Elemental Scientific

SampleSense *FAST* UHT-S with 4DXCi Autosampler on the Agilent 5900 ICP

Ultra-high Throughput Mehlich-3 ICP Analysis with SampleSense *FAST* UHT-S for Agilent 5000 Series 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 *FAST* UHT-S* 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 *FAST* UHT-S 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 *FAST* UHT-S 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 *FAST* UHT-S can double or even triple sample throughput

SampleSense *FAST* UHT-S 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
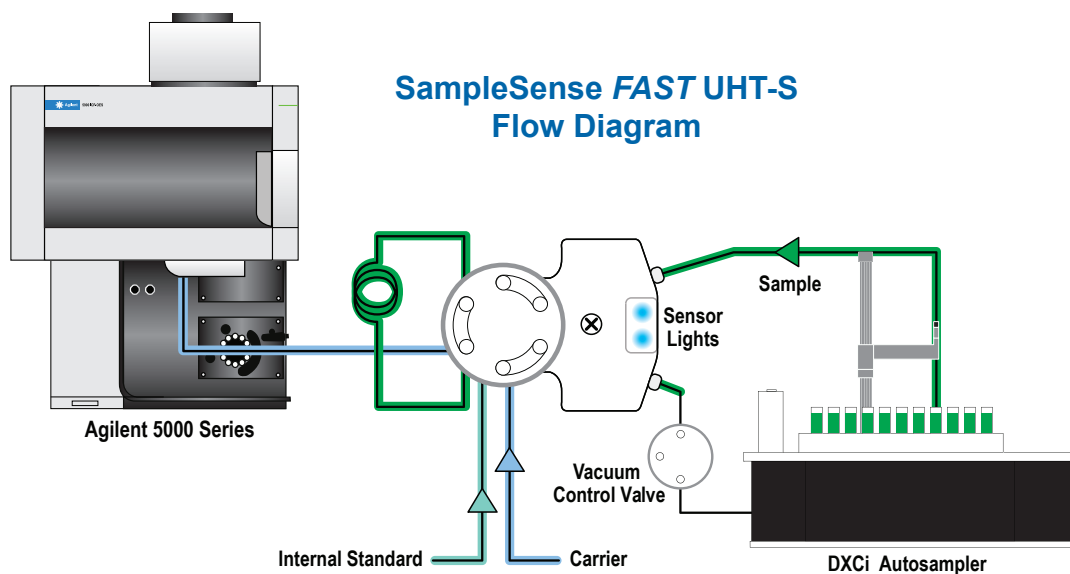- Doubles or triples throughput of ICP instrument
- Reduces argon consumption

