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SampleSense Oil – Ultra-high Throughput Automation System for In-Service Fluids Analysis – 3 Samples per Minute

- Unprecedented speed
- Rapid gas infusion sample mixing homogenizes sample immediately before analysis
- SampleSense valve eliminates method timing and accounts for varying sample viscosities
- Extremely stable and robust
- Clean system from probe to injector

Abstract

The SampleSense Oil system is uniquely optimized for oil and lubricant sample analysis through three main aspects.

1) Gas infusion mixing allows for controlled homogenization of settled oil samples immediately prior to analysis.

2) SampleSense optical valve provides dynamic sample detection, which helps to ensure analytical integrity by automatically accounting for variable sample viscosities, detecting missed sample events, minimizing sample consumption, and eliminating method timing.

3) DXCi autocorrecting autosampler provides sample position confirmation in addition to high speeds and smooth movements designed to reduce oil dripping.

The SampleSense Oil system provides high throughput, controlled sample mixing, positive confirmation of sample introduction and sample position, autosampler autocorrection, compatibility with multiple vial sizes, and stable performance, all while achieving three samples per minute.

Introduction

Laboratories throughout the world analyze engine oil and lubricants for wear metals, ingress elements, and additives to determine when to perform necessary maintenance on heavy machinery used in construction and mining applications. The cost to maintain and repair these machines can be very high, especially if the equipment must be removed from production. To reduce maintenance costs, testing fluids associated with different machine components provides valuable insight into needed maintenance and helps diagnose problems before they lead to an expensive mechanical failure. The ASTM D5185 method for ICPOES is commonly used to analyze these types of samples. A fast, accurate sample introduction system to perform this method saves the time, money, and labor associated with costly repairs.

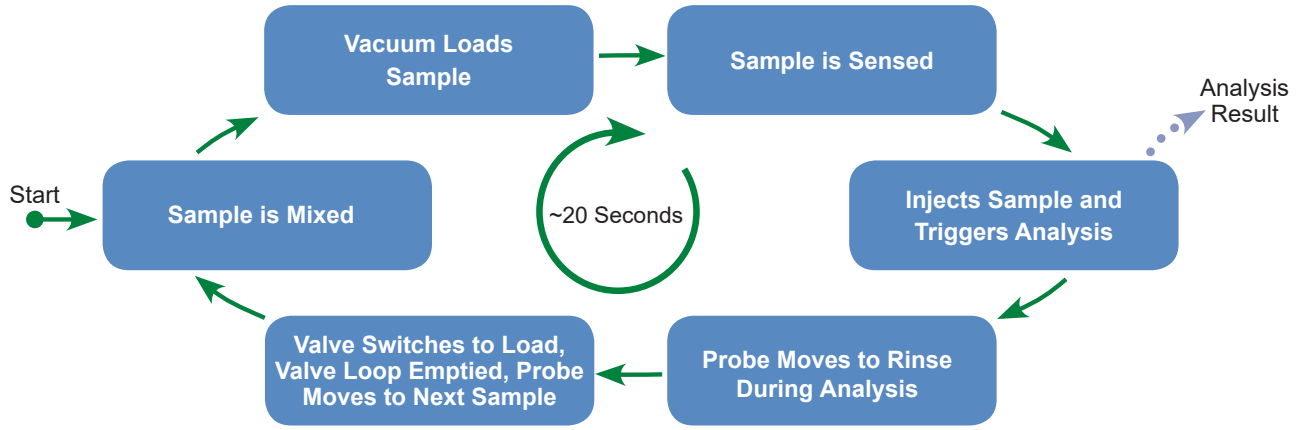
Gas Infusion Mixing with SampleSense Oil

SampleSense Oil provides a fast, accurate sample introduction system that incorporates a gas infusion mixing. This provides controlled sample mixing to homogenize the sample immediately before analysis, eliminating manual mixing of samples after dilution and resulting in significant time savings and improved productivity.

ASTM D5185 mandates that oil samples be diluted 10x by weight, and this is most often done with a clean kerosene diluent. Unfortunately, diluted samples, even when mixed by hand before analysis, will often settle and separate over time. This requires many laboratories to prepare samples and immediately mix and analyze them to prevent poor results due to settling. Even with this protocol, samples at the end of a long run may settle and consequently have poor analytical results. SampleSense Oil incorporates automatic mixing of each sample immediately before analysis through the gas infusion mixing. With this technology, samples may be diluted without mixing and may sit until the operator is ready to start the ICP analysis, saving substantial preparation time and allowing for flexibility in choosing when to analyze prepared samples.

