

Challenge

multi-element analysis of used and unused lubricating and base oils to evaluate contamination, wear, and overall equipment and lubricant condition

Solution

HR ICP-OES providing interference-free, accurate elemental analysis of lubricating oils after simple dilution in an organic solvent

Intended audience

Lubricant manufacturers and industrial, automotive, refinery, machinery, and power-generation labs needing routine elemental screening of lubricants

Analysis of new and used lubricating oils by high-resolution ICP-OES according to ASTM D5185

Introduction

Lubricating oils are the lifeblood of oil-wetted machinery. Elements such as barium, boron, calcium, copper, magnesium, molybdenum, phosphorus, sulfur, and zinc are used as additives in lubricating oils to improve their lubricating capability and properties, such as anti-wear, antioxidant, anticorrosive, and dispersing. The quantity of those elements highly affects a lubricant's performance. Therefore, a rapid, sensitive, and precise measurement is crucial as part of its production. For machinery monitoring and maintenance, used lubricating oil analysis closely resembles a blood test to determine the health of a machine. Elemental analysis of wear metals, contaminants and additives is an important component of oil condition analysis. It provides an efficient diagnostic tool for scheduling oil changes and predictive maintenance. Thus, routine elemental analysis helps improve profitability by increasing machinery's reliability, availability, and productivity.

Elemental analysis can be conducted with X-ray fluorescence, atomic absorption spectrometry (AAS), rotating electrode optical emission spectrometry (RDE-OES), or inductively coupled plasma optical emission spectrometry (ICP-OES). ICP-OES is a powerful tool for elemental analysis because of its capabilities for quick multi-elemental analysis, high sensitivity, and a wide linear dynamic analysis range. This prowess makes ICP-OES the prescribed method for new and used oil analysis in D5185 method. The challenges for ICP-OES, however, are lubricating oil's high viscosity, heterogeneity, and matrix complexity. These problems can be circumvented by diluting the sample with a less viscous solvent and applying a suitable sample introduction system, however at the cost of higher detection limits. The high organic load may cause plasma instability and can lead to soot formation on the torch injector, which requires immediate cleaning, causing downtime. In addition, spectral interferences can have a dramatic impact on the quality of

the baseline fitting routines, resulting in inferior precision and detection limits. As such, oil analysis by ICP-OES requires an instrument that offers excellent plasma performance, exceptional spectral resolution, and high sensitivity.

The Analytik Jena PlasmaQuant 9200 Elite is well-suited for cost-effective oil analysis, with its robust plasma, exceptional resolution, and unique sensitivity. The outstanding analytical capabilities arise from its four key components. First, the robust plasma can easily tolerate high organic sample loadings and provide long-term stability. Secondly, the highest optical resolution of 2 pm at 200 nm due to the unique double-echelle monochromator optics, combined with a charge-coupled device (CCD) detector provide supreme spectral resolution, which allows interference-free measurement by well-separated lines. The CCD detector captures emission line and background intensities simultaneously, which enables the automatic background correction. Thirdly, the vertical plasma geometry of the V Shuttle Torch makes soot formation and memory

effects a relic of the past. In routine analysis, the V Shuttle torch design makes maintenance effortless and reduces instrument downtime and consumable costs. Finally, yet importantly, the DualView PLUS plasma observation will improve the productivity of oil analysis significantly, because trace elements and major constituents can now be quantified from one sample measurement without tedious sample preparation.

Here, we present an analysis of lubricant oils performed using the ASTM D5185 method, which is designed for the "multielement determination of used and unused lubricating oils and base oils by inductively coupled plasma atomic emission spectrometry (ICP-AES)". This method is designed for both used and unused oil samples and can identify 22 additives, wear metals and contaminants. It is generally used as a rapid diagnostic method to determine contamination levels, wear rates and the overall condition of equipment and lubricants.

