

Improved Accuracy in the Measurement of Wear Metals and Additives in Lubricant Oils by ICP-OES

Using an Agilent 5800 Radial View ICP-OES and the ASTM D5185-18 standard method



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Introduction

Most types of machinery need some form of lubrication to work effectively and reliably over their lifetime, and to slow down wear and tear. Depending on the specific equipment and its operating conditions, additives are added to base lubricating or hydraulic oils to improve their properties. Lubricating oil manufacturers routinely assess the metal content of base oils and lubricants, as well as the homogeneity of any additive-blends. Equipment-owners and operators also regularly monitor the elemental profile of oils in service, as the elemental profile of the oils changes as machinery is used. This analysis is important for predictive/preventive maintenance and trend analysis. If certain metals are identified in the oil or are seen to be changing in concentration over time, action can be taken before the equipment is damaged or fails.

Standard method ASTM D5185-18 is used by laboratories around the world for the rapid determination of 22 elements in used and unused lubricating and base oils (1).

The method uses ICP-OES for the determination of additive elements, wear metals, and contaminants. Many laboratories that use this method process hundreds of samples per day, so achieving high sample throughput is critical. They also want to reduce the time spent remeasuring samples, so analysts can focus on more productive tasks and laboratories can minimize rework costs.

Typically, samples are remeasured because of an instrument-related problem or sample-related problem. To prevent the most common causes of having to remeasure a sample, the Agilent 5800 and 5900 ICP-OES use smart capabilities that deliver deeper insight into samples, processes, and operational status. This insight enables completely new and proactive approaches to reducing remeasurement and downtime, bringing greater confidence in both instrument operation and the results.

In this study, a series of used lubricant and hydraulic oil samples were analyzed using an Agilent 5800 Radial View (RV) ICP-OES. The IntelliQuant software routine was used to identify interferences and suggest the most appropriate wavelength for the analysis. The extra information provided by IntelliQuant ensures that high-quality results are produced first time, without rerunning samples or standards.

Agilent ICP Expert smart software tools

Agilent ICP Expert software developed for the Agilent 5900 and 5800 ICP-OES includes smart tools to simplify method development, verify results, and minimize sample remeasurements for this type of analysis.

- IntelliQuant uses data analytics to automatically identify spectral overlaps that can lead to false-positive results and recommends the emission wavelength that will give the most accurate result (2).
- Outlier Conditional Formatting (OCF) compares analytical results from different wavelengths for the same element, providing extra confidence in the results.
- Early Maintenance Feedback (EMF) allows the user to set up alerts that prompt analysts to perform maintenance based on instrument usage rather than simple time-based routines. Examples of EMF counters include the number of samples measured, advanced valve system (AVS) switches, plasma hours, etc. Maintaining the instrument at appropriate usage intervals ensures ongoing high-level analytical performance and minimal downtime. EMF provides seven default maintenance counter presets for general, high total dissolved solids (TDS), and organic applications, as the type of sample will affect the sample introduction and consumables differently. For example, for organic

samples, EMF will remind the user to clean the spray chamber after 1000 measured solutions. To reflect a laboratory sample load and experience, users can also customize their own maintenance counters to suit their needs.

Experimental

Instrumentation

All measurements were performed using an Agilent 5800 RV ICP-OES configured with an integrated AVS 7 port switching valve and an SPS 4 autosampler. The AVS switching valve decreases total run time enabling argon consumption to be decreased by at least 50% per sample, greatly reducing operating costs.

The standard sample introduction system was used with the Easy-fit fully demountable RV torch fitted with a removable 1.4 mm i.d. quartz injector (part number G8020-68002). The demountable torch is designed to improve laboratory workflows, reduce instrument downtime, and lower running costs by allowing the user to easily remove all the quartz components of the torch for maintenance (3). Instrument and AVS operating parameters are shown in Table 1 and 2, respectively.

Table 1. 5800 RV ICP-OES instrument and method parameters.

