Determination of Mercury in Soil Samples

Utilizing Direct Mercury Analysis in as little as 6 Minutes per Sample

Mercury analysis of soil and sediment samples is becoming increasingly important due to mercury's toxicity and the adverse eff ects it can have on human health. Dumping of contaminated wastes, effluents from manufacturing processes, and combustion of fossil fuels are a few common sources of soil contamination. Remediation of mercury contaminated sites require specific procedures which vary based on the levels of mercury found in the soil and sediment samples from the site.

Summary

Conventionally, to analyze mercury in soils and sediments, the samples need to be digested in acid and are then analyzed using either cold vapor atomic absorption spectroscopy (CVAA) or an ICP system. While these are tried and tested methodologies, the need to digest samples prior to analysis proves to be a very costly and a time and labor-intensive step. Additionally, the user is required to ensure that the digestion unit is properly maintained, safe working protocols are followed, and waste acids are properly disposed. This ultimately hinders the productivity of the lab. Direct mercury analysis eliminates these challenges completely, while providing accurate and reproducible data.

The main benefits of direct mercury analysis include:

- Reduced Sample Turnaround (6 Minutes)
- No Sample Preparation
- Reduced Hazardous Waste Generation
- Reduction of Analytical Errors
- General Cost Savings (70% versus CVAA)

Instrumentation

The DMA-80, Direct Mercury Analyzer, as referenced in EPA Method 7473, from Milestone (www.milestonesrl.com) was used in this study (Figure 1). Direct Mercury Analysis involves an integrated sequence of combustion, catalytic conversion, amalgamation and detection using Atomic Absorption.

Figure 1. Milestone's DMA-80 Direct Mercury Analyzer

The DMA-80 features a circular, stainless steel, interchangeable 40 position autosampler for virtually limitless throughput and can accommodate both nickel (500 mg) and quartz boats (1500 uL) depending on the requirements of the application. It operates from a single-phase 110/220V, 50/60 Hz power supply and requires regular grade oxygen as a carrier gas.

As the process does not require the conversion of mercury to mercuric ions, both solid and liquid matrices can be analyzed without the need for acid digestion or other sample preparation. The fact that zero sample preparation is required also eliminates all hazardous waste generation. All results, instrument parameters including furnace temperatures, are controlled and saved with easy export capabilities to Excel or LIMS.

Principles of Operation

Direct mercury analysis incorporates the following sequence: Thermal Decomposition, Catalytic Conversion, Amalgamation, and Atomic Absorption Spectrophotometry.
Controlled heating stages are implemented to first dry and then thermally decompose a sample introduced into a quartz tube.
A continuous flow of oxygen carries the decomposition products through a hot catalyst bed where halogens, nitrogen, and sulfur oxides are trapped.
All mercury species are reduced to Hg(0) and are then carried along with reaction gases to a gold amalgamator where the mercury is selectively trapped. All non-mercury vapors and decomposition products are flushed from the system by the continuous flow of gas.
The amalgamator is subsequently heated and releases all trapped mercury to the single beam, fixed wavelength atomic absorption spectrophotometer. Absorbance is measured at 253.7 nm as a function of mercury content.

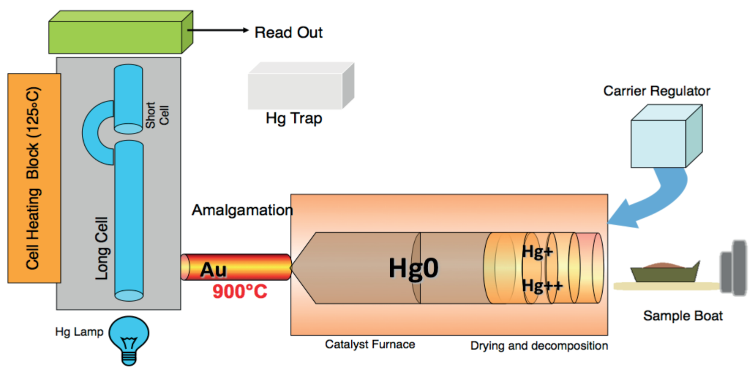

Figure 2. An Internal Schematic of Milestone's DMA-80.

Calibration

The DMA-80 can be calibrated using aqueous standards or Standard Reference Materials (SRM's). The DMA-80 used for this experiment had a tri-cell spectrophotometer and covered a dynamic range of 0.001-1500 ng Hg. Calibration was performed using diff erent volumes of 1ppm and 0.1 ppm stock solutions, prepared from an NIST traceable 1000 ppm stock solution (VHG Labs).

Experiment and Results

In this experiment, 5 diff erent soil samples were analyzed 4 times to study the concentration

range of mercury in soil.
A standard solution of 0.500 ppm was analyzed before and after the sample analysis. The results and temperature profile method are shown in Tables 1 and 2.

Sample	Concentration (ppm)
Standard Pre-check (0.500 ppm)	0.491
A	0.0940+/-0.02
B	0.1228+/-0.02
C	0.1423+/-0.02
D	0.0900+/-0.02
E	0.9620+/-0.02
Standard Post-check (0.500 ppm)	0.495

Table 1. Analysis of Unknown Soil Samples

Step	*Time (hh:mm:ss)*	*Temperature (°C)*
1	00:00:10	250
2	00:00:30	250
3	00:01:30	650
4	00:01:30	650
Max Start Temperature: 250°C Catalyst Temperature: 600°C Purge Time: 60 seconds Amalgamation Time: 12 seconds at 900°C Oxygen Flow: 120 mL/m		

Table 2. Temperature Profile Method

Conclusions

A lab analyzing mercury in soil samples is required to maintain high throughput while keeping its costs under control. The DMA-80 is an excellent tool as it yields results in ~6 min/sample and proves to be proficient, matrix-independent and cost effective while completely eliminating the sample preparation challenges posed by conventional mercury analysis techniques.

About Milestone
Our full suite of Microwave Sample Prep productivity tools is backed by over 50 patents and nearly 30 years of industry expertise. Milestone is committed to providing safe, reliable and fl exible platforms to enhance your productivity. Over 20,000 customers worldwide look to Milestone to improve their metals digestions, organic extractions, mercury analyzers or synthetic chemistry processes.

Further Reading

Please visit our Hg info center for complete access to application notes, technical papers, as well as links to valuable resouces for mercury testing.

Go to www.milestonesrl.com/library/mercury.

To learn more about mercury and other related topics, feel free to visit these websites.

EPA Method 7473
http://www.epa.gov/waste/hazard/testmethods/sw846/pdfs/7473.pdf

ASTM Method D6722-01
http://www.astm.org/Standards/D6722.htm

EPA Mercury
http://www.epa.gov/mercury/

Methyl Mercury
http://en.wikipedia.org/wiki/Methylmercury

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