Materials and Methods

Reagents

The following chemicals were used for standards and sample preparation:

- CONOSTAN® Standard S21 multi-elemental organometallic standards at 20, 100 and 900 mg/kg (SCP Science)
- 10000 mg/kg yttrium oil-based standard (SCP Science)
- 75 cSt blank oil (SCP Science)
- custom QC 1, 2 and 3 sample (LGC Group) and mixed xylenes (Fisher Scientific)

Sample preparation

Sample preparation was designed to be simple, reducing sample viscosity and minimizing the potential interferences. All samples and stock standards were homogenized in a heated ultrasonic bath for 30 minutes at 60 °C. Yttrium was used as the internal standard. The yttrium standard was diluted with mixed xylenes to obtain a final concentration of 2 mg/kg. This yttrium solution was used as a diluent for all samples and standards. To minimize the viscosity

difference between samples and standards, blank oil was added if needed, so that all the final solution contained 10% oil by weight. A blank was prepared by diluting the blank oil in the yttrium solution. All the standards were prepared by diluting the stock standards by weight to give the required concentration. The oil samples and quality control (QC) samples were diluted ten-fold by weight.

Calibration

For the D5185 method, the concentrations of standards are listed in Table 1, together with the coefficient of correlation (R2) and method detection limits. Linear calibrations were obtained with correlation coefficient greater than 0.9997 for all elements. Selected calibration curves for the methods are shown in Figure 1. ASTM D5185 expects detectability in the low mg/kg range for most elements to be sufficient. For most elements, the method detection limits (MDL) are below 5 µg/kg with the PlasmaQuant 9200 Elite in radial view.

Table 1: Calibration standards, coefficient of correlation (R2) and method detection limits (MDLs)

| Element | Cal.0 [mg/kg] | Cal.1 [mg/kg] | Cal.2 [mg/kg] | Cal. 3 [mg/kg] | Cal. 4 [mg/kg] | Cal. 5 [mg/kg] | Cal. 6 [mg/kg] | Cal. 7 [mg/kg] | R2 | MDL [mg/kg] |
|---------|------------------|------------------|------------------|-------------------|-------------------|-------------------|-------------------|-------------------|--------|----------------|
| Ag | 0 | 0.5 | 1 | 5 | 10 | 25 | 50 | | 1.0000 | 0.0016 |
| Al | 0 | 0.5 | 1 | 5 | 10 | 25 | 50 | | 1.0000 | 0.0043 |
| B | 0 | 0.5 | 1 | 5 | 10 | 25 | 50 | | 0.9999 | 0.0019 |
| Ba | 0 | 0.5 | 1 | 5 | 10 | 25 | 50 | | 1.0000 | 0.0026 |

| Element | Cal.0 [mg/kg] | Cal.1 [mg/kg] | Cal.2 [mg/kg] | Cal. 3 [mg/kg] | Cal. 4 [mg/kg] | Cal. 5 [mg/kg] | Cal. 6 [mg/kg] | Cal. 7 [mg/kg] | R2 | MDL [mg/kg] |
|---------|---------------|---------------|---------------|----------------|----------------|----------------|----------------|----------------|--------|-------------|
| Ca | 0 | 0.5 | 1 | 5 | 10 | 25 | 50 | 81.5 | 1.000 | 0.0033 |
| Cd | 0 | 0.5 | 1 | 5 | 10 | 25 | 50 | | 1.0000 | 0.0016 |
| Cr | 0 | 0.5 | 1 | 5 | 10 | 25 | 50 | | 0.9999 | 0.0017 |
| Cu | 0 | 0.5 | 1 | 5 | 10 | 25 | 50 | | 1.0000 | 0.0020 |
| Fe | 0 | 0.5 | 1 | 5 | 10 | 25 | 50 | | 0.9999 | 0.0009 |
| Mg | 0 | 0.5 | 1 | 5 | 10 | 25 | 50 | | 0.9999 | 0.0006 |
| Mn | 0 | 0.5 | 1 | 5 | 10 | 25 | 50 | | 0.9996 | 0.0002 |
| Mo | 0 | 0.5 | 1 | 5 | 10 | 25 | 50 | | 1.0000 | 0.0026 |
| Na | 0 | 0.5 | 1 | 5 | 10 | 25 | 50 | | 0.9998 | 0.0140 |
| Ni | 0 | 0.5 | 1 | 5 | 10 | 25 | 50 | | 1.0000 | 0.0021 |
| P | 0 | 0.5 | 1 | 5 | 10 | 25 | 50 | | 0.9999 | 0.0278 |
| Pb | 0 | 0.5 | 1 | 5 | 10 | 25 | 50 | | 1.0000 | 0.0056 |
| Si | 0 | 0.5 | 1 | 5 | 10 | 25 | 50 | | 1.0000 | 0.0024 |
| Sn | 0 | 0.5 | 1 | 5 | 10 | 25 | 50 | | 0.9999 | 0.0057 |
| Ti | 0 | 0.5 | 1 | 5 | 10 | 25 | 50 | | 0.9999 | 0.0010 |
| V | 0 | 0.5 | 1 | 5 | 10 | 25 | 50 | | 0.9999 | 0.0016 |
| Zn | 0 | 0.5 | 1 | 5 | 10 | 25 | 50 | | 0.9999 | 0.0005 |

