

# 70s Analysis of Mercury in Wastewater (ERM CA-713)

Method: US EPA 245.1

Category: Environmental

Technique: CVAA

## Summary

This technical note will describe the analysis of total mercury (Hg) in Wastewater (ERM CA-713) by USEPA 245.1 using the QuickTrace® M-7600 CVAA mercury analyzer's Green Chemistry configuration. This configuration reduces reagent consumption and waste generation and is validated in Application Note AN1905 ([Viewable Here](#)).

This method was optimized for speed with a total analysis time of 70s per sample. At this rate, 51 samples could be analyzed in 1 hour. It is also notable that only 1.18mL of sample and 3.15mL of stannous chloride (SnCl<sub>2</sub>) is needed for each analysis. Analysis of each sample generates <15 mL of waste, or ~760mL of waste per hour.

When comparing this Green Chemistry configuration to our standard method parameters and tubing set-up, SnCl<sub>2</sub> reagent consumption is reduced by ~53%, waste generation is reduced by ~40%, and the throughput is increased by more than 50%!

## Instrumentation

M-7600 analyzer and ASX-560 autosampler for automated analysis. SnCl<sub>2</sub> pump tubing was orange/yellow 0.51 mm (TLL PN 15-4309-102). Sample and waste tubing were white/white 1.02 mm (TLL PN 15-4308-102).

## Method Parameters

Parameter	Value
Sample Uptake (sec)	7
Rinse Time (sec)	63
Gas Flow (mL/min)	200
Pump Speed (%)	100
Quantitation Mode	Peak Area

## Calibration

Seven standards (0, 0.5, 1, 2, 5, 10 and 20 µg/L) were analyzed with the samples according to EPA 245.1. Calibration standards are not digested. The calibration curve must be linear with a correlation of 0.998 or better. The calibration was verified with a digested 2 µg/L second-source standard with a ± 10% acceptance limit.

## Sample Preparation

5mL of sample was added to digestion tubes containing 0.25 mL of conc. sulfuric acid and 0.125 mL of conc. nitric acid. Swirled to mix. Added 0.75 mL of 5% potassium permanganate (KMnO<sub>4</sub>) to each sample. After the purple color persisted, 0.4 mL of 5% potassium persulfate (K<sub>2</sub>S<sub>2</sub>O<sub>8</sub>) was added. Samples were digested in a graphite digestion block at 95 °C for 2 hours. After cooling to room temperature, 0.3 mL of 12%/12% hydroxylamine hydrochloride/sodium chloride (NH<sub>2</sub>OH HCl/ NaCl) was added to reduce the excess oxidizers. Samples were then vortexed and allowed to stand for 15min prior to analysis.

## Procedure

Samples and standards were loaded onto the ASX-560. Inorganic mercury was reduced to elemental mercury by excess online addition of 10% SnCl<sub>2</sub> in 7% HCl.

## Results

	µg/L
ICV (second source, 2 µg/L)	2.15 107.5 % Recovery
Wastewater (CRM CA-713) 1	2.07
Wastewater (CRM CA-713) 2	1.92
Wastewater (CRM CA-713) 3	1.88
Avg	1.96 ± 0.11 @ 95 %
STDEV	0.10
Min	1.88
Max	2.07
CCV (primary source, 2 µg/L)	2.12 106.0 % Recovery

An MDL study was performed in accordance with CFR 40 Part 136 Appendix B resulting in an MDL of 0.021 µg/L.

The results were calculated using blank subtraction and a force-through blank calibration algorithm.

## Conclusion

The QC recoveries of 107.5% and 106.0% demonstrate the system is in control and stable for analysis of mercury. The certified value for ERM CA-713 is 1.84 ± 0.11 µg/L. This analysis of ERM CA-713 had an average recovery of 1.96 ± 0.11 µg/L, which is 106.5% of the certified value.

Using reduced ID pump tubing and method parameters optimized for speed, samples were analyzed in 70s while reducing reagent consumption and waste generation and increasing throughput.