Lab Report XRF 168

S2 POLAR

- ISO 13032-compliant Analysis of Ultra-Low Concentration of Sulfur in Automotive Fuels

Automotive fuel specifications valid in the European Community are defined for diesel in the norm EN 590 and for gasoline fuels in EN 228. Within these fuel specifications the test method ISO 13032 is an approved standard for the determination of ultra-low sulfur contents in automotive fuels by energy-dispersive X-ray fluorescence (EDXRF) spectrometry.

The benchtop EDXRF S2 POLAR masters all the requirements for low and ultra-low sulfur analysis in various automotive fuels and is fully norm-compliant with standard ISO 13032-12, which has been re-confirmed in 2018.

Figure 1: S2 POLAR - Compact benchtop EDXRF developed for the fuel industry

S2 POLAR – A Powerful EDXRF for Automotive Fuel Analysis

The EDXRF S2 POLAR (Figure 1) with its polarizing HighSense™ beam path is optimized for petrochemical applications. The polarization results in excellent signal to background ratio, especially for elements like sulfur, where outstanding detection limits in the sub-ppm range are achieved. The instrument is equipped with a Pd X-ray tube and the HighSense ULS silicon drift detector. Compact and sturdy design make the S2 POLAR the ideal petrochemical analyzer for small labs or on-site process control at refineries, tank farms, and oil terminals. The easy-of-use multilingual TouchControl™ interface in combination with the factory pre-calibrated application for the norm ISO 13032 provide a 'One-Button' ready-to-analyze solution. This enables users to run routine samples from day one.

Minimal Sample Preparation – Advantages of XRF

A preparation kit for fuel samples contains all required parts to quickly prepare liquid cups for failsafe fuel sample analyses. The kit contains liquid cups with 40 mm outer diameter, SampleCare™ cups with 51 mm and XRF Mylar® foils with 3.6 µm thickness (Figure 2). The standardized liquid cup solution allows for a cost and time efficient operation. The liquid cup is filled with 7 g of fuel by using a pipette and simply placed into a larger SampleCare cup (Figure 3). The SampleCare cup itself is also prepared with a 3.6 µm Mylar® foil to prevent sample leakage and protect important system components. This guarantees maximum instrument availability, even with high throughput as required for process control at refineries.

Figure 2: Liquid sample cup (Ø 40 mm, left) and larger SampleCare cup (Ø 51 mm, right)

Figure 3: A preparation tool (black) is supplied for failsafe preparation of liquid cup

Calibration and Analyses

Due to the simple and straightforward sample preparation, the analytical results are available within minutes after taking the sample. Table 1 shows the optimized measurement parameters.

Table 1: Measurement parameters

Element	Tube voltage [kV]	Tube current [mA]	Measurement time [s]
S	25	1.7	300

In order to obtain best analytical results for ultra-low sulfur concentrations the measurements have been performed in 'Atmospheric Helium' mode. All measurements have been performed with liquid and SampleCare cup.

The S2 POLAR was calibrated by using a set of reference materials with 0, 5, 10, 30, 50, 70, und 100 ppm S, respectively. A typical calibration curve is shown in Figure 4 and sulfur signals for 0 to 25 ppm S in gasoline are shown in Figure 5.

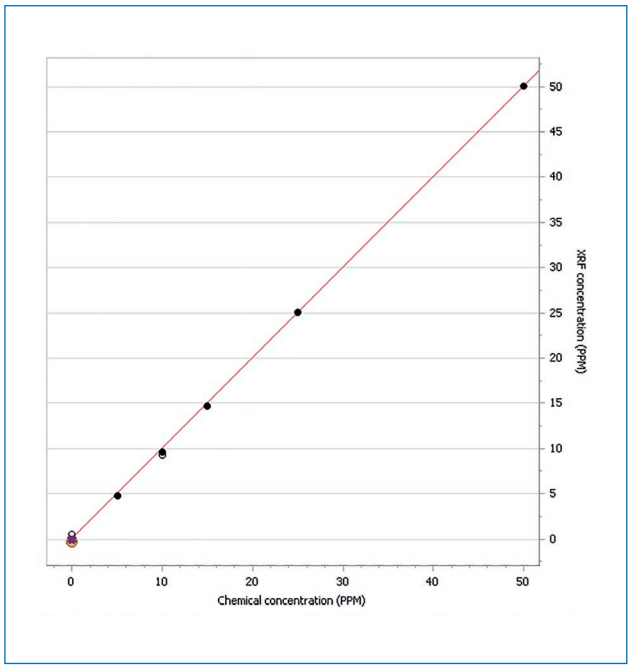

Figure 4: Calibration curve for sulfur in gasoline for the low concentration range between 0 to 50 ppm S

