

WEST VIRGINIA DEPARTMENT OF TRANSPORTATION
DIVISION OF HIGHWAYS
MATERIALS CONTROL, SOILS AND TESTING DIVISION

MATERIALS PROCEDURE

EVALUATION AND APPROVAL FOR LABORATORY TESTING OF SOLID
WASTE

1.0 PURPOSE

- 1.1 This procedure provides the criteria for evaluating and approving the technical competence of a solid waste testing laboratory; and the essential aspects of an accountability and quality control program for such a laboratory.

2.0 REFERENCED DOCUMENTS

- 2.1 ASTM Standards; E 548 Standard Guide for General Criteria Used for Evaluating Laboratory Competence, and E 882 Standard Guide for Accountability and Quality Control in the Chemical Analysis Laboratory.

2.2 USEPA SW 846

- 2.2.1 Method 3010: Acid Digestion of Aqueous Samples and Extracts for Total Metals for Analysis by Flame Atomic Absorption Spectroscopy or Inductively Coupled Plasma Spectroscopy.

- 2.2.2 Method 6010: Inductively Coupled Plasma Atomic Emission Spectroscopy

- 2.2.3 Method 7000: Atomic Absorption Methods

- 2.2.4 Method 7420: Lead (AA, Direct Aspiration)

2.3 40 CFR Chapter 1. Appendix II

- 2.3.1 Method 1311: Toxicity Characteristic Leaching Procedure

3.0 ORGANIZATION

3.1 The laboratory shall:

3.1.1 Use the proper method procedures as described by the United States Environmental Protection Agency (USEPA) or recognized by the West Virginia Department of Environmental Protection (WVDEP) for handling and testing of hazardous waste.

3.1.2 Have an organizational structure that shows the capability of satisfactorily performing the Toxicity Characteristic Leaching Procedure as specified by the United States Environmental Protection Agency.

3.1.3 Have a technical manager who has overall responsibility for the technical operation of the laboratory.

3.1.4 Be able to demonstrate, on request from the inspector or organization, that it is capable of performing solid waste tests by Environmental Protection Agency (EPA) approved methods.

4.0 QUALITY CONTROL SYSTEM

4.1 The laboratory shall operate a quality control system for the type, range, and volume of solid waste testing.

4.2 A relevant and current quality control manual shall be documented for the laboratory:

4.2.1 General and specific procedures for each test, as relevant for USEPA SW 846.

4.2.2 Proficiency testing using standard reference materials shall be necessary for certification.

4.2.3 Documentation of feedback and corrective action when test discrepancies are detected.

4.3 If the laboratory subcontracts any part of the solid waste testing, this work shall be placed with the quality control manual. The laboratory shall show that the subcontractors are competent in their testing and comply with the same regulations.

5.0 FACILITY AND EQUIPMENT

- 5.1 The laboratory shall contain all necessary equipment for proper performance for solid waste testing.
- 5.2 The laboratory environment shall not have adverse conditions such as dust, moisture, or vibration that could affect test results.
- 5.3 The equipment shall be properly maintained and calibrated on a regular basis.
- 5.4 Records shall be maintained on equipment. Each record should include:
 - 5.4.1 The name and manufacturer's identification model number.
 - 5.4.2 The date received, and the details of maintenance on equipment.
 - 5.4.3 For equipment that needs calibration; the date of last calibration, the calibration reports, and the maximum period of time between successive calibrations shall be documented.

6.0 TEST METHODS AND PROCEDURES

- 6.1 The laboratory shall:
 - 6.1.1 Have documented instructions on the use and operation of atomic absorption and/or inductively coupled plasma spectrophotometer; hot plate or microwave digestion system; and agitation apparatus.
 - 6.1.2 Use methods and procedures as discussed in reference documents in 2.0 or alternate procedures that through documentation show competence in both precision and accuracy that is allowable by USEPA SW 846 requirements.
- 6.2 All instructions, standards, manuals, and reference data relevant to the work of the laboratory shall be maintained up-to-date and be readily available.
- 6.3 All manual calculations and data transfers shall be subject to appropriate checks.

