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Application Note

# Automated Sample Preparation of Coolant Samples for Analysis by ICP-OES

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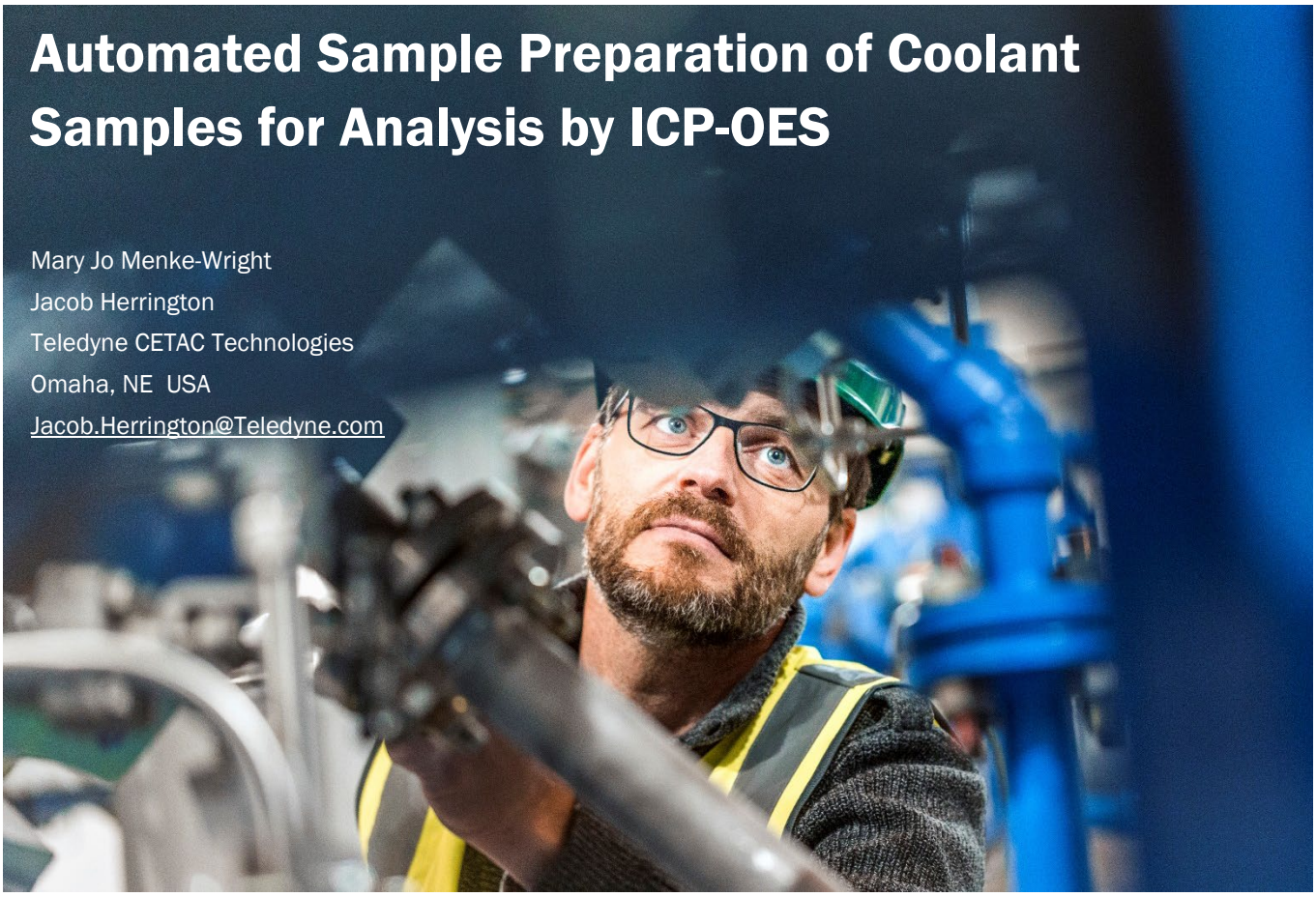


