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UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
REGION I
J.F. KENNEDY FEDERAL BUILDING, BOSTON, MASSACHUSETTS 02203-2211

April 21, 1993

Deborah Stockdale
Environmental Restoration Branch
Naval Facilities Engineering Command
U.S. Department of the Navy
10 Industrial Way
Mail Stop 82
Lester, PA 19113-2090

RE: Draft Action Memorandum for Building 31 - Naval Submarine Base - New London, Groton, Connecticut (NSBNL)

Dear Ms. Stockdale:

Per our telephone conversation this afternoon, the following comments were generated by EPA's Environmental Services Division specifically with regard to air monitoring issues. The following recommendations should amend EPA's comment package dated April 20, 1993.

Since the excavation of soils during the proposed removal action may release lead contaminated soil particulates into the air, EPA strongly recommends that air monitoring of the immediate and surrounding air environs be conducted to assess the presence of airborne lead concentrations.

The four attachments are offered as guidance for conducting air monitoring and the determination of lead particulate levels. Attachment A is a brief discussion of QA/QC features to be considered when designing an Air Monitoring Program. Attachment B provides useful information required when writing Standard Operating Procedures. Attachment C identifies elements of Air Monitoring Data Summaries and the NIOSH analytical method for determination of lead in airborne particulates is given in Attachment D.

In addition to the above referenced attachments, the following recommendations specific to Building 31 are offered for consideration:

- Air monitoring needs to be conducted for determination of lead in air both in and outside Building 31 for protection of at least on-site field workers.

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- Since much of the high lead levels are associated with soil within the building, air sampling equipment need to be operated in and around the high lead areas before, during, and after soil excavation.

- Air monitoring will also be needed in areas where the contaminated soil will be mixed with cement or otherwise treated. Such information will be of value in preparing the field Health and Safety Plan and for managing the treatment process to control lead in the ambient air.

Should you have any questions or need any additional information, please do not hesitate to call me at (617) 573-5764.

Sincerely,

Carol A. Keating

Carol A. Keating
Remedial Project Manager
Federal Facilities Superfund Section

Attachments

cc: Mr. William Mansfield, NSBNL
Mr. Adam Sullivan, CTDEP
Mr. Dale Weiss, TRC
Mr. Andy Miniuks, USEPA
Mr. Jack Harvanek, USEPA

**ATTACHMENT A**

Ambient Air Sampling Plan with QA/QC Procedures

A work plan documenting all aspects of sampling, analysis and associated QC/QA must be prepared, reviewed and approved prior to any sampling effort:

1. Data quality objectives must be established in order to determine whether any data collected will be relevant and useful. For example, if a risk assessment is to be performed, how many sampling stations and at which key locations will be required? Which species will be sampled for? Is the method to be utilized capable of quantifying those contaminants at the expected levels? Specify the detection limits expected under the proposed conditions.

2. Specification of the method to be utilized must include, for example, documentation of applicability to the species sought during sampling (provide a list of species expected to be found), and a detailed description of both sampling procedures and analytical procedures to be followed. Any deviations from referenced procedures must be thoroughly documented. Include the Standard Operating Procedures specified by the method. In addition, data must be presented demonstrating the capability of the method to be used to attain the required quality of data under the actual sampling and analysis conditions anticipated (see Performance Criteria and Quality Assurance requirements delineated in each method).

3. Sampling and analytical procedures should be described in a sufficient level of detail to provide assurance that they will be performed in accordance with accepted quality control standards. The same general level of scientific rigor as adhered to in the Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air (EPA-600/4-84-041) must be demonstrated for any technique utilized, in order to lend credibility to the results.

4. Sampling locations should be specified and identified on a site map, including sufficient detail to show sources and directions of potential receptors. The map should be north-oriented and include a scale. Specify the expected prevailing wind speed and direction during the proposed sampling period, including a wind rose. Address sampling station issues such as provisions for security and electrical power, as applicable. The sampling Standard Operating Procedure must list all necessary equipment and supplies.

   Specify how flow rates and sampling times will be established.

   - What is the rejection criteria for pre/post flow-rate calibration?

- How will the sampling equipment be cleaned, and how will the requisite degree of cleanliness be demonstrated?

- Will flow rates be corrected to standard conditions of temperature, pressure and humidity?

- Specify laboratory, trip and field blanks and quality control duplicates, as well as backup (secondary) cartridges where applicable.

5. Delineate the collection procedures for concurrent on-site meteorological data (specify equipment, siting criteria, calibration procedures, data recording and reduction, etc.). Attempt to conduct baseline ambient air monitoring under worst-case conditions (high temperatures, low humidity, low wind speeds).