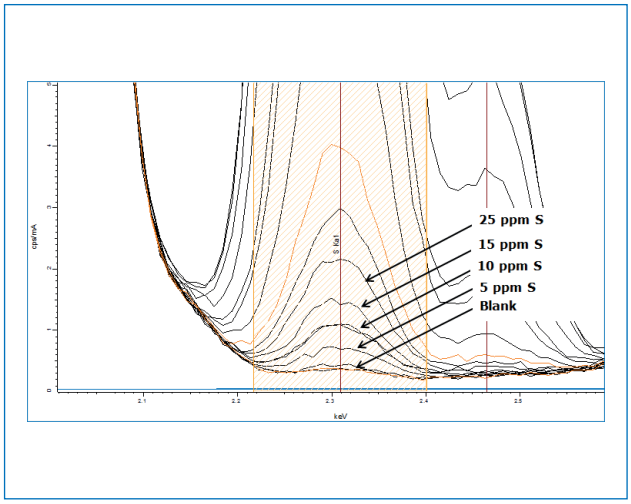

Figure 5: Overlaid sulfur signals in gasoline starting with a blank signal, followed here by 5, 10, 15 and 25 ppm S. The integration range is shown in orange.

Even the standard with the lowest S content (5 ppm S) shows a clear peak and confirms the excellent analytical performance of the S2 POLAR.

To ensure optimal performance the ISO 13032 solution contains the required quality control (QC) blank sample, one QC sample for the lower concentration range, and one QC sample for a higher concentration as well as a drift correction sample. There is no need for extensive method set-up and the instrument is immediately available for routine operation. In addition, there is no daily re-calibration needed in XRF.

Norm Compliance, Repeatability, and Precision

ISO 13032 defines a certain signal to background performance ratio. The polarized HighSense™ beam of the S2 POLAR easily fulfils this performance criterion. Repeatability tests proof the excellent performance of the S2 POLAR even for ultra-low sulfur levels (Table 2). The outstanding precision and accuracy of the system for S analyses easily complies with ISO 13032 and prepares your lab for even more stringent requirements.

Table 2: Repeatability of gasoline analyses

# Measurement	S [ppm]
1	11.7
2	11.3
3	11.9
4	12.1
5	11.7
6	12.2
7	11.3
8	11.6
9	11.4
10	11.2
Mean	11.64
Abs. Std. Dev.	0.33
Rel. Std. Dev. [%]	2.82

In order to determine the Lower Limit of Detection (LLD), 10 measurements of a blank gasoline sample containing no sulfur have been determined (Table 3). The LLD is then defined as three times the standard deviation of these measurements.

Table 3: Lower Limit of Detection (LLD) for gasoline

Element	LLD [ppm]	Measurement time [s]
S	0.7	300

Conclusion

The instrument is fully norm-compliant to the standard ISO 13032 and is even suitable to determine ultra-low sulfur levels much below the maximum regulated limit of 10 ppm S. The instrument is also compliant to international norms such as ISO 20847, ISO 8754, ASTM D7220, and ASTM D4294.

With factory pre-calibrated 'One-Button' TouchControl methods and the simple sample preparation, routine measurements are easy and straightforward. SampleCare prevents leakages of liquid samples and protects vital system components. This guarantees minimal maintenance and maximum instrument availability. The compact instrument is suitable for on-site process control at refineries or laboratories as well as for monitoring the downstream supply chain of pipelines, tank farms, oil terminals, and petrol stations.

Mylar® is a trademark of DuPont Nemours Inc.

Bruker AXS GmbH
info.baxs@bruker.com

Worldwide offices
bruker.com/baxs-offices

Online information
bruker.com/s2polar

www.bruker.com