

**LIST OF TABLES**

|                   |                                                                                                            |
|-------------------|------------------------------------------------------------------------------------------------------------|
| Table A-1         | Data Quality Objectives                                                                                    |
| Table A-2         | Decision Criteria used for Initial Disposal Classification of Sediments under RCRA and TSCA Disposal Rules |
| Table B-1         | Example Sample ID and Horizontal Coordinates                                                               |
| Table B-2         | River Section 1 Program Summary                                                                            |
| Table B-3         | River Section 2 Program Summary                                                                            |
| Table B-4         | River Section 3 Program Summary                                                                            |
| Table B-5         | Sample Container and Preservation Requirements                                                             |
| Table B-6a – B-6j | Reference Limit and Evaluation Tables for Analytical Methods                                               |
| Table B-7a – B-7n | Measurement Performance Criteria Tables for Analytical Methods                                             |
| Table B-8         | Data Collected During Sediment Core Collection                                                             |
| Table B-9         | Data Collected During Sample Processing in the Field Lab                                                   |
| Table B-10        | Valid Values for PCBs                                                                                      |
| Table C-1         | Summary of Analyses for Initial PE Acceptance Criteria Development                                         |
| Table C-2         | Summary of Analyses for Inter-Laboratory Comparison Study                                                  |
| Table D-1         | Format of an Environmental Standards Quality Assurance Review                                              |

**APPENDICES**

|             |                                                                                                                                                       |
|-------------|-------------------------------------------------------------------------------------------------------------------------------------------------------|
| Appendix 1  | SOP for Sediment Core Collection                                                                                                                      |
| Appendix 2  | SOP for Bathymetric Survey                                                                                                                            |
| Appendix 3  | SOP for Sub-Bottom Acoustic and Electromagnetic Surveying Equipment                                                                                   |
| Appendix 4  | SOP for Sediment Probing                                                                                                                              |
| Appendix 5  | SOP for the Analysis of PCBs by SW-846 Method 8082 (GEHR8082)                                                                                         |
| Appendix 6  | SOP for the Extraction and Cleanup of Sediment/Solid Samples for PCB Analysis Using the Pressurized Fluid Extraction by SW-846 Method 3545 (GEHR3545) |
| Appendix 7  | SOP for the Extraction and Cleanup of Sediment/Soil Samples for PCB Analysis Using the Soxhlet Extraction by SW-846 Method 3540C (GEHR3540C)          |
| Appendix 8  | SOP for Analysis of PCB Homologs by EPA Method 680 (GEHR680)                                                                                          |
| Appendix 9  | SOP for Data Package Deliverable (DPSOP)                                                                                                              |
| Appendix 10 | SOP for Grain Size                                                                                                                                    |
| Appendix 11 | SOP for Atterberg Limit                                                                                                                               |
| Appendix 12 | SOP for Specific Gravity                                                                                                                              |
| Appendix 13 | SOP for Bulk Density                                                                                                                                  |

**APPENDICES (Cont.)**

- Appendix 14 *(This appendix is no longer necessary. It has been left for convenience for potential future use).*
- Appendix 15 SOP for Total Organic Carbon
- Appendix 16 SOP for USCS Classification
- Appendix 17 SOP for Side Scan Survey Procedures
- Appendix 18 SOP for Core Processing
- Appendix 19 *(This appendix is no longer necessary. It has been left for convenience for potential future use).*
- Appendix 20 SOP for <sup>137</sup>Cs
- Appendix 21 SOP for TCLP Preparation by 1311
- Appendix 22 SOP for VOAs With TCLP Preparation
- Appendix 23 SOP for SVOAs With TCLP Preparation
- Appendix 24 SOP for Pesticides With TCLP Preparation
- Appendix 25 SOP for Herbicides With TCLP Preparation
- Appendix 26 SOP for Preparation of Metals and Mercury and Analysis of Mercury in Leachate  
(Refer to Appendix 29 for the Analysis of Metals by ICP)
- Appendix 27 SOP for Ignitability
- Appendix 28 SOP for PCDD/PCDF
- Appendix 29 SOP for Preparation and Analyses of Metals and Mercury in Sediment
- Appendix 30 Performance and Reporting of Field Audits
- Appendix 31 Performance and Reporting of Analytical Laboratory Audits
- Appendix 32 SOP for Data Validation of VOA Data (DV8260B)
- Appendix 33 SOP for Data Validation of SVOA Data (DV8270C)
- Appendix 34 SOP for Data Validation of Pesticide Data (DV8081A)
- Appendix 35 SOP for Data Validation of Herbicide Data (DV8151A)
- Appendix 36 SOP for Data Validation of PCBs (DV8082)
- Appendix 37 SOP for Data Validation of PCB (Homolog) Data (by GEHR680)
- Appendix 38 SOP for Data Validation of PCDD and PCDF Data (DV1613B)
- Appendix 39 SOP for Data Validation of ICP Metals Data (DV6010B)
- Appendix 40 SOP for Data Validation of Mercury Data (DV74707471)
- Appendix 41 SOP for Data Validation of TOC Data (DVTOC)
- Appendix 42 EDD Specifications

