksheets.

2.0 SAMPLE INFORMATION

Samples will be shipped to the laboratory during the period beginning May-June 2010.

The approximate number of samples to be submitted, the type of analyses to be conducted, and the analytical methods to be used are summarized in the attached Table 1.

Field duplicate samples will be submitted with "blinded" identification to the laboratory. The field crew will designate samples (one per twenty samples of like matrices) upon which matrix spike/matrix spike duplicate (organic analyses) and matrix spike/laboratory duplicate (inorganic analyses) are to be performed; additional volumes of these samples will be provided as necessary.

All samples are expected to contain low to moderate contaminant concentrations. The field crew will attempt to identify any potentially high concentration samples. Samples may be saturated and have high salinity concentrations.

For the ICP metals analyses, perform a matrix spike and a laboratory duplicate analysis instead of matrix spike/matrix spike duplicate. If the matrix spike recovery falls outside the control limits and the sample result does not exceed four times the spike added, a post-digestion spike analysis must be performed. In addition, a five-fold dilution analysis (serial dilution) must be performed on the field-designated QC sample. Results for these QC analyses must be summarized in the corresponding CLP-equivalent forms (Attachment B).

3.0 ANALYSIS/REPORTING INFORMATION

Data package deliverables must be submitted as a hardcopy and PDF (2 copies/2 CDs). The analytical requirements and approximate number of samples to be submitted for analysis are detailed in Table 1. Any analysis and reporting requirements addressed in the DOD Quality Systems Manual (January 2006 or April 2009 depending on current NFESC approval letter) and the requested methods must be followed. Additionally, it is a requirement of TtNUS that the associated PDF data packages for *PAHs and metals* analysis must meet Contract Laboratory Program (CLP) format, reporting, and PDF data package deliverable requirements. The PDF data packages for all the remaining analyses must be fully validatable and contain raw data, summary forms for all sample and laboratory method blank data, and summary forms containing all method specific quality control (results, recoveries, relative percent differences, relative standard deviations, and/or percent differences etc.).

Additionally, each PDF data package must contain a summary data package. This summary data package shall consist of only the summary forms (i.e., for CLP Forms 1 through 15 and non-CLP it shall be the CLP-like equivalent of Forms 1 through 15).

Attachment A details the required target compound list and required project action limits that must be met. The laboratory must submit its LOQs, LODs, and MDLs for all analyses and matrices requested by filling out the last two columns of Attachment A and including the completed attachment with the bid response.

Attachment B details the required summary forms for CLP-like data packages, the requirements for the organization, and bookmarking of PDF data packages.

Non-detected organic and metals results must be reported down to the LOD submitted by the laboratory in Attachment A with the bid response. Positive results above the MDL but below the laboratory's LOQ must be reported as estimated values qualified with a "J" **for both organic and metals** analyses. Soil samples must be reported on a dry-weight basis.

The PDF data package deliverable must contain a detailed case narrative for all analytical fractions. This case narrative must also include the Contract Task Order (CTO) number, the site name, and the TtNUS Project Manager's name. Data from all analytical runs (i.e., original, dilution, re-analysis) must be reported. All soil and sediment matrix sample results shall be reported on an adjusted dry-weight basis. Additionally, the data package deliverable must include the ORIGINAL chain-of-custody form (received with the samples and signed by the laboratory sample custodian).

As stipulated in the CLEAN Master Services Agreement (MSA), Sample Delivery Group (SDG) and fractionally-specific text (TXT) files containing all environmental sample and field quality control blank analysis results must be generated in accordance with the requirements outlined in Attachment C of this specification.

As part of the laboratory case narrative, it is required that the Laboratory Quality Assurance Manager sign an attestation statement verifying that all electronic diskette deliverables exactly match the data summary forms (i.e. Form Is).

Maximum holding time allowances, as defined in the following table, are to be strictly observed. Calculation of holding time is in calendar days and is to begin from the time of collection. The holding times are as follows:

Analyses	Holding Time
PAHs	14 days until extraction; 40 days to analysis
ETPH	14 days until extraction; 40 days to analysis
Metals (lead and antimony)	6 months to analysis
SPLP Leachate Metals (lead and antimony)	14 days until extraction; 6 months to analysis

These holding times are based on 40 CFR 136, data validation criteria, and method specific requirements, and are measured from date of collection. The holding time criteria depicted apply to all analyses necessary to successfully determine the contaminant level contained in the sample. Hence, the holding time criteria apply to any/all subsequent sample dilutions and re-analyses.

The TtNUS Project Manager for this project is Mr. Corey Rich and he must be contacted in the event of any laboratory problems that could impact project deadlines (i.e., late deliverables, technical problems in the lab that could lead to late deliverables.) To ensure good communication it is required that the laboratory's appointed project manager contact Mr. Rich once a week for the entire project duration.

Contact information for Mr. Rich is as follows:

Tetra Tech NUS, Inc.
Foster Plaza 7
661 Andersen Drive
Phone: 412-921-8984
Fax: 412-921-4040
e-mail: corey.rich@tetratech.com

Analytical data turnaround times are to be measured from receipt of each sample shipment. All PDF analytical data package (2 CDs) and associated electronic (TXT) deliverables are due to the TtNUS Sample Management Coordinator, Ms. Tobrena Skeen, within the standard MSA turnaround term of 21 calendar days from receipt of the last sample in a Sample Delivery Group (SDG). Additionally all SDGs must contain 20 samples. The frequency in which SDGs contain less than 20 samples should be minimal. All PDF data packages and electronic deliverables must be received at the same time or the deliverable will be considered incomplete and payment deductions may be imposed. **Additionally the laboratory Project Manager must fax copies of chain of custody forms to Ms. Skeen as samples are received by the laboratory.**

Contact information for Ms. Skeen follows:

Tetra Tech NUS, Inc.

Foster Plaza 7
661 Andersen Drive
Pittsburgh, PA 15220
Phone: 412-921-8582
Fax: 412-921-4040
e-mail: tobrena.skeen@tetratech.com

4.0 PERIOD OF PERFORMANCE/BOTTLEWARE INFORMATION

All samples will be shipped to the laboratory via express carrier within 48-hours of collection. Please circle the Yes or No at the bottom of Table 1 which will indicate if the laboratory will provide courier service at no extra charge. Samples will be shipped to the laboratory the period **beginning May-June 2010**.

Shipment of bottleware to support the entire project will be coordinated at a later date.

The laboratory is to provide all necessary sample containers **(plus approximately 10% extra for breakage)**. All sample containers must meet ICHEM series 300 cleanliness criteria (or equivalent), and documentation of certified cleanliness must be provided. All of the appropriate sample bottleware must be pre-preserved. The bottleware must be shipped to the designated location in Coleman-like coolers. **The laboratory must also provide any extra coolers needed for return shipment of sample to the laboratory for analysis.** The laboratory is also requested to provide a packing slip indicating the analytical parameters for which each container type is designated, sample labels, and chain-of-custody forms. The laboratory is to dispatch three to four EnCore samplers for each of the soil samples submitted for volatiles analysis. Upon receipt of the Encore samplers the laboratory shall prepare the soil samples in accordance with SW-846 Method 5035. In addition to the EnCore samplers the laboratory is to dispatch proper bottleware for the collection of additional soil, per sampling location, for dry-weight determination.

The laboratory must provide Material Safety Data Sheets (MSDSs) for all preservatives sent with each bottleware shipment to the field. MSDSs must be representative of the chemicals provided as preservatives with regard to mixtures and/or purity of the chemicals. For example if a 35% sulfuric acid solution is the preservative, the MSDS provided should be for 35% sulfuric acid solution not 96% sulfuric acid.

5.0 ADDITIONAL COMMENTS/CONTACTS

Within a laboratory, internal transfers of samples, extracts, and digestates must be accomplished and documented as controlled custody transfers. The laboratory must maintain documentation that supports an unbroken chain of custody for samples, digestates and extracts from time of receipt or production in the laboratory until disposal.

The laboratory is to provide a minimum of 60 days storage of sample extracts and 60 days storage of intact sample aliquots, as stipulated in the MSA. **Additionally, the laboratory must store hardcopy and/or PDF data packages for 5 years.**

All analyses conducted under this subcontract assignment are to be performed at the solicited facility only. The laboratory is not permitted to lower-tier subcontract these analyses, or analyze these samples at a

corporate facility other than the facility stipulated without prior notification and consent from the CLEAN Subcontracting Officer.

The unit cost for analysis is to include compensation for containers, preservatives, coolers, shipping costs, storage, disposal, and laboratory quality control analyses (such as matrix spike, matrix spike duplicate, laboratory duplicate, and laboratory control sample analyses.) These items are not to be billed as separate line items.

Technical, quality assurance, and data format concerns are to be directed to Ms. Kelly Carper at 412-921-7273 or via e-mail kelly.carper@tetratech.com. Ms. Carper must be contacted and informed of any difficulties encountered during the conduct of the requested analyses.

Questions regarding electronic diskette deliverable concerns are to be directed to Ms. Amy Thomson at 412-921-8182 or via e-mail amy.thomson@tetratech.com. Ms. Thomson must be contacted and informed of any difficulties encountered preparing the required electronic deliverables.

Contract concerns, and response to this solicitation, are to be directed to:

Ms. Meg Price
CLEAN Subcontracting Officer
Tetra Tech NUS, Inc.
234 Mall Boulevard, Suite 260
King of Prussia, PA 19406
Phone: 610-491-9688
Fax: 610-491-9645
e-mail: meg.price@tetratech.com

Triplicate copies of invoices associated with the analyses contracted herein are to be submitted to the attention of the Accounting Supervisor:

Tetra Tech NUS, Inc.
661 Andersen Drive, Foster Plaza 7
Pittsburgh, PA 15220
Phone: 412-921-8506
Fax: 412-921-4040

Please confirm the laboratory's ability to perform the methodologies requested at the analyte detection limits indicated. Also confirm available laboratory capacity, and complete/confirm the costing information indicated in Table 1. **All costing information must reflect the terms and conditions established by the 2009 CLEAN MSA.**

TABLE 1
NUMBER OF SAMPLES/ANALYTICAL METHODS
NAVAL SUBMARINE BASE (NSB) NEW LONDON, CONNECTICUT
CTO WE57, PRE-DESIGN INVESTIGATION

Matrix	Parameter	Method - *Please circle the method to be used*	# Samples[(1)]	Unit Price	Total Cost
Aqueous	SPLP Leachate Antimony	SW-846 1312/6010B, C or 6020	8	$	$
	SPLP Leachate Lead	SW-846 1312/6010B, C or 6020	25	$	$
	SPLP Leachate Antimony and Lead	SW-846 1312/6010B, C or 6020	13	$	$
Solid	PAHs	SW-846 8270C or D SIM	52	$	$
	ETPH	ETPH CT Method	62	$	$
	Antimony	SW-846 6010B, C or 6020	5	$	$
	Lead	SW-846 6010B, C or 6020	17	$	$
	Antimony and Lead	SW-846 6010B, C or 6020	12	$	$

TOTAL COST $

(1) The number of aqueous samples includes the field quality control blanks being taken.

Can the laboratory provide sample pick-up on site? YES or NO (circle one)
If yes is there an additional charge and what is that charge?_____
Name of Laboratory_____

Signature_____

ATTACHMENT A
Required Target Compound Lists and Quantitation Limits

Matrix: Soil
Analytical Group: PAHs

Analyte	CAS Number	Project Action Limit (mg/kg)	Project Action Limit Reference[1]	Project Quantitation Limit Goal (mg/kg)	Laboratory		
					Limit of Quantitation (mg/kg)	Limit of Detection (mg/kg)	Method Detection Limit (mg/kg)
Benzo(a)anthracene	56-55-3	1	CTDEP GB PMC	0.33			
Benzo(a)pyrene	50-32-8	1	CTDEP GBPMC	0.33			
Benzo(b)fluoranthene	205-99-2	1	CTDEP GB PMC	0.33			
Benzo(k)fluoranthene	207-08-9	1	CTDEP GB PMC	0.33			
Chrysene	218-01-9	1[2]	CTDEP GB PMC	0.33			
Dibenzo(a,h)anthracene	53-70-3	1[2]	CTDEP GB PMC	0.33			
Indeno(a,2,3-cd)pyrene	193-39-5	1[2]	CTDEP GB PMC	0.33			
Phenanthrene	85-01-8	40	CTDEP GB PMC	13.3			
Pyrene	129-00-0	40	CTDEP GB PMC	13.3			
Carbazole	86-74-8	1[2]	CTDEP GB PMC	0.33			

1. Connecticut Department of Environmental Protection Remediation Standards
2. Criteria based on detection limits

CTDEP GB PMC - Connecticut Department of Environmental Protection, Pollutant Mobility Criteria for groundwater classified as GB

Matrix: Soil
Analytical Group: TPH

Analyte	CAS Number	Project Action Limit (mg/kg)	Project Action Limit Reference[1]	Project Quantitation Limit Goal (mg/kg)	Laboratory		
					Limit of Quantitation (mg/kg)	Limit of Detection (mg/kg)	Method Detection Limit (mg/kg)
TPH	--	500	CTDEP RES DEC	167			

1. Connecticut Department of Environmental Protection Remediation Standards
CTDEP RES DEC - Connecticut Department of Environmental Protection, Direct Exposure Criterion for a residential scenario

Matrix: Soil
Analytical Group: Metals (antimony and lead)

Analyte	CAS Number	Project Action Limit (mg/kg)	Project Action Limit Reference[1]	Project Quantitation Limit Goal (mg/kg)	Laboratory		
					Limit of Quantitation (mg/kg)	Limit of Detection (mg/kg)	Method Detection Limit (mg/kg)
Antimony	7440-36-0	31	CTDEP RES DEC	10			
Lead	7439-92-1	400	CTDEP RES DEC	133			

1. Connecticut Department of Environmental Protection Remediation Standards
CTDEP RES DEC - Connecticut Department of Environmental Protection, Direct Exposure Criterion for a residential scenario

Matrix: SPLP Leachate
Analytical Group: Metals (antimony and lead)

Analyte	CAS Number	Project Action Limit (mg/kg)	Project Action Limit Reference[1]	Project Quantitation Limit Goal (mg/kg)	Laboratory		
					Limit of Quantitation (mg/kg)	Limit of Detection (mg/kg)	Method Detection Limit (mg/kg)
Antimony	7440-36-0	0.08	CTDEP GB PMC	0.027			
Lead	7439-92-1	0.15	CTDEP GB PMC	0.05			

1. Connecticut Department of Environmental Protection Remediation Standards
CTDEP GB PMC - Connecticut Department of Environmental Protection, Pollutant Mobility Criteria for groundwater classified as GB

ATTACHMENT B
Summary Form Requirements for PDF Deliverable and PDF Data Package Deliverables

PDF DATA PACKAGE DELIVERABLE REQUIREMENTS

The laboratory is to provide 2 compact disks (CDs) containing a PDF file in the following format:

1. Table of Contents
2. Case Narrative
3. Chain-of-Custody
4. Data Summary Package (contains summary of all CLP or CLP like Forms 1 through 15 per analytical fraction)
5. Analytical Fractions (VOA, SVOC, General Chemistry, etc.)
a. QC Summary (summary of all CLP or CLP like Forms 1 through 15 for a particular analytical fraction)
b. Raw Sample Data (includes all sample dilutions, sample re-analyses, QC samples, etc.)
c. Calibration Data (includes all initial and continuing calibrations)
d. Miscellaneous (includes extraction forms, IDLs, MDLs, etc.)

Each of the above sections should be bookmarked in the PDF for easy access.

Summary Form Requirements for PDF data package deliverable for non-CLP Methods:

The following summary forms are required as part of the data package deliverable for SW-846 6020/6010B/7000A series for metals:

Results Report - must present all information presented on CLP FORM I.
Initial and Continuing Calibration Summary -must present all information presented on CLP FORM 2A.
CRDL Summary - must present all information presented on CLP FORM 2B.
Blanks - must present all information presented on CLP FORM 3.
ICP Interference Check Sample Summary - must present all information presented on CLP FORM 4.
Matrix Spike Summary - must present all information presented on CLP FORM 5A.
Post Digestion Spike - must present all information presented on CLP FORM 5B.
Lab Duplicate Results - must present all information presented on CLP FORM 6.
LCS Summary - must present all information presented on CLP FORM 7.
MSA Summary (Method of Standard Addition) - must present all information presented on CLP FORM 8.
ICP Serial Dilution Summary - must present all information presented on CLP FORM 9.
Detection Limits - must present all information presented on CLP FORM 10.
Linear range – must present all information presented on CLP FORM 12
Prep Log - must present all information presented on CLP FORM 13.
Analysis Run Log - must present all information presented on CLP FORM 14.
ICP-MS Tune – must present all information on CLP FORM 14 (ILM05.4)
ICP/MS Internal Standard Relative Intensity Summary - must present all information presented on CLP FORM 15 (ILM05.4).

Also must include: Instrument Calibration Records, Chain-of-Custody Forms, and Case Narrative.

Summary Forms for SW-846, 8260B and 8270C (i.e., Any SW-846 GC/MS analysis of Volatile and Semivolatile Organic Compounds) should be presented in a CLP-Like format. The following Summary Forms must be included:

Result Summary	One Sample per summary page. Presentation of analytical results for both method blanks and environmental samples, date of collection, preparation, and analysis. Environmental samples should be identified

	with the field identification numbers on the COCs.
Surrogate Recovery Form	Present all information contained on CLP Form II.
Summary of Matrix Spike/Matrix Spike Duplicate Recovery	Present all information contained on CLP Form III.
Instrument Performance Check Summary Form - Mass Spec Tuning Form	Present all information Contained on CLP Form V.
Initial Calibration Summary	Present all information contained CLP Form VI.
Continuing Calibration Summary	Present All Information contained on CLP Form VII.
Internal Standard Area and Retention Time Summary	Present all information contained CLP Form VIII.

ATTACHMENT C
ELECTRONIC DATA DELIVERABLE REQUIREMENTS

ELECTRONIC DATA FORMAT REQUIREMENTS

1.0 INTRODUCTION

The laboratory is to submit text-based tab delimited EDD files for each SDG using Tetra Tech's laboratory data checker explained below. The files must be in the format specified in this Attachment. Additional information such as laboratory name, project name, fractions included, project number, site name/number, laboratory contact person and any specific comments related to the EDD should be included in the comments section of the EDD Submittal page.

The RESULT for nondetects should be populated with the project-specific sample quantitation reporting limits (i.e., either the sample quantitation limit or method detection limit, as specified in Section 3.0 of this scope of work. Any corrections made to the hardcopy data must also be made to the electronic file. Appropriate qualifiers as identified by the analytical protocol must also be designated; laboratory QC non-compliance codes are not to be depicted.

Tetra Tech's electronic EDD format follows the ADAPT structure and requires the A1 and A3 files. The A2 file is only required if the project is using ADAPT; and, for non-ADAPT EDD submittals the A2 file may be omitted. The EDD consists of separate, tab-delimited ASCII text files. Each file corresponds to a database table. The tables are identified as the Analytical Results Table (A1) and Sample Analysis Table (A3). A separate set of text files must be created and submitted for each sample delivery group (SDG). The files must be identified to correspond to the (A1) table and the (A3) table. The file naming convention is: the Sample Delivery Group (SDG) followed by the table identifier (A1 or A3), followed by the ".txt" extension. The file names must not contain spaces or special characters. For example, the EDD file names for a laboratory-reporting batch identified as SDG001 would be as follows:

SDG001A1.txt
SDG001A3.txt

On certain projects Tetra Tech will utilize the ADAPT Electronic Data Validation software, which will require the laboratory to use the ADAPT electronic data deliverable checker software prior to submitting the files through Tetra Tech's laboratory data checker (this will be clearly specified in the Tetra Tech laboratory statement of work). The ADAPT checker software can be downloaded from Laboratory Data Consultants' web site: http://www.lab-data.com. For projects which Tetra Tech is using the ADAPT software, Tetra Tech will provide the laboratory with the project library. The laboratory is not permitted to modify the project library. ADAPT projects will require the laboratory to export all three checked files (A1, A2, and A3) from the ADAPT software and submit them through Tetra Tech's laboratory data checker. **ADAPT error logs generated must be included with the electronic PDF data validation package and cannot be submitted through the laboratory data checker.**

The values reported in the EDD text files must agree exactly with the final values reported on the PDF data package sample result summaries. The details of file naming conventions, data structure and data checker use are discussed below.

Analytical Results Table (A1 File)

The Analytical Results table contains analytical results and related information for target analytes in field samples and associated laboratory quality control samples (excluding calibrations and tunes). Field samples and laboratory method blanks must report a result record for each analyte reported within a method. Laboratory control samples (LCS and LCSD) and matrix spike samples (MS and MSD) must report a result record for every analyte specified as a spiked analyte in the laboratory statement of work. Table A1 in this document lists the field names and data type descriptions for the Analytical Results Table (A1).

Lab Instrument Table (A2 File)

A2 file is only required if the project is using ADAPT. In all other EDD submittals, the A2 file may be omitted. Laboratories should refer to the ADAPT User Guide for populating the A2 Table.

Sample Analysis Table (A3 File)

The Sample Analysis table contains information specific to field environmental samples and laboratory quality control analyses (excluding calibrations and tunes). A sample record must exist for each sample/method/matrix/analysis type combination. Table A3 in this document lists the field names and data type descriptions for the Sample Analysis Table (A3).

All electronic data deliverables are due within the same time established for the associated hardcopy data packages.

In addition, the laboratory QC officer must read and sign a copy of the Quality Assurance Review Form displayed on the next page of this Attachment. Electronic deliverables are not considered to be complete without the accompanying Quality Assurance Review Form.

I _____, as the designated Quality Assurance Officer, hereby attest that all electronic deliverables have been thoroughly reviewed and are in agreement with the associated hardcopy data. The enclosed electronic files have been reviewed for accuracy (including significant figures), completeness and format. The laboratory will be responsible for any labor time necessary to correct enclosed electronic deliverables that have been found to be in error. I can be reached at
(____)_____if there are any questions or problems with the enclosed electronic deliverables.

Signature:_____ Title: _____Date: _____

2.0 EDD Field Properties

Tables A1 and A3 in this document specify the EDD field properties. Laboratories should refer to the ADAPT User Guide for populating the A2 Table. These include the field name, sequence order, field description, data type/length and reporting requirement for each field. Fields in the EDD **must** be sequenced according to the order that they appear below in Tables A1 and A3. For example, in the Analytical Results table (A1), the field "ClientSampleID" will always be the first piece of information to start every new line of data (or database record), followed by the field "LabAnalysisRefMethodID", "AnalysisType", etc.

When creating an EDD as a text file, use the ASCII character set in a file of lines terminated by a carriage return and line feed. No extra characters are allowed at the end of a line, after the carriage return and line feed. Enclose each data value with double quotes (text qualifier) and separate each field value with a **tab** character (tab delimiter). Data fields with no information (null) may be represented by two consecutive tabs. For example, in the Sample Analysis table, since the "Collected", "ShippingBatchID", and "Temperature" fields do not apply to laboratory generated QA/QC samples, the record for a Laboratory Control Sample by Method 8270C would be entered as follows. Note that the first two fields ("ProjectNumber" and "ProjectName") are omitted in this example.

…"LCSW100598" "AQ" "LCSW100598" "LCS" "8270C",…etc.

If a field is populated with less than the maximum allowed number of characters, do not pad the values with leading or trailing spaces. In the above example, although the "MatrixID" field can accommodate up to 10 characters, only 2 characters were entered in this field. **Do not include the delimiter (tab character) within any of the field values.** Example EDD files may be downloaded from the LEDD Checker application.

An example database shall be sent for review prior to the first electronic deliverable in the required .txt format. The example file will be examined for completeness and comments will be sent to the laboratory. Any questions regarding the electronic deliverable should be directed to LabSupport@tetratech.com.

Table A1
Field Descriptions for the Analytical Results Table (Table A1)
Contains laboratory test results and related information for field and QC samples (excluding instrument calibrations) on an analyte level for environmental chemistry including radiochemistry

Field Name	Field Name Description	Field Type	Field Length	Required Value
ClientSampleID	Client or contractor's identifier for a field sample If a sample is analyzed as a laboratory duplicate, matrix spike, or matrix spike duplicate, append suffixes DUP, MS and MSD respectively to the Client Sample ID with no intervening spaces or hyphens (i.e. MW01DUP, MW01MS, and MW01MSD). For Method Blanks, LCS, and LCSD enter the unique LaboratorySampleID into this field. Do not append suffixes to the ClientSampleID for dilutions, reanalyses, or re-extracts (the AnalysisType field is used for this distinction). For example, MW01DL and MW01RE are not allowed. Parent sample records must exist for each MS and MSD. If an MS/MSD is shared between two EDDs, records for the MS/MSD and its parent sample must exist in the Analytical Results table for both EDDs.	Text	25	X
LabAnalysisRefMethodID	Laboratory reference method ID. The method ID may be an EPA Method number or a Lab Identifier for a method such as a SOP Number, however; method ID is specified by the project. The method ID must be entered into the standard list.	Text	25	X
AnalysisType	Defines the analysis type (i.e., Dilution, Reanalysis, etc.). This field provides distinction for sample result records when multiple analyses are submitted for the same sample, method, and matrix; for example dilutions, re-analyses, and re-extracts.	Text	10	X
LabSampleID	Laboratory tracking number for field samples and lab generated QC samples such as method blank, LCS, and LCSD. There are no restrictions for the LabSampleID except for field length and that the LabSampleID must be distinct for a given field sample or lab QC sample and method. Suffixes may be applied to the LabSampleID to designate dilutions, reanalysis, etc.	Text	25	X
LabID	Identification of the laboratory performing the analyses.	Text	7	X
ClientAnalyteID	CAS Number or unique client identifier for an analyte	Text	12	X

Table A1
Field Descriptions for the Analytical Results Table (Table A1)
Contains laboratory test results and related information for field and QC samples (excluding instrument calibrations) on an analyte level for environmental chemistry including radiochemistry

Field Name	Field Name Description	Field Type	Field Length	Required Value
	or isotope. If a CAS Number is not available, use a unique identifier provided by the client or contractor. The ClientAnalyteID for a particular target analyte or isotope should be specified by the project and must exist in the standard value tables for Analytes. For the LCS, LCSD, MS, and MSD, it is only necessary to report the compounds designated as spikes in the library (and surrogates for organic methods.) For TICs from GC/MS analyses, enter the retention time in decimal minutes as the Client Analyte ID.			
AnalyteName	Chemical name for the analyte or isotope. The project specifies how an analyte or isotope is named. The analyte name must be associated to a ClientAnalyteID in the standard values table for Analytes (excluding compounds designated as TIC's).	Text	60	X
Result	Result value for the analyte or isotope. Entries must be numeric. For non-detects of target analytes or isotopes and spikes, **do not enter "ND" or "0". Do not leave this field blank**. If an analyte or spike was not detected, enter the reporting limit value corrected for dilution and percent moisture as applicable. Do not enter "0"	Numeric (1)	20(6)	X
ResultUnits	The units defining how the values in the Result, DetectionLimit, and ReportingLimit fields are expressed. For radiochemistry this also includes how the value in the Error field is expressed.	Text	10	X
LabQualifiers	A string of single letter result qualifiers assigned by the lab based on client-defined rules and values. <u>The "U" Lab Qualifier must be entered for all non-detects.</u> Other pertinent lab qualifiers may be entered with the "U" qualifier. Order is insignificant. Lab qualifiers other than those listed in the standard	Text	7	Q

Table A1
Field Descriptions for the Analytical Results Table (Table A1)
Contains laboratory test results and related information for field and QC samples (excluding instrument calibrations) on an analyte level for environmental chemistry including radiochemistry

Field Name	Field Name Description	Field Type	Field Length	Required Value
	values table may be used. If so, these must be added to the standard value table in the application.			
DetectionLimit	For radiochemistry methods, the minimum detectable activity for the isotope being measured. For all other methods: The minimum detection limit value for the analyte being measured.	Numeric (1)	10(6)	X
DetectionLimitType	Specifies the type of detection limit (i.e., MDA, MDL, IDL, etc.).	Text	10	X
RetentionTime or Error	For radiochemistry methods only, enter the 2 Sigma Counting Errors. The units for error are entered in the ResultUnits field. For GC/MS methods only, enter the time expressed in decimal minutes between injection and detection for GC/MS TICs only For target analytes in all other methods, leave this field blank. Note: GC retention times are not evaluated at this time.	Text	5	T
AnalyteType	Defines the type of result, such as tracer, surrogate, spike, or target compound.	Text	7	X
PercentRecovery	For radiochemistry methods: The tracer yield, if applicable. For all other analytical methods: The percent recovery value of a spiked compound or surrogate. If the spike or surrogate was not recovered because of dilution, enter “DIL”. If a spike or surrogate was not recovered because of matrix interference, enter “INT”. If a spike or surrogate was not recovered because it was not added to the sample, enter “NS”.	Numeric (1)	5(3)	R
RelativePercentDifference	The relative percent difference (RPD) of two QC results, such as MS/MSD, LCS/LCSD, and Laboratory Duplicates. Report RPD in Laboratory Duplicate, LCSD, and MSD records only.	Numeric (1)	5(3)	D
ReportingLimit	Reporting limit value for the measured analyte or isotope Factor in the dilution factor and percent moisture correction, if applicable. The Reporting Limit for each analyte and matrix in a given method is specified in	Numeric (1)	10(6)	X

Table A1
Field Descriptions for the Analytical Results Table (Table A1)
Contains laboratory test results and related information for field and QC samples (excluding instrument calibrations) on an analyte level for environmental chemistry including radiochemistry

Field Name	Field Name Description	Field Type	Field Length	Required Value
	the project library or QAPP.			
ReportingLimitType	Specifies the type of reporting limit (i.e., CRQL, PQL, SQL, RDL, etc). The Reporting Limit Type for each method and matrix is specified in the project library or QAPP.	Text	10	X
ReportableResult	This field indicates whether or not the laboratory chooses an individual analyte or isotope result as reportable. Enter "YES" if the result is reportable. Enter "NO" if the result is not reportable. If only one analysis is submitted for a particular sample and method, enter "YES" for all target compounds (where Analyte Type = TRG). For GC/MS methods enter yes for tentatively identified compounds (where Analyte Type = TIC). If two or more analyses are submitted for a particular sample and method (i.e. initial analysis, reanalysis and/or dilutions), enter "YES" from only <u>one</u> of the analyses for each target compound. For example: a sample was run a second time at dilution because benzene exceeded the calibration range in the initial, undiluted analysis. All target analytes are reported in each analysis. For the initial analysis, (Analysis Type = RES), enter "NO" for benzene and enter "YES" for all other compounds. For the diluted analysis (Analysis Type = DL), enter "YES" for benzene and enter "NO" for all other compounds. For TICs (Analyte Type = TIC), if more than one analysis is submitted for a particular sample and method, choose only one of the analyses where Reportable Result = YES for <u>all</u> TICs. For example, a sample was run a second time because one or more target compounds exceeded the calibration range in the undiluted analysis. Choose a particular analysis and enter "YES" for all TICS. In the other analysis enter "NO" for all TICs. Note that it is not necessary to report the full target analyte list for the initial result, dilution, re-analysis, or re-extraction. However, each target analyte must be reported YES once and once only in the case of multiple analyses for a given sample, method, and matrix. In the case of organics, all surrogates must be reported for all analyses submitted for a given sample, method, and, matrix.	Text	3	X

Table A1
Field Descriptions for the Analytical Results Table (Table A1)
Contains laboratory test results and related information for field and QC samples (excluding instrument calibrations) on an analyte level for environmental chemistry including radiochemistry

Field Name	Field Name Description	Field Type	Field Length	Required Value
SpkConcnAdded	The spike added. This value must be reported in the same units as the result. Where (SA) in the following equation: % Recovery = (SSA-SC)/SA x 100% where : SSA is the spiked sample concentration (amount) after spiking. SC is the sample concentration (amount) before spiking. SA is the the expected increase in sample concentration (amount) as a result of spiking. This value must incorporate all correction factors such as dilution factor and moisture content that are applied to the spiked sample when computing the spiked sample concentration or amount.	Numeric (1)	10(6)	R
SpkParentSampleID	The sampleID of a sample (often called the original sample) that receives a spike aliquot to form a spiked sample such as a matrix spike. This is not the same as the ID of the spiked sample (such as a matrix spike) after spiking. The result for SpkParentSampleID and the result (i.e., SpkConcnAdded) for the spiked sample are used to compute percent recovery of the analyte.	Text	25	R
SamplePrepInitial	The initial sample preparation volume in liters (L) for aqueous samples or grams (g) for solid samples.	Numeric (1)	20(6)	
SamplePrepFinal	The final sample preparation volume in liters (L) for aqueous samples or grams (g) for solid samples.	Numeric (1)	20(6)	
Comment	Add any comments or additional information specific to the analyte test result data record.	Text	200	

X Required field.
Q Only required if laboratory has qualified the result.
T Only required for tentatively identified compounds by GC/MS.
R Only required if Analyte_Type =”SPK” or “SURR”.
D Only required for spiked compounds in the LCSD and MSD.
(1) Field Length indicates decimal precision in parenthesis. For example, 5(2) = a total width of 5 numbers including a maximum of 2 decimal places.

Table A3
Field Description for the Sample Analysis (Table A3)
This table contains information related to analyses of field samples and laboratory QC samples (excluding calibrations and tunes) on a sample level for environmental chemical analyses including radiochemistry

Field Name	Field Name Description	Field Type	Field Length	Required Value
ProjectNumber	Project number assigned by the client.	Text	30	X
ProjectName	Project name assigned by the client.	Text	90	X
ClientSampleID	Client or contractor's identifier for a field sample If a sample is analyzed as a laboratory duplicate, matrix spike, or matrix spike duplicate, append suffixes DUP, MS and MSD respectively to the Client Sample ID with no intervening spaces or hyphens (i.e. MW01DUP, MW01MS, and MW01MSD). For Method Blanks, LCS, and LCSD enter the unique LaboratorySampleID into this field Do not append suffixes to the ClientSampleID for dilutions, reanalyses, or re-extracts (the Analysis_Type field is used for this distinction). For example, MW01DL and MW01RE are not allowed Parent sample records must exist for each MS and MSD. If an MS/MSD is shared between two EDDs, records for the MS/MSD and its parent sample must exist in the Sample Analysis table for both EDDs.	Text	25	X
Collected	Date and Time of sample collection. Refer to the date/time format at the end of this table. Leave this field blank for Method Blank, LCS, and LCSD	Date/ Time	16*	C
MatrixID	Sample matrix (i.e., AQ, SO, etc.)	Text	10	X
LabSampleID	Laboratory tracking number for field samples and lab generated QC samples such as method blank, LCS, and LCSD. There are no restrictions for the LabSampleID except field length and that the LabSampleID must be unique for a given field sample or lab QC sample and method.	Text	25	X
QCType	This record identifies the type of quality control sample QC (i.e., Duplicate, LCS, Method Blank, MS, or MSD). For regular environmental samples, populate this field with "NM".	Text	10	X
ShippingBatchID	Unique identifier assigned to a cooler or shipping container used to transport client or field samples. Links all samples to a cooler or shipping container. No	Text	25	C

Table A3
Field Description for the Sample Analysis (Table A3)
This table contains information related to analyses of field samples and laboratory QC samples (excluding calibrations and tunes) on a sample level for environmental chemical analyses including radiochemistry

Field Name	Field Name Description	Field Type	Field Length	Required Value
	value is required for method blanks, LCS, and LCSD.			
Temperature	Temperature (in centigrade degrees) of the sample as received. This field is not required for radiochemistry methods.	Numeric (1)	10(6)	C
LabAnalysisRefMethodID	Laboratory reference method ID. The method ID may be an EPA Method number or laboratory identifier for a method such as a SOP number, however; values used for Laboratory Method IDs are specified by the project and must in the in standard value list for method IDs.	Text	25	X
PreparationType	Preparation Method Number (i.e., 3010A, 3510C, 3550C, 5030B, etc.) For analytical procedures that do not have a specific preparation method number, use “Gen Prep”.	Text	25	X
AnalysisType	Defines the type of analysis such as initial analysis, dilution, re-analysis, etc. This field provides distinction for sample records when multiple analyses are submitted for the same sample, method, and matrix, for example: dilutions, re-analyses, and re-extracts.	Text	10	X
Prepared	For radiochemistry leave this field blank. For all other methods enter the date and time of sample preparation or extraction. Refer to the date/time format at the end of this table.	Date/ Time	16*	X
Analyzed	Date and time of sample analysis. Refer to the date and time format at the end of this table.	Date/ Time	16*	X
LabID	Identification of the laboratory performing the analysis.	Text	7	X
QCLevel	The level of laboratory QC associated with the analysis reported in the EDD. If only the Analytical Results Table (A1) and the Sample Analysis Table (A3) information are submitted for the sample, enter “COA”. If the Laboratory Instrument Table (A2) information is also submitted for the sample, enter “COCAL”	Text	6	X
ResultBasis	Indicates whether results associated with this sample records are reported as wet or percent moisture corrected. This field is only required for soils and sediments. Enter “WET” if results are not corrected for	Text	3	X

Table A3
Field Description for the Sample Analysis (Table A3)
This table contains information related to analyses of field samples and laboratory QC samples (excluding calibrations and tunes) on a sample level for environmental chemical analyses including radiochemistry

Field Name	Field Name Description	Field Type	Field Length	Required Value
	percent moisture. Enter "DRY" if percent moisture correction is applied to results.			
TotalOrDissolved	This field indicates if the results related to this sample record are reported as a total or dissolved fraction. This field is only required for metal methods. For all other methods leave this field blank.	Text	3	X
Dilution	Dilution of the sample aliquot. Enter "1" for method blanks, LCS, and LCSD, or if the field samples was analyzed without dilution.	Numeric (1)	10(6)	X
HandlingType	Indicates the type of leaching procedure, if applicable (i.e., SPLP, TCLP, WET). Leave this field blank if the sample analysis was not performed on a leachate.	Text	10	L
HandlingBatch	Unique laboratory identifier for a batch of samples prepared together in a leaching procedure (i.e., SPLP, TCLP, or WET preparation). The HandlingBatch links samples with leaching blanks. Leave this field blank if the sample analysis was not performed on a leachate	Text	12	L
LeachateDate	Date and time of leaching procedure (i.e., date for SPLP, TCLP, or WET preparation). Refer to the date and time format at the end of this table. Leave this field blank if the sample analysis was not performed on a leachate	Date /Time	16*	L
Percent_Moisture	Percent of sample composed of water. Enter for soil and sediment samples only.	Numeric (1)	10(6)	X
MethodBatch	Unique laboratory identifier for a batch of samples of similar matrices analyzed by one method and treated as a group for matrix spike, matrix spike duplicate, or laboratory duplicate association The method batch links the matrix spike and/or matrix spike duplicate or laboratory duplicates to associated samples. Note the MethodBatch association may coincide with the PreparationBatch association. The MethodBatch is specifically used to link the MS/MSD and/or DUP to associated samples.	Text	12	X
PreparationBatch	Unique laboratory identifier for a batch of samples	Text	12	X

Table A3
Field Description for the Sample Analysis (Table A3)
This table contains information related to analyses of field samples and laboratory QC samples (excluding calibrations and tunes) on a sample level for environmental chemical analyses including radiochemistry

Field Name	Field Name Description	Field Type	Field Length	Required Value
	prepared together for analysis by one method and treated as a group for method blank, LCS and LCSD association. The PreparationBatch links method blanks and laboratory control samples (blank spikes) to associated samples. Note, the PreparationBatch association may coincide with the MethodBatch association but the PreparationBatch specifically links the Method Blank and LCS to associated samples.			
RunBatch	For radiochemistry methods leave this field blank. For all other methods the RunBatch is the unique identifier for a batch of analyses performed on one instrument under the control of one initial calibration and initial calibration verification. The RunBatch links both the initial calibration and initial calibration verification to subsequently analyzed and associated continuing calibrations, field samples, and QC analyses. For GC/MS methods, the RunBatch also links a BFB or DFTPP tune. A distinct RunBatch must used with every new initial calibration within a method The value entered in this field links a particular sample/method/analysis type record to a set of associated initial calibration and initial calibration verification records from Table A2. This field is only required if the A2 table is included with the EDD.	Text	12	X
AnalysisBatch	For radiochemistry methods leave this field blank. For all other methods the AnalysisBatch is the unique identifier for a batch of analyses performed on one instrument and under the control of a continuing calibration or continuing calibration verification. The AnalysisBatch links the continuing calibration or calibration verification to subsequently analyzed and associated field sample and QC analyses. For GC/MS methods, the AnalysisBatch also links the BFB or DFTPP tune. A distinct AnalysisBatch must be used with every new continuing calibration or continuing calibration verification within a method The value entered in this field links a particular sample/method/analysis type record to a set of associated continuing calibration records in the Laboratory Instrument table.	Text	12	X

Table A3
Field Description for the Sample Analysis (Table A3)
This table contains information related to analyses of field samples and laboratory QC samples (excluding calibrations and tunes) on a sample level for environmental chemical analyses including radiochemistry

Field Name	Field Name Description	Field Type	Field Length	Required Value
	This field is only required if the A2 table is included with the EDD.			
LabReportingBatch	Unique laboratory identifier for the EDD. This is equivalent to the sample delivery group, lab work number, login ID, etc. The LabReportingBatch links all records in the EDD reported as one group. The value entered in this field must be the same in all records.	Text	12	X
LabReceipt	Date and time the sample was received in the lab. A time value of 00:00 may be entered. Refer to the date/time format at the end of this table.	Date/ Time	16*	X
LabReported	Date and time hard copy reported delivered by the lab. A time value of 00:00 may be entered. Refer to the date/time format at the end of this table.	Date/ Time	16*	X
Comment	Add any comments or additional information specific to the sample analysis data record.	Text	200	

C Only required for regular samples, duplicates and MS/MSDs.
L Only required if analysis performed is SPLP, TCLP or WET extracts.
X Required field.
(1) Field Length indicates decimal precision in parenthesis. For example, 5(2) = a total width of 5 numbers including a maximum of 2 decimal places.
* Format Date and Time as MM/DD/YYYY hh:mm; where MM = two digit month, DD = two digit day, and YYYY = four digit year, hh = hour in 24 hour format, and mm = minutes.

3.0 Laboratory Data Checker

The Laboratory Data Checker is a web-based application that will review Laboratory Electronic Data Deliverables (LEDDs) for adherence to Tetra Tech's EDD format requirements. EDDs will be reviewed for elements such as missing data and/or columns of data, and compliance of the data within each column to the required data types/lengths. Once an EDD passes through the checker with no errors, it must be submitted to Tetra Tech through the LEDD Checker application.

Access to the LEDD Checker application will be provided by an initial registration/approval process. An Information Systems Group (ISG) Administrator will approve requests for access**.** To access the site or begin the registration process, visit the ISG web site at http://isg.ttnus.com and select the "Laboratory Checker" link on the left of the home page. Registered users may access the checker immediately by logging in to the system using theircredentials. New users must select the "Register" button and provide all of the requested information**.**

After completing all fields on the registration form, select the "Submit" button to complete the request process. Upon verification by an ISG Administrator, an email notification will be sent verifying the user ID, password and account status. Forgotten passwords may be retrieved by using the "Forgot password?" link on the login page. Note that the email address that was provided for registration or password retrieval is the user ID and must be a valid e-mail address.

Attachment 3

Declaration of Conformance to the Department of Defense Quality Systems Manual for Environmental Laboratories Version 3 (DoD QSM)

Environmental laboratories performing services for the Department of Defense must possess any required state or host nation certification and/or be accredited for each applicable test method by a nationally recognized laboratory accreditation body (e.g. NELAP) compliant with ISO/IEC 17011:2004. The laboratory must demonstrate the ability to generate acceptable results from the analysis of proficiency-testing (PT) sample(s), subject to availability, using each applicable method in the specified matrix. Upon request, laboratories must make available, to the Department of Defense, the results of all PT samples analyzed by the laboratory during the contract-specified period of performance. Laboratories must have an established and documented laboratory quality system that conforms to ISO/IEC 17025 as implemented by the *DoD Quality Systems Manual for Environmental Laboratories*,[insert appropriate version number. [This document may be downloaded free of charge at https://www.denix.osd.mil/denix/Public/Library/Compliance/EDQW/admin.html. The laboratory must declare conformance to the DoD QSM using this form and must submit a copy of its laboratory quality manual (however named) to an authorized representative of the Department of Defense. All laboratories are subject to on-site assessments by authorized representatives of the Department of Defense. The laboratory must notify the Department of Defense, in writing, of any change in laboratory certification or accreditation status within 30 calendar days of the change.

Laboratory Information

Laboratory Name: Katahdin Analytical Services, Inc.
Address: 600 Technology Way
Scarborough, Maine 04074
Point of Contact: Ms. Leslie Dimond
Title: Quality Assurance Officer
Email: ldimond@katahdinlab.com
Phone: (207) 874-2400
Fax: (207) 775-4029

Title, date of laboratory quality manual

Quality Assurance Manual, November 2008.

List analytes/analyte groups, matrices, methods for which this declaration applies:

Please refer to the attached bid sheet.

Identify laboratory standard operating procedures (SOPs) for which this declaration applies (include number, title and date of last revision):

Please refer to the attached SOP list.

1. Quality System
   a. Does the laboratory quality manual meet the requirements of DoD QSM Sections 4.2.2, 4.2.3, and 4.2.4? Yes
   b. Do laboratory SOPs meet the requirements of DoD QSM Section 5.4.1? Yes
   c. Are laboratory records controlled in accordance with the requirements of DoD QSM Section 4.12? Yes
   d. Do your laboratory personnel structure and lines of responsibility meet the requirements of DoD QSM Section 4.1.5? Yes
   e. Does your laboratory Quality Manager meet requirements of DoD QSM Section 4.1.5(i)? Yes
   f. Does your laboratory perform internal audits, management reviews, and corrective action in accordance with DoD QSM Sections 4.13, 4.14, and 4.10? Yes
2. Training and Ethics
   a. Does your laboratory's technical training program meet the requirements of DoD QSM Section 5.2.6? Yes
   b. Does your laboratory ethics program comply with the requirements of DoD QSM Section 5.2.7? Yes
3. Proficiency Testing
   a. List all proficiency testing programs (e.g. UST, DMRQA) in which your laboratory participates that are relevant to the analytes/analyte groups, matrices, and methods included in the scope of this declaration: Yes
4. Resources
   a. Does your laboratory have adequate personnel, facilities, equipment, instrumentation, and other resources available, as required in DoD QSM Sections 5.2.1, 5.3.1, and 5.5, to perform the tests specified in this declaration? Yes
5. Please list any concerns/comments regarding conformance to the DoD QSM:
Please refer to Table 1 for KAS's proposed exceptions to the DOD QSM ver. 3. Please note, these are proposals only and KAS will make every effort to meet our client's data quality objective

The undersigned acknowledge the accuracy and correctness of this declaration. The undersigned acknowledge that laboratory engagement in any prohibited practice identified in Attachment 3a will be considered material non-compliance, which may result in contract termination or other remedies as appropriate.

Laboratory Quality Manager: Leslie Dimond
Signature: Leslie Dimond
Date: 06.11.09

Laboratory's Authorized Contract Representative: Deborah Nadeau
Signature: Deborah J. Nadeau
Date: 6.11.09

Attachment 3a
Prohibited Practices (Involving Environmental Sampling and Testing Activities)

The following prohibited practices will be considered material non-compliance, which may result in contract termination or other remedies as appropriate.

- Fabrication, falsification, or misrepresentation of data
  - Creating data for an analysis that was not performed
  - Creating information for a sample that was not collected
  - Using external analysts, equipment and/or laboratories to perform analyses when not allowed by contract
- Improper clock setting (time traveling) or improper date/time recording
  - Resetting the internal clock on an instrument to make it appear that a sample was analyzed within holding time when in fact it was not
  - Changing the actual time or recording a false time to make it appear that holding times were met, or changing the times for sample collection, extractions or other steps to make it appear that holding times were met
- Unwarranted manipulation of samples, software, or analytical conditions
  - Unjustified dilution of samples
  - Manipulating GC/MS tuning data to produce an ion abundance result that appears to meet specific QC criteria
  - Changing the instrument conditions for sample analysis from the conditions used for standard analysis (e.g., changing EM voltage)
  - Unwarranted manipulation of computer software, e.g. forcing calibration or QC data to meet criteria, removing computer operational codes such as the "M" flag, inappropriately subtracting background, or improperly manipulating the chromatographic baseline
  - Turning off, or otherwise disabling, electronic instrument audit/tracking functions
- Misrepresenting or misreporting QC samples
  - Representing spiked samples as being digested or extracted when this has not been done
  - Substituting previously generated runs for a non-compliant calibration or QC run to make it appear that an acceptable run was performed
  - Failing to prepare or analyze method blanks and LCS in the same manner that samples were prepared or analyzed
  - Tampering with QC samples and results, including over spiking and adding surrogates after sample extraction
  - Performing multiple calibrations or QC runs (including CCVs, LCSs, spikes, duplicates and blanks) until one meets criteria, rather than taking needed corrective action, and not documenting or retaining data for the other unacceptable data
  - Deleting or failing to record non-compliant QC data to conceal the fact that calibration or other QC analyses were non-compliant
- Improper calibrations
  - Discarding points in the initial calibration to force the calibration to be acceptable
  - Discarding points from an MDL study to force the calculated MDL to be higher or lower than the actual value
  - Using an initial calibration that does not correspond to the actual run sequence to make continuing calibration data look acceptable when in fact it was not
  - Performing improper manual integrations, including peak shaving, peak enhancing, or baseline manipulation to achieve QC criteria or to avoid corrective action
- Concealing a known analytical or sample problem
- Concealing a known improper or unethical behavior or action
- Failing to report the occurrence of a prohibited practice or known improper or unethical act to the appropriate laboratory or contract representative, or to an appropriate government official.

Table 1

KAS Proposed DoD QSM Version 3 Exceptions and Justification

QC check	DoD QSM criteria	KAS proposes	Reasoning
Metals Calibration Blank	No analyte detected > 2 x MDL	No analyte detected > PQL	Due to the instrument sensitivity of the ICP and ICPMS the MDL studies yield very low MDLs. In some cases they may be orders of magnitude below the client's project quantitation limits. It would be very difficult to use MDL criteria to assess blank acceptance.
Metals ICS-A	Absolute value of the concentration for all nonspiked analytes < 2 x MDL	Absolute value of the concentration for all nonspiked analytes < PQL	
Metals Method Blanks	No analyte detected > ½ PQL	No analyte detected > PQL	For most metals this would not be an issue but some metals such as calcium, magnesium, potassium and iron may pose a problem.
GC/MS Internal Standards	Compared to the mid point of the calibration	Compared to the opening CV.	This is a software issue. Currently our software does not produce the forms for comparing the IS to the midpoint of the calibration. We are working to correct this but as of now the form 8's would have to be generated by hand.
VOA and SVOA ICAL and ICV	No samples may be run until ICAL has passed and is verified.	Up to 10% compounds may not meet this criteria.	Due to the large number of compounds that may be analyzed by this method, some compounds may fail to meet the DoD Criteria, especially those that are known to be problematic. KAS narrates if any compound does not meet calibration criteria and in most cases the compound failing is not detected in the samples.

The general process for submitting EDD files through the LEDD Checker involves a 3-stage process that includes an upload stage, an error checking stage and a submittal stage.

Log into the LEDD Checker by typing your login credentials and select the “Login” button. The LEDD Checker home page provides a general overview of the checker functionality and EDD file format requirements. At the bottom of the home page, example EDDs are provided that may be viewed or downloaded. To download the files, right click on the link and select “Save target as” from the menu. Each LEDD Checker page includes a navigation bar with links to return to the home page or continue the checking and submittal process. Users should **NOT** use the back or forward buttons on the browser, instead use the links provided in the application to navigate through the site.

Detailed information regarding EDD preparation, formatting requirements and text file naming conventions are provided in the Electronic Data Format Requirements Section of the Laboratory SOW.

Begin the upload stage by selecting the “Upload/Check Files” link on the home page. Follow the steps on the upload page starting with the selection of the laboratory name that corresponds to your organization. If your organization is not listed, contact LabSupport@tetratech.com, and provide a full description of your organization name, contact information and include “Laboratory Contractor ID Request” in the subject line. An ISG Administrator will respond to the request via e-mail.

Load the appropriate A1, A2, or A3 target EDD files by clicking the “Browse” button next to each data table input box. A file browser dialog will appear allowing files to be selected from a local or network drive. After the EDD files are loaded, click the “Upload” button to complete the upload stage. Note that each table may be uploaded and checked separately; however, a minimum of the A1 and A3 files are required in order to submit the EDDs.

If the file upload was successful, the checking page will immediately load. Begin the checking stage by selecting the “Check Files” button. The LEDD Checker will begin validating the EDD files for compliance. Depending on file size and network activity the validation process may take several minutes. The progress should be displayed in the information bar at the bottom of the browser window. **Do not** select the “Check Files” button again or otherwise use the browser during this process. Other applications may be used; however, note that the LEDD Checker may not sit idle for more than 30 minutes. If the time is exceeded a new session must be started in a new browser window.

Any errors will be processed and returned on the error page. The following general errors may be returned.

- Column count / table structure errors – due to column header names being included, improper delimiter, extra tabs, extra or missing columns of data, spaces or other characters at the end of a row.
- Row and column value specific errors – may occur for one or more reasons including: data truncation, invalid date / time format, invalid decimal precision or field width exceedance, or if a value is not in a list of valid values or expected range.

If column count / table structure errors are encountered, the LEDD Checker will return an error and stop the checking process.

The EDDs will not be processed any further until the column errors are resolved. Text fields are validated for truncation. Date / Time fields are validated for truncation and format compliance. Numeric decimal fields are validated for truncation, character type compliance and decimal precision. All required fields are validated for null values or empty text strings (i.e. spaces). The LEDD Checker will return a list of all errors in and include a reference to the row number on which the error occurred. Note that consecutive EDD files may be loaded and checked, and submitted while logged in. However, no data may be submitted until all EDD files have passed through the LEDD Checker without errors. The list of errors may be printed by selecting the “Print this Page” button from the checker error page.

LABORATORY ACCREDITATION BUREAU

Certificate Number L2223

Certificate of Accreditation

Accredited to DoD ELAP and ISO/IEC 17025:2005

Katahdin Analytical Services, Inc.

600 Technology Way
Scarborough, ME 04074

has met the requirements set forth in L-A-B's policies and procedures, all requirements of ISO/IEC 17025:2005 "General Requirements for the competence of Testing and Calibration Laboratories" and the U.S. Department of Defense Environmental Laboratory Accreditation Program (DoD ELAP).*

The accredited lab has demonstrated technical competence to a defined "Scope of Accreditation" and the operation of a laboratory quality management system (refer to joint ISO-ILAC-IAF Communiqué dated 8 January 2009).

Accreditation Granted through: November 4, 2012

R. Douglas Leonard, Jr., Managing Director
Laboratory Accreditation Bureau
Presented the 4th of November, 2009

*See the laboratory's Scope of Accreditation for details of the DoD ELAP requirements
Laboratory Accreditation Bureau is found to be in compliance with ISO/IEC 17011:2004 and recognized by ILAC (International Laboratory Accreditation Cooperation) and NACLA (National Cooperation for Laboratory Accreditation).

LABORATORY ACCREDITATION BUREAU

Certificate # L2223

Scope of Accreditation For Katahdin Analytical Services

600 Technology Way
Scarborough, ME 04074
Leslie Dimond
1- 207-874-2400

In recognition of a successful assessment to ISO/IEC 17025:2005 and the requirements of the DoD Environmental Laboratory Accreditation Program (DoD ELAP) as detailed in the DoD Quality Systems Manual for Environmental Laboratories (DoD QSM v4.1) based on the National Environmental Laboratory Accreditation Conference Chapter 5 Quality Systems Standard (NELAC Voted Revision June 5, 2003), accreditation is granted to Katahdin Analytical Services to perform the following tests:

Accreditation granted through: **November 4, 2012**

Testing - Environmental

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/ECD	608 / 8081A,B/ SOM01.2	4 4`-DDD
GC/ECD	608 / 8081A,B/ SOM01.2	4 4`-DDE
GC/ECD	608 / 8081A,B / SOM01.2	4 4`-DDT
GC/ECD	608 / 8081A,B / SOM01.2	Aldrin
GC/ECD	608 / 8081A,B / SOM01.2	alpha-BHC (alpha-Hexachlorocyclohexane)
GC/ECD	8081A,B / SOM01.2	Alpha-Chlordane
GC/ECD	608 / 8081A,B / SOM01.2	beta-BHC (beta-Hexachlorocyclohexane)
GC/ECD	608 /8081A,B	Chlordane (tech.)
GC/ECD	608 / 8081A,B / SOM01.2	delta-BHC
GC/ECD	608 / 8081A,B / SOM01.2	Dieldrin
GC/ECD	608 / 8081A,B / SOM01.2	Endosulfan I
GC/ECD	608 / 8081A,B / SOM01.2	Endosulfan II
GC/ECD	608 / 8081A,B / SOM01.2	Endosulfan sulfate
GC/ECD	608 / 8081A,B / SOM01.2	Endrin
GC/ECD	608 / 8081A,B / SOM01.2	Endrin aldehyde
GC/ECD	8081A,B / SOM01.2	Endrin Ketone
GC/ECD	8081A,B / SOM01.2	gamma-BHC (Lindane gamma-Hexachlorocyclohexane)
GC/ECD	608 / 8081A,B / SOM01.2	Heptachlor
GC/ECD	608 / 8081A,B / SOM01.2	Heptachlor epoxide

LABORATORY ACCREDITATION BUREAU

Certificate # L2223

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/ECD	8081A,B / SOM01.2	Methoxychlor
GC/ECD	608 / 8081A,B / SOM01.2	Toxaphene (Chlorinated camphene)
GC/ECD	608 / 8082/8082A / SOM01.2	Aroclor-1221 (PCB-1221)
GC/ECD	608 / 8082/8082A / SOM01.2	Aroclor-1232 (PCB-1232)
GC/ECD	608 / 8082/8082A / SOM01.2	Aroclor-1242 (PCB-1242)
GC/ECD	608 / 8082/8082A / SOM01.2	Aroclor-1248 (PCB-1248)
GC/ECD	608 / 8082/8082A / SOM01.2	Aroclor-1254 (PCB-1254)
GC/ECD	608 / 8082/8082A / SOM01.2	Aroclor-1260 (PCB-1260)
GC/ECD	8082/8082A	Aroclor-1262 (PCB-1262)
GC/ECD	8082/8082A	Aroclor-1268 (PCB-1268)
GC/ECD	8082/8082A	2 2' 3 3' 4 4' 5 5' 6-Nonachlorobiphenyl (BZ 206)
GC/ECD	8082/8082A	2 2' 3 3' 4 4' 5 6-Octachlorobiphenyl (BZ 195)
GC/ECD	8082/8082A	2 2' 3 3' 4 4' 5-Heptachlorobiphenyl (BZ 170)
GC/ECD	8082/8082A	2 2' 3 3' 4 4'-Hexachlorobiphenyl (BZ 128)
GC/ECD	8082/8082A	2 2' 3 4 4' 5 5'-Heptachlorobiphenyl (BZ 180)
GC/ECD	8082/8082A	2 2' 3 4 4' 5' 6-Heptachlorobiphenyl (BZ 183)
GC/ECD	8082/8082A	2 2' 3 4 4' 5'-Hexachlorobiphenyl (BZ 138)
GC/ECD	8082/8082A	2 2' 3 4 4' 6 6'-Heptachlorobiphenyl (BZ 184)
GC/ECD	8082/8082A	2 2' 3 4' 5 5' 6-Heptachlorobiphenyl (BZ 187)
GC/ECD	8082/8082A	2 2' 3 4 5'-Pentachlorobiphenyl (BZ 87)
GC/ECD	8082/8082A	2 2' 3 5'-Tetrachlorobiphenyl (BZ 44)
GC/ECD	8082/8082A	2 2' 4 4' 5 5'-Hexachlorobiphenyl (BZ 153)
GC/ECD	8082/8082A	2 2' 4 5 5'-Pentachlorobiphenyl (BZ 101)
GC/ECD	8082/8082A	2 2' 4' 5-Tetrachlorobiphenyl (BZ 49)
GC/ECD	8082/8082A	2 2' 5 5'-Tetrachlorobiphenyl (BZ 52)
GC/ECD	8082/8082A	2 2' 5-Trichlorobiphenyl (BZ 18)
GC/ECD	8082/8082A	2 3 3' 4 4' 5-Hexachlorobiphenyl (BZ 156)
GC/ECD	8082/8082A	2 3 3' 4 4' 5'-Hexachlorobiphenyl (BZ 157)
GC/ECD	8082/8082A	2 3 3' 4 4'-Pentachlorobiphenyl (BZ 105)
GC/ECD	8082/8082A	2 3 3’ 4 4’ 5 5’-Heptachlorobiphenyl (BZ 189)
GC/ECD	8082/8082A	2 3' 4 4' 5 5'-Hexachlorobiphenyl (BZ 167)
GC/ECD	8082/8082A	2 3' 4 4' 5-Pentachlorobiphenyl (BZ 118)
GC/ECD	8082/8082A	2 3' 4 4'5-Pentachlorobiphenyl (BZ 123)
GC/ECD	8082/8082A	2 3' 4 4'-Tetrachlorobiphenyl (BZ 66)
GC/ECD	8082/8082A	2 3’ 4 4’ 5-Pentachlorobiphenyl (BZ 114)
GC/ECD	8082/8082A	2 4 4'-Trichlorobiphenyl (BZ 28)
GC/ECD	8082/8082A	2 4'-Dichlorobiphenyl (BZ 8)
GC/ECD	8082/8082A	3 3' 4 4' 5 5'-Hexachlorobiphenyl (BZ 169)
GC/ECD	8082/8082A	3 3' 4 4' 5-Pentachlorobiphenyl (BZ 126)
GC/ECD	8082/8082A	3 3' 4 4'-Tetrachlorobiphenyl (BZ 77)
GC/ECD	8082/8082A	3 4 4’ 5-Tetrachlorobiphenyl (BZ 81)
GC/ECD	8082/8082A	Decachlorobiphenyl (BZ 209)
GC/ECD	8151A	2 4 5-T
GC/ECD	8151A	2 4-D
GC/ECD	8151A	2 4-DB

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/ECD	8151A	Dalapon
GC/ECD	8151A	Dicamba
GC/ECD	8151A	Dichloroprop
GC/ECD	8151A	DInoseb
GC/ECD	8151A	MCPA
GC/ECD	8151A	MCPP
GC/ECD	8151A	Pentachlorophenol
GC/ECD	8151A	Silvex (2 4 5-TP)
GC/FID	8015B/C	Diesel range organics (DRO)
GC/FID	8015B/C	Gasoline range organics (GRO)
GC/FID	8011 / 504	1 2-Dibromoethane (EDB)
GC/FID	8011 / 504	1 2-Dibromo-3-chloropropane
GC/FID	RSK-175	Methane Ethane Ethene
GC/MS	8260B,C / 524.2	1 1 1 2-Tetrachloroethane
GC/MS	624 / 8260B,C / SOM01.2 / 524.2	1 1 1-Trichloroethane
GC/MS	624 / 8260B,C / SOM01.2 / 524.2	1 1 2 2-Tetrachloroethane
GC/MS	SOM01.2	1 1 2-Trichloro-1 2 2-trifluoroethane
GC/MS	624 / 8260B,C / SOM01.2 / 524.2	1 1 2-Trichloroethane
GC/MS	624 / 8260B,C / SOM01.2 / 524.2	1 1-Dichloroethane
GC/MS	624 / 8260B,C / SOM01.2 / 524.2	1 1-Dichloroethene
GC/MS	8260B,C / 524.2	1 1-Dichloropropene
GC/MS	8260B,C / SOM01.2 / 524.2	1 2 3-Trichlorobenzene
GC/MS	8260B,C / 524.2	1 2 3-Trichloropropane
GC/MS	8260B,C / SOM01.2 / 524.2	1 2 4-Trichlorobenzene
GC/MS	8260B,C / 524.2	1 2 4-Trimethylbenzene
GC/MS	8260B,C / SOM01.2 / 524.2	1 2-Dibromo-3-chloropropane
GC/MS	8260B,C / SOM01.2 / 524.2	1 2-Dibromoethane (EDB)
GC/MS	624 / 8260B,C / SOM01.2 / 524.2	1 2-Dichlorobenzene
GC/MS	624 / 8260B,C / SOM01.2 / 524.2	1 2-Dichloroethane
GC/MS	624 / 8260B,C / SOM01.2 / 524.2	1 2-Dichloropropane
GC/MS	8260B,C / 524.2	1 3 5-Trimethylbenzene
GC/MS	624 / 8260B,C / SOM01.2 / 524.2	1 3-Dichlorobenzene
GC/MS	8260B,C / 524.2	1 3-Dichloropropane
GC/MS	624 / 8260B,C / SOM01.2 / 524.2	1 4-Dichlorobenzene
GC/MS	8260B,C / SOM01.2	1 4-Dioxane
GC/MS	8260B,C / 524.2	2 2-Dichloropropane

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	8260B,C / SOM01.2 / 524.2	2-Butanone
GC/MS	624 / 8260B,C	2-Chloroethyl vinyl ether
GC/MS	8260B,C / 524.2	2-Chlorotoluene
GC/MS	8260B,C / SOM01.2 / 524.2	2-Hexanone
GC/MS	8260B,C / 524.2	4-Chlorotoluene
GC/MS	8260B,C / SOM01.2 / 524.2	4-Methyl-2-pentanone
GC/MS	8260B,C / SOM01.2 / 524.2	Acetone
GC/MS	8260B,C	Acetonitrile
GC/MS	624 / 8260B,C	Acrolein
GC/MS	624 / 8260B,C / 524.2	Acrylonitrile
GC/MS	8260B,C / 524.2	Allyl chloride
GC/MS	624 / 8260B,C / SOM01.2 / 524.2	Benzene
GC/MS	8260B,C / 524.2	Bromobenzene
GC/MS	8260B,C / SOM01.2 / 524.2	Bromochloromethane
GC/MS	624 / 8260B,C / SOM01.2 / 524.2	Bromodichloromethane
GC/MS	624 / 8260B,C / SOM01.2 / 524.2	Bromoform
GC/MS	8260B,C / SOM01.2 / 524.2	Carbon disulfide
GC/MS	624 / 8260B,C / SOM01.2 / 524.2	Carbon tetrachloride
GC/MS	624 / 8260B,C / SOM01.2 / 524.2	Chlorobenzene
GC/MS	624 / 8260B,C / SOM01.2 / 524.2	Chloroethane
GC/MS	624 / 8260B,C / SOM01.2 / 524.2	Chloroform
GC/MS	8260B,C	Chloroprene
GC/MS	8260B,C / SOM01.2 / 524.2	cis-1 2-Dichloroethene
GC/MS	624 / 8260B,C / SOM01.2 / 524.2	cis-1 3-Dichloropropene
GC/MS	SOM01.2	Cyclohexane
GC/MS	624 / 8260B,C / SOM01.2 / 524.2	Dibromochloromethane
GC/MS	8260B,C / 524.2	Dibromomethane
GC/MS	624 / 8260B,C / SOM01.2 / 524.2	Dichlorodifluoromethane
GC/MS	8260B,C / 524.2	Diethyl ether
GC/MS	8260B,C	Di-isopropylether
GC/MS	8260B,C / 524.2	Ethyl methacrylate
GC/MS	624 / 8260B,C / SOM01.2 / 524.2	Ethylbenzene
GC/MS	8260B,C	Ethyl-t-butylether
GC/MS	8260B,C / 524.2	Hexachlorobutadiene
GC/MS	8260B,C	Iodomethane
GC/MS	8260B,C	Isobutyl alcohol

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	8260B,C / SOM01.2 / 524.2	Isopropyl benzene
GC/MS	8260B,C / SOM01.2 / 524.2	m p-xylenes
GC/MS	8260B,C / 524.2	Methacrylonitrile
GC/MS	SOM01.2	Methyl acetate
GC/MS	624 / 8260B,C / SOM01.2 / 524.2	Methyl bromide (Bromomethane)
GC/MS	624 / 8260B,C / SOM01.2 / 524.2	Methyl chloride (Chloromethane)
GC/MS	8260B,C / 524.2	Methyl methacrylate
GC/MS	8260B,C / SOM01.2 / 524.2	Methyl tert-butyl ether
GC/MS	SOM01.2	Methylcyclohexane
GC/MS	624 / 8260B,C / SOM01.2 / 524.2	Methylene chloride
GC/MS	8260B,C / 524.2	Naphthalene
GC/MS	8260B,C / 524.2	n-Butylbenzene
Gc/ms	8260B,C / 524.2	n-Propylbenzene
GC/MS	8260B,C / SOM01.2 / 524.2	o-Xylene
GC/MS	8260B,C / 524.2	p-Isopropyltoluene
GC/MS	8260B,C / 524.2	Propionitrile
GC/MS	8260B,C / 524.2	sec-butylbenzene
GC/MS	8260B,C / SOM01.2 / 524.2	Styrene
GC/MS	8260B,C	t-Amylmethylether
GC/MS	8260B,C / 524.2	tert-Butyl alcohol
GC/MS	8260B,C	tert-Butylbenzene
GC/MS	624 / 8260B,C / SOM01.2 / 524.2	Tetrachloroethene (Perchloroethylene)
GC/MS	8260B,C / 524.2	Tetrahydrofuran
GC/MS	624 / 8260B,C / SOM01.2 / 524.2	Toluene
GC/MS	624 / 8260B,C / SOM01.2 / 524.2	trans-1 2-Dichloroethylene
GC/MS	624 / 8260B,C / SOM01.2 / 524.2	trans-1 3-Dichloropropylene
GC/MS	8260B,C / 524.2	trans-1 4-Dichloro-2-butuene
GC/MS	624 / 8260B,C / SOM01.2 / 524.2	Trichloroethene (Trichloroethylene)
GC/MS	624 / 8260B,C / SOM01.2 / 524.2	Trichlorofluoromethane
GC/MS	8260B,C	Vinyl acetate
GC/MS	624 / 8260B,C / SOM01.2 / 524.2	Vinyl chloride
GC/MS	624 8260B,C	Xylene
GC/MS	8270C,D / SOM01.2	1 2 4 5-Tetrachlorobenzene
GC/MS	625 / 8270C,D / SOM01.2	1 2 4-Trichlorobenzene
GC/MS	625 / 8270C,D / SOM01.2	1 2-Dichlorobenzene
GC/MS	8270C,D	1 2-Diphenylhydrazine

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	8270C,D	1 3 5-Trinitrobenzene
GC/MS	625 / 8270C,D / SOM01.2	1 3-Dichlorobenzene
GC/MS	8270C,D	1 3-Dinitrobenzene
GC/MS	625 / 8270C,D / SOM01.2	1 4-Dichlorobenzene
GC/MS	8270C,D	1 4-Dioxane
GC/MS	8270C,D	1 4-Naphthoquinone
GC/MS	8270C,D	1 4-Phenylenediamine
GC/MS	8270C,D	1-Naphthylamine
GC/MS	8270C,D / SOM01.2	2 3 4 6-Tetrachlorophenol
GC/MS	8270C,D / SOM01.2	2 4 5-Trochlorophenol
GC/MS	625 / 8270C,D / SOM01.2	2 4 6-Trichlorophenol
GC/MS	625 / 8270C,D / SOM01.2	2 4-Dichlorophenol
GC/MS	625 / 8270C,D / SOM01.2	2 4-Dimethylphenol
GC/MS	625 / 8270C,D / SOM01.2	2 4-Dinitrophenol
GC/MS	625 / 8270C,D / SOM01.2	2 4-Dinitrotoluene (2 4-DNT)
GC/MS	8270C,D	2 6-Dichlorophenol
GC/MS	625 / 8270C,D / SOM01.2	2 6-Dinitrotoluene (2 6-DNT)
GC/MS	8270C,D	2-Acetylaminofluorene
GC/MS	625 / 8270C,D / SOM01.2	2-Chloronaphthalene
GC/MS	625 / 8270C,D / SOM01.2	2-Chlorophenol
GC/MS	625 / 8270C,D / SOM01.2	2-Methyl-4 6-dinitrophenol
GC/MS	8270C,D / SOM01.2	2-Methylnaphthalene
GC/MS	8270C,D / SOM01.2	2-Methylphenol
GC/MS	8270C,D	2-Naphthylamine
GC/MS	8270C,D	2-Nitroaniline
GC/MS	625 / 8270C,D / SOM01.2	2-Nitrophenol
GC/MS	8270C,D	2-Picoline
GC/MS	625 / 8270C,D / SOM01.2	3 3'-Dichlorobenzidine
GC/MS	8270C,D	3 3'-Dimethylbenzidine
GC/MS	8270C,D	3-Methylcholanthrene
GC/MS	8270C,D / SOM01.2	3-Nitroaniline
GC/MS	8270C,D	4-Aminobiphenyl
GC/MS	625 / 8270C,D / SOM01.2	4-Bromophenyl phenyl ether
GC/MS	625 / 8270C,D / SOM01.2	4-Chloro-3-methylphenol
GC/MS	8270C,D / SOM01.2	4-Chloroaniline
GC/MS	625 / 8270C,D / SOM01.2	4-Chlorophenyl phenylether
GC/MS	8270C,D	4-Dimethyl aminoazobenzene
GC/MS	8270C,D / SOM01.2	4-Methylphenol
GC/MS	8270C,D / SOM01.2	4-Nitroaniline
GC/MS	625 / 8270C,D / SOM01.2	4-Nitrophenol
GC/MS	8270C,D	5-Nitro-o-toluidine
GC/MS	8270C,D	7,12-Dimethylphenethylamine
GC/MS	8270C,D	a a-Dimethylphenethylamine
GC/MS	625 / 8270C,D / SOM01.2	Acenaphthene
GC/MS	625 / 8270C,D / SOM01.2	Acenaphthylene

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	8270C,D / SOM01.2	Acetophenone
GC/MS	8270C,D	Aniline
GC/MS	625 / 8270C,D / SOM01.2	Anthracene
GC/MS	8270C,D	Aramite
GC/MS	8270C,D / SOM01.2	Atrazine
GC/MS	SOM01.2	Benzaldehyde
GC/MS	625 / 8270C,D	Benzidine
GC/MS	625 / 8270C,D / SOM01.2	Benzo(a)anthracene
GC/MS	625 / 8270C,D / SOM01.2	Benzo(a)pyrene
GC/MS	625 / 8270C,D / SOM01.2	Benzo(b)fluoranthene
GC/MS	625 / 8270C,D / SOM01.2	Benzo(g h i)perylene
GC/MS	625 / 8270C,D / SOM01.2	Benzo(k)fluoranthene
GC/MS	8270C,D	Benzoic Acid
GC/MS	8270C,D	Benzyl alcohol
GC/MS	8270C,D / SOM01.2	Biphenyl
GC/MS	625 / 8270C,D / SOM01.2	bis(2-Chloroethoxy)methane
GC/MS	625 / 8270C,D / SOM01.2	bis(2-Chloroethyl) ether
GC/MS	625 / 8270C,D / SOM01.2	bis(2-Chloroisopropyl) ether (2 2`-Oxybis(1-chloropropane))
GC/MS	625 / 8270C,D / SOM01.2	bis(2-Ethylhexyl) phthalate (DEHP)
GC/MS	625 / 8270C,D / SOM01.2	Butyl benzyl phthalate
GC/MS	SOM01.2	Caprolactam
GC/MS	8270C,D / SOM01.2	Carbazole
GC/MS	8270C,D	Chlorobenzilate
GC/MS	625 / 8270C,D / SOM01.2	Chrysene
GC/MS	8270C,D	Diallate
GC/MS	625 / 8270C,D / SOM01.2	Dibenz(a h)anthracene
GC/MS	8270C,D / SOM01.2	Dibenzofuran
GC/MS	625 / 8270C,D / SOM01.2	Diethyl phthalate
GC/MS	8270C,D	Dimethoate
GC/MS	625 / 8270C,D / SOM01.2	Dimethyl phthalate
GC/MS	625 / 8270C,D / SOM01.2	Di-n-butyl phthalate
GC/MS	625 / 8270C,D / SOM01.2	Di-n-octyl phthalate
GC/MS	8270C,D	Ethyl methanesulfonate
GC/MS	8270C,D	Famfur
GC/MS	625 / 8270C,D / SOM01.2	Fluoranthene
GC/MS	625 / 8270C,D / SOM01.2	Fluorene
GC/MS	625 / 8270C,D / SOM01.2	Hexachlorobenzene
GC/MS	625 / 8270C,D / SOM01.2	Hexachlorobutadiene
GC/MS	625 / 8270C,D / SOM01.2	Hexachlorocyclopentadiene
GC/MS	625 / 8270C,D / SOM01.2	Hexachloroethane
GC/MS	8270C,D	Hexachloropropene
GC/MS	625 / 8270C,D / SOM01.2	Indeno(1 2 3-cd)pyrene
GC/MS	8270C,D	Isodrin
GC/MS	625 / 8270C,D / SOM01.2	Isophorone

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	8270C,D	Isosafrole
GC/MS	8270C,D	Methapyriline
GC/MS	8270C,D	Methy methanesulfonate
GC/MS	8270C,D	Methyl parathion
GC/MS	625 / 8270C,D / SOM01.2	Naphthalene
GC/MS	625 / 8270C,D / SOM01.2	Nitrobenzene
GC/MS	8270C,D	Nitroquinoline-1-oxide
GC/MS	8270C,D	n-Nitrosodiethylamine
GC/MS	625 / 8270C,D / SOM01.2	n-Nitrosodimethylamine
GC/MS	8270C,D	n-Nitroso-di-n-butylamine
GC/MS	625 / 8270C,D / SOM01.2	n-Nitrosodi-n-propylamine
GC/MS	625 / 8270C,D / SOM01.2	n-Nitrosodiphenylamine
GC/MS	8270C,D	n-Nitrosomethylethylamine
GC/MS	8270C,D	n-Nitrosomorpholine
GC/MS	8270C,D	n-Nitrosopiperidine
GC/MS	8270C,D	n-Nitrosopyrrolidine
GC/MS	8270C,D	o o o-Triethyl phosphorothioate
GC/MS	8270C,D	o-Toluidine
GC/MS	8270C,D	Pentachlorobenzene
GC/MS	8270C,D	Pentachloronitrobenzene
GC/MS	625 / 8270C,D / SOM01.2	Pentachlorophenol
GC/MS	8270C,D	Phenacetin
GC/MS	625 / 8270C,D / SOM01.2	Phenanthrene
GC/MS	625 / 8270C,D / SOM01.2	Phenol
GC/MS	8270C,D	Phorate
GC/MS	8270C,D	Pronamide
GC/MS	625 / 8270C,D / SOM01.2	Pyrene
GC/MS	8270C,D	Pyrididne
GC/MS	8270C,D	Safrole
GC/MS	8270C,D	Thionazin
HPLC	8330/8330A/8330B	1 3 5-Trinitrobenzene
HPLC	8330/8330A/8330B	1 3-Dinitrobenzene
HPLC	8330/8330A/8330B	2 4 6-Trinitrotoluene
HPLC	8330/8330A/8330B	2 4-Dinitrotoluene
HPLC	8330/8330A/8330B	2 6-Dinitrotoluene
HPLC	8330/8330A/8330B	2-Amino-4 6 -dinitrotoluene
HPLC	8330/8330A/8330B	2-Nitrotoluene
HPLC	8330/8330A/8330B	3-Nitrotoluene
HPLC	8330/8330A/8330B	4-Amino-2,3-dinitrotoluene
HPLC	8330/8330A/8330B	4-Nitrotoluene
HPLC	8330/8330A/8330B	Hexahydro-1 3 5-trinitro-1 3 5-triazine (RDX)
HPLC	8330/8330A/8330B	Nitrobenzene
HPLC	8330/8330A/8330B	Nitroglycerin
HPLC	8330/8330A/8330B	Octahydro-1 3 5 7-tetrazocine (HMX)
HPLC	8330/8330A/8330B	Tetryl

Non-Potable Water		
Technology	**Method**	**Analyte**
CVAA	245.1 / 7470A / ILM05.3	Mercury
CVAF	1631E	Low Level Mercury
ICP	200.7 / 6010B,C / ILM05.3	Aluminum
ICP	200.7 / 6010B,C / ILM05.3	Antimony
ICP	200.7 / 6010B,C / ILM05.3	Arsenic
ICP	200.7 / 6010B,C / ILM05.3	Barium
ICP	200.7 / 6010B,C / ILM05.3	Beryllium
ICP	200.7 / 6010B,C	Boron
ICP	200.7 / 6010B,C / ILM05.3	Cadmium
ICP	200.7 / 6010B,C / ILM05.3	Calcium
ICP	200.7 / 6010B,C / ILM05.3	Chromium
ICP	200.7 / 6010B,C / ILM05.3	Cobalt
ICP	200.7 / 6010B,C / ILM05.3	Copper
ICP	200.7 / 6010B,C / ILM05.3	Iron
ICP	200.7 / 6010B,C / ILM05.3	Lead
ICP	200.7 / 6010B,C / ILM05.3	Magnesium
ICP	200.7 / 6010B,C / ILM05.3	Manganese
ICP	200.7 / 6010B,C	Molybdenum
ICP	200.7 / 6010B,C / ILM05.3	Nickel
ICP	200.7 / 6010B,C / ILM05.3	Potassium
ICP	200.7 / 6010B,C / ILM05.3	Selenium
ICP	200.7	Silicon
ICP	200.7 / 6010B,C / ILM05.3	Silver
ICP	200.7 / 6010B,C / ILM05.3	Sodium
ICP	6010B,C	Strontium
ICP	200.7 / 6010B,C / ILM05.3	Thallium
ICP	200.7 / 6010B,C	Tin
ICP	200.7 / 6010B,C	Titanium
ICP	200.7 / 6010B,C / ILM05.3	Vanadium
ICP	200.7 / 6010B,C / ILM05.3	Zinc
ICP/MS	200.8 / 6020/6020A / ILM05.3	Aluminum
ICP/MS	200.8 / 6020/6020A / ILM05.3	Antimony
ICP/MS	200.8 / 6020/6020A / ILM05.3	Arsenic
ICP/MS	200.8 / 6020/6020A / ILM05.3	Barium
ICP/MS	200.8 / 6020/6020A / ILM05.3	Beryllium
ICP/MS	200.8 / 6020/6020A	Boron
ICP/MS	200.8 / 6020/6020A / ILM05.3	Cadmium
ICP/MS	200.8 / 6020/6020A	Calcium
ICP/MS	200.8 / 6020/6020A / ILM05.3	Chromium
ICP/MS	200.8 / 6020/6020A / ILM05.3	Cobalt
ICP/MS	200.8 / 6020/6020A / ILM05.3	Copper
ICP/MS	200.8 / 6020/6020A	Iron
ICP/MS	200.8 / 6020/6020A / ILM05.3	Lead
ICP/MS	200.8 / 6020/6020A / ILM05.3	Magnesium
ICP/MS	200.8 / 6020/6020A	Manganese

Non-Potable Water		
Technology	**Method**	**Analyte**
ICP/MS	200.8 / 6020/6020A	Molybdenum
ICP/MS	200.8 / 6020/6020A / ILM05.3	Nickel
ICP/MS	200.8 / 6020/6020A	Potassium
ICP/MS	200.8 / 6020/6020A / ILM05.3	Selenium
ICP/MS	200.8 / 6020/6020A / ILM05.3	Silicon
ICP/MS	200.8 / 6020/6020A / ILM05.3	Silver
ICP/MS	200.8 / 6020/6020A	Sodium
ICP/MS	6020/6020A	Strontium
ICP/MS	200.8 / 6020/6020A / ILM05.3	Thallium
ICP/MS	200.8 / 6020/6020A	Tin
ICP/MS	200.8 / 6020/6020A	Titanium
ICP/MS	200.8	Uranium
ICP/MS	200.8 / 6020/6020A / ILM05.3	Vanadium
ICP/MS	200.8 / 6020/6020A / ILM05.3	Zinc
IC	300.0 / 9056/9056A	Bromide
IC	300.0 / 9056/9056A	Chloride
IC	300.0 / 9056/9056A	Nitrate as N
IC	300.0 / 9056/9056A	Nitrite as N
IC	300.0 / 9056/9056A	Nitrate + Nitrite
IC	300.0 / 9056/9056A	Orthophosphate as P
IC	300.0 / 9056/9056A	Sulfate
Titration	310.2 / 2320B	Alkalinity
Caculation	2340C	Hardness
Gravimetric	1664A	Oil and Grease
Gravimetric	2540 B, C, D	Solids
ISE	120.1 / 2510 B	Conductivity
ISE	2520B	Practical Salinity
ISE	4500F- C	Fluoride
ISE	4500H+ B	pH
ISE	5210B	TBOD / CBOD
Physical	1010 A	Ignitability
Physical	9040C	pH
Titration	2340B	Hardness
Titration	4500SO_3 B	Sulfite
Titration	9034 / 4500S^{2-} E	Sulfide
Titration	Chap. 7.3.4	Reactive Sulfide
TOC	9060A / 5310B	Total organic carbon
Turbidimetric	180.1 / 2130B	Turbidity
Turbidimetric	9038 / ASTM 516-02	Sulfate
UV/VIS	335.4 / 9012B / 4500-CN G	Amenable cyanide
UV/VIS	350.1 / 4500NH3 H	Ammonia as N
UV/VIS	3500Fe D	Ferrous Iron
UV/VIS	351.2	Kjeldahl nitrogen - total
UV/VIS	353.2 / 4500NO3 F	Nitrate + Nitrite

Non-Potable Water		
Technology	**Method**	**Analyte**
UV/VIS	353.2 / 4500NO3 F	Nitrate as N
UV/VIS	353.2 / 4500NO3 F	Nitrite as N
UV/VIS	365.1 / 4500P E	Orthophosphate as P
UV/VIS	365.4	Phosphorus total
UV/VIS	376.3	AVS-SEM
UV/VIS	410.4	COD
UV/VIS	420.1 / 9065	Total Phenolics
UV/VIS	4500Cl G	Total Residual Chlorine
UV/VIS	5540C	MBAS
UV/VIS	7196A / 3500-Cr D	Chromium VI
UV/VIS	9012B / ILM05.3/ 335.4	Total Cyanide
UV/VIS	9251 / 4500Cl E	Chloride
UV/VIS	Chap. 7.3.4	Reactive Cyanide
Preparation	**Method**	**Type**
Cleanup Methods	3640A	Gel Permeation Clean-up
Cleanup Methods	3630C	Silica Gel
Cleanup Methods	3660B	Sulfur Clean-Up
Cleanup Methods	3665A	Sulfuric Acid Clean-Up
Organic Preparation	3510C	Separatory Funnel Extraction
Organic Preparation	3520C	Continuous Liquid-Liquid Extraction
Inorganic Preparation	3010A	Hotblock
Volatile Organic Preparation	5030B,C	Purge and Trap

Solid and Chemical Waste		
Technology	**Method**	**Analyte**
GC/ECD	8081A,B/ SOM01.2	4 4`-DDD
GC/ECD	8081A,B / SOM01.2	4 4`-DDE
GC/ECD	8081A,B / SOM01.2	4 4`-DDT
GC/ECD	8081A,B / SOM01.2	Aldrin
GC/ECD	8081A,B / SOM01.2	alpha-BHC (alpha-Hexachlorocyclohexane)
GC/ECD	8081A,B / SOM01.2	Alpha-Chlordane
GC/ECD	8081A,B / SOM01.2	beta-BHC (beta-Hexachlorocyclohexane)
GC/ECD	608 /8081A,B	Chlordane (tech.)
GC/ECD	8081A,B / SOM01.2	delta-BHC
GC/ECD	8081A,B / SOM01.2	Dieldrin
GC/ECD	8081A,B / SOM01.2	Endosulfan I
GC/ECD	8081A,B / SOM01.2	Endosulfan II
GC/ECD	8081A,B / SOM01.2	Endosulfan sulfate
GC/ECD	8081A,B / SOM01.2	Endrin
GC/ECD	8081A,B / SOM01.2	Endrin aldehyde
GC/ECD	8081A,B / SOM01.2	Endrin Ketone

Solid and Chemical Waste		
Technology	**Method**	**Analyte**
GC/ECD	8081A,B / SOM01.2	gamma-BHC (Lindane gamma-Hexachlorocyclohexane)
GC/ECD	8081A,B / SOM01.2	Heptachlor
GC/ECD	8081A,B / SOM01.2	Heptachlor epoxide
GC/ECD	8081A,B / SOM01.2	Methoxychlor
GC/ECD	8081A,B / SOM01.2	Toxaphene (Chlorinated camphene)
GC/ECD	8082/8082A/ SOM01.2	Aroclor-1016 (PCB-1016)
GC/ECD	8082/8082A/ SOM01.2	Aroclor-1221 (PCB-1221)
GC/ECD	8082/8082A/ SOM01.2	Aroclor-1232 (PCB-1232)
GC/ECD	8082/8082A/ SOM01.2	Aroclor-1242 (PCB-1242)
GC/ECD	8082/8082A/ SOM01.2	Aroclor-1248 (PCB-1248)
GC/ECD	8082/8082A/ SOM01.2	Aroclor-1254 (PCB-1254)
GC/ECD	8082/8082A/ SOM01.2	Aroclor-1260 (PCB-1260)
GC/ECD	8082/8082A	Aroclor-1262 (PCB-1262)
GC/ECD	8082/8082A	Aroclor-1268 (PCB-1268)
GC/ECD	8082/8082A	2 2' 3 3' 4 4' 5 5' 6-Nonachlorobiphenyl (BZ 206)
GC/ECD	8082/8082A	2 2' 3 3' 4 4' 5 6-Octachlorobiphenyl (BZ 195)
GC/ECD	8082/8082A	2 2' 3 3' 4 4' 5-Heptachlorobiphenyl (BZ 170)
GC/ECD	8082/8082A	2 2' 3 3' 4 4'-Hexachlorobiphenyl (BZ 128)
GC/ECD	8082/8082A	2 2' 3 4 4' 5 5'-Heptachlorobiphenyl (BZ 180)
GC/ECD	8082/8082A	2 2' 3 4 4' 5' 6-Heptachlorobiphenyl (BZ 183)
GC/ECD	8082/8082A	2 2' 3 4 4' 5'-Hexachlorobiphenyl (BZ 138)
GC/ECD	8082/8082A	2 2' 3 4 4' 6 6'-Heptachlorobiphenyl (BZ 184)
GC/ECD	8082/8082A	2 2' 3 4' 5 5' 6-Heptachlorobiphenyl (BZ 187)
GC/ECD	8082/8082A	2 2' 3 4 5'-Pentachlorobiphenyl (BZ 87)
GC/ECD	8082/8082A	2 2' 3 5'-Tetrachlorobiphenyl (BZ 44)
GC/ECD	8082/8082A	2 2' 4 4' 5 5'-Hexachlorobiphenyl (BZ 153)
GC/ECD	8082/8082A	2 2' 4 5 5'-Pentachlorobiphenyl (BZ 101)
GC/ECD	8082/8082A	2 2' 4' 5-Tetrachlorobiphenyl (BZ 49)
GC/ECD	8082/8082A	2 2' 5 5'-Tetrachlorobiphenyl (BZ 52)
GC/ECD	8082/8082A	2 2' 5-Trichlorobiphenyl (BZ 18)
GC/ECD	8082/8082A	2 3 3' 4 4' 5-Hexachlorobiphenyl (BZ 156)
GC/ECD	8082/8082A	2 3 3' 4 4' 5'-Hexachlorobiphenyl (BZ 157)
GC/ECD	8082/8082A	2 3 3' 4 4'-Pentachlorobiphenyl (BZ 105)
GC/ECD	8082/8082A	2 3 3’ 4 4’ 5 5’-Heptachlorobiphenyl (BZ 189)
GC/ECD	8082/8082A	2 3' 4 4' 5 5'-Hexachlorobiphenyl (BZ 167)
GC/ECD	8082/8082A	2 3' 4 4' 5-Pentachlorobiphenyl (BZ 118)
GC/ECD	8082/8082A	2 3' 4 4'5-Pentachlorobiphenyl (BZ 123)
GC/ECD	8082/8082A	2 3' 4 4'-Tetrachlorobiphenyl (BZ 66)
GC/ECD	8082/8082A	2 3’ 4 4’ 5-Pentachlorobiphenyl (BZ 114)
GC/ECD	8082/8082A	2 4 4'-Trichlorobiphenyl (BZ 28)
GC/ECD	8082/8082A	2 4'-Dichlorobiphenyl (BZ 8)
GC/ECD	8082/8082A	3 3' 4 4' 5 5'-Hexachlorobiphenyl (BZ 169)
GC/ECD	8082/8082A	3 3' 4 4' 5-Pentachlorobiphenyl (BZ 126)
GC/ECD	8082/8082A	3 3' 4 4'-Tetrachlorobiphenyl (BZ 77)

Solid and Chemical Waste		
Technology	**Method**	**Analyte**
GC/ECD	8082/8082A	3 4 4' 5-Tetrachlorobiphenyl (BZ 81)
GC/ECD	8082/8082A	Decachlorobiphenyl (BZ 209)
GC/ECD	8151A	2 4 5-T
GC/ECD	8151A	2 4-D
GC/ECD	8151A	2 4-DB
GC/ECD	8151A	Dalapon
GC/ECD	8151A	Dicamba
GC/ECD	8151A	Dichloroprop
GC/ECD	8151A	DInoseb
GC/ECD	8151A	MCPA
GC/ECD	8151A	MCPP
GC/ECD	8151A	Pentachlorophenol
GC/ECD	8151A	Silvex (2 4 5-TP)
GC/FID	8015B,C	Diesel range organics (DRO)
GC/FID	8015B,C	Gasoline range organics (GRO)
GC/FID	8011	EDB
GC/FID	8011	1 2-Dibromo-3-chloropropane
GC/MS	8260B,C	1 1 1 2-Tetrachloroethane
GC/MS	8260B,C / SOM01.2	1 1 1-Trichloroethane
GC/MS	8260B,C / SOM01.2	1 1 2 2-Tetrachloroethane
GC/MS	SOM01.2	1 1 2-Trichloro-1 2 2-trifluoroethane
GC/MS	8260B,C / SOM01.2	1 1 2-Trichloroethane
GC/MS	8260B,C / SOM01.2	1 1-Dichloroethane
GC/MS	8260B,C / SOM01.2	1 1-Dichloroethylene
GC/MS	8260B,C	1 1-Dichloropropene
GC/MS	8260B,C / SOM01.2	1 2 3-Trichlorobenzene
GC/MS	8260B,C	1 2 3-Trichloropropane
GC/MS	8260B,C / SOM01.2	1 2 4-Trichlorobenzene
GC/MS	8260B,C	1 2 4-Trimethylbenzene
GC/MS	8260B,C / SOM01.2	1 2-Dibromo-3-chloropropane
GC/MS	8260B,C / SOM01.2	1 2-Dichlorobenzene
GC/MS	8260B,C / SOM01.2	1 2-Dichloroethane
GC/MS	8260B,C / SOM01.2	1 2-Dichloropropane
GC/MS	8260B,C	1 3 5-Trimethylbenzene
GC/MS	8260B,C / SOM01.2	1 3-Dichlorobenzene
GC/MS	8260B,C	1 3-Dichloropropane
GC/MS	8260B,C / SOM01.2	1 4-Dichlorobenzene
GC/MS	8260B,C / SOM01.2	1 4-Dioxane
GC/MS	8260B,C	2 2-Dichloropropane
GC/MS	8260B,C / SOM01.2	2-Butanone
GC/MS	8260B,C	2-Chloroethyl vinyl ether
GC/MS	8260B,C	2-Chlorotoluene
GC/MS	8260B,C / SOM01.2	2-Hexanone
GC/MS	8260B,C	4-Chlorotoluene
GC/MS	8260B,C / SOM01.2	4-Methyl-2-pentanone

Solid and Chemical Waste		
Technology	**Method**	**Analyte**
GC/MS	8260B,C / SOM01.2	Acetone
GC/MS	8260B,C	Acetonitrile
GC/MS	8260B,C	Acrolein
GC/MS	8260B,C	Acrylonitrile
GC/MS	8260B,C	Allyl chloride
GC/MS	8260B,C / SOM01.2	Benzene
GC/MS	8260B,C	Bromobenzene
GC/MS	8260B,C / SOM01.2	Bromochloromethane
GC/MS	8260B,C / SOM01.2	Bromodichloromethane
GC/MS	8260B,C / SOM01.2	Bromoform
GC/MS	8260B,C / SOM01.2	Carbon disulfide
GC/MS	8260B,C / SOM01.2	Carbon tetrachloride
GC/MS	8260B,C / SOM01.2	Chlorobenzene
GC/MS	8260B,C / SOM01.2	Chloroethane
GC/MS	8260B,C / SOM01.2	Chloroform
GC/MS	8260B,C	Chloroprene
GC/MS	8260B,C / SOM01.2	cis-1 2-Dichloroethene
GC/MS	8260B,C / SOM01.2	cis-1 3-Dichloropropene
GC/MS	SOM01.2	Cyclohexane
GC/MS	8260B,C / SOM01.2	Dibromochloromethane
GC/MS	8260B,C	Dibromomethane
GC/MS	624 / 8260B,C / SOM01.2	Dichlorodifluoromethane
GC/MS	8260B,C	Diethyl ether
GC/MS	8260B,C	Di-isopropylether
GC/MS	8260B,C / SOM01.2	EDB
GC/MS	8260B,C	Ethyl methacrylate
GC/MS	8260B,C / SOM01.2	Ethylbenzene
GC/MS	8260B,C	Ethyl-t-butylether
GC/MS	8260B,C	Hexachlorobutadiene
GC/MS	8260B,C	Iodomethane
GC/MS	8260B,C	Isobutyl alcohol
GC/MS	8260B,C / SOM01.2	Isopropyl benzene
GC/MS	SOM01.2	m p-xylenes
GC/MS	8260B,C	Methacrylonitrile
GC/MS	SOM01.2	Methyl acetate
GC/MS	8260B,C / SOM01.2	Methyl bromide (Bromomethane)
GC/MS	8260B,C / SOM01.2	Methyl chloride (Chloromethane)
GC/MS	8260B,C	Methyl methacrylate
GC/MS	8260B,C / SOM01.2	Methyl tert-butyl ether
GC/MS	SOM01.2	Methylcyclohexane
GC/MS	8260B,C / SOM01.2	Methylene chloride
GC/MS	8260B,C	Naphthalene
GC/MS	8260B,C	n-Butylbenzene
GC/MS	8260B,C	n-proplybenzene
GC/MS	8260B,C	o-Xylene

Solid and Chemical Waste		
Technology	**Method**	**Analyte**
GC/MS	8260B,C	p-Isopropyltoluene
GC/MS	8260B,C	Propionitrile
GC/MS	8260B,C	sec-butylbenzene
GC/MS	8260B,C / SOM01.2	Styrene
GC/MS	8260B,C	t-Amylmethylether
GC/MS	8260B,C	tert-Butyl alcohol
GC/MS	8260B,C	tert-Butylbenzene
GC/MS	8260B,C / SOM01.2	Tetrachloroethylene (Perchloroethylene)
GC/MS	8260B,C	Tetrahydrofuran
GC/MS	8260B,C / SOM01.2	Toluene
GC/MS	8260B,C / SOM01.2	trans-1 2-Dichloroethylene
GC/MS	8260B,C / SOM01.2	trans-1 3-Dichloropropylene
GC/MS	8260B,C	Trans-1 4-Dichloro-2-butuene
GC/MS	8260B,C / SOM01.2	Trichloroethene (Trichloroethylene)
GC/MS	8260B,C / SOM01.2	Trichlorofluoromethane
GC/MS	8260B,C	Vinyl acetate
GC/MS	8260B,C / SOM01.2	Vinyl chloride
GC/MS	8260B,C	Xylene
GC/MS	8270C,D	1-Naphthylamine
GC/MS	8270C,D	2-Acetylaminofluorene
GC/MS	8270C,D / SOM01.2	2-Chloronaphthalene
GC/MS	8270C,D / SOM01.2	2-Chlorophenol
GC/MS	8270C,D / SOM01.2	2-Methylnaphthalene
GC/MS	8270C,D / SOM01.2	2-Methylphenol
GC/MS	8270C,D	2-Naphthylamine
GC/MS	8270C,D	2-Nitroaniline
GC/MS	8270C,D / SOM01.2	2-Nitrophenol
GC/MS	8270C,D	2-Picoline
GC/MS	8270C,D	3-Methylcholanthrene
GC/MS	8270C,D / SOM01.2	3-Nitroaniline
GC/MS	8270C,D	4-Aminobiphenyl
GC/MS	8270C,D / SOM01.2	4-Bromophenyl phenyl ether
GC/MS	8270C,D / SOM01.2	4-Chloro-3-methylphenol
GC/MS	8270C,D / SOM01.2	4-Chloroaniline
GC/MS	8270C,D / SOM01.2	4-Chlorophenyl phenylether
GC/MS	8270C,D	4-Dimethyl aminoazobenzene
GC/MS	8270C,D / SOM01.2	4-Methylphenol
GC/MS	8270C,D / SOM01.2	4-Nitroaniline
GC/MS	8270C,D / SOM01.2	4-Nitrophenol
GC/MS	8270C,D	5-Nitro-o-toluidine
GC/MS	8270C,D	a a-Dimethylphenethylamine
GC/MS	8270C,D / SOM01.2	Acenaphthene
GC/MS	8270C,D / SOM01.2	Acenaphthylene
GC/MS	8270C,D / SOM01.2	Acetophenone
GC/MS	8270C,D	Aniline

Solid and Chemical Waste		
Technology	**Method**	**Analyte**
GC/MS	8270C,D / SOM01.2	Anthracene
GC/MS	8270C,D	Aramite
GC/MS	8270C,D / SOM01.2	Atrazine
GC/MS	SOM01.2	Benzaldehyde
GC/MS	8270C,D	Benzidine
GC/MS	8270C,D / SOM01.2	Benzo(a)anthracene
GC/MS	8270C,D / SOM01.2	Benzo(a)pyrene
GC/MS	8270C,D / SOM01.2	Benzo(b)fluoranthene
GC/MS	8270C,D / SOM01.2	Benzo(g h i)perylene
GC/MS	8270C,D / SOM01.2	Benzo(k)fluoranthene
GC/MS	8270C,D	Benzoic Acid
GC/MS	8270C,D	Benzyl alcohol
GC/MS	8270C,D / SOM01.2	Biphenyl
GC/MS	8270C,D / SOM01.2	bis(2-Chloroethoxy)methane
GC/MS	8270C,D / SOM01.2	bis(2-Chloroethyl) ether
GC/MS	8270C,D / SOM01.2	bis(2-Ethylhexyl) phthalate (DEHP)
GC/MS	8270C,D / SOM01.2	Butyl benzyl phthalate
GC/MS	SOM01.2	Caprolactam
GC/MS	8270C,D / SOM01.2	Carbazole
GC/MS	8270C,D	Chlorobenzilate
GC/MS	8270C,D / SOM01.2	Chrysene
GC/MS	8270C,D	Diallate
GC/MS	8270C,D / SOM01.2	Dibenz(a h)anthracene
GC/MS	8270C,D / SOM01.2	Dibenzofuran
GC/MS	8270C,D / SOM01.2	Diethyl phthalate
GC/MS	8270C,D	Dimethoate
GC/MS	8270C,D / SOM01.2	Dimethyl phthalate
GC/MS	8270C,D / SOM01.2	Di-n-butyl phthalate
GC/MS	8270C,D / SOM01.2	Di-n-octyl phthalate
GC/MS	8270C,D	Ethyl methanesulfonate
GC/MS	8270C,D	Famfur
GC/MS	8270C,D / SOM01.2	Fluoranthene
GC/MS	8270C,D / SOM01.2	Fluorene
GC/MS	8270C,D / SOM01.2	Hexachlorobenzene
GC/MS	8270C,D / SOM01.2	Hexachlorobutadiene
GC/MS	8270C,D / SOM01.2	Hexachlorocyclopentadiene
GC/MS	8270C,D / SOM01.2	Hexachloroethane
GC/MS	8270C,D	Hexachloropropene
GC/MS	8270C,D	Isodrin
GC/MS	8270C,D / SOM01.2	Isophorone
GC/MS	8270C,D	Isosafrole
GC/MS	8270C,D	Methapyriline
GC/MS	8270C,D	Methyl methanesulfonate
GC/MS	8270C,D	Methyl parathion
GC/MS	8270C,D / SOM01.2	Naphthalene

Solid and Chemical Waste		
Technology	**Method**	**Analyte**
GC/MS	8270C,D / SOM01.2	Nitrobenzene
GC/MS	8270C,D	Nitroquinoline-1-oxide
GC/MS	8270C,D	n-Nitrosodiethylamine
GC/MS	8270C,D / SOM01.2	n-Nitrosodimethylamine
GC/MS	8270C,D	n-Nitroso-di-n-butylamine
GC/MS	8270C,D / SOM01.2	n-Nitrosodi-n-propylamine
GC/MS	8270C,D / SOM01.2	n-Nitrosodiphenylamine
GC/MS	8270C,D	n-Nitrosomethylethylamine
GC/MS	8270C,D	n-Nitrosomorpholine
GC/MS	8270C,D	n-Nitrosopiperidine
GC/MS	8270C,D	n-Nitrosopyrrolidine
GC/MS	8270C,D	o o o-Triethyl phosphorothioate
GC/MS	8270C,D	o-Toluidine
GC/MS	8270C,D	Pentachlorobenzene
GC/MS	8270C,D	Pentachloronitrobenzene
GC/MS	8270C,D/ SOM01.2	Pentachlorophenol
GC/MS	8270C,D	Phenacetin
GC/MS	8270C,D / SOM01.2	Phenanthrene
GC/MS	8270C,D / SOM01.2	Phenol
GC/MS	8270C,D	Phorate
GC/MS	8270C,D	Pronamide
GC/MS	8270C,D / SOM01.2	Pyrene
GC/MS	8270C,D	Pyrididne
GC/MS	8270C,D	Safrole
GC/MS	8270C,D	Thionazin
GC/MS	8270C,D / SOM01.2	Indeno(1 2 3-cd)pyrene
GC/MS	8270C,D / SOM01.2	1 2 4-Trichlorobenzene
GC/MS	8270C,D	1 3 5-Trinitrobenzene
GC/MS	8270C,D / SOM01.2	1 2 4 5-Tetrachlorobenzene
GC/MS	8270C,D / SOM01.2	2 4 5-Trochlorophenol
GC/MS	8270C,D / SOM01.2	2 4 6-Trichlorophenol
GC/MS	8270C,D / SOM01.2	2 3 4 6-Tetrachlorophenol
GC/MS	8270C,D / SOM01.2	1 2-Dichlorobenzene
GC/MS	8270C,D	1 2-Diphenylhydrazine
GC/MS	8270C,D / SOM01.2	1 3-Dichlorobenzene
GC/MS	8270C,D	1 3-Dinitrobenzene
GC/MS	8270C,D / SOM01.2	1 4-Dichlorobenzene
GC/MS	8270C,D	1 4-Dioxane
GC/MS	8270C,D	1 4-Naphthoquinone
GC/MS	8270C,D	1 4-Phenylenediamine
GC/MS	8270C,D / SOM01.2	bis(2-Chloroisopropyl) ether (2 2`-Oxybis(1-chloropropane))
GC/MS	8270C,D / SOM01.2	2 4-Dichlorophenol
GC/MS	8270C,D / SOM01.2	2 4-Dimethylphenol
GC/MS	8270C,D / SOM01.2	2 4-Dinitrophenol

Solid and Chemical Waste		
Technology	**Method**	**Analyte**
GC/MS	8270C,D / SOM01.2	2 4-Dinitrotoluene (2 4-DNT)
GC/MS	8270C,D	2 6-Dichlorophenol
GC/MS	8270C,D / SOM01.2	2 6-Dinitrotoluene (2 6-DNT)
GC/MS	8270C,D / SOM01.2	3 3`-Dichlorobenzidine
GC/MS	8270C,D	3 3'-Dimethylbenzidine
GC/MS	8270C,D / SOM01.2	2-Methyl-4 6-dinitrophenol
GC/MS	8270C,D	7,12-Dimethylphenethylamine
HPLC	8330/8330A/8330B (Analysis Only)	1 3 5-Trinitrobenzene
HPLC	8330/8330A/8330B (Analysis Only)	1 3-Dinitrobenzene
HPLC	8330/8330A/8330B (Analysis Only)	2 4 6-Trinitrotoluene
HPLC	8330/8330A/8330B (Analysis Only)	2 4-Dinitrotoluene
HPLC	8330/8330A/8330B (Analysis Only)	2 6-Dinitrotoluene
HPLC	8330/8330A/8330B (Analysis Only)	2-Amino-4 6 -dinitrotoluene
HPLC	8330/8330A/8330B (Analysis Only)	2-Nitrotoluene
HPLC	8330/8330A/8330B (Analysis Only)	3-Nitrotoluene
HPLC	8330/8330A/8330B (Analysis Only)	4-Amino-2,3-dinitrotoluene
HPLC	8330/8330A/8330B (Analysis Only)	4-Nitrotoluene
HPLC	8330/8330A/8330B (Analysis Only)	Hexahydr-1 3 5-trinitro-1 3 5-triazine (RDX)
HPLC	8330/8330A/8330B (Analysis Only)	Nitrobenzene
HPLC	8330/8330A/8330B (Analysis Only)	Nitroglycerin
HPLC	8330/8330A/8330B (Analysis Only)	Octahydro-1 3 5 7-tetrazocine (HMX)
HPLC	8330/8330A/8330B (Analysis Only)	Tetryl
CVAA	7471B/ ILM05.3	Mercury
CVAF	1631E	Low Level Mercury
ICP	6010B,C /ILM05.3	Aluminum
ICP	6010B,C /ILM05.3	Antimony
ICP	6010B,C /ILM05.3	Arsenic
ICP	6010B,C /ILM05.3	Barium
ICP	6010B,C /ILM05.3	Beryllium
ICP	6010B,C	Boron
ICP	6010B,C /ILM05.3	Cadmium

Solid and Chemical Waste		
Technology	**Method**	**Analyte**
ICP	6010B,C /ILM05.3	Calcium
ICP	6010B,C /ILM05.3	Chromium
ICP	6010B,C /ILM05.3	Cobalt
ICP	6010B,C /ILM05.3	Copper
ICP	6010B,C /ILM05.3	Iron
ICP	6010B,C /ILM05.3	Lead
ICP	6010B,C /ILM05.3	Magnesium
ICP	6010B,C /ILM05.3	Manganese
ICP	6010B,C	Molybdenum
ICP	6010B,C /ILM05.3	Nickel
ICP	6010B,C /ILM05.3	Potassium
ICP	6010B,C /ILM05.3	Selenium
ICP	200.7	Silicon
ICP	6010B,C /ILM05.3	Silver
ICP	6010B,C /ILM05.3	Sodium
ICP	6010B,C	Strontium
ICP	6010B,C /ILM05.3	Thallium
ICP	6010B,C	Tin
ICP	6010B,C	Titanium
ICP	6010B,C /ILM05.3	Vanadium
ICP	6010B,C /ILM05.3	Zinc
ICP/MS	6020/6020A / ILM05.3	Aluminum
ICP/MS	6020/6020A / ILM05.3	Antimony
ICP/MS	6020/6020A / ILM05.3	Arsenic
ICP/MS	6020/6020A / ILM05.3	Barium
ICP/MS	6020/6020A / ILM05.3	Beryllium
ICP/MS	6020/6020A	Boron
ICP/MS	6020/6020A / ILM05.3	Cadmium
ICP/MS	6020/6020A	Calcium
ICP/MS	6020/6020A / ILM05.3	Chromium
ICP/MS	6020/6020A / ILM05.3	Cobalt
ICP/MS	6020/6020A / ILM05.3	Copper
ICP/MS	6020/6020A	Iron
ICP/MS	6020/6020A / ILM05.3	Lead
ICP/MS	6020/6020A / ILM05.3	Magnesium
ICP/MS	6020/6020A	Manganese
ICP/MS	6020/6020A	Molybdenum
ICP/MS	6020/6020A / ILM05.3	Nickel
ICP/MS	6020/6020A	Potassium
ICP/MS	6020/6020A / ILM05.3	Selenium
ICP/MS	6020/6020A / ILM05.3	Silver
ICP/MS	6020/6020A	Sodium
ICP/MS	6020/6020A	Strontium
ICP/MS	6020/6020A / ILM05.3	Thallium
ICP/MS	6020/6020A	Tin

LABORATORY ACCREDITATION BUREAU

Certificate # L2223

Solid and Chemical Waste		
Technology	**Method**	**Analyte**
ICP/MS	6020/6020A	Titanium
ICP/MS	6020/6020A / ILM05.3	Vanadium
ICP/MS	6020/6020A / ILM05.3	Zinc
IC	9056/9056A	Chloride
IC	9056/9056A	Fluoride
IC	9056/9056A	Nitrate as N
IC	9056/9056A	Nitrite as N
IC	9056/9056A	Sulfate
Gravimetric	9070A / 9071B	Oil and Grease
Physical	1010A	Ignitability
Physical	9045D	pH
Titration	Chap 7.3.4	Reactive Sulfide
TOC	Lloyd Kahn	Total organic carbon
TOC	9060A / 5310B	Total organic carbon
Turbidimetric	9038 / ASTM 516-02	Sulfate
UV/VIS	350.1 / 4500NH3 H	Ammonia as N
UV/VIS	9251 / 4500Cl E	Chloride
UV/VIS	Chap. 7.3.4	Reactive Cyanide
UV/VIS	376.3	AVS-SEM
UV/VIS	3500Fe D	Ferrous Iron
Cleanup Methods	3630C	Silica Gel
UV/VIS	7196	Chromium VI
UV/VIS	7196A	Chromium VI
UV/VIS	9012B / ILM05.3	Total cyanide
Preparation	**Method**	**Type**
Preparation	1311	Toxicity Characteristic Leaching Procedure
Preparation	1312	Synthetic Precipitation Leaching Procedure
Cleanup Methods	3660B	Sulfur
Cleanup Methods	3620C	Florsil
Cleanup Methods	3630C	Silica Gel
Cleanup Methods	3640A	GPC
Organic Preparation	3540C	Soxhlet Extraction
Organic Preparation	3545A	Pressurized Fluid Extraction
Organic Preparation	3550C	Sonication
Inorganics Preparation	3050B	Hotblock
Inorganics Preparation	3060A	Alkaline Digestion
Volatile Organics Preparation	5035/5035A	Closed System Purge and Trap

Notes:

1) This laboratory offers commercial testing service.

