
Testing Mercury (Hg) against soil organic matter

In the past few decades, mercury was found at concerning levels in many parts of the environment. Toxins like mercury are important to monitor in watersheds as they can be a sign of watershed quality. In order to manage watersheds with high mercury levels, specifically in fish, wildlife, water and soils, it's important to understand where it accumulates. The purpose of this lab is to look at different sample sites and to observe/compare the levels of Hg found.

The methods used to calculate mercury levels is by thermal decomposition, catalytic conversion, amalgamation and atomic absorption spectrophotometry.

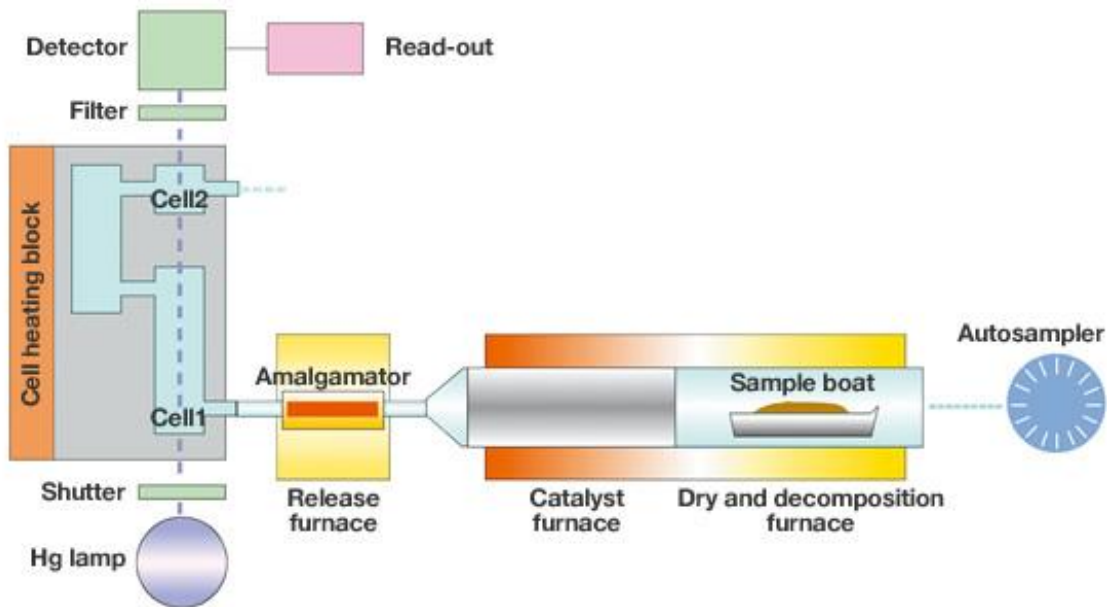


Figure 1. Schematic of DMA-80 instrument mercury sampling (Milestone, 2014).

EQUIPMENT AND SUPPLIES

- Soil samples
- Scale
- Mortar and pestle
- DMA-80 Direct Mercury Analyzer
- Nickel sample boat
- DORM-2, PACS-2 and LICHEN calibration standards

HG PROCEDURE

1. You will be measuring the Hg level within the **sample (ng)** and **concentration (mg/kg)**. In order to measure the Hg in the samples the samples need to be prepped. The samples were dried and need to be crushed using a mortar and pestle. Grind your dried sample to a fine powder. Before and after each sample, wash and dry the mortar and pestle with deionized water, this will prevent cross-contaminations.
2. To measure the Hg, a DMA-80 Direct Mercury analyzer will be used (Figure 2). Weigh out samples into nickel sample boat. Zero the scale with the boat on it and use the following weights for your samples:

Soil = 0.08 g - 0.09 g
Twigs = 0.08 g - 0.09 g
Hair = 0.0051 g - 0.011 g



Figure 2. DMA 80 Direct Mercury Analyzer.

