



# Rapid-Throughput Analysis for EPA Methods by ICP-AES and ICP-MS

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## Abstract

US Environmental Protection Agency (EPA) methods, which are relied upon by many regulatory agencies, are often lengthy and detailed. They provide specific instructions concerning sample collection, preservation, and treatment as well as required and recommended procedures for instrument calibration, tuning, and interference corrections. Compliance usually requires the methods be adhered to with no changes or modifications.

Even with rigorous compliance standards in place, increased productivity is desirable in most laboratories. An advanced sample introduction system, such as the CETAC **ASXPRESS<sup>®</sup> PLUS** Rapid Sample Introduction System can reduce time spent on sample delivery, stabilization, and washout.

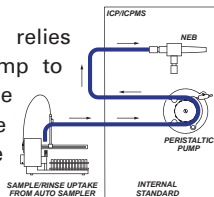


## Experimental

An accelerated sampling system, consisting of a CETAC **ASXPRESS<sup>®</sup> PLUS** Rapid Sample Introduction System coupled to a CETAC autosampler, was setup with various ICP-AES and ICP-MS instruments from multiple manufacturers. This system is introduced into the ICP-AES or ICP-MS analysis without altering method parameters such as elements, integration times, and number of replicates. The elimination of rinse times and a reduction of overall read delay times were the only changes.

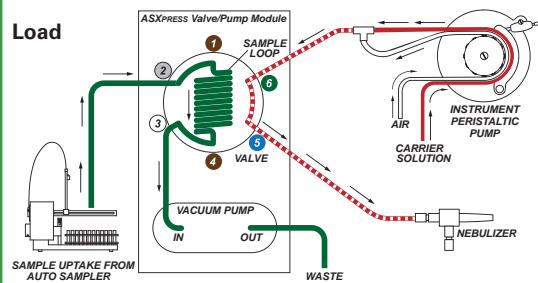
## Principle of Operation

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.



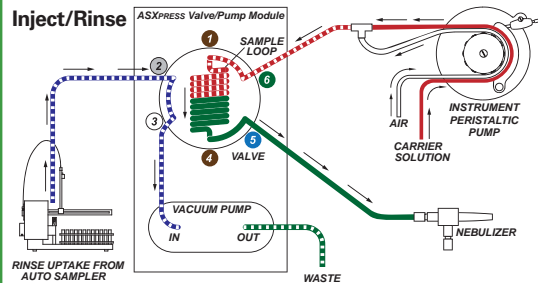
The **ASXPRESS<sup>®</sup> PLUS** utilizes a high speed vacuum pump in addition to the peristaltic pump to allow multiple functions to occur simultaneously which would otherwise take place separately. Analysis is divided into two stages:

### Load



The vacuum pump rapidly fills the sample loop while carrier solution is pumped by the ICP peristaltic pump to the nebulizer, maintaining plasma stability and rinsing the sample flow path.

### Inject/Rinse



The loaded sample is transported to the nebulizer by the carrier solution while the autosampler probe moves to the rinse station and the uptake flow path is flushed with rinse solution via the vacuum pump.

## ASXPRESS<sup>®</sup> PLUS Configuration Parameters

| Parameter            | Time (s) 6010C | Time (s) 200.7 | Time (s) 200.8 |
|----------------------|----------------|----------------|----------------|
| Loop Rinse           | 2.0            | 1.0            | 0.5            |
| Rinse Evacuation     | 2.0            | 1.0            | 1.0            |
| Loop Load            | 10.0           | 1.0            | 0.5            |
| Equalization         | 2.0            | 1.0            | 1.0            |
| Time to Evacuate     | 1.0            | 1.0            | 1.0            |
| Probe Rinse          | 5.0            | 1.0            | 1.0            |
| Rinse Station Refill | 4.0            | 3.5            | 3.0            |
| Loop Volume          | 5.0 mL         | 2.5 mL         | 1.5 mL         |

## Approximate analysis times, minutes per sample before & after implementation of ASXPRESS<sup>®</sup> PLUS

| EPA Method | Initial Analysis Time | Analysis Time w/ ASXPRESS <sup>®</sup> PLUS | Time Savings |
|------------|-----------------------|---|--------------|
| 6010C      | 6                     | 3.5   | 40%          |
| 200.7      | 2                     | 1   | 50%          |
| 200.8      | 7.5                   | 3.5   | 55%          |

