The Determination of Mercury in Sediments, Soils and Sludge using the Hydra-C Mercury Analyzer

Introduction

The accurate determination of mercury in sediments and sludges is important for the proper and safe disposition of these materials. The Hydra-C Direct Mercury Analyzer provides fast, simple and convenient analyses of these materials without sample pretreatment or production of hazardous chemical waste. A typical sample analysis takes only about 5 minutes and requires virtually no sample preparation. The Hydra-C employs U.S. EPA Method 7473 which has been approved for both laboratory and field analysis.

Instrumentation

The Hydra-C (shown to the right) is fully automated for unattended operation, comes complete with a 70 position autosampler and has on-the-fly loading capability for virtually unlimited sampler capacity. Hydra-C operates from a single 110/220V, 50/60 Hz power supply and oxygen supplied at 15-20 psig. All instrument operating parameters (e.g. furnace temperatures, gas flows, autosampler control) and process stages are computer controlled for ease-of-use.

Hydra-C Mercury Analyzer

Principle of Operation

Hydra-C operates on the principle of thermal decomposition to liberate elemental mercury from solid or liquid samples. Figure 1 shows a schematic diagram of Hydra-C's principle of Operation. First, a weighed sample is deposited into a sample boat and introduced into the decomposition furnace. After the furnace is closed, an oxidant (typically oxygen or compressed air) begins to flow over the sample and the furnace temperature is ramped in two stages; first to dry the sample, then to decompose it.

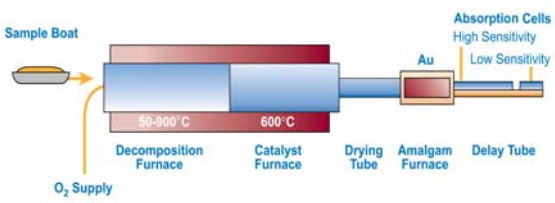

Figure 1. Schematic of Hydra C's Principle of Operation

The analytical process typically involves combusting (thermal decomposition) the sample at high temperatures with oxygen; although, for some applications gentle heating of the sample in air is adequate to release the mercury. During the combustion step the evolved gases are carried through a heated catalyst to produce free mercury while removing halogens, nitrogen oxides, and sulfur oxides. The remaining combustion products including elemental mercury (Hg) are swept first through a dryer and then through a gold amalgamation trap where all elemental mercury is captured. Following the decomposition step, the amalgamation trap is heated and the free mercury is carried into an

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atomic absorption spectrometer. The mercury level is reported using a wide dynamic range detection system that operates from 0.005 ng (its detection limit) to its upper limit of 1000 ng. For applications requiring significantly higher detection capability an optional high range detection system is available which can be used to analyze samples containing up to 20,000 ng of Hg

Experimental

Table I shows the instrument parameters employed for soils and sludges. For this analysis nickel boats were used for all samples.

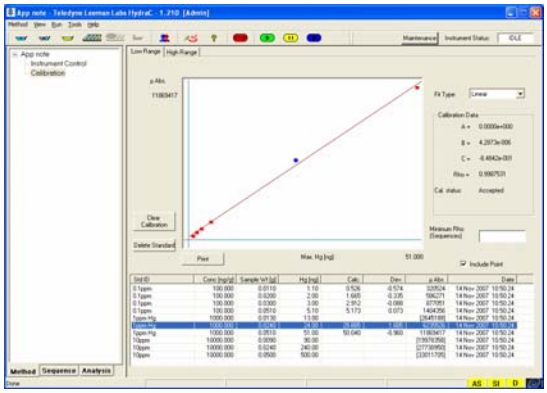
Table I: System Parameters

Parameter	Setting
Dry	300°C for 45 sec.
Decomposition	800°C for 150 sec.
Catalyst	600°C
Catalyst Wait Period	60 sec.
Gold Trap	700°C for 30 sec.
Measurement	90 sec.
Oxygen Flow	300 ml/min

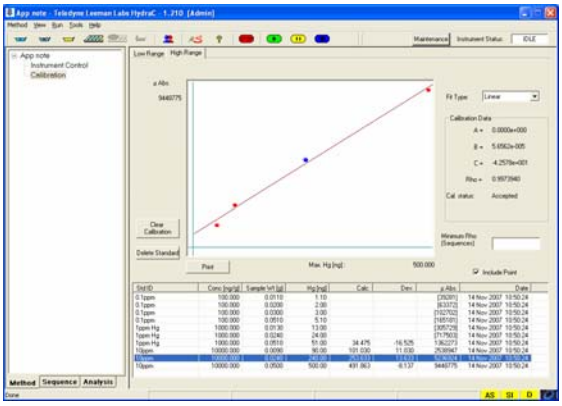
Calibration

Calibration was completed using aqueous standards prepared in 1% HNO_3. Working standards included a blank, 0.1, and 1.0 ppm solution. The calibration curve is displayed as microabsorbance vs. total mercury injected.

Low Concentration Range (0-50ng)



High Concentration Range (50-500ng)



Results

Two certified reference materials were analyzed for mercury and the results appear below in Table II.

Table II: Certified Reference Materials

Sample Matrix	CRM Name	Certified Concentration (PPM)	Measured Concentration (PPM)
Sediment	NIST 8406	0.060	0.0607
Soil	NIST 2709	1.4	1.52

Conclusion

Results obtained showed excellent correlation with certified values and were obtained without any sample treatment in about 5 minutes per sample.