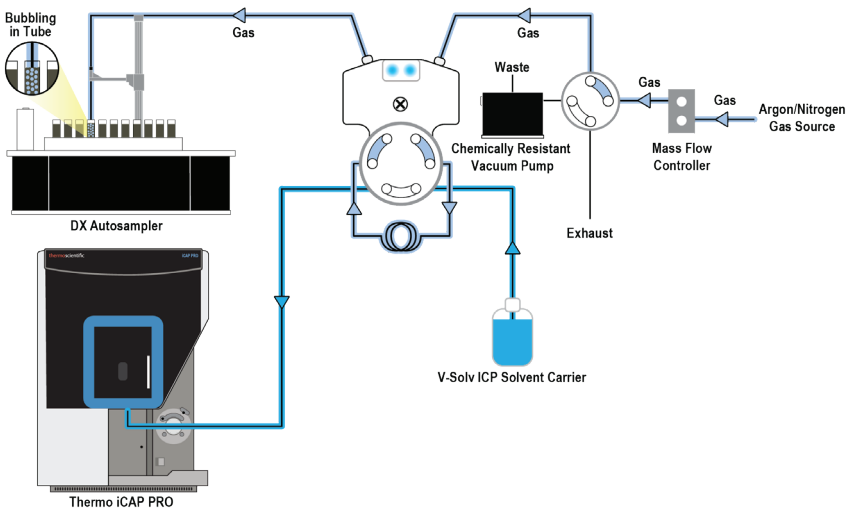
The sample mixing is performed by delivering a controlled gas stream through the probe, which causes the oil sample and solvent to be efficiently homogenized as the probe enters each tube. The valve then switches position to allow vacuum loading. With a mass flow controller connected, the gas flow can be software adjusted based on vial size and viscosity to ensure proper mixing for all vial sizes and varying oil viscosities.

SampleSense Oil Analytical Cycle

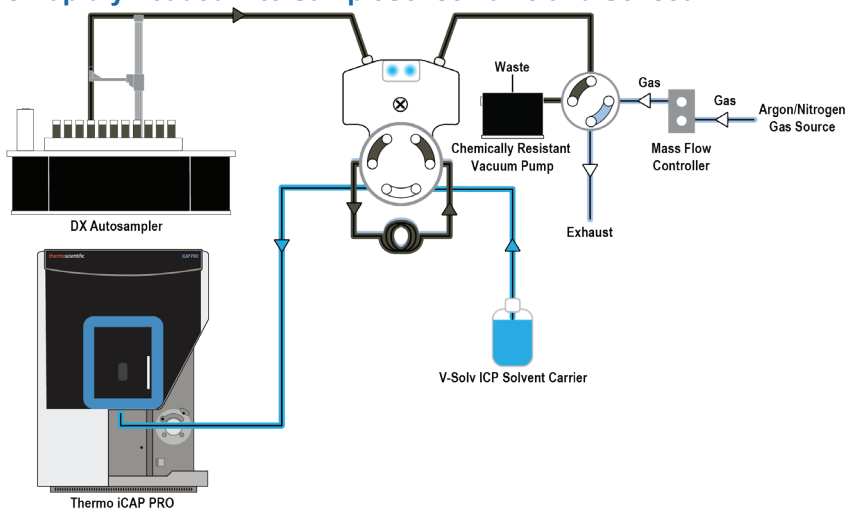


SampleSense Oil Controlled Mixing for Oil Samples

1. Probe Enters the Sample and Sample Is Mixed



2. Sample Is Rapidly Loaded into SampleSense Valve and Sensed



3. Discrete Sample Is Injected into Instrument and ICP Method Is Triggered

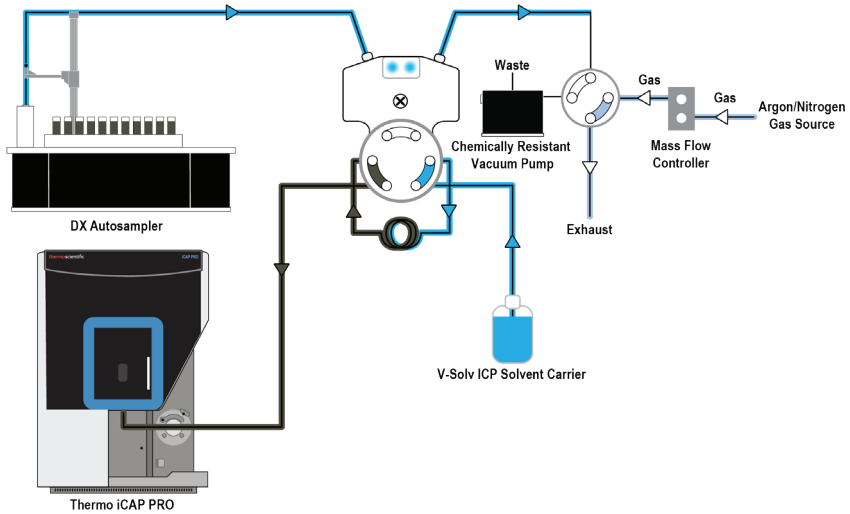


Figure 1. SampleSense Oil Controlled Mixing Diagram: (1) Gas infusion valve switches to allow gas flow through SampleSense valve and out the probe. Probe moves into the sample and mixes by gas infusion. (2) With the sample mixed, the gas infusion valve switches and vacuum loading of the loop begins. (3) Once the loop is filled and the sample is sensed, the SampleSense valve switches and sends the sample to the instrument while uptake path is rinsed.

