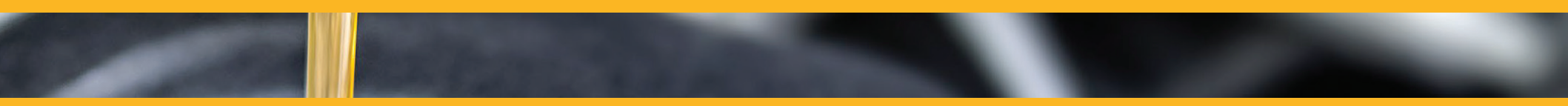
ASTM D7751: DETERMINATION OF ADDITIVE ELEMENTS IN LUBRICATING OILS BY EDXRF ANALYSIS



To guarantee optimal engine and machine performance and life-time, while maintaining the best-possible protection of our environment, today's lubricants are compositionally tuned to perfection. When producing, using, and recycling lubricating oils, it is inevitable to have a clear and precise understanding of the additive elements contents. X-ray fluorescence (XRF) analysis is the ideal method to accomplish this task. XRF elemental analysis is fast and precise, ensuring that additives are used economically while the properties of lubricants and fuels are ensured.

This reduces production costs and lowers environmental impact. The benchtop Energy Dispersive X-ray Fluorescence (EDXRF) S2 POLAR fulfills all requirements for the analysis of seven important additives (Mg, P, S, Cl, Ca, Zn, Mo) and is fully norm-compliant with ASTM D7751-16. The S2 POLAR allows for efficient quality control (QC) in base oil production, blending facilities or additive dosing stations.

Instrument

The EDXRF S2 POLAR (Fig. 1) with its polarizing HighSense™ beam path is optimized for multi-element petrochemical applications. The instrument is equipped with a high-power 50 W Pd X-ray tube and a HighSense ULS silicon drift detector with optimum spectral resolution. The easy-to-use, multilingual TouchControl™ interface in conjunction with the factory calibrated application packages for the norm ASTM D7751 ensure a 'One-Button' Ready-to-analyze solution (Fig. 2). This enables users with minimal training to run routine samples on day one.

Figure 1: S2 POLAR – Compact benchtop EDXRF designed for petrochemical applications

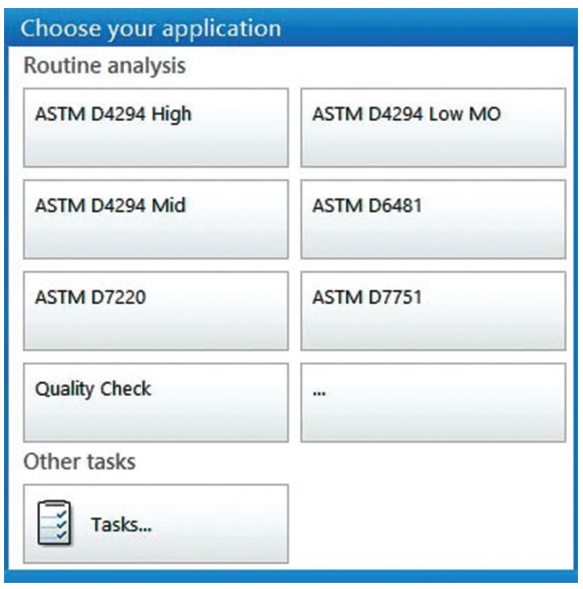


Figure 2: Ease and straightforward to use with factory precalibrated 'One-Button' TouchControl™ methods

Preparation

Bruker's dedicated preparation accessory kit for oil samples contains the required parts to prepare liquid cups for such samples. This kit includes liquid cups with 40 mm outer diameter, SampleCare™ cups with 51 mm and Prolene foils with 4.0 µm thickness. The SampleCare cup prevents sample leakages and protects important system components. This guarantees maximum instrument availability, even for high throughput lubricating oil analysis. In combination with the liquid cup preparation tool (provided with the S2 POLAR) fast and reproducible sample preparation is ensured. The use of standardized liquid cups results in low costs per sample. For sample preparation it is only required to weigh 7 g oil sample into a liquid cup and place it for the measurement into a larger SampleCare cup (Fig. 3).

