



S2 POLAR

DETERMINATION OF S, CL AND BR IN AROMATIC HYDROCARBONS (XYLENE)

Xylenes are an important petrochemical products generated by catalytic reforming and also by coal carbonisation in coke fuel manufacturing. This material is widely used as a solvent in the leather, rubber, and printing industry. Xylene is also used in laboratories for baths with dry ice, as a solvent, and as a substitute for toluene. The process and quality control of xylenes in large scale production is very important and requires a sensitive and versatile analytical technique for the determination of halides and sulphur.

Instrumentation

The benchtop EDXRF spectrometer S2 POLAR with its HighSense™ beam path masters all the requirements for petrochemical applications (Fig. 1). The X-rays are polarised using a graphite target resulting into an excellent signal to background ratio, especially for S, P and Cl, and outstanding detection limits in the sub-ppm range for diesel and gasoline, as shown in lab report XRF 157.

The S2 POLAR is equipped with a high-power 50 W Pd X-ray tube and a HighSense ULS silicon drift detector with optimal spectral resolution that allows to measure multiple elements at the same time (LR XRF 155).

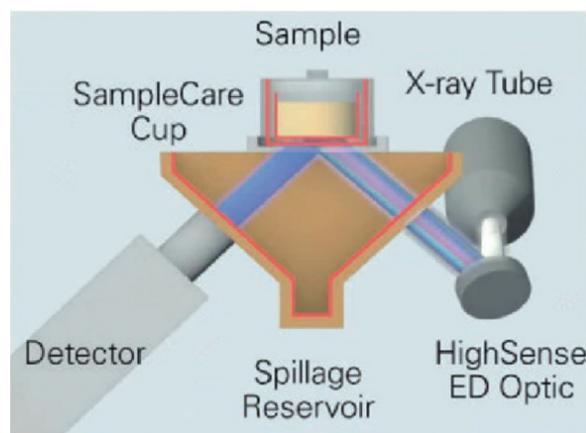


Figure 1: HighSense™ Technology in the S2 POLAR

Sample Preparation and Standards

For applications in the petro industry, the S2 POLAR offers a dedicated preparation accessory kit, that contains the required parts to prepare liquid samples. This kit includes liquid cups with 40 mm outer diameter (OD), SampleCare™ cups (OD = 51 mm) and Mylar® thin film (thickness 3.6 µm). The SampleCare™ cup prevents sample leakages into the system and thereby protects important system components, guaranteeing maximum instrument availability. Using of standardised liquid cups results in low costs per sample. For the sample preparation here, it is only required to weigh out 7.0 g liquid sample into the liquid cup and place it into a larger SampleCare™ cup for measurement (Fig. 2).



Figure 2: Preparation of liquid samples

Table 1: Concentration ranges of the standards

Elemental	Concentration range [ppm]
S	0 - 150
Cl	0 - 242
Br	0 - 240

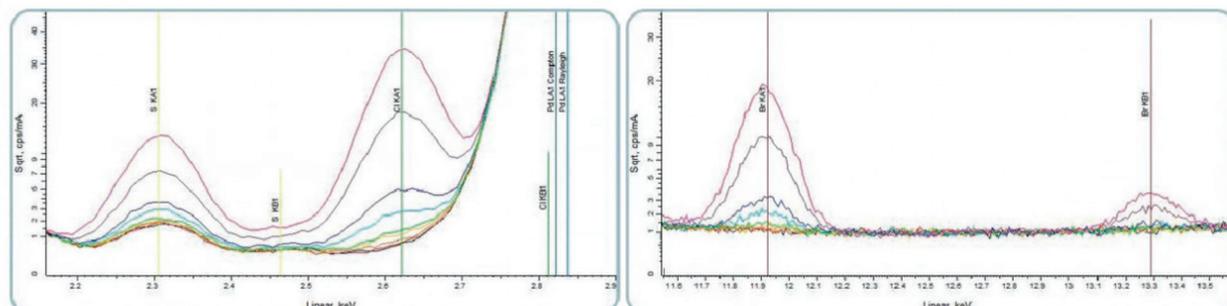


Figure 3: Scans of the S, Cl (left) and Br (left) characteristic lines

Measurement Method

For this application a method with two ranges was created, using atmospheric He as the measurement mode to ensure consistency of the volatile samples during the measurement process. Tab. 2 lists the measurement parameters. The automatic current mode adjusts the current until the maximum count rate is reached. The scans of the overlaid measurements are shown in Fig. 3.

