Lab Report XRF 158

S2 POLAR

- ASTM D6481: Determination of P, S, Ca, and Zn in Lubricating Oils

Introduction

Additives in base oils are used to optimize engine performance and its lifetime. It is crucial to follow the tight specifications when blending lubricating oils. Analytical accuracy and precision count to ensure high product quality in combination with low production cost. X-ray fluorescence (XRF) is the method of choice due to its ease of use and straightforward sample preparation. The Energy Dispersive X-ray Fluorescence (EDXRF) S2 POLAR is perfectly designed to master even challenging lubricating oil applications. Based on the HighSense™ beam path with the HighSense ULS detector, the S2 POLAR provides high sensitivity for excellent analytical precision, lowest detection limits and optimum spectral resolution. This enables S2 POLAR to perform multi-element oil analysis fully norm-compliant according to ASTM D6481-14. This allows tight quality control in base oil production, blending facilities or additive dosing stations.

Instrument

Figure 1: S2 POLAR - Compact, multi-element benchtop EDXRF

The EDXRF S2 POLAR (Fig. 1) with its polarizing HighSense™ beam bath is optimized for multi-element petrochemical applications. The instrument is equipped with a Pd X-ray tube and the HighSense ULS silicon drift detector. The easy-to-use multilingual TouchControl™ interface in combination with the factory pre-calibrated application package for the norm ASTM D6481 provides a 'One-Button' Ready-to-analyze solution (Fig. 2). This enables users with minimal training to run routine samples.

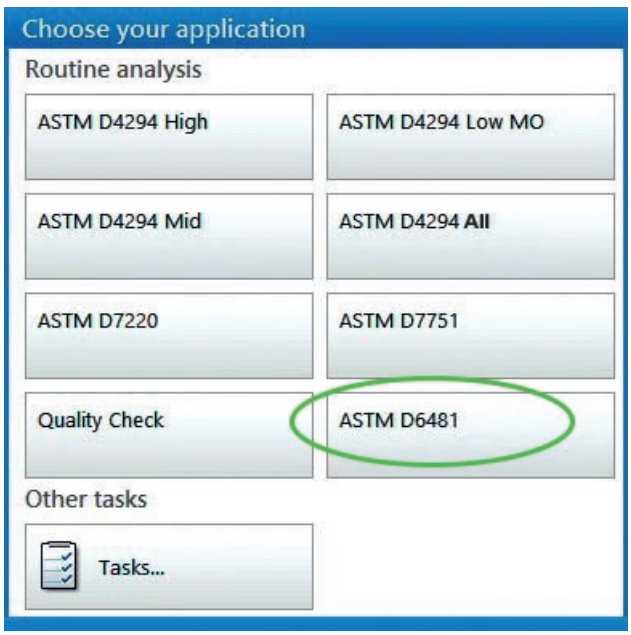

Figure 2: Ease to use factory pre-calibrated 'One-Button' TouchControl™ method for ASTM D6481

Preparation

The preparation accessory kit for oil samples (K410C215) contains the required parts to prepare liquid cups for fuel samples. This kit contains liquid cups with 40 mm outer diameter, SampleCare™ cups with 51 mm and Prolene foils with 4.0 µm thickness. The liquid cup preparation tool provided with the S2 POLAR is very helpful to prepare liquid cups in an optimal and efficient way. Due to the standardized liquid cups the costs per sample are low. For sample preparation it is only required to weigh 7 g fuel sample into a liquid cup and place it for the measurement into a larger SampleCare cup (Fig. 3). The SampleCare cup itself is also prepared with a 4.0 µm Prolene foil. The SampleCare cup prevents sample leakages and protects important system components. This guarantees maximum instrument availability, even for high throughput lubricating oil analysis.

Figure 3: Liquid cup (Ø 40 mm) in SampleCare cup (Ø 51 mm). The liquid cup preparation tool can also be used to transport the samples from lab bench to measurement chamber.

Measurement

One measurement region has been defined to cover all four elements. The tube current was optimized and fixed to gain maximum count rate for the various elements. Table 1 shows the detailed measurement parameters.