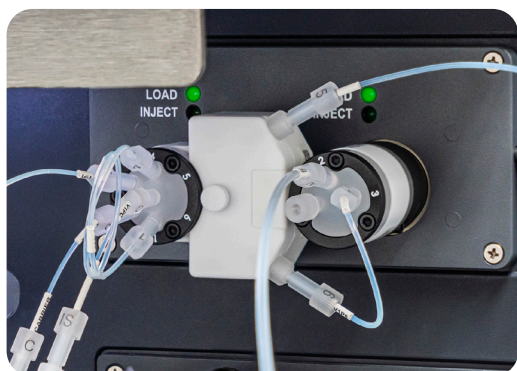

SampleSense valve with
dual optical sensors

SampleSense *FAST* UHT-S for Mehlich-3 ICP Soil Analysis

- SampleSense *FAST* UHT-S 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
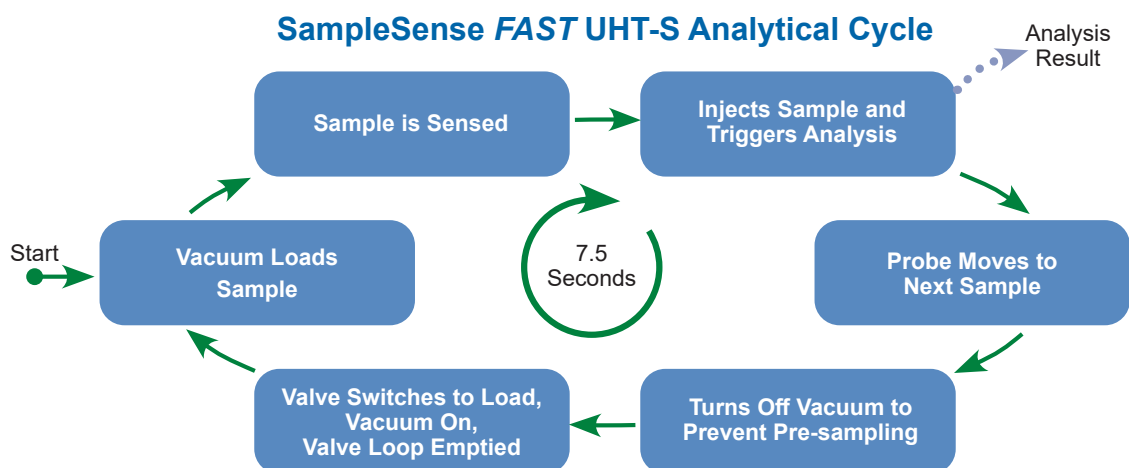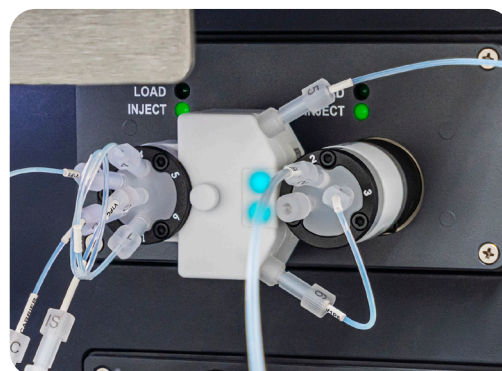- Most reliable high-throughput system on the market

SampleSense *FAST* UHT-S

SampleSense *FAST* UHT-S Flow Diagram

Agilent 5000 Series

Sensor Lights

Sample

Vacuum Control Valve

DXCi Autosampler

Internal Standard

Carrier

Sample not present, valve sensors not activated

Valve sensors activated, sample is sensed

SampleSense *FAST* UHT-S Analytical Cycle

Analysis Result

Sample is Sensed → **Injects Sample and Triggers Analysis**

7.5 Seconds

Start → **Vacuum Loads Sample**

Probe Moves to Next Sample

Valve Switches to Load, Vacuum On, Valve Loop Emptied ← **Turns Off Vacuum to Prevent Pre-sampling**

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

◢ Common Conditions

Replicates:	1	ⓘ
Pump speed (rpm):	20	ⓘ
Uptake delay (s):	0	ⓘ ✓ Fast Pump
Rinse time (s):	0	ⓘ ✓ Fast Pump
Enable Intelligent Rinse	☐	

◢ Measurement Conditions

Read time (s):	1	ⓘ	Nebulizer flow (L/min):	0.80
RF power (kW):	1.20		Plasma flow (L/min):	12.0
Stabilization time (s):	0	ⓘ	Aux flow (L/min):	1.00
Viewing mode:	Radial		Make up flow (L/min):	0.00
Viewing height (mm):	0			