Figure 3: Bruker's dedicated liquid sample cup (Ø 40 mm, left) and the larger SampleCare cup (Ø 51 mm, right)

Measurement

Two measurement regions have been defined to cover seven additive elements (Mg, P, S, Cl, Ca, Zn, Mo). The tube current was optimized and fixed to gain maximum count rate for the various elements. Table 1 lists the detailed measurement parameters.

To obtain best analytical results for light elements such as Mg and P, the measurements have been performed in 'reduced' Helium mode. This mode uses the vacuum pump to minimize helium consumption while ensuring excellent performance for light element analysis.

Table 1: Instrument measurement parameters

Element	Tube voltage [kV]	Tube current [mA]	Measurement time [s]
Mg, P, S, Cl, Ca	25	1700	240
Zn, Mo	50	850	240

Calibration

The Ready-to-analyze solution ASTM D7751 is a factory calibrated application package for unused lubricating oils and consists of a set of twelve validated multi-element standards. The solution covers the concentration ranges of the additive elements shown in Table 2. An exemplary calibration curve is shown in Figure 4.

For ease-of-use the ASTM D7751 solution also contains the required QC Blank sample, a QC sample as well as a Drift Correction sample. There is no need for extensive method set-up and the instrument is immediately available for routine samples.

Table 2: Concentration ranges of standards included in the ASTM D7751 solution package

Element	Min. concentration [%]	Max. concentration [%]
Mg	0.02	0.30
P	0.005	0.40
S	0.007	2.50
Cl	0.005	0.40
Ca	0.005	0.60
Zn	0.005	0.40
Mo	0.005	0.075

Results

Repeatability measurements have been performed to prove the norm-compliance with ASTM D7751-16.

Typical data obtained for a lubricating oil analyses are shown in Table 3.

Figure 5 illustrates the reproducibility for Mg in lubricating oil (blue line). The red bars at the bottom show that the difference between successive measurements is for all measurements lower than 0.006 %. The max. allowed difference is 0.01 % Mg which is easily achieved by the instrument.

This test proves its norm-compliance with ASTM D7751 and also demonstrates the excellent analytical performance of the S2 POLAR for all kinds of lubricating oil analysis.

Conclusion

The optimized, polarized HighSense™ beam path and the high-power 50 W X-ray tube of the S2 POLAR allows for precise monitoring of additive elements in lubricating oils and fuels. The instrument easily fulfills all requirements according to the international norm ASTM D7751-16. The shown data prove the excellent analytical performance of the instrument and ensure fast elemental analysis of additives.

With the simple sample preparation, routine sample measurements are easy and straightforward. High instrument uptime is ensured with Bruker's SampleCare technology. This guarantees utmost instrument availability and cost-effective QC, even with high throughput of lubricating oil samples.

The S2 POLAR Ready-to-analyze solutions minimize instrument set-up time and provide 'One Button' multielemental analysis. This leads to economic quality control and your investment pays back rapidly.

Table 3: Repeatability of lubricating oil analyses

	Mg [%]	P [%]	S [%]	Cl [%]	Ca [%]	Zn [%]	Mo [%]
Rep 1	0.099	0.111	0.511	0.0265	0.204	0.119	0.0097
Rep 2	0.097	0.112	0.514	0.0264	0.201	0.120	0.0095
Rep 3	0.102	0.111	0.509	0.0261	0.202	0.118	0.0097
Rep 4	0.101	0.112	0.514	0.0264	0.203	0.121	0.0096
Rep 5	0.104	0.111	0.514	0.0263	0.200	0.119	0.0094
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Rep 16	0.101	0.111	0.511	0.0264	0.202	0.120	0.0093
Rep 17	0.104	0.111	0.512	0.0263	0.202	0.118	0.0096
Rep 18	0.098	0.111	0.513	0.0265	0.202	0.121	0.0094
Rep 19	0.101	0.112	0.513	0.0264	0.203	0.119	0.0092
Rep 20	0.094	0.111	0.511	0.0264	0.203	0.119	0.0094
Mean value	0.100	0.111	0.512	0.0264	0.202	0.119	0.0094
Abs. Std. Dev.	0.0025	0.0004	0.0016	0.0001	0.0010	0.0009	0.0002
Rel. Std. Dev. [%]	2.49	0.36	0.31	0.40	0.51	0.78	1.98
Certified value	0.100	0.110	0.500	0.025	0.200	0.120	0.0100

