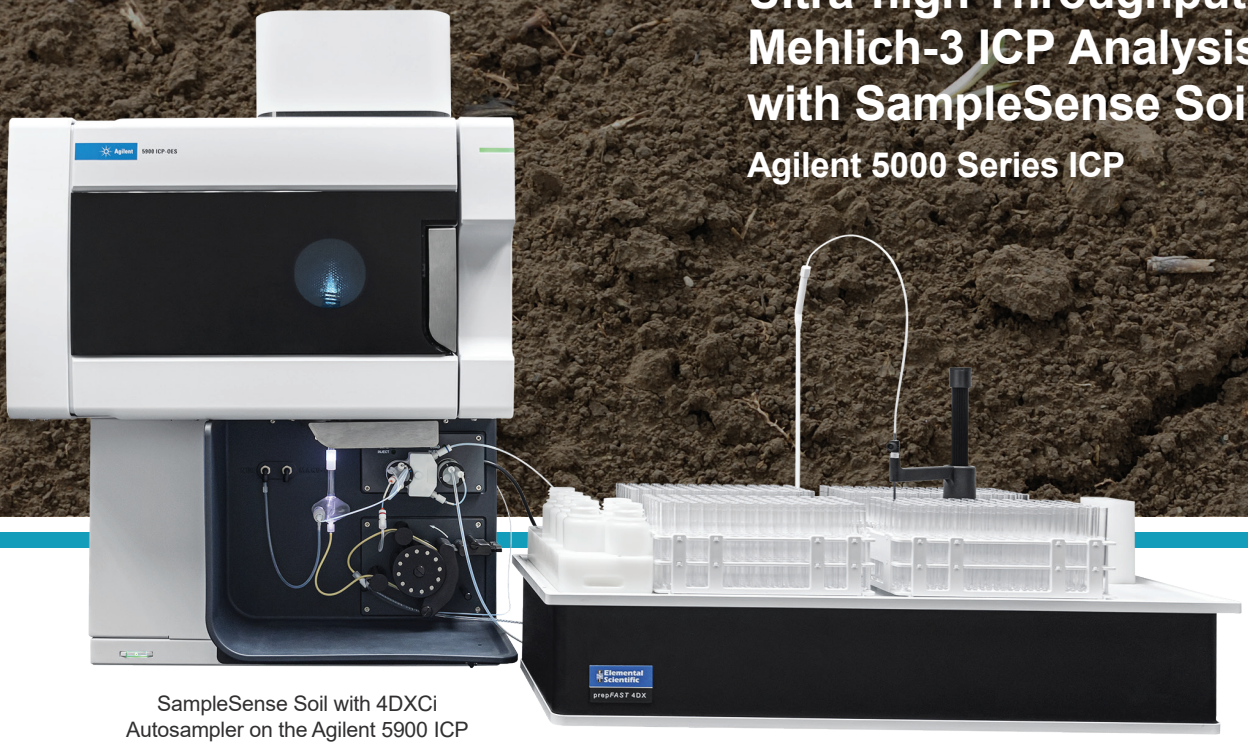
# Ultra-high Throughput Mehlich-3 ICP Analysis with SampleSense Soil

## Agilent 5000 Series ICP



SampleSense Soil with 4DXCi Autosampler on the Agilent 5900 ICP

## Introduction

Mehlich-3 ICP is a method for determining bioavailable concentrations of 11 extractable micronutrient elements in soil samples. Mehlich-3 ICP is invaluable for determining the amount of fertilizer to apply to farm fields. Because soil analyses must be completed in a narrow time window, ultra-high sample throughput with high-reliability is required.

SampleSense Soil uses an inert injection valve with built-in optical sensors that automatically detect the liquid sample, inject the valve and trigger the ICP read in a tightly-timed analytical sequence. SampleSense Soil eliminates wasted time from the ICP method and can double or even triple sample throughput on the Agilent 5000 Series ICP systems while recording missing or empty tubes.