6. Include procedures for sample collection, handling, storage and transportation, including preservation methods and holding times. Specify chain of custody procedures.

7. Additional Requirements:

   What are the calibration procedures for the analytical instruments to be used? How will standards be prepared?

   How will data from blank analysis be utilized? What is the limit of blank contamination for which data will be accepted?

   Will backup (series) cartridges be utilized? What is the criteria of acceptance for breakthrough from primary to backup cartridge? Specify the acceptance criteria (precision and accuracy) for duplicate cartridges.

   Will an internal standard be established by the spiking of blank, sample and calibration cartridges? Describe the spiking procedure.

   Are recovery and precision data available for the selected contaminants to establish the validity of quantitative data? Present all such data and all numerical criteria for quality control purposes.

8. In general, the proposal for ambient monitoring of air toxics must establish the scientific legitimacy of the sampling. Inadequately documented sampling and analytical procedures may necessitate discarding the resulting data.

9. The data package submitted should include, along with the raw data, all the information necessary to perform data validation, including standards preparation, calibration curves, all calculations used for the determination of detection limits and acceptance criteria to be applied (including precision and accuracy limits).

## ATTACHMENT B

### Standard Operating Procedures (SOPs)

Standard Operating Procedures (SOPs) must be prepared for all aspects of sampling, analysis and instrument calibration. An SOP is defined as a complete description of a sample collection, analysis or other operation whose mechanisms are thoroughly prescribed and which details a commonly accepted method of performing routine or repetitive tasks. Its purpose is to ensure consistency of application of a method and repeatability and comparability of results, regardless of which qualified person is performing the operation.

A SOP for sampling and analysis would include the following information:

- Method testing, including ruggedness testing
- Configuration and maintenance of sampling equipment
- Calibration of sampling equipment
- Cleaning and demonstration of cleanliness of sampling equipment
- Chain-of-custody
- Sample collection, including quality control samples such as blanks, duplicates, backups, etc.
- Sample handling/preservation/storage
- Configuration and maintenance of analytical equipment
- Tuning and calibration of analytical equipment
- Cleaning and demonstration of cleanliness of analytical equipment
- Standards preparation and control
- Sample preparation
- Spiking
- Introduction of samples
- Data reduction, processing (including uncertainty analysis), handling, storage and retrieval
- Data validation
- Reporting of results, including quality parameters
- Retention of samples and data
- Record-keeping

A calibration SOP would include:

- a definition of terms used in the procedure
- a description of the specific equipment to which the procedure is applicable, including model number and specifications
- a brief description of the scope, principle and/or theory of the calibration method

- fundamental calibration specifications, such as environmental conditions, calibration points and tolerances
- a description of standards required to perform an effective calibration, including source, identifying serial number, specified tolerance and expiration date
- a list of equipment necessary to perform a calibration, including manufacturer, model number, specified accuracy and maintenance status
- a cautionary list of possible impediments to a successful calibration, such as common procedural errors or interferences
- a clear, concise step-by-step breakdown of the calibration operation from beginning to end
- specific instructions for recording and reporting the calibration data and its use in qualifying the resultant experimental data

**ATTACHMENT C**

Air Monitoring Data Summaries

Air monitoring data summaries should include at least the following:

- A listing of concentrations measured by station and monitoring period indicating concentrations of all constituents for which monitoring was conducted. The listings should indicate detection limits for those cases in which a constituent is not detected, as well as upwind/downwind exposure classification and monitoring station operational data (e.g., sampling flow rates, station numbers, sampling start/end times);

- Summary tables of constituent-specific concentrations measured for each monitoring station, including the following:

  - Mean concentration
  - Minimum concentration
  - Maximum concentration
  - Detection limit
  - Frequency above and below detection limits
  - Number of samples
  - Number of occurrences of air concentrations exceeding selected values (e.g., health and safety criteria, ARARs and odor thresholds)
  - Upwind/downwind exposure summaries;

- A narrative discussion of sampling results, indicating problems encountered, the relationship of the sampling activity to unit operating conditions and meteorological conditions, sampling periods and times, background levels and other air emission sources, and interferences that may complicate data interpretation

- Data recovery parameters for all parameters

ATTACHMENT D

# NIOSH LEAD PROCEDURE: METHOD 7082

FORMULA: Pb

M.W.: 207.19 (Pb); 223.19 (PbO)

**LEAD**

METHOD: 7082
ISSUED: 2/15/84

OSHA: 0.05 mg/m³
NIOSH: 0.05 mg/m³ [1]
ACGIH: 0.15 mg/m³; STEL 0.45 mg/m³

PROPERTIES: soft metal;
d 11.3 g/cm³; MP 327.5 °C;
valences +2, +4 in salts

SYNONYMS: vary depending upon the chemical form (elemental lead and lead compounds except alkyl lead); CAS #1317-36-8 (PbO); CAS #7439-92-1 (Pb).

