

Rapid-Throughput Oil analysis for Inductively Coupled Plasma Atomic Emission 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-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 Systems



Figure 2. ASXpress™ plus with PerkinElmer Optima series 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 (Figure 3). 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 4).

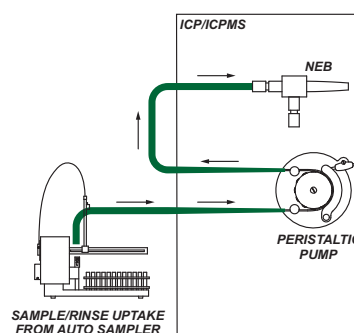


Figure 3. Standard analysis system setup

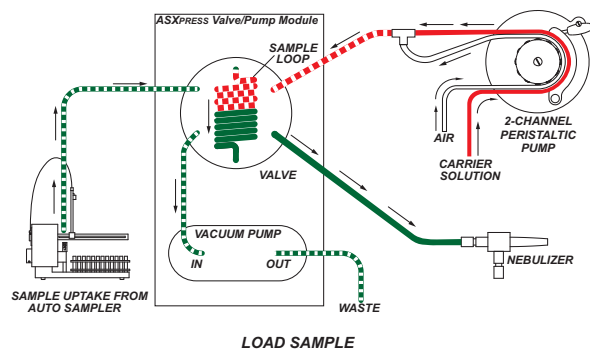


Figure 4. Analysis setup with ASXPRESS™ PLUS

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. 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.

The sample introduction approach used by the ASXPRESS™ PLUS has particular implications for oil analysis.

DATA QUALITY

Tests of the *ASXPRESS™ PLUS* system showed good precision with all RSD's below 5%, and typically below 2% for two replicate measurements across all wear metal and additive elements.

Calibration curves with correlation coefficients of 0.9999 or better are readily achievable over the concentration ranges expected in used oils. Proficiency testing with blind samples has shown achievable accuracy well within $\pm 10\%$.

Use of the *ASXPRESS™ PLUS* system increases the frequency of passing QC samples. Failures are greatly reduced as QC samples remain within passing range on a calibration for hundreds of samples before recalibration becomes necessary.

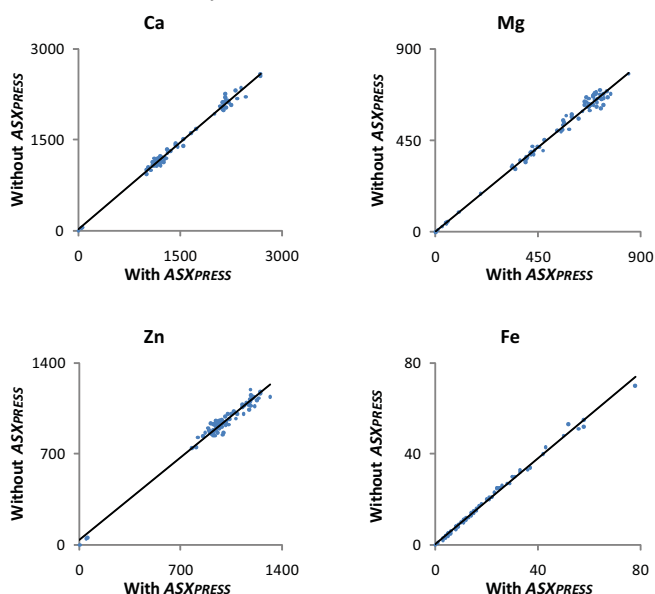


Figure 5. ICP data before and after *ASXPRESS™ PLUS*. Run comparison reflects 50% time savings when using *ASXPRESS™ PLUS*. All units are expressed in ppm.

Less than 0.1% carryover was observed with additive elements at multiple customer locations.

TIME SAVINGS

Existing oil analysis methods have been found to run samples on the order of 45 – 70 seconds 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 PerkinElmer Optima series ICP-AES, along with two different autosamplers. The CETAC ASX-520 is an XYZ autosampler. The CETAC ASX-1400 is an XYZ autosampler that has the added capability of stirring samples just prior to uptake/analysis.

Sampling time was cut to 20 – 21 seconds per sample when using the *ASXPRESS™ PLUS* and ASX-520 autosampler while still meeting all the criteria.

Times of 22 – 26 seconds per sample were achieved with the *ASXPRESS™ PLUS* and ASX-1400 mixing autosampler.

MINIMAL SAMPLE CONSUMPTION

The *ASXPRESS™ PLUS* also demonstrates great efficiency by using approximately 2 mL of sample per analysis depending on method setup. As such, multiple runs are possible from a single 5 mL of diluted sample solution while sample waste is minimized.

STABILITY AND LONGEVITY

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.

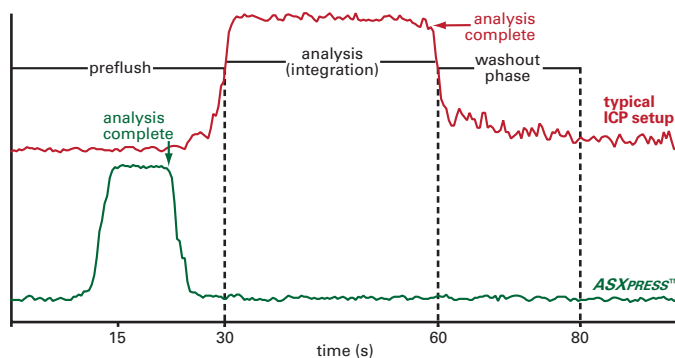


Figure 6. Time comparison, typical ICP setup vs. *ASXPRESS™ PLUS*

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.

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 PerkinElmer for details.

CONCLUSION

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