Elimination of Oil Dripping

SampleSense Oil nearly eliminates oil dripping through three unique additions. **1)** Constant vacuum pull forces oil droplets to be pulled back into the probe, including all autosampler movements. **2)** DXCi autocorrecting autosampler provides high speeds, but with smooth movements, turning, and homing. This allows the high throughput and speed that is necessary, but without oil droplets spreading from sample to sample and onto the autosampler deck. **3)** A unique autosampler probe with a V-Shaped tip allows for a greater opening at the tip of the probe and less surface area for the oil to accumulate (see Figure 2 below). As the probe moves to its next location, the constant vacuum is applied to the probe, causing any remaining oil to be extracted back into the probe and thus nearly eliminating any oil drops from the probe tip. Constant vacuum, high-speed DXCi autocorrecting autosampler, and the V-Shaped tip probe together offer very fast analysis time, without the oil droplets that typically accompany high-speed oil analysis.

Diagram of Droplet Resistant Probe Tip

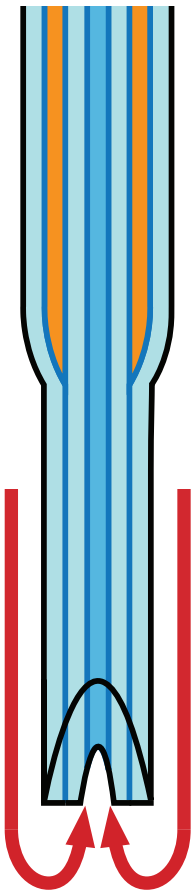


Figure 2. The figure above shows the V-shaped tapered tip probe. The figure shows the side view, where the cutouts reduce the oil-coated surface area and allows the vacuum to efficiently remove excess droplets.

Instrumentation

All samples were analyzed using SampleSense Oil in combination with a Thermo Fisher Scientific™ iCAP™ PRO XPS ICP-OES instrument.

Features

- 3 samples per minute
- Automatic sensing, injection and triggering of the ICP-OES analysis
- Detection and reporting of missing or empty sample tubes as “unsensed” samples
- Controlled gas infusion mixing of samples
- V-Shaped tapered tip probe

Sample Preparation

Oil samples used in analysis were diluted 10x using an organic solution of V-Solv ICP Solvent spiked with cobalt at 40 ppm for internal standard; V-Solv was also used as the rinse solution.

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)	8

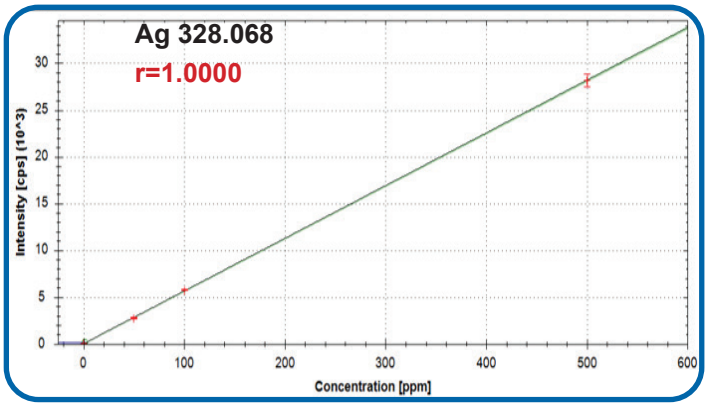
Nebulizer	MEINHARD® V-Groove Nebulizer (MP510-10QD)
Spray Chamber	Baffled Dual Pass Glass Zip Chamber (C3QX-37-P)
Torch	Quartz Torch (ML128074)
Injector	1.0 mm Demountable Quartz (ES-1623-0100-88)
Viewing Mode	Radial

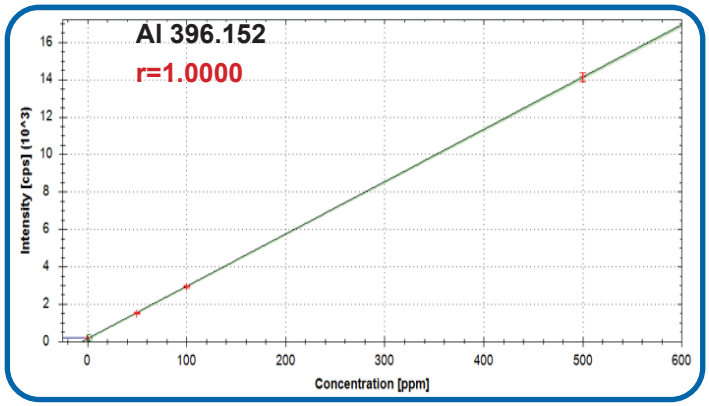
Analytes and Wavelengths Measured

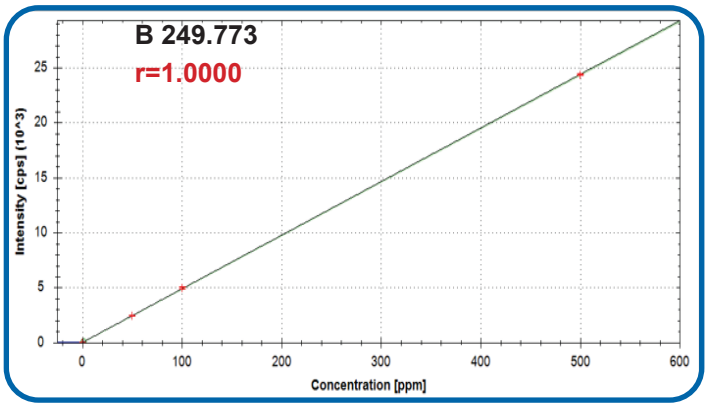
Analyte	Wavelength (nm)	Analyte	Wavelength (nm)
Co (IS)	228.616	Mn	257.610
Ag	328.068	Mo	202.030
Al	396.152	Na	589.592
B	249.773	Ni	231.604
Ba	230.424	P	177.495
Ca	315.887	Pb	220.353
Cd	226.502	Sb	206.833
Cr	283.563	Si	251.611
Cu	324.754	Sn	189.989
Fe	259.940	Ti	334.941
K	766.490	V	309.311
Mg	279.079	Zn	206.200