Parameter	Setting	
RF Power (kW)	1.40	
Plasma Gas Flow (L/min)	15.0	
Auxiliary Gas Flow (L/min)	1.50	
Torch	Easy fit fully demountable torch	
Tube Set	Quartz RV outer tube set for organic solvents	
Injector	Quartz 1.4 mm i.d., tapered	
Viewing Mode	Radial	
Viewing Height (mm)	7	
Nebulizer	SeaSpray U-series concentric glass nebulizer	
Nebulizer Gas Flow (L/min)	0.65	
Spray Chamber	Glass double pass	
Reading Time (s)	3	
Replicates	3	
Stabilization Time (s)	12	
Rinse Time (s)	5	
Peristaltic Pump Tubing	Sample	PVC Solvaflex black-black
	Waste	PVC Solvaflex blue-blue
	Internal standard	PVC Solvaflex black-black
Internal Standard	Co in A-Solv (25 µg/g)	
Oxygen Injection	Not required	
IntelliQuant	Yes	

Table 2. AVS 7 switching valve settings.

Parameter	Setting
Valve Uptake Delay (s)	10
Pump Rate - Uptake (mL/min)	29
Pump Rate - Inject (mL/min)	5.6
Sample Loop Size (mL)	1
Pre-emptive Rinse Time (s)	0
Bubble Inject Time (s)	2

Preparation of standards and samples

Four multi-element standards of 5, 10, 25, and 50 ppm were prepared by serial dilution of Agilent 900 µg/g A-21+K organometallic wear metal standard (part number 5190-8706). Three more high concentration multi-element standards containing different concentrations of P, Ca, Zn, and Mg were prepared from the corresponding Agilent 5000 µg/g single element organometallic oil standards prepared in 75 cSt hydrocarbon oil.

All standards and samples were prepared on a weight-weight (w/w) basis. To achieve consistent viscosity, additional 75 cSt base mineral oil (part number 5190-5715) was added when needed, to give a total oil concentration of 10% w/w. The calibration blank was prepared by dilution of the 75 cSt base mineral oil. Agilent A-Solv ICP solvent (part number 5190-8717) was used as the diluent for preparation of all blanks, standards, and oil samples.

A National Institute of Standards and Technology (NIST) standard reference material (SRM) 1085c Wear Metals in Lubricating Oil (Gaithersburg MD, USA) was analyzed to validate the method.

To assess the robustness of the method, used oil samples of three different oil types and viscosities were obtained and analyzed. The samples included a diesel engine oil SAE 15W-40, hydraulic oil SAE 10W, and gear oil SAE 85W-140.

An internal standard (ISTD) solution of 25 mg/kg cobalt in hydrocarbon oil was prepared by dilution of an Agilent 5000 µg/g organometallic oil ISTD (part number 5190-8714) on a w/w basis using A-Solv solvent.

Wavelength selection and background correction

The emission lines selected for the analysis and the background correction method used for each line are listed in Table 3. Some of the wavelengths were based on the lines suggested in ASTM D5185-18. Other lines were selected to provide minimal spectral interferences and a wide dynamic range, eliminating the need for time-consuming sample dilutions and reanalysis. Cadmium was included in the

analyte list despite not being included in the scope of D5185-18. Sulfur was not part of this study as this analyte was not present in the lubricating oil SRM.

The ISTD solution of 25 mg/kg cobalt in hydrocarbon oil was delivered online through the seventh AVS port.

Table 3. Line selection and calibration data.