Table 1: Instrument measurement parameters

Element	Tube voltage [kV]	Tube current [mA]	Measurement time [s]
P, S, Ca, Zn	25	1700	300

In order to obtain best analytical results for light elements such as P the measurements have been performed in Helium mode, in this particular case at reduced pressure conditions with the support of a vacuum pump to help minimize helium consumption.

Calibration

The Ready-to-analyze solution ASTM D6481 is a factory calibrated application package for lubricating oils and consists of a set of seventeen validated multi-element standards. The solution covers the concentration ranges of the additive elements shown in Tab. 2 while Fig. 4 shows an example calibration curve, in this case for P, and overlaid P signals are shown in Fig 5.

For ease-of-use the ASTM D6481 solution also contains the required QC Blank sample, one QC sample as well as a drift correction (DC) sample. There is no need for extensive method set-up and the instrument is immediately available for routine samples.

Table 2: Concentration ranges of included standards in the Ready-to-analyze solution ASTM D6481

Element	Min. concentration [%]	Min. concentration [%]
P	0.005	0.30
S	0.050	1.00
Ca	0.005	1.00
Zn	0.050	0.30

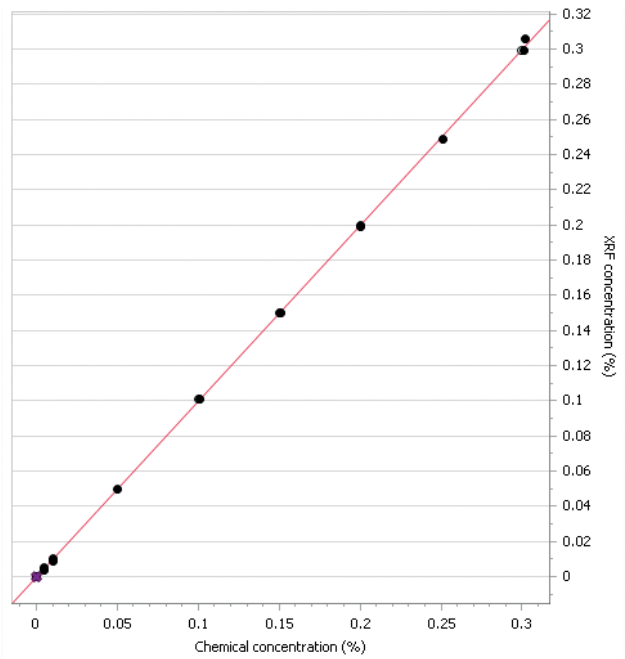

Figure 4: Calibration curve for P between 0 to 0.30 % P

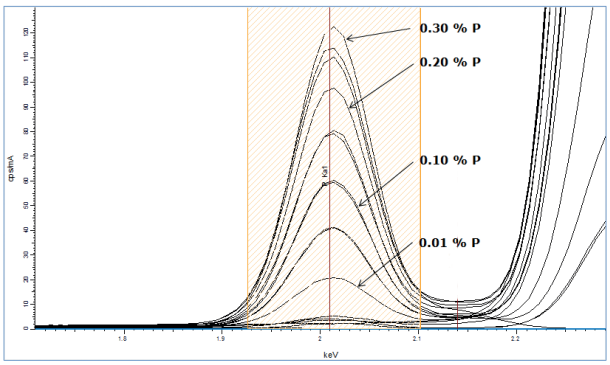

Figure 5: Overlaid P signals in lubricating oil between 0 to 0.30 % P

Results

Repeatability measurements have been performed to prove the norm-compliance with ASTM D6481. Some typical data for 20 measurements of a lubricating oil sample are shown in Tab. 3 and for Ca in Fig. 6.