## **APPENDICES**

## **APPENDIX 13**

**STANDARD OPERATING PROCEDURE**

**NORTHEAST ANALYTICAL, INC.**

**NE188\_01.DOC**

**REVISION NUMBER: 01**

**STANDARD OPERATING PROCEDURE FOR BULK DENSITY  
DETERMINATION**

**MAY 26, 2002**

**COPY #**

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## STANDARD OPERATING PROCEDURE

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Northeast Analytical, Inc.  
Issuing Section: Inorganics Laboratory  
SOP Name: NE188\_01.SOP  
Date: 5/26/2002  
Revision Number: 01

Approved by:

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Robert E. Wagner  
Laboratory Director

- 1.0 TITLE** Standard operating procedure for the determination of Bulk Density.
- 2.0 PURPOSE** To provide the SOP for the preparation and analyses of soil samples for the determination of Bulk Density.
- 3.0 SCOPE** This method is applicable to soil, peats and soil mixtures. This procedure is based on procedures found in ASTM Method D4531-86 (1996).
- 4.0 COMMENTS** The difference in the varying bulk density procedures is in the method in which the sample is obtained and the volumes that are used. Variation in bulk density is attributable to the relative proportion and specific gravity of solid organic and inorganic particles and to the porosity of the soil.
- 5.0 SAFETY**
- 5.1 Safety glasses, lab coat or lab apron and disposable gloves must be worn when handling chemicals and samples.
  - 5.2 Personnel should familiarize themselves with the necessary safety precautions by reading MSDS information covering any chemicals used to perform SOP.
  - 5.3 Samples that emit undersirable odors when heated should be placed in the oven at the end of the day. The oven used for drying samples is connected to a fume hood. The hood should be operational at all times. If the hood is not working properly, contact a member of the safety committee immediately.
  - 5.4 If samples contain known quantities of hazardous material, the dried samples and the drying dishes or cups are classified as hazardous waste and are subject to the procedures listed in SOP NE054.
- 6.0 REQUIREMENTS**
- 6.1 Knowledge on the operation and calibration of the analytical balance is required.
  - 6.2 Knowledge on the operation of the drying oven located in the Inorganics laboratory.
  - 6.3 Knowledge on the maintenance of the portable desiccator box.

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Northeast Analytical, Inc

Standard Operating Procedure  
SOP Name: NE188\_01.DOC  
Date: 5/26/2002  
Page: 1 of 5

## 7.0 EQUIPMENT

- 7.1 Apparatus and Equipment. Located in the Inorganics laboratory.
- 7.1.1 VWR model 1370FD model drying oven. Inside the oven is a calibrated thermometer placed in a sand filled bottle. Located in the metals laboratory.
  - 7.1.2 Analytical balance. Mettler model AG204. Located in the metals lab.
  - 7.1.3 Aluminum drying dishes. VWR p/n 25433-008. Located in the lab storage room.
  - 7.1.4 Plastic spoons. Located in the third floor storage room. Available at local stores.
  - 7.1.5 Bulk Density, % Moisture and % Total Solids logbook. Located in the metals laboratory. See attachment A for an example.
  - 7.1.6 Glass trays. Located throughout the laboratory.
  - 7.1.7 Portable desiccator. Located in the metals laboratory.
- 7.2 Operation of drying oven.
- 7.2.1 Before drying samples, the drying oven “set temperature” knob has to be adjusted so that the temperature is between 103° and 105° C.
  - 7.2.2 After adjusting the temperature, wait approximately 15 minutes and open the door and read the thermometer. If the temperature is not within the specified range, repeat 6.2.1. If the oven will not stabilize, contact the inorganics manager.
- 7.3 Calibration of GFAA cups
- 7.3.1 Calculate the average volume of the GFAA cups annually.
  - 7.3.2 Prepare 10 GFAA cups by numbering them “1” through “10”.
  - 7.3.3 Place the cups in a vial rack and place in the drying oven for a minimum of one hour at a temperature of 103° to 105° C.
  - 7.3.4 Each day that the cups are to be weighed, pull the desiccant material from the oven and place in the desiccator for a minimum of one hour before placing the rack and cups in the desiccator. At the end of the day the desiccant material is poured into a glass tray and placed in the drying oven at 103° to 105°C. Place the cups in the desiccator for exactly one hour.
  - 7.3.5 Place a cup on the balance and write the cup number in the logbook. Record the weight of the cup.
  - 7.3.6 Repeat the above process for each cup.
  - 7.3.7 Fill each cup to the top with laboratory grade water.
  - 7.3.8 Carefully record the weight of the cup and water under “WETSAW” column. Subtract the weight of the cup from the “WETSAW”. Record the water weight under the “Comments” column.
  - 7.3.9 Calculate the average water weight and record under the “Comments column”.

