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Automated Inline Preparation of Engine Coolant Samples for High Throughput ICP Analysis using prep*FAST* X

Abstract

prep*FAST* X highly automates ICP engine coolant analyses through high-speed in-line syringe-driven dilution. prep*FAST* 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, prep*FAST* X triggers the ICP read to complete the analysis. Automated analysis steps include:

- **Autodilution.** prep*FAST* X automatically dilutes coolant samples and spikes them with internal standard.
- **Autocalibration.** prep*FAST* 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.

prep*FAST* X dilutes and prepares coolant samples right at the moment of analysis, integrating perfectly and complementing the ICP instrument. prep*FAST* X provides improved laboratory efficiency and high analytical performance. This note describes the prep*FAST* X analytical procedure and ICP operating conditions, demonstrates linearity of autocalibration, shows stable results for >250 samples over > 9 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 prep*FAST* X Coolant Analysis System automates the coolant analysis process and enables laboratories to more effectively and confidently serve their customers.

prep*FAST* X - Fully Automated Single Valve Sample Preparation for ICP Analysis

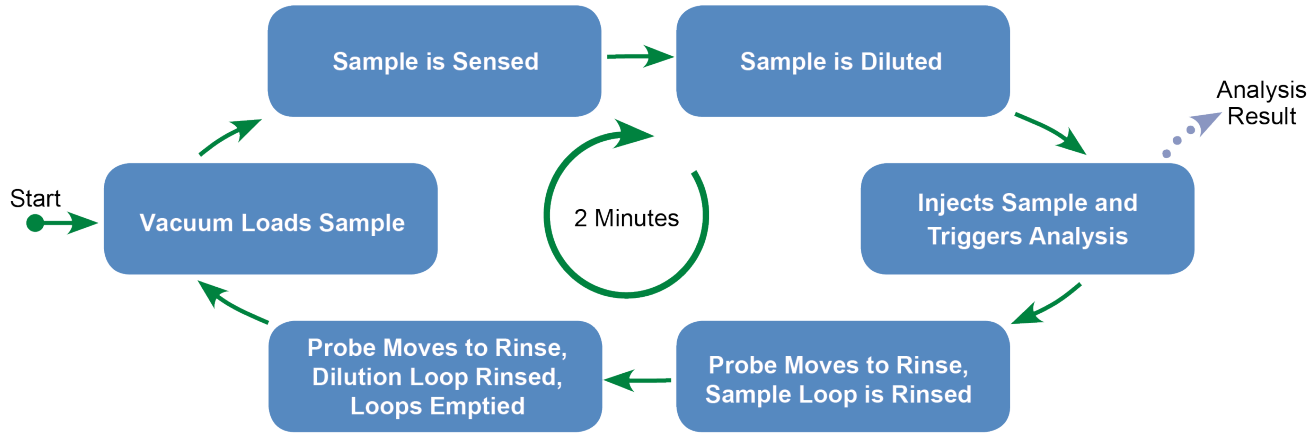
prep*FAST* X is a sample preparation system consisting of an intelligent autosampler coupled with a syringe pump module and a SampleSense valve integrated in the sample introduction FlexRack. The system fully automates laboratory dilutions while providing high sample throughput. It offers high-precision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

prep*FAST* 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 prep*FAST* 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.

