

**Mercury in Solids using RP-91C and LUMEX RA-915+
MERCURY ANALYZER**

Maine Department of Environmental Protection

Bureau of Remediation and Waste Management

Oil and Hazardous Waste Division

Standard Operating Procedure: Mercury in Solids

SOP Number: BRWM Hg02

REVISION: 1

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Approval:

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date

1.0 APPLICABILITY:

This SOP is applicable as a field test for mercury in solids. It does not contain a provision for reporting results on a dry weight basis, and therefore should not be used as the sole basis for making key project decisions. A percentage of the samples should be confirmed by fixed laboratory analysis.

For detailed operations of the RA 915 mercury analyzer please refer to SOP BRWM Hg01, MDEP Protocol For Collecting And Analyzing Mercury Vapor In Air With A Lumex Ra-915+ Mercury Analyzer.

2.0 PURPOSE

The purpose of this document is to describe the Maine Department of Environmental Protection, Bureau of Remediation and Waste Management [MDEP/BRWM] procedure for collecting and analyzing **solid** samples for mercury.

3.0 DEFINITIONS

- 3.1 SOP: Standard Operating Procedure
- 3.2 MDEP: Maine Department of Environmental Protection
- 3.3 Hg: Mercury

4.0 RESPONSIBILITIES

All Bureau Staff must follow this procedure when using the Lumex RA-915 for analysis of mercury in soil. Their respective supervisors and managers are responsible for ensuring that they are familiar with and adhere to this procedure, and receive the appropriate training and guidance to conduct fieldwork.

5.0 PROCEDURES:

- 5.1 Initial Instrument Check/ Maintenance
Follow instructions in SOP BRWM Hg01, MDEP Protocol For Collecting And Analyzing Mercury Vapor In Air With A Lumex Ra-915+ Mercury Analyzer for calibration check, background air, and instrument maintenance. These are considered basic instrument procedures undertaken each time the instrument is used to assure proper function.
- 5.2 Set up the equipment as described in section IV of OhioLumex Standard Operating Procedure OL-130.1, Using RP-91C Attachment for the Determination of Mercury in Solids, 09/02/2001.
- 5.3 Calibration
 - A calibration range must be set for the project. A blank plus at least three standards bracketing the applicable range should be run.
 - Calibration standards are produced as dilutions from purchased stock standards, and standard information recorded in the appropriate field notebook. All standards must be produced from NIST traceable material

using Hg free methanol. Using a 1000 ng/ul stock standard, the following dilutions may be used as working standards:

Standard Concentration [ng/ul]	Volume Methanol [ml]	Volume stock [ul]
2	1 ml [minus 2 ul]	2
5	1 ml [minus 5 ul]	5
10	1 ml [minus 10 ul]	10

- Follow steps A through E in section V of OhioLumex Standard Operating Procedure OL-130.1, Using RP-91C Attachment for the Determination of Mercury in Solids, 09/02/2001 [OhioLumex SOP].
- Add 0.1g blank soil into a quartz sample boat and introduce 10 ul Hg free methanol.
- Allow the methanol to evaporate and press Start in the Integration window; then insert the boat in the oven.
- Follow steps G, H & I in section V of OhioLumex SOP.
- Introduce the first standard into a room temperature boat containing 0.1g blank soil and allow it to evaporate completely.
- Follow steps K & L in the OhioLumex SOP.
- Remove the standard boat from the oven and click the Table button on the Program toolbar. Double click the Description field for entry number 2. Select Standard, and the cursor will be present after Std__ at this point type in the standard concentration in parts per billion [ppb]. Enter the number of microliters [ul] standard introduced in the M column and press Enter.
- Introduce the next standard into a room temperature boat containing 0.1g blank soil and allow it to evaporate completely. Then repeat the steps K through entering standard information in the Table. These steps are repeated until all standards have been run and recorded.
- Follow steps O & P in the OhioLumex SOP to complete the calibration.

5.4 Analysis

- Use an appropriate sample size for the detection limit required by the project. Sample size should be kept under 0.2g. A sample size of 0.1g will yield a detection limit well below 1 ppm.
- Follow all steps in section VI of the OhioLumex SOP.

5.5 Quality Control

- Initial instrument check samples must be performed as in section 5.1. Acceptance parameters are listed in SOP BRWM Hg01.
- Balance accuracy should be verified each day of use by measuring the mass of a class s 1g weight. Mass should be within 0.01g accuracy.
- A reagent blank must be analyzed and show no Hg contamination at or above the reporting level of the test.
- Initial calibration curve must include at least three calibration standards. The initial curve must have an r^2 value of 0.95 or higher.
- Continuing calibration standards must be analyzed every 8 hours of operation and should have a result $\pm 20\%$ true value.
- No results should be reported outside the range of the calibrants. If samples results are higher than the highest standard, either a smaller sample should be analyzed or a higher calibration standard should be analyzed and included in the calibration curve.
- Each person that analyzes samples must run an initial demonstration of capability that includes analyzing four samples with the same known

concentration of Hg. Acceptable accuracy [$\pm 20\%$ true value] and precision [$\leq 20\%$ RSD] must be demonstrated. This demonstration should be repeated on an annual basis.

- A percentage of filed test results should be confirmed by fixed laboratory analysis. The confirmation sample percentage should be set for each project.

5.6 Documentation

- Results of an initial demonstration of capability should be saved either as a hard copy report or in an electronic file by the analyst.
- All calibration standards should be recorded in bound field notebooks. Documentation should include concentration, expiration date and lot number of the stock standard as well as concentration and preparation steps for each of the working standards.
- Instrument check sample results should be recorded in bound field notebooks.
- Results of all samples with accompanying blank and calibration standards should be saved either as a hard copy report or in an electronic file by the analyst.

6.0 REFERENCES

OhioLumex Standard Operating Procedure OL-130.1, Using RP-91C Attachment for the Determination of Mercury in Solids, 09/02/2001

MDEP Protocol For Collecting And Analyzing Mercury Vapor In Air With A Lumex Ra-915+ Mercury Analyzer, Revision 1, 04/02/2002