◢ Condition Sets

☐ Use multiple conditions

Calibration Standards

Concentrations

Correlation coefficient limit	0.999 ⓘ	Standard additions	
Include blank in calibration	✓	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 *FAST* UHT-S 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.
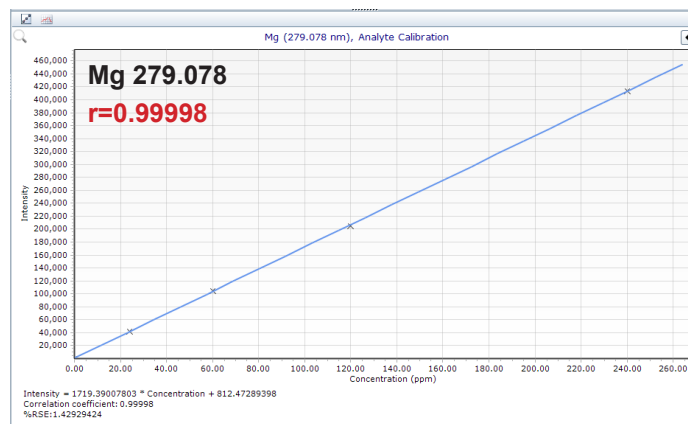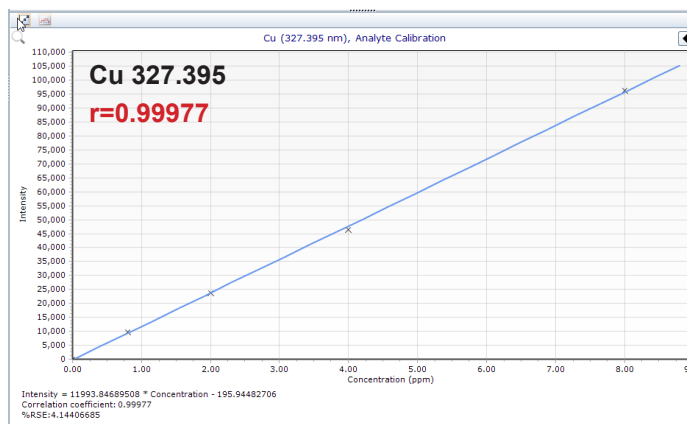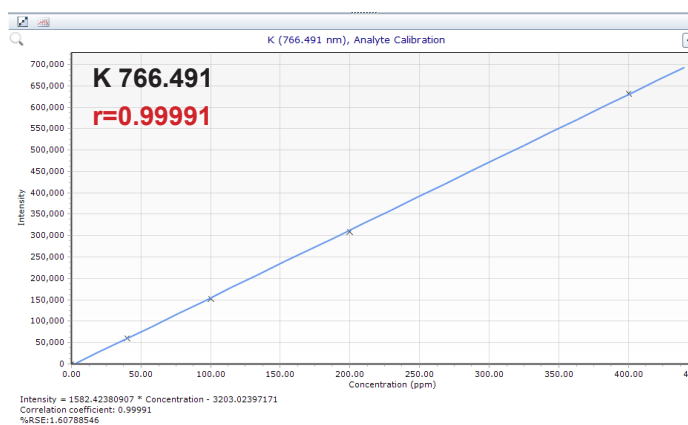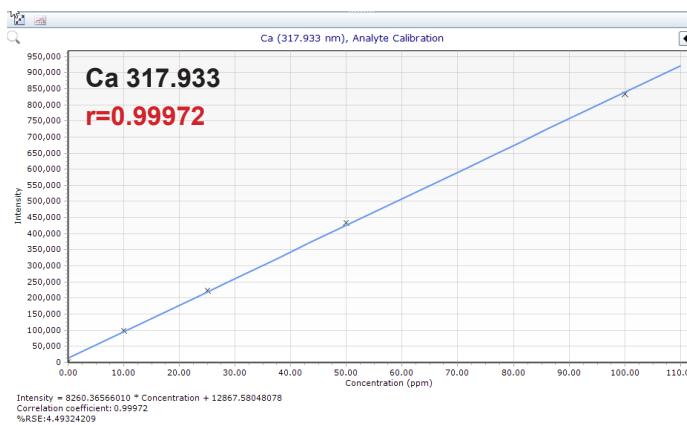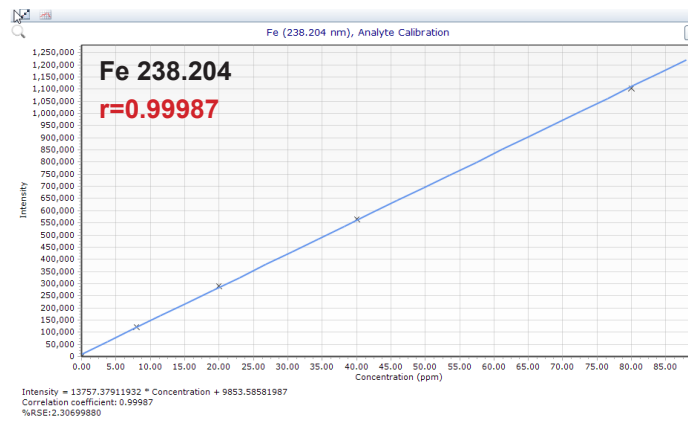
Low Sample Consumption

