

Mercury (Hg) in Soil (NIST 2710)

Method ID: USEPA 7473
Category: Environmental
Technique: CAA

Summary

This technical note will describe the analysis of total mercury in soil (NIST 2710) in the high mg/Kg range, using the Teledyne Leeman Labs Hydra IIc mercury analyzer. This method used 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 was completed at a rate of ~ 5 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)			500
Drying	300	30	
TempRamp*		60	
Decomposition	800	120	
Catalyst	600	20	
Amalgamator	700	30	
Integration		70	
LowPeakAbsLimit*			1100000
HighSampleAutoCleanAbsLimit*			30000000
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 used a quadratic fit.

Low Concentration	Blank, 1, 5, 10, 20, 50, 100 ng
High Concentration	100, 200, 600, 800, 1000, 1500, 2000 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 ~ 0.06 g.

Results

	mg/Kg	
NIST 2703 (0.474 mg/Kg)	0.526	111 % Recovery
NIST 2704 (1.44 mg/Kg)	1.262	88 % Recovery
NIST 2710 (1)	32.356	
NIST 2710 (2)	31.870	
NIST 2710 (3)	31.885	
NIST 2710 (4)	31.607	
NIST 2710 (5)	33.355	
NIST 2710 (6)	33.166	
NIST 2710 (7)	33.990	
Avg	32.604 ± 0.67 @ 95 % Confidence	
STDEV	0.905	
MDL	2.214 @ 95 % Confidence	
Min	31.607	
Max	33.990	
NIST 2403 (0.474 mg/Kg)	0.465	98 % Recovery
NIST 2704 (1.44 mg/Kg)	1.325	92 % Recovery

Conclusion

The calibration curve check standard of 111% and 98% recovery for NIST 2703, and 88% and 92% for NIST 2704 demonstrate the system is in control and stable in both the high and low sensitivity calibration ranges. The certified value for NIST 2710 is 32.6 ± 1.8 mg/Kg at 95% confidence. This analysis of NIST 2710 had a recovery of 32.604 ± 0.67 mg/Kg at 95% confidence, which is 100% of the certified value. The Hydra IIc in standard mode is an ideal system for the determination of total mercury in soil, NIST 2710 a high mercury content soil.