

Rapid-Throughput EPA 6010C Analysis by Inductively Coupled Plasma Atomic Emission Spectroscopy

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The CETAC ASXPRESS® PLUS Rapid Sample Introduction System, when coupled to a CETAC autosampler, optimizes sample introduction by significantly increasing sample throughput and reducing costs of materials, power, maintenance and labor for ICP-AES analysis. The system is designed to allow multiple functions to occur simultaneously which would otherwise take place separately.



Figure 1. ASXPRESS® PLUS Rapid Sample Introduction System

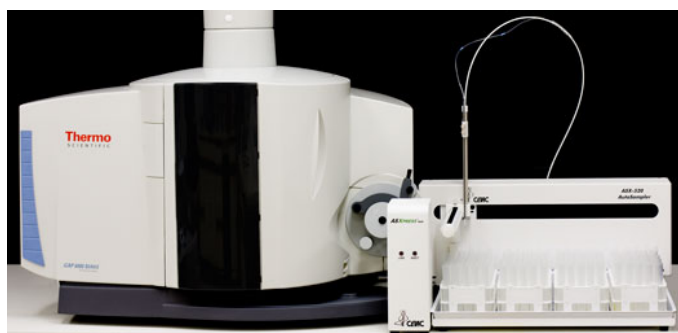


Figure 2. ASXPRESS® PLUS with Thermo Scientific iCAP 6500 ICP-AES

A standard analysis system relies upon a single peristaltic pump to both deliver samples to the nebulizer and rinse the sample flow path between sample deliveries. The ASXPRESS® PLUS system utilizes a high speed vacuum pump in addition to the ICP-AES peristaltic pump. The 6-port valve allows the use of both pumps simultaneously, reducing total sample analysis time significantly (Figure 3).

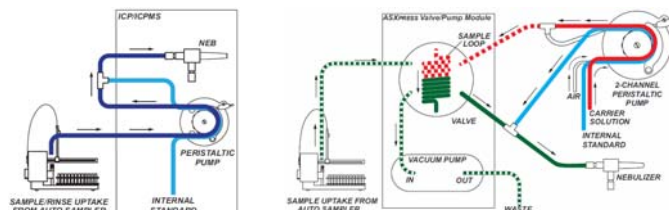


Figure 3. Standard analysis system setup (Left); Analysis setup with ASXPRESS® PLUS (Right)

The use of the valve effectively divides each analysis into two stages. First, while the valve is in the load position, the vacuum pump rapidly fills the sample loop, while the ICP-AES peristaltic pump simultaneously transports carrier solution, keeping the plasma stable and rinsing the nebulizer and spray chamber. In the second position, the loaded sample is pushed into the nebulizer for analysis via the carrier solution flowing through the ICP-AES peristaltic pump. Simultaneously, the autosampler probe is moved to the rinse station and the uptake flow path is flushed with rinse solution via the vacuum pump.

Since most environmental laboratories need to cope with high sample numbers, the sample introduction approach used by the ASXPRESS® PLUS has particular application to environmental analyses, dramatically increasing sample throughput without negatively impacting data quality.

DATA QUALITY

Using a 5 mL loop along with the concentric nebulizer and cyclonic spray chamber already in use, testing of the ASXPRESS® PLUS system showed good precision with RSDs less than 1% for 3 replicate measurements over all elements covered by EPA Method 6010C. Most are well below 1%.

EPA Method 6010C guidelines state that calibration correlation coefficients of 0.998 or better must be achieved. This is easily achieved and correlation coefficients for all elements are listed in Figure 4.

Element	Coefficient	Element	Coefficient
Al	0.99998	Mn	0.99993
Sb	0.99976	Mo	0.99983
As	0.99996	Ni	0.99995
Ba	0.99994	Pb	0.99989
Be	0.99977	K	0.99998
B	0.99998	Se	0.99982
Cd	0.99956	Si (SiO ₂)	0.99997
Ca	0.99911	Ag	0.99800
Cr	0.99994	Na	0.99986
Co	0.99999	Sr	0.99998
Cu	0.99958	Tl	0.99929
Fe	0.99974	Sn	0.99991
Pb	0.99992	Ti	0.99995
Li	0.99970	V	0.99997
Mg	0.99997	Zn	0.99942

Figure 4. Calibration correlation coefficients for EPA 6010C. (Data was supplied by a laboratory using CETAC QuickTrace® Mercury Analyzer for Hg. Hg not measured by ICP-AES.)

A set of eight production samples were analyzed on the ICP before and after the ASXPRESS® PLUS was installed. Correlation plots were then generated to establish how well the two sample introduction setups agreed with one another. The data obtained from the original setup was plotted against that obtained from the ASXPRESS® PLUS setup. The closer the slopes and correlation coefficients are to 1.0 for each element graphed, the closer the two sets of data are. A selection of graphs are displayed in Figure 5.