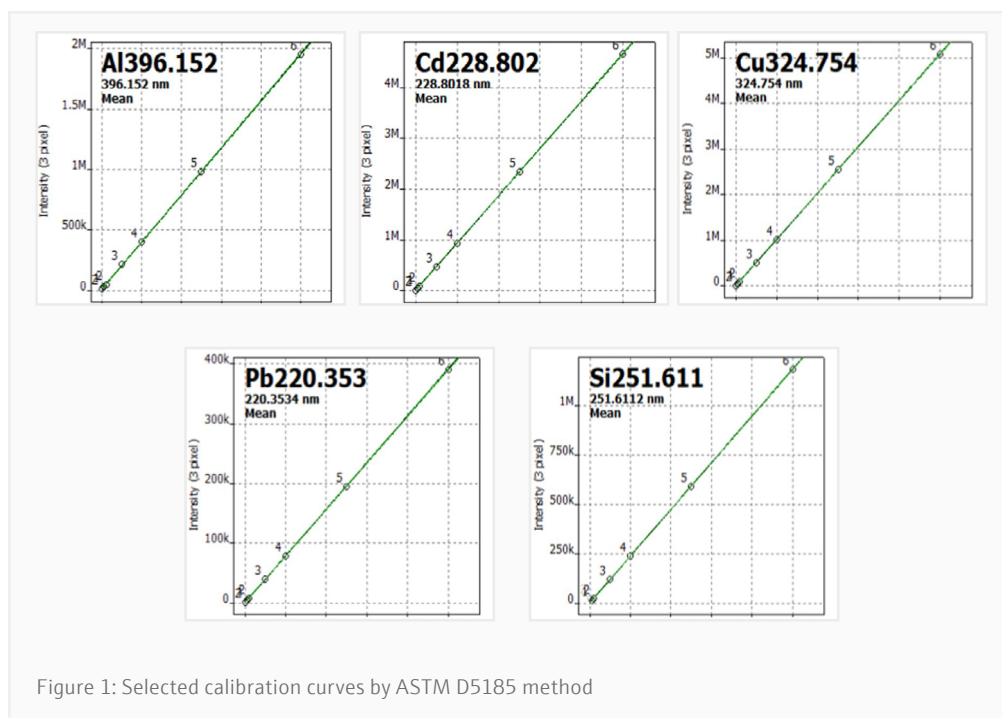


Figure 1: Selected calibration curves by ASTM D5185 method

Instrument settings

The PlasmaQuant 9200 Elite, equipped with Organic Sample Introduction Kit, was used for this analysis. Due to Ne correction and stable plasma generation, only 10 minutes of warm up time was needed before the sample analysis. No oxygen was added to the plasma. Adjusted plasma settings for the predecessor instrument are also given.

A Teledyne CETAC Oils 7400 Homogenizing Dual Matrix Autosampler was used to guarantee good homogeneity of the samples. The parameters are listed in Table 2.

