

## MDL Nota AP 021

# Determinación de Mercurio en aguas superficiales, estudio MDL, Método ISO 12846. Analizador QuickTrace™ M-7600 CVAAS

### PROCEDURE

The MDL study was performed using the CETAC QuickTrace™ M-7600 Cold Vapor Atomic Absorbance Spectrometry Mercury Analyzer. Calibration standards concentrations were 5.0, 10.0, 25.0, 50.0, and 100.0 ng/L. Standards were prepared from a 1000 ng/L mercury standard which was prepared from serial dilutions of a 1000 mg/L mercury standard. The final volume was 25.75 mL and they were prepared in 50 mL polypropylene tubes. Potassium bromate / potassium bromide solution was prepared by diluting purchased, pre-cleaned 1 N Br / 1 N BrO<sub>3</sub> solution with 90 mL of double distilled HCl to make a 0.1 N bromine monochloride (BrCl) solution. All calibration standards were matrix-matched, using 2.5 mL of 1:1 hydrochloric acid, 0.5 mL of 0.1 N BrCl solution, and 0.25 mL of 10% (w/v) L-ascorbic acid, according to performance based ISO Method 12846, Water quality - Determination of mercury – Method using atomic absorbance spectrometry (AAS) with and without enrichment. The calibration was analyzed beginning with one calibration blank and then proceeded from the lowest concentration standard to the highest concentration standard.

MDL standard concentrations were 5.0 ng/L and 2.0 ng/L. Replicate concentrations were prepared from a 100 ng/L mercury standard in 50 mL polypropylene tubes. Standard replicates were prepared with 2.5 mL of 1:1 hydrochloric acid, 0.5 mL of 0.1 N BrCl solution, 0.25 mL of 10% (w/v) L-ascorbic acid, and deionized water for a final volume of 25.75 mL.

Seven replicates of each MDL standard were analyzed using peak height. Sample uptake time was 28 seconds at 80% pump rate, utilizing approximately 6 mL of sample with 65 seconds of rinse time, for a total analysis time of 93 seconds per sample. The integration mode was set to

peak height utilizing 4 replicates at 1.0 second per replicate.

### RESULTS

Calculated MDL concentrations are based on a 99% uncertainty according to 40 CFR Appendix B to Part 136, "Definition and Procedure for the Determination of the Method Detection Limit," Revision 1.11.



**MDL Study Groundwater ISO 12846 40 CFR Ch.1 (7-1-91) Part. 136, App B**

Run 1 @ 5.0 ng/L	Run 2 @ 2.0 ng/L
5.2	1.95
5.27	2.35 4.94 2.63 5.42 2.67
5.64	1.9 4.47 2.4
5.54	1.86

0.40043 = Run 1 STDEV      0.34590 = Run 2 STDEV  
0.160348 = STDEV<sup>2</sup>      0.119648 = STDEV<sup>2</sup>

1.340 = F ratio

0.2799952 Sum STDEV<sup>2</sup> (Run 1 and 2)  
0.1399976 Sum STDEV<sup>2</sup> / 2 ((Run 1 and 2) / 2)  
0.3741626 STDEV<sub>Pooled</sub>

1.003 = MDL