prepFAST X Analytical Cycle



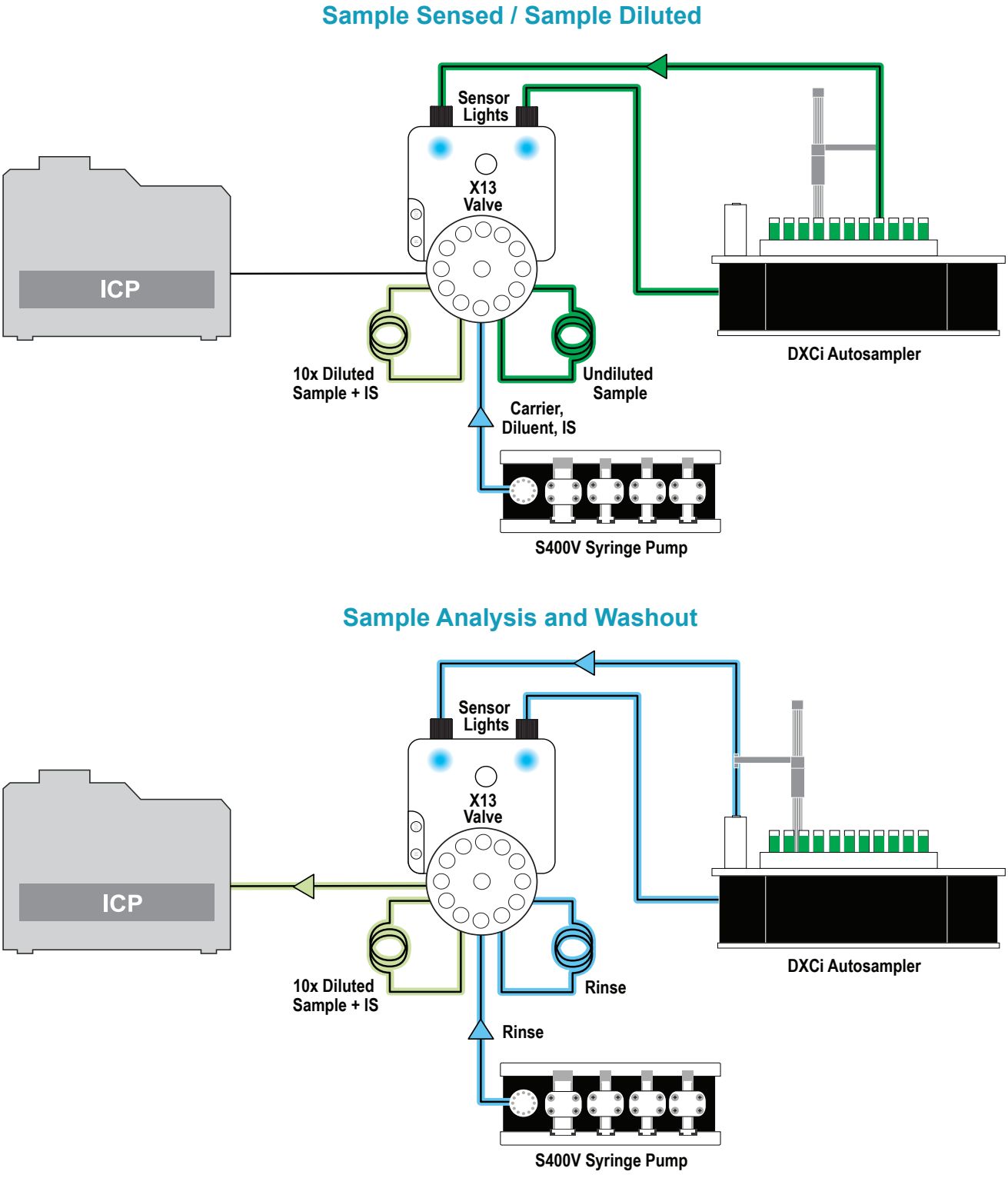


Figure 1. prep*FAST* X Coolants Flow Diagram: (1) Sample is loaded into loading loop and sensed by SampleSense valve. Sample is then pushed into sample loop and diluted inline with internal standard and diluent. (2) Diluted sample is pushed into the ICP for analysis. Once analysis is complete, both the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

Instrumentation

All samples were analyzed using prep*FAST* X in combination with a Thermo Fisher Scientific™ iCAP™ PRO XPS 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 prep*FAST* X with deionized water containing 10ppm 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

