Application Note

Rapid-Throughput Soil 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

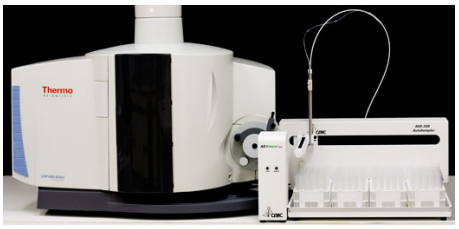

Figure 2. *ASXPRESS™ PLUS* with Thermo Scientific iCAP 6500 ICP-AES

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).

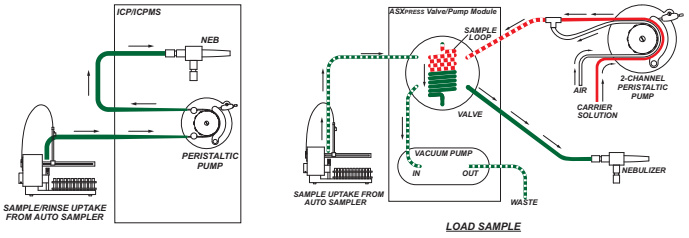

Figure 3. Standard analysis system setup (Left); Analysis setup with *ASXPRESS™ PLUS* (Right)

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 and agricultural laboratories need to cope with high sample volume, the sample introduction approach used by the *ASXPRESS™ PLUS* has particular application to soil analyses, dramatically increasing sample throughput without negatively impacting data quality.

DATA QUALITY

Most soil labs employ methods that take single replicate measurements. This is also the case in this example. Two methods from this lab are described here. The calibration schemes for these two methods are shown below in Figure 4. The yellow highlighting indicates which calibration standards were used to calibrate each line. Line-switching was used in these two methods. The calibration coefficients for these elements are tabulated in Figure 5.

Ammonium Acetate	Elemental Lines							
Soil Method	Ca 317.933	Ca 373.690	Ca 431.865	K 766.490	K 769.896	Mg 279.079	Mg 279.553	Na 589.592
Standard	(ppm)	(ppm)	(ppm)	(ppm)	(ppm)	(ppm)	(ppm)	(ppm)
1	0	0	0	0	0	0	0	0
2	50	50	50	5	5	5	5	2
3	150	150	150	15	15	15	15	7.5
4	300	300	300	30	30	30	30	15
5	500	500	500	50	50	50	50	25
6	700	700	700	70	70	70	70	35
7	1000	1000	1000	100	100	100	100	50
8	1500	1500	1500	150	150	150	150	75
9	2000	2000	2000	200	200	200	200	100

DTPA Soil	Elemental Lines							
Method	Cu 324.754	Cu 327.396	Fe 238.204	Fe 371.994	Mn 260.569	Mn 279.482	Zn 206.200	Zn 213.856
Standard	(ppm)	(ppm)	(ppm)	(ppm)	(ppm)	(ppm)	(ppm)	(ppm)
1	0	0	0	0	0	0	0	0
2	0.1	0.1	1	1	1	1	0.1	0.1
3	0.25	0.25	2.5	2.5	2.5	2.5	0.25	0.25
4	0.5	0.5	5	5	5	5	0.5	0.5
5	1	1	10	10	10	10	1	1
6	2.5	2.5	25	25	25	25	2.5	25
7	5	5	50	50	50	50	5	5
8	7.5	7.5	75	75	75	75	7.5	7.5
9	10	10	100	100	100	100	10	10

Figure 4. Calibration schemes

Ammonium Acetate		DTPA	
Line	Coefficient	Line	Coefficient
Ca 317.933	0.999930	Cu 324.754	0.999716
Ca 373.690	0.999907	Cu 327.396	0.999757
Ca 431.865	0.999965	Fe 238.204	0.999947
K 766.490	0.999968	Fe 371.994	0.999792
K 769.896	0.999011	Mn 260.569	0.999926
Mg 279.079	0.999905	Mn 279.482	0.999728
Mg 279.553	0.999702	Zn 206.200	0.999878
Na 589.592	0.999941	Zn 213.856	0.999449

Figure 5. Calibration correlation coefficients

The iCAP method analysis preferences and source settings are tabulated in Figure 6.

Analysis Preferences		Sample Pump		Source Settings	
Replicates	1	Flush Rate	22 RPM	RF Power	1150 W
Flush Time	10 s	Analysis Rate	22 RPM	Auxilliary Gas Flow	0.5 L/min
Analysis Mode	Speed	Stabilization	0 s	Nebulizer Gas Flow	0.45 L/min
Low WL Integration Time	1 s			Coolant Gas Flow	14 L/min
High WL Integration Time	1 s			Radial Viewing Height	12.0 mm

Figure 6. iCAP Method Parameters

A 0.7 mL loop was used and the CETAC Xpress configuration parameters are listed in Figure 7.

Xpress Config Settings	Time (s)
Loop Load Delay	1
Loop Load Delay	3.2
Probe Wash	1.4
Rinse Station Refill	5.6
Rinse Timeout	10

Figure 7. Xpress Configuration Settings

Accuracy was ensured by running a QC sample at the end of a calibration and run as well as every thirty samples. QC samples pass if the values are within 10% of the nominal value. A warning is issued if the values are not within 5%.

Precision data comes in the form of repeatability in methods such as these that only take one replicate per measurement. These methods are precise in so far as the several QC samples run each day consistently measure within 5% of the nominal values.

Carry-over was tested and found to be better with the CETAC *ASXPRESS™ PLUS* than with either the traditional ICP sample introduction setup or one that used another valve system.

STABILITY AND LONGEVITY

QC samples were found to pass for longer sample runs using the *ASXPRESS™ PLUS* system. Four *ASXPRESS™ PLUS* systems have been running on four Thermo Scientific iCAP 6000 Series instruments for well over a year.

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.

TIME SAVINGS

Existing analysis methods have been found to run samples on the order of 28 seconds per sample to meet analysis criteria such as throughput, precision, passing QC's and accuracy of results.

Using the CETAC *ASXPRESS™ PLUS* with the Thermo Scientific iCAP 6000 ICP-AES, sampling times were cut to 18 seconds per sample when using the *ASXPRESS™ PLUS* system while still meeting all the criteria. More than 35% time savings was realized. By using the *ASXPRESS™ PLUS* system carry-over improved.

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.

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. Installation by an authorized service representative is available; please contact CETAC or Thermo Fisher Scientific for details.

CONCLUSION

Use of the *ASXPRESS™ PLUS* results in the same quality of data, but only a fraction of the time is needed to gather it. In high throughput laboratory environments where turn-around-time plays the most vital role in success, the *ASXPRESS™ PLUS* is a crucial piece of equipment that can prevent the ICP analysis step from becoming the bottle neck of the operation.