6.4 Where tests are performed or results are derived by computerized systems, the laboratory shall initially and periodically validate the systems. This generally implies an ability to detect malfunctions in hardware or software during program execution and operation and the ability to take appropriate action.

7.0 SAMPLE HANDLING

7.1 A system for identifying the samples to be tested shall be applied, either through documents or through marking, to ensure direct correlation between the samples and the results of the measurements made.

7.2 There shall be clear procedures for the receipt, storage, retention, and disposal of samples.

8.0 RECORDS

8.1 The laboratory shall maintain for a definite period of time, a record of all original data observations, calculations, derived data calibrations, and final test reports.

8.2 All records and test reports shall be held secure and in confidence to the client, unless otherwise specified or required by law.

9.0 TEST REPORTS

9.1 The performed tests in the laboratory shall be clearly and accurately documented and shall include at least the following information:

9.1.1 Name and address of laboratory.

9.1.2 Specific identification of each report and of each page of the report.

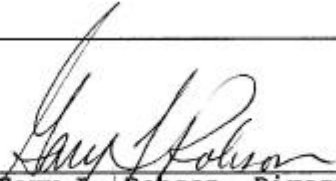
9.1.3 Name and address of the client.

9.1.4 Description and identification of the samples.

9.1.5 Project information.

9.1.6 The field samples, with identification numbers, that were analyzed.

- 9.1.7 Statistical information: the appropriate number and average of the samples analyzed, standard deviation, the standard error, and the confidence interval, and if more samples were analyzed to obtain accurate measure of the population.
- 9.1.8 The method of extraction, digestion, and analysis including the instrument employed and the sensitivity and minimum detection limits.
- 9.2 CHECKLIST FOR LABORATORY EVALUATION AND APPROVAL
- 9.2.1 Attached is a checklist for laboratory evaluation and approval.



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Attachment

CHECKLIST FOR THE EVALUATION AND APPROVAL FOR
LABORATORY TESTING OF SOLID WASTE

This checklist is Attachment One for the evaluation and approval for laboratory testing of solid waste.

ORGANIZATION

Does the laboratory have Standard Operating Procedures which that includes quality assurance, an organizational chart of responsible persons, and solid waste sampling and laboratory testing procedures?

YES ____ NO ____

Organizational chart or table showing the laboratory organization and line authority for the testing of hazardous solid waste?

YES ____ NO ____

The laboratory utilizes proper USEPA Solid Waste 846 method procedures in sample handling and testing of hazardous solid waste by the referenced documents in Section 2.0?

YES ____ NO ____

Does the laboratory subcontract any part of the solid waste testing? If so, who are the subcontractors and what tests do they perform? Does the subcontractor's laboratory use proper USEPA method procedures in solid waste?

YES ____ NO ____

Director or Supervisor who is ultimately responsible for all sample handling, quality assurance/control, final reports, and ensuring the production of valid measurement systems for precision and accuracy. Their qualifications and academic training.

Instrument operators who are performing the actual tests and the instrument being used. Their qualifications and academic training.

QUALITY ASSURANCE

Does the laboratory have a Quality Assurance and Quality Control (QA/QC) Plan available?

YES ____ NO ____

Does the Quality Assurance (QA) Plan include the following:

QA Officer or QA Manager?

YES ____ NO ____

NAME: _____

Corrective action procedures to eliminate any sample handling or laboratory problems?

YES ____ NO ____

Periodic reporting to management on the performance of the measurement system and the data quality?

YES ____ NO ____

Does the QC Plan for the analysis of RCRA samples include the following to establish analytical accuracy and precision of data? (QC PLAN MAY BE PART OF STANDARD OPERATING PROCEDURES (SOP)?

YES ____ NO ____

QUALITY CONTROL BLANKS

Calibration Blanks (One calibration blank should be analyzed with each analytical batch or every 20 samples, whichever is greater)?

YES ____ NO ____

Equipment blank to check on the field sampling device cleanliness?

YES ____ NO ____

Check standard of a material of known composition that is analyzed to evaluate the calibration of the analytical instrument?