Approved By: _____ Date: November 4, 2009

R. Douglas Leonard
Chief Technical Officer

Issued: 11/04/09

If the EDD files pass with no errors, the submittal page will immediately load. To complete the submittal stage, include the following information in the comment and additional information area of the form: laboratory name, laboratory contact person, project name, project number, site name/number, fractions included and any specific comments related to the EDD. Select the “Submit Files” button to continue the submittal process.

The submittal stage is not considered complete until a unique ticket key reference is returned in the browser window. The ticket key reference must be printed for record of submission and future reference. In addition, a copy of the ticket key reference must be included in the PDF data package.

STATEMENT OF WORK/PRICE TABLES

TECHNICAL SPECIFICATION FOR LABORATORY SERVICES
NAVAL SUBMARINE BASE (NSB) - NEW LONDON, GROTON, CT

COMPREHENSIVE LONG-TERM ENVIRONMENTAL ACTION - NAVY (CLEAN)
CONTRACT *N62470-08-D-1001,* CONTRACT TASK ORDER (CTO) NO. WE57

PRE-DESIGN SOIL REMEDIATION SAMPLING
CHROMIUM SPECIATION ANALYSES

1.0 INTRODUCTION

Tetra Tech NUS, Inc. (TtNUS) under CLEAN Contract ***N62470-08-D-1001*** is procuring laboratory analytical services for NSB-New London, Groton, CT in support of Pre-Design Activities. Requested analyses include total chromium, hexavalent chromium, trivalent chromium, and Oxidation-Reduction Potential (ORP.

The laboratory performing these analyses must be certified in the State of Connecticut. The laboratory must provide a copy of their DOD ELAP accreditation, all preparation and analytical methods requested must be specifically listed in the accreditation.

The responding laboratory must submit Method Detection Limits (MDLs), Limit of Detection (LOD), and Limits of Quantitation (LOQs) for all analyses and matrices requested by filling out the last two columns of Attachment A and including the completed attachment with the bid response. After award, the laboratory will be required to submit Standard Operating Procedures (SOPs) and relevant precision and accuracy limits for all preparation and analytical methods required under this scope of work. The laboratory will also be asked to complete Uniform Federal Policy (UFP) Sampling and Analysis Plan (SAP) Worksheets 19, and 23-28 for inclusion in the SAP. The SAP will be prepared according to the UFP for quality assurance project plans (March 2005) and utilize the 37 worksheets.

2.0 SAMPLE INFORMATION

Samples will be shipped to the laboratory during the period beginning Summer 2010. The schedule for sample shipment will be confirmed with the laboratory approximately 2 weeks prior to shipment.

The approximate number of samples to be submitted, the type of analyses to be conducted, and the analytical methods to be used are summarized in the attached Table 1.

Field duplicate samples will be submitted with “blinded” identification to the laboratory. The field crew will designate samples (one per twenty samples of like matrices) upon which matrix spike/matrix spike duplicate (organic analyses) and matrix spike/laboratory duplicate (inorganic analyses) are to be performed; additional volumes of these samples will be provided as necessary.

All samples are expected to be of low or moderate contaminant concentrations. The field crew will attempt to identify any potentially high concentration samples.

For the ICP metal analyses, perform a matrix spike and a laboratory duplicate analysis instead of matrix spike/matrix spike duplicate. If the matrix spike recovery falls outside the control limits and the sample result does not exceed four times the spike added, a post-digestion spike analysis must be performed. In addition, a five-fold dilution analysis (serial dilution) must be performed on the field-designated QC sample.

Results for these QC analyses must be summarized in the corresponding CLP-equivalent forms (Attachment B).

3.0 ANALYSIS/REPORTING INFORMATION

Data package deliverables must be submitted as a hardcopy and PDF (2 copies/2 CDs). The analytical requirements and approximate number of samples to be submitted for analysis are detailed in Table 1. Any analysis and reporting requirements addressed in the DOD Quality Systems Manual (April 2009) and the requested methods must be followed. Additionally, it is a requirement of TtNUS that the associated PDF data packages for metals analysis must meet Contract Laboratory Program (CLP) format, reporting, and PDF data package deliverable requirements. The PDF data packages for all the remaining analyses must be fully validatable and contain raw data, summary forms for all sample and laboratory method blank data, and summary forms containing all method specific quality control (results, recoveries, relative percent differences, relative standard deviations, and/or percent differences etc.).

Additionally, each PDF data package must contain a summary data package. This summary data package shall consist of only the summary forms (i.e., for CLP Forms 1 through 15 and non-CLP it shall be the CLP-like equivalent of Forms 1 through 15).

Attachment A details the required target compound list and required project action limits that must be met. The laboratory must submit its LOQs, LODs, and MDLs for all analyses and matrices requested by filling out the last two columns of Attachment A and including the completed attachment with the bid response.

Attachment B details the required summary forms for CLP-like data packages, the requirements for the organization, and bookmarking of PDF data packages.

Non-detected metals results must be reported down to the LOD submitted by the laboratory in Attachment A with the bid response. Positive results above the MDL but below the laboratory's LOQ must be reported as estimated values qualified with a "J" **for metals** analyses. Soil samples must be reported on a dry-weight basis.

The PDF data package deliverable must contain a detailed case narrative for all analytical fractions. This case narrative must also include the Contract Task Order (CTO) number, the site name, and the TtNUS Project Manager's name. Data from all analytical runs (i.e., original, dilution, re-analysis) must be reported. All soil and sediment matrix sample results shall be reported on an adjusted dry-weight basis. Additionally, the data package deliverable must include the ORIGINAL chain-of-custody form (received with the samples and signed by the laboratory sample custodian).

As stipulated in the CLEAN Master Services Agreement (MSA), Sample Delivery Group (SDG) and fractionally-specific text (TXT) files containing all environmental sample and field quality control blank analysis results must be generated in accordance with the requirements outlined in Attachment C of this specification.

As part of the laboratory case narrative, it is required that the Laboratory Quality Assurance Manager sign an attestation statement verifying that all electronic diskette deliverables exactly match the data summary forms (i.e. Form Is).

Maximum holding time allowances, as defined in the following table, are to be strictly observed. Calculation of holding time is in calendar days and is to begin from the time of collection. The holding times are as follows:

Analyses	Holding Time
Total chromium	6 months to analysis
Hexavalent chromium	30 days to analysis (after extraction 7 days to analysis)
Trivalent chromium	Calculation; Not applicable
ORP	24 hours to analysis

These holding times are based on 40 CFR 136, data validation criteria, and method specific requirements, and are measured from date of collection. The holding time criteria depicted apply to all analyses necessary to successfully determine the contaminant level contained in the sample. Hence, the holding time criteria apply to any/all subsequent sample dilutions and re-analyses.

The TtNUS Project Manager for this project is Mr. Corey Rich and he must be contacted in the event of any laboratory problems that could impact project deadlines (i.e., late deliverables, technical problems in the lab that could lead to late deliverables.) To ensure good communication it is required that the laboratory's appointed project manager contact Mr. Rich once a week for the entire project duration.

Contact information for Mr. Rich is as follows:

Tetra Tech NUS, Inc.
Foster Plaza 7
661 Andersen Drive
Pittsburgh, PA 15220
Phone: 412-921-8984
Fax: 412-921-4040
e-mail: corey.rich@tetratech.com

Analytical data turnaround times are to be measured from receipt of each sample shipment. All PDF analytical data package (2 CDs) and associated electronic (TXT) deliverables are due to the TtNUS Sample Management Coordinator, Ms. Tobrena Skeen, within the standard MSA turnaround term of 21 days from receipt of the last sample in a Sample Delivery Group (SDG). Additionally all SDGs must contain 20 samples. The frequency in which SDGs contain less than 20 samples should be minimal. All PDF data packages and electronic deliverables must be received at the same time or the deliverable will be considered incomplete and payment deductions may be imposed. **Additionally the laboratory Project Manager must fax copies of chain of custody forms to Ms. Skeen as samples are received by the laboratory.**

Contact information for Ms. Skeen follows:

Tetra Tech NUS, Inc.
Foster Plaza 7
661 Andersen Drive
Pittsburgh, PA 15220
Phone: 412-921-8582

Fax: 412-921-4040
e-mail: tobrena.skeen@tetratech.com

4.0 PERIOD OF PERFORMANCE/BOTTLEWARE INFORMATION

All samples will be shipped to the laboratory via express carrier within 48-hours of collection. Please circle the Yes or No at the bottom of Table 1 which will indicate if the laboratory will provide courier service at no extra charge. Samples will be shipped to the laboratory the period **beginning Summer 2010**.

Shipment of bottleware to support the entire project will be coordinated at a later date.

The laboratory is to provide all necessary sample containers **(plus approximately 10% extra for breakage)**. All sample containers must meet ICHEM series 300 cleanliness criteria (or equivalent), and documentation of certified cleanliness must be provided. All of the appropriate sample bottleware must be pre-preserved. The bottleware must be shipped to the designated location in Coleman-like coolers. **The laboratory must also provide any extra coolers needed for return shipment of sample to the laboratory for analysis.** The laboratory is also requested to provide a packing slip indicating the analytical parameters for which each container type is designated, sample labels, and chain-of-custody forms. The laboratory is to dispatch proper bottleware for the collection of additional soil, per sampling location, for dry-weight determination.

The laboratory must provide Material Safety Data Sheets (MSDSs) for all preservatives sent with each bottleware shipment to the field. MSDSs must be representative of the chemicals provided as preservatives with regard to mixtures and/or purity of the chemicals. For example if a 35% sulfuric acid solution is the preservative, the MSDS provided should be for 35% sulfuric acid solution not 96% sulfuric acid.

5.0 ADDITIONAL COMMENTS/CONTACTS

Within a laboratory, internal transfers of samples, extracts, and digestates must be accomplished and documented as controlled custody transfers. The laboratory must maintain documentation that supports an unbroken chain of custody for samples, digestates and extracts from time of receipt or production in the laboratory until disposal.

The laboratory is to provide a minimum of 60 days storage of sample extracts and 60 days storage of intact sample aliquots, as stipulated in the MSA. **Additionally, the laboratory must store hardcopy and/or PDF data packages for 5 years.**

All analyses conducted under this subcontract assignment are to be performed at the solicited facility only. The laboratory is not permitted to lower-tier subcontract these analyses, or analyze these samples at a corporate facility other than the facility stipulated without prior notification and consent from the CLEAN Subcontracting Officer.

The unit cost for analysis is to include compensation for containers, preservatives, coolers, shipping costs, storage, disposal, and laboratory quality control analyses (such as matrix spike, matrix spike duplicate, laboratory duplicate, and laboratory control sample analyses.) These items are not to be billed as separate line items.

Technical, quality assurance, and data format concerns are to be directed to Ms. Kelly Carper at 412-921-7273 or via e-mail kelly.carper@tetratech.com. Ms. Carper must be contacted and informed of any difficulties encountered during the conduct of the requested analyses.

Questions regarding electronic diskette deliverable concerns are to be directed to Ms. Amy Thomson at 412-921-8182 or via e-mail amy.thomson@tetratech.com. Ms. Thomson must be contacted and informed of any difficulties encountered preparing the required electronic deliverables.

Contract concerns, and response to this solicitation, are to be directed to:

Ms. Meg Price
CLEAN Subcontracting Officer
Tetra Tech NUS, Inc.
234 Mall Boulevard, Suite 260
King of Prussia, PA 19406
Phone: 610-491-9688
Fax: 610-491-9645
e-mail: meg.price@tetratech.com

Triplicate copies of invoices associated with the analyses contracted herein are to be submitted to the attention of the Accounting Supervisor:

Tetra Tech NUS, Inc.
661 Andersen Drive, Foster Plaza 7
Pittsburgh, PA 15220
Phone: 412-921-8506
Fax: 412-921-4040

Please confirm the laboratory's ability to perform the methodologies requested at the analyte detection limits indicated. Also confirm available laboratory capacity, and complete/confirm the costing information indicated in Table 1. **All costing information must reflect the terms and conditions established by the 2010 CLEAN MSA.**

TABLE 1
NUMBER OF SAMPLES/ANALYTICAL METHODS
NAVAL SUBMARINE BASE (NSB), NEW LONDON, CT
CTO WE57, CHROMIUM SPECIATION ANALYSES FOR SOIL

Matrix	Parameter	Method[1]	# Samples	Unit Price	Total Cost
Solid	Total chromium	SW-846 3050 prep./SW-846 6010C	11	$	$
	Hexavalent chromium	SW-846 3060A prep./SW-846 6800	11	$	$
	Trivalent chromium	Calculation	11	$	$
	ORP	SW-846 9045 prep./ASTM D1498-93	11	$	$

1. Please specify which method the lab will be performing.

TOTAL COST $

Can the laboratory provide sample pick-up on site? YES or NO (circle one)
If yes is there an additional charge and what is that charge?_____
Name of Laboratory_____

Signature_____

ATTACHMENT A
Required Target Compound Lists and Quantitation Limits

Matrix: Soil
Analytical Group: Select Metals

Analyte	CAS Number	Project Action Limit (mg/kg)	Project Action Limit Reference	Project Quantitation Limit Goal (mg/kg)	Laboratory		
					LOQ (mg/kg)	LOD (mg/kg)	MDL (mg/kg)
Total chromium	7440-47-3	NA	NA	NA			
Hexavalent chromium	18540-29-9	0.29	EPA RES RSL	0.097			
Trivalent chromium	16065-83-1	3900	CTDEP RES DEC	1300			

EPA RES RSL – US Environmental Protection Agency Residential Risk Screening Levels
CTDEP RES DEC - Connecticut Department of Environmental Protection Direct Exposure Criterion for a residential scenario
NA – Not available

ATTACHMENT B
Summary Form Requirements for PDF Deliverable and PDF Data Package Deliverables

PDF DATA PACKAGE DELIVERABLE REQUIREMENTS

The laboratory is to provide 2 compact disks (CDs) containing a PDF file in the following format:

1. Table of Contents
2. Case Narrative
3. Chain-of-Custody
4. Data Summary Package (contains summary of all CLP or CLP like Forms 1 through 15 per analytical fraction)
5. Analytical Fractions (VOA, SVOC, General Chemistry, etc.)
a. QC Summary (summary of all CLP or CLP like Forms 1 through 15 for a particular analytical fraction)
b. Raw Sample Data (includes all sample dilutions, sample re-analyses, QC samples, etc.)
c. Calibration Data (includes all initial and continuing calibrations)
d. Miscellaneous (includes extraction forms, IDLs, MDLs, etc.)

Each of the above sections should be bookmarked in the PDF for easy access.

Summary Form Requirements for PDF data package deliverable for non-CLP Methods:

The following summary forms are required as part of the data package deliverable for SW-846 6010 series for metals:

Results Report - must present all information presented on CLP FORM I.
Initial and Continuing Calibration Summary -must present all information presented on CLP FORM 2A.
CRDL Summary - must present all information presented on CLP FORM 2B.
Blanks - must present all information presented on CLP FORM 3.
ICP Interference Check Sample Summary - must present all information presented on CLP FORM 4.
Matrix Spike Summary - must present all information presented on CLP FORM 5A.
Post Digestion Spike - must present all information presented on CLP FORM 5B.
Lab Duplicate Results - must present all information presented on CLP FORM 6.
LCS Summary - must present all information presented on CLP FORM 7.
MSA Summary (Method of Standard Addition) - must present all information presented on CLP FORM 8.
ICP Serial Dilution Summary - must present all information presented on CLP FORM 9.
Detection Limits - must present all information presented on CLP FORM 10.
Linear range – must present all information presented on CLP FORM 12
Prep Log - must present all information presented on CLP FORM 13.
Analysis Run Log - must present all information presented on CLP FORM 14.
ICP-MS Tune – must present all information on CLP FORM 14 (ILM05.4)
ICP/MS Internal Standard Relative Intensity Summary - must present all information presented on CLP FORM 15 (ILM05.4).

Also must include: Instrument Calibration Records, Chain-of-Custody Forms, and Case Narrative.

Summary Forms for SW-846, 8260B and 8270C (i.e., Any SW-846 GC/MS analysis of Volatile and Semivolatile Organic Compounds) should be presented in a CLP-Like format. The following Summary Forms must be included:

Result Summary	One Sample per summary page. Presentation of analytical results for both method blanks and environmental samples, date of collection, preparation, and analysis. Environmental samples should be identified with the field identification numbers on the COCs.

Surrogate Recovery Form	Present all information contained on CLP Form II.
Summary of Matrix Spike/Matrix Spike Duplicate Recovery	Present all information contained on CLP Form III.
Instrument Performance Check Summary Form - Mass Spec Tuning Form	Present all information Contained on CLP Form V.
Initial Calibration Summary	Present all information contained CLP Form VI.
Continuing Calibration Summary	Present All Information contained on CLP Form VII.
Internal Standard Area and Retention Time Summary	Present all information contained CLP Form VIII.

Summary Forms for EPH should be presented in a CLP-Like format. The following Summary Forms must be included:

Result Summary	One Sample per summary page. Presentation of analytical results for both method blanks and environmental samples, date of collection, preparation, and analysis. Environmental samples should be identified with the field identification numbers on the COCs.
Surrogate Recovery Form	Present all information contained on CLP Form II
Summary of Matrix Spike/Matrix Spike Duplicate Recovery	Present all information contained on CLP Form III.
Summary of Initial Calibration	Present all information contained on CLP Form VI-PEST-2.
Summary of Calibration Verification	Present all information contained on CLP Form VII-PEST-1 and Form VII-PEST-2.
Analytical Sequence	Present all information contained on CLP Form VIII-PEST.

ATTACHMENT C
ELECTRONIC DATA DELIVERABLE REQUIREMENTS

ELECTRONIC DATA FORMAT REQUIREMENTS

1.0 INTRODUCTION

The laboratory is to submit text-based tab delimited EDD files for each SDG using Tetra Tech's laboratory data checker explained below. The files must be in the format specified in this Attachment. Additional information such as laboratory name, project name, fractions included, project number, site name/number, laboratory contact person and any specific comments related to the EDD should be included in the comments section of the EDD Submittal page.

The RESULT for nondetects should be populated with the project-specific sample quantitation reporting limits (i.e., either the sample quantitation limit or method detection limit, as specified in Section 3.0 of this scope of work. Any corrections made to the hardcopy data must also be made to the electronic file. Appropriate qualifiers as identified by the analytical protocol must also be designated; laboratory QC non-compliance codes are not to be depicted.

Tetra Tech's electronic EDD format follows the ADAPT structure and requires the A1 and A3 files. The A2 file is only required if the project is using ADAPT; and, for non-ADAPT EDD submittals the A2 file may be omitted. The EDD consists of separate, tab-delimited ASCII text files. Each file corresponds to a database table. The tables are identified as the Analytical Results Table (A1) and Sample Analysis Table (A3). A separate set of text files must be created and submitted for each sample delivery group (SDG). The files must be identified to correspond to the (A1) table and the (A3) table. The file naming convention is: the Sample Delivery Group (SDG) followed by the table identifier (A1 or A3), followed by the ".txt" extension. The file names must not contain spaces or special characters. For example, the EDD file names for a laboratory-reporting batch identified as SDG001 would be as follows:

SDG001A1.txt
SDG001A3.txt

On certain projects Tetra Tech will utilize the ADAPT Electronic Data Validation software, which will require the laboratory to use the ADAPT electronic data deliverable checker software prior to submitting the files through Tetra Tech's laboratory data checker (this will be clearly specified in the Tetra Tech laboratory statement of work). The ADAPT checker software can be downloaded from Laboratory Data Consultants' web site: http://www.lab-data.com. For projects which Tetra Tech is using the ADAPT software, Tetra Tech will provide the laboratory with the project library. The laboratory is not permitted to modify the project library. ADAPT projects will require the laboratory to export all three checked files (A1, A2, and A3) from the ADAPT software and submit them through Tetra Tech's laboratory data checker. **ADAPT error logs generated must be included with the electronic PDF data validation package and cannot be submitted through the laboratory data checker.**

The values reported in the EDD text files must agree exactly with the final values reported on the PDF data package sample result summaries. The details of file naming conventions, data structure and data checker use are discussed below.

Analytical Results Table (A1 File)

The Analytical Results table contains analytical results and related information for target analytes in field samples and associated laboratory quality control samples (excluding calibrations and tunes). Field samples and laboratory method blanks must report a result record for each analyte reported within a method. Laboratory control samples (LCS and LCSD) and matrix spike samples (MS and MSD) must report a result record for every analyte specified as a spiked analyte in the laboratory statement of work. Table A1 in this document lists the field names and data type descriptions for the Analytical Results Table (A1).

Lab Instrument Table (A2 File)

A2 file is only required if the project is using ADAPT. In all other EDD submittals, the A2 file may be omitted. Laboratories should refer to the ADAPT User Guide for populating the A2 Table.

Sample Analysis Table (A3 File)

The Sample Analysis table contains information specific to field environmental samples and laboratory quality control analyses (excluding calibrations and tunes). A sample record must exist for each sample/method/matrix/analysis type combination. Table A3 in this document lists the field names and data type descriptions for the Sample Analysis Table (A3).

All electronic data deliverables are due within the same time established for the associated hardcopy data packages.

In addition, the laboratory QC officer must read and sign a copy of the Quality Assurance Review Form displayed on the next page of this Attachment. Electronic deliverables are not considered to be complete without the accompanying Quality Assurance Review Form.

I _____, as the designated Quality Assurance Officer, hereby attest that all electronic deliverables have been thoroughly reviewed and are in agreement with the associated hardcopy data. The enclosed electronic files have been reviewed for accuracy (including significant figures), completeness and format. The laboratory will be responsible for any labor time necessary to correct enclosed electronic deliverables that have been found to be in error. I can be reached at
(____)_____if there are any questions or problems with the enclosed electronic deliverables.

Signature:_____ Title: _____Date: _____

2.0 EDD Field Properties

Tables A1 and A3 in this document specify the EDD field properties. Laboratories should refer to the ADAPT User Guide for populating the A2 Table. These include the field name, sequence order, field description, data type/length and reporting requirement for each field. Fields in the EDD **must** be sequenced according to the order that they appear below in Tables A1 and A3. For example, in the Analytical Results table (A1), the field "ClientSampleID" will always be the first piece of information to start every new line of data (or database record), followed by the field "LabAnalysisRefMethodID", "AnalysisType", etc.

When creating an EDD as a text file, use the ASCII character set in a file of lines terminated by a carriage return and line feed. No extra characters are allowed at the end of a line, after the carriage return and line feed. Enclose each data value with double quotes (text qualifier) and separate each field value with a **tab** character (tab delimiter). Data fields with no information (null) may be represented by two consecutive tabs. For example, in the Sample Analysis table, since the "Collected", "ShippingBatchID", and "Temperature" fields do not apply to laboratory generated QA/QC samples, the record for a Laboratory Control Sample by Method 8270C would be entered as follows. Note that the first two fields ("ProjectNumber" and "ProjectName") are omitted in this example.

…"LCSW100598" "AQ" "LCSW100598" "LCS" "8270C",…etc.

If a field is populated with less than the maximum allowed number of characters, do not pad the values with leading or trailing spaces. In the above example, although the "MatrixID" field can accommodate up to 10 characters, only 2 characters were entered in this field. **Do not include the delimiter (tab character) within any of the field values.** Example EDD files may be downloaded from the LEDD Checker application.

An example database shall be sent for review prior to the first electronic deliverable in the required .txt format. The example file will be examined for completeness and comments will be sent to the laboratory. Any questions regarding the electronic deliverable should be directed to LabSupport@tetratech.com

Table A1
Field Descriptions for the Analytical Results Table (Table A1)
Contains laboratory test results and related information for field and QC samples (excluding instrument calibrations) on an analyte level for environmental chemistry including radiochemistry

Field Name	Field Name Description	Field Type	Field Length	Required Value
ClientSampleID	Client or contractor's identifier for a field sample If a sample is analyzed as a laboratory duplicate, matrix spike, or matrix spike duplicate, append suffixes DUP, MS and MSD respectively to the Client Sample ID with no intervening spaces or hyphens (i.e. MW01DUP, MW01MS, and MW01MSD). For Method Blanks, LCS, and LCSD enter the unique LaboratorySampleID into this field. Do not append suffixes to the ClientSampleID for dilutions, reanalyses, or re-extracts (the AnalysisType field is used for this distinction). For example, MW01DL and MW01RE are not allowed.	Text	25	X

Table A1
Field Descriptions for the Analytical Results Table (Table A1)
Contains laboratory test results and related information for field and QC samples (excluding instrument calibrations) on an analyte level for environmental chemistry including radiochemistry

Field Name	Field Name Description	Field Type	Field Length	Required Value
	Parent sample records must exist for each MS and MSD. If an MS/MSD is shared between two EDDs, records for the MS/MSD and its parent sample must exist in the Analytical Results table for both EDDs.			
LabAnalysisRefMethodID	Laboratory reference method ID. The method ID may be an EPA Method number or a Lab Identifier for a method such as a SOP Number, however; method ID is specified by the project. The method ID must be entered into the standard list.	Text	25	X
AnalysisType	Defines the analysis type (i.e., Dilution, Reanalysis, etc.). This field provides distinction for sample result records when multiple analyses are submitted for the same sample, method, and matrix; for example dilutions, re-analyses, and re-extracts.	Text	10	X
LabSampleID	Laboratory tracking number for field samples and lab generated QC samples such as method blank, LCS, and LCSD. There are no restrictions for the LabSampleID except for field length and that the LabSampleID must be distinct for a given field sample or lab QC sample and method. Suffixes may be applied to the LabSampleID to designate dilutions, reanalysis, etc.	Text	25	X
LabID	Identification of the laboratory performing the analyses.	Text	7	X
ClientAnalyteID	CAS Number or unique client identifier for an analyte or isotope. If a CAS Number is not available, use a unique identifier provided by the client or contractor. The ClientAnalyteID for a particular target analyte or isotope should be specified by the project and must exist in the standard value tables for Analytes.	Text	12	X

Table A1
Field Descriptions for the Analytical Results Table (Table A1)
Contains laboratory test results and related information for field and QC samples (excluding instrument calibrations) on an analyte level for environmental chemistry including radiochemistry

Field Name	Field Name Description	Field Type	Field Length	Required Value
	For the LCS, LCSD, MS, and MSD, it is only necessary to report the compounds designated as spikes in the library (and surrogates for organic methods.) For TICs from GC/MS analyses, enter the retention time in decimal minutes as the Client Analyte ID.			
AnalyteName	Chemical name for the analyte or isotope. The project specifies how an analyte or isotope is named. The analyte name must be associated to a ClientAnalyteID in the standard values table for Analytes (excluding compounds designated as TIC's).	Text	60	X
Result	Result value for the analyte or isotope. Entries must be numeric. For non-detects of target analytes or isotopes and spikes, do not enter "ND" or "0". Do not leave this field blank. If an analyte or spike was not detected, enter the associated value specified in Section 3.0 of this scope of work (e.g., LOD, SQL, PQL, etc.), corrected for dilution and percent moisture as applicable. Do not enter "0". A "0" result may be acceptable for surrogate or internal standard percent recoveries; however, it should not be reported for any target compound.	Numeric (1)	20(6)	X

Table A1
Field Descriptions for the Analytical Results Table (Table A1)
Contains laboratory test results and related information for field and QC samples (excluding instrument calibrations) on an analyte level for environmental chemistry including radiochemistry

Field Name	Field Name Description	Field Type	Field Length	Required Value
ResultUnits	The units defining how the values in the Result, DetectionLimit, and ReportingLimit fields are expressed. For radiochemistry this also includes how the value in the Error field is expressed.	Text	10	X
LabQualifiers	A string of single letter result qualifiers assigned by the lab based on client-defined rules and values. The "U" Lab Qualifier must be entered for all non-detects. Other pertinent lab qualifiers may be entered with the "U" qualifier. Order is insignificant. Lab qualifiers other than those listed in the standard values table may be used. If so, these must be added to the standard value table in the application.	Text	7	Q
DetectionLimit	For radiochemistry methods, the minimum detectable activity for the isotope being measured. For all other methods: The minimum detection limit value for the analyte being measured. For surrogates, internal standards, etc. where detection limits are not applicable use the value -99.	Numeric (1)	10(6)	X
DetectionLimitType	Specifies the type of detection limit (i.e., MDA, MDL, IDL, etc.). If -99 is specified in the DetectionLimit field us the value NA.	Text	10	X
RetentionTime or Error	For radiochemistry methods only, enter the 2 Sigma Counting Errors. The units for error are entered in the ResultUnits field. For GC/MS methods only, enter the time expressed in decimal minutes between injection and detection for GC/MS TICs only For target analytes in all other methods, leave this field blank. Note: GC retention times are not evaluated at this time.	Text	5	T
AnalyteType	Defines the type of result, such as tracer, surrogate, spike, or target compound.	Text	7	X
PercentRecovery	For radiochemistry methods: The tracer yield, if applicable. For all other analytical methods: The percent	Numeric (1)	5(3)	X

Table A1
Field Descriptions for the Analytical Results Table (Table A1)
Contains laboratory test results and related information for field and QC samples (excluding instrument calibrations) on an analyte level for environmental chemistry including radiochemistry

Field Name	Field Name Description	Field Type	Field Length	Required Value
	recovery value of a spiked compound or surrogate. If the spike or surrogate was not recovered because of dilution, enter “DIL”. If a spike or surrogate was not recovered because of matrix interference, enter “INT”. If a spike or surrogate was not recovered because it was not added to the sample, enter “NS”.			
RelativePercentDifference	The relative percent difference (RPD) of two QC results, such as MS/MSD, LCS/LCSD, and Laboratory Duplicates. Report RPD in Laboratory Duplicate, LCSD, and MSD records only.	Numeric (1)	5(3)	X
ReportingLimit	Reporting limit value for the measured analyte or isotope Factor in the dilution factor and percent moisture correction, if applicable. The Reporting Limit for each analyte and matrix in a given method is specified in the project library or QAPP. For surrogates, internal standards, etc. where reporting limits are not applicable use the value -99.	Numeric (1)	10(6)	X
ReportingLimitType	Specifies the type of reporting limit (i.e., CRQL, PQL, SQL, RDL, etc). The Reporting Limit Type for each method and matrix is specified in the project library or QAPP. If -99 is specified in the ReportingLimit field us the value NA.	Text	10	X
ReportableResult	This field indicates whether or not the laboratory chooses an individual analyte or isotope result as reportable. Enter “YES” if the result is reportable. Enter “NO” if the result is not reportable. If only one analysis is submitted for a particular sample and method, enter “YES” for all target compounds (where Analyte Type = TRG). For GC/MS methods enter yes for tentatively identified compounds (where Analyte Type = TIC). If two or more analyses are submitted for a particular sample and method (i.e. initial analysis, reanalysis and/or dilutions), enter “YES” from only <u>one</u> of the analyses for each target compound. For example: a sample was run a second time at dilution because benzene exceeded the calibration range in the initial,	Text	3	X

Table A1
Field Descriptions for the Analytical Results Table (Table A1)
Contains laboratory test results and related information for field and QC samples (excluding instrument calibrations) on an analyte level for environmental chemistry including radiochemistry

Field Name	Field Name Description	Field Type	Field Length	Required Value
	undiluted analysis. All target analytes are reported in each analysis. For the initial analysis, (Analysis Type = RES), enter "NO" for benzene and enter "YES" for all other compounds. For the diluted analysis (Analysis Type = DL), enter "YES" for benzene and enter "NO" for all other compounds. For TICs (Analyte Type = TIC), if more than one analysis is submitted for a particular sample and method, choose only one of the analyses where Reportable Result = YES for all TICs. For example, a sample was run a second time because one or more target compounds exceeded the calibration range in the undiluted analysis. Choose a particular analysis and enter "YES" for all TICS. In the other analysis enter "NO" for all TICs. Note that it is not necessary to report the full target analyte list for the initial result, dilution, re-analysis, or re-extraction. However, each target analyte must be reported YES once and once only in the case of multiple analyses for a given sample, method, and matrix. In the case of organics, all surrogates must be reported for all analyses submitted for a given sample, method, and, matrix.			
SpkConcnAdded	The spike added. This value must be reported in the same units as the result. Where (SA) in the following equation: % Recovery = (SSA-SC)/SA x 100% where : SSA is the spiked sample concentration (amount) after spiking. SC is the sample concentration (amount) before spiking. SA is the the expected increase in sample concentration (amount) as a result of spiking. This value must incorporate all correction factors such as dilution factor and moisture content that are applied to the spiked sample when computing the spiked sample concentration or amount. Enter -99 where no spike was added.	Numeric (1)	10(6)	X
SpkParentSampleID	The sampleID of a sample (often called the original sample) that receives a spike aliquot to form a spiked sample such as a matrix spike. This is not the same as the ID of the spiked sample (such as a matrix spike) after spiking. The result for SpkParentSampleID and the result	Text	25	X

Table A1
Field Descriptions for the Analytical Results Table (Table A1)
Contains laboratory test results and related information for field and QC samples (excluding instrument calibrations) on an analyte level for environmental chemistry including radiochemistry

Field Name	Field Name Description	Field Type	Field Length	Required Value
	(i.e., SpkConcnAdded) for the spiked sample are used to compute percent recovery of the analyte.			
SamplePrepInitial	The initial sample preparation volume in liters (L) for aqueous samples or grams (g) for solid samples.	Numeric (1)	20(6)	
SamplePrepFinal	The final sample preparation volume in liters (L) for aqueous samples or grams (g) for solid samples.	Numeric (1)	20(6)	
LimitOfDetection	The smallest amount or concentration of a substance that must be present in a sample in order to be detected at a 99% confidence level. In other words, if a sample has a true concentration at the LOD, there is a minimum probability of 99% of reporting a "detection" (a measured value ≥ DL) and a 1% chance of reporting a non-detect (a false negative).	Numeric (1)	10(6)	N
Comment	Add any comments or additional information specific to the analyte test result data record.	Text	200	

X Required field.

Q Only required if laboratory has qualified the result.

T Only required for tentatively identified compounds by GC/MS.

(1) Field Length indicates decimal precision in parenthesis. For example, 5(2) = a total width of 5 numbers including a maximum of 2 decimal places.

Table A3
Field Description for the Sample Analysis (Table A3)
This table contains information related to analyses of field samples and laboratory QC samples (excluding calibrations and tunes) on a sample level for environmental chemical analyses including radiochemistry

Field Name	Field Name Description	Field Type	Field Length	Required Value
ProjectNumber	Project number assigned by the client.	Text	30	X
ProjectName	Project name assigned by the client.	Text	90	X
ClientSampleID	Client or contractor's identifier for a field sample If a sample is analyzed as a laboratory duplicate, matrix spike, or matrix spike duplicate, append suffixes DUP, MS and MSD respectively to the Client Sample ID with no intervening spaces or hyphens (i.e. MW01DUP, MW01MS, and MW01MSD). For Laboratory QC samples (i.e. Method Blanks, LCS, and LCSD, etc.) enter the unique LaboratorySampleID into this field Do not append suffixes to the ClientSampleID for dilutions, reanalyses, or re-extracts (the Analysis_Type field is used for this distinction). For example, MW01DL and MW01RE are not allowed Parent sample records must exist for each MS and MSD. If an MS/MSD is shared between two EDDs, records for the MS/MSD and its parent sample must exist in the Sample Analysis table for both EDDs.	Text	25	X
Collected	Date and Time of sample collection. Refer to the date/time format at the end of this table. Leave this field blank for Method Blank, LCS, and LCSD. For Collected values that are not applicable use the value of 00/00/0000 00:00.	Date/ Time	16*	X
MatrixID	Sample matrix (i.e., AQ, SO, etc.)	Text	10	X
LabSampleID	Laboratory tracking number for field samples and lab generated QC samples such as method blank, LCS, and LCSD. There are no restrictions for the LabSampleID except field length and that the LabSampleID must be unique for a given field sample or lab QC sample and method.	Text	25	X
QCType	This record identifies the type of quality control sample QC (i.e., Duplicate, LCS, Method Blank, MS, or MSD). For regular environmental samples, populate this field with "NM".	Text	10	X
ShippingBatchID	Unique identifier assigned to a cooler or shipping container used to transport client or field samples.	Text	25	X

Table A3
Field Description for the Sample Analysis (Table A3)
This table contains information related to analyses of field samples and laboratory QC samples (excluding calibrations and tunes) on a sample level for environmental chemical analyses including radiochemistry

Field Name	Field Name Description	Field Type	Field Length	Required Value
	Links all samples to a cooler or shipping container. No value is required for method blanks, LCS, and LCSD.			
Temperature	Temperature (in centigrade degrees) of the sample as received. The storage refrigerator or room temperature should be reported (in centigrade degrees) for laboratory QC samples (i.e. method blanks, laboratory control standards). Use -99 if temperature is not available. This field is not required for radiochemistry methods.	Numeric (1)	10(6)	X
LabAnalysisRefMethodID	Laboratory reference method ID. The method ID may be an EPA Method number or laboratory identifier for a method such as a SOP number, however; values used for Laboratory Method IDs are specified by the project and must in the in standard value list for method IDs.	Text	25	X
PreparationType	Preparation Method Number (i.e., 3010A, 3510C, 3550C, 5030B, etc.) For analytical procedures that do not have a specific preparation method number, use “Gen Prep”.	Text	25	X
AnalysisType	Defines the type of analysis such as initial analysis, dilution, re-analysis, etc. This field provides distinction for sample records when multiple analyses are submitted for the same sample, method, and matrix, for example: dilutions, re-analyses, and re-extracts.	Text	10	X
Prepared	Refer to the date/time format at the end of this table. If no sample preparation is involved enter the analysis date and time in this field. Refer to the date/time format at the end of this table.	Date/ Time	16*	X
Analyzed	Date and time of sample analysis. Refer to the date and time format at the end of this table. For Analyzed values that are not applicable use the value of 00/00/0000 00:00.	Date/ Time	16*	X
LabID	Identification of the laboratory performing the analysis.	Text	7	X
QCLevel	The level of laboratory QC associated with the analysis	Text	6	X

Table A3
Field Description for the Sample Analysis (Table A3)
This table contains information related to analyses of field samples and laboratory QC samples (excluding calibrations and tunes) on a sample level for environmental chemical analyses including radiochemistry

Field Name	Field Name Description	Field Type	Field Length	Required Value
	reported in the EDD. If only the Analytical Results Table (A1) and the Sample Analysis Table (A3) information are submitted for the sample, enter "COA". If the Laboratory Instrument Table (A2) information is also submitted for the sample, enter "COCAL"			
ResultBasis	Indicates whether results associated with this sample records are reported as wet or percent moisture corrected. Enter "WET" if results are not corrected for percent moisture. Enter "DRY" if percent moisture correction is applied to results. For aqueous samples, enter "WET". For other matrices where basis is not applicable enter "NA"	Text	3	X
TotalOrDissolved	This field indicates if the results related to this sample record are reported as a total or dissolved fraction. If not applicable please report "NA"	Text	3	X
Dilution	Dilution of the sample aliquot. Enter "1" for method blanks, LCS, and LCSD, or if the field samples was analyzed without dilution.	Numeric (1)	10(6)	X
HandlingType	Indicates the type of leaching procedure, if applicable (i.e., SPLP, TCLP, WET). Enter "NA" if the sample analysis was <u>not</u> performed on a leachate.	Text	10	X
HandlingBatch	Unique laboratory identifier for a batch of samples prepared together in a leaching procedure (i.e., SPLP, TCLP, or WET preparation). The HandlingBatch links samples with leaching blanks. Enter "NA" if the sample analysis was <u>not</u> performed on a leachate.	Text	12	X
LeachateDate	Date and time of leaching procedure (i.e., date for SPLP, TCLP, or WET preparation). Refer to the date and time format at the end of this table. . For Analyzed values that are not applicable use the value of 00/00/0000 00:00	Date /Time	16*	X
Percent_Moisture	For soil and sediment samples, enter the percent of sample composed of water. For aqueous samples enter "100". For other matrices where Percent_Moisture is not applicable use a value of -99	Numeric (1)	10(6)	X
MethodBatch	Unique laboratory identifier for a batch of samples of similar matrices analyzed by one method and treated	Text	12	X

Table A3
Field Description for the Sample Analysis (Table A3)
This table contains information related to analyses of field samples and laboratory QC samples (excluding calibrations and tunes) on a sample level for environmental chemical analyses including radiochemistry

Field Name	Field Name Description	Field Type	Field Length	Required Value
	as a group for matrix spike, matrix spike duplicate, or laboratory duplicate association The method batch links the matrix spike and/or matrix spike duplicate or laboratory duplicates to associated samples. Note the MethodBatch association may coincide with the PreparationBatch association. The MethodBatch is specifically used to link the MS/MSD and/or DUP to associated samples.			
PreparationBatch	Unique laboratory identifier for a batch of samples prepared together for analysis by one method and treated as a group for method blank, LCS and LCSD association. The PreparationBatch links method blanks and laboratory control samples (blank spikes) to associated samples. Note, the PreparationBatch association may coincide with the MethodBatch association but the PreparationBatch specifically links the Method Blank and LCS to associated samples.	Text	12	X
RunBatch	<u>For all other methods</u> the RunBatch is the unique identifier for a batch of analyses performed on one instrument under the control of one initial calibration and initial calibration verification. The RunBatch links both the initial calibration and initial calibration verification to subsequently analyzed and associated continuing calibrations, field samples, and QC analyses. For GC/MS methods, the RunBatch also links a BFB or DFTPP tune. A distinct RunBatch must used with every new initial calibration within a method The value entered in this field links a particular sample/method/analysis type record to a set of associated initial calibration and initial calibration verification records from Table A2. If Table A2 is not submitted enter a value of ‘NA” in this field.	Text	12	X
AnalysisBatch	<u>For radiochemistry methods</u> leave this field blank. <u>For all other methods</u> the AnalysisBatch is the unique identifier for a batch of analyses performed on one instrument and under the control of a continuing calibration or continuing calibration verification. The AnalysisBatch links the continuing calibration or	Text	12	X

Table A3
Field Description for the Sample Analysis (Table A3)
This table contains information related to analyses of field samples and laboratory QC samples (excluding calibrations and tunes) on a sample level for environmental chemical analyses including radiochemistry

Field Name	Field Name Description	Field Type	Field Length	Required Value
	calibration verification to subsequently analyzed and associated field sample and QC analyses. For GC/MS methods, the AnalysisBatch also links the BFB or DFTPP tune. A distinct AnalysisBatch must be used with every new continuing calibration or continuing calibration verification within a method The value entered in this field links a particular sample/method/analysis type record to a set of associated continuing calibration records in the Laboratory Instrument table.			
LabReportingBatch	Unique laboratory identifier for the EDD. This is equivalent to the sample delivery group, lab work number, login ID, etc. The LabReportingBatch links all records in the EDD reported as one group. The value entered in this field must be the same in all records.	Text	12	X
LabReceipt	Date and time the sample was received in the lab. A time value of 00:00 may be entered. Refer to the date/time format at the end of this table.	Date/ Time	16*	X
LabReported	Date and time hard copy reported delivered by the lab. A time value of 00:00 may be entered. Refer to the date/time format at the end of this table.	Date/ Time	16*	X
Comment	Add any comments or additional information specific to the sample analysis data record.	Text	200	

C Only required for regular samples, duplicates and MS/MSDs.
X Required field.

(1) Field Length indicates decimal precision in parenthesis. For example, 5(2) = a total width of 5 numbers including a maximum of 2 decimal places.

* Format Date and Time as MM/DD/YYYY hh:mm; where MM = two digit month, DD = two digit day, and YYYY = four digit year, hh = hour in 24 hour format, and mm = minutes.

3.0 Laboratory Data Checker

The Laboratory Data Checker is a web-based application that will review Laboratory Electronic Data Deliverables (LEDDs) for adherence to Tetra Tech's EDD format requirements. EDDs will be reviewed for elements such as missing data and/or columns of data, and compliance of the data within each column to the required data types/lengths. Once an EDD passes through the checker with no errors, it must be submitted to Tetra Tech through the LEDD Checker application.

Access to the LEDD Checker application will be provided by an initial registration/approval process. An Information Systems Group (ISG) Administrator will approve requests for access**.** To access the site or begin

the registration process, visit the ISG web site at http://isg.ttnus.com and select the “Laboratory Checker” link on the left of the home page. Registered users may access the checker immediately by logging in to the system using theircredentials. New users must select the “Register” button and provide all of the requested information**.**

After completing all fields on the registration form, select the “Submit” button to complete the request process. Upon verification by an ISG Administrator, an email notification will be sent verifying the user ID, password and account status. Forgotten passwords may be retrieved by using the “Forgot password?” link on the login page. Note that the email address that was provided for registration or password retrieval is the user ID and must be a valid e-mail address.

The general process for submitting EDD files through the LEDD Checker involves a 3-stage process that includes an upload stage, an error checking stage and a submittal stage.

Log into the LEDD Checker by typing your login credentials and select the “Login” button. The LEDD Checker home page provides a general overview of the checker functionality and EDD file format requirements. At the bottom of the home page, example EDDs are provided that may be viewed or downloaded. To download the files, right click on the link and select “Save target as” from the menu. Each LEDD Checker page includes a navigation bar with links to return to the home page or continue the checking and submittal process. Users should **NOT** use the back or forward buttons on the browser, instead use the links provided in the application to navigate through the site.

Detailed information regarding EDD preparation, formatting requirements and text file naming conventions are provided in the Electronic Data Format Requirements Section of the Laboratory SOW.

Begin the upload stage by selecting the “Upload/Check Files” link on the home page. Follow the steps on the upload page starting with the selection of the laboratory name that corresponds to your organization. If your organization is not listed, contact LabSupport@tetratech.com, and provide a full description of your organization name, contact information and include “Laboratory Contractor ID Request” in the subject line. An ISG Administrator will respond to the request via e-mail.

Load the appropriate A1, A2, or A3 target EDD files by clicking the “Browse” button next to each data table input box. A file browser dialog will appear allowing files to be selected from a local or network drive. After the EDD files are loaded, click the “Upload” button to complete the upload stage. Note that each table may be uploaded and checked separately; however, a minimum of the A1 and A3 files are required in order to submit the EDDs.

If the file upload was successful, the checking page will immediately load. Begin the checking stage by selecting the “Check Files” button. The LEDD Checker will begin validating the EDD files for compliance. Depending on file size and network activity the validation process may take several minutes. The progress should be displayed in the information bar at the bottom of the browser window. **Do not** select the “Check Files” button again or otherwise use the browser during this process. Other applications may be used; however, note that the LEDD Checker may not sit idle for more than 30 minutes. If the time is exceeded a new session must be started in a new browser window.

Any errors will be processed and returned on the error page. The following general errors may be returned.

- Column count / table structure errors – due to column header names being included, improper delimiter, extra tabs, extra or missing columns of data, spaces or other characters at the end of a row.
- Row and column value specific errors – may occur for one or more reasons including: data truncation, invalid date / time format, invalid decimal precision or field width exceedance, or if a value is not in a list of valid values or expected range.

If column count / table structure errors are encountered, the LEDD Checker will return an error and stop the checking process.

The EDDs will not be processed any further until the column errors are resolved. Text fields are validated for truncation. Date / Time fields are validated for truncation and format compliance. Numeric decimal fields are validated for truncation, character type compliance and decimal precision. All required fields are validated for null values or empty text strings (i.e. spaces). The LEDD Checker will return a list of all errors in and include a reference to the row number on which the error occurred. Note that consecutive EDD files may be loaded and checked, and submitted while logged in. However, no data may be submitted until all EDD files have passed through the LEDD Checker without errors. The list of errors may be printed by selecting the “Print this Page” button from the checker error page.

If the EDD files pass with no errors, the submittal page will immediately load. To complete the submittal stage, include the following information in the comment and additional information area of the form: laboratory name, laboratory contact person, project name, project number, site name/number, fractions included and any specific comments related to the EDD. Select the “Submit Files” button to continue the submittal process.

The submittal stage is not considered complete until a unique ticket key reference is returned in the browser window. The ticket key reference must be printed for record of submission and future reference. In addition, a copy of the ticket key reference must be included in the PDF data package.

From: Rich, Corey
Sent: Friday, July 23, 2010 12:58 PM
To: Balsamo, Nina; Carper, Kelly
Subject: FW: Test America - Chromium Speciation

FYI - An earlier email regarding accreditation.

Corey

-----Original Message-----
From: Nielsen, Janice L CIV NAVFAC, LANT [mailto:janice.nielsen@navy.mil]
Sent: Monday, July 12, 2010 8:20 AM
To: Gravette, James CIV NAVFAC; Rich, Corey
Cc: Tucker, Jonathan P CIV NAVFAC Lant; Ganser, Leanne; Johnston, Tom; Bowers,
Kenneth A CIV NAVFAC Lant; Adelson, Jordan M CIV SEA04X, LQAO
Subject: RE: Test America - Chromium Speciation

Jim and Corey: Sounds like we should be in ok shape based on Jordan's e-mail
below. Please let me know if you have concerns as your date approaches to
complete the work. Thanks, Jan

Jan Nielsen
NAVFAC LANT
Restoration Sciences Support Section
(757)322-8339

-----Original Message-----
From: Adelson, Jordan M CIV SEA04X, LQAO
Sent: Friday, July 09, 2010 12:31 PM
To: Gravette, James CIV NAVFAC; 'Rich, Corey'
Cc: Tucker, Jonathan P CIV NAVFAC Lant; Ganser, Leanne; Johnston, Tom; Bowers,
Kenneth A CIV NAVFAC Lant; Nielsen, Janice L CIV NAVFAC, LANT
Subject: RE: Test America - Chromium Speciation

I was copied on an email that was just sent from the Accreditation Body to the
laboratory indicating that the expansion to the scope for Cr speciation is
awaiting a edit to the laboratory's SOP. Once this edit is made the AB is
prepared to expand the scope.

It sounds like all of this should be resolved by early next week.

r/
Jordan

-----Original Message-----

From: Rich, Corey [mailto:Corey.Rich@tetratech.com]
Sent: Thursday, July 08, 2010 9:22
To: Adelson, Jordan M CIV SEA04X, LQAO
Cc: Gravette, James CIV NAVFAC; Tucker, Jonathan P CIV NAVFAC Lant; Ganser,
Leanne; Johnston, Tom; Bowers, Kenneth A CIV NAVFAC Lant; Nielsen, Janice L
CIV NAVFAC, LANT
Subject: RE: Test America - Chromium Speciation

Jordan,

Per your request to Jim Gravette, please find below the contact information
for Test America. Let me know if you need anything else.

Regards,
Corey

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING
301 Alpha Drive
Pittsburgh PA 15238
www.testamericainc.com

TestAmerica is DoD ELAP accredited!

Test America Contacts:
Pittsburgh office: 412-963-7058
Dave Miller - Product Specialist for method 6800
(412-963-2424)
Dave.Miller@testamericainc.com
Rusty Vicinie -General Manager
Rusty.vicinie@testamericainc.com
Larry Matko - Lab director
Larry.matko@testamericainc.com
Barbara Hall - Account executive
Barb.Hall@testamericainc.com

-----Original Message-----
From: Nielsen, Janice L CIV NAVFAC, LANT [mailto:janice.nielsen@navy.mil]
Sent: Wednesday, July 07, 2010 5:38 PM
To: Rich, Corey; Adelson, Jordan M CIV SEA04X, LQAO
Cc: Gravette, James CIV NAVFAC; Tucker, Jonathan P CIV NAVFAC Lant; Ganser,
Leanne; Johnston, Tom; Balsamo, Nina; Bowers, Kenneth A CIV NAVFAC Lant
Subject: RE: Test America - Chromium Speciation
Importance: High

Corey: Since the accrediting board does not work for the navy we have very
little control over the timing of the accreditation effort but I will work to

find out the status and make sure we do what we can to support the efforts of
the team. With this in mind I am sending to Jordan Adelson of NAVSEA who works
with us on this program and is the primary point of contact for the DOD ELAP
program to see what we can find out. Jan

Jordan: The lab is ELAP accredited but not for the chromium speciation method
SW846 6800. They have submitted the request to have this added to their
accreditation but the accreditation process has not been completed yet. Is
there any way you can let us know of the status of this effort? I know you
don't control the timing or work of the ABs but thought you might have some
insight or suggestions on next steps. The project needs to make progress and
may be put in a position that they will be sending out the SAP before the lab
is fully accredited for this additional method. The SAP is anticipated to be
sent out for regulatory review next week with the field work planned for
August 2010. My understanding is that this is typically a pretty quick
process for a lab already in the program and I thought they may be able to
keep moving forward assuming the process will be completed with good results
before the lab receives the samples but wanted to discuss with you and get
your
input. I was not sure if we should try to get a project specific approval
for this project or if you thought we might be ok. Appreciate any insight and
advice. V/R Jan Nielsen

Jan Nielsen
NAVFAC LANT
Restoration Sciences Support Section
(757)322-8339

-----Original Message-----
From: Rich, Corey [mailto:Corey.Rich@tetratech.com]
Sent: Wednesday, July 07, 2010 5:06 PM
To: Nielsen, Janice L CIV NAVFAC, LANT
Cc: Gravette, James CIV NAVFAC; Tucker, Jonathan P CIV NAVFAC Lant; Ganser,
Leanne; Johnston, Tom; Balsamo, Nina
Subject: Test America - Chromium Speciation

Jan,

Per Jim Gravette's request and our discussion this afternoon, I am providing you with the following information to hopefully expedite Test America's audit and approval to perform chromium speciation for soil samples to be collected and analyzed at the Lower Subase at Naval Submarine Base - New London.

Laboratory - Test America

Analyte - Hexavalent Chrome

Method - SW846 Method 6800

I cc'd Jon Tucker, the Navy chemist that has been involved on this project, to keep him informed of our efforts.

Tetra Tech NUS is currently preparing the draft final version of the UFP Soil Pre-Design Investigation SAP and is hopeful to submit it to the team for review/concurrence next week. The Navy would like field work initiated in August 2010 (ASAP). Based on discussions with the laboratory today, they have submitted all of the required paperwork, but they have not heard back regarding the timing of the audit. Anything you can do to help expedite this process would be appreciated.

Thank you,

Corey Rich | Senior Project Manager/Civil Engineer
Direct: 412.921.8984 | Main: 412.921.7090 | Fax: 412.921.4040
Corey.Rich@tetratech.com

Tetra Tech NUS, Inc. | Technical Support Services Group
661 Andersen Drive Foster Plaza 7 | Pittsburgh, PA 15220

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confidential and/or inside information. Any distribution or use of this
communication by anyone other than the intended recipient is strictly
prohibited and may be unlawful. If you are not the intended recipient, please
notify the sender by replying to this message and then delete it from your
system.

From: Miller, Dave [Dave.Miller@testamericainc.com]
Sent: Monday, July 26, 2010 10:41 AM
To: Ganser, Leanne
Cc: Hall, Barb
Subject: FW: Request for Expanded Scope

Leanne:

The email I referred to is attached below.

Dave

From: larry jackson [mailto:lpeytonj@comcast.net]
Sent: Friday, July 16, 2010 6:20 AM
To: DeRubeis, Nasreen; bill.hirt@aclasscorp.com; 'Collins, Erica F.'
Cc: Reinheimer, Bill; jordan.adelson@navy.mil
Subject: RE: Request for Expanded Scope

I have reviewed the revised SOP provided by the laboratory to cover the issue of dead time correction in the detector response. It adequately discusses the laboratory procedure to adjust for detector/drift/decay and meets the requirements of the reference method, EPA 6800.

I recommend that the laboratory scope of accreditation be expanded to include EPA Method 6800 at this time.

larry

> -----Original Message-----
> **From:** DeRubeis, Nasreen [mailto:Nasreen.DeRubeis@testamericainc.com]
> **Sent:** Wednesday, July 14, 2010 2:02 PM
> **To:** larry jackson
> **Cc:** Reinheimer, Bill; bill.hirt@aclasscorp.com; jordan.adelson@navy.mil; Hall, Barb; Vicinie, Rusty; Matko, Larry
> **Subject:** RE: Request for Expanded Scope
>
> Hi Larry,
> Enclosed is our revised SOP for method 6800. Please let me know if you have any questions.

Nasreen K. DeRubeis
Quality Assurance Manager

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

301 Alpha Drive
Pittsburgh, PA 15238
412-963-2425
412-963-2468 - fax
www.testamericainc.com

From: larry jackson [mailto:lpeytonj@comcast.net]
Sent: Monday, July 12, 2010 1:50 PM
To: DeRubeis, Nasreen
Cc: Reinheimer, Bill; bill.hirt@aclasscorp.com; jordan.adelson@navy.mil; 'larry jackson'
Subject: RE: Request for Expanded Scope

Nasreen --

Thank you for your prompt response to my question. I called Bill right away and discussed the issue with him. As I suspected, this correction for 'dead time' is software related and based on the daily analysis of a certified reference sample and can be thought of as an auto-tune type of procedure where the software alerts the analyst if detector response has changed beyond the level of linearity and cross calibration required over the calibration concentration range. The analyst then performs corrective action to return the instrument to the required level of performance. Bill assures me that this procedure is required by the instrument manufacturer and has been in place over the lifetime of the procedure in your facility. Therefore, your current PT performance, DL, LOD, and LOQ based on the ICP-MS determinative method are considered current and applicable to this analysis.

My assessment of your procedure indicates it meets the technical requirements of the method but your laboratory has not documented this important step/ requirement in your quality system documents such as an SOP. You can satisfy the ACLASS expectation for a corrective action by revision of your document(s) to include this requirement and a description of the procedure, including corrective actions [sample re-analysis, etc.] when linearity and cross calibration does not meet specifications. Full revision of your SOP at this time is not necessary as long as you provide, in writing, a one to two page "Change Notice" identifying the document(s) affected, the language to be inserted / revised, its location in the document, the effective date of the change, and a description of how the analyst(s) currently trained to the method have been brought up to date with the change. You will also need to address the issue of documenting this procedure whenever it is performed in a manner similar to the way you document the BFB tune for a GC/MS procedure.

This documentation requirement must also be described in your "change notice". The 'change notice' will have to go through the same document control / document approval process described in DoD QSM section 4.3. The change must be incorporated in the full QS document(s) during the next scheduled review/revision cycle.

Upon receipt of the approved change notice and ACLASS review and approval, I expect we immediately will issue you a revised certificate reflecting the addition. We shall also undertake to inform the DoD ELAP of the update and provide the documentation they require to support the update at the same time.

Awaiting your reply, -- respectfully,

larry jackson,
ACLASS

-----Original Message-----
From: DeRubeis, Nasreen [mailto:Nasreen.DeRubeis@testamericainc.com]
Sent: Monday, July 12, 2010 12:57 PM
To: larry jackson
Cc: Reinheimer, Bill
Subject: FW: Request for Expanded Scope

Hi Larry,
Here is the explanation regarding the dead time. If you would like to speak to Bill, he can be reached at the lab @ 412-963-7058.

Nasreen K. DeRubeis
Quality Assurance Manager

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

301 Alpha Drive
Pittsburgh, PA 15238
412-963-2425
412-963-2468 - fax
www.testamericainc.com

From: Reinheimer, Bill
Sent: Monday, July 12, 2010 12:45 PM
To: DeRubeis, Nasreen
Subject: RE: Request for Expanded Scope

Nas-

Attached is a copy of what our Cross calibration looks like.
We do it on a daily basis. It is a solution containing all the analytes we test for at concentrations that are at levels where the detector reads in both pulse-count and analogue modes. The instrument has a default dead time set at 30 nanoseconds. If the cross calibration would fail the detector voltages are then automatically adjusted to achieve the dead time setting.
Bill.

From: DeRubeis, Nasreen
Sent: Friday, July 09, 2010 2:53 PM
To: Reinheimer, Bill; Vicinie, Rusty; Matko, Larry
Subject: FW: Request for Expanded Scope

Hi Bill, I need to speak to you on Monday. Could you let me know when you get this message so I can call you. Larry Jackson who is doing the approval for ACLASS for 6800 has a question that I need to disuses with you. Once this is resolved, he will be able to approve us for 6800 for DoD.
Thanks

Nasreen K. DeRubeis
Quality Assurance Manager

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Pittsburgh, PA 15238
412-963-2425
412-963-2468 - fax
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From: larry jackson [mailto:lpeytonj@comcast.net]
Sent: Friday, July 09, 2010 12:18 PM
To: DeRubeis, Nasreen
Cc: 'Hirt, Bill'; jordan.adelson@navy.mil; 'larry jackson'
Subject: Request for Expanded Scope

Nasreen--

Thank you for your return phone call from my voice mail of yesterday.

I have completed my review of the materials you have submitted to support your application to expand your ACLASS accreditation under the DoD ELAP

for EPA method 6800. As I explained in our conversation, I have one question about your SOP. EPA method 6800, section 4.2.2 places considerable emphasis on the "dead time correction" of MS detector bias which must be performed daily. This required step is not included in your SOP; section 4.7 provides some very limited mention of the subject but otherwise the SOP is silent on how this requirement is satisfied.

I understand that your technical staff is not available to help answer this issue because of vacation schedules. I am prepared to approve the addition of this method to your scope based on the technical review when the question of "dead time correction" is resolved. I assume you have made the necessary arrangements with ACLASS to complete any administrative matters. If so, I believe we can finish all steps required to add this method to your scope and send you a revised scope within one day.

I will be available Monday to talk with your staff should they call me. For Tuesday and Wednesday, an e-mail response will be best since I will be out of the office. At a minimum, I believe your SOP will have to be revised to address the "dead time correction" issue. It may be embedded in the instrument software as a type of 'auto-tune' function or it may have to be preformed by the analyst but either way, the SOP should address how it is done and it's frequency.

Call me if I can provide you more information should you need it,

larry

tel: 603-924-6852
email: lpeytonj@comcast.net

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Please consider the environment before printing this e-mail.

CERTIFICATE OF ACCREDITATION

ANSI-ASQ National Accreditation Board/ACLASS
500 Montgomery Street, Suite 625, Alexandria, VA 22314, 877-344-3044

This is to certify that

Test America - Pittsburgh
301 Alpha Drive
Pittsburgh, PA 15238

has been assessed by ACLASS
and meets the requirements of

DoD-ELAP

while demonstrating technical competence in the field(s) of

TESTING

Refer to the accompanying Scope(s) of Accreditation for information regarding the types of tests to which this accreditation applies.

ADE-1422
Certificate Number

ACLASS Approval

Certificate Valid: 03/12/2010-03/12/2012
Version No. 001

SCOPE OF DoD-ELAP ACCREDITATION

TestAmerica Pittsburgh

301 Alpha Drive, Pittsburgh PA 15238
Nasreen K. DeRubeis Phone: 412-963-7058

TESTING

Valid to: March 12, 2012 Certificate Number: ADE - 1442

I. Environmental

MATRIX	SPECIFIC TEST or ANALYTE GROUP	SPECIFICATION OR STANDARD METHOD (all SW846 unless specified)	* KEY EQUIPMENT OR TECHNOLOGY USED
Water and Solids	Metals	6010B / C	ICP-AES
Water and Solids	Metals	6020 / 6020A	ICP-MS
Water and Solids	Mercury	7470A and 7471A / B	CVAA
Water and Solids	Hexavalent Chromium with Alkaline Digestion	7196A	Spectrophotometer
Water and Solids	Total Cyanide	9012A / B 9010B / C / 9013	
Water and Solids	Anions	9056A	IC
Water and Solids	Oil and Grease	9071B / 9070A EPA 1664A	Gravimetric
Water and Solids	Organochlorine Pesticides	8081A / B	GC
Water and Solids	Organo-Phosphorus Compounds	8141A / B	GC
Water, Solids, and Oil	PCBs	8082 / 8082A	GC
Water and Solids	Chlorinated Herbicides	8151A	GC

MATRIX	SPECIFIC TEST or ANALYTE GROUP	SPECIFICATION OR STANDARD METHOD (all SW846 unless specified)	* KEY EQUIPMENT OR TECHNOLOGY USED
Water and Solids	Volatiles	8260B	GC/MS
Water and Solids	Semi-Volatiles	8270C / D	GC/MS
Water	Total Organic Carbon	9060/9060A	TOC Analyzer
Water	EDB and DBCP	8011	GC
Water and Solids	PAHs	8310	HPLC
Solids	Total Organic Carbon	Lloyd Kahn	TOC Analyzer
Water and Solids	Sulfide	9030B / 9034	Titration
Water	pH	9040B / C	pH Meter
Solids	pH	9045C / D	pH Meter
Water and Solids	Flashpoint	1020B / AST- D3278-96	Setaflash closed tester
Water and Solids	Flashpoint	1010A / AST- D93-08	Pensky-Martens Closed Flash Tester.
Solids	Percent Moisture	SM 2540G	Balance
Water	Acid Digestion	3005A	FLAA / ICP
Water	Acid Digestion	3010A	FLAA / ICP
Solids	Acid Digestion	3050B	
Solids	Alkaline Digestion	3060A	
Water	Purge-and-Trap	5030B	
Solids	Closed-system Purge-and-Trap	5035	
Solids	Waste Dilution	3585	
Solids	Automated Soxhlet Extraction	3541	

MATRIX	SPECIFIC TEST or ANALYTE GROUP	SPECIFICATION OR STANDARD METHOD (all SW846 unless specified)	* KEY EQUIPMENT OR TECHNOLOGY USED
Water	Liquid-Liquid Extraction	3510C	
Water	Continuous Liquid-Liquid Extraction	3520C	
Solids	Ultrasonic Extraction	3550C	
Solids	Waste Dilution	3580A	
Water and Solids	Sulfur Cleanup	3660B	
Water and Solids	Gel Permeation Cleanup	3640A	
Water and Solids	TCLP Toxicity Leaching	1311	

Notes:

1. ** = As Applicable*
2. *This scope is part of and must be included with the Certificate of Accreditation No. ADE-1442*

Vice President

APPENDIX F

LABORATORY STANDARD OPERATING PROCEDURES
KATAHDIN ANALYTICAL SERVICES, INC.

KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

SOP Number: CA-101
Revision History
Cover Page
Page 1

TITLE: **EQUIPMENT MAINTENANCE**

Prepared By: Julie Ricardi Date: 8·96

Approved By:

Group Supervisor: _____ Date: _____

Operations Manager: Joel C. Burton Date: 12/15/00

QA Officer: Deborah J. Nadeau Date: 12·13·00

General Manager: JC Burton for D. McGrath Date: 12/15/00

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01	Added requirements for recording maintenance in appropriate log. Added Archon maintenance. Modified Lachat maintenance. Attached equipment list.	DN	12·13·00	12·13·00
02	Removed references to General Manager. Removed furnace (GFAA) maintenance from Figure 1 and added ICP-MS. Updated equipment list.	DN	10·22·02	10·22·02
03	Changed Supervisor to department manager. Added maintenance for the ASE and general lab maintenance. Updated equipment list.	HRC	06.03.04	06.03.04
04	Added new equipment list.	LAD	01·28·05	01·28·05
05	Added Horizon SPE extractor & HPLC maintenance to Table 1. Added TOC analyzer, IC & miscellaneous instrument maintenance to Table 2. Updated equipment list.	DN	03·06	03·06

TITLE: **EQUIPMENT MAINTENANCE**

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
06	Updated ICP maintenance to include maintenance for new ICP (7500). Updated equipment list.	DR	04.07	04.07
07	Updated equipment list.	DR	07.08	07.08
08	Added more detail to section 7.3.3 describing corrective action documentation. Added maintenance for GPC. Updated equipment list. Added sections on unscheduled maintenance and troubleshooting and on service contracts and outside maintenance	DR	08/09	08/09

TITLE: EQUIPMENT MAINTENANCE

Please acknowledge receipt of this standard operating procedure by signing and dating both of the spaces provided. Return the bottom half of this sheet to the QA Department.

I acknowledge receipt of copy ___ of document **CA-101-08**, titled **EQUIPMENT MAINTENANCE**.

Recipient: _____Date:_____

KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy ___ of document **CA-101-08**, titled **EQUIPMENT MAINTENANCE**.

Recipient: _____Date:_____

TITLE: EQUIPMENT MAINTENANCE

1.0 SCOPE AND APPLICATION

The consistent generation of high quality analytical results requires properly operating analytical equipment. Accordingly, Katahdin Analytical Services, Inc. has an equipment maintenance program, which prescribes the necessary maintenance measures required and also schedules the frequency of conduct and documentation of those measures.

The purpose of this SOP is to describe the routine scheduled maintenance procedures employed for analytical and support equipment and to discuss non-scheduled maintenance of analytical and support equipment utilized by Katahdin Analytical personnel. It is applicable to all equipment utilized by laboratory personnel for analytical and support work. The procedures outlined in this SOP provide general guidance for equipment maintenance and documentation. The appropriate equipment manufacturer's manual(s) and specific analytical SOPs must be available and may provide specific maintenance and cleaning procedures.

1.1 Definitions

1.2 Responsibilities

It is the responsibility of each technical employee to carry out and document the required routine equipment maintenance at the required frequency for the instrumentation that they use.

It is the responsibility of each Department Manager to ensure that maintenance performed in-house is conducted properly by experienced personnel familiar with the equipment. On a routine basis, the department manager or qualified designee reviews equipment maintenance activities and recordkeeping requirements. Deficiencies are brought to the attention of the analyst who is responsible for an unacceptable procedure or entry, and any corrections are made by the appropriate analyst.

The Quality Assurance Officer is responsible for conducting periodic audits of the equipment maintenance program and documentation in each analytical area. Maintenance logs and contracted vendor maintenance records are reviewed for completeness and frequency of maintenance. Deficiencies are immediately brought to the attention of the Supervisor who is responsible for informing his/her staff and initiating the necessary corrective or improved actions in his/her analytical area.

2.0 SUMMARY OF METHOD

Not applicable.

3.0 INTERFERENCES

Refer to specific instrument manufacturer's operating manuals.

TITLE: EQUIPMENT MAINTENANCE

4.0 APPARATUS AND MATERIALS

Not applicable.

5.0 REAGENTS

Not applicable.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

Not applicable.

7.0 PROCEDURES

The laboratory is equipped with all equipment needed for analysis and support activities. All equipment is maintained in working condition through a schedule of preventive maintenance and/or outside service contracts or appointments as needed. All maintenance, including internal maintenance and outside service calls, is documented. Any out-of-service equipment is marked as such until it can be repaired and shown by calibration or testing to perform correctly.. In cases where outside equipment is rented or borrowed, any records necessary to demonstrate equipment calibration or maintenance will be requested from the provider.

All equipment must be maintained so that its performance is acceptable for its intended use. For example, an instrument that does not have sufficient sensitivity should not be used for low-level analysis or a balance not verified above 100 grams should not be used to weigh items over 100 grams.

7.1 ROUTINE PREVENTIVE MAINTENANCE

Preventive maintenance (PM) may be conducted by qualified Katahdin personnel or may be performed under contract by authorized service technicians. Between service visits, instruments having PM service contracts are cleaned and maintained according to manufacturer's specifications by experienced Katahdin personnel. Instruments not under service contracts are cleaned and maintained by experienced laboratory personnel according to manufacturer's specifications and based on professional experience. The frequency of maintenance performed depends on the equipment. Preventive maintenance in each analytical section at Katahdin Analytical is scheduled, conducted and documented on a regular basis by the analytical section staff. Many maintenance needs (e.g., accidental breakage, part failure) are not satisfied by scheduled maintenance and are performed as needed. This would apply to routine daily instrument maintenance as well as non-scheduled instrument maintenance (e.g.,

replacement of GC injection port liners, replacement of volatiles Tekmar traps, etc.). Maintenance procedures indicated in Tables 1 and 2 are documented in appropriate logbooks maintained in each analytical area. Included in the documentation are general remarks describing the maintenance operations performed, the date maintenance was performed, and the signature or initials of the individuals who completed the maintenance. If a specific instrument problem prompted the need for maintenance, the problem and its resolution are documented along with the description of maintenance performed. Specific requirements for maintenance logs are described in Section 7.3.

7.2 PREVENTIVE MAINTENANCE SCHEDULES

Tables 1 and 2 summarize the routine preventive maintenance procedures and schedules followed within Katahdin Analytical for major laboratory instrumentation. These items are required to be documented in the applicable instrument or maintenance log. Refer to Section 7.3 for specific requirements for maintenance log documentation.

7.3 UNSCHEDULED MAINTENANCE & TROUBLESHOOTING

In addition to preventive maintenance, analysts are often required to perform unscheduled maintenance due to sporadic instrument problems. The instrument problems could be obvious, such as those cause by "nasty" samples, or not so obvious. The problem could be more mechanical, such as an autosampler jamming or a needle clogging, or the problems could be more technical such as failing QC for no apparent reason. In all cases, analysts shall proceed through a series of troubleshooting steps to investigate the problem. Analysts should consult the manufacturer's guidelines for troubleshooting certain equipment problems. Changing only one parameter and/or part at a time is critical to ascertain which change may improve or correct a problem.

All troubleshooting steps and outcomes must be recorded in the applicable instrument maintenance logbook for future reference and guidance. The steps taken, which correct the problem should be noted as such. If it is determined that a problem with the equipment exists, and that the problem has had an impact on the quality of past completed data, an immediate Corrective Action Report (CAR) must be initiated. A thorough investigation of all impacted data must be completed and corrective action must be implemented. Refer to the current revision of Katahdin SOP, QA-803, Laboratory Quality Assurance Self Inspection System, for further details.

7.4 MAINTENANCE LOG (or equivalent) REQUIREMENTS

Maintenance must be recorded in either the instrument run log or in a separate maintenance log. For equipment that does not have a run log or does not necessitate a separate maintenance log (i.e. little maintenance required), any maintenance may be recorded in the appropriate logbook (i.e. balance maintenance recorded in the balance calibration log). Attachment A provides a list of analytical equipment for which maintenance activities are recorded. Balances are not listed, although maintenance activities are recorded for these.

7.4.1 Maintenance logs (or equivalent) shall include the type of equipment and the analytical group/analysis for which it is designated, the manufacturer's model number and serial number, as well as any laboratory-assigned identifier (e.g., GC08, MS2, etc.). Each lab-assigned identifier must be unique and each instrument must have only one lab-assigned identifier. As new instrumentation is received, the date acquired, condition when received (new, used, reconditioned), and date put into use should be recorded in the new instrument's maintenance log.

7.4.2 Each page of the maintenance logbook (or equivalent) must clearly indicate the type of equipment and laboratory-assigned identifier in the header information. Maintenance entries shall include the date and signature or initials of the individual performing the maintenance as well as a brief explanation of the maintenance performed.

7.4.3 If maintenance is conducted as the result of a suspected or known instrument problem, the maintenance record must clearly describe the following:

- Statement of the Problem: brief description of the problem (i.e calibrations are failing)
- Cause Analysis: what might be causing the problem. This could be something that occurred just prior to the problem (i.e. electrical outage, analysis of nasty samples) or something that is unknown.
- Corrective Action Plan: What might fix the problem (i.e replacing liners, tubing or column that could be "damaged" from a nasty sample)
- CAP Implementation: an indication of when the maintenance is performed
- Demonstration of CAP Implementation: an indication of whether the problem was resolved and the date when return to control was noted. Document any changes resulting from the CAP.

7.4.4 On a routine basis the Department Manager or qualified designee shall review equipment maintenance activities and adherence to recordkeeping requirements. Deficiencies are brought to the attention of the analyst who was responsible for the unacceptable procedure or entry, and any corrections are made by the appropriate analyst. Documentation of the maintenance log review can be made by initialing and dating each page of the log or by indicating "reviewed by", initials and date on the front cover of the maintenance log (or equivalent).

7.4.5 On a periodic basis the laboratory QA Officer shall conduct audits of the equipment maintenance program and documentation in the analytical areas. Maintenance logs and contracted vendor maintenance records are reviewed for completeness and adherence to this SOP. Deficiencies are immediately brought to the attention of the Department Manager who is responsible for informing his/her staff and initiating the necessary corrective actions in his/her analytical area.

7.5 SERVICE CONTRACTS

Katahdin will maintain service contracts for major instrumentation as needed. The need for a service contract will depend on factors such as the cost, the estimated down time, the availability of back-up instrumentation, and the availability of skilled analysts to perform non-routine maintenance. The decision to purchase a service contract is made by the management team of the Vice President, the President, the Operations Manager and the Department Managers.

In cases where a service contract is not in place, and a problem with an instrument or piece of support equipment cannot be corrected in-house, Katahdin will use reputable service companies to perform the repair and/or troubleshooting work. Relationships are maintained with several companies to perform these services.

Occasionally, equipment may need to be sent out to the service company for repair. All equipment must be packaged in large enough boxes to leave room for at least four inches of bubble wrap on every side, including the top and bottom. Any small movable parts must be removed and wrapped separately. When available, a manufacturer's box with appropriate Styrofoam inserts should be used. All equipment returning to the laboratory must be checked and recalibrated to be sure all instrument capabilities and conditions have been maintained.

A preventive maintenance contract is in place for all support equipment and thermometers for cleaning, calibrating to manufacturer's specifications and verifying against NIST traceable sources. The support equipment includes all pH meters, conductivity meters, turbidity meters, balances, spectrophotometers, autoclaves, ovens, and incubators. Field equipment meters are included in this annual verification. All equipment supported under this contract are labeled by the outside calibration company with the last calibration date, and when the next calibration is due. All additional comments of the outside calibration are maintained in files with the QA Officer.

Records of all service contract visits, third party services and preventive maintenance services are maintained to document support equipment performance. All equipment not meeting manufacturer's specifications or those required by the test are removed from service until appropriate corrective actions can be taken.

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

Not applicable.

9.0 METHOD PERFORMANCE

Not applicable.

10.0 APPLICABLE DOCUMENTS/REFERENCES

Applicable equipment manufacturer's operating manuals.

LIST OF TABLES & ATTACHMENTS

TABLE 1 – ROUTINE MAINTENANCE SCHEDULE FOR ORGANICS INSTRUMENTATION
TABLE 2 - ROUTINE MAINTENANCE SCHEDULE FOR INORGANICS INSTRUMENTATION
TABLE 3 - ROUTINE MAINTENANCE SCHEDULE FOR GENERAL LABORATORY EQUIPMENT
ATTACHMENT A – ANALYTICAL EQUIPMENT

TABLE 1

ROUTINE MAINTENANCE SCHEDULE FOR ORGANICS INSTRUMENTATION

GC/MS

Regularly performed maintenance includes, but is not limited to, the following for GC/MS instrumentation:

- Check to ensure the pressure on the primary regulator never runs below 100 psi
- Check to ensure the gas supply is sufficient for the day's activity, and delivery pressures are set as described in the SOP.
- Change septa weekly or as needed.
- Replace/cut GC column as needed.
- Replace GC injector glass liner weekly or as needed.
- Replace glass jet splitter as needed.
- Replace pump oil as needed.
- Change gas line dryers as needed.
- Replace electron multiplier as needed.

GC

Regularly performed maintenance includes, but is not limited to, the following for GC instrumentation:

- Check to ensure the pressure on the primary regulator never runs below 100 psi.
- Check to ensure the gas supply is sufficient for the day's activity, and delivery pressures are set as described in the SOP.
- Change septa weekly or as needed.
- Replace/cut GC column as needed.
- Replace GC injector glass liner as needed.
- Change O_2/moisture traps as needed
- Clean/replace GC detector as needed.

TABLE 1, cont'd

ROUTINE MAINTENANCE SCHEDULE FOR ORGANICS INSTRUMENTATION

Purge and Trap Sample Concentrator

Regularly performed maintenance includes, but is not limited to, the following:

- Check to ensure the gas supply is sufficient for the day's activity and delivery pressures are set as described in the SOP.
- Replace trap as needed.
- Decontaminate the system after running high concentration samples or as required by blank analysis.
- Check system for leaks when problem suspected.

Archons

Regularly performed maintenance includes, but is not limited to, the following:

- Visually inspect sampler for corrosion or significant water seepage past plunger.
- Check system pressure (denotes leaks).
- Check for sufficient standard materials in standard vials.
- Recalibrate x,y,z components, i.e. robotic arm, as needed.

Accelerated Solvent Extractor (ASE)

Regularly performed maintenance includes, but is not limited to, the following:

- Check for leaks at the pump solvent reservoir, valves and other components.
- Inspect needle alignment of source needle.
- Check alignment of autoseal arms.
- Change peek seals and o-rings on cell caps after about 50 extractions per cell.
- Inspect cell edges for nicks and gouges on cell body.
- Inspect stainless steel frits and sonicate in solvent if needed.

TABLE 1, cont'd

ROUTINE MAINTENANCE SCHEDULE FOR ORGANICS INSTRUMENTATION

Horizon SPE Automated Extractor System

Regularly performed maintenance includes, but is not limited to, the following:

- Check and clean sensors with a KIMWipe.
- Change sensors as needed.
- Purge system with solvent before use and after use.
- Clean system with hot water by running method 15 after samples are analyzed and before purging system.

HPLC

Regularly performed maintenance includes, but is not limited to, the following for HPLC instrumentation:

- Check and sonicate pump valves as needed.
- Backflush column as needed.
- Replace analytical column or guard column as needed.
- Filter and replace solvent with every use.
- Replace the UV lamp as needed.
- Perform a column wash at the end of each run
- Change needle and/or needle seat as needed

GPC

Regularly performed maintenance includes, but is not limited to, the following for GPC instrumentation:

- Fill solvent reservoir as needed.
- Empty waste container as needed.

TABLE 2

ROUTINE MAINTENANCE SCHEDULE FOR INORGANICS INSTRUMENTATION

ICP/MS

Regularly performed maintenance includes, but is not limited to, the following for ICP instrumentation:

- Clean torch assembly and spray chamber when discolored.
- Clean nebulizer as needed.
- Check to ensure that the argon supply is sufficient for the day's activity, and that delivery pressures are set as described in the SOP. Change argon tanks as necessary.
- Replace peristaltic pump tubing when it becomes stretched or develops flat spots.
- Check coolant water level weekly; replenish as necessary.
- Check rinse solution level daily; replenish as necessary.
- Check waste container level daily; empty as necessary.
- Check cleanliness of instrument air filters weekly; clean or replace as necessary.
- Check instrument computer date and time at the start of each day; correct as necessary.
- Check condition of vacuum pump oil weekly, replace as necessary.
- Clean sampling and skimmer cones every 2-4 weeks.
- Clean extraction lenses monthly, or as needed.
- Clean Einzel lenses every 3-6 months.

ICP

Regularly performed maintenance includes, but is not limited to, the following for ICP instrumentation:

- Clean torch assembly and spray chamber when discolored.
- Clean nebulizer as needed.

TABLE 2, cont'd

ROUTINE MAINTENANCE SCHEDULE FOR INORGANICS INSTRUMENTATION

ICP, cont'd

- Check to ensure that the argon supply is sufficient for the day's activity, and that delivery pressures are set as described in the SOP. Change argon tanks as necessary.
- Replace peristaltic pump tubing when it becomes stretched or develops flat spots.
- Check coolant water level weekly; replenish as necessary.
- Check rinse solution level daily; replenish as necessary.
- Check waste container level daily; empty as necessary.
- Check instrument computer date and time at the start of each day; correct as necessary.

Mercury Analyzer

Regularly performed maintenance includes, but is not limited to, the following:

- Replace drying tube as necessary
- Replace peristaltic pump tubing when it becomes stretched or develops flat spots.
- Replace mercury lamp as necessary.
- Clean optical cell quarterly or as needed.
- Clean liquid/gas separator when it becomes cloudy. Replace as needed.
- Check waste container level before each use; empty as necessary.
- Check exhaust system integrity before each use; correct as necessary.
- Check instrument computer date and time at the start of each day; correct as necessary.

TABLE 2, cont'd

ROUTINE MAINTENANCE SCHEDULE FOR INORGANICS INSTRUMENTATION

Lachat Autoanalyzer

Regularly performed maintenance includes, but is not limited to, the following:

- Check pump tubing; replace as needed.
- Clean interference filter with Kimwipe.
- Check reagent levels and expiration dates; refill or replace as needed.
- Rinse manifolds with water after analysis.
- Check manifold board surfaces; clean as needed by running under tap water.
- Check supplies and reagents; order as needed
- Check for leaks
- Check autosampler and autosampler trays;clean and/or lubricate as needed.
- Check fittings and o-rings on boards; replace as needed.
- Check peristaltic pump rollers; clean and lubricate as needed.
- Inspect manifold tubing for kinks and/or stains; replace as needed.
- Inspect valve flares; clean or replace as needed.
- Inspect reagent and waste lines; replace as needed.
- Check flow cell; clean as needed with Kimwipe.

Konelab AutoAnalyzer

Regularly performed maintenance includes, but is not limited to, the following:

- Rinse and refill distilled water container weekly.

TABLE 2, cont'd

ROUTINE MAINTENANCE SCHEDULE FOR INORGANICS INSTRUMENTATION

Konelab AutoAnalyzer, cont'd

- Check cleanness of segments weekly.
- Wash reagent tubes monthly.
- Change lamp as needed.
- Change diluent and wash tubes as needed.
- Change mixing paddles as needed.
- Change syringes as needed.
- Change dispensing needles as needed.
- Change drain and waste tubes as needed.
- Check used cuvettes and waste daily.

TOC Combustion Analyzer

Regularly performed maintenance includes, but is not limited to, the following:

- Check level of dilution water, drain vessel water, humidifier water, autosampler rinse water, and phosphoric acid vessel and fill as needed.
- Replace oxygen cylinder as needed.

IC

Regularly performed maintenance includes, but is not limited to, the following for IC instrumentation:

- Check regenerate pump tubing and replace as needed.
- Clean or regenerate column as needed.

TABLE 2, cont'd

ROUTINE MAINTENANCE SCHEDULE FOR INORGANICS INSTRUMENTATION

IC, cont'd

- Replace analytical column or guard column as needed.
- Change suppressor as needed.

Miscellaneous Support Instrumentation (including field testing equipment)

Regularly performed maintenance includes, but is not limited to, the following:

- Replace spectrophotometer lamps as needed.
- Inspect DO probe membrane for tears and check for air bubbles under the membrane.
- Replace the DO probe membrane cap and electrolyte solution as needed.
- Clean pH electrode as needed.
- Change the autotitrator filling solution as needed.
- Clean, check, calibrate to manufacturers' specifications all pH, DO, conductivity, and turbidity meters annually (minimum); clean, check, calibrate to manufacturers' specifications all spectrophotometers and balances annually. All calibrations must be NIST traceable.

TABLE 3

ROUTINE MAINTENANCE SCHEDULE FOR GENERAL LABORATORY EQUIPMENT

General Laboratory Areas

- Clean and calibrate balances annually (minimum).
- Check balance calibration each day of use.
- Clean balance pan prior to each use.
- Calibrate automatic pipettes with each use.
- Calibrate spirit thermometers yearly against an NIST traceable thermometer; calibrate digital thermometers quarterly.
- Record refrigerator, freezer, incubator and oven temperatures each day, when appropriate.
- General housekeeping: keep counter tops, hoods, and floors clean.
- Check airflow in hoods once a week.

ATTACHMENT A

ANALYTICAL EQUIPMENT LIST

ANALYTICAL INSTRUMENTATION

KEY INSTRUMENTATION	DATE IN SERVICE	USED FOR ANALYSIS	Instrument Identification
Hewlett Packard 5973 GC/MS with EPC, Archon Autosampler with foam sensor option and EST ENCON Evolution Purge and Trap Concentrator with heated purge capability	**2008**	5030/8260, 5035/8260, 624, Current versions of CLP SOWs (OLM, OLC, & SOM) (VOA), & 524.2	D
Hewlett Packard 5972 GC/MS with EPC, Centurion Autosampler with foam sensor option and EST ENCON Purge and Trap Concentrator	**2006**	5030/8260, 624, Current versions of CLP SOWs (OLM & SOM) (VOA)	T
Hewlett Packard 5972 GC/MS with EPC; Tekmar LSC-3100 Purge and Trap concentrator; Archon autosampler capable of low soils per Method 5035.	**1998**	5030/8260, 5035/8260, 624, Current versions of CLP SOWs (OLM, OLC, & SOM) (VOA), & 524.2	S
Hewlett Packard 5972 GC/MS with EPC; Tekmar LSC - 3000 purge and trap concentrator; Archon autosampler capable of low soils per Method 5035.	**1996**	5030/8260, 5035/8260, 624, Current versions of CLP SOWs (OLM, OLC, & SOM) (VOA), & 524.2	Z
Hewlett Packard 5972 GC/MS with EPC; Encon Purge and Trap concentrator; Archon autosampler capable of low soils per method 5035.	**1995**	5030/8260, 5035/8260, 624, Current versions of CLP SOWs (OLM, OLC, & SOM) (VOA), & 524.2	M
Hewlett Packard 5972 GC/MS with EPC; Tekmar LSC-3000 Purge and Trap concentrator; Archon autosampler capable of low soils per Method 5035.	**1993**	5030/8260, 5035/8260, 624, Current versions of CLP SOWs (OLM, OLC, & SOM) (VOA), & 524.2	F
Hewlett Packard 5973 GC/MS with EPC and Model 6890 GC and Agilent 7683 autosampler.	**2008**	8270, 625 & Current versions of CLP SOWS (OLM, OLC, & SOM) (SVOA)	G
Hewlett Packard 5973 GC/MS with EPC and Model 6890 GC and Agilent 7683 autosampler.	**2006**	8270, 625 & Current versions of CLP SOWS (OLM, OLC, & SOM) (SVOA)	R

ATTACHMENT A

ANALYTICAL EQUIPMENT LIST, cont'd

ANALYTICAL INSTRUMENTATION

KEY INSTRUMENTATION	DATE IN SERVICE	USED FOR ANALYSIS	Instrument Identification
Hewlett Packard 5973 GC/MS with EPC and Model 6890 GC and Agilent 7673 autosampler.	**1999/ 2001**	8270, 625 & Current versions of CLP SOWS (OLM, OLC, & SOM) (SVOA)	U
Hewlett Packard Model 5890 gas chromatograph with flame ionization detector; Tekmar ALS 2016 autosampler; Tekmar LSC 2000 purge and trap.	**1991**	8015 MOD., GRO	GC04
Hewlett Packard Model 5890 gas chromatograph with a flameionization detector; Agilent Technologies G1888 Network Headspace Analyzer.	**1991/ 2005**	Methane, Ethane and Ethene	GC05
Hewlett Packard Model 5890 gas chromatograph with EPC and dual electron capture detectors(ECD); Hewlett Packard Model 7673 autosampler.	**1993**	8081, 8082, 608	GC06
Agilent Model 6890 gas chromatograph with dual EPC injection ports and micro electron capture detectors; Agilent Model 7683 autosampler	**2000**	504, 608, 8011, 8081, 8082 (including congeners), 8151, current versions of CLP SOWs (OLM, OLC, & SOM) (P/P)	GC07
Agilent Model 6890 gas chromatograph with one EPC injection ports and micro electron capture detectors; Agilent Model 7683 autosampler	**2009**	608, 8081, 8082 (including congeners), current versions of CLP SOWs (OLM, OLC, & SOM) (P/P)	GC08
Hewlett Packard Model 5890 gas chromatograph with flame ionization detector and photo ionization detector; Tekmar ALS 2016 autosampler; Tekmar LSC 3000 purge and trap.	**1988**	8015 MOD., MAVPH, GRO	GC09

ATTACHMENT A

ANALYTICAL EQUIPMENT LIST, cont'd

Katahdin ANALYTICAL SERVICES

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ANALYTICAL INSTRUMENTATION

KEY INSTRUMENTATION	DATE IN SERVICE	USED FOR ANALYSIS	Instrument Identification
Hewlett Packard Model 5890 gas chromatograph with EPC and dual flame ionization detectors; Hewlett Packard Model 7673 autosampler.	**1993/ 1996**	8015 MOD., MAEPH, DRO	GC10
Hewlett Packard Model 5890 gas chromatograph with flame ionization and nitrogen-phosphorous detectors; Hewlett Packard Model 7673 autosampler.	**1992/ 1996**	ALCOHOLS, DMF, GLYCOLS	GC11
Hewlett Packard Model 5890 gas chromatograph with EPC and dual flame ionization detectors; Hewlett Packard Model 7673 autosampler.	**1987**	8015 MOD., MAEPH, DRO	GC12
Agilent Model 6890 gas chromatograph with one EPC injection ports and micro electron capture detectors; Agilent Model 7683 autosampler	**2008**	608, 8081, 8082 (including congeners), current versions of CLP SOWs (OLM, OLC, & SOM) (P/P)	GC01
Hewlett Packard series 1100 HPLC with Quaternary pump, Multiwavelength detector and autosampler	**2008**	8330, 8332	HPLC03
J2 Scientific AccuPrep MPS™ GPC with internal UV detection	**2009**	GPC-3640 Current versions of CLP SOWS (OLM, OLC, & SOM)	GPC01
Horizon SPE-DEX 4790 Automated Extractor System equipped with 4 extractors.	**2001/ 2005**	3535 method development	Horizon #1
Horizon SPE-DEX 3000 Automated Extractor System equipped with 3 extractors.	**2006**	1664, 9070, 9071	Horizon #2
Thermo ICAP 6500 ICP Emission Spectrometer with autosampler.	**2006**	6010, 200.7, ILM05.4	I

TITLE: EQUIPMENT MAINTENANCE

ATTACHMENT A

ANALYTICAL EQUIPMENT LIST, cont'd

ANALYTICAL INSTRUMENTATION

KEY INSTRUMENTATION		DATE IN SERVICE	USED FOR ANALYSIS	Instrument Identification
Agilent 7500a ICP-MS with autosampler.		**2007**	6020, 200.8, ILM05.4	J
CETAC M-6100 Automated Mercury Analyzer with Autosampler		**2004**	7470/7471, 245.1, 245.5, ILM05.4	H
Tekran Series 2600 automated mercury analyzer with gold amalgam preconcentration and atomic fluorescence detector; Model 2620 autosampler.		**2000**	Ultra-trace level mercury (1631)	G
CPI ModBlock™ Metals Digestion Unit – Two 48 Place Units.		**2000**	Metals Aqueous Digestions	Digestion Unit #1 Digestion Unit #2
LACHAT Quickchem 8000 Continuum Series – Automated Ion Analyzer and autosampler.		**2000**	Various	WC1
Shimadzu TOC-V Combustion Analyzer, PC Controlled, High Sensitivity, Auto-Aqueous TOC Autosampler w/ 40 mL vials; Model SSM-5000A Solid Sample Module.		**2002**	TOC	WC2
Waters 717 Plus Autosampler, Waters 431 conductivity detector, Spectra System P4000 pump.	Dionex IonPak AS4A column; Dionex AMMS III chemical suppression system	**1991/ 2004**	Ion chromatography – Various Anions	See "Key Instrumentation " column for identification
	Dionex AS16 column; Dionex ultra ASRS electronic suppression system.		Ion Chromatography - Perchlorate	See "Key Instrumentation " column for identification
Scanning Fluorescence Detector		**1997**	Out of Service	NA
10 position Lab Crest Cyanide Midi-Distillation system.		**1998**	Cyanide	WC3

ATTACHMENT A

ANALYTICAL EQUIPMENT LIST, cont'd

ANALYTICAL INSTRUMENTATION

KEY INSTRUMENTATION	DATE IN SERVICE	USED FOR ANALYSIS	Instrument Identification
Spectronic 401 spectrophotometer.	**2000**	Various	WC4
HACH Turbidimeter, Model 2100A.	**1993**	Turbidity	WC6
Accumet pH/Conductivity Meter, Model 20.	**1998**	Various	WC8
Mettler DL25 Autotitrator & Mettler ST20A sample changer.	**1993**	Alkalinity	WC9
YSI Dissolved Oxygen Meter.	**1990**	Dissolved Oxygen & Biochemical Oxygen Demand	WC10
Konelab 20 Multi-Wavelength Photometric Analyzer.	**2003**	Various	Konelab #1
Dionex Accelerated Solvent Extractor ASE 200.	**2003**	3545	ASE #1
Waters HPLC system with 600E multisolvent delivery system, 717 plus autosampler, Spectra System UV200 detector	**2003/2004**	Food Testing	HPLC01
Thermo Spectronic Genesys 10uv spectrophotometer	**2005**	Food Testing	Food #1
DryVap Concentrator System 5000	**2005**	GC and GC/MS Extractables	DryVap #1

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY: SW 846 METHOD 8270 – Modified for Selected Ion Monitoring (SIM)

Prepared By: GC/MS Department Date: 6/98

Approved By:

Group Supervisor: A Haley Date: 020101

Operations Manager: John C. Benton Date: 1/31/01

QA Officer: Deborah J. Nadeau Date: 1.31.01

General Manager: Dennis P. Lufan Date: 2/01/01

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01 8270C Mod.	Format changes, added pollution prevention, added instrument and other calibration options. Other minor changes to sections 7, 8 & QA Table.	DN	1.31.01	1.31.01
02 8270C	Many changes in formatting. Some additions to section 8 + Table 1 to comply with Navy.	DN	09.30.04	09.30.04
03 8270C	Sect. 7.2: Removed "K" Instrument & added "R" instrument. Added Pentafluorophenol surr. to Tables 3, 5 and Sect. 8.2. Removed all References to TIC's.	LAD	04/06	04/06
04 8270C	Sect. 8.2 - changed 5 to 4 and removed pentachlorophenol. Table 3 and 5 - removed pentachlorophenol. Changed linear regression correlation coefficient criteria. Added MI SOP reference. Added LCS exceedance criteria. Added ICV requirement and criteria. Added RT window procedure.	LAD	06/07	06/07
05 8270C	Added "G" instrument, Removed "X" instrument Edited Section 7.5.1 - initial cal table	LAD	02/08	02/08

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY: SW 846 METHOD 8270 – Modified for Selected Ion Monitoring (SIM)

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
06	Section 5.3.2.3 - Added calibration Mix B. Section 7.5.1 - Edited to address diffent SIM compounds may need to be calibrated at different levels depending on the compound and project requirements	LAD	04/09	04/09
07	Changes made for compliance with DoD QSM version 4.1	LAD	08/09	08/09

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY: SW 846 METHOD 8270 – Modified for Selected Ion Monitoring (SIM)

Please acknowledge receipt of this standard operating procedure by signing and dating both of the spaces provided. Return the bottom half of this sheet to the QA Department.

I acknowledge receipt of copy ___ of document SOP CA-213-07, titled "ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY METHOD 8270 – Modified for Selected Ion Monitoring (SIM)".

Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy ___ of document SOP CA-213-07, titled "ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY METHOD 8270 – Modified for Selected Ion Monitoring (SIM)".

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TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY: SW 846 METHOD 8270 – Modified for Selected Ion Monitoring (SIM)

1.0 SCOPE AND APPLICATION

The purpose of this SOP is to describe the procedures utilized by Katahdin Analytical Services, Inc. laboratory personnel to prepare and analyze water and soil sample extracts for semivolatile organics by EPA SW-846 Method 8270, current revision, modified for selected ion monitoring.

In order to maintain consistency in data quality, this SOP consolidates all aspects of the analyses in one working document, to be revised as necessary.

1.1 Definitions

ANALYTICAL BATCH: 20 or fewer samples which are analyzed together with the same method sequence and the same lots of reagents and with the manipulations common to each sample within the same time period or in continuous sequential time periods.

METHOD BLANK (laboratory reagent blank): An artificial sample designed to determine if method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus. For aqueous samples, reagent water is used as a blank matrix; for soil samples, baked organic-free sand is used as a blank matrix. The blank is taken through the appropriate steps of the process.

CALIBRATION CHECK: Verification of the ratio of instrument response to analyte amount, a calibration check is done by analyzing for analyte standards in an appropriate solvent. Calibration check solutions are made from a stock solution that is different from the stock used to prepare standards.

CALIBRATION STANDARD (WORKING STANDARD): A solution prepared from the stock standard solution that is used to calibrate the instrument response with respect to analyte concentration.

LABORATORY CONTROL SAMPLE (LCS): A blank that has been spiked with the analyte(s) from an independent source and is analyzed exactly like a sample. Its purpose is to determine whether the methodology is in control, and whether the laboratory is capable of making accurate and precise measurements. The matrix used should be phase matched with the samples and well characterized.

MATRIX SPIKE/MATRIX SPIKE DUPLICATE (MS/MSD): Predetermined quantities of stock solutions of certain analytes are added to a sample matrix prior to sample extraction and analysis. Samples are split into duplicates, spiked and analyzed. Percent recoveries are calculated for each of the analytes detected. The relative percent difference between the samples is calculated and used to assess analytical precision.

STANDARD CURVE (CALIBRATION CURVE): A curve that plots concentration of known analyte standard versus the instrument response to the analyte.

STOCK STANDARD SOLUTION: A concentrated solution containing a single certified standard that is a method analyte, or a concentrated solution of a single analyte prepared in the laboratory with an assay reference compound. Stock standard solutions are used to prepare calibration standards.

SURROGATES: Organic compounds which are similar to analytes of interest in chemical composition, extraction and chromatography, but which are not normally found in environmental samples. These compounds are spiked into all blanks, standards, samples and spiked samples prior to analysis. Percent recoveries are calculated for each surrogate.

TARGET: A software system that combines full processing, reporting and comprehensive review capabilities, regardless of chromatographic vendor and data type.

TARGET DB: An oracle database used to store and organize all Target data files.