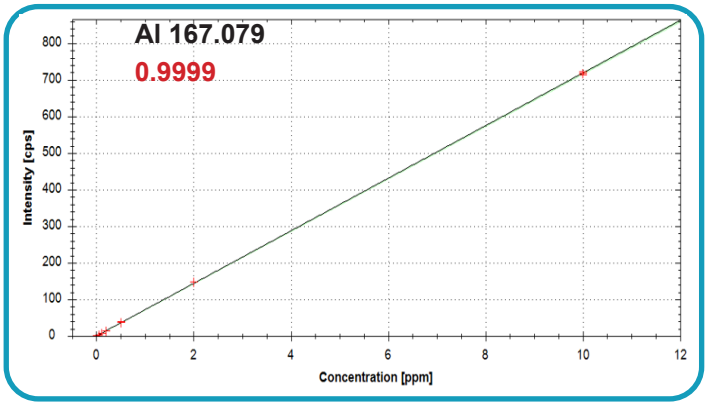
Analyte	Wavelength (nm)	Analyte	Wavelength (nm)
Co (IS)	228.616	Mo	202.030
Al	167.079	Na	588.995
B	249.773	P	177.495
Ca	393.366	Pb	220.353
Cu	324.754	Si	251.611
Fe	259.940	Sn	189.989
K	766.490	Zn	206.200
Mg	279.553		

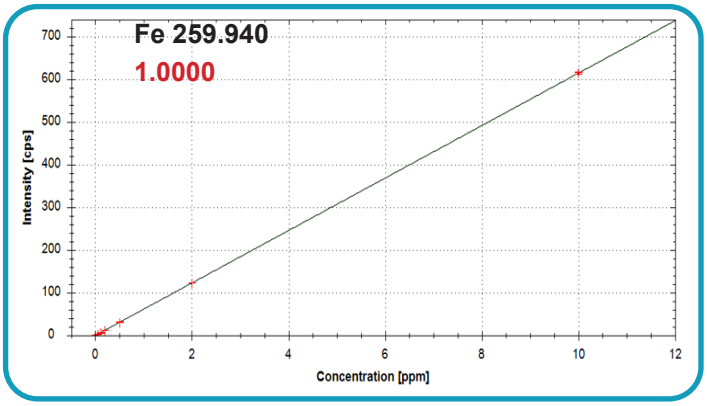
Instrument settings

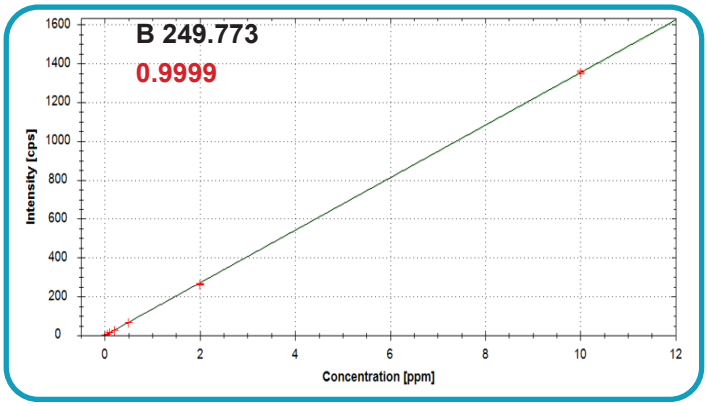
Parameter	Value
ICP RF Power (W)	1150
Nebulizer Gas Flow (L/min)	0.45
Auxillary Gas Flow (L/min)	1.0
Plasma Gas Flow (L/min)	12
Nebulizer	MEINHARD® V-Groove Nebulizer
Spray Chamber	Baffled Dual Pass Glass Zip Chamber
Torch	Quartz Torch
Injector	2.0 mm ID Demountable Quartz
Viewing Mode	Radial

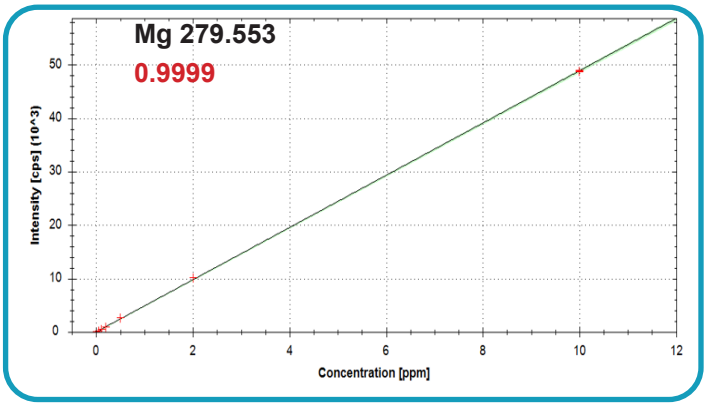
Figure 3. prep*FAST* X with FlexRack. Autosampler model options include 2DXCi, 4DXCi, 8DXCi, and 14DXCi to support various sample capacity requirements.

