

Application Note

A Novel Approach to Increase Sample Throughput on a CVAA Mercury Analyzer using an Injection Valve

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ABSTRACT

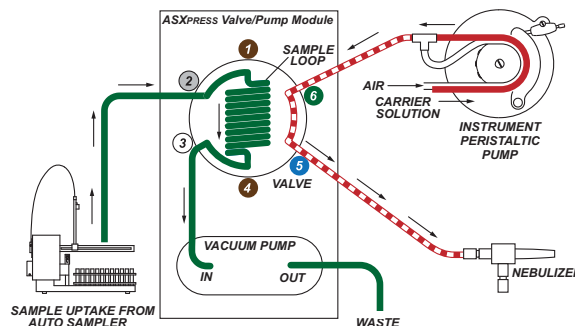
Mercury determination in soil plays a crucial role in agriculture, environmental status, and human health. The detection of mercury allows for long-term and short-term analysis of each of these systems, and it gives information on specific sources of mercury contamination. The analysis of soil yields quantitative data for water quality, air quality, and crops. The concentration of mercury in soil depends on numerous variables. These include the industry, human impact, and mercury volatility conditions. In this study we utilize our CETAC QuickTrace™ M-7600 Cold Vapor Atomic Absorbance Analyzer in conjunction with our ASXPRESS® PLUS to increase throughput while maintaining low detection limits.

INTRODUCTION AND OBJECTIVES

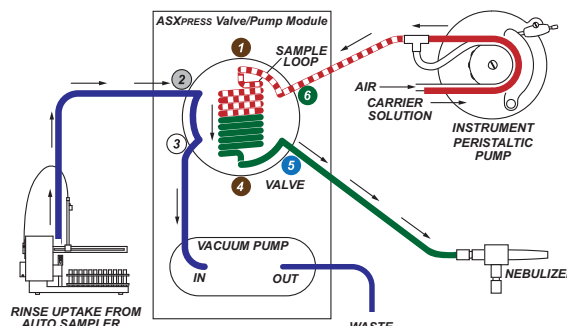
Governing bodies like the EPA, MOE, and EU are focusing more time and effort on the testing and monitoring of mercury's impact on the environment and human health. As the demand for new and more frequent testing rises, environmental testing facilities will need to increase their sample load capabilities. One method for increasing the sample capacity of a laboratory is to increase the sample capacity of current mercury analyzers. The coupling of an ASXPRESS® PLUS to a CETAC QuickTrace™ M-7600 can increase sample throughput up to five times, without sacrificing analytical precision and accuracy.



Figure 1: QuickTrace™ M-7600 coupled with ASXPRESS® PLUS



LOAD SAMPLE



INJECT/RINSE

Figure 2 & 3: ASXPRESS® PLUS diagram with ISTD Load/Inject

The *ASXPRESS® PLUS* is a rapid sample introduction system originally designed for the ICP – OES/MS market. The *ASXPRESS® PLUS* utilizes a 6-port valve and a high speed vacuum pump to rapidly fill a sample loop. In the load configuration the sample probe and loop are connected to the vacuum pump, while the Gas-Liquid Separator (GLS) line is being rinsed by the peristaltic pump on the M-7600. Once the loop is filled the valve will switch to inject. The peristaltic pump is now pushing the sample out of the loop and into the GLS line. While the sample is being analyzed, the sample probe line is being rinsed with vacuum pump.

MATERIALS & METHODS

The digestion method used to prepare samples was EPA 245.5. This method starts with a 0.2g soil sample added to 1.8 mL aqua regia. This mixture is then placed in a heat block and heated to 95°C for 2 minutes. The mixture is

allowed to cool to room temperature and 5.3 mL of potassium permanganate is added. The mixture is heated once again to 95°C and held at temperature for 30 minutes. The mixture is allowed to cool to room temperature and 2.2 mL of 12% hydroxylamine is added and the mixture is diluted to 50mL with deionized 18 MΩ water. All calibration, ICB, ICV, CCB and CCV standards were matrix matched using similar preparation methodology.

Table 1: Samples analyzed in this study

Sample	Hg Concentration
Montana Soil, SRM 2711a	7.42 mg/Kg ±0.18

The M-7600 and *ASXPRESS® PLUS* were optimized to provide the fastest sample analysis time. The operating conditions for the QuickTrace™ M-7600 and the *ASXPRESS® PLUS* can be found in Figures 3 and 4.