## Introduction

The analysis of coolant samples taken from heavy machinery is a growing part of the in-service marketplace. Similar to the analysis of lubricating oils, the analysis of coolant samples provides critical information regarding the health of the engine and needed maintenance. Assessing the elements present in these samples allows the user to test for contamination with other fluids, for excessive dilution of the coolant, for the quality of the additive package and for particulate contamination either from environmental sources or component failure. This essential information when combined with a proactive maintenance program can improve the longevity of the cooling system.

Coolant samples are typically diluted at 10× to mitigate matrix effects and reduce analyte concentrations such that they are within the instrument's detection range. These dilutions can be time consuming and prone to user error. The SimPrep is stand-alone auto-diluter which dilutes samples, prepares calibration curves, and adds reagents. The accuracy of the system is equivalent to samples prepared by hand. In addition to the SimPrep, Teledyne CETAC offers the *ASXPRESS PLUS* Rapid Sample Introduction system which can increase sample throughput by reducing sample uptake times and eliminating rinse times. Combining these two products with the Oils 7400 homogenizing autosampler, laboratories can increase their production capability without significantly impacting their labor costs. In this note, the combination of the *ASXPRESS PLUS*, Oils 7400 and the SimPrep will be shown to facilitate coolant analysis.

## Sample Preparation

A local laboratory supplied various types of used coolants from commercial vehicles. A coolant of each type was randomly selected for analysis. Samples were diluted at 10× in triplicate by the SimPrep. They were also spiked for various elements in duplicate. One coolant (not shown) was aliquoted 10× by the SimPrep to demonstrate the accuracy of the system.

## Instrument Conditions

Sample analysis was accomplished using a Perkin Elmer Avio ICP-OES with a Teledyne CETAC Oils 7400 Autosampler connected to an *ASXPRESS PLUS* Rapid Sample Introduction Accessory. A 2.5 mL aqueous loop was used for the sample aliquot. The software settings are in Figure 2.

In-service laboratories are facing a growing need to test both oils and coolant samples. The Oils 7400 autosampler features a dual chamber rinse station with separate peristaltic pumps for different matrices. This allows the user to switch between oils and coolant analysis without having to replumb tubing or replace the rinse station. Simply moving the rinse station from one position to the other as seen in Figure 3, switches the active pump.

The ICP was calibrated using a blank and 4 standards prepared in 5% glycol. For the internal standard, 2 ppm of Cobalt was added to all samples and standards using the 7th port of the *ASXPRESS PLUS* valve. The SimPrep prepared the calibration curve as shown in Figure 4. For the Cal 1 Standard, 50 µL of the Cal Parent standard was added to the vial and filled to a final volume (Volume [µL] column) of 10,000 µL.

The calibration curves for Copper and Boron are shown in Figure 5. The SimPrep accurately diluted the calibration curve while the *ASXPRESS PLUS* precisely delivered the samples. Thus, correlation coefficients for all elements were 0.999 or greater.

