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DEPARTMENT OF THE NAVY  
NORTHERN DIVISION  
NAVAL FACILITIES ENGINEERING COMMAND  
PHILADELPHIA PENNSYLVANIA 19112-5094

IN REPLY REFER TO  
5090  
Ser 1080/1421/RF  
30 July 1990

MEMORANDUM

FOR THE MEMBERS OF THE TECHNICAL REVIEW COMMITTEE (TRC) REMEDIAL INVESTIGATION/FEASIBILITY STUDY AT NETC NEWPORT, RI

Enclosed is a copy of the minutes from the fourteenth Technical Review Committee (TRC) meeting held on 21 June 1990 at NETC Newport, RI. Any comments or corrections may be forwarded to Northern Division prior to the next meeting.

The fifteenth Technical Review Committee Meeting is scheduled for 10:00 on Thursday 13 September 1990 at NETC Newport, RI. Topics for discussion will include the Final Community Relations Plan, the removal project of oily soils at Melville North Landfill (site 02) and status of remaining fieldwork.

The 13 September 1990 meeting will be held at the Officers Club located near the main gate and pass office. If there are any questions concerning the meeting please contact me at (215) 897-6431. It is suggested that you contact Ms. Rachel Marino at NETC Newport if you need directions to the Officers Club. Ms. Marino can be reached at (401) 841-3735.

Sincerely,

R. H. FISH  
Remedial Project Manager  
By Direction of the Commanding Officer

DISTRIBUTION  
TECHNICAL REVIEW COMMITTEE MEMBERS

- US EPA Region I, Ann Fenn
- US EPA Region I, Carol Cody
- RI DEM, Jeffrey Crawford
- RI DEM, Warren S. Angell, II
- RI DEM, Joseph Migliori
- Narragansett Bay Project, Jennifer Martin
- US EPA-ERL, Wayne Munns
- City of Newport, RI, Roy Anderson
- Planning Board of Portsmouth, Joe Marshall
- Town of Middletown, Fire Chief, Donald Ardito
- Town of Middletown, Charles Silvia
- NETC Newport, Rachel Marino
- NETC Newport, Mary Silvia

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FOR THE MEMBERS OF THE TECHNICAL REVIEW SCOMMITTEE (TRC) REMEDIAL  
INVESTIGATION/FEASIBILITY STUDY AT NETC NEWPORT, RI

NETC Newport, LCDR Robert Humphreys  
NETC Newport, Robert Moore  
NETC Newport, LCDR Howard Goodman  
Hood Enterprises, Inc. Dean Coker  
TRC-ECI, Robert Smith  
TRC-ECI, Jim Peronto  
Newport Fire Dept., Capt. David Lemler  
Save the Bay, Director, Mr. Curt Spalding

Copy to:

Naval Ocean Systems Center, Bob Johnston  
EPA-ERL Narragansett, Tim Gleason  
US EPA Region 1, Douglas Gutro  
Chairperson, Portsmouth Citizen Advisory Committee

*Internal Copy:*

*Code 1422/FL*

*Code 1421/RF*



TECHNICAL REVIEW COMMITTEE MEETING MINUTES

NAVY INSTALLATION RESTORATION PROGRAM  
NETC, Newport, Rhode Island

JUNE 21, 1990

TRC Environmental Consultants, Inc.  
Contract No. N62472-86-C-1282  
TRC Project No. 5383-N81-00

Prepared by:

Robert C. Smith, P.E.  
Program Manager  
and  
James Peronto, P.E.  
Project Manager

Prepared for:

Russell Fish  
Project Manager  
U.S. Navy - Northern Division



MINUTES OF THE THIRTEENTH TRC MEETING

The Fourteenth Technical Review Committee meeting (TRC) for the Newport Installation Restoration Program (IRP) studies was held at NETC in Newport, Rhode Island from 10:00 a.m. to 11:45 a.m. on June 21, 1990. The 21 June 1990 meeting was held at the Officers Club located near the main gate and pass office. The primary objective was to discuss the status of the Remedial Investigation (RI) work completed at Site 01, McAllister Point; and the field investigations currently underway at the remaining sites: Melville North Landfill (Site 02), Old Fire Fighting Training Area (Site 09), Tank Farm Number 4 (Site 12) and Tank Farm Number 5 (Site 13); the Community Relations Plan; the Oil-Soaked Piles - Melville North Landfill removal status and the Closure Plan for Tanks 53 and 56 (Tank Farm 5). Also, coordination efforts with the USEPA Region I and their oversight contractor (CDM-Federal Programs) were discussed. The TRC meeting attendees are listed on Attachment A.