Method parameters

In the ASTM D185 method, analysis wavelengths are suggested as a guideline. With the exceptional spectral resolution of the Analytik Jena PlasmaQuant 9200 Elite, the most sensitive line can be selected without any concern for interference. In the ASpect PQ software, the automatic background correction (ABC) function was applied for data evaluation. The ABC function automatically fits a baseline to the spectral background during the sample analysis. The evaluation parameters for are listed in Table 3.

Table 2: Instrument settings

| Parameter | Setting 9200 Elite | Setting 9100 Elite |
|------------------------|--|--------------------|
| RF power | 1450 W | 1400 W |
| Plasma gas flow | 10 L/min | 15 L/min |
| Nebulizer gas flow | 0.45 L/min | 0.35 L/min |
| Auxiliary gas flow | 0.6 L/min | |
| Spray chamber | borosilicate cyclonic spray chamber with dip tube, 50 mL | |
| Injector | quartz, inner diameter 1 mm | |
| Nebulizer | borosilicate concentric nebulizer, 1 mL/min | |
| Outer tube/ inner tube | quartz/quartz | |
| Pump tubing | Viton (black/black) | |
| Sample pump rate | 1 mL/min | |
| Read delay | 40 s | |
| Rinse time | 45 s | |
| Torch position | -2 mm | |

Table 3: Overview of evaluation parameters

| Element | Wavelength [nm] | Plasma view | Integrati-on mode | Replicates | Read time [s] | Evaluation | | | |
|---------|-----------------|-------------|-------------------|------------|---------------|--------------|--------------|--------------------|------------|
| | | | | | | No. of pixel | Baseline fit | Poly-nomial degree | Correction |
| Ag | 328.068 | radial | peak | 3 | 1 | 3 | ABC | auto | Y |
| Al | 396.152 | radial | peak | 3 | 1 | 3 | ABC | auto | Y |
| B | 249.773 | radial | peak | 3 | 1 | 3 | ABC | auto | Y |
| Ba | 455.403 | radial | peak | 3 | 1 | 3 | ABC | auto | Y |
| Ca | 317.933 | radial | peak | 3 | 1 | 3 | ABC | auto | Y |
| Cd | 228.802 | radial | peak | 3 | 1 | 3 | ABC | auto | Y |
| Cr | 267.716 | radial | peak | 3 | 1 | 3 | ABC | auto | Y |
| Cu | 324.754 | radial | peak | 3 | 1 | 3 | ABC | auto | Y |
| Fe | 238.204 | radial | peak | 3 | 1 | 3 | ABC | auto | Y |
| Mg | 285.210 | radial | peak | 3 | 1 | 3 | ABC | auto | Y |
| Mn | 257.610 | radial | peak | 3 | 1 | 3 | ABC | auto | Y |
| Mo | 202.030 | radial | peak | 3 | 1 | 3 | ABC | auto | Y |
| Na | 589.590 | radial | peak | 3 | 1 | 3 | ABC | auto | Y |
| Ni | 221.648 | radial | peak | 3 | 1 | 3 | ABC | auto | Y |
| P | 178.224 | radial | peak | 3 | 1 | 3 | ABC | auto | Y |
| Pb | 220.353 | radial | peak | 3 | 1 | 3 | ABC | auto | Y |

| Element | Wavelength [nm] | Plasma view | Integrati-on mode | Replicates | Read time [s] | Evaluation | | | |
|---------|-----------------|-------------|-------------------|------------|---------------|--------------|--------------|--------------------|------------|
| | | | | | | No. of pixel | Baseline fit | Poly-nomial degree | Correction |
| Si | 251.611 | radial | peak | 3 | 1 | 3 | ABC | auto | Y |
| Sn | 189.927 | radial | peak | 3 | 1 | 3 | ABC | auto | Y |
| Ti | 334.941 | radial | peak | 3 | 1 | 3 | ABC | auto | Y |
| V | 309.311 | radial | peak | 3 | 1 | 3 | ABC | auto | Y |
| Zn | 213.856 | radial | peak | 3 | 1 | 3 | ABC | auto | Y |
| Y | 371.030 | radial | peak | 3 | 1 | 3 | ABC | auto | - |