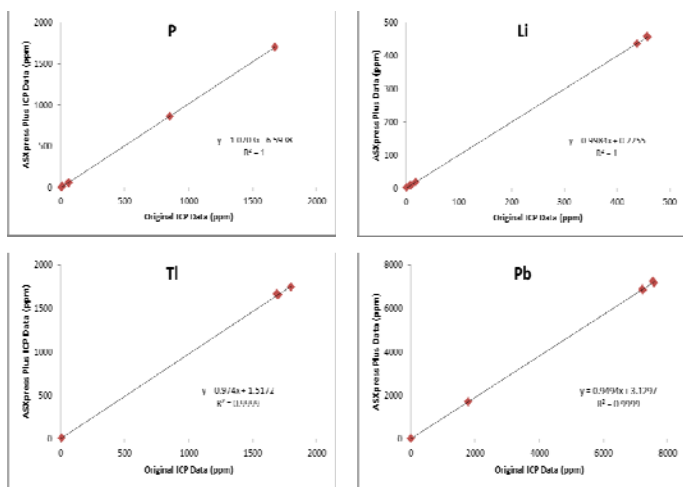


Figure 5. Stability for EPA 6010C elements with ASXPRESS® PLUS

STABILITY AND LONGEVITY

Precision and accuracy were tested by running a continuing calibration verification (CCV) standard five consecutive times. The precision was 1% or below for all elements analyzed across the five samples. The stability can be better visualized with the following graphs shown below in Figure 6.

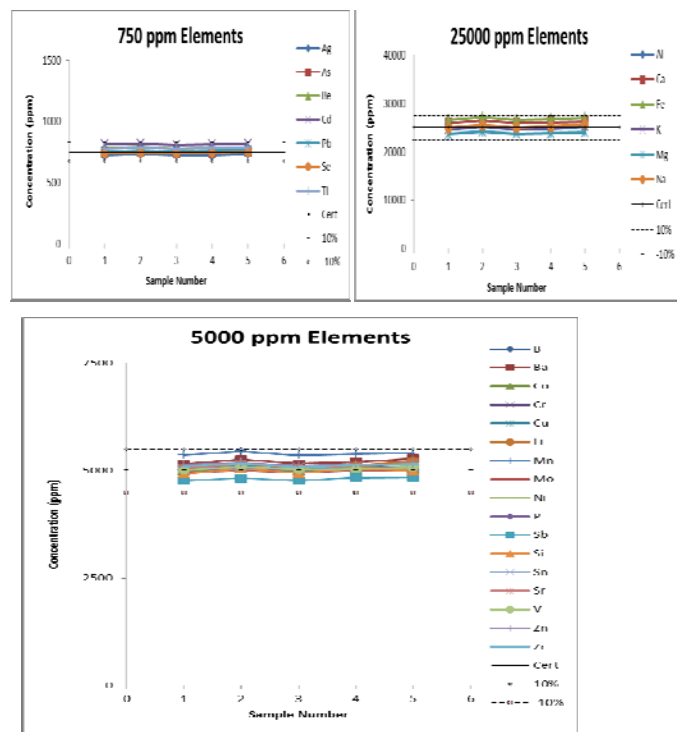


Figure 6. Stability for EPA 6010C elements with ASXPRESS® PLUS

TIME SAVINGS

Existing analysis methods have been found to run samples on the order of 6 minutes per sample to meet analysis criteria such as throughput, precision, passing QCs and accuracy of results.

Various time tests were conducted using the CETAC ASXPRESS® PLUS with the Thermo Scientific iCAP 6500 ICP-AES. Sampling time was cut to ~3.5 minutes per sample when using the ASXPRESS® PLUS system while still meeting all the criteria. A time saving of greater than 40% was realized.

Using the ASXPRESS® PLUS system improved carryover. Carryover was tested using highly concentrated Fe and Na samples and found to be less than 0.02%.

LOW MAINTENANCE COST – TIME & MONEY

Only simple and quick maintenance procedures are required for the *ASXPRESS® PLUS* system. Routine maintenance includes disassembling the valve body and using compressed air to blow out the sampling ports and the rotor on a weekly to bi-weekly basis, depending on sample volume and matrix.

Operation with the *ASXPRESS® PLUS* greatly extends the service life of ICP components, reducing nebulizer and spray chamber maintenance by reducing exposure to the sample matrix. Since peristaltic pump tubing is never exposed to the sample matrix and is used at a constant analytical pump rate, its service life is also greatly extended.

The *ASXPRESS® PLUS* equipment itself is very stable and the system can be taken apart and reassembled or even stored for extended periods of time realizing the same data quality once reintegrated into the system.

Depending upon sample matrix, the 6-port valve is capable of lasting well over 100,000 samples. Low cost service components are readily available.

EASE OF INSTALLATION

An easy, out-of-the-box set of instructions and initial configuration parameters have been developed for the *ASXPRESS® PLUS* to allow the utmost ease of installation. The *ASXPRESS® PLUS* integrates quickly and easily into the sample flow path, without modification to the analysis method. A simple and convenient Windows® based configuration tool is used to store parameters to the system's on-board processor. No additional software is required. Loop Rinse, Loop Evacuation, Loop Load, Equalization, Probe Evacuation, Probe Rinse, and Rinse Station Fill times were 2, 2, 10, 2, 1, 5, and 4 seconds respectively. Installation by an authorized service representative is available; please contact CETAC or Thermo Fisher Scientific for details.

CONCLUSION

As shown in the data comparison, use of the *ASXPRESS® PLUS* results in the same quality of data, but only a fraction of the time is needed to gather it.