**Figure 1: Coolant Samples**

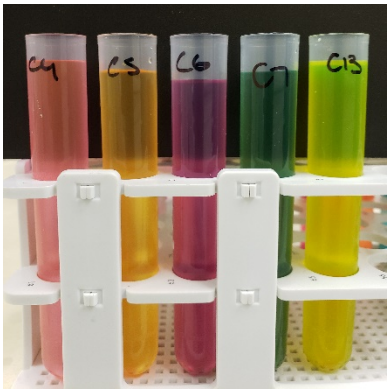



**Figure 2: *ASXPRESS PLUS* Settings**

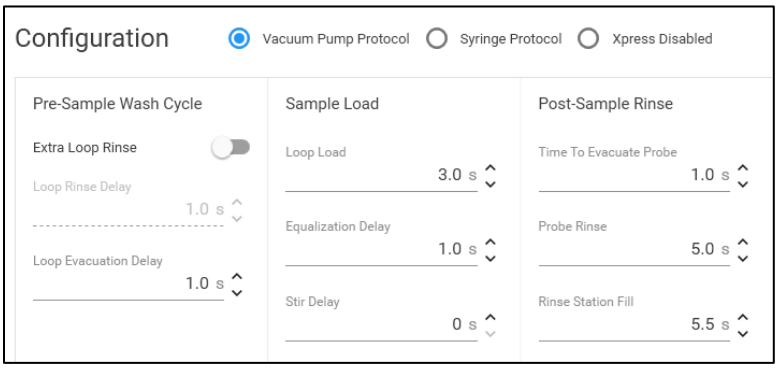



**Figure 3: Oils 7400 Rinse Station Positions**

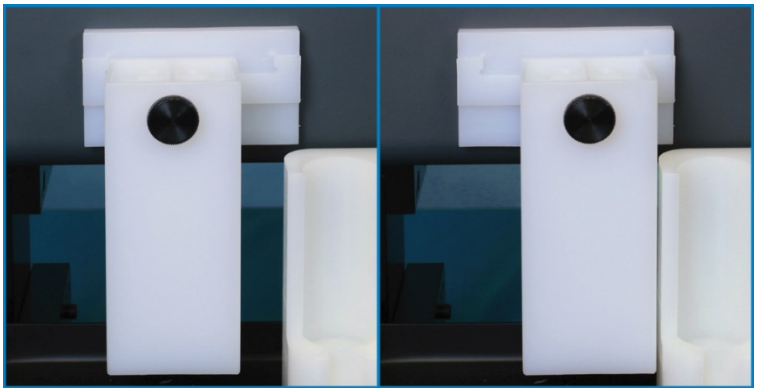
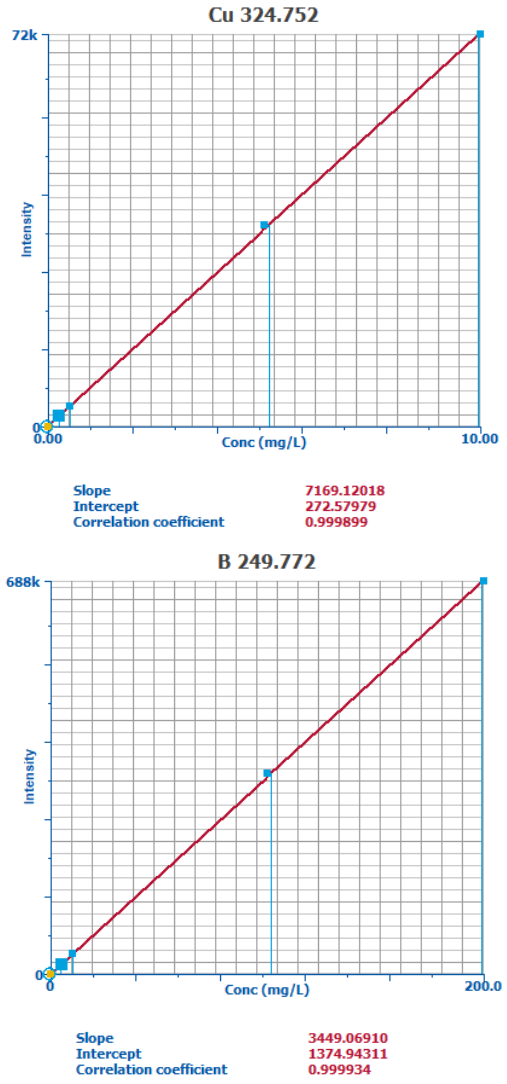