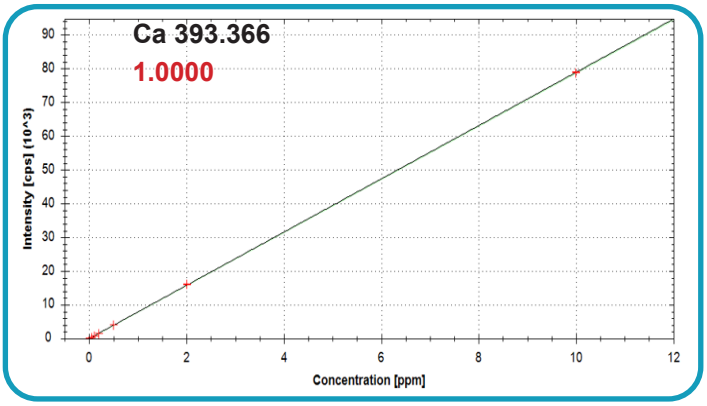
Calibration Curves for All Analytes

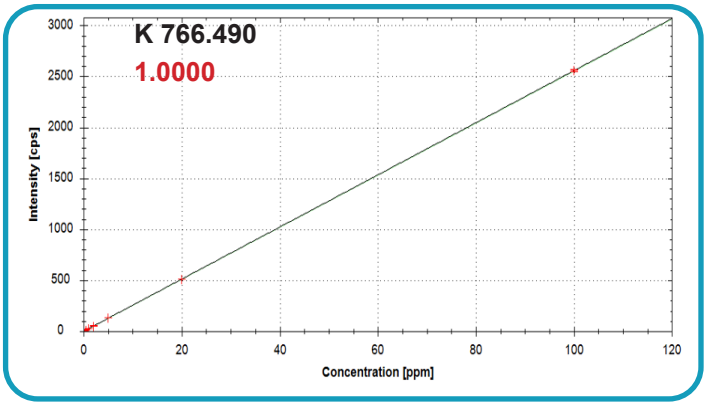


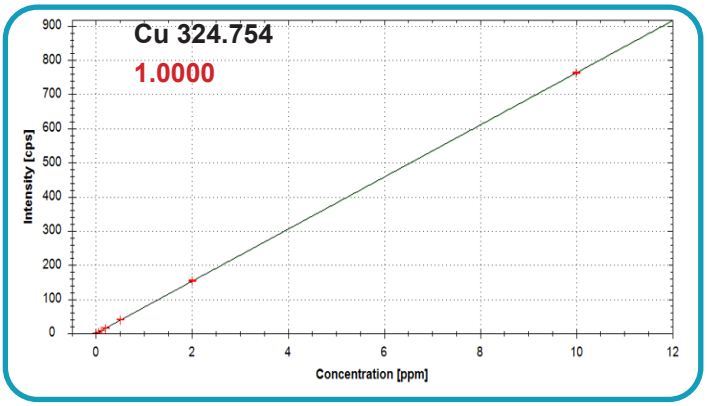


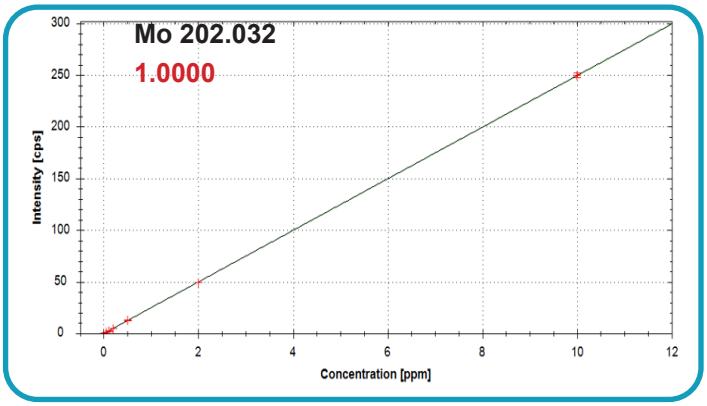




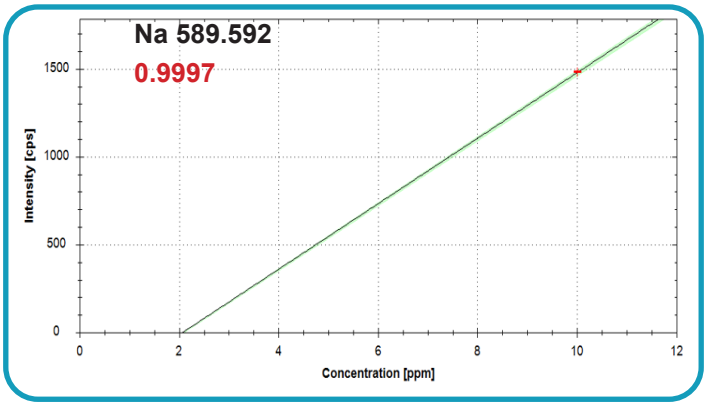


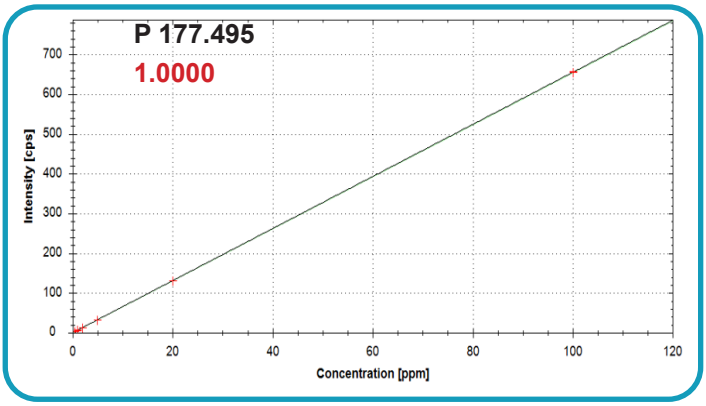


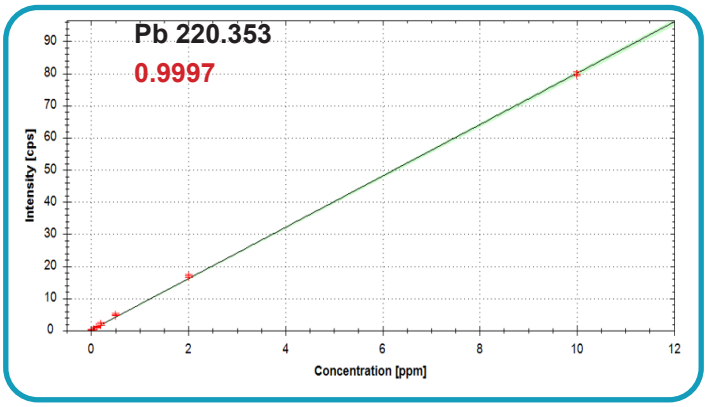


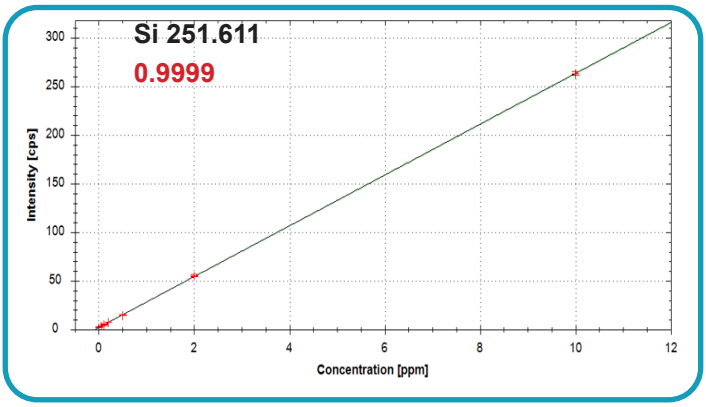


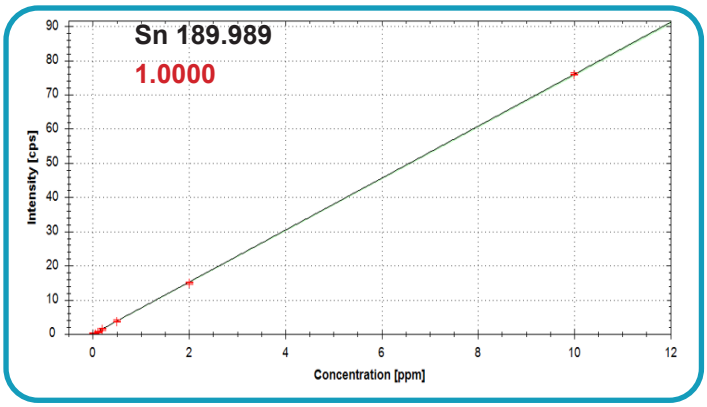
Calibration Curves for All Analytes











Results

The system was autocalibrated using a single standard made in 0.5% HNO_3 at 10 ppm for all elements except for P and K, which were prepared at 100 ppm. prep*FAST* X generated linear calibration curves (Figure 4) for all elements using multiple dilution factors of 1x, 5x, 20x, 50x, 100x, and 200x. The system then autodiluted and analyzed 250 samples in just over 9 hours. The stability was excellent, with RSDs for all elements in sample under 2% (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).