QUICKFORMS: A laboratory reporting software for Target and Target DB. The QuickForms report module for Target is preconfigured with generalized forms and US EPA CLP report forms and disk deliverables, which can be customized.

1.2 Responsibilities

This method is restricted to use by, or under the supervision of analysts experienced in the analysis of semivolatile organic compounds by EPA Method 8270. Each analyst must demonstrate and document their ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, "Personnel Training & Documentation of Capability," current revision.

It is the responsibility of all Katahdin technical personnel involved in analysis of semivolatiles by Method 8270 to read and understand this SOP, to adhere to the procedures outlined, and to properly document their data in the appropriate lab notebook. Any deviations from the test or irregularities with the samples should also be recorded in the lab notebook and reported to the Department Manager or designated qualified data reviewer responsible for this data.

It is the responsibility of the Department Manager to oversee that members of their group follow this SOP, to ensure that their work is properly documented and to initiate periodic review of the associated logbooks.

1.3 Safety

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDSs for all the materials used in this procedure.

Each qualified analyst or technician must be familiar with the Katahdin Analytical Health and Safety Manual including the Katahdin Hazardous Waste Management Plan and follow appropriate procedures. These include the use of appropriate personal protective equipment (PPE) such as safety glasses, gloves, and lab coats when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location of a respirator and all safety equipment. Each analyst shall receive a safety orientation from their Department Manager, or designee, appropriate for the job functions they will perform.

1.4 Pollution Prevention/Waste Disposal

Whenever possible, laboratory personnel should use pollution prevention techniques to address their waste generation. Refer to the current revision of the Katahdin Hazardous Waste Management Program for further details on pollution prevention techniques.

After analysis, autosampler vials containing sample extracts in methylene chloride are returned to the SVOA hood, and the contents transferred to a labeled waste container. The contents of this container are disposed of in accordance with the Katahdin Hazardous Waste Management Plan and Safety Manual and SOP SD-903, "Sample Disposal," current revision. Expired standards are lab packed, placed in the Katahdin hazardous waste storage area, and disposed of in accordance with this SOP.

2.0 SUMMARY OF METHOD

The process involves the extraction of semivolatiles from a sample using an appropriate solvent followed by clean up steps (where applicable) and concentration of the extract (refer to Katahdin SOP CA-502, "Preparation of Aqueous Samples for Extractable Semivolatile Analyses", SOP CA-512, "Preparation of Sediment/Soil Samples by Sonication Using Method 3550 for Subsequent Extractable Semi-Volatiles Analysis" and SOP CA-526, "Preparation of Sediment/Soil Samples by Soxhlet Extraction Using Method 3540 for Subsequent Extractable Semivolatile Analysis"). An aliquot of the final extract is injected into the gas chromatograph

for compound separation by capillary column, followed by the electron impact mass spectrometer for identification and quantitation.

3.0 INTERFERENCES

Interfering contamination may occur when a sample containing low concentrations of SVOCs is analyzed immediately after a sample containing high concentrations of SVOCs. Any samples that have suspected carryover must be reanalyzed.

4.0 APPARATUS AND MATERIALS

4.1 GC: Hewlett Packard 5890 and/or 6890

4.2 Mass Spectrometers (MS): HP5973, HP5972 and/or HP5970

4.3 Helium: Carrier gas for routine applications. All carrier gas lines must be onstructed from stainless steel or copper tubing; non-polytetrafluoroethylene (non-PTFE) thread sealant or flow controllers with rubber component are not to be used.

4.4 Autosamplers: HP 7673As

4.5 Hamilton syringes: 2.00 uL to 10 mL

4.6 Volumetric glassware: Grade A or equivalent

4.7 Columns: DB-5MS 30m, 0.25mm I.D., 25um film thickness, columns (J&W Scientific) or equivalent.

4.8 Acquisition System: The acquisition system must be interfaced to the MS and allow continuous acquisition of data throughout the duration of the chromatographic program. It must permit, at a minimum, the output of time vs. intensity (peak height or peak area). Hewlett Packard Chemstation or equivalent.

4.9 Data System: The Target software is used for processing data and generating forms.

5.0 REAGENTS

5.1 J.T. Baker Ultra Resi-Analyzed methylene chloride (or equivalent)

5.2 Purge and trap grade methanol

5.3 Standards: Stock standards and working standards are received and recorded in accordance with SOP CA-106 "Standard Preparation and Documentation".

5.3.1 The expiration date for all standards is one year from date of opening the ampule. If the manufacturer's expiration date is before this one year date, the manufacturer's expiration must be followed. New standards must be opened if degradation is observed.

5.3.2 Secondary dilution standards

5.3.2.1 The standards are prepared on an as needed basis (or every 6 months) and stored in screw-cap amber bottles with Teflon liners in the BNA standards freezer between uses. Standards prepared from various stock solutions must always use the first expiration date of any of the solutions used for preparation.

5.3.2.2 Calibration Mix A – Prepare standards in methylene chloride containing the compounds listed in Table 3. The final concentration of each compound is 20 ug/mL.

5.3.2.3 Calibration Mix B - Some compounds must be calibrated at higher concentrations. For these compounds a secondary standard is prepared which will "boost" the concentration of these compounds in the initial calibration. The concentration of this standard is determined on a project to project basis.

5.3.2.4 Internal Standard Solution – Prepare standard in methylene chloride containing 1,4-dichlorobenzene-d4, naphthalene-d8, acenaphthene-d10, phenanthrene-d10, chrysene-d12, and perylene-d12 at a final concentration of 80 ug/mL.

5.3.2.5 DFTPP Solution – Prepare standard in methylene chloride containing DFTPP at a final concentration of 25 ug/mL.

5.3.2.6 Independent Calibration Verification (ICV) Standard – From a source independent of the calibration standards, prepare a standard in methylene chloride containing the compounds listed in Table 3. The final concentration of each compound is 2 ug/mL.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

All semivolatile sample extracts must be analyzed within forty days following the date of extraction.

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY: SW 846 METHOD 8270 – Modified for Selected Ion Monitoring (SIM)

7.0 PROCEDURES

7.1 NAMING AND CODING CONVENTIONS FOR ANALYTICAL STANDARDS – Used in accordance with SOP CA-106 "Standard Preparation and Documentation".

7.2 COMPUTER (DATA SYSTEM) CONVENTIONS -

Conventions for all instruments are as follows:

Sub-Directory for data acquisition and storage: C:\HPCHEM\1\DATA
Tune file: DFTPP.U

Method files: LSPSIMXX.M (all samples and standards)
Where:
XX = the calibration number in chronological order
L = instrument ID (R, U, or G)
DFTPP390.M (DFTPP tuning acquisition)

NOTE: All acquisition parameters must be identical for LSPSIMXX.M and DFTPP2. M.

Data Files: L_ _ _ _.D, where _ _ _ _ is a number in chronological order from 0001 to 9999 and L is the instrument ID (R, U, or G). This file also contains the Quantitation output file.

Data Files for DFTPP: LD_ _ _.D, where _ _ _ is a number in chronological order from 001 to 999 and L is the instrument ID (R, U, or G).

7.3 INSTRUMENT SPECIFIC PROCEDURES

It is the policy of the GC/MS group that all data be acquired in the batch mode. The following items must be checked prior to data acquisition in the batch mode:

- Ensure that the proper sequence and tune files are being used.
- Check the autosampler syringe (Is it clean? Does the plunger move freely? etc.), its alignment and make sure the solvent rinse vial is full. Ensure that the knurled nut holding the top of the syringe plunger is tight.
- Look at the batch to be analyzed and check the following:

  -Make sure that the data files are in numerical order with no duplication and that the method file is the same as that used for ICAL or Continuing Calibration analysis.

-Bottle numbers match with the numbers on the autosampler tray.

After the batch has been deemed free of errors, start the batch by using the "Position and run" command under the SEQUENCE menu in MSTop.

7.4 INSTRUMENT TUNING - Prior to the analysis of any calibration standards, blanks or samples, the GC/MS system must be shown to meet the mass spectral key ion and ion abundance criteria for decafluorotriphenylphosphine (DFTPP) tabulated below. Pentachlorophenol, benzidine and DDT are also present in this standard.

DFTPP Key Ions and Ion Abundance Criteria	
Mass	**Criteria**
51	30.0-80.0 percent of mass 198
68	less than 2.0 percent of mass 69
69	present
70	less than 2.0 percent of mass 69
127	40.0 – 60.0 percent of mass 198
197	less than 1.0 percent of mass 198
198	base peak, 100 percent of mass 198
199	5.0-9.0 percent of mass 198
275	10.0-30.0 percent of mass 198
365	greater than 1.00 percent of mass 198
441	present, but less than mass 443
442	greater than 40.0 percent of mass 198
443	17.0-23.0 percent of mass 442

All ion abundances must be normalized to m/z 198, the nominal base peak.

The following are the GC/MS operating conditions for injection of DFTPP.

GC/MS OPERATING CONDITIONS - DFTPP	
Initial column temperature hold	140°C for 3 minutes
Column temperature program	140-275°C at 15 degrees/minute
Final column temperature hold	275°C
Injection port temperature	280°C
Transfer line/source temperature	285°C
Injector - splitless, valve time	0.18 minutes
EPC	inlet B
Constant flow	ON
Constant flow pressure	10psi
Constant flow temperature	30°C

GC/MS OPERATING CONDITIONS – DFTPP (CONT.)	
Vacuum comp.	ON
Run time	10-12 minutes
Scan start time	5.0 minutes
Sample volume	2.0 uL of 25 ng/uL DFTPP solution
Carrier gas	helium at @ 1.0 mL/minute
Mass range	35 to 500 amu
Number of A/D samples	4
GC Peak threshold	500 counts
Threshold	10 counts

Set up the run on the Enviroquant system using "Edit Sample Log Table". For a more detailed explanation of the Enviroquant software, consult the appropriate manual, Organic Department Manager, or senior chemist within the GC/MS group.

The DFTPP solution must be analyzed once at the beginning of each twelve hour period during which standards and/or samples are analyzed. The 12 hour time period for GC/MS system begins at the moment of injection of the DFTPP analysis. The time period ends after twelve hours has elapsed according to the system clock. The last injection must be accomplished prior to the expiration of 12 hours; conceivably, the run-time of an injection could end after the twelve hours.

When the DFTPP has concluded, the run must be evaluated to determine if sample analysis can proceed. The chromatography and the ion ratios must be examined. The DFTPP run is processed using the current algorithms in the Target software.

If the results indicate the system does not meet acceptance criteria, the GC/MS must be manually tuned. Once the manual tune procedure is completed, DFTPP must be re-injected and reevaluated. If the instrument still does not meet criteria, notify your Department Manager. Under no circumstances should calibration proceed if the instrument DFTPP is not in criteria.

7.5 INSTRUMENT CALIBRATION

7.5.1 Initial Calibration for Method 8270-SIM

Prior to the analysis of samples and required method blanks, and after the instrument DFTPP tuning criteria have been met, the GC/MS system must be calibrated at six different concentrations, typically, 0.20, 0.50, 1.0, 2.0, 5.0 and 8.0 ng/uL. This is done to determine instrument sensitivity and the linearity of GC/MS response for the semivolatile target and surrogate compounds.

Some SIM compounds may need to be calibrated at higher concentrations. A second standard is prepared containing these compounds. The two

standards are combined as in the example below. A 100 uL aliquot of each of the standards above is spiked with 1 uL of internal standards and analyzed.

Example –
For a calibration at the following levels:
Calibration mix A would be prepared containing ALL analytes at 20 ng/ul
Calibration Mix B would be prepared containing PCP, HCB and BEHP at 20 ng/ul.

Final PAH conc. (ng/uL)	Final PCP, HCB, BEHP Conc. (ng/ul)	Cal-Mix A Added (uL)	Cal-Mix B Added (uL)	$MeCl_2$ Added (uL)	Final Volume (uL)
0.20	1.0	10	40	950	1000
0.50	2.0	25	75	900	1000
1.0	3.0	50	100	850	1000
2.0	4.0	100	100	800	1000
5.0	5.0	250	0	750	1000
8.0	8.0	400	0	600	1000

Note: Calibration Mix B only is used to boost the PCP, HCB and BEHP concentrations in Cal. levels 1 through 4.

The GC/MS operating conditions for the calibration standards injections are the same as for the DFTPP with the following exceptions:

GC/MS OPERATING CONDITIONS – CALIBRATION and SAMPLES	
Column temperature program	40°C for 3 min. to 300°C at 10°/min.
Final column temperature hold	300°C
Run time	35 minutes (time may vary dependent upon column length)
Scan start time	2.0-6.0 minutes (time may vary dependent upon column length)
Sample volume	1 uL

The conditions are set up in the method file LSPSIMXX.M

After analysis of the five calibration points, they must be quantitated and evaluated for adherence to QC criteria. Minimum requirements of ID files are the use of specific quantitation ions and quantitating a specific set of targets and surrogates with a set internal standard. Of particular importance when performing SIM analysis are the ion ratios. These requirements are found in Tables 3 and 5.

7.5.2 Initial Calibration Criteria

Relative response factors (RRFs) must be calculated and evaluated for each target compound and surrogate. The RRF is defined as follows:

$$RRF = \frac{Ax}{A_{IS}} \times \frac{C_{IS}}{Cx}$$

where:
- Ax = area of the primary ion for the target compound
- A_{IS} = area of the primary ion for the corresponding istd
- C_{IS} = concentration of the istd (ng/uL)
- C_x = concentration of the target compound

After the calibration points have been quantitated, update the calibration curve points using the Target data processing software to generate the RRF's and %RSD's for all analytes. If information is needed concerning the use of these programs, consult the Organic Department Manager or a senior chemist within the group.

Response factor criteria have been established for the calibration of the semivolatile target and surrogate compounds. These criteria must be met in order for the calibration curve to be considered valid. The percent RSD for each calibration check compound (CCC) must be less than or equal to 30 percent. There are three CCC's: Acenaphthene, Fluoranthene, and Benzo(a)pyrene. There are no criteria for the SPCC compounds. **This is also applicable to clients that request DOD criteria.**

7.5.2.1 Linearity of Target Analytes **(This is also applicable to clients that request DOD criteria.)**

If the RSD of any target analyte is 15% or less, then the response factor is presumed to be constant over the calibration range, and the average response factor may be used for quantitation.

If the RSD of any target analyte exceeds 15%, then a calibration option outlined in section 7.0 of method 8000 will need to be employed.
Option 1 (Section 7.5.2 of method 8000 - Rev. 2, 12/96), is a linear regression of instrument response versus the standard concentration.

The correlation coefficient (r) for each target analyte and surrogate must be greater than or equal to 0.995. Target software calculates the

correlation coefficient squared (r^2). This must be equal to or greater than 0.990.

Option 2 (Section 7.5.3 of method 8000 - Rev. 2, 12/96), is a non-linear calibration model not to exceed a third order polynomial. The lab would use a quadratic model or second order polynomial. The use of a quadratic model requires six calibration points. In order for the quadratic model to be acceptable, the coefficient of determination must be greater than or equal to 0.990.
If time remains in the clock after meeting the initial calibration acceptance criteria, samples may be analyzed. The calibration must be verified each twelve hour time period (time period starts from the moment of the DFTPP injection) for Method 8270-SIM. The SSTD1.0 in the curve may be used as the continuing calibration standard as long as it meets the continuing calibration acceptance criteria. All sample results must be quantitated using the initial calibration response factors.

7.5.2.2 Immediately following calibration an Independent Calibration Verification Standard must be analyzed. For clients requiring DOD criteria, all project analytes must be within +/- 20% of true value.

7.5.3 Continuing Calibration

A check of the calibration curve must be performed once every twelve hours immediately following analysis of the tuning compound DFTPP. This check contains all target compounds and surrogates at a concentration of 1.0 ng/uL.

After quantitation of the 1.0 ng/uL continuing calibration check, response factors must be calculated and compared to the average response factors in the initial calibration. The Target program calculates the calibration check response factors and compares them to the average RFs in the calibration curve by calculating percent differences. The method 8270 CCC's must have a % difference of +/- 20%D in order to be considered in criteria. These conditions must be met before method blank and/or sample analysis can begin. **For clients requiring DOD criteria, all project analytes and surrogates must be within +/- 20%.**

If the continuing calibration check does not meet criteria, corrective action must be taken. Depending on the situation, corrective action may be as follows:

- Re-analyze the 1.0 ng/uL continuing calibration check.

- Change the septum; clean the injection port; install a clean, silanized quartz liner; cut off a small portion (1" to 3") of the front end of the capillary column. This is usually performed when chromatography is poor. Record any of these actions in the appropriate instrument maintenance logbook.

- Analyze a new initial calibration curve.

The last option, the generation of a new initial calibration curve, is usually chosen when percent difference are >30%. In these instances, there is little or no chance of a continuing calibration reanalysis meeting criteria. If there is any doubt concerning which corrective action to undertake, consult the Organic Department Manager or a senior chemist within the group.

If the continuing calibration does meet the criteria specified above then analysis may proceed using initial calibration response factors.

7.5.4. Retention Time Windows

Retention time windows are set at the midpoint standard of the calibration curve, following every ICAL. When a CV is analyzed (and not an ICAL), the retention time windows of the daily CV must be within 30 seconds of the midpoint calibration standard of the most recent ICAL. The samples analyzed following the daily CV must have retention times within 30 seconds of those for the daily CV. Each successive daily CV must be compared to the most recent ICAL midpoint standard.

7.6 SAMPLE ANALYSIS

Sample extracts may be analyzed only after the GC/MS system has met tuning criteria, initial calibration and continuing calibration requirements. Ensure that the same instrument conditions are being used for tuning, calibration and sample analysis by reviewing the GC parameters using the "Edit entire method" option under the Method menu in MSTOP. Note that you can not edit a method if the instrument is running.

Extracts are stored in the refrigerator in the organics extraction laboratory at 4°C ±2°C. Remove them from the refrigerator and place them in the GC/MS laboratory semivolatile hood when ready for analysis.

Prepare a 1.8 mL clear glass vial (crimp top) with a disposable insert (350 uL). Add 100 uL of sample extract and 1.0 uL of the 80 ng/uL IS stock to the vial and then cap. This gives a 0.8 ng/uL final concentration for the internal standard compounds. The samples are topped with Teflon lined crimp top caps.

7.7 FINAL DATA PACKAGE

7.7.1 Initial Data Review (IDR)

The initial data review is accomplished by the analyst who ran the samples and is a review of sufficient quality and detail to provide a list of samples that need to be reanalyzed or diluted and reanalyzed. The initial data review is performed on the detailed quantitation reports of the analyzed sample. This data review examines criteria that directly impact whether or not the sample needs to be reanalyzed:

- Surrogate Recoveries
- Internal Standard Area Stability
- Method Blank Acceptance
- Chromatography
- Target Compound Detection/Quantitation/Review for false positives
- Laboratory Control Sample Recoveries
- Matrix Spike/Matrix Spike Duplicate Recoveries

The analyst must evaluate all data using the QA Acceptance Criteria table found within this SOP (Table 1). This table gives acceptance criteria and corrective actions for criteria that are not met. In addition to evaluating QC elements, the chromatography and quantitation of target analytes must be reviewed. During this review, the analyst checks the integration of each individual peak. The hardcopy has false positives crossed out so they can be reviewed for appropriateness by the Organic Department Manager.

7.7.2 Chromatography

The chromatography should be examined for the presence or absence of any ghost peaks and can also be used as an indication of whether or not matrix interferences might be affecting surrogate recoveries and/or istd area recoveries. Whether or not the chromatography is acceptable is a judgment call on the part of the analyst and should be used in conjunction with other monitored QC (e.g. surrogate recoveries) to determine the necessity of reanalyzing.

Manual integrations are to be performed when chromatographic conditions preclude the computer algorithm from correctly integrating the peak of

concern. In no instance shall a manual integration be performed solely to bring a peak within criteria.

Each peak of concern is examined by the primary analyst to ensure that the peak was integrated properly by the computer algorithm. Should a manual integration be necessary (for instance, due to a split peak, peak tailing, or incomplete resolution of isomeric pairs), an "m" qualifier will automatically be printed on the quantitation report summary.

This manual integration package must then be submitted to the Department Manager or his/her designee, who will review each manual integration.

For specific manual integration procedures, refer to Katahdin SOP QA-812, "Manual Integration", current revision.

7.7.3 Target Compound Detection/Quantitation

The semivolatile ID files have been set up to err on the side of false positives; that is to identify and quantitate peaks as target compounds that may not necessarily be valid hits. It is the responsibility of the GC/MS analyst to use his/her technical judgment to determine if the identification of a target compound is correct or not.

If any target concentration exceeds the upper limit, a dilution must be made and analyzed. The dilution chosen should keep the concentration of the largest target compound hit in the upper half of the initial calibration range. LCS and MS/MSD samples need not be diluted to get spiked analytes within the calibration range.

The requirements for qualitative verification by comparison of mass spectra are as follows:

- All ions present in the standard mass spectra at a relative intensity > 10% must be present in the sample spectrum.
- The relative intensities of primary and secondary ions must agree within ±20% between the standard and sample spectra.
- Ions greater than 10% in the sample spectrum but not present in the standard spectrum must be considered and accounted for by the analyst.

If a compound cannot be verified by all three criteria above, but, in the technical judgment of the mass spectral interpretation specialist, the

identification is correct, then the laboratory shall report that compound on the Form 1 as a valid hit.

The GC/MS laboratory initial data review must be completed within twelve hours of batch completion; in the majority of instances, the initial review should be accomplished at the beginning of a work shift for the previous set of analyses.

7.7.4 Reporting

After the chromatograms have been reviewed and any target analytes have been quantitated using Target, the necessary files are brought into QuickForms. Depending on the QC label requested by the client, a Report of Analysis (ROA) and additional reports, such as LCS forms and chronology forms, are generated. The package is assembled to include the necessary forms and raw data. The data package is reviewed by the primary analyst and then forwarded to the secondary reviewer. The secondary reviewer validates the data and checks the package for any errors. When completed, the package is sent to the department manager for final review. A complete review checklist is provided with each package. The final data package from the Organics department is then processed by the Data Management department.

7.8 INJECTION PORT LINER CLEANING AND SILANIZING PROCEDURE

- Remove the rubber o-ring from the liner and place the liner in a large Erlenmeyer flask.
- In the hood, pour nitric acid into the flask until the liner is covered. Place the flask on a hotplate and boil for 2-3 hours.
- Let cool; drain nitric acid and thoroughly flush the liner with water.
- Bake briefly in the muffle oven until liner is dry and cool to room temperature.
- Place the liner in a beaker, fill with Sylon and let it soak for at least two hours.
- Take out the liner and rinse it thoroughly with toluene.
- Rinse the liner thoroughly with purge and trap grade methanol.
- Bake the liner in the muffle oven for a minimum of three hours.

7.9 Instrument Maintenance

Instrument preventative maintenance is performed on a semi-annual basis by GC/MS chemists. This maintenance includes a thorough inspection and cleaning of all parts, including changing rough and turbopump oils. GC/MS analysts perform other maintenance on an as-needed basis. Typically, routine maintenance involves clipping off the front end of the DB-5MS column, replacing the injection port septum, and installing a freshly silanized quartz liner after sample analysis.

All maintenance must be documented in the instrument-specific maintenance log, whether it is routine or not. The Department Manager must authorize any maintenance over and above a routine source cleaning.

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

Refer to Table 1 and to details in this section for a summary of QC requirements, acceptance criteria, and corrective actions. These criteria are intended to be guidelines for analysts. The criteria does not cover all possible situations. If any of the QC requirements are outside the recovery ranges listed in this section or in Table 1, all associated samples must be evaluated against all the QC. In some cases data may be reported, but may be reanalyzed in other cases. Making new reagents and standards may be necessary if the standardization is suspect. The corrective actions listed in this section and in Table 1 may rely on analyst experience to make sound scientific judgements. These decisions are based on holding time considerations, client and project specific Data Quality Objectives and on review of chromatograms. The supervisor, Operations Manager, and/or Quality Assurance Officer may be consulted to evaluate data. Some samples may not be able to be reanalyzed within hold time. In these cases "qualified" data with narration may be advisable after consultation with the client.

In some cases the standard QC requirements listed in this section and in Table 1 may not be sufficient to meet the Data Quality Objectives of the specific project. Much of the work performed at the lab is analyzed in accordance with specific QC requirements spelled out in a project specific Quality Assurance Project Plan (QAPP) or in a program specific Quality Systems Manual (QSM). The reporting limits, acceptance criteria and/or corrective actions may be different than those specified in this SOP. In these cases the appropriate information will be communicated to the Department Manager and/or senior chemists before initiation of the analyses so that specific product codes can be produced for the project. In addition, the work order notes for each project will describe the specific QAPP or QSM to be followed.

8.1 Method Blank Criteria

A method blank is defined as a volume of a clean reference material (deionized distilled water for water samples, baked organic-free sand for soil/sediment matrices)

that is carried through the entire analytical procedure. One method blank must be extracted with each group of samples of a similar matrix and must be analyzed on the GC/MS system that was used to analyze the samples.
An acceptable method blank must contain less than or equal to the PQL of any target compound. **For clients requiring DOD criteria, no analytes detected at > ½ PQL and > 1/10 the amount measrued in any sample or 1/10 the regulatory limit.**

If the method blank exceeds these contamination levels, the analytical system is considered out of control and corrective action must be taken before sample analysis.

Reanalysis of the blank is the first step of the corrective action; if that does not solve the problem, a Katahdin Corrective Action Report (CAR) will be initiated. Corrective action will be specified after consultation including the Department Manager, Operations Manager, and QA Officer.

8.2 Surrogate Recoveries

The four surrogates (2-Methylnaphthalene-d10, 2,4-Dibromophenol, Fluorene-d10 and Pyrene-d10) must meet the current statistically derived acceptance limits. If statistical limits have not been established then the surrogate recovery must meet the nominal limits of 30-150%. **For clients requiring DOD criteria, use acceptance limits specified by DOD or use in-house limits where none are specified.**

If specifications are not met, the sample (or blank) should be reanalyzed. If specifications are met in the reanalysis, this reanalysis should only be submitted. If surrogate specifications are not met in the sample or method blank reanalysis, a Corrective Action Report (CAR) should be initiated. Corrective action will be specified after consultation including the Department Manager and Operations Manager.

For further information regarding the acceptance of surrogate recoveries, consult the Organic Department Manager.

8.3 Internal Standard Responses

Internal standard responses and retention times (RT) in all samples and blanks must be evaluated as part of the technical data review. The method files have been set up to only detect compounds that fall within a set RT window. For Method 8270-SIM analysis, if the extracted ion current profile (EICP) area for any internal standard changes by more than a factor of two (-50% to +100%) as compared to the daily continuing calibration standard, reanalysis must occur. If the reanalysis meets criteria, only the in-criteria run should be reported. If the reanalysis is still out of criteria, both analyses should be included in the sample package set.
MS/MSD samples that do not meet the EICP area criteria above do not have to be reanalyzed.

8.4 Laboratory Control Sample (LCS)

An LCS must be performed for each group of samples of a similar matrix, for the following, whichever is more frequent:

- Every 20 samples of a similar matrix or similar concentration, or
- Every batch of samples extracted.

Statistical limits are compiled annually for LCS recoveries (archived in QA office). Statistical limits are only calculated when at least 20 usable data points are obtained for any given compound. If insufficient data points are available, nominal limits are set by the section supervisor, Laboratory Operations Manager and Quality Assurance Officer. Refer to Katahdin SOP QA-808, "Generation and Implementation of Statistical QC Limits and/or Control Charts", current revision.

The use of statistical limits versus nominal limits is dependent on the client and project. This information is communicated to the Organic Department Manager through the Katahdin project manager. It is standard practice to use statistical limits for reporting purposes and to evaluate any QC criteria exceedances. However, nominal limits of 30-150% may be used for some projects or states (i.e. South Carolina). **For clients requiring DOD criteria, use acceptance limits specified by DOD or use in-house limits where none are specified.**

The LCS recoveries for all analytes are evaluated. All of the compounds of interest must fall within the established statistical limits with the following sporadic exceedance allowances.

Number of Analytes	Number of Allowable Exceedances
> 90	5
71 – 90	4
51 – 70	3
31 – 50	2
11 – 30	1
<11	0

If less than the number of allowable exceedances fail the statistical limits, no corrective action is needed. If greater than the number of allowable exceedances fail the statistical limits, corrective action may be taken. Corrective actions may vary with each situation. However, in the case where the failures are high and the samples are non-detect for those compounds, then no corrective action is required. Otherwise, corrective action may involve reanalysis or recalibration. The specific corrective actions taken will rely on analyst experience to make sound scientific judgments while

considering client objectives, other quality control indicators and/or the ability to reanalyze a sample within holding time

Please note that for compounds with only nominal limits (i.e. insufficient data points were available to generate statistical limits), no corrective action is required for out-of-criteria recoveries until enough data points are established to generate statistical limits.

8.5 Matrix Spike/Matrix Spike Duplicate (MS/MSD) Criteria

Matrix Spike and Matrix Spike Duplicates must be extracted and analyzed for each group of up to 20 samples of a similar matrix or similar concentration. In the event insufficient sample volume is available an LCS/LCS Duplicate is extracted and analyzed in place of the MS/MSD.

Statistical limits are compiled annually for MS/MSD recoveries for a short list of the spiked compounds (Acenaphthene, Pentachlorophenol and Pyrene). Nominal limits of 30-130% are used for all other compounds. Generally, corrective action is only taken for the short list of the spiked compounds. The specific corrective actions will rely on analyst experience to make sound scientific judgments while considering client objectives, other quality control indicators and/or the ability to reanalyze a sample within holding time. **For clients requiring DOD criteria, use acceptance limits specified by DOD or use in-house limits where none are specified.**

A Corrective Action Report (CAR) must be filled out and filed if any criteria for percent recovery or relative percent difference are not met to document any decisions with reporting data.

9.0 METHOD PERFORMANCE

The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. The MDLs are determined annually per type of instrument and filed with the Organic Department Manager and with the QAO. Refer to the current revision of Katahdin SOP QA-806, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications, for procedures on determining the MDL.

Refer to the current revision of Method 8270 for other method performance parameters and requirements.

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY: SW 846 METHOD 8270 – Modified for Selected Ion Monitoring (SIM)

10.0 APPLICABLE DOCUMENTS/REFERENCES

"Test Methods for the Evaluation of Solid Waste: Physical/Chemical Methods", SW-846, third Edition, Final Update III, December 1996, Method 8270C.

"USEPA Contract Laboratory Program Statement of Work for Organics Analysis," Rev. 02/88.

Code of Federal Regulations (40 CFR), Part 136, Appendix A, Rev. June, 1998.

Katahdin SOP CA-101, Equipment Maintenance

Department of Defence Quality Systems Manual for Environmental Laboratories (DoD QSM), Version 4.1, 04/22/09

The National Environmental Laboratory Accreditation Conference (NELAC) Standards, June 2003

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TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY: SW 846 METHOD 8270 – Modified for Selected Ion Monitoring (SIM)

TABLE 1

QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Check of mass spectral ion intensities using DFTPP	Prior to initial calibration and calibration verification	Refer to the criteria listed in Section 7.4	Retune instrument, and verify
Six-point initial calibration for all analytes	Initial calibration prior to sample analysis	RSD ≤30 for RFs of the CCCs; Average %RSD < 15% for all compounds. Refer to section 7.5.2.1 for more details.	Repeat calibration if criterion is not met
Independent calibration verification	Once after Initial calibration	± 20 % D	1) Reanalyze standard 2) Reprep standard 3) Reprep standard from fresh stock.
Continuing calibration verification	Once per each 12 hours, prior to sample analysis	CCCs ≤ 20%D	Repeat initial calibration and reanalyze all samples analyzed since the last successful calibration verification
ISs	Immediately after or during data acquisition of calibration check standard	Retention time ± 30 seconds; EICP area within -50% to +100% of last calibration verification (12 hours) for each IS	Inspect mass spectrometer or GC for malfunctions; mandatory reanalysis of samples analyzed while system was malfunctioning
Demonstration of ability to generate acceptable accuracy and precision	Once per analyst initially and annually thereafter	All recoveries within method QC acceptance limits.	Recalculate results; locate and fix problem; reextract/reanalyze P&A study for those analytes that did not meet criteria
Method blank	One per prep batch	No analytes detected > PQL	(1) Investigate source of contamination (2) Evaluate the samples and associated QC: i.e.If the blank results are above the PQL, report samples that are <PQL or > 10X the blank result. Reprep a blank and the remaining samples.
LCS for all analytes	One LCS per prep batch	Statistically derived from lab data or nominal limits depending on the project. . See also section 8.4 of this SOP for more information on allowable exceedances.	(1) Evaluate the samples and associated QC: i.e.If an MS/MSD was performed and acceptable, narrate. If an LCS/LCSD was performed and only one was unacceptable, narrate. If the surrogate recoveries in the LCS are low but are acceptable in the blank and samples, narrate. If the LCS rec. is high but the sample results are <PQL, narrate. Otherwise, reprep a blank and the remaining samples.

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY: SW 846 METHOD 8270 – Modified for Selected Ion Monitoring (SIM)

TABLE 1

QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Surrogate spike	Every sample, control, standard, and method blank	Statistically derived limits.	(1) Check chromatogram for interference; if found, flag data (2) If not found, check instrument performance; if problem is found, correct and reanalyze (3) If still out reextract and analyze sample (4) If reanalysis is out, flag data
MS/MSD	One MS/MSD per every 20 samples	Statistically derived from lab data or nominal limits depending on the project. Nominal limits are used as default limits.	(1) Evaluate the samples and associated QC: i.e. If the LCS results are acceptable, narrate. (2) If both the LCS and MS/MSD are unacceptable reprep the samples and QC.
MDL study	Refer to KAS SOP QA-806, "Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications", current revision.		

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY: SW 846 METHOD 8270 – Modified for Selected Ion Monitoring (SIM)

TABLE 2

SUMMARY OF METHOD MODIFICATIONS

TOPIC	KATAHDIN SOP CA-213-07	METHOD 8270, current revision
Apparatus/Materials	none	
Reagents	none	
Sample preservation/ handling	none	
Procedures	none	
QC - Spikes	none	
QC - LCS	none	
QC - Accuracy/Precision	none	
QC - MDL	none	

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY: SW 846 METHOD 8270 – Modified for Selected Ion Monitoring (SIM)

TABLE 3

ANALYTE QUANITIATION AND INTERNAL STANDARDS

Internal Standard: 1,4-dichlorobenzene-d4	Phenanthrene
Target and Surrogates:	Hexachlorobenzene (special)
Hexachloroethane (special)	Anthracene
Internal Standard: Naphthalene-d8	Fluoranthene
Target and Surrogates:	Carbazole (special)
Naphthalene	Di-n-butylphthalate (special)
1-Methylnaphthalene (dredge)	**Internal Standard: Chrysene-d12**
2-Methylnaphthalene	Target and Surrogates:
2-Methylnaphthalene-D10 (surrogate)	Pyrene
Internal Standard: Acenaphthene-d10	Benzo(a)Anthracene
Target and Surrogates:	Chrysene
Biphenyl (dredge)	Bis-(2-ethylhexyl)phthalate (special)
2,6 Dimethylnapthalene (dredge)	Pyrene-d10 (surrogate)
Acenaphthylene	**Internal Standard: Perylene-d12**
Acenaphthene	Target and Surrogates:
Fluorene	Perylene (dredge)
2-Fluorene-d10 (surrogate)	Benzo(b)fluoranthene
2,4-Dibromophenol (surrogate)	Benzo(k)fluoranthene
2-Chloronaphthalene (special)	Benzo(e)pyrene (dredge)
Internal Standard: Phenanthrene-d10	Benzo(a)pyrene
Target and Surrogates:	Indeno(1,2,3-cd)pyrene
Pentachlorophenol (special)	Dibenz(a,h)anthracene
1-Methylphenanthrene (dredge)	Benzo(ghi)perylene

`TABLE 4

PROCEDURE CONDENSATION

Clock

12 hours from injection of 50ng DFTPP.

Calibration Curve Criteria

<30% RSD for CCCS
<15% RSD average for all analytes in calibration standard

Continuing Calibration Check Criteria

<20% D for CCC compounds

Additional QC

LCS every extraction batch
MS/MSD every 20 samples

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY: SW 846 METHOD 8270 – Modified for Selected Ion Monitoring (SIM)

TABLE 5

SVOA COMPOUNDS AND CHARACERISTIC IONS

COMPOUND	PRIMARY ION	SECONDARY IONS
Naphthalene	128	129,127
2-Methylnaphthalene	142	115
Acenaphthylene	152	151,153
Acenaphthene	153	152,154
Fluorene	166	165,167
Phenanthrene	178	179,176
Anthracene	178	179,176
Fluoranthene	202	200,203
Pyrene	202	200,203
Benzo(a)anthracene	228	226
Chrysene	228	226
Benzo(b)fluoranthene	252	253,125
Benzo(k)fluoranthene	252	253,125
Benzo(a)pyrene	252	253,250
Indeno(1,2,3-cd)pyrene	276	277
Dibenz(ah)anthracene	278	279
Benzo(ghi)perylene	276	277
1-Methyl naphthalene (dredge)	142	115
Biphenyl (dredge)	154	76
2,6-Dimethyl Naphthalene (dredge)	156	141
1-Methyl phenanthrene (dredge)	192	191,193
Benzo (e) pyrene (dredge)	252	125
Perylene (dredge)	252	125
Carbazole	167	166,139
Pentachlorophenol	266	264,268
Hexachlorobenzene	284	282, 286
Bis(2-ethylhexyl)phthalate	149	167
2-Chloronaphthalene	162	127, 164
Di-n-butylphthalate	149	104 150
Hexachloroethane	117	201, 199
2-methylnaphthalene-d10 (surrogate)	152	125
Fluorene-d10 (surrogate)	176	175, 177
Pyrene-d10 (surrogate)	212	210, 213
2,4-Dibromophenol (surrogate)	252	63, 143
1,4-Dichlorobenzene-d4 (istd.)	152	115,150
Naphthalene-d8 (istd.)	136	134,137
Acenaphthene-d10 (istd.)	164	162,160
Phenanthrene-d10 (istd.)	188	189
Chrysene-d12 (istd.)	240	241,236
Perylene-d12 (istd.)	264	260

Primary ions must not be changed except in unusual instances where interference occurs with a co-eluting non-target analyte. In this case, a secondary ion may be used for quantitation with the following rules:

(1) The corresponding standard(s) (initial calibration curve and continuing calibration standard) must be re-quantitated with the secondary ion.

(2) Approval must be obtained from the Organic Department Manager or the laboratory Operations Manager.

The quantitation ion must then be changed back to the one specified in the table above after quantitation of the samples(s).

Secondary ions are recommended only and may be changed depending upon instrument conditions (sensitivity, etc.). However, it is Katahdin policy that a minimum of 2 ions (primary and one secondary) be used for all GC/MS analyses.

TITLE: **ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY: SW 846 METHOD 8270 – Modified for Selected Ion Monitoring (SIM)**

FIGURE 1

EXAMPLE OF RUNLOG LOGBOOK PAGE

KATAHDIN ANALYTICAL SERVICES
GC/MS SVOA INJ LOG INSTRUMENT: 5970-X

DATE OF DFTPP INJECTION: 03/009

JOB	SAMPLE	DATAFILE	DF	ALS #	METHOD	UL INJ	CHEMIST	COMMENTS
	50 ng DFTPP	X0600	1	1	DFTP0390	2.0	JK	OK
	SSTD060X0310	X9437		2	X625A022	1.0		✓
	150	38		3				✓
	100	39		4				✓ Curve OK
	30	40		5				✓
	05	41		6				✓
	3510 MIX 1	42		7				OK
	2	43		8				
	3	44		9				
	4	45		10				
	5	46		11				
	6	47		12				
	7	48		13				
	8	49		14				
	9	50		15				
	BLANK 1	51		16				
	2	52		17				

STANDARD	CODE
DFTPP	50856
CAL. STD.	50829-30 50847-49
IS MIX	AHP0934

REVIEWED AND APPROVED BY:
DATE:

QAMS302

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY: SW 846 METHOD 8270 – Modified for Selected Ion Monitoring (SIM)

FIGURE 2

EXAMPLE OF GC/MS STANDARDS RECEIPT LOGBOOK ENTRY

KATAHDIN ANALYTICAL SERVICES

STOCK STANDARDS RECEIVED

GCMS LABORATORY
REVIEWED BY/DATE:

AMP0946

AccuStandard 125 Market St. • New Haven, CT 06513 • USA Tel. 203-786-5290 • www.accustandard.com

APP-9-176-D-20X
Pentachlorophenol
2.0 mg/mL in CH2Cl2
Lot: B3010100
Exp. Jan 10, 2013

1 mL

FOR LABORATORY USE ONLY
WARNING: This product contains a chemical(s) known to the State of California to cause cancer

STORAGE Ambient

POISON [redacted]

Dual 8/16/06 JCA

AMP0947

AccuStandard 125 Market St. • New Haven, CT 06513 • USA Tel. 203-786-5290 • www.accustandard.com

APP-9-090-50X
4,6-Dinitro-o-cresol
5.0 mg/mL in MeOH
Lot: B1100296
Exp. Aug 16, 2012

1 mL

FOR LABORATORY USE ONLY

STORAGE Ambient

FLAMMABLE [redacted]

AMP0948

AccuStandard 125 Market St. • New Haven, CT 06513 • USA Tel. 203-786-5290 • www.accustandard.com

APP-9-145-50X
p-Nitrophenol
5.0 mg/mL in MeOH
Lot: B5050205
Exp. May 18, 2015

1 mL

FOR LABORATORY USE ONLY

STORAGE Ambient

POISON [redacted]

TITLE: **ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY: SW 846 METHOD 8270 – Modified for Selected Ion Monitoring (SIM)**

FIGURE 3

EXAMPLE OF SVOA STANDARDS PREPARATION LOGBOOK ENTRY

GC/MS SVOA STANDARD PREP LOGBOOK

S0863	8270 Stock (w/o MeOH)	3-15-06	7-7-06	JLK	AMP0844	8270 Acetaphthene	300	2-22-07	4.2ml	150 ng/ml
					AMP0837	↓	350	3-15-07		
					AMP0911	App IX #2	600	3-2-07		
					AMP0910	↓ 1	100	3-9-07		
					AMP0570	↓ 1	200	7-7-06		
					AMP0659	Organophosphorus pest	300	8-14-06		
					AMP0931	Benzoic Acid	↓	3-9-07		
					AMP0907	Hexachlorophene		2-22-07		
					AMP0906	Benzidine		3-9-07		
					AMP0936	3,3'-Dichlorobenzidine	↓	3-14-07		
					AMP0932	8270 Surr	150	3-9-07		
					S0861	DEA	300	3-13-07		
					B43290	$MeCl_2$	550	—		
S0864	8270 level 1	3-15-06	7-7-06	JLK	S0863	8270 Stock	70	7-7-06	1.05ml	10 ng/ml
					B43290	$MeCl_2$	980			
S0865	8270 level 2	3-15-06	7-7-06	JLK	S0863	8270 Stock	150	7-7-06	0.90ml	25 ng/ml
					B43290	$MeCl_2$	750			
S0866	8270 level 3	3-15-06	7-7-06	JLK	S0863	8270 Stock	600	7-7-06	1.8ml	50 ng/ml
					B43290	$MeCl_2$	1200			
S0867	8270 level 4	3-15-06	7-7-06	JLK	S0864	8270 Stock	700	7-7-06	1.05ml	100 ng/ml
					B43290	$MeCl_2$	350			

Reviewed by/Date:

GATMS284

0000057

TITLE: **DETERMINATION OF EXTRACTABLE TOTAL PETROLEUM HYDROCARBONS BY STATE OF CONNECTICUT ETPH METHOD (CT-ETPH)**

Prepared By: _____ Date: 3-12-10

Approved By:

Department Manager: _____ Date: 3-12-10

Operations Manager: Deborah J. Madden Date: 3.12.10

QA Officer: Lesie Dimond Date: 03.12.10

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date

Please acknowledge receipt of this standard operating procedure by signing and dating both of the spaces provided. Return the bottom half of this sheet to the QA Department.

I acknowledge receipt of copy ____ of document **SOP CA-342-00**, titled **DETERMINATION OF EXTRACTABLE TOTAL PETROLEUM HYDROCARBONS BY STATE OF CONNECTICUT ETPH METHOD (CT-ETPH).**

Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy ____ of document **SOP CA-342-00**, titled **DETERMINATION OF EXTRACTABLE TOTAL PETROLEUM HYDROCARBONS BY STATE OF CONNECTICUT ETPH METHOD (CT-ETPH).**

Recipient: _____ Date: _____

TITLE: **DETERMINATION OF EXTRACTABLE TOTAL PETROLEUM HYDROCARBONS BY STATE OF CONNECTICUT ETPH METHOD (CT-ETPH)**

1.0 SCOPE AND APPLICATION

This SOP describes all aspects of the analysis of extracts of solid and aqueous samples for Extractable Total Petroleum Hydrocarbons by the State of Connecticut, department of Health, ETPH Method as performed by Katahdin Analytical Services, Inc. including sample analysis, data review, standard preparation and instrument calibration. These compounds correspond to a hydrocarbon range of C_9-C_{36} inclusive and a boiling point range between approximately 170°C and 430°C.

This method is based on a solvent extraction, Gas Chromatography (GC) procedure. The method is designed to measure mid-range petroleum products such as diesel (#2 fuel oil) or kerosene (#1 fuel oil). Components greater than C_{28} present in products such as motor oils or lubricating oils are detectable under the conditions of the method. Additional analyses may be performed including, but not limited to, analysis of additional reference materials. These additional efforts are not contained within this method.

1.1 Definitions

EXTRACTABLE TOTAL PETROLEUM HYDROCARBONS (TPH): All resolved and unresolved material eluting from n-nonane (n-C_9) through n-hexatriacontane (n-C_{36}), inclusive. Quantitation is based on direct comparison of the area within this range to the total area of the 14 components in the TPH/Diesel Component Standard.

TPH/DIESEL COMPONENT STANDARD: A fourteen-component blend of alkanes. This standard serves as a quantitation standard and a retention time window for TPH/DRO.

INDEPENDANT CALIBRATION VERIFICATION: A standard obtained from a source different from the calibration standards.

DIESEL COMPONENT SPIKE: A method blank sample spiked with the Diesel Component Standard and run through the method (including extraction) with samples as a quality control check. See Section 8.2 & 8.3.

SURROGATE CONTROL SAMPLE (SURROGATE BLANK): A laboratory reagent grade water or soil blank spiked with the surrogate compound and run through the method (including extraction). The surrogate recovery is used as a laboratory control. See Section 8.4.

METHOD DETECTION LIMIT (MDL): Minimum concentration that an analyte can be measured and reported with a 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix

containing the analyte. The MDL is determined using EPA Appendix B to Part 136, CFR 40 Ch. 1(7-1-94) using the Student t Test.

MINIMUM REPORTING LEVEL (MRL): Lab reporting level for a commercial diesel fuel oil mixture must be equal to or greater than the MDL and meet the regulatory requirements for fuel oil. The reporting limits are 75 ug/L for aqueous samples and 5.0 mg/kg for soil samples.

TEMPERATURE BLANK (If required by project): A vial of water supplied by the laboratory, treated in the same manner as sample vials and carried along with samples, to determine if proper cooling of samples has been achieved (less than 6°C). A 40 ml or 60 ml vial will be adequate for this purpose.

KATAHDIN INFORMATION MANAGEMENT SYSTEM (KIMS): A complete multi-user system with the capabilities of integrating laboratory instrumentation, generating laboratory worksheets, providing complete Lab Order status and generating reports. LIMS utilizes these features through a database.

PE NELSON TURBOCHROM: A data acquisition system that is used to collect, chromatographic data. The system can also be used to archive raw data files.

TARGET: A software system that combines full processing, reporting and comprehensive review capabilities, regardless of chromatographic vendor and data type.

TARGET DB: An oracle database used to store and organize all Target data files.

QUICKFORMS: A laboratory reporting software for Target and Target DB. The QuickForms report module for Target is preconfigured with generalized forms and US EPA CLP report forms and disk deliverables, which can be customized.

1.2 Responsibilities

This method is restricted to use by, or under the supervision of analysts experienced in the analysis of ETPH by Method CT-ETPH. Analysts should be skilled in the interpretation of gas chromatograms and their use as a quantitative tool. Each analyst must demonstrate and document their ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, current revision, "Personnel Training & Documentation of Capability".

It is the responsibility of all Katahdin technical personnel involved in analysis of ETPH by Method CT-ETPH to read and understand this SOP, to adhere to the procedures outlined, and to properly document their data in the appropriate lab notebook. Any deviations from the test or irregularities with the samples should also

be recorded in the lab notebook and reported to the Department Manager or designated qualified data reviewer responsible for this data.

It is the responsibility of the Department Manager to oversee that members of their group follow this SOP, to ensure that their work is properly documented and to initiate periodic review of the associated logbooks.

1.3 Safety

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDSs (material safety data sheets) for all the materials used in this procedure.

Each qualified analyst or technician must be familiar with Katahdin Analytical Environmental Health and Safety Manual including the Katahdin Hazardous Waste Plan and must follow appropriate procedures. These include the use of appropriate personal protective equipment (PPE) such as safety glasses, gloves and lab coats when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location of all safety equipment. Each analyst shall receive a safety orientation from their Department Manager, or designee, appropriate for the job functions they will perform.

1.4 Pollution Prevention/Waste Disposal

Whenever possible, laboratory personnel should use pollution prevention techniques to address their waste generation. Refer to the current revision of the Katahdin Hazardous Waste Management Program for further details on pollution prevention techniques.

Wastes generated during the preparation of samples must be disposed of in accordance with the Katahdin Analytical Environmental Health and Safety Manual and SOP SD-903, "Sample Disposal," current revision. Expired standards are lab packed, placed in the Katahdin hazardous waste storage area, and disposed of in accordance with this SOP.

Any Methylene chloride solvent waste generated during the preparation of standards etc. should be disposed of in the Acid/Solvent or "D" waste stream satellite accumulation area nearest the point of generationTPH sample vials are Extract Vials/Mixed Flammables or "P" waste and should be disposed of in the

corresponding satellite waste accumulation area nearest the point of generation. Please refer to the current revision of SOP CA-107 for the location of satellite accumulation areas.

2.0 SUMMARY OF METHOD

This method provides gas chromatographic conditions for the detection of semi-volatile petroleum fractions such as diesel, fuel oil #2 or kerosene. Samples are analyzed utilizing extraction to dissolve the organic constituents. The extract is dried, concentrated and injected into a capillary column gas chromatograph. The gas chromatograph is temperature programmed to facilitate separation of organic compounds. Detection and quantitation is based on FID detector response compared to a diesel component standard using an external standard technique.

This method is suitable for the analysis of waters, soils or wastes.

3.0 INTERFERENCES

3.1 Other organic compounds including chlorinated hydrocarbons, phenols, and phthalate esters are measurable. As defined in the method, the TPH results include these compounds. Spills of known specific constituents should be analyzed and quantified by a method specific for those compounds.

3.2 Method interferences are reduced by washing all glassware with hot soapy water and then rinsing it sequentially with tap water, methanol or acetone, and Methylene chloride. Method /reagent blanks (Surrogate Control Samples) must be analyzed with each batch to demonstrate that the samples are free from method interferences.

3.3 High purity reagents such as Burdick and Jackson GC Methylene chloride or Baker capillary grade Methylene chloride must be used to minimize interferences.

3.4 Contamination by carryover can occur whenever high-level and low-level samples are sequentially analyzed. Whenever an unusually concentrated sample is encountered, it should be followed by analysis of a solvent blank to check for cross-contamination.

3.5 The flame ionization detector (FID) is a non-selective detector. There is a potential for many non-target compounds present in samples to interfere with this analysis. There is also the potential for analytes to be resolved poorly, especially in samples that contain many analytes. The data user should consider this and may wish to alter the target analyte list accordingly.

TITLE: **DETERMINATION OF EXTRACTABLE TOTAL PETROLEUM HYDROCARBONS BY STATE OF CONNECTICUT ETPH METHOD (CT-ETPH)**

4.0 APPARATUS AND MATERIALS

4.1 Gas Chromatograph: Analytical system complete with gas chromatograph and all required accessories, including a detector, column supplies, recorder, gases, and syringes. A capillary split/splitless injector operating in the splitless mode is recommended. A data system capable of determining peak areas by integrating from baseline to baseline is required.

4.2 Column 1: 30 m x 0.32 mm ID Rtx-1, 0.25 micron film thickness (or equivalent). The column must be capable of resolving typical diesel components, and the solvent front from C_9.

4.3 Detector: Flame ionization detector (FID).

4.4 Microsyringes: 1 ul, 5 ul, 10 ul, 25 ul, and 100 ul.

4.5 Disposable pipettes: Pasteur.

4.6 2 ml (and larger) vials with Teflon lined caps for storage of extracts.

5.0 REAGENTS

5.1 Solvent: Methylene chloride: Pesticide grade or equivalent. Stored away from other solvents.

5.2 Stock Standards: Aliphatic Hydrocarbon standard mix from a vendor like UltraScientific at a concentration of 1000 ug/ml (each of the 14 components from C_9 to C_{36}). Surrogate mix containing 1-Chloro-octadecane and o-Terphenyl from a vendor like AccuStandard at a concentration of 2000 ug/ml in acetone.

5.3 Calibration Standards: The Aliphatic Hydrocarbon standards are prepared at the following five different concentrations: 200 ug/ml, 100 ug/ml, 50 ug/ml, 20 ug/ml, and 5 ug/ml (per each component). The commercial diesel fuel oil standard is prepared at the following five different concentrations: 2000 ug/ml, 1000 ug/ml, 500 ug/ml, 200 ug/ml, and 50 ug/ml. The 200 ug/ml (per component) standard is prepared by adding 2.0 ml of the aliphatic hydrocarbon stock standard (1000 ug/ml) and 400 ul of the surrogate mix stock standard at 2000 ug/ml and diluting to 10 ml with Methylene chloride to arrive at a final surrogate concentration of 80 ug/ml. The other four Aliphatic Hydrocarbon calibration standards are prepared accordingly:

5.4

TITLE: **DETERMINATION OF EXTRACTABLE TOTAL PETROLEUM HYDROCARBONS BY STATE OF CONNECTICUT ETPH METHOD (CT-ETPH)**

AMT of Standard	AMT $MeCL_2$	FINAL CONC. (each component)
2500 µl of 200/80 ug/ml std.	2500 µl	100/40 µg/ml
2500 µl of 200/80 ug/ml std.	7500 µl	50/20 µg/ml
1000 µl of 200/80 ug/ml std.	9000 µl	20/8 µg/ml
500 µl of 100/40 ug/ml std.	9500 µl	5/2 µg/ml

5.3.1 Transfer the stock standard solution into a Teflon-sealed screw-cap/crimp cap bottle. Store, with minimal headspace, at 6°C or less and protect from light.

5.3.2 Working standards must be replaced after 6 months, or sooner if comparison with check standards indicates a problem.

5.4 Independent Calibration Verification standard: A standard obtained from a source different from the calibration standards. Prepared by diluting the stock mix of 1000 ug/ml that contains the 14 Aliphatic Hydrocarbon components from C_9 to C_{36} into methylene chloride to give final concentration of 50 ug/ml. Expiration date is 6 months or sooner. Information is documented in the standards prep logbook.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

6.1 Water samples must be extracted within 7 days. The extracts must be refrigerated and analyzed within 40 days of extraction.

6.2 Soil samples must be extracted within 14 days. The extracts must be refrigerated and analyzed within 40 days of extraction.

7.0 PROCEDURES

7.1 Waters are extracted using a separatory funnel or continuous liquid liquid extraction technique. Soils are extracted using a sonication extraction technique. Alternatively, soils may be extracted by a Soxhlet or Sonication extraction technique. Refer to the current revision of Katahdin SOP's CA-547 and CA548 for sample preparation procedures. After the extracts are concentrated, an appropriate volume (usually 1ul) is injected directly into the GC. (Recommend using splitless injection techniques).

NOTE: NaCl may be added to water samples to improve extraction efficiency.

If the sample concentration exceeds the calibration range for TPH an appropriate dilution should be used. An appropriate dilution is one that keeps the response of major constituents (previously saturated peaks) in the linear range of the detector. If

an initial dilution does not accomplish this then an intermediate dilution should be performed.

7.2 Samples are analyzed by GC/FID. Gas Chromatography:

7.2.1 Conditions (For both column 1 and 2): Set column temperature to 60°C for 2 minutes, then 10°C/min. to 300°C and hold for 14 min. Set FID Detector to 310°C and injector to 300°C. Conditions may be altered to improve resolution or recovery of diesel range organics.

7.2.2 Other columns-set GC conditions to meet the criteria in 4.2.

7.3 Retention Time Window and Quantitation

7.3.2 Total Petroleum Hydrocarbons (TPH): All chromatographic material eluting from n-nonane through n-hexatriacontane inclusive. Quantitation is based on direct comparison of the total area within this range including all resolved and unresolved components to the total area of the 14 components in the TPH/Diesel Component Standard. (Using a "baseline to baseline" integration as opposed to a "valley to valley" integration.)

7.3.4 The TPH retention time window is defined as beginning approximately .1 minutes before the retention time of n-nonane and ending .1 minutes after the retention time of n-hexatriacontane in the calibration run.

The retention time windows must be established as described below.

7.3.3.1 Three injections of the TPH component standard mix are made throughout the course of a 72-hour period.

7.3.3.2 The standard deviation of the three retention times for n-decane (C_{10}) and n-hexatriacontane (C_{36}) are calculated.

7.3.3.3 Plus or minus three times the standard deviation of the retention times for each standard is used to define the retention time window.

7.3.3.4 Retention time windows are calculated for each standard on each GC column and whenever a new GC column is installed. The data is kept on file in the laboratory.

7.3.3.5 If the calculated retention time window results in a value of 0.03 minutes or less, the laboratory will apply a nominal window of 0.03 minutes.

7.3.5 The laboratory must determine retention time windows for the first and last standard on each GC column and whenever a new GC column is installed. This data must be retained by the laboratory.

7.3.6 Quantify by summing all resolved and unresolved chromatographic material from n-nonane through n-hexatriacontane, inclusive.

7.4 TPH Calibration

7.4.1 Run the TPH/Diesel Component Standard mix or the commercial diesel fuel at the five concentration levels listed in section 5.7. Each time a new calibration curve is established the curve should be verified with a standard from an independent source.

NOTE: 50 ug/ml in the concentrate equals 50 ug/L in the sample when concentrated to 1.0 ml assuming 100% recovery.

7.4.2 Inject each calibration standard. Tabulate the total peak area for the fourteen components against the mass injected. The results are used to prepare a calibration curve by linear regression.

7.4.3 Immediately following calibration an Independent Calibration Verification Standard must be analyzed

7.4.4 The working calibration curve must be verified at the beginning and end of each 12-hour work shift by the injection of a TPH component calibration verification standard (CV). If the response for the CV varies from the predicted response by more than 30%, the analytical system should be examined to determine the cause and corrective action should be performed and/or a new continuing calibration standard should be prepared and analyzed. If the 30% criteria is still not met, the system must be recalibrated. All samples must be reanalyzed that fall within the standard that exceeded criteria and the last standard that was acceptable. Closing calibration verifications should be analyzed at an alternate level than the opening CV.

A discrimination check standard must be analyzed at the beginning of sample analysis. The %D is calculated using the RF from each hydrocarbon as compared to the average RF for all of the hydrocarbons. They must be within 20%. One hydrocarbon is allowed out if <50%.

7.5 Calculations

7.5.1 TPH Calculation: The concentration of TPH in the sample is determined from a summation of the total peak area of all resolved and unresolved chromatographic material eluting from n-nonane through n-hexatriacontane, inclusive, using the calibration curve. (Using a "baseline to baseline" integration as opposed to a "valley to valley" integration.) Refer to Section 7.3 (Retention Time Windows and Quantification).

7.5.2 The concentration of an analyte is calculated by using the calibrated curve that is prepared in Target. When an analyte is identified, Target displays a concentration when the file is processed through the appropriate calibrated method.

7.5.3 The concentrations from the reports are then incorporated with the extraction data to arrive at a final concentration.
Water: Concentration (ug/L) = (C) (Vt)/(Vs)
Soil/Sediment: Concentration (mg/kg) = (C) (Vt)/(Ws) (D)

where, C = concentration calculated by Target in ug/ml
Vt = Volume of total extract including any instrument dilutions
Vs = Volume of sample extracted
Ws = Weight of sample extracted
D = Decimal total solids

7.5.4 Extraction blank areas cannot be subtracted from the sample areas. Baseline correction is allowed to correct for rises due to temperature programming.

7.5.5 All area detected in the TPH window must be reported as “TPH”. ".

7.6 Data Review

7.6.1 Initial Data Review
The initial data review is accomplished by the analyst who ran the samples. This review is of sufficient quality and detail to provide a list of samples that need to be reanalyzed or diluted and reanalyzed. The initial data review is performed in Target review. This data review examines criteria that directly impact whether or not the sample needs to be reanalyzed and/or extracted. These criteria include:

- QC criteria for method blank, LCS, MS/MSD, and calibration – refer to section 8.0.

- Surrogate recovery
- Chromatography: manual integration.
- Target compound detection: quantitation, false positives.

The requirement of the GC laboratory is that this initial data review be completed no later than the end of the next work day. After the analyst has completed his or her initial data review, the information is then ready to be processed for reporting. Refer to section 7.7.

7.6.2 Surrogate recovery

All recoveries must meet the most recent laboratory established acceptance limits, which are listed on the GC Laboratory Surrogate Acceptance Limit sheet.

The sample is evaluated for recoveries of the surrogate OTP. If the recovery is low and there is no apparent matrix effect, the sample should be reanalyzed. If the reanalysis is still low, re-extract. If the recovery is low and there may be a matrix effect, reanalyze to confirm a matrix effect and narrate. If the surrogate is high and the sample results are less than the PQL, or there is likely a matrix effect, narrate.

7.6.3 Chromatography

The chromatography should be examined for the presence of any non-target peaks, which can be used as an indication of whether or not matrix interference might be influencing surrogate recoveries.

Manual integrations are to be performed when chromatographic conditions preclude the computer algorithm from correctly integrating the peak of concern. In no instance shall a manual integration be performed solely to bring a peak within criteria.

Each peak of concern is examined by the primary analyst to ensure that the peak was integrated properly by the computer algorithm. Any manual integrations that are necessary (for instance, if the sample contains a concentration of TPH which was integrated "valley to valley" instead of a "baseline to baseline"), are performed in Target Review. A "m" qualifier will automatically be printed on the quantitation report summary. For specific procedures on how to manually integrate, refer to Katahdin SOP QA-812, Manual Integration, current revision.

7.6.4 Target Compound Detection

To correctly quantitate the TPH concentration, the area of the surrogate, OTP, is not included in the TPH timed range area. In some instances, OTP may need to be manually integrated in Target Review to correctly quantitate the OTP and TPH concentration.

7.7 Reporting

After the chromatograms have been reviewed and any target analytes have been quantitated using Target, the necessary files are brought into QuickForms. Depending on the QC level requested by the client, a Report of Analysis (ROA) and additional reports, such as LCS forms and chronology forms, are generated. The package is assembled to include the necessary forms and raw data. The data package is reviewed by the primary analyst and then forwarded to the secondary reviewer. The secondary reviewer validates the data and checks the package for any errors. When completed, the package is sent to the department manager for final review. A completed review checklist is provided with each package. The final data package from the Organics department is then processed by the Data Management department.

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

Refer to Table 1 and to details in this section for a summary of QC requirements, acceptance criteria, and corrective actions. These criteria are intended to be guidelines for analysts. The criteria does not cover all possible situations. If any of the QC requirements are outside the recovery ranges listed in this section or in Table 1, all associated samples must be evaluated against all the QC. In some cases data may be reported, but may be reanalyzed in other cases. Making new reagents and standards may be necessary if the standardization is suspect. The corrective actions listed in this section and in Table 1 may rely on analyst experience to make sound scientific judgments. These decisions are based on holding time considerations, client and project specific Data Quality Objectives and on review of chromatograms. The supervisor, Operations Manager, and/or Quality Assurance Officer may be consulted to evaluate data. Some samples may not be able to be reanalyzed within hold time. In these cases "qualified" data with narration may be advisable after consultation with the client.

In some cases the standard QC requirements listed in this section and in Table 1 may not be sufficient to meet the Data Quality Objectives of the specific project. Much of the work performed at the lab is analyzed in accordance with specific QC requirements spelled out in a project specific Quality Assurance Project Plan (QAPP) or in a program specific Quality Systems Manual (QSM). The reporting limits, acceptance criteria and/or corrective actions may be different than those specified in this SOP. In these cases the appropriate information

will be communicated to the Department Manager and/or senior chemists before initiation of the analyses so that specific product codes can be produced for the project. In addition, the work order notes for each project will describe the specific QAPP or QSM to be followed.

8.1 The analyst must make an initial demonstration of the ability to generate acceptable accuracy and precision with this method by successful analysis of the following:

8.1.1 Replicate commercial diesel oil spikes in water: Analysis of at least 4 replicates at a concentration of 100 ug/L (in laboratory reagent grade water) with accuracy of the replicates falling between 60% to 140% of the known concentration. The precision of all replicates should be within 20%.

8.1.2 Replicate commercial diesel oil spikes in soil: Analysis of at least 4 replicates at a concentration of 10 mg/kg with the accuracy of the replicates falling between 60% and 140% of the known concentration. The precision of all the replicates should be within 20%. Soil spikes should be prepared and analyzed as described in section 8.3.

8.2 For every 20 water samples analyzed, the lab must analyze a set of duplicate diesel component spikes in laboratory reagent grade water. The Duplicate spikes must be run through the method in the same manner as samples. The accuracy of the two water spikes should fall within the laboratory established acceptance limits of the known concentration with a relative % difference of 30% or less.

Alternatively duplicate samples and spiked samples can be substituted for the laboratory-spiked duplicates at a frequency of 10%. Care must be taken to ensure that the samples are homogeneous before analyzing duplicates and spikes.

8.3 For every 20 soil samples analyzed, the lab must analyze a set of duplicate Diesel Component Spikes in clean sand or soil. The duplicate spikes must be run through the method in the same manner as samples. The accuracy of the two soil spikes should fall within the laboratory established acceptance limits of the known concentration and the relative % difference should be 50% or less.

Soil spikes are prepared by spiking 30 g of clean soil or sand with the component standard in methylene chloride (use 1 ml of a standard at a concentration of 500 ug/ml [50 ug/ml each component] is recommended). The component standard spike should be added at the same step as the surrogate standard.

Alternatively duplicate samples and spiked samples can be substituted for the laboratory-spiked duplicates at a frequency of 10%. Care must be taken to ensure that the samples are homogeneous before analyzing duplicates and spikes.

8.4 A Surrogate Control Sample (Method Blank) must be extracted and analyzed with each batch of water or soil samples extracted. The amount of material in the blank should not exceed the minimum reporting level.

8.5 Calibration requirements:

8.5.1 When linear regression analysis is used for calculations the correlation coefficient must be at least 0.995. For linear models, Target reports r^2. This is calculated by either calculating r and squaring the result or by calculating the coefficient of determination. For a linear calibration, the equation for either is the same. The value for r^2 must greater than or equal to 0.990.

8.5.2 A Quality Control Check Standard (obtained from a source independent of the calibration standards) should be analyzed concurrent with the calibration standards in order to confirm the validity of the calibration curve. The QC check should fall within 20% of the expected value using the calibration data.

8.5.3 The working calibration curve must be verified at the beginning and end of each 12-hour work shift by the injection of a calibration verification standard (CV). The response for the CV must not vary from the predicted response by more than 30%.

A discrimination check standard be analyzed at the beginning of sample analysis. The %D is calculated using the RF from each hydrocarbon as compared to the average RF for all of the hydrocarbons. They must be within 20%. One hydrocarbon is allowed out if <50%.

8.6 If any of the criteria above are not met, the problem must be corrected before further samples are analyzed. Any samples run between the last QC samples that meet the criteria and those that have fallen out should be rerun. If this is not possible, that data must be flagged.

8.7 Laboratory spiked duplicates prepared by spiking fuel oil into laboratory reagent grade water must be run at a minimum frequency of 5%.

8.8 Alternatively duplicate samples and spiked samples can be substituted for the laboratory-spiked duplicates at a frequency of 10%. Care must be taken to ensure that the samples are homogeneous before analyzing duplicates and spikes.

8.9 Trip blanks, field blanks, field duplicates, matrix spikes and/or temperature blanks may be recommended or required for specific sampling programs.

8.10 Methylene chloride blanks should be run after samples suspected of being highly concentrated to prevent carryover.

8.11 It is recommended that acceptance criteria be established for recoveries of surrogates. Collect recoveries from 30 samples where no interference is suspected and calculate the mean recovery (X) and standard deviation (S). The acceptance limits for samples not exhibiting matrix interference will be X-3S to X+3S. The warning limits will be X-2S to X+2S. Plotting the surrogate recoveries on a control chart will make checking recoveries easier and is highly recommended. Refer to the current revision of Katahdin SOP QA-808, "Generation and Implementation of Statistical QC Limits and/or Control Charts".

In samples containing TPH, the surrogate peak may need to be "skimmed" off of an envelope of unresolved material in order to get an accurate peak area. If surrogate recovery is outside of the established limits, verify calculations, dilutions, and standard solutions. Verify instrument performance. Low recovery may be due to sample matrix. The analysis should be repeated to confirm matrix problems if the surrogate recovery is less than 50% or, if the analysis cannot be repeated, the data should be flagged.

High recoveries may be due to co-eluting matrix interference. Surrogate recoveries may be reported as "masked" in high level samples exhibiting matrix interference. Additionally, the surrogate may be reported as "diluted out" in high level water or soil samples that must be diluted for analysis. These samples do not need to be rerun solely to try to bring surrogate recovery into acceptance limits.

8.12 It is highly recommended that gasoline, diesel (#2 fuel oil), kerosene (#1 fuel oil), motor oil or other petroleum products be run on a periodic basis for identification purposes, at a minimum with each new column, a new initial calibration, and following any significant retention time changes.

9.0 METHOD PERFORMANCE

9.1 Detection limit for waters: The laboratory must be able to achieve a detection limit of 75 ug/L using a commercial diesel fuel oil mixture spiked into laboratory blank water and calculated against the TPH component standard.

9.2 Detection limit for soils: The laboratory must be able to achieve a detection limit of 5.0 mg/kg for a commercial diesel fuel oil mixture spiked into blank sand and calculated against the TPH component standard.

9.3 The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. The MDLs are determined annually per type of instrument and filed with the Organic Department Manager and with the QAO.

TITLE: **DETERMINATION OF EXTRACTABLE TOTAL PETROLEUM HYDROCARBONS BY STATE OF CONNECTICUT ETPH METHOD (CT-ETPH)**

The Practical Quantitation Limit (PQL) concentration for TPH is listed in Figure 3.

10.0 APPLICABLE DOCUMENTS/REFERENCES

ASTM "Standards Methods for Comparison of Waterborne Petroleum Oils by Gas Chromatography," 3328-78.

The National Environmental Laboratory Accreditation Conference (NELAC) Standards, June 2003.

Katahdin SOP CA-101, Equipment Maintance, Current revision.

Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, USEPA SW846, 3rd Edition, Final Updates I, II, IIA, IIB, III, IIIA, IIIB and IV, February 2007, Method 6010C.

Katahdin SOP QA-806, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications, current revision.

Analysis of Extractable Total Petroleum Hydrocarbons (ETPH) Using Methylene Chloride, prepared by Environmental Research Institute, University of Connecticut, March 1999.

LIST OF TABLES AND FIGURES

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Figure 3 PQLs

TITLE: **DETERMINATION OF EXTRACTABLE TOTAL PETROLEUM HYDROCARBONS BY STATE OF CONNECTICUT ETPH METHOD (CT-ETPH)**

TABLE 1

QC REQUIREMENTS

Parameter/ Method	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Method CT-ETPH	Method blank	One per prep batch	No analyte detected >PQL	(1) Investigate source of contamination (2) Evaluate the samples and associated QC: i.e. If the blank results are above the PQL, report sample results which are < PQL or > 10X the blank concentration. Otherwise, reprep a blank and the remaining samples. (3) If reanalysis cannot be performed, data must be qualified and explained in the narrative. Apply B-flag to all results for the specific analyte in all samples in the associated prep batch.
	LCS	One per prep batch	Laboratory established acceptance limits	Evaluate the samples and associated QC: i.e. (1) If an MS/MSD was performed and acceptable, narrate. (2) If an LCS/LCSD was performed and only one of the set was unacceptable, narrate. (3) If the surrogate recoveries in the LCS are low but are acceptable in the blank and samples, narrate. (4) If the LCS recovery is high but the sample results are < PQL, narrate (5) Otherwise, reprep a blank and the remaining samples.
	Initial Calibration	Initial cal prior to sample analysis	Correlation coefficient (r) ≥ 0.995 or r^2 ≥ 0.990. See sect. 8.5.1 for further clarification.	(1) Perform instrument maintenance as needed. (2) Reanalyze and or reprep calibration standards.
	Discrimination Check	At the beginning of each 12-hour work shift	+/- 20 %D, one can be out if <50%	(3) Perform instrument maintenance as needed. (4) Reanalyze CCAL and/or recalibrate.
	ICV-Independent Calibration Verification	Immediately after Calibration	± 20 % D	(1) Reanalyze standard (2) Reprep standard (3) Reprep standard from fresh stock. (4) Repeat ICAL

TITLE: **DETERMINATION OF EXTRACTABLE TOTAL PETROLEUM HYDROCARBONS BY STATE OF CONNECTICUT ETPH METHOD (CT-ETPH)**

TABLE 1 (cont.)

QC REQUIREMENTS

Parameter/ Method	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Method CT-ETPH	CV(At or near the midpoint of the ICAL with the closing CV at an alternate level from the opening CV)	At the beginning of each 12-hour work shift or every 10 samples whichever comes first if an ICAL was previously analyzed	CT-DEP criteria is +/- 30%D	(1) Evaluate the samples: If the %D>+20% and sample results are <PQL, narrate. If %D>±20% and is likely a result of matrix interference, narrate. All samples must be reanalyzed that fall within the standard that exceeded criteria and the last standard that was acceptable. (2) Repeat ICAL
	Matrix Spike/Matrix Spike Duplicate	One for every set of 20 samples if client requested or if sample aliquot is available. LCSD may be required if MS/MSD analysis not performed.	Laboratory established acceptance limits RPD< 30 % for waters and < 50 % for solids	(1) (1) Evaluate the samples and associated QC: i.e. If the LCS results are acceptable, narrate. (2) If both the LCS and MS/MSD are unacceptable, reprep the samples and QC.
	Sample Duplicate (If required in lieu of MSD)	One sample duplicate per twenty samples	RPD ≤20	(1) Evaluate data for matrix interference homogeneity of sample.
	Demonstration of analyst proficiency; accuracy and precision	Minimum frequency, 1 time per analyst initially and annually there after.	Must pass all applicable QC for method	Repeat analysis until able to perform passing QC; document successful performance in personal training file
	MDL study	Refer to KAS SOP QA-806, "Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications", current revision.		

TITLE: **DETERMINATION OF EXTRACTABLE TOTAL PETROLEUM HYDROCARBONS BY STATE OF CONNECTICUT ETPH METHOD (CT-ETPH)**

TABLE 2

SUMMARY OF METHOD MODIFICATIONS

TOPIC	KATAHDIN SOP CA-342-00	CT-ETPH
Apparatus/Materials		
Reagents		
Sample preservation/ handling		
Procedure	8 7.3.4 The TPH retention time window is defined as beginning approximately .1 minutes before the retention time of n-nonane and ending .1 minutes after the retention time of n-hexatriacontane in the calibration run.	7.7.1 . . . starts at the beginning of the n-C9 peak and terminates at the end of the n-C36 peak.
QC - Spikes		
QC – LCS		
QC - Accuracy/Precision		
QC – MDL		

TITLE: **DETERMINATION OF EXTRACTABLE TOTAL PETROLEUM HYDROCARBONS BY STATE OF CONNECTICUT ETPH METHOD (CT-ETPH)**

FIGURE 1

EXAMPLE OF RUNLOG PAGE

KATAHDIN ANALYTICAL SERVICES, INC. - GC LABORATORY RUN LOG

Instrument GC010

Methods – **Circle Applicable Methods**

Amount Injected: 1 µl

DRO/TPH – **8015Mod.** / **MDEP 4.1.25** / **8100Mod.**

Date	Init	Result File	Sample #	Accept Y/N	Method	Column	Comments
02-11-04	SDW	AUB1038	MeCl2 lot#Y52E36	Y	DRO007A	216	
		39	WG5812-1 3540				
		40	-2				
		41	-3				
		42	WG5974-1	Y			
		43	-2	Y			
		44	-3				OTP high
		45	WG5967-1 3520	Y			
		46	-2				
		47	-3				
		48	WU0256-2 3540	Y			
		49	-1	N			Rerun 1:10
		50	MeCl2	N			
02-12-04		51	FD # 2	Y			
		52	Thermal Bleed				
		53	PHC 50 µg/mL H1241				
		54	WU0225-3DL	N			1:10 100 µL/1mL Rerun 1:50
		55	WU0333-2				Rerun 1:20
		56	-1 3520				Rerun 1:30
		57	WU0358-2 3520				Rerun 1:3
		58	-3DL	Y			1:10 100 µL/1mL
		59	-4DL	N			1:10 Rerun 1:1
		60	WU0333-3DL				1:10 Rerun 1:200
		61	-4DL				1:10 Rerun 1:200
		62	MeCl2				
		63	FD # 2 H1221	Y			
		64	Thermal Bleed				
		65	PHC 50 µg/mL H1241				

REVIEWED BY DL ON 5/5/04

TITLE: **DETERMINATION OF EXTRACTABLE TOTAL PETROLEUM HYDROCARBONS BY STATE OF CONNECTICUT ETPH METHOD (CT-ETPH)**

FIGURE 2

EXAMPLE OF REVIEW CHECKLIST

Verbal Due Date _____ **Due Date**_____

Client:		Primary	Secondary
Method:		Date:	Date:
SDG No:	Level:	Initials:	Initials:
KAS No:			Approved : ☐ Yes

PRIMARY REVIEW CHECKLIST

Highlight Method / project specific information. _____

All needed forms are present . _____

Correct Work Order Number or SDG name (all forms). _____

Correct project name and spelling (all forms). _____

Correct file numbers (all forms). _____

Analysis Date Correct. _____

Extraction Method & Analysis Method Correct. _____

Product list compared to ROAs (compounds & PQLs). _____

Chromatogram reviewed for unlabeled peaks (check product list). _____

Flagging of all ROAs correct (Florida Flagging ☐). _____

All tunes included (level IV) . _____

All log book pages included (Soil weights,TCLP & SPLP). _____

Verify quant results for CLP. _____

Update sample history files. _____

Sign & Date Manual integration **(CLP only).** _____

Sample I.D's Truncated (NARRATE). YES ☐ Please list KAS # below :

First correction ☐ → Review and replace appropriate SDS Forms ☐.

Second correction ☐ → Review and replace appropriate SDS Forms ☐.

TITLE: **DETERMINATION OF EXTRACTABLE TOTAL PETROLEUM HYDROCARBONS BY STATE OF CONNECTICUT ETPH METHOD (CT-ETPH)**

FIGURE 3

PQLS

Analyte	Water ug/L	Soil mg/Kg
ETPH	75	5.0

TITLE: PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT EXTRACTABLE SEMI-VOLATILES ANALYSIS

Prepared By: Mike Thomas Date: 09/96

Approved By:

Group Supervisor: Michael F. Thomas Date: 11/15/00

Operations Manager: J C Burton Date: 10/25/00

QA Officer: Deborah J. Nadeau Date: 10·24·00

General Manager: Dennis P. Lufgan Date: 11/16/00

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01	Minor changes throughout. Clarifications to procedure section.	DN	10·24·00	10/24/00
02	Addition of compounds to Figure 2.	DN	3·28·02	3·28·02
03	Definitions added to section 1.1. Wording was added or changed to clarify sections 4, 5, 6, 7, 8 + 9. Minor changes throughout. New figures.	HRC	11.08.04	11.08.04
04	Updated Sect. 5.0 with current spike solutions prep. Removed section on medium level soil extraction. Replaced figure 3 and 4 with current LCS/MS spike components. Minor corrections to sect. 1.3, 4.24, 6.0 and 7.12. Updated logbook	LAD	04/06	04/06
05	Many changes made throughout, including but not limited to, waste information, updated spikes and surrogates, added SIM LCS/D and MS/D information, updated Table 1. Please refer to the QAM/SOP change form filed w/ SOP in QA for a detailed list of changes.	LAD	09/07	09/07

TITLE: PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT EXTRACTABLE SEMI-VOLATILES ANALYSIS

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
06	Updated logbook example. Added adipate compounds to fig. 2. Added necessity of recording lot numbers of consumables in logbook. Added to record the temperature of the nitrogen evaporation water bath.	LAD	07/08	07/08
07	Added requirement to add spike before Na_2SO_4. Changed N_2 water bath temperature from <39°C to <30°C. Removed respirator reference. Added KAEHS manual. Added KAS SOP CA-1008 reference for additional subsampling information.	LAD	02/09	02/09

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KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

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TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT EXTRACTABLE SEMI-VOLATILES ANALYSIS**

1.0 SCOPE AND APPLICATION

The purpose of this SOP is to describe the procedures and requirements for the preparation of solid samples for analysis of extractable semivolatile organic compounds. This SOP is specifically applicable to EPA Method 3550B in accordance with SW-846 Method 8270, current revision.

1.1 Definitions

METHOD BLANK (laboratory reagent blank): An artificial sample designed to determine if method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus. For aqueous samples, reagent water is used as a blank matrix; for soil samples, baked organic-free sand is used as a blank matrix. The blank is taken through the appropriate steps of the process.

LABORATORY CONTROL SAMPLE (LCS): A blank that has been spiked with the analyte(s) from an independent source, and is analyzed exactly like a sample. Its purpose is to determine whether the methodology is in control, and whether the laboratory is capable of making accurate and precise measurements. The matrix used should be phase matched with the samples and well characterized.

MATRIX SPIKE/MATRIX SPIKE DUPLICATE (MS/MSD): Predetermined quantities of stock solutions of certain analytes are added to a sample matrix prior to sample extraction and analysis. Samples are split into duplicates, spiked and analyzed. Percent recoveries are calculated for each of the analytes detected. The relative percent difference between the samples is calculated and used to assess analytical precision.

SURROGATES: Organic compounds which are similar to analytes of interest in chemical composition, extraction and chromatography, but which are not normally found in environmental samples. These compounds are spiked into all blanks, standards, samples and spiked samples prior to analysis. Percent recoveries are calculated for each surrogate.

1.2 Responsibilities

This method is restricted to use by, or under the supervision of analysts experienced in the extraction of samples for semivolatile analysis. Each analyst must demonstrate and document their ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, current revision, "Personnel Training".

It is the responsibility of all Katahdin technical personnel involved in the extraction of samples for semivolatile analysis to read and understand this SOP, adhere to the procedures outlined, and to properly document their data in the appropriate lab

notebook. Any deviations from the test or irregularities with the samples should also be recorded in the lab notebook and reported to the Department Manager or designated qualified data reviewer responsible for this data.

It is the responsibility of the Department Manager to oversee that members of their group follow this SOP, to ensure that their work is properly documented and to indicate periodic review of the associated logbooks.

1.3 Safety

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDSs for all the materials used in this procedure.

Each qualified analyst or technician must be familiar with Katahdin Analytical Environmental Health and Safety Manual including the Katahdin Hazardous Waste Plan and must follow appropriate procedures. These include the use of appropriate personal protective equipment (PPE) such as safety glasses, gloves and lab coats when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location of all safety equipment. Each analyst shall receive a safety orientation from their Department Manager, or designee, appropriate for the job functions they will perform.

1.4 Waste Disposal

Wastes generated during the preparation of samples must be disposed of in accordance with the procedures described in the current revision of the Katahdin Analytical Environmental Health and Safety Manual and SOP SD-903, "Sample Disposal," current revision. Expired standards are lab packed, placed in the Katahdin hazardous waste storage area, and disposed of in accordance with this SOP.

Any methylene chloride solvent waste generated during the rinsing of glassware etc. should be disposed of in the "D" waste stream satellite accumulation area nearest the point of generation. Acetone and methanol are considered flammable waste, and should be disposed of in the "O" waste stream satellite accumulation area nearest the point of generation. Post-extraction soil samples and sodium sulfate waste should be disposed of in the soil with organics "I" waste stream satellite accumulation area nearest the point of generation. Please refer to the

current revision of SOP CA-107 for the location of satellite waste accumulation areas.

2.0 SUMMARY OF METHOD

A 30 gram portion of sediment/soil is mixed with anhydrous powdered sodium sulfate and extracted with 1:1 methylene chloride/acetone (v/v) using an ultrasonic probe. The methylene chloride extract is dried and concentrated to a volume of 1.0 mL.

3.0 INTERFERENCES

Contaminants in solvents, reagents, glassware, and other sample processing hardware may cause method interferences such as discrete artifacts and/or elevated baselines in the total ion current profiles (TICPs). All of these materials routinely must be demonstrated to be free from interferences under the conditions of the analysis by running laboratory method blanks. Interferences caused by phthalate esters can pose a major problem in semivolatile organics analysis because many phthalates are also target analytes. Common flexible plastics contain varying amounts of phthalates that are easily extracted during laboratory operations, so cross-contamination of glassware frequently occurs when plastics are handled. Interferences from phthalates can best be minimized by avoiding the use of such plastics in the laboratory.

At no time may gloves that have not been tested for phthalates or gloves known to contain phthalates be used or stored in the organic extraction lab. Additionally, whenever possible plastic items in this lab must be replaced with metal or Teflon or other non-phthalate plastic substitute.

Special care should be taken to ensure clean glassware and apparatus are used, pre-rinsed with the appropriate solvent prior to use. Solvents should be analyzed prior to use to demonstrate that each lot is free of contaminants that may interfere with the analysis. Interferences coextracted from the samples will vary considerably from source to source. If analysis of an extracted sample is prevented due to inteferences, further cleanup of the sample extract may be needed to minimize interferences.

4.0 APPARATUS AND MATERIALS

Prior to use, all glassware must be rinsed three times with methylene chloride. Brand names and catalog numbers are included below for illustration purposes only.

4.1 Syringe - gas tight, 1.0 mL, solvent rinsed between each use.

4.2 Sonicator ultrasonic processor XL – Misonix (or equivalent) equipped with dual titanium 3/4" horn extenders for extracting two samples at a time.

4.3 Powder funnels, 100 mm diameter, 35 mm stem

4.4 Kuderna-Danish (KD) apparatus - Concentrator tube - 10 mL
Evaporative flask - 500 mL
Snyder column - 3-ball macro

4.5 Filter paper, 7.0 cm, Whatman #4

4.6 Vacuum filtration flask - 500 mL Erlenmeyer

4.7 Buchner funnel, porcelain, Coors® with 85 mm plate diameter (or equivalent)

4.8 Beakers - 400 mL

4.9 Boiling chips - approximately 12 mesh, silicon carbide (carborundum or equivalent). Soxhlet extract overnight in methylene chloride.

4.10 Water bath - eight position concentric ring bath, or equivalent, equipped with a calibrated thermometer. The bath should be used in a hood.

4.11 Balance - capable of accurately weighing ± 0.1 g.

4.12 Vials and caps – 1.8 mL with PTFE/silicone septa and 12 mL with Teflon-lined caps for extracts designated for GPC cleanup.

4.13 Filter paper, 18.5 cm, Fisherbrand or Whatman 114V (or equivalent)

4.14 Pasteur pipets - disposable, 5 ¾ ".

4.15 Nitrogen evaporation apparatus.

4.16 Muffle oven – capable of maintaining 400 °C for baking glass wool and organic-free sand.

5.0 REAGENTS

5.1 Sodium Sulfate - anhydrous powdered and granular crystals, reagent grade, certified by the manufacturer/vendor as purified heating to 400°C prior to receipt by the laboratory. Solvent rinse immediately prior to use by rinsing three times with pesticide

grade methylene chloride. (Jost Chemical anhydrous powder, catalog #2797 or equivalent, and Jost Chemical granular crystals, catalog #2796 or equivalent).

5.2 Methylene chloride, methanol, and acetone - pesticide residue analysis grade or equivalent. Methylene chloride and acetone are evaluated by lot prior to use by concentration of approximately 400 mL to 1.0 mL followed by GC/MS analysis. The lot numbers of all solvents used during an extraction must be recorded in the extraction logbook.

5.3 Organic-free sand, purified by baking at 400 °C. Method blanks serve as checks on the baked sand.

5.4 Base/Neutral and Acid (SVOA) Surrogate Spiking Solution - Surrogate standards are added to all samples and calibration solutions. Prepare a surrogate standard spiking solution that contains the following compounds at the indicated concentrations in acetone.

Compound	Conc.
phenol-$_{d6}$	100 ug/mL
2,4,6-tribromophenol	100 ug/mL
2-fluorophenol	100 ug/mL
nitrobenzene-$_{d5}$	50 ug/mL
p-terphenyl-$_{d14}$	50 ug/mL
2-fluorobiphenyl	50 ug/mL

Store the spiking solution at -10°C to -20°C in Teflon-sealed containers. These solutions must be replaced after six months, or sooner if comparison with quality control check samples indicates a problem. If reanalysis of a method blank still indicates surrogates out of criteria, a new surrogate solution must be used.

5.5 SIM Surrogate Spiking Solution- Surrogate Standards are added to all samples and calibration solutions. Prepare a surrogate solution that contains the following compounds at a concentration of 2 ug/mL in acetone.

Compound	Conc. ug/mL
Fluorene-d10	2.0 ug/mL
2-Methylnaphthalene-d10	2.0 ug/mL
Pyrene-d10.	2.0 ug/mL
2,4-Dibromophenol	2.0 ug/mL

Store the spiking solution at -10°C to -20°C in Teflon-sealed containers. These solutions must be replaced after six months, or sooner if comparison with quality control check samples indicates a problem. If reanalysis of a method blank still indicates surrogates out of criteria, a new surrogate solution must be used.

5.6 Base/Neutral and Acid (SVOA) Matrix Spike/Lab Control Sample Spiking Solution - Prepare a spiking solution in methanol that contains the compounds listed in Figure 2 at a concentration of 50 ug/mL for base/neutrals and 100 ug/mL for acids. Store the spiking solution at -10°C to -20°C in Teflon-sealed containers. These solutions must be replaced after six months, or sooner if comparison with quality control check samples indicates a problem.

5.7 Base/Neutral and Acid (SVOA APPENDIX IX) Matrix Spike/Lab Control Sample Spiking Solution. Prepare a spiking solution in methanol that contains the compounds listed in Figure 3 at a concentration of 100 µg/mL for each compound. Store the spiking solution at -10°C to -20°C in Teflon-sealed containers. These solutions must be replaced after six months, or sooner if comparison with quality control check samples indicates a problem

5.8 Base/Neutral (SIM) Matrix Spike/ Lab Control Sample Spike Solution for SIM-SVOA. Prepare a spiking solution in methanol that contains the compounds listed in Figure 2 at a concentration of 2 ug/mL for base/neutral. Take out 1.0 mL of Base/Neutral and Acid Matrix Spike/Lab Control Spiking Solution for SVOA and dilute it to 25.0 mL in methanol. Store the solution Spiking at -10°C to -20°C in Teflon-sealed containers. These solutions must be replaced after six months, or sooner if comparison with quality control check samples indicates a problem.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

Sediment/soil samples must be collected in a soil jar and must be maintained at 4°C (±2°C).

Holding time for extraction of sediment/soil samples for Method 3550 is 14 days from date of sample collection, although the analyst should be aware that actual holding times employed may be project/program specific.

Store all extracts at 4°C (±2°C) in the dark in labeled Teflon-sealed containers. See SOP SD-902, "Sample Receipt and Internal Control," current revision, for storage areas and temperature maintenance procedures.

7.0 PROCEDURES

Some solid samples may need to be cleaned up to reduce matrix interferences. The cleanup procedure employed is gel permeation chromatography (GPC). The organic department manager should be consulted to determine if a particular sample should be

subjected to further cleanup procedures; the decision should consider sample history, sample appearance, and project/client needs.

Sign chain-of-custody when removing and replacing samples in storage locations, and fill out the sample preparation/extraction log with the necessary information before starting the extraction. Prerinse all glassware three times with methylene chloride.

7.1 Decant and discard any water layer on a sediment sample. Mix with a stainless steel spatula to ensure homogeneity of the sample. If the sample container is full to the extent that stirring the sample is impractical, try to remove the "best representative" aliquot from the jar based on color, particle size, moisture, etc. Remove any foreign objects such as sticks, leaves, and rocks, and note actions taken in the appropriate extraction logbook. Please refer to the current revision of Katahdin Analytical Services SOP CA-108, "Basic Laboratory Technique ", for more detailed guidance on subsampling to ensure reproducibility.

7.2 The following steps should be performed rapidly to avoid loss of the more volatile extractable. Weigh out a 30.0 ± 0.05 g portion of sample into a labeled 400-mL beaker. Record sample weight to the nearest 0.1 g in appropriate extraction logbook. Refer to Add between 30 g and 60 g of anhydrous powdered sodium sulfate as required for producing a "free-flowing" mixture. The amount of sodium sulfate added will depend upon the moisture content of the sample (e.g., low moisture content will require less sodium sulfate). Mix well with a spatula. Keep the spatula in the sample beaker and cover the beaker with aluminum foil.

7.3 A method blank must be prepared with each extraction batch, not to exceed 20 client samples. To prepare a method blank, weigh out one 30.0 ± 0.05 g portion of purified sand in a labeled 400 mL beaker. Refer to sections 7.7 and 7.7 for spike and surrogate addition instructions. Add 60 g sodium sulfate and mix well. Although a "free-flowing" mixture can be achieved with less than 60 g sodium sulfate, the method blank must contain 60 g in order to evaluate the sodium sulfate as a potential source of contamination.

7.4 A laboratory control sample (LCS) must be prepared with each extraction batch, not to exceed 20 client samples. To prepare LCS, weigh out one 30.0 ± 0.05 g portion of purified sand in a labeled 400 mL beaker. Refer to sections 7.7 and 7.7 for spike and surrogate addition instructions. Add 30 g sodium sulfate and mix well. If combined SIM-SVOA analysis is requested, a separate LCS must be prepared for each analysis. If an MS/MSD pair is not extracted on a particular day, an LCS/LCSD pair may be required in order to meet client-specific or program-specific requirements. This information will be disseminated from the project manager or Department Manager.

7.5 A matrix spike/matrix spike duplicate (MS/MSD) set must be prepared for every 20 samples. To prepare MS/MSD, weigh out 30.0 ± 0.05 g portions of the sample designated for MS/MSD into each of two labeled 400 mL beakers. Record sample weights to nearest 0.1 g in appropriate extraction logbook. Refer to sections 7.7 and 7.7 for spike and surrogate addition instructions. Add 30 - 60 g sodium sulfate to each to produce a free-flowing mixture, and mix well. If combined SIM-SVOA analysis is requested, a separate MS/MSD must be prepared for each analysis.

7.6 Record all weights to one decimal place in the extraction logbook.

7.7 To all samples, method blank, LCS/LCSD, and MS/MSD add 1.0 mL of the appropriate base/neutral and acid surrogate spiking solution listed below using the pre-rinsed 1.0 mL gas tight syringe. The surrogate spike should be added **prior** to the addition of the sodium sulfate. Record surrogate spike volume and identification code in extraction logbook. Thoroughly rinse syringe with solvent prior to using it for another spiking solution.

7.7.1 If the request is for SVOA or SVOA Appendix IX, use the SVOA surrogate solution (sect. 5.4).

7.7.2 If the request is for SIM, use the SIM surrogate solution (sect. 5.5).

7.7.3 If the request is for SIM-SVOA, use both the SIM and SVOA surrogate solutions. NOTE: Separate LCS/LCSD and/or MS/MSD are needed for each analysis and should be spiked with the appropriate surrogate.

7.8 To the LCS/LCSD and the MS/MSD add 1.0 mL of the appropriate base/neutral and acid (SVOA) matrix spike/LCS spiking solution listed below using a 1.0 mL gas tight syringe. The LCS/MS spike should be added **prior** to the addition of the sodium sulfate. Record the matrix spike/LCS spiking solution volume and identification code in extraction logbook. Thoroughly rinse the syringe with solvent when spiking is completed.

7.8.1 If the request is for SVOA, add 1 mL of SVOA Spiking Solution (sect 5.6).

7.8.2 If the request is for SIM, add 1 mL SIM Spiking solution (sect 5.8).

7.8.3 If the request is for SVOA and SIM, add 1mL of SVOA Spiking Solution and 1 mL SIM Spiking solution (sect 5.6 and 5.8).

7.8.4 If the request is for SVOA Appendix IX, add 1mL of SVOA Spiking Solution and 1 mL of SVOA Appendix IX Spiking solution (sect 5.6 and 5.7).

7.9 To assure optimum operation and maximum energy output, the sonicators must be tuned daily prior to extracting samples. The following tuning procedure must be performed with the sonicator probes vibrating in air.

7.9.1 Turn OUTPUT CONTROL knob counter-clockwise to zero. This automatically switches the duty cycle to continuous mode.

7.9.2 Press and hold down the power switch to on.

7.9.3 Press and hold down the TUNE switch. Check if the counter is less or equal to 20%; otherwise, rotate the Tuning Knob (tuning button) clockwise until a reading of 20%or less is obtained.

7.9.4 Release the TUNE switch.

7.9.5 Turn OUTPUT CONTROL KNOB counter-clockwise to 50 and the power switch off.

7.9.6 Confirm that the sonicators were tuned by recording the date and/or percent in the extractions logbook.

7.10 Prior to extracting any samples, ensure that the sonicator probes are decontaminated by rinsing three times with a methylene chloride wash bottle. Collect the waste in a waste beaker. It may sometimes be necessary to wipe the upper part of each probe with a methylene chloride dampened KimWipe. Repeat this decontamination step between each sample on each probe.

7.11 To the mixed and spiked blank and LCS, add 100 mL of the 1:1 methylene chloride/acetone (V/V) solution and proceed with steps 7.11 through 7.14. Record the lot numbers of the solvents in the extraction logbook.

7.12 It may be necessary at this time to stir the sample/sodium sulfate mixture with the spatula to loosen up the mixture prior to extracting. Rinse the spatula with methylene chloride and collect the rinsing into a correspondent beaker. Position the beaker in the ultrasonic cell disruptor so that the bottom surface of the tip of the 3/4 inch disruptor horn is about halfway below the surface of the solvent and above the sediment layer.

7.13 Sonicate for 3 minutes with the output control knob set at 10, and mode switch on "pulsed" and % duty cycle knob set at 50%. While the mixture is sonicating, one should be able to see all, or most of the material, moving in the beaker under the influence of the energized probes. If not, stir the mixture again.

7.14 Prepare a filter flask fitted with a Buchner funnel. The Buchner funnel should contain a 7.0 cm Whatman #4 filter. Prerinse the flask, funnel and filter with

methylene chloride and discard rinsings into solvent waste container. Decant extract into the filter flask and Buchner funnel. A vacuum pump may be used to facilitate filtration or the extract may be gravity filtered. The lot number of the filter paper must be written ti the extraction logbook.

7.15 Repeat the extraction two more times (sec 7.11 – 7.14) using 100 mL portions of 1:1 methylene chloride: acetone. Before each extraction, make certain that the sodium sulfate is still free-flowing and not a consolidated mass. As required, break up large lumps with the spatula. Decant the extraction solvent into the Buchner funnel after each sonication. On the final sonication, pour the entire sample contents into the Buchner funnel and rinse thoroughly with methylene chloride to complete the quantitative transfer of the extract. Use the vacuum pump to pull all the extract into the flask

CONCENTRATION OF LOW LEVEL EXTRACTS

7.16 Rinse the K-D glassware (flask, concentration tube, and Snyder column, including the ground glass joints on the flask and columns) three times with methylene chloride before assembling. Add two boiling chips to the K-D. Insert fluted 18.5 cm filter papers into short stem powder funnels and add ~ 2 inches of sodium sulfate crystals. Rinse the sodium sulfate in the assembled funnels with ~20-30 mLs of methylene chloride. Place the assembled K-D's under the funnels. The lot number of the filter paper must be written ti the extraction logbook.

7.17 Transfer the extracts to the K-D concentrator setups through the sodium sulfate in the funnels. This is the drying step, which is required to remove residual water from the extracts. Any large water layers must be removed by other means, prior to pouring through the sodium sulfate. After pouring all of the extract volume through the sodium sulfate, rinse the extract flask three times with ~ 2 – 3 mls of methylene chloride. Add the rinsings through the sodium sulfate to complete a quantitative transfer. Rinse the sodium sulfate with ~ 15 mls of methylene chloride and allow to drain.

7.18 If samples are to be GPC'd, refer to the current revision of Katahdin SOP CA-513, Extract Cleanup Using Gel Permeation Chromatography, for appropriate concentrating procedures.

7.19 If samples are not to be GPC'd, follow Steps 7.19 through 7.24 to concentrate extracts to final volume of 1 mL. Otherwise proceed to GPC cleanup procedure as described in the current revision of Katahdin SOP CA-513.

7.20 Transfer the labels from the extract flasks to the K-Ds. Remove the funnel and attach a 3-ball macro Snyder column. Pre-wet the Snyder column with 1 mL of methylene chloride.

7.21 Place the K-D in a hot water bath (75-85°C). Gently swirl K-D in the water until boiling begins. At the proper distillation rate, the Snyder balls should chatter but the chambers should not flood with condensed solvent. The K-D should be kept in a vertical orientation while on the bath. When the apparent volume in the concentrator tube reaches ≈ 6 mL, remove the K-D from the water bath. **Do not allow the evaporator to go dry. If the sample extract does go dry, re-extraction must occur immediately.** Allow the K-D to cool for 10 minutes. Rinse the Snyder column lower joint with ≈ 1 mL of methylene chloride. Remove the Snyder column. Wipe off any water from the neck above the lower joint of the flask. Separate the K-D flask from the concentrator tube, rinsing the ground glass joint with ≈ 1 mL methylene chloride.

7.22 Reduce the extract in the concentrator tube to approximately 1 mL using the nitrogen blow-down apparatus. The bath temperature must be < 30°C. Turn the gas to 3 psi. Be careful not to splash the extract out of the tube. During concentration on the N-evap, the internal wall of the concentrator tube and the N-evap sparging pipet must be rinsed down at least once or twice with ≈1 mL of methylene chloride. The solvent level in the concentrator tube must be positioned below the level of the water bath as much as possible to prevent water from condensing into the sample extract. As the extract volume is reduced, lower the N_2 sparging pipet closer to the surface of the extract to expedite the concentration. Record the temperature of the water in the nitrogen evaporation water bath in the logbook also note any problems or extract losses, if they occur, in the extractions logbook.

7.23 When the apparent volume reaches slightly less than 1 mL, remove the concentrator tube and allow it to cool.

7.24 Complete the quantitative transfer of the extract to a 1.8 mL vial by using methylene chloride. Adjust the volume of the methylene chloride extract to 1.0 mL using the 1.8 mL reference vial for volume comparison.

7.25 Label the vial with lab sample number, extraction date, matrix and analysis. Store extract vials at a temperature of 4 ± 2 °C until ready for analysis. Indicate in the extraction logbook the box number and "tray location" of the individual extract vials.

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

A method blank must be extracted for each and every item listed below:

- Each sample matrix
- Each extraction method or level
- Every extraction batch of twenty or fewer samples

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A laboratory control sample (LCS) is required for each and every item listed below:

- Each sample matrix
- Each extraction method or level
- Every extraction batch of twenty or fewer samples

Refer to the current revision of the applicable Katahdin SOP for analysis of Semivolatile Organics for quality control acceptance criteria.

Each extractions analyst must demonstrate proficiency in performing the extractions that prepare samples for analysis. Demonstration consists of preparation (extraction), by the analyst, of at least four aliquots which are then analyzed according to the analytical method in question. These QC samples must meet all quality control acceptance limits. Demonstration must be documented by use of a form which summarizes the results of the analysis of these aliquots, calculated percent recoveries, and standard deviation. Demonstration of proficiency must be done one time per analyst initially and then annually thereafter. Refer to SOPs QA-805 and QA-807, current revision.

If, upon analysis of the extracted samples, it is discovered that quality control acceptance criteria have not been met, all associated samples must be evaluated against all the QC. In some cases data may be reported, perhaps with narration, while in other cases, other corrective action may be taken. The corrective actions may include re-extraction of the samples associated with the quality control sample that did not meet acceptance criteria, or may include making new reagents and standards if the standardization is suspect. These decisions are based on holding time considerations, client and project specific Data Quality Objectives and on review of chromatograms. The supervisor, Operations Manager, and/or Quality Assurance Officer may be consulted to evaluate data. Some samples may not be able to be reanalyzed within hold time. In these cases "qualified" data with narration may be advisable after consultation with the client.

Much of the work performed at the lab is analyzed in accordance with specific QC requirements spelled out in a project specific Quality Assurance Project Plan (QAPP) or in a program specific Quality Systems Manual (QSM). The reporting limits, acceptance criteria and/or corrective actions may be different than those specified in this SOP. In these cases the appropriate information will be communicated to the Department Manager and/or senior chemists before initiation of the analyses so that specific product codes can be produced for the project. In addition, the work order notes for each project will describe the specific QAPP or QSM to be followed.

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9.0 METHOD PERFORMANCE

The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. The MDLs are determined annually per type of instrument and filed with the Organic Department Manager and with the QAO. Refer to the current revision of Katahdin SOP QA-806, Method Detection Limit and Instrument Detection Limit Studies, for procedures on determining the MDL.

Refer to the applicable analytical SOP for other method performance parameters and requirements.

10.0 APPLICABLE DOCUMENTS/REFERENCES

Test Methods for Evaluating Solid Waste-Physical/Chemical Methods, Method 3550C, USEPA SW-846, Third Edition, Update IV, February 2007.