**Figure 4: SimPrep Calibration Curve Preparation**

| Sample | | Dilution 1 | | | | |
|---|---|---|---|---|---|---|
| Position | Name | Position | Volume [µl] | Vol Sample | Vol Std | Name Std |
| | Blank | R1-01 | 10000 | | | |
| | Cal 1 | R1-02 | 10000 | | 50 | Cal Parent |
| | Cal 2 | R1-03 | 10000 | | 100 | Cal Parent |
| | Cal 3 | R1-04 | 10000 | | 1000 | Cal Parent |
| | Cal 4 | R1-05 | 10000 | | 2000 | Cal Parent |

**Figure 5: Calibration Curves**




## Results

### Time Savings

Once the SimPrep software is programmed, the same program can be reused for subsequent calibration curves and samples. Time to set-up the SimPrep (including filling the diluent bottle) is only 3.5 minutes using a saved method. The SimPrep can operate unattended allowing the analyst more time to focus on other tasks. Additional time savings are achieved by adding an *ASXPRESS PLUS* to an existing coolant method. In Table 1, the *ASXPRESS PLUS* method is compared to a customer method. The *ASXPRESS PLUS* saved 50 seconds of analysis time per sample. For this application note, additional delay time was added to improve RSDs. The delay time can be as low as 15 seconds for trend analysis.

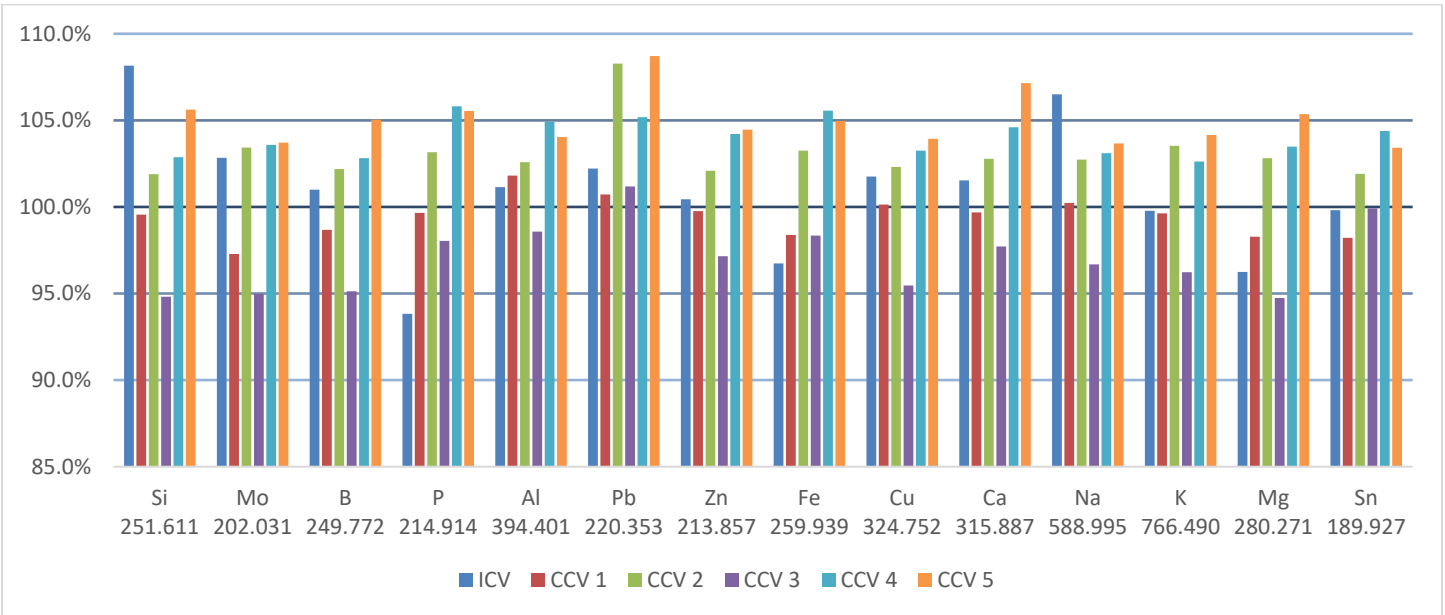
**Table 1: Sample Introduction Method Settings**

| | Customer Method Time (s) | *ASXPRESS PLUS* Method (s) |
|---|---|---|
| Flush Time | 35 | 0 |
| Delay Time | 15 | 30 |
| Rinse Time | 30 | 0 |
| Total Time | 80 | 30 |