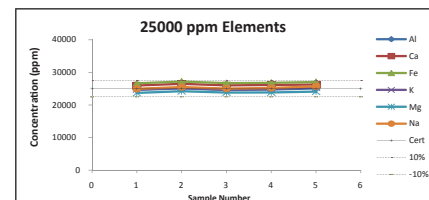
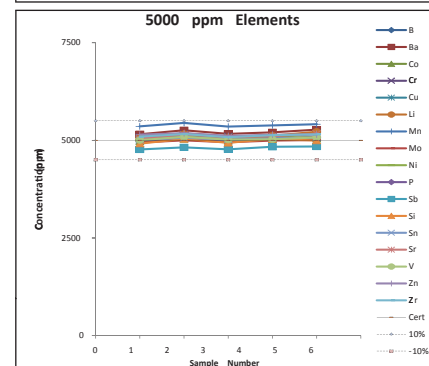
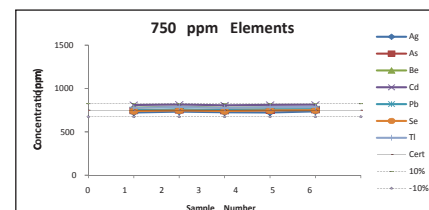
## Carryover Data for EPA 200.8 using ASXPRESS<sup>®</sup> PLUS

| 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                        |         |     |                                |

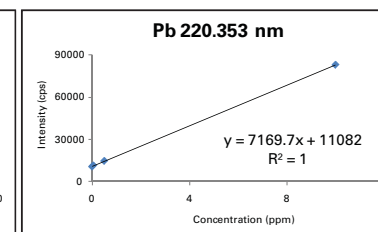
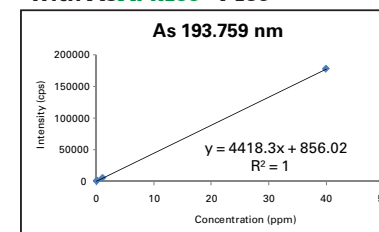
## Repeatability Data for EPA 200.8

| 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                |

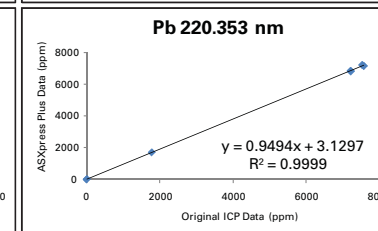
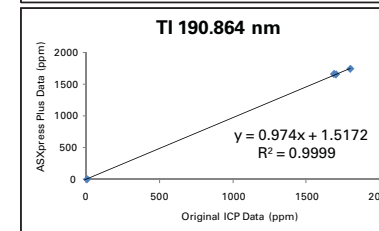
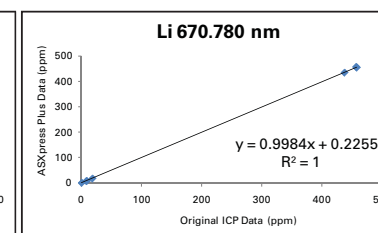
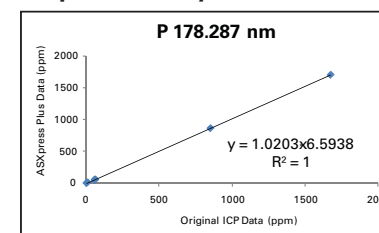
## Stability for EPA 6010C elements with ASXPRESS<sup>®</sup> PLUS



## Calibration Plots Showing Linearity for EPA 200.7 elements with ASXPRESS<sup>®</sup> PLUS



## Reproducibility for EPA 6010C elements with ASXPRESS<sup>®</sup> PLUS



## Results

The **ASXPRESS<sup>®</sup> PLUS** system achieved a time savings of 40–55% while preserving the original data quality.

## References

EPA Method 200.7, "Determination of Metals and Trace Metals in Water and Wastes by Inductively Coupled Plasma-Atomic Emission Spectrometry," Revision 4.4, 1994, Environmental Monitoring Systems Laboratory, Office of Research and Development, United States Environmental Protection Agency, Cincinnati, OH 45268.

EPA Method 200.8, "Methods for the Determination of Metals in Environmental Samples – Supplement 1", EPA-600/R-94-111, May 1994, Available at NTIS, PB 94-184942.

USEPA SW-846 Method 6010C, Revision 3, November 2000.

| Element | m/z | Average Measured Result (ppb) | RSD (%) | Known Value (ppb) | Average Recovery (%) |
|---------|-----|-------------------------------|---------|-------------------|----------------------|
| 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                 |