APPLICATION NOTE

Single Particle Optical Sizing and ICP-OES

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A Novel Approach to Analysis of In-Service Lubricants for Particle Size/Count and Metal Content

Introduction

Lubricant contamination testing has continued to expand in its ability to measure, count and identify the impacts of smaller and lower concentrations of contaminants for in-service lubricants. With smaller and lower concentrations of particulates being identified as a root cause of mechanical wear, increasingly cleaner fluids are being recommended to maintain reasonable levels of wear. An increase in particulate levels during use of the lubricant can lead to failure of components or lubricant breakdown.

Particle counting is becoming a more routine tool for lubricant condition monitoring and maintenance programs to better identify particles of certain sizes and concentrations that can contribute to machine failure. In-service oils and other lubricants are monitored for particle size similarly to how they are monitored for elemental concentration. An increase in the ISO code (ISO 4406) for particle count or concentration of key wear metals indicates when maintenance is required.

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Previous work[1] has demonstrated the ability of PerkinElmer's Avio® 550 Max ICP-OES Oils system to measure in-service oil samples in accordance with ASTM method D5185[2] in a high-throughput environment. By adding the LPC 500™ liquid particle counter, in-line with the Avio 550 Max, oil and lubricant samples can be analyzed for both wear metals and particles all in one run.

This application discusses the hyphenation of an in-line particle counter to the ICP sampling system between the autosampler and the ICP nebulizer. This approach brings several benefits to particle counting by doing the measurements on diluted lubricants while not impacting the ICP analysis:

- High sample throughput for particle counting
- Two analyses, one run
- Reproducibility of the particle numbers
- Small footprint to save laboratory bench space
- Smaller sample size for particle counting

This is all achieved with the combination of the Avio 550 Max ICP-OES Oils system, LPC 500 liquid particle counter, Teledyne CETAC Oils 7400 dual homogenizing autosampler, and the Teledyne CETAC Particle Xpress rapid sample introduction system for oils.

Experimental

Samples and Sample Preparation

In-service lubricant samples were prepared by diluting 1:10 with Ultra Low Particulate (ULP) V-Solv™ + Co, which is refined kerosene that has been prefiltered to ensure low background counts of particles. This solvent comes pre-spiked with Cobalt (Co) at 40 ppm. The Co serves as an internal standard to correct for sample transport variations and matrix differences in the ICP analysis. Adding the internal standard into the diluent is the fastest, most accurate way to add it to all standards and samples equally. Since these samples will also be analyzed for particle content, the solvent must be free of particles to a level capable of supporting particle count results at the desired ISO codes. To provide the lowest level of background contamination, ULP V-Solv™ with Co added is available, which meets a cleanliness requirement for low baseline results for the >4, >6, and >14 µm particle sizes (Part No. N9308750 for five gallons). Please refer to the Consumables Used table at the end of this document for all consumable part numbers.

ICP quantitative measurements were made against external calibration curves prepared from three V-23 Oil Stock Solutions at 50, 100, and 500 ppm, along with a Metals Additive Oil Standard (MA4), which contains Ca at 5000 ppm, P and Zn at 1600 ppm each and Mg at 1600 ppm. The blank for the ICP results was 75 cST base oil. The QC standards consisted of the 50 ppm standard for all wear metals and the MA4 standard for the additive elements.

The LPC 500 Liquid Particle Counter is calibrated at the factory; recalibration is recommended once every six months with a secondary reference standard. A verification fluid for particle counting/sizing (Part No. N9308753) can be used to verify the calibration of the sensor from time to time.

Instrumental Conditions

All analyses were performed on the Avio 550 Max ICP-OES Oils system running in radial mode using the conditions in Table 1 and the elements and wavelengths in Table 2. The nebulizer flow was adjusted so that the tip of the green carbon "bullet" in the center of the plasma was just below the top flat plate.

