TELEDYNE LEEMAN LABS
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Technical Note: HG/ENV-2101

Mercury (Hg) in Soil (NIST 2710)

Method: EN 16175-1 **Category: Environmental** **Technique: CVAA**

Summary

This technical note demonstrates the analysis of Soil (NIST 2710) using the Teledyne Leeman Labs QuickTrace® M-7600 CVAA mercury analyzer and following the guidance in EN 16175-1.

Instrumentation

QuickTrace® M-7600 CVAA mercury analyzer (without enrichment), autosampler for unattended analysis and autosampler enclosure to prevent contamination. Stannous chloride ($SnCl_2$) reagent pump tubing was orange-yellow 0.51 mm ID (PN 15-4309-102). Sample and waste tubing was white-white 1.02 mm ID (PN 15-4308-102), QuickTrace® software version 3.2, digest tubes, analytical balance, pipette and tips, labware and method reagents for digestion and calibration standard preparation. Refer to Application Note: AN1905 – Green Chemistry: Decreased Reagent Consumption and Waste Using Reduced ID Tubing on the QuickTrace® M-7600 CVAA Mercury Analyzer (Viewable Here).

Method Parameters

Parameter	Value
Sample Uptake (sec)	25
Rinse Time (sec)	60
Gas Flow (mL/min)	300
Pump Speed (%)	100
Read Delay Time (sec)	36
Replicate Read Time (sec)	1
Number of Replicates	4

Reagents

The preparation of reagents followed EN 16174 and EN 16175-1 with the exception of the $SnCl_2$ reagent, which was prepared according to the instrument manufacturer's specification of 10% $SnCl_2$ in 7% hydrochloric acid (HCl).

Calibration

Calibration standards (0, 1, 3, 6, 12 and 20 µg/L) were prepared by adding 0, 0.1, 0.3, 0.6, 1.2 and 2.0 mL volumes of a 1000 µg/L intermediate standard to 100 mL volumetric flasks containing ~50 mL of 2.8% aqua regia, and then bringing to volume with 2.8% aqua regia.

Sample Preparation

Samples were prepared by weighing ~1.5 g of the SRM into each of seven 50 mL digest tubes. Approximately 2 mL of deionized water was added to wet the soil in each tube. In a fume hood, 10.5 mL of HCl was slowly added, followed by 3.5 mL of nitric acid (HNO_3). The tubes were gently swirled, loosely capped and then placed in a hot block for a 2-hour digestion at 95 °C. The digests were cooled, filtered and brought up to 50 mL. Dilutions (factor of 10) were prepared from the filtrates. Due to the SRM concentration, additional dilutions (factor of 10) were made using a matrix-matched 2.8% aqua-regia solution. The tubes were mixed and loaded onto the autosampler and the autosampler enclosure sealed.

Procedure

1. Prepare the samples and standards according to EN 16174 and 16175-1.
2. Perform instrument set-up and warm-up according to the *QuickTrace® M-7600 Operator's Manual*.
3. Perform a Peak Profile to optimize detection times for baseline correction and peak signal.
4. Verify tube positions and initiate the sequence.

Results

ICV (2.0 µg/L; 2nd source)	2.03	101.5% Recovery
CCV (2.0 µg/L)	2.00	100% Recovery
	mg/kg	
NIST 2710	32.80	
NIST 2710	33.28	
NIST 2710	32.89	
NIST 2710	33.56	
NIST 2710	33.47	
NIST 2710	33.17	
NIST 2710	32.81	
Avg	33.14	±0.230 @ 95%
STDEV	0.31	
MDL	0.77	@ 95%
Min	32.80	
Max	33.56	
CCV (2.0 µg/L)	2.00	100% Recovery

Conclusion

A linear calibration curve fit was used and the calibration coefficient (R^2) was 0.99989. Quality control (QC) check standard recoveries of 100.0% demonstrate that the system was in control and stable during analysis. The certified value for NIST 2710 is 32.6 ±1.8 mg/kg. The calculated recovery was 101.7%.