

# Analysis of Sulfur in Oils according to ASTM D2622 Method

ARL ADVANT'X Series with IntelliPower™  
Sequential X-Ray Fluorescence Spectrometer

## Introduction

In order to demonstrate the analysis of sulfur (S) in oil, a calibration curve has been constructed according to the ASTM D2622 norm

## Instrumentation

The powerful and stable Thermo Scientific ARL ADVANT'X WDXRF instrument has been used. It includes an option for fast change-over of the analysis environment from vacuum to helium atmosphere (< 2 min.) which allows liquids analysis.



## Sulfur analysis in oils

The ASTM D2622 norm has been followed and six standard samples have been prepared in order to construct a calibration curve. The analytical conditions were set according to the ASTM norms.

The S net intensities have been determined with the following formula:

$$S \text{ net} = (S \text{ Ka1, 2} - S \text{ background})$$

The intensity of each standard has been reported to the S content in ppm, which resulted in the calibration curve shown in Figure 1. Table 2 gives the numerical results including the absolute difference between nominal and calculated concentrations, as well as the Standard Error of Estimate.

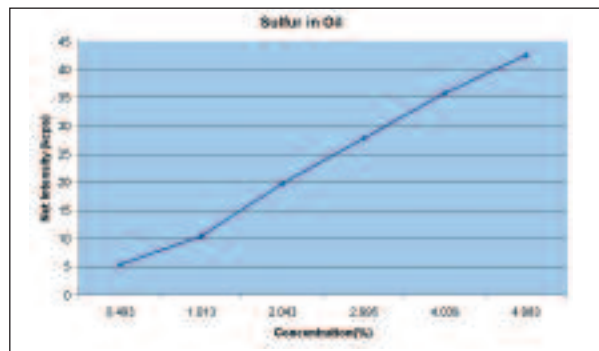


Figure 1

SAMPLE #	S NET KILOCOUNTS/S	NOM. CONCENTRATION %	CALCULATED CONCENTRATION %	ABSOLUTE DIFFERENCE %
1	5.370	0.502	0.493	-0.009
2	10.447	0.998	1.013	0.015
3	19.804	2.045	2.043	-0.0016
4	27.740	3.009	2.995	-0.014
5	35.814	4.027	4.039	0.012
6	42.506	4.986	4.983	-0.0033
Standard error of estimate =				0.013 %

Table 1: Sulfur calibration data for high concentration levels

## Key Words

- ARL ADVANT'X
- ASTM D2622
- Oil
- Sulfur
- XRF

## Stability test on a sample of oil residue containing sulfur

A sulfur stability test has been made on an oil residue sample, loaded into three different liquid cells: A, B and C.

The instrument settings were the following:

	Tube volt. KV	Tube curr. mA	Detector	Collimator	Crystal
S Ka1,2	40	70	FPC	Coarse	Ge111
S background	40	70	FPC	Coarse	Ge111

FPC = Flow Proportional Counter

Table 2: Analytical conditions

Each cell was analyzed three times; the results are shown in Table 3.

Run #	Cell A S concentration in %	Cell B S concentration in %	Cell C S concentration in %
1	3.60	3.59	3.55
2	3.61	3.60	3.56
3	3.59	3.58	3.57
Average	3.60	3.59	3.56
St.Dev.	0.008	0.011	0.008
RSD	0.235	0.315	0.233

Table 3: Stability test on an oil residue sample

Examination of these results reveals the excellent repeatability of analysis that can be achieved for S in oil with the ARL ADVANT'XP XRF.

## Analysis of unknown oil samples

Four unknown samples were prepared and each was loaded into three different cells: crude oil, heavy oil and two different oil residues.

Table 4 shows the repeatability results obtained as well as a comparison with the certified sulfur concentration.

Sample	Crude oil	Heavy oil	Oil residue 1	Oil residue 2
Certified value %	2.05	2.10	4.6	3.6
1 <sup>st</sup> run %	1.998	2.072	4.785	3.612
2 <sup>nd</sup> run %	2.000	2.063	4.772	3.616
3 <sup>rd</sup> run %	1.999	2.068	4.771	3.600
Average %	1.999	2.068	4.776	3.609
St.Dev. %	0.002	0.004	0.008	0.008
RSD %	0.121	0.215	0.169	0.235

Table 4: Analytical results and comparisons for 4 unknown oil samples

## Conclusion

Good calibration curves can be obtained with the ARL ADVANT'X for sulfur determination in oil when applying the appropriate ASTM standard method.

Thanks to the reproducibility of loading and to the Thermo Scientific helium shutter protecting the goniometer chamber from the helium environment, high repeatability of analysis can be demonstrated for S analysis.

The results obtained either for concentrations or for repeatability show that very good accuracy and precision can be achieved with the ARL ADVANT'X instrument. This instrument is well suited for the analysis of S in petrochemical products.

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