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## Automated Inline Preparation of Engine Coolant Samples for High Throughput ICP Analysis using prepFAST

### Abstract

prepFAST X highly automates ICP engine coolant analyses through high-speed in-line syringe-driven dilution. prepFAST X includes four inert syringes coupled to a SampleSense X valve having integrated sample-sensing technology. The system also includes a DXCi autocorrecting autosampler with position detection and autocorrection capability. After sensing and preparing each sample, prepFAST X triggers the ICP read to complete the analysis. Automated analysis steps include:

- **Autodilution.** prepFAST X automatically dilutes coolant samples and spikes them with internal standard.
- **Autocalibration.** prepFAST X automatically calibrates the ICP instrument by inline dilution of a stock standard.
- **Automated Sample Detection.** SampleSense valve technology optically senses samples, accounting for the range of sample viscosities, minimizing sample consumption, detecting missing samples, and eliminating method timing variables.

prepFAST X dilutes and prepares coolant samples right at the moment of analysis, integrating perfectly and complementing the ICP instrument. prepFAST X provides improved laboratory efficiency and high analytical performance. This note describes the prepFAST X analytical procedure and ICP operating conditions, demonstrates linearity of autocalibration, shows stable results for 200 samples in under 8 hours, and sample-to-sample rinseout factors >1000x for all 14 monitored elements in coolant samples.



## Introduction

Varying elemental concentrations in coolant samples indicate potential breakdown of not only the coolant system but also the engine components that the coolant system is designed to regulate. ICP coolant analysis can determine the coolant condition over time as additive elemental concentrations change. Coolant analysis typically makes up approximately 10% of the overall ICP sample volume for many in-

service laboratories. Consequently, laboratories analyzing hundreds of oil samples each day must switch to aqueous coolant ICP analysis. This presents sample preparation and analysis challenges that are vastly different from oil analysis. The prepFAST X Coolant Analysis System automates the coolant analysis process and enables laboratories to more effectively and confidently serve their customers.

## prepFAST X - Fully Automated Single Valve Sample Preparation for ICP Analysis

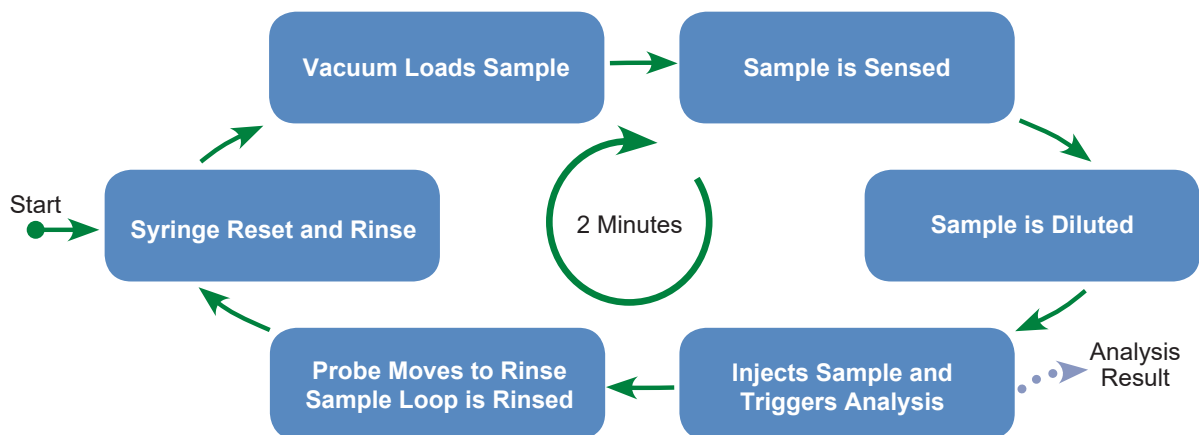
prepFAST X is a sample preparation system consisting of an intelligent autosampler coupled with a syringe pump module and a SampleSense valve integrated in a DXi. The system fully automates laboratory dilutions while providing high sample throughput. It offers high-precision inline autodilution up to 400x and autocalibration from one or more stock standards.

