Application Brief

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# Improved Measurement Accuracy of Wear Metals in Lubricant Oils by ICP-OES

## Following ASTM D5185-18 method using an Agilent 5800 RV ICP-OES and IntelliQuant

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## Get the right answer the first time

The Agilent 5800 Radial View (RV) ICP-OES incorporates smart capabilities, including IntelliQuant, Outlier Conditional Formatting (OCF), and Early Maintenance Feedback (EMF), that deliver deeper insight into methods, samples, and operational status. This insight enables new proactive approaches to reduce remeasurement and downtime, bringing you greater confidence in your results.

Identifying wear metals in lubricant oils can prevent damage to important equipment. Many labs use ASTM D5185-18 standard method for the rapid determination of 22 elements in lubricating oils. Although the ICP-OES method is well-established, labs sometimes need to remeasure samples. In one case, an oils analysis lab had problems with carbon forming in the torch during analysis by ICP-OES. Replacing the torch with an Agilent Easy-fit fully demountable torch for oil analysis reduced the carbon build-up.

In another example, a small contracting lab specializing in the analysis of lubricating oils occasionally received specialty oils containing a high Mo content. Colloidal Mo compounds are often used as antifriction additives in lubricant oils and greases. At high concentrations, Mo will interfere with other elements at the typical emission lines specified in ASTM D5185-18. IntelliQuant is a software function that can accurately identify potential interferences on analytical wavelengths. The function will suggest the most appropriate wavelength based on the elements present in the sample, without remeasuring the samples.

## Automatic flagging of outlying results

The OCF feature in the ICP Expert software makes it quick and easy to find potentially problematic results in a large data set. To simulate an oil sample containing a high concentration of Mo, engine oil samples were spiked at 250 mg/kg and analyzed using IntelliQuant. As shown in Figure 1, OCF highlighted inconsistencies in the results between three Si wavelengths, indicating a potential interference on one of the wavelengths.

| Rack:Tube | Solution Label | Outlier Summary | Si 251.432 nm ppm | Si 251.611 nm ppm | Si 288.158 nm ppm | V 309.310 nm ppm | V 311.837 nm ppm |
|---|---|---|---|---|---|---|---|
| 3:24 | EngineOil-A21_Spkd-1 | F | 2.9625 | 2.9717 | 2.9796 | 2.3414 | 2.3338 |
| 3:24 | EngineOil-A21_Spkd-2 | F | 2.9865 | 2.9878 | 3.0141 | 2.3536 | 2.3380 |
| 3:24 | EngineOil-A21_Spkd-3 | F | 3.0333 | 3.0252 | 3.0538 | 2.3683 | 2.3503 |
| 3:25 | EngineOil-A21_Mo10_Spkd-1 | F | 2.9581 | 3.0367 | 3.0053 | 2.3503 | 2.3314 |
| 3:25 | EngineOil-A21_Mo10_Spkd-2 | F | 2.9561 | 3.0136 | 2.9878 | 2.3138 | 2.2879 |
| 3:25 | EngineOil-A21_Mo10_Spkd-3 | F | 2.9801 | 3.0195 | 2.9881 | 2.3302 | 2.3055 |
| 3:26 | EngineOil-A21_Mo100_Spkd-1 | F | 2.9539 | 3.5467 | 2.9931 | 2.3196 | 2.2878 |
| 3:26 | EngineOil-A21_Mo100_Spkd-2 | F | 2.9382 | 3.5247 | 2.9709 | 2.3009 | 2.2674 |
| 3:26 | EngineOil-A21_Mo100_Spkd-3 | F | 2.9704 | 3.5642 | 2.9952 | 2.2982 | 2.2720 |
| 3:27 | EngineOil-A21_Mo250_Spkd-1 | F | 2.9277 | 4.3820 | 2.9956 | 2.2628 | 2.2340 |
| 3:27 | EngineOil-A21_Mo250_Spkd-2 | F | 2.9004 | 4.3732 | 3.0015 | 2.2350 | 2.2110 |
| 3:27 | EngineOil-A21_Mo250_Spkd-3 | F | 2.9986 | 4.4219 | 3.0785 | 2.2438 | 2.2083 |

**Figure 1.** Flagging of Mo-spiked oil sample results informs the user of a difference in the Si concentrations determined using the different wavelengths.