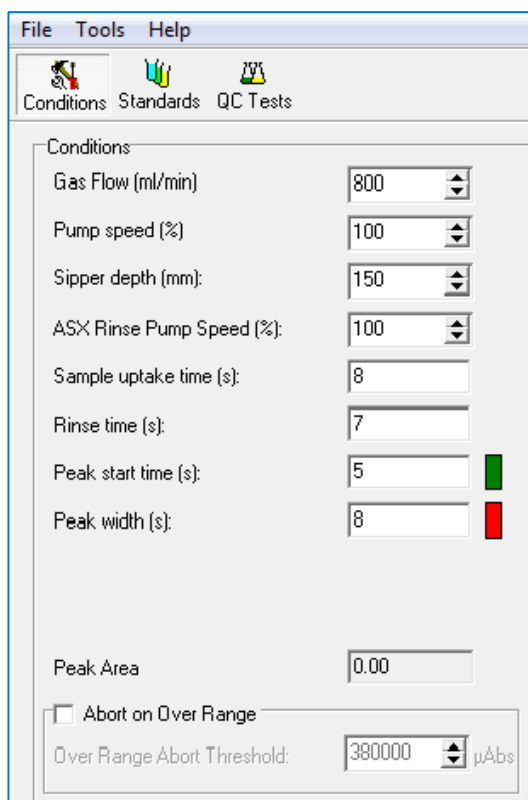


Figure 4: QuickTrace™ M-7600 Method Settings

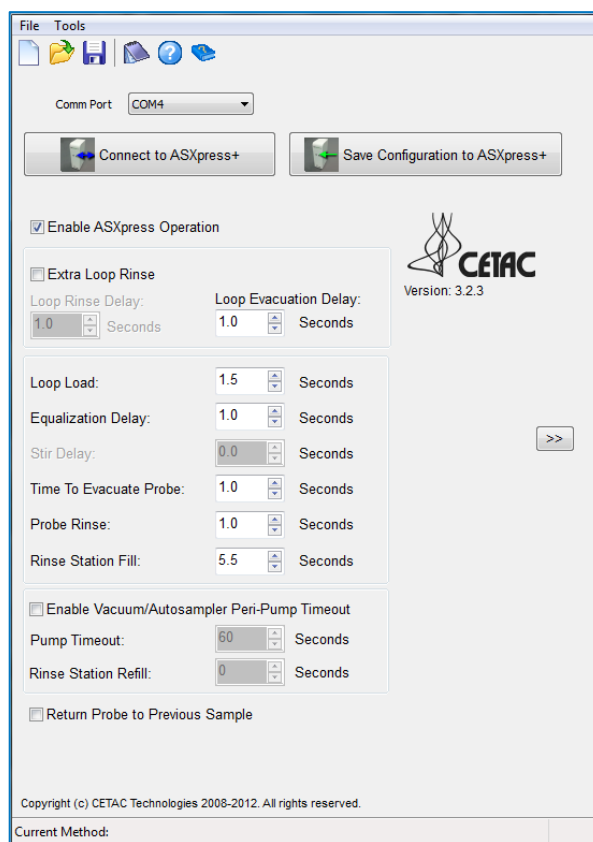


Figure 5: ASXPRESS® PLUS Configuration Settings.

RESULTS AND DISCUSSION

The objective of this study was to increase the sample throughput of the CETAC QuickTrace™ M-7600 CVAA analyzer, while maintaining low detection limits and sample accuracy. Prior to analysis, a MDL study was performed to determine the sensitivity of the method. Calibration data and a peak profile are shown in Figures 6 and 7. The MDL results are found in Table 2.

NIST Montana Soil 2711a Standard Reference Material (SRM) was used to test precision and accuracy of the method. Seven samples were weighed and digested according to EPA 245.5 methodology. The samples were then analyzed following the EPA 245.5 analytical and control criteria. Montana soil 2711a has a certified mercury concentration of 7.42mg/Kg with an uncertainty of ± 0.18mg/Kg at a 95% confidence limit. The results for the Montana soil samples can be found in Table 3.