## SampleSense Soil Benefits

- 8 samples per minute Mehlich-3 ICP
- Automatic sensing, injection, and triggering of the ICP analytical read
- Detection and reporting of missing or empty sample tubes as “unsensed” samples
- DXCi autosampler automatically goes to the correct sample position – even when accidentally obstructed
- Adding SampleSense Soil can double or even triple sample throughput

## SampleSense Soil Advantages for Mehlich-3 ICP

### Ease of Use

- Optical detection of the filled sample loop automatically triggers ICP analysis
- Removes timing and read delay adjustments
- Eliminates method development when changing sample loop size for other methods
- Automatic in-line addition of internal standard(s) if desired

### Automated Compensation of Physical Clogs and Timing Variables

- Reliable timing down to 0.05 s and better
- Compensates for:
  - Partial clogs from filter paper fibers, particles, line kinks, etc.
  - Timing variables caused by high or low sample tube levels
  - ICP computer slow-down from software and data storage

### Increased Productivity

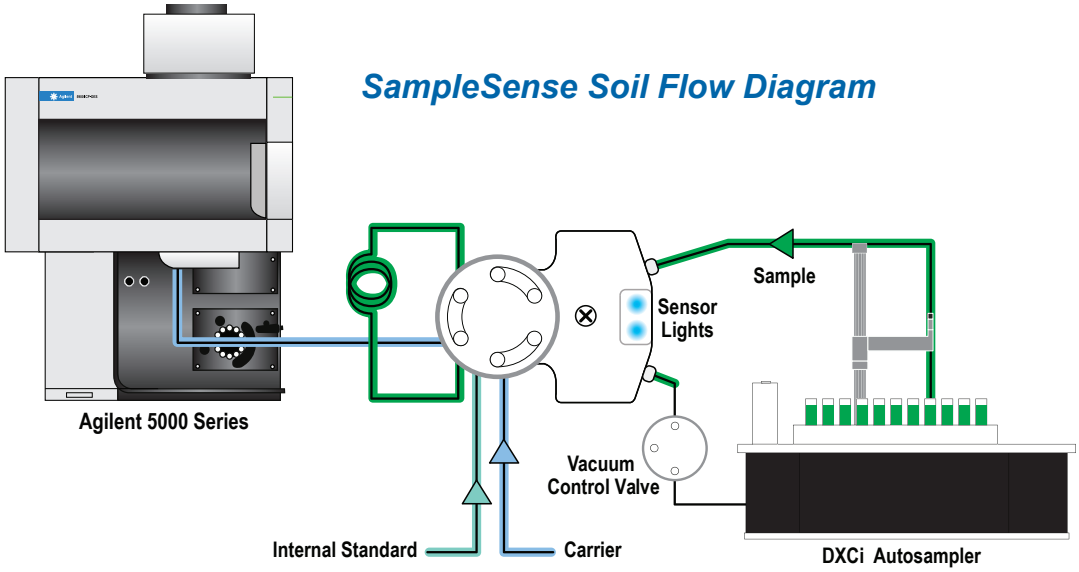
- Produces long analytical runs without operator intervention
- Minimized sample consumption allows reanalysis
- Doubles or triples throughput of ICP instrument
- Reduces argon consumption

SampleSense valve with dual optical sensors

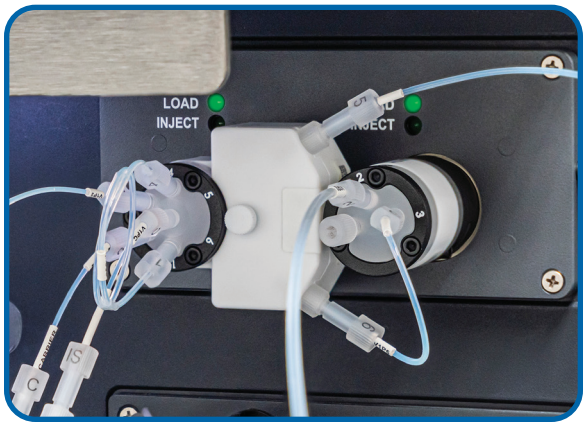
## SampleSense Soil for Mehlich-3 ICP Soil Analysis

- SampleSense Soil ICP (Agilent 5900 SVDV/VDV)
- 11 Elements determined: B, Ca, Cu, Fe, K, Mg, Mn, Na, S, P, Zn in radial view mode
- ICP read triggered from dual sensor optical sample detection
- 360 samples analyzed in <45 minutes
- 7.5 second sample-to-sample time
- <2 mL of sample consumed with vacuum control
- Automatic detection of empty or missing sample tubes
- Most reliable high-throughput system on the market

