



# High Throughput Automation for EPA Method 200.7 & 200.8

NEMC Conference 2021



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# New Automation Creating High Throughput

- SampleSense Enviro
  - Reduces EPA methods sample to sample time
  - Provides Quality Assurance for sample loading
  - Eliminates method timing
  - Eliminates loading variability due to sample viscosity

Improves Sample throughput without data compromise



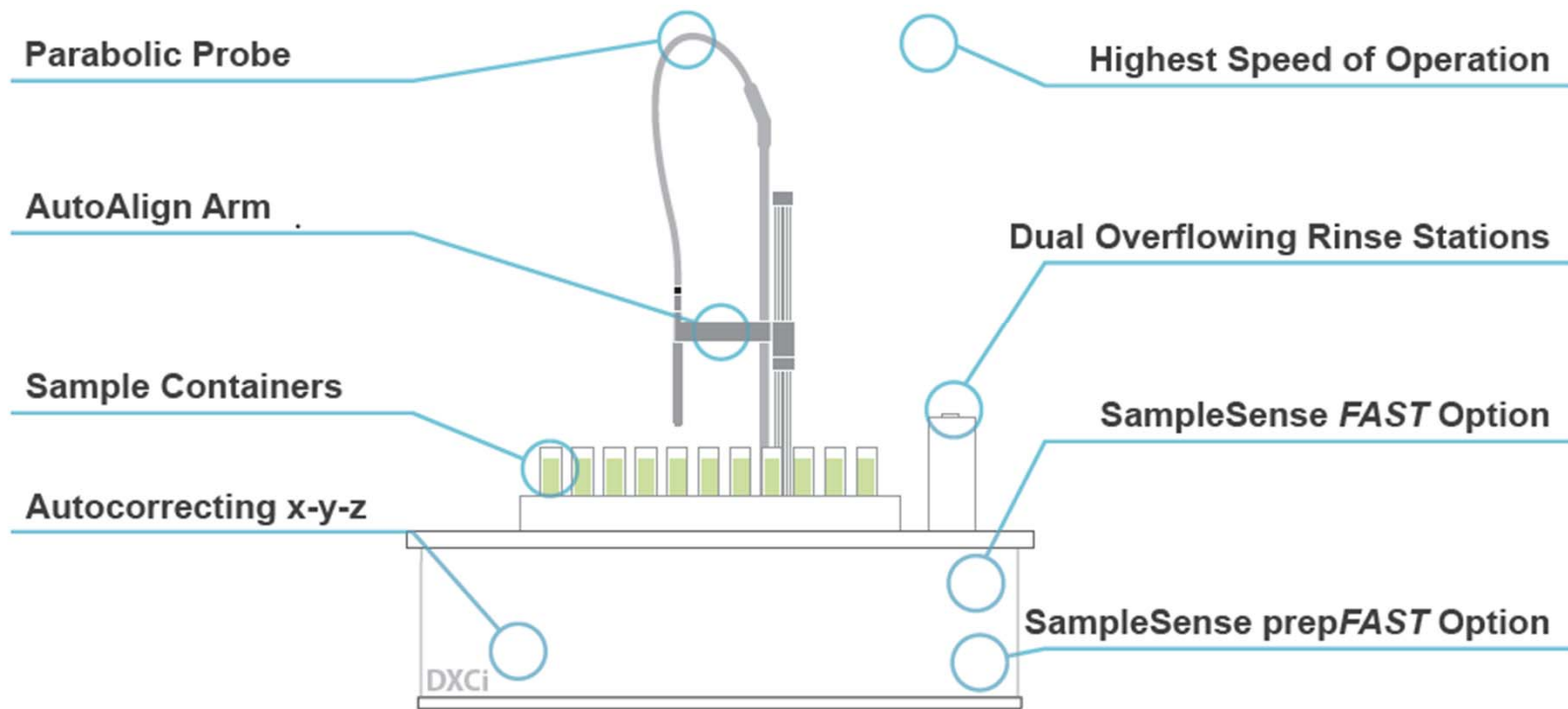
# SampleSense Enviro

- **What is SampleSense Enviro?**

- **DXCi Intelligent Autosampler**
- **SampleSense *FAST***
  - *Vacuum control valve*
- **Pergo Argon Humidifier**