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TABLE 1

SUMMARY OF METHOD MODIFICATIONS

TOPIC	KATAHDIN SOP CA-512-07	METHOD 3550, current revision
Apparatus/Materials	1) short stem funnels	1) drying columns
Reagents		
Sample preservation/ handling		
Procedures	1) extract dried using Na_2SO_4 in short stem funnels 2) place sonicator horns ½ way between the surface of the solvent and the sediment layer 3) no apparatus height specification for concentration on water bath 4) water bath at 75-85 deg C 5) sample removed from water bath when volume reaches ~6 mL	1) extract dried using Na_2SO_4 in drying columns 2) place sonicator horns ½ inch below the solvent surface but above sediment layer 3) partially immerse concentrator tube in water and lower apparatus to complete concentration in 10-15min 4) water bath at 80-90 deg C 5) sample removed from water bath when volume reaches 1-2 mL
QC - Spikes	1) Acid surrogate and spike components at 100 ug/mL; base/neutral surrogate and spike components at 50 ug/mL	1) Acid surrogate and spike components at 200 ug/mL; base/neutral surrogate and spike components at 100 ug/mL
QC - LCS	1) Acid surrogate and spike components at 100 ug/mL; base/neutral surrogate and spike components at 50 ug/mL	1) Acid surrogate and spike components at 200 ug/mL; base/neutral surrogate and spike components at 100 ug/mL
QC - Accuracy/Precision		
QC - MDL		

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FIGURE 1

EXAMPLE OF LOGBOOK PAGE

SV-SON

KATAHDIN ANALYTICAL SERVICES, INC.
ORGANIC EXTRACTIONS LOG - SOIL SEMIVOLATILE

Extraction Method: (check one)	SW846 3550 (SONIC.) ✓	SW846 3540 (SOX)	SW846 3535 (ASE)
Analytical Method: (check one)	SW846 8270 ✓	SW846 3580 (OILS/WIPES)	OTHER
Standards	Surrogate ID (1): SV2239	Spike ID (1): SV2231	
	Surrogate ID (2):	Spike ID (2):	
Solvents	Solvent Lot # (MeCl2): G22E23	Solvent Lot # (Acetone): E46E42	
Consumables	Filter Paper Lot # (SON) 41119164	Filter Paper Lot # (KD) J1137505	
Misc.	Nitrogen Bath Temperature: 39°C	Sonicator Horns Tuned:	

Date Extracted	Ext. Init.	Sample ID	Initial Weight (g)	Surr. Vol. (mL)	Spike Vol. (mL)	Final Vol. (mL)	Date Conc.	Tray Location	Initials	Comments
7-17-08	GN	WG53548-1	30.01	1mL	NR	1mL	7-17-08	SV324 C4	KF	R53707
		-2	30.03		1mL			C5		
		-3	29.97					C6		
						KF	7-17-08			

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Date Extracted	Ext. Init.	Sample ID	Initial Weight (g)	Surr. Vol. (mL)	Spike Vol. (mL)	Final Vol. (mL)	Date Conc.	Tray Location	Initials	Comments
7-17-08	GN	SB3798-2	30.00	1mL	NR	1mL	7-17-08	SV324 C7	KF	
		SB801-1	29.99			2mL		FP740 E3		Cleaned Chips
		-2	29.96					E4		
		-3	30.03					E5		Cleaned chips
		SB3845-1	30.01			1mL		SV324 C8		Reg-SB3217-6
						KF	7-17-08			

Reviewed By_____ Date_____

QAEX147 0000068

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT EXTRACTABLE SEMI-VOLATILES ANALYSIS**

FIGURE 2

LCS/MATRIX SPIKE COMPONENT LIST

BASE/NEUTRALS	
1-Methylnaphthalene	Bis (2-chloroethoxy) methane
1,1-Biphenyl	Bis (2-chloroethyl) ether
1,2,4-Trichlorobenzene	Bis (2-Chloroisopropyl) ether)
1,2-Dichlorobenzene	Bis (2-ethylhexyl) adipate
1,3-Dichlorobenzene	Bis (2-ethylhexyl) phthalate
1,4-Dichlorobenzene	Butylbenzyl phthalate
1,4-Dioxane	Caprolactam
2,4-Dinitrotoluene	Carbazole
2,6-Dinitrotoluene	Chrysene
2-Chloronaphthalene	Dibenz (a, h) anthracene
2-Methylnaphthalene	Dibenzofuran
2-Nitroaniline	Diethyl adipate
3,3'-Dichlorobenzidine	Diethyl phthalate
3-Nitroaniline	Dimethyl phthalate
4-Bromophenylphenyl ether	Di-n-butylphthalate
4-Chloroaniline	Di-n-octyl phthalate
4-Chlorophenylphenyl ether	Fluoranthene
4-Nitroaniline	Fluorene
Acenaphthene	Hexachlorobenzene
Acenaphthylene	Hexachlorobutadiene
Acetophenone	Hexachlorocyclopentadiene
Aniline	Hexachloroethane
Anthracene	Indeno (1,2,3-cd) pyrene
Atrazine	Isophorone
Azobenzene	Naphthalene
Benzaldehyde	Nitrobenzene
Benzidine	N-Nitrosodimethylamine
Benzo (a) Anthracene	N-Nitroso-di-n-propylamine
Benzo (a) pyrene	N-Nitrosodiphenylamine
Benzo (b) fluoranthene	Phenanthrene
Benzo (ghi) perylene	p-toluidine
Benzo (k) fluoranthene	Pyrene
Benzyl alcohol	Pyridine

ACIDS		
2, 3, 4, 6-Tetrachlorophenol	2-Chlorophenol	Benzoic acid
2,4,5-Trichlorophenol	2-Methylphenol	Ethyl methanesulfonate
2,4,6-Trichlorophenol	2-Nitrophenol	Methyl methanesulfonate
2,4-Dichlorophenol	4,6-Dinitro-2-methylphenol	Pentachlorophenol
2,4-Dimethylphenol	4-Chloro-3-methylphenol	Phenol
2,4-Dinitrophenol	4-Methylphenol	
2,6-Dichlorophenol	4-Nitrophenol	

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT EXTRACTABLE SEMI-VOLATILES ANALYSIS**

FIGURE 3

APPENDIX IX LCS/MATRIX SPIKE COMPONENT LIST

1,2,4,5-Tetrachlorobenzene	Hexachloropropene
1,3,5-Trinitrobenzene	Isodrin
1,4-Naphthoquinone	Isosafrole
1-Chloronaphthalene	Kepone
1-Naphthylamine	m-Dinitrobenzene
2,4-D	Methapyrilene
2-Acetyl aminofluorene	Methyl parathion
2-Naphthylamine	n-Nitrosodiethylamine
2-Picoline	n-Nitrosodi-n-butylamine
3,3-Dimethylbenzidine	n-Nitrosomethylethylamine
3-Methylcholanthrene	n-Nitrosomorpholine
4-Aminobiphenyl	n-Nitrosopyrrolidine
4-Nitroquinoline-1-oxide	n-Nitrotrosopiperidine
5-Nitro-o-toluidine	O,O,O-Triethyl phosphorothioate
7,12-Dimethylbenz(a)anthracene	o-Toluidine
a,a-Dimethylphenethylamine	Parathion
Acetophenone	p-Dimethylaminoazobenzene
Aramite	Pentachlorobenzene
Chlorobenzilate	Pentachloronitriobenzene
Diallate	Phenacetin
Dibenz(a,j)acridine	Phorate
Dimethoate	p-Phenylenediamine
Dinoseb	Pronamide
Diphenylamine	Safrole
Disulfoton	Silvex (2,4,5-TP)
Famphur	Sulfotep
Hexachlorophene	Thionazin

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR SUBSEQUENT EXTRACTABLE SEMIVOLATILE ANALYSIS**

Prepared By: Mike Thomas Date: 7/98

Approved By:

Group Supervisor: Michael Thomas Date: 11/15/00

Operations Manager: [illegible] Date: 11/15/00

QA Officer: Deborah J. Nadeau Date: 11·16·00

General Manager: [illegible] Date: 11/20/00

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01	Minor changes throughout. Clarifications to procedure section.	DN	11·16·00	11/16/00
02	Definitions added to section 1.1. Wording was added or changed to clarify sections 4, 5, 6, 7, 8 + 9. Minor changes throughout. New figures.	MRC	11.09.04	11.09.04
03 LAD 6-26-06	Updated Sect. 7.0 to include SIM. Updated figures 2 and 3 to include current SVOA ~~mixes~~ compounds used. Updated Sect. 5.0 to include all compounds analyzed for. Updated logbook page. minor edits throughout.	LAD	04/06	04/06
04	Added waste generated information. Updated Spikes and Surrogates. Added SIM LCSD and MS/D requirements. Updated Table 1. Added GPC references. Added LCSD after LCS.	LAD	09/07	09/07
05	Updated logbook page. Added adipate compounds to Fig. 2. Added recording of consomable's lot #'s and recording the nitrogen water bath temp. in logbook	LAD	07/08	07/08

TITLE: PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR SUBSEQUENT EXTRACTABLE SEMIVOLATILE ANALYSIS

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
06	Added balance criteria. Changed all weight criteria to +/- 0.05g. Revised section 7 to reflect current techniques. Added SOP CA-108 reference for sub-sampling. Updated logbook example	LAD	08/09	08/09

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR SUBSEQUENT EXTRACTABLE SEMIVOLATILE ANALYSIS**

Please acknowledge receipt of this standard operating procedure by signing and dating both of the spaces provided. Return the bottom half of this sheet to the QA Department.

I acknowledge receipt of copy ___ of document **SOP CA-526-06**, titled **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR SUBSEQUENT EXTRACTABLE SEMIVOLATILE ANALYSIS.**

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STANDARD OPERATING PROCEDURE

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Recipient: _____ Date: _____

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR SUBSEQUENT EXTRACTABLE SEMIVOLATILE ANALYSIS**

1.0 SCOPE AND APPLICATION

The purpose of this SOP is to describe the procedures and requirements for extracting semivolatile organic compounds from solids such as soils, sludges, and wastes using Method 3540. The Soxhlet extraction process ensures intimate contact the sample matrix with the extraction solvent.

This method is applicable to the isolation and concentration of water-insoluble and slightly water soluble organics in preparation for a variety of chromatographic procedures.

1.1 Definitions

METHOD BLANK (laboratory reagent blank): An artificial sample designed to determine if method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus. For aqueous samples, reagent water is used as a blank matrix; for soil samples, baked organic-free sand is used as a blank matrix. The blank is taken through the appropriate steps of the process.

LABORATORY CONTROL SAMPLE (LCS): A blank that has been spiked with the analyte(s) from an independent source, and is analyzed exactly like a sample. Its purpose is to determine whether the methodology is in control, and whether the laboratory is capable of making accurate and precise measurements. The matrix used should be phase matched with the samples and well characterized.

MATRIX SPIKE/MATRIX SPIKE DUPLICATE (MS/MSD): Predetermined quantities of stock solutions of certain analytes are added to a sample matrix prior to sample extraction and analysis. Samples are split into duplicates, spiked and analyzed. Percent recoveries are calculated for each of the analytes detected. The relative percent difference between the samples is calculated and used to assess analytical precision.

SURROGATES: Organic compounds which are similar to analytes of interest in chemical composition, extraction and chromatography, but which are not normally found in environmental samples. These compounds are spiked into all blanks, standards, samples and spiked samples prior to analysis. Percent recoveries are calculated for each surrogate.

1.2 Responsibilities

This method is restricted to use by, or under the supervision of analysts experienced in the extraction of samples for semivolatile analysis. Each analyst must demonstrate and document their ability to generate acceptable results with this method. Refer to

Katahdin SOP QA-805, current revision, "Personnel Training and Documentation of Capability".

It is the responsibility of all Katahdin technical personnel involved in the extraction of samples for semivolatile analysis to read and understand this SOP, adhere to the procedures outlined, and to properly document their data in the appropriate lab notebook. Any deviations from the test or irregularities with the samples should also be recorded in the lab notebook and reported to the Department Manager or designated qualified data reviewer responsible for this data.

It is the responsibility of the Department Manager to oversee that the members of his/her group follow this SOP, to assure that their work is properly documented, and to indicate periodic review of the pertinent logbooks.

1.3 Safety

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDSs for all the materials used in this procedure.

Each qualified analyst or technician must be familiar with Katahdin Analytical Environmental Health and Safety Manual including the Katahdin Hazardous Waste Plan and must follow appropriate procedures. These include the use of appropriate personal protective equipment (PPE) such as safety glasses, gloves and lab coats when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location of all safety equipment. Each analyst shall receive a safety orientation from their Department Manager, or designee, appropriate for the job functions they will perform.

1.4 Waste Disposal

Wastes generated during the preparation of samples must be disposed of in accordance with the procedures described in the current revision of the Katahdin Hazardous Waste Plan and Safety Manual and SOP SD-903, "Sample Disposal," current revision. Expired standards are lab packed, placed in the Katahdin hazardous waste storage area, and disposed of in accordance with this SOP.

Any methylene chloride solvent waste generated during the rinsing of glassware etc. should be disposed of in the "D" waste stream satellite accumulation area nearest the point of generation. Acetone and methanol are considered flammable waste,

and should be disposed of in the "O" waste stream satellite accumulation area nearest the point of generation. Post-extraction soil samples, used glass wool, and sodium sulfate waste should be disposed of in the soil with organics "I" waste stream satellite accumulation area nearest the point of generation. Please refer to the current revision of SOP CA-107 for the location of satellite waste accumulation areas.

2.0 SUMMARY OF METHOD

2.1 The solid sample is mixed with anhydrous sodium sulfate, placed in a Soxhlet extractor and extracted with methylene chloride.

2.2 The extract is then dried and concentrated for subsequent 8270 Semivolatile Organics analysis.

3.0 INTERFERENCES

Contaminants in solvents, reagents, glassware, and other sample processing hardware may cause method interferences such as discrete artifacts and/or elevated baselines in the total ion current profiles (TICPs). All of these materials routinely must be demonstrated to be free from interferences under the conditions of the analysis by running laboratory method blanks. Interferences caused by phthalate esters can pose a major problem in semivolatile organics analysis because many phthalates are also target analytes. Common flexible plastics contain varying amounts of phthalates that are easily extracted during laboratory operations, so cross-contamination of glassware frequently occurs when plastics are handled. Interferences from phthalates can best be minimized by avoiding the use of such plastics in the laboratory. At no time may gloves that have not been tested for phthalates or gloves known to contain phthalates be used or stored in the organic extraction lab. Additionally, whenever possible plastic items in this lab must be replaced with metal or Teflon or other non-phthalate plastic substitute.

Special care should be taken to ensure clean glassware and apparatus are used, pre-rinsed with the appropriate solvent prior to use. Solvents should be analyzed prior to use to demonstrate that each lot is free of contaminants that may interfere with the analysis.

Interferences coextracted from the samples will vary considerably from source to source. If analysis of an extracted sample is prevented due to interferences, further cleanup of the sample extract may be needed to minimize interferences.

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR SUBSEQUENT EXTRACTABLE SEMIVOLATILE ANALYSIS**

4.0 APPARATUS AND MATERIALS

4.1 Soxhlet apparatus:
- a) Soxhlet extractor – 45/50 top joint and 24/40 lower joint.
- b) 500 mL flat-bottom boiling flask
- c) Allihn cooling water condenser

4.2 Powder Funnels – 100 mm top diameter, 35 mm stem

4.3 Kuderna-Danish (K-D) apparatus

4.3.1 Concentrator tube - 10-mL

4.3.2 Evaporation flask - 500-mL

4.3.3 Snyder column - Three-ball macro

4.4 Nitrogen evaporation (N-EVAP) apparatus.

4.5 Boiling stones, 12 mesh silicon carbide (carborundum) – pre-purified by Soxhlet extraction in methylene chloride

4.6 Water bath - Heated, with concentric ring cover, capable of temperature control (± 5°C). The bath should be used in a hood.

4.7 Vials - Glass, 1.8-mL capacity, with polytetrafluoroethylene (PTFE)-lined septum vials, and 12 mL with Teflon-lined caps for extracts designated for GPC cleanup.

4.8 Glass wool (fiberglass) - baked at 400°C for a minimum of 4 hours or overnight.

4.9 Heating mantles - Rheostat controlled.

4.10 Disposable glass pasteur pipets, 5 ¾" and bulbs.

4.11 Drying oven - capable of maintaining 105°C for glassware drying.

4.12 Muffle oven – capable of maintaining 400 °C for baking glass wool and organic-free sand.

4.13 Beakers, 250 or 400 mL

4.14 Top-loading balance - capable of weighing to 0.**01** g.

4.15 Spatulas, stainless-steel

4.16 Long forceps, stainless-steel

4.17 Metal clips – for securing Soxhlets to boiling flasks

4.18 Filter Paper, 18.5 cm, Fisherbrand or Whatman 114V (or equivalent)

5.0 REAGENTS

5.1 Sodium Sulfate - anhydrous powdered and granular crystals, reagent grade, certified by the manufacturer/vendor as purified heating to 400°C prior to receipt by the laboratory.

5.2 Methylene chloride, methanol, and acetone - pesticide residue analysis grade or equivalent. Methylene chloride and acetone are evaluated, by lot, prior to use, by concentration of approximately 400 mL to 1.0 mL followed by GC/MS analysis.

5.3 Organic-free sand, purified by baking at 400 °C. Method blanks serve as checks on the baked sand.

5.4 Base/Neutral and Acid (SVOA) Surrogate Spiking Solution - Surrogate standards are added to all samples and calibration solutions. Prepare a surrogate standard spiking solution that contains the following compounds at the indicated concentrations in acetone.

Compound	Conc.
phenol-$_{d6}$	100 ug/mL
2,4,6-tribromophenol	100 ug/mL
2-fluorophenol	100 ug/mL
nitrobenzene-$_{d5}$	50 ug/mL
p-terphenyl-$_{d14}$	50 ug/mL
2-fluorobiphenyl	50 ug/mL

Store the spiking solution at -10°C to -20°C in Teflon-sealed containers. These solutions must be replaced after six months, or sooner if comparison with quality control check samples indicates a problem. If reanalysis of a method blank still indicates surrogates out of criteria, a new surrogate solution must be used.

5.5 SIM Surrogate Spiking Solution- Surrogate Standards are added to all samples and calibration solutions. Prepare a surrogate solution that contains the following compounds at a concentration of 2 ug/mL in acetone.

Compound	Conc. ug/mL
Fluorene-d10	2.0 ug/mL
2-Methylnaphthalene-d10	2.0 ug/mL
Pyrene-d10.	2.0 ug/mL
2,4-Dibromophenol	2.0 ug/mL

Store the spiking solution at -10°C to -20°C in Teflon-sealed containers. These solutions must be replaced after six months, or sooner if comparison with quality control check samples indicates a problem. If reanalysis of a method blank still indicates surrogates out of criteria, a new surrogate solution must be used.

5.6 Base/Neutral and Acid (SVOA) Lab Control Sample / Matrix Spike Spiking Solution - Prepare a spiking solution in methanol that contains the following mixes listed in Figure 2 at a concentration of 50 ug/ml for the base/neutral compounds and 100 ug/ml for the acid compounds. Store the spiking solution at -10°C to -20°C in Teflon-sealed containers. These solutions must be replaced after six months, or sooner if comparison with quality control check samples indicates a problem.

5.7 Base/Neutral (SIM) Matrix Spike/ Lab Control Sample Spike Solution for SIM-SVOA. Prepare a spiking solution in methanol that contains the compounds listed in Figure 2 at a concentration of 2 ug/mL for base/neutral. Take out 1.0 mL of Base/Neutral and Acid Matrix Spike/Lab Control Spiking Solution for SVOA and dilute it to 25.0 mL in methanol. Store the solution Spiking at -10°C to -20°C in Teflon-sealed containers. These solutions must be replaced after six months, or sooner if comparison with quality control check samples indicates a problem.

5.8 Base/Neutral and Acid (SVOA) Appendix IX Lab Control Sample / Matrix Spike Spiking Solution – Prepare a spiking solution in methanol that contains the compounds listed in Figure 3 at concentrations of 100 ug/ml. Store the spiking solution at -10°C to -20°C in Teflon-sealed containers. These solutions must be replaced after six months, or sooner if comparison with quality control check samples indicates a problem.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

Sediment/soil samples must be collected in a soil jar and must be maintained at 4°C (±2°C).

Holding time for extraction of sediment/soil samples for Method 3540 is 14 days from date of sample collection, although the analyst should be aware that actual holding times employed may be project/program specific.

Store all extracts at 4°C (±2°C) in the dark in labeled Teflon-sealed containers. See SOP SD-902, "Sample Receipt and Internal Control," current revision, for storage areas and temperature maintenance procedures.

7.0 PROCEDURES

All solid samples need to be cleaned up to reduce matrix interferences, time permitting. The cleanup procedure employed is gel permeation chromatography (GPC).

Sign chain-of-custody when removing and replacing samples in storage locations, and fill out the sample preparation/extraction log with the necessary information before starting the extraction. Prerinse all glassware three times with methylene chloride.

7.1 Preparing the Soxhlet Extraction Apparatus

7.1.1 Rinse the Soxhlet extractors and 500 mL flat-bottom boiling flasks three times with methylene chloride. Be sure that the solvent rinses through the large vapor tube and smaller siphon tubes of the Soxhlet. Inspect these for tiny cracks. Also rinse the 24/40 lower joint.

7.1.2 Add ~ 250 mLs of methylene chloride to the 500 mL boiling flask. Add several boiling stones. Using stainless steel forceps and working in a hood, place a plug of the pre-baked glass wool at the bottom of the Soxhlet so that the siphon tube hole is covered. Insert the 24/40 joint of the Soxhlet extractor into the 500 mL boiling flask and secure with a metal clip. Cover the top of the Soxhlet extractor with a piece of aluminum foil until ready to begin loading the sample. Record the solvent lot number in the extraction logbook.

7.2 Sample Handling

7.2.1 Sediment/soil samples - Decant and discard any water layer on a sediment sample. Mix the sample thoroughly with the stainless steel spatula. If the sample container is full to the extent that stirring the sample is impractical, try to remove the “best representative” aliquot from the jar based on color, particle size, moisture, etc. Discard any foreign objects such as sticks, leaves, and rocks.

7.2.2 Gummy, fibrous, or oily materials not amenable to mixing should be cut, shredded, or otherwise reduced in size to allow for maximum exposure of the sample surfaces to the extraction solvent. Materials such as glass, rubber, metal, etc. may not require mixing with powdered sodium sulfate to

disperse the sample. Plastic materials must be tested for degradation (melting) in methylene chloride prior to Soxhlet extraction.

7.2.3 Refer to Katahdin SOP CA-108, current revision, "Basic Laboratory Technique" for more information on subsampling.

7.3 The following steps should be performed rapidly to avoid loss of the more volatile extractables. Weigh out a 30.00 ± 0.05 g portion of sample into a labeled 400-mL beaker. Record sample weight to the nearest 0.05 g in appropriate extraction logbook. Add between 30 g and 60 g of anhydrous powdered sodium sulfate as required to produce a "free-flowing" mixture. The amount of sodium sulfate added will depend upon the moisture content of the sample (e.g., low moisture content will require less sodium sulfate). Mix well with a spatula. Keep the spatula in the sample beaker and cover the beaker with aluminum foil.

7.4 A method blank must be prepared with each extraction batch, not to exceed 20 client samples. To prepare method blank, weigh out one 30.00 ± 0.05 g portion of purified sand in a labeled 400 mL beaker. Add 60 g sodium sulfate and mix well. Although a "free-flowing" mixture can be achieved with less than 60 g sodium sulfate, the method blank must contain 60 g in order to evaluate the sodium sulfate as a potential source of contamination.

7.5 A laboratory control sample (LCS) must be prepared with each extraction batch, not to exceed 20 client samples. To prepare LCS, weigh out one 30.00 ± 0.05 g portion of purified sand in a labeled 400 mL beaker. Add 30 g sodium sulfate and mix well. If combined SIM-SVOA analysis is requested, a separate LCS must be prepared for each analysis. If an MS/MSD pair is not extracted on a particular day, an LCS/LCSD pair may be required in order to meet client-specific or program-specific requirements. This information will be disseminated from the project manager or Department Manager.

7.6 A matrix spike/matrix spike duplicate (MS/MSD) set must be prepared for every 20 samples. To prepare MS/MSD, weigh out 30.00 ± 0.05 g portions of the sample designated for MS/MSD into each of two labeled 400 mL beakers. Record sample weights to nearest 0.05 g in appropriate extraction logbook. Add 30 - 60 g sodium sulfate to each to produce a free-flowing mixture, and mix well. If combined SIM-SVOA analysis is requested, a separate MS/MSD must be prepared for each analysis.

7.7 Once all of the QC and field samples have been weighed and mixed with sodium sulfate, begin adding each to the assembled and appropriately labeled Soxhlet extractors using the stainless steel spatulas. Carefully scrape all of the mixtures from the beaker walls so that no more than 1% remains behind in the beaker. Be

careful not to have any of the solid material fall into the extract flask through the large vapor tube.

7.8 To all samples, method blank, LCS/LCSD, and MS/MSD add 1.0 mL of the appropriate base/neutral and acid surrogate spiking solution listed below using the pre-rinsed 1.0 mL gas tight syringe. Record surrogate spike volume and identification code in extraction logbook. Thoroughly rinse syringe with solvent prior to using it for another spiking solution.

7.8.1 If the request is for SVOA, use the SVOA surrogate solution (sect. 5.4)

7.8.2 If the request is for SIM, use the SIM surrogate solution (sect. 5.5).

7.8.3 If the request is for SIM-SVOA, use both the SIM and SVOA surrogate solutions. NOTE: Separate LCS/LCSD and/or MS/MSD are needed for each analysis and should be spiked with the appropriate surrogate.

7.9 To the LCS/LCSD and the MS/MSD add 1.0 mL of the appropriate base/neutral and acid (SVOA) matrix spike/LCS spiking solution listed below using a 1.0 mL gas tight syringe. Record matrix spike/LCS spiking solution volume and identification code in extraction logbook. Thoroughly rinse syringe with solvent when spiking is completed.

7.9.1 If the request is for SVOA, add 1 mL of the SVOA spiking solution (sect. 5.6).

7.9.2 If the request is for SIM, add 1 mL of the SIM Spiking solution (sect. 5.7).

7.9.3 If the request is for SVOA/SIM, add 1 mL of the SVOA spiking solution and 1 mL of the SIM Spiking solution to appropriate LCS/LCSD and/or MS/MSD. (sect's 5.6 and 5.7).

7.9.4 If the request is for SVOA Appendix IX, add 1 mL of the SVOA Appendix IX spiking solution and 1 mL of the SVOA spiking solution (sect's 5.6 and 5.8).

7.10 Place each of the Soxhlet extractors in a heating mantle and lower the Allihn cooling water condensers into the 45/50 joints of the extractors. Save the pieces of aluminum foil for covering the Soxhlets when the extraction is complete. Switch on the individual heating mantles and be sure that the Rheostat of the variable transformer is set to 55-60% of the output voltage. Once the methylene chloride begins to boil and the Soxhlet begins to cycle (solvent will immerse the sample and collect in the Soxhlet until the level reaches that of the small siphon tube and then begin to spill over into the extract flask), re-check the apparatus' for leaks. Allow the samples to extract for 18-24 hours. Be sure the chiller/recirculator temperature

is set low enough to provide enough cooling capacity for the number of extractions in the batch.

7.11 When the extraction is complete, allow the extracts to cool before dismantling. Tilt each extractor slightly to cause any remaining solvent in the sample chamber to drain through the siphon tube into the extract flask. This will help to cool the extract flask and make the apparatus easier to dismantle. Remove the Allihn condenser and replace the aluminum foil on top of the extractor. Move the extractors to a hood and detach the extractor from the extract flask. Try to drain as much solvent as possible from the extractor into the flask. **This is done by rinsing a glass tube in methylene chloride and pressing on the sample slightly so that as solvent as possible is drained into the extract flask.** Cover the flask with aluminum foil and store in the interim extract storage refrigerator unless the extracts are to be concentrated the same day.

7.12 Immediately remove the extracted soil/sodium sulfate mixtures from the extractors using a square edge spatula, and dispose of in an appropriate solid waste container. It is important to do this soon after the extractors are dismantled, as the sample mixture will tend to "freeze" into a solid mass in the Soxhlet as the solvent dries.

CONCENTRATION OF EXTRACTS

7.13 Rinse the K-D glassware (flask, concentration tube, and Snyder column, including the ground glass joints on the flask and columns) three times with methylene chloride before assembling. Add two boiling chips to the K-D. Insert 18.5 cm filter papers into short stem powder funnels and add ~ 2 inches of sodium sulfate crystals. Rinse the sodium sulfate in the assembled funnels with ~20-30 mLs of methylene chloride. Place the assembled K-D's under the funnels. Record the filter paper and sodium sulfate lot numbers in the extraction logbook.

7.14 Transfer the extracts to the K-D concentrator setups through the sodium sulfate in the funnels. This is the drying step, which is required to remove residual water from the extracts. Any large water layers must be removed by other means, prior to pouring through the sodium sulfate. After pouring all of the extract volume through the sodium sulfate, rinse the extract flask three times with ~ 2 – 3 mLs of methylene chloride. Add the rinsings through the sodium sulfate to complete a quantitative transfer. Rinse the sodium sulfate with ~ 15 mLs of methylene chloride and allow draining.

7.15 All samples should go through GPC cleanup except if time does not permit. Refer to the current revision of Katahdin SOP CA-513, Extract Cleanup Using Gel Permeation Chromatography, for appropriate concentrating procedures.

7.16 If samples are not to be GPC'd, when time does not permit, follow Steps 7.17 through 7.22 to concentrate extracts to final volume of 1 mL.

7.17 Transfer the labels from the extract flasks to the K-Ds. Remove the funnel and attach a 3-ball macro Snyder column. Pre-wet the Snyder column with 1 mL of methylene chloride.

7.18 Place the K-D in a hot water bath (75-85°C). Gently swirl K-D in the water until boiling begins. At the proper distillation rate, the Snyder balls should chatter but the chambers should not flood with condensed solvent. The K-D should be kept in a vertical orientation while on the bath. When the apparent volume in the concentrator tube reaches ≈ 4-6 mL, remove the K-D from the water bath. **Do not allow the evaporator to go dry. If the sample extract does go dry, re-extraction must occur immediately.** Allow the K-D to cool for 10 minutes. Rinse the Snyder column lower joint with ≈ 1 mL of methylene chloride. Remove the Snyder column. Wipe off any water from the neck above the lower joint of the flask. Separate the K-D flask from the concentrator tube, rinsing the ground glass joint with ≈ 1 mL methylene chloride.

7.19 Reduce the extract in the concentrator tube to approximately 1 mL using the nitrogen blow-down apparatus. The bath temperature must be no higher than the boiling point of the solvent (39°C for methylene chloride). Turn the gas to 3 psi. Be careful not to splash the extract out of the tube. During concentration on the N-evap, the internal wall of the concentrator tube and the N-evap sparging pipet must be rinsed down at least once or twice with ≈1 mL of methylene chloride. The solvent level in the concentrator tube must be positioned below the level of the water bath as much as possible to prevent water from condensing into the sample extract. As the extract volume is reduced, lower the N_2 sparging pipet closer to the surface of the extract to expedite the concentration. Record the temperature of the water in the nitrogen evaporation water bath in the extraction logbook, also note any problems or extract losses, if they occur.

7.20 When the apparent volume reaches slightly less than 1 mL, remove the concentrator tube and allow it to cool.

7.21 Complete the quantitative transfer of the extract to a 1.8 mL vial by using methylene chloride. Adjust the volume of the methylene chloride extract to 1.0 mL using the 1.8 mL reference vial for volume comparison.

7.22 Label the vial with lab sample number, extraction date, matrix and analysis. Store extract vials at a temperature of 4 ± 2 °C until ready for analysis. Indicate in the extractions logbook the box number and "tray location" of the individual extract vials.

8 QUALITY CONTROL AND ACCEPTANCE CRITERIA

A method blank must be extracted for each and every item listed below:

- Each sample matrix (soil, water)
- Each day of extraction (24 hours midnight - midnight)
- Each extraction method or level
- Every 20 samples extracted in a 24-hour period

A laboratory control sample (LCS) is required for each and every item listed below:

- Each sample matrix
- Each extraction method or level
- Every extraction batch of twenty or fewer samples

Refer to the current revision of the applicable Katahdin SOP for analysis of extractable semivolatile organics for quality control acceptance criteria.

Each extractions analyst must demonstrate proficiency in performing the extractions that prepare samples for analysis. Demonstration consists of preparation (extraction), by the analyst, of at least four aliquots which are then analyzed according to the analytical method in question. These QC samples must meet all quality control acceptance limits. Demonstration must be documented by use of a form which summarizes the results of the analysis of these aliquots, calculated percent recoveries, and standard deviation. Demonstration of proficiency must be done one time per analyst initially and then annually thereafter. Refer to SOPs QA-805 and QA-807, current revision.

If, upon analysis of the extracted samples, it is discovered that quality control acceptance criteria have not been met, all associated samples must be evaluated against all the QC. In some cases data may be reported, perhaps with narration, while in other cases, other corrective action may be taken. The corrective actions may include re-extraction of the samples associated with the quality control sample that did not meet acceptance criteria, or may include making new reagents and standards if the standardization is suspect. These decisions are based on holding time considerations, client and project specific Data Quality Objectives and on review of chromatograms. The supervisor, Operations Manager, and/or Quality Assurance Officer may be consulted to evaluate data. Some samples may not be able to be reanalyzed within hold time. In these cases "qualified" data with narration may be advisable after consultation with the client.

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR SUBSEQUENT EXTRACTABLE SEMIVOLATILE ANALYSIS**

Much of the work performed at the lab is analyzed in accordance with specific QC requirements spelled out in a project specific Quality Assurance Project Plan (QAPP) or in a program specific Quality Systems Manual (QSM). The reporting limits, acceptance criteria and/or corrective actions may be different than those specified in this SOP. In these cases the appropriate information will be communicated to the Department Manager and/or senior chemists before initiation of the analyses so that specific product codes can be produced for the project. In addition, the work order notes for each project will describe the specific QAPP or QSM to be followed.

9.0 METHOD PERFORMANCE

The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. The MDLs are determined annually per type of instrument and filed with the Organic Department Manager and with the QAO. Refer to the current revision of Katahdin SOP QA-806, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications, for procedures on determining the MDL.

Refer to the applicable analytical SOP for other method performance parameters and requirements.

10.0 APPLICABLE DOCUMENTS/REFERENCES

Test Methods for Evaluating Solid Waste-Physical/Chemical Methods, Method 3540C, SW-846, Third Edition, Updates I, II, IIA, IIB, and III Revised December 1996, US EPA.

Department of Defense Quality Systems Manual for Environmental Laboratories (DOD QSM), Version 4.1, 04/22/09.

The National Environmental Laboratory Accreditation Conference (NELAC) Standards, June 2003.

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Figure 2 LCS/Matrix Spike Component List
Figure 3 Appendix IX LCS/Matrix Spike Component List

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR SUBSEQUENT EXTRACTABLE SEMIVOLATILE ANALYSIS**

TABLE 1

SUMMARY OF METHOD MODIFICATIONS

TOPIC	KATAHDIN SOP CA-526-06	METHOD 3540, current revision
Apparatus/Materials	1. short stem funnels	2. drying columns
Reagents		
Sample preservation/ handling		
Procedures	1. Use 30 grams of sample and 30 grams of sodium sulfate 2. Place a plug of glass wool in soxhlet then add sample 3. Use 250 mL of methylene chloride for extraction 4. Extract the sample for 18 - 24 hours 5. Extract dried using Na_2SO_4 in short stem funnels 6. Rinse the extract flask three times with ~ 2 – 3 mLs of methylene chloride then rinse the sodium sulfate with ~ 15 mLs of methylene chloride to complete a quantitative transfer 7. no apparatus height specification for concentration on water bath 8. Water bath at 75-85 deg C 9. Sample removed from water bath when volume reaches ~6 mL	1. Use 10 grams of sample and 10 grams of sodium sulfate. 2. Place sample between 2 plugs of glass wool 3. Use 300 mL of methylene chloride for extraction 4. Extract the sample for 16 - 24 hours at 4 - 6 cycles/hour 5. Extract dried using Na_2SO_4 in drying columns 6. Wash the extractor flask and sodium sulfate column with 100 to 125 mL of extraction solvent to complete the quantitative transfer 7. partially immerse concentrator tube in water and lower apparatus to complete concentration in 10-20min 8. Water bath at 15-20 deg C above solvent boiling point 9. Sample removed from water bath when volume reaches 1-2 mL
QC - Spikes	1. Acid surrogate and spike components at 100 ug/mL; base/neutral surrogate and spike components at 50 ug/mL	1. Acid surrogate and spike components at 200 ug/mL; base/neutral surrogate and spike components at 100 ug/mL
QC - LCS	1. Acid surrogate and spike components at 100 ug/mL; base/neutral surrogate and spike components at 50 ug/mL	1. Acid surrogate and spike components at 200 ug/mL; base/neutral surrogate and spike components at 100 ug/mL
QC - Accuracy/Precision		
QC – MDL		

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR SUBSEQUENT EXTRACTABLE SEMIVOLATILE ANALYSIS**

FIGURE 1

EXAMPLE OF LOGBOOK PAGE

SV SOX

KATAHDIN ANALYTICAL SERVICES, INC.
ORGANIC EXTRACTIONS LOG - SOIL SEMIVOLATILE

Extraction Method: (check one)	SW846 3550 (SONIC.)	SW846 3540 (SOX) ✓	SW846 3545 (ASE)
	SW846 3580 (OIL / WIPES)	OTHER	
Analytical Method: (check one)	SW846 8270 ✓	OTHER	
Standards	Surrogate ID (1): SV2324	Spike ID (1): SV2320	Spike ID (3):
	Surrogate ID (2):	Spike ID (2):	
Solvents / Chemicals / Consumables	Solvent Lot # (Mecl2): H196008	Solvent Lot # (Acetone): —	Sodium Sulfate (granular) Lot # ~~2769~~ 279690D2
	Filter Paper Lot # (SON) —	Filter Paper Lot # (KD) K1672363	Sodium Sulfate (powder) Lot # ~~2769~~ 279690D1
Misc.	Nitrogen Bath Temperature: 35°C	Sonicator Horns Tuned: —	Balance ID: Mettler R3400

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Ext. Date	Ext. Int.	Sample ID	Initial Weight (g)	Surr. Vol (mL)	Spk Vol. (mL)	Fraction		Pre-GPC			Post-GPC				Comments
						B/N	A	Date Conc.	Conc Init.	Final Vol (mL)	Date Conc	Conc Init	Final Vol. (mL)	Tray Loc	
6-23-09	KF	1665386-1	30.00	1mL	1mL	✓		6-26-09	KF	5mL	6-29-09	KF	1mL	SV451 B6	R102843
		-2	29.98		1mL									B7	
		-3	29.96											B8	
		GPC Blank 6-29-09												B9	
										KF	6-26-09				

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Ext. Date	Ext. Int.	Sample ID	Initial Weight (g)	Surr. Vol (mL)	Spk Vol. (mL)	Fraction		Pre-GPC			Post-GPC				Comments
						B/N	A	Date Conc.	Conc Init.	Final Vol (mL)	Date Conc.	Conc Init.	Final Vol. (mL)	Tray Loc	
6-23-09	KF	SC3258-11F	31.04	1mL	1mL	✓		6-26-09	KF	5mL	6-30-09	GN	1mL	SV451 B10	
		-12F	30.02											C1	
		-13F	30.00											C2	
		-14F	30.01											C3	
		-15F	29.96											C4	
		-16F	30.05											C5	
		-17F	30.02											C6	
		-18F	29.99											C7	
		-19F	29.99											C8	
		-20F	30.00											C9	
		SC3259-3F	31.99											C10	
		-4F	29.96											D1	
		-5F	30.00											D2	
		SC3305-3F	30.01											D3	
										KF	6-26-09				

QAEX169

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Reviewed By_____ Date_____

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR SUBSEQUENT EXTRACTABLE SEMIVOLATILE ANALYSIS**

FIGURE 2

LCS/MATRIX SPIKE COMPONENT LIST

BASE/NEUTRALS	
1-Methylnaphthalene	Bis (2-chloroethoxy) methane
1,1-Biphenyl	Bis (2-chloroethyl) ether
1,2,4-Trichlorobenzene	Bis (2-Chloroisopropyl) ether)
1,2-Dichlorobenzene	Bis (2-ethylhexyl) adipate
1,3-Dichlorobenzene	Bis (2-ethylhexyl) phthalate
1,4-Dichlorobenzene	Butylbenzyl phthalate
1,4-Dioxane	Caprolactam
2,4-Dinitrotoluene	Carbazole
2,6-Dinitrotoluene	Chrysene
2-Chloronaphthalene	Dibenz (a, h) anthracene
2-Methylnaphthalene	Dibenzofuran
2-Nitroaniline	Diethyl adipate
3,3'-Dichlorobenzidine	Diethyl phthalate
3-Nitroaniline	Dimethyl phthalate
4-Bromophenylphenyl ether	Di-n-butylphthalate
4-Chloroaniline	Di-n-octyl phthalate
4-Chlorophenylphenyl ether	Fluoranthene
4-Nitroaniline	Fluorene
Acenaphthene	Hexachlorobenzene
Acenaphthylene	Hexachlorobutadiene
Acetophenone	Hexachlorocyclopentadiene
Aniline	Hexachloroethane
Anthracene	Indeno (1,2,3-cd) pyrene
Atrazine	Isophorone
Azobenzene	Naphthalene
Benzaldehyde	Nitrobenzene
Benzidine	N-Nitrosodimethylamine
Benzo (a) Anthracene	N-Nitroso-di-n-propylamine
Benzo (a) pyrene	N-Nitrosodiphenylamine
Benzo (b) fluoranthene	Phenanthrene
Benzo (ghi) perylene	p-toluidine
Benzo (k) fluoranthene	Pyrene
Benzyl alcohol	Pyridine

ACIDS		
2, 3, 4, 6-Tetrachlorophenol	2-Chlorophenol	Benzoic acid
2,4,5-Trichlorophenol	2-Methylphenol	Ethyl methanesulfonate
2,4,6-Trichlorophenol	2-Nitrophenol	Methyl methanesulfonate
2,4-Dichlorophenol	4,6-Dinitro-2-methylphenol	Pentachlorophenol
2,4-Dimethylphenol	4-Chloro-3-methylphenol	Phenol
2,4-Dinitrophenol	4-Methylphenol	
2,6-Dichlorophenol	4-Nitrophenol	

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR SUBSEQUENT EXTRACTABLE SEMIVOLATILE ANALYSIS**

FIGURE 3

APPENDIX IX LCS/MATRIX SPIKE COMPONENT LIST

1,2,4,5-Tetrachlorobenzene	Hexachloropropene
1,3,5-Trinitrobenzene	Isodrin
1,4-Naphthoquinone	Isosafrole
1-Chloronaphthalene	Kepone
1-Naphthylamine	m-Dinitrobenzene
2,4-D	Methapyrilene
2-Acetyl aminofluorene	Methyl parathion
2-Naphthylamine	n-Nitrosodiethylamine
2-Picoline	n-Nitrosodi-n-butylamine
3,3-Dimethylbenzidine	n-Nitrosomethylethylamine
3-Methylcholanthrene	n-Nitrosomorpholine
4-Aminobiphenyl	n-Nitrosopyrrolidine
4-Nitroquinoline-1-oxide	n-Nitrotrosopiperidine
5-Nitro-o-toluidine	O,O,O-Triethyl phosphorothioate
7,12-Dimethylbenz(a)anthracene	o-Toluidine
a,a-Dimethylphenethylamine	Parathion
Acetophenone	p-Dimethylaminoazobenzene
Aramite	Pentachlorobenzene
Chlorobenzilate	Pentachloronitriobenzene
Diallate	Phenacetin
Dibenz(a,j)acridine	Phorate
Dimethoate	p-Phenylenediamine
Dinoseb	Pronamide
Diphenylamine	Safrole
Disulfoton	Silvex (2,4,5-TP)
Famphur	Sulfotep
Hexachlorophene	Thionazin

KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

SOP Number: CA-535
Revision History
Cover Page
Page 1

TITLE: PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT DIESEL RANGE ORGANICS (DRO), TOTAL PETROLEUM HYDROCARBONS (TPH), or EXTRACTABLE TOTAL PETROLEUM HYDROCARBONS ANALYSIS

Prepared By: Charlie Gomez Date: 3-4/02

Approved By:

Group Supervisor: [signature] Date: 4-4-02

Lab Operations Mgr: [signature] Date: 4/5/02

QA Officer: Deborah J. Nadeau Date: 4.4.02

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01	Many edits to be consistent with other Sonication SOP's.	LAD	030805	030805
02	Updated Materials in Sect. 4.0 Sect. 5.2 added Sodium Sulfate crystals removed fluted from filter paper.	LAD	04/06	04/06
03	Added waste generated information. Updated Table 1. Added definitions. Added LCSD after LCS. Minor changes throughout to provide continuity with other DRO SOP's.	LAD	09/07	09/07
04	Updated logbook page. Added recording of Solvent, Na_2SO_4, filter paper lot #'s as well as the temperature of the nitrogen water bath in the logbook	LAD	07/08	07/08
05	Changed balance criteria to 0.01g. Changed the weight criteria to ±0.05g. Minor changes to Section 7 to reflect current techniques. Updated logbook example.	LAD	08/09	08/09

TITLE: PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT DIESEL RANGE ORGANICS (DRO), TOTAL PETROLEUM HYDROCARBONS (TPH), or EXTRACTABLE TOTAL PETROLEUM HYDROCARBONS ANALYSIS

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
06	Added ETPH references, Added ETPH silica gel clean up procedure. Added ETPH to title	LAD	03/2010	03/2010

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT DIESEL RANGE ORGANICS (DRO), TOTAL PETROLEUM HYDROCARBONS (TPH), or EXTRACTABLE TOTAL PETROLEUM HYDROCARBONS ANALYSIS**

Please acknowledge receipt of this standard operating procedure by signing and dating both of the spaces provided. Return the bottom half of this sheet to the QA Department.

I acknowledge receipt of copy _____ of document **SOP CA-535-06**, titled **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT DIESEL RANGE ORGANICS (DRO), TOTAL PETROLEUM HYDROCARBONS (TPH), or EXTRACTABLE TOTAL PETROLEUM HYDROCARBONS ANALYSIS.**

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TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT DIESEL RANGE ORGANICS (DRO), TOTAL PETROLEUM HYDROCARBONS (TPH), or EXTRACTABLE TOTAL PETROLEUM HYDROCARBONS ANALYSIS**

1.0 SCOPE AND APPLICATION

The purpose of this SOP is to describe the procedures and requirements for the preparation of solid samples using SW-846 method 3550 for subsequent DRO/TPH/ETPH analysis by gas chromatography with a flame ionization detector.

1.1 Definitions

TOTAL PETROLEUM HYDROCARBONS (TPH): All resolved and unresolved material eluting from n-nonane (n-C9) through n-hexatriacontane (n-C36), inclusive.

EXTRACTABLE TOTAL PETROLEUM HYDROCARBONS (ETPH): All resolved and unresolved material eluting from n-nonane (n-C9) through n-hexatriacontane (n-C36), inclusive.

DIESEL RANGE ORGANICS (DRO): All resolved and unresolved material eluting from decane (n-C10) through n-octacosane (n-C28), inclusive.

ANALYTICAL BATCH: 20 or fewer samples which are analyzed together with the same method sequence and the same lots of reagents and with the manipulations common to each sample within the same time period or in continuous sequential time periods.

METHOD BLANK (laboratory reagent blank): An artificial sample designed to determine if method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus. For aqueous samples, reagent water is used as a blank matrix; for soil samples, baked organic-free sand is used as a blank matrix. The blank is taken through the appropriate steps of the process.

LABORATORY CONTROL SAMPLE (LCS): A blank that has been spiked with the analyte(s) from an independent source, and is analyzed exactly like a sample. Its purpose is to determine whether the methodology is in control, and whether the laboratory is capable of making accurate and precise measurements. The matrix used should be phase matched with the samples and well characterized.

MATRIX SPIKE/MATRIX SPIKE DUPLICATE (MS/MSD): Predetermined quantities of stock solutions of certain analytes are added to a sample matrix prior to sample extraction and analysis. Samples are split into duplicates, spiked and analyzed. Percent recoveries are calculated for each of the analytes detected. The relative percent difference between the samples is calculated and used to assess analytical precision.

SURROGATES: Organic compounds which are similar to analytes of interest in chemical composition, extraction and chromatography, but which are not normally found in environmental samples. These compounds are spiked into all blanks, standards, samples and spiked samples prior to analysis. Percent recoveries are calculated for each surrogate.

1.2 Responsibilities

This method is restricted to use by, or under the supervision of analysts experienced in the extraction of samples for petroleum hydrocarbon analysis using method 3550. Each analyst must demonstrate and document their ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, current revision, "Personnel Training & Documentation of Capability".

It is the responsibility of all Katahdin technical personnel involved in the extraction of samples for petroleum hydrocarbon analysis to read and understand this SOP, to adhere to the procedures outlined, and to properly document their data in the appropriate lab notebook. Any deviations from the test or irregularities with the samples should also be recorded in the lab notebook and reported to the Department Manager or designated qualified data reviewer responsible for this data.

It is the responsibility of the Department Manager to oversee that the members of his/her group follow this SOP, to assure that their work is properly documented, and to indicate periodic review of the pertinent logbooks.

1.3 Safety

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDSs for all the materials used in this procedure.

Each qualified analyst or technician must be familiar with Katahdin Analytical Environmental Health and Safety Manual including the Katahdin Hazardous Waste Management Plan and must follow appropriate procedures. These include the use of appropriate personal protective equipment (PPE) such as safety glasses, gloves and lab coats when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location of all safety

equipment. Each analyst shall receive a safety orientation from their Department Manager, or designee, appropriate for the job functions they will perform.

1.4 Pollution Prevention/Waste Disposal

Whenever possible, laboratory personnel should use pollution prevention techniques to address their waste generation. Refer to the current revision of the Katahdin Hazardous Waste Management Plan for further details on pollution prevention techniques.

Wastes generated during the preparation of samples must be disposed of in accordance with the Katahdin Hazardous Waste Management Plan and Safety Manual and SOP SD-903, "Sample Disposal," current revision. Expired standards are lab packed, placed in the Katahdin hazardous waste storage area, and disposed of in accordance with this SOP.

Any methylene chloride solvent waste generated during the rinsing of glassware etc. should be disposed of in the "D" waste stream satellite accumulation area nearest the point of generation. Acetone is considered flammable waste, and should be disposed of in the "O" waste stream satellite accumulation area nearest the point of generation. Post-extraction soil samples and sodium sulfate waste should be disposed of in the soil with organics "I" waste stream satellite accumulation area nearest the point of generation. Please refer to the current revision of SOP CA-107 for the location of satellite waste accumulation areas.

2.0 SUMMARY OF METHOD

Soil samples are extracted with a methylene chloride/acetone solution (1:1 by volume) according to SW-846 Method 3550. The resulting extract is dried, concentrated, and then analyzed by GC-FID. For ETPH, silica gel is added to the extract before concentrating.

3.0 INTERFERENCES

3.1 Other organic compounds including chlorinated hydrocarbons, phenols, and phthalate esters are measurable. As defined in the method, the DRO results include these compounds. Spills of known specific constituents should be analyzed and quantified by a method specific for those compounds.

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT DIESEL RANGE ORGANICS (DRO), TOTAL PETROLEUM HYDROCARBONS (TPH), or EXTRACTABLE TOTAL PETROLEUM HYDROCARBONS ANALYSIS**

3.2 Method interferences are reduced by washing all glassware with hot soapy water and then rinsing it sequentially with tap water, methanol or acetone, and methylene chloride. Method /reagent blanks must be analyzed with each batch to demonstrate that the samples are free from method interferences. Solvents should be analyzed prior to use to demonstrate that each lot is free of contaminants that may interfere with the analysis.

4.0 APPARATUS AND MATERIALS

Prior to use, all glassware must be rinsed three times with methylene chloride.

4.1 Beakers - 400 Ml

4.2 Kuderna-Danish (KD) apparatus -

- Concentrator tube - 10 mL
- Evaporative flask - 500 mL
- Snyder column - 3-ball macro

4.3 Powder funnels, 100 mm diameter, 35 mm stem

4.4 Vacuum filtration flask - 500 mL Erlenmeyer

4.5 Buchner funnel, porcelain, Coors® with 85 mm plate diameter (or equivalent)

4.6 Sonic disruptors – Misonix XL2015 W-375, (or equivalent), equipped with dual titanium horn extenders for extracting two samples at a time.

4.7 Spatula - stainless steel

4.8 Filter paper, 18.5 cm, Fisher brand or Whatman 114V (or equivalent)

4.9 Boiling chips - 12 mesh, silicon carbide (or equivalent)

4.10 Water bath - eight position concentric ring bath or equivalent, equipped with a calibrated thermometer

4.11 Filter paper - 7.0 cm, Whatman, #4, or equivalent

4.12 Syringe - gas tight, 1.0 mL, solvent rinsed between each use

4.13 Balance – top-loading, capable of weighing to 0.01 g

4.14 Nitrogen evaporation apparatus

4.15 Vials - 1.8mL, 12-mL, and/or 16-mL screw cap w/PTFE coated septa

5.0 REAGENTS

5.1 Sodium sulfate - (ACS reagent grade) powdered, anhydrous, certified by the manufacturer/vendor as purified by heating to 400 °C prior to receipt by the laboratory.

5.2 Sodium sulfate - (ACS reagent grade) crystals, anhydrous, purified as described in section 5.1.

5.3 Methylene chloride - (pesticide grade or equivalent) purchased by lot, evaluated prior to use by concentration of 300mLs to 1.0 mL followed by GC-FID analysis.

5.4 Acetone - pesticide grade or equivalent

5.5 Methanol – pesticide grade or equivalent.

5.6 Organic-free sand, purified by baking at 400 °C overnight. Method blanks serve as checks on the baked sand.

5.7 Surrogate spiking solution - Prepare a solution of o-Terphenyl at a concentration of 20 ug/mL in acetone. Store the solution at –10 to -20 °C (±2 °C) in a Teflon sealed container. Solution must be verified by GC-FID prior to use, and must be replaced every 6 months or sooner if degradation is evident.

5.8 Matrix Spike/Lab Control Sample spiking solution - Prepare a spiking solution in methanol that contains all target analytes listed below:

Analyte	ug/mL	Analyte	ug/mL
Decane	50	Ecosane	50
Dodecane	50	Docosane	50
Tetradecane	50	Tetracosane	50
Hexadecane	50	Hexacosane	50
Octadecane	50	Octacosane	50

Store the solution at –10 to -20 °C (±2 °C) in a Teflon sealed container. Solution must be verified by GC-FID prior to use, and must be replaced every 6 months or sooner if degradation is evident.

5.9 Silica gel, 60-200 mesh, Davidson Grade 950 or equivalent.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

Samples are collected in soil jars and held at 4 (±2) °C until time of extraction.

Holding time for extraction of sediment/soil samples for Method 3550 is 14 days from date of sample collection, although the analyst should be aware that actual holding times employed may be project/program specific.

Store all extracts at 4°C (±2°C) in the dark in labeled Teflon-sealed containers. See SOP SD-902, "Sample Receipt and Internal Control," current revision, for storage areas and temperature maintenance procedures.

7.0 PROCEDURES

The procedure involves extraction of DRO/TPH/ETPH from an initial sample weight of 30.0 ± 0.05 g using an ultrasonic cell disruptor.

7.1 Discard any excess water on the sediment sample. Mix with a stainless steel spatula to ensure homogeneity of the sample. If the sample container is full to the extent that stirring the sample is impractical, try to remove the "best representative" aliquot from the jar based on color, particle size, moisture, etc. Remove any foreign objects such as sticks, leaves, or rocks, and note actions taken in the appropriate extraction logbook. Please refer to Katahdin Analytical Services SOP CA-108, "Basic Laboratory Technique", current revision, for more information on subsampling.

7.2 Weigh out a 30.0 ± 0.05g portion of sample into a labeled 400 mL beaker. Record sample weight to nearest 0.05 g in appropriate extraction logbook. Add between 30 g and 60 g of powdered sodium sulfate, as required, to produce a "free-flowing" mixture. The amount of sodium sulfate added will depend upon the moisture content of the sample (e.g., low moisture content will require less sodium sulfate). Mix well with a spatula. Keep the spatula in the sample beaker and cover the

beaker with aluminum foil. Record the lot number of the sodium sulfate in the extraction logbook.

7.3 A method blank must be prepared with each extraction batch, not to exceed 20 client samples. To prepare method blank, weigh out one 30.0 ± 0.05 g portion of purified sand in a labeled 400 mL beaker. Add 60 g sodium sulfate and mix well. (Although a "free-flowing" mixture can be achieved with less than 60 g sodium sulfate, the method blank must contain 60 g in order to evaluate the sodium sulfate as a potential source of contamination.)

7.4 A laboratory control sample (LCS) must be prepared with each extraction batch, not to exceed 20 client samples. To prepare LCS, weigh out one 30.0 ± 0.05 g portion of purified sand in a labeled 400 mL beaker. Add 30 g sodium sulfate and mix well. If an MS/MSD pair is not extracted on a particular day, an LCS/LCSD pair may be required in order to meet client-specific or program-specific requirements. This information will be disseminated from the project manager or Department Manager.

7.5 A matrix spike/matrix spike duplicate (MS/MSD) set must be prepared for every 20 samples. To prepare MS/MSD, weigh out 30.0 ± 0.05 g portions of the sample designated for MS/MSD into each of two labeled 400 mL beakers. Record sample weights to nearest 0.05 g in appropriate extraction logbook. Add 30 - 60 g sodium sulfate to each to produce a free-flowing mixture, and mix well.

7.6 To all samples, method blank, LCS/LCSD, and MS/MSD add 1.0 mL DRO/TPH surrogate spiking solution using a 1.0 mL gas tight syringe. Record surrogate spike volume and identification code in extraction logbook. Thoroughly rinse syringe with solvent prior to each use.

7.7 To LCS/LCSD and MS/MSD pairs add 1.0 mL of DRO/TPH matrix spike/LCS spiking solution using a 1.0 mL gas tight syringe. Record matrix spike/LCS spiking solution volume and identification code in extraction logbook. Thoroughly rinse syringe with solvent prior to spiking a different solution and when spiking is completed.

7.8 To assure optimum operation and maximum energy output, the sonicators <u>must</u> be tuned daily prior to extracting samples. The following tuning procedure must be performed with the sonicator probes vibrating in air.

7.8.1 Turn OUTPUT CONTROL knob counter-clockwise to zero. This automatically switches the duty cycle to continuous mode.

7.8.2 Press and hold down the power switch to on.

7.8.3 Press and hold down the TUNE switch. Check if the counter is less or equal to 20%; otherwise, rotate the Tuning Knob (tuning button) clockwise until a reading of 20%or less is obtained.

7.8.4 Release the TUNE switch.

7.8.5 Turn OUTPUT CONTROL KNOB counter-clockwise to 50 and the power switch off.

7.8.6 Confirm that the sonicators were tuned by recording the date and/or percent in the extractions logbook.

7.9 Prior to extracting any samples, ensure that the sonicator probes are decontaminated by rinsing with a methylene chloride wash bottle. Collect the waste in a waste beaker. It may sometimes be necessary to wipe the upper part of each probe with a methylene chloride dampened KimWipe. Repeat this decontamination step between each sample on each probe.

7.10 To the mixed and spiked blank and LCS, add 100 mL of the 1:1 methylene chloride/acetone (V/V) solution and proceed with steps 7.11 through 7.14. After step 7.14, repeat these steps with two more mixed and spiked samples. Record the lot numbers of the solvents in the extraction logbook.

7.11 It may be necessary at this time to stir the sample/sodium sulfate mixture with the spatula to loosen up the mixture prior to extracting. Position beaker in the ultrasonic cell disruptor so that the bottom surface of the tip of the 3/4 inch disruptor horn is about halfway below the surface of the solvent and above the sediment layer.

7.12 Sonicate for 3 minutes with the output control knob set at 10, and mode switch on "pulsed" and % duty cycle knob set at 50%. While the mixture is sonicating, one should be able to see all, or most of the material, moving in the beaker under the influence of the energized probes. If not, stir the mixture again.

7.13 Prepare a filter flask fitted with a Buchner funnel. The Buchner funnel should contain a 7.0 cm Whatman #4 filter; prerinsed flask, funnel and filter with methylene chloride and discard rinsings into solvent waste container. Decant extract into the filter flask and Buchner funnel. A vacuum pump may be used to facilitate filtration

or the extract may be gravity filtered. Record the filter paper number lot number in the extraction logbook.

7.14 Repeat the extraction two additional times using 100 mL portions of 1:1 methylene chloride/acetone. Before each extraction, make certain that the sodium sulfate is free-flowing and not a consolidated mass. As required, break up large lumps with the clean spatula. Decant the extraction solvent into the Buchner funnel after each sonication. On the final sonication, pour the entire sample contents into the Buchner funnel and rinse thoroughly with methylene chloride to complete the quantitative transfer of the extract.

7.15 Concentration - Rinse the K-D glassware (flask, concentration tube, and Snyder column, including the ground glass joints on the flask and columns) three times with methylene chloride before assembling. Add two boiling chips to the K-D. Insert 18.5 cm filter papers into short stem powder funnels and add ~ 2 inches of sodium sulfate crystals. Rinse the sodium sulfate in the assembled funnels with ~20-30 mLs of methylene chloride. Place the assembled K-D's under the funnels. Record the lot numbers of the sodium sulfate crystals and filter paper in the extraction logbook.

7.16 For ETPH, before the extract is transferred to the funnel, 3 grams of silica gel per 100mls of sample are added to the filter flask.

7.17 Transfer the extracts to the K-D concentrator setups through the sodium sulfate in the funnels. This is the drying step, which is required to remove residual water from the extracts. Any large water layers must be removed by other means, prior to pouring through the sodium sulfate. After pouring all of the extract volume through the sodium sulfate, rinse the extract flask three times with ~ 2 – 3 mL of methylene chloride. Add the rinsings through the sodium sulfate to complete a quantitative transfer. Rinse the sodium sulfate with ~ 15 mL of methylene chloride and allow to drain.

7.18 Transfer the labels from the extract flasks to the K-Ds. Remove the funnel and attach a 3-ball macro Snyder column. Pre-wet the Snyder column with 1 mL of methylene chloride.

7.19 Place the K-D in a hot water bath (75-85°C). Gently swirl K-D in the water until boiling begins. At the proper distillation rate, the Snyder balls should chatter but the chambers should not flood with condensed solvent. The K-D should be kept in a vertical orientation while on the bath. When the apparent volume in the

concentrator tube reaches ≈ 6 mL, remove the K-D from the water bath. **Do not allow the evaporator to go dry. If the sample extract does go dry, re-extraction must occur immediately.** Allow the K-D to cool for 10 minutes. Rinse the Snyder column lower joint with ≈ 1 mL of methylene chloride. Remove the Snyder column. Wipe off any water from the neck above the lower joint of the flask. Separate the K-D flask from the concentrator tube, rinsing the ground glass joint with ≈ 1 mL methylene chloride.

7.20 Reduce the extract in the concentrator tube to approximately 0.8 mL using the nitrogen blow-down apparatus. The bath temperature must be no higher than the boiling point of the solvent (39°C for methylene chloride). Turn the gas to 3 psi. Be careful not to splash the extract out of the tube. During concentration on the N-evap, the internal wall of the concentrator tube and the N-evap sparging pipet must be rinsed down at least once or twice with ≈1 mL of methylene chloride. The solvent level in the concentrator tube must be positioned below the level of the water bath as much as possible to prevent water from condensing into the sample extract. As the extract volume is reduced, lower the N_2 sparging pipet closer to the surface of the extract to expedite the concentration. Record the temperature of the water in the nitrogen evaporation water bath in the extraction logbook, also note any problems or extract losses, if they occur.

7.21 When the apparent volume reaches slightly less than 1 mL, remove the concentrator tube and allow it to cool.

7.22 Complete quantitative transfer of the extract to a vial by using methylene chloride. Adjust the volume of the methylene chloride extract to 1.0 mL in a 1.8 mL vial using the appropriate "reference vial" for volume comparison. If the extract is highly colored or a precipitate forms during concentration, the final volume should be adjusted and noted in the extractions logbook with a comment.

7.23 Label the vial with lab sample number, extraction date, matrix and analysis. Store extract vials at a temperature of 4 ± 2 °C until ready for analysis. Indicate in the extractions logbook the "tray location" of the individual extract vials.

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

A method blank and laboratory control sample must be extracted for every extraction batch of twenty or fewer samples.

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT DIESEL RANGE ORGANICS (DRO), TOTAL PETROLEUM HYDROCARBONS (TPH), or EXTRACTABLE TOTAL PETROLEUM HYDROCARBONS ANALYSIS**

Refer to the current revision of the applicable Katahdin SOP for analysis of Diesel Range Organics, Total Petroleum Hydrocarbons or Extractable Total Petroleum Hydrocarbons for quality control acceptance criteria.

Each extractions analyst must demonstrate proficiency in performing the extractions that prepare samples for analysis. Demonstration consists of preparation (extraction), by the analyst, of at least four aliquots which are then analyzed according to the analytical method in question. These QC samples must meet all quality control acceptance limits. Demonstration must be documented by use of a form which summarizes the results of the analysis of these aliquots, calculated percent recoveries, and standard deviation. Demonstration of proficiency must be done one time per analyst initially and then annually thereafter. Refer to SOPs QA-805 and QA-807, current revision.

If, upon analysis of the extracted samples, it is discovered that quality control acceptance criteria have not been met, all associated samples must be evaluated against all the QC. In some cases data may be reported, perhaps with narration, while in other cases, other corrective action may be taken. The corrective actions may include re-extraction of the samples associated with the quality control sample that did not meet acceptance criteria, or may include making new reagents and standards if the standardization is suspect. These decisions are based on holding time considerations, client and project specific Data Quality Objectives and on review of chromatograms. The supervisor, Operations Manager, and/or Quality Assurance Officer may be consulted to evaluate data. Some samples may not be able to be reanalyzed within hold time. In these cases "qualified" data with narration may be advisable after consultation with the client.

Much of the work performed at the lab is analyzed in accordance with specific QC requirements spelled out in a project specific Quality Assurance Project Plan (QAPP) or in a program specific Quality Systems Manual (QSM). The reporting limits, acceptance criteria and/or corrective actions may be different than those specified in this SOP. In these cases the appropriate information will be communicated to the Department Manager and/or senior chemists before initiation of the analyses so that specific product codes can be produced for the project. In addition, the work order notes for each project will describe the specific QAPP or QSM to be followed.

9.0 METHOD PERFORMANCE

The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. The MDLs are determined annually per type of instrument and filed with the Organic Department Manager and with the QAO. Refer to the current revision of Katahdin SOP

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT DIESEL RANGE ORGANICS (DRO), TOTAL PETROLEUM HYDROCARBONS (TPH), or EXTRACTABLE TOTAL PETROLEUM HYDROCARBONS ANALYSIS**

QA-806, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications, for procedures on determining the MDL.

Refer to the applicable analytical SOP for other method performance parameters and requirements.

10.0 APPLICABLE DOCUMENTS/REFERENCES

Test Methods for Evaluating Solid Waste - Physical/Chemical Methods, Method 3550B, USEPA SW-846, Third Edition, Final Update III, December 1996.

Maine HETL, Method 4.1.25, Modified Method for the Determination of DROs, 9/95.

Department of Defense Quality Systems Manual for Environmental Laboratories (DOD QSM), Version 4.1, 04/22/09.

The National Environmental Laboratory Accreditation Conference (NELAC) Standards, June 2003.

Analysis of Extractable Total Petroleum Hydrocarbons (ETPH) Using Methylene Chloride, prepared by Environmental Research Institute, University of Connecticut, March 1999.

LIST OF TABLES AND FIGURES

Table 1 Summary of Method Modifications
Figure 1 Example of DRO/TPH/ETPH Sample Prep Logbook Page

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT DIESEL RANGE ORGANICS (DRO), TOTAL PETROLEUM HYDROCARBONS (TPH), or EXTRACTABLE TOTAL PETROLEUM HYDROCARBONS ANALYSIS**

TABLE 1

SUMMARY OF METHOD MODIFICATIONS

TOPIC	KATAHDIN SOP CA-535-06	METHOD 3550, current revision
Apparatus/Materials	1) 1.0 mL syringe	1) 5.0 mL syringe
Reagents		
Sample preservation/ handling		
Procedures	1) extract dried using Na_2SO_4 in short stem funnels 2) place sonicator horns ½ way between the surface of the solvent and the sediment layer 3) no apparatus height specification for concentration on water bath 4) water bath at 75-85 deg C 5) sample removed from water bath when volume reaches ~6 mL	1) extract dried using Na_2SO_4 in drying columns 2) place sonicator horns ½ inch below the solvent surface but above sediment layer 3) partially immerse concentrator tube in water and lower apparatus to complete concentration in 10-15min 4) water bath at 80-90 deg C 5) sample removed from water bath when volume reaches 1-2 mL
QC - Spikes		
QC - LCS		
QC - Accuracy/Precision		
QC - MDL		

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT DIESEL RANGE ORGANICS (DRO), TOTAL PETROLEUM HYDROCARBONS (TPH), or EXTRACTABLE TOTAL PETROLEUM HYDROCARBONS ANALYSIS**

FIGURE 1

EXAMPLE OF LOGBOOK PAGE

DRO Son

KATAHDIN ANALYTICAL SERVICES, INC.
ORGANIC EXTRACTIONS LOG - SOIL FUEL OILS

Extraction Method: (check one)	SW846 3550 (SONIC.) X	SW846 3540 (SOX)	SW846 3535 (ASE)
Analytical Method: (check one)	SW846 8015M -DRO X	ME DEP 4.1.25 - DRO	SW8100M - TPH
	FLO PRO - TPH	CT-ETPH	TX 1005 - TPH
Standards	Surrogate ID: GCO 1013	Spike ID: GCO 1004	Spike ID:
Solvents	Solvent Lot # (Mecl2): H17E31	Solvent Lot # (Acetone): G51E06	Solvent Lot # (n-Pentane):
Consumables	Filter Paper Lot # (SON) J115889781	Filter Paper Lot # (KD) K1162273363	Acid Lot # ——
Misc.	Nitrogen Bath Temperature: 350C	Sonicator Horns Tuned: <20%	Sulfate: 27969002
		Scale Mettler PJ400	Powder 27979001

QAEX152

Date Extracted	Ext. Init.	Sample ID	Initial Vol. ~~mL~~ g	Surr. Vol.	Spike Vol.	Final Vol. mL	Date Conc.	Tray Location	Initials	Comments
7-23-09	AC	WG66542-1	30.04	1mL	NR	1mL	7/23/09	PO 499 D9	CB	R104791
		-2	30.04		0.5mL			D10		
		-3	30.00					G1		
							CB 7/23/09			

0000139

Date Extracted	Ext. Init.	Sample ID	Initial Vol. ~~mL~~ g	Surr Vol.	Spike Vol.	Final Vol. mL	Date Conc.	Tray Location	Initials	Comments
7-23-09	AC	SC4082-1b	30.01	1mL	NR	1mL	7/23/09	PO 499 E2	CB	
		-2b	30.00					E3		
		-3b	29.96					E4		
		-4b	30.02					E5		
		-5b	30.02					E6		
							CB 7/23/09			

QAEX152

0000140

Reviewed By_____ Date_____

TITLE: ACID DIGESTION OF SOLID SAMPLES BY USEPA METHOD 3050 FOR METALS ANALYSIS BY ICP-AES AND GFAA

Prepared By: George Brewer Date: 3/98

Approved By:

Group Supervisor: George Brewer Date: 01/24/01

Operations Manager: Joel C. Burton Date: 1/24/01

QA Officer: Deborah J. Nadeau Date: 1.24.01

General Manager: Duncan F. Lufkin Date: 1/25/01

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01 3050B	Format changes, added pollution prevention, added MSD, added spiking instruction tables.	DN	1.24.01	1/24/01
02 3050B	Removed all references/procedures devoted to GFAA. Added use of digestates for ICP-MS analysis. Revised standard solution names + concs. in Tables 3 + 4 to reflect current practice.	DN	8.29.02	8.29.02
03 3050B	New Title to include ILMO5.3. Use of digestion block and polyethylene digestion tubes added to sections 4.0, 7.0 and Table 1. PBS changed from 1.0g water to 1.0g boiling chips. H_2O_2 addition from 3.0ml then 2.0mls to 2.0ml, 2.0ml then 7.0ml. Figures and Tables updated to reflect current practices.	LAD	03/08	03/08
04	Updated Tables 3 and 4 with current spike concentrations and volumes added. Updated Logbook page. Added CA-108 reference for subsampling information.	LAD	08/09	08/09

TITLE: ACID DIGESTION OF SOLID SAMPLES BY USEPA METHOD 3050 FOR METALS ANALYSIS BY ICP-AES, ICP-MS

Please acknowledge receipt of this standard operating procedure by signing and dating both of the spaces provided. Return the bottom half of this sheet to the QA Department.

I acknowledge receipt of copy ___ of document **SOP CA-605-04**, titled **ACID DIGESTION OF SOLID SAMPLES BY USEPA METHOD 3050 FOR METALS ANALYSIS BY ICP-AES, ICP-MS AND ILM05.3.**

Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy ___ of document **SOP CA-605-04**, titled **ACID DIGESTION OF SOLID SAMPLES BY USEPA METHOD 3050 FOR METALS ANALYSIS BY ICP-AES, ICP-MS AND ILM05.3.**

Recipient: _____ Date: _____

TITLE: ACID DIGESTION OF SOLID SAMPLES BY USEPA METHOD 3050 FOR METALS ANALYSIS BY ICP-AES, ICP-MS

1.0 SCOPE AND APPLICATION

The purpose of this SOP is to describe the Katahdin Analytical Services, Inc. procedure utilized to dissolve solid matrices and solubilize metals from solid samples prior to analysis for metals by ICP-AES and ICP-MS. This SOP applies to samples prepared by EPA Method 3050, with method modifications as summarized in Table 2.

This procedure applies to all solid sample (e.g. sediments, sludges, soils, and ashes) preparations for ICP-AES and ICP-MS analyses. This method is not a total digestion technique for most samples. It is a very strong acid digestion that will dissolve almost all elements that could become "environmentally available". By design, elements bound in silicate structures are not normally dissolved by this procedure as they are not usually mobile in the environment.

1.1 Definitions

ICP-AES – Inductively Coupled Plasma Atomic Emission Spectroscopy.

ICP-MS – Inductively Coupled Plasma Mass Spectrometry.

LCSS – Laboratory Control Sample for Solids – A standard or solid reference material that has been brought through the sample preparation process.

Matrix Spike – An aliquot of a sample to which a known amount of analyte has been added before digestion.

PBS – Preparation Blank for Solids – An aliquot of reagent water that has been brought through the sample preparation process.

1.2 Responsibilities

This method is restricted to use by, or under the supervision of analysts experienced in the acid digestion of solid samples by USEPA Method 3050 for metals analysis. Each analyst must demonstrate the ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, current revision, "Personnel Training & Documentation of Training".

It is the responsibility of all Katahdin technical personnel involved in the acid digestion of solid samples by USEPA Method 3050 to read and understand this SOP, to adhere to the procedures outlined, and to properly document their data in the appropriate lab notebook. Any deviations from the procedure or irregularities with the samples should also be recorded in the lab notebook and reported to the responsible Department Manager or designated qualified data reviewer.

It is the responsibility of the Department Manager to ensure that technical personnel perform acid digestions in accordance with this SOP and to confirm that their work is properly documented through periodic review of the associated logbooks.

1.3 Safety

The acids used in this procedure are highly corrosive and reactive, and spiking standards contain toxic metals. The toxicity and reactivity of client samples are usually unknown, so samples should always be assumed to present a contact hazard. To reduce or eliminate exposure to potentially harmful chemicals, lab coats, gloves, and safety glasses or goggles must be worn whenever handling samples or reagents. Additional safety apparel, including face shields, aprons, dust masks, and shoe protectors, is available in the Metals prep lab and should be worn whenever circumstances warrant.

Acids should be added to samples slowly and carefully, while watching for reactions. This should be done under a hood, in case harmful fumes are evolved.

Hood sashes should be lowered as far as possible whenever beakers are being heated on a hot plate. Use caution when handling hot beakers.

Personnel are required to read the Katahdin Hazrdous Waste Management Plan and Safety Manual before performing this procedure, and must be familiar with the general rules for laboratory safety, personal hygiene, housekeeping, and use of protective clothing and equipment.

1.4 Pollution Prevention/Waste Disposal

Whenever possible, laboratory personnel should use pollution prevention techniques to address their waste generation. Refer to the current revision of the Katahdin Hazardous Waste Management Program for further details on pollution prevention techniques.

Excess spiking solutions must be emptied into the corrosive waste carboy located in the Metals prep lab for subsequent appropriate disposal in accordance with the Katahdin Hazardous Waste Management Plan and Safety Manual.

Sample digestates should be stored for a minimum of 60 days after digestion to allow for analysis, and reanalysis if necessary. Digestates older than 60 days may be emptied into the corrosive waste carboy in the Metals prep lab for subsequent appropriate disposal in accordance with the Katahdin Hazardous Waste Mnagement Plan and Safety Manual.

TITLE: ACID DIGESTION OF SOLID SAMPLES BY USEPA METHOD 3050 FOR METALS ANALYSIS BY ICP-AES, ICP-MS

2.0 SUMMARY OF METHOD

A representative 1 to 2 g (wet weight) sample is digested with repeated additions of nitric acid and hydrogen peroxide. Hydrochloric acid is added to the initial digestate and the sample is refluxed. The digestate is then filtered and diluted to a final volume of 100 mL.

3.0 INTERFERENCES

Interferences are discussed in the applicable analytical SOPs.

4.0 APPARATUS AND MATERIALS

4.1 Digestion vessels. If digestion is performed using a hot plate, the appropriate digestion vessels are 100 mL pre-cleaned Griffin beakers (cleaned according to the current revision of SOP CA-100, "Labware Cleaning" and CA-602, "Glassware Preparation and Sample Preservation for Trace Element Analyses"). If digestion is performed using a block digester, the appropriate digestion vessels are new 70 mL disposable graduated polyethylene digestion tubes with attached snap lids.

4.2 Ribbed watch glasses. If digestion is performed using a hot plate, 75 mm diameter glass watch glasses (pre-cleaned as above) are used. If digestion is performed using a block digester, 40 mm diameter disposable polyethylene watch glasses are used.

4.3 Adjustable volume automatic pipets covering the range from 10 uL to 1000 uL and disposable pipet tips; calibrated Finn pipets or Eppendorf pipets are acceptable.

4.4 Disposable graduated polystyrene specimen containers with pouring lips, 200 mL capacity.

4.5 Hot plate or block digester, griddle, or other heating source - adjustable and capable of maintaining a temperature of 95°C ± 5°C. Heating sources must be numbered for easy identification.

4.6 Device for measuring hot plate temperature, consisting of a flask or digestion vessel in which the bulb of a thermometer is immersed in sand or water. The temperature of each hot plate used is measured and recordedeach day. The hot plate identification number and the measured temperature are recorded on the sample preparation logbook sheet.

4.7 Plastic funnels, pre-cleaned as in Section 4.1.

4.8 Filter funnel holders, capable of suspending plastic funnels above disposable specimen containers.

4.9 Polyethylene wash bottles for dispensing reagent water and 5% HNO_3.

4.10 Filter paper, Whatman No. 41 or equivalent. Filters are acid-washed immediately prior to use as follows. Place a pre-cleaned funnel in the funnel holder and put a disposable plastic specimen container under the funnel to collect the rinsates. Place a folded filter in the funnel and rinse three times with approximate 10 mL volumes of 5% HNO_3, making sure the entire surface of the filter is wetted each time and allowing each rinse to drain completely before continuing. Then rinse three times with approximate 25 mL volumes of reagent water, again allowing each rinse to drain completely. Discard the rinsates into the appropriate waste container. The acid-washed filter is now ready for use.

4.11 Polyethylene sample containers with screw caps or graduated polyethylene sample containers with attached snap lids, 125 mL capacity.

4.12 Repipetters (adjustable repeating pipetters with reservoirs) for dispensing concentrated nitric acid, 1:1 HNO_3, and concentrated HCl.

4.13 Analytical balance capable of reading to 0.01 gram.

4.14 Spatulas, scoops, or spoons; plastic or stainless steel, rinsed with 5% HNO_3 and reagent water. Disposable tongue depressors may be used and do not requrire to be rinsed.

5.0 REAGENTS

5.1 Concentrated nitric acid, HNO_3 – trace metals grade.

5.2 Concentrated hydrochloric acid, HCl – trace metals grade.

5.3 Reagent water - water that meets the performance specifications of ASTM Type II water (ASTM D1193).

5.4 Nitric acid, 1:1. Add a volume of concentrated HNO_3 to an equivalent volume of reagent water and swirl gently to mix.

5.5 Nitric acid, 5% v/v. Add 25 mL concentrated HNO_3 to 475 mL reagent water in a 500 mL wash bottle. Cap, point the dispensing tip into a sink, and shake gently to mix.

5.6 30% hydrogen peroxide (H_2O_2) - spectrometric grade.

5.7 Multielement spiking solutions (see Table 3 for a list of required spiking solutions).

5.8 Solid reference material – a soil containing all the elements of interest, with empirically established method-specific recoveries and acceptance limits for all analytes. Solid reference materials are purchased with documentation of analysis provided by the vendor. See Figure 4 for an example certificate of analysis for a solid reference material.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

Samples should be collected in clean plastic or glass containers. Samples must be refrigerated (4°C ±2°C) upon receipt by the laboratory. The holding time for solid samples is 6 months from the date of sample collection.

7.0 PROCEDURE

The procedure described below is condensed for quick reference in Table 3.

SAMPLE PREPARATION

7.1 Prior to performing the digestion, make a list of the samples that are to be digested. Enter digestion information (Katahdin Sample Numbers, QC Batch ID, preparation date, analyst initials, etc.) into the ACCESS computer spreadsheet. Print out a copy of the spreadsheet (see Figure 2 for an example). Hand label the digestate vessels

7.2 If using glass beakers as the digestion vessels, submerge previously cleaned beakers and watch glasses three times into a 10% nitric acid bath, then rinse three times with reagent water. The polyethylene digestion tubes used in conjunction with the block digeter do not require acid rinsing or precleaning. Label the digestion vessels with sample numbers.

7.3 Weigh 1 to 2 g of well-mixed sample into a properly cleaned, labeled, and tared Griffin beaker or polyethylene digestion tube. Record (hand write) the weight of each sample on the printout of the digestion spreadsheet. Refer to Katahdin Analytical Services SOP CA-108, current revision "Basic Laboratory Technique" for more information on subsampling.

7.4 Weigh an appropriate amount of solid reference material to a clean, labeled, and tared Griffin beaker or polyethylene digestion tube to serve as a laboratory control sample.

7.5 Add spike solutions to matrix spike samples (refer to Tables 3 and 4 for spiking instructions).

7.6 Using repipetters, add 10 mL of 1:1 HNO_3, mix the slurry. Cover with a ribbed watch glass and place on heat source. Gently heat the sample to 95°C ± 5 °C and reflux for 10 to 15 minutes without boiling. Remove the digestion vessel from the heat source and cool the sample.

7.7 Add 5 mL of concentrated HNO_3 to the sample, replace the watch glass, and reflux for 30 minutes. If brown fumes are generated, indicating oxidation of the sample by HNO_3, repeat this step (addition of 5 mL of concentrated HNO_3) until no brown fumes are given off by the sample, indicating complete reaction by HNO_3.

7.8 Continue heating the sample at 95°C ± 5°C without boiling until the digestate has evaporated to approximately 5 to 10 mL or until two hours have elapsed, whichever occurs first. Do not allow the sample to go to dryness. Remove the digestion vessel from the heat source and cool the sample.

7.9 Add 2 mL of reagent water and 2 mL of 30% H_2O_2 to the sample, replace the watch glass, and heat gently on the heat source to start the peroxide reaction. Continue heating until effervescence subsides.

7.10 Add an additional 2 mL of 30% H_2O_2 to the sample, replace the watch glass, and heat gently on the heat source to start the peroxide reaction. Continue heating until effervescence subsides.

7.11 Add an additional 6 mL of 30% H_2O_2 in 1-mL aliquots with warming until the effervescence is minimal or until the general sample appearance is unchanged.

7.12 Continue heating the sample at 95°C ± 5°C without boiling until the digestate has evaporated to approximately 5 to 10 mL or until two hours have elapsed, whichever occurs first. Do not allow the sample to go to dryness. Remove the sample from the heat source and cool.

7.13 Add 10 mL of concentrated HCl to the digest from 7.12, replace the watch glass, and reflux at 95°C ± 5°C for 15 minutes. Remove the sample from the heat source and cool.

7.14 Use a pre-cleaned funnel and acid-rinsed filter paper to filter the digestate into a clean graduated polystyrene specimen container or graduated polyethylene sample container with attached snap lid. Using a wash bottle, rinse the digestion vessel with reagent water and add the rinsates to the filter apparatus. After all of the liquid in the filter has drained into the specimen container, thoroughly rinse the filter three times with small (5-10 mL) volumes of reagent water, allowing the liquid to drain completely after each rinse. Using the graduations on the specimen container or

snap-lid container, dilute to 100 mL with reagent water. If a specimen container has been used, transfer the contents to the corresponding labeled polyethylene sample bottle, cap the bottle, and discard the empty specimen container. If a snap-lid container has been used, close and secure the snap-lid. Shake the container gently to mix. The digestate is now ready for ICP-AES or ICP-MS analysis.

7.15 Review the ACCESS computer spreadsheet for accuracy. If any information is incorrect, make the necessary changes to the computer spreadsheet and print out a corrected copy. Do not discard the original copy of the spreadsheet. Record (hand write) reagent lot numbers, spiking information, and heat source temperature in the appropriate spaces on the spreadsheet. Record any method deviations, irregularities with the samples, or other pertinent observations at the bottom of the page, and sign and date the spreadsheet. Bind all copies of the spreadsheet in the sample preparation log. An example sample preparation logbook page (ACCESS spreadsheet) is included as Figure 2.

7.15 Reopen the electronic ACCESS spreadsheet for the digestion and transcribe the sample weights from the handwritten, bound copy into the electronic copy. The information in this electronic spreadsheet will later be imported into the ACCESS metals database and used to calculate sample concentrations on a weight basis.

7.16 Place each batch of digestates in a box labeled with the QC Batch ID, and put the box of digestates in the metals digestates storage area.

CALCULATIONS

7.17 Analytical results for solid samples are reported on a dry weight basis. Total solids are determined by the Wet Chemistry Group, and are recorded in spreadsheets that are electronically imported into the Access metals database. Final dry weight concentrations are calculated by the Access database as follows:

$$\text{Concentration (mg/kg dry weight)} = (C \times V) / (W \times S)$$

where: C = Measured concentration (mg/L)
V = Digestate final volume (L)
W = Sample wet weight (kg)
S = % Solids/100

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

USEPA Method 3050 requires the laboratory to perform specific quality control checks to assess laboratory performance and data quality. Minimum frequencies, acceptance criteria, and corrective actions for these control checks are tabulated in Table 1 and are described below. Table 1 criteria are intended to be guidelines for analysts. The table does

not cover all possible situations. If any of the QC requirements are outside the recovery ranges listed in Table 1, all associated samples must be evaluated against all the QC. In some cases data may be reported, but may be reanalyzed in other cases. Making new reagents and standards may be necessary if the standardization is suspect. The corrective actions listed in Table 1 may rely on analyst experience to make sound scientific judgements. These decisions are based on holding time considerations and client and project specific Data Quality Objectives. The Depatment Manager, Operations Manager, and/or Quality Assurance Officer may be consulted to evaluate data. Some samples may not be able to be reanalyzed within hold time. In these cases "qualified" data with narration may be advisable after consultation with the client.

8.1 At least one preparation blank for soils (PBS) is processed concurrently with each digestion batch of 20 or fewer samples, and is used to assess contamination resulting from the digestion procedure. The PBS consists of a 1.0 g of boiling stones that is digested using the same reagents as those used to digest associated samples. Refer to the appropriate analytical SOP for PBS acceptance criteria and corrective actions.

8.2 At least one laboratory control sample for soils (LCSS) is processed concurrently with each digestion batch of 20 or fewer samples. The LCSS consists of an aliquot of a solid reference material for which the concentrations of the analytes of interest have been empirically established (solid-matrix LCSS), or an aliquot of reagent water that is spiked to contain all analytes of interest at known concentrations (aqueous-matrix LCSS). The solid reference material should normally be used as the LCSS, unless a particular client or analytical program requires that spiked reagent water be used. The LCSS is digested using the same reagents as those used to digest associated samples. Directions for spiking the aqueous-matrix LCSS are contained in Table 3. The measured analyte recoveries for the LCSS are used to assess digestion method performance. Refer to the appropriate analytical SOP for LCSS recovery acceptance criteria and corrective actions.

8.3 Matrix spike samples are processed along with each digestion batch at a minimum frequency of one per digestion batch. A matrix spike sample consists of an aliquot of a sample that is fortified with known amounts of all analytes of interest prior to digestion. Matrix spike recoveries are used to assess the biasing effects of sample matrix on digestion and analysis performance. Directions for spiking matrix spike samples are contained in Figure 2. Refer to the appropriate analytical SOP for matrix spike recovery acceptance criteria and corrective actions.

8.4 Matrix spiked duplicate samples are processed concurrently with each digestion batch at a minimum frequency of one per digestion batch. Matrix spiked duplicate samples are used to assess the precision of the digestion and analysis methods. Refer to the appropriate analytical SOP for matrix spike duplicate precision acceptance criteria and corrective actions.

NOTE: Clients may choose specific samples for matrix spike and duplicate analysis; otherwise, the choice is left to the person performing the digestion.

8.5 The quality control measures and frequencies described above are minimum requirements. Individual clients and analytical programs may impose additional QC requirements.

9.0 METHOD PERFORMANCE

Refer to the applicable instrumental analysis SOP for method performance information.

10.0 APPLICABLE DOCUMENTS/REFERENCES

"Test Methods for the Evaluation of Solid Waste," United States Environmental Protection Agency, SW-846, Third Edition, Final Update III, 12/96, Method 3050B.

Department of Defense Quality Systems Manual for Environmental Laboratories (DOD QSM), Version 4.1, 04/22/09.

The National Environmental Laboratory Accreditation Conference (NELAC) Standards, June 2003.

LIST OF TABLES AND FIGURES

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Table 2 Summary of Method Modifications – Method 3050
Table 3 Preparation of Matrix Spikes and Spiking Solutions
Table 4 Element Concentrations in ICP-AES Matrix Spikes and Their Component Spiking Solutions
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Figure 2 Example Page from Metals Sample Preparation Logbook
Figure 3 Example Certificate of Analysis for Solid Reference Material

TABLE 1

QC REQUIREMENTS – METHOD 3050

Method	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
3050	Preparation Blank for Solids (PBS)	One per prep batch of 20 or fewer samples.	Refer to analytical method.	Refer to analytical method.
	Laboratory Control Sample for Solids (LCSS)	One per prep batch of 20 or fewer samples.	Refer to analytical method.	Refer to analytical method.
	Matrix Spike Sample	One per prep batch.	Refer to analytical method.	Refer to analytical method.
	Matrix Spike Duplicate Sample	One per prep batch.	Refer to analytical method.	Refer to analytical method.
	Demonstration of analyst proficiency	One-time demonstration by each analyst performing the method.	Must pass all applicable QC for method.	Repeat analysis until able to perform passing QC; document successful performance in personal training file.

TITLE: ACID DIGESTION OF SOLID SAMPLES BY USEPA METHOD 3050 FOR METALS ANALYSIS BY ICP-AES, ICP-MS

TABLE 2

SUMMARY OF METHOD MODIFICATIONS – METHOD 3050

Topic	Katahdin SOP CA-605-02	Method 3050, current revision
Apparatus /Materials	1) Digestion performed in 100 mL Griffin beaker or 70 mL polyethylene tube. 2) Graduated disposable plastic cup or 120 mL polyethylene tube used to bring digestate to final volume.	1) Digestion performed in 250 mL Griffin beaker. 2) Volumetric flask used to bring digestate to final volume.
Procedure	1) Digestate volume reduced to 5 to 10 mL prior to filtering. 2) After filtration, the filters are rinsed three times with reagent water. 3) 30% H_2O_2 is added in two 2 mL aliquots and then six 1 mL aliquots.	1) Digestate volume reduced to 5 mL prior to filtering. 2) After filtration, the filters are rinsed twice with reagent water. 3) 30% H_2O_2 is added in one 3 mL aliquot and then seven 1 mL aliquots.

TABLE 3

PREPARATION OF MATRIX SPIKES AND SPIKING SOLUTIONS FOR DIGESTION OF SOLID SAMPLES BY USEPA METHOD 3050

Sample or Solution Name	Component Solution Name	Source of Component	Amount of Component Added per 100 mL Final Volume (mL)
Matrix Spike for ICP-AES	CLPP-SPK-1	Inorganic Ventures	0.10
	CLPP-SPK-INT1	Lab Prepared (see below)	1.00
	CLPP-SPK-INT2	Lab Prepared (see below)	1.00
	1000 mg/L Uranium Std.	High Purity Standards	0.01

Sample or Solution Name	Component Solution Name	Source of Component	Amount of Component Added per 100 mL Final Volume (mL)
CLPP-SPK-INT1	1000 mg/L Se	High Purity Standards	5.0
	1000 mg/L As	High Purity Standards	5.0
	1000 mg/L Pb	High Purity Standards	5.0
	1000 mg/L Cd	High Purity Standards	2.5
	1000 mg/L Sb	High Purity Standards	5.0
	1000 mg/L K	High Purity Standards	10.0
	1000 mg/L Na	High Purity Standards	7.5
	1000 mg/L Mg	High Purity Standards	5.0
	1000 mg/L Ca	High Purity Standards	2.5
CLPP-SPK-INT2	2007ICS-1	Inorganic Ventures	10.0
	1000 mg/L Sr	High Purity Standards	5.0
	1000 mg/L Sn	High Purity Standards	5.0
	10000 mg/L Si	High Purity Standards	5.0

TITLE: ACID DIGESTION OF SOLID SAMPLES BY USEPA METHOD 3050 FOR METALS ANALYSIS BY ICP-AES, ICP-MS

TABLE 4

ELEMENT CONCENTRATIONS IN ICP-AES MATRIX SPIKES AND THEIR COMPONENT SPIKING SOLUTIONS FOR DIGESTION OF SOLID SAMPLES BY METHOD 3050

Element	CONCENTRATION IN SOLUTION, mg/L							
	Matrix Spike (ICP-AES)	CLPP-SPK-1	CLPP-SPK-4	CLPP-SPK-INT1	CLPP-SPK-INT2	QCP-CICV-3 SPK-3	2007 ICS- 1	1000 mg/L U
Aluminum	2.000	2000						
Antimony	0.500		100	50				
Arsenic	0.500		4	50		500		
Barium	2.000	2000						
Beryllium	0.050	50						
Boron	0.500		50		50		500	
Cadmium	0.250		5	25		250		
Calcium	2.500			250				
Chromium	0.200	200						
Cobalt	0.500	500						
Copper	0.250	250						
Iron	1.000	1000						
Lead	0.500		2	50		500		
Magnesium	5.000			500				
Manganese	0.500	500						
Molybdenum	0.300		30		30		300	
Nickel	0.500	500						
Potassium	10.000			1000				
Selenium	0.500		5	50		500		
Silicon	5.230				523		230	
Silver	0.050	50						
Sodium	7.500			750				
Strontium	0.500		50		50			
Thallium	0.500		5	250		500		
Tin	0.500		50		50			
Titanium	1.000		100		100		1000	
Uranium	0.100							1000
Vanadium	0.500	500						
Zinc	0.500	500						

TITLE: ACID DIGESTION OF SOLID SAMPLES BY USEPA METHOD 3050 FOR METALS ANALYSIS BY ICP-AES, ICP-MS

FIGURE 1

PROCEDURE CONDENSATION – METHOD 3050

1. Prepare and print out ACCESS spreadsheet.
2. If performing digestion on a hot plate, rinse 250 mL Griffin beakers and ribbed watch glasses 3 times in acid bath. Then rinse beakers and watch glasses 3 times with laboratory reagent grade water. If perfoming digestion with block digester, polyethylene digestion tubes do not require precleaning.
3. Label digestion vessels (beakers or polyethylene sample tubes) with sample numbers.
4. Weigh 1 to 2 g of well-mixed sample into tared digestion vessels. Record sample weights.
5. Add spike solutions to matrix spike samples.
6. Add 10 mL 1:1 HNO_3 to samples and cover with watch glasses.
7. Reflux for 10 to 15 minutes at $95^{o} \pm 5^{o}$ C. without boiling. Cool samples.
8. Add 5 mL conc. HNO_3, cover beakers, and reflux for 30 minutes.
9. Repeat Step 8 as necessary until digestion is complete.
10. Reduce sample volumes to 5 to 10 mL or heat for 2 hours, whichever occurs first.
11. Cool sample and add 2 mL reagent water and 2 mL 30% H_2O_2. Heat gently until effervescence subsides.
12. Cool sample and add 2 mL 30% H_2O_2. Heat gently until effervescence subsides.
13. Cool samples and add 6 mL of 30% H_2O_2 in 1 mL aliquots. Heat gently until effervescence subsides.
14. Reduce sample volumes to 5 to 10 mL or heat for 2 hours, whichever occurs first.
15. Add 10 mL conc. HCl and reflux for 10 to 15 minutes at $95^{o} \pm 5^{o}$ C.
16. Cool sample and filter into graduated specimen container. Bring to volume with reagent water and transfer to labeled polyethylene bottle.
17. Enter sample weights into ACCESS spreadsheet.

TITLE: ACID DIGESTION OF SOLID SAMPLES BY USEPA METHOD 3050 FOR METALS ANALYSIS BY ICP-AES, ICP-MS

FIGURE 2

EXAMPLE PAGE FROM METALS SAMPLE PREPARATION LOGBOOK

Katahdin Analytical Services, Inc.

Metals Preparation Benchsheet

Reagent Information:
JT Baker HNO3: H12022 JT Baker HCL: H64040 KMG H2O2: 62022 Method: 3050H

LCS / Spike — LCS/Spiking Information:
- ☐ ☑ I.V. CLPP-SPK-1 (ID/Vol): MS1569 / 0.1 mL
- ☐ ☑ CLPP-SPK-INT1 (ID/Vol): MW11404 / 1.0 mL
- ☐ ☑ CLPP-SPK-INT2 (ID/Vol): MW11864 / 1.0 mL
- ☐ ☑ Uranium Spike (ID/Vol): MS1594 / ~~0.01~~ 0.1 mL
- ☐ ☐ CLPP-SPK-4 (ID/Vol): N/A / mL

LCSS: MS1578 Balance ID: Ohaus Galaxy 400 / Mettler AE260

Hot Plate/Block ID: B
Start Temp.: 95 °C
End Temp.: 95 °C
Thermometer ID/Pos.: ALC 8 / 1:4
Fisher Filter Paper: K1167223 23B

REVIEWED
KATAHDIN ANALYTICAL METALS SECTION

Sample ID	Batch ID	Initial Wt/Vol	Initial Units	Final Vol	Final Units	MX	Meth	Anal.	Date	Initial Color	Initial Texture	Final Color	Final Clarity	Artifacts	Bottle
LC2SZG311CS0	ZG311CS0	0.5074	g	0.10	L	SL	IC	AJB	07/31/2009	N/A	N/A	N/A	N/A	N/A	N/A
LCSSZG311CS0	ZG311CS0	0.5034	g		L	SL	IC	AJB	07/31/2009	N/A	N/A	N/A	N/A		
PBSZG311CS0	ZG311CS0	1.00	g		L	SL	IC	AJB	07/31/2009	N/A	N/A	N/A	N/A		
SC4328-001	ZG311CS0	1.79	g		L	SL	IC	AJB	07/31/2009						A
SC4328-001P	ZG311CS0	1.05	g		L	SL	IC	AJB	07/31/2009						
SC4328-001S	ZG311CS0	1.04	g		L	SL	IC	AJB	07/31/2009						
SC4348-001	ZG311CS0	1.35	g		L	SL	IC	AJB	07/31/2009						
SC4348-002	ZG311CS0	1.24	g		L	SL	IC	AJB	07/31/2009						
SC4348-003 7/31/09	ZG311CS0	1.11	g		L	SL	IC	AJB	07/31/2009						
SC4357-001 AJB	ZG311CS0	1.46	g		L	SL	IC	AJB	07/31/2009						
SC4357-~~002~~ 002	ZG311CS0	1.17	g		L	SL	IC	AJB	07/31/2009						
SC4357-~~003~~ 003	ZG311CS0	1.52	g	0.10	L	SL	IC	AJB	07/31/2009						A

AJB 7/31/09

Digestion performed by: AJB Date: 7/31/09

Page: ZG104 Revision: 00

TITLE: ACID DIGESTION OF SOLID SAMPLES BY USEPA METHOD 3050 FOR METALS ANALYSIS BY ICP-AES, ICP-MS

FIGURE 3

EXAMPLE CERTIFICATE OF ANALYSIS FOR SOLID REFERENCE MATERIAL

ENVIRONMENTAL RESOURCE ASSOCIATES®
The Industry Standard™

M51475

DataPacK™
Lot No. D051-540

Trace Metals in Soil
Catalog No. 540

Certification

Method 3050 HNO3, H2O2, HCl Parameter	Total Concentration [1] (mg/Kg)	Certified Value [2] (mg/Kg)	Performance Acceptance Limits™ [3] (mg/Kg)
aluminum	55600*	7870	4630 - 11100
antimony	160	70.5	D.L. - 149
arsenic	316	289	234 - 344
barium	869	211	174 - 247
beryllium	60.9	54.4	45.2 - 63.6
boron	129	91.3	58.8 - 124
cadmium	114	101	82.9 - 119
calcium	9750*	3680	2970 - 4390
chromium	249	224	180 - 268
cobalt	113	101	82.7 - 119
copper	94.9	88.0	73.3 - 103
iron	24400*	15700	6610 - 24900
lead	184	158	129 - 187
magnesium	3780*	2260	1760 - 2750
manganese	703	420	343 - 497
mercury	5.32	5.18	3.42 - 6.87
molybdenum	80.2	69.6	55.5 - 83.7
nickel	137	120	99.1 - 141
potassium	33000*	3000	2200 - 3800
selenium	146	130	101 - 159
silver	127	104	68.9 - 139
sodium	15600*	1080	692 - 1470
strontium	326	113	90.5 - 135
thallium	106	94.0	72.8 - 115
tin	175	149	104 - 194
titanium	3100*	284	116 - 453
vanadium	151	111	85.1 - 137
zinc	311	272	215 - 329

Method 3050 HNO3, H2O2 Parameter	Total Concentration [1] mg/Kg	Certified Value [2] mg/Kg	Performance Acceptance Limits™ [3] mg/Kg
aluminum	55600*	7380	4440 - 10300
antimony	160	75.2	D.L. - 198
arsenic	316	284	225 - 343
barium	869	217	177 - 257
beryllium	60.9	53.6	42.7 - 64.5
boron	129	89.5	58.9 - 120
cadmium	114	103	83.6 - 122
calcium	9750*	3540	2800 - 4270
chromium	249	224	172 - 275
cobalt	113	101	82.0 - 120
copper	94.9	85.5	70.4 - 100
iron	24400*	12500	5480 - 19500
lead	184	162	132 - 192
magnesium	3780*	2160	1650 - 2670
manganese	703	415	330 - 500
mercury	5.32	5.18	3.42 - 6.87
molybdenum	80.2	68.8	52.7 - 84.9
nickel	137	119	98.5 - 140
potassium	33000*	2840	2160 - 3520
selenium	146	135	104 - 166
silver	127	107	49.8 - 164
sodium	15600*	1010	709 - 1310
strontium	326	111	89.0 - 133
thallium	106	99.3	76.8 - 122
tin	175	148	70.6 - 225
titanium	3100*	283	104 - 463
vanadium	151	104	70.5 - 138
zinc	311	275	222 - 328

TITLE: TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010

Prepared By: George Brewer Date: 7/98

Approved By:

Group Supervisor: George Brewer Date: 01/23/01

Operations Manager: John C. Burton Date: 1/23/01

QA Officer: Deborah J. Nadeau Date: 1·23·01

General Manager: Dennis P. Hufnagle Date: 1/25/01

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01 6010B	Format changes, added pollution prevention, expanded procedure and QC sections. Added tables.	DN	1·23·01	1/23/01
02 6010B	Calibration begins with analysis of S0 (Cal. blank) followed by S1 (Mixed Cal. Std.) Changes to section 7.5 and Table 8 to reflect this. Made changes to element concs. in Tables 3, 4, 5, 6 to reflect current practices.	DN	10·21·02	10·21·02
03 6010B	Added MN-IEC to standards run. Changed frequency of LRS. Changed concentration of HNO_3 in calibration blank. CRI changed from three separate solutions to one. Changed CRI vendor.	MRC	04.15.04	04.15.04
04	Updated ICV, CCV, ICB, PQL Chk std. PBW, PBS, MS & MSD acceptance criteria updated Table 1	LAD	05/06	05/06
05	Updated Tables 3, 4, 5, 6 and 7 with current standard concentrations and prep. Updated Table 1 with current practices including NAVY audit findings. Updated sections 2, 7.2, 7.6 and Table 1 with new ICP information. Updated Table 8 with current sequence requirements.	LAD	07/07	07/07

TITLE: TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
06	Added hardness definition and calculation (APP. 1)	LAD	09/07	09/07
07	Updated Summary to reflect new ICP functions. Removed ICP set-up updated tables to reflect changes in standard concentrations and preparation	LAD	11/08	11/08
08	Updates to Sections 8 and 10, Tables 1 and 2 to reflect changes from 6010B to 6010C. Added LLQC information and criteria to Sect. 8 and Table 1. Added criteria to analyze PQL standard at the beginning and END of each run.	LAD	02/09	02/09
09	Updated Sections 8, 9, 10 and Table 1 for compliance with DoD QSM version 4.1.	LAD	08/09	08/09

TITLE: **TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010**

Please acknowledge receipt of this standard operating procedure by signing and dating both of the spaces provided. Return the bottom half of this sheet to the QA Department.

I acknowledge receipt of copy ___ of document **SOP CA-608-09**, titled **TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010.**

Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy ___ of document **SOP CA-608-09**, titled **TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010.**

Recipient: _____ Date: _____

500 mg/L); solution ICSAB contains interferents at the same concentrations as well as analytes at low (20 mg/L or less) concentrations.

ICV - Initial Calibration Verification - A standard made from a source independent from the calibration standards and with analyte concentrations different from those in the CCV; used to verify the accuracy of the instrument calibration.

IDL - Instrument Detection Limit - The lowest concentration of an analyte that can be determined with 99% confidence.

LOD – Limit of Detection – An estimate of the minimum amount of a substance that an analytical process can reliably detect. An LOD is analyte and matrix-specific and is used for DoD QSM acceptance criteria.

LOQ – Limit of Quantitation.- The minimum concentration of a target analyte that produces a quantitative result within specified limits of precision and bias.

LCS - Laboratory Control Sample - A standard or solid reference material that has been brought through the sample preparation process.

LRS - Linear Range Standard - A high-concentration standard used to determine the upper reporting limit of the ICP calibration.

PB - Preparation Blank - Reagent water that has been brought through the sample preparation process.

PQL - Practical Quantitation Limit - The lowest concentration of an analyte that is routinely reported by the laboratory; nominally three to five times the IDL.

Matrix Spike - An aliquot of a sample to which a known amount of analyte has been added before digestion.

Serial Dilution - The dilution of a sample by a factor of five. When corrected by the dilution factor, the measured analyte concentrations of the diluted sample should agree with those of the original undiluted sample within specified limits. Serial dilution may reflect the influence of interferents.

Hardness – The sum of the calcium and magnesium concentrations, both expressed as calcium carbonate, in mg/L.

1.2 Responsibilities

This method is restricted to use by, or under the supervision of, analysts experienced in ICP analysis by EPA Method 6010. Each analyst must demonstrate and document their ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, current revision, "Personnel Training & Documentation of Capability".

It is the responsibility of all Katahdin technical personnel involved in ICP analysis by Method 6010 to read and understand this SOP, to adhere to the procedures outlined, and to properly document their data in the appropriate lab notebook. Any deviations from the test or irregularities with the samples should also be recorded in the lab notebook and reported to the group supervisor or designated qualified data reviewer responsible for this data.

It is the responsibility of the Group Supervisor to oversee that members of their group follow this SOP, to ensure that their work is properly documented and to initiate periodic review of the associated logbooks.

1.3 Safety

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDSs for all the materials used in this procedure.

Each qualified analyst or technician must be familiar with Katahdin Analytical Environmental Health and Safety Manual including the Katahdin Hazardous Waste Plan and must follow appropriate procedures. These include the use of appropriate personal protective equipment (PPE) such as safety glasses, gloves and lab coats when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location of all safety equipment. Each analyst shall receive a safety orientation from their Department Manager, or designee, appropriate for the job functions they will perform.

Samples, sample digestates, standards, and other reagents used in ICP analysis may contain high concentrations of acids and toxic metals. Safety glasses should be worn when changing or adjusting argon tanks.