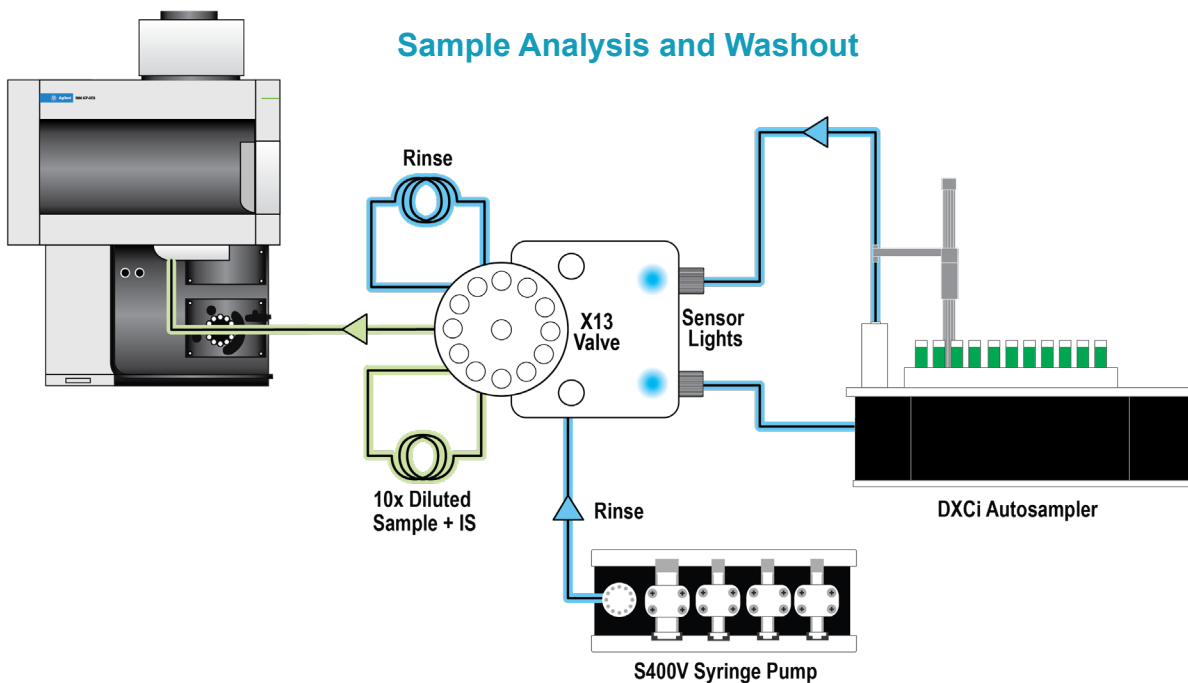
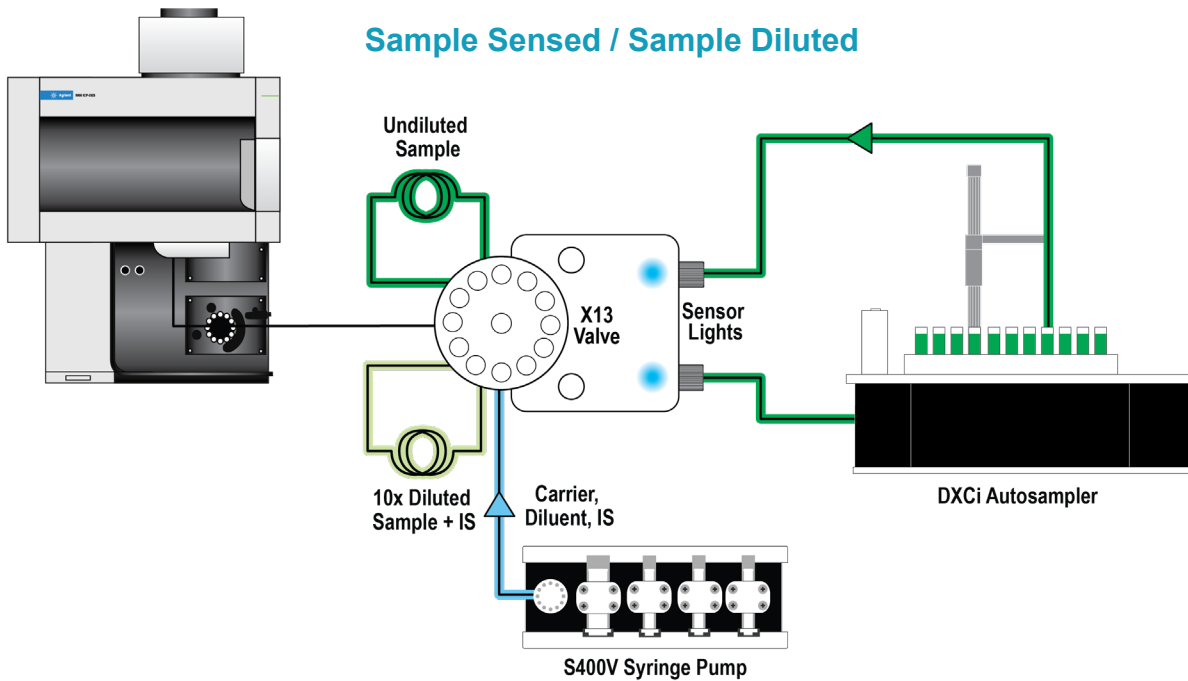
prepFAST X combines an autocorrecting DXCi autosampler with an inert injection valve featuring integrated optical sensors that automatically detect both the arrival of a sample in the valve and the subsequent sample loop fill completion. The undiluted sample is quickly vacuum loaded, automatically adjusting for varying sample viscosities. Next the sample is diluted inline and transferred into a second loop. The prepFAST X injects the diluted sample into the ICP where analysis is automatically triggered in a tightly timed analytical sequence free of predetermined delay timings.