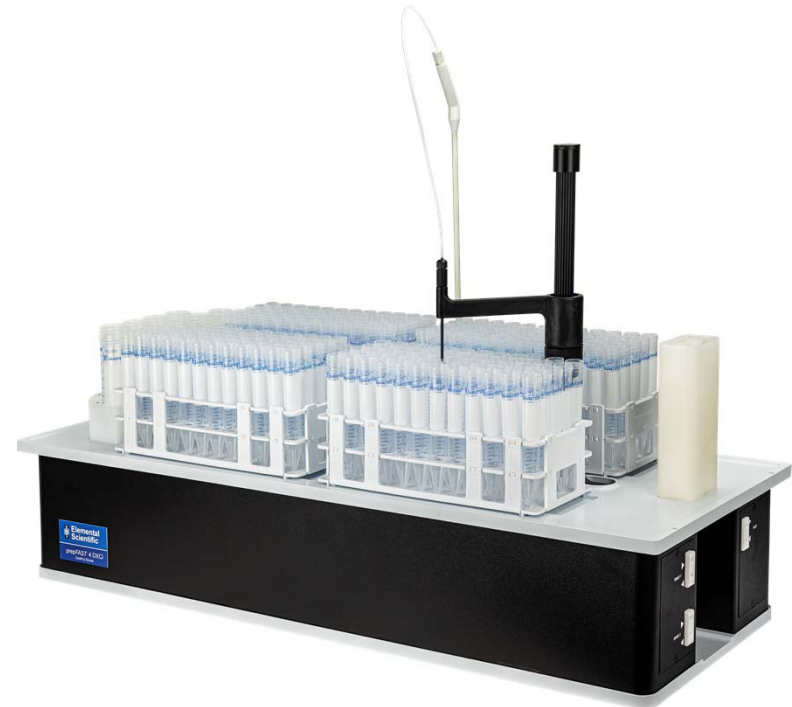


# DXCi: The Intelligent Autocorrecting Autosampler



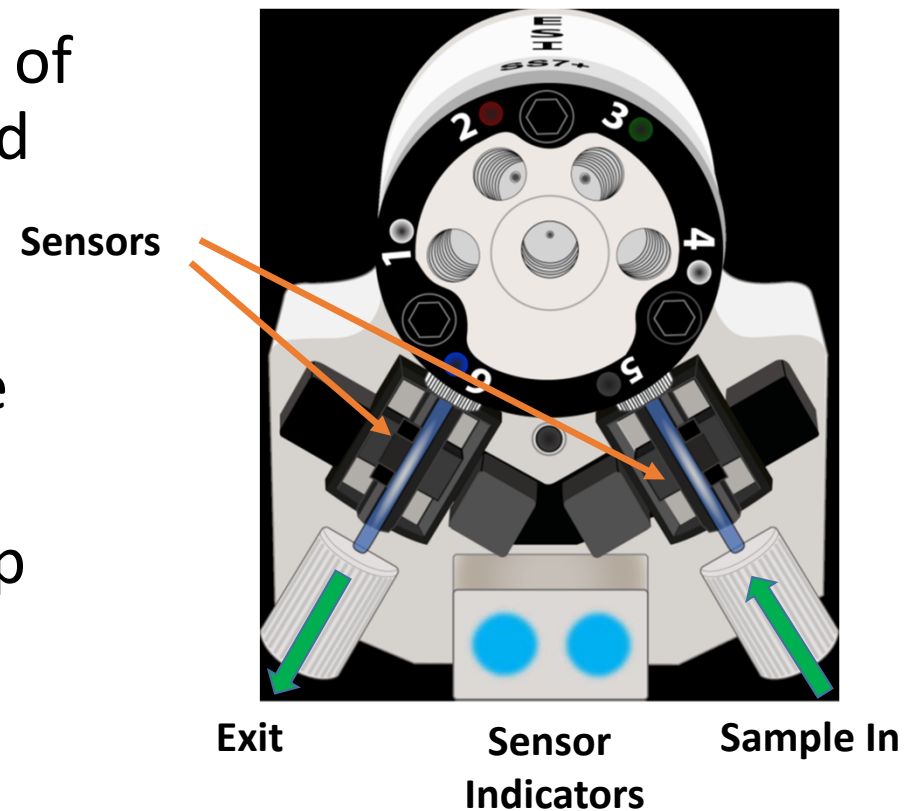
## New DXCi Intelligent Position-Correcting Autosampler

- Monitors autosampler probe position in 3 axes of motion
- Intelligently corrects the position
- Reports unmoved obstructions
- Eliminates analytical interruptions
- Available in 2DXCi, 4DXCi, 8DXCi, 14DXCi
- All SampleSense systems will include new DXCi



# SampleSense Valve – how does it work?

- Optical sensors monitor for arrival of solution front at both entrance and exit.
- Solution arrival at both locations provides confirmation that sample loop is fully loaded
- Air bubbles during the sample loop loading are detected

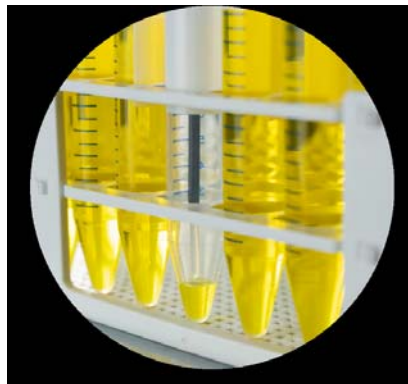
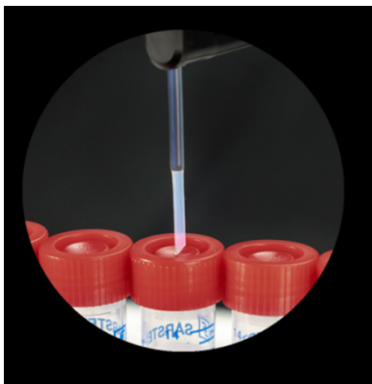




# SampleSense Enviro: Optimized QC Workflow

- **Intelligent Sample Loading Verification**

- SampleSense Enviro reports non-sample events:
  - Capped Sample Vials
  - Underfilled Sample Vials
  - Underfilled/Empty QC Standards
- Log of “Un-Sensed” Samples is Generated



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

## SampleSense Enviro: Low Sample Consumption



- Vacuum-Control Valve:
  - Trapping valve minimizes sample consumption.
  - Automatically shutting off the vacuum to conserve valuable sample.
- SampleSense Enviro consumes < 2.5 mL of sample\*.
  - The black line shows the original level of 6 mL sample
  - Post analysis, 3.5 mL of sample remains for a potential reanalysis.