1.4 Pollution Prevention/Waste Disposal

Whenever possible, laboratory personnel should use pollution prevention techniques to address their waste generation. Refer to the current revision of the Katahdin Hazardous Waste Management Program for further details on pollution prevention techniques.

Wastes from ICP analysis should be disposed of in a manner appropriate to the hazards they present. Wastes generated during the preparation of samples must be disposed of in accordance with the Katahdin Analytical Environmental Health and Safety Manual I and SOP SD-903, "Sample Disposal," current revision. Expired

standards are lab packed, placed in the Katahdin hazardous waste storage area, and disposed of in accordance with this SOP.

2.0 SUMMARY OF METHOD

This method describes multielemental determinations by ICP-AES using simultaneous optical systems and radial and axial viewing of the plasma. The basis of the method is the measurement of atomic emission from sample atoms entrained in an argon plasma by optical spectroscopy. Samples are nebulized and the aerosol that is produced is transported to the plasma torch where thermal excitation of entrained atoms and ions occurs. Characteristic atomic-line and ionic-line emission spectra are produced by a radio-frequency inductively coupled plasma (ICP). The spectra are dispersed by a grating and the intensities of the emitted lines are monitored by a solid state charge injection device (CID) camera system. Photocurrents from the CID camera system are measured by a computer system. Element concentrations of unknown samples are quantitated by comparison of sample emission intensities to emission intensities of standards of known concentration. A background correction technique is used to compensate for variable background contribution to the determination of trace elements. Background is measured adjacent to the analyte lines on samples during analysis. The position selected for the background intensity measurement, on either or both sides of the analytical line, has been determined by the complexity of the spectrum adjacent to the analytical line. The position used must be relatively free of spectral interference and must reflect the same change in background intensity as occurs at the analyte wavelength. Physical interferences are corrected through the use of an internal standard (yttrium) that is automatically added to all samples and standards prior to nebulization. The possibility of additional interferences (noted in section 3) must be recognized and appropriate corrections applied.

3.0 INTERFERENCES

Several types of interference effects may contribute to inaccuracies in the determination of trace elements. They can be summarized as spectral interferences, physical interferences, and chemical interferences.

Spectral interferences can be categorized as 1) overlap of a spectral line from another element; 2) unresolved overlap of molecular band spectra; 3) background contribution from continuous or recombination phenomena; and 4) background from stray light from the line emission of high concentration elements. The first of these effects is compensated by utilizing the computer correction of raw data, requiring the monitoring and measurement of the interfering element (interelement correction). The second effect is controlled by choosing analytical wavelengths that are free from overlapping molecular emission spectra. The third and fourth effects are usually compensated by a background correction adjacent to the analyte line. Uncorrected spectral interferences may be detected through examination of serial dilution and matrix spike data.

Physical interferences are generally considered to be effects associated with sample nebulization and transport processes. Such properties as changes in viscosity and surface tension can cause significant inaccuracies, especially in samples that may contain high dissolved solids and/or acid concentrations. Matrix matching of standards and samples and the use of a peristaltic pump may lessen these interferences. If these types of interferences are operative, they must be reduced by dilution of the sample and/or utilization of standard addition techniques. Another problem that can occur from high dissolved solids is salt buildup at the tip of the nebulizer. This affects aerosol flow rate causing instrumental drift. Regular cleaning of nebulizer tips and dilution of samples with high dissolved solids contents are used to control this problem. Physical interferences are also corrected by this laboratory through the use of an internal standard. Uncorrected physical interferences may be detected through examination of serial dilution and matrix spike data. Instrument drift caused by the salting up of nebulizer tips may also be detected by looking for oriented drift in calibration verification standards analyzed regularly throughout the run.

Chemical interferences are characterized by molecular compound formation, ionization effects, and solute vaporization effects. Normally these effects are not pronounced with the ICP technique; however, if observed they can be minimized by careful selection of operating conditions (i.e., incident power, observation position, etc.), by matrix matching, and by standard addition procedures. These types of interferences can be highly dependent on matrix type and the specific analyte element. Uncorrected chemical interferences may be detected through examination of serial dilution data.

4.0 APPARATUS AND MATERIALS

4.1 Computer-controlled inductively-coupled plasma atomic emission spectrometer (plasma viewed radially or axially) equipped for internal standardization, and capable of performing automatic background correction and interelement correction. For more information refer to the current revision of Katahdin SOP CA-632, "Operation and Maintenance of the Thermo ICAP 6500 ICP Spectrophotometer".

4.2 Computer-controlled autosampler.

4.3 Argon gas supply – high purity.

4.4 Volumetric glassware of suitable precision and accuracy.

4.5 Automatic pipets of suitable precision and accuracy. Calibrated Eppendorf Reference pipets and Finn digital pipets are appropriate.

Refer to the appropriate instrument-specific SOP for additional required equipment.

5.0 REAGENTS

5.1 Hydrochloric acid, concentrated (HCl) – spectroscopic grade.

5.2 Nitric acid, concentrated (HNO_3) – spectroscopic grade.

5.3 Reagent water, trace metals free.

5.4 Calibration blank – reagent water containing HCl (5% v/v) and HNO_3 (5% v/v). Calibration blank solution is prepared in large volumes (up to 20 liters) and stored in a carboy. Calibration blank solution is used in establishing the analytical curve, and in all initial and continuing calibration blank determinations. This solution is also used to flush the system between standards and samples. Intermediate and working standards are prepared by diluting stock standards and intermediate standards with calibration blank solution so that all standards and blanks are acid matrix-matched to sample digestates.

5.5 Single element and multielement stock standard solutions – purchased standards prepared from high purity salts or metals, and supplied by the vendors with certificates of purity and analysis. Refer to Tables 3 and 4 for a listing of stock standards required, and to Table 7 for element concentrations in stock standards.

5.6 Intermediate standard solutions – laboratory-prepared multielement standards that are used in the subsequent preparation of working standards. Refer to Table 4 for a listing of intermediate standards required and for preparation instructions. Refer to Table 6 for element concentrations in intermediate standards.

5.7 Working standard solutions – laboratory-prepared multielement standards that are used to calibrate the instrument and to perform all necessary QC checks. Refer to Table 3 for a listing of working standards and for preparation instructions. Refer to Table 5 for element concentrations in working standards.

5.8 5 mg/L yttrium internal standard solution – add 0.5 mL 10000 mg/L yttrium stock standard to a 1000 mL volumetric flask half filled with calibration blank solution. Bring to volume with calibration blank solution.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

Samples to be analyzed for trace metals by ICP should be collected and preserved as described in the following table.

Matrix	Container [1]	Collection Volume/ Weight	Preservation/ Treatment	Holding Time
Aqueous (total)	P, G	250 mL	HNO_3 to pH < 2	6 months
Aqueous (dissolved)	P, G	250 mL	Filter, HNO_3 to pH < 2	6 months
Solid	P, G	10 g	Cool, 4°C	6 months

[1] P = polyethylene or , G = glass

7.0 PROCEDURES

7.1 Begin by following the startup and calibration instructions provided in the current revision of Katahdin SOP CA-632, "Operation and Maintenance of the Thermo ICAP 6500 ICP Spectrophotometer"

7.2 Analysis must proceed in the sequence described in Table 8 to ensure that all necessary quality control samples are analyzed at the appropriate frequencies. A minimum of two replicate integrations is required for all standards and samples. Analysis always begins with the analysis of a calibration blank solution (S0) followed by analysis of a multielement calibration standard (S1 in Table 3) to calibrate the instrument. The system is flushed with calibration blank for two minutes between each sample and standard, and each sample and standard is aspirated for one minute prior to the beginning of emission measurements.

7.3 Analysis continues with analysis of the initial calibration verification standard (ICV) and the initial calibration blank (ICB) to verify the accuracy of the calibration. Refer to Section 8 and Table 1 for additional information.

7.4 A continuing calibration verification standard (CCV) and a continuing calibration blank (CCB) must be analyzed at the beginning of the run, after every ten samples, and at the end of the run to verify the continued accuracy of the calibration. Refer to Section 8 and Table 1 for additional information.

7.5 Interference check standard solutions (ICSA and ICSAB) must be analyzed at the beginning, end, and at periodic intervals (4-6 hours, 30-40 analytical samples) throughout the sample run to verify the accuracy of the IEC factors. Refer to Section 8 and Table 1 for additional information.

7.6 A practical quantitation limit standard (PQL) must be analyzed at the beginning of each run to determine the accuracy of the calibration at the reporting limit. Refer to Section 8 and Table 1 for additional information.

7.7 All sample analytical results for a particular element that are bracketed (preceded or followed) by failing results in a QC sample (ICV, ICB, CCV, CCB, ICSA, or ICSAB)

for that element must not be reported. The sample must be reanalyzed for the element in question.

7.8 All samples that exceed the linear dynamic range must be diluted and reanalyzed. This includes samples with interfering elements that exceed the calibration ranges, because accurate quantitation of interfering elements is necessary for reliable interelement correction. For example, if a sample has been submitted to the laboratory for lead analysis, and the measured aluminum concentration of that sample exceeds the calibration range for aluminum, it must be diluted sufficiently to bring aluminum within the linear dynamic range and the lead result must be reported from that dilution analysis.

7.9 If dilutions of digested samples are performed, the measured element concentrations must be multiplied by the dilution factor prior to reporting. This is accomplished automatically by entering the dilution factor in the autosampler table prior to initiation of analysis.

7.10 All analyses are performed using yttrium as an internal standard to compensate for enhancement or depression of the analytical signal due to matrix effects. Yttrium solution is pumped at a constant rate through one channel of the peristaltic pump. Samples and standards are pumped through a second channel of the pump. The tubing carrying the internal standard is connected to the tubing carrying samples and standards downstream from the pump, and mixing of the two streams is accomplished in a mixing coil downstream from the connection, prior to nebulization. For each sample or standard, the computer that controls the spectrometer divides the detected emission signal for each element by the detected yttrium emission signal prior to quantitation, thus normalizing all emission signals to that of yttrium.

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

USEPA Method 6010 requires the laboratory to perform specific quality control checks to assess laboratory performance and data quality. Minimum frequencies, acceptance criteria, and corrective actions for these control checks are tabulated in Table 1 and are described below. Table 1 criteria are intended to be guidelines for analysts. The table does not cover all possible situations. If any of the QC requirements are outside the recovery ranges listed in Table 1, all associated samples must be evaluated against all the QC. In some cases data may be reported, but may be reanalyzed in other cases. Making new reagents and standards may be necessary if the standardization is suspect. The corrective actions listed in Table 1 may rely on analyst experience to make sound scientific judgments. These decisions are based on holding time considerations and client and project specific Data Quality Objectives. The supervisor, Operations Manager, and/or Quality Assurance Officer may be consulted to evaluate data. Some samples may not be able to be reanalyzed within hold time. In these cases "qualified" data with narration may be advisable after consultation with the client.

In some cases the standard QC requirements listed in this section and in Table 1 may not be sufficient to meet the Data Quality Objectives of the specific project. Much of the work performed at the lab is analyzed in accordance with specific QC requirements spelled out in a project specific Quality Assurance Project Plan (QAPP) or in a program specific Quality Systems Manual (QSM). The reporting limits, acceptance criteria and/or corrective actions may be different than those specified in this SOP. In these cases the appropriate information will be communicated to the Department Manager and/or senior chemists before initiation of the analyses so that specific product codes can be produced for the project. In addition, the work order notes for each project will describe the specific QAPP or QSM to be followed.

INITIAL DEMONSTRATION OF PERFORMANCE

8.1 Instrument detection limits (IDL) are determined quarterly for each analyte analyzed on each instrument. This determination requires seven replicate analyses of a reagent water spiked at 3-5 times the anticipated detection limit for each analyte, performed on three non-consecutive days. The standard deviation of the 21 analyses is multiplied by three to obtain the IDL. For more information on performing IDL determinations, refer to the current revision of Katahdin SOP QA-806.

8.2 Method detection limits (MDL) are determined annually for each analyte analyzed on each instrument. This determination requires at least seven replicate digestions and analyses of a reagent water spiked at 3-5 times the anticipated MDL for each analyte. MDLs differ from IDLs in that the seven replicates are digested prior to analysis, and they may be analyzed on a single day. The standard deviation of the 7 (or more) replicate analyses is multiplied by the Student's t-value to obtain the MDL. For more information on performing MDL determinations, refer to the current revision of Katahdin SOP QA-806.

8.3 Limits of Detection (LOD) are used when evaluating data using DoD QSM. The LOD is established by spiking a quality system matrix at 2-3 times the detection limit for a single analyte standard and 1-4 times the detection limit for a multi-analyte standard. The LOD must be verified quarterly. For more information on performing LOD determinations, refer to the current revision of Katahdin SOP QA-806.

8.4 Limits of Quantitation (LOQ) are used when evaluating data using DoD QSM. The LOQ must be above the LOD.

8.5 A Lower Limit of Quantitation Check (LLQC) sample must be prepared and analyzed annually or on an as-needed basis to confirm the laboratory's Practical Quantitation Limits (PQLs). The LLQC sample is equivalent to the PQL standard (Section 8.10) but is carried through the entire sample preparation and analysis process. Element recoveries for the LLQC sample must fall within 70% to 130% of the expected concentrations to confirm the previously established PQLs.

8.6 The upper limit of the linear dynamic range (LDR) must be established for each wavelength utilized. It must be determined from a linear calibration prepared in the normal manner using the established analytical operating procedure for the instrument. The LDR should be determined by analyzing succeedingly higher standard concentrations of the analyte until the observed analyte concentration differs by no more than 10% from the stated concentration of the standard. Determined LDRs must be documented and kept on file. The LDR which may be used for the analyses of samples should be judged by the analyst from the resulting data. Determined sample analyte concentrations that are greater than the determined upper LDR limit must be diluted and reanalyzed. The LDRs should be verified **every six months** or whenever, in the judgment of the analyst, a change in analytical performance caused by either a change in instrument hardware or operating conditions would dictate they be redetermined.

8.7 The alkali and alkaline earth metals may have non-linear response curves due to ionization and self-absorption effects. These curves may be used for quantitation of samples if the effective range is checked and if the second order curve fit has a correlation coefficient of 0.998 or better. Third order fits are not acceptable. Non-linear response curves must be revalidated and recalculated every six months.

ANALYTICAL RUN QC SAMPLES

8.8 An Initial Calibration Verification (ICV) solution is analyzed after the initial calibration to check calibration accuracy. The ICV solution is prepared by combining compatible elements from a standard source different than that of the calibration standard and at concentrations within the linear working range of the instrument. The results of the ICV must fall within 90% to 110% of the expected values. If the ICV fails, result for the failing elements may not be reported from the run unless the ICV recovery is greater than 110% and the sample result is less than the PQL.

No results may be accepted for failing elements if DoD QSM acceptance criteria are being used.

8.9 Continuing Calibration Verification (CCV) solutions are analyzed after the initial calibration, after every ten samples, and at the end of the analytical run. The CCV solution is prepared using the same standards used for calibration at concentrations near the mid-point of the calibration curve. Results of the CCVs must fall within 90% to 110% of the expected values. If a CCV fails, results for the failing elements may not be reported from the run unless the CCV recovery is greater than 110% and the sample result is less than the PQL (less than reporting limit for DoD QSM). Also, for failing elements, all samples analyzed after the last passing CCV must be reanalyzed.

8.10 Calibration blank solution is analyzed after each ICV and CCV. A calibration blank that is analyzed after the ICV is called an Initial Calibration Blank (ICB). A

calibration blank that is analyzed after a CCV is called a Continuing Calibration Blank (CCB). The absolute values of results of ICBs and CCBs must be less than the Practical Quantitation Level (PQL) for each element. If an ICB or a CCB fails, results for the failing elements may not be reported from the run until the problem is corrected and a passing ICB or CCB has been analyzed, with the following exception. If the result for a CCB or ICB is greater than the PQL, sample results that are less than the PQL or greater than or equal to ten times the measured CCB concentration may be reported. Also, for failing elements, all samples analyzed after the last passing CCB must be reanalyzed, with the exception noted above.

If DoD QSM acceptance criteria are being used, the absolute values of results of ICBs and CCBs must be less than the Limit of Detection (LOD). If an ICB or a CCB fails, results for the failing elements may not be reported from the run until the problem is corrected and a passing ICB or CCB has been analyzed.

8.11 Interference check solutions ICSA and ICSAB (refer to Section 1.1) are analyzed at the beginning of each run to verify interelement correction factors and background correction. ICSA contains interferent elements (Al, Ca, Fe, and Mg) only, at concentrations of 200 mg/L to 500 mg/L. Results for interfering elements in the ICSA must fall within 80% to 120% of the expected values. Results for unspiked elements in ICSA must fall within ± PQL if the PQL is greater than 0.01 mg/L, within ± 2xPQL if the PQL is less than or equal to 0.01 mg/L. If DoD QSM acceptance criteria are being used, the absolute value of unspiked elements must be less than the LOD. ICSAB contains interferent elements at concentrations of 200 mg/L to 500 mg/L, and analytes at concentrations of 20 mg/L or less. Results for all elements (interferents and analytes) in ICSAB must fall within 80% to 120% of the expected values. If the ICSA or ICSAB fails, results for the failing elements may not be reported from the run until the problem is corrected and a passing ICSA or ICSAB has been analyzed.

8.12 A Practical Quantitation Limit (PQL) Check Standard or low level continuing calibration verification (LLCCV) is analyzed at the beginning (after the ICV and ICB samples) and at the end of each run. Element concentrations in this solution are at the laboratories practical quantitation limit. Element recoveries for the PQL check Standard must fall between 70-130% of the expected values. If the PQL Check Standard fails, the results for the failing elements may not be reported from the run, unless the PQL Check Standard recovery is greater than 130% and the samples results are less than the PQL.

If DoD QSM acceptance criteria are being used, recoveries must fall between 80-120%. If the PQL Check Standard fails, the results for the failing elements may not be reported from the run.

PREPARATION BATCH QC SAMPLES

8.13 Each digestion batch of twenty or fewer samples will contain a preparation blank and a laboratory control sample. Each batch will also contain one or more of the following QC samples: laboratory control sample duplicate, sample duplicate, matrix spike sample or matrix spike sample duplicate.

8.14 A preparation blank (PBW or PBS), consisting of reagent water carried through the same process as associated samples, is prepared with each digestion batch of twenty or fewer samples. The results of preparation blanks must be less than the Practical Quantitation Level (PQL) for each element. For DoD QSM acceptance criteria the results must be less than ½ the PQL except for common contaminants which must be less than the PQL. If a preparation blank fails, results for the failing elements may not be reported from the digestion batch, and all associated samples must be redigested, with the following exception. If the result for a preparation blank is greater than the PQL (greater than ½ PQL for DoD), associated sample results that are less than the PQL (less than ½ PQL for DoD) or greater than or equal to ten times the measured preparation blank concentration may be reported.

8.15 A laboratory control sample (LCS), consisting of spiked reagent water or a solid reference material carried through the same process as associated samples, is prepared with each digestion batch of twenty or fewer samples. Results for laboratory control samples must fall within 80% to 120% of the expected value, unless vendor-supplied limits (for solid reference materials) or laboratory-generated statistical limits are available. If a laboratory control sample fails, results for the failing elements may not be reported from the digestion batch, and all associated samples must be redigested with the following exception. If the LCS fails high, sample results less than the PQL may be reported.

If DoD QSM acceptance criteria are being used, recovery for solid matrix samples must fall between 80% to 120% except for Ag, which must fall between 75% and 120%. Results may not be reported without a valid LCS and will be qualified and explained if reanalysis cannot be performed.

SAMPLE MATRIX QC SAMPLES

8.16 Matrix spiked duplicate samples are prepared at a minimum frequency of one per digestion batch. The recovery for each element in a spiked sample or spiked duplicate sample must fall within 75% to 125% of the actual value if the result for the unspiked sample is less than four times the amount of spike added. If one or both spike recoveries fail, the associated sample result must be flagged on the report of analysis. If DoD QSM acceptance criteria are being used, recoveries must be the same as stated for laboratory control samples.

The relative percent difference between sample duplicate, matrix spiked duplicate or LCS duplicate, is calculated as follows:

$$RPD\ (\%) = \frac{|D_1 - D_2|}{(|D_1 + D_2|)/2} \times 100$$

Where: D_1 = sample result
D_2= duplicate sample result

A control limit of 20% RPD is applied to duplicate analysis if the original sample result is greater than 50X the IDL. If the matrix spike duplicate analysis fails, the associated sample result must be flagged on the report of analysis.

8.15 A serial dilution is analyzed to check for chemical or physical interferences. If the analyte concentration of a sample is sufficiently high (minimally, 50 x IDL or 50 x LOQ if using DoD QSM acceptance criteria), the measured concentration of a serial dilution (1:5 dilution) of the sample should agree within 90% to 110% of the original determination. The percent difference between the original sample and the serial dilution should be calculated as follows:

$$Difference\ (\%) = \frac{|L-S|}{S} * 100\%$$

where:
L = Serial dilution result (corrected for dilution)
S = Original sample result

If the serial dilution analysis fails, a matrix interference should be suspected. The associated sample result should be flagged on the report of analysis or the sample should be reanalyzed at dilution to eliminate the interference.

For DoD QSM samples a Post-digestion Spike (PDS) addition must be performed if the serial dilution is not within acceptance criteria.

8.16 Post-digestion Spike (PDS) additions must be performed for DoD QSM samples if the serial dilution is not within acceptance criteria or if the analyte concentrations in all samples are less than 50x the LOD. The spike addition should produce a concentration that is between 10 and 100x the LOQ. The recovery of the PDS must be within 75-125%. If the PDS fails, all samples must be run by method of standard additions or appropriately flagged.

9.0 METHOD PERFORMANCE

The method detection limit (MDL) and the limit of detection (LOD) are defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. The MDLs and LODs are determined annually per

type of instrument and filed with the Metals Supervisor and with the QAO. Refer to the current revision of Katahdin SOP QA-806, Method Detection Limit and Instrument Detection Limit Studies, for procedures on determining the MDL.

Refer to the current revision of Method 6010 for other method performance parameters and requirements.

10.0 APPLICABLE DOCUMENTS/REFERENCES

Department of Defense Quality Systems Manual for Environmental Laboratories (DOD QSM), Version 4.1, 04/22/09

Katahdin SOP CA-101, Equipment Maintance

Katahdin SOP QA-806, Method Detection Limit and Instrument Detection Limit Studies

The National Environmental Laboratory Accreditation Conference (NELAC) Standards, June 2003

Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, USEPA SW846, 3rd Edition, Final Updates I, II, IIA, IIB, III, IIIA, IIIB and IV, February 2007, Method 6010C.

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TABLE 1

QC REQUIREMENTS

Method	QC Sample	Minimum Frequency	Acceptance Criteria	Corrective Action
USEPA 6010	Initial Calibration, minimum 1 point plus a calibration blank.	Daily prior to sample analysis.	Correlation coefficient (r) ≥ 0.998	Recalibrate
	Initial Calibration Verification (ICV), prepared from a second source.	Before beginning a sample run.	Recovery within ± 10% of true value.	1) Do not use results for failing elements unless the ICV > 110% and the sample < the PQL. 2) Investigate and correct 3) DoD: No samples may be run until calibration is verified
	Initial Calibration Blank (ICB)	Immediately after the ICV.	Absolute value of ICB < PQL. DoD: no analytes detected > LOD	1) Do not use results if ≥ PQL and 10x< CCB level. 2) Investigate and correct problem. 3) DoD: Do not report results without valid ICB
	Continuing Calibration Verification (CCV)	At beginning of run, after every 10 samples, and at end of run.	Recovery within ± 10% of true value.	1) Do not use results for failing elements unless the CCV > 110% and the sample < the PQL. 2) Investigate and correct problem. 3) DoD: Do not use results for failing elements unless CCV > 110% and the sample is below reporting limit.
	Continuing Calibration Blank (CCB)	After every 10 samples and at end of the run.	Absolute value of CCB < PQL. DoD: no analytes detected > LOD	1) Do not use results if ≥ PQL and < 10x CCB level. 2) Investigate and correct problem. 3) DoD: Do not report results without valid CCB
	Practical Quantitation Level Check Standard (PQL) (LLCCV)	At beginning and end of run.	Recovery within ± 30% of true value. DoD: Recovery within 20% of true value	1) Do not use results for failing elements unless the ICV > 110% and the sample < the PQL. 2) Investigate and correct problem. 3) DoD: No samples may be run without valid PQL
	Interference Check Solution A (ICSA)	At beginning and end of run.	For Al, Ca, Fe, and Mg, recovery within ± 20% of true value. For analytes not spiked, ± PQL, or, if PQL ≤ 0.01 mg/L, ± 2x PQL. DoD: Absolute value for all non-spiked analytes < LOD, unless verified trace impurity	1) Do not use results for failing elements. 2) Investigate and correct problem.

TABLE 1 (cont.)

QC REQUIREMENTS

Method	QC Sample	Minimum Frequency	Acceptance Criteria	Corrective Action
USEPA 6010 (cont.)	Interference Check Solution AB (ICSAB)	At beginning and end of run.	Recovery of each analyte within ± 20% of true value.	1) Do not use results for failing elements. 2) Investigate and correct problem.
	Preparation Blank (PBW/PBS)	One per digestion batch of 20 or fewer samples.	Less than PQL. DoD: No analytes detected >1/2 RL and greater than 1/10 the amount measured in any sample or 1/10 the regulatory limit. (whichever is greater) For common contaminants, no analytes detected >RL	1) Investigate source of contamination. 2) Redigest and reanalyze all associated samples if sample concentration ≥ PQL and <10x the blank concentration. 3) DoD: Report flagged results for samples greater than 10x target analyte in blank. Report results for target analytes less than ½ RL or less than RL for common contaminants. Redigest and reanalyze all other associated samples.
	Laboratory Control Sample (LCSW/LCSS)	One per digestion batch of 20 or fewer samples.	Recovery within ± 20% of true value, unless vendor-supplied or statistical limits have been established. DoD: Recovery within ± 20% of true value; for Ag in solid matrix, recovery within 75 – 120%.	1) Investigate source of problem. 2) Redigest and reanalyze all associated samples. 3) DoD: Flag specific analytes if samples cannot be reanalyzed.
	Matrix Spike Sample (S)	One per digestion batch of 20 or fewer samples.	Recovery ± 25% of true value, if sample < 4x spike added. DoD: Use QC acceptance criteria specified for LCS.	1) Flag results. 2) DoD: Perform additional project specific quality control tests.
	Matrix Spike Duplicate Sample (P) or sample duplicate	One per digestion batch of 20 or fewer samples.	Recovery ± 25% of true value, if sample < 4x spike added. DoD: Use QC acceptance criteria specified by DoD for LCS. RPD ≤20% for duplicate spikes and sample duplicates.	1) Flag results. 2) DOD: Perform additional project specific quality control tests.

TABLE 1 (cont.)

QC REQUIREMENTS

Method	QC Sample	Minimum Frequency	Acceptance Criteria	Corrective Action
USEPA 6010 (cont.)	Serial Dilution (L)	One per digestion batch.	If original sample result is at least 50x IDL (LOQ if DoD), 5-fold dilution must agree within ± 10% of the original result. Flag result or dilute and reanalyzed sample to eliminate interference	DoD: Perform post digestion spike addition (PDS)
	Post-Digestion Spike Sample (A)	When dilution test fails or analyte concentration in all samples <50x LOD	Recovery within ± 25%.	Run associated samples by method of standard addition or flag results.
	Instrument Detection Limit (IDL) Study	Quarterly.	IDL < MDL PQL > 2-3 * the IDL	1) Repeat IDL study. 2) Raise PQL.
	Method Detection Limit (MDL) Study	Refer to KAS SOP QA-806, "Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications", current revision.		
	Lower Limit of Quantitation Check (LLQC) Sample	Digest and analyze annually or as needed to confirm PQLs	0% - 130% of true value	Re-evaluate PQLs
	Linear Range Study	Every six months	Run succeedingly higher stds until recovery not within ± 10%. Use highest passing concentration as upper limit of linear range.	Only accept data to highest passing concentration until next linear range study.
	Limit of Detection (LOD) Determination	Quarterly	LOD = 1-4X MDL	Repeat LOD Determination
	Limit of Quantification (LOQ) Determination	Quarterly	LOQ > LOD	

TABLE 2

SUMMARY OF METHOD MODIFICATIONS

TOPIC	KATAHDIN SOP CA-608-09	METHOD 6010, current revision
Apparatus/Materials		
Reagents		
Sample preservation/ handling		
Procedures		
QC - Spikes		
QC - LCS		
QC - Accuracy/Precision		
QC - MDL		
QC - Calibration Blanks	Acceptance criteria employed for 6010: ± PQL	Acceptance criteria stated in 6010: less than 10% of PQL

TITLE: **TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010**

TABLE 3

PREPARATION OF CALIBRATION AND QUALITY CONTROL STANDARDS

Sample or Solution Name	Component Solution Name	Source of Component	Amount of Component Added per 100 mL Final Volume (mL)
Calibration Standard (STD1 or S1)	ICP- intermediate Standard	Lab Prepared (see Table 4)	10.0
	QCS 26	High Purity Standards	1.0
Initial Calibration Verification (ICV)	QCP-CICV-3	Inorganic Ventures	0.96
	1000 mg/L Si	Inorganic Ventures	0.98
	1000 mg/L Al	High Purity Standards	0.96
	IV-28	Inorganic Ventures	0.4
	1000 mg/L Sn	Inorganic Ventures	0.04
Interference Check Sample A (ICSA)	CLPP-ICS-A	Inorganic Ventures	10.0
Interference Check Sample AB (ICSAB)	CLPP-ICS-A	Inorganic Ventures	10.0
	CLPP-ICS-B4	Inorganic Ventures	1.0
	ICSAB-INT	Lab Prepared (see Table 4)	5.0
Continuing Calibration Verification (CCV)	ICP intermediate standard	Lab Prepared (see Table 4)	5.0
	QCS 26	High Purity Standards	0.5
Practical Quantitation Limit Sample (PQL)	PQL-INT	Lab Prepared (see Table 4)	1.0

TITLE: **TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010**

TABLE 4

PREPARATION OF INTERMEDIATE STANDARDS

Sample or Solution Name	Component Solution Name	Source of Component	Amount of Component Added per 100 mL Final Volume (mL)
PQL-INT	1000 mg/L B,Li,Sn,Sr, W, U	H-P or IV	1.0 each
	10000 mg/L K, Na	H-P or IV	1.0 each
	1000 mg/L Ni	High Purity Standards	0.4
	1000 mg/L Co	High Purity Standards	0.3
	1000 mg/L Cu,V,Zn	High Purity Standards	0.25 each
	1000 mg/L Si	High Purity Standards	2.0
	1000 mg/L Cr,Ti,Tl,Ag	High Purity Standards	0.15 each
	1000 mg/L Cd,Se, Mo	High Purity Standards	0.1 each
	10000 mg/L Al	High Purity Standards	0.3
	1000 mg/L As,Sb	High Purity Standards	0.08 each
	1000 mg/L Ba,Be,Mn,Pb	High Purity Standards	0.05 each
	10000 mg/L Ca,Mg	High Purity Standards	0.05 each
	10000 mg/L Fe	High Purity Standards	0.1
ICSAB-INT	10000 mg/L K,Na	H-P or IV	4.0 each
	10000 mg/L B, Li, Mo,Sr,Sn,Ti, W, U	High Purity Standards	1.0 each
	1000 mg/L Si	High Purity Standards	4.0

TITLE: **TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010**

TABLE 5

ELEMENT CONCENTRATIONS IN WORKING STANDARDS

	CONCENTRATION IN SOLUTION, mg/L								
Element	**STD1**	**ICV**	**PQL**	**ICSA**	**ICSAB**	**CCV**	**AL_IEC**	**FE_IEC**	**MN_IEC**
Aluminum	25	10	0.3	500	500	12.5	500		
Antimony	1	0.4	0.008		0.6	0.5			
Arsenic	1	0.4	0.008		0.1	0.5			
Barium	1	0.4	0.005		0.5	0.5			
Beryllium	1	0.4	0.005		0.5	0.5			
Boron	1	0.4	0.1		0.5	0.5			
Cadmium	1	0.4	0.01		1.0	0.5			
Calcium	25	10	0.05	500	500	12.5			
Chromium	1	0.4	0.015		0.5	0.5			
Cobalt	1	0.4	0.03		0.5	0.5			
Copper	1	0.4	0.025		0.5	0.5			
Iron	25	10	0.1	200	200	12.5		200	
Lead	1	0.4	0.005		0.05	0.5			
Lithium	1	0.4	0.1		0.5	0.5			
Magnesium	25	10	0.05	500	500	12.5			
Manganese	1	0.4	0.005		0.5	0.5			10
Molybdenum	1	0.4	0.01		0.5	0.5			
Nickel	1	0.4	0.04		0.5	0.5			
Potassium	25	13.6	1		20	12.5			
Selenium	1	0.4	0.01		0.05	0.5			
Silicon	1	0.4	0.2		2	0.5			
Silver	1	0.4	0.015		0.2	0.5			
Sodium	25	10	1		20	12.5			
Strontium	1	0.4	0.1		0.5	0.5			
Thallium	1	0.4	0.015		0.1	0.5			
Tin	1	0.4	0.1		0.5	0.5			
Titanium	1	0.4	0.015		0.5	0.5			
Tungsten	1	0.4	0.1		0.5	0.5			
Uranium	1	0.4	0.1		0.5	0.5			
Vanadium	1	0.4	0.025		0.5	0.5			
Zinc	1	0.4	0.025		1.0	0.5			

TITLE: **TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010**

TABLE 6

ELEMENT CONCENTRATIONS IN INTERMEDIATE STANDARDS

	CONCENTRATION IN SOLUTION, mg/L			
Element	**ICP Intermed STD**		**PQL-INT**	**ICSAB-INT**
Aluminum	240		30	
Antimony			0.8	
Arsenic			0.8	
Barium			0.5	
Beryllium			0.5	
Boron			10	10
Cadmium			1.0	
Calcium	240		5.0	
Chromium			1.5	
Cobalt			3.0	
Copper			2.5	
Iron	240		10	
Lead			0.5	
Lithium	10		10	10
Magnesium	240		5.0	
Manganese			0.5	
Molybdenum			1.0	10
Nickel			4.0	
Potassium	150		100	400
Selenium			1.0	
Silicon	250		20	40
Silver			1.5	
Sodium	240		100	400
Strontium	10		10	10
Thallium			1.5	
Tin	10		10	10
Titanium			1.5	10
Tungsten	10		10	10
Uranium	10		10	10
Vanadium			2.5	
Zinc			2.5	

TITLE: **TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010**

TABLE 7

ELEMENT CONCENTRATIONS IN STOCK STANDARDS

Element	CONCENTRATION IN SOLUTION, mg/L					
	IV-28	QCS-26	2007 ICS-1	CLPP-ICS-A	CLPP-ICS-B4	QCP-CICV-3
Aluminum	100	100		5000		
Antimony	100	100			60	
Arsenic	100	100			10	500
Barium	100	100			50	
Beryllium	100	100			50	
Boron	100	100	500			
Cadmium	100	100			100	250
Calcium	100	100		5000		
Chromium	100	100			50	
Cobalt	100	100			50	
Copper	100	100			50	
Iron	100	100		2000		
Lead	100	100			5	500
Lithium	100					
Magnesium	100	100		5000		
Manganese	100	100			50	
Molybdenum	100	100	300			
Nickel	100	100			100	
Potassium	1000	1000				
Selenium	100	100			5	500
Silicon	50	50	230			
Silver	100	100			20	
Sodium	100	100				
Strontium	100					
Thallium	100	100			10	500
Tin						
Titanium	100	100	1000			
Vanadium	100	100			50	
Zinc	100	100			100	

TABLE 8

REQUIRED ANALYTICAL SEQUENCE

Sequence Number	Standard/Sample	Purpose
1	Blank (Calibration Blank)	Initial calibration
2	S1 (Calibration Standard)	Initial calibration
3	ICV (Initial Calibration Verification)	Check calibration accuracy
4	ICB (Initial Calibration Blank)	Check calibration accuracy
5	PQL (Practical Quantitation Level Sample)	Check calibration accuracy near PQL, repeat before final CCV, CCB
6	ICSA (Interference Check Solution A)	Verify accuracy of IEC factors, repeat before final CCV, CCB
7	ICSAB (Interference Check Solution AB)	Verify accuracy of IEC factors, repeat before final CCV, CCB
8	CCV (Continuing Calibration Verification)	Check calibration stability
9	CCB (Continuing Calibration Blank)	Check calibration stability
10-19	Analyze up to 10 samples	
20	CCV (Continuing Calibration Verification)	Check calibration stability
25	CCB (Continuing Calibration Blank)	Check calibration stability
...	Continue analyzing sequences of up to 10 samples, followed by a CCV and a CCB	

ATTACHMENT 1

HARDNESS BY CALCULATION

As referenced in "Standard Methods for the Examination if Water and Wastewater," Methods 2340 A & B, Hardness Introduction and Hardness by Calculation, American Public Health Association, 18th Edition, Revised 1992, total hardness is the sum of the calcium and magnesium concentrations, both expressed as calcium carbonate, in milligrams per liter.

Once the calcium and magnesium concentrations have been determined by EPA methods 6010, 6020, 200.7 or 200.8, the total hardness of an aqueous sample may be calculated as follows:

$$\text{Total Hardness, mg equivalent } CaCO_3/L = 2.497\ (Ca, mg/L) + 4.118\ (Mg, mg/L)$$

The calcium hardness of an aqueous sample may also be calculated as follows:

$$\text{Calcium Hardness, mg equivalent } CaCO_3/L = 2.497\ (Ca, mg/L)$$

TITLE: **SYNTHETIC PRECIPITATION LEACHING PROCEDURE (SPLP) FOR INORGANIC AND NON-VOLATILE ORGANIC ANALYTES**

Prepared By: George Brewer Date: 03/14/05

Approved By:

Department Manager: George Brewer Date: 03/14/05

Operations Manager: Deborah J. Nadeau Date: 3.14.05

QA Officer: Leslie Dimond Date: 031405

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01	Added expirations dates (1yr) for SPLP fluid s Added DOC requirements Revised TCLP/SPLP logbooks - making it usable for both TCLP/SPLP and record pH, expiration dates	LAD	01/07	01/07
02	Updated figures 6 → 8 with current logbook pages.	LAD	04/08	04/08

TITLE: SYNTHETIC PRECIPITATION LEACHING PROCEDURE (SPLP) FOR INORGANIC AND NON-VOLATILE ORGANIC ANALYTES

Please acknowledge receipt of this standard operating procedure by signing and dating both of the spaces provided. Return the bottom half of this sheet to the QA Department.

I acknowledge receipt of copy ___ of document **SOP CA-620-02**, titled **Synthetic Precipitation Leaching Procedure (SPLP) For Inorganic and Non-Volatile Organic Analytes**.

Recipient: _____Date:_____

KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy ___ of document **SOP CA-620-02**, titled **Synthetic Precipitation Leaching Procedure (SPLP) For Inorganic and Non-Volatile Organic Analytes.**

Recipient: _____Date:_____

TITLE: SYNTHETIC PRECIPITATION LEACHING PROCEDURE (SPLP) FOR INORGANIC AND NON-VOLATILE ORGANIC ANALYTES

1.0 SCOPE AND APPLICATION

The purpose of this SOP is to define the procedures used by Katahdin Analytical Services, Inc., personnel for SPLP extraction of samples for inorganic and non-volatile organic components using USEPA Method 1312 (Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, US EPA SW846), with the modifications discussed in Table 2.

The SPLP (Synthetic Precipitation Leaching Procedure) is designed to determine the mobility of both organic and inorganic analytes present in liquid, solid, and multiphasic wastes.

1.1 Definitions - None.

1.2 Responsibilities

This method is restricted to use by, or under the supervision of, analysts experienced in SPLP extractions. Each analyst must demonstrate the ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, current revision, Personnel Training and Demonstration of Capability.

It is the responsibility of all Katahdin technical personnel involved in SPLP extractions to read and understand this SOP, to adhere to the procedures outlined, and to properly document their data in the appropriate lab notebook. Any deviations from the test or irregularities with the samples should also be recorded in the lab notebook and reported to the Department Manager or designated qualified data reviewer responsible for this data.

It is the responsibility of the Department Manager to ensure that members of their group follow this SOP, to ensure that their work is properly documented, and to initiate periodic review of the associated logbooks.

1.3 Safety

Users of this procedure must be aware of inherent laboratory hazards, proper disposal procedures for contaminated materials, and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method may not be precisely known; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets (MSDS) is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDSs for all the materials used in this procedure.

Each qualified analyst or technician must be familiar with Katahdin Analytical safety procedures and the Katahdin Hazardous Waste Plan and must follow appropriate procedures. These include the use of appropriate personal protective equipment (PPE) such as safety glasses, gloves and lab coats when working with chemicals or

near an instrument and not taking food or drink into the laboratory. Each analyst should know the location of a respirator and all safety equipment. Each analyst shall receive a safety orientation from their Department Manager, or designee, appropriate for the job functions they will perform.

1.4 Pollution Prevention/Waste Disposal

Whenever possible, laboratory personnel should use pollution prevention techniques to address their waste generation. Refer to the current revision of the Katahdin Hazardous Waste Plan for further details on pollution prevention techniques.

Wastes from SPLP extraction may contain acids, heavy metals, toxic organics, and other toxic components and should be disposed of in a manner appropriate to the hazards they present. Further information regarding waste classification and disposal may be obtained by consulting the Katahdin Hazardous Waste Plan and the Department Manager.

2.0 SUMMARY OF METHOD

2.1 For liquid wastes (i.e., those containing less than 0.5% dry solid material), the waste, after filtration through a 0.6 to 0.8 µm glass fiber filter, is defined as the SPLP extract.

2.2 For wastes containing greater than or equal to 0.5% solids, the liquid phase is first separated from the solid phase and stored for later analysis. The particle size of the solid phase is reduced, if necessary, and the solid phase is extracted with an amount of extraction fluid equal to 20 times its weight. If the sample is a soil, the composition of the extraction fluid employed depends on the region of the country where the sample site is located. If the sample is a waste or a wastewater, the extraction fluid used is a pH 4.2 solution. After extraction, the liquid extract is separated from the solid phase by filtration through a 0.6 to 0.8 µm glass fiber filter.

2.3 If they are compatible (i.e., multiple phases will not form on combination), the initial liquid phase of the waste is added to the liquid extract and these are analyzed together. If they are incompatible, the liquids are analyzed separately and the results are mathematically combined to yield a volume-weighted average concentration.

3.0 INTERFERENCES

Because the dissolved solids contents of SPLP extracts are typically high, analyses of these extracts are often troubled by matrix interferences. Methods to detect and overcome matrix interferences are integral to the SPLP procedure and are discussed in detail in Section 8.0, Quality Control.

Potential interferences that may be encountered during analysis are discussed in the individual analytical methods.

4.0 APPARATUS AND MATERIALS

4.1 Agitation apparatus (rotary extractor) - The agitation apparatus must be capable of rotating the extraction vessel in an end-over-end fashion at 30±2 revolutions per minute (rpm) – see Figure 2. Each of the laboratory's rotary extractors is equipped with a device that displays the actual rotation rate in rpm. The rotation rate of each extractor is monitored before each use, and the measured rotation rates are recorded in a logbook maintained for that purpose (see Figure 8). If the measured rotation rate of an extractor is outside the range 30±2 rpm, it must be taken out of service until it can be repaired.

4.2 Extraction vessels - must fit the rotary extractor and have sufficient capacity to hold the sample and the extraction fluid (jars with capacities of 2.2 L are normally used). The vessel must be made of borosilicate glass or fluorinated plastic (e.g. Teflon) if the extract is to be analyzed for organics. If the extract is to be analyzed only for inorganics, polyethylene or polypropylene containers may be used.

4.3 Filter Holder - Filter holders for pressure filtration are used. They are constructed of type 316 stainless steel (with or without PTFE linings) and are capable of sustaining internal pressures exceeding 50 psi. These devices have an internal capacity of 1.5 L and accommodate glass fiber filters 142 mm in diameter.

4.4 Filters - Borosilicate glass fiber filters containing no binder materials and having an effective pore size of 0.6 to 0.8 µm, 142 mm diameter or equivalent. Prefilters must not be used. Glass fiber filters are fragile and should be handled with care. Filters should be acid-washed with 1N HNO_3 and triple rinsed with laboratory reagent grade water (minimum 500 mL/ rinse) prior to use.

4.5 pH meter accurate to ±0.05 units at 25°C. The pH meter must be calibrated on each day of use.

4.6 Laboratory balance accurate to within ±0.01 grams (all weight measurements are to be within ±0.1 grams).

4.7 Beakers, glass, 500 mL.

4.8 Watch glasses, appropriate diameter to cover beakers.

4.9 Magnetic stirrer.

5.0 REAGENTS

Reagent grade chemicals shall be used in all tests. Other grades may be used only if it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

5.1 Laboratory reagent grade water - Reagent water should be monitored periodically for impurities.

5.2 Sulfuric acid, concentrated (H_2SO_4) – reagent grade.

5.3 Nitric acid, concentrated (HNO_3) – reagent grade.

5.4 Nitric acid, 1N, for acid-washing filters. Dilute 63 mL reagent grade HCl to 1000 mL with laboratory reagent grade water.

5.5 Sulfuric acid (0.6 weight percent) / nitric acid (0.4 weight percent) mixture – Add 0.6 g concentrated H_2SO_4 and 0.4 g HNO_3 to a 100 mL volumetric flask half-filled with laboratory reagent grade water. Swirl to mix and bring to a final volume of 100 mL with laboratory reagent grade water.

5.6 SPLP Fluid #1 – Add approximately 15 L of laboratory reagent grade water to a clean graduated 20 L polyethylene carboy reserved for this fluid. Add approximately 5 mL of 0.6% H_2SO_4 / 0.4% HNO_3 solution to the carboy, and fill with laboratory reagent grade water to the 20 L graduation. Cap the carboy tightly and agitate it until the fluid is well mixed. Dispense approximately 30 mL of fluid from the carboy's spigot into a disposable cup and measure the pH of the fluid. The pH of the fluid must be 4.20 ± 0.05. If necessary, add more acid solution to lower the pH, or remove some of the fluid and replace it with laboratory reagent grade water to raise the pH, until the correct pH is obtained. SPLP Fluid #1 is used to determine the leachability of soils from sites east of the Mississippi River, and the leachability of wastes and wastewaters. The fluid may be used for up to one year from the preparation date.

5.7 SPLP Fluid #2 - Add approximately 4 L of laboratory reagent grade water to a clean graduated 5 L polyethylene carboy reserved for this fluid. Add approximately 1.5 mL of 0.6% H_2SO_4 / 0.4% HNO_3 solution to the carboy, and fill with laboratory reagent grade water to the 5 L graduation. Cap the carboy tightly and agitate it until the fluid is well mixed. Dispense approximately 30 mL of fluid from the carboy's spigot into a disposable cup and measure the pH of the fluid. The pH of the fluid must be 5.00 ± 0.05. If necessary, add more acid solution to lower the pH, or dump out some of the fluid and replace it with laboratory reagent grade water to raise the pH, until the correct pH is obtained. SPLP Fluid #1 is used to determine the leachability of soils from sites west of the Mississippi River. The fluid may be used for up to one year from the preparation date.

5.8 SPLP Fluid #3 – This fluid is laboratory reagent grade water (Section 5.1) and is used to determine cyanide leachability.

NOTE: The pH of each extraction fluid must be checked prior to each use to ensure that it has been prepared accurately, and the measured pH is recorded in the Non-Volatile SPLP Extraction Logbook (Figure 9) for each sample extracted. Details of the preparation of these fluids (reagent lot numbers, volumes, and masses; measured pH; etc.) are recorded in the SPLP Fluid Preparation and Use Logbook (Figure 7). Upon preparation, each new batch of extraction fluid is assigned a 3-digit batch number by the analyst (batches are numbered consecutively), and the Katahdin Sample Number of each client sample extracted with a particular fluid batch is recorded in the SPLP Fluid Preparation and Use Logbook. Extraction fluids are monitored for impurities as described in Section 8.0 of this SOP.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

All samples shall be collected using an appropriate sampling plan.

6.1 Sufficient sample must be collected to support the preliminary determinations and to provide an extract volume adequate for all analytical and quality control purposes. The necessary sample size will depend on the solids content of the waste, but in no instance should less than 250 g of waste be provided to the laboratory.

6.2 Preservatives shall not be added to samples before extraction. Samples should be stored at 4°C and opened immediately prior to SPLP extraction.

6.3 SPLP extracts should be prepared for analyses and analyzed as soon as possible following SPLP extraction. Extracts for metals analysis must be acidified to a pH < 2 with nitric acid. Extracts for other analyses should be preserved according to the guidance given in the individual analytical methods. Extracts for organic analyte determinations shall not be allowed to come into contact with the atmosphere (i.e., no headspace) to prevent losses.

6.4 Sample holding times for non-volatile SPLP extraction and analysis summarized in the following table:

SPLP PARAMETER	FROM COLLECTION TO SPLP EXTRACTION	FROM SPLP EXTRACTION TO PREPARATIVE EXT'N	FROM PREP EXT'N TO ANALYSIS
PEST/HERBS	14	7	40
SEMIVOLATILES	14	7	40
MERCURY	28	N/A	28
METALS EXCEPT MERCURY	180	N/A	180

7.0 PROCEDURES

The procedure consists of a series of preliminary evaluations of the waste, followed by the actual extraction. Flow charts summarizing the procedure appear as Figures 3 and 4. Preliminary evaluations are to be performed on a minimum 100 g aliquot of the waste. This aliquot may not actually undergo SPLP extraction. These preliminary evaluations include: (1) determination of the percent solids, Section 7.1; (2) determination of whether the waste contains insignificant solids and is, therefore, its own extract after filtration, Section 7.2; (3) particle size evaluation, Section 7.3; and (4) determination of the appropriate extraction fluid to be used for the SPLP extraction, Section 7.4.

All information and measurements pertaining to SPLP extractions are recorded in the Non-Volatile SPLP Extraction Logbook (Figure 9). In the following procedure, the section or line of the Non-Volatile SPLP Extraction Logbook page in which the pertinent information should be recorded is indicated in bold, e.g. **Section II** or **Line C**.

PRELIMINARY EVALUATIONS

7.1 Determination of Percent Solids (**Section I**) - Percent solids is defined for SPLP as that fraction of a waste sample (as a percentage of the total sample) from which no liquid may be forced out by an applied pressure, as described below.

If the waste will obviously yield no liquid when subjected to pressure filtration (i.e., is 100% solids) the percent solids determination may be omitted. Proceed to Section 7.3, Particle Size Evaluation.

If the sample is liquid or multiphasic, liquid/solid separation by filtration is required to make a preliminary determination of percent solids. This involves the filtration device. The procedure is as follows, Sections 7.1.1 through 7.1.9:

7.1.1 Pre-weigh the filter (**Line A**) and the container that will receive the filtrate (filtrate vessel) (**Line B**).

7.1.2 Assemble the filter holder and filter following the manufacturer's instructions. Place the filter on the support screen and secure.

7.1.2 Weigh out a subsample of the waste (100 gram minimum) and record the combined weight of the weigh boat and waste (**Line C**).

7.1.4 Allow slurries to stand to permit the solid phase to settle. Wastes that settle slowly may be centrifuged, prior to filtration. Centrifugation is to be used only as an aid to filtration. If centrifugation is used, the liquid should be decanted and filtered followed by filtration of the solid portion of the waste through the same filtration system.

7.1.5 Quantitatively transfer the waste sample (liquid and solid phases) to the filter holder, spreading the waste sample evenly over the surface of the filter. If filtration of the waste at 4°C reduces the amount of expressed liquid over what would be expressed at room temperature, then allow the sample to warm up to room temperature in the device before filtering.

7.1.6 Weigh the weigh boat and any residue clinging to it (**Line D**). Determine the total weight of waste to be filtered by subtracting the weight of the weigh boat and residue from the weight of the weigh boat and waste (**Line E**).

7.1.7 Gradually apply vacuum or gentle pressure of 1-10 psi until air or pressurizing gas moves through the filter, collecting any filtrate in the pre-weighed filtrate vessel. If this point is not reached under 10 psi, and if no additional liquid has passed through the filter in any 2-minute interval slowly increase the pressure in 10-psi increments to a maximum of 50 psi. After each incremental increase of 10 psi, if the pressurizing gas has not moved through the filter, and if no additional liquid has passed through the filter in any 2-minute interval, proceed to the next 10-psi increment. When the pressurizing gas begins to move through the filter, or when liquid flow has ceased at 50 psi (i.e., filtration does not result in any additional filtrate within any 2 minute period), stop the filtration.

The material in the filter holder is defined as the solid phase of the waste, and the filtrate is defined as the liquid phase.

7.1.8 Weigh the filtrate vessel and its contents (**Line F**). Determine the weight of the liquid phase by subtracting the weight of the filtrate vessel from the total weight of the filtrate-filled container (**Line G**).

7.1.9 Calculate the percent wet solids as follows (**Line H**):

$$\text{Percent wet solids} = \frac{\text{(Total weight of waste)-(Weight of liquid phase)}}{\text{Total weight of waste}}$$

7.2 If the percent solids determined in Section 7.1.9 above is equal to or greater than 0.5% and the weight of water entrained in the filter is small in comparison with the weight of the solid phase, then proceed to Section 7.3 to determine whether the solid material requires particle size reduction. Continue with Section 7.2 if it is noticed that the amount of the filtrate entrained in wetting the filter is significant in proportion to the weight of the solid phase. If the percent solids determined in Section 7.1.9 is less than 0.5%, then proceed to Section 7.5.4 using a fresh portion of the waste.

7.2.1 Remove the solid phase and filter from the filtration apparatus.

7.2.2 Dry the filter and solid phase at 100± 20°C until two successive weighings yield the same value within ±1%. Record the weight of the filter and dry solids (**Line I**).

NOTE: Caution should be taken to ensure that the subject solid will not flash upon heating. It is recommended that the drying oven be vented to a hood or other appropriate device.

7.2.3 Calculate the weight of dry solids by subtracting the weight of the filter from the weight of the filter and dry solids (**Line J**).

7.2.4 Calculate the percent dry solids as follows (**Line L**):

$$\text{Percent dry solids} = \frac{\text{Weight of dry solids}}{\text{Total weight of waste}} \times 100$$

Note: Non-aqueous liquid samples (e.g. oils) may be entrained in the filter, and may remain in the filter after drying, contributing weight to the dried filter. If this is the case, the surface of the filter should be examined for apparent solids or particulate material. If none are found, a comment to that effect should be made in the Comments section of the Non-Volatile SPLP Extraction Logbook (e.g. "No apparent solids present – dry solid weight is due to entrained non-volatile liquid"), and the sample should be treated as if it contains less than 0.5% dry solids.

7.2.5 If the percent dry solids is less than 0.5%, then proceed to Section 7.5.4. If the percent dry solids is greater than or equal to 0.5%, proceed to Section 7.3.

7.3 Particle Size Evaluation - Visually evaluate the particle size of the solid phase of the waste. Filamentous material (cloth, paper, etc.) will require particle size reduction if it has a surface area per gram of less than 3.1 cm^3. Other solid materials require particle size reduction if the particles are greater than 1 cm in their narrowest dimension (i.e. if they will not pass through a 9.5 mm standard sieve). Particle size reduction may be accomplished by cutting, crushing, or grinding the waste to a surface area or particle size as described above. Perform particle size reduction on the solid material that will actually undergo extraction, not on that used for the preliminary determinations.

7.4 Determination of Appropriate Extraction Fluid - If the solid content of the waste is greater than or equal to 0.5%, determine the appropriate fluid for the non-volatiles extraction as follows:

7.4.1 For soils, if the sample is from a site that is east of the Mississippi River, SPLP Fluid #1 should be used. If the sample is from a site that is west of the Mississippi River, SPLP Fluid #2 should be used.

7.4.2 For wastes and wastewaters, SPLP Fluid #2 should be used.

7.4.3 For cyanide containing wastes or soils, SPLP Fluid #3 must be used because leaching of cyanide-containing samples under acidic conditions may result in the formation of hydrogen cyanide gas.

SPLP EXTRACTION FOR NON-VOLATILES

7.5 A minimum sample size of 100 grams (solid and liquid phases) is recommended. In some cases, a larger sample size may be appropriate, depending on the solids content of the waste sample, whether the initial liquid phase of the waste will be miscible with the aqueous extract of the solid, and whether inorganics, semivolatile organics, pesticides, and herbicides are all analytes of concern. Enough solids should be generated for extraction such that the volume of SPLP extract will be sufficient to perform all of the required analyses. If necessary, multiple extractions may be performed and the extracts combined and aliquoted for analysis.

7.5.1 If the waste will obviously yield no liquid when subjected to pressure filtration (i.e., is 100% solid), weigh out a subsample of the waste (100 g minimum), record the weight (**Section III**), and proceed to Section 7.5.11. If the sample is liquid or multiphasic, liquid/solid separation is required - proceed to Section 7.5.2.

7.5.2 Pre-weigh the container that will receive the filtrate (filtrate vessel) (**Line M**).

7.5.3 Assemble the filter holder and filter following the manufacturer's instructions. Place the filter on the support screen and secure. Acid-wash the filter if extracting for metals components. Acid-washed filters may be used for non-volatile extractions even when metals are not of concern.

7.5.4 Weigh out a subsample of the waste (100 gram minimum) and record the combined weight of the waste and weigh boat (**Line N**). If the waste contains <0.5% dry solids, the liquid portion of the waste, after filtration, is defined as the SPLP extract. Therefore, enough of the sample should be filtered so that the amount of filtered liquid will support all of the required analyses. For wastes containing >0.5% dry solids, information is obtained in Section 7.1 to determine the optimum sample size (100 gram minimum) for filtration. Enough solids should be generated by filtration to support the analyses to be performed on the SPLP extract.

7.5.5 Allow slurries to stand to permit the solid phase to settle. Wastes that settle slowly may be centrifuged prior to filtration. Use centrifugation only as an aid to filtration. If centrifugation is used, the liquid should be decanted and filtered followed by filtration of the solid portion of the waste through the sample filtration system.

7.5.6 Quantitatively transfer the waste sample (liquid and solid phases) to the filter holder. Spread the waste sample evenly over the surface of the filter. If filtration of the waste at 4°C reduces the amount of expressed liquid over what would be expressed at room temperature, then allow the sample to warm up to room temperature in the device before filtering.

7.5.7 Weigh the weigh boat and any residue clinging to it (**Line O**). Determine the total weight of waste to be filtered by subtracting the weight of the weigh boat and residue from the weight of the weigh boat and waste (**Line P**).

7.5.8. Gradually apply vacuum or gentle pressure of 1-10 psi until air or pressurizing as moves through the filter, collecting any filtrate in the pre-weighed filtrate vessel. If this point is reached under 10 psi, and if no additional liquid has passed through he filter in any 2-minute interval, slowly increase in pressure in 10-psi increments to a maximum of 50 psi. After each incremental increase of 10 psi, if the pressurizing gas has not moved through the filter, and if no additional liquid has passed through the filter in any 2-minute interval, proceed to the next 10-psi increment. When the pressurizing gas begins to move through the filter, or when the liquid flow has creased at 50 psi (i.e., filtration does not result in any additional filtrate within a 2 minute period), stop the filtration.

The material in the filter holder is defined as the solid phase of the waste, and the filtrate is defined as the liquid phase.

7.5.9 Weigh the filtrate vessel and its contents (**Line Q**). Determine the weight of the liquid phase by subtracting the weight of the filtrate vessel from the total weight of the filtrate-filled container (**Line R**). Decant the liquid phase into a graduated cylinder and measure and record its volume (**Line S**). Pour the liquid phase back into the filtrate vessel for storage. The liquid phase may now either be analyzed or stored at 4°C until time of analysis.

NOTE: Some wastes, such as oily wastes and some paint wastes, will obviously contain some material that appears to be a liquid. Even after applying pressure filtration, as outlined in Section 7.5.8, this material may not filter. If this is the case, the material within the filtration device is defined as a solid and is carried through the extraction as a solid. Do not replace the original filter with a fresh filter under any circumstances. Use only one filter.

7.5.10 Calculate the weight of wet solids by subtracting the weight of the liquid phase from the total weight of waste (**Line T**).

7.5.11 If necessary, prepare the solid portion of the waste for extraction by crushing, cutting, or grinding the waste to a surface area or particle size as described in Section 7.3. Describe the particle size reduction process in **Section IV** of the

logbook. When the surface area or particle size has been appropriately altered, quantitatively transfer the solid material into an extractor bottle. Include the filter used to separate the initial liquid from the solid phase.

7.5.12 Determine the amount of extraction fluid to add to the extractor vessel as follows:

$$\text{Weight of extraction fluid} = \frac{(20)\ (\text{Weight of wet solids})}{100}$$

Slowly add this amount of appropriate extraction fluid to the extractor vessel. Record the fluid batch ID and the amount used in **Section III** of the logbook. Close the extractor bottle tightly (Teflon tape may be used to ensure a tight seal), secure in rotary agitation device, and rotate at 30± 2 RPM during the extraction period of 18 ± 2 hours. Record the extraction start and end times and the room temperatures in **Section IV** of the logbook.

NOTE: As agitation continues, pressure may build within the extractor bottle for some types of wastes (e.g., limed or calcium carbonate containing waste may evolve gases such as carbon dioxide). To relieve excess pressure, the extractor bottle may be periodically opened (e.g., after 15 minutes, 30 minutes, and 1 hour) and vented into a hood.

7.5.13 Following the extraction, separate the contents of the vessel into its component liquid and solid phases by filtering through a new acid-washed glass fiber filter, as outlined in Section 7.5.6. For final filtration of the SPLP extract, the glass fiber filter may be changed, if necessary, to facilitate filtration.

NOTE: If the waste contained no initial liquid phase, it is only necessary to filter enough extract to support the required analyses. However, if the waste contained an initial liquid phase, the entire contents of the extraction vessel must be filtered.

7.5.14 Prepare the SPLP extract as follows:

7.5.14.1 If the waste contained no initial liquid phase, the filtered liquid material obtained from Section 7.5.13 is defined as the SPLP extract. Proceed to Section 7.5.15.

7.5.14.2 If compatible (e.g., multiple phases will not result on combination), combine the filtered liquid resulting from Section 7.5.13 with the initial liquid phase of the waste obtained in Section 7.5.8. This combined liquid is defined as the SPLP extract. Proceed to Section 7.5.15.

7.5.14.3 If the initial liquid phase of the waste, as obtained from Section 7.5.8, is not or may not be compatible with the filtered liquid resulting from Section 7.5.13, do not combine these liquids. Measure the volume of filtrate obtained in Section 7.5.13 and record in **Section IV** of the logbook. Individually analyze these two liquids, collectively defined as the SPLP extract, and combine the results mathematically, as described in Section 7.6.

7.5.15 Following collection of the SPLP extract, the pH of the extract should be measured and recorded (**Section IV**). Immediately aliquot and preserve the extract for analysis. Metals aliquots must be acidified with nitric acid to pH <2. All other aliquots must be stored under refrigeration (4°C) until analyzed.

7.6 The SPLP extract shall be prepared and analyzed according to appropriate analytical methods. SPLP extracts to be analyzed for metals shall be acid digested except in those instances where digestion causes loss of metallic analytes. If an analysis of the undigested extract shows that the concentration of any regulated metallic analyte exceeds the regulatory level, then the waste is hazardous and digestion of the extract is not necessary. However, data on undigested extracts alone cannot be used to demonstrate that the waste is not hazardous. If the individual phases are to be analyzed separately, determine the volume of the individual phases (to ± 0.5%), conduct the appropriate analyses, and combine the results mathematically by using a simple volume-weighted average:

$$\text{Final Analyte Concentration} = \frac{(V_1)(C_1) + (V_2)(C_2)}{V_1 + V_2}$$

where: V_1 = The volume of the first phase (L).
C_1 = The concentration of the analyte of concern in the first phase (mg/L).
V_2 = The volume of the second phase (L).
C_2 = The concentration of the analyte of concern in the second phase (mg/L).

7.6 Compare the analyte concentrations in the SPLP extract with the levels identified in the appropriate regulations. Refer to Section 8.0 for quality control requirements.

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

USEPA Method 1312 requires the laboratory to perform specific quality control checks to assess laboratory performance and data quality. Minimum frequencies, acceptance criteria, and corrective actions for these control checks are listed in Table 1 and are described below. Table 1 criteria are intended to be guidelines for analysts. The table does not cover all possible situations. If any of the QC requirements are outside the recovery

ranges listed in Table 1, all associated samples must be evaluated against all the QC. In some cases data may be reported, but may be reanalyzed in other cases. Making new reagents and standards may be necessary if the standardization is suspect. The corrective actions listed in Table 1 may rely on analyst experience to make sound scientific judgments. These decisions are based on holding time considerations and client and project specific Data Quality Objectives. The Department Manager, Operations Manager and/or Quality Assurance Officer may be consulted to evaluate data. Some samples may not be able to be reanalyzed within hold time. In these cases "qualified" data with narration may be advisable after consultation with the client.

In some cases the standard QC requirements listed in this section and in Table 1 may not be sufficient to meet the Data Quality Objectives of the specific project. Much of the work performed at the lab is analyzed in accordance with specific QC requirements spelled out in a project specific Quality Assurance Project Plan (QAPP) or in a program specific Quality Systems Manual (QSM). The reporting limits, acceptance criteria and/or corrective actions may be different than those specified in this SOP. In these cases the appropriate information will be communicated to the Department Manager and/or senior chemists before initiation of the analyses so that specific product codes can be produced for the project. In addition, the work order notes for each project will describe the specific QAPP or QSM to be followed.

8.1 A minimum of one method blank for every 20 extractions performed using a particular batch of extraction fluid <u>and</u> per 20 extractions performed in a particular extraction vessel must be extracted and analyzed for the same contaminants as all associated samples. The method blanks are analyzed to check for laboratory contamination. A count of extractions performed in each extraction vessel is maintained in order to monitor the frequency of method blanks (1 per 20 extractions per vessel) required for each extraction vessel.

8.1.1 After SPLP extraction, SPLP method blanks must undergo preparative extraction and analysis within method holding times (refer to Section 6.4). For this reason it may be necessary to extract more than one method blank using a particular batch of extraction fluid. For example, suppose that a sample requiring analysis for SPLP metals and semivolatiles is extracted using freshly prepared fluid from Batch 300. Because the fluid is new, a method blank is extracted with the sample and analyzed for the same components as the sample. Eight days later, a different sample requiring full SPLP analysis (metals, semivolatiles, pesticides, and herbicides) is extracted using fluid from Batch 300. Because the holding time for the previous SPLP method blank for pesticides and herbicides has expired, a new SPLP method blank must be extracted and analyzed for pesticides and herbicides. The new method blank need not be analyzed for metals and semivolatiles, because the first method blank that was prepared with fluid from Batch 300 has already been analyzed for these constituents.

8.1.2 Each SPLP method blank is identified in the SPLP extraction logbooks by a seven-character code. The first three characters are "PBP", which stands for "Preparation Blank - SPLP". Characters 4 through 6 consist of the three-digit preparation number of the extraction fluid. The seventh character is a letter, starting with "A" and proceeding alphabetically, which is unique to the extraction date for a particular batch of fluid. For example, "PBP316A" refers to the first SPLP method blank extracted using fluid from Batch 316; "PBP316B" refers to the second SPLP method blank extracted using the same fluid. The extraction date of each SPLP method blank is recorded in the SPLP Fluid Preparation and Use Logbook.

8.2 The laboratory recommends that a matrix spike be performed for each waste type (e.g., wastewater treatment sludge, contaminated soil, etc.) unless the result exceeds the regulatory level and the data are being used solely to demonstrate that the waste property exceeds the regulatory level. Because the laboratory charges for the preparation and analysis of SPLP matrix spikes, selection of samples for SPLP matrix spiking is left to the discretion of the client. A minimum of one SPLP matrix spike must be analyzed for each batch of 20 SPLP extractions. As a minimum, follow the matrix spike addition guidance provided in each analytical method. Additional matrix spiking directions and guidance are provided in Table 4 and Figures 5 and 6.

8.2.1 Matrix spikes are to be added after filtration of the SPLP extract and before any preservation. Matrix spikes should not be added prior to SPLP extraction of the sample.

8.2.2 Instructions for preparing SPLP matrix spikes for metals analysis are contained in Table 3. Instructions for preparing SPLP matrix spikes for organics analyses are contained in Figures 5 and 6. In order to avoid differences in matrix effects, the matrix spikes must be added to the same nominal volume of SPLP extract as that which was analyzed for the unspiked sample.

8.2.3 Matrix spike recoveries are calculated by the following formula:

$$\text{Recovery (\%)} = 100\ (X_s - X_u)/K$$

where:
X_s = measured value for the spiked sample,
X_u = measured value for the unspiked sample, and
K = known value of the spike in the sample

8.2.4 The purpose of the matrix spike is to monitor the performance of the sample preparation and analytical methods used and to determine whether matrix interferences exist. Use of internal calibration methods (e.g. the method of standard additions [MSA]), modification of the analytical methods, or use of alternate analytical methods may be needed to accurately measure the

analyte concentration of the SPLP extract when the recovery of the matrix spike is below the expected analytical method performance. Metallic analytes must be quantitated by the method of standard additions if the SPLP matrix spike recovery for the analyte is less than 50% and the measured concentration of the analyte in the unspiked aliquot is within 20% of the regulatory level.

8.3 Each new analyst must demonstrate her/his ability to perform the method acceptably by while being witnessed by an analyst who is experience in performing the method. To successfully demonstrate the method, the analyst must perform the method in conformance with all the requirements of the SOP, referring to the SOP for guidance as necessary. In addition, each analyst must demonstrate the ability to produce TCLP Extraction Blanks that are free of contamination. This demonstration will require the analyst to collect and file the analytical results from four Extraction Blanks that he/she has generated.

8.4 All quality control measures described in the appropriate analytical methods shall be followed.

9.0 METHOD PERFORMANCE

The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. The MDLs are determined annually per type of instrument and filed with the Department Manager and with the QAO.

Refer to the current revisions of USEPA Method 1312 for other method performance parameters and requirements.

10.0 APPLICABLE DOCUMENTS/REFERENCES

Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, USEPA SW846, Third Edition, Final Update I (7/92), Method 1312

TITLE: SYNTHETIC PRECIPITATION LEACHING PROCEDURE (SPLP) FOR INORGANIC AND NON-VOLATILE ORGANIC ANALYTES

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TITLE: SYNTHETIC PRECIPITATION LEACHING PROCEDURE (SPLP) FOR INORGANIC AND NON-VOLATILE ORGANIC ANALYTES

TABLE 1

QC REQUIREMENTS

Parameter/ Method	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Synthetic Precipitation Leaching Procedure (SPLP) /EPA 1312	Method Blanks	One per 20 samples extracted using a particular batch of extraction fluid.	Refer to individual analytical methods.	Prepare fresh extraction fluid and repeat SPLP extraction of all associated samples.
		One per 20 samples extracted in a particular extraction vessel.	Refer to individual analytical methods.	Remove extraction vessel from service.
	Matrix Spike	One per 20 SPLP extractions performed (required). One per waste type (suggested, left to discretion of client).	For metallic analytes, >50% if native analyte concentration is within ± 20% of regulatory level. For other analytes, refer to appropriate analytical methods.	For metallic analytes, quantitate by method of standard additions. For other analytes, refer to appropriate analytical methods.
	Demonstration of analyst proficiency; accuracy and precision	One time demonstration by each analyst performing the method	New analyst's performance of the method is witnessed by an experienced analyst. New analyst must produce method blanks that meet all method and laboratory acceptance criteria.	Repeat analysis until able to demonstrate acceptable performance of the method to witnessing analyst and by producing acceptable method blanks; document successful performance in personal training file.

TABLE 2

SUMMARY OF METHOD MODIFICATIONS

TOPIC	KATAHDIN SOP CA-620-02	EPA METHOD 1312
QC - Method Blanks	Frequency of one method blank per 20 extractions performed using a particular batch of extraction fluid and per 20 extractions performed in a particular extraction vessel.	Frequency of one method blank per 20 extractions performed in a particular extraction vessel.
QC - Spikes	Matrix spike **recommended** for each waste type.	Matrix spike **required** for each waste type.

TITLE: SYNTHETIC PRECIPITATION LEACHING PROCEDURE (SPLP) FOR INORGANIC AND NON-VOLATILE ORGANIC ANALYTES

TABLE 3

SPLP MATRIX SPIKING FOR METALLIC ANALYTES

SPIKING INSTRUCTIONS			
Sample or Solution Name	**Component Solution Name**	**Source of Component**	**Amount of Component Added per 50 mL Final Volume (mL)**
SPLP Matrix Spike (ICP)	CLPP-SPK-1	Inorganic Ventures	0.050
	CLPP-SPK-INT1	Lab Prepared (see below)	0.50
SPLP Matrix Spike (Mercury)	1000 ug/L Hg Standard	Prepared from 1000 mg/L stock standard	0.10

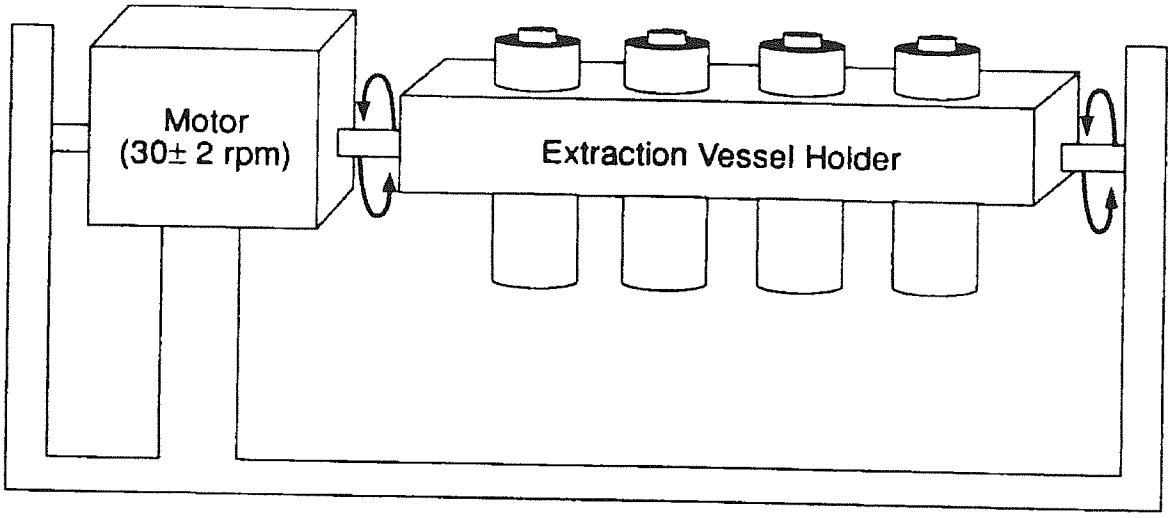
Note: Spiking must be performed after SPLP extraction and before preservation.

PREPARATION OF INTERMEDIATE SPIKING SOLUTIONS			
Solution Name	**Component Solution Name**	**Source of Component**	**Amount of Component Added per 100 mL Final Volume (mL)**
CLPP-SPK-INT1	QCP-CICV-3	Inorganic Ventures	10.0
	1000 mg/L Sb	High Purity Standards	5.0
	10000 mg/L K	High Purity Standards	10.0
	10000 mg/L Na	High Purity Standards	7.5
	10000 mg/L Mg	High Purity Standards	5.0
	10000 mg/L Ca	High Purity Standards	2.5
1000 ug/L Hg Standard	1000 mg/L Hg	Inorganic Ventures	0.10

ELEMENT CONCENTRATIONS IN MATRIX SPIKES AND SPIKING SOLUTIONS				
Element	**CONCENTRATION IN SOLUTION, mg/L**			
	SPLP Matrix Spike	**CLPP-SPK-1**	**CLPP-SPK-INT1**	**1000 ug/L Hg Std.**
Arsenic	2.000		200	
Barium	2.000	2000		
Cadmium	0.050		5	
Chromium	0.200	200		
Lead	0.500		50	
Selenium	2.000		200	
Silver	0.050	50		
Mercury	0.0020			1000

FIGURE 1

ROTARY AGITATION APPARATUS



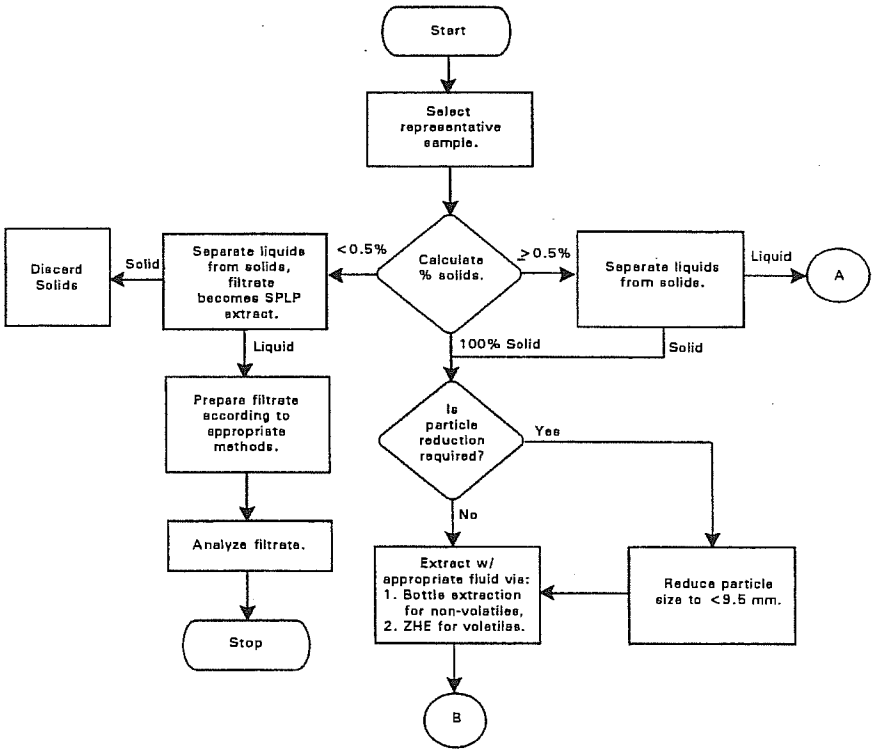
TITLE: SYNTHETIC PRECIPITATION LEACHING PROCEDURE (SPLP) FOR INORGANIC AND NON-VOLATILE ORGANIC ANALYTES

FIGURE 2

SPLP FLOW CHARTS

METHOD 1312

SYNTHETIC PRECIPITATION LEACHING PROCEDURE



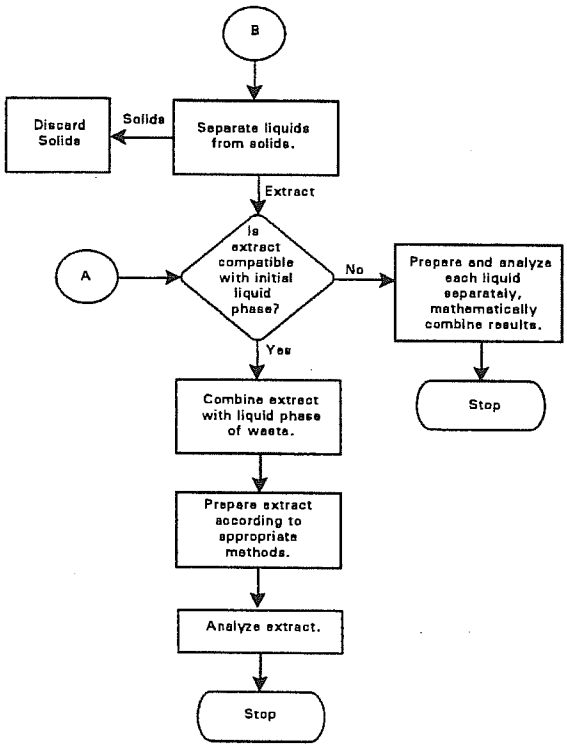
TITLE: SYNTHETIC PRECIPITATION LEACHING PROCEDURE (SPLP) FOR INORGANIC AND NON-VOLATILE ORGANIC ANALYTES

FIGURE 3

SPLP FLOW CHARTS

METHOD 1312

SYNTHETIC PRECIPITATION LEACHING PROCEDURE (continued)



TITLE: SYNTHETIC PRECIPITATION LEACHING PROCEDURE (SPLP) FOR INORGANIC AND NON-VOLATILE ORGANIC ANALYTES

FIGURE 4

SVOA SPLP MATRIX SPIKE AND SURROGATE GUIDELINES

MATRIX SPIKE

The following compounds are reported for SPLP matrix spikes, although a full list spike solution is utilized (refer to SOP CA-502, current revision). Acid extractable compounds are at 100 ug/mL and base/neutral extractable compounds are at 50 ug/mL. 1.0 mL of this mix is added to the sample designated for the SPLP matrix spike.

Pyridine
1,4-Dichlorobenzene
2-Methylphenol
3-,4-Methylphenol*
Hexachloroethane
Nitrobenzene
Hexachlorobutadiene
2,4,6-Trichlorophenol
2,4,5-Trichlorphenol
2,4-Dinitrotoluene
Hexachlorobenzene
Pentachlorophenol

* Due to coelution on the GC/MS, 3-methylphenol and 4-methylphenol are reported as the combined concentration for the two isomers; the matrix spike solution contains 4-methylphenol at 100 ug/mL.

SURROGATE

The following surrogate compounds are reported for SPLP samples, although the surrogate mix also includes one additional surrogate (refer to SOP CA-502, current revision). Acid extractable surrogates are at 100 ug/mL and base/neutral extractable surrogates are at 50 ug/mL. 1.0 mL of this mix is added to all samples.

2-Fluorophenol	100 ug/mL
Phenol-d5	100 ug/mL
Nitrobenzene-d5	50 ug/mL
2-Fluorobiphenyl	50 ug/mL
2,4,6-Tribromophenol	100 ug/mL
Terphenyl-d14	50 ug/mL

FIGURE 5

PESTICIDE SPLP MATRIX SPIKE AND SURROGATE GUIDELINES

MATRIX SPIKE

The following compounds are reported for SPLP matrix spikes, although a full list spike solution is utilized (refer to SOP CA-515, current revision). All compounds are at 0.5 ug/mL. 1.0 mL of this mix is added to the sample designated for the SPLP matrix spike.

Endrin
Heptachlor
Methoxychlor
Lindane
Heptachlor Epoxide

SURROGATE

Surrogates are at 1.0 ug/mL. 1.0 mL of this mix is added to all samples.

Decachlorobiphenyl (DCB)	1.0 ug/mL
Tetrachloro-m-xylene (TCMX)	1.0 ug/mL

TITLE: SYNTHETIC PRECIPITATION LEACHING PROCEDURE (SPLP) FOR INORGANIC AND NON-VOLATILE ORGANIC ANALYTES

FIGURE 6

EXAMPLE PAGE FROM SPLP FLUID USE LOGBOOK

KATAHDIN ANALYTICAL SERVICES, INC.
Non–Volatile TCLP Extraction Fluid Preparation and Use Log

FLUID PREPARATION

SPLP ~~TCLP~~ Fluid # 1 Fluid Batch # 834

Reagent	Manufacturer's Lot Number	Reagent Volume (mL)	Reagent Mass (g)	Fluid Final Volume (L)
Glacial Acetic Acid			~~N.A.~~	20
Sodium Hydroxide	HWH 4-2-08	N.A.		
0.4% HNO_3/0.6% H_2SO_4		5mL	—	

Preparation Date: 21-2-08 Prepared by: DWM Measured pH: 4.15

FLUID USE LOG

	KATAHDIN Sample Number	TCLP Extraction Start Date	Extract To Be Analyzed For: Metals	SVOA	Pest	Herb
1)	SB1596-5A	4-2-08		X		
2)	-2A	↓		↓		
3)	PBT834A					
4)						
5)						
6)						
7)						
8)						
9)						
10)						
11)						
12)						
13)						
14)						
15)						
16)						
17)						
18)						
19)						
20)						

DISK:METFORMS\FLUIDLOG.WK1

TITLE: SYNTHETIC PRECIPITATION LEACHING PROCEDURE (SPLP) FOR INORGANIC AND NON-VOLATILE ORGANIC ANALYTES

FIGURE 7

EXAMPLE PAGE FROM ROTARY EXTRACTOR RPM VERIFICATION LOGBOOK

KATAHDIN ANALYTICAL SERVICES, INC.

ROTARY EXTRACTOR RPM VERIFICATION LOGBOOK

EXTRACTOR #1: TOP SHELF SERIAL NUMBER: NONE
EXTRACTOR #2: MIDDLE SHELF SERIAL NUMBER: 1173
EXTRACTOR #3: BOTTOM SHELF SERIAL NUMBER: 1169

Please record the number of RPMs for each extractor each time they are used.

Date	Initials	Extractor #1	Extractor #2	Extractor #3	Comments
10/30/07	DJJ	Out of Service	Not in use	30	
10/31/07	DJJ	↓	↓	30	
11/1/07	DMP	↓	↓	30	
11/4/07	DJJ	↓	↓	30	
11/6/07	DJJ	↓	↓	30	
11/7/07	DJJ	↓	↓	30	
11/8/07	DJJ	↓	↓	30	
11/13/07	DWM	↓	↓	30	
11/14/07	DMP	↓	↓	30	
11/15/07	DJJ	↓	↓	30	
11/18/07	DJJ	↓	↓	30	
11/19/07	DMP	↓	↓	30	
11/20/07	DJJ	↓	↓	30	
11/26/07	DJJ	↓	↓	30	
11/27/07	DJJ	↓	↓	30	

Acceptance Range is 28-32 RPMS.
Meters Should Be Verified Against A Wrist Watch Annually And Recorded In The Comments Section.

TITLE: SYNTHETIC PRECIPITATION LEACHING PROCEDURE (SPLP) FOR INORGANIC AND NON-VOLATILE ORGANIC ANALYTES

FIGURE 8

EXAMPLE PAGE FROM NON-VOLATILE SPLP EXTRACTION LOGBOOK

KATAHDIN ANALYTICAL SERVICES, INC.
Non-Volatile TCLP / SPLP Extraction Log

Katahdin Sample No.: _____ Extraction Type (Check one): ☐ TCLP ☐ SPLP
Client: _____ Matrix: _____

I. SOLIDS DETERMINATION (Check one box below):

☐ 100% wet solids - waste will obviously yield no liquid upon pressure filtration (proceed to Section II).
☐ <100% solids (perform solids determination below):

A. Weight of filter	_____ g	G. Weight of liquid phase (F-B)	_____ g
B. Weight of filtrate vessel	_____ g	H. Percent wet solids [(E-G)/E x 100]	_____ %
C. Weight of weigh boat + waste	_____ g	Note: Steps I - L are optional. Refer to SOP.	
D. Weight of weigh boat + residue	_____ g	I. Weight of filter + dry solids	_____ g
E. Total weight of waste (C-D)	_____ g	J. Weight of dry solids (I - A)	_____ g
F. Weight of filtrate vessel + filtrate	_____ g	L. Percent dry solids (J/E x 100)	_____ %

II. pH DETERMINATION AND FLUID SELECTION

Initial pH of solid phase: _____ (For TCLP, if <5, proceed to Section III using TCLP Fluid #1. If >5, proceed to next step.)
TCLP only: pH after addition of 3.5 mL of 1 N HCl: _____ (If <5, use TCLP Fluid #1. If >5, use TCLP Fluid #2)
SPLP only: Sample from ☐ east (use SPLP Fluid #1) ☐ west (use SPLP Fluid #2) of Mississippi River.

III. EXTRACTION SETUP (Check one box below):

☐ 100% wet solids: _____ mL of Fluid # _____ (Batch _____) added to _____ g unfiltered waste.
☐ <0.5% dry solids: _____mL of waste filtered (filter sufficient volume to support all required analyses).
☐ >0.5% dry solids and <100% wet solids (perform phase separation below):
_____mL of Fluid # _____ (Batch _____) added to _____ g solid phase of waste.

M. Weight of filtrate vessel	_____ g	Q. Weight of filtrate vessel + filtrate	_____ g
N. Weight of weigh boat + waste	_____ g	R. Weight of liquid phase (Q - M)	_____ g
O. Weight of weigh boat + residue	_____ g	S. Volume of liquid phase	_____ mL
P. Total weight of waste (N - O)	_____ g	T. Weight of wet solids (P - R)	_____ g

Associated Extraction Blank ID: _____ Fluid pH on day of use: _____
Extraction Bottle ID: _____ Fluid expiration date: _____

IV. ROTARY EXTRACTION CONDITIONS (Rotary extraction not required if waste <0.5% dry solids)

Rotary extraction started: Date_____ Time_____ Analyst_____ Room Temp. (degrees C)_____
Rotary extraction completed: Date_____ Time_____ Analyst_____ Room Temp. (degrees C)_____
Elapsed extraction time (HH:MM): _____:_____ pH of extract after extraction: _____
Rotary extraction filtered: Date_____ Analyst_____ Filter Lot Number_____
Was pre-extn. filtrate from Section III combined with rotary extract (check one)? ☐ Yes ☐ No ☐ N.A.
If no, enter the volume of filtrate obtained from the rotary extraction: _____mL
This TCLP/SPLP extract to be analyzed for (check all that apply):
☐ Metals ☐ Semivolatiles ☐ Pesticides ☐ Herbicides

Comments:

ADDENDUM

SOP NO CHANGE FORM

KATAHDIN ANALYTICAL SERVICES, INC.
SOP "REVIEW WITH NO CHANGES" FORM

Name of Person Reviewing SOP: Adrian Biscontini

Review Date: 3/11/09

SOP Number: CA-620

SOP Title: Synthetic Precipitation Leaching Procedure (SPLP) for Inorganic and Non-Volatile Organic Analytes

THE ABOVE REFERENCED SOP HAS BEEN REVIEWED BY A QUALIFIED AND TRAINED ANALYST OR SUPERVISOR. NO CHANGES ARE REQUIRED TO THE SOP AT THIS TIME.

Department Supervisor Signature: A. Drewer

Date: 03/16/09

QAO Signature: Leseio Dimond

Date: 03.17.09

KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

SOP Number: SD-902
Revision History
Cover Page
Page 1

TITLE: **SAMPLE RECEIPT AND INTERNAL CONTROL**

Prepared By: Andrea Colby Date: 6/2002

Approved By:

Group Supervisor: Andrea Colby Date: 6/6/02

Lab Operations Mgr: J. C. Burton Date: 6/5/02

QA Officer: Deborah J. Nadeau Date: 6.6.02

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
04	Changed cover sheet, minor changes to sections 7.1, 7.6, 7.7.4, 7.10 + 7.20. Complete rewrite of sections 7.11 + 7.12 to comply with new KIMS	DN	6.6.02	6.6.02
05	Added verbal date entry to KIMS. Added reference to immediate internal COC book. Added Department Manager reference. Added section 7.7.3. Updated new incoming	DN	05.04	05.04
06	Added procedure + logbook page for checking turbidity of drinking water samples. Changed wet chem shorts board to a book (included example page). Added custody procedures for food/micro. Added VOA soil freezer storage.	DN	01.26.04	01.26.04
07	Added instructions to create lettered labels. Changed sample locations to reflect new building. Removed Figures 8 and 10. Updated Table and Figures w/ current ones. Added wording to sect. 7.7.5 to clarify how pH measurements are taken.	LAD	02/07	02/07
08	Added summary stating sample acceptance policy. Deleted all references to radiation checks (not performed). Add IR gun usage. Reorganized section 7.0 to prioritize time sensitive tasks. Added wireless thermometer monitoring. Updated SRCR. Other minor changes.	DN	~~05/09~~ 08/09 8.4.09	~~05/09~~ 08/09

Added section concerning locking of coolers. Added more detail to 7.18 on unique container IDs. Added more detail on immediate COCs + a section on retention of samples.

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

Please acknowledge receipt of this standard operating procedure by signing and dating both of the spaces provided. Return the bottom half of this sheet to the QA Department.

I acknowledge receipt of copy ___ of document **SD-902-08**, titled **Sample Receipt and Internal Control**.

Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy ___ of document **SD-902-08**, titled **Sample Receipt and Internal Control**.

Recipient: _____ Date: _____

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

1.0 SCOPE AND APPLICATION

Katahdin Analytical Services, Inc. requires the use of specific receiving, acceptance, identification, storage, and distribution procedures for samples it accepts for analyses. These procedures assure that:

- samples are uniquely identified,
- samples are protected from loss or damage,
- essential sample characteristics are preserved,
- any alteration of samples (e.g., filtration, preservation) is documented,
- the correct samples are analyzed, and
- a record of continuous sample custody and utilization is established.

The purpose of this SOP is to describe the procedures used for the receipt and tracking of samples received by Katahdin Analytical Services, Inc. (Katahdin).

1.1 Definitions

SDG: Sample Delivery Group – A group of samples to be reported as one data package.

1.2 Responsibilities

It is the responsibility of all Katahdin staff who receive samples or handle samples in the course of analysis to follow the procedures set forth in this SOP, to document their understanding of the procedures in their training files (refer to Katahdin SOP QA-805, current revision, "Personnel Training & Documentation of Capability"), and to suggest changes and revisions when appropriate. All technical staff are responsible for monitoring their immediate areas, stopping an activity when a problem is detected or suspected, initiating corrective action when needed, documenting any actions taken, and notifying the appropriate individual (e.g., Department Manager, Operations Manager, QAO). The primary responsibility for implementing real-time corrective actions and maintaining an effective QA self-inspection system resides with Katahdin staff. When problems are identified Katahdin personnel are expected to attempt to resolve situations within the scope of their technical knowledge, and to seek assistance from peers and the Department Manager as necessary.

It is the responsibility of Department Managers to oversee the adherence to Katahdin QC practices and internal documentation of laboratory activities within their area, to take corrective actions where needed and communicate problems to the Operations Manager, QAO or Vice President/President when warranted.

It is the responsibility of the Operations Manager to oversee adherence to Katahdin QA/QC practices by all laboratory groups under his/her authority, to help identify

problems and assure resolution, to facilitate corrective action where needed, and to communicate problems and concerns to the QAO and Vice President/President.

It is the responsibility of the Quality Assurance Officer (QAO) to oversee adherence to this SOP, to conduct periodic audits of each laboratory, to track corrective action reports, resolution, and documentation, and to communicate concerns and report findings to the Vice President/President. The QA Officer shall function independently from laboratory operations and be able to evaluate data objectively and perform assessments without outside influence. The QA Officer has the authority to independently halt production operations (including data reporting) if warranted by quality problems.

1.3 Safety

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDSs for all the materials used in this procedure.

Each qualified analyst or technician must be familiar with Katahdin Analytical safety procedures and the Katahdin Environmental Health & Safety Manual and must follow appropriate procedures. These include the use of appropriate personal protective equipment (PPE) such as safety glasses, gloves and lab coats when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location of all safety equipment. Each analyst shall receive a safety orientation from their Department Manager, or designee, appropriate for the job functions they will perform.

1.4 Pollution Prevention/Waste Disposal

Whenever possible, laboratory personnel should use pollution prevention techniques to address their waste generation. Refer to the current revision of the Katahdin Hazardous Waste Management Program for further details on pollution prevention techniques.

Wastes generated during the receipt of samples must be disposed of in accordance with the Katahdin Environmental Health & Safety Manual and SOPs SD-903, "Sample Disposal" and CA-107, "The Management of Hazardous Waste as it Relates to the Disposal of Laboratory Process Waste, Reagents, Solvents and

Standards," current revisions. Expired standards are placed in the Katahdin hazardous waste storage area, and disposed of in accordance with these SOPs.

2.0 SUMMARY OF METHOD

Regulatory, program, and/or method requirements dictate the specifics of sample acceptance. These requirements include, but are not limited to, temperature upon receipt, chemical preservation, container type, sample amount, holding time considerations and complete and accurate documentation of all of these conditions, as well as sample identification. Katahdin's sample acceptance policy is to note any anomalies, discrepancies or non-compliances concerning the receipt of samples. The client is always notified with these issues to direct Katahdin on how and whether to proceed with analysis. All guidance from the client is recorded in the project phone logs and/or on the Sample Receipt Condition Report, which becomes part of the final report. Conditions or analyses performed which do not meet the necessary requirements are narrated or notated as described in the individual analytical SOPs.

3.0 INTERFERENCES

Not applicable.

4.0 APPARATUS AND MATERIALS

4.1 Thermometer – Oakton® Non-Contact Infrared Thermometer, or equivalent, capable of reading 0.1°C and digital probe style capable of reading 0.1°C (used for back-up).

4.2 Capillary tubes – 75 mm Hematocrit Tubes, disposable

4.3 Wide range pH test strips, pH 0 to 14 pH, EMD ColorpHast or equivalent.

4.4 Narrow range pH test strips, pH 0 to 2.5 pH, EMD ColorpHast or equivalent.

4.5 Narrow range pH test strips, pH 11 to 13 pH, EMD ColorpHast or equivalent.

5.0 REAGENTS

Preservatives - refer to Table 1, Sampling and Preservation Requirements, for specifics.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

Refer to Table 1, Sampling and Preservation Requirements, for specifics.

7.0 PROCEDURES

PROCEDURES FOR SAMPLE CUSTODIAN

The following procedures include all steps to be completed for satisfactory receipt and acceptance of samples at Katahdin. These steps do not necessarily have to be performed in the exact order as described. Sample deliveries occur constantly throughout the day, so the sample custodian must multi-task and move back and forth between different procedures to accomplish the most critical tasks of checking receipt temperatures and checking for "RUSH" or quick hold time parameters.

7.1 When samples (except for non-environmental food samples) are dropped off, by either a delivery service (i.e. FEDEX or UPS) or by the client, the Chain-of-Custody (COC) should be signed immediately. The client (who is delivering or that has shipped samples with a delivery service) shall sign (at the lab upon delivery or prior to shipment of samples) that they have relinquished custody to the laboratory. The laboratory shall sign and record the date and time that custody is accepted. (Refer to Figures 1-3 for a Katahdin standard COC, a Katahdin Homeowner COC, and a Katahdin Food/Microbiology COC).

7.2 Cut custody seals and open all coolers. Remove the packets containing the client Chains-of-Custody (COCs).

7.3 Using the COCs, enter the date and time of sample receipt and the client name into the next available work order/login number in the sample receipt logbook (Figure 4). Initial each entry (line) to maintain a record of the individual who assigned each group of samples a discreet lab work order/login number. Record the assigned work order numbers in the appropriate space on the client COCs. Complete the log-in entry date and time once samples are logged in as described below.

7.4 Inventory the COCs for any "RUSH" or quick hold time analyses. Notify the appropriate section managers of these analyses. List any samples for analyses that have short hold times in the "Wet Chemistry Shorts and Rushes Logbook" (Figure 5) in the wet chemistry laboratory. Be sure to list the client, number of samples and date and time of the earliest sample. GC or GC/MS personnel must be informed when ENCORES are received so that they may be scheduled for extrusion. Microbiology personnel should also be informed of any microbiology samples that arrive. Parameters that routinely require short analytical hold times are:

Coliforms	Color	pH
Nitrate/Nitrite	Dissolved Oxygen	Turbidity
Ferrous iron	Orthophosphate	Hex. Chromium
MBAS	TBOD	Free CO_2
Sulfite	ENCORE soil samples	Settleable Solids
Odor	Residual Chlorine	CBOD

7.5 Inspect the condition of custody seals, cooler, ice condition and samples received. Note any non-intact conditions on the Sample Receipt Condition Report (SRCR - Figure 6). Notify the Katahdin project manager (PM) of any discrepancies or problems with sample receipt. The PM contacts the client as necessary. If breakage of a potentially hazardous sample is discovered, close and seal the packing container with all the samples inside and move to a hood in the organic extractions area or to the smaller hood in the login area if space permits. One of the three Katahdin Emergency Response Coordinators or the Katahdin Environmental Health & Safety Manager must be notified. Disposition of the broken and other possibly contaminated samples will be determined on a case-by-case basis in accordance with the laboratory's handling procedures for hazardous waste as outlined in the Katahdin Environmental Health & Safety Manual. Generally, when a sample has broken and has mixed with any ice in the cooler, that liquid will be poured off into 2 liter plastic containers and labeled as "do not use". These containers will be disposed of as soon as the disposition of the appropriate samples has been determined through analysis.

7.6 If there is no breakage of a potentially hazardous sample:

Check cooler temperatures using the IR thermometer assigned to the sample receipt area. If a cooler temperature blank is present, aim the IR gun at the temperature blank; otherwise aim the IR gun at any sample in the cooler if no temperature blank is present. Be sure that the IR gun is within 6 inches of the bottle and not aimed at a label on the bottle. Press the trigger on the handle and be sure the red dot is visible on the bottle surface. The IR gun has been set to read in degrees celcius. If checking the temperature of a plastic bottle, set the emissivity at 0.90. If checking the temperature of a glass bottle (either amber or clear), set the emissivity at 0.85. Refer to Figure 7 for manufacturer's instructions on changing the emissivity. Record the temperature on the Sample Receipt Condition Report. Receipt temperatures should be <6 °C, without freezing. Any temperature falling outside of this range must be noted on the SRCR and reported to the appropriate Katahdin project manager.

Note: Samples received for metals analysis only do not have to meet any temperature receipt requirements.

Note: A probe type thermometer is retained as back-up in case there is a problem with the IR thermometer.

7.7 Note the condition of the ice or ice packs. If the ice has melted and the temperature is out of acceptance criteria, note this on the SRCR. For samples that are hand delivered to the laboratory immediately after collection (i.e. sample collection times are <6 hours old), the temperature blank and/or cooler temperature will most likely not meet the acceptance criteria. The samples shall be considered acceptable if there is evidence that the chilling process has begun such as arrival on ice. Note this on the SRCR. If samples (that were just collected) have not arrived on ice, note this on the SRCR, and start the cooling process as soon as possible after arrival at the laboratory.

Note: All clients must be notified when samples are received that do not meet the appropriate temperature requirements. In these cases, certain regulatory requirements may not be met and may invalidate certain data.

7.8 Inventory the samples against the chain of custody (COC). If the COC is incomplete, the sample custodian must inform the appropriate Katahdin project manager (PM). The PM may make changes to correct or complete the COC, but all changes must be initialed and dated. Changes must be noted on the SRCR. Any discrepancies between the samples and the COC must also be noted on the SRCR.

7.9 Using the Sampling and Preservation Requirements Table (Table 1) as a reference, check if samples are in proper containers and received correct pretreatment (e.g., filtration, preservation) for the analyses requested. For aqueous parameters requiring preservation, check pH by inserting a clean capillary tube into the sample and dabbing the tube on wide range pH paper. If the pH is not clearly either less than 2 or greater than 12, the appropriate narrow range pH paper must be used. NOTE: The pH of volatile organic (VOA) samples is checked and recorded by the analyst after completion of analysis and not by sample receipt personnel. The used capillary tube is discarded and a new capillary tube is used for each sample.

Additional preservative is added to samples if the pH is not in the range specified in the Sampling and Preservation Requirements Table. No more than 10% of the original sample volume should be added as preservative. If the client has noted that the sample reacts violently (i.e., foams and bubbles) upon preservation, add no more preservative to the sample. Some clients may wish to be contacted if their samples are found to be improperly preserved. Record all preservation discrepancies on the Sample Receipt Condition Report including the lot number of the preservative added. If additional preservative is added, a sticker with the type of preservative must be placed on the sample container.

Note: Preservatives are obtained from the larger containers in the bottle preparation area.

Note: If samples are received unpreserved for 200.7 or 200.8 analysis, the samples must be preserved to pH <2 with nitric acid. Samples must be held for 16 hours after preservation before sample preparation can begin.

7.10 For samples requiring filtration as pretreatment (i.e. for dissolved metals), the work order/login numbers are recorded in the filtration logbook (see Figure 8). The samples are filtered by the Metals Group.

7.10.1 A 500 mL filter flask and filter funnel are acid rinsed three times in a 10% nitric acid bath, then three times with Laboratory Reagent Grade Water.

7.10.2 A vacuum pump is attached.

7.10.3 A 0.45 micron filter is rinsed three times with 5% nitric acid and three times with Laboratory Reagent Grade Water. The rinsate is discarded.

7.10.4 A sufficient sample aliquot is filtered and preserved with concentrated nitric acid to pH <2.

7.10.5 The bottles are labeled with the work order/login number and other sample information and stored at <6 ° C until the time of digestion.

7.11 Using the Sampling and Preservation Requirements Table (Table 1) as a reference, determine if sufficient volume of sample is present for analysis. Note discrepancies on the SRCR.

7.12 For drinking water samples, enter the appropriate information (work order, date, etc.) into the Measured Turbidity and Preservation of Incoming Samples Logbook. Inform the appropriate analyst of the sample. The turbidity must be measured prior to sample preparation. If the turbidity is <1 NTU, the sample does not have to be digested prior to metals analysis. If the turbidity is >1 NTU, the sample must be digested prior to metals analysis. The sample must be preserved after the turbidity measurement is taken. Record the appropriate information in the logbook (Figure 9).

7.13 Notify the PM immediately if there are any discrepancies or problems with sample receipt. The PM will contact the client for information and resolution as necessary. All decisions to proceed or not to proceed with analysis associated with samples received that do not meet specified acceptance criteria (i.e. cooler temperature, preservation, container, etc.) must be fully documented on the SRCR. Although this form is included with all client reports, additional narration or flagging of data may be necessary.

7.14 Review any additional paperwork that accompanies the sample(s) submitted for analysis along with laboratory-generated information. This includes shipping forms, letters, chain-of-custody forms, sample labels, Incoming Sample Information Sheets (ISIS), quotes, memos, etc. These forms may provide details on specific client

requests. The ISIS will provide information on specifics for log-in. Refer to Figure 10 for an example.

7.15 Resolve any questions or concerns raised by steps 7.1-7.14 by consulting the correspondence files or client services personnel or communicating directly with the client. Note in the notes section of the SRCR any deviations from normal sample handling or analytical procedures (e.g., client requests analysis although hold-time expired).

7.16 When non-environmental food samples are delivered to the laboratory, they are taken immediately to the food/microbiology laboratory and stored in the refrigerators there. A copy of the Chain-of-Custody is left with the analysts. The original paperwork is forwarded to sample log in where the job is logged into the KIMS system.

7.17 The following information is documented via the Katahdin Information Management System (KIMS) and a work order/login COC report (Figure 11) is generated for the samples received:

7.17.1 Log onto KIMS by entering employee ID under "Username", employee specific password under "Password" and KIMS under "Database".

7.17.2 Once logged onto KIMS select "Sample Management" and then "Login".

7.17.3 Select "New" and the next available Login ID number will automatically be entered. Select "OK" and the Sample Definition screen will open.

Note: If a Work Order number has already been opened, select "change" and type in the appropriate number to access the information.

7.17.4 In the Sample Definition Screen, enter the following information.

Client ID -	Enter client sample description.
ReceiveDate -	Enter in date that samples were received in the lab in the format YY-Month-DD.
CollectDate -	Enter in date that samples were collected in the format YY-Month-DDTIME.
TAT -	Enter TAT for hardcopy report.
DueDate -	Due date will automatically be calculated based on calendar days.
VerbalDate -	Manually type in verbal due date.

QuoteRef -	Enter quote number if applicable.
Project -	Enter project number if applicable.
Account -	Enter client specific account number.
Account Name -	Account name will automatically be entered.
Collected By -	Enter name/initials of sampler listed on COC. If unknown, enter "Client".
Locator -	May be used for client ID information when requested by the project manager.
Site -	Enter project site name.
Description -	May be used for long client Ids when requested by the project manager.
Discount -	No entry-not currently used.
Priority -	No entry-not currently used.
Fact. -	No entry-not currently used.
Expected -	No entry-not currently used.
Comments -	Enter MS/MSD, verbal due date and any sample irregularities if applicable.
OrderDate -	Current date is automatically entered.
Matrix -	Enter sample matrix code where

AQ = Aqueous	SLD = Food Solid
SL = Solid, Soil, Sludge	AR = Air
FP = Free Product	SWAB = Swab
WP = Wipe	SAL = Saline
NOAQ = NonAqueous	TIS = Tissue
DW = Drinking Water	

Product Code -	Enter analysis code per test requested on COC.
Type -	Product code type will automatically be entered where S = Stand alone

P = Parent
C = Children

Fact. - No entry-default is 1.