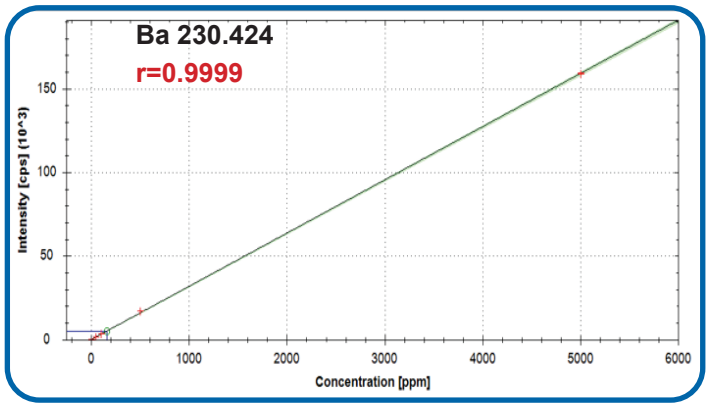
Figure 3. SampleSense Oil on the iCAP PRO XPS ICP-OES with four 90 vial racks and a 4DXCi (4F-SS-Oil-68). Autosampler options also include 2DXCi, 8DXCi, and 14DXCi.

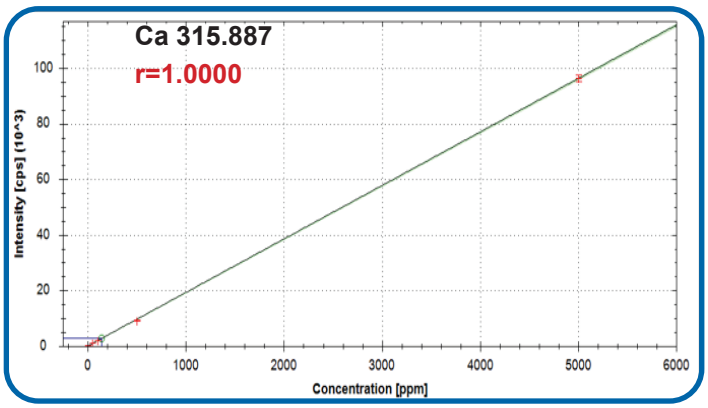
Calibration Curves for All Analytes

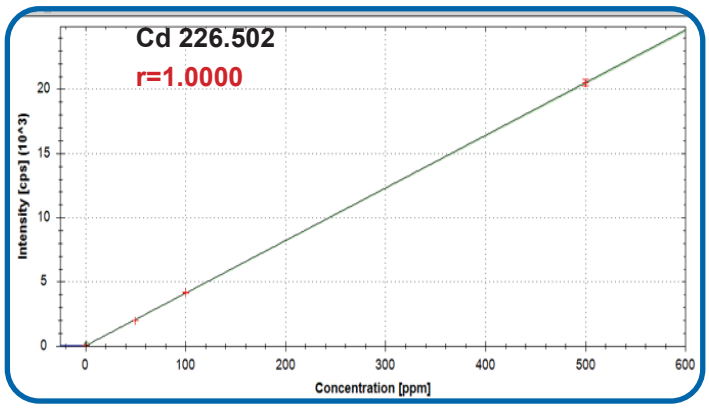


