

# **Mercury in Water using the Lumex RA 915+Mercury Analyzer and RP-91 Attachment for Liquids**

**Maine Department of Environmental Protection**

**Bureau of Remediation and Waste Management**

Standard Operating Procedure: Mercury in Water  
SOP Number: BRWM Hg03  
REVISION: 1  
DATE: April 26, 2005  
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## APPLICABILITY:

This SOP is applicable as a field lab test for mercury in water. 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.

## 1.0 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 field mercury analysis of water samples.

## 2.0 DEFINITIONS

- 2.1 SOP: Standard Operating Procedure
- 2.2 MDEP: Maine Department of Environmental Protection
- 2.3 Hg: Mercury

## 3.0 RESPONSIBILITIES

All Bureau Staff must follow this procedure when using the Lumex RA-915 RP-91 attachment for analysis of mercury in water. Bureau supervisors and managers are responsible for ensuring that staff is familiar with and adheres to this procedure, and receive the appropriate training and guidance to conduct fieldwork.

Staff must complete an initial demonstration of capability as defined in section 5.5 before using this procedure in the field.

## 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 verification, background air analysis, and instrument maintenance. These basic instrument procedures should be undertaken before the instrument is transported to the field to assure proper function.
- 5.2 Set-up [on site]
  - Field requirements include a 20A/120V AC circuit and a supply of mercury free water.
  - Set up the equipment as described in section IV of OhioLumex Standard Operating Procedure OL-120, Using RP-91 Attachment for the Determination of Mercury in Liquids, 09/10/2001.
  - For step H, add 20 ml SnCl<sub>2</sub> reagent instead of 15 ml.

- Set the Lumex RA 915+ Optical Bridge to II.
- Connect the computer and make sure RA-915P software has been installed [look for the icon on desktop]. Software has been installed on Technical Services T-7 laptop computer.

### 5.3 Calibration

- A calibration range must be set for the project. A blank plus three standards bracketing the applicable range will typically 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 water. Dilute the 1000 ng/μl stock standard to 0.5 ng/μl by adding 0.5μl stock to 1 ml mercury free water. Serial dilutions can be made from the 0.5 ng/μl stock standard as follows:

Standard Concentration	Volume Water	Volume stock
1 μg/L	1 ml	2 μl
2 μg/L	1 ml	4 μl
4 μg/L	1 ml	8 μl
5 μg/L	1 ml	10 μl

- Standards are made in a 5ml syringe by drawing in 1 ml of mercury free water and adding the appropriate volume of stock using a 10 μl syringe.
- Open the RA-915P software and select the **Liquid** mode from the main menu.
- Familiarize yourself with the toolbar. The name of the function will appear as text as you place the cursor over each tool icon.
- Click **Run**  button on the **Graph** toolbar. The integration window will pop up.
- Click the **Baseline Check**  button on the **Control** toolbar. Wait 15 seconds and then click the button again to establish baseline.
- Press **Start** in the Integration window then inject 1 ml blank into impinger # 1. Wait 60 seconds and press **End**.
- Press the **Table** button on the Program toolbar. This will bring up the Liquid Analysis window.
- In row 1 of the table, double click on the *Description* field, which will bring up a dialog box with two choices, BLANK and Std\_\_. Click on **BLANK** to choose this description. Be careful to follow this step carefully. The BLANK and Std\_\_ descriptions need to be inserted exactly into the *Description* field or the computer software will be unable to correctly recognize them.
- In the *V, ml* field type 1.
- Click **Graph**  on the **Table** toolbar. This will return you to the integration window.
- Repeat the baseline check step: Click the **Baseline Check** button on the **Control** toolbar. Wait 15 seconds and then click the button again to establish baseline.
- Press **Start** in the Integration window then inject 1 ml of the 1 μg/L standard into impinger # 1. Wait 60 seconds and press **End**.

- Press the **Table** button on the **Program** toolbar. In row 2, double click on the *Description* field and choose **Std\_\_**. Make sure the cursor is flashing at the end of **Std\_\_** and add the concentration in ng/L. In the case of the 1 µg/L standard the entry would look like **Std\_\_1000**. Again, this description needs to be exactly correct for the computer software to recognize it.
- Repeat the previous four steps for the remaining standards.
- When all standards are run and logged into the Liquid Analysis window table click the **Select** button on the **Table** toolbar. Click on the row containing the first standard then hold down the Shift button and press the down arrow to highlight all the standards.
- Click the **Cal Graph** button on the **Calculation** toolbar. At this point a calibration graph will appear. Click the **Apply** button. Save the calibration graph as a bitmap file, click **Exit** and click the **Yes** button to save the calibration coefficients.

#### 5.4 Analysis

- Click the **Graph**  button on the **Table** toolbar in the Liquid Analysis window to bring up the integration window.
- Click the **Baseline Check**  button on the **Control** toolbar. Wait 15 seconds and then click the button again to establish baseline.
- Press **Start** in the Integration window and inject 1 ml sample into impinger #1. Wait 60 seconds [until baseline returns to normal] and press **End**.
- Press the **Table** button on the **Program** toolbar to bring up the Liquid analysis table.
- Type in the sample description and volume into the correct fields in the table. **Note:** if you try to click on the *Description* field and the entire row is highlighted, click the **Edit/Select** button on the **Table** toolbar.
- Click the **Select** button and highlight the row containing sample data.

Click the **Calculation**  button on the **Table** toolbar and the Hg concentrations in ng/L will be reported in the C field.

- Analyze a check sample for every 10 samples.
- After 10 samples and the check sample turn off the pump and drain contents of impinger #1 into a waste bottle. To analyze more samples, add more SnCl<sub>2</sub> reagent [1 ml reagent for every sample—20 ml maximum] to impinger #1 and turn the pump back on. Wait five minutes before analyzing more samples.
- Export data to an EXCEL spreadsheet to save.

#### 5.5 Quality Control

- Initial instrument check samples must be performed as in section 5.1. Acceptance parameters are listed in SOP BRWM Hg01.
- A reagent blank must be analyzed and show no Hg contamination at or above the reporting level of the test.
- A calibration curve is generated each day samples are analyzed. A curve generally includes three calibration standards with an r<sup>2</sup> value of 0.90 or higher.

- Continuing calibration standards must be analyzed every 10 samples 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 [ $\pm 30\%$  true value] and precision [ $\leq 20\%$  RSD] must be demonstrated. This demonstration should be repeated on an annual basis.
- A percentage of field 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 EXCEL file.

## 6.0 REFERENCES

- OhioLumex Standard Operating Procedure OL-120, 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
- RP-91 Attachment User's Manual, Lumex Ltd. Company, 2001