Before samples were introduced into the Avio system, they passed through the LPC 500 liquid particle counter optical sensor that is mounted on the autosampler arm. Liquid particle counter parameters used for analysis of the particle count and size are listed in Table 3. The Oils 7400 dual homogenizing autosampler stirs the diluted samples just before analysis, ensuring that all samples are mixed and particles have not settled over time. Sample throughput is increased through the incorporation of a valve-and-loop system – a Particle Xpress sample introduction system was used with the parameters listed in Table 4. Overall throughput using this system for ICP and particle count analysis was measured at an average of 45 seconds per sample.

Table 1. Analytical parameters.

Parameter	Value
Nebulizer	GemCone™, Low Flow (Modified Babbington)
Spray Chamber	Baffled Glass Cyclonic
RF Power	1500 W
Torch	3-slot Avio Torch for Organics
Injector	1.2 mm Ceramic
Plasma Gas Flow	10 L/min
Aux. Gas Flow	0.8 L/min
Nebulizer Gas Flow	0.40 L/min
Torch Position	-4
Sample Uptake Rate	2.30 mL/min
Sample Uptake Tubing	Black/Black (0.76 mm id), SolvaFlex Flared Ends
Drain Tubing	Red/Red (1.14 mm i.d), SolvaFlex
Read Delay	21 sec
Replicates	2
Integration Range	0.2-2 sec

Table 2. Analytes and Wavelengths.

Analyte	Wavelength	Analyte	Wavelength
Ag	328.063	Mn	257.612
Al	394.398	Mo	203.845
B	249.675	Na	588.974
Ba	233.529	Ni	232.002
Ca	315.889	P	214.913
Cd	228.799	Pb	220.352
Co (int std)	228.613	Si	288.151
Cr	205.558	Sn	189.927
Cu	324.749	Ti	334.936
Fe	259.937	V	292.397
K	766.508	Zn	213.861
Mg	279.085		

Table 3. LPC 500 Liquid Particle Counter Parameters and Conditions.

Parameter	Value
Replicates	2
Time per Replicate	3 sec
Channels Measured	>4, >6, >14 µm
Data Delay	4 sec

Table 4. Particle Xpress and Oils 7400 Autosampler Parameters.

Parameter	Value
System Flowrate	20 mL/min
Total Diluted Sample Volume	4.0 mL per analysis
Backflush Rinse Volume	5.0 mL per analysis
Stir Speed	15%
Autosampler Peristaltic Pump Speed	25%

Results and Discussion

To assess the accuracy of the LPC 500, the verification fluid was analyzed periodically throughout an eight-hour analysis. Table 5 shows the expected particle counts per mL from the certificate of analysis (COA) along with the corresponding ISO 4406 code for the >4, >6 and >14 µm sizes.

Table 5. Verification Fluid COA Results and Corresponding ISO 4406 Code.

Particle Diameter (µm (c))	Particle Count (# of Particles/mL)	ISO 4406 Code
>4	12,540	21
>6	5,186	20
>14	440	16

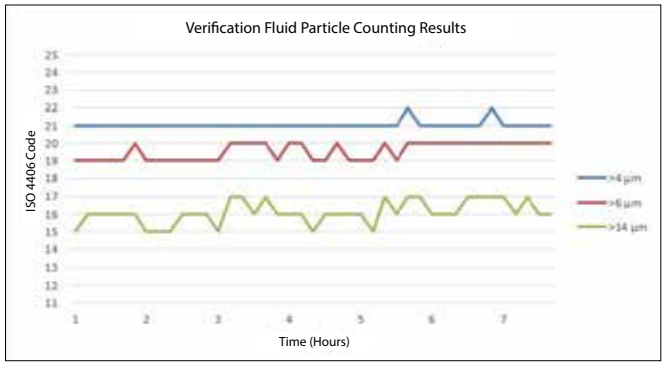

Figure 1. Accuracy for the particle counting analysis of the verification fluid, where the >4, >6, >14 µm results come within +/- 1 ISO code.

To assess stability of the LPC 500, a composite transmission and composite gear oil sample were split into 96 aliquots for measurement.