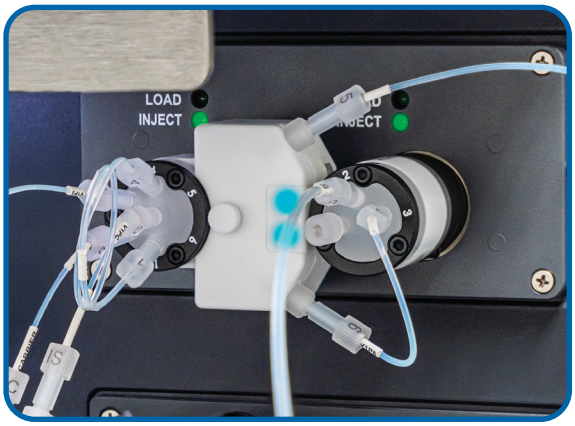
## SampleSense Soil Flow Diagram



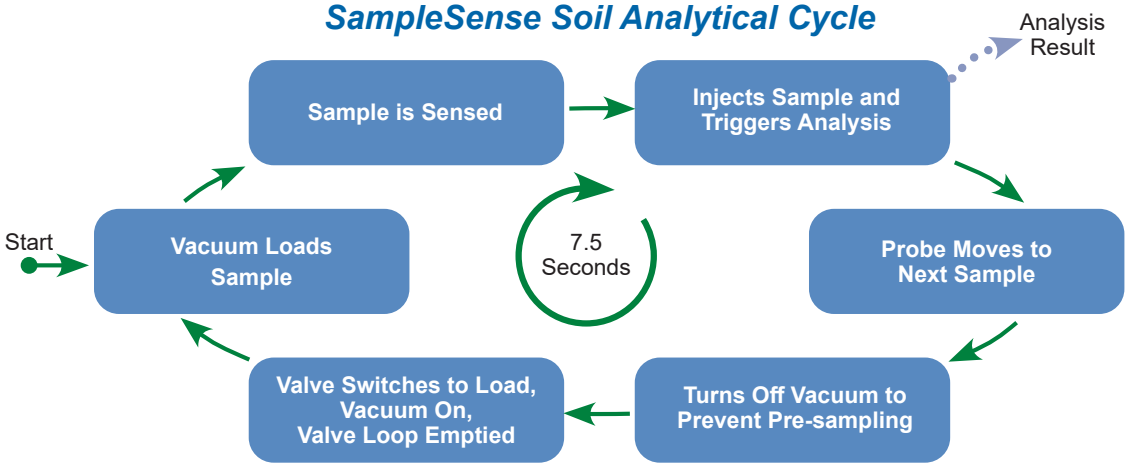

### Sample not present, valve sensors not activated




### Valve sensors activated, sample is sensed




## SampleSense Soil Analytical Cycle



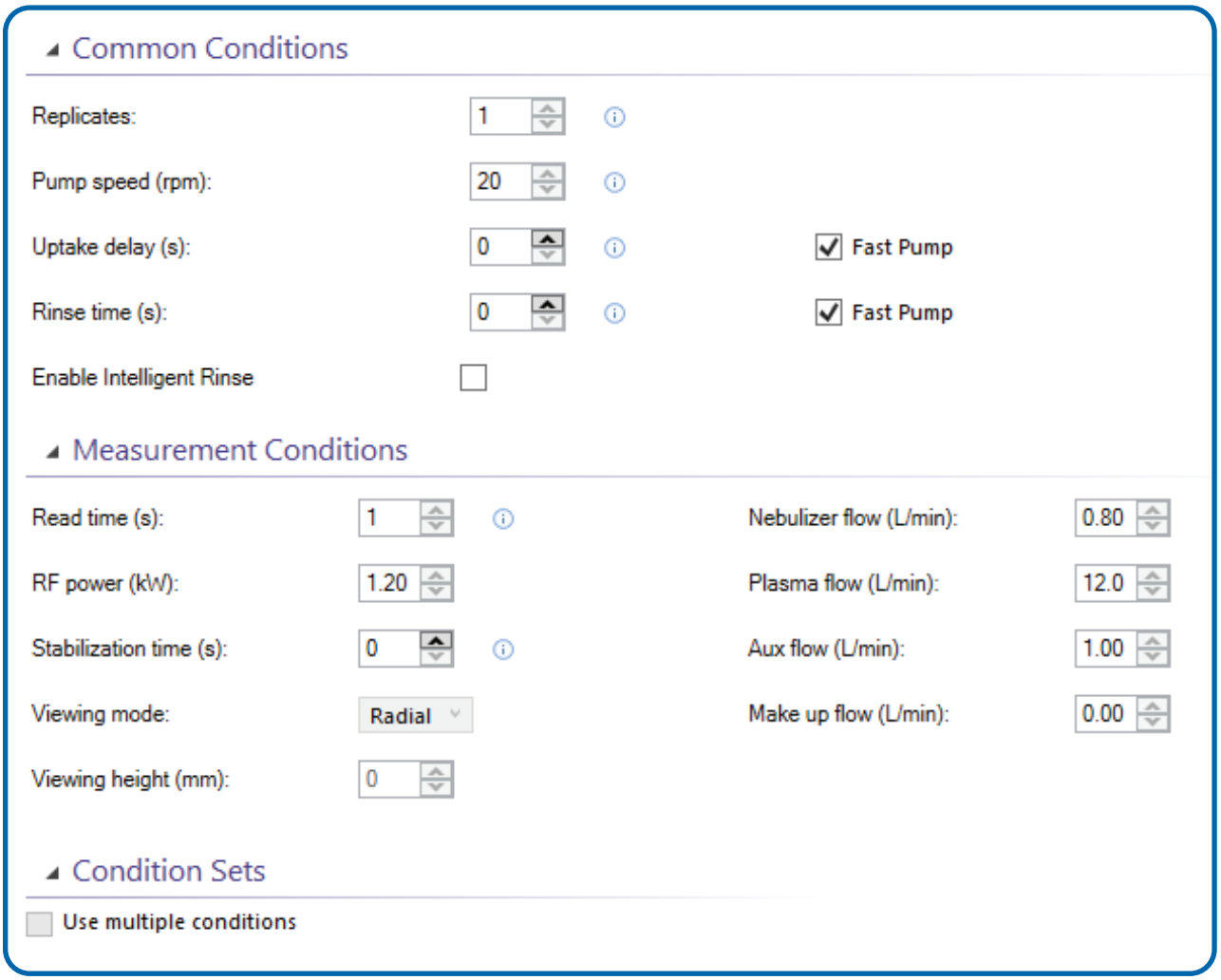
## Method Conditions

### ICP Conditions

- Plasma Gas Flow: 12 L/min
- Aux Gas Flow: 1 L/min
- Nebulizer Gas Flow: 0.8 L/min
- Plasma Power: 1.2 kW
- Plasma View: Radial
- Replicates: 1
- Total Analysis Time ~7.5 sec per sample

### Mehlich-3 Soil Analytes (nm):

- B 249.678
- Ca 317.933
- Cu 327.395
- Fe 238.204
- K 766.491
- Mg 279.078
- Mn 257.610
- Na 589.592
- P 214.914
- S 180.669
- Zn 213.857




Agilent 5900 ICP method conditions

## Calibration Standards

Concentrations

Correlation coefficient limit: 0.999

Include blank in calibration

Standard additions

Use reagent blank

Enable Reslope:

Number of standards: 4

| Solution Label | B | Ca | Cu | Fe | K | Mg | Mn | Na | P | S | Tm | Zn |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | ppm | ppm | ppm | ppm | ppm | ppm | ppm | ppm | ppm | ppm | ppm | ppm |
| Blank | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| Bottom Standard | 0.20 | 10.00 | 0.80 | 8.00 | 40.00 | 24.00 | 2.00 | 6.00 | 8.00 | 4.00 | | 0.40 |
| Lower Mid Standard | 0.50 | 25.00 | 2.00 | 20.00 | 100.00 | 60.00 | 5.00 | 15.00 | 20.00 | 10.00 | | 1.00 |
| Higher Mid Standard | 1.00 | 50.00 | 4.00 | 40.00 | 200.00 | 120.00 | 10.00 | 30.00 | 40.00 | 20.00 | | 2.00 |
| Top Standard | 2.00 | 100.00 | 8.00 | 80.00 | 400.00 | 240.00 | 20.00 | 60.00 | 80.00 | 40.00 | | 4.00 |

The ICP system was calibrated for the 11 elements of interest with a blank and 4 standards at varying concentrations across the ppm range. The additional element Tm was introduced to assess injection completeness and stability. The linearity of the resulting calibrations are shown on pages 6 and 7.

