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Application Note

Analysis of Lubricating Oils following ASTM D5185[1]

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Introduction

There is an ever-growing demand for heavy machinery in the global market. This demand stretches across many industries including mining, construction, agriculture and more. As the need for equipment increases, it becomes ever more important to properly maintain what has already been produced. One key method for ensuring that heavy machinery is properly maintained is the analysis of lubricants.

By analyzing the metals present in the gear, engine, and lube oils, as well as transmission fluids, we are able to predict wear and tear of the engine and predict what parts will fail. This helps to minimize downtime by allowing maintenance to be done in a controlled and planned timeline, ensuring the equipment is ready to run when needed. This type of analysis has become so common and important to the marketplace that ASTM created method D5185[1] specifically for quantifying the amount of wear metals in lubricating oils.

The testing herein was done to show that a new Stirring autosampler, the Teledyne CETAC Oils 7400, can meet the requirements of the ASTM methodology by homogenizing samples and facilitating fast analysis while still maintaining proper data quality.

Instrumentation

Monitoring wear metals presents a set of challenges which can be met with the right equipment. Typical sample concentrations range from 1 ppm to 500 ppm. This makes ICP-OES an excellent fit due to its speed, ease of use, and robustness. The Avio 500 has previously been shown[2] to be able to meet the requirements of method D5185, and many of the details from that application note were replicated for this experiment.

Teledyne CETAC Oils 7400 with *ASXPRESS PLUS*

PerkinElmer Avio 500 ICP-OES

The Avio 500 was paired with the Oils 7400 autosampler for its unique capabilities which are key to wear metals analysis. Features include a built-in stirring motor and paddle for mixing samples prior to analysis, a sample drip cup to maintain cleanliness and prevent cross contamination, and a filter probe to maintain the health of the sample introduction glassware on the ICP-OES. The Oils 7400 simplifies the requirements for homogenization, reproducibility, and repeatability by removing human error. In addition to the autosampler, the *ASXPRESS PLUS* Rapid Sample Introduction System was used to reduce sample run times and simulate conditions for a high throughput laboratory doing wear metals analysis.

Sample Preparation

A 4 point calibration curve from 0 to 500 ppm was prepared from a VHG-V23 multi-element standard for oils analysis. A 75 cSt base oil was used for the blank and all samples were diluted by weight with Kerosene. Samples were spiked with 40 ppm Cobalt internal standard to show consistency of the analysis. We used a 50 ppm standard for our QC sample, which was prepared in a larger quantity so it could be analyzed many times to show the reproducibility of the system.

Methodology

All samples were analyzed in radial viewing mode with 2 replicates per sample. The elements and wavelengths analyzed are specified in table 1. A GemCone™ nebulizer was coupled with a baffled spray chamber and Avio organics torch for sample aspiration. This setup, when combined with the *ASXPRESS PLUS* and Oils 7400 autosampler allowed for fast and reliable sample introduction or uptake.

Table 1: Analysis Elements and Wavelengths

Element	Wavelength (nm)	Element	Wavelength (nm)
Al	394.401	Mn	257.61
Ag	328.068	Na	588.995
B	249.677	Ni	232.003
Ba	232.527	P	214.914
Ca	315.887	Pb	220.353
Cd	228.802	Sb	217.582
Cr	205.56	Si	288.158
Cu	324.752	Sn	189.927
Fe	259.939	Ti	334.94
K	766.49	V	292.464
Mg	279.077	Zn	213.857
Mo	203.845	Co*	228.616

*Internal standard

Results and Discussion

For high throughput labs, sample to sample time is critical. Every minute that the ICP is running is cost to the laboratory and with that in mind this experiment was conducted with the *ASXPRESS PLUS* Rapid Sample Introduction System. The *ASXPRESS PLUS* uses a vacuum pump to quickly load all sample necessary for analysis into a sample loop which is then pushed, via the ICP peri-pump to the nebulizer for aspiration and analysis. During analysis the *ASXPRESS PLUS* rinses the probe and loop so that no rinse time is needed between samples. This reduces the time per sample thus greatly increasing the operational efficiency of the ICP.

Here we define operation efficiency for the ICP-OES to be the percent of time it is analyzing samples out of total operational time. Figure 1 shows a graphical representation of how the *ASXPRESS PLUS* affects the efficiency of the analyzer. Note that the rinse time is removed and in ten minutes time more than 20 samples can be run as compared to only 10 samples previously. Also, the operational efficiency improves from 15% to 32%. With the *ASXPRESS PLUS* we were able to average a time of 27 seconds per sample even with all 23 elements being analyzed as stipulated by D5185.

