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Application Note

Using Automated Sample Mixing to Improve the Quality of Engine Oils Analysis with ICP-OES

Introduction

Analysis of wear metal samples provides critical information in determining if and when heavy equipment needs to undergo expensive maintenance procedures. The value of this testing has been well proven over the last two decades, and the accuracy of this testing is paramount to save companies time and money.

When analyzing oils by ICP-OES, samples are typically diluted prior to analysis with an organic solvent such as kerosene. The oil samples and solvent have different viscosities, resulting in separation over time. Ideally, samples would be analyzed immediately after preparation and prior to analysis. However, this is not always practical as laboratories run multiple instruments and may have difficulty finding time to mix samples properly.

To ensure analytical accuracy, the Teledyne CETAC Oils 7400 Autosampler is equipped with a stirring paddle which mixes each sample prior to analysis. In this application note, we will demonstrate the improved accuracy and reliability of data analyzed with the Oils 7400 Autosampler.

Sample Preparation

Two used engine oils samples were obtained from a local laboratory. Samples were diluted on a per weight basis at a 1:10 dilution with V-Solv™ ICP solvent. A Conostan™ S-21 100 cSt Oil Standard was diluted in a similar manner to make a 10 ppm standard. The samples and 10 ppm standard were prepared in triplicate and allowed to settle overnight (see Figure 1).

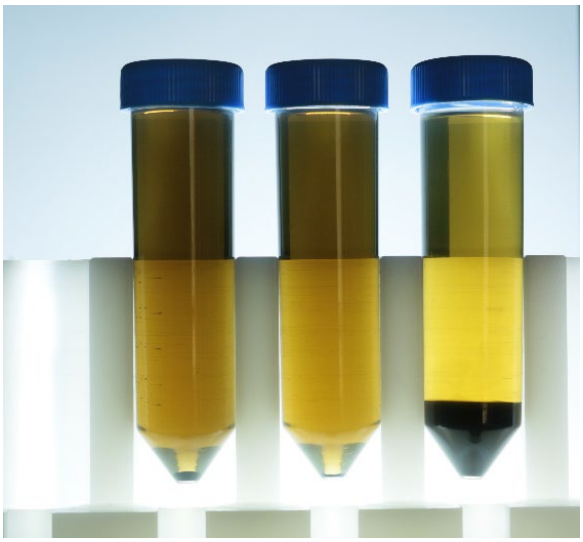

Figure 1: Oil Sample As It Settles

Each sample type was analyzed using three different final preparation steps. The first set of samples were analyzed without any mixing. The next set of samples were stirred by the autosampler prior to analysis. The last set of samples acted as the control set and were stirred by the autosampler and hand mixing.

Instrument Conditions

Samples were analyzed on a Thermo iCAP™ 7600 radial ICP-OES. Instrument conditions are listed in Table 1.

Table 1: Thermo iCAP Instrument Conditions

Instrument Parameters	Setting
Exposure Time	1 second
RF Power	1150 W
Nebulizer Gas Flow	0.35 L/min
Coolant Gas Flow	12 L/min
Auxiliary Gas Flow	1.5 L/min
Pump Speed	40 rpm
Viewing Height	12 mm
Slit Position	High

The instrument was calibrated using 10, 25, and 50 mg/L standards prepared from a Conostan oil standard (different than the sample standard). Calibration coefficients were 0.9995 or better. Reported elements and their wavelengths are listed in Table 2. A continuing calibration verification (CCV) at 25 mg/L was analyzed after the curve and after every 3 analyses of each sample type. All CCVs came back within 10% of the target value.

Table 2: Elements and Wavelengths

Element	Wavelength
Aluminum	308.215
Barium	455.403
Boron	249.773
Calcium	317.933
Iron	259.940
Magnesium	285.213
Manganese	257.610
Nickel	231.604
Titanium	334.941
Vanadium	309.311
Potassium	766.490
Silicon	288.158
Tin	283.999

The Oils 7400 Autosampler was run using the default movement (5/5) and stirrings speeds (5%). The iCAP peristaltic pump was used to drive sample aspiration. Each sample tube was analyzed until fully aspirated, resulting in a minimum of 50 replicates for each sample.

