



Lab Report XRF 164

S6 JAGUAR

- Accurate Analyses of Additives and Contaminants in Lubricating Oil – ASTM D6443 compliant!

Engine and lubricating oils are inevitable for most motors and engines, ensuring proper performance and lifetime. Lubricating oils can be tuned to perfection for specific applications and engines. The properties and performance of lubricating oils depend significantly on the additives (e.g., Ca, Zn, P, S) as well as on unwanted contaminants (e.g., Cl, which can cause corrosion). Certain wear metals, such as copper (Cu), can indicate wear in an engine. Thus, reliable determination of additives, contaminants, and wear metal concentrations is

important to use additive compounds economically and to optimize the oil and engine performance and lifetime.

This report demonstrates the outstanding capabilities of the wavelength-dispersive X-ray fluorescence (WDXRF) benchtop spectrometer S6 JAGUAR to analyze lubricating and engine oils. X-ray fluorescence (XRF) combines ease-of-use and low cost of operation with excellent accuracy and precision.



Figure 1: Powerful 400 W HighSense™ X-ray tube.



Figure 2: Dedicated liquid cups for lubricant oils.

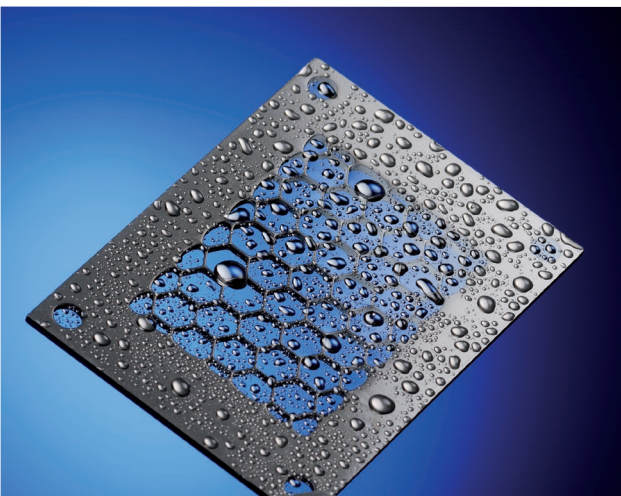


Figure 3: Vacuum Seal for protection of the HighSense™ detector.

S6 JAGUAR – A Powerful Benchtop System with Full WDXRF performance

The WDXRF spectrometer S6 JAGUAR is equipped with Bruker's 400 W HighSense™ X-ray tube (Figure 1). This makes the S6 JAGUAR the most powerful benchtop WDXRF unit on the market! Modern software and state-of-the-art hardware enable best-in-its-class analytical performance. The S6 JAGUAR achieves outstanding sensitivity for a wide range of elements (F to U) and the various configuration options allow us to optimize the system for your needs.

S6 JAGUAR – Ready for Petrochemical Applications

- Dedicated liquid cups for simple and rapid loading of oil samples (Figure 2)
- SampleCare™ technology, protecting critical system components for low maintenance (Figure 1)
- (optional) Cup-in-cup technology for additional protection
- Intuitive software SPECTRA.ELEMENTS with "one-button" solutions
- Sturdy design and robust, high quality components for long lifetime
- (optional) Ergonomic TouchControl™ display for operation w/o PC peripherals

Calibration and Analyses

We used 22 lubricating oil certified reference materials (CRMs) for the calibration; including one blank. The compositional range is listed in Table 1. The analytical conditions and the crystal setup were optimized for lubricating oil analysis (Table 2). The Ge(111) analyzer crystal significantly improves the sensitivity for S, Cl, and P in oil, when compared to a conventional PET crystal. The XS-55 analyzer crystal provides optimal performance for Mg analysis.

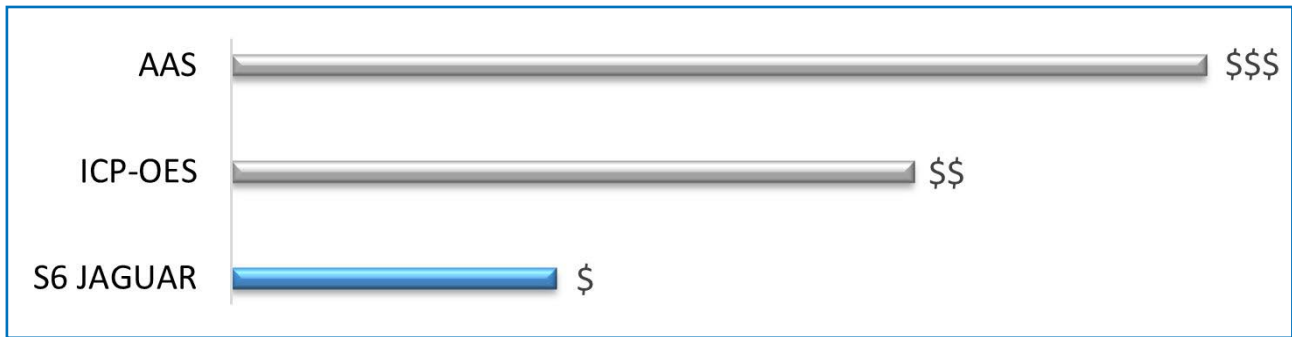


Figure 4: Relative estimation of operation costs. The consumption of chemicals (AAS, ICP-OES), expensive accessories (AAS) and noble gas (ICP-OES) results in much higher costs per sample for AAS and ICP-OES analyses when compared to XRF.