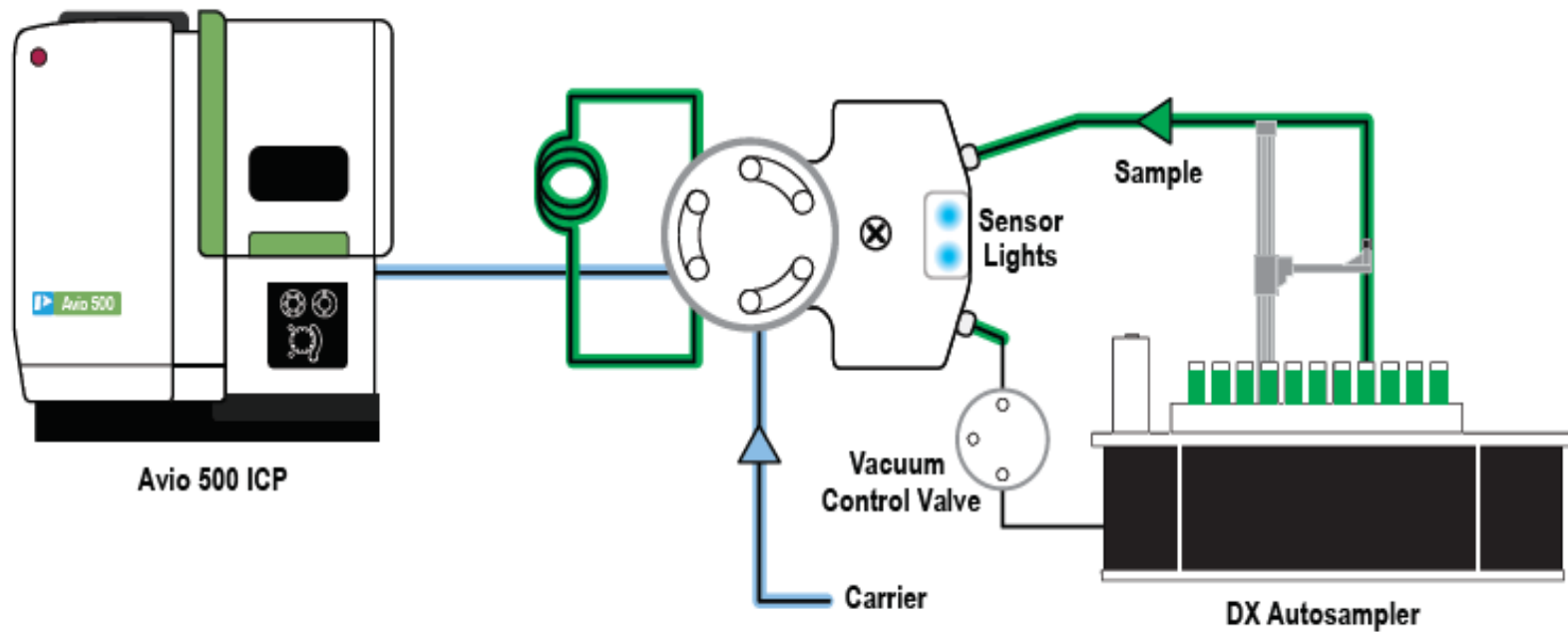


\* System using 1.5 mL loop size



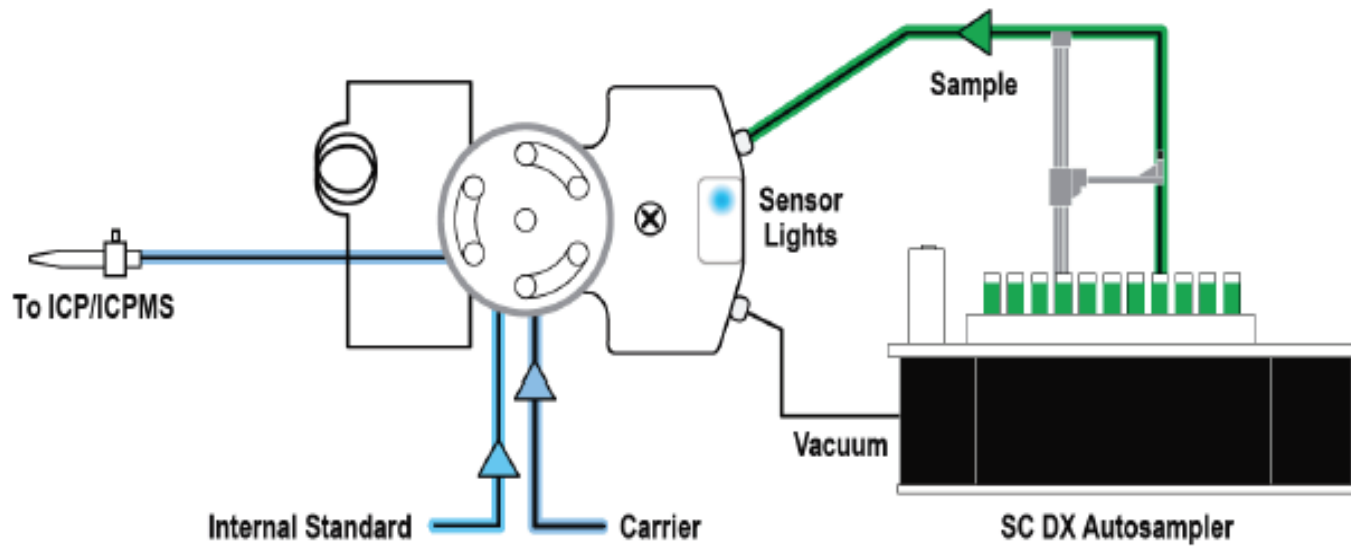