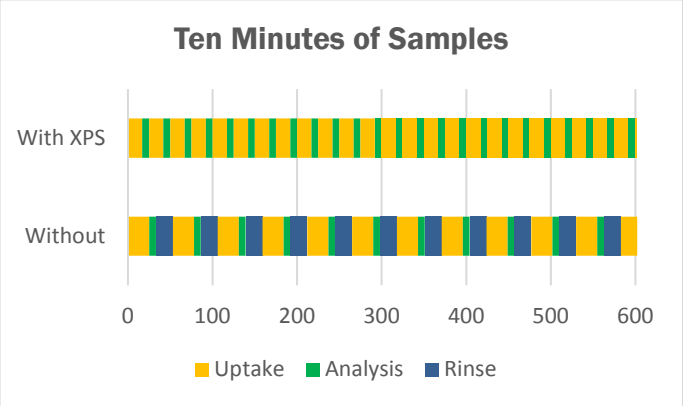

Figure 1: Analyzer Efficiency with and without *ASXPRESS PLUS* (XPS)

While speed is paramount, data quality cannot be ignored. Across all of the elements analyzed calibration coefficients of .9999 or greater were achieved. A few of these calibrations are shown on the next page in figure 2. This test was repeated three times and the R^2 values remained consistent. Just as we reused the same standards multiple times, many labs reuse their standards across multiple days. To assist with this, the Oils 7400 has the capability to stir standards before analysis. This ensures that the standards are well mixed, even if they were not prepared fresh.

Consistency is critical in not only our standard curve. To show the reproducibility of the system the same 50 ppm QC was run 30 times across three different sample batches. On the next page, figure 3 shows the consistency of the data for a number of analytes. From all of the elements being examined we saw excellent reproducibility with RSD values for the many repeats at less than 3%. Another avenue used to show consistency is our Cobalt Internal Standard. The intensity of the internal standard across all 60 plus samples and standards can be seen in figure 4. Across all of those samples, there was only one point where the IS came in a more than a 5% variation from the baseline. This low variation shows the consistency of both the analyzer and the sample preparation.