This technology provides many benefits including:

- Eliminates all sample uptake method development – no uptake delays required
- Optimizes loading conditions for each sample matrix with no method adjustments
- Allows sample loop sizes to be changed without needing to alter method settings
- Automatically compensates for drift caused by kinked lines or partial blockages
- Provides positive confirmation of sample loading – if a sample fails to load for any reason, the failed sample is logged and the user is alerted.
- Automatically goes to the correct sample location every time – even if the autosampler probe is accidentally obstructed.

### SampleSense prepFAST X Analytical Cycle





**Figure 1.** prepFAST X Coolants Flow Diagram: (1) Sample is loaded into loading loop and sensed by SampleSense valve. Sample is then pushed into sample loop and diluted inline with internal standard and diluent. (2) Diluted sample is pushed into the ICP for analysis. Once analysis is complete, both the loading loop and sample loop are rinsed with rinse solution.

## Instrumentation

All samples were analyzed using prepFAST X in combination with an Agilent 5900 ICP-OES system.

### Features

- 2 minutes a sample
- Automatic sensing, injection and triggering of the ICP-OES analysis
- Detection and reporting of missing or empty sample tubes as “unsensed” samples
- Autocalibration from single stock standard
- Autodilution up to 400x

## Sample Preparation

Coolant samples were inline diluted 10-fold using prepFAST X with 0.5% nitric acid and 40ppm Yttrium internal standard. The autosampler rinse station was supplied with deionized water to rinse the probe and uptake line between samples.

## Analytes and Wavelengths Measured

Analyte	Wavelength (nm)	Analyte	Wavelength (nm)
Y (IS)	371.029	Mo	201.512
Al	396.152	Na	589.592
B	249.772	P	213.618
Ca	315.887	Pb	220.353
Cu	327.395	Si	251.611
Fe	234.350	Sn	189.925
K	766.491	Zn	334.502
Mg	279.800		

