CETAC www.cetac.com

Agilent Technologies

Rapid-Throughput EPA 200.8 Analysis for Inductively Coupled Plasma Mass Spectroscopy

Michael Sgroi, Automation Product Line Manager, CETAC Technologies, Omaha, NE, (402) 738-5415, msgroi@cetac.com

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-MS 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

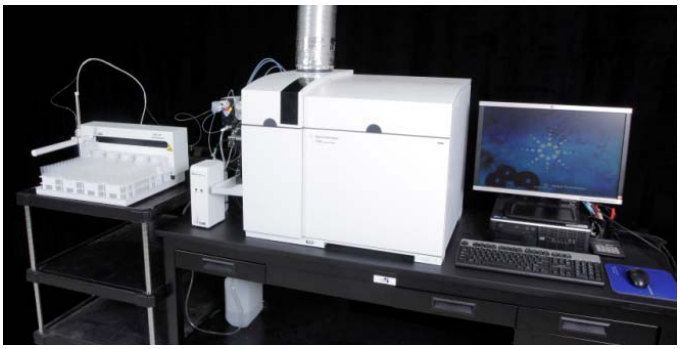

Figure 2. *ASXPRESS® PLUS* with Agilent 7700

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-MS peristaltic pump. The 6-port valve allows the use of both pumps simultaneously, reducing total sample analysis time significantly (Figure 3).

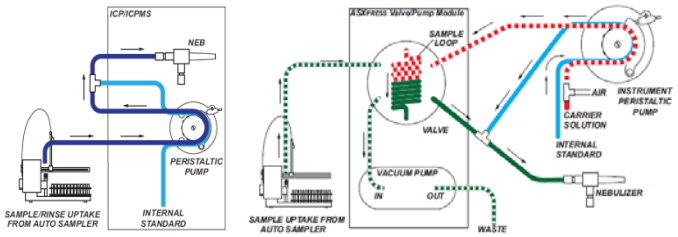

Figure 3. Standard analysis system setup (Left); Analysis setup with *ASXPRESS® PLUS* (Right)

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-MS peristaltic pump simultaneously transports carrier solution, keeping the plasma stable. In the second position, the loaded sample is pushed into the nebulizer for analysis via the carrier solution flowing through the ICP-MS 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.

The sample introduction approach used by the *ASXPRESS® PLUS* has particular implications for environmental applications.

DATA QUALITY

Instrument parameters such as gas flows, plasma power, mode stabilizations times and data collection settings were not manipulated with the addition of the *ASXPRESS® PLUS*. The flush and rinse times were not necessary with the *ASXPRESS® PLUS* system and the delay time was reduced by ten seconds. The end result was a time savings of over 50% and while preserving the original data quality.

Using a 1.5 mL loop along with the Micro Mist concentric nebulizer and quartz low-volume Scott's Double Pass cooled spray chamber already in use, testing of the *ASXPRESS® PLUS* system showed good precision for 3 replicate measurements over all elements covered by the EPA 200.8 Method. Most RSDs are well below 1%.

Carryover was also tested with the *ASXPRESS® PLUS* system by running a high standard followed by a blank five times. The formula 100% * (blank value / standard value) was used to calculate the results which are tabulated below in Figure 4.

In addition to the use of a segmented carrier stream and a simultaneous analysis and rinsing scheme, the sample never comes into contact with the peristaltic pump tubing. This further helps to minimize memory effects.

Element	m/z	Average Measured Carryover (%)	Element	m/z	Average Measured Carryover (%)
Be	9	0.0148	As	75	0.0130
Na	23	-0.0080	Se	78	0.0499
Mg	24	0.0054	Mo	95	0.0151
Al	27	-0.5152	Mo	98	0.0122
K	39	0.0050	Ag	107	-0.0068
Ca	44	0.0047	Cd	111	-0.1023
V	51	0.0059	Sb	121	0.0070
Cr	52	0.0044	Sb	123	0.0075
Mn	55	0.0031	Ba	137	0.0040
Fe	56	0.0056	Tl	205	0.0111
Co	59	0.0051	Pb	206	0.0040
Ni	60	-0.0161	Pb	207	0.0048
Cu	63	0.0094	Pb	208	0.0047
Zn	66	-0.0046			

Figure 4. Carryover Data for EPA 200.8

STABILITY AND LONGEVITY

Over time or sample volume, an ICP-MS signal can drift. One of the main reasons for such drift is directly related to the sample matrix coming into contact with the cones. With the use of the *ASXPRESS® PLUS*, the instrument only comes into contact with the matrix during data acquisition. This dramatically reduces the instrument exposure to the matrix thereby reducing drift. Since the system is also able to run many more samples per unit time, the drift per unit number of samples is also reduced.

Precision and accuracy were tested by running a continuing calibration verification (CCV) standard thirty consecutive times. This data is shown in Figure 5.

TIME SAVINGS

The existing analysis method was found to run on the order of 7.5 minutes per sample to meet analysis criteria such as throughput, precision, passing QC's and accuracy of results.

Various time tests were conducted using the CETAC *ASXPRESS® PLUS* with the Agilent 7700 ICP-MS. Sampling time was cut to 3.5 minutes per sample when using the *ASXPRESS® PLUS* system while still meeting all the criteria. A 55% time savings was realized.

Element	m/z	Average Measured Result (ppb)	RSD (%)	Known Value (ppb)	Average Recovery (%)
Be	9	23.1	1.2	25	92.2
Na	23	2491.8	0.8	2500	99.7
Mg	24	2507.5	0.8	2500	100.3
Al	27	23.9	2.2	25	95.4
K	39	2516.2	1.4	2500	100.6
Ca	44	2426.2	0.9	2500	97.0
V	51	23.5	0.7	25	94.1
Cr	52	23.9	0.9	25	95.7
Mn	55	23.4	0.7	25	93.5
Fe	56	2491.0	0.6	2500	99.6
Co	59	24.8	0.9	25	99.2
Ni	60	25.1	1.0	25	100.6
Cu	63	24.5	1.0	25	98.1
Zn	66	25.2	1.1	25	100.8
As	75	24.8	0.7	25	99.2
Se	78	25.7	2.2	25	102.7
Mo	95	22.9	1.2	25	91.6
Mo	98	23.1	1.2	25	92.3
Ag	107	2.5	3.2	2.5	101.3
Cd	111	24.9	1.2	25	99.7
Sb	121	24.2	1.1	25	96.8
Sb	123	24.3	1.1	25	97.1
Ba	137	24.5	1.1	25	98.2
Tl	205	25.1	1.1	25	100.2
Pb	206	24.5	1.1	25	98.1
Pb	207	24.5	1.1	25	97.9
Pb	208	24.6	1.0	25	98.5

Figure 5. Repeatability Data from 30 CCV Samples for EPA 200.8

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, 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 only to realize the same resulting 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 shown in the following table (Figure 6.) to the system's on-board processor. No additional software is required.

Parameter	Time (s)
Loop Rinse	0.5
Rinse Evacuation	1.0
Loop Load	0.5
Equalization	1.0
Time to Evacuate	1.0
Probe Rinse	1.0
Rinse Station Refill	3.0

Figure 6. *ASXPRESS® PLUS* Configuration Parameters

Installation by an authorized service representative is available; please contact CETAC or Agilent 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.