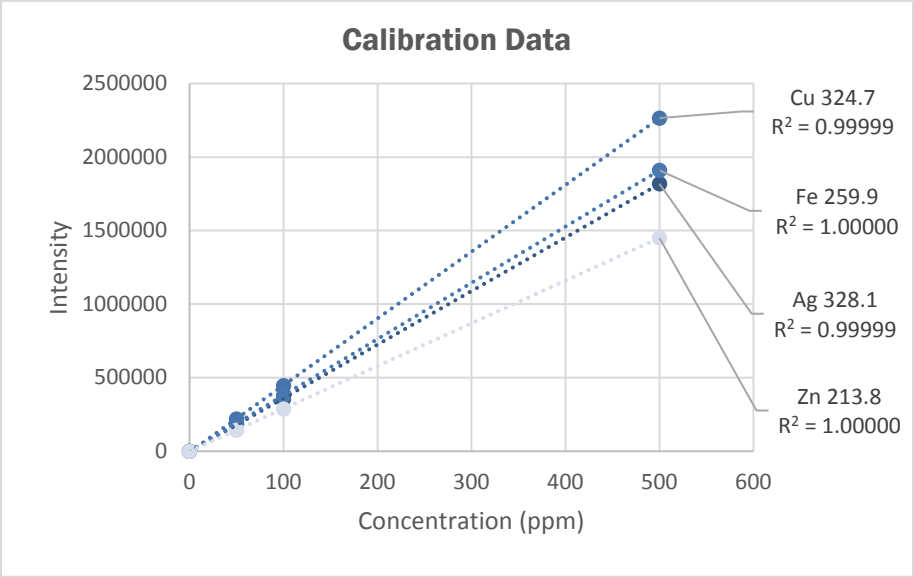

Figure 2: Calibration Data

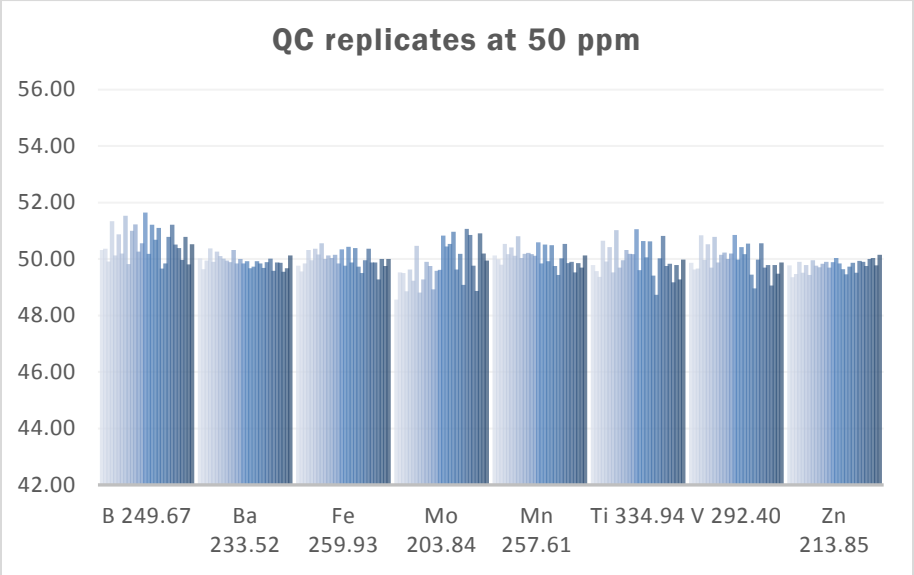

Figure 3: Reproducibility across 30 repeats

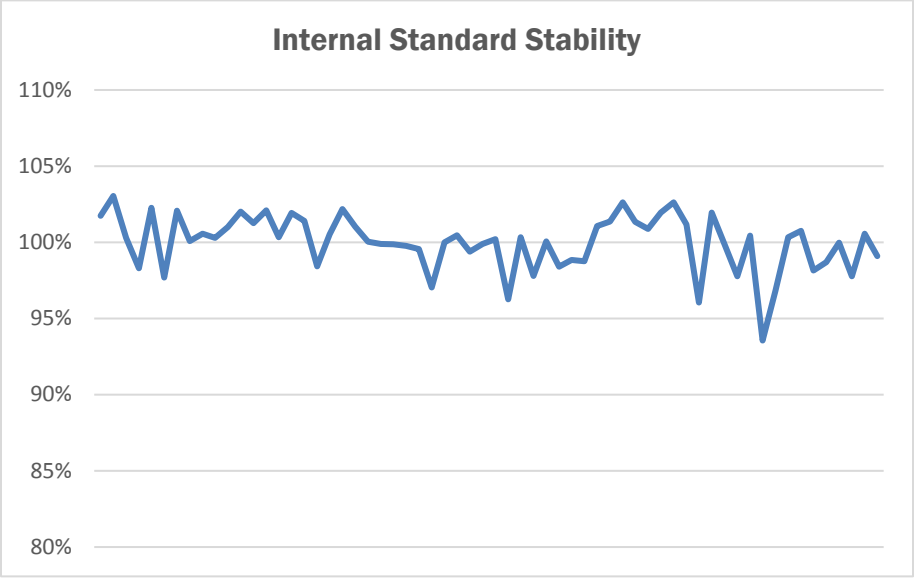

Figure 4: Internal Standard Stability

Another element of data quality which is critical and can sometimes be jeopardized when minimizing the sample run time is the sample to sample carryover. Table 2 shows the sample list for each batch. Three initial blanks provide our baseline for analyzing sample to sample carryover. Blank 4 was analyzed directly after the 500 ppm high standard, and the data from this was compared with the average value of blanks 1, 2, 3, 5, 7, and 8. While there was carryover at slightly elevated levels on Boron and Potassium all values came in under 1% and the average carryover was .1%. These levels could be lowered by adding additional rinse time, but they are acceptable for the wear metals industry.

Table 2: Sample List

Blank 1
Blank 2
Blank 3
50 ppm Standard
100 ppm Standard
500 ppm Standard
Blank 4
Blank 5
QC 1
QC 2
QC 3
QC 4
QC 5
QC 6
QC 7
QC 8
QC 9
QC 10
Blank 6
Blank 7
Blank 8

Conclusion

This work has demonstrated the ability of the Oils 7400 autosampler in conjunction with the *ASXPRESS PLUS* and the Avio 500 to meet the needs of a high throughput laboratory while upholding the standards of ASTM D5185. The specialized equipment is designed in every aspect to meet the challenges of oils samples and provide a clean, robust, and consistent sample introduction and analysis for the wear metals market.

References

1. ASTM D5185 “Standard Test Method for Multielement Determination of Used and Unused Lubricating Oils and Base Oils by Inductively Coupled Plasma Atomic Emission Spectrometry”, ASTM.
2. Hilligoss, D. “Analysis of In-Service Oils Following ASTM D5185 with the Avio 500 ICP-OES” PerkinElmer application note, 2017.

Automation Systems Used

Description	Part Number
Oils 7400 Homogenizing Autosampler	A74-99-1413HR
ASXPRESS PLUS Rapid Sample Introduction System	XPU-99-7026

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