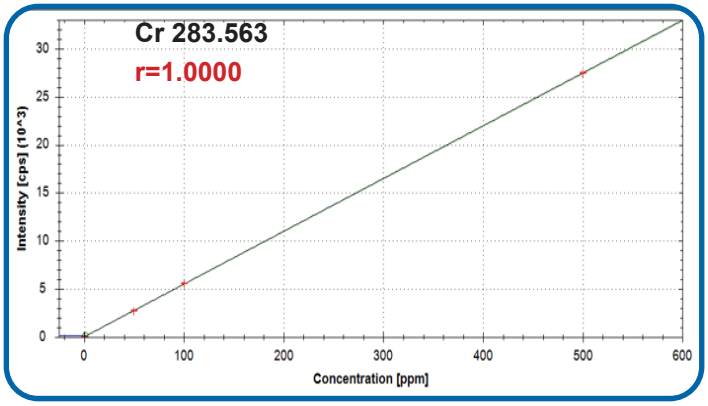


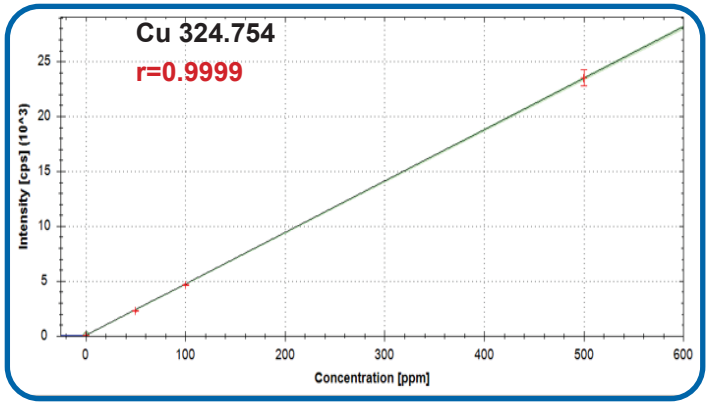


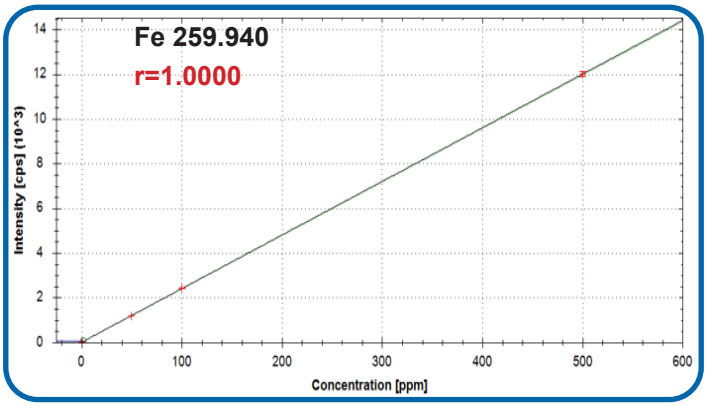








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r=1.0000
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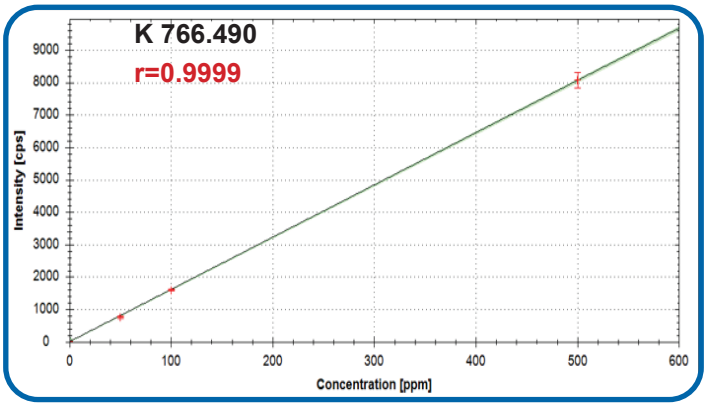
Concentration [ppm]


