

AN APPLICATION REPORT FROM
SPECTRO ANALYTICAL INSTRUMENTS

XRF-130, REV. 0



SPECTRO XEPOS XEP05C

ASTM D4294: Analysis of Sulfur in Petroleum and Petroleum Products According to ASTM D4294 With a Benchtop ED-XRF Spectrometer

Introduction

X-ray fluorescence spectrometry is a very powerful analytical technique for the analysis of various elements in petroleum products. Energy dispersive X-ray fluorescence spectrometry (ED-XRF) is widely accepted for the analysis of sulfur in petroleum products.

One of the test methods describing this application is ASTM D4294.

This report describes the performance achievable with the benchtop ED-XRF analyzer SPECTRO XEPOS.

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The determination of sulfur in petroleum and petroleum products is an important analytical application because the sulfur dioxide resulting from fuel emissions reacts with oxygen and water vapor. This reaction causes sulfuric acid and, as a consequence, acid rains. Also for this application SPECTRO is driven to offer the best performance with its analytical instruments.

ED-XRF is a well approved technology in the petrochemical field. The SPECTRO XEPOS is well suited to meet and top the performance data for the analysis of sulfur as described in test methods.

In general, the SPECTRO XEPOS handles multiple applications at peak performance, demonstrating the analytical capabilities of ED-XRF technique at superior levels.

Instrumentation

The SPECTRO XEPOS C is equipped with an air-cooled, 50 W end-window X-ray tube which uses a binary-alloy cobalt-palladium anode. Up to two different excitation modes are applicable, consisting of an adaptive direct excitation system with optimized filters and a HAPG polarizer.

For the detection of fluorescence X-rays a high-resolution large area silicon drift detector (SDD) with Peltier cooling is used.

The spectral resolution (FWHM) of the SDD amounts to < 130 eV ($\text{Mn K}\alpha$) and the input count rate can be up to 1,000,000 cps.

The instrument is equipped with a vacuum pump and helium flush, utilizing the instrument's low-consumption helium purge. The used measurement parameters are displayed in the following Table 1.

kV	Mode	Meas.Time*
22.5	Polarization	100/300s

Table 1: Measurement parameters

*The measurement time was adjusted according to the sulfur concentration in the standards as recommended in the ASTM D4294.

Sample and sample preparation

Approx. 4 g of the liquid sample were filled into a disposable XRF sample cup with an outer diameter of 32 mm. The cup was closed on the analytical side with a 3 μm thin Etnom film. All standards have been analyzed using an additional safety cup also sealed with 3 μm thin Etnom film.

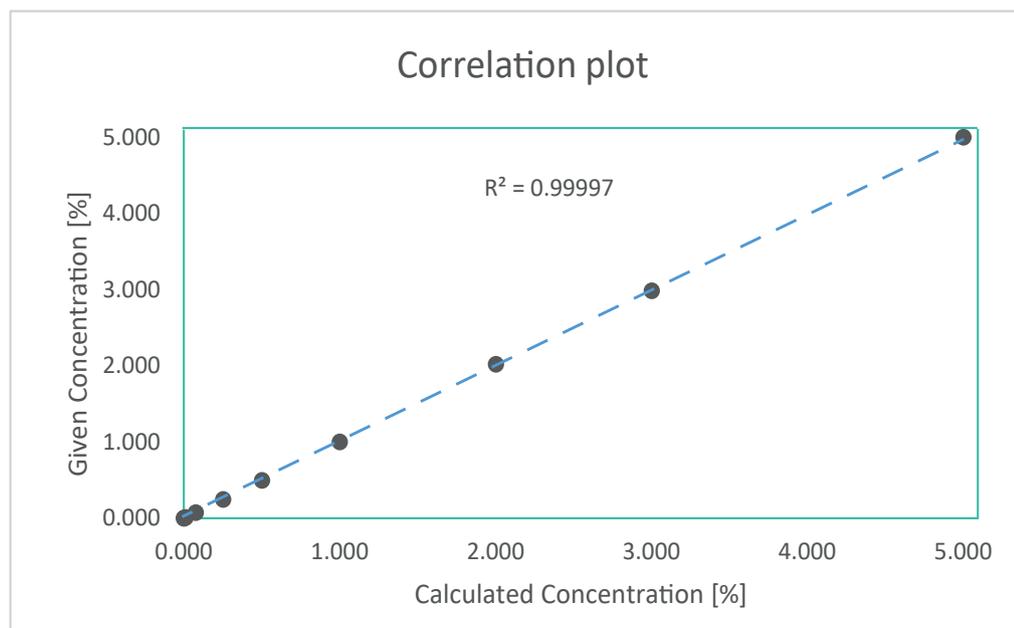
Calibration

Calibration for sulfur was performed by measuring a series of reference samples prepared according to ASTM D4294.

The calibration data are summarized in Table 2.

No. of Standards	Range [%]	Corr. Coef.
10	0-5	0.999985

Table 2: Calibration data



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Analytical performance

The reproducibility of the analyzer was tested by analyzing two samples at 0.02 % and 2.00 % sulfur content. For the measurements, each sample was freshly prepared.

Table 3: Analytical results of the repeated analysis of the sample containing 0.02 % S

Sample name	S in %
QC low (1)	0.01954 ± 0.00005
QC low (2)	0.01969 ± 0.00005
QC low (3)	0.01969 ± 0.00005
QC low (4)	0.01972 ± 0.00005
QC low (5)	0.01957 ± 0.00005
QC low (6)	0.01963 ± 0.00005
QC low (7)	0.01958 ± 0.00005
QC low (8)	0.01961 ± 0.00005
QC low (9)	0.01967 ± 0.00005
QC low (10)	0.01971 ± 0.00005
Average	0.01964
Std. dev.	0.00006

Table 4: Analytical results of the repeated analysis of the sample containing 2.00 % S

Sample name	S in %
QC High (1)	2.002 ± 0.001
QC High (2)	1.987 ± 0.001
QC High (3)	2.002 ± 0.001
QC High (4)	2.004 ± 0.001
QC High (5)	2.007 ± 0.001
QC High (6)	2.006 ± 0.001
QC High (7)	1.996 ± 0.001
QC High (8)	1.998 ± 0.001
QC High (9)	2.002 ± 0.001
QC High (10)	2.003 ± 0.001
Average	2.001
Std. dev.	0.006

Both results from the reproducibility test show compliance with the statements from ASTM D4294. Based on the measured standards the limit of detection for sulfur is estimated to be 0.2 mg/kg.

Summary

The SPECTRO XEPOS simultaneous XRF spectrometer provides a fast, precise, accurate and economic solution for the analysis of sulfur in petroleum and petroleum products according to ASTM D4294.



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