SUMMARY OF FIELD ACTIVITIES  
COMPLETED AT MCALLISTER POINT LANDFILL  
(As of June 20, 1990)

RI/FS PROJECT

It was reported by Jim Peronto, Project Manager for TRC-ECI, that a deep well (MW-1D) and three shallow wells (MW-1S, MW-3S and MW-5S) had been vandalized (week of 6 June, 1990) at Tank Farm Four. All wells were re-developed and the depths of the wells checked. Cement had been poured down the deep well and had "set-up" to a depth of 7 feet. Approximately 8 feet of open screen remains. Measurements indicate that the pH of ground water has not been impacted to date. The NETC base security has been notified and increased surveillance is anticipated. A recommendation will be provided by TRC-ECI regarding the use of this well for study purposes. The remaining three wells appear to be unaffected. Special "heavy-duty" security locks and well covers will be substituted for the typical protective measures.

The Remedial Investigation field work is substantially complete at Site 01 - McAllister Point Landfill. The work program was described in the RI/FS Work Plan and consisted of the following activities:

- Land survey
- Geophysical Surveys
- Surface Soil Sampling
- Test Borings
- Monitoring Wells

Off-shore sampling will be scheduled for summer (1990). All laboratory data (Site 01) has been sent to Environmental Standards Inc. for required data validation.

TRC-ECI is generally meeting the Project Schedule for the remaining RI field program and associated laboratory analysis and data validation effort.

A summary of field activities is presented below:

Site reconnaissance work included a detailed visual survey, ambient air and radiological surveys, geophysical survey and soil gas surveys (Sites 09, 12 and 13).

Soil Gas Surveys are completed for:

- Site 09 - Old Fire Fighting Training Area
- Site 12 - Tank Farm Four
- Site 13 - Tank Farm Five

*How was this determine?*

At Sites 12 and 13 it was determined in the field that a soil gas survey at shallow depth, typically 1 to 3 feet, would not provide meaningful data. Therefore, small diameter borings (2½" I.D.) were drilled to approximately 10 to 15 feet and samples obtained for field GC analysis.



• Surface Soils:

- Site 02 - Melville North Landfill
- Site 09 - Old Fire Fighting Training Area
- Site 12 - Tank Farm Four
- Site 13 - Tank Farm Five

Surface soil sampling at Sites 02 and 09 is complete (split samples with USEPA oversight contractor - CDM-Federal Programs). Surface soil samples were collected at two locations at each tank farm site for the purpose of splitting samples with USEPA. Remaining surface soil sampling at the tank farms were conducted as required in the Field Sampling Plan.

Test Pits:

- Site 02 - Melville North Landfill

Test pitting is complete. Samples split with USEPA.

Test Borings:

- Site 02 - Melville North Landfill
- Site 09 - Old Fire Fighting Training Area

Test borings complete at Sites 02 and 09. Borings completed at tank farms in connection with monitor well construction. Samples from selected borings (Sites 12 and 13) were split with USEPA. Samples from test borings at Sites 02 and 09 were not split with USEPA. However, USEPA did collect a sample from a split spoon retrieved at boring MW-1 (Depth 0'-2') at Site 9. This sample was taken from a previously-filled "ziploc" baggie and placed directly into glass sample containers using a dedicated stainless-steel spoon. It will be analyzed for dioxins and furans.

Monitoring Wells:

- Site 02 - Melville North Landfill
- Site 09 - Fire Fighting Training Area
- Site 12 - Tank Farm Four
- Site 13 - Tank Farm Five

Monitoring well installation is underway. Complete at Sites 09, 12 and 13.

Surface Water, Sediments, and Mussels:

- Site 01 - McAllister Point Landfill
- Site 02 - Melville North Landfill
- Site 09 - Old Fire Fighting Training Area
- Site 12 - Tank Farm Four
- Site 13 - Tank Farm Five

The surface water and sediment sampling at Tank Farms Four and Five was conducted for June 7 and June 8, 1990 to allow for split sampling with USEPA. Off-shore biota/sediment samples to be obtained during the July 1990 time frame.



Tanks and Structures:

- Site 12 - Tank Farm Four
- Site 13 - Tank Farm Five

Sampling scheduled for week of 11 June 1990 and is continuing through week of July 2, 1990. Samples will be split with USEPA.

Well Development

- Site 01 - McAllister Point Landfill
- Site 02 - Melville North Landfill
- Site 09 - Old Fire Fighting Training Area
- Site 12 - Tank Farm Four
- Site 13 - Tank Farm Five

All wells have been developed in accordance with procedures discussed in the approved Field Sampling Plan (combination surge block techniques and pumping).

*Follow-up  
in FSP.*

Well Permeability Testing

- Site 01 - McAllister Point Landfill
- Site 02 - Melville North Landfill
- Site 09 - Old Fire Fighting Training Area
- Site 12 - Tank Farm Four
- Site 13 - Tank Farm Five

Well permeability testing will be conducted the week of July 16, 1990. Permeability test methods will be in accordance with procedures discussed in the approved Field Sampling Plan (see Table 1 - Permeability Test Methods).