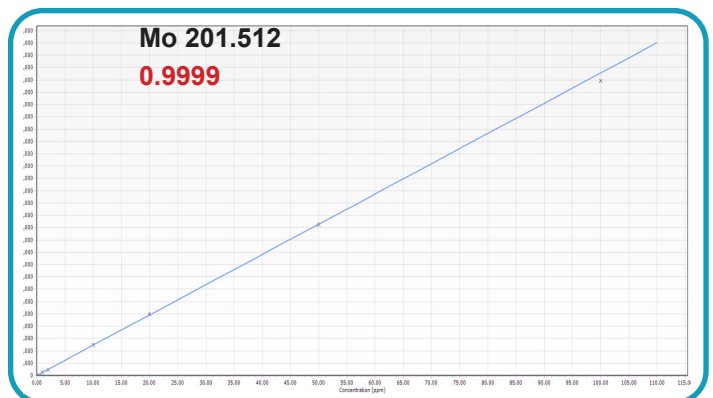
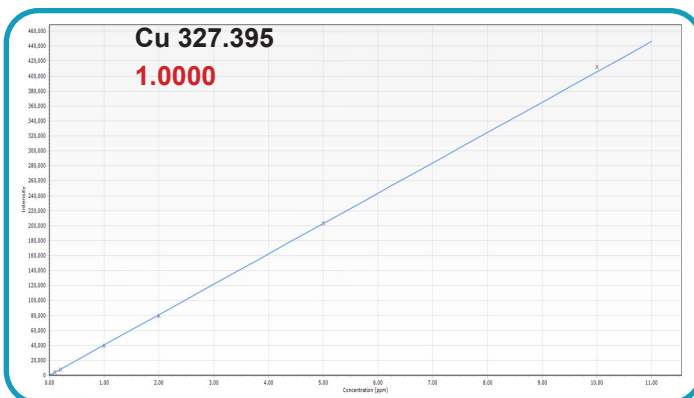
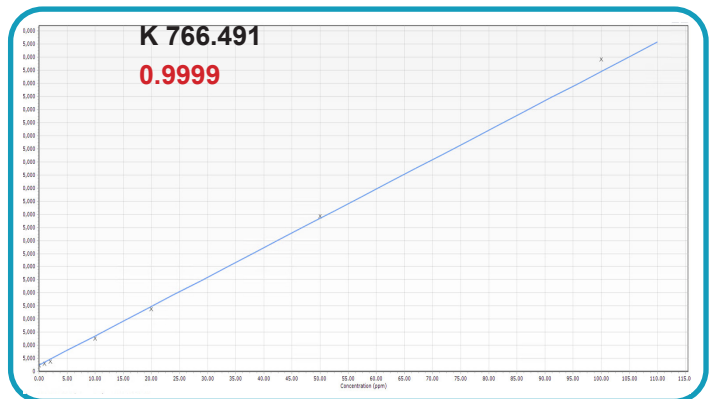
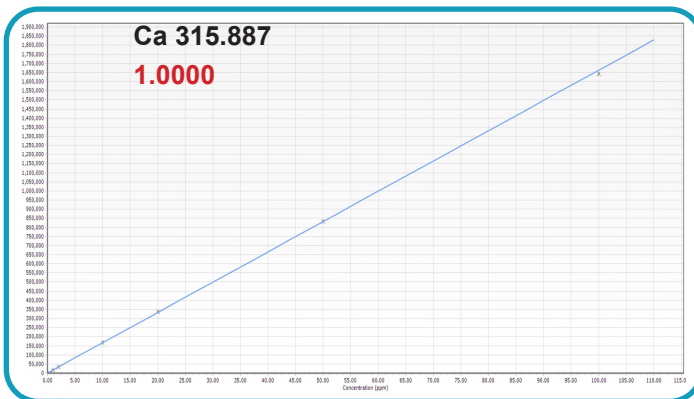