prep*FAST* X Element Stability over 9 Hours and 30 Minutes for 250 Samples

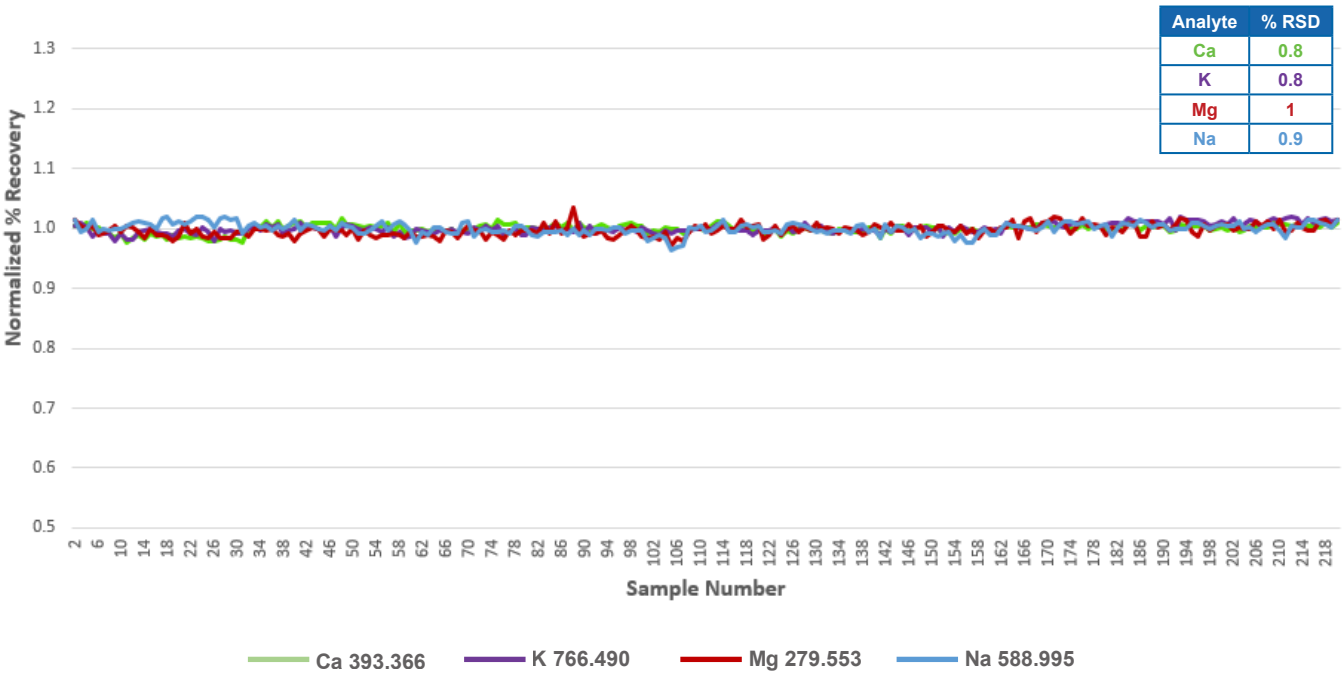


Figure 5. New coolant samples autodiluted by 10x with prep*FAST* X and analyzed by ICP for 15 elements. 250 coolant samples diluted and analyzed in 9 hours and 30 minutes. Stability of four elements with highest concentrations shown above; lower concentrations exhibited similar excellent long term stability.

