

Mercury (Hg) in Sediment (NIST 2703)

Method ID: USEPA 7473
Category: Environmental
Technique: CAA

Summary

This technical note will describe the analysis of total mercury in sediment (NIST 2703), using the Teledyne Leeman Labs Hydra IIc mercury analyzer. This method utilized the moisture control system as described in AN1701 ([viewable here](#)) to enhance the performance of the Hydra IIc in standard, direct combustion mode for total mercury in this matrix.

Direct combustion mercury analysis, as described in USEPA 7473, is a simple method which eliminates lengthy sample preparations and the hazardous wastes generated from wet chemistry techniques. With no sample pretreatment, total mercury results are obtained faster and at less expense using the Teledyne Leeman Labs' Hydra IIc mercury analyzer for direct combustion atomic absorption (CAA).

Weighed samples were introduced into the analyzer using an automated sequence. The unattended analysis of samples were completed at a rate of ~ 6 min / sample.

Instrumentation

Hydra IIc CAA mercury analyzer, Envoy software version 2.2, quartz boats (calibration), nickel boats (samples), analytical balance, hot plate, watch glasses, disposable spatulas, pipettes/tips and labware/reagents for aqueous calibration standard preparation.

Method Parameters

	°C	Seconds	Other
Oxygen Flow (mL/min)			350
Drying	300	30	
TempRamp*		60	
Decomposition	800	120	
Catalyst	600	20	
Amalgamator	700	30	
Integration		80	
LowPeakAbsLimit*			380000
NafionFurnaceTemp*			600
EluteWarmTempOverride*			175

*Adjusted in, or added to, the startup.ini text file located in the Envoy folder.

Calibration

Aqueous intermediate standards were prepared in 1% HNO₃ acid for mercury stability. Various weights of

intermediate calibration standards were added to quartz boats for total mass in ng of Hg, as listed below. Both Low and High Concentration ranges utilized a quadratic fit.

Low Concentration	Blank, 0.1, 0.5, 1, 5, 10, 20, 50 ng
High Concentration	Blank, 50, 100, 200, 400, 600, 800, 1000 ng

Procedure

1. Homogenized sample in the container
2. Tare boats and add sample to boats
3. Load boats onto the sample boat shuttle
4. Run Hydra IIc using parameters listed with an automated sequence
5. Determine percent moisture in CRM for data analysis

Sample Weight

Average sample weight range was > 0.08 g but less than 0.12 g.

Results

	mg/Kg	
Mess-1 (0.091 mg/Kg)	0.096	105.5 % Recovery
SRM 1633c (1.005 mg/Kg)	0.957	95.2 % Recovery
NIST 2703 (1)	0.456	
NIST 2703 (2)	0.443	
NIST 2703 (3)	0.457	
NIST 2703 (4)	0.457	
NIST 2703 (5)	0.449	
NIST 2703 (6)	0.487	
NIST 2703 (7)	0.506	
Avg	0.465 ± 0.017 @ 95 % Confidence	
STDEV	0.023	
MDL	0.056 @ 95 % Confidence	
Min	0.443	
Max	0.506	
Mess-1 (0.091 mg/Kg)	0.096	105.5 % Recovery
SRM 1633c (1.005 mg/Kg)	0.956	95.2 % Recovery

Conclusion

The calibration curve check standard of 105.5% and 105.5% recovery for MESS-1, and 95.2% and 95.2% for NIST 1633c demonstrate the system is in control and stable in both the high and low sensitivity calibration ranges. The certified value for NIST 2703 is 0.474 ± 0.066 mg/Kg at 95%. This analysis of NIST 2703 had a recovery of 0.465 ± 0.017 mg/Kg, which is 98.1% of the certified value. The Hydra IIc in standard mode is an ideal system for the determination of total mercury in sediment, NIST 2703.