## SAMPLING

SAMPLER: FILTER
(0.8-µm cellulose ester membrane)

FLOW RATE: 1 to 4 L/min

VOL-MIN: 200 L @ 0.05 mg/m³
-MAX: 1200 L

SHIPMENT: routine

SAMPLE STABILITY: stable

BLANKS: 2 to 10 field blanks per set

## ACCURACY

RANGE STUDIED: 0.13 to 0.4 mg/m³ [2];
0.15 to 1.7 mg/m³ (fume) [3]

BIAS: not significant [2]

OVERALL PRECISION ($s_r$): 0.072 [2];
0.068 (fume) [3]

## MEASUREMENT

TECHNIQUE: ATOMIC ABSORPTION, FLAME

ANALYTE: lead

ASHING: conc. $HNO_3$, 6 mL; 140 °C

FINAL SOLUTION: 10% $HNO_3$, 10 mL

FLAME: air-acetylene, oxidizing

WAVELENGTH: 283.3 nm

BACKGROUND CORRECTION: $D_2$ or $H_2$ lamp

CALIBRATION: $Pb^{++}$ in 10% $HNO_3$

RANGE: 10 to 200 µg per sample [3,8]

ESTIMATED LOD: 2.6 µg per sample [9]

PRECISION ($s_r$): 0.03 [2]

APPLICABILITY: The working range is 0.025 to 0.5 mg/m³ for a 400-L air sample. The method is applicable to elemental lead, including Pb fume, and all other aerosols containing lead. This is an elemental analysis, not compound specific. Aliquots of the samples can be analyzed separately for additional elements.

INTERFERENCES: Use $D_2$ or $H_2$ continuum background correction to control flame or molecular absorption. High concentrations of calcium, sulfate, carbonate, phosphate, iodide, fluoride, or acetate can be corrected.

OTHER METHODS: This method combines and replaces P&CAM 173 [8] and S341 [7,9] for lead. Method 7300 (ICP-AES) is an alternate analytical method. Method 7505 is specific for lead sulfide. The following have not been revised: the dithizone method, which appears in P&CAM 102 [4] and the lead criteria document [1]; P&CAM 191 (ASV) [5]; and P&CAM 214 (graphite furnace-AAS) [6].

REAGENTS:

1. Nitric acid, conc.
2. Nitric acid, 10% (w/v). Add 100 mL conc. $HNO_3$ to 500 mL water; dilute to 1 L.
3. Hydrogen peroxide, 30% $H_2O_2$ (w/w), reagent grade.
4. Calibration stock solution, 1000 µg Pb/mL. Commercial standard or dissolve 1.00 g Pb metal in minimum volume of (1+1) HCl and dilute to 1 L with 1% (v/v) HCl. Store in a polyethylene bottle. Stable ≥ one year.
5. Air, compressed, filtered.
6. Acetylene.
7. Distilled or deionized water.

EQUIPMENT:

1. Sampler: Cellulose ester filter, 0.8-µm pore size, 37-mm diameter; in cassette filter holder.
2. Personal sampling pump, 1 to 4 L/min, with flexible connecting tubing.
3. Atomic Absorption Spectrophotometer with an air-acetylene burner head.
4. Lead hollow cathode lamp or electrode dischargeless lamp.
5. Regulators, two-stage, for air and acetylene.
6. Beakers, Phillips, 125 mL, or Griffin, 50 mL with watchglass covers.*
7. Volumetric flasks, 10- and 100-mL.*
8. Assorted volumetric pipets as needed.*
9. Hotplate, surface temperature 140° C.
10. Bottles, polyethylene, 100-mL.

*Clean all glassware with conc. nitric acid and rinse thoroughly with distilled or deionized water before use.

SPECIAL PRECAUTIONS: Perform all acid digestions in a fume hood.

SAMPLING:

1. Calibrate each personal sampling pump with a representative sampler in line.
2. Sample at an accurately known flow rate between 1 and 4 L/min for up to 8 hrs for TWA measurements. Do not exceed a filter loading of ca. 2 mg total dust.

SAMPLE PREPARATION:

NOTE: The following sample preparation gave quantitative recovery (see EVALUATION OF METHOD) [9]. Steps 4 through 9 of Method 7300 or other quantitative ashing techniques may be substituted, especially if several metals are to be determined on a single filter.