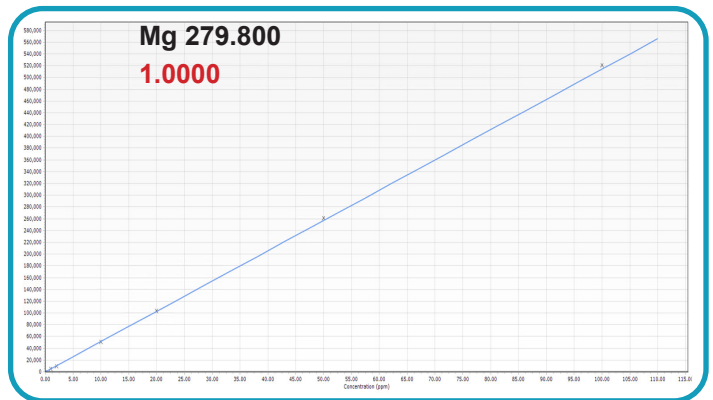
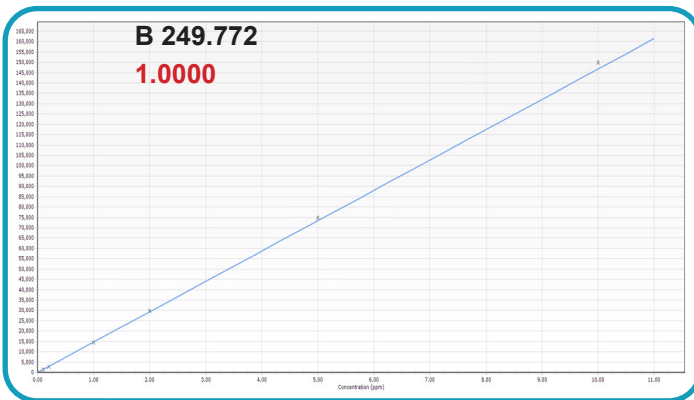
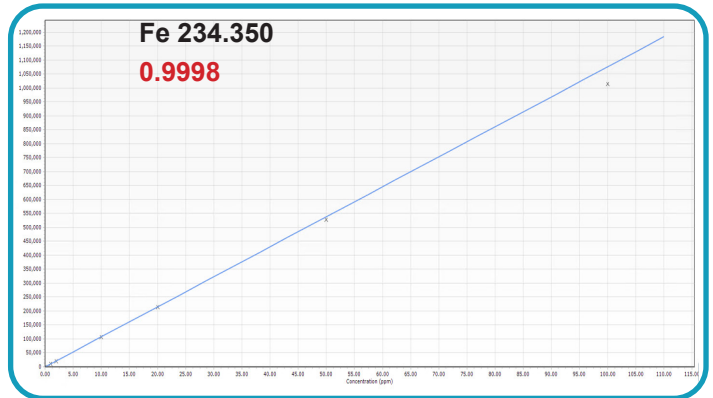
## Instrument settings

