ASOMA® PHOENIX II

Determination of Sulfur in Crude Oil Using Direct Excitation

Summary

This report demonstrates the capability of the PHOENIX II benchtop XRF analyzer to determine sulfur content in crude oil using direct excitation optics. The performance shown also applies to other hydrocarbon oils, such as residual oil and bunker fuels.

The PHOENIX II quickly and precisely gives results for sulfur in diesel fuel at-line during monitoring and quality control of the fuel. In addition to ensuring the quality of the oil, the XRF measurement requires little if any sample preparation. These benefits work together to maximize quality and reduce operational costs.

The PHOENIX II complies with:

- ASTM D4294
- IP 336
- IP 496
- ISO 8754
- ISO 20847

Introduction

The PHOENIX II direct excitation system is an excellent QC benchtop analyzer that offers a fast, precise, simple and non-destructive analysis technique well suited for the analysis of sulfur in crude oil and petroleum products.

The PHOENIX II is a powerful tool for monitoring sulfur. The analyzer uses a rugged, time-proven proportional counter as its detection system and a direct excitation X-ray tube. This combination of ruggedness and simplicity using a small benchtop analyzer enables fast and precise results.

Data handling and results storage can be obtained on a paper print out and are stored in the hard drive of the PHOENIX II. The data can be readily transferred to a USB thumbdrive or accessed via an Ethernet connection.

Calibration is easily carried out with certified assayed standards. This ensures traceability of results for quality purposes. The calibration process is a “once only” procedure in which the curve can be revalidated by using a simple standardization procedure.

The PHOENIX II offers power, versatility and performance all in a small, compact, easy-to-use design.

Experimental Portion

Equipment

All measurements were conducted using a PHOENIX II XRF analyzer using direct excitation optics. Performance is shown for a measurement time of 200 seconds.

Sample Preparation

Sample preparation is minimal. Ensure the oil sample is well-mixed and stable. A sample is simply poured into a commercially available XRF sample cup. The instrument’s optical system is protected from inadvertent oil spillage by an easily changeable safety window.

Measurement Parameters

All measurement parameters are easily controlled using the touch screen PC. Operators simply choose the correct method from the analysis screen (there

may be more than one method stored, e.g. for crude oil, diesel fuel, bunker fuel, residual oil, etc.) and then press the green ANALYZE button.

The results can be reported using a variety of different options: results are reported on the display screen; on a thermal paper printout; and in the database history within the analyzer.

Instrument Configuration

ASOMA PHOENIX II

Excitation: Direct excitation 30 kV 9 W Air-cooled X-ray tube
Detection: Gas-filled Proportional Counter
Analytes Optimization: X-ray voltage, current and X-ray filters
Atmosphere: Air
Options: None

Note: No consumable gases required.

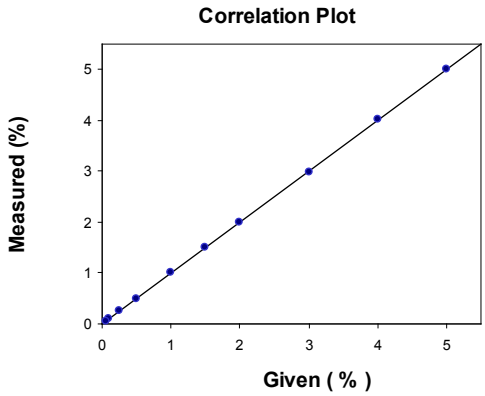
XRF Sample Cup

Easy assembly with film window

Results for S in Crude Oil

Calibration for S in Crude Oil

Element: S	Std. Error of Estimate: 0.0077	
Units: %	R.M.S.: 0.0026	
Sample	**Given**	**Measured**
1	5.0	5.00
2	4.0	4.01
3	3.0	2.98
4	2.0	2.01
5	1.5	1.50
6	1.0	1.01
7	0.5	0.50
8	0.25	0.249
9	0.10	0.099
10	0.05	0.048



Precision for S in Crude Oil

10 repeat analyses at 200 seconds per measurement

Element: S				Units: %
Sample	**Given**	**Mean**	**Std. Dev.**	**%Rel.**
1	5.0	4.992	0.008	0.2
6	1.0	1.019	0.003	0.3
10	0.05	0.0510	0.0005	1.0

Minimum Detection Limit (MDL)
S in Used Oil

The Minimum Detection Limit (MDL) for an element is determined as three times the standard deviation of ten analyses of the blank mineral oil sample containing no sulfur. The following MDL was derived using this empirical method and applies to this matrix and concentration range.

Element	Count Time	MDL
S	200 sec	21 ppm

Conclusion

As can be seen from the above data, the use of the PHOENIX II XRF system using direct excitation gives excellent performance when applied to the determination of sulfur in crude oil. Results are rapid, precise and analysis is easily carried out, even by non-laboratory personnel. Because no consumable chemicals are used, the relative "cost of ownership" is much lower than other analytical techniques.

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Application Note

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