# SampleSense Enviro Flow Diagram



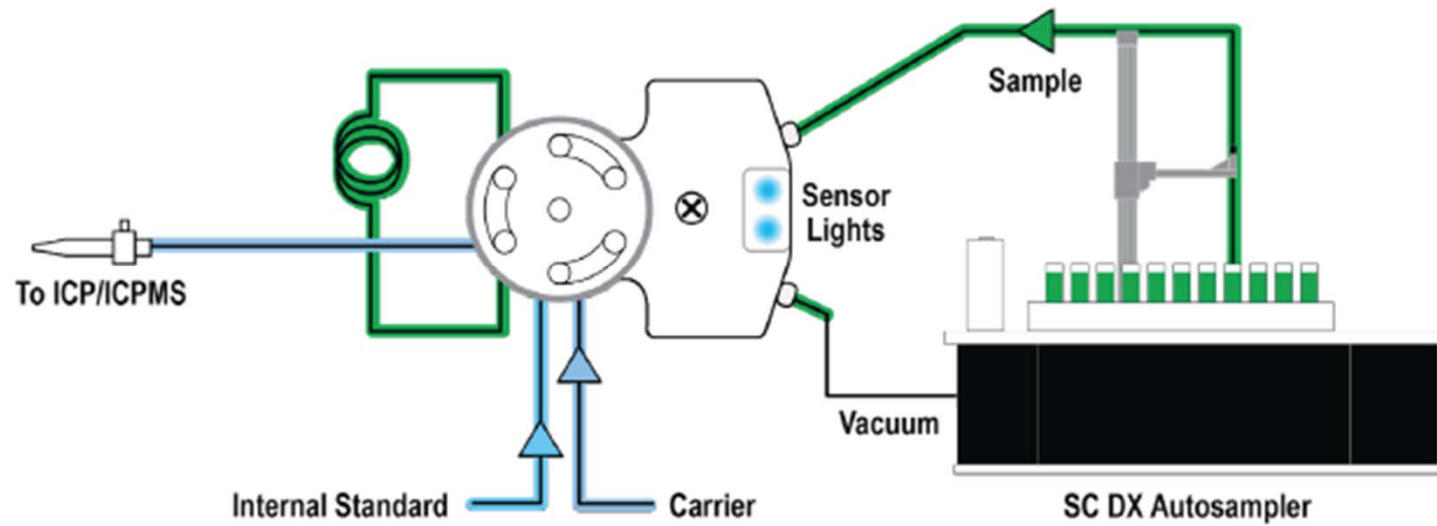
# Step 1:

## Initiate Sample Loading - Inlet Sensor Activated



## Step 2:

### Sample Loading Complete - Exit Sensor Activated

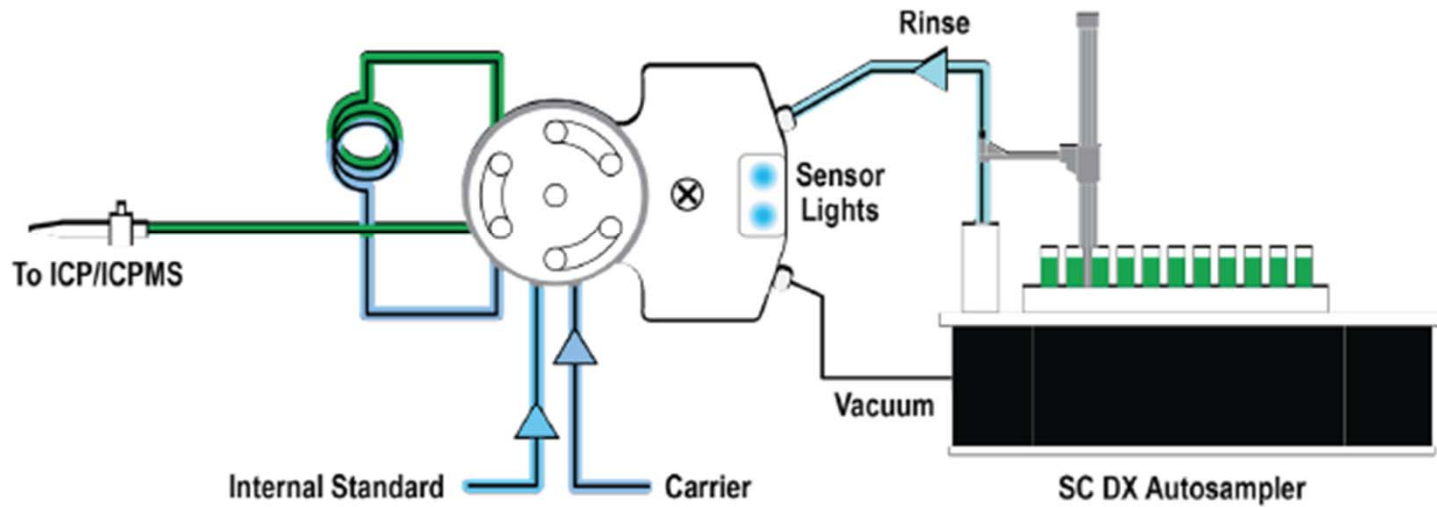


*When both sensors are triggered, vacuum valve closes and then sample valve switches to inject.*

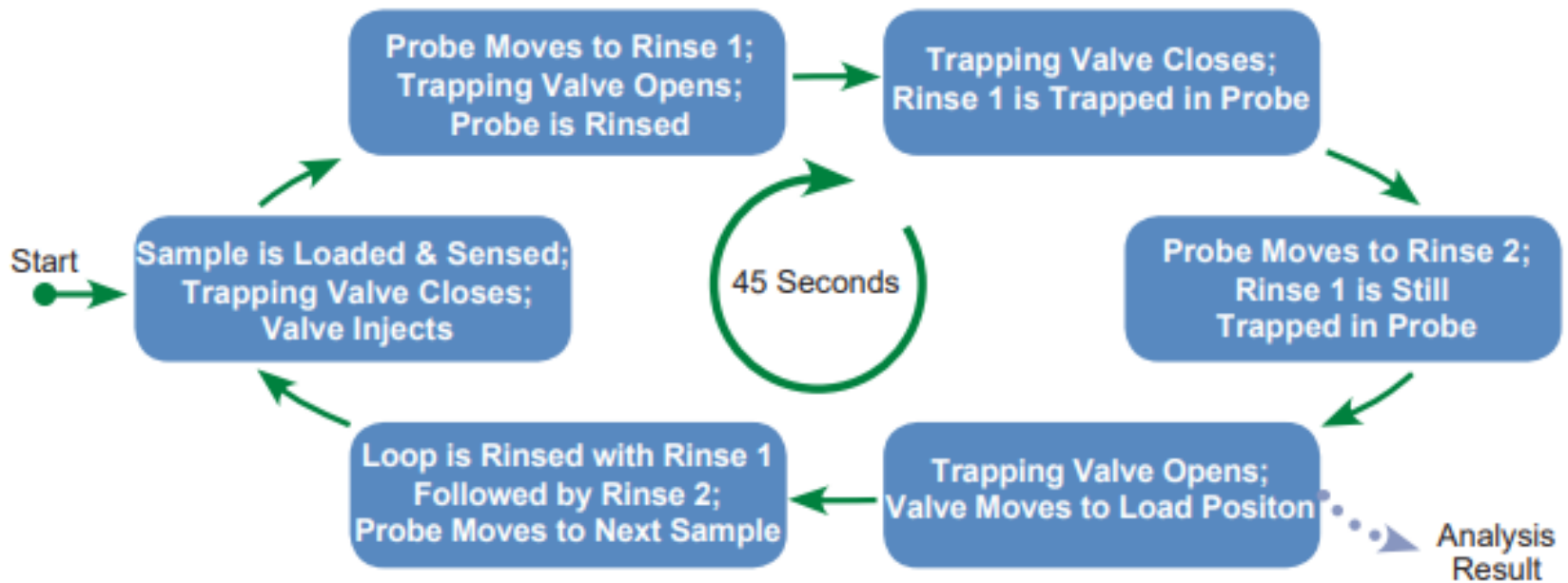


## Step 3:

### Sample Analysis Automatically Triggered by SampleSense



# SampleSense Enviro Analytical Cycle



# Experimental: Instrument Setup & Method conditions

**Table 1.** Instrument analysis settings.

Parameter	Value
Nebulizer	PFA ST 3-90
Spray Chamber	Unbaffled Quartz Cyclonic
Sample Flow Rate	1 mL/min
RF Power	1500 W
Torch/Injector	ESI One-piece Quartz Ziptorch with 2 mm id injector
Argon Humidifier	<i>pergo</i> 500
Nebulizer Gas Flow	0.6 L/min
Auxiliary Gas Flow	0.2 L/min
Plasma Gas Flow	8 L/min
Sample Uptake Tubing	Black/Black PVC (0.76 mm id), Flared
Drain Tubing	Grey/Grey Santoprene (1.14 mm id)
Integration Time	0.2-5 s
Replicates	3



- Method settings use standard analytical conditions (Analytes, replicates, sample flow, etc.)
- Method uses 3 replicates
- Uses < 2.5 mL sample/injection
- Probe washout and loop rinse are part of FAST method



## Example ICP-OES Time Savings (U.S. EPA Method 200.7)

### Standard autosampler

- 3:12 per sample

Sample Id	Acquisition Time
Blank	8/22/2018 12:16:46 PM
Cal-1	8/22/2018 12:20:32 PM
Cal-2	8/22/2018 12:24:18 PM
Cal-3	8/22/2018 12:28:06 PM
Cal-4	8/22/2018 12:31:18 PM
Cal-5	8/22/2018 12:34:31 PM