Element and Wavelength (nm)	Calibration Fit	Background Correction	Internal Standard Correction	Calibration Range (mg/kg)	Correlation Coefficient (R)
Ag 328.068	Linear	Fitted	None	0–50	1.0000
Al 308.215	Linear	Fitted	None	0–50	1.0000
B 249.678	Linear	Fitted	None	0–50	0.9999
Ba 493.408	Linear	Fitted	None	0–50	1.0000
Ca 422.673	Linear	Fitted	Co (258.033)	0–380	0.9997
Cd 228.802	Linear	Fitted	None	0–50	1.0000
Cr 267.716	Linear	Fitted	None	0–50	1.0000
Cu 324.754	Linear	Fitted	None	0–50	0.9999
Fe 259.940	Linear	Fitted	None	0–50	1.0000
K 766.491	Linear	Fitted	None	0–50	0.9999
Mg 285.213	Linear	Fitted	Co (258.033)	0–120	1.0000
Mn 293.305	Linear	Fitted	None	0–50	1.0000
Mo 202.032	Linear	Fitted	None	0–50	1.0000
Na 589.592	Linear	Fitted	None	0–50	0.9999
Ni 231.604	Linear	Off-peak right	None	0–50	1.0000
P 178.222	Linear	Fitted	Co (258.033)	0–160	0.9997
Pb 220.353	Linear	Fitted	None	0–50	1.0000
Si 251.611	Linear	Fitted	None	0–50	0.9999
Sn 242.950	Linear	Fitted	None	0–50	0.9999
Ti 334.941	Linear	Fitted	None	0–50	1.0000
V 310.229	Linear	Fitted	None	0–50	1.0000
Zn 206.200	Linear	Fitted	Co (237.863)	0–220	0.9994

Calibration linearity

A wide linear dynamic range is needed for efficient analysis of lubricating oils. Wear metals are typically present in used oils at low concentrations, while elements in oil additive packages are usually present in concentrations up to several hundreds of mg/kg. The wide linear dynamic range of the 5800 RV ICP-OES allows quantification of all elements in one determination. The calibration curves of Cr 267.716 and Ca 422.673, which are shown in Figure 1, included concentrations up to 50 and 380 mg/kg, respectively. Correlation coefficients were greater than 0.9999 for all elements (Table 3) with less than 3% calibration error on each calibration point.

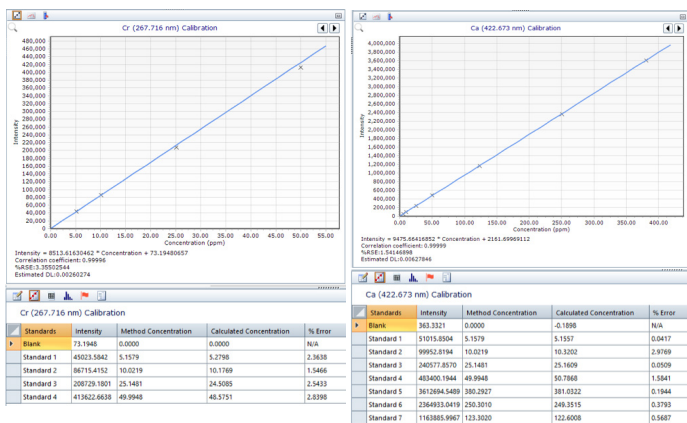


Figure 1. Calibration curves for Cr at 267.716 nm and Ca at 422.673 nm.

Method detection limits

Method detection limits (MDLs) were determined by running the full calibration, followed by 10 repeat analyses of the sample blank. The MDL is defined as three times the mean standard deviation of the concentration readings for each element. All MDLs were well below 0.5 mg/kg, allowing wear metals to be detected and monitored at low levels (Table 4).

The limit of quantification (LOQ) for this analysis was estimated as 10 times the standard deviation of the concentration readings multiplied by the dilution factor (10 x).

Results and discussion

Flagging outlying results using Outlier Conditioning Formatting

The Outlier Conditioning Formatting function in the ICP Expert software makes it quick and easy to find potentially problematic results in a large set of quantitative data, using color-coded flags. OCF can be configured to highlight over ranges, poor RSDs, internal standard failures, or inconsistent results across wavelengths of the same element.