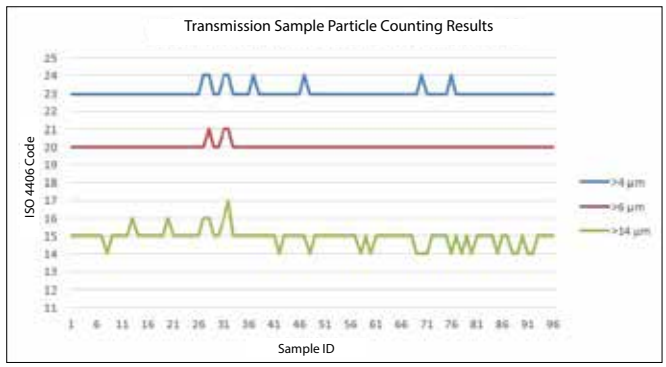

Figure 2. Stability for the particle counting analysis of the transmission oil sample, where both the >4 µm and >6 µm results come within +/- 1 ISO code and the >14 µm results are within one ISO code for all but one instance.

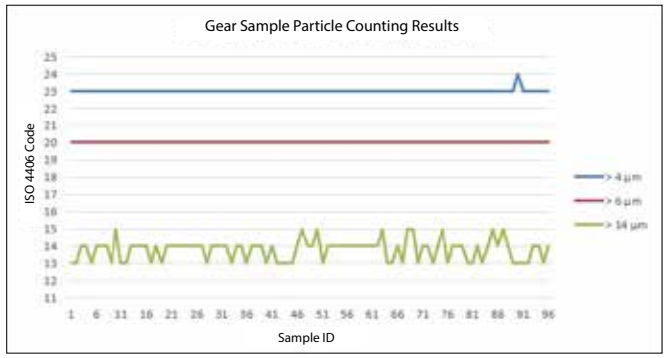

Figure 3. Stability for the particle counting analysis of the gear oil sample, where all of the >4 µm, >6 µm and >14 µm results come within +/- 1 ISO code.

ICP stability was also analyzed with QC analyzed throughout an eight-hour analysis. 50 ppm QC percent recovery is within 10% during an eight-hour analytical run of 576 in-service oil samples.

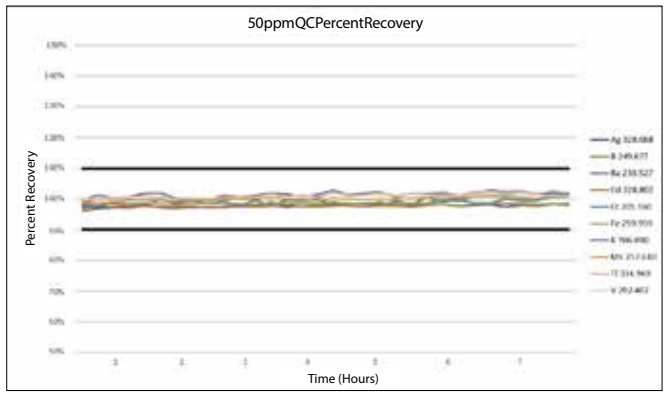

Figure 4. 50 ppm QC stability during an eight-hour analytical run of 576 in-service oil samples.

The results demonstrate the robustness and stability of the Avio 550 Max ICP-OES Oils system in accordance with the ASTM method D5185, and the LPC 500 liquid particle counter with the ISO 4406 method in a high-throughput environment.

Exporting the Results

Much like the customized template created for the elemental concentration results, the Data Manager application also includes a guided workflow to create an export template for the particle counting results. This template, along with the template created for the wear metals results, can later be used for automatic exporting at the end of the analysis. And since these templates can create files in various formats and data layouts, seamless uploading of the data to a LIMS can be achieved.