Results and Discussion

Quality control (QC) samples, used oil samples, and spiked samples were analyzed. The results are shown in Table 4. Excellent precision and recovery rates of all QC and spiked samples were obtained with the PlasmaQuant 9200 Elite ICP-OES. All QC and spike recovery values are within 5% of the expected values. To achieve consistent and precise results and reduce the need for recalibration or remeasurement of samples, the ICP-OES system must provide good long-term stability. Long-term stability was tested by measuring lubricating oil samples and QC 4 with continuous aspiration over a period of 10 hours. QC 1 was analyzed every 5 samples and no recalibration was performed. For all the elements, the recovery of QC 1 was within 5% of the certified value, with RSD smaller than 2.5% (Figure 2 and 3). Result consistency was investigated by analyzing two different emission lines for each element. Even lines with a high risk of spectral interferences, such as the P/Cu line pair at 213.9 nm, can be used for correct quantification, since they are baseline separated by the high-resolution optics of the PlasmaQuant 9200 Elite (Figure 4). There was no soot formation over this period even without applying oxygen, thus, no cleaning of injector, torch and entrance window was needed. Those results demonstrate that the PlasmaQuant 9200 Elite ICP-OES can be easily used for routine oil analysis with excellent plasma and signal stability.

Table 4: Sample analysis results by ASTM D5185 method

| Element | QC3 | | Used oil 1 | | | Used oil 2 | | |
|---------|---------------|--------------|---------------|----------------------|--------------|---------------|----------------------|--------------|
| | Conc. [mg/kg] | Recovery [%] | Conc. [mg/kg] | Spiked conc. [mg/kg] | Recovery [%] | Conc. [mg/kg] | Spiked conc. [mg/kg] | Recovery [%] |
| Ag | 3 | 103 | 0.12 | 5 | 100 | <MDL | 5 | 103 |
| Al | 3 | 104 | 2.63 | 5 | 99 | 17.91 | 5 | 101 |
| B | 3 | 105 | 15.66 | 25 | 105 | 22.4 | 25 | 104 |
| Ba | 3 | 102 | 0.38 | 5 | 101 | 0.84 | 5 | 102 |
| Ca | 3 | 101 | 1355.10 | 25 | 99 | 1566.8 | 25 | 95 |
| Cd | 3 | 99 | 0.14 | 5 | 97 | <MDL | 5 | 95 |
| Cr | 3 | 102 | 1.14 | 5 | 99 | 1.48 | 5 | 100 |
| Cu | 3 | 103 | 0.41 | 5 | 98 | 6.48 | 5 | 100 |
| Fe | 3 | 101 | 0.45 | 5 | 98 | 9.38 | 5 | 99 |
| Mg | 3 | 101 | 3.38 | 5 | 98 | 10.71 | 5 | 95 |
| Mn | 3 | 101 | <MDL | 5 | 103 | <MDL | 5 | 103 |
| Mo | 3 | 102 | 0.12 | 5 | 100 | 377.46 | 5 | 105 |

| Element | QC3 | | Used oil 1 | | | Used oil 2 | | |
|---------|---------------|--------------|---------------|----------------------|--------------|---------------|----------------------|--------------|
| | Conc. [mg/kg] | Recovery [%] | Conc. [mg/kg] | Spiked conc. [mg/kg] | Recovery [%] | Conc. [mg/kg] | Spiked conc. [mg/kg] | Recovery [%] |
| Na | 3 | 99 | 0.03 | 5 | 102 | 4.22 | 5 | 99 |
| Ni | 3 | 102 | <MDL | 5 | 98 | <MDL | 5 | 100 |
| P | 3 | 98 | 268.83 | 25 | 101 | 321.00 | 5 | 104 |
| Pb | 3 | 101 | 0.06 | 5 | 96 | 7.61 | 5 | 97 |
| Si | 3 | 95 | 2.76 | 5 | 96 | 5.37 | 5 | 96 |
| Sn | 3 | 102 | <MDL | 5 | 99 | <MDL | 5 | 101 |
| Ti | 3 | 104 | 0.35 | 5 | 100 | 0.06 | 5 | 102 |
| V | 3 | 102 | 0.83 | 5 | 99 | 0.50 | 5 | 99 |
| Zn | 3 | 101 | 305.07 | 25 | 99 | 378.80 | 25 | 100 |