### Quality Control

To verify the calibration curve, calibration verification standards were analyzed after calibration, every 10 samples, and at the end of analysis. The initial calibration verification (ICV) was prepared by hand from a second source. The continuing calibration verifications (CCV) were prepared by the SimPrep from the same source as the curve at a concentration of ½ the highest calibration standard. Results are in Figure 6. Recoveries for all elements were between 90–110%.

**Figure 6: ICV and CCV Recoveries**



## Hand Prep vs SimPrep

Coolant samples can be challenging to dilute as many of the samples are viscous. Traditional pipetting techniques can be subject to error as the sample viscosity interferes with the ability of the user to consistently expel all of the sample. Moreover, different analysts can have different technique, affecting the accuracy of dispensing. One mL of a coolant sample was aliquoted 10× by the SimPrep, Analyst A (uses a pipette daily), and Analyst B (uses a pipette infrequently). Each sample aliquot was weighed, then the RSD of the 10 measurements was calculated. The SimPrep gave the most repeatable sample delivery. Incorporating this system in a laboratory workflow allows for consistent sample preparation regardless of the operator.

## Spiked Results

The coolant samples and a 5% glycol blank were spiked with 0.5 mL of a second source standard. Final concentrations are in Table 2. Some elements were not included in the spike as the samples had high concentrations of those elements.

The SimPrep was able to dilute the sample and spike it in the same preparation step. For the matrix spike (MS) in Figure 8, 1000 µL of R1-49 and 500 µL of the spike standard were added to a vial and filled to a final volume of 10,000 µL.

Laboratory Control Sample (LCS) recoveries were between 95–106%. Sample recoveries for all samples except Pb for C4 were between 81–113%. As the other elements in C4 were within 20%, the failure is most likely due to matrix effect and not an inaccurate spike aliquot.

**Figure 7: Ten 10x Sample Preparations**

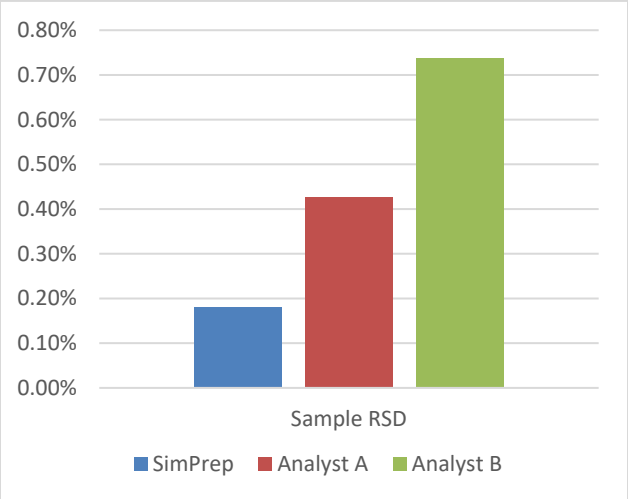



**Table 2: Final Concentration LCS/MS/MSD**

| Element | Spike (mg/L) |
|---|---|
| Copper | 1.0 |
| Tin | 2.0 |
| Silicon | 2.3 |
| Zinc | 2.5 |
| Al, Pb, Fe, Ca, Mg | 5 |