K 766.490
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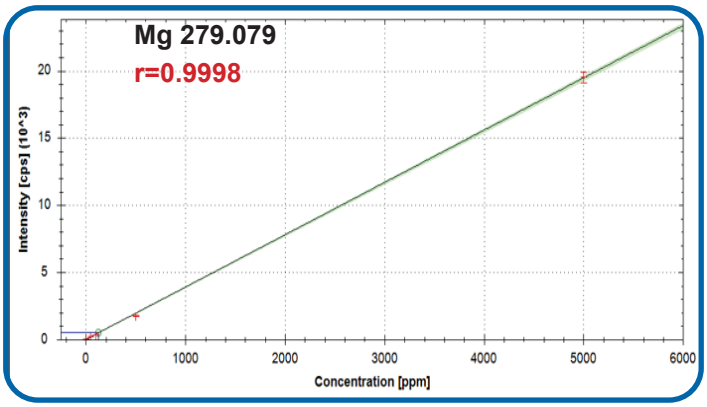
Intensity [cps]
Concentration [ppm]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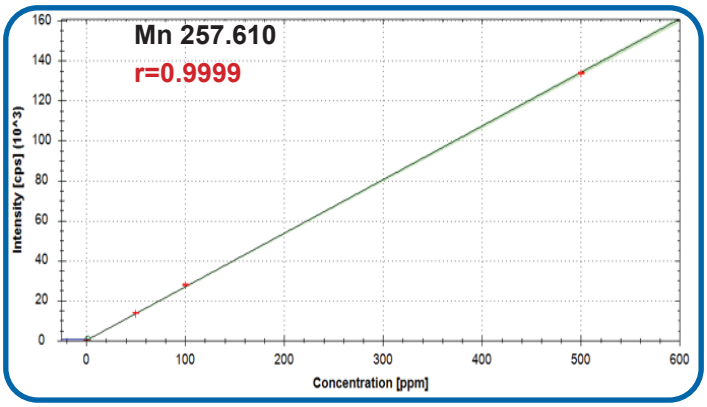
r=0.9998
Intensity [cps] (10^3)
Concentration [ppm]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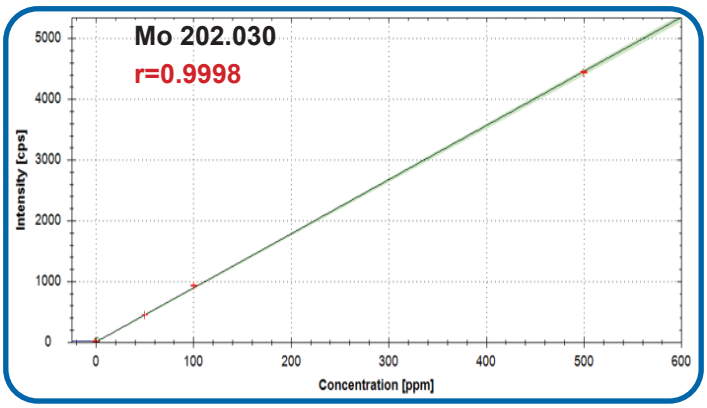
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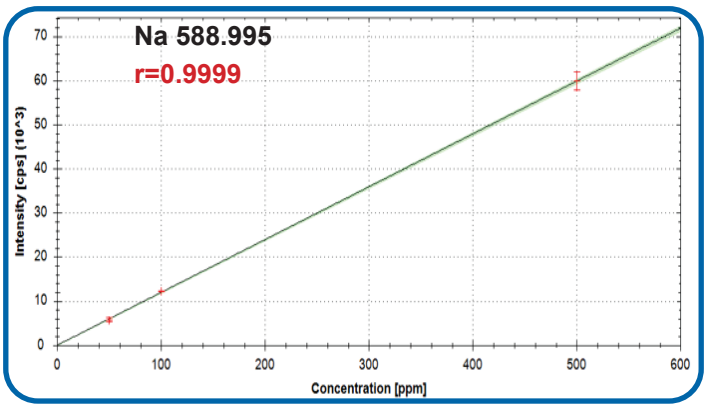
Concentration [ppm]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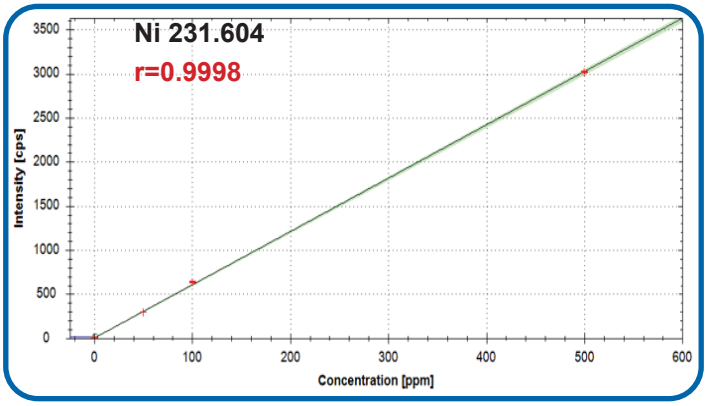
Concentration [ppm]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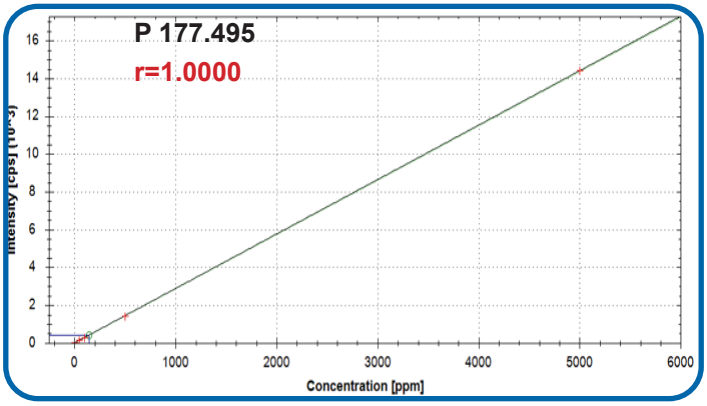
Concentration [ppm]


P 177.495
r=1.0000
Intensity [cps] (10^3)
Concentration [ppm]