Results

Used Engine Oil

The engine oils did not have detection of all elements analyzed; therefore, we chose elements with high intensities for comparison in Figure 2. The unmixed samples results were highly inconsistent with RSDs of 120% for engine oil 1 and 17% for engine oil 2. The stirred only results had an RSD of 2.2% for both samples which was similar to the 1.5% average RSD for the hand mixed/stirred samples.

Evidence to the importance of proper mixing can be seen in Figure 2. The heavier sample oil settles to the bottom of the tube, which is where the probe on the autosampler goes to when aspirating the sample. This results in the most concentrated sample being pulled up first and then the intensity decays to a much lower level than what is seen in the mixed samples. So, the concern becomes, what part of the sample is being analyzed?

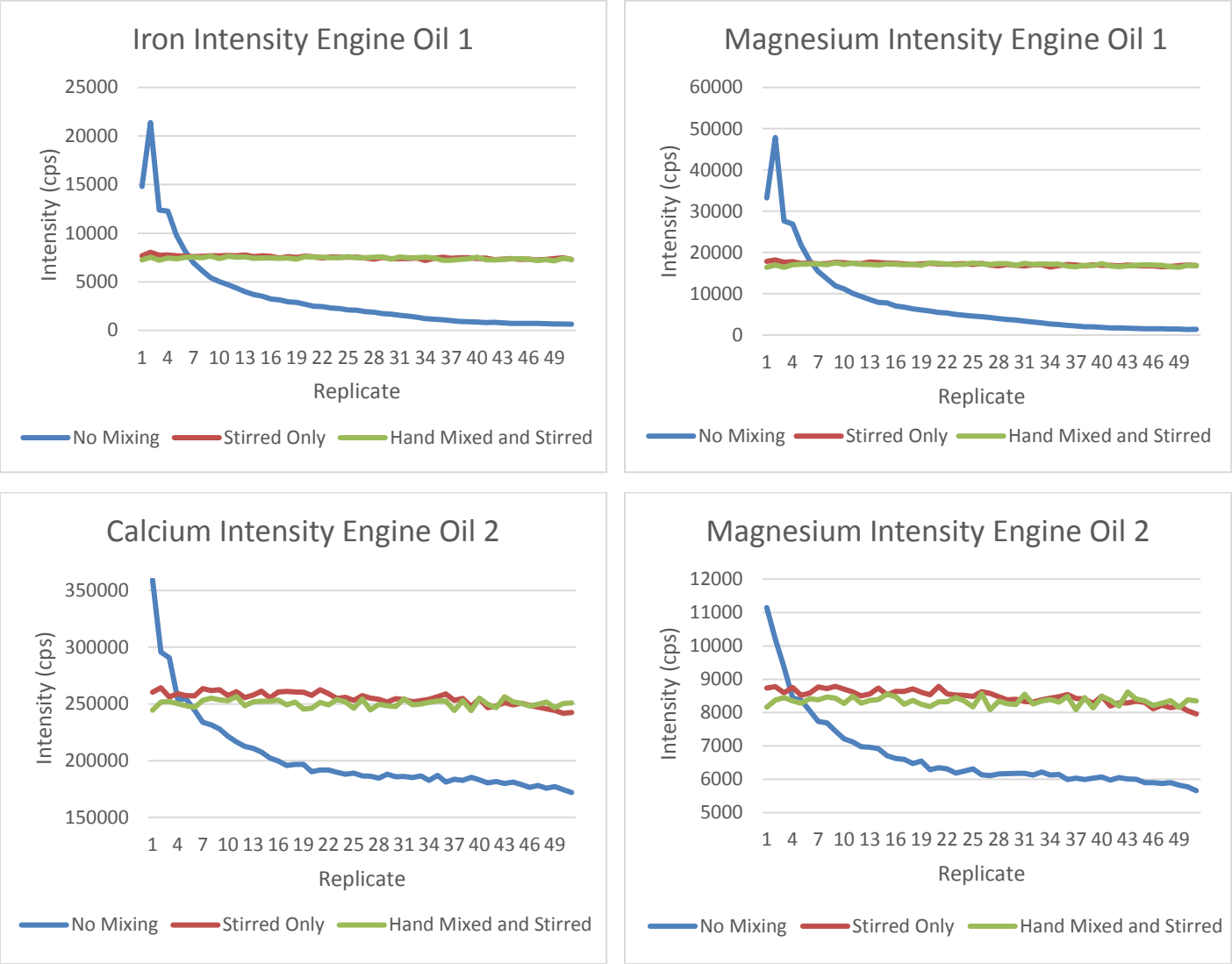

Figure 2: Select Elements with 50 Replicate Intensities for Engine Oils

Since most laboratories do not run more than 3 replicates per sample, we used the first 3 replicates when calculating the concentration of these samples. The results in Figure 3 show the sample concentration for engine oil 1 without mixing are almost double the concentration with mixing. Engine Oil 2 has better results; however, the unmixed samples are roughly 30% higher than the mixed samples. The autosampler (stirred only) achieved similar results as the hand mixed samples.