Table 3: Repeatability for 20 measurements of a lubricating oil sample

	Date Time	P [%]	S [%]	Ca [%]	Zn [%]
Rep 01	13.06.2018 14:14	0.100	0.502	0.251	0.101
Rep 02	13.06.2018 14:25	0.100	0.504	0.252	0.101
Rep 03	13.06.2018 14:36	0.100	0.501	0.251	0.100
Rep 04	13.06.2018 14:47	0.101	0.504	0.251	0.100
…	…	…	…	…	…
…	…	…	…	…	…
Rep 17	13.06.2018 17:50	0.100	0.500	0.251	0.102
Rep 18	13.06.2018 18:00	0.100	0.501	0.251	0.101
Rep 19	13.06.2018 18:11	0.100	0.500	0.251	0.101
Rep 20	13.06.2018 18:21	0.100	0.500	0.251	0.101
Mean		**0.100**	**0.500**	**0.251**	**0.101**
Abs. Std. Dev.		**0.001**	**0.002**	**0.001**	**0.001**
Rel. Std. Dev. [%]		**0.50%**	**0.35%**	**0.26%**	**0.50%**
Certified		**0.100**	**0.500**	**0.250**	**0.100**

The data show the excellent precision of the S2 POLAR and confirm the analytical performance of the instrument.

Reproducibility measurements over a longer period of time have been performed and are shown in Tab. 4.

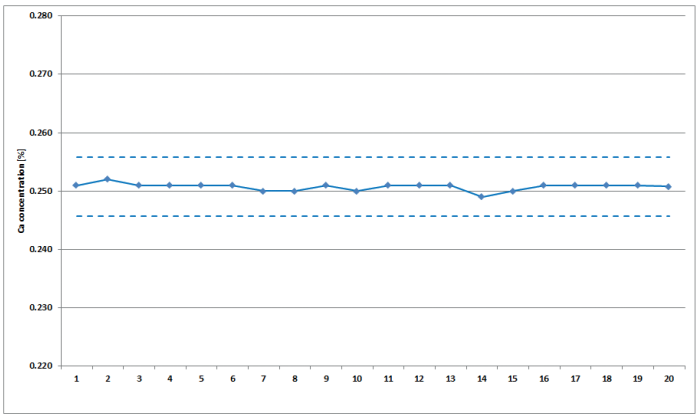

Figure 6: Repeatability for Ca of a lubricating oil sample, according to ASTM D6481. The doted lines denote the allowed limits by the norm.

Table 4: Reproducibility of a lubricating oil sample

	Date Time	P [%]	S [%]	Ca [%]	Zn [%]
Rep 01	27.02.2018 17:05	0.100	0.496	0.247	0.099
Rep 02	02.03.2018 12:42	0.100	0.500	0.248	0.101
Rep 03	05.03.2018 11:58	0.101	0.503	0.254	0.101
Rep 04	06.03.2018 11:12	0.100	0.501	0.253	0.101
Rep 05	07.03.2018 15:18	0.100	0.501	0.250	0.098
Rep 06	08.03.2018 12:37	0.100	0.498	0.251	0.100
Rep 07	12.03.2018 12:58	0.098	0.492	0.249	0.098
Rep 08	13.03.2018 10:02	0.101	0.497	0.251	0.098
Mean		**0.100**	**0.498**	**0.250**	**0.100**
Abs. Std. Dev.		**0.0009**	**0.0035**	**0.0024**	**0.0014**
Rel. Std. Dev. [%]		**0.93**	**0.70**	**0.95**	**1.42**
Certified		**0.100**	**0.500**	**0.250**	**0.100**

The data show the excellent precision of the S2 POLAR and confirm the analytical performance of the instrument.

Reproducibility measurements over a longer period of time have been performed and are shown in Tab. 4.

With the shown repeatability the S2 POLAR proves its norm-compliance with ASTM D6481 and also demonstrates the excellent analytical performance for all kinds of lubricating oil analysis.

Conclusion

With its optimized HighSense beam path the S2 POLAR allows accurate and precise quality control of additive elements in lubricating oils. The instrument fulfills all requirements according to the international norm ASTM D6481-14 and the data shown prove the excellent analytical performance of the instrument. With the factory pre-calibrated 'One-Button' Touch Control method and the facilitated sample preparation, routine measurements are easy and straightforward.

The S2 POLAR SampleCare technology prevents leakages of liquid samples and protects important system components. This guarantees maximum instrument availability.

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