Colloidal molybdenum (Mo) compounds are often used as antifriction additives in lubricant oils and greases (4). Mo concentration levels can be high enough to interfere with other elements at the typical emission lines specified in ASTM D5185-18. To simulate an oil sample containing a high concentration of Mo, engine oil samples were spiked at 10, 100, and 250 mg/kg and analyzed using IntelliQuant as part of the quantitative method. The difference between the results obtained using three Si and three Sn wavelengths exceeded the specified precision threshold, so were flagged, as shown in Figure 2. The IntelliQuant function was then used to identify which Si and Sn results to report. The Si 251.432 and Sn 189.925 nm emission lines were given the highest confidence rating by the IntelliQuant algorithm.

Table 4. Method detection limits and estimated limits of quantification.

Element and Wavelength (nm)	MDL (µg/kg)	LOQ (mg/kg)
Ag 328.068	3.0	0.08
Al 167.019	4.3	0.14
Al 308.215	13.9	0.46
B 249.678	5.4	0.18
Ba 233.527	2.7	0.09
Ca 422.673	4.9	0.16
Cd 214.439	1.6	0.05
Cr 267.716	2.5	0.08
Cu 324.754	2.8	0.09
Fe 259.940	4.1	0.14
K 766.491	48.9	1.63
Mg 280.270	1.6	0.05
Mn 257.610	1.6	0.05
Mo 202.032	5.8	0.19
Na 588.995	20.7	0.69
Ni 231.604	10.5	0.35
P 177.434	22.6	0.75
Pb 220.353	29.0	0.97
Si 251.432	24.4	0.81
Si 251.611	8.2	0.27
Sn 189.925	17.2	0.57
Sn 242.950	30.6	1.02
Ti 334.941	1.7	0.06
V 311.837	2.2	0.07
Zn 213.857	1.9	0.06

Rack/Tube	Solution Label	Outlier Summary	Si	Si	Si	Sn	Sn	Sn
			251.432 nm ppm	251.611 nm ppm	288.158 nm ppm	189.925 nm ppm	242.950 nm ppm	283.998 nm ppm
3:19	GearOil-A21_Spkd-2	F	3.1239	3.1107	3.1356	2.5911	2.4698	2.4740
3:19	GearOil-A21_Spkd-3	F	3.0910	3.1150	3.1163	2.6138	2.4825	2.5014
3:20	GearOil-A21_Mo10_Spkd-1	F	3.0987	3.1376	3.0906	2.6543	2.4761	2.5052
3:20	GearOil-A21_Mo10_Spkd-2	F	3.0761	3.1306	3.0650	2.6139	2.4378	2.5066
3:20	GearOil-A21_Mo10_Spkd-3	F	3.0671	3.1346	3.0751	2.5930	2.4440	2.4871
3:21	GearOil-A21_Mo100_Spkd-1	F	3.0091	3.4853	3.0045	2.6041	2.7432	2.5048
3:21	GearOil-A21_Mo100_Spkd-2	F	2.9707	3.4642	3.0059	2.5635	2.6982	2.4851
3:21	GearOil-A21_Mo100_Spkd-3	F	2.9887	3.4762	3.0168	2.6211	2.7519	2.4642
3:22	GearOil-A21_Mo250_Spkd-1	F	3.0175	4.1546	3.0279	2.5768	3.2929	2.5041
3:22	GearOil-A21_Mo250_Spkd-2	F	2.9844	4.1528	2.9918	2.5910	3.3709	2.4941
3:22	GearOil-A21_Mo250_Spkd-3	F	2.9508	4.1206	2.9912	2.5657	3.1480	2.4858

Figure 2. Flagging of Mo-spiked oil sample results using Outlier Conditioning Formatting. The flags alert the user of a difference in the Si and Sn concentrations determined using Si 251.432, Si 251.611, Si 288.158, and Sn 189.925, Sn 283.998, Sn 249.950 wavelengths, respectively.