Calibration Curves for All Analytes

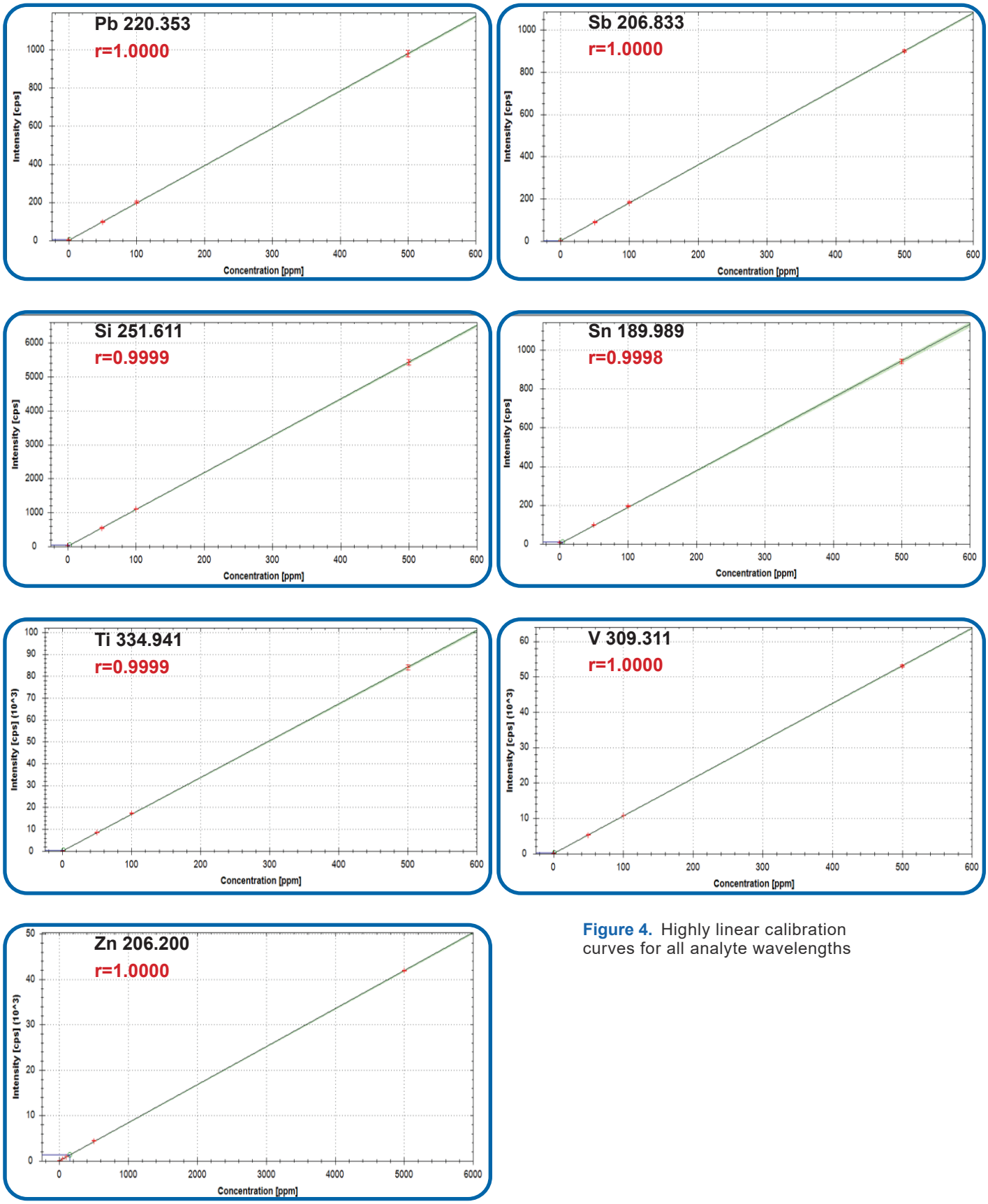


Figure 4. Highly linear calibration curves for all analyte wavelengths

SampleSense Oil Element Stability over 9 Hours and 30 Minutes for 1671 Samples

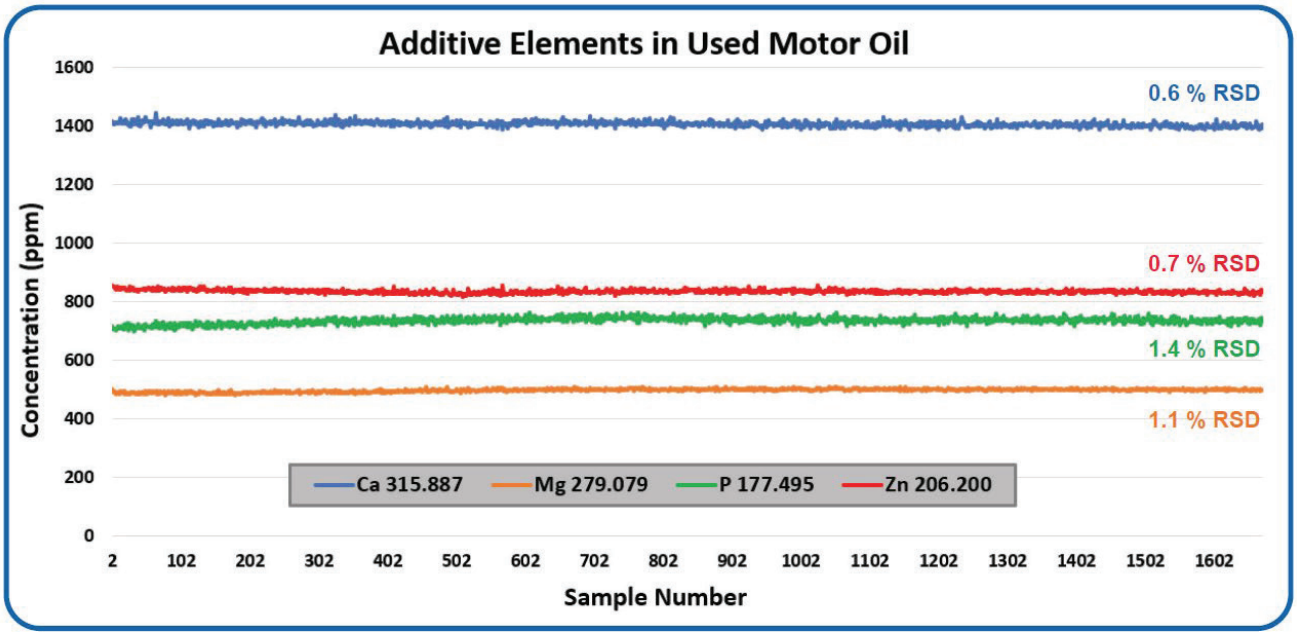


Figure 5. SampleSense Oil with 4DXCi autosampler using controlled gas infusion mixing for 23 elements measured over 1671 used motor oil samples in 9 hours and 30 minutes. Stability of four elements with higher concentrations shown above. Lower concentration elements showed similar stability.