## Missing Samples Detected

Message

**Unsensed Samples**

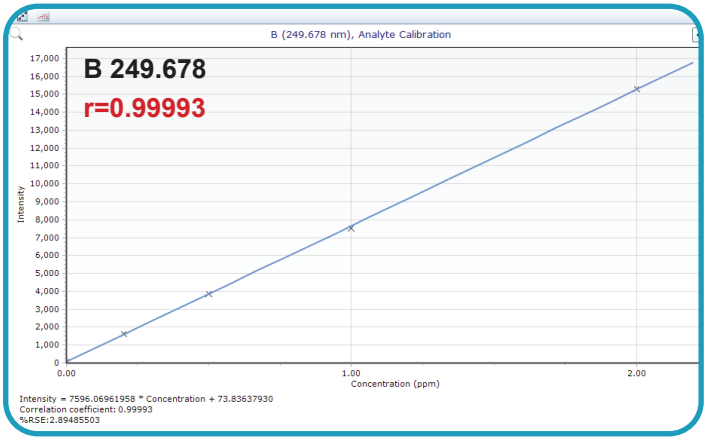
| | SC Rack Number | SC Vial Number | Instrument Rack | Instrument Vial | Time |
|---|---|---|---|---|---|
| ▶ | 1 | 45 | 1 | 45 | 20190813 9:05:00 |
| | 1 | 90 | 1 | 90 | 20190813 9:09:34 |
| | 2 | 45 | 2 | 45 | 20190813 9:14:08 |
| | 2 | 90 | 2 | 90 | 20190813 9:18:42 |
| | 3 | 45 | 3 | 45 | 20190813 9:23:16 |
| | 3 | 90 | 3 | 90 | 20190813 9:27:50 |

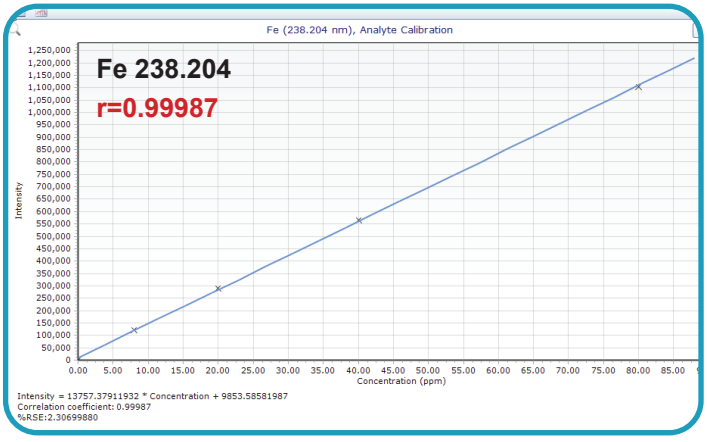
This table demonstrates the automatic missed sample logging capabilities provided by the SampleSense Soil sensors. Empty sample vials were placed in the first three sample racks at positions 45 and 90. SampleSense identified the missing samples and provides this information in the software log shown here.

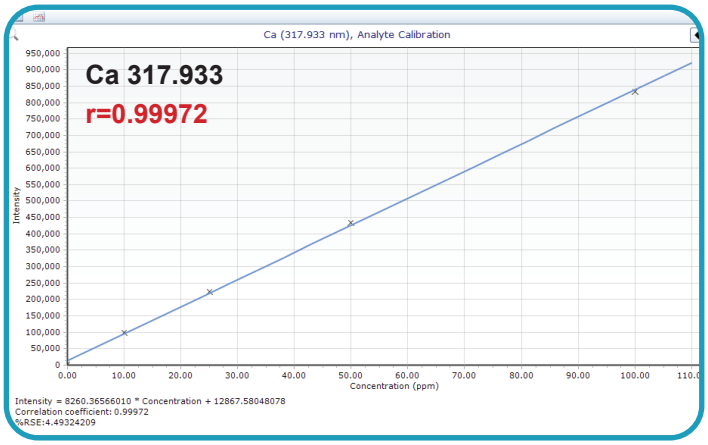
## Low Sample Consumption

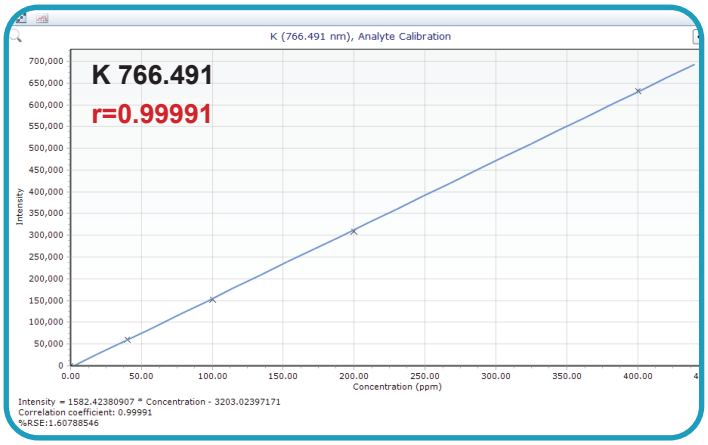
SampleSense Soil consumes < 2 mL of sample. The black line shows the original level of 5 mL extract. Post analysis 3.5 mL remains. Samples can be reanalyzed without re-extraction