3. Prior to running samples, the machine needs to be calibrated. Use a standard to calibrate the Direct Mercury Analyzer prior to analysis. LICHEN for twigs, PACS-2 for soil, DORM-2 for Hair and Fish. Each standard will be weighed and analyzed in the DMA 80, prior to running samples to make sure the machine is calibrated to the type of samples.

Table 1. Calibration parameters for standards.

Standard	C mg/kg	Weight, g
LICHEN	0.20	0.1
DORM-2	4.64	0.0040
PACS-2	3.04	0.0070

4. Load all of the samples into the auto sampler for analysis. Make sure to note the order the samples are placed in the auto sampler. Place a blank boat between every few samples. This will clean out any contaminant buildup between samples. Total analysis for each sample will take approximately 5 minutes (Table 2). The outputs will be in measured Hg in parts per million (ppm).

Table 2. Analysis of operating parameters

Parameter	Settings
Drying temperature/time	10 sec @ 160 °C
Decomposition time	2 min 30 sec
Waiting time	60 sec

SOIL ORGANIC MATTER (Carbon and LOI)

Organic matter increases water and nutrient retention in soils and can act as a buffer against soil acidification. It is an important source of nitrogen and phosphorus and aids in the detoxification of pesticides. The productivity of a soil generally increase with an increasing content of organic matter. DOM (or dissolved organic matter) is found to have a strong affinity to Hg and given it is often found in aquatic environments, it is known to bind to metals strongly, affecting **solubility**, **mobility**, and **toxicity**.

Methods of organic matter determination are based on one of the following principles:

1. **LOI (loss of ignition)** - The weight loss of soil on removal of the organic fraction of the soil. Removal of the organic fraction can be accomplished by ignition of soil samples in a muffle furnace. This method, however is subject to considerable errors cause by volatilization of substances other than organic materials (e.g., crystalline H₂O).

To calculate LOI the following procedures are done:

- I. Weighing out approximately 2.0g of dried soil sample into a ceramic crucible. Place the crucibles into a muffle furnace and ash the samples at 600°C for 6 hours. Remove from muffle furnace and allow to cool before weighing again.
 - II. $LOI = \frac{\text{mass of crucible \& ash} - \text{mass of empty crucible}}{\text{mass of crucible \& sample} - \text{mass of empty crucible}} \times 100$
2. **Carbon %** - The determination of a constituent which comprises a relatively constant percentage of soil organic matter. For example, the percentage of carbon in soil organic matter is relatively constant. Therefore, carbon determinations are extensively used to estimate the organic matter content in soil. Analyses of a large number of samples have indicated that soil organic matter contains, on the average, 58% carbon. From this value, a conversion factor of 1.72 (=100/58) is obtained.

To calculate the Carbon the following is done:

- I. The sample is combusted with oxygen in a LECO Induction furnace. Approximately 97% of the carbon is oxidized to CO₂, and about 3% combusts to CO, which is catalytically converted to CO₂. During combustion, the developing gasses are trapped in a closed loop at the exit of the combustion chamber. Only the CO₂ gas is detected in the resulting gas mixture by a CO₂ detector. Here, a special light source is used to pass light with an appropriate infrared (IR) wavelength to the detector. In the absence of CO₂, the light energy received by the detector is maximum. During combustion, the IR absorption of CO₂ causes a loss of energy, resulting in a loss of signal. This loss is proportional to the concentration of the CO₂ gas evolved. This proportional change is electronically processed to be displayed as percent carbon (% C) on of the instrumental readouts.

NOTE: For this lab, you will be receiving both the **%C** and the **LOI** content based on weight loss of soil OM.

REPORT

Look at the Hg levels in the soil samples across the 10 sites in Fredericton. Also look at the statistical correlation between the Hg levels and OM/Carbon %. There should be some sort of correlation. Also look at previous lab results to see if there are any relationships. The report should contain valid research with properly references to back up your findings.