YES ____ NO ____

Method Detection Limit (MDL) for the minimum concentration of a substance that can be measured and that the analyte concentration is greater than zero?

YES ____ NO ____

FIELD SAMPLES

Integrity of the samples has been maintained upon receipt?

YES ____ NO ____

Samples are checked to ensure that the number of samples, time of collection, and project information agrees with the information on the sample tags and the Chain of Custody sheets?

YES ____ NO ____

Field duplicate samples taken from the same sampling area (for QC of comparability between similar samples)?

YES ____ NO ____

Reagent blank that is carried throughout the entire analytical procedure?

YES ____ NO ____

LABORATORY QUALITY CONTROL SAMPLES

Standard curve for inorganic analytes using one calibration blank and three concentrations prepared for each standard. The response of each standard is based on three replicates of each standard concentration?

YES ____ NO ____

Standard curve is generated by computerized instrumentation?

YES ____ NO ____

If the standard curve is calculated manually, does it meet USEPA SW 846; with the linear equation which describes the calibration curve?

YES ____ NO ____

Concentration/response data (or relative response data) of the calibration check standards, along with dates on which they were analytically determined?

YES ____ NO ____

Reference sample prepared from a standard at a concentration other than that used for calibration, but within the calibration range?

YES ____ NO ____

Documentation of the holding times for field samples; that is, the date samples were received by a laboratory to the date the samples were prepared?

YES ____ NO ____

Check standard of known concentration prepared daily by the analyst to monitor the instrument's performance?

YES ____ NO ____

EXTRACTION FOR TOTAL METAL ANALYSIS

The entire sample is quartered and reduced to obtain a 100 gram sample that is used for extraction?

YES ____ NO ____

The sample is oven dried for 16 to 24 hours at 104 +/-2 degrees Celsius then reweighed to obtain the solid content of the sample?

YES ____ NO ____

A 5 gram subsample is dried and tested for pH by acid digestion to determine the extraction fluid used for leaching the metals out of the sample?

YES ____ NO ____

The extraction fluid #1 is made up fresh using 5.7 mL glacial acetic acid to 500 mL of deionized distilled water, adding 64.3 mL of 1.0N NaOH, and diluted to a volume of 1L (pH 4.93 ± 0.05)?

YES ____ NO ____

The extraction fluid #2 is made up fresh using 5.7mL glacial acetic acid to 1L of deionized distilled water (pH 2.88 ± 0.05)?

YES ____ NO ____

A 100 gram sample on a dry weight basis is used for each extraction?

YES ____ NO ____

The extraction solid to liquid ratio is 1:20 in the extraction vessel (a 100 gram sample of solid would require 2000mL of extraction liquid)?

YES ____ NO ____

The extraction vessels are such that the extraction fluid and sample occupy approximately 95 percent the container?

YES ____ NO ____

The agitation equipment rotates the extraction vessel in an end-over-end fashion at a rate of 30 +/- 2 rotations per minute?

YES ____ NO ____

Samples are agitated continuously for 18 +/- 0.25 hours at 18 to 27 degrees Celsius?

YES ____ NO ____

The waste/leaching solution is transferred through a large funnel to a pressure filtration device equipped with a 0.45um filter?

YES ____ NO ____

A plastic or glass container, large enough to hold the entire extraction solution, is used and is stored at 4 degrees Celsius?

YES ____ NO ____

ACID DIGESTION FOR EXTRACTION SOLUTIONS

The digestion of the extraction solution is performed by Method 3010 of USEPA SW 846, "ACID DIGESTION OF AQUEOUS SAMPLES AND EXTRACTS FOR TOTAL METALS FOR ANALYSIS BY FLAA OR ICP SPECTROSCOPY"?

YES ____ NO ____

100mL of extraction solution is placed in a beaker with the addition of 3mL of nitric acid. The beaker is covered with a ribbed watch glass and refluxed on a hotplate with additional portions of nitric acid until the digestate is light in color or until its color has stabilized?

YES ____ NO ____

The 100 mL of extraction solution is measured by pipette?

YES ____ NO ____

The extraction solution is not boiling at any time while refluxing on the hotplate?