500x Washout After High Calibration Standard

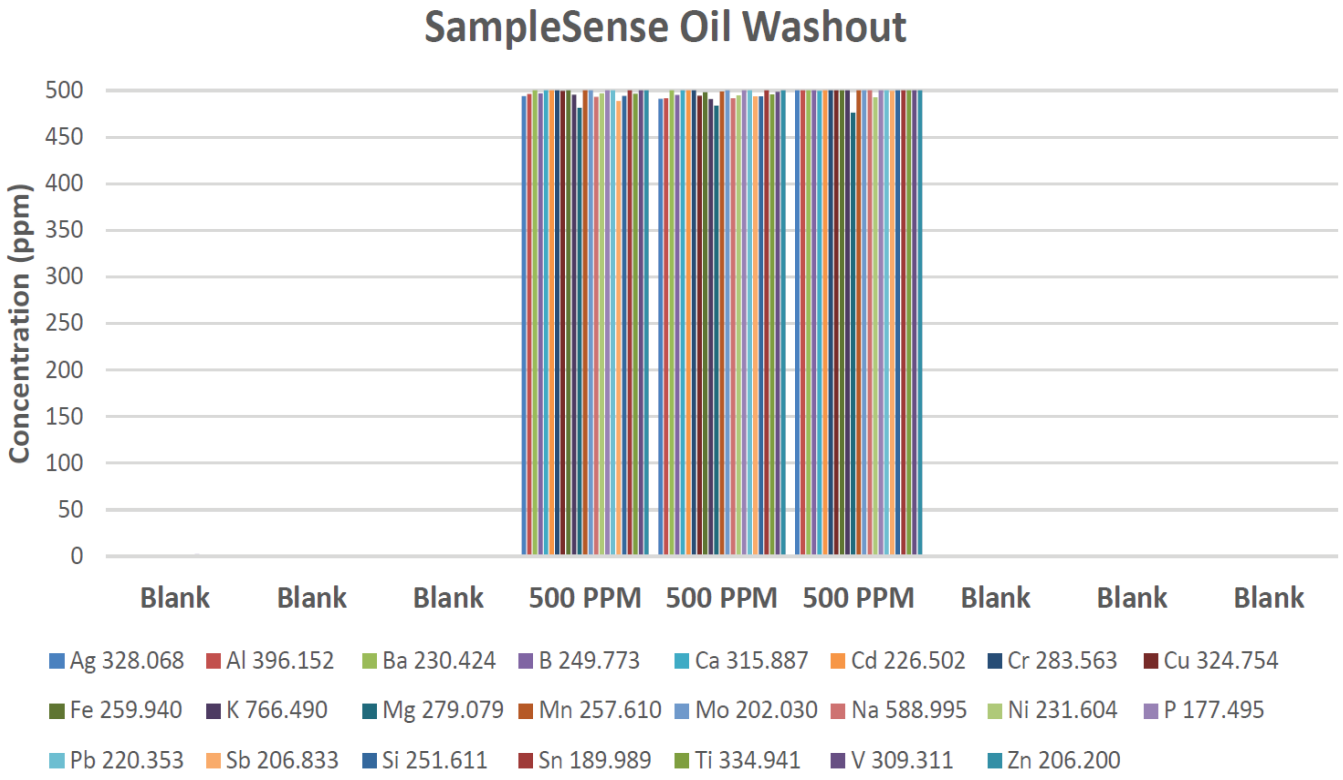


Figure 6. 500x washout for all wear metals.

Results

The system was calibrated at 50, 100, and 500 mg/L using three V-23 oil stock solutions in addition to an additives stock solution at 5000 mg/L for elements considered additives (B, Ca, Mg, P, and Zn). A 75 cSt base oil blank was also added to maintain the organic matrix present throughout the calibration. SampleSense Oil generated linear calibration curves (Figure 4) for all elements in under two minutes. The system then analyzed 1,671 samples in just over 9.5 hours. The stability was excellent, with RSDs for all the main elements under 2% (see Figure 5). The washout in the blank immediately after analyzing three high standards was >500x, showing suitable performance for wear metals oil analysis (see Figure 6).

SampleSense Oil Sample Throughput Performance

	1 Minute	1 Hour	8 Hours	24 Hours
# Oil Samples Analyzed per	3	180	1440	4320

Conclusion

SampleSense Oil completely optimizes the analysis of oils and lubricants for high-throughput laboratories using an automated valve injection sample introduction system. The use of a controlled gas infusion mixing allows for complete sample homogenization for various sample sizes and viscosities 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. The probe design combined with the DXCi autosampler allows for high-speed movement while eliminating oil droplets and the resulting cross-contamination. SampleSense Oil is a highly effective automation system that improves analytical efficiency in a production laboratory environment, providing quick and accurate determination of trace elements in all oil and lubricant fluids.