

ASOMA® PHOENIX II

Determination of Sulfur in Petroleum Coke Using Direct Excitation

Summary

This report demonstrates the capability of the ASOMA PHOENIX II benchtop XRF analyzer to determine sulfur content in petroleum coke using direct excitation optics.

The PHOENIX II quickly and precisely gives results for sulfur in petroleum coke at-line during monitoring and quality control of the cracking process, and is monitored by endusers to ensure product quality. In addition to ensuring the quality of the coke, the XRF measurement requires little if any sample preparation. These benefits work together to maximize quality and reduce operational costs.

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 petroleum coke.

The PHOENIX II is a powerful tool for monitoring sulfur in oils, fuels and petroleum coke. The analyzer uses a rugged, time-proven proportional counter as its detection system and a direct excitation X-ray tube. This combination of ruggedness, power 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 thumb-drive 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. The samples were ground into a homogenous powder; poured into a prepared sample cup; pressed with 250 in-lb of torque using a manual sample press; and placed on the aperture for analysis. The instrument's optical system is protected from inadvertent powder 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 diesel fuel, crude oil, 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

Analyte Optimization: X-ray voltage, current and X-ray filters

Atmosphere: Air

Options: Manual sample press

Note: No consumable gases required.

XRF Sample Cup



Manual Sample Press

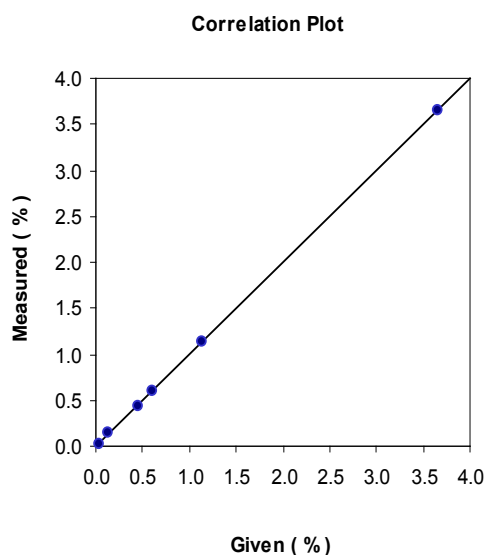


The following results section shows performance for typical sulfur in petroleum coke samples, submitted by an ASOMA user.

Results

Calibration for Sulfur in Petroleum Coke

Element: S		Std. Error of Estimate: 0.013	
Units: %		RMS: 0.004	
Sample	Given	Measured	
1	0.03	0.028	
2	0.14	0.154	
3	0.45	0.434	
4	0.60	0.596	
5	1.13	1.139	
6	3.66	3.659	



The achievable accuracy for the analysis of S in petroleum coke depends on the matrix matching of standards and samples.

Precision Sulfur in Petroleum Coke
10 repeat analyses at 200 seconds per measurement

Element: S		Units: %		
Sample	Given	Mean	Std. Dev.	% Rel.
1	0.03	0.028	0.0005	1.61
5	1.13	1.142	0.0038	0.34
6	3.66	3.653	0.0045	0.12

Minimum Detection Limit
Sulfur in Petroleum Coke

The Minimum Detection Limit (MDL) for an element is determined as three times the standard deviation of ten analyses of the blank 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	10 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 petroleum coke. 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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F-0290 Rev. 2 (0511)

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