Table 2: Measurement parameters

Range - Element	Voltage [kV]	Current [mA]	Count Rate [Cps]	Time [s]
# 1 - S, Cl	25	fix, 100	300,000	180
# 2 - Br	50	auto, 850	300,000	120

Calibration

The intensity was read at fixed peak positions and the background was calculated from the full measured spectrum. The calibration curves of S and Cl show excellent linearity. The calibration curve for Br exhibits high correlation as well and shows the versatility of the S2 POLAR even for heavier elements, see Fig. 4.

Precision Testing

A sample with known concentrations (not part of the calibration set) was measured 10 times. For each measurement the sample was prepared using fresh raw material and a new liquid cup. The results demonstrate high accuracy of the S2 POLAR (Tab. 3).

Table 3: Analytical precision test

Rep.	S [ppm]	Cl [ppm]	Br [ppm]
#01	23.7	30.5	29.3
#02	23.4	31.3	29.9
#03	23.4	31.3	29.0
#04	22.8	30.4	30.0
#05	23.5	29.9	27.6
#06	23.3	30.2	29.9
#07	23.3	31.1	29.2
#08	24.6	28.4	28.1
#09	24.4	29.7	28.9
#10	24.7	30.2	27.4
Average	23.7	30.3	28.9
Std. Dev.	0.6	0.8	0.9
Rel. Std. Dev.	2.70 %	2.74 %	3.25 %
Certified	25.0	30.0	30.0

Conclusion

The shown data proves the excellent analytical performance of the S2 POLAR for the analysis of S, Cl and Br in Xylenes. High instrument uptime is ensured with Bruker's SampleCare™ technology permitting cost-effective quality control of petroleum products. The easy-to-use, multilingual TouchControl™ in combination with the intuitive software SPECTRA.ELEMENTS and

the simple sample preparation allows using the S2 POLAR for routine analysis on day one with minimal training.

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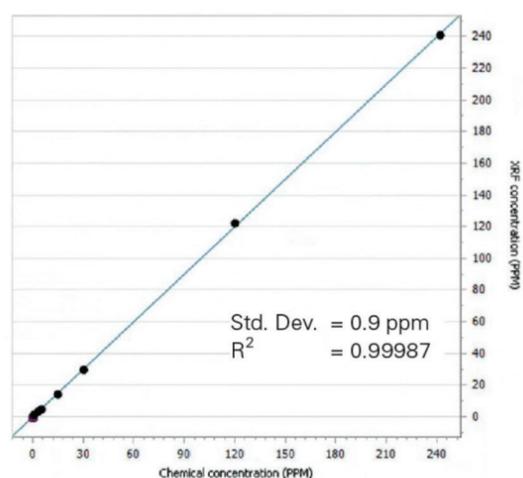
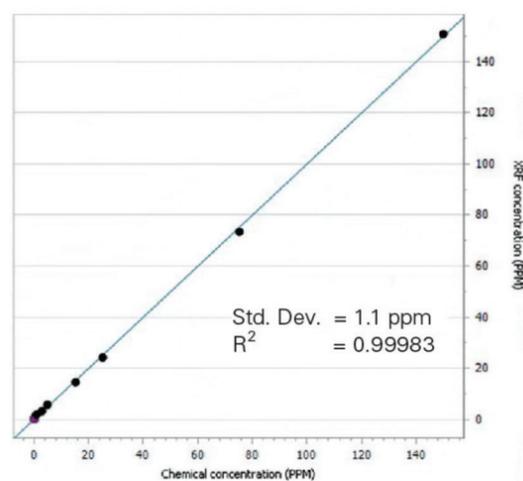
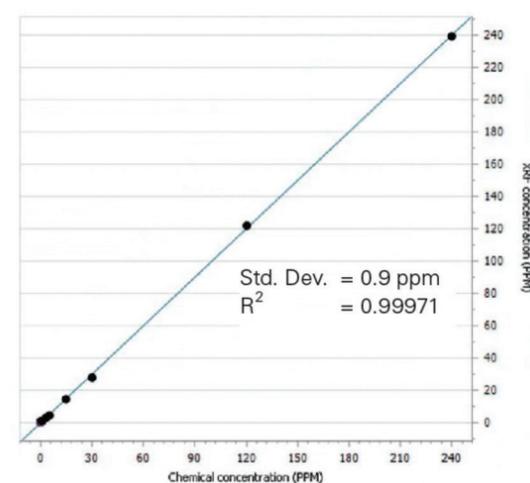


Figure 4: Calibration curves for S, Cl and Br