SampleSense *FAST* UHT-S 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.
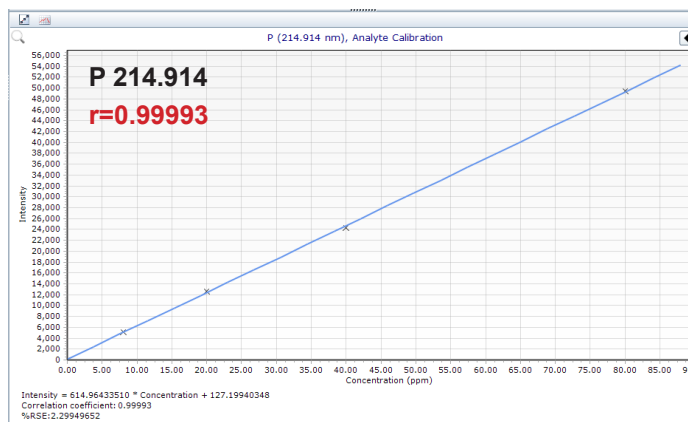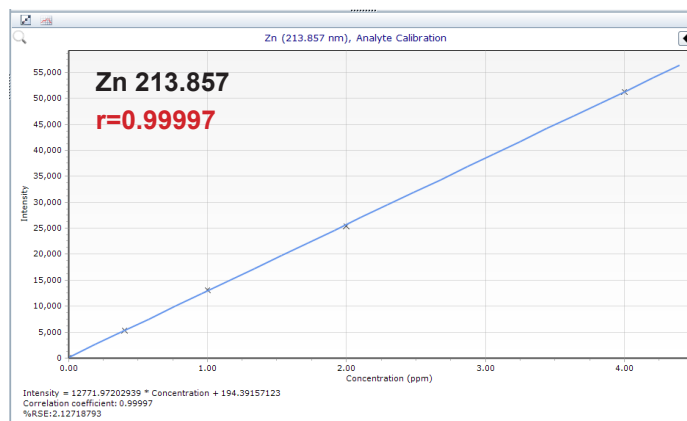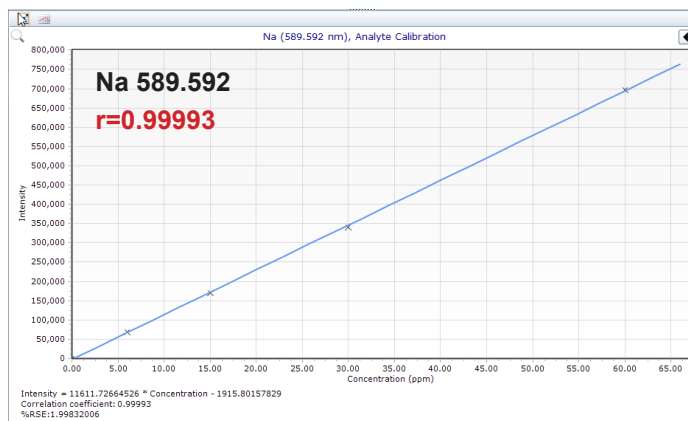
Experimental *(Continued)*

Calibration Curves

B 249.678
r=0.99993

B (249.678 nm), Analyte Calibration

Intensity = 7596.06961958 * Concentration + 73.83637930
Correlation coefficient: 0.99993
%RSE:2.89485503

Fe 238.204
r=0.99987

Fe (238.204 nm), Analyte Calibration

Intensity = 13757.37911932 * Concentration + 9853.58581987
Correlation coefficient: 0.99987
%RSE:2.30699880

Ca 317.933
r=0.99972

Ca (317.933 nm), Analyte Calibration

Intensity = 8260.36566010 * Concentration + 12867.58048078
Correlation coefficient: 0.99972
%RSE:4.49324209

K 766.491
r=0.99991

K (766.491 nm), Analyte Calibration

Intensity = 1582.42380907 * Concentration - 3203.02397171
Correlation coefficient: 0.99991
%RSE:1.60788546

Cu 327.395
r=0.99977

Cu (327.395 nm), Analyte Calibration

Intensity = 11993.84689508 * Concentration - 195.94482706
Correlation coefficient: 0.99977
%RSE:4.14406685

Mg 279.078
r=0.99998

Mg (279.078 nm), Analyte Calibration

Intensity = 1719.39007803 * Concentration + 812.47289398
Correlation coefficient: 0.99998
%RSE:1.42929424

Calibration Curves

Mn 257.610
r=0.99990

Intensity = 50144.99951781 * Concentration + 8048.07039917
Correlation coefficient: 0.99990
%RSE:1.93897174

S 180.669
r=0.99991

Intensity = 823.20890498 * Concentration - 11.40958178
Correlation coefficient: 0.99991
%RSE:1.82236670