Advantages of XRF

XRF sample preparation and analysis is simple and cheap, when compared to alternative wet-chemical methods, such as atomic absorption spectrometry (AAS) or inductively coupled plasma optical emission spectrometry (ICP-OES). The latter require skilled operators and consume pricy and/or hazardous chemicals and accessories (see Figure 4). When using the S6 JAGUAR for your oil analysis, you can also decrease the time from sampling to result, considering the required dilution step for AAS and ICP-OES, which adds another benefit to the XRF side. In contrast to AAS and ICP-OES, a simple one-time calibration makes the S6 JAGUAR ready for your application. Only occasional analysis of a drift correction sample is required: No re-calibration necessary!

Table 1: Compositional range of the lubricant oil CRMs

Elements	Oil standards [wt.%]
Mg	0 – 0.2
P	0 – 0.15
S	0 – 0.75
Cl	0 – 0.15
Ca	0 – 0.5
Cu	0 – 0.05
Zn	0 – 0.15

Norm Compliance, Repeatability, Precision and Accuracy

The compliance of the S6 JAGUAR with the lubricating oil norm ASTM D6443 is illustrated in Figure 5. Clearly, the S6 JAGUAR meets the requirements easily! A repetition test demonstrates the exceptional stability and accuracy of the S6 JAGUAR for trace element analyses in oil (Table 3). The samples were unloaded and reloaded between the measurements.

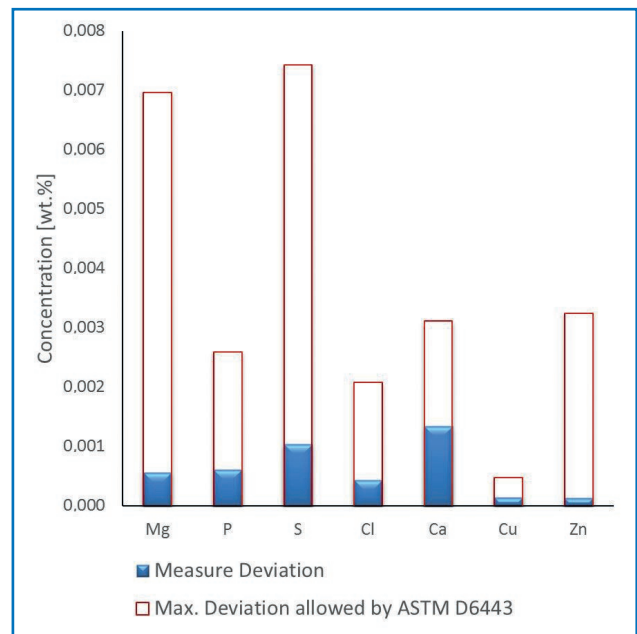


Figure 5: Norm Compliance with ASTM D6443

Table 2: Analytical conditions

Elements	Voltage [kV]	Current [mA]	Power [W]	Analyzer crystal	Filter	Detector
Mg	25	16	400	XS-55	none	FlowCounter
P, S, Cl	30	13.3	400	Ge(111)	none	FlowCounter
Ca	50	8	400	LiF(200)	none	FlowCounter
Cu, Zn	50	8	400	LiF(200)	Al (500 µm)	HighSense XE

Table 3: Stability, Precision, Accuracy, and Norm Compliance

[wt.%]	Mg	P	S	Cl	Ca	Cu	Zn
Rep-1	0.0786	0.0501	0.2781	0.0505	0.2034	0.0203	0.0503
Rep-2	0.0797	0.0513	0.2802	0.0505	0.2039	0.0203	0.0501
Rep-3	0.0780	0.0494	0.2782	0.0501	0.2028	0.0201	0.0501
Rep-4-12
Rep-13	0.079	0.0511	0.2789	0.0516	0.2041	0.0199	0.0505
Average	0.0787	0.0501	0.2793	0.0507	0.2037	0.0201	0.0503
Abs. Std. Deviation	0.0006	0.0006	0.0010	0.0004	0.0014	0.0001	0.0001
ASTM D6443	✓	✓	✓	✓	✓	✓	✓
Rel. Std. Deviation [%]	0.71	1.23	0.37	0.86	0.66	0.70	0.27
Certified composition	0.0800	0.0500	0.2754	0.0500	0.2024	0.0200	0.0501
Difference	0.0013	0.0001	0.0039	0.0007	0.0013	0.0001	0.0002
Rel. Difference [%]	1.62	0.20	1.43	1.40	0.64	0.50	0.38

Conclusion

This report highlights the outstanding performance of the S6 JAGUAR for lubricating and engine oil analysis. The system easily fulfils the requirements for the WDXRF oil norm ASTM D6443. The S6 JAGUAR brings several advantages to your lab, when compared to AAS and ICP-OES; including lower costs of operation and minimal calibration effort.

The S6 JAGUAR enables time-efficient and accurate monitoring of additives, contaminants, and wear metals in lubricating and engine oils. When installed at oil-production plants, you can easily test the quality of incoming materials and optimize the use additives. Accurate compositional monitoring of used oils from your engines allows you to forecast potential part failures to minimize downtime and maintenance costs.

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