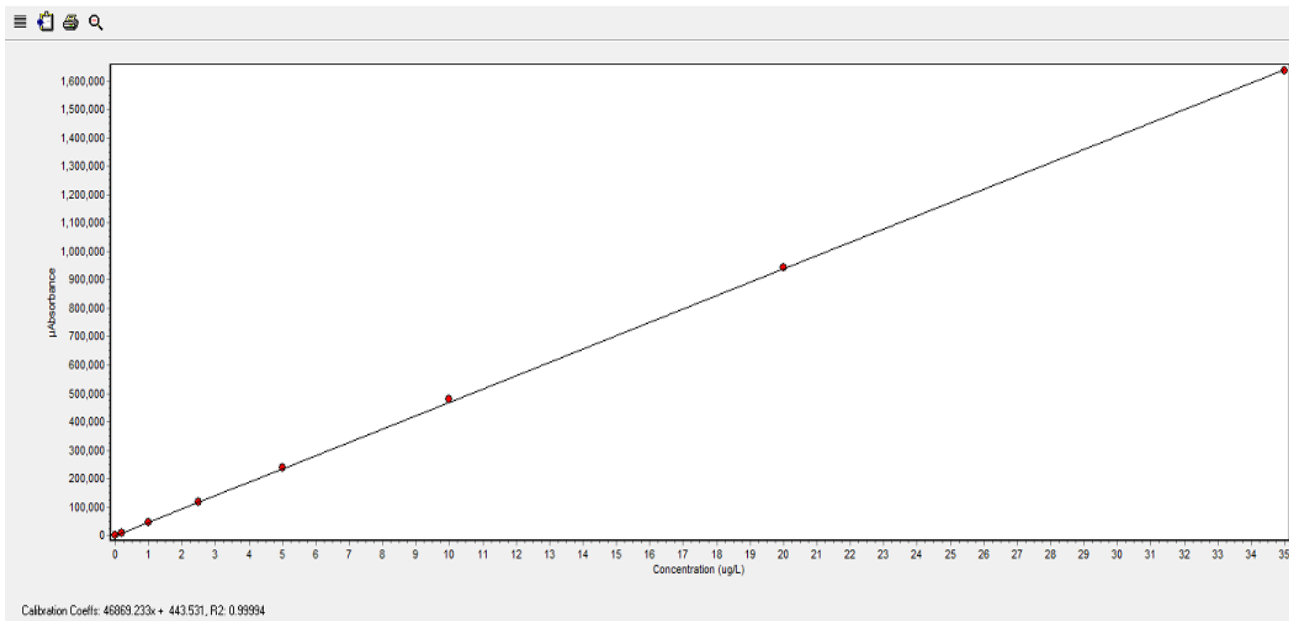


Figure 6: Calibration data

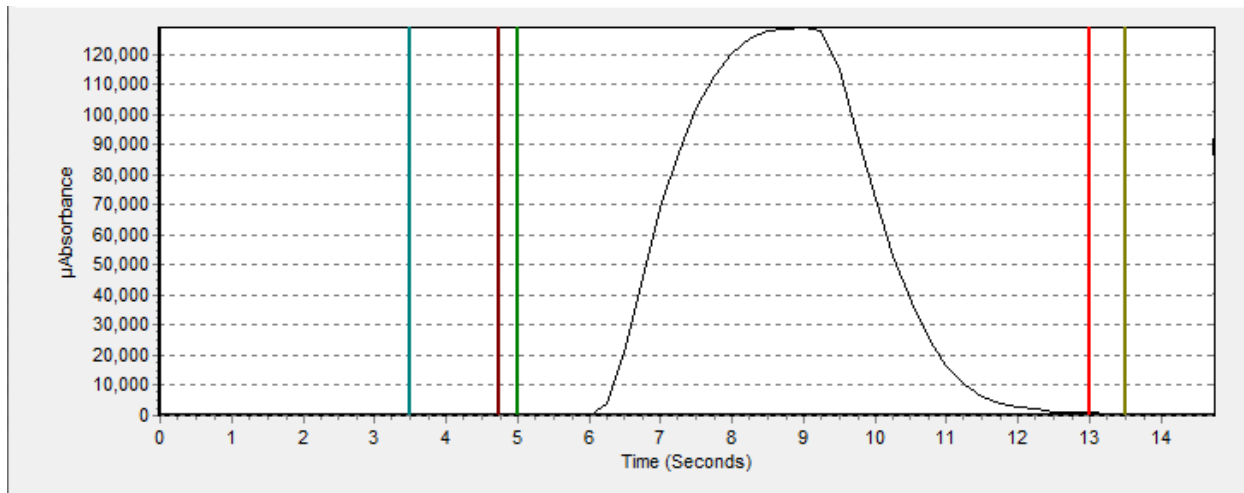


Figure 7: Peak Profile for 35μg/L Standard

Table 2: MDL results, Montana Soil EPA 245.5 40 CFR Ch.1 (7-1-91) Part. 136, App B

Run 1 @ 0.05 µg/L	Run 2 @ 0.02µg/L
0.068	0.031
0.065	0.038
0.068	0.048
0.061	0.044
0.085	0.033
0.070	0.046
0.071	0.026
MDL = 0.021 µg/L	

Table 3: Montana Soil SRM 2711a results.

Digest	Mercury (mg/Kg)
1	7.25
2	7.28
3	7.21
4	7.18
5	7.22
6	7.25
7	7.29
Mean =	7.24
STDEV =	0.039
RSD % =	0.536
Uncertainty =	0.076

Conclusions

Using the ASXPRESS® PLUS had the following effects:

- Increased sample throughput
 - Normal sampling times for similar samples are ≥ 60sec vs. 15sec with the ASXPRESS® PLUS, a savings of 75% in analysis time.
- Decreased rinse solution usage
 - Valve design uses minimal amount of rinse to clean system
- Increased efficiency
 - Run more samples per shift