Ground Water Sampling

- Site 01 - McAllister Point Landfill
- Site 02 - Melville North Landfill
- Site 09 - Old Fire Fighting Training Area
- Site 12 - Tank Farm Four
- Site 13 - Tank Farm Five

Ground water samples have been collected from Site 01. Ground water samples will be collected from all of the newly installed monitoring wells and the previously installed (existing) wells at each site (Sites 02, 09, 12 and 13). Ground water samples will be collected the week of July 16, 1990. Samples from selected wells will be split with USEPA on July 18 and July 19, 1990.

TANKS 53 AND 56 - STATUS

*obtain a copy of work plan*

A work program for a ground water assessment has been developed (NETC and RIDEM).

A fee proposal for the work to be performed by TRC-ECI was submitted on May 30, 1990 and will be negotiated with NORTHDIV as soon as possible. It is anticipated that negotiations will be conducted in late July. Well installation is expected by late August or September, 1990.



## MEEVILLE NORTH LANDFILL - OIL-SOAKED PILES

Twelve additional soil samples were obtained on April 4, 1990 to characterize the oily soil piles in the Melville North Landfill for disposal. Samples were analyzed for EP Toxicity for 8 heavy metals, volatile organic compounds (standard Method 8240) PCBs, reactivity (including cyanide and sulfide), corrosivity and flashpoint. Based on results of the laboratory analysis, these soils do not exhibit any of the characteristics of a hazardous waste.

Jeff Crawford, RIDEM, asked if Total Petroleum Hydrocarbon (TPH) analysis was conducted on the soil pile samples. The Scope of Work requirements (see Technical Review Committee Meeting minutes dated January 11, 1990) did not require TPH analysis. The scope was developed based on disposal characterization requirements of the NETC selected disposal site.

## COMMUNITY RELATIONS PLAN - STATUS

The CRP prepared for the Installation Restoration Program - NETC had been finalized (January 1990). However, additional comments have been provided by EPA - Region I that are now under consideration (March 30, 1990). An update on the status of the CRP was presented by Mary Silvia - Public Affairs Officer for NETC.

Final USEPA - Region I review (Addendum to Comments) have been received by NETC (June 20, 1990). A suggestion by USEPA to release the CRP for public comment is being evaluated by NETC. The Public Affairs Officer will be briefing the command regarding community relations and a final decision relative to a comment period for the Final CRP is expected in the near future.

## OTHER ISSUES

Off-shore sampling/analysis to be conducted adjacent to Sites 01, 02 and 09. Although NORTHDIV discussed with Naval Ocean Systems Center (NOSC) the potential for a concurrent study of Narragansett Bay, it was decided that this would not be performed at this time. However, NOSC will provide technical input as appropriate during the proposed off-shore investigations.

NOSC believes that the analytical methods proposed to be used for the marine sediment analysis are not really appropriate for marine samples because of difficulties with sample matrix interferences. The detection limits of Contract Laboratory Protocol (CLP) methods are not appropriate for determining the level of contaminants that may be harmful to marine organisms. For example, the CLP detection limit for copper in seawater is 25 µg/l, while the EPA water quality criteria for copper is 3 µg/l. However, for the purposes of the current study, use of CLP methods may be appropriate (concentration of contaminants in the areas to be sampled are high above CLP detection limits, based on the previous data). The Standard Operating Procedure for tissue extraction provided by NOSC is included as Attachment B.



NEXT MEETING

The next TRC meeting will be held on September 13, 1990 at the NETC Officers Club at 10:00 a.m.



# ATTACHMENT A

TECHNICAL REVIEW COMMITTEE MEETING  
NETC NEWPORT RHODE ISLAND  
June 21, 1990

	Name	Organization	Phone
1.	RUSSELL FISH	NORTH DIV NAVFAC	[REDACTED]
2.	RACHEL MARINO	NETC - PWD	[REDACTED]
3.	Robert K Johnston	NOSC San Diego	[REDACTED]
4.	Joe Marshall	Town of Portsmouth	[REDACTED]
5.	Howard Goodman	NETC - SJTB	[REDACTED]
6.	LCDR R. HUMPHREYS	NETC - PWD	[REDACTED]
7.	Patricia J. Chalfant	North Div - Counsel Office	[REDACTED]
8.	ROBERT C. SMITH	TRC - ECI	[REDACTED]
9.	JIM PERONTO	TRC - ECI	[REDACTED]
10.	FRANCISCO A. LAGRECA	NORTH DIV NAVFAC	[REDACTED]
11.	Michael Kulbersh	CDM Federal Programs	[REDACTED]
12.	Leuel Uedy	EPA-Region 1	[REDACTED]
13.	Douglas S. Gutro	EPA Region 1	[REDACTED]
14.	JEFFREY CRAWFORD	RIDEM - DAHM	[REDACTED]
15.	Mary K Silvia	NETC - PAO	[REDACTED]
16.	Paul Kelpa	RIDEM - DAHM	[REDACTED]
17.			
18.			
19.			
20.			
21.			
22.			
23.			
24.			