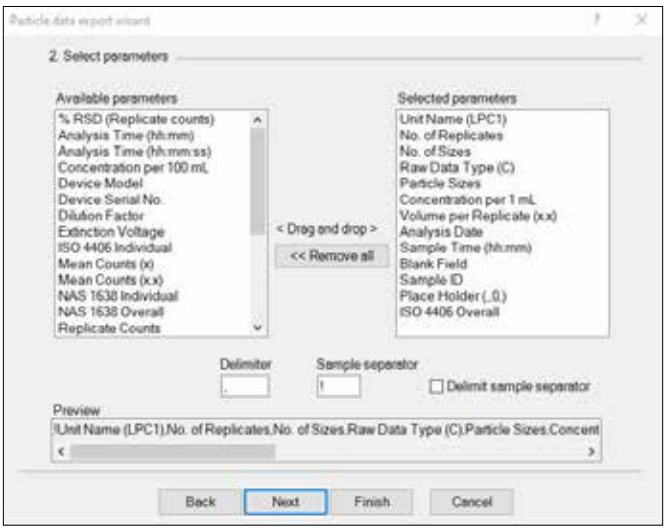

Figure 5. The Select Parameters step of the guided workflow for the particle data export uses drag-and-drop capabilities to select and order the various parameters needed in the export, while a preview area provides a rough idea as to how the export will look in a .txt format complete with delimiters and sample separators.

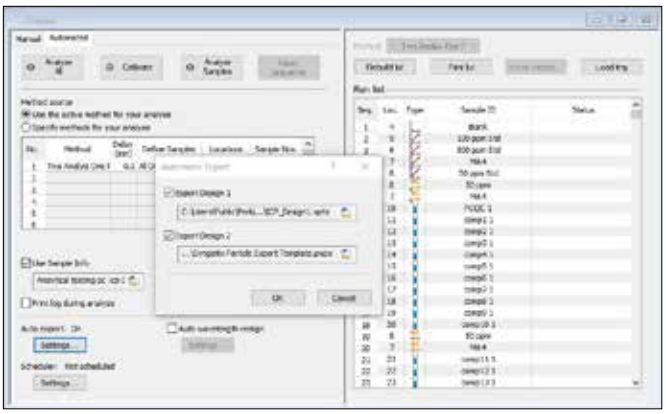

Figure 6. In the Analysis window, clicking on the Settings button allows the user to select two templates: one for elemental concentration results and the other for particle counting results. At the end of the analysis, the system will automatically export separate export files for the two types of results based on the selected templates.

Conclusions

By adding the LPC 500 liquid particle counter in-line with the Avio 550 Max ICP-OES Oils system, lubricant samples can be analyzed for both wear metals and particles all in one run with results repeatably within one ISO code for the >4, >6 µm sizes for transmission and gear oil. Throughput of 45 seconds on average per sample with less than 1 mL is achieved for both ICP as well as particle counting and sizing, all with the smallest automated particle counting system in the lubricants testing industry.

References

1. Hilligoss, D. "Analysis of In-Service Oils Following ASTM D5185 with the Avio 550 Max ICP-OES", PerkinElmer Application Note, 2020.
2. 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.

Consumables Used

Component	Part Number
Sample Uptake Tubing, Black/ Black (0.76 mm id.) SolvaFlex, Flared End	N0811914
Drain Tubing, Red/Red (1.14 mm id), SolvaFlex	09923037
Metal Additives Standard, MA4	N9308259 (100 g) N0776108 (200 g) N9308333 (400 g)
V-23 Wear Metals Standard, 50 µg/g	N9308243 (100 g) N0776104 (200 g) N9308317 (400 g)
V-23 Wear Metals Standard, 100 µg/g	N9308245 (100 g) N0776105 (200 g) N9308318 (400 g)
V-23 Wear Metals Standard, 500 µg/g	N9308249 (100 g) N0776106 (200 g) N9308320 (400 g)
UltraLowParticulateV-SolvSolvent+Co,ForDilution	N9308750 (5 gal)
Ultra Low Particulate V-Solv Solvent, For Rinse	N9308749 (5 gal)
Parti-Count™ Particle Count Verification Fluid	N9308753 (400 g)

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