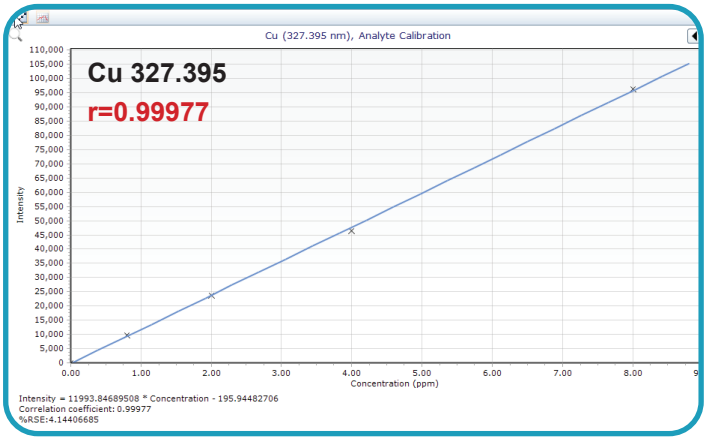
## Calibration Curves

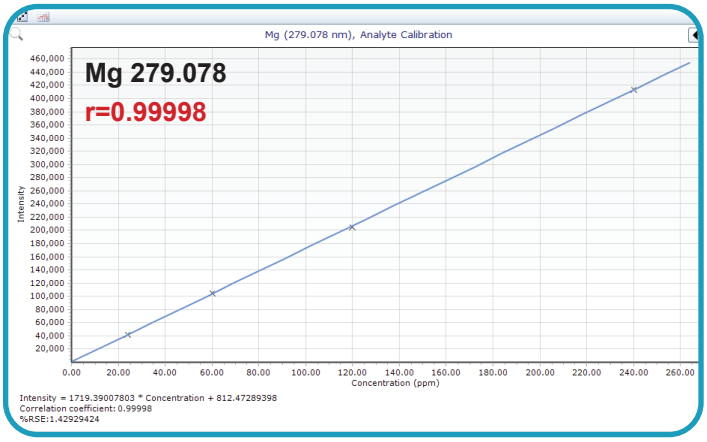


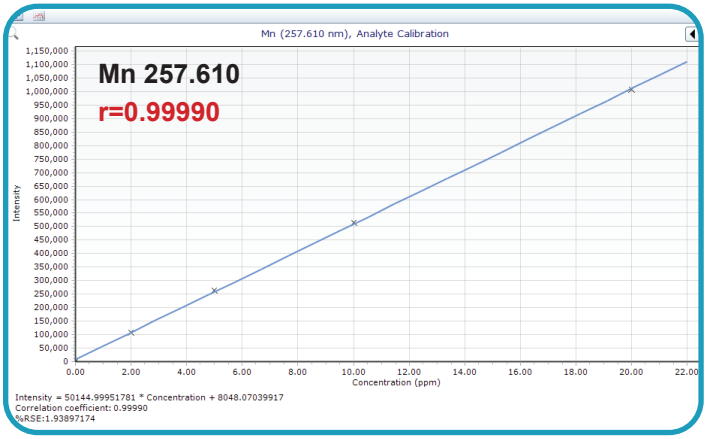



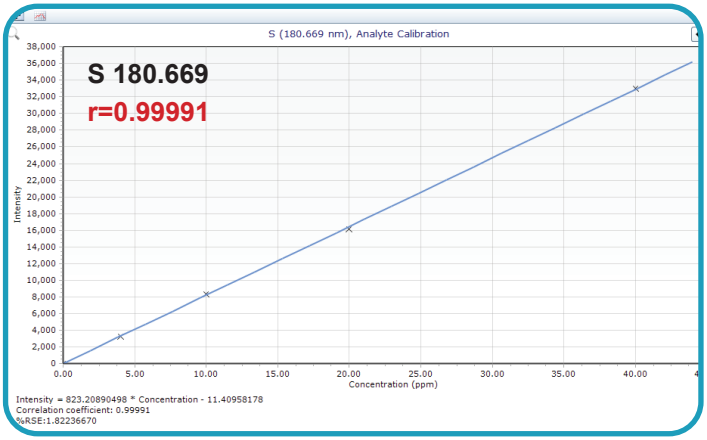



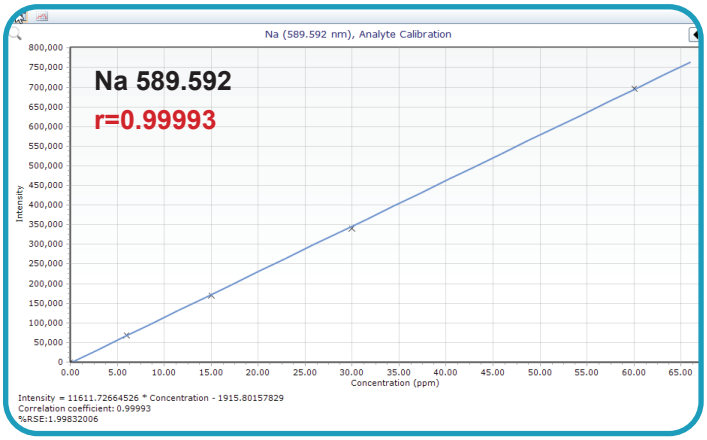



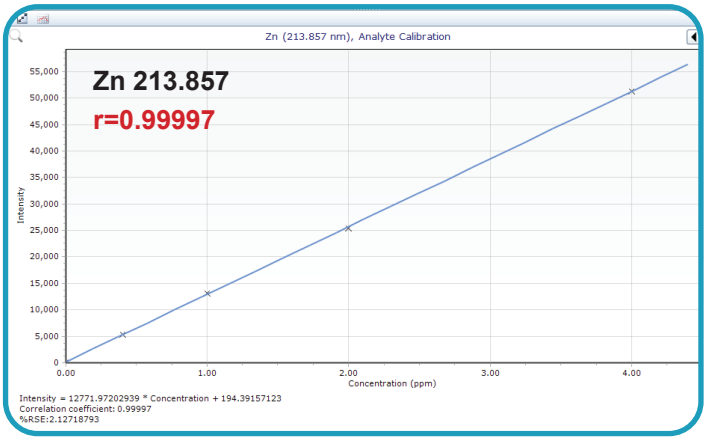



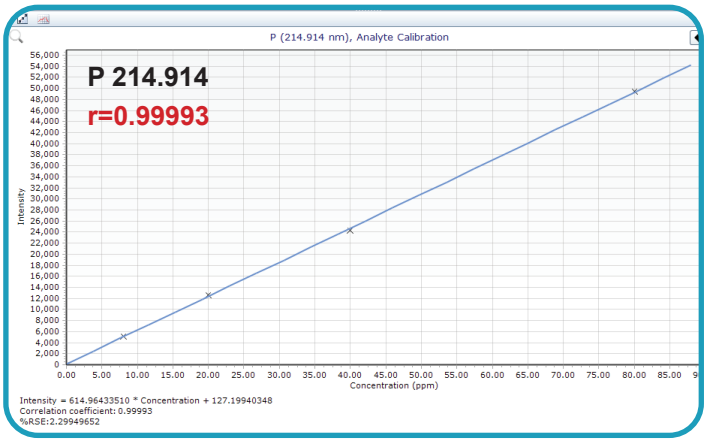





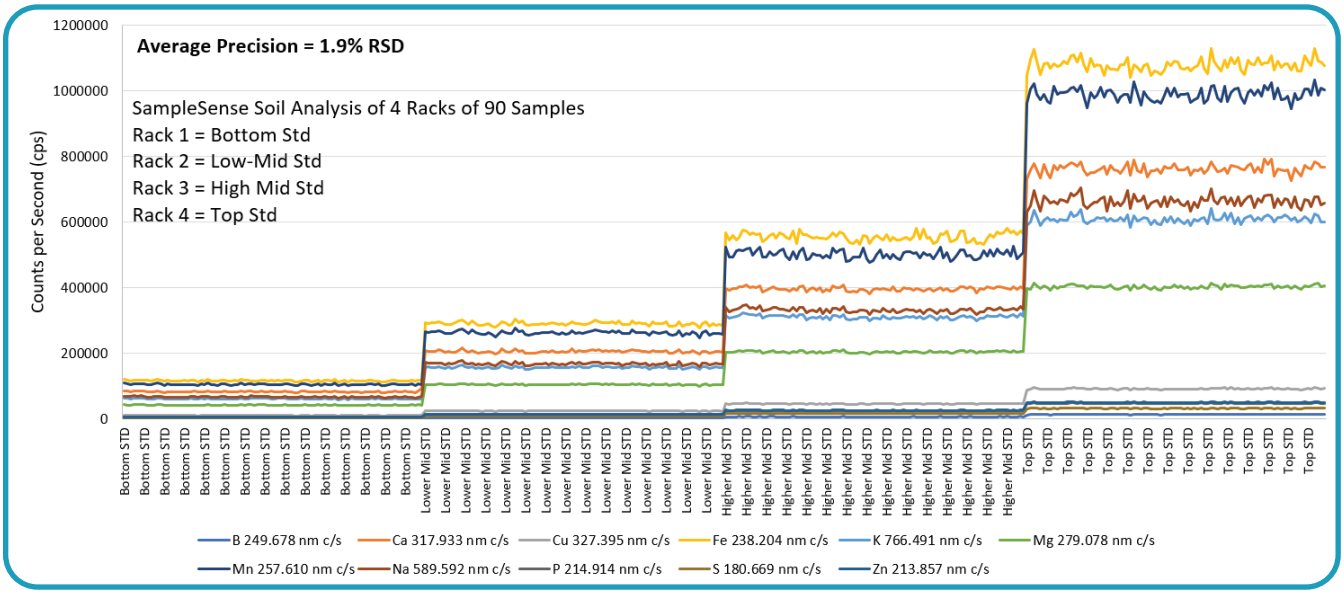




## Correlation Coefficient Data

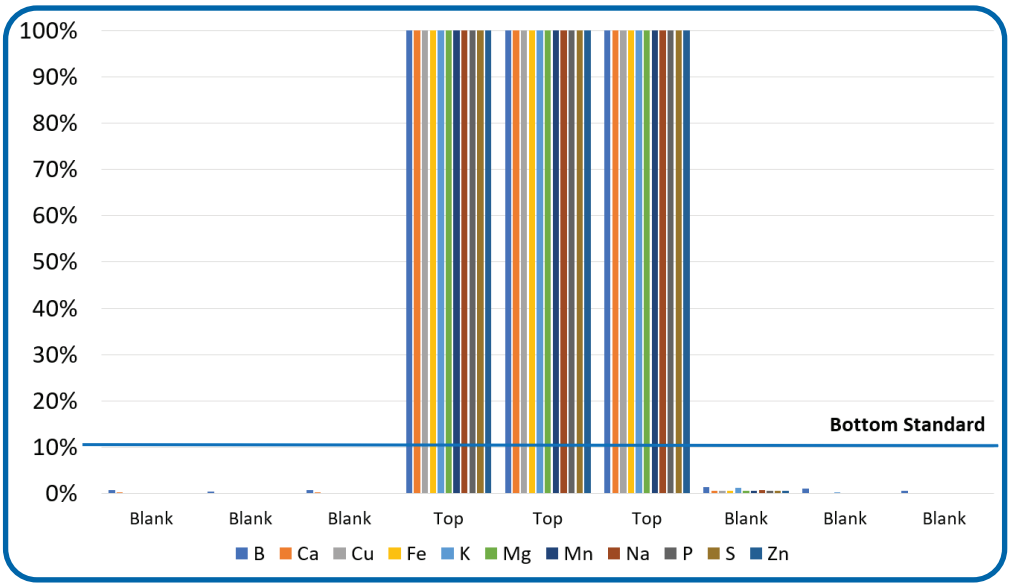
| Element | Correlation Coefficient |
|---|---|
| B | 0.9999 |
| Ca | 0.9997 |
| Cu | 0.9997 |
| Fe | 0.9998 |
| K | 0.9999 |
| Mg | 0.9999 |
| Mn | 0.9999 |
| Na | 0.9999 |
| P | 0.9999 |
| S | 0.9999 |
| Zn | 0.9999 |

## Throughput - 360 samples in 45 minutes




Analysis of 360 samples in 45 minutes. The four levels represent a rack of 90 samples, with each of the four calibration standards levels individually loaded with 5 mL of liquid into each of the 90 positions contained in each rack. Excellent stability observed from within each of the four concentration levels.

## Rinseout




The SampleSense Soil rinse-out immediately reduces all elements to concentrations well below the bottom standard when using the seven and one half second Mehlich-3 ICP procedure. Additional seconds can be added to the rinse-out to meet lab requirements. The system can deliver rinse-out factors of 1,000x, 10,000x or more depending on how clear the spray chamber needs to be before the next sample is introduced.

## Conclusion

At 8 samples per minute, SampleSense Soil for Mehlich-3 ICP method can more than double the productivity of the ICP instrument. It delivers reliable and reproducible data, while providing quick and effective sample rinse out.

| Description | Part Numbers |
|---|---|
| SampleSense Soil 2DXCi | 2F-SS-Soil-65 |
| SampleSense Soil 4DXCi | 4F-SS-Soil-65 |
| SampleSense Soil 8DXCi | 8F-SS-Soil-65 |
| SampleSense Soil 14DXCi | 14F-SS-Soil-65 |

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