Parameter	Value
ICP RF Power (W)	1200
Nebulizer Gas Flow (L/min)	0.45
Auxillary Gas Flow (L/min)	1.0
Plasma Gas Flow (L/min)	12
Nebulizer	MEINHARD® V-Groove Nebulizer
Spray Chamber	Baffled Dual Pass Glass Zip Chamber
Torch	Quartz Torch
Injector	2.0 mm ID Demountable Quartz
Viewing Mode	Radial

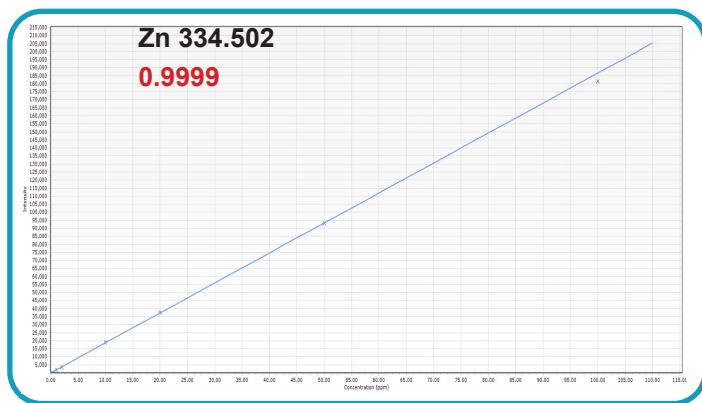
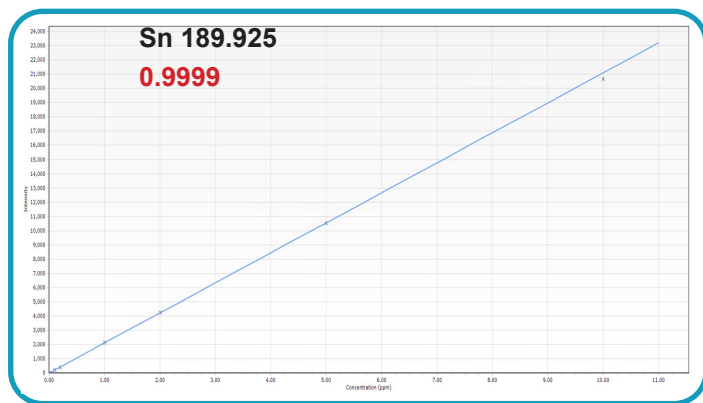
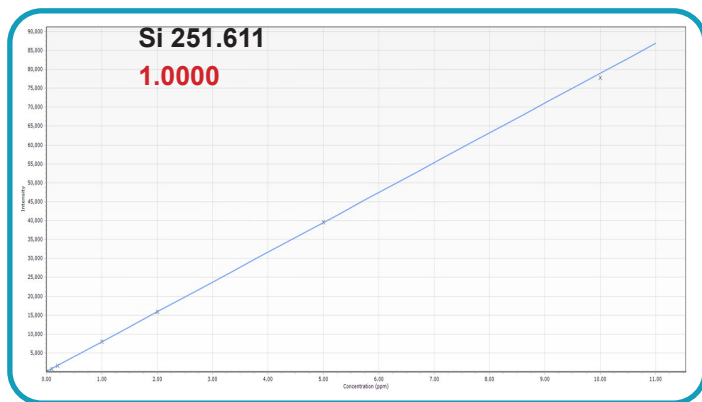
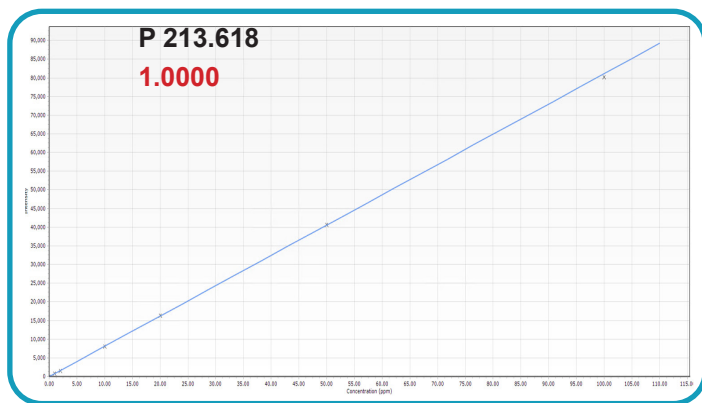
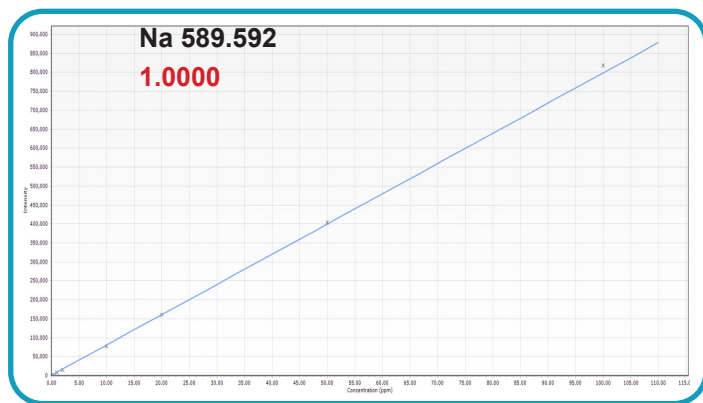


**Figure 3.** 4DXCi prepFAST X on the Agilent 5900 ICP-OES (PN: 4PFSS-X-88). Model options include 2DXCi, 4DXCi, 8DXCi, and 14DXCi to support various sample capacity requirements.

