TELEDYNE LEEMAN LABS
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Technical Note: HG/ENV-2004

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

Method: ISO 12846:2012(E) **Category: Environmental** **Technique: CVAA**

Summary

This technical note demonstrates the analysis of Wastewater (ERM®-CA713) using the Teledyne Leeman Labs QuickTrace® M-7600 CVAA mercury analyzer, with digestion by ISO 12846:2012(E).

Instrumentation

QuickTrace® M-7600 CVAA Mercury Analyzer (without enrichment), autosampler with enclosure. QuickTrace® software Version 3.2, digest tubes, analytical balance, pipette and tips, labware and method reagents for digestion and calibration standard preparation. Stannous chloride ($SnCl_2$) reagent pump tubing was orange/yellow 0.51 mm (PN 15-4309-102), while sample and waste tubing were white/white 1.02 mm (PN 15-4308-102) to reduce reagent/waste. 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)	50
Gas Flow (Regulator at 120 PSI)	200
Pump Speed (%)	100
Read Delay Time (sec)	36
Replicate Read Time (sec)	1
Number of Replicates	4

Reagents

Preparation of reagents followed ISO 12846. A premixed ampoule of potassium bromide potassium bromate ($KBr/KBrO_3$) reagent was used. Method volume for $KBr/KBrO_3$ and hydrochloric acid (HCl) were 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 hydroxylammonium chloride ($NH_2OH \bullet HCl$) was unaltered and followed method guidelines at 0.1 mL per 10 mL sample.

Calibration

Calibration standards were prepared by adding 0.1 mL of HCl 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 by adding 0.1 mL of HCl and 0.20 mL of $KBr/KBrO_3$ reagent to seven sample tubes. 10 mL Volumes of ERM®-CA713 were then added to each tube. All tubes were capped, mixed and allowed to digest at room temperature for 24 hours. The yellow color of the solution was monitored to ensure the reagent remained in excess for the full digestion period. After 24 hours, 0.1 mL of $NH_2OH \bullet HCl$ reagent was added to reduce the excess bromine. Tubes were capped and mixed again.

Procedure

1. Perform the digestion protocol in ISO 12846.
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. Load and start.

Results

	µg/L	
ICV (2.0 µg/L; 2nd source)	2.03	101.5 % Recovery
CCV (2.0 µg/L)	2.03	101.6 % Recovery
ERM®-CA713	1.985	
ERM®-CA713	1.986	
ERM®-CA713	1.988	
ERM®-CA713	1.987	
ERM®-CA713	1.989	
ERM®-CA713	1.992	
ERM®-CA713	1.996	
Avg	1.989 ± 0.0030 @ 95 %	
STDEV	0.004	
MDL	0.009 @ 95 %	
Min	1.985	
Max	1.996	
CCV (2.0 µg/L)	2.03	101.5 % Recovery

Conclusion

A linear calibration curve fit was used and the calibration coefficient (R^2) was 0.99990. Quality control (QC) check standard recoveries of 101.6% to 101.5% demonstrate that the system was in control and stable during analysis. The certified value for ERM®-CA713 was 1.84 ±0.11 µg/L. The calculated recovery was 108.1%. **Due to the small positive bias, the entire analytical batch was analyzed on the QuickTrace® M-8000 CVAF mercury analyzer. The positive bias was not observed, indicating the possible presence of bromoform, a potential absorbing compound for this CVAA method. The calculated recovery using CVAF was 101.7%.**