3. Open the cassette filter holders and transfer the samples and blanks to clean beakers.
4. Add 3 mL conc. $HNO_3$, and 1 mL 30% $H_2O_2$ and cover with a watchglass. Start reagent blanks at this step.
   NOTE: If $PbO_2$ is not present in the sample, the 30% $H_2O_2$ need not be added [3,9].
5. Heat on hotplate (140 °C) until most of the acid has evaporated.
6. Repeat two more times using 2 mL conc. $HNO_3$ and 1 mL 30% $H_2O_2$ each time.
7. Heat on 140 °C hotplate until a white ash appears.
8. When sample is dry, rinse the watchglass and walls of the beaker with 3 to 5 mL 10% $HNO_3$. Allow the solution to evaporate to dryness.
9. Cool each beaker and dissolve the residues in 1 mL conc. $HNO_3$.
10. Transfer the solution quantitatively to a 10-mL volumetric flask and dilute to volume with distilled water.
    NOTE: If the concentration (M) of any of the following is expected to exceed the lead concentration (M) by 10-fold or more, add 1 mL 1 M $Na_2EDTA$ to each flask before dilution to volume: $CO_3^{=}$, $NO_3^-$, $I^-$, $F^-$, $CH_3COO^-$. If $Ca^{++}$ or $SO_4^{=}$ are present in 10-fold excess, make all standards and samples 1% (w/w) in $La^{+++}$ [8].

CALIBRATION AND QUALITY CONTROL:

11. Prepare a series of working standards covering the range 1 to 20 µg Pb/mL (1 to 200 µg Pb per sample) by adding aliquots of calibration stock solution to 100-mL volumetric flasks. Dilute to volume with 10% $HNO_3$. Store the working standards in polyethylene bottles and prepare fresh weekly.
12. Analyze the working standards together with the blanks and samples (steps 17 and 18).
13. Prepare a calibration graph of absorbance vs. solution concentration (µg/mL).
14. Aspirate a standard for every 10 samples to check for instrument drift.
15. Check recoveries with at least one spiked media blank per 10 samples.
16. Use method of additions occasionally to check for interferences.

MEASUREMENT:

17. Set spectrophotometer as specified by the manufacturer and to conditions on page 7082-1.
    NOTE: An alternate wavelength is 217.0 nm [10]. Analyses at 217.0 nm have slightly greater sensitivity, but poorer signal-to-noise ratio compared to 283.3 nm. Also, non-atomic absorption is significantly greater at 217.0 nm, making the use of $D_2$ or $H_2$ continuum background correction mandatory at that wavelength.
18. Aspirate standards, samples, and blanks. Record absorbance readings.
    NOTE: If the absorbance values for the samples are above the linear range of the standards, dilute with 10% $HNO_3$, reanalyze, and apply the appropriate dilution factor in the calculations.

CALCULATIONS:

19. Using the measured absorbances, calculate the corresponding concentrations (µg/mL) of lead in the sample, $C_s$, and average media blank, $C_b$, from the calibration graph.
20. Using the solution volumes (mL) of the sample, $V_s$, and media blanks, $V_b$, calculate the concentration, C (mg/m³), of lead in the air volume sampled, V (L):

$$C = \frac{C_s V_s - C_b V_b}{V}, \text{ mg/m}^3.$$

EVALUATION OF METHOD:

Method S241 [7] was issued on October 24, 1975, and validated over the range 0.13 to 0.4 mg/m³ for a 180-L air sample, using generated atmospheres of lead nitrate [2]. Recovery in the range 18 to 72 µg Pb per sample was 98%, and collection efficiency of 0.8-µm mixed cellulose ester filters (Millipore Type AA) was 100% for the aerosols. Subsequent studies on analytical recovery of 200 µg Pb per sample gave the results [3,9]:

| Species | Digestion Method | Analytical Recovery, % |
|---|---|---|
| Pb metal | $HNO_3$ only | 92 ± 4 |
| Pb metal | $HNO_3 + H_2O_2$ | 103 ± 3 |
| PbO | $HNO_3$ only | 93 ± 4 |
| PbS | $HNO_3$ only | 93 ± 5 |
| $PbO_2$ | $HNO_3$ only | 82 ± 3 |
| $PbO_2$ | $HNO_3 + H_2O_2$ | 100 ± 1 |
| Pb in paint* | $HNO_3$ only | 95 ± 6 |
| Pb in paint* | $HNO_3 + H_2O_2$ | 95 ± 6 |

*Standard Reference Material #1579, U.S. National Bureau of Standards.