Price - This is left as is by sample log-in. During project management review of the work order, the prices are entered based on quotes or standard prices.

Cost - No entry needed.

Lev - No entry needed.

Type - Container type will automatically be entered.

Bot - Enter number of containers per test for printing of labels.

Login Info - Parameter Data Screen will open. Enter following information

KAS Proj. Manager-	Initials of Katahdin person overseeing the project.
Client PO#-	Client purchase order.
Project-	Project name.
Cooler Temperature-	Temperature blanks or cooler temps.
Delivery Services-	Method of delivery to the lab.
QC Level-	QC Level of report and regulatory agency (ie., IV NFESC).
SDG ID-	Sample Delivery Group ID if applicable.
SDG Status-	Begin, Continue or End.
Analysis Instructions-	PM will enter special instructions regarding project.
Report Instructions-	PM will enter special instructions regarding project.
Regulatory List-	Used for federal programs.
EDD Format-	Specific KAS EDD format.

Select "SAVE" and then "CANCEL".

Addresses - Select "Addresses" and the Address Links screen will open. The billing address is the default address of the

account. Enter the client account code under "Project/Account" and select the report to contact under "Address Type". Select the appropriate boxes for report, report CC and invoice CC. Select "SAVE" and then "CLOSE".

Create Containers - Select "Create Containers". Letters will be assigned to each sample number. Select "OK" until letters have been assigned to each sample number. To manually assign letters, Select "Enter Container IDs" and "OK". Enter sample numbers including letters and select "OK", "Close", "Yes" to save changes, "Cancel" and "Cancel".

7.17.5 To print the login report, select "Reports", "Login" and "Login COC". Enter login number under "Login Number". Select "OK", "Run Report" and then "Print".

7.18 To print labels unique to each bottle, select "Reports", "Login" and "Labels". Enter login number under "Login/Prelogin", select "Background (IDXL)" and select F9 on keyboard under "Select Sample Label". Select "OK" and then "Print". After labels print out select "Cancel".

Note: As stated in "create containers" above, each sample bottle is assigned a unique ID. The job is given a work order number. Each different client sample ID is given a numerical number following the work order number and each sample container with the same client ID is given a container ID using alphabetical letters. This series of work order, sample number and container ID is transcribed throughout the raw data for traceability purposes.

Example: One job containing one client sample with 3 different containers:

SC9001-001(a), SC9001-001(b), SC9001-001(c)

Example: One job containing two client samples with 2 different containers for each:

SC9002-001(a), SC9002-001(b), SC9002-002(a), SC9002-002(b)

7.19 Affix permanent sample number labels to sample containers, assuring that sample IDs on labels correspond to sample bottle IDs. Do not obscure client ID on the bottles.

7.20 Place samples in their designated storage locations and log them in, noting initials, date and time, work order/login and sample numbers, and storage location on the internal laboratory chain of custody form (Figure 12). Place form in the appropriate

binder in the log in area. Non-environmental food samples do not get an internal COC and are taken immediately to the food/microbiology lab for storage.

Storage location of the samples is determined by type of sample and/or type of analysis, as outlined below. Most samples are stored in the walk-in cooler, which is organized by test type and work order/login number.

Specific storage locations are described below.

7.20.1 Aqueous samples for wet chemistry (except hardness, see 7.19.4 below) - left aisle, both sides, as you enter walk-in cooler. TOC vials are to be stored in the trays designated for TOC samples.

7.20.2 Aqueous samples for organic extractions – right aisle, left side, as you enter walk-in cooler.

7.20.3 Non-aqueous samples (all analyses except volatile organics) - to the right and towards the back as you enter walk-in cooler. Non-aqueous samples for volatile organics are stored in "VOA Refrigerator 2" located in the Volatiles Laboratory.

7.20.4 Aqueous samples for metals and/or hardness analyses – right aisle, right side towards the front as you enter walk-in cooler.

7.20.5 Samples (aqueous and solid) for volatile organics analyses (VOA) – All aqueous samples and soil samples in VOA vials (preserved with methanol or sodium bisulfate) are stored in "VOA Refrigerator 1" in the Volatiles Laboratory. VOA soils in jars or ENCORE samplers are stored in "VOA Refrigerator 2" in the Volatiles Laboratory. VOA samples known or suspected to be hazardous (such that cross-contamination of other samples might occur) are placed in a "paint can" and stored in the walk-in.

7.20.6 Soil samples for volatile organics analyses (VOA) that are unpreserved or preserved with Laboratory Reagent Grade Water are stored in "VOA Freezer 1" in the volatiles laboratory.

Sample storage coolers are not locked, but internal chain-of-custody is documented with respect to native samples, extracts and digestates within the laboratory. The laboratory maintains a secure facility with respect to unauthorized personnel, as described in the current revision of Katahdin SOP, AD-004, Laboratory Facility Security and Confidentiality. All sample storage coolers are equipped with locks if specific project or regulatory requirements deem it necessary.

7.21 Sample Receipt gives the Work order/login COC report and confirmation of the job, as logged-in, to the appropriate Katahdin project manager. All chain-of-custody and

other receipt documentation must accompany the job. The project manager reviews the job for accuracy and completeness. Any unresolved issues should be resolved at this time. Any project or program specific forms should be included with the paperwork at this time. These forms may include CLP forms or state-specific forms. The project manager then dispatches the work order/login to the individual department worklists. The dispatched work order/login package is then filed in Data Management where the complete package will eventually be compiled.

7.22 The temperature of all sample storage refrigerators and freezers is recorded daily by assigned individuals. Notebooks containing a record of each refrigerator and freezer temperature history are used for this purpose and are maintained by the assigned individuals. Temperatures above or below the acceptance range are to be brought to the attention of a Department Manager, Operations Manager, or Quality Assurance Officer. Such an occurrence and the actions taken to correct it must be noted in the comments column of the temperature recording notebook next to the temperature measurement. (See Figure 13).

Additionally, temperatures of storage units are monitored continuously by wireless thermometers. A temperature is recorded electronically every 10 minutes. The QAO can generate a specified report as needed, including every reading or maximum/minimum temperatures for a given timeframe. These monitoring devices ensure continual compliance seven days per week. The data can be used to check for problems.

PROCEDURES FOR CHEMISTS

7.23 When removing a sample from its storage location, record on the laboratory internal chain-of-custody (from the appropriate department) the sample number, date and time it was removed, chemist who removed it, and the analysis to be conducted or reason for removal.

7.24 If the samples have not been logged in yet and they need to be pulled in order to analyze short holding time parameters, the analyst taking the sample must use the designated logbook (Immediate Internal COC – Figure 14) to sign the samples out. Many circumstances lead to analysts having to pull samples before they are logged into the KIMS system. It is everyone's responsibility to ensure that all samples can be accounted for at all times. Failure to do so can create confusion and bottle necks for others trying to access the samples. Samples that are pulled before log-in must be returned to the designated bin in the sample receipt area. When the logbook for Immediate Internal COC's is used, the standard internal COC's do not have to be signed at a later date. The Immediate Internal COC Logbook must always be consulted if there is ever a question about whether an internal COC has been completed.

7.25 If a sample is not consumed by an analysis, return the remaining sample to its assigned storage location and enter the date and time returned on the laboratory internal chain-of-custody record.

7.26 If analysis consumes the entire sample, indicate this on the laboratory internal chain-of-custody record.

7.27 After the completion of all analyses, the original "left over" sample containers will remain in sample storage until their final disposal. Samples are held during this period for the purposes of retesting if required by a laboratory corrective action or by a client. Refer to the current revision of Katahdin SOP, SD-903, Sample Disposal, for details on final disposal of samples.

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

Each thermometer used to monitor sample storage or cooler temperatures must be calibrated annually against a NIST traceable thermometer. The QAO is responsible for ensuring that the thermometer(s) are scheduled for annual calibration and for maintaining the calibration records. All other procedures and documentation listed in this SOP must be followed at all times.

9.0 METHOD PERFORMANCE

Not applicable.

10.0 APPLICABLE DOCUMENTS/REFERENCES

"Handbook for Analytical Quality Control in Water and Wastewater Laboratories," U.S. EPA EMSL Office of Research and Development, March 1979.

Code of Federal Regulations 40, Parts 136 and 141.

"Test Methods for Evaluating Solid Waste: Physical/Chemical Methods," SW-846 Chapters 1 & 2, USEPA, Third Edition, including Updates I, II, IIA, and IIB, III June, 1997.

Katahdin Analytical Services, Inc., Environmental Health & Safety Manual, current revision.

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Figure 14 Example of Immediate Internal COC Logbook

TABLE 1

SAMPLING AND PRESERVATION REQUIREMENTS

PARAMETER – AQUEOUS MATRICES	METHOD	QUANTITY	CONTAINER	PRSV	HOLD TIME
GENERAL CHEMICAL ANALYSES					
Acidity	305.1	100 mL	P,G	1,2	14 days
Alkalinity-Manual Titrimetric	310.1	100 mL	P,G	1,2	14 days
Ammonia-Nitrogen with distill-Auto. Phenate	350.1	1 L	P,G	1,3	28 days
Ammonia-Nitrogen-Automated Phenate	350.1, 350.2	250 mL	P,G	1,3	28 days
Anions (Cl, Br, SO4, NO2, NO3)	300.0	250 mL	P, G	1	48hr/28days
Bicarbonate, Carbonate (see pH & alkalinity)	calc.				
Biochemical Oxygen Demand-Carbonaceous	405.1	1 L	P,G	1	48 hours
Biochemical Oxygen Demand-Total	405.1	1 L	P,G	1	48 hours
Bromide	320.1	500 mL	P,G	1	28 days
Chemical Oxygen Demand-Manual Colorimetric	410.4	100 mL	P,G	1,3	28 days
Chloride-Automated Ferricyanide	325.2	100 mL	P,G	1	28 days
Chlorine, Residual	SM4500-Cl G	100 mL	P,G	1,9	ASAP
Chromium, Hexavalent	SM3500Cr D / SW7196	200 mL	P,G	1,9	24 hours
Color, Apparent	110.2	100 mL	P,G	1,2	48 hours
Cyanide, Amenable-Spectrophotometric	335.1	250 mL	P,G	1,5	14 days
Cyanide, Total-Spectrophotometric	SM4500CN C 335.3, 335.4	250 mL	P,G	1,5	14 days
Dissolved Oxygen(Lab)-Membrane Electrode	360.1	500 mL	G	1	ASAP
Ferrous Iron - Colorimetric	SM3500-Fe D	250mL	P	1	24 hrs
Fluoride with distillation, Potentiometric ISE	SM4500F C/340.2	500 mL	P only	1	28 days
Fluoride, Potentiometric ISE	340.2	200 mL	P only	1	28 days
Free CO_2	SM4500-CO_2 C	250mL	P	1	24 hrs.
Hardness, Total-Manual Titrimetric	130.2,SM2340C	250 mL	P,G	4	6 months
MBAS, Extraction-Colorimetric	SM5540C	1 L	P,G	1	48 hours
Nitrate+Nitrite-Automated Cadmium Reduction	353.2	100 mL	P,G	1,3	28 days
Nitrate-Automated Cadmium Red./Diazotization	353.2	100 mL	P,G	1	48 hours
Nitrite-Automated Diazotization	353.2	100 mL	P,G	1	48 hours
Oil & Grease-Total Recoverable, Gravimetric N-Hexane extractable material N-Hexane extractable material w/ silica gel cleanup	1664	(2) 1 L	glass only	1,11	28 days
pH (Laboratory)	150.1	100 mL	P,G	1,2	24 hours
Phenolics, Total Recoverable-Manual 4AAP	420.1	1000 mL	glass only	1,3	28 days
Phosphate, Ortho- Ascorbic Acid	365.2	100 mL	P,G	1	48 hours
Phosphate,Total	365.4	100 mL	P,G	1,3	28 days
Solids-Filterable Residue (TDS),Gravimetric180	160.1	250 mL	P,G	1	7 days
Solids-Nonfilterable Residue (TSS)	160.2	500 mL	P,G	1	7 days
Solids-Settleable Solids (SS)	160.5	1 L	P,G	1	48 hours

TABLE 1 (cont.)

SAMPLING AND PRESERVATION REQUIREMENTS

PARAMETER – AQUEOUS MATRICES	METHOD	QUANTITY	CONTAINER	PRSV	HOLD TIME
GENERAL CHEMICAL ANALYSES					
Solids-Total Solids	160.3	250 mL	P,G	1	7 days
Solids-Total Volatile (TVS)	160.4	250mL	P,G	1	7 days
Solids-Volatile Filterable Residue (VDS)	160.1/160.4	250 mL	P,G	1	7 days
Solids-Volatile Nonfilterable Residue (VSS)	SM 2540 F	500 mL	P,G	1	7 days
Specific Conductance-Wheatstone Bridge	120.1	100 mL	P,G	1,2	28 days
Sulfate-Turbidimetric	375.4	100 mL	P,G	1	28 days
Sulfide-Iodometric	376.1	500 mL	P,G	1,7	7 days
Sulfite-Titrimetric	377.1	500 mL	P,G	1,9	ASAP
Tannin/Lignin-Colorimetric	SM 5550 B	100 mL	P,G	1	7 days
TKN-Auto Block Digest, Spect.	351.2	100 mL	P,G	1,3	28 days
Total Inorganic Carbon	415.1	(2) 40 mL	VOA vial	1	28 days
Total Inorganic Carbon if with TOC	415.1	(2) 40 mL	VOA vial	1	28 days
Total Organic Carbon-Oxidation	415.1	(2) 40 mL	VOA vial	1,3	28 days
Total Organic Halogen	9020	500 mL	Amber Glass	1,3	28 days
Turbidity	180.1	100 mL	P,G	1	48 hours
ELEMENTAL ANALYSES					
Chromium, Hexavalent	7196/6010	500 mL	P,G	1,9	24 hrs
GFAA(Furnace) Elements	SM 3113/ 200 series	500 mL	P,G	4	6 months
ICP Elements	200.7/6010	500 mL	P,G	4	6 months
ICP MS Elements	200.8/6020	500 mL	P,G	4	6 months
Low Level Mercury	1631	500 mL	G	NA	90 days
Mercury	245.1/7470	500 mL	P,G	4	28 days
GC ORGANIC ANALYSES					
BTEX & MTBE	602 & 8021	(2) 40 mL	VOA vial	1,8,9	14 days(~)
EDB, DBCP & 1,2,3-TCP	504.1	(2) 40 mL	VOA vial	1,8,9	14 days(~)
Extractable Petroleum Hydrocarbons	MADEP/EPH	(2) 1000 mL	Amber Glass	12	14days/40days
Formaldehyde	556	(2) 40 mL	VOA vial	1,8,9	14 days(~)
Fuel Oil in Water	8015Modified	(2) 1000 mL	Amber Glass	1,8	7days/40days
Fuel Oil in Water	ME HETL 4.1.25	(2) 1000 mL	Amber Glass	1,8	7days/40days
Gasoline in Water	8015Modified	(2) 40 mL	VOA vial	1,8	14 days
Gasoline in Water	ME HETL 4.2.17	(2) 40 mL	VOA vial	1,8	14 days
Glycols	8015Modified	(2) 40 mL	VOA vial	1,8,9	14 days(~)
Herbicides	8151	(2) 1000 mL	Amber Glass	1	7days/ 40days
Methane, Ethane & ethene	RSK 175	(2) 40 mL	VOA vial	1,8,9	14 days(~)
PCB's (& Congeners)	608 & 8082	(2) 1000 mL	Amber Glass	1	7days/40days

TABLE 1 (cont.)

SAMPLING AND PRESERVATION REQUIREMENTS

PARAMETER – AQUEOUS MATRICES	METHOD	QUANTITY	CONTAINER	PRSV	HOLD TIME
GC ORGANIC ANALYSES					
Pesticides	608 & 8081	(2) 1000 mL	Amber Glass	1	7days/40days
Pesticides and PCB's	608 & 8081/8082	(2) 1000 mL	Amber Glass	1	7days/40days
Purgeable Aromatics	602 & 8021	(2) 40 mL	VOA vial	1,8,9	14 days(~)
Purgeable Halocarbons	601 & 8021	(2) 40 mL	VOA vial	1,8,9	14 days(~)
Purgeables, Total	601 & 602	(2) 40 mL	VOA vial	1,8,9	14 days(~)
Purgeables, Total	8021	(2) 40 mL	VOA vial	1,8,9	14 days(~)
Solvents (Direct Injection)	8015M	(2) 40 mL	VOA vial	1	14 days
Volatile Petroleum Hydrocarbons	MADEP/VPH	(2) 40 mL	VOA vial	11	14days
GC/MS ORGANIC ANALYSES					
Acid Extractables-Priority Pollutants	625	(2) 1000 mL	Amber Glass	1	7days/40days
Acid Extractables-TCL	8270	(2) 1000 mL	Amber Glass	1	7days/40days
Base Neutral Extract.-Priority Pollutants	625	(2) 1000 mL	Amber Glass	1	7days/40days
Base Neutral Extractables-TCL	8270	(2) 1000 mL	Amber Glass	1	7days/40days
Drinking Water Volatiles - Low Level	524.2	(3) 40 mL	VOA vial	1,8,9,10	14 days(~)
PCB Homologues	680	(2) 1000 mL	Amber Glass	1	7days/40days
Polyaromatic Hydrocarbons	8270/8270 SIM	(2) 1000 mL	Amber Glass	1	7days/40days
Semivolatile Extractables-Priority Pollutants	625	(2) 1000 mL	Amber Glass	1	7days/40days
Semivolatile Extractables-TCL	8270	(2) 1000 mL	Amber Glass	1	7days/40days
Volatile Organics	8260	(2) 40 mL	VOA vial	1,8,9	14 days(~)
Volatile Organics-Priority Pollutants	624	(2) 40 mL	VOA vial	1,8,9	14 days(~)
HPLC ANALYSES					
HPLC-Explosives	8330, 8332	(2) 1000 mL	Amber Glass	1	7days/40days
MICROBIOLOGICAL ANALYSES					
Coliform, Fecal	SM 9222D, SM 9213D Mod.	100 mL	P,G	1,6	6 hours
Coliform, Total	SM 9222B	100 mL	P,G	1,6	30 hours
Coliform and E-coli, Total	SM9223B/Colitag	100 mL	P,G	1,6	30 hours
E-coli	SM9213D, Colilert/Quantitray	100 mL	P,G	1,6	6 hours
Heterotrophic Plate Count	SM9215B SIMPLATE	100 mL	P,G	1,6	30 hours

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

TABLE 1 (cont.)

SAMPLING AND PRESERVATION REQUIREMENTS

PARAMETER – SOLID MATRICES	METHOD	QUANTITY	CONTAINER	PRSV	HOLD TIME
GENERAL CHEMICAL ANALYSES		**4 oz=100 g**			
% Carbon	9060 mod.	4 oz	Soil Jar	1	28 days
Ammonia-Nitrogen-Automated Phenate	350.1 mod.	4 oz	Soil Jar	1	28 days (^)
Anions	9056	4 oz	Soil Jar	1	48hrs to 28 days from slurry (^)
Cation Exchange Capacity	9081	4 oz	Soil Jar	1	14days/7days (^)
Chloride-Automated Ferricyanide	9251/300.0	4 oz	Soil Jar	1	28days from slurry (^)
Cyanide, Amenable-Spectrophotometric	9012	4 oz	Soil Jar	1	14 days
Cyanide, Total-Spectrophotometric	9012	4 oz	Soil Jar	1	14 days
Fluoride, Potentiometric ISE	300.0 mod./340.2	4 oz	Soil Jar	1	28 days (^)
Lime Equivalency	310.1 mod.	4 oz	Soil Jar	1	28 days (^)
Nitrate+Nitrite-Automated Cadmium Reduction	300.0 mod./353.2	4 oz	Soil Jar	1	28 days (^)
Nitrate-Automated Cadmium Red./Diazotization	300.0 mod./353.2	4 oz	Soil Jar	1	48 hrs from slurry (^)
Nitrite-Automated Diazotization	300.0 mod./353.2	4 oz	Soil Jar	1	48 hrs from slurry (^)
Oil & Grease-Total Recoverable, Gravimetric N-Hexane extractable material N-Hexane extractable material w/ silica gel cleanup	9071	4 oz	Soil Jar	1	28 days (^)
Organic Nitrogen-Auto. Block Digest.,Spectro.	350.1/351.2 mod.	4 oz	Soil Jar	1	28 days (^)
pH (Laboratory)	9045	4 oz	Soil Jar	1	24 hours (^)
Phenolics, Total Recoverable-Manual 4AAP	Mod. 9065	4 oz	Soil Jar	1	28 days (^)
Phosphate, Ortho- Ascorbic Acid	300.0 mod./365.2	4 oz	Soil Jar	1	48 hrs from slurry (^)
Phosphate,Tot.-Auto Ascorbic Acid/Block Dig.	Mod. 365.4	4 oz	Soil Jar	1	28 days (^)
Solids-Ash	SM 2540 F	4 oz	Soil Jar	1	28 days (^)
Solids-Total Solids	CLP-CIP	4 oz	Soil Jar	1	28 days (^)
Solids-Volatile Solids	SM 2540 F	4 oz	Soil Jar	1	28 days (^)
Specific Conductance-Wheatstone Bridge	Mod. 9050	4 oz	Soil Jar	1	28 days (^)
Sulfate-Turbidimetric	9036/9038	4 oz	Soil Jar	1	28 days from slurry (^)
Sulfide-Iodometric	9030	4 oz	Soil Jar	1	7days from slurry (^)
Sulfide-Monier-Williams	40CFR-425	4 oz	Soil Jar	1	28 days (^)
Sulfite-Titrimetric	ASTM D3987/377.1 mod.	4 oz	Soil Jar	1	24 hrs from slurry (^)
TKN-Auto Block Digest,Spectro.	351.2 mod.	4 oz	Soil Jar	1	28 days (^)
Total Organic Halogen	9020/9021	4 oz	Soil Jar	1	28 days (^)
Total Petroleum Hydrocarbons-Extraction, IR	9071	4 oz	Soil Jar	1	28 days (^)
ELEMENTAL ANALYSES					
ICP Elements	6010	4 oz	Soil Jar	1	6 months
ICP MS ELements	6020	4 oz	Soil Jar	1	6 months
GFAA(Furnace) Elements	7000series	4 oz	Soil Jar	1	6 months
Mercury	7471	4 oz	Soil Jar	1	28 days

TABLE 1 (cont.)

SAMPLING AND PRESERVATION REQUIREMENTS

PARAMETER – SOLID MATRICES	METHOD	QUANTITY	CONTAINER	PRSV	HOLD TIME
ELEMENTAL ANALYSES **(cont.)**		**4 oz=100 g**			
Chromium, Hexavalent	3060/7196	4 oz	Soil Jar	1	30dys/24hrs
GC ORGANIC ANALYSES					
BTEX & MTBE	8021	(2) 40 mL	VOA Vial	1	14 days
Explosives - HPLC	8330, 8332	4 oz	Soil Jar	1	14days/40days
Extractable Petroleum Hydrocarbons	MADEP/EPH	4 oz	Soil Jar	1	7days/40days
Fuel Oil	ME HETL 4.1.25	4 oz	Soil Jar	1	14days/40days
Fule Oil	8015 mod.	4 oz	Soil Jar	1	14days/40days
Gasoline	ME HETL 4.2.17	(2) 40 mL	VOA Vial	1	14 days
Gasoline	8015 mod.	(2) 40 mL	VOA Vial	1	14 days
Herbicides	8151	4 oz	Soil Jar	1	14days/40days
PCB's (& Congeners)	8082	4 oz	Soil Jar	1	14days/40days
PCB's in Oil	8082	4 oz	VOA Vial	1	40 days
Pesticides	8081	4 oz	Soil Jar	1	14days/40days
Pesticides and PCB's	8081/8082	4 oz	Soil Jar	1	14days/40days
Purgeable Aromatics	8021	(2) 40 mL	VOA Vial	1	14 days
Purgeable Halocarbons	8021	(2) 40 mL	VOA Vial	1	14 days
Purgeables, Total	8021	(2) 40 mL	VOA Vial	1	14 days
Solvents (Direct Injection)	8015M	(2) 40 mL	VOA Vial	1	14 days
Volatile Petroleum Hydrocarbons	MADEP/VPH	(2)40 mL	VOA vial	13	28days
HPLC ANALYSES					
HPLC-Explosives	8330, 8332	4 oz	Soil Jar	1	7days/40days
GC/MS ANALYSES					
Acid Extractables-Priority Pollutants	8270	4 oz	Soil Jar	1	14 days/40 days
Acid Extractables-TCL	8270	4 oz	Soil Jar	1	14 days/40 days
Base Neutral Extractables-Priority Pollutants	8270	4 oz	Soil Jar	1	14 days/40 days
Base Neutral Extractables-TCL	8270	4 oz	Soil Jar	1	14 days/40 days
Polyaromatic Hydrocarbons	8270/8270SIM	4 oz	Soil Jar	1	14 days/40 days
Semivolatile Extractables-Priority Pollutants	8270	4 oz	Soil Jar	1	14 days/40 days
Semivolatile Extractables-TCL	8270	4 oz	Soil Jar	1	14 days/40 days
Volatile Organics – High Soil (>200 ug/kg)	5035/8260	Please refer to Table 6-2	Encore or similar sampler or VOA Vial or soil jar	14	Extruded w/in 48 hrs. Analyzed w/in 14 days
Volatile Organics – Low Soil (<200 ug/kg)	5035/8260	Please refer to Table 6-2	Encore or similar sampler or VOA Vial	14 or 15	Extruded w/in 48 hrs. Analyzed w/in 14 days
Volatile Organics-Priority Pollutants	8260	(2) 40 mL	VOA Vial	1	14 days
Volatile Organics-TCL	8260	(2) 40 mL	VOA Vial	1	14 days

TABLE 1 (cont.)

SAMPLING AND PRESERVATION REQUIREMENTS

PARAMETER – SOLID MATRICES	METHOD	QUANTITY	CONTAINER	PRSV	HOLD TIME
RCRA - HAZARDOUS WASTE CHARAC.					
Corrosivity-pH	9045	4 oz	Soil Jar	1	24 hours (^)
Ignitability-Flash Point (closed cup)	1010	4 oz	Soil Jar	1	14 days (^)
Reactivity-Reactive Cyanide	7.3.3.2	4 oz	Soil Jar	1	14 days
Reactivity-Reactive Sulfide	7.3.4.1	4 oz	Soil Jar	1	7 days
TCLP					
TCLP Extraction-Volatile Organics	1311	100 g	Soil Jar	1	14 days
TCLP Extraction-Semivolatiles	1311	200 g	Soil Jar	1	14 days
TCLP Extraction-Pesticides & Herbicides	1311	400 g	Soil Jar	1	14 days
TCLP Extraction-Metals	1311	200 g	Soil Jar	1	28 days
TCLP Analysis-Volatile Organics	8260	see above	Soil Jar	1	14 days
TCLP Analysis-Metals	6010/6020	see above	Soil Jar	1	180 days
TCLP Analysis-Mercury	7470	see above	Soil Jar	1	28 days
TCLP Analysis-Semivolatiles	8270	see above	Soil Jar	1	7 days/40 days
TCLP Analysis-Pesticides	8081	see above	Soil Jar	1	7 days/40 days
TCLP Analysis-Herbicides	8151	see above	Soil Jar	1	7 days/40 days

METHODS OF PRESERVATION
1 = Cool at 4 Degrees Celsius
2 = Settled
3 = H_2SO_4 to pH<2
4 = HNO_3 to pH<2
5 = NaOH to pH>12
6 = 1 mL 0.1M $Na_2S_2O_3$ or 1 10 mg pellet
7 = 1 m/L 2NZnAc/L & NaOH
8 = 2 drops 1:1 HCl
9 = No headspace
10 = $Na_2S_2O_3$, if chlorinated
11 = HCl to pH < 2
12 = 5 mL of HCL
13 = 15 mL of methanol
14 = methanol
15 = sodium bisulfate

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

FIGURE 1

EXAMPLE OF STANDARD KATAHDIN CHAIN-OF-CUSTODY FORM

Katahdin ANALYTICAL SERVICES
600 Technology Way
Scarborough, ME 04074
Tel: (207) 874-2400
Fax: (207) 775-4029

CHAIN of CUSTODY

PLEASE BEAR DOWN AND PRINT LEGIBLY IN PEN

Page ____ of ____

Client | Contact | Phone # ( ) | Fax # ( )

Address | City | State | Zip Code

Purchase Order # | Proj. Name / No. | Katahdin Quote #

Bill (if different than above) | Address

Sampler (Print / Sign) | Copies To:

LAB USE ONLY | WORK ORDER #:
KATAHDIN PROJECT NUMBER _____

REMARKS: _____

SHIPPING INFO: ☐ FED EX ☐ UPS ☐ CLIENT

AIRBILL NO: _____

TEMP°C _____ ☐ TEMP BLANK ☐ INTACT ☐ NOT INTACT

ANALYSIS AND CONTAINER TYPE PRESERVATIVES

#	Sample Description	Date / Time coll'd	Matrix	No. of Cntrs.	Filt. ☐Y ☐N	Filt. ☐Y ☐N	Filt. ☐Y ☐N	Filt. ☐Y ☐N	Filt. ☐Y ☐N	Filt. ☐Y ☐N	Filt. ☐Y ☐N	Filt. ☐Y ☐N	Filt. ☐Y ☐N	Filt. ☐Y ☐N
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COMMENTS

Relinquished By: (Signature)	Date / Time	Received By: (Signature)	Relinquished By: (Signature)	Date / Time	Received By: (Signature)
Relinquished By: (Signature)	Date / Time	Received By: (Signature)	Relinquished By: (Signature)	Date / Time	Received By: (Signature)

THE TERMS AND CONDITIONS ON THE REVERSE SIDE HEREOF SHALL GOVERN SERVICES, EXCEPT WHEN A SIGNED CONTRACTUAL AGREEMENT EXISTS

ORIGINAL

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

FIGURE 2

EXAMPLE OF HOMEOWNER KATAHDIN CHAIN-OF-CUSTODY FORM

Katahdin
ANALYTICAL SERVICES

600 Technology Way
P.O. Box 540
Scarborough, ME 04070
Tel: (207) 874-2400 Fax: (207) 775-4029

Homeowner Chain of Custody

Client:	Contact:	Phone:	Fax:
Address:	City:	State:	Zip:
Purchase Order #:	Project Name/No.:	E-mail:	
Billing Address (if different):			
Sampler (Print/Sign):		Copies To:	

***Test results are for compliance and will be reported to the state (see statement below). yes no | Compliance samples must be received on ice.

Lab Use Only | Work Order #: | KAS Project Manager:

Shipping: UPS Fed-Ex Mail Drop-Off

Sample(s) Received on Ice? Yes No Temperature if Iced:

Sample Description (Sample Identification and/or Lot #)	Date Collected	Time Collected	No. of Cntrs.	Standard Homeowner	Arsenic	Total Coliforms	Lead	Safety Test – coliform & N+N	FHA/MSH	Fluoride	Uranium		What's Included in the Standard Test and the FHA/MSH Test.
													Standard Homeowner Total Coliform/e-coli Nitrate, Nitrite Chloride, pH Hardness Copper, Iron Manganese Sodium
													FHA/MSH Standard plus Lead Turbidity Color Odor

Relinquished By:	Date/Time:	Received By:	Relinquished By:	Date/Time:	Received By:

Per the National Environmental Laboratory Accreditation Program (NELAP) Standards, Katahdin is required to accept samples that have been properly preserved. All sample containers provided to you have been properly preserved, but the proper preservation also requires samples to be received at <6 degrees celcius. The Safe Drinking Water Act regulations only require this for compliance samples (i.e., results that are submitted to the state). By circling no for compliance (above), you acknowledge that the samples described above are not for compliance purposes, and thus may not meet the temperature receipt requirements.

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

FIGURE 3

EXAMPLE OF FOOD/MICROBIOLOGY KATAHDIN CHAIN-OF-CUSTODY FORM

Katahdin
ANALYTICAL SERVICES

600 Technology Way
P.O. Box 540
Scarbrough, ME 04070
Tel: (207) 874-2400 Fax: (207) 775-4029

Chain of Custody

Client: Contact: Phone: Fax:

Address: City: State: Zip:

Purchase Order #: Project Name/No.: E-mail:

Billing Address (if different):

Sampler (Print/Sign): Copies To:

Lab Use Only | Work Order #: KAS Project Manager:

Shipping: UPS Fed-Ex Airbill No.:

Temperature:

Food & Microbiological Services

Sample Description (Sample Identification and/or Lot #)	Date/Time Collected	Matrix	No. of Cntrs.	Plate Count (A/H/S)	Listeria	Yeast and Mold	Salmonella	E-Coli	E-Coli 0157:H7	Staph	Vibrio	Total Coliforms	Campylobacter	Shelf Life	Challenge Study		

Relinquished By:	Date/Time:	Received By:	Relinquished By:	Date/Time:	Received By:

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

FIGURE 4

EXAMPLE OF KATAHDIN SAMPLE RECEIPT LOGBBOK

KATAHDIN ANALYTICAL SERVICES, INC.

SAMPLE LOG IN

Date Received	Time Received	Date Logged In	Time Logged In	Work Order	Client	Initials
				SA 0094		
				SA 0095		
				SA 0096		
				SA 0097		
				SA 0098		
				SA 0099		
				SA 0100		
				SA 0101		
				SA 0102		
				SA 0103		
				SA 0104		
				SA 0105		
				SA 0106		
				SA 0107		
				SA 0108		
				SA 0109		
				SA 0110		
				SA 0111		
				SA 0112		
				SA 0113		
				SA 0114		
				SA 0115		
				SA 0116		
				SA 0117		
				SA 0118		
				SA 0119		
				SA 0120		
				SA 0121		
				SA 0122		
				SA 0123		
				SA 0124		

Signed By:_____ Date:_____

Reviewed By:_____ Date:_____

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

FIGURE 5

EXAMPLE OF WET CHEMISTRY SHORTS AND RUSHES LOGBOOK

WET CHEMISTRY SHORTS & RUSHES **Receipt Date:**

HOLDING TIME					Immediate			24 Hr		48 Hr								
Work Order / Client	Matrix	Earliest Sampling Date	Earliest Sampling Time	Rush Parameters	pH	DO	Sulfite	Fe +2	Cr+6	Total BOD	Carbon BOD	Color	Nitrate	Nitrite	OPO4	Set Solids	Turbidity	Comments (Quick TAT, MS/MSD, etc.)

00000004

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

FIGURE 6

EXAMPLE OF SAMPLE RECEIPT CONDITION REPORT FORM

Katahdin Analytical Services, Inc. **Sample Receipt Condition Report**

Client:	KAS PM:	Sampled By:
Project:	KIMS Entry By:	Delivered By:
KAS Work Order#:	KIMS Review By:	Received By:
SDG #:	Cooler: _____ of _____	Date/Time Rec.:

Receipt Criteria	Y	N	EX*	NA	Comments and/or Resolution
1. Custody seals present / intact?					
2. Chain of Custody present in cooler?					
3. Chain of Custody signed by client?					
4. Chain of Custody matches samples?					
5. Temperature Blanks present? If not, take temperature of any sample w/ IR gun.					Temp (°C):
Samples received at <6 °C w/o freezing?					Note: Not required for metals analysis.
Ice packs or ice present?					The lack of ice or ice packs (i.e. no attempt to begin cooling process) may not meet certain regulatory requirements and may invalidate certain data.
If not, has the cooling process begun (i.e. ice or packs present) and sample collection times <2hrs., but samples are not yet cool?					Note: No cooling process required for metals analysis.
6. Volatiles free of headspace: **Aqueous:** No bubble larger than a pea **Soil/Sediment:**					
Received in airtight container?					
Received in methanol?					
Methanol covering soil?					
7. Trip Blank present in cooler?					
8. Proper sample containers and volume?					
9. Samples within hold time upon receipt?					
10. Aqueous samples properly preserved? Metals, COD, NH3, TKN, O/G, phenol, TPO4, N+N, TOC, DRO, TPH – pH <2					
Sulfide - >9					
Cyanide – pH >12					

* Log-In Notes to Exceptions: document any problems with samples or discrepancies or pH adjustments

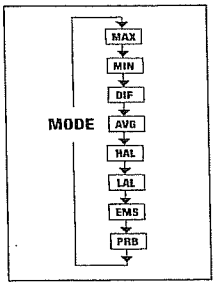
TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

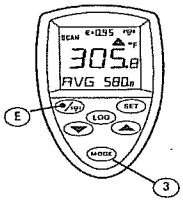
FIGURE 7

IR THERMOMETER MANUFACTURER'S INSTRUCTIONS FOR CHANGING EMISSIVITY

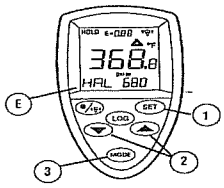
MODE Button Functions
Your infrared thermometer measures Maximum (MAX), Minimum (MIN), Differential (DIF)*, and Average (AVG)** temperatures each time you take a reading. This data is stored and can be recalled with the MODE button (3) until a new measurement is taken. (See "Hold and Recall" for information on how to recall stored data.) When the trigger is pulled again, the unit will begin measuring in the last mode selected. Pressing the MODE button also allows you to access the High Alarm (HAL), Low Alarm (LAL), Emissivity (EMS), Probe temperature (PRB—only available when the probe is connected), and Data logger (LOG). Each time you press MODE, you advance through the mode cycle. The diagram shows the sequence of functions in the Mode cycle.
Note: PRB (probe) is only available in the MODE loop when the contact probe is connected to the unit.

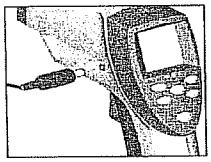
*DIF shows the difference between the maximum and minimum temperatures measured.
**AVG shows the average temperature reading for each time the trigger is pulled or the unit is locked on.

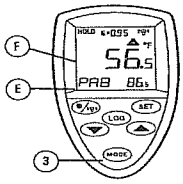




Selecting a Function
To Select the MAX, MIN, DIF, or AVG mode, pull the trigger. While holding the trigger, press the MODE button (3) until the appropriate code appears in the lower left corner of the display (E). Each time you press MODE, you advance through the MODE cycle. The MODE cycle is shown above.







Setting the High Alarm, Low Alarm, and Emissivity
To set values for the High Alarm (HAL), Low Alarm (LAL), and Emissivity, pull the trigger or press the MODE button (3) to activate the display. Press the MODE button until the appropriate code appears in the lower left corner of the display (E). Use the up and down keys (2) to adjust the desired values. To activate the alarms, press SET (1). To deactivate the alarms, press SET again.

Using a Probe (PRB)
Connect the probe to the input on the side of the unit (as shown).
PRB automatically appears in the lower left corner of the display (E, below). The probe temperature is shown in the lower right part of the display. The current infrared temperature continues to show in the center of the display (F). While the probe is connected, you may still cycle through the mode functions by pressing MODE (3).

Note: PRB is only available in the MODE loop when a probe is connected to the unit; the probe temperature will not activate the high alarm or low alarm.

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

FIGURE 8

EXAMPLE OF KATAHDIN SAMPLE FILTRATION LOGBOOK

KATAHDIN ANALYTICAL SERVICES, INC.
Sample Filtration Logbook

Katahdin Sample No.	Site ID	Filtration Requested By:		Filtered and Preserved By:		
List Individually	(Optional)	Initials	Date	Initials	Date	Time

Reviewed and Approved by: _____ Date: _____

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

FIGURE 9

MEASURED TURBIDITY AND PRESERVATION OF INCOMING SAMPLES LOGBOOK

KATAHDIN ANALYTICAL SERVICES

Measured Turbidity and Preservation of Incoming Samples

KAS Lab Sample ID	Measured Turbidity (NTU)	Turbidity Date	Turbidity Analyst	Preservation Date	Preservation Time	Preservation Analyst
WW6757-9A	0.22	12-19-06		12/14/06	~~1830~~ 1400	DM
↓ -10A	0.18	↓		↓	↓	↓
WW6758-1A	4.80	↓		↓	↓	↓
WW6776-1A	0.68	12-20-06		12/20/06	1000	DM
WW6855-1A	0.92	12-26-06		12/26/06	1600	DM
WW6860-1A	2.40	↓		↓	↓	↓
SA0027-1A	0.52	1-3-07		01/03/07	1445	DM
SA0028-1A	0.72	1-3-07		↓	↓	↓
SA0087-1A	0.26	1-5-07		01/05/07	1645	DM
SA0088-1A	10.4	1-5-07		↓	↓	↓
SA0110-1A	0.47	1-8-07		01/08/07	1430	DM
SA0109-1A	0.32	1-8-07		↓	↓	↓
SA0138-1A	0.12	1-09-07		01/09/07	1700	DM
SA0262-1A	0.14	1-18-07		01/18/07	0930	DM
SA0263-1A	0.33	1-18-07		↓	↓	↓
SA0267-1A	0.30	1-18-07		01/18/07	0950	DM
SA0313-1A	0.58	1-22-07		01/22/07	1030	↓
SA0316-1A	1.70	1-22-07		01/22/07	1345	DM

REVIEWED BY: DATE:

FIGURE 10

EXAMPLE OF LABORATORY INCOMING SAMPLE INFORMATION SHEET (ISIS)

KATAHDIN ANALYTICAL SERVICES, INC. – INCOMING SAMPLE INFORMATION SHEET

CLIENT:	ATTENTION:
ADDRESS:	
TELEPHONE:	FAX:
OTHER CLIENT CONTACTS:	
REPORT CC:	PM/SALES CONTACT:
PO #:	CLIENT ACCOUNT:
KAS QUOTE #:	KIMS QUOTE #:
PROJECT NAME:	KIMS PROJECT REF.:
SDG:	APPLICABLE CERTIFICATION:
RECEIPT DATE:	MEANS OF DELIVERY:

METHOD OR KIMS PRODUCT	MATRIX	# OF SAMPLES	SPECIAL INSTRUCTIONS (I.E LIMITS, PREPS)

SUBCONTRACT PARAMETER	SUBCONTRACT LABORATORY

QC LEVEL:	EDD:
RUSH/VERBAL TAT:	HARDCOPY TAT:
HISTORY:	
OTHER:	

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

FIGURE 11

EXAMPLE OF KATAHDIN WORK ORDER/LOGIN COC REPORT

Katahdin
ANALYTICAL SERVICES

Katahdin Analytical Services
Login Chain of Custody Report (lno1)
Jan. 26, 2007
03:51 PM

Page: 1 *of* 1

Login Number: SA0395
Account:KATAHD001 Web
Katahdin Analytical Services

Project:

Primary Report Address:
Leslie Dimond
Katahdin Analytical Services
600 Technology Way
P.O. Box 540
Scarborough,ME 04070

Primary Invoice Address:
Accounts Payable
Katahdin Analytical Services
600 Technology Way
P.O. Box 540
Scarborough,ME 04070

Report CC Addresses:
Invoice CC Addresses:

Login Information

ANALYSIS INSTRUCTIONS	:
CHECK NO.	:
CLIENT PO#	:
COOLER TEMPERATURE	: n/a
DELIVERY SERVICES	: In House
EDD FORMAT	:
MAIL DATE	:
PM	: LAD
PROJECT NAME	: QC Holding Blanks
QC LEVEL	: I
REGULATORY LIST	:
REPORT INSTRUCTIONS	:
SDG ID	:
SDG STATUS	:

Laboratory Sample ID	Client Sample Number	Collect Date/Time	Receive Date	PR	Verbal Date	Due Date	Comments
SA0395-1	WHITE FRIDGE	26-JAN-07 15:50	26-JAN-07			08-FEB-07	
Matrix	*Product*	*Hold Date (shortest)*	*Bottle Type*		*Bottle Count*		
Aqueous	S SW8260FULL5ML	09-FEB-07			2		
SA0395-2	BLUE FRIDGE	26-JAN-07 15:50	26-JAN-07			08-FEB-07	
Matrix	*Product*	*Hold Date (shortest)*	*Bottle Type*		*Bottle Count*		
Aqueous	S SW8260FULL5ML	09-FEB-07			2		

Total Samples: 2 ***Total Analyses:*** 2

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

FIGURE 12

EXAMPLE OF LABORATORY INTERNAL CHAIN-OF-CUSTODY FORM

Katahdin Analytical Services, Inc.
Internal Custody Record

EXTRACTIONS (AQ) Work Order #:_____

Sample Numbers Involved	Placed in refrigeration by: Initials
	Date
	Time

Sample Numbers	Analysis	Removed			Returned			Consumed?	
		Initials	Date	Time	Initials	Date	Time	Yes	No

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

FIGURE 13

EXAMPLE OF REFRIGERATOR TEMPERATURE LOGBOOK

KATAHDIN ANALYTICAL SERVICES, INC.

SAMPLE RECEIPT WALK-IN TEMPERATURE LOG

Corrective Action: Note in the "comments" column and notify the QAO or supervisor; document corrective actions taken and return to control.

Thermometer Location		Sample Receipt Walk-in 1	Comments
Acceptance Criteria		2 to 6 °C	
Date	Initials	Temp (°C)	
02/2/07	NDA	3.7	
02/05/07	DWM	5.8	
02/06/07	DWM	5.8	
02/07/07	DWM	5.8	
02/08/07	DWM	3.8	
02/09/07	DWM	3.5	
02/12/07	DWM	3.5	
02/13/07	DWM	3.5	

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

FIGURE 14

EXAMPLE OF IMMEDIATE INTERNAL COC LOGBOOK

KATAHDIN ANALYTICAL SERVICES, INC.
INTERNAL CUSTODY RECORD FOR IMMEDIATES

CLIENT	PROJECT	CLIENT ID &/or WORK ORDER #	ANALYSIS	OUT date/time	IN date/time	INIT	Consumed?
Jacobs		WW4813-1A, -2A	ICP	9/13/06 0930 →	0935	DJJ	yes no
Jacobs		WW4883-1A	ICP	9/16/06 0900 →	1000	DJJ	yes no
CES		WW4965	BOD	9/20/06 0830	9/20/06 1000	CP	yes no
CCAR		WW4969	BOD	9/20/06 1000	↓	CP	yes no
GEMF		WW4970	BOD	↓	↓	CP	yes no
Jacobs		WW4962-1A, -2A	ICP	9/20/06 0900 →	1000	DJJ	yes no
Irving		WW4994	BOD	9/21/06 1000	9/21/06 1015	CP	yes no
Highliner		WW4992 ②	BOD	9/21/06 1015		CP	yes no
NATIONAL		WW5000	TS, PERCHLORE PH, SP.COND	9/21/06 1100	9/21/06 1217	↓	yes no
W&C		WW5001	BOD	9/21/06 1300		CP	yes no
Arlens		WW5016	NO7	9/22/06 1300 NO7	09/22/06 1100	[illegible]	yes no
RAISOM		WW5010	↓	↓ ↓	↓	↓	yes no
EcoMaine		WW5029	BOD	9/22/06 1100		CP	yes no

QAQC143

0000095

KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

SOP Number: SD-903
Revision History
Cover Page
Page 1

TITLE: **SAMPLE DISPOSAL**

Prepared By: _____ Date: 2/01

Approved By:

Group Supervisor: _____ Date: _____

Operations Manager: Jal C. Burton Date: 2/01

QA Officer: Deborah J. Nadeau Date: 2.01

General Manager: _____ Date: 2/01

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01	Format changes, ~~added pollution prevention~~, added updated log-book and greater detail on dis-posal.	DN	2.01	2/01
02	Major rewrite to include more detail on hazardous waste regu-lations + to reflect current practices.	DN	02/05	02/05
03	Rewrite of section 7 to comply with current practices in new facility. Updated Figures 1 to 3.	DN	02.08	02.08
04	Added elementary neutralization to section 7.0. Other minor edits.	DN	05.09	0509

TITLE: SAMPLE DISPOSAL

Please acknowledge receipt of this standard operating procedure by signing and dating both of the spaces provided. Return the bottom half of this sheet to the QA Department.

I acknowledge receipt of copy ___ of document **SD-903-04**, titled **SAMPLE DISPOSAL**.

Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy ___ of document **SD-903-04**, titled **SAMPLE DISPOSAL**.

Recipient: _____ Date: _____

TITLE: SAMPLE DISPOSAL

1.0 SCOPE AND APPLICATION

Katahdin Analytical Services, Inc. requires strict adherence to specific procedures for the disposal of samples. The procedures are designed to categorize waste materials, provide for their safe and timely disposal and to ensure compliance with local and federal regulations pertaining to disposal of chemicals and environmental samples. Any other means of disposal not described in this SOP is prohibited without consent from the Katahdin Environmental Health & Safety Officer and/or the Katahdin Environmental Compliance Officer.

The purpose of this SOP is to describe the procedures utilized by Katahdin Analytical personnel for the disposal of samples. These procedures apply to the disposal of all samples received or processed by Katahdin. Refer to the current revision of Katahdin SOP CA-107 regarding the disposal of spent preparation and analysis reagents, standards, sample extracts, distillates, or digestates.

1.1 Definitions

Hazardous Waste – A "Solid Waste" which displays a hazardous characteristic or is specifically listed as hazardous waste.

Solid Waste – Any discarded material that is not excluded from the definition of hazardous waste.

Discarded Material – Material that is abandoned, recycled or inherently waste-like.

Waste (State of Maine) –

- Any useless, unwanted, or discarded substance or material, whether or not such substance or material has any other future use.
- Any substance or material that is spilled, leaked, pumped, poured, emptied or dumped onto the land or into the water or ambient air.
- Materials which are used in a matter constituting disposal, burned for energy recovery, reclaimed, or accumulated speculatively.

Ignitable Hazardous Waste – EPA Waste Code D001

- Liquids with a flash point less than 140°F or 60°C.
- Solids capable of spontaneous combustion under normal temperature and pressure.
- Ignitable compressed gas.
- Oxidizers.

Corrosive Hazardous Waste - Liquids with a pH less than or equal to 2.0 or greater than or equal to 12.5. EPA waste code D002.

Reactive Hazardous Waste – EPA waste code D003.

- A material that reacts violently with water.
- A material that generates toxic gases or fumes.
- Explosives.

Toxic Hazardous Waste – A material that exceeds certain concentration levels based on the toxicity characteristic leaching procedure (TCLP). See Figure 3 for the chemicals and concentration levels covered under this definition.

Listed Wastes – Lists of chemicals that are considered hazardous based on the following criteria

- Virgin chemical or unused product.
- Sole active ingredient.
- Single substance spill debris.

Listed wastes are divided into 5 subcategories

- F-wastes – Describe hazardous waste from non-specific sources usually containing halogenated and non-halogenated solvents.
- K-wastes – Describe hazardous wastes created by specific processes.
- U-wastes – Describe toxic or non-acute hazardous wastes.
- P-wastes – Describe acute hazardous wastes. (Note: Maine considers a material to be a P-listed waste if it contains 10% or more of any P-listed chemical.
- State listed wastes – Maine lists any material with a concentration of greater than 50 ppm Polychlorinated Biphenyls (PCB) as a hazardous waste.

Organics hit – A liquid sample containing greater than 1 mg/L of organic contaminants or a soil sample containing greater than 20 mg/kg of organic contaminants.

1.2 Responsibilities

Only designated analysts/technicians trained in these procedures may dispose of samples or analytical by-products. Each analyst or technician must be familiar with Katahdin Analytical safety procedures. Gloves, safety glasses, lab coats and/or other protective clothing must be worn at all times.

It is the responsibility of the designated Katahdin personnel involved in the disposal of samples to read and understand this SOP, to adhere to the procedures outlined, to properly document their activities in the appropriate lab notebook and file the necessary manifests and reports to outside agencies in the required manner. Refer to

Katahdin SOP QA-805, "Personnel Training & Documentation of Capability," current revision.

It is the responsibility of the Department Managers to oversee that members of their group follow this SOP, to ensure that their work is properly documented and to initiate periodic review of the associated logbooks.

It is the responsibility of the Katahdin Environmental Health & Safety Officer (EHSO) to manage the proper classification and disposal of samples. Katahdin is responsible for regulatory compliance of Katahdin's waste storage areas (less than 90 day storage). The EHSO ensures compliance of the waste storage areas with applicable state and federal regulations. The EHSO is responsible for providing the appropriate training to all individuals involved in the proper classification and/or disposal of samples. The EHSO is responsible for working with the Laboratory Operations Manager/Environmental Compliance Officer to help identify problems and assure resolution, to facilitate corrective action where needed, and to communicate unresolved problems and concerns to the Laboratory Vice President.

It is the responsibility of the Operations Manager/Environmental Compliance Officer to oversee adherence to Katahdin sample disposal and hazardous waste practices by all laboratory groups under his/her authority, to help identify problems and assure resolution, to facilitate corrective action where needed, and to communicate problems and concerns to the EHSO and/or the Laboratory Vice President.

It is the responsibility of the Laboratory Vice President to provide the necessary resources to meet the regulatory requirements of proper classification and disposal of samples.

2.0 SUMMARY OF METHOD

Not applicable.

3.0 INTERFERENCES

Not applicable.

4.0 APPARATUS AND MATERIALS

Not applicable.

TITLE: SAMPLE DISPOSAL

5.0 REAGENTS

Not applicable.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

Not applicable.

7.0 PROCEDURES

7.1 Sample purging is the removal of samples from laboratory refrigerated storage. Sample storage areas where samples are removed (purged) from include wet chemistry, organic extractables, metals, volatiles, total organic carbon and soils. Wet chemistry, aqueous metals, organic extractables, total organic carbon, and soils can all be found in the walk-in refrigerator. Aqueous and soil volatiles can be found in the volatiles laboratory refrigerators/freezer.

7.2 Samples are purged from storage, after analysis and reporting, on a routine basis to make room for incoming samples. Samples are to be kept in storage for a duration of 30 days past the report mailed date. Some samples must be kept for 60 or 90 days beyond the report mailed date, depending on specific client requests and contracts.

7.3 The first step in disposing of samples is to generate a disposal list. The disposal list contains sample analysis information stored in the Katahdin Information Management System (KIMS). The analytical data for the samples is compared to the hazardous waste criteria specified in 40CFR Part 261 and to local wastewater discharge criteria. Refer to Figure 4 for 40 CFR Part 261 Characteristic Hazardous Waste Criteria. Based on this comparison, the report displays information on the classification/category for disposal of each sample. The disposal report should be reviewed against the data reports for accuracy. Refer to Figure 2 for an example of a KIMS generated disposal list. The primary disposal categories listed in the report are: non-hazardous, high organics, high metals, flashpoint, high mercury, high PCBs, and high cyanide. Katahdin has established 14 waste stream profiles with a 3rd party waste transporter/waste disposal firm for sample disposal based on these categories. As required, new or special temporary waste profiles are established based on the characteristics of samples.

7.4 Sorting through samples and preparing them for disposal is a crucial quality checkpoint. Samples put into the incorrect waste stream could not only produce adverse environmental effects, but, could also interrupt the 3rd party's waste treatment efficiency, or endanger an individual handling the waste stream. Therefore, when sorting through samples pay close attention to which waste stream each sample falls into.

7.5 Once you are ready to dispose of the samples of interest (the oldest samples that have been purged), these samples must be sorted, logged, and the classification/category (sample knowledge) information recorded.

Sample storage times (as listed in section 7.2) and space should be taken into consideration when purging samples. It is important to make room for future samples, but to make sure that samples are not purged too early. Samples should be pulled from the walk-in or the volatiles refrigerators to make room for new samples. When purging, chose a section that needs extra space the most and remove the oldest samples.

Safety glasses, nitrile gloves, lab coat, and a splash apron must be worn when handing samples during disposal

7.6 Remove the designated purge samples from the shelf one by one and line them up on the countertop in the log-in area. Generally, removing two cartloads at a time is a good amount to purge at one time. For volatile samples in 40mL vials, 5 or 6 vial trays should be purged at a time. Samples should be lined up across the counter with the earliest sample to the left and building up to the right, organizing the samples according to work order and sample number. After the samples are lined up, they should be recorded in the Sample Disposal Logbook (SDL). Refer to Figure 1 for an example SDL page. The location the samples were removed from should also be recorded. Sample storage areas are recorded with the following designations:

VOA (Aq)	Aqueous Volatiles(VOA)
VOA (SL)	Solid Volatiles(VOA)
M	Metals
EXT	Extractables (Organic)
TOC	Total Organic Carbon
WC	Wet Chemistry
S	Soils

7.7 The next step is to use the sample disposal list to determine the earliest release date of the reports and to determine each samples appropriate waste classification/characterization. As stated in section 7.3, the primary disposal categories listed in the report are: non-hazardous, high organics, high metals, flashpoint, high mercury, high PCBs, and high cyanide.

Using the information from the KIMS disposal list, record the appropriate classification for each sample in the SDL. If multiple categories are identified as being present then a single category is selected as controlling. The order of precedence is PCB's, metals and then organics. If another scenario is found, the individual should bring it to the EHSO for a determination of the acceptable waste stream designation or a determination that it should be lab packed separately.

If samples have been sorted that have not been in storage for the 30 days beyond the release date (60 or 90 for certain clients), then these samples need to be placed back in storage and it should be noted in the SDL.

7.8 As stated above, a sample may be categorized into a waste stream based upon the analytes it contains as determined by laboratory testing. In addition, many samples are also categorized as hazardous waste based upon the preservative that they contain. Since many samples contain preservatives, caution must be used when dumping samples. It is also important to ensure that the sample container is empty. This can be accomplished by holding the container upside down and shaking gently until liquid is no longer observed coming out of the container.

7.9 Once waste categories have been determined and entered into the SDL, The following waste categories are disposed of as follows:

7.9.1 Dumping non-hazardous samples (as determined by laboratory testing)

Non-hazardous samples (non-preserved) are poured directly into the sink in the warehouse.

Non-hazardous solid samples are disposed of with the general trash, which is picked up by commercial trash collectors and ultimately disposed of in a waste-to-energy incinerator.

Sample containers from non-hazardous samples are disposed of with the general trash.

7.9.2 Dumping Samples with high Organics (as determined by laboratory testing)

Aqueous samples get dumped into waste stream "K". Containers are disposed of with general trash. Solid samples are placed into waste stream "I" with their containers. The disposal date is recorded in the SDL.

7.9.3 Dumping samples high in metals, including mercury (as determined by the by laboratory testing)

Aqueous samples get disposed of in waste stream "A". Containers are disposed of with general trash. Solid samples are placed in waste stream "L" with their containers. The disposal date is recorded in the SDL.

7.9.4 Dumping Acidic Samples that do not contain any other hazardous waste constituents (as determined by the acidic preservative or by laboratory testing)

Refer to section 7.10 below.

7.9.5 Dumping Basic samples (as determined by the basic preservative or by laboratory testing)

Aqueous samples get disposed of in waste stream "NHi". Containers are disposed of with general trash. The disposal date is recorded in the SDL.

7.9.6 Dumping samples with high PCBs (as determined by laboratory testing)

Aqueous samples are disposed of in waste stream "Q". Containers are disposed of with general trash. Solid samples get disposed of in waste stream "F" with their containers. The disposal date is recorded in the SDL.

7.9.7 Dumping samples with low flashpoints (as determined by laboratory testing)

Aqueous samples are disposed of in waste stream "O". Containers are disposed of with general trash. Solid samples get disposed of in waste stream "I" with their containers. The disposal date is recorded in the SDL.

7.9.8 Dumping samples with high cyanide (as determined by laboratory testing)

Aqueous samples are disposed of in waste stream "NHi". Containers are disposed of with general trash. Solid samples should be set aside for labpack. The disposal date is recorded in the SDL.

7.9.9 Miscellaneous Disposal (as determined by the preservative)

Sodium Bisulfate: Sodium Bisulfate often comes in vials, but may also come in the 2-4oz glass jars. Dump the Sodium Bisulfate out of the container into waste stream "A". There may be remaining soil left in the sample container. The soil's waste stream and dump date will be dictated by the SDL. The disposal date is recorded in the SDL.

Methanol / Free Products: This often comes in vials, but may also come in the 2-4oz glass jars. Dump the methanol out of the container into the mix-flammables accumulation. When this satellite accumulation container gets full it can be dumped into the "O" waste stream. There may be remaining soil left in the sample container. The soil's waste stream and dump date will be dictated by the SDL. Lastly, samples marked "free product" on the Katahdin sample ID label can be dumped into the mixed flammables stream. The disposal date is recorded in the SDL.

7.10 Pursuant to Maine DEP regulations, Katahdin has the necessary agreements, processes and documentation in place to neutralize samples without a license. Refer to the current revision of the Katahdin Environmental Health & Safety Manual for additional information. Generally, the following procedures are followed.

7.10.1 Samples that have been determined to be hazardous due **solely** to the corrosivity characteristic are neutralized using sodium hydroxide pellets. In the warehouse, samples are emptied into a five gallon heavy duty carboy to about 60% capacity. The carboy is kept in a secondary container. Sodium hydroxide pellets are added slowly to the carboy (about 5 grams at a time) and stirred with a long glass stirring rod. The pH is checked with pH paper.

7.10.2 This process is continued until the pH is between 7 and 8. This normally takes about 30-40 grams of sodium hydroxide pellets, but may vary depending on the buffering capacity of the individual samples.

7.10.3 The carboy is emptied into the sink in the warehouse. The tap water is run at the same time as the neutralized material is disposed of. An eyewash station and spill material is located at this sink.

7.10.4 All neutralization activities are documented, including the date and time of neutralization, the name of the person doing the neutralizing, the amount of neutralized liquid discharged, details on the inspection of the drain area and the date and nature of any significant repairs or corrective actions. This documentation is maintained by the EHSO. Refer to Figure 5 for an example logbook page of neutralization documentation.

7.11 Every 3 to 5 weeks a pickup of hazardous waste is scheduled with the 3rd party waste transporter/waste disposal firm. An inventory is faxed to the transporter summarizing the number of drums and waste streams/profiles. As required, a "lab pack" of expired chemicals or orphan samples is organized as necessary. A designated individual, with applicable Hazardous Waste (RCRA) and Department of Transportation (DOT) training, oversees the waste pickup and signs the hazardous manifests and land ban documentation. Within 7 days a copy is forwarded to the Maine Department of Environmental Protection (MEDEP) and the environmental agency in the designation state (if required by that state). Once the report is received at the disposal facility a copy is returned to KATAHDIN and the MEDEP.

7.12 Prior to March 31 of each year, the laboratory prepares the Annual Hazardous Waste Report (i.e., MEDEP modified EPA Form 8700-13A) as required by MEDEP Hazardous Waste Management Rules. The complete report is reviewed by the Katahdin Environmental Compliance Officer and then forwarded to the following address:

Maine Department of Environmental Protection
Bureau of Remediation & Waste Management
State House Station #17
Augusta, ME. 04333
Attn: Annual Hazardous Waste Report

TITLE: SAMPLE DISPOSAL

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

On a daily basis, a designated individual performs quality checks in all hazardous waste storage areas. The daily check documentation is located in login. Any discrepancy is copied to the Operations Manager and the Katahdin Vice President for corrective action. Refer to the current revision of Katahdin SOP CA-107, *The Management of Hazardous Waste as it Relates to the Disposal of Laboratory Process Waste, Reagents, Solvents & Standards*, for more information. Refer to Figure 3 for a copy of the daily check documentation.

9.0 METHOD PERFORMANCE

Not applicable.

10.0 APPLICABLE DOCUMENTS/REFERENCES

USEPA Code of Federal Regulations, 40 CFR Part 261.

Maine Department of Environmental Protection (ME DEP) Hazardous Waste Management Rules

ME DEP modified EPA Form 8700-13A

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TITLE: SAMPLE DISPOSAL

FIGURE 1

EXAMPLE OF SAMPLE DISPOSAL LOGBOOK (SDL)

KATAHDIN ANALYTICAL SERVICES, INC. -SAMPLE STORAGE/DISPOSAL LOGBOOK

WORK ORDER/ SAMPLE NUMBERS	DEPARTMENT	EARLIEST RELEASE DATE	< CRITERIA	SAMPLE KNOWLEDGE									DATE DISPOSED	INITIALS
			CLEAN	WL	ORG	METS	CN	FP	HG	PCBS				
SAS783-1	WC	10-17-07	✓										1-22-08	GN
SAS786-1		10-17-07	✓											
SAS787-1,2,4		10-17-07	✓											
SAS790-1		10-19-07		✓										
SAS793-1		10-17-07	✓											
SAS795-1-9		10-23-07	✓											
SAS797-1		10-23-07	✓											
SAS798-1,2		10-25-07			✓									
SAS799-1-5		10-31-07	✓											
SAS804-1,2		10-25-07	✓											
SAS809-1,2		10-23-07	1			2				2				
SAS810-1-4	✓	10-23-07	✓										✓	

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TITLE: SAMPLE DISPOSAL

FIGURE 2

EXAMPLE OF KIMS GENERATED WASTE DISPOSAL REPORT

SAMPLE DISPOSAL REPORT

Query by: Login SA6501 to SA7000
Date : 15-JAN-08

Sample	SDG	Status	Mail Date	Parameter	Value		
SA6605-1		NEED	12/02/07				
SA6606-1		NEED	12/02/07				
SA6607-1		NEED	11/15/07				
SA6608-1		NEED	12/06/07				
				ORG	1.17	MG/L	(HIGH)
SA6608-1		NEED	12/06/07				
SA6608-2		NEED	12/06/07				
				AA	13	MG/KG	(HIGH)
SA6609-1		NEED	11/26/07				
SA6609-1		NEED	11/26/07				
SA6610-1		NEED	11/30/07				
SA6611-1	FCS-020	NEED	12/07/07				
SA6611-2	FCS-020	NEED	12/07/07				
SA6611-3	FCS-020	NEED	12/07/07				
SA6611-4	FCS-020	NEED	12/07/07				
SA6611-5	FCS-020	NEED	12/07/07				
SA6611-6	FCS-020	NEED	12/07/07				
SA6611-7	FCS-020	NEED	12/07/07				
SA6611-8	FCS-020	NEED	12/07/07				
SA6612-1	NSA-030	NEED	12/07/07				
SA6612-2	NSA-030	NEED	12/07/07				
SA6612-3	NSA-030	NEED	12/07/07				
SA6612-4	NSA-030	NEED	12/07/07				
				ORG	1.70735	MG/L	(HIGH)
SA6612-5	NSA-030	NEED	12/07/07				
				ORG	1.0481	MG/L	(HIGH)

TITLE: SAMPLE DISPOSAL

FIGURE 3

EXAMPLE OF HAZARDOUS WASTE STORAGE AREA DAILY CHECK

Daily Checklist for
HAZARDOUS WASTE STORAGE AREA

Week of: 1-28, 20 08

Item/Day:	Monday	Tuesday	Wednesday	Thursday	Friday
1. Are containers closed? (Except when waste is being added)	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No
2. Are containers properly labeled with a hazardous waste label?	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No
3. Do you have access to each container and can you read the label? (36" aisle?)	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No
4. Is each container marked with the date storage began?	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No
5. Are the dates on the containers less than 90 days old?	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No
6. Is container free of dents, bulges, rust, spills or leaks?	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No
7. Are all containers on a firm working surface?	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No
8. Inspection by: Name (No Initials)	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
9. Time of Inspection	16:35	15:00	14:45	14:15	16:25
10. Verification of Inspection (Name/Date)	[illegible] 1-28-08	[illegible] 1-29-08	[illegible] 1-30-08	[illegible] 1-31-08 14:15	[illegible] 1-02 17:20

Deficiency noted:					
Corrective action:					
By (Name/Date):					

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FIGURE 4

CHARACTERISTIC TOXIC HAZARDOUS WASTE AND TCLP CONCENTRATIONS

Chemical Name	CAS Number	Waste Code	TCLP conc. liquid	Equivalent conc. In Soil
Arsenic	7440-38-2	D004	5.0 mg/L	100 mg/kg
Barium	7440-39-3	D005	100 mg/L	2000 mg/kg
Cadmium	7440-43-9	D006	1.0 mg/L	20 mg/kg
Chromium	7440-47-3	D007	5.0 mg/L	100 mg/kg
Lead	7439-92-1	D008	5.0 mg/L	100 mg/kg
Mercury	7439-97-6	D009	0.2 mg/L	4 mg/kg
Selenium	7782-49-2	D010	1.0 mg/L	100 mg/kg
Silver	7440-22-4	D011	5.0 mg/L	20 mg/kg
Endrin	72-20-8	D012	0.02 mg/L	0.4 mg/kg
Lindane	58-89-9	D013	0.4 mg/L	8 mg/kg
Methoxychlor	72-43-5	D014	10 mg/L	200 mg/kg
Toxaphene	8001-35-2	D015	0.5 mg/L	10 mg/kg
2,4-D	94-75-7	D016	10 mg/L	200 mg/kg
2,4,5-TP (Silvex)	93-72-1	D017	1.0 mg/L	20 mg/kg
Benzene	71-43-2	D018	0.5 mg/L	10 mg/kg
Carbon Tetrachloride	56-23-5	D019	0.5 mg/L	10 mg/kg
Chlordane	57-74-9	D020	0.03 mg/L	0.6 mg/kg
Chlorobenzene	108-90-7	D021	100 mg/L	2000 mg/kg
Chloroform	67-66-3	D022	6.0 mg/L	120 mg/kg
o-Cresol	95-48-7	D023	200 mg/L	4000 mg/kg
m-Cresol	108-39-4	D024	200 mg/L	4000 mg/kg
p-Cresol	106-44-5	D025	200 mg/L	4000 mg/kg
Cresol	1319-77-3	D026	200 mg/L	4000 mg/kg
1,4-Dichlorobenzene	106-46-7	D027	7.5 mg/L	150 mg/kg
1,2-Dichloroethane	107-06-2	D028	0.5 mg/L	10 mg/kg
1,1-Dichloroethylene	75-35-4	D029	0.7 mg/L	14 mg/kg
2,4-Dinitrotoluene	121-14-2	D030	0.13 mg/L	2.6 mg/kg
Heptachlor	76-44-8	D031	0.008 mg/L	0.16 mg/kg
Hexachlorobenzene	118-74-1	D032	0.13 mg/L	2.6 mg/kg
Hexachlorobutadiene	87-68-3	D033	0.5 mg/L	10 mg/kg
Hexachloroethane	67-72-1	D034	3.0 mg/L	60 mg/kg
Methyl Ethyl Ketone	78-93-3	D035	200 mg/L	4000 mg/kg
Nitrobenzene	98-95-3	D036	2.0 mg/L	40 mg/kg
Pentachlorophenol	87-86-5	D037	100 mg/L	2000 mg/kg
Pyridine	110-86-1	D038	5.0 mg/L	100 mg/kg
Tetrachloroethylene	127-18-4	D039	0.7 mg/L	14 mg/kg
Trichloroethylene	79-01-6	D040	0.5 mg/L	10 mg/kg

FIGURE 4, cont'd

CHARACTERISTIC TOXIC HAZARDOUS WASTE AND TCLP CONCENTRATIONS

Chemical Name	CAS Number	Waste Code	TCLP conc. liquid	Equivalent conc. In Soil
2,4,5-Trichlorophenol	95-95-4	D041	400 mg/L	8000 mg/kg
2,4,6-Trichlorophenol	88-06-2	D042	2.0 mg/L	40 mg/kg
Vinyl Chloride	75-01-4	D043	0.2 mg/L	4.0 mg/kg

TITLE: SAMPLE DISPOSAL

FIGURE 5

EXAMPLE OF ELEMENTARY NEUTRALIZATION LOGBOOK

Katahdin Analytical Services, Inc. – Elementary Neutralization Logbook

Date: 3-4-09		**Time:** 12:00	**Analyst:** GN
# of gallons neutralized	Final pH	Condition of drain and sink area before and after neutralization.	Significant Repairs or Corrective Actions
5	5	good	
6	7	good	
6	5	good	
6	6	good	
2	8	good	

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Date: 3-10-09		**Time:** 13:45	**Analyst:** GN
# of gallons neutralized	Final pH	Condition of drain and sink area before and after neutralization.	Significant Repairs or Corrective Actions
6	7	good	
6	6	good	
5	6	good	
6	8	good	
6	5	good	
6	8	good	
5	5	good	
6	7	good	
3	5	good	

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ERA

A Waters Company

M51623

Certificate of Analysis

Lot No. D061-540

Metals in Soil

Catalog No. 540
Issue Date: May 1, 2008
Revision Date: Original

Certification

Parameter	Total Concentration [1] (mg/kg)	Certified Value [2] (mg/kg)	Uncertainty [3]	QC PALs™ [4] (mg/kg)	PT PALs™ [5] (mg/kg)
aluminum	61800	10600	5.1%	5750 - 15400	4880 - 16300
antimony	267	126	1.6%	63.3 - 189	26.5 - 317
arsenic	253	225	3.8%	181 - 270	160 - 290
barium	1160	565	4.6%	461 - 669	422 - 709
beryllium	178	162	5.4%	134 - 190	122 - 202
boron	129	107	9.3%	74.3 - 139	63.1 - 151
cadmium	79.5	69.1	6.3%	58.1 - 80.1	50.6 - 87.6
calcium	18100	10000	7.1%	8310 - 11700	7540 - 12400
chromium	356	124	5.4%	101 - 147	86.7 - 161
cobalt	132	115	6.2%	95.6 - 135	85.7 - 145
copper	78.8	66.7	5.3%	53.9 - 79.5	48.8 - 84.6
iron	34400	17600	2.4%	8930 - 26400	7320 - 28000
lead	272	223	3.6%	183 - 264	168 - 279
magnesium	7440	4260	5.6%	3290 - 5230	2980 - 5540
manganese	695	368	4.8%	304 - 433	279 - 458
mercury	5.20	5.15	20.1%	3.69 - 6.61	2.63 - 7.67
molybdenum	136	107	6.2%	83.8 - 130	75.0 - 141
nickel	202	172	7.0%	140 - 204	127 - 218
potassium	24200	4090	4.4%	2960 - 5220	2660 - 5520
selenium	166	147	3.5%	114 - 180	98.7 - 195
silver	40.1	35.2	7.0%	23.3 - 47.1	23.1 - 47.3
sodium	13500	538	7.1%	366 - 710	280 - 796
strontium	294	117	6.1%	94.8 - 139	82.3 - 151
thallium	197	173	3.2%	140 - 205	119 - 227
tin	189	164	5.3%	121 - 207	99.1 - 229
titanium	2920	381	2.8%	116 - 647	0.00 - 779
vanadium	155	93.9	4.4%	72.1 - 116	60.6 - 127
zinc	424	349	5.8%	280 - 418	252 - 446

Please see footnotes on back

Certified value used as the true value.

QC PALS used for the acceptance range.

6000 West 54th Ave., Arvada, CO 80002 800-372-0122 fax: 303-421-0159 www.eraqc.com

Katahdin Analytical Services
SVOC QC Limits

Target List	EPA Method	Soil Accuracy % Recovery	Soil Precision RPD (1)	Soil PQLs	Soil MDLs
Semivolatile Organic SIM Compounds				**ug/Kg**	**ug/Kg**
Benzo(a)anthracene	8270C SIM	50-138	0-50 (nominal)	20	1.9
Benzo(a)pyrene	8270C SIM	52-113	0-50 (nominal)	20	3.3
Benzo(b)fluoranthene	8270C SIM	44-119	0-50 (nominal)	20	2.4
Benzo(k)fluoranthene	8270C SIM	40-120	0-50 (nominal)	20	3.1
Chysene	8270C SIM	44-132	0-50 (nominal)	20	1.7
Dibenzo(a,h)anthracene	8270C SIM	30-134	0-50 (nominal)	20	1.8
Indeno(1,2,3-cd)pyrene	8270C SIM	32-134	0-50 (nominal)	20	1.9
Phenanthrene	8270C SIM	58-122	0-50 (nominal)	20	1.8
Pyrene	8270C SIM	33-133	0-50 (nominal)	20	2.1
Carbazole	8270C SIM	30-150 (nominal)	0-50 (nominal)	20	1.3
Surrogates:					
2-Methylnaphthalene-D10	8270C SIM	33-125	---	---	---
Fluorene-D10	8270C SIM	53-136	---	---	---
Pyrene-D10	8270C SIM	16-162	---	---	---
2,4-Dibromophenol	8270C SIM	30-150 (nominal)	---	---	---

APPENDIX G

LABORATORY STANDARD OPERATING PROCEDURES
TESTAMERICA

G-1 SOP NO. PT-MT-001, REV. 11, EFFECTIVE DATE: 07/27/09, INDUCTIVELY COUPLED PLASMA-ATOMIC EMISSION SPECTROMETRY (ICP), METHODS: SW-846 6010B, 6010C AND EPA 200.7

G-2 SOP NO. PT-IP-002, REV. 8, EFFECTIVE DATE: 04/28/09, ACID DIGESTION OF SOILS, METHOD: SW846 METHOD 3050B

G-3 SOP NO. PT-QA-027, REV. 14, EFFECTIVE DATE: 05/23/2010, SAMPLE RECEIPT AND CHAIN OF CUSTODY

G-4 SOP NO. PT-QA-029, REV. 2, EFFECTIVE DATE: 01/22/2010, QA/QC REQUIREMENTS FOR DOD QSM

G-1 SOP NO. PT-MT-001, REV. 11, EFFECTIVE DATE: 07/27/09,

INDUCTIVELY COUPLED PLASMA-ATOMIC

EMISSION SPECTROMETRY (ICP),

METHODS: SW-846 6010B, 6010C AND EPA 200.7

TITLE: Inductively Coupled Plasma-Atomic Emission Spectrometry (ICP)

METHODS: SW-846 6010B, 6010C AND EPA 200.7

Approvals (Signature/Date):			
	07/17/09		07/20/09
William Reinheimer Technical Manager	Date	Steve Jackson Health & Safety Manager / Coordinator	Date
	07/07/09		07/17/09
Nasreen K. DeRubeis Quality Assurance Manager	Date	Larry Matko Laboratory Director	Date

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Controlled Source: Intranet

1. SCOPE AND APPLICATION

1.1. This procedure describes the analysis of trace elements including metals in solution by Inductively Coupled Plasma -Atomic Emission Spectroscopy (ICP-AES) using SW-846 Method 6010B, 6010C and EPA Method 200.7. Table I of Appendix A lists the elements appropriate for analysis by Methods 6010B/6010C and 200.7. Additional elements may be analyzed under Methods 6010B, 6010C and 200.7 provided that the method performance criteria presented in Section 13.0 are met. In addition to SOW ILMO4.0, this SOP is also compliant with the requirements of CLP SOWs 7/88, 3/90, ILMO1.0 and ILMO2.1.

1.2. ICP analysis provides for the determination of metal concentrations over several orders of magnitude. Detection limits, sensitivity and optimum concentration ranges of the metals will vary with the matrices and instrumentation used. For instance, in comparison to conventional ICP technique, ICP-Trace can achieve detection levels comparable to those determined using the graphite furnace atomic absorption spectroscopy (GFAAS) technique.

1.3. Method 6010B and 6010C are applicable to the determination of dissolved, suspended, total recoverable and total elements in ground water, aqueous samples, soils, sludges, wastes, sediments, tissues, wipes and TCLP, EP and other leachates/extracts. All matrices require digestion prior to analysis with the exception of analyses for dissolved metals in filtered and acidified aqueous samples. Although digestion is not specifically required by the method, some clients and regulators may require digestion of dissolved samples and this must be clarified and documented before project initiation. Silver concentrations must be below 2.0 mg/L in aqueous samples and 100 mg/kg in solid matrix samples. Precipitation may occur in samples where silver concentrations exceed these levels and lead to the generation of erroneous data.

1.4. Method 200.7 is applicable to the determination of dissolved, suspended, total recoverable, and total elements in water, waste water, and solid wastes. All matrices require digestion prior to analysis with the exception of analyses for dissolved metals in filtered and acidified aqueous samples if the criteria in Section 11.1 are met. Silver concentrations must be below 0.1 mg/L in aqueous samples and 50 mg/kg in solid matrix samples.

1.5. For DoD QSM Version 3 requirements, refer to SOP PT-QA-025. For DoD V4.1 refer to SOP PT-QA-029.

1.6. On occasion clients may request modifications to this SOP. These modifications are handled following the procedures outlined in Section 12.1 in the Quality Assurance Manual.

2. SUMMARY OF METHOD

2.1. This method describes a technique for the determination of multi elements in solution using sequential or simultaneous optical systems and axial or radial viewing of the plasma. The basis of the method is the measurement of atomic emission by an optical spectroscopic technique. Samples are nebulized and the aerosol that is produced is transported to the plasma torch where excitation occurs. Characteristic atomic-line emission spectra are produced by a radio frequency inductively coupled plasma (ICP). The spectra are dispersed by a grating spectrometer and the intensities of the emission lines are monitored by photomultiplier tubes. The photocurrents from the photomultiplier tubes are processed and controlled by a computer system. A background correction technique is required to compensate for variable background contribution to the determination of trace elements. Background must be measured adjacent to analyte lines during analysis. The position selected for the background intensity measurement, on either or both sides of the analytical line, will be determined by the complexity of the spectrum adjacent to the analyte line. The position used must be free of spectral interferences and reflect the same change in background intensity as occurs at the analyte wavelength measured. Background correction is not required in cases of line broadening where a background correction measurement would actually degrade the analytical result. The possibility of additional interferences should also be recognized and appropriate actions taken. Alternatively, multivariate calibration methods may be chosen for which point selection for background correction is superfluous since whole spectral regions are processed. Consult the appropriate SOP's for details on sample preparation methods.

2.2. Refer to the appropriate SOPs for details on sample preparation methods.

3. DEFINITIONS

3.1. Dissolved Metals: Those elements which pass through a 0.45 um membrane. (Sample is acidified <u>after</u> filtration).

3.2. Suspended Metals: Those elements which are retained by a 0.45 um membrane.

3.3. Total Metals: The concentration determined on an unfiltered sample following vigorous digestion.

3.4. Total Recoverable Metals: The concentration determined on an unfiltered sample following treatment with hot, dilute mineral acid.

4. INTERFERENCES

4.1. Spectral, physical and chemical interference effects may contribute to inaccuracies in the determinations of trace elements by ICP. Spectral interferences are caused by:

- Overlap of a spectral line from another element.
- Unresolved overlap of molecular band spectra.
- Background contribution from continuous or recombination phenomena.
- Stray light from the line emission of high concentration elements.

4.1.1. Chemical interferences are characterized by molecular compound formation, ionization effects and solute vaporization effects. Normally these effects are not significant with the ICP technique but if observed can be minimized by buffering the sample, matrix matching or standard addition procedures.

4.1.2. A background correction technique is required to compensate for variable background contribution to the determination of trace elements. Background correction is not required in cases where a background corrective measurement would actually degrade the analytical result.

4.1.3. Inter-element correction factors (IECs) are necessary to compensate for spectral overlap. Inter-element interferences occur when elements in the sample emit radiation at wavelengths so close to that of the analyte that they contribute significant intensity to the analyte channel. If such conditions exist, the intensity contributed by the matrix elements will cause an excessively high (or sometimes low) concentration to be reported for the analyte. Inter-element corrections IECs must be applied to the analyte to remove the effects of these unwanted emissions.

4.1.4. Physical interferences are generally considered to be effects associated with sample transport, nebulization and conversion within the plasma. These interferences may result in differences between instrument responses for the sample and the calibration standards. Physical interferences may occur in the transfer of solution to the nebulizer (e.g., viscosity effects), at the point of aerosol formation and transport to the plasma (e.g., surface tension) or during excitation and ionization processes within the plasma itself. Changes in viscosity and surface tension can cause significant inaccuracies, especially in samples containing high dissolved solids or high acid concentrations. If

physical interferences are present, dilution of the sample, use of a peristaltic pump, mass flow controller, use of an internal standard and/or use of a high solids nebulizer can reduce the effect.

5. SAFETY

5.1. Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001), Radiation Safety Manual and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.2 The ICP plasma emits strong UV light and is harmful to vision. All analysts must avoid looking directly at the plasma.

5.3 The following is a list of the materials used in this method, which have a serious or significant hazard rating. **NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Nitric Acid	Corrosive Oxidizer Poison	2 ppm-TWA 4 ppm-STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Hydrochloric Acid	Corrosive Poison	5 ppm-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

5.4 Eye protection that protects against splash, laboratory coat, and appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled. Cut resistant gloves must be worn doing any other task that presents a strong possibility of getting cut. Disposable gloves that have been contaminated will be removed and discarded; other gloves will be cleaned immediately.

5.5 The RF generator produces strong radio frequency waves, most of which are unshielded. People with pacemakers should not go near the instrument while in operation.

5.6 Exposure to chemicals must be maintained **as low as reasonably achievable**, therefore, unless they are known to be non-hazardous, all samples should be opened, transferred and prepared in a fume hood, or under other means of mechanical ventilation. Metals digestates can be processed outside of a fume hood. Solvent and waste containers will be kept closed unless transfers are being made.

5.7 The preparation of standards and reagents will be conducted in a fume hood or well-ventilated area.

5.8 All work must be stopped in the event of a known or potential compromise to the health and safety of a TestAmerica associate. The situation must be reported **immediately** to a laboratory supervisor or EH&S coordinator.

6. EQUIPMENT AND SUPPLIES

6.1. Inductively Coupled Plasma Atomic Emission Spectrometer equipped with autosampler and background correction.

6.2. Radio Frequency Generator.

6.3. Nitrogen or argon gas supply, welding grade or equivalent.

6.4. Cool flow or appropriate water cooling device.

6.5. Peristaltic Pump.

6.6. Calibrated automatic pipettes or Class A glass volumetric pipettes.

6.7. Class A volumetric flasks.

6.8. Autosampler tubes.

7. REAGENTS AND STANDARDS

7.1. Intermediate standards are purchased as custom TestAmerica multielement mixes or as single element solutions. All standards must be stored in FEP fluorocarbon or previously unused polyethylene or polypropylene bottles. Intermediate standard solutions must be replaced prior to the expiration date provided by the manufacturer. If no expiration date is provided, the intermediate solutions may be used for up to one year and must be replaced sooner if verification from an independent source indicates a problem. Expiration dates can be extended provided that the acceptance criteria described in laboratory-specific SOPs are met.

7.2. Working calibration and calibration verification solutions may be used for up to 3 months and must be replaced sooner if verification from an independent source indicates a problem. Standards should be prepared in a matrix of 5% hydrochloric and 5% nitric acids. An exception to this is in the event the Trace ICP is utilized without the internal standard. In this case, the standard acid matrix must be matched to the final preparation matrix.

7.3. Refer to Tables III, IV, IVA, V and VI (Appendix A) for details regarding the working standard concentrations for calibration, calibration verification, interference correction and spiking solutions.

7.4. Concentrated nitric acid (HNO_3), trace metal grade or better.

7.5. Concentrated hydrochloric acid (HCl), trace metal grade or better.

7.6. Reagent water must be produced by a Millipore DI system or equivalent. Reagent water must be free of the analytes of interest as demonstrated through the analysis of method blanks.

8. SAMPLE COLLECTION, PRESERVATION, SHIPMENT AND STORAGE

8.1. Sample holding times for metals are six months from time of collection to the time of analysis.

8.2. Aqueous samples are preserved with nitric acid to a pH of <2 and may be stored in either plastic or glass. If boron or silica are to be determined, plastic containers are preferred. Refrigeration is not required. Preservation must be verified prior to analysis.

8.3. Soil and wipe samples do not require preservation but must be stored at 4°C ± 2° until the time of preparation. Tissue samples are stored frozen until preparation.

8.4. Dissolved metals samples that are filtered and preserved at the laboratory with concentrated Nitric acid will be held for 24 hours before digestion.

9. QUALITY CONTROL

9.1. Table VII (Appendix A) provides a summary of quality control requirements including type, frequency, acceptance criteria and corrective action. See SOP PT-QA-021"TestAmerica Quality Control Program" for additional detail on criteria and corrective actions.

9.2. Each laboratory must have initial demonstration of performance data on file for each analyte of interest as described in Section 12.0.

9.3. Method Blank (MB) - One method blank must be processed with each preparation batch of up to 20 samples. The method blank consists of reagent water containing all reagents specific to the method that is carried through the entire analytical procedure, including preparation and analysis. The method blank is used to identify any system and process interferences or contamination of the analytical system that may lead to the reporting of elevated analyte concentrations or false positive data. The method blank should not contain any analyte of interest at or above the reporting limit (exception: common laboratory contaminants, see below) or at or above 5% of the measured concentration of that analyte in associated samples, whichever is higher (sample result must be a minimum of 10X higher than the blank

contamination level). **Refer to PT-QA-025 for specific DoD requirements for the method blank. For DoD V4.1 refer to SOP PT-QA-029.**

- If the analyte is a common laboratory contaminant (copper, iron, lead (Trace only) or zinc) the data may be reported with qualifiers if the concentration of the analyte in the method blank is less than two times the RL. **Such action must be taken in consultation with the client and must be addressed in the project narrative.**
- Repreparation and reanalysis of all samples associated with an unacceptable method blank is required when reportable concentrations are determined in the samples (see exception noted above).
- If there is no analyte greater than the RL in the samples associated with an unacceptable method blank, the data may be reported with qualifiers. **Such action must be taken in consultation with the client and must be addressed in the project narrative.**
- If the above criteria are not met and reanalysis is not possible, then the sample data must be qualified. **This anomaly must be addressed in the project narrative and the client must be notified.**
- For dissolved metals samples, which have not been digested, a CCB result is reported as the method blank. The CCB run immediately prior to the start of the dissolved sample analyses must be used for this purpose. No more than 20 samples can be associated with one CCB.

9.4. Laboratory Control Sample (LCS) - One aqueous LCS (referred to as a Laboratory Fortified Blank in 200.7) must be processed with each preparation batch of up to 20 samples. The LCS must contain all analytes of interest and must be carried through the entire analytical procedure. Aqueous LCS spike levels are provided in Table III (Appendix A). The LCS is used to monitor the accuracy of the analytical process. On-going monitoring of the LCS results provides evidence that the laboratory is performing the method within acceptable accuracy and precision guidelines.

- If any analyte is outside established control limits the system is out of control and corrective action must occur. Control limits are established, for method 6010B/6010C, a control limit of 80 - 120% (85-115% for 200.7) recovery must be applied.
- In the event that an MS/MSD analysis is not possible a Laboratory Control Sample Duplicate (LCSD) must be analyzed. The RPD of the LCS and LCSD must be compared to the matrix spike RPD limits.

- In the instance where the LCS recovery is greater than 120% (115% for 200.7) and the sample results are < RL, the data may be reported with qualifiers. **Such action must be taken in consultation with the client and must be addressed in the report narrative.**

- Corrective action will be repreparation and reanalysis of the batch unless the client agrees that other corrective action is acceptable.

- For dissolved metals samples, which have not been digested, a CCV result is reported as the LCS. The CCV run immediately prior to the start of the dissolved sample analyses must be used for this purpose. No more than 20 samples can be associated with one CCV.

9.5. Matrix Spike/Matrix Spike Duplicate (MS/MSD) - One MS/MSD pair must be processed for each preparation batch of up to 20 samples (6010B and 6010C) or one MS for every 10 or fewer samples (200.7). A matrix spike (MS) is a field sample to which known concentrations of target analytes have been added (referred to as a Laboratory Fortified Matrix in 200.7). A matrix spike duplicate (MSD) is a second aliquot of the same sample (spiked identically as the MS) prepared and analyzed along with the sample and matrix spike. Some client specific data quality objectives (DQO's) may require the use of sample duplicates in place of or in addition to MS/MSDs. The MS/MSD results are used to determine the effect of a matrix on the precision and accuracy of the analytical process. Due to the potential variability of the matrix of each sample, these results may have immediate bearing only on the specific sample spiked. Samples identified as field blanks cannot be used for MS/MSD analysis. Spiking levels are provided in Tables III and VI (Appendix A).

- If any analyte recovery or RPD falls outside the acceptance range, the recovery of that analyte must be in control for the LCS. For method 6010B and 6010C, control limits of 75 - 125% (70 – 130% for 200.7) recovery and 20% RPD or historical acceptance criteria must be applied to the MS/MSD. **Refer to PT-QA-025 for specific DoD requirements for the MS.** For DoD V4.1 refer to SOP PT-QA-029. If the LCS recovery is within limits, then the laboratory operation is in control and the results may be accepted. If the recovery of the LCS is outside limits corrective action must be taken. Corrective action will include repreparation and reanalysis of the batch. MS/MSD results which fall outside the control limits must be addressed in the narrative.

- If the native analyte concentration in the MS/MSD exceeds 4x the spike level for that analyte, the recovery data are reported as NC (i.e., not calculated). If the reporting software does not have the ability to report NC then the actual recovery must be reported and narrated as follows: "Results outside of limits

do not necessarily reflect poor method performance in the matrix due to high analyte concentrations in the sample relative to the spike level.".

- If an MS/MSD is not possible due to limited sample volume then a laboratory control sample duplicate (LCSD) should be analyzed. The RPD of the LCS and LCSD must be compared to the matrix spike RPD limits.
- For dissolved metals samples by 200.7, which have not been digested, a MS must be performed per every 10 or fewer samples by spiking an aliquot of the sample at the levels specified in Table III (Appendix A).
- For Methods **6010B/ 6010C/DoD** samples- If the MS/MSD recoveries are unacceptable, the same sample from which the MS/MSD aliquots were prepared should also be spiked with a post digestion spike. Otherwise, another sample from the same preparation batch should be used as an alternative. An analyte spike is added to a portion of a prepared sample, or its dilution, and should be recovered to within 80% to 120% (for Method 6010C) of the known value. The spike addition should produce a minimum level of 10 times and a maximum of 100 times the lower limit of quantitation. The spike recovery from the post digestion spiked sample **for method 6010B/DoD** the spike recovery should be within the range 75-125%. If this spike fails, then the dilution test (Sec. 9.6 should be run on this sample. If both the MS/MSD and the post digestion spike fail, then matrix effects are confirmed.

9.6 Dilution test – A dilution test is performed to determine whether significant physical or chemical interferences exist due to the sample matrix. One sample per preparation batch must be processed as a dilution test. The test is performed by running a sample at a 5x (1:5) dilution. Samples identified as field blanks cannot be used for dilution tests. The results of the diluted sample, after correction for dilution, should agree within 10% of the original sample determination when the original sample concentration is greater than 50x the MDL. If the results are not within 10%, the possibility of chemical or physical interference exists.

9.7 Initial Calibration Verification (ICV/ICB) - Calibration accuracy is verified by analyzing a second source standard (ICV). For analyses conducted under Method 200.7, the ICV result must fall within 5% of the true value for that solution with relative standard deviation <3% from replicate readings of four exposures of the ICV standard. For Method 6010B and 6010C, the ICV must fall within 10% of the true value for that solution with relative standard deviation <5% from replicate (minimum of three) exposures. An ICB is analyzed immediately following the ICV to monitor low level accuracy and system cleanliness. The ICB result must fall within +/- the RL from zero. **Refer to PT-QA-025 for specific DoD requirements for the ICB. For DoD V4.1 refer to SOP PT-QA-029.** If either the ICV or ICB fail to meet criteria, the analysis should be terminated, the problem corrected, the instrument recalibrated

and the calibration reverified. This standard is equivalent to the Quality Control Standard (QCS) and the first Instrument Performance Check (IPC) specified in 200.7.

9.8 Continuing Calibration Verification (CCV/CCB) - Calibration accuracy is monitored throughout the analytical run through the analysis of a known standard after every 10 samples. The CCV is a mid-range standard made from a dilution of the calibration standard. The CCV for both methods must fall within 10% of the true value for that solution with relative standard deviation <5% from replicate (minimum of three) exposures. A CCB is analyzed immediately following each CCV. The CCB result must fall within +/- RL from zero. **Refer to PT-QA-025 for specific DoD requirements for the CCB.** For DoD V4.1 refer to SOP PT-QA-029. If the blank is less than 1/10 the concentration of the action level of interest, and no sample is within 10% of the action limit, reanalysis and recalibration are not required before continuation of the run. If a mid-run CCV or CCB fails, the analytical run may be continued; however, the result(s) for the affected element(s) may only be reported when bracketed by valid CCV/CCB pairs. If analytical results for one or more elements are not bracketed by valid CCV/CCB pairs, the problem must be corrected, the instrument recalibrated, the calibration verified and the affected samples reanalyzed for those elements only.

9.9 Reporting Limit Verification Standard (RLV)/CRA or LLICV/LLCCV (6010C) – Calibration accuracy at the laboratory reporting limit is verified after the analysis of the ICB by running the RLV, CRA or LLICV/LLCCV. For method 6010C it must be analyzed at beginning and end of the analytical run. This standard is at the reporting limit. For Methods 200.7 and 6010B the control limit is 50 – 150%. For Method 6010C LLICV/LLCCV the control limit is 70-130%. For method 6010C the RLV/CRA which is the low level quantitation check sample is prepared and analyzed quarterly or as needed. The control limit is 70-130%. Please note the RLV/CRA (undigested) is still analyzed at the beginning and end of the analytical run. **Refer to PT-QA-025 for specific DoD requirements for the RLV standard.** For DoD V4.1 refer to SOP PT-QA-029.

9.10. Interference Check Analysis (ICSA/ICSAB) - The validity of the interelement correction factors is demonstrated through the successful analysis of interference check solutions. The ICSA contains only interfering elements, the ICSAB contains analytes and interferents. Refer to Table V (Appendix A) for the details of ICSA and ICSAB composition. Custom TestAmerica multielement ICS solutions must be used. All analytes should be spiked into the ICSAB solution therefore, if a non-routine analyte is required then it should be manually spiked into the ICSAB using a certified ultra high purity single element solution or custom lab-specific mix. If the ICP will display over correction as a negative number then the non-routine elements can be controlled from the ICSA as described in section 9.10.3. Elements known to be

interferents on a required analyte must be included in the ICP run when that analyte is determined. Aluminum, iron, calcium and magnesium must always be included in all ICP runs.

9.10.1. The ICSA and ICSAB solutions must be run at the beginning of the run. (See Section 10.11 for required run sequence).

9.10.2. The ICSAB results for the interferents must fall within 80 - 120% of the true value. If any ICSAB interferent result fails criteria, the analysis should be terminated, the problem corrected, the instrument recalibrated and the samples rerun.

9.10.3. ICSA results for the non-interfering elements with reporting limits ≤ 10 ug/L must fall within the TestAmerica guidelines of ± 2x RL from zero. ICSA results for the non-interfering elements with RLs > 10 µg/L must fall within the TestAmerica guidelines of ± 1x RL from zero. **Refer to PT-QA-025 for specific DoD requirements for the ICSA.** For DoD V4.1 refer to SOP PT-QA-029. If the ICSA results for the non-interfering elements do not fall within +/- 2x RL (RL ≤10) or ± 1xRL (RL>10) from zero the field sample data must be evaluated as follows:

- If the non-interfering element concentration in the ICSA is the result of contamination versus a spectral interference, and this reason is documented, the field sample data can be accepted.
- If the affected element was not required then the sample data can be accepted.
- If the interfering elements are not present in the field sample at a concentration, which would result in a false positive or negative result greater than +/- 2x RL from zero then the field sample data can be accepted.
- If the interfering element is present in the field sample at a level which would result in a false analyte signal greater than ± 2x RL from zero, the data can be accepted only if the concentration of the affected analyte in the field sample is more than 10x the analyte signal in the ICSA.
- If the data does not meet the above conditions then the IECs must be re-evaluated and corrected if necessary and the affected samples reanalyzed or the sample results manually corrected through application of the new IEC to the raw results. If the results are

recalculated manually the calculations must be clearly documented on the raw data.

9.12 Method of Standard Addition (MSA) -This technique involves adding known amounts of standard to one or more aliquots of the processed sample solution. This technique compensates for a sample interferent that may enhance or depress the analyte signal, thus producing a different slope from that of the calibration standards. It will not correct for additive interferences, which cause a baseline shift. Refer to Section 10.15 for additional information on when MSA is required as well as Appendix D for specific MSA requirements.

9.13 Quality Assurance/Project Summaries - Certain clients may require project- or program-specific QC, which may supersede this SOP's requirements. Quality Assurance Summaries (QASs) or equivalent documents providing project-specific requirements should be developed so that project staff clearly understands the special project requirements.

10. PROCEDURE

10.1. Calibration and Standardization

10.1.1. Set up the instrument with the operating parameters recommended by the manufacturer. Allow the instrument to become thermally stable before beginning calibration (approximately 30 minutes of warm-up is required).

10.1.2. The instruments are profiled and calibrated according to the manufacturer's recommended procedures. Thermo has set up the ICP 61E to be profiled on Cu and the Trace ICPs are to be profiled on As. All other lines are preset by Thermo and should not be adjusted by the user. Flush the system with the calibration blank. The calibration curve must consist of a minimum of a blank and a standard. Refer to the facility-specific instrument SOP or ICP instrument manual for a detailed set up and operation protocols.

10.1.3. Calibration must be performed daily and each time the instrument is set up. Instrument runs may be continued over periods exceeding 24 hours as long as all calibration verification (CCV) and interference check QC criteria are met. The instrument standardization date and time must be included in the raw data.

10.1.4. Refer to Section 9.0 for calibration verification procedures, acceptance criteria and corresponding corrective actions. The NELAC requirement for verification of the initial calibration at varied concentrations is met daily since the ICVs, CCVs, and RLVs/CRA are all at different concentrations.

10.2. For 200.7 analyses, dissolved (preserved) samples must be digested unless it can be documented that the sample meets all of the following criteria:

A. Visibly transparent with a turbidity measurement of 1 NTU or less.

B. Is of one liquid phase and free of particulate or suspended matter following acidification.

C. Is NOT being analyzed for silver.

If the above criteria are met, the dissolved samples can be analyzed directly after an appropriate amount of 1:1 nitric acid is added to an aliquot of sample to adjust the acid concentration to approximately a 1% (v/v) nitric acid solution. Allowance for sample dilution should be made in the calculation.

10.3. A minimum of <u>three</u> <u>exposures</u> for each standard, field sample and QC sample is required. The average of the exposures is reported. For Trace ICP analyses, the results of the sum channel must be used for reporting.

10.4. Prior to calibration and between each sample/standard the system is rinsed with the calibration blank solution. The minimum rinse time between analytical samples is 60 seconds unless following the protocol outlined in 10.1.2 it can be demonstrated that a shorter rinse time may be used. Triton-X can be added to the rinse solution to facilitate the rinse process.

10.5. The use of an autosampler for all runs is strongly recommended.

10.6. The use of automated QC checks through the instrument software is highly recommended for all calibration verification samples (ICV, CCV, RLV/CRA/LLICV/LLCCV), blanks (ICB, CCB, PB), interference checks (ICSA, ICSAB) and field samples (linear range) to improve the data review process.

10.7. To facilitate the early identification of QC failures and samples requiring rerun it is strongly recommended that sample data be reviewed periodically throughout the run.

10.8. To facilitate the data review and reporting processes it is strongly recommended that all necessary dilutions be performed before closing out the instrument run. If any digestate for Method 200.7 has silver detected above 100 mg/L, add 1.0 ml of concentrated HCl to the digestate, mix and reanalyze. If the second analysis yields a higher value for silver, the second analysis is reported and discussed in the report narrative.

10.9. The use of an internal standard is recommended on the conventional, non-Trace ICPs as an alternative to using the method of standard additions. This technique is useful in overcoming matrix interferences especially in high solids matrices.

However, for conventional ICP techniques, internal standards may not be necessary provided that one of the following is performed to minimize physical interferences: (1) peristaltic pump is used, (2) high solids nebulizer is used, or (3) high solids samples are diluted and reanalyzed.

10.10. The use of an internal standard is **required** on any axial ICP unless the calibration and QC standards are matrix matched to each digestion procedure. The following procedural guidelines must be followed when using an internal standard:

10.10.1. Typically used internal standards are: yttrium or scandium. (Note: Any element can be used that is not typically found in environmental samples at a high rate of occurrence.)

10.10.2. The internal standard (IS) must be added to every sample and standard at the same concentration. It is recommended that the IS be added to each analytical sample automatically through use of a third pump channel and mixing coil. Internal standards should be added to blanks, samples and standards in a like manner, so that dilution effects resulting from the addition may be disregarded.

10.10.3. The concentration of the internal standard should be sufficiently high to obtain good precision in the measurement of the IS analyte used for data correction and to minimize the possibility of correction errors if the IS analyte is naturally present in the sample.

10.10.4. The internal standard raw intensity counts must be printed on the raw data.

10.10.5. The analyst must monitor the response of the internal standard throughout the sample analysis run. This information is used to detect potential problems and identify possible background contributions from the sample (i.e., natural occurrence of IS analyte).

10.10.5.1. If the internal standard counts fall within ±30% of the counts observed in the ICB then the data is acceptable.

10.10.5.2. If the internal standard counts in the field samples are more than ±30% higher than the expected level, the field samples must then be:

(1) Diluted and reanalyzed;

(2) The IS concentrations must be raised; or

(3) A different internal standard must be used.

10.11. The following analytical sequence must be used for Methods 6010B/6010C and 200.7:

Instrument Calibration

ICV

ICB

RLV/CRA/LLICV

ICSA

ICSAB

7 Samples

CCV

CCB

10 samples

CCV

CCB

Repeat sequence of up to 10 samples between CCV/CCB pairs as required to complete run

CCV

CCB

RLV/CRA/LLCCV

CCV

CCB

Refer to Quality Control Section 9.0 and Table VII (Appendix A) for Method 6010B/6010C and 200.7 quality control criteria.

10.12. Full method required QC must be available for each wavelength used in determining reported analyte results.

10.13. Guidelines are provided in the appendices on procedures to minimize contamination of samples and **standards, preventive maintenance and troubleshooting.**

10.14. All measurements must fall within the defined linear range where spectral interference correction factors are valid. Dilute and reanalyze all samples for required analytes that exceed the linear range or use an alternate wavelength for which QC data are established. If an interelement correction exists for an analyte, which exceeds the linear range, the IEC may be inaccurately applied. Therefore, even if an overrange analyte may not be required to be reported for a sample, if that analyte is a interferent for any requested analyte in that sample, the sample must be diluted. Acid strength must be maintained in the dilution of samples.

10.15. For TCLP samples, full four-point MSA will be required if all of the following conditions are met:

- recovery of the analyte in the matrix spike is not at least 50%,
- the concentration of the analyte does not exceed the regulatory level, and,
- the concentration of the analyte is within 20% of the regulatory level.

The reporting and regulatory limits for TCLP analyses as well as matrix spike levels are detailed in Table VI (Appendix A). Appendix D provides guidance on performing MSA analyses.

10.16. Any variation in procedure shall be completely documented using instrument run logs, maintenance logs, report narratives, a Nonconformance Memo, or an anomaly report and is approved by a Supervisor/Group Leader and QA Manager. If contractually required, the client shall be notified by the Project Manager.

10.17. Nonconformance documentation shall be filed in the project file.

10.18. Any unauthorized deviations from this procedure must also be documented as a nonconformance, with a cause and corrective action described.

11. DATA ANALYSIS AND CALCULATIONS

11.1. ICV percent recoveries are calculated according to the equation:

$$\%R = 100\left(\frac{Found(ICV)}{True(ICV)}\right)$$

11.2. CCV percent recoveries are calculated according to the equation:

$$\%R = 100\left(\frac{Found(CCV)}{True(CCV)}\right)$$

11.3. RLV/CRA percent recoveries are calculated using the same equation as the ICV or CCV (replace ICV or CCV with RLV/CRA in the above equations).