1000x Washout After High Calibration Standard

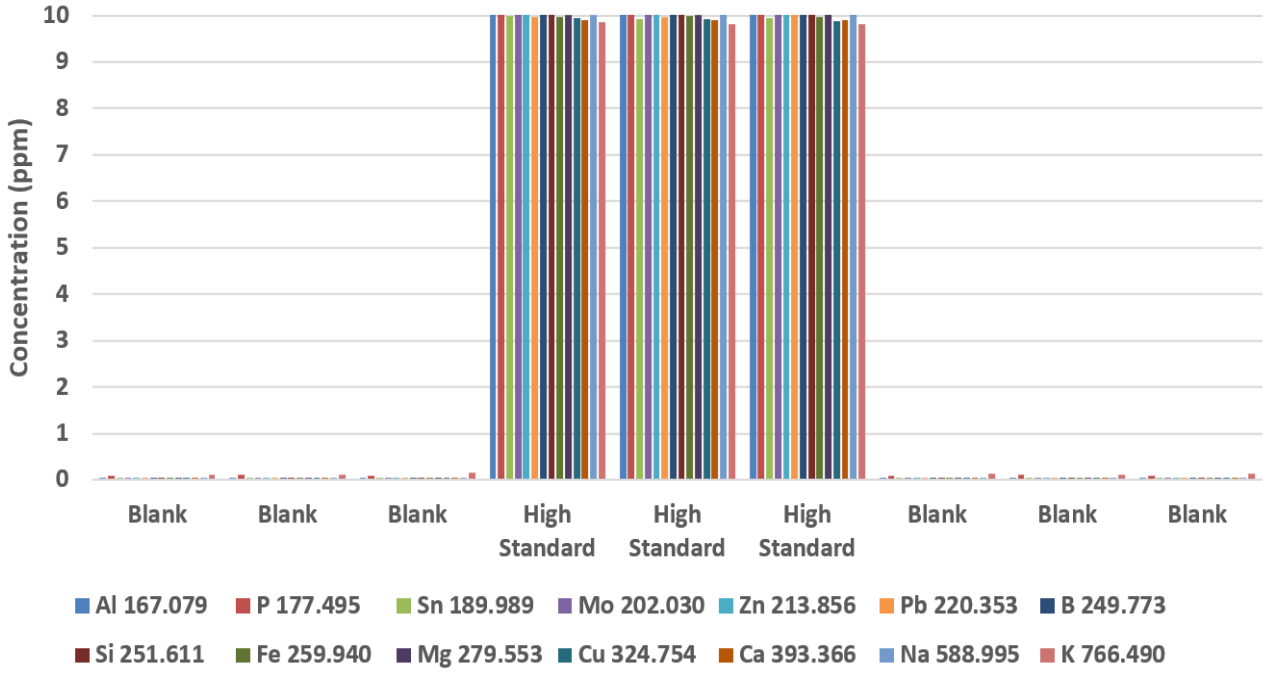


Figure 6. 1000x washout for all elements. Three blanks analyzed, then three high standards, followed immediately by three blanks.

Conclusion

prep*FAST* 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.

prep*FAST* 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 prep*FAST* 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.