Investigating interferences using IntelliQuant

IntelliQuant can accurately identify wavelengths with potential interferences and suggest the most appropriate line based on the elements present in the sample, without remeasuring the samples. To test the capabilities of IntelliQuant, the following experiment was carried out:

- Used gear and engine oil samples were spiked with known concentrations of 22 elements (≈ 2.5 mg/kg)
- The spiked samples were then spiked with Mo at 10, 100, and 250 mg/kg
- All samples were analyzed using the quantitative method with IntelliQuant enabled

The results for the used gear oil (Table 5 and Figure 3) show quantitative results for Al, Si, and Sn lines in the unspiked sample, spiked sample, and spiked sample with Mo added at three concentration levels. The measured concentrations of Al 308.215, Si 251.611, and Sn 242.950 were higher than expected in the presence of 100 and 250 mg/kg Mo, suggesting a Mo-based interference. The measured results for Al 167.019, Si 251.432, and Sn 189.925 were as expected, suggesting no interference from Mo.

Table 5. Used gear oil sample spiked with Mo at 10, 100, and 250 mg/kg.

	Measured concentrations (mg/kg)					
	Al 308.215	Al 167.019	Si 251.611	Si 251.432	Sn 242.950	Sn 189.925
Sample	0.1	0.1	1.1	1.0	0.0	0.0
Spiked sample	2.5	2.5	3.3	3.3	2.4	2.3
Spiked sample + 10 mg/kg Mo	2.6	2.5	3.3	3.2	2.4	2.3
Spiked sample + 100 mg/kg Mo	3.4	2.5	3.8	3.2	2.6	2.3
Spiked sample + 250 mg/kg Mo	4.6	2.5	4.6	3.2	2.9	2.2

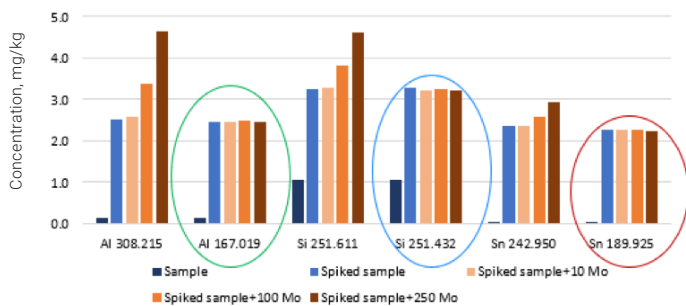


Figure 3. Measured concentrations for the elements examined in this interference study, highlighting the effects of Mo concentration on different wavelengths of the same element in gear oil.

IntelliQuant uses data analytics to rank different emission wavelengths for the same element and identifies likely interferences based on all elements present in the sample. Based on signal intensity, background structure, and potential interferences, IntelliQuant recommended Al 167.019, Si 251.432, and Sn 189.925 as the best wavelengths for the analysis, as shown in Figure 4. The recommended wavelengths agree with the wavelengths suggested by the results reported in Table 5.

By hovering over the "?" symbol next to the various lines (Figure 4), IntelliQuant provides extra information. Using Si as an example, IntelliQuant identified an interference on Si 251.611 from Mo 251.609, while Si 184.685 and Si 185.005 were considered concentration outliers (Figure 5). 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).

Element	Used	Flags	Wavelength	Rating	Concentration	Intensity	Background
Ag	✓		328.068	★★★★★	2.40	47172.3	3686.0
			338.289	★★★★	2.42	6186.5	1436.2
			211.383	★★★	28.04	917.1	734.9
Al			396.152	* ?	9.11	50980.9	9436.5
	✓		167.019	★★★★★	2.11	4403.4	400.1
			237.312	★★★	2.59	807.0	996.1
			309.271	★★★★★	2.22	4793.4	3725.9
			236.705	* ?	5.31	757.8	793.0
Si			251.611	* ?	4.62	16577.8	1218.5
			288.158	★★★★	3.23	9412.1	1408.3
			185.005	* ?	4.15	3504.1	581.4
	✓		251.432	★★★★★	3.17	3728.4	1218.0
			184.685	* ?	2.99	1552.8	618.7
Sn	✓		189.925	★★★★	2.11	2363.1	704.0
			283.998	* ?	2.38	2865.7	1527.3
			224.606	★★★★	2.86	574.5	919.0

Figure 4. 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.