Na 589.592
r=0.99993

Intensity = 11611.72664526 * Concentration - 1915.80157829
Correlation coefficient: 0.99993
%RSE:1.99832006

Zn 213.857
r=0.99997

Intensity = 12771.97202939 * Concentration + 194.39157123
Correlation coefficient: 0.99997
%RSE:2.12718793

P 214.914
r=0.99993

Intensity = 614.96433510 * Concentration + 127.19940348
Correlation coefficient: 0.99993
%RSE:2.29949652

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

Results

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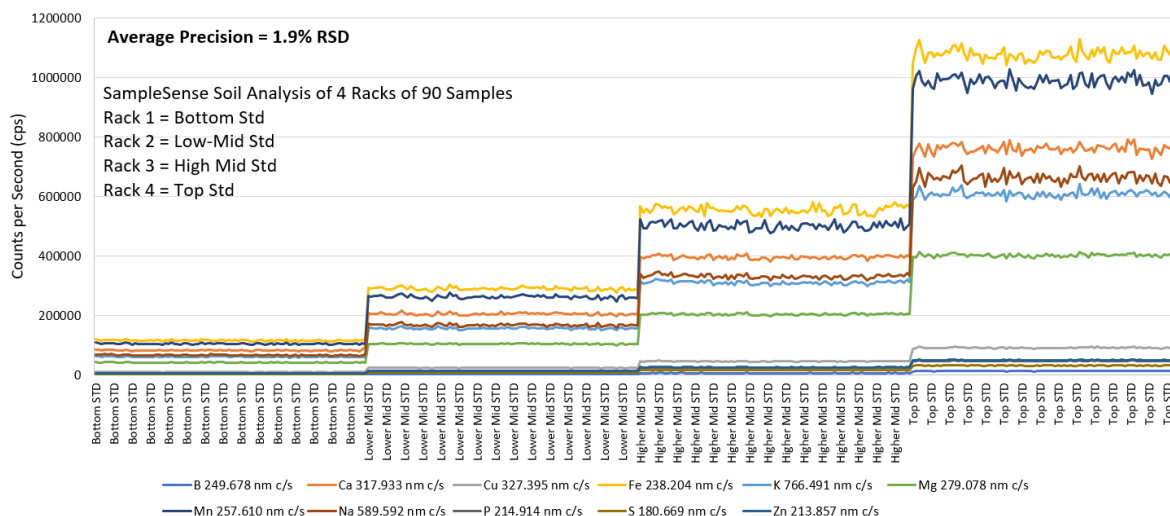
Analysis of 360 samples in 45 minutes. The four levels represent a rack of 90 samples, with each of the four calibration standards levels individually loaded with 5 mL of liquid into each of the 90 positions contained in each rack. Excellent stability observed from within each of the four concentration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

Rinseout
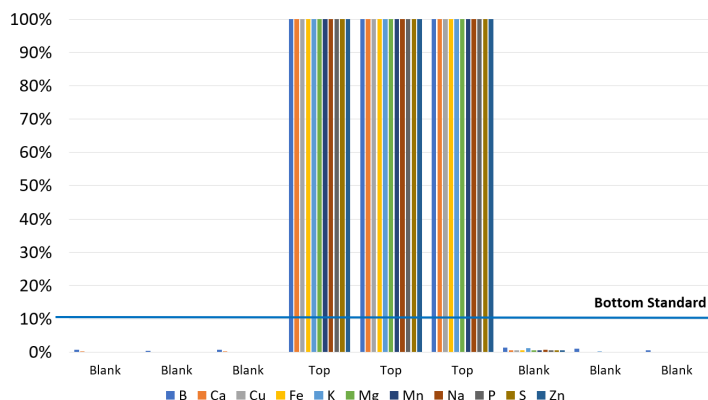
The SampleSense *FAST* UHT-S rinse out immediately reduces all elements to concentrations well below the bottom standard when using the seven and one half second Mehlich-3 ICP procedure. Additional seconds can be added to the rinse-out to meet lab requirements. The system can deliver rinse-out factors 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 *FAST* UHT-S 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 *FAST* UHT-S 2DXCi	2F-SS-Soil-65
SampleSense *FAST* UHT-S 4DXCi	4F-SS-Soil-65
SampleSense *FAST* UHT-S 8DXCi	8F-SS-Soil-65
SampleSense *FAST* UHT-S 14DXCi	14F-SS-Soil-65