

Aplicación 025

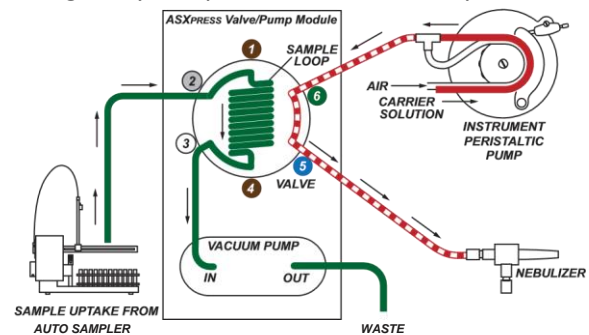
Analizador de Mercurio CVAA asociado a una válvula de inyección

RESUMEN

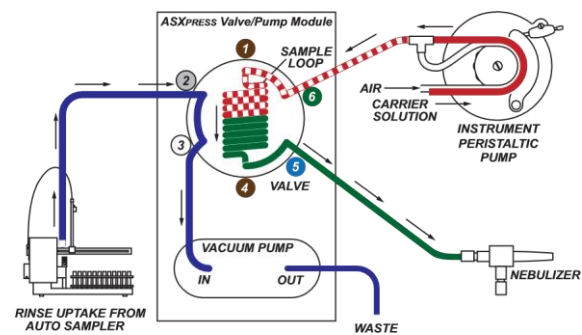
La determinación de mercurio en suelos es muy importante en agricultura, medioambiente y la salud humana. El análisis de mercurio da información sobre las fuentes de contaminación de Mercurio y permite seguir la estabilidad de estos sistemas en el largo y corto plazo. El análisis de suelos genera datos cuantitativos sobre la calidad del agua, el aire y las cosechas. La concentración de mercurio depende de la actividad industrial, el impacto humano y la volatilidad del mismo. En esta aplicación el Analizador CVAA QuickTrace™ M-7600 junto con el sistema ASXPRESS® PLUS se muestra la productividad con el aseguramiento de los límites de detección..

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.



LOAD SAMPLE



INJECT/RINSE

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

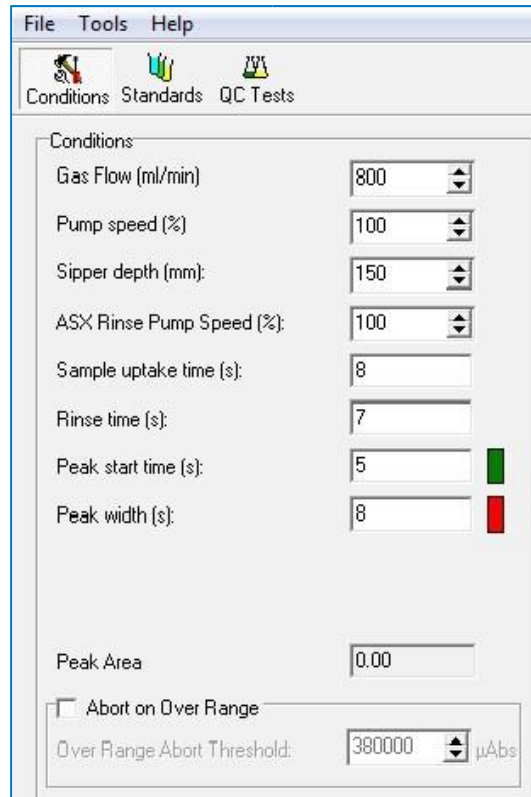
Figure 2 & 3: ASXPRESS® PLUS diagram with ISTD Load/Inject

M-7600-007

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.

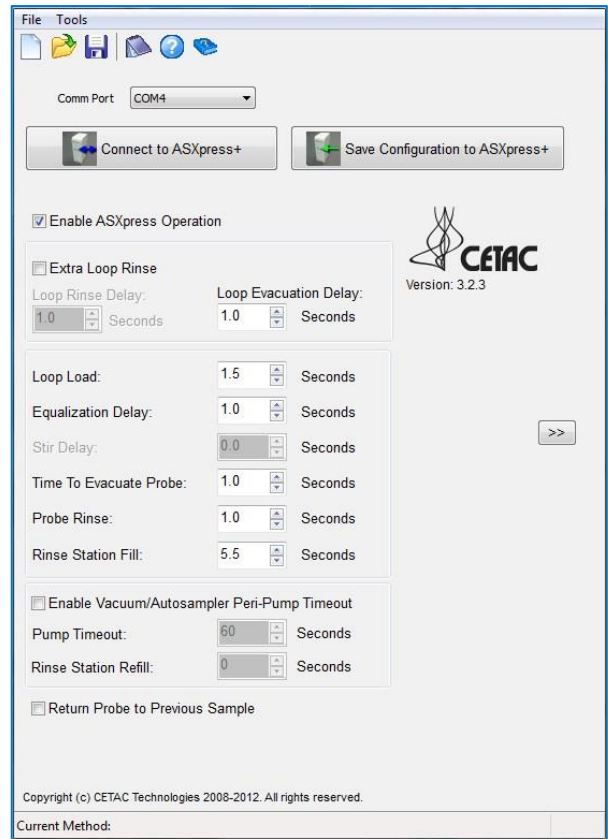


Figure 4: QuickTrace™ M-7600 Method Settings

Figure 5: ASXPRESS® PLUS Configuration Settings.