Analyte: Si(251.611) Confidence: very weak Interference: Mo(251.609) Confidence: very strong	Analyte: Si(185.005) Confidence: very weak Concentration outlier	Analyte: Si(184.685) Confidence: very weak Concentration outlier
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Figure 5. Information provided by IntelliQuant for the poor rating on Si 251.611, 185.005, and 184.685 wavelengths.

The effects of Mo interferences on the measured concentration of Al 308.215, Si 251.611, and Sn 242.950 in the used engine oil samples were similar to the gear oil results. As shown in Figure 6, the IntelliQuant function's full wavelength spectrum provides quick sample insight about potential interferences based on the elements present in the sample. This insight helps with technically challenging and time-consuming method development. Once identified, any interferences can be avoided by selecting a different wavelength for an element, such as Al 167.019, Si 251.432, and Sn 189.925 in this example. If a specific wavelength must be used, interferences can be corrected using fitted background correction (FBC), Fast Automated Curve-fitting Technique (FACT), or Inter Element Correction (IEC).

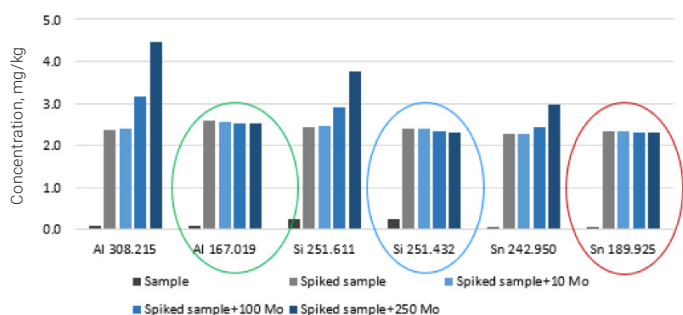


Figure 6. Measured concentrations for the elements examined in this interference study, highlighting the effects of Mo concentration on different wavelengths of the same element in engine oil.

SRM recoveries

The recoveries for the 22 elements measured in NIST 1085c Wear Metals in Lubricating Oil SRM were all within $\pm 10\%$ (Table 6). The results are the average of three determinations of the SRM. The relative standard deviation of the results was below 3%.

Table 6. Mean measured recoveries for elements determined in the NIST 1085c Wear Metals in Lubricating Oil SRM, n = 3.

Element	Certified concentration ($\mu\text{g/g}$)	Measured Concentration ($\mu\text{g/g}$)	Recovery %
Ag	298	295	99
Al	292	309	106
B	304	280	92
Ba	306	289	94
Ca	299	305	102
Cd	301	311	103
Cr	302	293	97
Cu	298	307	103
Fe	301	299	99
K	295	303	103
Mg	300	294	98
Mn	299	300	100
Mo	305	296	97
Na	300	299	100
Ni	306	289	94
P	304	286	94
Pb	303	293	97
Si	293	286	98
Sn	298	289	97
Ti	300	302	101
V	285	288	101
Zn	285	275	97

* Reference value.

Spike recoveries

To assess the robustness of the method, used oil samples of different types and viscosities were obtained. Samples (2 g) were spiked with known amounts of the organometallic oil standards. Low concentration spikes were made for the wear metal elements, and high concentration spikes were made for Ca, Mg, P, and Zn. Base mineral oil (75 cSt) was added to give a total oil concentration of 10% w/w and the samples were diluted to 40 g using the A-Solv solvent.

Excellent spike recoveries were obtained in all the different oil sample types (Tables 7 to 9). The recoveries for all elements were within $\pm 10\%$ of the expected values, despite the different viscosities of the oil samples tested.