## ATTACHMENT B

### ERLN CHEMISTRY GROUP STANDARD OPERATING PROCEDURE FOR TISSUE EXTRACTION - DEPARTMENT

#### 1.0 OBJECTIVES

The objective of this document is to define the standard operating procedure for the extraction of semi-volatile organic compounds from marine tissue samples. The extracts will be further cleaned up by silica gel chromatography procedures prior to analysis by gas chromatography (GC) or gas chromatography/mass spectrometry (GC/MS).

#### 2.0 MATERIALS AND EQUIPMENT

Apparatus for homogenizing tissue

Brinkman Polytron  
100- or 150-ml glass centrifuge tubes

Apparatus for determining weight and dry weight

Top-loading balance capable of weighing to 0.01 g  
Aluminum weighing pans  
Stainless steel spatula  
Drying oven maintained at 105-120°C

Kuderna-Danish (K-D) apparatus

Reservoir, 250 or 500 ml  
Snyder column, three-ball macro  
Micro-snyder column  
Concentrator tube, 10 ml

Heating mantle block capable of heating to 100°C, in fume hood

Tube heater, Kontes or equivalent, capable of 110°C

Boiling beads, solvent rinsed and muffled at 450°C

Glass graduated cylinders, 100- and 500-ml

Glass erlenmeyer flasks, 250- and 500-ml

Glass separatory funnels, 1000-ml

Borosilicate glass vials with Teflon-lined screw caps, 1.8 ml



Microliter syringes or micropipets, solvent rinsed

Reagents

Pentane, pesticide grade or equivalent  
Acetonitrile, pesticide grade or equivalent

Sodium sulfate-anhydrous, reagent grade. Heated to 750°C for at least 4 hours, then cooled and stored in a tightly-sealed glass container at room temperature.

Internal Standards, to be added to each sample during extraction.

3.0 METHODS

3.1 Weigh approximately 10.0g of sample into a solvent rinsed centrifuge tube. Weigh approximately 1.0 gram into a preweighed aluminum pan for dry/wet determination.

3.2 Add Internal Standards: OCN for PCB Analysis; Gamma Chlordene for Pesticides; and d12 Benzo(a)Anthracene and d10 Phenanthrene mix for PAHs. The amount of IS added is dependent on the expected contaminant concentrations and should be equivalent to those concentrations.

3.3 Add 50 ml acetonitrile.

3.4 Polytron the samples for 20 seconds, at a speed setting of ~ 5. Centrifuge for 10 minutes at 1750 rpm and pour off the supernatant into a separatory funnel containing 300ml pentane extracted deionized water (DI). Repeat this step two more times.

3.5 Back extract the DI/ACETONITRILE phase in the separatory funnel with 3X 50 ml pentane. After each addition of pentane has been shaken, draw off the bottom layer into a 500 ml erlenmeyer flask. Decant the Pentane layer into a 250 ml erlenmeyer flask by pouring it out the top of the separatory funnel. This way the transfer of water into the pentane extract will be avoided.

3.6 Transfer the water layer from the 500 ml erlenmeyer flask back into the separatory funnel for every addition of pentane. Rinse the 500 ml flask 3 x 5 ml with Pentane and add the rinses to the separatory funnel.

3.7 Combine the pentane extracts and dry over Sodium Sulfate.

3.8 Transfer the sample to a 500 ml round bottom flask.



Rinse the erlenmeyer sample flask 3x with pentane and add the rinses to the round bottom flask. Add several boiling beads. Fit the round bottom with Kuderna-Danish (K-D) and Snyder column apparatus and reduce the volume to about 10 ml.

3.9 Transfer the sample to a 10 ml graduated concentrator tube and rinse the round bottom flask 3x with 5 ml pentane. Add the rinses to the concentrator tube. Add an ebulator to the tube and fit with a micro-Snyder column and reduce the volume to about 0.5 ml in a tube heater. Adjust the volume to 1 ml with pentane. Remove 0.1 ml of sample into a preweighed aluminum pan for lipid weight determination. Allow it to dry at room temperature for at least 24 hours. Record the weight of the pan plus the sample. Transfer the 0.9 ml sample to a vial with a screw cap with a micropipet.

3.10 Fractionate the sample following the Column Chromatography SOP.



ERLN CHEMISTRY GROUP  
STANDARD OPERATING PROCEDURE FOR COLUMN CHROMATOGRAPHY

1.0 OBJECTIVES

The objective of this document is to define the standard operating procedure for the preparation of columns for the cleanup and chemical class separation of semi-volatile organic compounds from marine tissue samples. The extract fractions will be analyzed by gas chromatography (GC) or gas chromatography/mass spectrometry (GC/MS).