Report Outline

The purpose of this lab is to compare the results of mercury to organic matter across all of the study sites. In your analysis, try and showcase any trends that form (i.e. quality trends by soil type, by location to pollutants, etc...). Look at correlations between Hg and OM (statistically) and also try to see any relationships with past data (i.e. EC?). In your **introduction**, introduce the effects of Hg, specifically on the soil and also introduce any hypothesis/expectations for the results. Your introduction should have good background research with *quality* references. **Methods** should include a detailed overall explanation at what was done in the lab with the DMA, the loss of ignition method, as well as the dry combustion LECO method. You should go over the internal functions of the DMA and make sure to explain all the steps of how it works. Also include any explanations needed for statistical analysis work. **Results** should include a table of results (might be suitable for an appendix depending on size), potential maps showing visual trends of data results, as well as any statistical results. Statistical results would include a table or mention of trend, and graphics with trends. (*Please do not forget to talk about results in this section. Don't just plop tables and graphs here, you need to introduce and talk about them*). Your **discussion** section is where you talk about your results, and elaborate on the trends/relationships found. This section is important and should include research and references to support your findings. You should also focus on the belief and trust regarding the outcomes of the results i.e. reliability, uncertainty, representativeness, appropriateness. Discuss the results but also the methods in which you found them and their reliability. **Conclude** your report with stated objectives and the overall lesson learnt. (Don't forget appendices and reference)

Report worth:

Introduction - 20

Methods - 15

Results - 20

Discussion - 30

Conclusion - 5

Title, References, Appendix together: 10

Notes: You need a title page with a title that is descriptive and creative. Make sure to have **appropriate** references. We discourage the use of web based references. Utilize the library and the online archives to find actual scientific references and books. Make sure to have a properly formatted list of references. Appendices should each have a title. Number your pages, and remember that page 1 starts with your writing, not with the table of contents (if you chose to add one). This report is marked from a 3rd year university level, therefore be sure to write and format appropriately.

Marking Guide

Introduction (20)

- Did they introduce the topic and do some background research on the impact of mercury in soil?
- Did they talk about the relationship between organic matter and Hg?
- Are there references in their research? (Proper references)
- Did they state the objective of the report? (Where? Why? How?)

Methods (15)

- How were samples prepared for processing? What machine was used to calculate mercury and OM?
- How does the machine work? (Explain the internal processes involved with calculating Hg from soil)
- Did they mention about how to calibrate the DMA-80?
- Did they keep the writing in the proper tense? No first person.

Results (20)

- Are the figures introduced before they are placed in text? (i.e. need to reference them before they show up)
- Are the figures formatted properly? (Proper axis, significant digits, axis labels, colour/pattern to differentiate, clean looking?)
- Did they do any statistical analysis between OM and Hg? This is an important step in results as it shows critical thought. They need to look at the relationship and do some regression/statistics to show if their data shows trends to support literature.
- Quick mention of trends in the results? Elaborate on this in the discussion but a quick mention of drastic differences. The “what” goes in results, the “why” in discussion.

Discussion (30)

- Is there a discussion about the results? Do they go into detail about the “WHY”?
- Did they go over any trends found and explain them with research to support their explanations?
- Are there appropriate references backing up their discussion?
- Did they deal with the matter of belief and trust regarding the results? (Reliability, uncertainty, representativeness, context)
- Did they suggest ways to enhance reliability? How would they make the lab better? (Sampling? Procedure? Human errors?)
- Why is mercury in soil important? Why is it important to know the relationship between OM and Hg? How does this all effect watershed management?

Conclusion (5)

- What was learned from looking into the mercury/OM levels at each site?
- No personal comments about what they feel, but just what was accomplished in the lab?

Title, Appendix & References (10)

- Is the title creative and informative?
- Does it specifically state what will be looked at in the report?
- Is the bibliography formatted properly? (Alphabetical order, APA, consistent format)
- Are all of the reference academic? (I.e. books and journals, no URL/wiki references)
- Is the appendix formatted properly? (I.e. proper titles for each appendix)
- If necessary, data should be in appendix if it's too large or complex for the body of the report.
- Is the paper grammatically correct? Does it flow well? Proper paragraph and sentences? Spelling?