Table 7. Spike concentrations and spike recoveries for a sample of used hydraulic oil SAE 10W (1:10 solution).

Element	Hydraulic Oil (mg/kg)	Spiked Hydraulic Oil (mg/kg)	Measured Spike Concentration (mg/kg)	Spike Concentration (mg/kg)	Recovery %
Ag	0.048	5.143	5.095	5.266	97
Al	0.198	5.477	5.279	5.266	100
B	0.089	5.091	5.002	5.266	95
Ba	0.022	5.057	5.035	5.266	96
Ca	9.149	24.50	15.36	15.72	98
Cd	0.005	4.854	4.849	5.266	92
Cr	0.049	4.987	4.938	5.266	94
Cu	0.279	5.402	5.123	5.266	97
Fe	0.644	5.549	4.905	5.266	93
K	0.110	5.323	5.213	5.266	99
Mg	0.256	15.46	15.21	15.41	99
Mn	0.016	5.162	5.146	5.266	98
Mo	0.046	5.096	5.050	5.266	96
Na	0.129	5.361	5.232	5.266	99
Ni	0.026	4.862	4.836	5.266	92
P	24.42	39.29	14.87	15.40	97
Pb	0.051	4.847	4.796	5.266	91
Si	0.517	5.506	4.988	5.266	95
Sn	0.009	4.948	4.939	5.266	94
Ti	0.014	5.073	5.059	5.266	96
V	0.006	4.985	4.979	5.266	95
Zn	26.54	40.99	14.45	15.42	94

Table 8. Spike concentrations and spike recoveries for a sample of used gear oil SAE 85W-140 (1:10 solution).

Element	Gear Oil (mg/kg)	Spiked Gear Oil (mg/kg)	Measured Spike Concentration (mg/kg)	Spike Concentration (mg/kg)	Recovery %
Ag	0.002	5.056	5.054	5.138	98
Al	0.133	5.268	5.135	5.138	100
B	0.150	5.136	4.986	5.138	97
Ba	0.002	4.946	4.944	5.138	96
Ca	200.8	213.2	12.46	13.74	91
Cd	0.003	4.719	4.716	5.138	92
Cr	0.017	4.847	4.831	5.138	94
Cu	0.705	5.799	5.094	5.138	99
Fe	1.589	6.546	4.957	5.138	96
K	0.045	5.248	5.203	5.138	101
Mg	0.881	15.69	14.81	13.49	110
Mn	0.031	5.079	5.048	5.138	98
Mo	0.013	4.976	4.964	5.138	97
Na	0.113	5.334	5.221	5.138	102
Ni	0.017	4.768	4.751	5.138	92
P	44.16	58.07	13.91	13.47	103
Pb	0.007	4.640	4.633	5.138	90
Si	1.122	6.084	4.961	5.138	97
Sn	0.027	4.851	4.824	5.138	94
Ti	0.008	5.002	4.994	5.138	97
V	0.006	4.927	4.922	5.138	96
Zn	49.50	62.29	12.79	13.50	95

Table 9. Spike concentrations and spike recoveries for a sample of used diesel engine oil SAE 15W-40 (1:10 solution).

Element	Engine Oil (mg/kg)	Spiked Engine Oil (mg/kg)	Measured Spike Concentration (mg/kg)	Spike Concentration (mg/kg)	Recovery %
Ag	0.001	4.876	4.874	5.323	92
Al	0.085	5.371	5.286	5.323	99
B	1.049	5.940	4.891	5.323	92
Ba	0.007	4.964	4.957	5.323	93
Ca	134.8	149.8	15.01	14.97	100
Cd	0.002	4.862	4.860	5.323	91
Cr	0.020	4.882	4.862	5.323	91
Cu	0.240	5.137	4.897	5.323	92
Fe	0.617	5.434	4.817	5.323	90
K	0.106	5.103	4.997	5.323	94
Mg	0.659	15.74	15.08	14.68	103
Mn	0.014	5.053	5.039	5.323	95
Mo	1.554	6.513	4.959	5.323	93
Na	0.187	5.219	5.032	5.323	95
Ni	0.017	4.846	4.829	5.323	91
P	50.33	64.72	14.39	14.67	98
Pb	0.053	4.888	4.834	5.323	91
Si	0.298	5.118	4.820	5.323	91
Sn	0.019	4.987	4.969	5.323	93
Ti	0.002	4.900	4.898	5.323	92
V	0.004	4.852	4.848	5.323	91
Zn	55.21	68.93	13.72	14.69	93