2.0 MATERIALS AND EQUIPMENT

9.5-mm ID X 45-cm glass chromatography column with 200 ml reservoir

Apparatus for determining weight

Top-loading balance capable of weighing to 0.01 g

Kuderna-Danish (K-D) apparatus

Reservoir, 250 or 500 ml  
Snyder column, three-ball macro  
Micro-snyder column  
Concentrator tube, 10 ml

Heating mantle block capable of heating to 100°C, in fume hood

Tube heater, KONTES or equivalent, capable of 110°C

Tumbler, ball-mill

Boiling beads, solvent rinsed and muffled at 450°C

Glass graduated cylinders, 100- and 500-ml

Glass round bottom flasks, 250- or 500-ml

Glass beakers, 50-ml

Borosilicate glass vials with Teflon-lined screw caps, 2-ml

Micropipets, solvent rinsed or muffled at 400°C



## Reagents

Pentane, pesticide grade or equivalent  
Methylene Chloride (CH<sub>2</sub>Cl<sub>2</sub>), pesticide grade or equivalent  
Hexane, pesticide grade or equivalent  
Heptane, pesticide grade or equivalent  
Deionized water, pentane-extracted  
  
BioSil A silicic acid, 100-200 mesh  
  
Glass wool, silanized

## 3.0 METHODS

### 3.1 Silica gel preparation

3.1.1 Approximately 150 grams of fully activated silica gel is accurately weighed and transferred to a glass jar.

3.1.2 The silica gel is deactivated by adding 7.5% (weight basis) of pentane-extracted deionized water. The water is weighed accurately and an appropriate amount is added dropwise, ~ 1 ml at a time, to the silica gel. After the water has been added, the jar is hand-shaken vigorously.

3.1.3 The glass jar is then placed on a ball-mill tumbler and allowed to tumble overnight.

3.1.4 After tumbling, the jar is removed from the tumbler. The silica gel is stored tightly sealed in the jar at room temperature until use.

### 3.2 Column preparation

3.2.1 The glass columns are set up in ring stands in a fume hood.

3.2.2 Glass wool, sufficient to create a 1 cm thick plug in the column is placed into the reservoir of the column. A glass rod is used to push the glass wool to the bottom of the column.

3.2.3 11.5 g of the 7.5% deactivated silica gel is weighed out in a beaker. Approximately 30 ml of CH<sub>2</sub>Cl<sub>2</sub> is added to the beaker to form a slurry. The slurry is then carefully poured into the column. The beaker is rinsed with additional CH<sub>2</sub>Cl<sub>2</sub>, as are the inner walls of the reservoir to ensure all silica is introduced to the column. The total volume of CH<sub>2</sub>Cl<sub>2</sub> should be approximately 50 ml.



3.2.4 The column is allowed to drip, with the eluate being collected and discarded. When the level of the  $\text{CH}_2\text{Cl}_2$  just reaches the top of the silica gel, 50 ml of pentane is slowly added to the column. This eluate is also collected and discarded.

### 3.3 Chemical class separations

3.3.1 The 0.9 ml sample extract is introduced to the column just as the pentane rinse level reaches the silica gel. The vial is then rinsed with an additional 1 ml of pentane which is also introduced to the column just before the silica gel is exposed. The eluate is collected in a clean round bottom flask.

3.3.2 As the sample rinse level reaches the silica gel, 45 ml of pentane is added to the column. The eluate is collected as the F-1 fraction in a clean round bottom flask.

3.3.3 As the pentane level reaches the top of the silica, 36 ml of a 70:30 (volume:volume) pentane: $\text{CH}_2\text{Cl}_2$  mixture is introduced to the column. The flask is replaced with a clean one, and the F-2 fraction is collected. After collection, the flasks are kept tightly capped with aluminum foil. At no time should the column flow rate exceed 6 ml/min.

3.3.4 After the F-2 fraction has been collected from the column, the flasks are fitted with a K-D and Snyder column. Boiling beads are added to the flask. The fractions are then placed on a heating mantle and the solvent is reduced to approximately 10 ml. The samples are then solvent-exchanged to hexane and concentrated to about 10 ml. The samples are then removed from the heating mantle and allowed to cool for at least 30 minutes.

3.3.5 The cooled fractions are then transferred to graduated concentrator tubes fitted with micro-Snyder columns and ebullators. Each flask is rinsed with heptane, and the rinse is then added to the sample. The tubes are then placed in a tube heater which is set at  $110^\circ\text{C}$ . An aluminum foil cape is fitted to surround all tubes in the heater. The solvent is then reduced to approximately 0.5 ml and the tubes removed from the heater. Each ebullator is rinsed with heptane and the final volume of the each fraction is adjusted to 1.0 ml with heptane.

3.3.6 The fractions are then transferred to bor silicate glass vials fitted with Teflon-lined screw caps for storage until analysis.



**ERLN CHEMISTRY GROUP  
GCMS STANDARD OPERATING PROCEDURE**

**1.0 OBJECTIVES**

The objective of this document is to define the standard procedure for analyzing marine environmental samples using GCMS in electron impact/positive ion mode.

**2.0 EQUIPMENT USED**

Finnigan MAT 4530 GCMS system with PPINICI, Data General Nova 3 computer and Control Data CMD disk drive utilizing Super-Incos 5.5 software.