YES ____ NO ____

COMMENTS ON ACID DIGESTION PROCEDURE:

After refluxing, the beaker is uncovered, the solution is evaporated to a low volume 5 to 10 mL not allowing any portion of the bottom of the beaker to go dry. The beaker is cooled and a small quantity of 1:1 hydrochloric acid is added 10/100mL then warm the beaker for an additional 15 minutes to dissolve any precipitate?

YES ____ NO ____

COMMENTS:

The solution is never boiling while refluxing and evaporating?

YES ____ NO ____

ACID DIGESTION BY MICROWAVE (ALTERNATIVE METHOD PROCEDURE)

The acid digestion of waste solid extracts for total metals is performed by "MICROWAVE DIGESTION"?

YES ____ NO ____

The microwave is manufactured for the primary purpose in digesting, dissolving, extracting, and drying materials for sample preparation?

YES ____ NO ____

Does the microwave use computerized fiberoptic control for the temperature and pressure parameters?

YES ____ NO ____

If it is a microwave sample preparation system; has it been approved by the USEPA in solid waste digestion for Flame Atomic Absorption or Inductively Coupled Plasma Atomic Emission analysis?

YES ____ NO ____

If a microwave sample preparation system is not used by the laboratory; is a conventional microwave used?

YES ____ NO ____

Does the conventional microwave procedure follow ASTM Standard Practice "Sample Digestion using Closed Vessel Microwave Heating Technique for the Determination of Total Recoverable Metals in Water" (ASTM D4309)?

YES ____ NO ____

Has the laboratory performed sufficient QC samples to meet precision and accuracy by EPA when using a conventional microwave for digestion procedures?

YES ____ NO ____

ANALITICAL INSTRUMENT METHOD

What type of analytical instrument is used when analyzing samples for trace metals?
CHECK ALL THAT APPLY AND INDICATE METHOD PROCEDURE USED.

FLAME ATOMIC ABSORPTION SPECTROPHOTOMETER
FURNACE ATOMIC ABSORPTION SPECTROPHOTOMETER

INDUCTIVELY COUPLED PLASMA ATOMIC EMISSION
SPECTROPHOTOMETER

COMMENTS OF INSTRUMENTATION:
MANUFACTURER/DATE-OF-PURCHASE/MAINTENANCE-OF-INSTRUMENT

SW 846 METHODS

METHOD 7000 - ATOMIC ABSORPTION METHODS

METHOD 6010 - INDUCTIVELY COUPLED PLASMA ATOMIC
EMISSION SPECTROSCOPY METHOD 7420

METHOD FOR DETERMINATION OF LEAD (AA, DIRECT ASPIRATION)

METHOD 7421 - METHOD FOR DETERMINATION OF LEAD(AA, FURNACE
TECHNIQUE)

COMMENTS ON METHOD PROCEDURES AND ALTERNATIVE METHOD
PROCEDURES:

Does the instrumentation and the method procedures meet the detection limits and
instrument sensitivity in the determination of the analyte as specified by USEPA Solid
Waste Guidelines?

YES ____ NO ____

STATISTICAL DATA REPORTING AND FINAL REPORTS

The statistics used are either computer generated or calculated; and are in the guidelines for statistical inferences as described in SW 846 Part I: Chapter One?

YES ____ NO ____

Does the final report include the following information?

YES ____ NO ____

The project information (Name of project; field sampler; date and time sampled; sample number, lot number; type of waste material; analysis to be performed; observations and remarks?

YES ____ NO ____

The statistical data on samples; the average of 4 samples, standard deviation, the appropriate number of samples, the standard error and the confidence interval?

YES ____ NO ____

The sample spike and recovery percentages for QC?

YES ____ NO ____

The minimum detection limits for sensitivity of the instrument?

YES ____ NO ____

The method of extraction (CFR 40 Method 1311)?

YES ____ NO ____

The method of digestion (USEPA SW 846 Method 3010)?

YES ____ NO ____

The method of analysis and instrument used: (USEPA SW 846 Method 6010 for ICP or Method 7000 and 7420 for FLAA)?

YES ____ NO ____