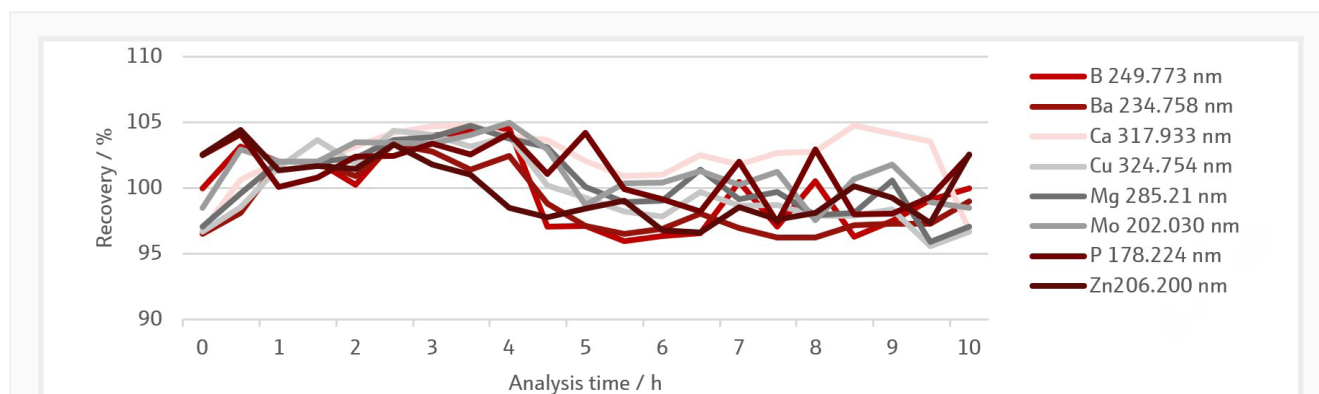


Figure 2: Percentage recoveries of QC 1 sample during a 10-hour measurement period