NIST Montana Soil 2711a Standard Reference Material (SRM) was used to test precision and accuracy of the

RESULTS AND DISCUSSION

The objective of this study was to increase the sample method. Seven samples were weighed and digested according to EPA 245.5 methodology. The samples were lyzer, while maintaining low detection limits and sample then analyzed following the EPA 245.5 analytical and con- accuracy. Prior to analysis, a MDL study was performed to trol criteria. Montana soil 2711a has a certified mercury determine the sensitivity of the method. Calibration data concentration of 7.42mg/Kg with an uncertainty of and a peak profile are shown in Figures 6 and 7. The MDL

± 0.18mg/Kg at a 95% confidence limit. The results for the

results are found in Table 2.

Montana soil samples can be found in Table 3.

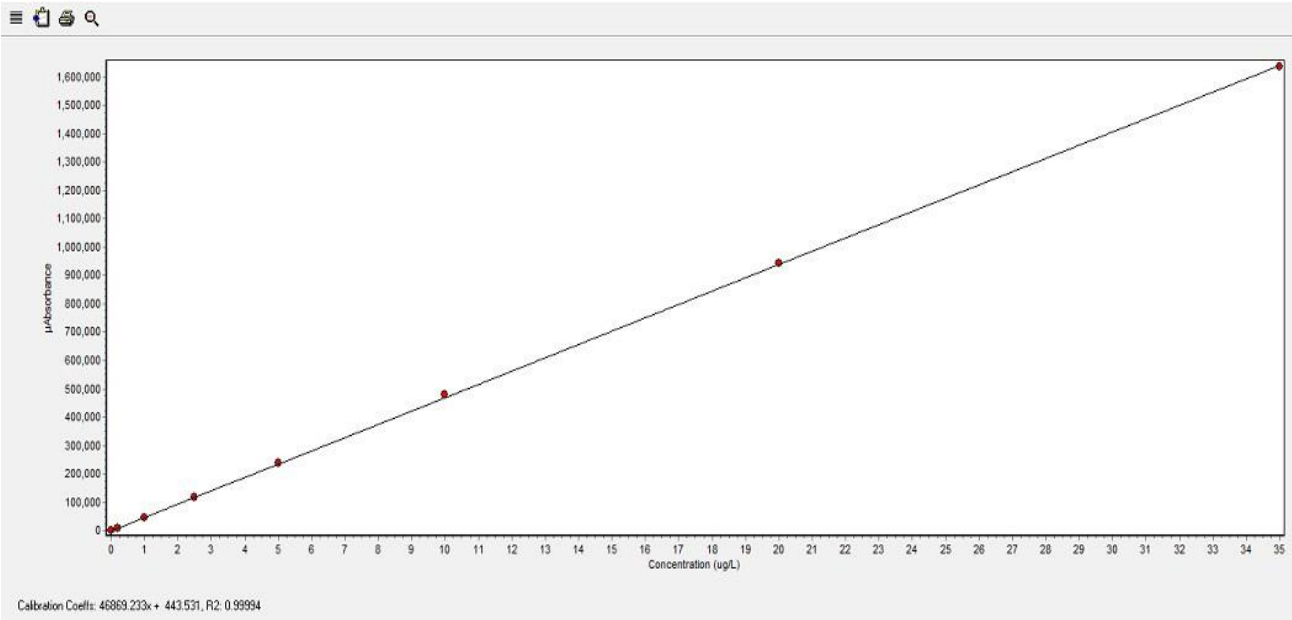


Figure 6: Calibration data

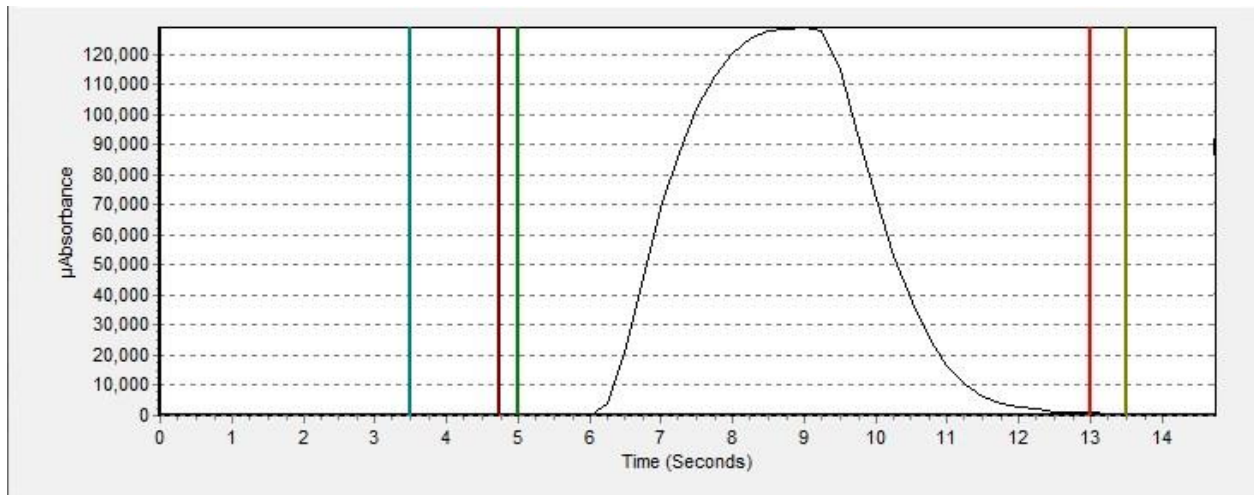


Figure 7: Peak Profile for 35μg/L Standard

Table 2: MDL results, Montana Soil EPA 245.5 40 CFR Conclusions 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	

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

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