**3.0 DAILY SYSTEM QUALITY ASSURANCE**

The following series of system checks is performed at least once a day, i.e. at least once during every 12 hour period of instrument operation. Each check has a previously established acceptable range that can be found in the GCMS logbook. When values are not acceptable, the operator records those values in the logbook and proceeds to make adjustments to correct the condition before continuing with system checks. After the correction has been made, the new values are recorded.

- A. Check interface (separator oven), transfer line and manifold temperatures.
- B. Check source vacuum (at GC oven temperature 60) and GC column pressure.
- C. Establish zero setting using the procedure ZDPLAY. Check instrument sensitivity by admitting perfluorotributylamine (FC-43) to the detector at a pre-determined rate. Record the electron multiplier voltage required to give a 1.6 volt signal at m/e 219 with SENS set at  $10^{*-8}$ . Check peak resolution at low (69/70), mid (219/220) and high (414 or 502) masses. Check the ratios of the peak heights (low to mid, mid to high, etc.). If peak resolution or height ratio adjustments are necessary, record all changes in source tuning in the logbook. Calibrate the instrument using the procedure CALI. Acceptable ranges are Projection Error -150 to +150 MMU, RMS Fit Error < 50 MMU (or < 5%



peak width), and Elimination Factor (S.D.) RMS < 10. Make a hard copy and file it under CALIBRATIONS.

- D. Inject 1 ul of decafluorotriphenylphosphine [DFTPP-the EPA GCMS Analytical Quality Control (AQC) compound] at a concentration of 40 ng/ul. Compare fragment peak intensity and relative abundance to the Key Ion Abundance Criteria established by EPA (see attached sheet) for DFTPP. If unacceptable, repeat system checks beginning at "D" above. Create hard copies of Mass Spectrum and Mass List of DFTPP and file with raw data reports. Check intensity of air peak (28) and column bleed (207).
- E. Inject appropriate analytical standard, based on analysis to be performed. Check early, middle and late eluting compounds for peak shape. Choose two compounds with the same mass and strong molecular ions that elute within 8 scans of each other and check resolution.

#### 4.0 IDENTIFICATION CRITERIA

- A. Identification of peaks run full scan is established by retention times and Extracted Ion Current Profiles (EICP). Compounds must elute within three scans of expected time. If an EICP for a group of related compounds differs markedly from what is expected (based on typical EICP templates), the spectrum of the peak in question is checked to determine it's identity.
- B. Identification of peaks run using the multiple ion detection (MID) mode is established by retention times and EICP as above. Due to the lack of a full spectrum, final confirmation is made by monitoring ion masses which are characteristic of the the compound in question. Characteristic ion ratios must match standards to within 20% before making final identification.

#### 5.0 QUANTITATION CRITERIA

- A. All standards are detailed in the Standards Logbook, which contains information about sources of compounds and methods used to prepare the standards.

#### 5.1 INTERNAL STANDARD(S) QUANTITATION / USE OF AUTHENTIC STANDARD

- A. Co-inject the authentic standard and the internal



standard to determine both of the RFs. Record in the file labelled RESPONSE FACTORS.

- B. Measure the ion peak area of the compound and of the internal standard in the sample run.
- C. Calculate the concentration of the compound in the original matrix, based on the ratio of the RFs, the measured areas, the amount of internal standard added to the extract, and the dry weight or volume of the sample (see calculation A).
- D. Record the results either in the individual file for the sample, or in the file grouping the results from a number of similar samples.

**5.2 INTERNAL STANDARD(S) QUANTITATION / USE OF A SIMILAR COMPOUND AS A STANDARD**

A similar compound is one (or more) which elutes within a few minutes of the compound of interest, belongs to the same chemical class, and has a key fragment ion within 50 amu of that of the compound of interest.

- A. Co-inject the similar compound and the internal standard to determine both of the RFs. Record in the file labelled RESPONSE FACTORS.
- B. Measure the ion peak area of the compound and of the internal standard in the sample run.
- C. Determine the fragment ion percentages (FIPs) for the key ions in both the compound of interest and the similar standard.
- D. Assume the Total Ion Current Response Factors (RFTs) are the same for the similar compound and the standard.
- E. Calculate the concentration of the compound in the original matrix, based on the ratios of the internal standard response factors (RFIs) and FIPs., the measured areas, the amount of internal standard added to the extract, and the dry weight or volume of the sample (see calculation B).
- F. Record the results either in the individual file for the sample or in the file grouping the results from a number of similar samples.

**5.4 INTERNAL STANDARD QUANTITATION/ NO SIMILAR COMPOUND IS AVAILABLE FOR USE AS A STANDARD**



- A. Choose the closest eluting internal standard which has a key ion(s) similar in mass to a key ion(s) in the compound of interest in the sample.
- B. Measure the ion peak area of the compound and of the internal standard in the sample run.
- C. Determine the FIPs for the key ions in both the compound of interest and the internal standard.
- D. Assume the RFTs are the same for these two compounds.
- E. Calculate the concentration of the compound in the original matrix based on the ratio of the FIPs, the measured areas, the amount of internal standard added to the extract, and the dry weight or volume of the sample (see calculation C).
- F. Record the results either in the individual file for the sample or in the file grouping the results from a number of similar samples.