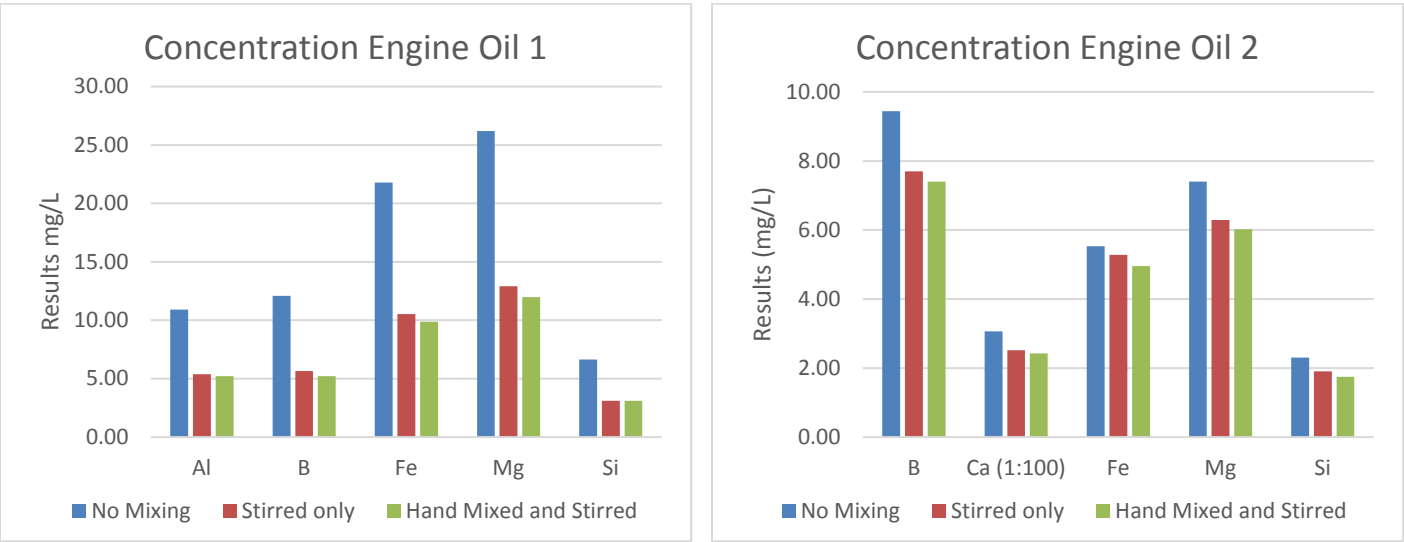

Figure 3: Average Concentration First 3 Replicates for Both Engine Oils

10 ppm Standard

A 10 ppm standard was analyzed to demonstrate the improved reliability and accuracy for a known sample. The recovery of the first 3 replicates is seen in Figure 4. The unmixed standard had an average of 30% higher response mixing the standard by hand. Mixing the standards by hand or autosampler resulted in similar recoveries between 90—110% shown by the black lines in Figure 4.