11.4. Matrix Spike Recoveries are calculated according to the following equation:

$$\%R = 100\left(\frac{SSR - SR}{SA}\right)$$

Where:

SSR = Spike Sample Result

SR = Sample Result

SA = Spike Added

11.5. The relative percent difference (RPD) of matrix spike/matrix spike duplicates are calculated according to the following equations:

$$RPD = 100\left[\frac{|MSD - MS|}{\left(\frac{MSD + MS}{2}\right)}\right]$$

Where:

MS = determined spiked sample concentration

MSD = determined matrix spike duplicate concentration

11.6. The final concentration for a digested aqueous sample is calculated as follows:

$$mg/L = \frac{CxV1xD}{V2}$$

Where:

C = Concentration (mg/L) from instrument readout

D = Instrument dilution factor

V1 = Final volume in liters after sample preparation

V2 = Initial volume of sample digested in liters

11.7. The final concentration determined in digested solid samples when reported on a dry weight basis is calculated as follows:

$$mg/Kg, dryweight = \frac{CxVxD}{WxS}$$

Where:

C = Concentration (mg/L) from instrument readout

D = Instrument dilution factor

V = Final volume in liters after sample preparation

W = Weight in Kg of wet sample digested

S = Percent solids/100

Note: A Percent Solids determination must be performed on a separate aliquot when dry weight concentrations are to be reported. If the results are to be reported on wet weight basis the "S" factor should be omitted from the above equation.

11.8. The final concentration determined in digested wipe samples is calculated as follows:

ug/wipe = (C x V x D x 1000)

Where:

C = Concentration (mg/L) from instrument readout

V = Volume of digestate (L)

D = Instrument dilution factor

11.9. The LCS percent recovery is calculated according to the following equation:

$$\%R = 100\left(\frac{Found(LCS)}{True(LCS)}\right)$$

11.10. The dilution test percent difference for each component is calculated as follows:

$$\%Difference = \frac{|I - S|}{I} x100$$

Where:

I = Sample result (Instrument reading)

S = Dilution test result (Instrument reading × 5)

11.11. Appropriate factors must be applied to sample values if dilutions are performed.

11.12. Sample results should be reported with up to three significant figures in accordance with the TestAmerica significant figure policy.

12. METHOD PERFORMANCE

12.1. Initial Demonstration of Capability

12.1.1. An initial demonstration of capability for each method must be performed prior to analyzing samples. For the standard analyte list, the initial demonstration consists of the preparation and analysis of an LCS sample containing all of the standard analytes for the method, as well as a method detection limit (MDL) study (described below).

12.1.2. Four LCS samples are analyzed with the same procedures used to analyze samples, including sample preparation.

12.1.3. The mean recovery and standard deviation are calculated for each analyte of interest. These results are compared with the established or project-specific acceptance criteria. All four results must meet acceptance criteria before the method can be used to analyze samples. For further detail refer to SOP PT-QA-001.

12.2. Prior to analysis of any analyte using either Method 200.7, Method 6010B or 6010C, the following requirements must be met.

12.2.1. Method Detection Limit (MDL) - An MDL must be determined for each analyte prior to the analysis of any client samples. The MDL is determined using seven replicates of reagent water, spiked with all the analytes of interest, that have been carried through the entire analytical procedure. MDLs must be determined in accordance with 40 CFR Part 136 Appendix B requirements as

detailed in TestAmerica QA SOP PT-QA-007. The result of the MDL determination must be below the TestAmerica reporting limit (RL).

12.2.2. Instrument Detection Limit (IDL) 200.7/6010B - The IDL for each analyte must be determined for each analyte wavelength used on each instrument. The IDL will be determined quarterly (every 3 months). If the instrument is adjusted in any way that may affect the IDL, the IDL for that instrument must be redetermined. The IDL shall be determined by multiplying by 3, the average of the standard deviations obtained on three nonconsecutive days from the analysis of a standard solution (each analyte in reagent water) at a concentration 3x - 5x the previously determined IDL, with seven consecutive measurements per day. Each measurement must be performed as though it were a separate analytical sample (i.e., each measurement must be followed by a rinse and/or any other procedure performed between the analysis of separate samples). The IDL must be < MDL.

12.2.3. Instrument detection limits (IDLs) – Method 6010C – IDLs are useful means to evaluate the instrument noise level and response changes over time for each analyte from a series of reagent blank analyses to obtain a calculated concentration. They are not to be confused with the lower limit of quantitation, nor should they be used in establishing this limit. It may be helpful to compare the calculated IDLs to the established lower limit of quantitation, however, it should be understood that the lower limit of quantitation needs to be verified according to the guidance in Sec. 9.9.

12.2.4. IDLs in µg/L can be estimated by calculating the average of the standard deviations of three runs on three non-consecutive days from the analysis of a reagent blank solution with seven consecutive measurements per day. Each measurement should be performed as though it were a separate analytical sample (i.e., each measurement must be followed by a rinse and/or any other procedure normally performed between the analysis of separate samples). IDLs should be determined at least every three months.

12.2.4.1. **DoD samples cannot be analyzed without a valid IDL**.

12.2.4.2. **For DoD, the established IDL must be less than the MDL for each analyte**.

12.2.5. Linear Range Verification (LR) - The linear range will be determined on a quarterly basis for each analyte wavelength used on each instrument as per CLP requirements. Method 6010B/6010C require that the linear range only be determined semiannually. Method 200.7 requires linear ranges to be determined annually. The standards used to define the linear range limit

must be analyzed during a routine analytical run. For the **initial** determination of the upper limit of the linear dynamic range (LDR) for each wavelength, determine the signal responses from a minimum of three to five different concentration standards across the estimated range. One standard should be near the upper limit of the estimated range. The determined concentration of the linear range standards must be within 5% of the true value. The linear range is the concentration above which results cannot be reported without dilution of the sample. If the instrument is adjusted in any way that may affect the LR's, the LR's must be redetermined. The LR data must be documented and kept on file.

12.2.6. Background Correction Points - To determine the appropriate location for off-line background correction when establishing methods, the user must scan the area on either side adjacent to the wavelength and record the apparent emission intensity from all other method analytes. This spectral information must be documented and kept on file. The location selected for background correction must be either free of off-line interelement spectral interference or a computer routine must be used for automatic correction on all determinations. Tests to determine spectral interference must be done using analyte concentrations that will adequately describe the interference. Background correction points must be set prior to determining IECs. Refer to the facility-specific instrument operation SOP and ICP instrument manual for specific procedures to be used in setting background correction points.

12.2.7. Inter-element Corrections (IECs) - ICP interelement correction factors must be determined prior to the analysis of samples and every six months thereafter. If the instrument is adjusted in any way that may affect the IECs, the IECs must be redetermined. When initially determining IECs for an instrument, wavelength scans must be performed to ensure that solutions in use are free from contaminants. If an IEC varies significantly from the previously determined IEC then the possibility of contamination should be investigated. The purity of the IEC check solution can be verified by using a standard from a second source or an alternate method (i.e., GFAA or ICP-MS). Published wavelength tables (e.g. MIT tables, Inductively Coupled Plasma-Atomic Spectroscopy: Prominent Lines) can also be consulted to evaluate the validity of the IECs. Refer to the facility specific instrument operation SOP and instrument manufacturer's recommendations for specific procedures to be used in setting IECs. An IEC must be established to compensate for any interelement interference which results in a false analyte signal greater than ± the RL as defined in Tables I, IA or II. To determine IECs, run a single element standard at the established linear range. To

calculate an IEC, divide the observed concentration of the analyte by the observed concentration of the "interfering element."

Note: Trace ICP IECs are more sensitive to small changes in the plasma and instrument setup conditions. Adjustments in the IECs will be required on a more frequent basis for the Trace as reflected by the ICSA response.

12.2.8. Rinse Time Determination - Rinse times must be determined whenever a new instrument is set up. . To determine the appropriate rinse time for a particular ICP system, the linear range verification standard should be aspirated as a regular sample followed by the analysis of a series of rinse blanks. The length of time required to reduce the analyte signals to < RL will define the rinse time for a particular ICP system. For some analytes it may be impractical to set the rinse time based on the linear range standard result (i.e., analyte not typically detected in environmental samples at that level and an excessive rinse time would be required at the linear range level). Until the required rinse time is established, the method recommends a rinse period of at least 60 seconds between samples and standards. If a memory effect is suspected, the sample must be reanalyzed after a rinse period of sufficient length. Rinse time studies can be conducted at additional concentration levels. These additional studies must be documented and kept on file, if a concentration other than the linear range level is used to set the rinse time. The concentration levels used to establish the rinse time must be taken into consideration when reviewing the data. Linear Range Verifications are performed at a minimum of every six months. Whenever Linear Range Verifications are performed the suitability of the rinse time settings will be evaluated and the rinse time determination will be repeated when necessary.

12.3. Method performance is determined by the analysis of matrix spike and matrix duplicate samples as well as preparation blanks and laboratory control samples. The matrix spike recovery should fall within +/- 25 % and the matrix duplicates should compare within 20% RPD. Preparation blanks must meet the criteria specified in Section 9.3. The LCS should recover within 20% of the true value for aqueous LCS and within the control limits supplied by the manufacturer of the soil LCS.

12.4. Training Qualification:

12.4.1. The group/team leader has the responsibility to ensure that this procedure is performed by an associate who has been properly trained in its use and has the required experience.

13. POLLUTION PREVENTION

13.1. It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

13.2. This method does not contain any specific modifications that serve to minimize or prevent pollution.

14. WASTE MANAGEMENT

14.1. Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to CW-E-M-001. The following waste streams are produced when this method is carried out.

14.1.1. Acid waste consisting of sample and rinse solution, this waste is collected in waste containers identified as "Acid Waste", Waste #33.

14.1.2. Expired Metals Standards – This waste is collected in containers identified as "Acid Waste with Metals", Waste #6.

15. REFERENCES/CROSS REFERENCES

15.1. 40 CFR Part 136, Appendix B, 7-5-95, Determination of Method Detection Limits.

15.2. Test Methods for Evaluating Solid Waste , Physical/Chemical Methods, SW-846, 3rd Edition, Final Update III, Method 6010B,Revision 2, December 1996.

15.3. Test Methods for Evaluating Solid Waste , Physical/Chemical Methods, SW-846, 3rd Edition, Final Update IV, Method 6010C, Revision 3, February, 2007.

15.4. Determination of Metals and Trace Elements in Water and Wastes by Inductively Coupled Plasma-Atomic Emission Spectrometry, Revision 4.4, May 1994. Method 200.7.

15.5. Standard Methods 20th Edition 2340B; Hardness by Calculation.

15.6. SOP PT-QA-001, Employee Orientation and Training, current version.

15.7. SOP PT-HS-001, Waste Collection, Accumulation and Storage, current version.

15.8. SOP PT-QA-007, Determination of Method Detection Limits (MDLs), current version.

15.9. SOP PT-QA-009, Rounding and Significant Figures.

15.10. SOP PT-QA-016, Nonconformance & Corrective Action System, current version.

15.11. SOP PT-QA-018, Technical Data Review Requirements, current version.

15.12. SOP PT-QA-021, Quality Control Program, current version.

15.13. SOP PT-QA-025, Implementation of the DoD QSM Version 3, January 2006, current version.

15.14. SOP PT-QA-029, DoD QSM Version 4.1 Requirements, current version.

16. METHOD MODICATIONS

16.1. Modifications/Interpretations from reference method

16.1.1. Modifications/interpretations from both Methods 6010B and 200.7.

16.1.1.1. TestAmerica laboratories use mixed calibration standard solutions purchased from approved vendors instead of using individual mixes prepared in house as recommended by the subject methods.

16.1.1.2. Methods 200.7 and 6010B state that if the correction routine is operating properly, the determined apparent analyte(s) concentration from analysis of each interference solution should fall within a specific concentration range around the calibration blank. In determining IECs, because of lack of definition clarification for "concentration range around the calibration blank," TestAmerica has adopted the procedure in EPA CLP ILM04.1.

16.1.1.3. Section 8.5 of Method 6010B and Section 9.5 of Method 200.7 recommend that whenever a new or unusual matrix is encountered, a series of tests be performed prior to reporting concentration data for that analyte. The dilution test helps determine if a chemical or physical interference exists. Because TestAmerica laboratories receive no prior information from clients regarding when to expect a new or unusual matrix, TestAmerica may select to perform a dilution test on one sample in each prep batch. According to the method, the post digestion spike (PDS) determines any potential matrix interferences. At TestAmerica labs, matrix interference is determined by evaluating data for the LCS and MS/MSD. TestAmerica requires documented, clear guidance when a new or

unusual matrix will be received for a project and a request to perform the dilution test or PDS on a client-identified sample.

16.1.2. Modifications from Method 200.7.

16.1.2.1. The calibration blank is prepared in an acid matrix of 5% HNO_3/5% HCl instead of the specified 2% HNO_3/10% HCl matrix as the former matrix provides for improved performance relative to the wide variety of digestate acid matrices which result from the various EPA preparation protocols applied.

16.1.2.2. Section 7.12 of 200.7 indicates that the QCS (ICV) should be prepared at a concentration near 1 ppm. The ICV specified in this SOP accommodates the 1 ppm criteria for the majority of analytes. For the remaining analytes, this SOP specifies ICV concentrations, which are appropriate to the range of calibration. The intent of the ICV, verification of calibration standard accuracy, is independent of the ICV concentration used.

16.1.2.3. The ICS criteria applied by this SOP differ from those stated in the method. Method 200.7 section 10.4 states that results should fall within the established control limits of 3 times the standard deviation of the calibration blank for that analyte. TestAmerica Pittsburgh follows the CLP ICS procedures because it is applicable to a wider number of programs. Therefore, we feel it is a more conservative approach.

16.1.2.4. Method 200.7 section 9.3.4 states the CCB should be less than the IDL, but > the lower 3-sigma control limit of the calibration blank. The intent of this requirement is to ensure that the calibration is not drifting at the low end. TestAmerica has adopted an absolute control limit of +/- RL from zero for calibration blank criteria. SOP section 9.8 provides the detailed corrective action criteria that must be followed. **Refer to PT-QA-025 for specific DoD requirements for the CCB.** For DoD V4.1 refer to SOP PT-QA-029.

16.1.3. Modifications from Method 6010B.

16.1.3.1. Chapter 1 of SW-846 states that the method blank should not contain any analyte of interest at or above the MDL. This SOP states that the method blank must not contain any analyte of interest at or above the reporting limit. Common lab contaminants are allowed up to two times the reporting limit in the blank following consultation with the client. **Refer to PT-QA-025 for specific DoD**

requirements for the method blank. For DoD V4.1 refer to SOP PT-QA-029.

16.1.3.2. Method 6010B section 8.6.1.3 states that the results of the calibration blank are to agree within 3x the IDL. If not, repeat the analysis two or more times and average the results. If the average is not within three standard deviations of the background mean, terminate the analysis, correct the problem, recalibrate, and reanalyze the previous 10 samples. The intent of this requirement is to ensure that the calibration is not drifting at the low end. TestAmerica has adopted an absolute control limit of +/- RL from zero for calibration blank criteria. See SOP Section 9.8 for a detailed description of the required corrective action procedures. **Refer to PT-QA-025 for specific DoD requirements for the calibration blanks.** For DoD V4.1 refer to SOP PT-QA-029.

17. ATTACHMENTS

17.1. Documentation and Record Management

- The following documentation comprises a complete ICP raw data package:
- Raw data (direct instrument printout).
- Relevant sample preparation benchsheets.
- Run log printout from instrument software where this option is available (TJA) or manually generated run log (i.e., Ward WSL printout).
- Data review checklist - See Appendix B.
- Standards documentation (including prep and expiration dates, source, and lot #).
- Nonconformance/anomaly documentation (if applicable).

17.2. FIGURE 1 – FLOW DIAGRAM

17.3. APPENDIX A - TABLES

17.4. APPENDIX B - TESTAMERICA ICP DATA REVIEW CHECKLIST

17.5. APPENDIX C - TROUBLESHOOTING GUIDE

17.6. APPENDIX D - CONTAMINATION CONTROL GUIDELINES

17.7. APPENDIX E - PREVENTIVE MAINTENANCE

18. REVISION HISTORY

18.1. Revision 10, 03/31/09

18.1.1. Updated into the new Corporate format; added the appropriate Corporate text to the Scope, Safety, Waste Management and Pollution Control sections; added text to section 9 concerning the Initial Demonstration of Capability being moved to section 12; changed STL to TestAmerica throughout the SOP; updated reference numbers and SOP references throughout the document; updated the Reference section SOP numbers; in section 9.3 changed 20X to 10X; in section 10.10 changed the Trace to any axial ICP.

18.1.2. 6010C method reference added. SOP references updated.

18.1.3. Updated SOP for requirement of Method 6010C, see SOP areas highlighted referring to 6010C.

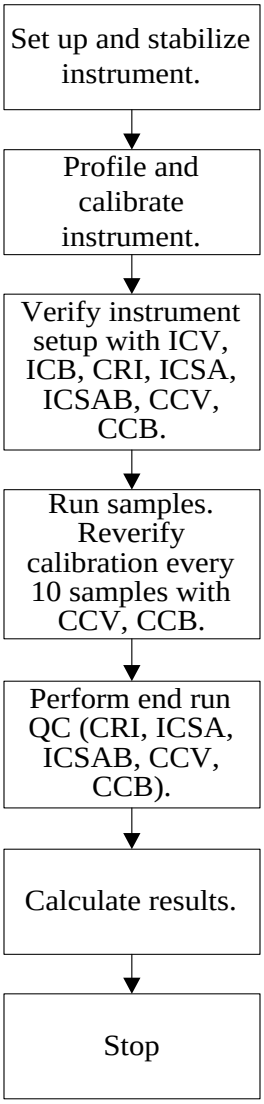
18.1.4. Added to section 8: Dissolved metals samples that are filtered and preserved at the laboratory with concentrated Nitric acid will be held for 24 hours before digestion.

18.2. Revision 11:

18.2.1. Added to section 9.9: For method 6010C the RLV/CRA which is the low level quantitation check sample is prepared and analyzed quarterly or as needed. The control limit is 70-130%. Please note the RLV/CRA (undigested) is still analyzed at the beginning and end of the analytical run.

18.2.2. Added references to SOP PT-QA-029.

Figure 1. Flow Diagram



APPENDIX A

TABLES

TABLE I. Method 200.7, 6010B and 6010C Target Analyte List

ELEMENT	Symbol	CAS #	6010B/6010 C analyte	200.7 analyte	Reporting Limit (ug/L) Water	Reporting Limit (mg/kg) Soil	Reporting Limit (ug/wipe) Wipe
Aluminum	Al	7429-90-5	X	X	200	20	10
Antimony	Sb	7440-36-0	X	X	60	6	3
Arsenic	As	7440-38-2	X	X	300	30	15
Barium	Ba	7440-39-3	X	X	200	20	10
Beryllium	Be	7440-41-7	X	X	4	0.4	0.25
Boron	B	7440-42-8	X	X	200	20	10
Cadmium	Cd	7440-43-9	X	X	5	0.5	0.25
Calcium	Ca	7440-70-2	X	X	5000	500	250
Chromium	Cr	7440-47-3	X	X	10	1	0.5
Cobalt	Co	7440-48-4	X	X	50	5	2.5
Copper	Cu	7440-50-8	X	X	25	2.5	1.25
Iron	Fe	7439-89-6	X	X	100	10	5
Lead	Pb	7439-92-1	X	X	100	10	5
Lithium	Li	7439-93-2	X	X	50	5	2.5
Magnesium	Mg	7439-95-4	X	X	5000	500	250
Manganese	Mn	7439-96-5	X	X	15	1.5	0.75
Molybdenum	Mo	7439-98-7	X	X	40	4	2
Nickel	Ni	7440-02-0	X	X	40	4	2
Phosphorus	P	7723-14-0	X	X	300	30	NA
Potassium	K	9/7/7440	X	X	5000	500	250
Selenium	Se	7782-49-2	X	X	250	25	12.5
Silicon	Si	7631-86-9	X	X	500	N/A	N/A
Silver	Ag	7440-22-4	X	X	10	1	0.5
Sodium	Na	7440-23-5	X	X	5000	500	250
Strontium	Sr	7440-24-6	X	X	50	5	2.5
Thallium	Tl	7440-28-0	X	X	2000	200	100
Vanadium	V	7440-62-2	X	X	50	5	2.5
Zinc	Zn	7440-66-6	X	X	20	2	1

Note: Where reporting “Hardness” by ICP use the following equations per SM20th ed. 2340B:

Calcium Hardness = 2.497 [Ca, mg/L]
Total Hardness = 2.497 [Ca, mg/L] + 4.118 [Mg, mg/L]
Where reporting "Silica" by ICP use the following equation:

Silica = Silicon * 2.14

TABLE IA. Method 200.7, 6010B and 6010C Trace ICP Target Analyte List

ELEMENT	Symbol	CAS #	Reporting Limit (ug/L) Water	Reporting Limit (mg/kg) Soil	Reporting Limit (ug/wipe) Wipe
Arsenic	As	7440-38-2	10	1.0	0.5
Lead	Pb	7439-92-1	3.0	0.3	0.15
Selenium	Se	7782-49-2	5.0	0.5	0.25
Thallium	Tl	7440-28-0	10	1.0	0.5
Antimony	Sb	7440-36-0	10	1.0	0.5
Cadmium	Cd	7440-43-9	5.0	0.5	0.25
Silver	Ag	7440-22-4	5.0	0.5	0.25
Chromium	Cr	7440-47-3	5.0	0.5	0.25

TABLE II. Non-Routine Analyte List

			Reporting	Reporting	Reporting

ELEMENT	Symbol	CAS #	Limit (ug/L) Water	Limit (mg/kg) Soil	Limit (ug/wipe) Wipe
Tin	Sn	7440-31-5	100	10	5
Titanium	Ti	7440-32-6	50	5	2.5

TABLE III. Matrix Spike and Aqueous Laboratory Control Sample Levels

ELEMENT	LCS Level (ug/L)	Matrix Spike Level (ug/L)
Aluminum	2000	2000
Antimony	500	500
Arsenic	2000	2000
Barium	2000	2000
Beryllium	50	50
Cadmium	50	50
Calcium	50000	50000
Chromium	200	200
Cobalt	500	500
Copper	250	250
Iron	1000	1000
Lead	500	500
Lithium	1000	1000
Magnesium	50000	50000
Manganese	500	500
Molybdenum	1000	1000
Nickel	500	500
Potassium	50000	50000
Selenium	2000	2000
Silver	50	50
Sodium	50000	50000
Strontium	1000	1000
Thallium	2000	2000
Vanadium	500	500
Zinc	500	500

ELEMENT	LCS Level (ug/L)	Matrix Spike Level (ug/L)
Boron	1000	1000
Silicon	10000	10000
Tin	2000	2000
Titanium	1000	1000

TABLE IV. ICP Calibration and Calibration Verification Standards

Element	Calibration Level	RL (ug/L)	ICV (ug/L)	CCV (ug/L)
Aluminum	100000	200	25000	50000
Antimony	10000	60	1000	5000
Arsenic	10000	300	1000	5000
Barium	10000	200	1000	5000
Beryllium	10000	4	1000	5000
Cadmium	10000	5	1000	5000
Calcium	100000	5000	25000	50000
Chromium	10000	10	1000	5000
Cobalt	10000	50	1000	5000
Copper	10000	25	1000	5000
Iron	100000	100	25000	50000
Lead	10000	100	1000	5000
Lithium	10000	50	1000	5000
Magnesium	100000	5000	25000	50000
Manganese	10000	15	1000	5000
Molybdenum	10000	40	1000	5000
Nickel	10000	40	1000	5000
Potassium	100000	5000	25000	50000
Selenium	10000	250	1000	5000
Silver	2000	10	500	1000
Sodium	100000	5000	25000	50000
Strontium	10000	50	1000	5000
Thallium	20000	2000	5000	10000
Vanadium	10000	50	1000	5000
Zinc	10000	20	1000	5000

Element	Calibration Level	RL (ug/L)	ICV (ug/L)	CCV (ug/L)
Boron	10000	200	1000	5000
Silicon	10000	500	1000	5000
Tin	10000	100	1000	5000
Titanium	10000	50	1000	5000

TABLE IVA. Trace ICP Calibration and Calibration Verification Standards

Element	Calibration Level	RL (ug/L)	ICV (ug/L)	CCV (ug/L)
Aluminum	50000	200	12500	25000
Antimony	1000	10	250	500
Arsenic	1000	10	250	500
Barium	4000	10	1000	2000
Beryllium	4000	4	1000	2000
Cadmium	1000	5	250	500
Calcium	100000	5000	25000	50000
Chromium	4000	5	1000	2000
Cobalt	4000	50	1000	2000
Copper	4000	25	1000	2000
Iron	50000	100	12500	25000
Lead	1000	3	250	500
Magnesium	100000	5000	25000	50000
Manganese	4000	15	1000	2000
Molybdenum	4000	40	1000	2000
Nickel	4000	40	1000	2000
Potassium	250000	5000	50000	125000
Selenium	1000	5	250	500
Silver	2000	5	500	1000
Sodium	250000	5000	50000	125000
Thallium	2000	10	500	1000
Vanadium	4000	50	1000	2000
Zinc	4000	20	1000	2000
Boron	4000	200	1000	2000

Element	Calibration Level	RL (ug/L)	ICV (ug/L)	CCV (ug/L)
Silicon	4000	500	1000	2000
Tin	4000	100	1000	2000
Titanium	4000	50	1000	2000

TABLE V. Interference Check Sample Concentrations*

Element	ICSA (ug/L)	ICSAB (ug/L)
Aluminum	500000	500000
Antimony	-	1000
Arsenic	-	1000
Barium	-	500
Beryllium	-	500
Cadmium	-	1000
Calcium	500000	500000
Chromium	-	500
Cobalt	-	500
Copper	-	500
Iron	200000	200000
Lead	-	1000
Magnesium	500000	500000
Manganese	-	500
Molybdenum	-	1000
Nickel	-	1000
Potassium	-	10000
Selenium	-	1000
Silver	-	1000
Sodium	-	10000
Thallium	-	10000**
Vanadium	-	500
Zinc	-	1000
Tin	-	1000

* Custom TestAmerica solutions contain common analytes. Non-routine elements not listed above should be spiked into the ICSAB at 1000 ug/L.
** Thallium level for Trace ICP should be at 1000 ug/L.

TABLE VI. TCLP Reporting Limits, Regulatory Limits and Matrix Spike Levels

ELEMENT	Reporting Level (ug/L)	Regulatory Limit (ug/L)	Spike Level (ug/L)
Arsenic	500	5000	5000
Barium	10000	100000	50000
Cadmium	100	1000	1000
Chromium	500	5000	5000
Lead	500	5000	5000
Selenium	250	1000	1000
Silver	500	5000	1000

TABLE VII. Summary of Quality Control Requirements

QC PARAMETER	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
Two-point Initial Calibration	Beginning of every analytical run, every 24 hours, whenever instrument is modified, or CCV criterion is not met	RSD between duplicate exposures ≤5%	Terminate analysis; Correct the problem; Prepare new standards; Recalibrate following system performance.
ICV	Beginning of every analytical run.	Method 200.7: 95 - 105 % recovery. Method 6010B & 6010C: 90 - 110 % recovery.	Terminate analysis; Correct the problem; Recalibrate.
ICB	Beginning of every analytical run, immediately following the ICV.	The result must be within +/- RL from zero. (1)	Terminate analysis; Correct the problem; Recalibrate.
RLV/CRA/LLICV/LLCCV	Beginning of every analytical run, immediately following the ICB. Method 6010C also requires analysis at the end of the analytical sequence.	50 – 150% recovery. (1) Method 6010C: 70 – 130% recovery	Terminate analysis; Correct the problem; Recalibrate.
CCV	Every 10 samples and at the end of the run.	Method 200.7, 6010B & 6010C: 90 - 110 % recovery.	Terminate analysis; Correct the problem; Recalibrate and rerun all samples not bracketed by acceptable CCV.
CCB	Immediately following each CCV.	The result must be within +/- RL from zero. (1)	Terminate analysis; Correct the problem; Recalibrate and rerun all samples not bracketed by acceptable CCB.

TABLE VII. Summary of Quality Control Requirements

QC PARAMETER	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
ICSA	Beginning of every run	See Section 9.10.3 (1)	See Section 9.10.3.
ICSAB	Immediately following each ICSA.	Results must be within 80 - 120% recovery.	See Section 9.10.2.
Dilution Test	One per prep batch.	For samples > 50x MDL, dilutions must agree within 10%.	Narrate the possibility of physical or chemical interference per client request.
Method Blank	One per sample preparation batch of up to 20 samples.	The result must be less than or equal to the RL. (1) Common lab contaminants may be accepted up to 2x the RL after consultation with the client (See 9.3). Sample results greater than 20x the blank concentration are acceptable. Samples for which the contaminant is < RL may not require redigestion or reanalysis (see Section 9.3).	Redigest and reanalyze samples. Note exceptions under criteria section. See Section 9.3 for additional requirements.
Laboratory Control Sample (LCS)	One per sample preparation batch of up to 20 samples.	Aqueous LCS must be within 80 - 120% recovery or in-house control limits. (85-115% for 200.7)	Terminate analysis; Correct the problem; Redigest and reanalyze all samples associated with the LCS.

TABLE VII. Summary of Quality Control Requirements			
QC PARAMETER	**FREQUENCY**	**ACCEPTANCE CRITERIA**	**CORRECTIVE ACTION**
		Samples for which the contaminant is < RL and the LCS results are > 120% (115% for 200.7) may not require redigestion or reanalysis (see Section 9.4)	
Matrix Spike	One per sample preparation batch of up to 20 samples (6010B/6010C) or one per every 10 or fewer samples (200.7).	75 - 125 % (6010B & 6010C) or 70 – 130% (200.7) recovery or in-house control limits. (1) For TCLP See Section 10.15.	In the absence of client specific requirements, flag the data; no flag required if the sample level is > 4x the spike added. For TCLP see Section 10.15.
Matrix Spike Duplicate	See Matrix Spike	75 - 125 % recovery; RPD ≤ 20%. (1)	See Corrective Action for Matrix Spike.

(1) For specific DoD requirements, refer to PT-QA-025. For DoD V4.1 refer to SOP PT-QA-029.

APPENDIX B

TESTAMERICA ICP DATA REVIEW CHECKLIST

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

TestAmerica Pittsburgh ICP Data Review Checklist

Run Date: _____ 3. _____ 10. _____ 17. _____ 24. _____ 31. _____
Analyst: _____ 4. _____ 11. _____ 18. _____ 25. _____ 32. _____
Inst: _____ 5. _____ 12. _____ 19. _____ 26. _____ 33. _____
Meth: _____ 6. _____ 13. _____ 20. _____ 27. _____ 34. _____
Lots Analyzed: 7. _____ 14. _____ 21. _____ 28. _____ 35. _____
1. _____ 8. _____ 15. _____ 22. _____ 29. _____ 36. _____
2. _____ 9. _____ 16. _____ 23. _____ 30. _____ 37. _____

Review Item	Yes (✓)	No (✓)	N/A (✓)	1st Lv (✓)	Comments
A. Calibration/Instrument Run QC 1. Instrument calibrated per manufacturer's instructions and at SOP specified levels?					
2. ICV/CCV analyzed at appropriate frequency and within control limits? (6010B, CLP=90-110%, 200.7=95-105%[ICV])?					
3. ICB/CCB analyzed at appropriate frequency and within +/- RL or +/- CRDL (CLP)?					
4. CRA/RLV/CRI analyzed? (CRI for CLP only)					
5. ICSA/ICSAB run at required frequency and within SOP limits?					
B. Sample Results 1. Were samples with concentrations > the linear range for any parameter diluted and reanalyzed?					
2. All reported results bracketed by in control QC?					
3. Sample analyses done within holding time?					
C. Preparation/Matrix QC 1. LCS done per prep batch and within QC limits?					
2. Method blank done per prep batch and < RL or CRDL (CLP)?					
3. MS run at required frequency and within limits?					
4. MSD or DU run at required frequency and RPD within SOP limits?					
5. Dilution Test done per prep batch (or per SDG for CLP)?					
6. Post digestion spike analyzed if required (CLP only)?					
D. Other 1. Are all nonconformances documented appropriately?					
2. Current IDL/LR/IEC data on file?					
3. Calculations checked for error?					
4. Transcriptions checked for error?					
5. All client/project specific requirements met?					
6. Date/Time of analysis verified as correct?					

General Comments: _____

Analyst & Date: _____ Second-Level Review & Date: _____

Quantera PT09-ICPM-001\OCMQ\ICP\ICP.DOC

APPENDIX C

CROSS REFERENCE OF TERMS USED IN METHODS 6010B, 6010C, 200.7 AND BY TESTAMERICA

CROSS REFERENCE OF TERMS COMMONLY USED IN

METHODS EPA 200.7, SW846 6010B/6010C AND TESTAMERICA INC. SOP

EPA 200.7	SW6010B/6010C	TestAmerica Inc. SOP
Calibration blank (CB)	Calibration blank	Initial and continuing calibration blanks (ICB/CCB)
Dilution test	Dilution test	Dilution Test
Instrument detection limit (IDL)	Instrument detection limit (IDL)	Instrument detection limit (IDL)
Instrument performance check (IPC)	Continuing calibration verification (CCV)	Continuing calibration verification (CCV)
Internal standard	Internal standard	Internal standard (IS)
Laboratory duplicates	n/a	n/a
Laboratory fortified blank (LFB)	n/a	Laboratory control sample (LCS)
Laboratory fortified sample matrix (LFM)	Matrix spike and matrix spike duplicate (MS/MSD)	Matrix spike and matrix spike duplicate (MS/MSD)
Laboratory reagent blank (LRB)	Method blank	Method or Prep blank (MB)
Linear dynamic range (LDR)	Linear dynamic range (LDR)	Linear dynamic range (LDR)
Method detection limit (MDL)	Method detection limit (MDL)	Method detection limit (MDL)

EPA 200.7	SW6010B/6010C	TestAmerica Inc. SOP
Quality control sample (QCS)	Check standard or Initial calibration verification (ICV)	Initial calibration verification (ICV)
Spectral interference check solution (SIC)	Interference check solution (ICS)	Interference check solution (ICSA/ICSAB)

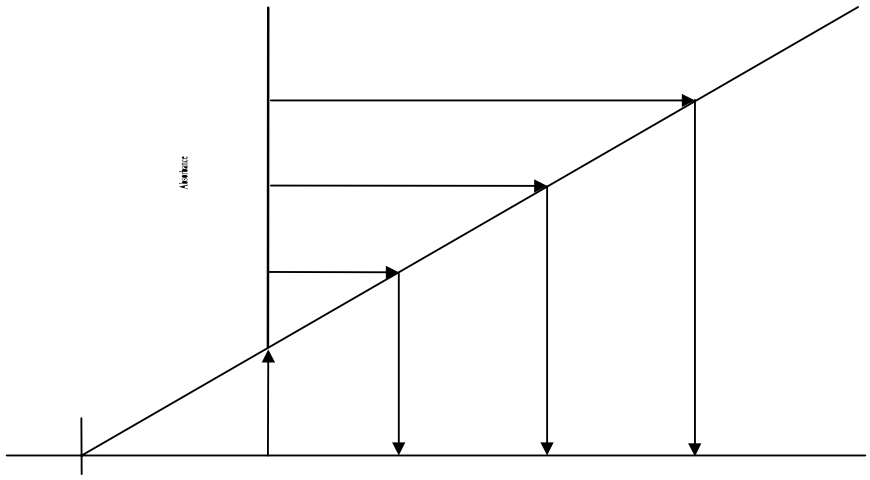
APPENDIX D

MSA GUIDANCE

Method of Standard Addition

Four equal volume aliquots of sample are measured and known amounts of standards are added to three aliquots. The fourth aliquot is the unknown and no standard is added to it. The concentration of standard added to the first aliquot should be 50% of the expected concentration. The concentration of standard added to the second aliquot should be 100% of the expected concentration and the concentration of standard added to the third aliquot should be 150% of the expected concentration. The volume of the unspiked and spiked standard should be the same.

In order to determine the concentration of analyte in the sample, the analytical value of each solution is determined and a plot or linear regression performed. On the vertical axis the analytical value is plotted versus the concentrations of the standards on the horizontal axis. An example plot is shown in Figure 1. When the resulting line is extrapolated back to zero absorbance, the point of interception of the horizontal axis is the concentration of the unknown.



- For the method of standard additions to be correctly applied, the following limitations must be taken into consideration:

- The plot of the sample and standards must be linear over the concentration range of concern. For best results, the slope of the curve should be similar to that of a plot of the aqueous standard curve.

- The effect of the interference should not vary as the ratio of the standard added to the sample matrix changes.

APPENDIX E

TROUBLESHOOTING GUIDE

Problem	Possible Cause/Solution
High Blanks	Increase rinse time Clean or replace tip Clean or replace torch Clean or replace sample tubing Clean or replace nebulizer Clean or replace mixing chamber Lower Torch
Instrument Drift	RF not cooling properly Vacuum level is too low Replace torch (Crack) Clean or replace nebulizer (blockage) Check room temperature (changing) Replace pump tubing Room humidity too high Clean torch tip (salt buildup) Check for argon leaks Adjust sample carrier gas Reprofile Horizontal Mirror Replace PA tube
Erratic Readings, Flickering Torch or High RSD	Check for argon leaks Adjust sample carrier gas Replace tubing (clogged) Check drainage(back pressure changing) Increase uptake time (too short) Increase flush time (too short) Clean nebulizer, torch or spray chamber Increase sample volume introduced Check that autosampler tubes are full Sample or dilution of sample not mixed Increase integration time (too short) Realign torch Reduce amount of tubing connectors
Cu/Mn Ratio Outside Limits or Low Sensitivity	Plasma conditions changed Clean nebulizer, torch or spray chamber Replace tubing (clogged)

Problem	Possible Cause/Solution
	Realign torch Check IECs
Standards reading twice normal absorbance or concentration	Incorrect standard used Incorrect dilution performed

APPENDIX F

CONTAMINATION CONTROL GUIDELINES

APPENDIX F. CONTAMINATION CONTROL GUIDELINES

The following procedures are strongly recommended to prevent contamination:

All work areas used to prepare standards and spikes should be cleaned before and after each use.

All glassware should be washed with detergent and tap water and rinsed with 20% nitric acid followed by deionized water.

Proper laboratory housekeeping is essential in the reduction of contamination in the metals laboratory. All work areas must be kept scrupulously clean.

Powdered or Latex Gloves must not be used in the metals laboratory since the powder contains silica and zinc as well as other metallic analytes. Only vinyl or nitrile gloves should be used in the metals laboratory.

Glassware should be periodically checked for cracks and etches and discarded if found. Etched glassware can cause cross contamination of any metallic analytes.

Autosampler trays should be covered to reduce the possibility of contamination. Trace levels of elements being analyzed in the samples can be easily contaminated by dust particles in the laboratory.

The following are helpful hints in the identification of the source of contaminants:

Yellow pipette tips and volumetric caps can sometimes contain cadmium.

Some sample cups have been found to contain lead.

The markings on glass beakers have been found to contain lead. If acid baths are in use for glassware cleaning, they should be periodically checked for contaminants since contaminant concentrations will increase over time.

New glassware especially beakers can be a source of silica and boron.

Reagents or standards can contain contaminants or be contaminated with the improper use of a pipette.

Improper cleaning of glassware can cause contamination.

Latex gloves contain over 500 ppb of zinc.

APPENDIX G

PREVENTATIVE MAINTENANCE

APPENDIX G. PREVENTIVE MAINTENANCE

A maintenance log is used to record when maintenance is performed on instruments. When an instrument problem occurs indicate the date, time and instrument number, then identify the problem and corrective action in the maintenance log.

The following procedures are required to ensure that that the instrument is fully operational.

Daily
Change sample pump tubing and pump windings
Check argon gas supply level
Check rinse solution and fill if needed
Check waste containers and empty if needed
Check sample capillary tubing is clean and in good condition
Check droplet size to verify nebulizer is not clogged.
Check sample flow for cross flow nebulizer
Check Cu/Mn ratio-should be 30% of value at date that IECs were performed
Check pressure for vacuum systems

As Needed
Clean plasma torch assembly to remove accumulated deposits
Clean nebulizer and drain chamber; keep free-flowing to maintain optimum performance
Replace peristaltic pump tubing, sample capillary tubing, and autosampler sipper probe

Weekly
Apply silicon spray on autosampler tracks
Check water level in cool flow

Monthly
Clean air filters on back of power unit to remove dust
Check D mirror for air instruments

Bi-yearly
Change oil for vacuum systems
Replace coolant water filter (may require more or less frequently depending on quality of cooling water)

G-2 SOP NO. PT-IP-002, REV. 8, EFFECTIVE DATE: 04/28/09,

ACID DIGESTION OF SOILS,

METHOD: SW846 METHOD 3050B

Title: Acid Digestion of Soils

Method: SW846 Method 3050B

Approvals (Signature/Date):

	07/17/09		07/20/09
William Reinheimer	Date	Steve Jackson	Date
Technical Manager		Health & Safety Manager	
	07/17/09		07/17/09
Nasreen DeRubeis	Date	Larry Matko	Date
Quality Assurance Manager		Laboratory Director	

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1. SCOPE AND APPLICATION

1.1. This procedure describes the preparation of soil samples for the analysis of certain metals by Inductively Coupled Plasma Atomic Emission Spectroscopy (ICP) and Inductively Coupled Plasma/Mass Spectrometry (ICP/MS) as specified in SW846 Method 3050B.

1.2. Samples prepared by the protocols detailed in this SOP may be analyzed by ICP or ICP/MS for the elements listed in Table I (Appendix A). Other elements and matrices may be analyzed following digestion by these protocols provided that the method performance criteria specified in Section 12.0 of this SOP are met.

1.3. This method is not a total digestion, but will dissolve almost all metals that could become "environmentally available". By design, metals bound in silicate structures are not dissolved by this procedure, as they are not usually mobile in the environment. This SOP can be applied to metals in solids, sludges, wastes, sediments, wipes and tissues.

2. SUMMARY OF METHOD

A representative 1-gram (wet weight) portion of sample is digested in nitric acid and hydrogen peroxide. The digestate is refluxed with hydrochloric acid for ICP analysis. The digestates are then filtered and diluted to 100 mL/100 g.

3. DEFINITIONS

Additional definitions of terms used in this SOP may be found in the glossary of the PT-LQAM.

3.1. Total Metals: The concentration determined on an unfiltered sample following digestion. Note that this method is designed to determine the total *environmentally available* metals.

4. INTERFERENCES

4.1. There are numerous routes by which samples may become contaminated. Potential sources of trace metals contamination include: metallic or metal-containing labware (e.g., talc gloves which contain high levels of zinc), containers, impure reagents, dirty glassware, improper sample transfers, dirty work areas, atmospheric inputs such as dirt and dust, etc. Be aware of potential sources of contamination and take appropriate measures to minimize or avoid them.

4.2. The entire work area, including the bench top and fume hood, should be thoroughly cleaned on a routine schedule in order to minimize the potential for environmental contamination. Refer to Appendix C for additional contamination control guidelines.

4.3. Boron and silica from the glassware will grow into the sample solution during and following sample processing. For critical low-level determinations of boron and silica, only quartz and/or plastic labware should be used.

4.4. Physical interference effects may contribute to inaccuracies in the determinations of trace elements. Oils, solvents and other matrices may not be digested using these methods if they are not soluble with acids. If physical interferences are present, they should be documented.

4.5. Visual interferences or anomalies (such as foaming, emulsions, precipitates, etc.) must be documented.

4.6. Allowing samples to boil or go dry during digestion may result in the loss of volatile metals. If this occurs the sample must be reprepared. Antimony is easily lost by volatilization from hydrochloric media.

4.7. Specific analytical interferences are discussed in each of the determinative methods.

5. SAFETY

5.1. Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001), Radiation Safety Manual and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.2. Samples that contain high concentrations of carbonates or organic material or samples that are at elevated pH can react violently when acids are added.

5.3. All heating of samples must be carried out in a fume hood.

5.4. The acidification of samples containing reactive materials may result in the release of toxic gases, such as cyanides or sulfides. Acidification of samples should be done in a fume hood. The analyst should also be aware of the potential for a vigorous reaction.

5.5. The following is a list of the materials used in this method, which have a serious or significant hazard rating. **NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS

for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Hydrochloric Acid	Corrosive Poison	5 ppm-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Hydrogen Peroxide	Oxidizer Corrosive	1 ppm-TWA	Vapors are corrosive and irritating to the respiratory tract. Vapors are very corrosive and irritating to the eyes and skin.
Nitric Acid	Corrosive Oxidizer Poison	2 ppm-TWA 4 ppm-STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

5.6. Eye protection that protects against splash, laboratory coat, and appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled. Cut resistant gloves must be worn doing any other task that presents a strong possibility of getting cut. Disposable gloves that have become contaminated will be removed and discarded; other gloves will be cleaned immediately.

5.7. The preparation of standards and reagents will be conducted in a fume hood with the sash closed as far as the operation will permit or under other means of mechanical ventilation.

5.8. All work must be stopped in the event of a known or potential compromise to the health and safety of a TestAmerica associate. The situation must be reported **immediately** to a laboratory supervisor or EH&S coordinator.

6. EQUIPMENT AND SUPPLIES

6.1. Hot plate, hot block, or other heating source capable of maintaining a temperature of 90-95°C.

6.2. Thermometer that covers a temperature range of 0-150°C.

6.3. Hot block Disposable Digestion Cup (from Environmental Express).

6.4. Vapor recovery device (Watch glasses, ribbed or other device).

6.5. Whatman No. 41 filter paper or equivalent.

6.6. Funnels or equivalent filtration apparatus.

6.7. Centrifugation equipment (if desired method of removing particulates is centrifugation).

6.8. Graduated cylinder or equivalent capable of measuring 100 mL within 3% accuracy.

6.9. Analytical balance capable of accurately weighing to the nearest 0.01 grams.

6.10. Repipetors or suitable reagent dispensers.

6.11. Calibrated automatic pipettes with corresponding pipette tips or Class A glass volumetric pipettes.

6.12. Class A volumetric flasks.

6.13. pH indicator strips (pH range 0 - 6).

6.14. Plastic bottles.

6.15. Teflon chips.

7. REAGENTS AND STANDARDS

7.1. Reagent water must be produced by a Millipore DI system or equivalent. Reagent water must be free of the analytes of interest as demonstrated through the analysis of method blanks as defined in the determinative SOPs.

7.2. Laboratory Control Sample (LCS) and matrix spike (MS) solutions are purchased as custom TestAmerica solutions. All standards must be stored in FEP fluorocarbon or previously unused polyethylene or polypropylene bottles. Stock standard solutions must be replaced prior to the expiration date provided by the manufacturer. If no expiration date is provided, the stock solutions may be used for up to one year and must be replaced sooner if verification from an independent source indicates a problem.

7.3. Working ICP LCS/MS spike solution: The ICP LCS/MS working spike solution is provided directly by the vendor, no further standard preparation is necessary.

7.4. The LCS and MS samples must contain all the elements designated for analysis in each batch of samples. If a non-routine element is required that is not contained in the custom TestAmerica solution, a solution must be purchased from a designated vendor that will cover the additional analyte(s) of interest and provide for a final spike concentration that is appropriate to the determinative method.

7.5. Laboratory Control Samples (LCSs) and matrix spike samples are prepared as described in Sections 9.5 and 9.6. Refer to Table II (Appendix A) for details regarding the stock, working standard and final digestate spike concentrations for the ICP LCS and matrix spike preparations.

7.6. Nitric acid (HNO_3), concentrated, trace metal grade or better. For ICPMS analysis, Optima reagents (ultra pure) are used for sample preparation.

7.7. Nitric acid, 1:1 - dilute concentrated HNO_3 with an equal volume of reagent water.

Note: When preparing diluted acids always add acid to water. If the water is added to the acid a violent reaction may occur.

7.8. Hydrochloric acid (HCl), concentrated, trace metal grade or better.

7.9. Hydrochloric acid, 1:1 - dilute concentrated HCl with an equal volume of reagent water.

Note: When preparing diluted acids always add acid to water. If the water is added to the acid a violent reaction may occur.

7.10. 30% Hydrogen peroxide (H_2O_2), reagent or ultrex grade.

8. SAMPLE COLLECTION, PRESERVATION, SHIPMENT AND STORAGE

8.1. Sample holding time for metals included under the scope of this SOP is 180 days from the date of collection to the date of analysis.

8.2. Soil and wipe samples do not require preservation but must be stored at 4 °C ± 2 °C until the time of analysis. Tissue samples will be stored frozen.

9. QUALITY CONTROL

Table III (Appendix A) provides a summary of quality control requirements including type, frequency, acceptance criteria and corrective action.

9.1. The initial demonstration study as detailed in Section 12.1 must be acceptable before the analysis of field samples under this SOP may begin. The results of the initial demonstration study may be used to extend a method for the analysis of other elements provided all acceptance criteria are met.

9.2. Preparation Batch - A group of up to 20 samples that are of the same matrix and are processed together using the same procedures and reagents. The preparation batch must contain a method blank, a LCS and a matrix spike/matrix spike duplicate. In some cases, at client request, it may be appropriate to process a matrix spike and sample duplicate in place of the MS/MSD. If clients specify specific samples for MS/MSD, the batch may contain multiple MS/MSD pairs.

9.3. Sample Count - Laboratory generated QC samples (method blanks, LCS, MS/MSD) are not counted towards the maximum 20 samples in a batch. Field QC samples are included in the batch count.

9.4. Method Blank (MB) - One method blank must be processed with each preparation batch. The method blank consists of Teflon chips, to which all reagents specific to the method are added and then carried through the entire analytical procedure, including preparation and analysis. The method blank is used to identify any system and process interferences or contamination of the analytical system that may lead to the reporting of elevated analyte concentrations or false positive data. Criteria for the acceptance of blanks are contained within the individual analytical method SOP's. If the method blank does not meet the criteria contained within the analytical method SOPs, the blank and all associated samples in the batch must be redigested.

9.4.1. Soil method blanks are prepared by taking 1 g of Teflon chips through the procedure described in Section 10.10.

9.5. Laboratory Control Sample (LCS) - One LCS must be processed with each preparation batch. The LCS must contain all analytes of interest and must be carried through the entire analytical procedure. The LCS is used to monitor the accuracy of the analytical process. On-going monitoring of the LCS results provides evidence that the laboratory is performing the method within acceptable accuracy and precision guidelines. Criteria for the acceptance of LCS results are contained within the individual analytical method SOP's. Corrective action when LCS results fail to meet control limits will be repreparation and reanalysis of the batch. Table II provides the details regarding the stock, working standards and final spike concentrations for ICP. Refer to Section 7.3 for instructions on preparation of the LCS.

9.5.1. The LCS is prepared by spiking a 1 g aliquot of Teflon chips with 1 mL of the working LCS/MS spike solution (7.3). The LCS is then processed as described in Section 10.10.

9.6. Matrix Spike/Matrix Spike Duplicate (MS/MSD) - One MS/MSD pair must be processed for each preparation batch. A matrix spike (MS) is a field sample to which known concentrations of target analytes have been added. A matrix spike duplicate (MSD) is a second aliquot of the same sample (spiked identically as the MS) prepared and analyzed along with the sample and matrix spike. Some client specific data quality objectives (DQO's) may require the use of sample duplicates in place of or in addition to MS/MSD's. The MS/MSD results are used to determine the effect of a matrix on the precision and accuracy of the analytical process. Samples identified as field blanks cannot be used for MS/MSD analysis. If any analyte recovery or RPD falls outside the acceptance range, the recovery of that analyte must be in control for the LCS. If the recovery of the LCS is outside limits, corrective action must be taken. Corrective action will include repreparation and reanalysis of the batch. Corrective action when MS results fail to meet control limits does not include repreparation of samples unless the results indicate that a spiking error may have occurred. Table II provides the details regarding the stock, working standards and final matrix spike concentrations for ICP. Refer to Section 7.3 for instructions on preparation of the working matrix spike solutions.

9.6.1. The soil matrix spike sample is prepared by spiking a 1 g aliquot of a sample with 1 mL of the working LCS/MS spike solution (7.3). The matrix spike sample is then processed as described in Section 10.10.

9.7. Quality Assurance Summaries - Certain clients may require specific project or program QC, which may supersede the SOP requirements. Quality Assurance Summaries (QAS) should be developed to address these requirements.

10. PROCEDURE

10.1. One time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance Memo and is approved by a Technical Specialist and QA Manager. If contractually required, the client shall be notified. The Nonconformance Memo shall be filed in the project file.

10.2. Any unauthorized deviations from this procedure must also be documented as a nonconformance, with a cause and corrective action described.

10.3. Hotplate or hot block temperature must be verified daily for each unit used and must be recorded on either the metals preparation log or in a hotplate/hotblock temperature logbook. The hotplate/hotblock temperature should be verified by measuring the temperature of a beaker of reagent water placed on each hotplate/hotblock. For block digestors, use a tube containing water.

10.4. All preparation procedures must be carried out in a properly functioning hood.

10.5. All samples are to be checked out of sample control with the chain of custody documentation filled out completely.

10.6. Proper sample identification is extremely important in any preparation procedure. Labeling of beakers and bottles must be done in a manner to ensure connection with the proper sample. The use of automatic label printing programs is recommended to reduce transcription errors (Quantims option).

10.7. Samples are typically logged in as either waters or soils. Wastes such as organic liquids or sludges and tissues (animal/vegetable) are usually logged in with solid test codes. When initiating prep examine the sample to see if the sample matches the matrix designation. If the sample is logged in as aqueous but it appears more like a waste (biphasic, sludge like, organic liquid, lots of sediment etc.) contact the lab supervisor or project administrator for further instructions. In some cases it may be more appropriate to process these samples as solids.

10.8. If possible prepare all the samples of a project at the same time to minimize the QC required and streamline the flow of the project through the lab and reporting group.

10.9. Guidelines are provided in the appendices on procedures to minimize contamination of samples and standards.

10.10. Preparation of Soils, Sediments, Sludges,Tissues or Wipes for Analysis by ICP or ICP/MS.

10.10.1. Mix sample thoroughly by stirring with a clean plastic or wooden spoon or spatula.

10.10.1.1. Refer to PT-QA-024 for subsampling procedures.

10.10.1.2. Mixing is not required for wipe samples because the entire wipe is used in the digestion.

10.10.2. For each sample, weigh a 1.0 gram portion of solid and record the exact weight to the nearest 0.01 g. A 2 gram sample size may also be used if needed to meet the reporting limits.

10.10.2.1. For wipe samples, the entire sample is digested and no subsampling is required.

10.10.3. Measure additional aliquots of the designated samples for the MS and MSD analyses.

10.10.3.1. Due to the unique nature of wipe samples, an MS and MSD are not practical. An LCS/LCSD is used to satisfy precision and accuracy requirements for wipes.

10.10.4. Spike each of the MS and MSD aliquots with 1 mL of the working LCS/MS spiking solution (7.3).

10.10.5. Measure 1 g of Teflon chips into a beaker for the method blank.

10.10.5.1. Measure 0.5 mL of reagent water into a beaker for the method blank for wipe samples.

10.10.6. Measure 1 g of Teflon chips into a beaker for the LCS. Spike the LCS aliquot with 1 mL of the working LCS/MS spiking solution (7.3).

10.10.6.1. Measure 0.5 mL of reagent water into a beaker for the LCS and LCSD for wipe samples. Spike the LCS and LCSD aliquots with 0.5 mL of the working LCS/MS spiking solution (7.3).

10.10.7. Add 10 mL of 1:1 HNO_3 and mix the sample.

10.10.7.1. For wipes, add 5 mL of 1:1 HNO_3 and mix the sample.

10.10.8. Cover with a ribbed watch glass.

10.10.9. Heat sample to 95°C and reflux for 10 minutes without boiling, using a vapor recovery device.

Note: DO NOT ALLOW SAMPLE TO BOIL OR GO DRY during any part of the digestion. Doing so will result in the loss of analyte and the sample must be reprepared.

10.10.10. Allow sample to cool.

10.10.11. Add 5 mL of concentrated HNO_3 and replace vapor recovery device.

10.10.11.1. For wipes, add 2.5 mL of concentrated HNO_3 and replace vapor recovery device.

10.10.12. Reflux at 95°C for 30 minutes. (Add reagent water as needed to ensure that the volume of solution is not reduced to less than 5 mL.)

10.10.13. If brown fumes are observed, repeat steps 10.10.11 and 10.10.12 until no more fumes are evolved.

10.10.14. Using a vapor recovery device, allow the sample to evaporate to 5 - 10 mL while ensuring that no portion of the bottom of the beaker is allowed to go dry. Alternatively heat at 95°C for 2 hours.

10.10.15. Allow the samples to cool.

10.10.16. Add 2 mL of reagent water and 3 mL of 30 % H_2O_2. Care must be taken to ensure that losses do not occur due to excessively vigorous effervescence.

10.10.16.1. For wipes, add 1 mL of reagent water and 1.5 mL of 30 % H_2O_2. Care must be taken to ensure that losses do not occur due to excessively vigorous effervescence.

10.10.17. Replace the vapor recovery device and heat sample until effervescence subsides.

10.10.18. Allow the sample to cool.

10.10.19. Continue adding 30% H_2O_2 in 2 mL aliquots with warming until effervescence is minimal or sample appearance is unchanged.

10.10.19.1. For wipes, continue adding 30% H_2O_2 in 1 mL aliquots with warming until effervescence is minimal or sample appearance is unchanged.

Note: Do not add more than a total of 10 mL (5 ml for wipe samples) of 30 % H_2O_2.

10.10.20. Continue heating at 95°C until the volume is reduced to approximately 5 mL. Alternatively the sample may be heated for 2 hours.

10.10.21. Add 10 mL of concentrated HCl and reflux for an additional 15 minutes without boiling.

10.10.21.1. For wipes, add 5 mL of concentrated HCl and reflux for an additional 15 minutes without boiling.

Note: Antimony and silver have poor solubility in dilute nitric acid solution. Therefore it is strongly recommended that these elements are determined by the ICP procedure that includes HCl as the final digestion acid.

10.10.22. Allow the sample to cool.

10.10.23. Wash down beaker walls and vapor recovery device with reagent water.

10.10.24. Filter sample through Whatman 41 filter paper or equivalent into a pre-weighed bottle. Other measuring bottles (for example, Corning Snap Seals™) may be used if their average error is documented and is better than ± 2%. Rinse beaker and filter paper with reagent water to ensure complete sample transfer.

Note: In place of filtering, the samples, after dilution and mixing, may be centrifuged or allowed to settle by gravity overnight to remove insoluble material

10.10.25. Dilute sample to 100 mL or 100g with reagent water. The sample is now ready for analysis.

10.10.25.1. For wipe samples, dilute sample to 50 mL or 50g with reagent water. The sample is now ready for analysis.

Note: This SOP allows for samples to be weighed instead of measured volumetrically. This assumes the density of the diluted sample is close to 1.0 g/mL (See Section 16.1.2).

10.11. Documentation and Record Management

10.11.1. The preparation benchsheet should, at a minimum, include the following information:

- Preparation date, analyst name, matrix, prep type (ICP or ICP/MS), SOP reference.
- Sample ID, initial weight/volume and final weight/volume.
- Standards Documentation (source, lot, prep date, volume added).
- Analyst Signature.
- Reviewer's Signature and date.

11. CALCULATIONS / DATA REDUCTION

Not Applicable.

12. METHOD PERFORMANCE

12.1. Initial Demonstration of Capability

Prior to analysis of any analyte using Method 3050B the following requirements must be met.

12.1.1. Method Detection Limit (MDL) - An MDL must be determined for each analyte/matrix prior to the analysis of any samples. The MDL is determined using seven replicates of reagent water, spiked with all the analytes of interest that have been carried through the entire analytical procedure. MDL's must be determined in accordance with 40 CFR Part 136 Appendix B requirements as detailed in TestAmerica QA SOP PT-QA-007. The spike level should be between the calculated MDL and 10X the MDL to be valid. The result of the MDL determination must be below the TestAmerica reporting limit.

12.1.2. Initial Demonstration Study- This requires the analysis of four QC check samples. The QC check sample is a well-characterized laboratory generated sample used to monitor method performance, which should contain all the analytes of interest. The results of the initial demonstration study must be acceptable before analysis of samples may begin. The results of the initial demonstration study may be used to extend a method for the analysis of other elements provided all acceptance criteria are met.

12.1.2.1. Four aliquots of the check sample (LCS) are prepared and analyzed using the procedures detailed in this SOP and the determinative SOPs.

12.1.2.2. Calculations and acceptance criteria for QC check samples are given in the determinative SOPs (PT-MT-001 and PT-MT-002).

12.2. Method performance is determined by the analysis of matrix spike and matrix spike duplicate samples as well as method blanks and laboratory control samples. In general, the matrix spike recovery should fall within +/- 25% and the matrix spike duplicates should compare within 20% RPD. Method blanks must meet the criteria specified in the determinative SOPs. The laboratory control samples should recover within 20% of the true value until in house control limits are established. Acceptance criteria are given in the determinative SOPs. **Refer to PT-QA-025 for specific DoD QC requirements.**

12.3. Training Qualification:

12.3.1. The group/team leader has the responsibility to ensure that this procedure is performed by an associate who has been properly trained in its use and has the required experience.

13. POLLUTION CONTROL

13.1. It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

13.2. This method does not contain any specific modifications that serve to minimize or prevent pollution.

14. WASTE MANAGEMENT

14.1. Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to CW-E-M-001. The following waste streams are produced when this method is carried out.

14.1.1. Contaminated disposable materials utilized for the analysis. These items are placed in trash containers which are emptied in the general trash dumpster located near the shipping/receiving dock.

15. REFERENCES

15.1. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition, Final Update III, December 1996. Method 3050B.

15.2. PT-MT-001, Inductively Coupled Plasma-Atomic Emission Spectroscopy, Spectrometric Method for Trace Element Analysis of Water and Wastes, Method 6010B and Method 200.7.

15.3. PT-MT-002, Analysis of Metals by Inductively Coupled Plasma/Mass Spectrometry (ICPMS) for Methods 200.8, 6020 & ILM05.2.

15.4. PT-QA-021, QC Program.

15.5. PT-QA-009, Rounding and Significant Figures.

15.6. PT-QA-007, Method Detection Limits.

15.7. PT-QA-024, Subsampling.

15.8. PT-QA-025, Implementation of the DoD QSM Version 3.

16. METHOD MODIFICATIONS

16.1. Modifications/Interpretations from reference method.

16.1.1. Chapter 1 of SW-846 states that the method blank should not contain any analyte of interest at or above the MDL. This SOP states that the method blank must not contain any analyte of interest at or above the reporting limit. Common lab contaminants, as defined in the determinative SOPs, are allowed up to two times the reporting limit in the blank following consultation with the client. **Refer to PT-QA-025 for specific DoD QC requirements.**

16.1.2. This SOP allows for aqueous samples to be weighed instead of measured volumetrically. This assumes the density of the sample is close to 1.0 g/mL. Samples with large amounts of sediment or suspended solids, sludges, non-aqueous liquids must be processed volumetrically. Weighing samples directly into the digestion vessel minimizes the potential for cross contamination, offers improved

accuracy over the use of graduated cylinders (comparable to volumetric flask accuracy), uses less glassware and is more efficient.

17. ATTACHMENTS

17.1. Figure 1 – Soil Sample Preparation

17.2. Figure 2 – Soil Sample Preparation (Continued)

17.3. Appendix A – Tables

17.3.1. Table I – Method 3050B Approved Analyte List

17.3.2. Table II – ICP & ICPMS Soil Matrix Spike and LCS Levels

17.3.3. Table III – Summary of Quality Control Requirements

17.4. Appendix B – Metals Preparation Bench Sheet

17.5. Appendix C – Contamination Control Guidelines

18. REVISION HISTORY

18.1. Revision 7, 09/12/07

18.1.1. Changed laboratory name to TestAmerica.

18.1.2. Changed the format of the SOP to correspond to the new Corporate SOP format.

18.1.3. Added the requirement to use a solid matrix for the method blanks and Laboratory Control Samples.

18.2. Revision 8, 04/28/09:

18.2.1. Updated the SOP reference numbers throughout the SOP; added Corporate text to the Scope, Safety, Quality Control, Pollution Control and Waste Management sections.

18.2.2. Added section 7.6, For ICPMS analysis, Optima reagents (ultra pure) are used for sample preparation.

Figure 1. Soil Sample Preparation (Section 10.10)

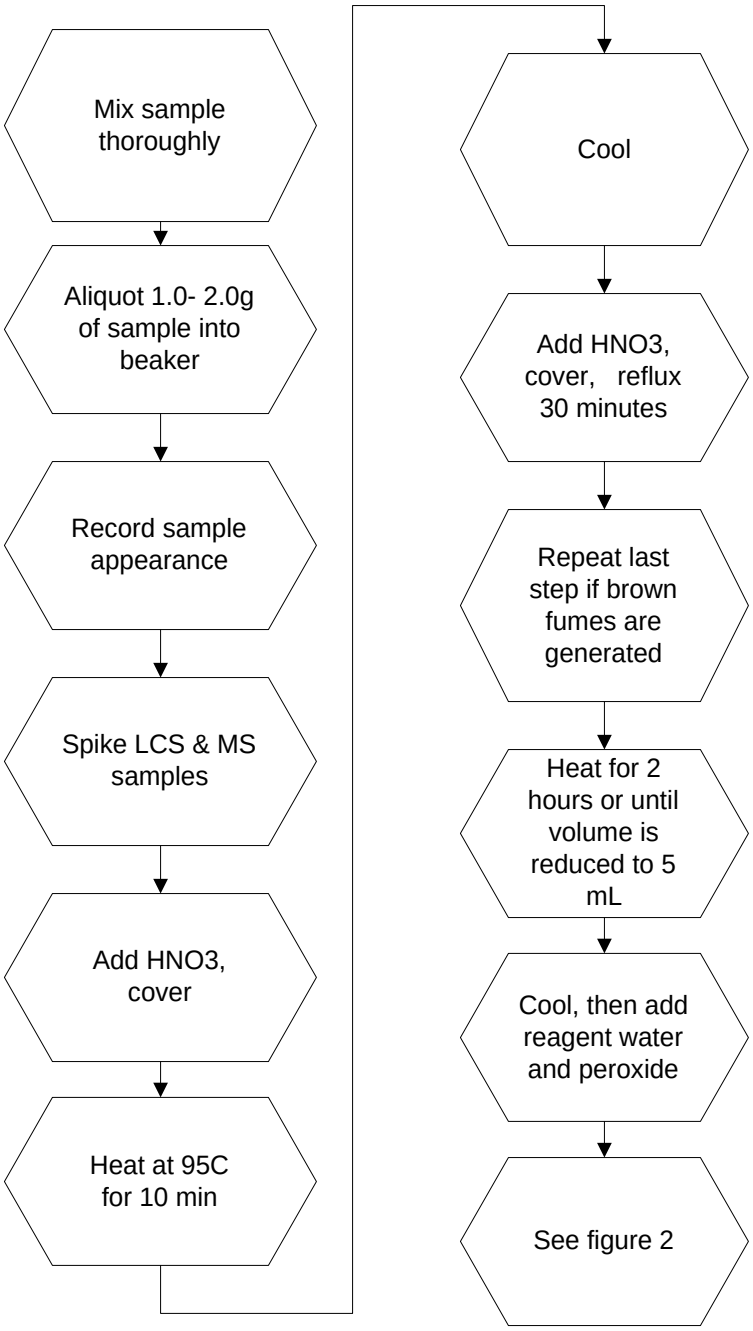
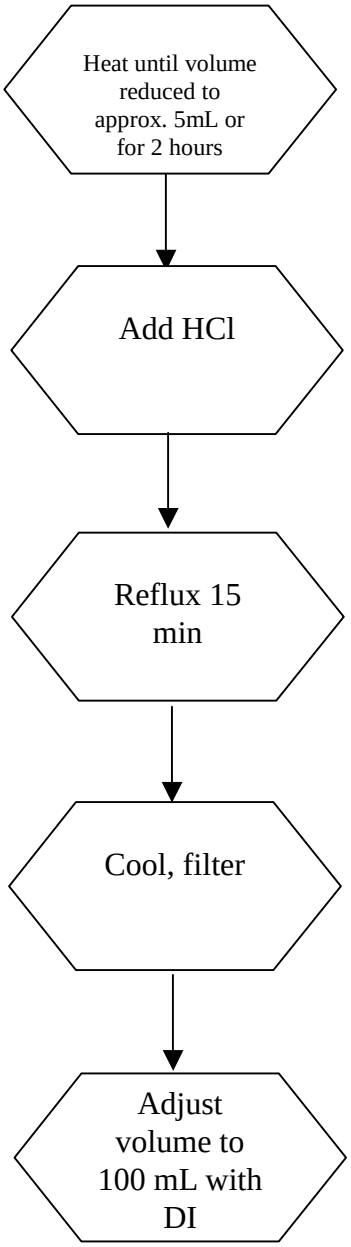


Figure 2. Soil Sample Preparation (continued)



APPENDIX A

TABLES

TABLE I. Method 3050B Approved Analyte List

ELEMENT	Symbol	CAS Number
Aluminum	Al	7429-90-5
Antimony	Sb	7440-36-0
Arsenic	As	7440-38-2
Barium	Ba	7440-39-3
Beryllium	Be	7440-41-7
Cadmium	Cd	7440-43-9
Calcium	Ca	7440-70-2
Chromium	Cr	7440-47-3
Cobalt	Co	7440-48-4
Copper	Cu	7440-50-8
Iron	Fe	7439-89-6
Lead	Pb	7439-92-1
Magnesium	Mg	7439-95-4
Manganese	Mn	7439-96-5
Molybdenum	Mo	7439-98-7
Nickel	Ni	7440-02-0
Potassium	K	7440-09-7
Selenium	Se	7782-49-2
Silver	Ag	7440-22-4
Sodium	Na	7440-23-5
Thallium	Tl	7440-28-0
Vanadium	V	7440-62-2
Zinc	Zn	7440-66-6

TABLE II. ICP & ICPMS Soil Matrix Spike and LCS Levels

ELEMENT	Working LCS/MS Standard (mg/L)	Aqueous LCS/MS Level* (ug/L)	Soil LCS/MS Level ** (mg/Kg)
Aluminum	200	2000	200
Antimony	50	500	50
Arsenic	200 (ICP); 4 (ICPMS)	2000 (ICP); 40 (ICPMS)	200 (ICP); 4 (ICPMS)
Barium	200	2000	200
Beryllium	5	50	5
Cadmium	5	50	5
Calcium	5000	50000	5000
Chromium	20	200	20
Cobalt	50	500	50
Copper	25	250	25
Iron	100	1000	100
Lead	50 (ICP); 2 (ICPMS)	500 (ICP); 20 (ICPMS)	50 (ICP); 2 (ICPMS)
Lithium	100	1000	100
Magnesium	5000	50000	5000
Manganese	50	500	50
Molybdenum	100	1000	100
Nickel	50	500	50
Phosphorous	1000	10000	1000
Potassium	5000	50000	5000
Selenium	200 (ICP); 1 (ICPMS)	2000 (ICP); 10 (ICPMS)	200 (ICP); 1 (ICPMS)
Silver	5	50	5
Sodium	5000	50000	5000
Strontium	100	1000	100
Thallium	200 (ICP); 5 (ICPMS)	2000 (ICP); 50 (ICPMS)	200 (ICP); 5 (ICPMS)
Vanadium	50	500	50
Zinc	50	500	50
Boron	100	1000	100
Silica	1000	10000	1000
Tin	200	2000	200
Titanium	100	1000	100

* Levels shown indicate the spike concentration in the final digestate of the aqueous LCS or matrix spike based on the addition of 1.0 mL working spike (7.3) to 100 mL of sample.

** Final soil spike concentration based on the addition of 1.0 mL working spike (7.3) to 1.0 g of sample (or 1.0 g of Teflon chips for the LCS)/100 mL final volume (assumes 100% solids).

TABLE III. Summary Of Quality Control Requirements

QC PARAMETER	FREQUENCY	ACCEPTANCE CRITERIA[1]	CORRECTIVE ACTION
Method Blank	One per sample preparation batch of up to 20 samples.	Refer to determinative SOPs: PT-MT-001 & PT-MT-002	Redigest and reanalyze samples.
Laboratory Control Sample (LCS)	One per sample preparation batch of up to 20 samples.	Refer to determinative SOPs: PT-MT-001 & PT-MT-002	Redigest and reanalyze all samples associated with the LCS.
Matrix Spike	One per sample preparation batch of up to 20 samples.	Refer to determinative SOPs: PT-MT-001 & PT-MT-002	Reprep not required unless preparation error suspected.
Matrix Spike Duplicate	See Matrix Spike	Refer to determinative SOPs: PT-MT-001 & PT-MT-002	See Corrective Action for Matrix Spike.

[1] For specific DoD requirements, refer to PT-QA-025.

APPENDIX B - EXAMPLE METALS PREPARATION BENCHSHEET

TestAmerica Pittsburgh ***Metals Preparation Log*** Batch No. 7232156 Page 1 of 1

V1.0.5 - Last Revised 12/30/2005

Method 3050B	Matrix Solid	Start Time 0945	SDG -
Analyst example	Date 082007	Lot Numbers	
MS STLPA-MS-A, STLPA-MS-B, STLPA-MS-C		C7H030157	
Lab Lot # C7H030157			
Analyst Sign:			

Sample ID	Initial Wt/Vol g/mL	Final Vol mL	Comments
J3500			
J3501			
J3503			
J3505			
J3507			
J3509			
J350A			
J350C			
J350F			
J350H			
J350K			
J350N			
J350R			
J350W			
J351D			
J35X4			
J35X6			
J35X8			
J35X9			
J35XM			
J35XM S			
J35XM D			
J47LA B			
J47LA C			

SAMPLE CODING: B-Blank C-Check L-Check Duplicate S-Matrix Spike D-Matrix Spike Duplicate X-Sample Duplicate

NOTE: Samples marked with an asterisk (*) required filtration after digestion and prior to analysis
Samples marked with a plus sign (+) required additional Conc.HNO3 in digestion process, brown fumes were observed

Reagents: 10mL 1:1 HNO3, Met 4475-07, Standards Log
5 mL Conc.HNO3, 6623E20033, Mallinckrodt
10mL 30% H2O2, 42H15, Mallinckrodt
10mL Conc.HCl, 5587C47A01, Mallinckrodt

Hot Plate/Block Temp	Correction Factor

Minimum digestion times have been met (analyst initials):

Digestate(s)	Date	Time	(Received)	Analyst	Location	Date	Time	(Relinquished)	Analyst	Location

	Lot Number	Manufacturer	
Initial Digestion Vessel:	A603LP021	Environmental Express	Pipet #: 30450H
Final Digestion Vessel	070603	Corning	Balance #: C94817
Filter Paper:	G1822665	Whatman	Printed on: 24-Aug-07 1:38:47 PM

Reviewed By: Date: MET-86-7232156

APPENDIX C

CONTAMINATION CONTROL GUIDELINES

APPENDIX C. CONTAMINATION CONTROL GUIDELINES

The following procedures are strongly recommended to prevent contamination:

All work areas used to prepare standards and spikes should be cleaned before and after each use.

All glassware should be washed with detergent and tap water and rinsed with 1:1 nitric acid followed by deionized water.

Proper laboratory housekeeping is essential in the reduction of contamination in the metals laboratory. All work areas must be kept scrupulously clean.

Powdered or Latex Gloves must not be used in the metals laboratory since the powder contains silica and zinc, as well as other metallic analytes. Only vinyl or nitrile gloves should be used in the metals laboratory.

Glassware should be periodically checked for cracks and etches and discarded if found. Etched glassware can cause cross contamination of any metallic analytes.

Autosampler trays should be covered to reduce the possibility of contamination. Trace levels of elements being analyzed in the samples can be easily contaminated by dust particles in the laboratory.

The following are helpful hints in the identification of the source of contaminants:

Reagents or standards can contain contaminants or be contaminated with the improper use of a pipette.

Improper cleaning of glassware can cause contamination.

Separate glassware if an unusually high sample is analyzed and soak with sulfuric acid prior to routine cleaning.

G-3 SOP NO. PT-QA-027, REV. 14, EFFECTIVE DATE: 05/23/2010, SAMPLE RECEIPT AND CHAIN OF CUSTODY

Title: Sample Receipt and Chain of Custody

Approvals (Signature/Date):

Anthony Lee	05/20/10	Steve Jackson	05/21/10
Technical Manager	Date	Health & Safety Manager / Coordinator	Date
Nasreen DeRubeis	05/20/10	Larry Matko	05/23/10
Quality Assurance Manager	Date	Laboratory Director	Date

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1. PURPOSE

1.1. This SOP details procedures for receiving samples into the laboratory and describes laboratory custody practices. For DoD QSM 4.1 requirements refer to SOP PT-QA-029.

2. SCOPE

2.1. This protocol described here complies with accepted laboratory custody procedures and regulatory requirements.

3. SAFETY

3.1. Employees must abide by the policies and procedures in the Corporate Safety Manual, Radiation Safety Manual, the Waste Management SOP, and this document.

3.2. During the course of performing this procedure it may be necessary to go into laboratory areas to consult with appropriate staff members, therefore employees performing this procedure must be familiar with the Laboratory Health & Safety Plan, and take appropriate precautions and wear appropriate attire and safety glasses.

3.3. Sample bottles containing samples suspected to contain high levels of cyanide or sulfide shall be marked with a sticker marked “High Cyanide” or “High Sulfide”. These sample bottles shall only be opened in a fume hood.

3.4. The following is a list of the materials used in this method, which have a serious or significant hazard rating. NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table. A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Hydrochloric Acid	Corrosive Poison	5 ppm-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Nitric Acid	Corrosive Oxidizer Poison	2 ppm-TWA 4 ppm-STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Sodium Hydroxide	Corrosive Poison	2 ppm, 5 mg/m^3	This material will cause burns if comes into contact with the skin or eyes. Inhalation of Sodium Hydroxide dust will cause irritation of the nasal and respiratory system.
Sulfuric Acid	Corrosive Oxidizer Dehydrator Poison Carcinogen	1 Mg/M3-TWA	Inhalation produces damaging effects on the mucous membranes and upper respiratory tract. Symptoms may include irritation of the nose and throat, and labored breathing. Symptoms of redness, pain, and severe burn can occur. Contact can cause blurred vision, redness, pain and severe tissue burns. Can cause blindness.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

3.5. Eye protection that protects against splash, laboratory coat, and appropriate latex gloves must be worn while samples, standards, solvents, and reagents are being handled. Cut resistant gloves must be worn doing any other task that presents a strong possibility of getting cut. Disposable gloves that have become contaminated will be removed and discarded; other gloves will be cleaned immediately.

3.6. Exposure to chemicals must be maintained **as low as reasonably achievable**, therefore, unless they are known to be non-hazardous, all samples must be opened, transferred, and prepared in a fume hood or under other means of mechanical ventilation. Solvent and waste containers will be kept closed unless transfers are being made. If waste is generated during operations, it will be segregated and disposed of according to the facility hazardous wastes procedures as appropriate. The Environmental Health and Safety Coordinator will be contacted if related information is required.

3.7. All work must be stopped in the event of a known or potential compromise to the health and safety of a TestAmerica associate. The situation must be reported **immediately** to a laboratory supervisor or the EH&S coordinator.

3.8. It is TestAmerica policy that no employee should lift over 50 pounds without help. The weight of the coolers must be evaluated, and if over 50 pounds, the employee must get assistance before lifting.

4. DEFINITIONS

4.1. Custody: The definition of custody may be found in Section 6.1.

4.2. Internal Chain of Custody: Records generated by various departments internal to the laboratory and which document custody of samples or sample preparations for transfers within the laboratory.

4.3. LIMS: Laboratory Information Management System. The computer system used to track information about samples after being received by the laboratory.

5. PROCEDURE

5.1. Samples will be received at the laboratory facility during normal working hours (8 a.m. to 5 p.m.). If sample receipt is anticipated outside normal working hours, the project manager must notify and coordinate sample receipt with the sample custodian, who in turn will designate personnel to receive the samples. The sample custodian examines the shipping container (cooler, box, or other shipping container) and completes the header information on a Cooler Receipt Form (see “ATTACHMENTS,” Section 8.0). Information recorded at this time will include: client name and project, quote number, TestAmerica lot number, the date the shipping container was received and temperature checked, and the date that the shipping container was unpacked. Samples received after normal working hours are left in their coolers and placed in a cold room. The person receiving the samples must record the date and time received, the presence or absence of ice and custody seals, the temperature of samples, presence and type of packing material, and initials. **NOTE:** For samples requiring **DoD QSM protocol**, all samples received after normal business hours will be directly placed into the cold room. The next day

the sample receiving personnel will check the cooler temperature, sample preservation, etc. and record all pertinent information on the Cooler Receipt Form, Figure 1.

5.2. For DoD Work, the Project Manager will notify the client that samples are received after hours, the lab will not accept the samples until the following day. This is included in the sample acceptance policy.

5.3. The temperature of the cooler may be taken either by using an enclosed temperature blank, if supplied, or by inserting a thermometer between samples in the cooler (the thermometer must not come in contact with the ice). If the temperature of the cooler is not 0 - 6°C, this must be noted on a Condition Upon Receipt Variance Report (see "ATTACHMENTS," Section 8.0). The temperature will be recorded on the Cooler Receipt Form. The reading that is recorded will take into consideration the relevant thermometer correction factor.

5.4. Samples shall be deemed acceptable if arrival temperature is just above freezing and less than or equal to 6.0 °C (0-6 °C). Samples that are hand-delivered immediately after collection may not be at the required temperatures; however, if there is evidence that the chilling process has begun, such as the arrival on ice, the samples shall be considered acceptable. This will be documented on the Cooler Receipt Form.

5.5. If the samples were shipped in ice and solid ice is still present and in direct contact with samples, report the samples as "received on ice." Direct contact means samples must be surrounded by ice cubes or crushed ice. Ice present in a plastic bottle or other container does not constitute direct contact.

5.6. If the cooler temperature is >6°C, the sample custodian will document which sample containers were inside each specific cooler where the temperature was >6°C. This will be documented on the Condition Upon Receipt Variance Report and the project manager will be notified.

5.7. Other information to be noted at this time is: presence/absence of custody seal(s) on the shipping container(s), and their condition.

5.8. If samples are received without a COC, TestAmerica will provide a generic COC form to be completed by the client when the samples are brought to the laboratory. The client is always provided with a copy of the completed COC form for their records.

5.9. The sample custodian opens the shipping container and removes the entire contents, assuring that all enclosed samples and documents are retained. After reviewing the entire contents of the shipping container and carefully checking sample integrity (intact, broken, leaking, etc.), labels, and shipment completeness, the remaining information is placed on the Cooler Receipt Form (see "ATTACHMENTS," Section 8.0).

5.10. The sample custodian must compare the field chain(s) of custody, the sample labels, and airbills or bills of lading. If there are any inconsistencies between the forms, the discrepancies are documented on the Condition Upon Receipt Variance Report (see "ATTACHMENTS," Section 8.0). If samples are received out of holding time, a Nonconformance Memo must be completed and the project manager notified. The project manager then notifies the client and a decision is made concerning whether to proceed with analysis.

5.11. The sample custodian measures the pH of preserved aqueous samples (with the exception of samples that have been submitted for the testing of volatile organic compounds). This is done by placing a few drops of the sample on *narrow*-range pH paper using a clean disposable Pasteur pipette. This measurement determines whether the sample has been properly preserved. (For Oil & Grease pour in cap and then pipette.) The used pipette is then discarded. **Note:** The pH of samples to be analyzed for VOAs will be measured by the volatiles analytical group after removing an aliquot for analysis.

5.12. Residual chlorine will be checked when sample receiving is notified by client/PM that samples contain residual chlorine. Semivolatile organic samples containing residual chlorine should be preserved with sodium thiosulfate. (Add 3-mL 10% sodium thiosulfate solution per gallon (or 0.008%). (Addition of sodium thiosulfate solution to sample container may be performed in the laboratory prior to field use.) For all potentially chlorinated samples the sample custodian checks for residual chlorine by testing the sample with KI-starch paper. If residual chlorine is present, the sample custodian must indicate so on the <u>sample condition upon receipt form and issue a NCM.</u> Also, the client, the PM, and the group responsible for analysis of the sample must be notified. Steps for removal of residual chlorine are included in the SOP for the method the sample is to be analyzed by.

5.13. If all samples recorded on the field chain(s) of custody were received by the laboratory and there are no problems observed with the sample shipment, the sample custodian signs the field chain(s) of custody in the "Received for Laboratory by:" box on the document. Problems such as broken bottles and pH or temperature outside criteria are noted on the Condition Upon Receipt Variance Report (see "ATTACHMENTS," Section 8.0). Where approved by the project manager, unpreserved samples will be preserved by the sample custodian. This action will be documented on the Condition Upon Receipt Variance Report (CURV) and where required by the PM in a non-conformance memo. Where lab reagents are used to make a preservative adjustment, record the reagent, lot number and amount of reagent added in the comment field on the CURV report. Whenever the laboratory preserves samples for metals analysis, the samples must be held for 24 hours after preservation before analysis may be performed.

5.14. The project manager's Quote form (the LIMS description of the sampling program) is reviewed to assure that the field chain(s) of custody is in agreement. If the documents do not agree, the project manager is notified of the discrepancy. Samples are not logged in until all discrepancies are resolved.

5.15. If it has been necessary to complete a Condition Upon Receipt Variance Report, this will be forwarded to the project manager for review. The project manager will determine the action required and contact the client as necessary or otherwise agreed.

5.16. Any deviations from the checks described above that question the suitability of the sample for analysis, or incomplete documentation as to the tests required will be resolved by consultation with the client. If the sample acceptance criteria (Appendix 2) are not met, the laboratory shall either:

5.17. Retain all correspondence and/or records of communications with the client regarding the disposition of rejected samples (The project manager may be able to make decisions on samples with prior knowledge from the client, but documentation of acceptable scenarios must be provided and acknowledged by the client, and records of these decisions must be documented), or

5.18. Fully document any decision to proceed with sample analysis that does not meet sample acceptance criteria.

5.19. Note: North Carolina requires that they be notified when samples processed do not meet sample acceptance criteria.

5.20. If the sample does not meet the sample receipt acceptance criteria listed in the sample acceptance policy (Appendix 2), the laboratory shall either:

5.20.1. Retain correspondence and/or records of conversations concerning the final disposition of rejected samples; or

5.20.2. Fully document any decision to proceed with the analysis of samples not meeting acceptance criteria.

5.20.3. The condition of these samples shall, at a minimum, be noted on the chain of custody or laboratory Condition Upon Receipt Variance Report (Figure2).

5.20.4. If the conditions listed on the Acceptance Policy are not satisfactory and when lacking direction or agreement with the client, the sample will be rejected by the laboratory. The client is notified of sample acceptance policy in the TestAmerica's terms and conditions, section 1.3.

5.21. Once the project manager has resolved any discrepancies between the field chain(s) of custody and Quote and has taken the appropriate action for any Condition Upon Receipt Variance Report that has been generated, the samples are logged in using the Laboratory Information Management System (LIMS). A unique laboratory identification number is

assigned to each sample (i.e., C6B060001-001: C denotes Pittsburgh; 6 references the year [2006]; B signifies the month [February]; 06 means the sixth day of the month; 0001 is the first lot logged in; and -001 is the first sample of that lot).

5.22. The Sample Control person will enter each sample into the laboratory computer (QuantIMS), where a unique lot number is assigned to each project received, and sequential sample numbers are designated for each client identification within the lot.

5.23. **Lot Numbers**: The lot number is nine characters in length and is based on the date of receipt. Lot number A5J010021 is described as follows:

A - TestAmerica location where the samples were received.
(A = North Canton, B = Tampa, C = Pittsburgh, etc.)
5 - Last digit of the year (i.e. 2005).
J - Month (i.e. A = January, B = February, J = October, etc.)
01 - The next 2 numeric characters identify the day of the month, in this case, the first day of the month.
0021 - The next 4 numeric characters are the sequential assignment of numbers specific to each lot received. Each day the first lot logged in receives the number "0001", the second lot receives the number "0002", etc..

For example:
If four bottles were submitted under Client ID numbers AB100-AB103 and the laboratory identification number generated by the computer is A2K100001, then the assigned laboratory number recorded on the Sample Log-In Sheet would be as follows:

Client ID Sample Number	Assigned Laboratory Number
AB 100	A2K100001-001
AB 101	A2K100001-002
AB 102	A2K100001-003
AB 103	A2K100001-004

5.24. **Sample Numbers**: The samples in each lot are assigned a sample number that is attached to the lot number and are reset at each new lot. For example: the first and second samples in the lot above are labeled A5J010121-001 and A5J010121-002.

5.25. **Sample Suffixes**: Each sample also has a 1-character field (which is not a required field for all samples) called the suffix, which identifies the sample as specified below.

Client Sample	No. Suffix
Method Blank	B
Laboratory Control Sample	C
Laboratory Control Sample Duplicate	L

Matrix Spike	S
Matrix Spike Duplicate	D
Sample Duplicate	X
Serial Dilution	P
Sample Confirmation	Y
Post Digestion Spike	Z
Re-analysis	I

Example: A5J010121-001X is a sample duplicate for sample A5J010121-001.

5.26. **Work Order Numbers**: Each test requested by the client for an individual sample receives an individual 8 digit work order number assigned by QuantIMS. Work order number A5WE1-2-1C is described as follows:

A5WE1 - In addition to the three digit sample (i.e. - 001 and - 002), the first 5 characters of the work order number also identifies each unique sample. This identification is generated in QuantIMS using a sequential logic.

2 - The “modifier” indicates the type of run. In this case this is the second time the sample had to be run. If it needs reprepared and run again, the number would indicate a “3”. The original analysis work order number assigns “1” to the modifier position.

1C - The “suffix” is the identification of the specific test for that sample. The suffix in this case is not always sequential, but is unique to the test to be performed on the sample.

5.27. Worksheets and sample container labels are printed. The sample custodian will assure that each sample container is labeled with a unique laboratory identification number that cannot be accidentally removed in the laboratory.

5.28. The label will contain the following information:

5.28.1. The laboratory information management system unique Lab ID number.

5.28.2. Sampling date.

5.28.3. Client ID.

5.28.4. Storage location.

5.28.5. Client code.

5.28.6. Number and size of bottles received.

5.29. The sample custodian will put the samples into secure storage areas. Samples for tests other than volatile organics are placed on numbered shelves inside secure walk-in refrigerators. Samples received for volatile organics are placed in separate secure refrigerators that have been designated for volatile sample storage only and are located within the volatiles lab area. From the time of login and refrigerator assignment by the sample receiving personnel, the volatiles personnel become the designated sample custodians for the volatile samples. As custodians they must document custody within their workgroup. In both cases temperature is maintained at 0 – 6 °C. The storage location for all received samples is entered in the laboratory information management system.

5.30. The sample custodian files the shipping documents, field chain(s) of custody, quote, related correspondence, cooler receipt form, analytical summary, along with preliminary invoice. The summary and supporting documentation are reviewed for accuracy by the sample custodian and placed in a project folder before being submitted to Project Management. Laboratory information management system worksheets are generated by the sample custodian and distributed to the analytical group leaders.

5.31. Internal Chain of Custody

5.31.1. The sample custodian transfers the custody of requested raw samples to the analytical staff using a COC/Sample Request form (Figure 3). If the analyst needs the sample right away, this form is used for transfer of raw samples prior to login. After samples are logged into LIMS the internal chain of custody form is printed from LIMS (Figure 4) (see “ATTACHMENTS,” Section 8.0), which lists the laboratory identification number of

the samples being transferred. The sample custodian checks that the sample labels and the COC/Sample Request are in agreement, dates, records the time of transfer (using military time), and signs the COC/Sample Request as having relinquished the samples. The analyst will then sign the COC/Sample Request as having received the listed samples.

5.31.2. The COC/Sample Request form must accompany the raw samples while they are being analyzed. The analyst may transfer the samples to a second analyst using a second COC/Sample Request form that has been generated listing the samples and purpose of the transfer. All samples listed on the initial COC/Sample Request must be present and listed on the second COC/Sample Request. Transferring less than all of the samples listed on the initial COC/Sample Request is prohibited. If only several of the samples listed on the initial form are required, then all samples on the initial form will be returned to the laboratory sample custodian who will relinquish only those samples required, as described in the above section. The relinquishing analyst assures that all samples are present and correctly identified on the second COC/Sample Request before signing, dating, and recording the time of transfer (military time) on the second COC/Sample Request form. The second analyst signs the second COC/Sample Request form to accept the samples. The original COC/Sample Request from the laboratory sample custodian must be relinquished to the second analyst and will remain with the samples.

5.31.3. Custody of raw samples may be maintained if the analyst receiving custody as described above places the samples in a secure storage area accessible only to authorized personnel for the purpose of analysis. Storage conditions must be such that the integrity of the sample will be maintained for the required analyses.

5.31.4. Raw samples (whether the containers have sample volume remaining or are empty) shall be returned to the sample custodian with the original COC/Sample Request and any additional COC/Sample Request forms that were required to document subsequent transfers. The COC/Sample Request must be reconciled with the samples being returned before the sample custodian accepts receipt of the samples. (The sample custodian will not accept the samples unless <u>all</u> of the containers listed on the COC/Sample Request form are present.) The analyst then signs as relinquishing the samples and sample custodian accepts custody of returned raw samples by signing, dating, and recording the time of receipt (using military time) on the COC/Sample Request form that documents the most recent transfer of the samples.

5.31.5. The sample custodian will record which samples have been completely expended in analyses by making an entry on the COC/Sample Request form.

5.31.6. Custody information for extracts, distillates, and digestates prepared from raw samples shall be recorded on the sample preparation benchsheet used to document the preparation. (A COC/Sample Request will not be used for sample preparations.) The

individual who has conducted the preparation is responsible for completing custody transfer information. The relinquishing analyst assures that all sample preparations are present and correctly identified before signing, dating, and recording the time of transfer (military time) on the benchsheet. Prepared samples are transferred to and kept in a secure area accessible only to authorized personnel for the purpose of analysis. If further custody transfers are necessary, these are recorded in a similar manner on the same benchsheet. The benchsheet with completed custody information must accompany the prepared samples whenever custody transfers are necessary.

5.31.7. The approved sample custodians, other than Sample Receiving personnel, are listed in the Work Instruction for Approved Sample Custodian Designees, PT-QA-W-001. If there is no sample custodian in the building during weekends, early morning or late evening hours to relinquish or receive samples and sign the Internal COC form, the analyst will have the authority to sign and date the COC and put on the paperwork no custodian available.

5.32. If after analysis, a non-volatile sample is found to contain high levels of contamination for a particular analyte or group of analytes the sample receiving personnel will place the raw sample into a paint can and seal it to prevent cross-contamination of other samples. The sealed paint can will be stored on a shelf in the cold room. The appropriate laboratory department will facilitate this process by notifying sample receiving personnel which raw samples contain high levels of non-volatile contamination and should be segregated.

5.33. For DoD Work or other projects as applicable: Upon notification from client of high concentration samples, prior to analyses, the project manager will notify the lab. Sample receiving will segregate the samples by placing the samples into a closed secondary container. The container will be placed in the cold room.

5.34. Samples will be properly disposed of by the sample custodian thirty days after the final report unless the laboratory has been otherwise notified in writing. All samples in a single TestAmerica laboratory lot will be disposed of at the same time, and a record documenting the lot disposal will be kept in a logbook maintained by the sample custodian.

5.35. The temperature of each walk-in cooler is monitored daily using an electronic Temperature Guard monitoring system. Refer to SOP PT-QA-008.

5.36. **Cooler Handling Procedure**: All coolers should be rinsed out after receipt and unloading. If a cooler smells or is visibly soiled it should be soaped and warm water rinsed. If a cooler cannot be cleaned up or remains odorous after cleaning, it should be discarded.

5.37. Any deviation from the conditions and handling described in the “PROCEDURES” Section will be documented using a Nonconformance Memo (NCM) with distribution to the Project Manager

and Quality Assurance Department. Corrective action must be fully explained in the Nonconformance.

5.38. Holding Times are noted in the TestAmerica Pittsburgh Laboratory Quality Assurance Manual, current version. Please note, client specific or project specific holding times would supersede the holding times listed in the QA manual.

6. RESPONSIBILITIES

6.1. It is the responsibility of the Sample Receiving Department to receive samples in full compliance with this SOP. Sample receiving duties of the sample custodian (and other designated individuals) are described in the "PROCEDURE" Section. Following this SOP assures that all samples are properly received and will always be under the custody of a person or stored in a secure area while at the laboratory.

6.2. For the purpose of this SOP, a sample is under custody if:

6.3. It is in your possession, or

6.4. It is in your view after being in your possession, or

6.5. It was in your possession and you locked it up, or

6.6. It is in a designated secure area. (Secure areas shall be accessible only to authorized personnel.)

6.7. It is the responsibility of each analyst or any TestAmerica Pittsburgh staff member to maintain sample custody for all entrusted samples as described herein in full compliance with this SOP.

6.8. It is the responsibility of the laboratory director, QA manager, and supervisors to assure that operations are conducted in full compliance with this SOP. It is also their responsibility to supply training, materials, and equipment so that the laboratory staff can comply with all SOP requirements.

7. REFERENCES / CROSS-REFERENCES

7.1. TestAmerica Pittsburgh Laboratory Quality Assurance Manual, PT-LQAM, current version.

7.2. PT-QA-W-001, Work Instruction for Approved Sample Custodian Designees, current version.

7.3. SOP# PT-QA-008, Thermometer Calibration and Temperature Monitoring.

7.4. SOP # PT-QA-025, DoD QSM 3.0 Requirements.

7.5. SOP # PT-QA-029, DoD QSM 4.1 Requirements.

8. ATTACHMENTS

8.1. Appendix 1 - Disaster Recovery Plan

8.2. Appendix 2 - Sample Acceptance Policy

8.3. Figure 1 – Example Cooler Receipt Form, Page 1 and Page 2

8.4. Figure 2 – Example Condition Upon Receipt Variance Report

8.5. Figure 3 – Example COC/Sample Request

9. REVISION HISTORY

9.1. Revision 9, 03/13/2008:

9.1.1. Changed SOP to match new corporate SOP format.

9.1.2. Replaced the Sample Acceptance Policy with the policy in the LQAM.

9.1.3. Updated several parts of the Procedure Section.

9.1.4. Updated the Cooler Receipt Form, the Condition Upon Receipt Variance Report and the COC/Sample Request form.

9.1.5. All changes have been highlighted in yellow.

9.2. Revision 10, 08/11/08:

9.2.1. SOP renumbered as PT-QA-027.

9.2.2. Section 3.5 added latex gloves.

9.2.3. Section 5.5, removed requirement for pouring samples into cups for DoD, this is not a DoD requirement.

9.2.4. Section 5.19.7 added: The approved sample custodians are listed in the Work Instruction for Approved Sample Custodian Designees, PT-QA-W-001. If there is no sample custodian in the building during weekends or late evening hours to relinquish or receive samples and sign the Internal COC form, the analyst will have the authority to sign and date the COC and put on the paperwork no custodian available.

9.3. Revision 11, 09/09/2009

9.3.1. Updated section 5.21 to reflect the use of the electronic Temperature Guard monitoring system. Added references for SOPs PT-QA-008 and PT-QA-029.

9.3.2. Updated section 5.7 last sentence: Whenever the laboratory preserves samples for metals analysis, the samples must be held for 24 hours after preservation before analysis may be performed.

9.3.3. Temperature ranges updated from 4 ± °C ± 2 °C to 0 – 6 °C.

9.4. Revision 12, 10/07/09

9.4.1. Added Section 5.10.4: If the sample does not meet the sample receipt acceptance criteria listed in the sample acceptance policy (Appendix 2), the laboratory shall either:

9.4.2. Retain correspondence and/or records of conversations concerning the final disposition of rejected samples; or

9.4.3. Fully document any decision to proceed with the analysis of samples not meeting acceptance criteria.

9.4.4. The condition of these samples shall, at a minimum, be noted on the chain of custody or laboratory Condition Upon Receipt Variance Report (Figure2).

9.5. Revision 13, 1/18/2010

9.5.1. Added text to section 5.1 concerning the after hours procedure when receiving samples for analysis by DoD protocol.

9.5.2. Added to section 5.10.4.4: If the conditions listed on the Acceptance Policy are not satisfactory and when lacking direction or agreement with the client, the sample will be rejected by the laboratory.

9.5.3. Added to section 5.11: Residual chlorine will be checked when the sample receiving is notified by client/PM that samples contain residual chlorine. Semivolatile organic samples containing residual chlorine should be preserved with sodium thiosulfate. (Add 3-mL 10% sodium thiosulfate solution per gallon (or 0.008%). (Addition of sodium thiosulfate solution to sample container may be performed in the laboratory prior to field use.)

9.5.4. Added section 5.17 which details the procedure for notification of sample receiving when raw samples have high concentrations for a particular analyte and how sample receiving is to segregate this sample from the other samples in the cold room.

9.5.5. Added section 10 to Sample Acceptance policy, Appendix 2: If the conditions listed on the Acceptance Policy are not satisfactory and when lacking direction or agreement with the client, the sample will be rejected by the laboratory.

9.5.6. Added column for residual chlorine check in Cooler Receipt form, Figure 1.

9.6. Revision 14:

9.6.1. Added to section 5.2: For DoD work, the Project Manager will notify the client that samples are received after hours, the lab will not accept the samples until the following day. This is included in the sample acceptance policy.

9.6.2. Added to section 5.20.4: The client is notified of sample acceptance policy in the TestAmerica's terms and conditions, section 1.3.

9.6.3. Added to section 5.33: For DoD work or other projects as applicable, upon notification from client of high concentration samples, prior to analyses, the project manager will notify the lab. Sample receiving will segregate the samples by placing the samples into a closed secondary container. The container will be placed in the cold room.

9.6.4. Added to Appendix 2: For DoD work, the Project Manager will notify the client that samples are received after hours they lab will not accept the samples until the following day.

Appendix 1 - Disaster Recovery Plan

1. When sample location transfers are required due to cooler malfunction or power failure, all transfers must be properly documented as described in the above SOP sections. Sample security must be properly maintained with access to samples limited, controlled, and documented. Temperature of the relocated samples will be documented and controlled to 0 – 6 °C.

2. In the event of a power failure limiting available cold storage capacity to less than that required to adequately store all samples at the necessary temperature, backup refrigeration trucks will be called to give assistance on site to store samples and maintain proper temperature at 0 -6°C. Trucking companies that could be called are:

   Thermo King Transport Refrigeration
   Ryder Trucks
   Budget Trucks

3. If refrigeration trucks are not available during a power failure, dry ice will be used to maintain proper temperature.

4. When cooling units malfunction, a local heating, ventilation, and air conditioning contractor will immediately be called to correct the problem. Sample Receiving will coordinate all service calls using a 24-hour servicing company to minimize impact on sample integrity.

Appendix 2 - TestAmerica Pittsburgh Sample Acceptance Policy

All incoming work will be evaluated against the criteria listed below. Where applicable, data from any samples that do not meet the criteria listed below will be noted on the laboratory report defining the nature and substance of the variation. In addition the client will be notified either by telephone, fax or e-mail ASAP after the receipt of the samples.

1) Samples must arrive with labels intact with a Chain of Custody filled out completely. The following information must be recorded.
   - ***Client name, address, phone number and fax number (if available)***
   - ***Project name and/or number***
   - ***Unique sample identification***
   - ***Date, time and location of sampling***
   - ***The collectors name***
   - ***The matrix description***
   - ***The container description***
   - ***The total number of each type of container***
   - ***Preservatives used***
   - ***Analysis requested***
   - ***Requested turnaround time (TAT)***
   - ***Any special instructions***
   - ***Purchase Order number or billing information (e.g. quote number) if available***
   - ***The date and time that each person received or relinquished the sample(s), including their signed name.***
   - **Information must be legible**

2) Samples must be properly labeled.
   - Use durable labels (labels provided by TestAmerica are preferred)
   - Include a unique identification number
   - Include sampling date and time & sampler ID
   - Include preservative used.
   - Use indelible ink
   - **Information must be legible**

3) Proper sample containers with adequate volume for the analysis and necessary QC are required for each analysis requested.

4) Samples must be preserved according to the requirements of the requested analytical method. Most analytical methods require chilling samples to 4° C (other than water samples for metals analysis). For these methods, the criteria are met if the samples are chilled to below 6° C and above freezing (0°C). For methods with other temperature criteria (e.g. some bacteriological methods require $\leq$ 10 ^{o}C), the samples must arrive within $\pm$ 2° C of the required temperature or within the method specified range. **Note:** Samples that are hand delivered to the laboratory immediately after collection may not have had time to cool sufficiently. In this case the samples will be considered acceptable as long as there is evidence that the chilling process has begun (arrival on ice).

- Chemical preservation (pH) will be verified prior to analysis and the project manager will be notified immediately if there is a discrepancy. If analyses will still be performed, all affected results will be flagged to indicate improper preservation.

5) *Matrix Spikes* are required for your project, separate sample volumes must be available for the requested analyses.

6) For Volatile Organic analyses: Efforts should be made to minimize any air bubbles in aqueous volatile samples. Air bubbles also the escape of volatile organics. This is especially important because air bubbles tend to form in iced samples. Volatile vials containing air bubbles larger than a pea will be treated as non-conformances.

7) All samples submitted for Volatile Organic analyses must have a Trip Blank submitted at the same time. TestAmerica will supply a blank with the bottle order.

8) Sample Holding Times

- TestAmerica will make every effort to analyze samples within the regulatory holding time. Samples must be received in the laboratory with enough time to perform the sample analysis. Except for short holding time samples (< 48hr HT) sample must be received with at least 48 hrs (working days) remaining on the holding time for us to ensure analysis.

- Analyses that are designated as "field" analyses (Odor, pH, Dissolved Oxygen, Disinfectant Residual; a.k.a. Residual Chlorine, and Redox Potential) should be analyzed ASAP by the field sampler prior to delivering to the lab (within 15 minutes). However, if the analyses are to be performed in the laboratory, TestAmerica will make every effort to analyze the samples within 24 hours from receipt of the samples in the testing laboratory. Samples for "field" analyses received after 4:00 pm on Friday or on the weekend will be analyzed no later than the next business day after receipt (Monday unless a holiday). Samples will remain refrigerated and sealed until the time of analysis. Samples analyzed in the laboratory will be qualified on the final report to indicate holding time exceedance.

9) The project manager will be notified if any sample is received in damaged condition. TestAmerica will request that a sample be resubmitted for analysis. The laboratory will notify the client upon sample receipt if the samples exhibit obvious signs of damage, contamination or inadequate preservation.

   NOTE: Never affix a label directly on an Encore Volatile sampler.

10) If the conditions listed on the Acceptance Policy are not satisfactory and when lacking direction or agreement with the client, the sample will be rejected by the laboratory.

11) For DoD work, the Project Manager will notify the client that samples are received after hours they lab will not accept the samples until the following day.

Figure – 1
Example Cooler Receipt Form

Client: _____ Project: _____ Quote: _____

Cooler Rec'd & Opened for Temp. Check on: _____

Coolers Opened and Unpacked on: _____ By: _____
(Signature)

TestAmerica Pittsburgh Lot Number: _____

		Yes	No	NA
1.	Were custody seals on the outside of the cooler? _____	____	____	____
	If YES, how many and where? Quantity __ Location _____			
	Were signatures and date correct? _____	____	____	____
2.	Were custody papers included inside the cooler? _____	____	____	____
3.	Were custody papers properly filled out (ink, signed, match labels)? _____	____	____	____
4.	Did you sign the custody papers in the appropriate place? _____	____	____	____
5.	Was shippers packing slip attached to this form? _____	____	____	____
6.	Were packing materials used? _____	____	____	____
	If YES, what type? _____			
7.	Were the samples received within the acceptable temperature range? _____	____	____	____
8.	Were the samples appropriately preserved? _____	____	____	____
9.	Were all bottles sealed in separate plastic bags? _____	____	____	____
10.	Did all bottles arrive in good condition (unbroken)? _____	____	____	____
11.	Were all bottle labels complete (sample ID, preservatives, etc.)? _____	____	____	____
12.	Did all bottle labels and/or tags agree with custody papers? _____	____	____	____
13.	Were correct bottles used for tests indicated? _____	____	____	____
14.	Were all VOA vials checked for the presence of air bubbles? _____	____	____	____
15.	Was a sufficient amount of sample sent in each bottle? _____	____	____	____

16. Samples received by: FEDEX UPS CLIENT DROP-OFF OTHER DHL US CARGO

Explain any discrepancies: _____

_____

_____

Level 2 Review _____

Was contacted on _____ by _____ to resolve discrepancies.