## 6.0 CALCULATIONS

A.  $CONC = AREAC * RFI * NGI / (AREAI * DWORVOL * RFC)$

AREAC = area counts of the quantitation ion of the compound of interest

AREAI = area counts of the quantitation ion of the internal standard

CONC = concentration of the compound of interest in the sample

= nanograms/(gm dry weight) for organisms and sediments

= parts per trillion for water samples

DWORVOL = dry weight (grams) or volume (liters) of sample

RFI = response factor of the internal standard

RFC = response factor of the compound of interest

NGI = nanograms of internal standard added to the extract.

B.  $CONC = AREAC * RFI * NGI / (AREAI * DWORVOL * RFS * FIPC / FIPS)$



RFS = response factor of the similar compound

FIPC = fragment ion percentage of the quantitation ion in the spectrum of the compound of interest.

FIPS = fragment ion percentage of the quantitation ion in the spectrum of the similar compound

C.  $CONC = AREAC * FIPI * NGI / (AREAI * FIPS * DWORVOL)$

FIPI = fragment ion percentage of the quantitation ion in the spectrum of the internal standard

Table II. Reference Compound Key Ions and Ion Abundance Criteria

<u>Mass</u>	<u>Ion Abundance Criteria</u>
51	30-60% of mass 198
68	Less than 2% of mass 69
70	Less than 2% of mass 69
127	40-60% of mass 198
197	Less than 1% of mass 198
198	Base Peak, 100% Relative Abundance
199	5-9% of mass 198
275	10-30% of mass 198
365	1% of mass 198
441	Less than mass 443
442	40-60% of mass 198 - this ion is very sensitive to spectrum number chosen and condition of equipment. If greater than 60% equipment is OK if all other criteria are met.
443	17-23% of mass 442



## ERLN Standard Operating Protocol for Ultrasonic Extraction of Sediment Samples for Inorganic Analysis

## I. Sample preparation

1. Samples should be thawed and homogenized using appropriate equipment prior to subsampling for analysis.
2. Label or number dry, acid-washed polycarbonate centrifuge tubes, e.g. with peel-off labels, and obtain the tare weight of each tube.
3. Add approximately 5 g of wet sediment ( 2 - 2.5 g dry) to each tube and reweigh, obtaining the wet gross weight. Place the tubes upright in freezer until sediments are frozen solid.
4. Freeze-dry the samples using the Virtis lyophilizer.
  - a. Drain the condensor, then precool to  $-50^{\circ}$  C. Refrigerate sample compartment below  $0^{\circ}$  C.
  - b. Place frozen specimens in sample compartment. Seal door, close vacuum release clamp and start vacuum pump. Verify that vacuum is being drawn (pressure  $<1.5$  torr).
  - c. Freeze-dry specimens for 16 hr. at  $-40^{\circ}$  C, then turn on shelf heat and hold for 24 hr at  $45^{\circ}$  C.
5. Remove the tubes from the freeze dryer and weigh again, obtaining the dry gross weights for the samples.

## II. Ultrasonic Extraction

1. Add 25 ml of 2M  $\text{HNO}_3$  to each sample tube and replace cap hand-tight.
2. Place tubes in ultrasonic bath rack. Insure that all tubes are completely vertical and contained within openings on bottom of rack. Place cover over rack and fasten, using nylon wing nuts, hand-tight. Place covered rack into ultrasonic bath and insure that bath level is at or above level of acid in the tubes.
3. Turn on ultrasonic bath and ultrasonicate samples approximately 16 hours (overnight). Bath will heat to about  $75$  deg. C.
4. Remove rack and tubes from ultrasonic bath, allow to drain and cool.

## III. Specimen centrifugation and dilution

1. Weigh centrifuge tubes. Pair tubes as closely as possible by gross weight. Centrifug at 10,000 rpm for 15 minutes.



2. Decant supernatant from tube into 50 ml volumetric flask. Add 15 ml of 2M  $\text{HNO}_3$  to residue in tube and ultrasonicate for another 15 minutes. Repeat centrifugation and decant into flask. Dilute solution in flask to the volume mark. Pour sample solution into 60 ml bottle and label bottle appropriately.