**Figure 8: SimPrep Spike Sequence**

| Sample | | Dilution 1 | | | | |
|---|---|---|---|---|---|---|
| Position | Name | Position | Volume [µl] | Vol Sample | Vol Std | Name Std |
| | LCS | R1-01 | 10000 | | 500 | Spike |
| | LCSD | R1-02 | 10000 | | 500 | Spike |
| R1-49 | Coolant | R1-03 | 10000 | 1000 | | |
| R1-49 | MS | R1-04 | 10000 | 1000 | 500 | Spike |
| R1-49 | MSD | R1-05 | 10000 | 1000 | 500 | Spike |

**Figure 9: LCS and Matrix Spike/Matrix Spike Duplicate Recoveries**

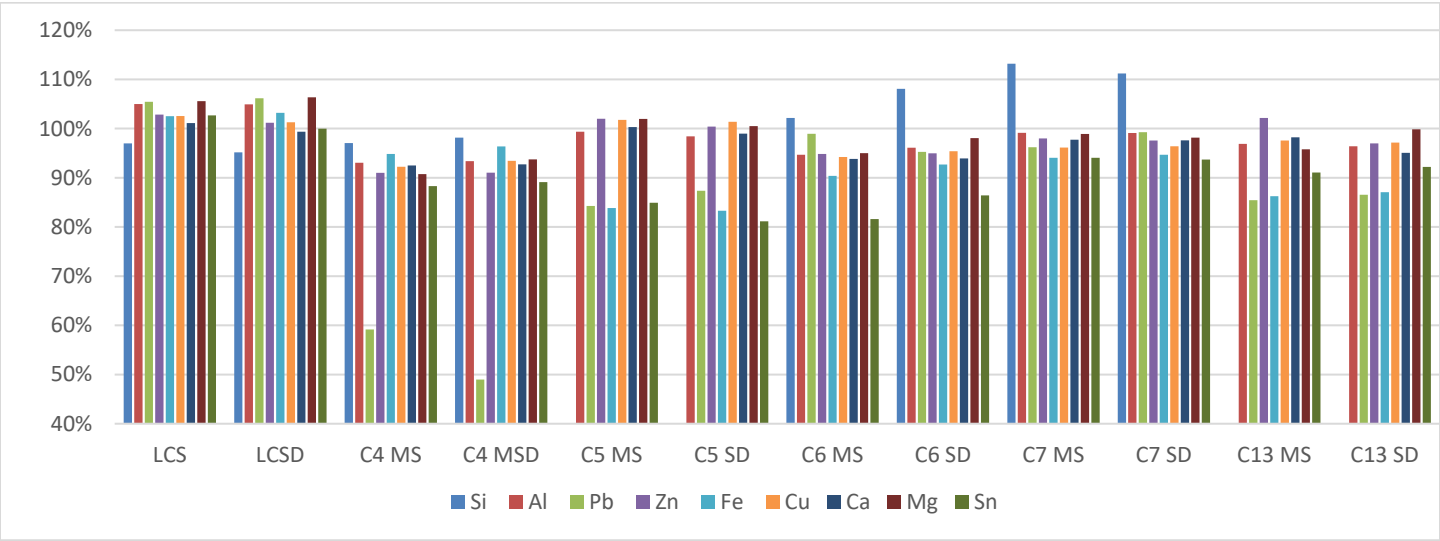


**Figure 10: Relative Percent Difference between Spike Duplicates**

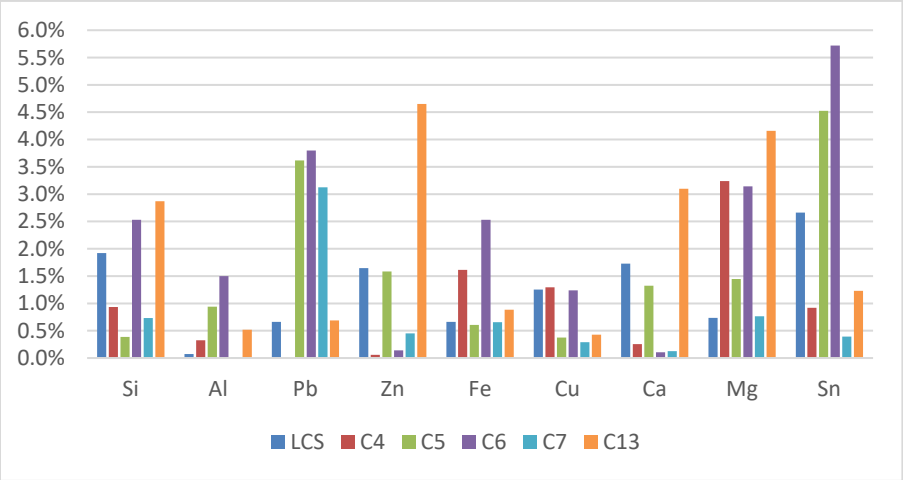
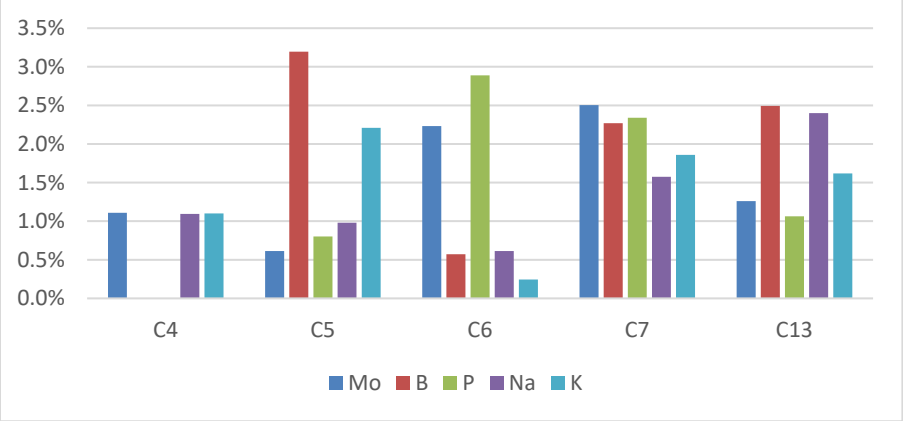



**Table 3: Sample Concentrations (mg/L)**

| | Mo | B | P | Na | K |
|---|---|---|---|---|---|
| C4 | 56.3 | - | - | 74.6 | 579.8 |
| C5 | 13.9 | 85.4 | 10.6 | 449 | 59.3 |
| C6 | 12.2 | 19.9 | 12.6 | 169.5 | 42.5 |
| C7 | 2.1 | 3.4 | 110.5 | 276.2 | 448.5 |
| C13 | 9.2 | 75.6 | 12.9 | 370.8 | 55.5 |

**Figure 11: RSDs 3 Sample Replicate Measurements**




## Sample Precision

The RPD was calculated for the LCS and spiked samples. LCS RPDs were less than 3% while sample RPDs were less than 6%. Sample results easily meet the 10% RSD expectation. Many elements had RPDs of less than 0.5% indicating excellent spike preparation precision by the SimPrep.

Each coolant sample was also prepared at a 10x dilution in triplicate using the SimPrep. Sample concentrations are in Table 3 and RSDs are in Figure 11. RSDs are less than 5% across all elements, demonstrating not only the reliability of the SimPrep, but also the precision of the sample introduction by the *ASXPRESS PLUS* and Oils 7400.

## Conclusion

The SimPrep and the *ASXPRESS PLUS* are valuable additions to any laboratory to facilitate faster and more reliable analysis of coolant samples. The SimPrep is able to accurately and precisely dilute coolant samples, saving analyst preparation time, while the *ASXPRESS PLUS* reduces analysis time allowing for higher sample throughput. These accessories can be used in conjunction with the Oils 7400 homogenizing autosampler providing a versatile automation package for the fast-paced in-service laboratory. With these Teledyne CETAC sample preparation and introduction accessories, production can be maximized while maintaining the highest quality data.

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