### Standard Valving System

- 1:16 per sample

Sample Id	Acquisition Time
BLANK	8/22/2018 5:27:48 PM
Cal-1	8/22/2018 5:29:45 PM
Cal-2	8/22/2018 5:31:40 PM
Cal-3	8/22/2018 5:33:00 PM
Cal-4	8/22/2018 5:34:16 PM
Cal-5	8/22/2018 5:35:32 PM

### SampleSense Enviro System

- 0:45 per sample

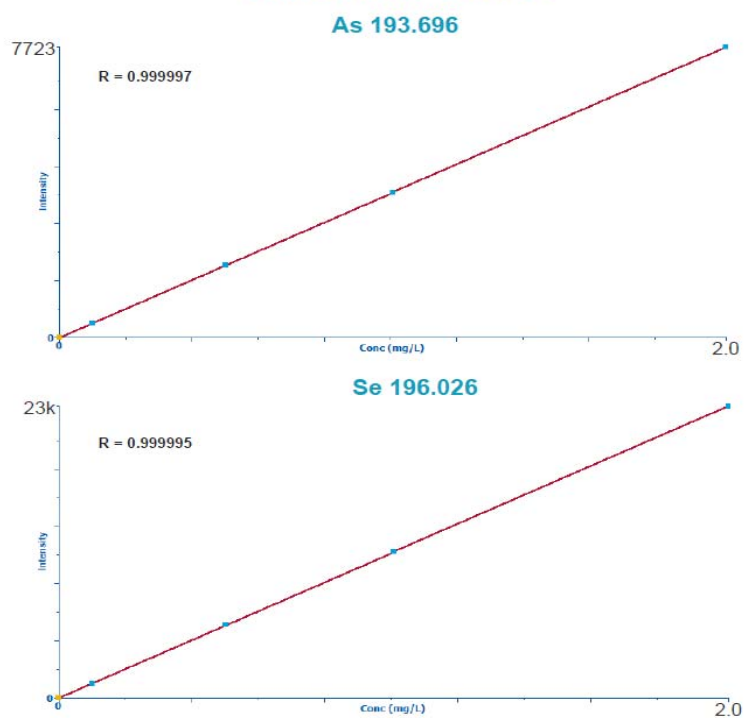
Sample Id	Acquisition Time
Blank	1/22/2021 10:10:13 AM
Cal-1	1/22/2021 10:10:56 AM
Cal-2	1/22/2021 10:11:38 AM
Cal-3	1/22/2021 10:12:23 AM
Cal-4	1/22/2021 10:13:07 AM
LRB	1/22/2021 10:13:53 AM
IPC	1/22/2021 10:14:38 AM
QCS	1/22/2021 10:15:24 AM
LFB	1/22/2021 10:16:07 AM
Rinse	1/22/2021 10:16:49 AM
Rinse	1/22/2021 10:17:34 AM
TMDW-500	1/22/2021 10:18:18 AM



# A Few Examples of Calibrations



## Calibration Curves



**Table 4.** Detection Limits and Calibration Linearity. Summary of estimated detection limits, calculated as 3\*standard deviation of 10 blank measurements, and calibration linearity, shown by the calibration coefficient, R.

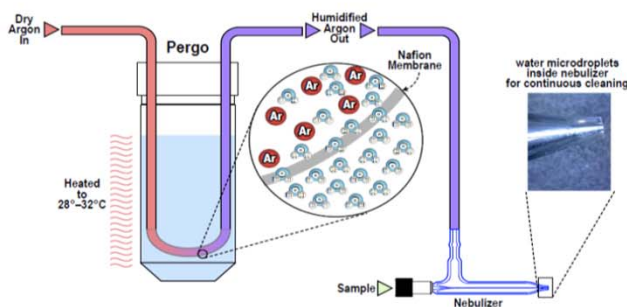
Element	Detection Limit	Correlation Coefficient (R)
Ag	1.5 ppb	0.999834
As	5.7 ppb	0.999997
Be	1.5 ppb	0.999956
Cd	0.9 ppb	0.999905
Cr	1.1 ppb	0.999913
Mo	3.5 ppb	0.999979
Pb	1.5 ppb	0.999832
Se	5.6 ppb	0.999995
Tl	3.4 ppb	0.999732
Zn	1.7 ppb	0.999954



# Pergo - Argon Humidifier

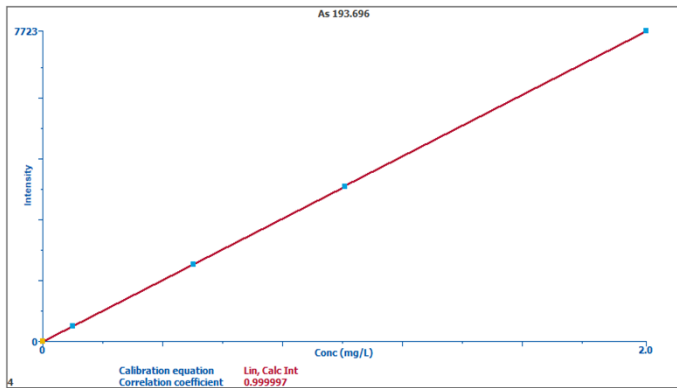
## Benefits

- Dissolves salt crystals at nebulizer tip
- Reduces long term drift
- Improves stability
- Improves detection limits