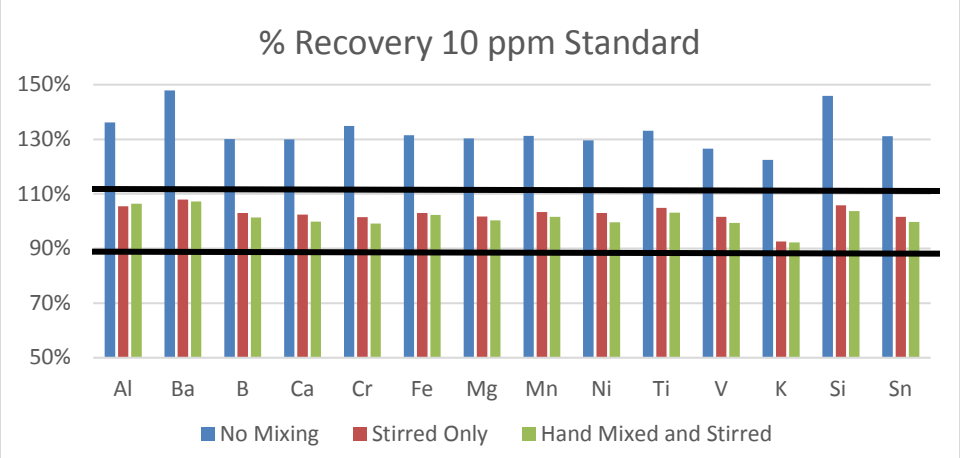

Figure 4: % Recovery for First 3 Replicates of 10 ppm Standard

The average recovery per replicate was calculated by averaging the percent recovery for each element per replicate (Figure 5). The standards mixed either by hand or autosampler remained stable throughout the analysis compared to unmixed standards which showed extreme variability.

The RSD for the 50 replicates is in Figure 6. Results for both standards mixed by hand and standards mixed by the autosampler are below the 5% threshold.

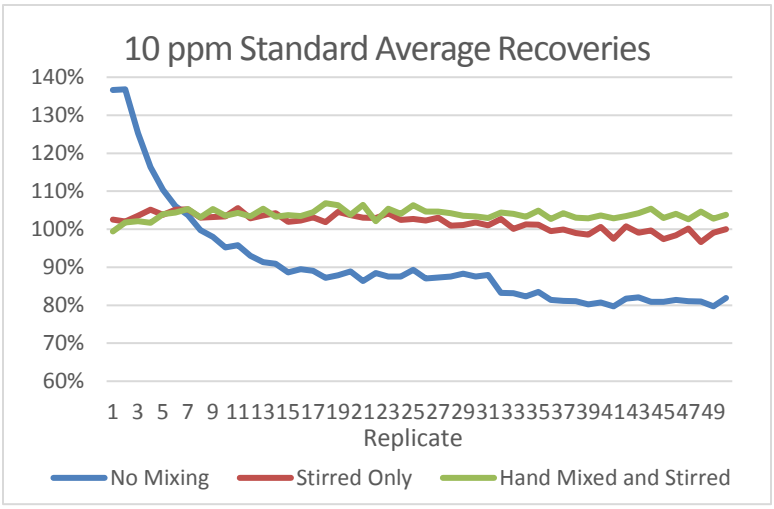

Figure 5: Average Recoveries Per Replicate

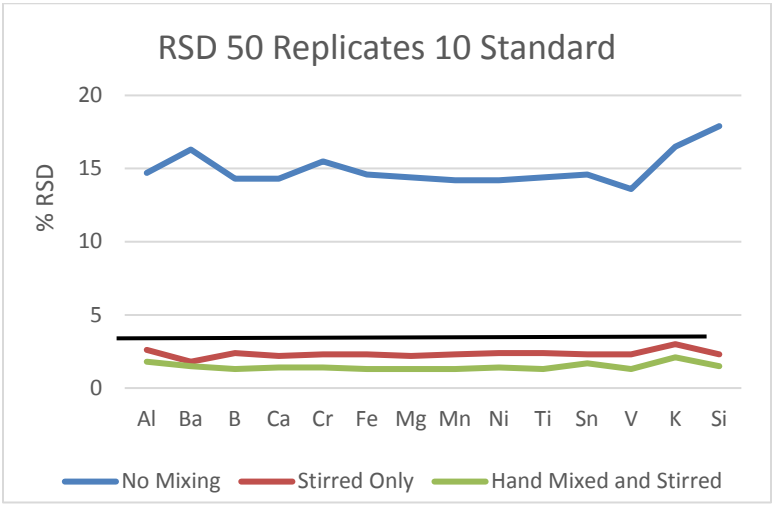

Figure 6: Percent RSD for 50 Replicates of the 10 ppm Standard

Conclusion

This application note has demonstrated that oil samples allowed to settle can have results which are 30-50% higher than the value of a properly homogenized sample. Hand mixing produces accurate results; however, it is time consuming and not always feasible when samples are run overnight. The Oils 7400 Autosampler achieved similar results as the hand mixed samples without user intervention. With its simple set-up and robust operation, the Oils 7400 Autosampler is an ideal addition to engine oil analysis.

Trademarks

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