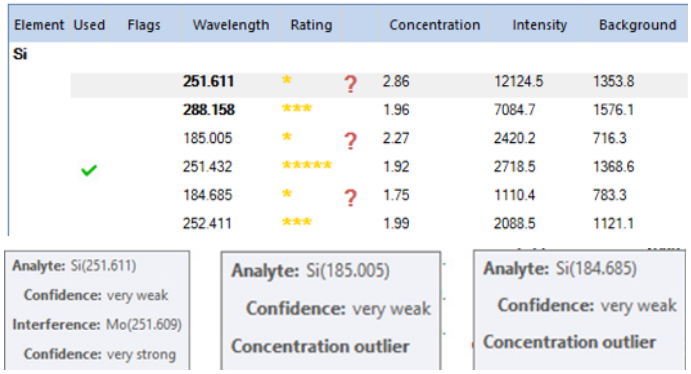


**Figure 2.** IntelliQuant ranks different emission wavelengths for the same element using a star-ranking system. Hovering over the "?" symbol displays reasons for the poor rating on a wavelength.

IntelliQuant uses data analytics to rank different emission wavelengths for the same element and identifies likely interferences. By hovering over the "?" symbol next to three Si lines, IntelliQuant identified an interference on Si 251.611 from Mo 251.609, and Si 184.685 and Si 185.005 were considered concentration outliers (Figure 2). IntelliQuant suggested 251.432 nm as the best wavelength to use for the quantitative analysis of Si, indicated by the highest confidence ranking (five stars).

## Oil SRM recoveries

The 5800 RV ICP-OES fitted with an integrated AVS 7 valve system was used for the fast analysis of lubricating oil samples following ASTM D5185-18. The sample introduction system included the Easy-fit fully demountable RV torch fitted with a removable 1.4 mm i.d. quartz injector. NIST 1085c Wear Metals in Lubricant Oil SRM was used to validate the method. As shown in Table 1, recoveries for all elements were within ±10% of the certified or reference values. The data shows the effectiveness of the 5800 RV ICP-OES for the analysis of wear metals and additives in lubricating oils according to the ASTM method.

## Minimizing downtime

To keep the 5800 running over long periods, smart EMF diagnostics and counters alert the analyst when maintenance is needed. Ensuring peak instrument performance minimizes QC failures and reduces the need for sample reruns.

**Table 1.** Recoveries of elements in oil SRM.

| Element and Wavelength (nm) | Certified Concentration (mg/kg) | Measured Concentration (mg/kg) | Recovery % |
|---|---|---|---|
| Ag 328.068 | 298 | 295 | 99 |
| Al 167.019 | 292 | 309 | 106 |
| B 249.678 | 304 | 280 | 92 |
| Ba 233.527 | 306 | 289 | 94 |
| Ca 422.673 | 299 | 305 | 102 |
| Cd 214.439 | 301 | 311 | 103 |
| Cr 267.716 | 302 | 293 | 97 |
| Cu 324.754 | 298 | 307 | 103 |
| Fe 259.940 | 301 | 299 | 99 |
| K 766.491 | 295 | 303 | 103 |
| Mg 280.270 | 300 | 294 | 98 |
| Mn 257.610 | 299 | 300 | 100 |
| Mo 202.032 | 299 | 296 | 99 |
| Na 588.995 | 300 | 299 | 100 |
| Ni 231.604 | 306 | 289 | 94 |
| P 177.434 | 304 | 286 | 94 |
| Pb 220.353 | 303 | 293 | 97 |
| Si 251.432 | 293 | 286 | 98 |
| Sn 189.925 | 298 | 289 | 97 |
| Ti 334.941 | 300 | 302 | 101 |
| V 311.837 | 285* | 288 | 101 |
| Zn 213.857 | 285 | 275 | 97 |

**Reference value*

Full details of this application can be found in Agilent publication 5994-1671EN.

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