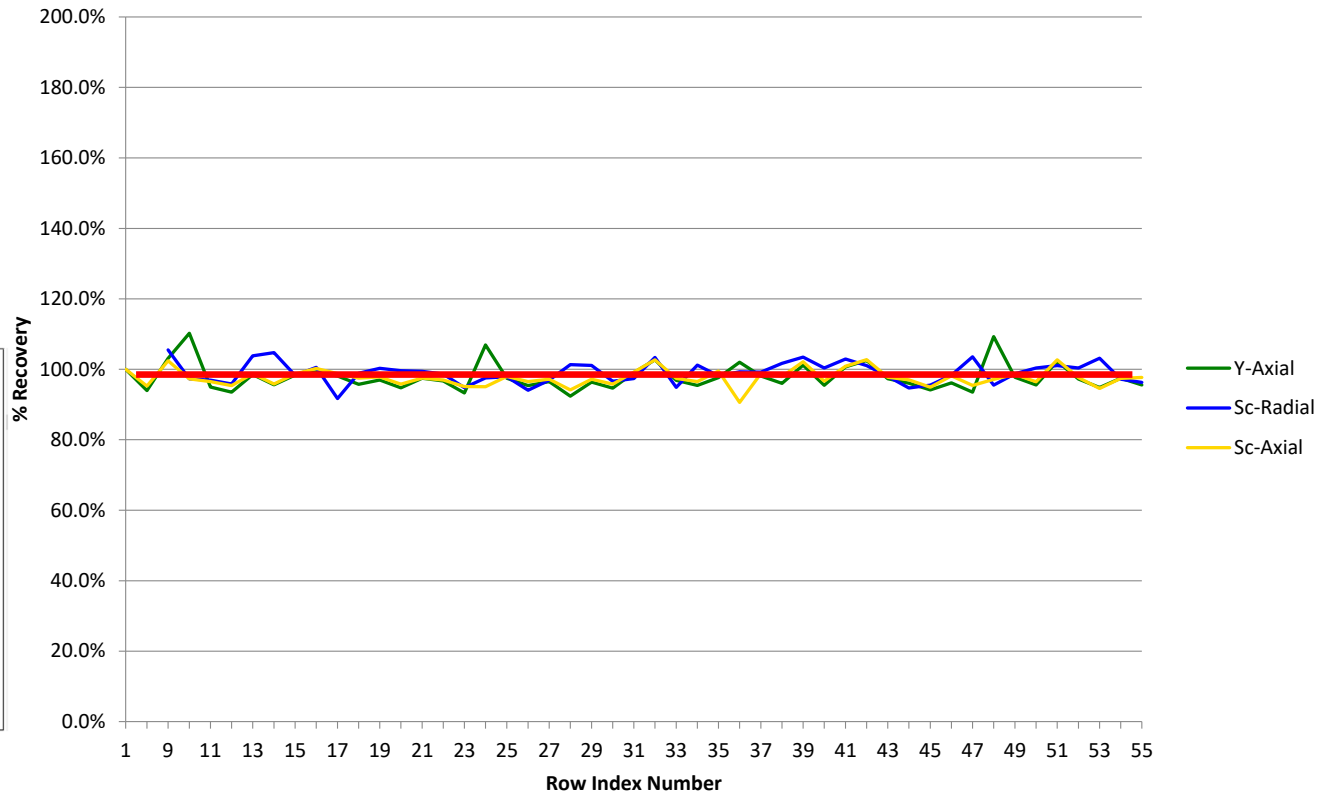


# Experimental: Calibration and Data

- Calibration for As 193
- IS plot over sample run

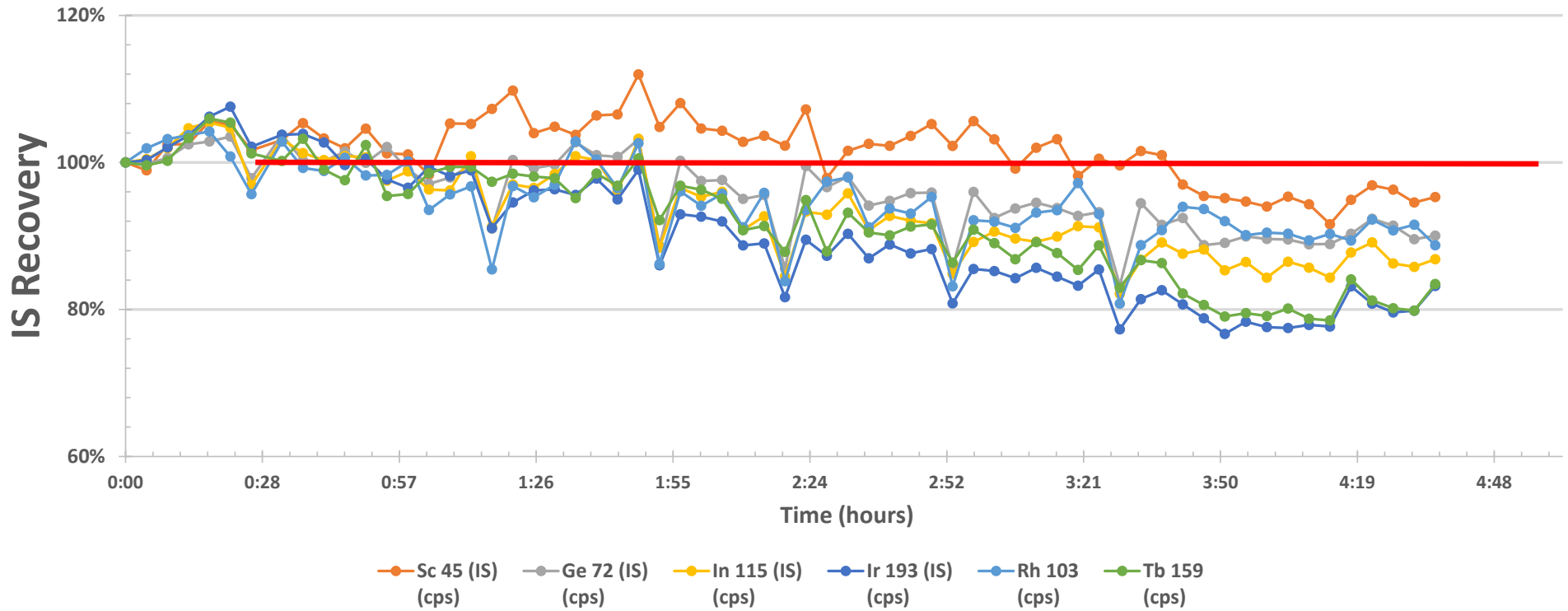


0.999997



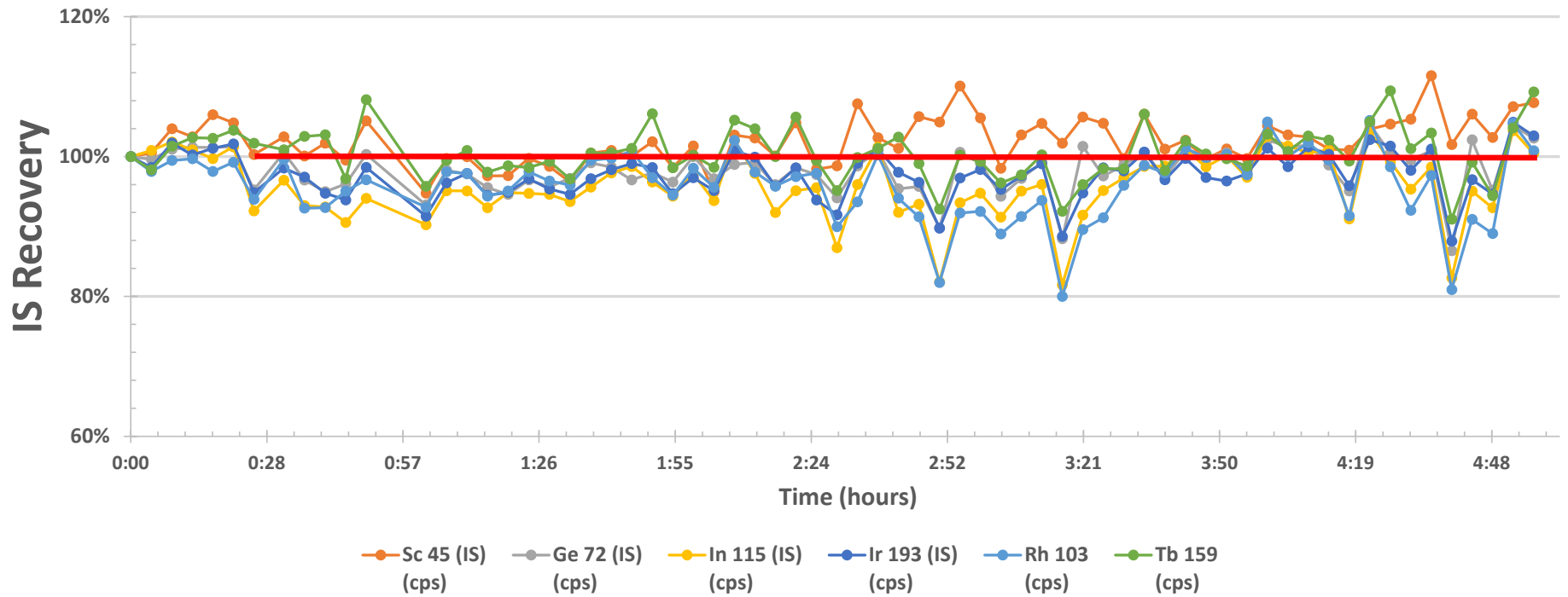
# Internal Standard Recovery

## Drift without pergo



# Internal Standard Recovery

Stable Signal with pergo





# Benefits Summary



## Viscosity Differences

One valve, one loop and one method for multiple sample types. SampleSense accounts for viscosity and automatically adjusts timing.



## Time Savings

Operator time for sample introduction method development is eliminated. Just run samples.



## Sample Savings

The sample is injected at precisely the right time. Every time. Even for small samples.



## Error Notification

Incomplete sample loading or bubbles in the sample are detected and logged.

# Questions ??

SampleSense Enviro – provides fastest, most economical, most intelligent analysis



Unsensed Samples					
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