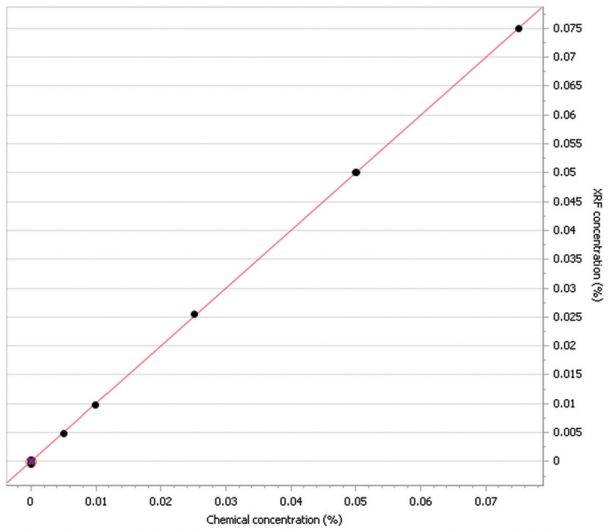

Figure 4: Calibration curve for Mo in mineral oil covering the range from 0.005 % to 0.075 % Mo

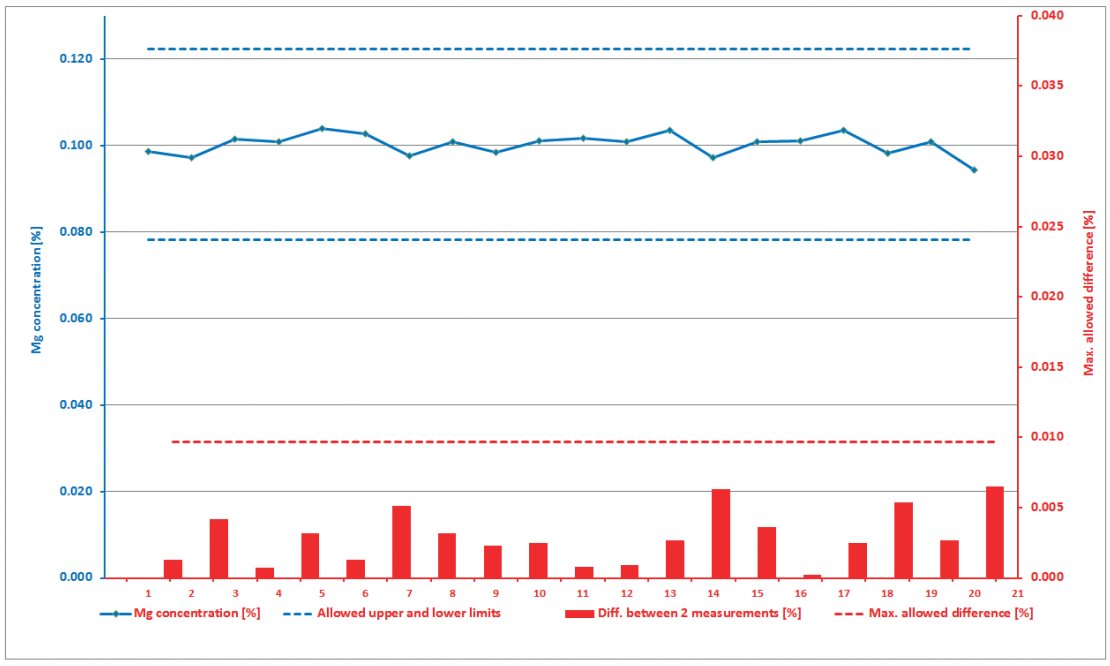

Figure 5: Repeatability test (n = 20) for Mg in lubricating oil and compliance with ASTM D7751

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