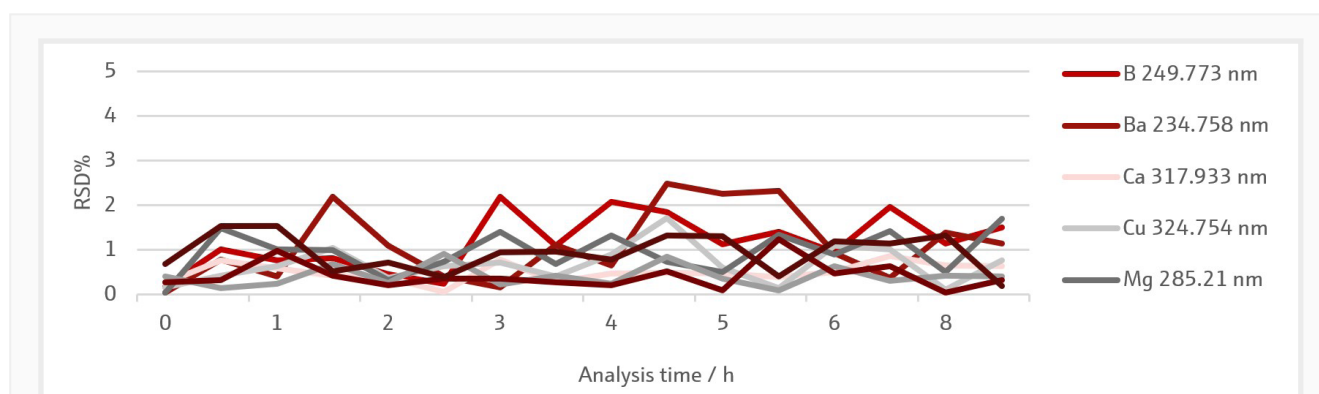
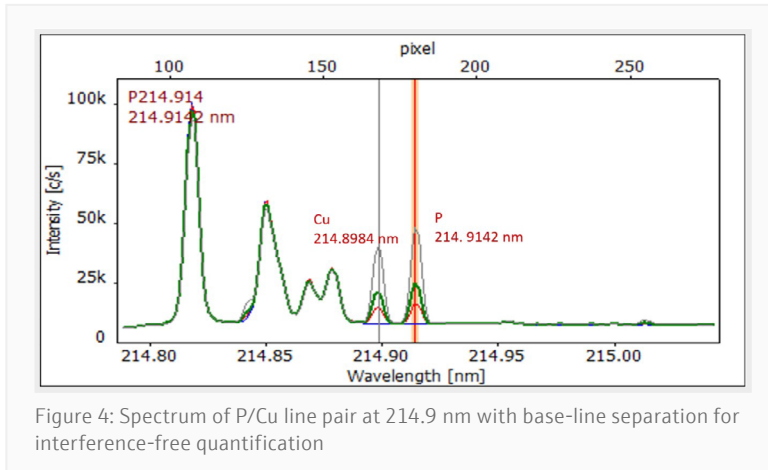
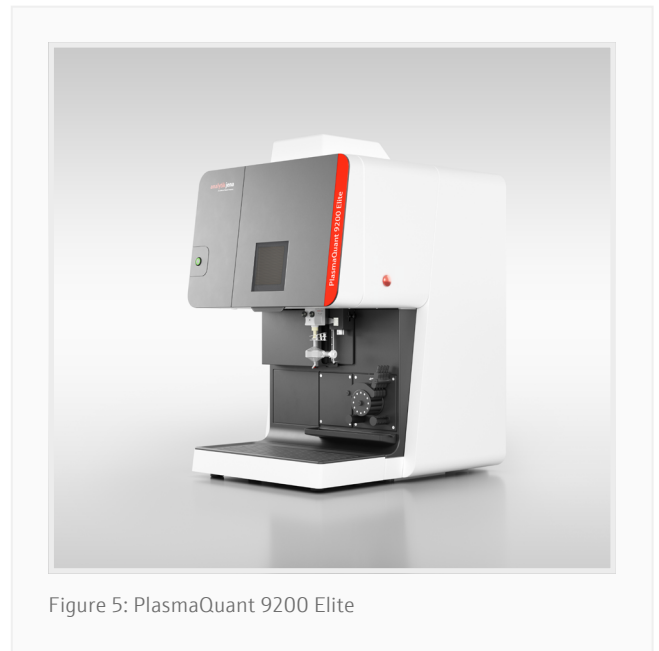


Figure 3: RSD of QC 1 sample during a 10-hour measurement period



Summary

The exceptional precision and accuracy in lubricating oil analysis has been demonstrated by Analytik Jena's PlasmaQuant 9200 Elite ICP-OES in this study. The PlasmaQuant 9200 Elite can easily fulfill the requirements of ASTM 4951 and D5185 methods. The robust plasma and the high matrix tolerance make PlasmaQuant 9200 Elite the ideal instrument for routine oil analysis. The unique V shuttle torch minimizes maintenance due to minimal carbon deposition. High resolution optics easily separate analytical lines from interferences, such as carbon-based emission. In summary, the PlasmaQuant 9200 Elite ICP-OES provides an easy solution for evaluating lubricating oil accurately, effectively and timely.



Recommended device configuration

Table 5: Overview of devices, accessories, and consumables

| Article | Article number | Description |
|----------------------------------|----------------|---|
| PlasmaQuant 9200 Elite | 818-09201-2 | High resolution ICP-OES |
| ORGANIC KIT for PlasmaQuant 9200 | 810-88562-0 | Sample introduction kit for organic samples |
| Teledyne Cetac Oils 7400 | 810-88122-0 | Autosampler for oil samples with stirring functionality, dual port rinse station and drip capture |

References

- [1] ASTM D5185-13(2018), Standard Test Method for Multielement Determination of Used and Unused Lubricating Oils and Base Oils by Inductively Coupled Plasma Atomic Emission Spectrometry (ICP-AES)

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