- 7.3.10 Divide the average weight of the water in each cup by the density of water (1 g/ml) to calculate the average volume of water in each cup. Record the average volume (ml) under “Comments” column.
- 7.3.11 Sample cups may be purchased from Perkin Elmer p/n B008-7056 or any other vendor of 2ml GFAA cups.

## 8.0 PROCEDURE

### 8.1 Sample Analysis.

- 8.1.1 Prepare the GFAA sample cups by writing the last three digits of the NEA sample ID on the cups, for example, the sample cup for NEA sample AB01234 would be labeled as ‘234’. Prepare extra sample cups with their own unique numbers.
- 8.1.2 Place the cups in a vial rack and place in the drying oven for a minimum of one hour at a temperature of 103° to 105°C.
- 8.1.3 Each day that the cups are to be weighed, pull the desiccant material from the oven and place in the desiccator for a minimum of one hour before placing the rack and cups in the desiccator. At the end of the day the desiccant material is poured into a glass tray and placed in the drying oven at 103° to 105°C. Place the cups in the desiccator for exactly one hour.
- 8.1.4 Set up the logbook by writing the NEA#’s under the appropriate column.
- 8.1.5 Access LIMs and go to ‘WIN RESUTS’. Select “SAMPLE DESIGNATION” and type the NEA#’s in the white box. Select the ‘TEST’ template and click ‘OK’.
- 8.1.6 Place a cup on the balance and write the cup number in the logbook. See the following table to determine what column heading and cell to use.
- 8.1.7 Right click and select “Take BOAT”. Copy the value from the cell into the logbook. Repeat process for all the samples.
- 8.1.8 Using a tongue depressor or spatula, carefully place a portion of well mixed sample into the cup (do not use rocks or stones). Remove air pockets in the cup by gently tapping the cup on the counter. Fill the cups to the top.
- 8.1.9 Access LIMs and go to ‘WIN RESULTS’. Select “SAMPLE DESIGNATION” and type the NEA#’s in the white box. Select the “TEST” template and click “OK”.3
- 8.1.10 Place each cup on the balance. Right click and select “Take “WETSAW”. Coopy the value from the cell into thelogbook. Repeat process for all the samples.
- 8.1.11 Place the cups in a vial rack and place in the drying oven OVERNIGHT at a temperature of 103° to 105°C.
- 8.1.12 Place the cups in the desiccator for exactly one hour.
- 8.1.13 Access LIMs and go to “WIN RESULTS”. Select “SAMPLE DESIGNATION” and type the NEA#’s in the white box. Select the “TEST” template and click “OK”.
- 8.1.14 Place a cup on the balance. See the following table to determine what column heading and cell to use.
- 8.1.15 Right click and select “Take DRYSAW”. Copy the value from the cell into the logbook. Repeat process for all samples.
- 8.1.16 Enter the aver volume of the cups under “BDMCVO”.

8.1.17 The results are automatically calculated.

## 9.0 SAMPLE COLLECTION AND STORAGE

9.1 No preservation is required.

9.2 Samples can be collected in plastic or glass bottles with Polyseal caps.

## 10.0 QUALITY CONTROL

10.1 Duplicates:

10.1.1 One duplicate sample is processed each day that samples are prepared or every 10 samples, whichever occurs first.

10.2 Criteria:

10.2.1  $\% \text{RPD} = \text{ABS}[(X1-X2)/(X1+X2)]*200$ .

10.3 Limits:

10.3.1 The % RPD must be less than 20 %. If not samples need to be reanalyzed.

## 11.0 DOCUMENTATION

11.1 In the logbook, record the temperature of the oven and the date and time that the sample cups were placed in the oven.