Early Maintenance Feedback

Analyzing complex sample types such as lubricating oils can be tough on the sample introduction system of an ICP-OES. This can result in deteriorating analytical performance, high consumable costs, and unplanned instrument downtime. Scheduling maintenance tasks according to the number of solutions measured, rather than elapsed time, can reduce these impacts. The EMF function allows the user to set up an alert to prompt maintenance after a specified number of samples. Recommended alert settings for specific sample types can be generated automatically. The alert system will result in more frequent instrument maintenance when measuring complex samples, ensuring consistent analytical performance.

Long-term stability

To check instrument stability, a 5 mg/kg organometallic calibration standard was analyzed after every 25 measurements of the used oil samples, over a period of 10 hours (> 700 samples). No recalibration or reslope was applied. Excellent long-term stability was achieved over 10 hours, as shown in Figure 7. All measurements were within $\pm 10\%$ of the expected value, with precision better than 5% RSD in most cases.

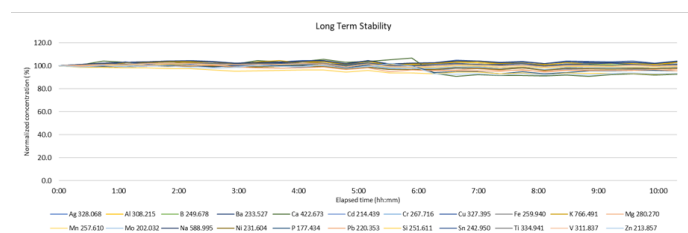


Figure 7. Long-term stability showing recoveries of a 5 mg/kg organometallic calibration standard analyzed over the course of the 10-hour sequence.

Conclusion

The study shows the suitability of the Agilent 5800 RV ICP-OES for oil-testing laboratories running multi-element analysis of oils according to ASTM D5185-18. The 5800 ICP-OES uses smart software tools to provide insight about samples and instrument performance, so users can get the right answer the first time.

To test the Outlier Conditional Formatting and IntelliQuant software functions, oil samples were spiked with increasing concentrations of Mo. The Outlier Conditional Formatting flagged discrepancies in the results for Si and Sn, and IntelliQuant identified Al, Si, and Sn wavelengths subject to Mo interference. IntelliQuant suggested the most appropriate wavelengths for the accurate measurement of these elements in used gear and engine oils, without rerunning the samples.

Excellent recoveries were obtained for all elements in the NIST Wear Metals in Lubricating Oil SRM. The results of the spike recoveries of 22 elements in used oil samples and the long-term stability test demonstrated the robustness of the method and the sample introduction system.

The Easy-fit fully demountable torch reduced carbon build-up during the analysis of the oil samples. Also, to keep the 5800 running over long periods, smart Early Maintenance Feedback diagnostics and counters were used to alert the analyst when maintenance was needed. Ensuring peak instrument performance minimizes QC failures and reduces the need to rerun samples—saving time.

The work demonstrated that the 5800 RV ICP-OES can accurately detect low range concentrations of wear metals and high concentrations of the elements from the additive package in a single analysis. For high throughput lubricant oil analysis, the 5800 can be fitted with the integrated AVS 7 sampling valve. The AVS maximizes sample throughput and reduces argon consumption, improving productivity, while reducing operating costs.

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