TELEDYNE LEEMAN LABS
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Technical Note: HG/ENV-2003

Mercury (Hg) in Wastewater (ERM®-CA713)

Method: ISO 17852 **Category: Environmental** **Technique: CVAF**

Summary

This technical note demonstrates the analysis of Wastewater (ERM®-CA713) using the Teledyne Leeman Labs QuickTrace® M-8000 CVAF mercury analyzer, with digestion by ISO 17852:2006(E).

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 pump 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)	22
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

Note: The photomultiplier tube (PMT) was manually adjusted down to attain a fluorescence response of ~1500 hF units for the highest calibration standard.

Reagents

Preparation of reagents followed ISO 17852. A premixed ampoule of potassium bromide potassium bromate ($KBr/KBrO_3$) reagent was used. Method volume for $KBr/KBrO_3$ and hydrochloric acid (HCl) are based on 100 mL calibration and sample volumes. Because this study used 10 mL calibration and sample volumes, $KBr/KBrO_3$ and HCl volumes were adjusted accordingly (reduced by 90%). The quantity of L-ascorbic acid was unaltered, and followed method guidelines at 0.1 mL per 10 mL sample.

Calibration

Calibration standards were prepared by adding 1.50 mL of HCl reagent and 0.20 mL of $KBr/KBrO_3$ reagent to each standard tube. 10 mL Volumes of six standards (0, 1.0, 3.0, 5.0, 7.0 and 10.0 µg/L) were then added to each tube.

Sample Preparation

Samples were prepared in the same manner as standards. 1.50 mL of HCl Reagent and 0.20 mL of $KBr/KBrO_3$ reagent were added to seven sample tubes. Due to limited quantity, 10 mL of ERM®-CA713 was then added. All tubes were capped, mixed and allowed to digest at room temperature for 1 hour. The yellow color of the solution was monitored to ensure the reagent remained in excess for the full digestion period. After 1 hour, 0.10 mL of L-ascorbic acid ($C_6H_8O_6$) reagent was added to reduce excess bromine. Again, tubes were capped, mixed and then loaded onto the autosampler. The autosampler enclosure was then sealed.

Procedure

1. Perform the digestion protocol in ISO 17852.
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 correct positioning of all standards/samples in the autosampler and initiate the sequence.

Results

	µg/L	
ICV (2.0 µg/L; 2nd source)	2.00	100.0 % Recovery
CCV (2.0 µg/L)	2.01	100.5 % Recovery
ERM®-CA713	1.85	
ERM®-CA713	1.86	
ERM®-CA713	1.85	
ERM®-CA713	1.85	
ERM®-CA713	1.86	
ERM®-CA713	1.88	
ERM®-CA713	1.86	
Avg	1.86 ± 0.0074 @ 95 %	
STDEV	0.01	
MDL	0.03 @ 95 %	
Min	1.85	
Max	1.88	
CCV (2.0 µg/L)	2.02	101.0 % Recovery

Conclusion

A linear calibration curve fit was used and the calibration coefficient (R^2) result was 0.99998. Quality control (QC) check standard recoveries of 100.5% to 101% demonstrate that the system was in control and stable during analysis. The certified value for ERM®-CA713 was 1.84 ±0.11 µg/L. Calculated recovery for this SRM was 101%.