## ERLN Standard Operating Protocol for Microwave Digestion of Organism Samples for Inorganic Analysis

## I. Sample preparation

1. Organism samples should be thawed prior to dissection. Removed tissue specimens from shell or skin using stainless steel instruments. Rinse instruments between samples by with deionized water. If required, homogenize samples using appropriate tissue homogenizing system (do not use stainless steel generators if chromium and/or nickel are to be analyzed).
2. Number the empty Teflon digestion vessels with peel-off labels and obtain the tare weight of each vessel (without the pressure relief disk).
3. Add approximately 15 g of wet tissue (approximately 2.5 g dry tissue) to each vessels and reweigh, obtaining the wet gross weight. Place the vessels upright in freezer until specimens are frozen solid.
4. Freeze-dry the samples using the Virtis lyophilizer.
  - a. Drain the condensor, then precool to  $-50^{\circ}$  C. Refrigerate sample compartment below  $0^{\circ}$  C.
  - b. Place frozen specimens in sample compartment. Seal door, close vacuum release clamp and start vacuum pump. Verify that vacuum is being drawn (pressure  $<1.5$  torr).
  - c. Freeze-dry specimens for 48 hr. at  $-40^{\circ}$  C, then turn on shelf heat and hold for 24 hr at  $45^{\circ}$  C.
5. Remove the vessels from the freeze dryer and weigh again, obtaining the dry gross weights for the samples.

## II. Microwave digestion

## A. Open-vessel digestion

1. Add 15 ml of concentrated  $\text{HNO}_3$  (Instra-Analyzed grade) to each sample vessel and close cap, without pressure relief disks, hand-tight. If bubbling or foaming occurs, allow samples to sit at room temperature until foaming subsides (4-1 hr.).
2. Load vessels into carousel, place carousel into microwave oven and close door. Begin carousel rotation, making sure oven exhaust fan is operating.



3. Program MDS-81:

	Time(min)	Power level		
# of vessels:		6	8	12
S-1:	3:00	25%	30%	35%
S-2:	5:00	35%	40%	55%
S-3:	5:00	50%	60%	75%

and press START to initiate microwave digestion.

3. After program has completed run, remove sample carousel from MDS-81 and place in hood to cool.

B. Closed vessel digestion

1. Remove cap from each vessel and add 3 ml H<sub>2</sub>O<sub>2</sub> to vessel. Place pressure relief disk, ring side up, on top of lower portion of vessel and replace cap hand-tight. Tighten caps to correct torque using MDS-81 capping station.

2. Place vessels in carousel. Insert vent tube into each vessel neck and tighten nut. Insert free end of tube into vent trap in center of carousel and return carousel to oven. Insure that venting fan is operating and begin carousel rotation.

3. Program MDS-81:

# of vessels:	6	8	12	
	Time(min) Power level			
S-1:	6:00	40%	50%	60%
S-2:	2:00	0%	0%	0%
S-3:	5:00	50%	60%	80%

and press START to initiate microwave digestion.

4. After program is completed, remove carousel from MDS-81 and place in hood to cool (minimum 1 hour). When vessels are cool to touch, remove vent tubes and CAREFULLY vent vessels manually to release pressure. If venting is too vigorous, allow to cool longer and vent again. Repeat until no more venting occurs.

5. Remove caps from vessels using MDS-81 capping station. Invert cap and pressure relief disk over vessel and rinse with deionized water, allowing rinse to drain into vessel. Add 15 ml of deionized water to vessel.

III. Sample filtration and dilution

1. Using plastic tweezers, place circle of Whatman 42 filter paper into filtration apparatus. Wash filter with 2 M HNO<sub>3</sub>. Place 60-ml acid-cleaned, polyethylene bottle and vacuum gasket under filtration apparatus and apply vacuum. Filter digested sample solution through filter paper into bottle. Rinse the digestion vessel with deionized water and pour through filter as well. Repeat rinse/filtration. Holding



sample bottle, release vacuum and remove bottle.

2. Pour combined filtrates from bottle into 50 ml volumetric flask. Rinse bottle and use the rinse to dilute solution in flask to the volume mark. Discard any remaining rinse solution in bottle. Return the sample solution to the bottle and label bottle appropriately.



Direct determination of trace metals in sea water samples.

#### SAMPLE PREPARATION

1. Samples should be collected in acid cleaned polyethelene bottles. The preparation procedure is the same for either soluble or total metals determinations; samples intended for soluble metals analyses should be filtered prior to acidification.
2. Samples should be acidified to a pH of approximately 2-2.5 with 1 microlitre of conc. HNO<sub>3</sub> (Seastar brand or equivalent) per millilitre of sample. Samples should sit for at least 1 hour before proceeding in order to completely recover particulate metals and metals adsorbed to the container walls (although ultrasonic agitation may reduce the amount of time necessary). Once acidified, the samples can be stored for long periods of time before analysis.
3. Pipette one millilitre (1 ml) of each sample into acid clean d polyethylene vials. Add 100 microlitres of conc. HNO<sub>3</sub> (Seastar brand or equivalent), close the vial and shake well.
4. Standards should be prepared from 1 millilitre of Chelex-100 stripped seawater or the NASS-1 open ocean reference seawater and 100 microlitres of conc. HNO<sub>3</sub> (Seastar brand or equivalent).

#### INSTRUMENTAL ANALYSIS

Atomic absorption (AA) or inductively coupled plasma emission (ICP) spectrochemical analyses or performed according to ERLN standard operating parameters or manufacturers' recommended operating conditions when standard ERLN parameters do not exist.