TELEDYNE LEEMAN LABS
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Technical Note: HG/ENV-2006

Mercury (Hg) in Soil (NIST 2711a)

Method: ISO 11466 & ISO 16722 **Category: Environmental** **Technique: CVAF**

Summary

This technical note demonstrates the analysis of Soil (NIST 2711a) using the Teledyne Leeman Labs QuickTrace® M-8000 CVAF mercury analyzer, following the guidance in ISO 16722.

Instrumentation

QuickTrace® M-8000 CVAF mercury analyzer in non-gold trap mode, autosampler for unattended analysis and autosampler enclosure to prevent contamination. Stannous chloride ($SnCl_2$) reagent pump tubing was black-black with a 0.76 mm ID (PN SP5705B). Sample and waste tubing was yellow-yellow with a 1.42 mm ID (PN SP5705A). QuickTrace® software version 3.2, digest tubes, analytical balance, pipette and tips, labware and method reagents for digestion and calibration standard preparation.

Method Parameters

Parameter	Value
Sample Uptake (sec)	23
Rinse Time (sec)	60
Gas Flow (Regulator at 35 PSI)	High Flow
Pump Speed (%)	100
Read Delay Time (sec)	34
Replicate Read Time (sec)	1
Number of Replicates	4

Reagents

The preparation of reagents followed ISO 11466 and ISO 16722 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, 0.5, 1.5, 3, 6 and 10 mL volumes of a 100 µg/L intermediate standard to tubes containing 40 mL of 2.8% aqua regia, then brought up to 50 mL.

Sample Preparation

Samples were prepared by weighing ~1.5 g of the SRM into 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 allowed to pre-digest for 16 hours before 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 2) 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 ISO 11466 and ISO 16722.
2. Perform instrument set-up and warm-up according to the *QuickTrace® M-8000 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)	1.99	99.5% Recovery
CCV (2.0 µg/L)	1.99	99.5% Recovery
	mg/kg	
NIST 2711A	7.60	
NIST 2711A	7.53	
NIST 2711A	7.76	
NIST 2711A	7.62	
NIST 2711A	7.55	
NIST 2711A	7.51	
NIST 2711A	7.58	
Avg	7.59	±0.059 @ 95%
STDEV	0.08	
MDL	0.20	@ 95%
Min	7.51	
Max	7.76	
CCV (2.0 µg/L)	1.99	99.5% Recovery

Conclusion

A linear calibration curve fit was used and the calibration coefficient (R^2) was 0.99999. Quality control (QC) check standard recoveries of 99.5% demonstrate that the system was in control and stable during analysis. The certified value for NIST 2711a was 7.42 ±0.18 mg/kg. The calculated recovery was 102.3%.