# Calibration Curves for All Analytes



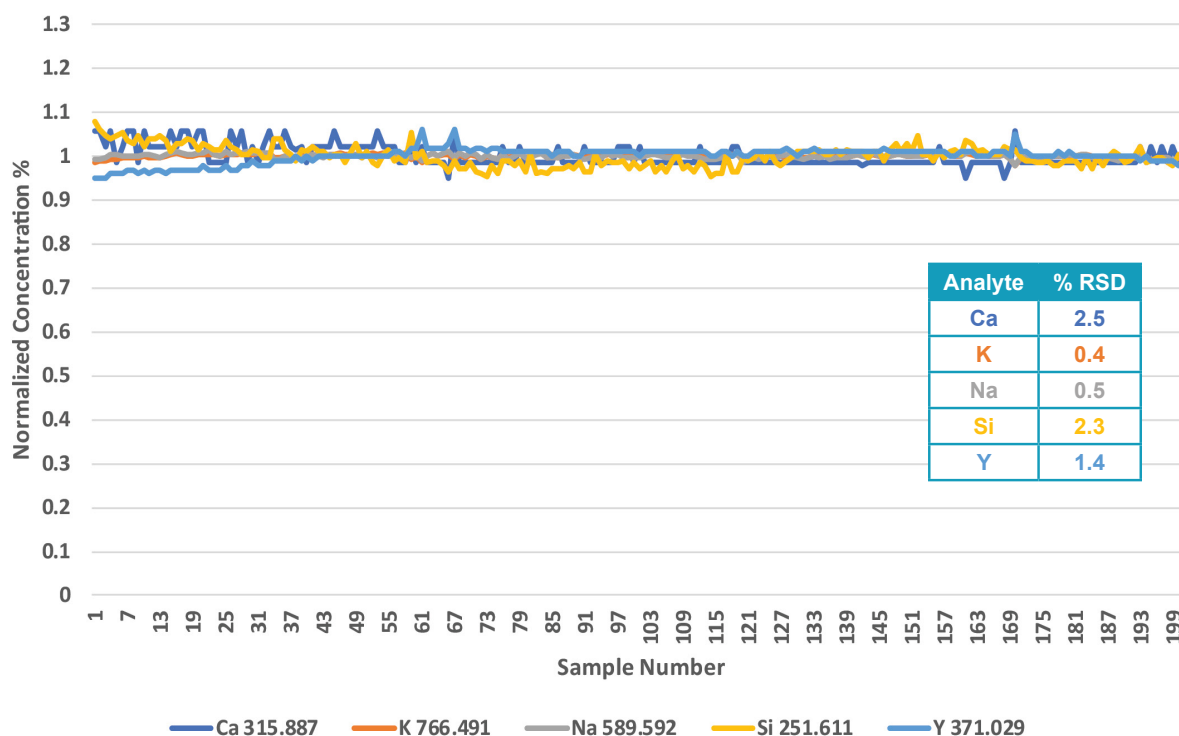
## Calibration Curves for All Analytes



## Results

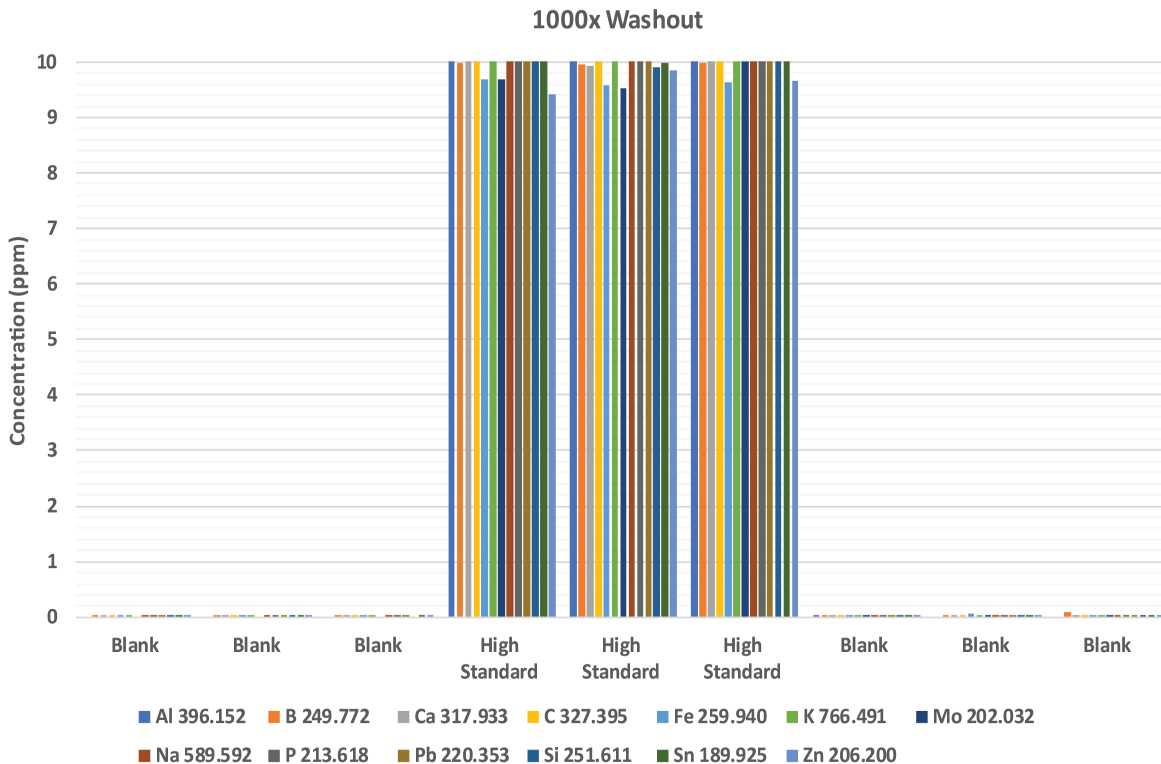
The system was autocalibrated using a single standard made in 0.5% HNO<sub>3</sub> at 100 ppm for all elements except for B, Pb, Si, and Sn, which were prepared at 10 ppm. prepFAST X generated linear calibration curves (Figure 4) for all elements using multiple dilution factors of 1x, 2x, 5x, 10x, 50x, and 100x. The system then autodiluted and analyzed 200 samples in just under 8 hours. The stability was excellent, with RSDs for all elements in sample under 3% (see Figure 5). The washout in the blank immediately after analyzing three undiluted standard samples was >1000x, showing suitable performance for new and used coolant analysis (see Figure 6).

### prepFAST X Element Stability over 7 Hours and 45 Minutes for 200 Samples



**Figure 5.** New coolant samples autodiluted by 10x with prepFAST X and analyzed by ICP for 15 elements. 200 coolant samples diluted and analyzed in 7 hours and 45 minutes. Stability of four elements with highest concentrations shown above; lower concentrations exhibited similar excellent long-term stability.

## 1000x Washout After High Calibration Standard



**Figure 6.** 1000x washout for all elements. Three blanks analyzed, then three high standards, followed immediately by three blanks.

## Conclusion

prepFAST X completely optimizes the analysis of new and used coolants for laboratories using a sample preparation system consisting of a syringe pump module, intelligent autocorrecting autosampler, and an optically sensing SampleSense valve for sample injection. The use of a syringe pump module allows for both autocalibration from a single standard as well as inline autodilution immediately before analysis. The SampleSense valve eliminates timing parameters and method adjustments needed to account for varying sample viscosity. The optical sensors in the SampleSense valve also provide essential information to the laboratory through positive confirmation that the sample was loaded into the valve for analysis and by logging any missed samples.

prepFAST X is a highly effective sample preparation and introduction system that improves analytical efficiency in a production laboratory environment, providing quick and accurate determination of elements in all coolant fluids. Significant operational savings are realized from the prepFAST X by removing the need for laboratory staff to clean volumetric labware, reducing laboratory waste streams, eliminating the preparation of daily calibration standards, and automatically diluting all incoming coolant samples.



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