Figure – 1
Example Cooler Receipt Form Continued

P: Preserved
UP: Unpreserved

Sample ID	TMET PH<2	DMET PH<2	HG PH<2	NUT(1) PH<2	CN PH ≥12	OG TPHC PH<2	PHEN PH<2	SULF PH ≥12	TOC PH<2	TOX PH<2	VOA P/UP	Hardness PH<2	Residual CL	

(1) "NUT" could include sample bottles for ammonia, chemical oxygen demand, nitrate/nitrite, TKN, or total phosphorus

Comments: _____

Cooler Number	Temperature*	Thermometer ID

Sample	Lot Number**

***Acceptable Temperature Range: < 0-6 °C**

**Please use an asterisk if bottle lot number was covered by the label

Figure – 2
Example Condition Upon Receipt Variance Report

Work Order No.: _____

Condition Upon Receipt Variance Report
TestAmerica Pittsburgh Laboratory

Client: _____ Date: _____
Project No.: _____ Initiated by: _____
Analysis Requested: _____ RFA/COC: _____
Client Sample Numbers Affected: _____

Condition/Variance (Check all that apply):

1. ☐ Not enough sample received for proper analysis. Received approx. _____
2. ☐ Sample received broken/leaking.
3. ☐ Sample received without proper preservative.
   - ☐ Cooler temperature not within 4°C ± 2°C.
     - Record temperature: _____
     - ☐ pH _____
     - ☐ other: _____
4. ☐ Sample received in improper container.
5. ☐ Sample received without proper paperwork. _____
6. ☐ Paperwork received without sample.
7. ☐ No sample ID on sample container.
8. ☐ Custody tape disturbed/broken/missing.
9. ☐ Sample splits performed by lab.
10. ☐ Volatile sample received with approximately _____ mm headspace.
11. ☐ Sample ID on container does not match on paperwork. Explain: _____
12. ☐ All coolers on airbill not received with
13. ☐ Other (explain below): _____

Notes: _____

Corrective Action:

☐ Client's Name: _____ Informed verbally on: _____ By: _____

☐ Client's Name: _____ Informed in writing on: _____ By: _____

☐ Sample(s) processed "as is" _____

☐ Sample(s) on hold until: _____ If released: _____

Sample Control Supervisor Review: _____ Date: _____

Project Management Review: _____ Date: _____

SIGNED ORIGINAL MUST BE RETAINED IN THE PROJECT FILE

TA PT/05-10/96-005/CURVR.DOC

Figure – 3
Example COC/Sample Request

COC/Sample Request

TestAmerica Pittsburgh
301 Alpha Drive
Pittsburgh, PA 15238
Phone: 412-963-7058
FAX: -412-963-2468

Project Name: Site:

Lot Number/Sample Number	Analysis	Matrix

	Raw Relinquished by				Raw Received by			
Raw Sample	Date	Time	Analyst	Location	Date	Time	Analyst	Location

G-4 SOP NO. PT-QA-029, REV. 2, EFFECTIVE DATE: 01/22/2010, QA/QC REQUIREMENTS FOR DOD QSM

Title: QA/QC Requirements for DoD QSM

Approvals (Signature/Date):

Nasreen K. DeRubeis	01/20/10	Larry Matko	01/21/10
Nasreen K. DeRubeis Quality Assurance Manager	Date	Larry Matko Laboratory Director	Date

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1.0 PURPOSE

This policy describes TestAmerica Pittsburgh requirements for ensuring compliance with Federal Program quality assurance (QA) and quality control (QC) requirements for environmental analyses as dictated in the Department of Defense Quality Systems Manual for Environmental Laboratories (DoD QSM). Variance requests, if needed, will be provided to TestAmerica Pittsburgh clients on a project-specific basis for their approval.

2.0 SCOPE

2.1 This policy shall be enforced and followed throughout the laboratory.

2.2 This policy includes QA/QC requirements of the following Federal environmental analysis program requirements documents:

Federal Department	Document Title	Version
Department of Defense (DoD)	Quality Systems Manual for Environmental Laboratories (QSM)	Version 3 Final, January 2006
Department of Defense (DoD)	Quality Systems Manual for Environmental Laboratories (QSM)	Version 4.1 Final, April 2009

2.3 Any additional or contradictory instructions in approved project planning documents take precedence over those given in this policy.

2.4 General requirements for review of project objectives for any program prior to work acceptance are described in Section 7.0 of the TestAmerica Pittsburgh Quality Assurance Manual (PT-LQAM).

2.5 Further details concerning contract review, verification of certification requirements, determination of project-specific technical requirements (e.g., methods, analyte lists, QC requirements), TAT, deliverables, documentation of project requirements, and organizational responsibilities are given in SOP PT-PM-001, “Project Information Requirements.”

3.0 SAFETY

3.1 There are no specific safety hazards associated with this SOP.

3.2 During the course of performing this procedure it may be necessary to go into laboratory areas to consult with appropriate staff members, therefore employees performing this procedure must be familiar with the Laboratory Health & Safety Plan, and take appropriate precautions and wear appropriate attire and safety glasses.

4.0 DEFINITIONS

4.1 Detection Limit (DL): The smallest analyte concentration that can be demonstrated to be different from zero or a blank concentration at 99% confidence. At the DL, the false positive rate (Type 1 error) is 1%. This is equivalent to TestAmerica's MDL (see below).

4.2 Grey Boxes: The DoD QSM are based on the NELAC Quality Standards, Chapter 5 and use the same formatting and numbering. The DoD documents use boxes shaded in grey to add clarifications or additional requirements that are more explicit or go beyond those in the NELAC Standard.

4.3 Initial Calibration Verification (ICV): This term has a slightly different meaning in the DoD QSM than typical usage. In the DoD QSM, it refers to the first calibration verification standard run each day. TestAmerica Pittsburgh SOPs use this term to mean a second-source standard analyzed immediately following the initial calibration.

4.4 Limit of Detection (LOD): The smallest amount or concentration of a substance that must be present in a sample in order to be detected at a high level of confidence (99%). At the LOD, the false negative rate (Type II error) is 1%. This is equivalent to the MDL verification standards are consistently detected, as discussed in SOP PT-QA-007.

4.5 Limit of Quantitation (LOQ): The lowest concentration that produces a quantitative result within specified limits of precision and bias. For DoD projects, the LOQ shall be set at or above the concentration of the lowest initial calibration standard. The laboratory's routine reporting limit is equal to the LOQ, unless project documents specifies a higher concentration to be used as the project-specific reporting limit.

4.6 Marginal Exceedances: Sporadic failures of LCS results outside of the 3-standard-deviation control limits, but less than 4 standard deviations. The NELAC Standard and DoD QSM, define the number of marginal exceedances allowed as a function of the number of analytes in the LCS.

4.7 Method Detection Limit (MDL): Detection limits as determined according to testAmerica Pittsburgh SOP PT-QA-007.

4.8 Limit of Detection Verification (LODV): LOD verification is the process of conducting a test to prove that the calculated MDL is reliable. The verification of the LOD shall be confirmed by qualitative identification of the analytes in a QC sample in each quality system matrix at 2-3 times the LOD for single analyte tests or 3-4 times the LOD for multiple analyte methods. The LOD shall be verified quarterly.

4.9 Limit of Quantitation Verification (LOQV): LOQ verification is the process of conducting a test to demonstrate that the initial characterization of bias and precision at the LOQ concentration continues to be reliable. The LOQ shall be confirmed by successful analysis of a QC sample containing each analyte of concern in each quality system matrix at 1-2 times the claimed LOQ.

4.10 Reporting Limit (RL): The laboratory's routine RLs are determined according to TestAmerica Pittsburgh SOP PT-QA-014. Project teams can instruct the laboratory to use

higher reporting limits for individual projects, but reporting limits cannot be lower than the laboratory's LOQ.

4.11 NELAC: National Environmental Laboratory Accreditation Conference. A voluntary association of state and federal officials who foster the generation of laboratory data of known and documented quality through adoption of national standards.

4.12 QAS – A Quality Assurance Summary is an outline of project-specific requirements that are different than the laboratory's routine practices. It is prepared by the laboratory Project Manager or designee and can be used by the operational sections of the laboratory as a reference when performing the analytical work for a given project

4.13 QAPP: Quality Assurance Project Plan

4.14 USACE: United States Army Corps of Engineers, one of the DoD armed services.

5.0 PROCEDURE

5.1 Work falling under the requirements of any of the Federal programs listed in Section 2.2 will be conducted in accordance with the practices given in this policy, unless changes are specifically discussed in advance with a client and approved in writing.

5.2 Any requirements in this policy that are more stringent or otherwise different than the laboratory standard operating procedures (SOPs) or the PT-LQM take precedence and must be followed, unless superseded by approved project-specific instructions.

5.3 Although this policy presents the requirements of the most current version of each of the guidance documents listed in Section 2.2, the laboratory may still receive samples that are governed by a previous version, which may differ from the current version. Project Managers must include a notice to analysts in the Client Requirements section of quotes in the LIMS to inform analysts which version of the guidance document applies.

5.4 Methods and compounds beyond those given in the respective Federal guidance documents may require changes to some of the general QC requirements. For example, special consideration would be needed for analysis of the full Appendix IX list of compounds. All such changes will be discussed with the client in advance, and will not be used until approved.

5.5 The following tables describe the requirements for QSM 4.1.

6.0 RESPONSIBILITIES

6.1 The bench level analysts, group leaders, and data reviewers are responsible for following the requirements specified in this policy for DoD projects.

6.2 The Project Manager is responsible for informing the laboratory via LIMS test codes, special instructions in LIMS, and QASs, the necessity of applying DoD requirements, as

appropriate. In the communication to the analysts, the Project Manager must differentiate work involving different versions of the guidance documents. The Project Manager is also responsible for a final review of the data package. The Project Manager reviews for completeness, accuracy of deliverables, and correct invoicing. In addition, the Project Manager must inform the QA Manager when the first data packages will be available for the required QA review.

6.3 The Customer Service Manager is responsible for communicating the laboratory's capabilities to clients, including making arrangements for review and approval of all required variances and clarifications, as specified in "TestAmerica Pittsburgh Technical Approach to the DoD QSM".

6.4 The Quality Assurance Manager (QAM) is responsible for ensuring that work performed by the laboratory complies with the DoD QSM. The QAM is responsible for setting up structured analysis codes (SACs) in the LIMS that incorporate the appropriate MDLs, RLs, data flagging, and control limits as specified in the applicable requirements document, i.e., DoD QSM. The QAM is also responsible for QA oversight, which includes review of non-conformances, review of control charts, and other performance indicators. These QA activities are explained in more detail in the Quality Assurance Manual (QAM). At least 10% of DoD packages will be reviewed by the QA department. This review is part of the QA program and does not need to be completed before the data package is issued to the client.

7.0 REVISION HISTORY

7.1 Revision 0.

7.2 Revision 1:

7.2.1 Added the following methods and tables to specify requirements for: Sulfide 9030B/9034, Flash Point methods 1010 and 1020, pH methods 9040 and 9045, TOC in soil by Lloyd Kahn and percent moisture method 2540G.

7.2.2 Added Tin to metals list. Deleted the following compounds for 8270: 1,2,4,5-Tetrachlorobenzene, 2-Naphthylamine, 7,12-Dimethylbenz (a) anthrancene, Diphenylamine, Benzaldehyde, Atrazine, Acetophenone, Indene and Methyl methanesulfonate. Deleted the following for 8260: 1,3,5-Trichlorobenzene and Pentafluorobenzene (Surrogate). Deleted Carbazole for 8310.

7.3 Revision 2:

7.3.1 Updated Attachment 1, Flagging Protocols for LIMS

7.3.2 Updated flags throughout the SOP to match what is in Attachment 1.

8.0 Attachments

8.1 Attachment 1: Flagging Protocols for LIMS

8.2 Attachment 2: Organic Targert Analyte Lists/Methods for DoD QSM 4.1

8.3 Attachment 3: Inorganic Target Analyte Lists/Methods for DoD QSM 4.1

GENERAL REQUIREMENTS FOR ALL LAB SECTIONS

The following table summarizes DoD requirements that are applicable to all areas of the laboratory.

GENERAL REQUIREMENTS FOR ALL LAB SECTIONS	
Requirement	**DoD QSM v3 and v4.1**
Detection Limits Limits of Detection (LOD) Method Detection Limit (MDL) Detection Limit (DL)	Establish MDLs for all matrix-method-analyte combinations. Determine MDLs according to TestAmerica Pittsburgh SOP PT-QA-007. If a method employs a cleanup step for only some samples, then determine an MDL for the method with and without the cleanup. Run an **MDLV** check sample at approximately **2X** the current MDL immediately following the MDL study. • The analyte must be reliably detected **and** identified using method-specific criteria. • All requirements for analyte detection must be met, e.g., ion abundance, second column confirmation, or pattern recognition. • If the method does not include confirmation of an analyte, then the MDLV check sample must produce a signal that is 3X the instrument's noise. 10X rule: The ratio of the ***mean recovered concentration*** (not the spiked concentration) must be between 1 - 5 for a water matrix, and between 1 - 10 for other matrices. An annual MDL study is **not** required as long as MDLV check samples are successfully measured **quarterly**. The MDL must be ≤ $^{1}/_{3}$ RL. **Version 4.1 DL** Establish DLs for all matrix-method-analyte combinations. Determine DLs according to 40 CFR 136B or an acceptable alternative method. **Version 4.1: LOD** Run an **LOD** (equivalent to the MDLV spike) check sample at approximately **2-3X** the current DL (1-4 times for multi-analyte methods) immediately following the DL study. The LOD must be analyzed on each instrument for a given method. This spike concentration establishes the LOD. • The analyte must be reliably detected **and** identified using method-specific criteria. • The LOD must be analyzed on each instrument. • All requirements for analyte detection must be met, e.g., ion abundance, second column confirmation, or pattern recognition. • If the method does not include confirmation of an analyte, then the MDLV check sample must produce a signal that

GENERAL REQUIREMENTS FOR ALL LAB SECTIONS	
Requirement	**DoD QSM v3 and v4.1**
	is 3X the instrument's noise. • If the LOD fails, then the LOD must be performed at a higher concentration and pass two consecutive LOD verifications at the higher concentration. The LOD will be set at the higher concentration. An annual DL study is **not** required as long as LOD check samples are successfully measured **quarterly**.
Reporting Limits (RLs) Limit of Quantitation (LOQ)	RLs are defined by the client and related to project-specific action levels. Must be at least 3 times the MDL. A small number of marginal exceptions may be allowed. Exceptions are allowed for multi-component analytes such as PCBs, toxaphene, GRO, and DRO: The LOQ is not based solely on the LOD of the various components, but on the concentration of the mixture at which the pattern becomes recognizable to the analyst. The RL must lie within the calibration range, at or above the LOQ. If a client requires an RL below the lowest standard of the calibration curve and below the LOQ, then method modification is required. For methods that require only one standard (i.e., lower limit of curve is the origin), the RL shall be no lower than the low-level check standard, designed to verify the integrity of the curve at the lower limits. The lowest standard of the calibration establishes the LOQ, which must be ≥ 3X LOD. The quantitation range is defined by the low and high calibration standards. Results reported to the client that are below the RL must be flagged appropriately: ND ≤ MDL: U-flag, report LOD adjusted by any dilution factors used. MDL - RL: J-flag, reported as estimation **Version 4.1 – LOQ** The validity of the LOQ shall be confirmed by successful analysis of a QC sample containing the analytes of concern in each quality system matrix 1-2 times the claimed LOQ. • The LOQ must be set within the calibration range. • The LOQ must be verified quarterly. • The procedure for determining the LOQ must empirically demonstrate precision and accuracy.

GENERAL REQUIREMENTS FOR ALL LAB SECTIONS	
Requirement	**DoD QSM v3 and v4.1**
	• If the method is modified, precision and bias at the new LOQ must be demonstrated and reported.
Initial Calibration Criteria	Include all reported target analytes and surrogates in the initial calibration. Use a minimum of 5 contiguous calibration points for organics and 3 points for inorganics. The RL must lie within the calibration range, at or above the LOQ. Do not exclude any initial calibration points without technical justification. Acceptance Criteria: • Linear regression correlation coefficient ≥0.995 • Maximum %RSD of 20% for each analyte Deviations permitted only with DoD approval. When sample concentrations exceed the upper limit of the calibration curve, samples shall be diluted and reanalyzed to bring them within the calibration curve. When sample concentrations fall outside the calibration range, high or low, the resulting data shall be qualified as having estimated values and shall be flagged.
Second-Source Standard (Initial Calibration Verification or ICV)	Measure a second-source standard after the ICAL to verify the ICAL (initial calibration verification or ICV). The concentration of the second-source standard shall be at or near the middle of the calibration range. Acceptance criteria must be at least as stringent as those established for the CCV. CCV must pass acceptance criteria before analyzing samples. Can use standard from a second lot when only one manufacturer (i.e., producer of standard and not the vendor) of the calibration standard exists. The requirement for a second-source standard for ICV is waived if a second-source standard is used for the CCV. Deviations require project-specific approval from appropriate DoD personnel.
Continuing Calibration Verification (CCV)	Verify the initial calibration prior to sample analysis with an acceptable CCV in each analytical batch. As long as the CCV is acceptable, a new ICAL is not necessary. When reference methods specify a CCV frequency in terms of a per sample basis, this shall be field samples only. However, QC samples must be run with their associated batches. The grouping of QC samples from more than one batch is not acceptable. If the method does specify a frequency, each batch of 20 samples shall be bracketed by CCVs.

GENERAL REQUIREMENTS FOR ALL LAB SECTIONS	
Requirement	**DoD QSM v3 and v4.1**
	CCV ≤ middle of the calibration range The CCV may be from the same source as the calibration standards. Compare CCVs to the ICAL. Criteria may include % difference from the mean response factor for the ICAL, minimum response factor checks, and confirmation that the RT is within an acceptable window. The % difference must be <15% of the ICAL for organic methods, and 10% for inorganic methods, unless otherwise specified by the reference method. If CCV fails: • Need project-specific permission to report data • Use CV flag and explain in the case narrative. If initial corrective action attempts fail and the CCV results are still outside established acceptance criteria, and the laboratory chooses to demonstrate the success of routine corrective action through the use of 2 consecutive CCVs, then the concentrations of the 2 CCVs must be at 2 different levels within the original calibration curve. At least one of the CCV standards shall fall below the middle of the initial calibration range.
Control Limits	In order of priority: • Use project-specific limits, if specified. • Use control limits in the QSM (Appendix DoD-D v3 and G v4.1), unless other limits have been approved. • If no DoD limits, then use lab in-house limits. The laboratory limits must be within the DoD limits where they exist, or within published limits for similar methods. The lower LCS control limit must be ≥ 10%. Requirements for in-house limits: • Must be statistically derived. Limits at ±3 times the standard deviation of the recovery data are recommended. • Must meet limits specified in the method, if available • Must be updated annually and re-established after major changes in the analytical process (e.g., new instrument, new analyst). • Must be based on at least 30 data points generated under the same analytical process. • Must not exclude failed LCS recovery data and statistical outliers unless there is a documented and scientifically valid reason. • Using control charts to detect trends and prevent out-of-control conditions is recommended. **NOTE**: The TA Pittsburgh QA Office maintains separate LIMS QC programs with verified MDLs and with the appropriate control limits, i.e., QSM limits or in-house control limits. The LIMS flags out-of-control data based on the limits

GENERAL REQUIREMENTS FOR ALL LAB SECTIONS	
Requirement	**DoD QSM v3 and v4.1**
	entered for the QC program. Analysts are responsible for investigating out-of-control data and implementing corrective actions.
Marginal Exceedances (ME)	MEs apply to methods with more than 10 analytes. • Set the ME limit at 4 standard deviations around the mean. • MEs must be random. • Allowed 5 MEs if > 90 analytes; 4 MEs if 71–90 analytes; 3 MEs if 51–70 analytes; 2 MEs if 31–50 analytes; 1 ME if 11-30 analytes. • Designated poor performing analytes (see LCS below) do not control batch acceptance. The same analyte exceeding the LCS control limits 2 out of 3 consecutive LCSs indicates non-random behavior. **DoD does not allow any project-specific analytes of concern to exceed its LCS control limits, even marginally. The use of marginal exceedences should be addressed with the client prior to the beginning of a project.**
Control Charts	The quality manager must ensure continuous improvement through the use of control charts and other method performance indicators. Control limits must be monitored for shifts in mean recovery, changes in standard deviation, and development of trends.
Batch	When there is no separate preparation method used, e.g., volatiles in water, the batch is defined as environmental samples that are analyzed together with the same method and personnel, using the same lots of reagents, not to exceed the analysis of 20 environmental samples.
Laboratory Control Sample (LCS)	Include all target analytes in the LCS. For multi-component analytes, the LCS should be spiked with the same constituents as the calibration standard. Analyze 1 LCS per preparation batch. Use evaluation and acceptance criteria in QSM Appendixes DoD-B and DoD-D (version 3) and DoD-F and G (version 4.1). No project-specific analyte of concern can exceed its LCS control limits, even marginally. DoD-defined poor performers, listed below, should not be used to control the batch: 8270 Water 4-nitrophenol benzoic acid phenol phenol-d5 or d6 (surrogate) 8270 Solid 3,3'-dichlorobenzidine benzoic acid

GENERAL REQUIREMENTS FOR ALL LAB SECTIONS	
Requirement	**DoD QSM v3 and v4.1**
	4-chloroaniline 8151 Solid dinoseb
LCS Corrective Action	Samples analyzed along with an LCS that is determined to be "out of control" shall be considered suspect and the samples reprocessed and re-analyzed or the data reported with appropriate data qualifying codes.
Matrix Spike and Duplicate (MS and MSD)	DoD clients should provide a minimum of 3 MS/MSD samples for a new or unknown matrix. In this case, compare the % mean recoveries and standard deviations for each analyte recovered in the new matrix to the DoD LCS means and control limits generated for clean matrices. These should be within the limits published in Appendix DoD-D (v3) and G (v4.1). Analyze one MS and MSD using the same environmental matrix collected for the specific DoD project in each prep batch of DoD samples. If there is insufficient sample material, it must be noted in the case narrative. All target analytes must be spiked in the project-specific MS and MSD. The RPD must be calculated as a comparison of measured concentrations. Results for MS and MSD must be evaluated against DoD acceptance criteria for the LCS. Corrective actions for matrix spike failures can vary from project to project and depend on project-specific data quality objectives (DQOs). • Organic cleanup procedures should be considered, i.e., sulfur removal for 8081A and 8082, sulfuric acid washing for 8082, Florisil column cleanup for 8081A, and GPC for biological or soil samples. • Dilution of the sample can reduce the level of interferences, but will result in higher reporting limits and so should be compared to DQOs. • Re-preparation and reanalysis to confirm matrix interference is not beneficial if the laboratory has objective evidence of matrix interferences. This should be discussed in advance. If reanalysis is required when documented evidence of interference is present, this should be specified in the project documents and communicated to the analysts accordingly.
Surrogates	Compare surrogate spike recoveries to project-specific limits specified by the client or the QSM limits, if project-specific limits are not available. If DoD limits are not available, use in-house limits.
Internal Standards	Add internal standards to samples, controls, and blanks per reference Method. Reanalyze any samples associated with out-of-control results.
Method Blanks (MB)	Analyze one blank per batch. Acceptance criteria for MB:

GENERAL REQUIREMENTS FOR ALL LAB SECTIONS	
Requirement	**DoD QSM v3 and v4.1**
	• < ½ of the reporting limit for each analyte, • or < $^{1}/_{10}$ of the regulatory limit, • or < $^{1}/_{10}$ of the sample results, whichever is greater. For common lab contaminants: 8260B: Methylene Chloride, Acetone and 2-Butanone. 8270C: Phthalates. 6010B/6020: Copper, Iron, Lead (Trace ICP only), Zinc, Chromium and Tin, the concentration cannot exceed the reporting limit. If the blank is contaminated: • Investigate and take measures to minimize or eliminate contamination. • Any sample associated with a failed blank must be reprocessed, unless the sample is ND. • If insufficient volume remains to reprocess the sample, then apply a B-flag to all results for the specific analyte(s) in all samples associated with the failed blank. • Document any corrective action. **NOTE**: TA Pittsburgh's LIMS automatically applies a B flag to results when the method blank exceeds the MDL, rather than ½ RL (i.e., we are over-flagging). However, analysts take corrective action only if the blank result exceeds ½ RL and the situation is explained in the final report case narrative.
Sample Duplicates	"Matrix duplicates" are replicate aliquots of the same sample taken through the entire analytical procedure. The matrix duplicate provides a usable measure of precision only when target analytes are found in the selected sample. The frequency of duplicate analysis is determined by the project or as specified in the mandated method. If the known concentration of concern is >5 times the LOQ, a matrix duplicate may be analyzed in place of the MSD. A matrix spike is still required. Duplicate analyses should be performed at a minimum frequency of one per preparatory batch per matrix type. Results are used to assess analytical precision for a given matrix and are expressed as RPD, which must be calculated as a comparison of measured concentrations. Acceptance criteria are either specified in the mandated method or established by the lab.
Holding Time (HT)	Holding time is the time elapsed from the time of sampling to the time of extraction or analysis, as appropriate. Include both date and time of analysis as part of the laboratory report. If the time of the sample collection is not provided, assume the most conservative, i.e., the earliest, time of day. **NOTE**: TA Pittsburgh routinely reports date and time of analysis. However, TA Pittsburgh normally calculates holding time to the same units as expressed in the reference methods. For example, if the holding time is expressed in hours, then TA Pittsburgh measures to the hour; and if expressed in days, then to the day. TA Pittsburgh practice is consistent with the reference methods. For most projects, this is not considered a variance, TA

GENERAL REQUIREMENTS FOR ALL LAB SECTIONS	
Requirement	**DoD QSM v3 and v4.1**
	Pittsburgh ensures that this practice is brought to the attention of project personnel.
Client Approval Needed for Some Preferred Methods	Any modifications to existing method requirements require project-specific approval by DoD personnel. Soxhlet extraction (SW3540/3541) is preferred by the USACE. Written approval is needed to use sonication (SW3550B) instead. The laboratory performs SW3550B/C only for Method 8151A.
Aliquotting and Subsampling	Procedures shall address laboratory practices for the handling, subsampling, and documenting of extraneous materials (e.g., rocks, twigs, vegetation, etc.) present in samples. Subsampling must be consistent with ASTM D6323 and EPA/660/R-03/027. Pittsburgh SOP# PT-QA-024. Targeting weights is not allowed. Water samples must be aliquotted by volume using Class A glassware. Do not decant or filter samples, unless required by the method or approved for the project. Sample containers for extractable organics must be solvent rinsed, and the rinse included in the extraction.
Data Qualifier Flags	Flagging should be used only as a last resort. Data should be flagged only after corrective action has been performed. U - Undetected at the LOD – the associated data is the LOD, adjusted by any dilution factor used in the analysis. J - Estimated: The analyte was positively identified; quantitation is an estimation; e.g., matrix interference, below standard, outside the calibration range, between MDL and RL. B - Blank contamination: The analyte was detected above ½ the RL in an associated blank. N - Non-target analyte: The analyte is a tentatively identified compound (TIC) using mass spec. LC -LCS recovery * - surrogate recovery failed. Z- Internal Standard failure. AB – ICSA/ICASAB – Quality control failed. CV – CCV failure. Qualifiers may be combined when appropriate. **See flagging protocols for the LIMS in Attachment 1.**
Manual Integration	When manual integrations are performed, raw data records shall include the following: • A complete audit trail for those manipulations. • Raw data output showing the results of the manual integration (i.e., chromatograms or manually integrated peaks) • Notation of rationale. • Date • Signature/initials of person performing manual operation (electronic signature is acceptable). **Identify all manual integrations in the case narrative:**

GENERAL REQUIREMENTS FOR ALL LAB SECTIONS	
Requirement	**DoD QSM v3 and v4.1**
	The laboratory provides this information as an attachment to the case narrative. For acceptable manual integration procedures also refer to policy CA-Q-S-002, Manual Integration.
Reporting & Records	Report all results for solids on a dry-weight basis. Project managers must consider those methods that include a drying step in order to avoid double correction for moisture content (LIMS set-up) e.g. TOC. Report the date and time of analysis for all tests. Report DLs, LODs, and LOQs that are adjusted for dilutions with sample results. Report all dilutions. Both date and time of analysis are considered to be essential information and shall be included as part of the analytical record. Identify all samples for which manual integration was necessary. **Version 4.1 Reporting Results:** Guidance: Detection limit (DL) = 2, Limit of Detection (LOD) = 4, Limit of Quantitation (LOQ) = 15, sample is undiluted Sample #1: Analytical result: Not detected; reported result : 4U Sample #2: Analytical result: 2; reported result : 4J Sample #3: Analytical result: 10; reported result : 10J Sample #4: Analytical result: 15; reported result : 15
Proficiency Testing	The lab must participate in a PT program as defined by NELAC. Successful PT performance is required for all fields of testing for which commercial PT materials are available.
Corrective Actions	Managers, including the quality manager, are responsible for acting upon corrective action report reviews and are ultimately accountable for the follow-through, verification, and evaluation of these corrective actions.
Measuring & Test Equipment	Check balance calibration daily or before use with 2 weights that bracket target weight(s). Balances shall be calibrated annually by a certified technician. Acceptance criteria for analytical balance checks are ±0.1% or ±0.5 mg, whichever is larger, unless method-specific guidance exists. For top-loading balances, see ASTM D 4753. (v4.1) Standard weights must be calibrated every 5 years and require a third-party certificate of acceptance. Monitor refrigerator/freezer temperatures daily. Refrigerators are controlled to 4 ± 2 °C, and freezers to -10 to -20 °C (≤-

GENERAL REQUIREMENTS FOR ALL LAB SECTIONS	
Requirement	**DoD QSM v3 and v4.1**
	10°C v4.1). Check the calibration of liquid-in-glass thermometers when received and annually. Check the accuracy of electronic thermometers against a NIST-traceable thermometer quarterly at 1 temperatures that bracket the target temperatures (check at 1 temperature allowed in V4.1). Check the accuracy of mechanical pipettes at least quarterly; the volume must be within ±2% of the true volume. Precision, RSD < 1%. Check the accuracy of non-volumetric glassware/labware that is used for measuring initial sample and final extract/digestate volumes on a per lot basis at the time of purchase. The volume must be within ±3% of the true value. RSD ≤3%. Check the temperature accuracy of drying ovens before and after. Temperatures must comply with method-specific requirements or be within ±5% of set temperature.

REQUIREMENTS FOR ORGANIC ANALYSIS BY GC AND HPLC (METHODS 8011, 8015B/C, 8021B, 8081A/B, 8082/A, 8141A/B, 8151A, 8310)

The following table summarizes the DoD requirements that are specific to the GC and HPLC SW 846 Methods 8011, 8081, 8082, 8141, 8151 and 8310. These requirements are in addition to or in place of the requirements listed in section 4.

Requirements for ORGANIC Analysis BY GC AND HPLC (Methods 8011, 8081A/B, 8082/A, 8141A/B, 8151A and 8310)	
Requirement	**DoD QSM v3 and v4.1**
Method 8081 (only) Endrin/DDT Breakdown Check	Performed daily prior to analysis of samples. Degradation must be ≤15% for both Endrin and DDT. Any problems must be corrected. Do not run samples until degradation is ≤15%.
Initial Calibration (ICAL)	Perform a minimum 5 point calibration for all analytes prior to sample analysis including chlordane and toxaphene. Acceptance Criteria options: 1. RSD for each analyte ≤ 20%. 2. Linear least squares regression: r ≥ 0.995. 3. Non-linear regression: coefficient of determination (COD) r^2 ≥ 0.99 (6 points must be used for second order). [*This option cannot be used for Method 8082, but is allowed for 8082A.*]

Requirements for ORGANIC Analysis BY GC AND HPLC (Methods 8011, 8081A/B, 8082/A, 8141A/B, 8151A and 8310)	
Requirement	**DoD QSM v3 and v4.1**
	Any problems must be corrected and ICAL repeated. No sample can be run until ICAL is successful. For PCB analysis, a mixture of Aroclors 1016 and 1260 is normally used to establish detector calibration linearity, unless project-specific data suggest the presence of other Aroclors. Also analyze a mid and low standard for each of the remaining Aroclors for pattern recognition and response factor. If Aroclors other than 1016 and 1260 are detected, a multi-point calibration will be performed for those Aroclors and the associated samples will be reanalyzed. Quantitation for multi-component analytes such as chlordane, toxaphene, and Aroclors must be performed using a 5-point calibration. Results may not be quantitated using a single point.
Second-Source Calibration Verification	Measure a second-source standard once after each ICAL. Acceptance limits for all analytes is ± 20% of expected value. Correct any problems and verify second-source standard. Rerun standard. If that fails, correct problem and repeat ICAL. No samples may be analyzed until the second-source calibration verification is successful. **Version 4.1:** Acceptance limits for HPLC Methods: All analytes within ± 15% of expected value. For GC methods ± 20% of expected value.
RT Window Position	Establish the RT window position for each analyte and surrogate once per ICAL and at the beginning of the analytical shift. Set position using the midpoint standard of the calibration curve or the value in the CCV run at the beginning of the analytical shift.
RT Window Verification	Verify RT window for each analyte and surrogate for each calibration verification. The analyte must be within the established window. Any problems must be corrected and all samples reanalyzed since the last acceptable RT check. If they fail, redo the ICAL and reset RT window. If CCV, apply a CV-flag to all results for analytes outside the established window. Must have verified RT window at the initial calibration verification.
Retention Time (RT) Window Width	Perform 72-hour study at method set-up and after major maintenance (e.g., column change) to calculate the RT window width for each analyte and surrogate. RT width is ± 3 times the standard deviation for each analyte RT.
Continuing Calibration Verification (CCV)	CCV concentration ≤ midpoint of calibration range. Run at beginning of day, after every 10 field samples, and at the end of the analysis sequence. All analytes must be within ± 20% of expected value from ICAL. Correct any problems, repeat CCV, and reanalyze all samples since last successful CCV. Apply Q-flag to all results for the specific analyte(s) in all samples since the last acceptable CCV if reanalysis is not possible. If %D for an individual analyte > 20%, no samples may be analyzed until the problem has been corrected. **Version 4.1:**

Requirements for ORGANIC Analysis BY GC AND HPLC (Methods 8011, 8081A/B, 8082/A, 8141A/B, 8151A and 8310)	
Requirement	**DoD QSM v3 and v4.1**
	Acceptance limits for GC Methods: All analytes within ± 20% of expected value. Acceptance limits for HPLC Methods: All analytes within ± 15% of expected value. Corrective Action: Correct problem, then rerun calibration verification. If that fails, then repeat ICAL. Reanalyze all samples since the last successful calibration verification. If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply CV-flag to all results for the specific analyte(s) in all samples since the last acceptable calibration verification. Problem must be corrected. Results may not be reported without a valid CCV. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
LCS	Include all analyte(s) in LCS that are required to be reported, including surrogates. Run one LCS per prep batch. Acceptance criteria are specified by DoD, if available. Correct any problems then re-prep and reanalyze the LCS and all associated samples for failed analytes. If insufficient sample, then notify the client and apply LC-flag to specific analyte(s) in all samples in the associated prep batch.
Matrix Spike	Run one per prep batch. Use DoD-specific criteria for LCS. For failures, consult project-specific DQOs and contact client for additional measures to be taken. For specific analyte(s) in parent sample, apply a-flag for Organics if acceptance criteria are not met. The MS is for matrix evaluation only. If MS falls outside LCS limits, evaluate data to determine the source of the difference and to determine if there is a matrix effect or analytical error.
MSD or Sample Duplicate	Run one per prep batch. RPD should be ≤ 30%. For failures, consult project-specific DQOs and contact client for additional measures to be taken. For specific analyte(s) in parent sample, apply a-flag for Organics if acceptance criteria are not met. The MS is for matrix evaluation only. If MS falls outside LCS limits, evaluate data to determine the source of the difference.
Surrogates	Spike all field and QC samples with analytes identified in DoD QSM Appendix DoD-D. Must meet DoD LCS acceptance criteria, otherwise method-specified criteria or lab's in-house criteria. For QC and field samples, correct any problems, then re-prep and reanalyze all failed samples for failed surrogates in the associated prep batch. If obvious chromatographic interference with surrogate is present, reanalysis may not be necessary. For specific analyte(s) in the effected field sample, apply *-flag if acceptance criteria are not met. For 8082 PCB analysis, if both surrogates TCMX and DCBP are analyzed and results are processed for both, then report both even if only one is included in the project work plan.
Confirmation of Positive Results	All positive results must be confirmed except toxaphene and technical chlordane in QSM Version 3 (version 4.1 requires confirmation for these analytes). Calibration and QC criteria are the same as for the primary column analysis. The RPD between results for the primary and

Requirements for ORGANIC Analysis BY GC AND HPLC (Methods 8011, 8081A/B, 8082/A, 8141A/B, 8151A and 8310)	
Requirement	**DoD QSM v3 and v4.1**
	secondary columns must be ≤ 40%. Apply J-flag if RPD > 40% or Q-flag if sample is not confirmed and discuss in case narrative. Report the higher of the 2 confirmed results unless overlapping peaks are causing erroneously high results then report the unaffected result and document in case narrative. **Version 4.1:** If the method does not include reporting requirements, then report the results from the primary column or detector, unless there is a scientifically valid and documented reason for not doing so. Apply PG-flag if RPD > 40%. Confirmation is required for all methods except 8015.
Results Between DL (MDL) and LOQ (RL) **Version 4.1** Results between DL, LOD, and LOQ	Apply J-flag to all results between DL and LOQ.

REQUIREMENTS FOR ORGANIC ANALYSIS BY GC/MS (METHODS 8260B AND 8270C/D)

The following table summarizes the DoD requirements that are specific to the GC/MS SW 846 Methods 8260 and 8270. These requirements are in addition to or in place of the requirements listed in section 4.

Requirements for ORGANIC Analysis BY GC/MS (Methods 8260B and 8270C/D)	
Requirement	**DoD QSM v3 and v4.1**
Tuning	Perform tuning prior to calibration and every 12 hours during sample analysis. Use method specified tuning and criteria. If unsuccessful, retune and verify. Rerun any affected samples. No samples may be accepted without a valid tune.
Method 8270 (only) Breakdown Check (GC Performance Check)	Performed daily prior to analysis of samples. Degradation must be ≤20% for DDT. Any problems must be corrected. Do not run samples until degradation is ≤ 20%. Benzidine and pentachlorophenol should be present at their normal responses and peak tailing should not exceed a factor of 2.
Initial Calibration (ICAL)	Perform a minimum 5 point calibration for all analytes prior to sample analysis. Acceptance Criteria options: 1. Average response factor (RF) for SPCCs: VOCs ≥ 0.30 for chlorobenzene and 1,1,2,2-tetrachloroethane ≥ 0.1 for chloromethane, bromoform, and 1,1-dichloroethane SVOCs ≥ 0.050 2. RSD for RFs for CCCs: VOCs and SVOCs ≤ 30% and one option below: Option 1: RSD for each analyte ≤ 15% Option 2: linear least squares regression r ≥ 0.995 Option 3: non-linear regression coefficient of determination (COD) r^2 ≥ 0.99 (6 points for second order) Any problems must be corrected and ICAL repeated. No sample can be run until ICAL is successful.
Second-Source Calibration Verification	Measure a second-source standard once after each ICAL. Acceptance limits for all analytes is ± 25% of expected value. Any problems must be corrected. No samples may be analyzed until the second-source calibration verification is successful.

Requirements for ORGANIC Analysis BY GC/MS (Methods 8260B and 8270C/D)	
Requirement	**DoD QSM v3 and v4.1**
	Version 4.1: All analytes must be within ± 20% of the true value.
RT Window Position	Establish the RT window position for each analyte and surrogate once per ICAL and at the beginning of the analytical shift. Set position using the midpoint standard of the calibration curve.
Relative Retention Time (RRT) Verification	Evaluate RRTs for each sample. RRT of each target analyte in each calibration standard must be within ± 0.06 RRT units. Correct any problems, then rerun ICAL.
Continuing calibration Verification (CCV)	CCV concentration ≤ midpoint of calibration range. Run before sample analysis and every 12 hours of analysis time. The average RF for SPCCs must meet following criteria: VOCs ≥ 0.30 for chlorobenzene and 1,1,2,2-tetrachloroethane ≥ 0.1 for chloromethane, bromoform, and 1,1-dichloroethane SVOCs ≥ 0.050 The %Difference/Drift for CCCs for both VOCs and SVOCs must meet the following criteria: ≤ 20%. Note that "D" is "difference" when using RFs or "drift" when using regression or non-linear calibration. Correct any problems then rerun the CCV. If that fails, repeat the ICAL. Apply Q-flag if no sample material remains and analyte fails criteria. **Version 4.1:** Acceptance limits all analytes and surrogates ≤ 20% D. Corrective Action: DoD project level approval must be obtained for each of the failed analytes or corrective action must be taken. Correct problem, then rerun calibration verification. If that fails, then repeat ICAL. Reanalyze all samples since last acceptable CCV. If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply CV-flag to all results for the specific analyte(s) in all samples since last acceptable CCV. Problem must be corrected. Results may not be reported without a valid CCV. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Internal Standards Verification	Use internal standard in all field samples and standards. RT must be ± 30 seconds from RT of the midpoint standard in the ICAL. EICP area must be within -50% to 100% of ICAL midpoint standard. For any failures, inspect mass spec and GC for malfunctions. Samples analyzed while measurement system was malfunctioning must be reanalyzed. If corrective action fails in field samples, apply Z-flag to analytes associated with the non-compliant IS. Flagging is not appropriate for failed standards.

Requirements for ORGANIC Analysis BY GC/MS (Methods 8260B and 8270C/D)	
Requirement	**DoD QSM v3 and v4.1**
	Sample results are not acceptable without a valid IS verification.
Method Blank	Every field sample, standard, and QC sample. No analytes detected > ½ RL and > 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater). Blank result must not otherwise affect sample results. For common laboratory contaminants, no analytes detected > RL (see Box D-1). Corrective Action: Correct problem, then see criteria in Box D-1. If required, reprep and reanalyze method blank and all samples processed with the contaminated blank. If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch. Problem must be corrected. Results may not be reported without a valid method blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
LCS	Include all analyte(s) in LCS that are required to be reported, including surrogates. Run one LCS per prep batch. Acceptance criteria are specified by DoD, if available. Correct any problems then re-prep and reanalyze the LCS and all associated samples for failed analytes. If insufficient sample or corrective action fails, then apply LC-flag to specific analyte(s) in all samples in the associated prep batch.
Matrix Spike	Run one pre prep batch. Use DoD-specific criteria for LCS. For failures, consult project-specific DQOs and contact client for additional measures to be taken. For specific analyte(s) in parent sample, apply a-flag for Organics if acceptance criteria are not met. The MS is for matrix evaluation only. If MS falls outside LCS limits, evaluate data to determine the source of the difference and to determine if there is a matrix effect or analytical error.
MSD or Sample Duplicate	Run one per prep batch. RPD should be ≤ 30%. For failures, consult project-specific DQOs and contact client for additional measures to be taken. For specific analyte(s) in parent sample, apply a-flag for Organics if acceptance criteria are not met. The MS is for matrix evaluation only. If MS falls outside LCS limits, evaluate data to determine the source of the difference.
Surrogates	Spike all field and QC samples with analytes identified in DoD QSM Appendix DoD-D. Must meet DoD LCS acceptance criteria, otherwise method-specified criteria or lab's in-house criteria. For QC and field samples, correct any problems, then re-prep and reanalyze all failed samples for failed surrogates in the associated prep batch. If obvious chromatographic interference with surrogate is present, reanalysis may not be necessary. For specific analyte(s) in the effected field sample, apply *-flag if acceptance criteria are not met.

Requirements for ORGANIC Analysis BY GC/MS (Methods 8260B and 8270C/D)	
Requirement	**DoD QSM v3 and v4.1**
Results Between LOD (MDL) and LOQ (RL)	Apply J-flag to all results between LOD and LOQ. **Version 4.1:** Apply J-flag to all results between DL and LOQ.

Requirements for INORGANIC Analysis (metals) BY ICP, ICPMS, AND CVAA (Methods 6010B/C, 6020/A, and 7000 series)

The following table summarizes the DoD requirements that are specific to the ICP, ICP/MS, and CVAA. SW-846 Methods 6010B/C, 6020/A, and 7000 series, respectively.

Requirements for INORGANIC Analysis (metals) BY ICP, ICPMS, AND CVAA (Methods 6010B, 6020, and 7000 series)	
Requirement	**DoD QSM v3 and v4.1**
Instrument Detection Limit Study (ICP)	Perform an IDL study every 3 months. IDLs must be ≤ MDL. Samples cannot be analyzed without a valid IDL. For ICPMS IDLs to be done at initial set-up and after significant change in instrument type, personnel, test method, or sample matrix. **Version 4.1:** IDL shall be ≤ LOD
Linear Dynamic Range (LDR) or High Level Check Standard (ICP & ICP/MS)	Run an LDR or high-level check standard at least once every 6 months. Must be within ± 10% of expected value.
MS Tuning	Tuning must be performed prior to initial calibration. The following acceptance criteria apply: Mass calibration: ≤ 0.1 amu from the true value Resolution: < 0.9 amu full width at 10% peak height Stability: RSD ≤ 5% for at least 4 replicate analytes If tuning fails, then retune instrument and reanalyze tuning solutions. Do not analyze samples without a valid MS tune.
Initial Calibration (ICAL)	**ICP**: Measure a minimum of one high standard and a calibration blank. If more than one standard used, then r must be ≥ 0.995, otherwise no acceptance criteria. **ICP/MS**: If more than one standard used, then r must be ≥ 0.995 **CVAA**: Measure a minimum of 5 standards and a calibration blank; r must be ≥ 0.995. The ICAL must pass before running any samples. **NOTE**: The laboratory currently performs three burns for the ICAPES method.
Second-Source Calibration Verification (Initial Calibration	Run second-source standard once after each ICAL and prior to sample analysis.

Requirements for INORGANIC Analysis (metals) BY ICP, ICPMS, AND CVAA (Methods 6010B, 6020, and 7000 series)	
Requirement	**DoD QSM v3 and v4.1**
Verification or ICV)	Must be within ± 10% of expected value for all analytes. Correct any problems, verify standard, and rerun ICV. If that fails, correct problem and rerun ICAL. Verification must pass before running any samples.
Continuing Calibration Verification (CCV)	Run CCV at beginning of day, after every 10 samples, and at the end of the analysis sequence. **ICP**:within ± 10% of expected value **ICP/MS**:within ± 10% of expected value **CVAA**: within ± 20% of expected value Correct any problems, then rerun CCV. If that fails, then repeat ICAL. Reanalyze all samples since last successful CCV. Results cannot be reported without a valid CCV.
Low-Level Calibration Check (ICP & ICP/MS)	Run low-level standard at a concentration ≤ RL daily after one-point ICAL. Must be within ± 20% of expected value. Correct any problems, then reanalyze. Results cannot be reported without a valid low-level check.
Calibration Blank	Analyze calibration blank before analyzing samples, after every 10 samples, and at the end of the analysis sequence. No analytes detected > LOD (MDLV). Correct any problems, then re-prep and reanalyze the calibration blank and 10 previous samples. Apply CB-flag to all results for specific analyte(s) in all samples associated with the failed blank.
Interference Check Solution (ICS) (ICP & ICP/MS)	Run the ISC at the beginning of an analytical run. **ICS-A**:Absolute value of concentration for non-spiked analytes must be < LOD (except for the following trace impurities verified by the vendor Inorganic Ventures: Ag, As, Ba, Fe, Cd, Cu, Mn, Ni, Pb, Sb, Se, Sr, Tl, V and Zn). **ICS-AB**:Within ± 20% of expected value. Correct any problems and reanalyze ICS. Do not analyze samples without a valid ICS. Terminate analysis; locate and correct problem; reanalyze ICS, reanalyze all samples. If corrective action fails, apply AB-flag to all results for specific analyte(s) in all samples associated with the ICS. **NOTE**: TA Pittsburgh has a letter from the ICSA standards manufacturer for many of the elements. The current version of TA Pittsburgh's variance requests for this issue should be included for each project.
Method Blank	One per preparatory batch. No analytes detected > ½ RL and greater than 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater). Blank result must not otherwise affect sample results. For common laboratory contaminants, no analytes detected > RL (see Box D-1).

Requirements for INORGANIC Analysis (metals) BY ICP, ICPMS, AND CVAA (Methods 6010B, 6020, and 7000 series)	
Requirement	**DoD QSM v3 and v4.1**
	Correct problem, then see criteria in Box D-1. If required, reprep and reanalyze method blank and all samples processed with the contaminated blank. If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch. Problem must be corrected. Results may not be reported without a valid method blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed
LCS	Run one LCS will all analytes required to be reported per prep batch. Recovery must meet DoD QSM limits, if available. **6010 & 7470 Water:** 80-120% (ME limits are 80-120%, except Mo, Se, and Ag, which have limits 75-120%.) **6010 & 7471 Soil:** 80-120%, except Ag 75-120% (ME limits are 80-120%, except Al, SB, Mo, Se, and Zn, which have limits 75-120%, and Ag 70-125%.) **6020:** Water/Solid: 80-120% Correct any problems, then re-prep and reanalyze LCS and associated samples for failed analytes in all samples in the associated batch. If corrective action fails, apply LC-flag to specific analyte(s) in all samples in associated batch.
Dilution Test (ICP only)	Run dilution test with each prep batch. Five-fold dilution must agree within ± 10% of original determination. If test fails criteria, then perform following corrective action: **ICP**: Perform post-digestion spike (PDS) addition. Flagging criteria is not appropriate. Only applicable for samples with concentrations > 50 x LOQ.
Post-Digestion Spike (PDS) Addition (ICP)	Perform PDS when dilution test fails or analyte conc in all samples is <50 x LOD for ICP. Spike addition must result in level between 10 - 100 x MDL. Recovery must be within 75 - 125 % of expected result. If PDS fails, then run samples by method of standard addition (MSA) or apply W-flag to all sample results (for same matrix) for specific analyte(s) for all samples associated with the PDS. Spike addition should produce a concentration of 10 – 100 x LOQ.
MSA or Internal Standard Calibration	Run MSA or internal standard calibration when matrix interference is confirmed. Document use of MSA in case narrative.
Matrix Spike (MS)	Run one MS per prep batch. Use DoD acceptance criteria for LCS. If MS fails, consult project-specific DQOs and contact client to see if additional measures need to be taken. For specific analyte(s) in parent sample, apply N-flag or Inroganics if acceptance criteria are not met. The MS is for matrix evaluation only. If MS falls outside LCS limits, evaluate data to determine the source of the difference and to determine if there is a matrix effect or analytical error.

Requirements for INORGANIC Analysis (metals) BY ICP, ICPMS, AND CVAA (Methods 6010B, 6020, and 7000 series)	
Requirement	**DoD QSM v3 and v4.1**
MSD or Sample Duplicate	Analyze one MSD or sample duplicate per prep batch per matrix. RPD between duplicates must be ≤ 20%. For failures, consult project-specific DQOs and contact client for additional measures to be taken. For specific analyte(s) in parent sample, apply N-flag or Inroganics if acceptance criteria are not met. The MS is for matrix evaluation only. If MS falls outside LCS limits, evaluate data to determine the source of the difference.
Internal Standard (IS) (ICP/MS Only)	Add IS to every sample. IS intensity must be within 30 - 120% of intensity of the IS in the ICAL. Perform corrective action as described in Method 6020. **Note**: TA Pittsburgh uses internal standard for method 6010 as well.
Results Between LOD (MDL) and LOQ (RL)	Apply J-flag to all results between LOD and LOQ.

REQUIREMENTS FOR WET CHEMISTRY (METHODS 7196, 9010/9012, AND 9056)

The following tables summarize the DoD requirements that are specific to Wet Chemistry Methods 7196, 9010/9012, and 9056.

Requirements for COLORIMETRIC HEXAVALENT CHROMIUM Analysis (Method 7196)	
Requirement	**DoD QSM v4.1**
Reference Blank (Reagent Water)	Analyze before beginning standards or sample analysis. Use for blank subtraction of standards, field samples, and QC samples. For turbid field samples, a turbidity blank must be used instead of the reference blank.
Initial Calibration	Perform ICAL daily prior to sample analysis using a minimum of 3 standards plus a calibration blank. Correlation coefficient r must be ≥ 0.995. Do not analyze samples unless ICAL passes criteria.
Second-Source Calibration Verification (ICV)	Run ICV before beginning a sample run. Value of ICV must be within ± 10% of expected value. Correct any problems and verify second-source standard. Rerun ICV. If that fails, correct any problems and repeat ICAL. Reanalyze all samples since last successful calibration.
Continuing Calibration Verification (CCV)	Run CCV after every 15 samples and at the end of the analysis sequence. Value must be within ± 10% of expected value. Correct any problems then repeat CCV and reanalyze all samples since the last successful calibration verification. If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply CV-flag to all results for the specific analyte(s) in all samples since the last acceptable calibration verification. Problem must be corrected. No samples may be run until calibration has been verified. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Method Blank	One per prep batch. No analyte detected > 1/2 the reporting limit and > 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater). If the method blank is contaminated as described above, then the laboratory shall reprocess affected samples in a subsequent preparation batch, except when sample results are below the LOD. If insufficient sample volume remains for reprocessing, the results shall be reported with appropriate data qualifiers. (Box D-1). If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch. Problem must be corrected. Results may not be reported without a valid method blank. Flagging is only appropriate in cases

Requirements for COLORIMETRIC HEXAVALENT CHROMIUM Analysis (Method 7196)	
Requirement	**DoD QSM v4.1**
	where the samples cannot be reanalyzed.
LCS	One per prep batch. QC acceptance criteria 85-115%. Corrective Action: Correct problem, then reprep and reanalyze the LCS and all samples in the associated batch for the failed analyte in all samples in the associated preparatory batch, if sufficient sample material is available (see full explanation in Appendix G. If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply LC-flag to specific analyte(s) in all samples in the associated preparatory batch. Problem must be corrected. Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Sample Matrix Verification (also known as Matrix Spike)	Once for every sample matrix analyzed. Spike recovery within 85–115%. If check indicates interference, dilute and reanalyze sample; persistent interference may indicate the need to use alternative method or analytical conditions, or to use method of standard additions. Flagging is not appropriate. Verification check ensures lack of reducing condition or interference from matrix. Additional corrective actions are identified in Method 7196A (7.4 and 7.5) and SOP # PT-WC-015.
MSD or Sample Duplicate	For aqueous matrix, run one MS/MSD pair per every 10 samples per matrix. For solid matrix, run one MS/MSD pair per prep batch per matrix. Spike recovery within 85–115%. RPD between duplicates must be ≤ 20% for aqueous matrix, and ≤ 30% for solid matrix. Examine project-specific DQOs. Contact the client to determine if additional measures are to be taken. Flagging criteria are not appropriate.
Pre-digestion Matrix Spikes Method 3060 Solid Matrix Only (Soluble and Insoluble)	Analyze one soluble and one insoluble pre-digestion MS pre prep batch prior to analysis. MS recoveries must be within 75 - 125%. Correct any problems and rehomogenize, redigest, and reanalyze samples. If that fails, evaluate against LCS results. If corrective action fails, apply N-flag to specific analyte(s) in all samples in the associated prep batch.
Results Between LOD (MDL) and LOQ (RL)	Apply J-flag to all results between LOD and LOQ.

Requirements for CYANIDE Analysis (Methods 9010/9012)	
Requirement	**DoD QSM v4.1**
Initial Calibration	Run initial calibration, using at least 6 standards and a calibration blank, daily prior to sample analysis. Correlation coefficient r must be ≥ 0.995 for linear regression. Do not analyze samples unless ICAL passes criteria.
Distilled Standards	Analyze one high and one low distilled standard once per multipoint calibration. Values must be within ± 15% of true value. Correct any problems, then repeat. Do not analyze samples until distilled standards have passed.
Second-Source Calibration Verification	Analyze second-source calibration verification check standard once after each multipoint calibration. Value must be within ± 15% of expected value. Correct any problems and verify standard. Rerun verification. If that fails, correct any problems and repeat ICAL.
Method Blank	One per prep batch. No analyte detected > 1/2 the reporting limit and > 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater). If the method blank is contaminated as described above, then the laboratory shall reprocess affected samples in a subsequent preparation batch, except when sample results are below the LOD. If insufficient sample volume remains for reprocessing, the results shall be reported with appropriate data qualifiers. (Box D-1). If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch. Problem must be corrected. Results may not be reported without a valid method blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
LCS	One per prep batch. QC acceptance criteria 85-115%. Corrective Action: Correct problem, then reprep and reanalyze the LCS and all samples in the associated batch for the failed analyte in all samples in the associated preparatory batch, if sufficient sample material is available (see full explanation in Appendix G. If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply LC-flag to specific analyte(s) in all samples in the associated preparatory batch. Problem must be corrected. Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
MS/MSD	For 9010, run one MS/MSD pair per prep batch per matrix. For 9012, run one MS/MSD pair per every 10 samples per matrix. Use LCS QC acceptance criteria. % Recovery within 85-115%, RPD ≤ 20% RPD between MS and MSD. Apply N-flag Inorganics for the specific analyte(s) in parent sample if acceptance criteria not met. Contact client to determine if

Requirements for CYANIDE Analysis (Methods 9010/9012)	
Requirement	**DoD QSM v4.1**
	additional measures are desired.
Sample Duplicate (or Matrix Spike duplicate (MSD))	Analyze duplicate sample once per every 20 samples. % Difference between MS or MSD or sample and sample duplicates must be ± 20%. Correct any problems and reanalyze sample and duplicate. Apply N-flag Inorganics if sample cannot be rerun or reanalysis does not correct problem.
Results Between LOD (MDL) and LOQ (RL)	Apply J-flag to all results between LOD and LOQ.

Requirements for COMMON ANIONS Analysis (Method 9056)	
Requirement	**DoD QSM v4.1**
Initial Calibration	Run initial calibration for all analytes, using at least 3 standards and a calibration blank, prior to sample analysis. Correlation coefficient r must be ≥ 0.995 for linear regression. Corrective Action: If fails, correct problem and repeat ICAL. Flagging criteria are not appropriate. NO samples can be run until ICAL passes.
Second-Source Calibration Verification	Analyze second-source calibration verification check standard once after each multipoint calibration. Value for all analytes must be within ± 10% of expected value. Correct any problems and verify standard. Rerun verification. If that fails, correct any problems and repeat ICAL.
RT Window Position	Establish RT window position for each analyte once per multipoint calibration. Position shall be at set using the midpoint standard of the ICAL curve when ICAL is performed. On days when ICAL is not performed, the initial CCV is used.
Retention Time (RT) Window Width	Calculate RT window width for each analyte after method set-up and after major maintenance (e.g., column change). RT width is ± 3 times standard deviation for each analyte over 24-hour period.
RT Window Verification	RT window verification is performed for each CCV. Each analyte peak must fall within the established window. Correct any problems, then reanalyze all samples since the last RT check. If they fail, redo the ICAL and reset RT window.
Initial Calibration Verification (ICV) (second source)	Perform ICV daily before sample analysis, after each ICAL. All analytes must be within ± 10% of expected value AND RTs must be within established windows. Corrective Action: Correct any problems, rerun ICV. If that fails, then repeat ICAL. Flagging is not appropriate.
Method Blank	One per prep batch.

Requirements for COMMON ANIONS Analysis (Method 9056)	
Requirement	**DoD QSM v4.1**
	No analyte detected > 1/2 the reporting limit and > 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater). If the method blank is contaminated as described above, then the laboratory shall reprocess affected samples in a subsequent preparation batch, except when sample results are below the LOD. If insufficient sample volume remains for reprocessing, the results shall be reported with appropriate data qualifiers. (Box D-1). If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch. Problem must be corrected. Results may not be reported without a valid method blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
LCS	One per prep batch. QC acceptance criteria 80-120%. Corrective Action: Correct problem, then reprep and reanalyze the LCS and all samples in the associated batch for the failed analyte in all samples in the associated preparatory batch, if sufficient sample material is available (see full explanation in Appendix G. If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply LC-flag to specific analyte(s) in all samples in the associated preparatory batch. Problem must be corrected. Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Continuing Calibration Verification (CCV)	Run midrange CCV standard after every 10 samples and at the end of the analytical sequence. Instrument response must be within ± 10% of expected value. Correct any problems, then repeat CCV and reanalyze all samples since last successful calibration verification. If reanalysis cannot be performed, data must be qualified. Apply CVQ-flag to all results for the specific analyte(s) in all samples since the last acceptable calibration verification.
MS/MSD	Run one MS/MSD pair per prep batch per matrix. Use LCS QC acceptance criteria. % Recovery within 80-120%. RPD between MS and MSD must be ≤ 20%. Apply N-flag Inorganics for the specific analyte(s) in parent sample if acceptance criteria not met. Contact client to determine if additional measures are desired.
Sample Duplicate	Analyze one sample duplicate per every 10 samples. % Difference between sample and its duplicate must be ≤ 10%. Corrective Action: Correct any problems and reanalyze sample and duplicate. If corrective action fails, apply *-flag to specific analyte(s) in the sample.
Results Between LOD (MDL) and LOQ (RL)	Apply J-flag to all results between LOD and LOQ.

Requirements for TOC Analysis (Method 9060)	
Requirement	**DoD QSM v4.1**
Initial Calibration	Run initial calibration using 5 standards and a calibration blank, prior to sample analysis. Correlation coefficient r must be ≥ 0.995 for linear regression. Corrective Action: Correct any problems, rerun ICV. If that fails, then repeat ICAL. Flagging is not appropriate.
Initial Calibration Verification (ICV) (second source)	Analyze second-source calibration verification check standard once after each multipoint calibration. Perform ICV before sample analysis, after each ICAL. All analytes must be within ± 10% of expected value. Correct any problems and verify standard. Rerun verification. If that fails, correct any problems and repeat ICAL.
Method Blank	One per prep batch. No analyte detected > 1/2 the reporting limit and > 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater). If the method blank is contaminated as described above, then the laboratory shall reprocess affected samples in a subsequent preparation batch, except when sample results are below the LOD. If insufficient sample volume remains for reprocessing, the results shall be reported with appropriate data qualifiers. (Box D-1). If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch. Problem must be corrected. Results may not be reported without a valid method blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
LCS	One per prep batch. QC acceptance criteria 80-120%. Corrective Action: Correct problem, then reprep and reanalyze the LCS and all samples in the associated batch for the failed analyte in all samples in the associated preparatory batch, if sufficient sample material is available (see full explanation in Appendix G. If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply LC-flag to specific analyte(s) in all samples in the associated preparatory batch. Problem must be corrected. Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Continuing Calibration Verification (CCV)	Run midrange CCV standard after every 10 samples and at the end of the analytical sequence. Instrument response must be within ± 10% of expected value. Correct any problems, then repeat CCV and reanalyze all samples since last successful calibration verification. If reanalysis cannot be performed, data must be qualified. Apply CV-flag to all results for the specific analyte(s) in all samples since the last acceptable calibration verification.

Requirements for TOC Analysis (Method 9060)	
Requirement	**DoD QSM v4.1**
MS/MSD	Run one MS/MSD pair per 10 samples. Use LCS QC acceptance criteria. % Recovery within 80-120%. RPD between MS and MSD must be ≤ 20%. Apply N-flag Inorganics for the specific analyte(s) in parent sample if acceptance criteria not met. Contact client to determine if additional measures are desired. Apply *-flag for RPD failure.
Sample Duplicate	Analyze one sample duplicate per every 10 samples. % Difference between sample and its duplicate must be ≤ 20%. Corrective Action: Correct any problems and reanalyze sample and duplicate. If corrective action fails, apply *-flag to specific analyte(s) in the sample.
Results Between LOD (MDL) and LOQ (RL)	Apply J-flag to all results between LOD and LOQ.

Requirements for Oil & Grease/TPH Analysis (Methods 1664A/9070B, 9071B)	
Requirement	**DoD QSM v4.1**
Method Blank	One per prep batch. No analyte detected > 1/2 the reporting limit and > 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater). If the method blank is contaminated as described above, then the laboratory shall reprocess affected samples in a subsequent preparation batch, except when sample results are below the LOD. If insufficient sample volume remains for reprocessing, the results shall be reported with appropriate data qualifiers. (Box D-1). If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch. Problem must be corrected. Results may not be reported without a valid method blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
LCS	One per prep batch. QC acceptance criteria HEM 78-114%. SGT-HEM 64-132%. Corrective Action: Correct problem, then reprep and reanalyze the LCS and all samples in the associated batch for the failed analyte in all samples in the associated preparatory batch, if sufficient sample material is available (see full explanation in Appendix G. If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply LC-flag to specific analyte(s) in all samples in the associated preparatory batch. Problem must be corrected. Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
MS/MSD	Run one MS/MSD pair per batch of samples. Use LCS QC acceptance criteria. Percent recovery criteria HEM 78-114%, RPD HEM 18%. Percent recovery SGT-HEM 64-132%, RPD 34%. Apply N-flag Inorganics for the specific analyte(s) in parent sample if acceptance criteria not met. Contact client to determine if additional measures are desired. Apply *-flag for RPD failure.
Results Between LOD (MDL) and LOQ (RL)	Apply J-flag to all results between LOD and LOQ.

Requirements for Sulfide Analysis (Method 9034/9030B)	
Requirement	**DoD QSM v4.1**
Method Blank	One per prep batch. No analyte detected > 1/2 the reporting limit and > 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater). If the method blank is contaminated as described above, then the laboratory shall reprocess affected samples in a subsequent preparation batch, except when sample results are below the LOD. If insufficient sample volume remains for reprocessing, the results shall be reported with appropriate data qualifiers. (Box D-1). If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch. Problem must be corrected. Results may not be reported without a valid method blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
LCS	One per prep batch. QC acceptance criteria 85-115%. Corrective Action: Correct problem, then reprep and reanalyze the LCS and all samples in the associated batch for the failed analyte in all samples in the associated preparatory batch, if sufficient sample material is available (see full explanation in Appendix G. If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply LC-flag to specific analyte(s) in all samples in the associated preparatory batch. Problem must be corrected. Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
ICV/CCV	Run ICV at the beginning of analysis. Run CCV standard after every 10 samples and at the end of the analytical sequence. Criteria must be within ± 15% of expected value. Correct any problems, then repeat CCV and reanalyze all samples since last successful CCV. If reanalysis cannot be performed, data must be qualified. Apply CV-flag to all results for the specific analyte(s) in all samples since the last acceptable CCV.
MS/MSD	Run one MS/MSD pair per 20 samples. % Recovery within 85-115%. RPD between MS and MSD must be ≤ 20%. Apply N-flag Inorganics for the specific analyte(s) in parent sample if acceptance criteria not met. Contact client to determine if additional measures are desired. Apply *-flag for RPD failure.
Results Between LOD (MDL) and LOQ (RL)	Apply J-flag to all results between LOD and LOQ.

Requirements for TOC Analysis in Soil (Method Lloyd Kahn)	
Requirement	**DoD QSM v4.1**
Initial Calibration	Run initial calibration using 7standards, prior to sample analysis. Correlation coefficient r must be ≥ 0.995 for linear regression. Corrective Action: Correct any problems, rerun ICV. If that fails, then repeat ICAL. Flagging is not appropriate.
Initial Calibration Verification (ICV) (second source)	Analyze second-source calibration verification check standard once after each multipoint calibration. All analytes must be within ± 15% of expected value. Correct any problems and verify standard. Rerun verification. If that fails, correct any problems and repeat ICAL.
ICB/CCB	Analyze calibration blank before analyzing samples, after every 20 drops, and at the end of the analysis sequence. No analytes detected > LOD (MDLV). Correct any problems, then re-prep and reanalyze the calibration blank and 20 previous drops. Apply CB-flag to all results for specific analyte(s) in all samples associated with the failed blank
Method Blank	One per prep batch. No analyte detected > 1/2 the reporting limit and > 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater). If the method blank is contaminated as described above, then the laboratory shall reprocess affected samples in a subsequent preparation batch, except when sample results are below the LOD. If insufficient sample volume remains for reprocessing, the results shall be reported with appropriate data qualifiers. (Box D-1). If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch. Problem must be corrected. Results may not be reported without a valid method blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
LCS	One per prep batch. QC acceptance criteria 75-125%. Corrective Action: Correct problem, then reprep and reanalyze the LCS and all samples in the associated batch for the failed analyte in all samples in the associated preparatory batch, if sufficient sample material is available (see full explanation in Appendix G. If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply LC-flag to specific analyte(s) in all samples in the associated preparatory batch. Problem must be corrected. Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Continuing Calibration Verification (CCV)	Run midrange CCV standard after every 20 drops and at the end of the analytical sequence. Instrument response must be within ± 15% of expected value.

Requirements for TOC Analysis in Soil (Method Lloyd Kahn)	
Requirement	**DoD QSM v4.1**
	Correct any problems, then repeat CCV and reanalyze all samples since last successful calibration verification. If reanalysis cannot be performed, data must be qualified. Apply CV-flag to all results for the specific analyte(s) in all samples since the last acceptable calibration verification.
MS/MSD	Run one MS/MSD pair per 20 samples. Use LCS QC acceptance criteria. % Recovery within 75-125%. RPD between MS and MSD must be ≤ 20%. Apply N-flag Inorganics for the specific analyte(s) in parent sample if acceptance criteria not met. Contact client to determine if additional measures are desired. Apply *-flag for RPD failure.
Results Between LOD (MDL) and LOQ (RL)	Apply J-flag to all results between LOD and LOQ.

Requirements for Flash Point Analysis (Methods 1010/ASTM D93-85)	
Requirement	**DoD QSM v4.1**
LCS	One per prep batch of 20 samples. QC acceptance criteria 96-103 °F. Corrective Action: Correct problem, then reprep and reanalyze the LCS and all samples in the associated batch for the failed analyte in all samples in the associated preparatory batch, if sufficient sample material is available (see full explanation in Appendix G. If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply LC-flag to specific analyte(s) in all samples in the associated preparatory batch. Problem must be corrected. Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
CCV	CCV which the same standard as the LCS is analyzed every 10 samples. Criteria same as LCS. Corrective Action: Correct any problems, then repeat CCV and reanalyze all samples since last successful calibration verification. If reanalysis cannot be performed, data must be qualified. Apply CV-flag to all results for the analyte in all samples since the last acceptable calibration verification.
Sample Duplicate	Analyze one sample duplicate per 20 samples. % Difference between sample and its duplicate must be ≤ 10%. Corrective Action: Correct any problems and reanalyze sample and duplicate. If corrective action fails, apply *-flag to specific analyte(s) in the sample.

Requirements for Flash Point Analysis (Methods 1020/ASTM D3278-96)

Requirement	DoD QSM v4.1
Sample Duplicate	Analyze one sample duplicate per 20 samples. % Difference between sample and its duplicate must be ≤ 20%. Corrective Action: Correct any problems and reanalyze sample and duplicate. If corrective action fails, apply *-flag to specific analyte(s) in the sample.

Requirements for pH/Corrosivity Analysis (Methods 9040 and 9045)	
Requirement	**DoD QSM v4.1**
Initial Calibration	pH meter is calibrated daily using buffer 4, 7 and 10. The manufacturer recommended slope is 95.0 %. If the slope is not met, pour fresh buffer solutions into the cups and try again. If this does not resolve the problem, change the KCL solution in the electrode. If still having a problem the probe may need to be changed. Data cannot be reported without a valid calibration. The pH readings must be within 0.05 pH units of the buffer solution value. If not repeat adjustments on successive portions of the two buffer solutions until the readings are within 0.05 pH units (s.u.).
ICV (Second Source)	A second source pH 7 buffer is analyzed after calibration before sample analysis. The pH meter must be recalibrated if the buffer is not within 0.05 pH units.
CCV	CCV same source as calibration standards is analyzed every ten samples. The pH meter must be recalibrated if the buffer is not within 0.05 pH units. If this range is exceeded, reanalyze all samples analyzed since the last pH buffer that met criteria.
Sample Duplicate	Analyze one sample duplicate per 20 samples. The acceptance criterion for the SD is ± 0.05 units. Corrective Action: Correct any problems and reanalyze sample and duplicate. If corrective action fails, apply *-flag to specific analyte(s) in the sample.

Requirements for Percent Moisture Analysis (Method SM 2540G)	
Requirement	**DoD QSM v4.1**
Sample Duplicate	Analyze one sample duplicate per 20 samples. % Difference between sample and its duplicate must be ≤ 20%. Corrective Action: Correct any problems and reanalyze sample and duplicate. If corrective action fails, apply *-flag to specific analyte(s) in the sample.

1. Example Variances and clarifications to the QSM v4.1

TestAmerica Pittsburgh will work with our DoD clients to develop a QAPP that meets data quality objectives for the project and that meets the laboratory's capabilities.

If TestAmerica Pittsburgh is not able to meet project requirements or requirements are unclear and may be subject to a range of interpretations. To avoid misunderstandings and potential project failures, the laboratory will supply the document tiled "TestAmerica Pittsburgh Technical Approach to the DoD QSM" during the project setup phase, which details the issues requiring variances or clarifications. The current document is saved to TestAmerica Pittsburgh's Public folder N:\QA\01_Facility_QA_Documents\09_DoD, and an example document is as follows:

TestAmerica Pittsburgh requests the following example variances and clarifications to the DoD QSM Version 4.1 April 22, 2009.

Variance Requests

1. Holding Time – Box 46**:**

QSM Requirement: Both date and time of preparation and analysis are considered essential information.

Proposal: The laboratory does routinely report date and time of analysis. However, TestAmerica Pittsburgh normally calculates holding time to the same units as expressed in the methods. For example, if the holding time is expressed in hours, then we measure to the hour; and if expressed in days, then we measure to the day. We believe our practice is consistent with the methods and the standard practice of EPA data validators. For most projects this is not considered a variance, but we want to ensure that this practice is brought to the attention of project personnel.

2. Reporting:

Method Blank flagging:

Requirement: Method blanks must be ≤ ½ x RL or < 1/10 x concentration found in samples, with allowances for common lab contaminants controlled to the RL. See SOP PT-QA-029, DoD QSM Version 4.1 Requirements.

Our current LIMS system has no provisions for automatically flagging samples where the blank contamination is > one-half the RL. However we will include a note of the exceedances in the report narrative. Generally where contamination occurs between the RL and ½ the RL the client will be contacted before re-preparation and reanalysis is performed.

Example

Common Laboratory contaminants are: 8260B: Methylene Chloride, Acetone and 2-Butanone. 8270C: Phthalates. 6010B/6020: Copper, Iron, Lead (Trace ICP only), Zinc, Chromium and Tin.

3. Pesticides Method 8081A:

Toxaphene -

(A) Toxaphene and technical chlordane are not included in the spike list

Requirement: "All target analytes must be spiked." QSM Boxes D-2.

The recoveries for the single component chlorinated pesticides encompass the range of conditions that can affect toxaphene recovery. This is analogous to using AR-1016 and AR-1260 as bracketing spiking compounds for method 8082. Typically we would not spike with toxaphene. But if requested the laboratory will meet this requirement. Additional sample volume will be required since two sets of MS/MSDs will be needed.

(B) A multipoint calibration can be performed for Toxaphene and technical chlordane if required by the project.

Requirement: "The initial calibration range shall consist of a minimum of five calibration points for organic analytes and three calibration points for inorganic analytes and IH samples (unless otherwise stated in the method). All reported target analytes and surrogates (if applicable) shall be included in the initial calibration. Reported results for all target analytes and surrogates shall be quantified using a multipoint calibration curve. Exclusion of calibration points without technical justification is not permitted. " QSM Box 34 and Table F-2.

Typically we would not perform a 5 point calibration for Toxaphene and technical chlordane. TestAmerica Pittsburgh requests a variance to run a single low calibration standard with the single peak pesticide calibration curve. If toxaphene and/or technical chlordane are not detected in the samples, the data will be reported. If toxaphene or technical chlordane is detected, a multipoint calibration curve for the detected analyte will be run and the samples reanalyzed and quantitated from the calibration curve.

4. <u>Second-Column Confirmation:</u>

Requirement: For GC & HPLC methods, a second column or second detector must confirm all positive results, with exceptions allowed for toxaphene and Technical Chlordane.

Note: The laboratories standard practice is that second column confirmation of Aroclors (PCBs) by Method 8082 are not normally performed unless set as a project requirement; hits are considered to be confirmed by fingerprint identification. Confirmation must be specified up front. However, the laboratory is prepared to perform dual-column analysis for PCBs by method 8082 if this variance request is not approved.

Example

Clarifications:

TestAmerica Pittsburgh believes that the following items do not constitute variances, but are issues open to a range of interpretations. Our goal here is to clarify all potential technical issues at the outset of projects to ensure that there are no misunderstandings. If these clarifications need to be submitted to DoD for approval, we are prepared to defend them.

1. **8270C SIM (Selective Ion Monitoring) Analysis**: For method 8270C SIM, a non-QSM method, a mass calibration is performed in place of the DFTPP tune standard. For 8270C SIM analysis the mass calibration will be performed before SIM analysis begins to demonstrate that the mass axis and peak width are within calibration. A report will be retained to document the calibration. A 12 hour operation period will commence with the injection of the first calibration standard.

2. **Metals 6010 and 6020**: **ICS-A**: Absolute value of concentration for all nonspiked analytes < 2 x LOD, except for the following trace impurities verified by the vendor Inorganic Ventures: Ag, As, Ba, Fe, Cd, Cu, Mn, Ni, Pb, Sb, Se, Sr, Tl, V and Zn. (Ref: Page 154,157 Table B-7 DoD QSM V 3.0)

3. Marginal Exceedances for LCS Failures - Grey Box D-5
   QSM 4.1 Requirement: DoD does not allow any target analyte to exceed its LCS control limits, even marginally..
   Clarification: The phrase "project-specific analytes of concern" is not defined and is unclear. Based on discussions with Dr. Joseph Solsky and Dr. Chung-Rei Mao at the USACE/CX branch, TestAmerica Pittsburgh understands that the intent was not to imply all target analytes. Instead, it was meant to apply to analytes that are identified site contaminants and are identified as primary drivers for environmental decisions. For example, if vinyl chloride had already been identified as a special analyte of concern, a risk-driver, and this is discussed in the approved project documents, then vinyl chloride cannot exceed the LCS control limits, even marginally. In the more common situation where special analytes of concern have not been identified (e.g., investigation phase of work), the concept of marginal exceedances can be applied when evaluating LCSs for all target analytes. TestAmerica has requested written verification from USACE, confirming that we understand this concept correctly.

4. Acceptable Corrective Action for Noncompliant CCV – Section 5.5.10.e & Grey Box 36-37
   Sect. 5.5.10.e: If the continuing calibration verification results obtained are outside established acceptance criteria, corrective actions must be performed. If routine corrective action procedures fail to produce a second consecutive (immediate) calibration verification within acceptance criteria, then either the laboratory has to demonstrate acceptable performance after corrective action with two consecutive calibration verifications, or a new initial instrument calibration must be performed..
   Grey Box 37 Adds: The laboratory shall reanalyze CCVs and all samples analyzed since last successful calibration verification. If reanalysis is not possible, the

Example

laboratory must notify the client prior to reporting data associated with a noncompliant CCV. If these data are reported, the data must be qualified and explained in the case narrative.

Clarification: TestAmerica Pittsburgh does not employ the additional attempt to verify calibration as described in Sect. 5.5.10.e. Instead, the laboratory makes only one attempt to pass consecutive CCVs at the same concentration. If this attempt fails, the laboratory would take appropriate corrective actions and recalibrate.

5. Common Laboratory Contaminants – Section D.1.1.1, d, Grey Box D-1

QSM 4.1 Requirement: For DoD samples, the method blank will be considered to be contaminated if:

- The concentration of any target analyte in the blank exceeds 1/2 the reporting limit and is greater than 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater);
- The concentration of any common laboratory contaminant in the blank exceeds the reporting limit and is greater than 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater); or
- The blank result otherwise affects the samples results as per the test method requirements or the project-specific objectives.

Clarification: The common laboratory contaminants for TestAmerica Pittsburgh comprise the following:

Volatile Organics (8260): Methylene Chloride, Acetone and 2-Butanone.

GC/MS Semivolatiles (8270): Phthalates (bis-2-ethylhexylphthalate, di-n-octyl phthalate)

Metals (6010 & 6020): Copper, Iron, Lead (Trace ICP only), Zinc, Chromium and Tin.

Example

6. RPD Calculation – Section D.1.1.3.1.d, Grey Box D-13

QSM 4.1 Requirement: For DoD, RPD must be calculated as a comparison of measured concentrations.

Clarification: For most DoD projects, TestAmerica Pittsburgh's analytical reports are produced by a CLP-style report generator that does calculate the RPD as defined. However, some clients prefer the presentation on the reports generated directly from our LIMS, and if aliquots for the unspiked sample and/or replicate spikes (e.g., MS/MSD) are different then the RPD calculation will produce a different RPD value because it is based on recovered amounts, rather than recovered concentrations.

Attachment 1

Flagging Protocols for LIMS

TestAmerica QuantIMS Flagging for DoD QSM Version 4.1 – QC Program D4					
Condition	**Method Group**	**Flag ➔ Remap**	**DoD QSM Qualifier Equivalent**		**Qualifier Type**
Analyte < DL	All areas	ND ➔ U	U	Automatic	Not Applicable
Estim. value between MDL & RL	Inorganic areas	B ➔ J	J	Automatic	01
Estim. value between MDL & RL	Organic areas	J	J	Automatic	01
Result above working range	Organic areas	E	J	Manual	31
Result above working range	Inorganic areas	I	J	Manual	31
CCV failure	All areas	CV (new)	Q	Manual	31
Calibration blank > LOD	Inorganic areas	CB (new)	B	Manual	31
Method blank > MDL	Organic areas	B	B	Automatic	31
Method blank > MDL	Inorganic areas	J ➔ B	B	Automatic	31
LCS failure, flag sample result	All areas (*)	LC (new)	Q	Manual**	31
MS failure, flag on summary report	Organic areas	a	J	Automatic	07/08
MS failure, flag on summary report	Inorganic areas	N	J	Automatic	07/08
MS/MSD RPD failure (on summary)	Organic areas	p	J	Automatic	09
MS/MSD RPD failure (on summary)	Inorganic areas	*	J	Automatic	09
Duplicate RPD failure (on summary)	Inorganic areas	*	J	Automatic	09?
Internal standard failure (next to analytes in affected samples)	Organic areas	Z	Q	Manual	31
Surrogate standard failure (next to surrogate)	Organic areas	*	Q	Automatic	31
Confirm>40%RPD	Organic areas	PG	J	Manual	31
TIC	MSSEMI	N	N	Automatic - upload	31
EMPC	Dioxin	Q	None	Manual	31

TestAmerica QuantIMS Flagging for DoD QSM Version 4.1 – QC Program D4					
Condition	**Method Group**	**Flag ➔ Remap**	**DoD QSM Qualifier Equivalent**		**Qualifier Type**
ICSA/ICSAB (affected samples/analytes)	ICP Metals (MET)	AB (new)	Q	Manual	31
PDS (affected analyte/parent sample)	Metals	W	J	Manual	31

*Note: QuantIMS overflags, B applied when MB > MDL, rather than ½ RL. We will continue to overflag and narrate when B flag indicates DoD method blank criteria failure.
**Note: Request programming in QuantIMS to automatically qualify associated sample results.
Qualifier Type: are QuantIMS types that determine whether it is automatically applied or manually applied and under what conditions it shows up on the report.

TestAmerica QuantIMS Flagging for DoD QSM Version 4.1 – QC Program D4 Footnotes associated with Qualifiers			
Condition	**Method Group**	**Flag ➔ Remap**	**Footnote**
Analyte < DL	All areas	ND ➔ U	Analyte was not detected.
Estim. value between MDL & RL	Inorganic areas	B ➔ J	Estimated Result. Result is less than RL.
Estim. value between MDL & RL	Organic areas	J	Estimated Result. Result is less than RL
Result above working range	Organic areas	E	Estimated result. Result concentration exceeds the calibration range.
Result above working range	Inorganic areas	I	Estimated result. Result concentration exceeds the calibration range.
CCV failure	All areas	CV (new)	Quality control criteria failed (CCV)
Calibration blank > LOD	Inorganic areas	CB (new)	Blank contamination. The recorded result is associated with a contaminated calibration blank.
Method blank > MDL	Organic areas	B	Method blank contamination. The associated method blank contains the target analyte at a reportable level.

TestAmerica QuantIMS Flagging for DoD QSM Version 4.1 – QC Program D4 Footnotes associated with Qualifiers			
Method blank > MDL	Inorganic areas	J ➔ B	Method blank contamination. The analyte was found at a reportable level in the blank.
LCS failure, flag sample result	All areas	LC (new)	Quality control criteria failed (LCS)
MS failure, flag on summary report	Organic areas	a	Spiked analyte recovery is outside stated control limits.
MS failure, flag on summary report	Inorganic areas	N	Spiked analyte recovery is outside stated control limits.
MS/MSD RPD failure (on summary)	Organic areas	p	Relative percent difference (RPD) is outside stated control limits.
MS/MSD RPD failure (on summary)	Inorganic areas	*	Relative percent difference (RPD) is outside stated control limits.
Duplicate RPD failure (on summary)	Inorganic areas	*	Relative percent difference (RPD) is outside stated control limits.
Internal standard failure (next to analytes in affected samples)	Organic areas	Z➔Z	Quality control criteria failed (IS)
Surrogate standard failure (next to surrogate)	Organic areas	*	Surrogate recovery is outside stated control limits.
Confirm>40%RPD	Organic areas	PG	The relative percent difference between the original and confirmation analyses is greater than 40%.
TIC	MSSEMI	N	Estimated result. Analyte is a Tentative ly Identified Compound (TIC).
EMPC	Dioxin	Q	Estimated maximum possible concentration (EMPC).
ICSA/ICSAB (affected samples/analytes)	ICP Metals	AB (new)	Quality control criteria failed (ICSA and/or ICSAB)
PDS (affected analyte/parent sample)	Metals	W	Post digestion spike recovery is outside control limits.

Discussion:

Using the DoD QSM V4.1 generic qualifiers does not provide sufficient information to data validators to make decisions on the impact of the qualification on data quality and usability. Following the minimum qualifications required by the DoD QSM may result in the same flag being used to reflect multiple issues on one sample report. This leaves the data user no way to easily see the reason for the qualification. Fortunately the DoD QSM allows the use of different letters and more qualifiers.

The only remaps done for this program code are changing the “B” inorganics qualifier to “J” and “J” to “B.”

Existing QuantIMS qualifiers are used with a more detailed description than the generic qualifiers suggested by the DoD QSM V4.1.

The table shows the relationship of the qualifiers to be used by the lab to the associated DoD QSM V4.1 qualifier. Some new qualifiers (labeled as “new”) will be used to provide qualification required by the DoD QSM tables and provide more detailed description of the event.

Attachment 2- Organic Methods Target Analytes for DoD QSM 4.1		
Target Analyte	**Analytical Method**	**Test Matrix**
Organochlorine Pesticide		
4,4'-DDD	8081A/8081B	Water and Solid
4,4'-DDE	8081A/8081B	Water and Solid
4,4'-DDT	8081A/8081B	Water and Solid
Aldrin	8081A/8081B	Water and Solid
alpha-BHC	8081A/8081B	Water and Solid
alpha-Chlordane	8081A/8081B	Water and Solid
beta-BHC	8081A/8081B	Water and Solid
Chlordane (Technical)	8081A/8081B	Water and Solid
delta-BHC	8081A/8081B	Water and Solid
Dieldrin	8081A/8081B	Water and Solid
Endosulfan I	8081A/8081B	Water and Solid
Endosulfan II	8081A/8081B	Water and Solid
Endosulfan sulfate	8081A/8081B	Water and Solid
Endrin	8081A/8081B	Water and Solid
Endrin aldehyde	8081A/8081B	Water and Solid
Endrin ketone	8081A/8081B	Water and Solid
gamma-BHC (Lindane)	8081A/8081B	Water and Solid
gamma-Chlordane	8081A/8081B	Water and Solid
Heptachlor	8081A/8081B	Water and Solid
Heptachlor epoxide	8081A/8081B	Water and Solid
Methoxychlor	8081A/8081B	Water and Solid
Toxaphene	8081A/8081B	Water and Solid
Surrogates		
Decachlorobiphenyl (surr)	8081A/8081B	Water and Solid
Tetrachloro-m-xylene (TCMX) (surr)	8081A/8081B	Water and Solid
PCBs		
Aroclor 1016	8082/8082A	Water and Solid
Aroclor 1221	8082/8082A	Water and Solid
Aroclor 1232	8082/8082A	Water and Solid
Aroclor 1242	8082/8082A	Water and Solid
Aroclor 1248	8082/8082A	Water and Solid
Aroclor 1254	8082/8082A	Water and Solid
Aroclor 1260	8082/8082A	Water and Solid
Aroclor 1262	8082/8082A	Water and Solid
Aroclor 1268	8082/8082A	Water and Solid
Aroclor 1016/1260	8082/8082A	Water and Solid
Surrogates		

Attachment 2- Organic Methods Target Analytes for DoD QSM 4.1		
Target Analyte	**Analytical Method**	**Test Matrix**
Decachlorobiphenyl (surr)	8082/8082A	Water and Solid
Tetrachloro-m-xylene (surr)	8082/8082A	Water and Solid
Organophosphorus Pesticides		
Azinphos-methyl	8141A/8141B	Water and Solid
Bolstar (Sulprofos)	8141A/8141B	Water and Solid
Chlorpyrifos	8141A/8141B	Water and Solid
Coumaphos	8141A/8141B	Water and Solid
Demeton-O	8141A/8141B	Water and Solid
Demeton-S	8141A/8141B	Water and Solid
Demeton (total)	8141A/8141B	Water and Solid
Diazinon	8141A/8141B	Water and Solid
Dichlorvos (DDVP)	8141A/8141B	Water and Solid
Disulfoton	8141A/8141B	Water and Solid
Ethoprop	8141A/8141B	Water and Solid
Dimethoate	8141A/8141B	Water and Solid
EPN	8141A/8141B	Water and Solid
Ethyl parathion (Parathion)	8141A/8141B	Water and Solid
Famphur	8141A/8141B	Water and Solid
Fensulfothion	8141A/8141B	Water and Solid
Fenthion	8141A/8141B	Water and Solid
Malathion	8141A/8141B	Water and Solid
Mevinphos	8141A/8141B	Water and Solid
O,O,O-Triethyl phosphorothioate	8141A/8141B	Water and Solid
Parathion, methyl	8141A/8141B	Water and Solid
Phorate	8141A/8141B	Water and Solid
Ronnel	8141A/8141B	Water and Solid
Sulfotepp (Tetraethyldithiopyrophosphate)	8141A/8141B	Water and Solid
Stirophos (Tetrachlorovinphos)	8141A/8141B	Water and Solid
Thionazin		
Tokuthion (Protothiofos)	8141A/8141B	Water and Solid
Trichlornate	8141A/8141B	Water and Solid
Surrogates		
Tributyl phosphate (surr)	8141A/8141B	Water and Solid
Triphenyl phosphate (surr)	8141A/8141B	Water and Solid
Herbicides		
2,4,5-T	8151A	Water and Solid
2,4,5-TP (Silvex)	8151A	Water and Solid
2,4-D	8151A	Water and Solid

Attachment 2- Organic Methods Target Analytes for DoD QSM 4.1

Target Analyte	Analytical Method	Test Matrix
2,4-DB	8151A	Water and Solid
Dalapon	8151A	Water and Solid
Dicamba	8151A	Water and Solid
Dichlorprop	8151A	Water and Solid
Dinoseb	8151A	Water and Solid
MCPA	8151A	Water and Solid
MCPP	8151A	Water and Solid
Pentachlorophenol	8151A	Water and Solid
Surrogates		
2,4-Dichlorophenylacetic acid (DCAA) (surr)	8151A	Water and Solid
Volatile Organics by GC/MS		
1,1,1,2-Tetrachloroethane	8260B	Water and Solid
1,1,1-Trichloroethane	8260B	Water and Solid
1,1,2,2-Tetrachloroethane	8260B	Water and Solid
1,1,2-Trichloroethane	8260B	Water and Solid
1,1,2-Trichloro-1,2,2-trifluoroethane	8260B	Water and Solid
1,1-Dichloroethane	8260B	Water and Solid
1,1-Dichloroethene	8260B	Water and Solid
1,1-Dichloropropene	8260B	Water and Solid
1,2,3-Trichlorobenzene	8260B	Water and Solid
1,2,3-Trichloropropane	8260B	Water and Solid
1,2,4-Trichlorobenzene	8260B	Water and Solid
1,2,4-Trimethylbenzene	8260B	Water and Solid
1,2-Dibromo-3-chloropropane (DBCP)	8260B	Water and Solid
1,2-Dibromoethane (EDB)	8260B	Water and Solid
1,2-Dichlorobenzene	8260B	Water and Solid
1,2-Dichloroethane	8260B	Water and Solid
1,2-Dichloropropane	8260B	Water and Solid
1,2-Dichloroethene (total)	8260B	Water and Solid
1,3,5-Trimethylbenzene	8260B	Water and Solid
1,3-Dichlorobenzene	8260B	Water and Solid
1,3-Dichloropropane	8260B	Water and Solid
1,4-Dichlorobenzene	8260B	Water and Solid
2,2-Dichloropropane	8260B	Water and Solid
2-Butanone (MEK)	8260B	Water and Solid
2-Chlorotoluene	8260B	Water and Solid
2-Hexanone	8260B	Water and Solid

Attachment 2- Organic Methods Target Analytes for DoD QSM 4.1		
Target Analyte	**Analytical Method**	**Test Matrix**
4-Chlorotoluene	8260B	Water and Solid
4-Methyl-2-pentanone (MIBK)	8260B	Water and Solid
Acetone	8260B	Water and Solid
Benzene	8260B	Water and Solid
Bromobenzene	8260B	Water and Solid
Bromochloromethane	8260B	Water and Solid
Bromodichloromethane	8260B	Water and Solid
Bromoform	8260B	Water and Solid
Bromomethane	8260B	Water and Solid
Carbon disulfide	8260B	Water and Solid
Carbon tetrachloride	8260B	Water and Solid
Chlorobenzene	8260B	Water and Solid
Chloroethane	8260B	Water and Solid
Chloroform	8260B	Water and Solid
Chloromethane	8260B	Water and Solid
cis-1,2-Dichloroethene	8260B	Water and Solid
cis-1,3-Dichloropropene	8260B	Water and Solid
Cyclohexane	8260B	Water and Solid
Dibromochloromethane	8260B	Water and Solid
Dibromomethane	8260B	Water and Solid
Dichlorodifluoromethane	8260B	Water and Solid
Ethylbenzene	8260B	Water and Solid
Hexachlorobutadiene	8260B	Water and Solid
Isopropylbenzene	8260B	Water and Solid
m&p-Xylenes	8260B	Water and Solid
Methylene chloride	8260B	Water and Solid
Methyl-tert-butyl-ether (MTBE)	8260B	Water and Solid
Methyl acetate	8260B	Water and Solid
Methylcyclohexane	8260B	Water and Solid
Naphthalene	8260B	Water and Solid
n-Butylbenzene	8260B	Water and Solid
n-Propylbenzene	8260B	Water and Solid
o-Xylene	8260B	Water and Solid
p-Isopropyltoluene	8260B	Water and Solid
Sec-Butylbenzene	8260B	Water and Solid
Styrene	8260B	Water and Solid
Tert-Butylbenzene	8260B	Water and Solid
Tetrachloroethene	8260B	Water and Solid
Toluene	8260B	Water and Solid
trans-1,2-Dichloroethene	8260B	Water and Solid

Attachment 2- Organic Methods Target Analytes for DoD QSM 4.1		
Target Analyte	**Analytical Method**	**Test Matrix**
trans-1,3-Dichloropropene	8260B	Water and Solid
Trichloroethene	8260B	Water and Solid
Trichlorofluoromethane	8260B	Water and Solid
Vinyl chloride	8260B	Water and Solid
Total Xylenes	8260B	Water and Solid
Surrogates		Water and Solid
1,2-Dichloroethane-d4 (surr)	8260B	Water and Solid
4-Bromofluorobenzene (surr)	8260B	Water and Solid
Dibromofluoromethane (surr)	8260B	Water and Solid
Toluene-d8 (surr)	8260B	Water and Solid
1,2-Dichlorobenzen-d4 (surr)	8260B	Water and Solid
Fluorobenzene (surr)	8260B	Water and Solid
Semivolatile Organics by GC/MS		
1-Methylnaphthalene	8270C/8270D	Water and Solid
1,1'-Biphenyl	8270C/8270D	Water and Solid
1,2,4-Trichlorobenzene	8270C/8270D	Water and Solid
1,2-Dichlorobenzene	8270C/8270D	Water and Solid
1,2-Diphenylhydrazine	8270C/8270D	Water and Solid
1,3-Dichlorobenzene	8270C/8270D	Water and Solid
1,4-Dichlorobenzene	8270C/8270D	Water and Solid
1,4-Dioxane	8270C/8270D	Water and Solid
2,2-oxybis (1-chloropropane)	8270C/8270D	Water and Solid
2,3,4,6-Tetrachlorophenol	8270C/8270D	Water and Solid
2,3,5,6-Tetrachlorophenol	8270C/8270D	Water and Solid
2,4,5-Trichlorophenol	8270C/8270D	Water and Solid
2,4,6-Trichlorophenol	8270C/8270D	Water and Solid
2,4-Dichlorophenol	8270C/8270D	Water and Solid
2,4-Dimethylphenol	8270C/8270D	Water and Solid
2,4-Dinitrophenol	8270C/8270D	Water and Solid
2,4-Dinitrotoluene	8270C/8270D	Water and Solid
2,6-Dichlorophenol	8270C/8270D	Water and Solid
2,6-Dinitrotoluene	8270C/8270D	Water and Solid
2-Chloronaphthalene	8270C/8270D	Water and Solid
2-Chlorophenol	8270C/8270D	Water and Solid
2-Methylnaphthalene	8270C/8270D	Water and Solid
2-Methylphenol (o-cresol)	8270C/8270D	Water and Solid
2-Nitroaniline	8270C/8270D	Water and Solid
2-Nitrophenol	8270C/8270D	Water and Solid

Attachment 2- Organic Methods Target Analytes for DoD QSM 4.1		
Target Analyte	**Analytical Method**	**Test Matrix**
3,3-Dichlorobenzidine	8270C/8270D	Water and Solid
3-Methylphenol	8270C/8270D	Water and Solid
4-Methylphenol	8270C/8270D	Water and Solid
3/4-Methylphenol (m/p-cresol)	8270C/8270D	Water and Solid
3-Nitroaniline	8270C/8270D	Water and Solid
4,6-Dinitro-2-methylphenol	8270C/8270D	Water and Solid
4-Bromophenyl phenyl ether	8270C/8270D	Water and Solid
4-Chloro-3-methylphenol	8270C/8270D	Water and Solid
4-Chloroaniline	8270C/8270D	Water and Solid
4-Chlorophenyl phenyl ether	8270C/8270D	Water and Solid
4-Nitroaniline	8270C/8270D	Water and Solid
4-Nitrophenol	8270C/8270D	Water and Solid
Acenaphthene	8270C/8270D	Water and Solid
Acenaphthylene	8270C/8270D	Water and Solid
Aniline	8270C/8270D	Water and Solid
Anthracene	8270C/8270D	Water and Solid
Benzidine	8270C/8270D	Water and Solid
Benzo(a)anthracene	8270C/8270D	Water and Solid
Benzo(a)pyrene	8270C/8270D	Water and Solid
Benzo(b)fluoranthene	8270C/8270D	Water and Solid
Benzo(ghi)perylene	8270C/8270D	Water and Solid
Benzo(k)fluoranthene	8270C/8270D	Water and Solid
Benzoic Acid	8270C/8270D	Water and Solid
Benzyl alcohol	8270C/8270D	Water and Solid
Bis(2-chloroethoxy)methane	8270C/8270D	Water and Solid
Bis(2-chloroethyl)ether	8270C/8270D	Water and Solid
Bis(2-ethylhexyl)phthalate	8270C/8270D	Water and Solid
Bis(2-chloroisopropyl)ether	8270C/8270D	Water and Solid
Butyl benzyl phthalate	8270C/8270D	Water and Solid
Carbazole	8270C/8270D	Water and Solid
Caprolactam	8270C/8270D	Water and Solid
Chrysene	8270C/8270D	Water and Solid
Dibenzo(a,h)anthracene	8270C/8270D	Water and Solid
Dibenzofuran	8270C/8270D	Water and Solid
Diethyl phthalate	8270C/8270D	Water and Solid
Dimethyl phthalate	8270C/8270D	Water and Solid
Di-n-butyl phthalate	8270C/8270D	Water and Solid
Di-n-octyl phthalate	8270C/8270D	Water and Solid
Fluoranthene	8270C/8270D	Water and Solid

Attachment 2- Organic Methods Target Analytes for DoD QSM 4.1		
Target Analyte	**Analytical Method**	**Test Matrix**
Fluorene	8270C/8270D	Water and Solid
Hexachlorobenzene	8270C/8270D	Water and Solid
Hexachlorobutadiene	8270C/8270D	Water and Solid
Hexachlorocylcopentadiene	8270C/8270D	Water and Solid
Hexachloroethane	8270C/8270D	Water and Solid
Indeno(1,2,3-cd)pyrene	8270C/8270D	Water and Solid
Isophorone	8270C/8270D	Water and Solid
Naphthalene	8270C/8270D	Water and Solid
Nitrobenzene	8270C/8270D	Water and Solid
N-Nitrosodimethylamine	8270C/8270D	Water and Solid
N-Nitroso-di-n-propylamine	8270C/8270D	Water and Solid
N-Nitrosodiphenylamine	8270C/8270D	Water and Solid
N-Nitrosopyrrolidine	8270C/8270D	Water and Solid
Pentachlorophenol	8270C/8270D	Water and Solid
Phenanthrene	8270C/8270D	Water and Solid
Phenol	8270C/8270D	Water and Solid
Pyrene	8270C/8270D	Water and Solid
Pyridine	8270C/8270D	Water and Solid
Surrogates		Water and Solid
2,4,6-Tribromophenol (surr)	8270C/8270D	Water and Solid
2-Fluorobiphenyl (surr)	8270C/8270D	Water and Solid
2-Fluorophenol (surr)	8270C/8270D	Water and Solid
Nitrobenzene-d5 (surr)	8270C/8270D	Water and Solid
Phenol-d5/d6 (surr)	8270C/8270D	Water and Solid
Terphenyl-d14 (surr)	8270C/8270D	Water and Solid
Polynuclear Aromatic Hydrocarbons (HPLC)		
Acenaphthene	8310	Water and Solid
Acenaphthylene	8310	Water and Solid
Anthracene	8310	Water and Solid
Benzo(a)anthracene	8310	Water and Solid
Benzo(a)pyrene	8310	Water and Solid
Benzo(b)fluoranthene	8310	Water and Solid
Benzo(ghi)perylene	8310	Water and Solid
Benzo(k)fluoranthene	8310	Water and Solid
Chrysene	8310	Water and Solid
Dibenzo(a,h)anthracene	8310	Water and Solid
Fluoranthene	8310	Water and Solid
Fluorene	8310	Water and Solid

Attachment 2- Organic Methods Target Analytes for DoD QSM 4.1		
Target Analyte	**Analytical Method**	**Test Matrix**
Indeno(1,2,3-cd)pyrene	8310	Water and Solid
Naphthalene	8310	Water and Solid
Phenanthrene	8310	Water and Solid
Pyrene	8310	Water and Solid
Surrogates	8310	Water and Solid
Benzo(e)Pyrene	8310	Water and Solid
Terphenyl-d14	8310	Water and Solid
1,2-Dibromoethane (EDB)	8011	Water
1,2-Dibromo-3-chloropropane (DBCP)	8011	Water
Surrogate		
1,1,1,2-Tetrachloroethane	8011	Water

Attachment 3 - Inorganic Analytes for DoD QSM 4.1		
Analyte	**Method**	**Matrix**
Aluminum	6010B/6010C, 6020/6020A	Water and Solid
Antimony	6010B/6010C, 6020/6020A	Water and Solid
Arsenic	6010B/6010C, 6020/6020A	Water and Solid
Barium	6010B/6010C, 6020/6020A	Water and Solid
Beryllium	6010B/6010C, 6020/6020A	Water and Solid
Boron	6010B/6010C, 6020/6020A	Water and Solid
Cadmium	6010B/6010C, 6020/6020A	Water and Solid
Calcium	6010B/6010C, 6020/6020A	Water and Solid
Chromium	6010B/6010C, 6020/6020A	Water and Solid
Cobalt	6010B/6010C, 6020/6020A	Water and Solid
Copper	6010B/6010C, 6020/6020A	Water and Solid
Iron	6010B/6010C, 6020/6020A	Water and Solid
Lead	6010B/6010C, 6020/6020A	Water and Solid
Magnesium	6010B/6010C, 6020/6020A	Water and Solid
Manganese	6010B/6010C, 6020/6020A	Water and Solid
Molybdenum	6010B/6010C, 6020/6020A	Water and Solid
Nickel	6010B/6010C, 6020/6020A	Water and Solid

Attachment 3 - Inorganic Analytes for DoD QSM 4.1		
Potassium	6010B/6010C, 6020/6020A	Water and Solid
Selenium	6010B/6010C, 6020/6020A	Water and Solid
Silica	6010B/6010C, 6020/6020A	Water and Solid
Silicon	6010B/6010C, 6020/6020A	Water and Solid
Silver	6010B/6010C, 6020/6020A	Water and Solid
Sodium	6010B/6010C, 6020/6020A	Water and Solid
Strontium	6010B/6010C, 6020/6020A	Water and Solid
Thallium	6010B/6010C, 6020/6020A	Water and Solid
Tin	6010B/6010C, 6020/6020A	Water and Solid
Titanium	6010B/6010C, 6020/6020A	Water and Solid
Vanadium	6010B/6010C, 6020/6020A	Water and Solid
Zinc	6010B/6010C, 6020/6020A	Water and Solid
Mercury	7470A/7471A/7471B	Water and Solid
Bromide	9056A	Water and Solid
Chloride	9056A	Water and Solid
Fluoride	9056A	Water and Solid
Nitrate	9056A	Water and Solid
Nitrite	9056A	Water and Solid
Phosphate	9056A	Water and Solid
Sulfate	9056A	Water and Solid
Total cyanide	9010/9012	Water and Solid
Oil and Grease	1664A/9070A, 9071B	Water and Solid
Hexavalent Chromium	3060A/7196A	Water and Solid
Total Organic Carbon	9060	Water
Total Organic Carbon	Lloyd Kahn	Solid
Sulfide	9030B and 9034	Water and Solid
pH	9040	Water
pH	9045	Solid
Flash Point	1020/ASTM D3278-96	Water and Waste
Flash Point	1010/ASTM D93-85	Water and Waste
Percent Moisture	SM 2540G	Solid