# Low Sulphur Analysis of Petroleum Products According to ASTM 2622 with the S8 TIGER

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*Sulphur in automotive fuels is a source of toxic air pollutants like sulphur dioxide and sulphates. Legislation enforces lower residual sulphur concentrations in fuels, like the actual limit of 10 ppm in Europe. Future regulations may drive these limits down even further. Wavelength-dispersive X-ray fluorescence (WDXRF) analysis is today the method of choice for accurately and precisely analysing low sulphur concentrations in petroleum products. WDXRF is a non-destructive method that is easily integrated into industrial environments such as refineries and laboratories. Simple sample preparation, reliability, ease-of-use and accurate analytical results are the main advantages of WDXRF. The outstanding analytical performance of the S8 TIGER exceeds the requirements of actual standards like ASTM 2622 and already meets future demands – a safe investment. This report shows how the S8 TIGER excels at low sulphur analysis of petroleum products.*


## Instrument

The S8 TIGER WDXRF spectrometer is the perfect solution to perform elemental analysis for the hydrocarbon processing industry. With the most flexible and compact beampath, high-performance X-ray tube and advanced analyser crystals, the S8 TIGER offers superior analytical performance in detection limits, precision and accuracy. The S8 TIGER is optimised for highest instrument uptime, lowest cost of ownership and ultimate reliability. SampleCare™ with 4x protection (including the unique vacuum seal) reduces the helium consumption and protects spectrometer components against damage from droplets or particles, even during measurements. For the determination of low sulphur concentrations in petroleum products, the S8 TIGER is equipped with a 4 kW rhodium X-ray tube, two collimators (0.23° and 0.46°) and four analyser crystals to cover the elemental range from N to U. The curved germanium crystal XS-GE-C provides 20-40% more intensity for the elements P, S and C, and is the best choice for the analysis of sulphur in fuels.

## Standard and Sample Preparation

Calibration according to ASTM 2622 was performed with ten standard samples prepared by using mineral oil and di-n-butyl sulfide covering a concentration range from 0 – 1000 ppm. The standards are commercially available and traceable to the NIST reference sample SRM 1616 a – sulphur in kerosene. Seven grams of the standard or sample were pipetted into a liquid cell prepared with m 3.6 ∝m Mylar film support. The samples were measured directly after preparation.

## Measurement

Measurements were performed on the S8 TIGER using the curved germanium crystal XS-GE-C, 0.46° collimator and 4 kW excitation with the settings 30 kV and 135 mA. Peak intensities were recorded to an amount of 1 x $10^5$ counts to meet the desired statistical error. The adjusted peak position of S Kα1 for the XS-GE-C crystal was set to a 2θ value of 110.698°, the background position was set at 115.22°. The measurement time was set to 25s for the peak and 25s for the background.

## Results

The calibration curve was calculated from the intensity of the ten standard samples by multiple regression. Calibration details are shown in Table 1, the curve is shown in *Figure 2*.

| Analyte S | Kα1 [XS-GE-C] |
|---|---|
| Peak [° 2θ] | 110.698° |
| Bkg [° 2θ] | 115.220° |
| Sensitivity | 387.2 kcps/% |
| Cal.Offs. | 0.4 ppm |
| Cal. Dev. | 2.0 ppm |
| $r_2$ | 0.999961 |
| LLD | 0.2 ppm [3σ, 100 s] |

To test the precision of the method, two samples with sulphur concentrations of 5 and 25 ppm were measured twenty times. The results and the statistical parameters are shown in *Figure 3*.

## Conclusions

The analysis of low sulphur concentrations in automotive fuels with the S8 TIGER is fast, precise and accurate. The high analytical performance with the optimised excitation and the increased sensitivity using the new analyser crystal XS-GE-C speeds up the analysis considerably. The presented data prove that today's and future regulations can be mastered by the S8 TIGER. The high precision of the S8 TIGER allows the safe identification of fuel sample very close to the federal limit values. The process to reduce the sulphur level in fuel therefore can be controlled better and doesn't require high safety ranges – a big cost advantage. The S8 TIGER is the perfect tool for elemental analysis of hydrocarbon samples especially in laboratory and industrial environments, due to the ease-of-use with TouchControl™ and the 4x protection of spectrometer components by SampleCare™.

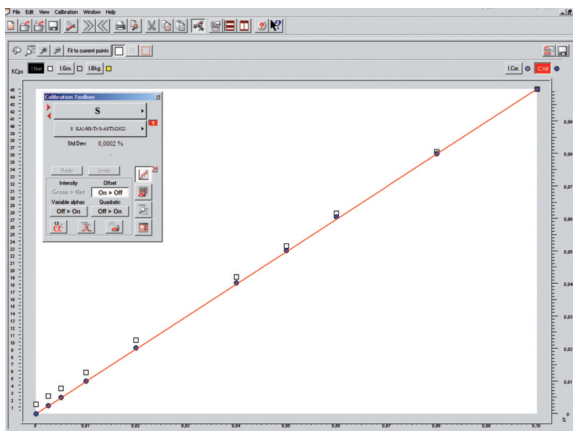

*Figure 2: Calibration curve for sulphur in petroleum products of concentratrion range 0 - 1000 ppm*

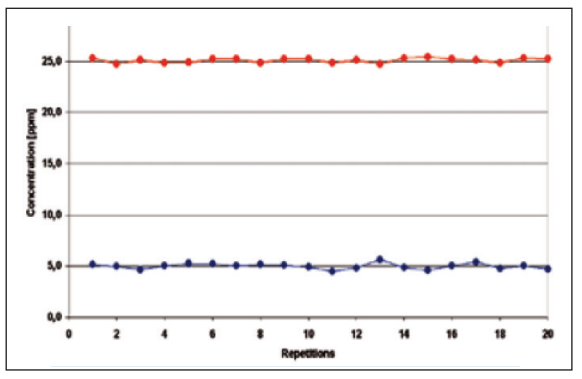


*Figure 3: Precision data for sulphur in fuels at two concentration levels*

## Revolutionary Addition to Range of Portable FT-IR Spectrometers Launched at Pittcon 2008

**A2 Technologies** (USA) showcased an exciting new addition to its innovative new range of FTIR spectrometers at Pittcon 2008, New Orleans, Louisiana. Further extending its range of portable FTIR spectrometers, A2 Technologies will be launching a revolutionary handheld portable FTIR spectrometer specifically designed for field-based surface analysis applications. Weighing only six pounds, the innovative light-weight analyser can be fully operated using only one hand and is also capable of handling Attenuated Transmission Reflectance (ATR) applications for users in the general laboratory. This FTIR analyzer was unveiled at Pittcon 2008. Dr. John Seelinbinder presented a product forum on behalf of A2 Technologies on this new development at Pittcon. Featuring interchangeable internal reflectance (ATR) and external reflectance sampling interfaces, this system is designed for sampling almost any material. Designed to analyze solids, pastes, gels and liquids with a traditional ATR, the novel spectrometer is also capable of analyzing large surface analysis with the A2 Technologies' unique diamond internal reflection sampling system. This highly compact spectrometer combines all the capabilities of a laboratory based FTIR with the advantages of a portable instrument that can be used in the field.

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