11.2 In the logbook, record the temperature of the oven and the date and time that the sample cups were taken out of the oven.

11.3 Copies of the LIMs sheets are to be reviewed against the logbook by the analyst and placed in the folder.

11.4

**LIMS RESULT TEMPLATE "TEST" COLUMN HEADINGS AND DESCRIPTIONS**

| Column         | Description           | Column          | Description         | Column           | Description            |
|----------------|-----------------------|-----------------|---------------------|------------------|------------------------|
| <b>%SOLIDS</b> | Percent solids        | <b>BOATWT</b>   | Cup or boat wt.     | <b>WETSAW</b>    | Cup and wet sample wt. |
| <b>BULK</b>    | Bulk density          | <b>BDMCVO</b>   | Cup volume ml.      | <b>MOIST</b>     | % Moisture             |
| <b>D_BULK</b>  | Dup. Bulk density     | <b>BDMCDVOL</b> | Cup volume ml.      | <b>DUPBOATWT</b> | Dup. boat wt.          |
| <b>P_BULK</b>  | Precision calculation | <b>D_MOIST</b>  | Dup. %Moist.        | <b>WETDUPWT</b>  | Dup. wet sample wt.    |
| <b>P_MOIST</b> | Precision calculation | <b>DRYDUPWT</b> | Dup. dry sample wt. | <b>DRYSAW</b>    | Dry sample wt.         |

## 12.0 POLLUTION PREVENTION/WASTE MANAGEMENT.

12.1 Refer to NEA168.SOP for instructions for pollution prevention.

12.2 Refer to NEA089.SOP and NEA054.SOP for instructions for disposal of waste generated during the procedures previously mentioned.

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Northeast Analytical, Inc

Standard Operating Procedure  
SOP Name: Ne188\_01.DOC  
Date: 5/26/2002  
Page: 4 of 5

### 13.0 DEFINITIONS

- 13.1 Analytical Batch – The basic unit for analytical quality control is the analytical batch. The analytical batch is defined as samples that 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. Samples in each batch should be of similar composition (e.g. groundwater, sludge, ash, etc.)
- 13.2 Bulk Density – Is the measure of the weight of the soil per unit volume (g/ml), usually given on an oven dry (110° C) basis.
- 13.3 Matrix – The predominant material of which the sample to be analyzed is composed.
- 13.4 MSDS – Material safety data sheet. OSHA has established guidelines for the descriptive data that should be concisely provided on a data sheet to serve as the basis for written hazard communication programs. The thrust of the law is to have those who make, distribute, and use hazardous materials responsible for effective communication.
- 13.5 Relative Percent Difference (RPD) – To compare two values, the relative percent difference is based on the mean of the two values, and is reported as an absolute value, i.e., always expressed as a positive number or zero.
- 13.6 Replicate – Repeated operation occurring within an analytical procedure. Two or more analyses for the same constituent in an extract of a single sample constitute replicate extract analyses.
- 13.7 RCRA – Resource Conservation and Recovery Act, PL 94-580. Found at 40 CFR 240-271. EPA has jurisdiction. Enacted November 21, 1976, and amended since. RCRA's major emphasis is the control of hazardous waste disposal. It controls all soil-waste disposal and encourages recycling and alternative energy sources.
- 13.8 RCRA Hazardous Waste – A material designated by RCRA as hazardous waste and assigned a number to be used in record keeping and reporting compliance.
- 13.9 Reagent Water – Water in which an interferent is not observed at or above the minimum quantitation limit of the parameters of interest.
- 13.10 Rounding Rules – If the figure following those to be retained is less than 5, the figure is dropped, and the retained figures are kept unchanged. If the figure following those to be retained is greater than 5, the figure is dropped, and the last retained figure is raised by
- 13.10.1 If the figure following those to be retained is 5, and if there are no figures other than zeros beyond the five, the figure 5 is dropped, and the last-place figure retained is increased by one if it is an odd number or it is kept unchanged if an even number.
- 13.10.2 If a series of multiple operations is to be performed (add, subtract, divide, multiply), all figures are carried through the calculations. Then the final answer is rounded to the proper number of significant figures.
- 13.11 Sample Delivery Group (SDG) – Unit within a single case that is used to identify a group of samples for delivery. An SDG is a group of 20 or fewer field samples within a case, received over a period of up to 14 calendar days (7 calendar days for 14-day data turnaround contracts). Data from all samples in an SDG are due concurrently.

### 14.0 REFERENCES

- 14.1 ASTM methods D4531-86(1996).