ASOMA® Application Note

ASOMA PHOENIX II

Determination of Sulfur in Crude Oil and Fuel Oils

Summary

Samples were analyzed using the ASOMA PHOENIX II XRF Benchtop system. This report demonstrates the capability of the ASOMA PHOENIX II to analyze sulfur in fuels from 15 ppm to 5%. The PHOENIX II with its polarized source x-rays and proportional counter, complete with programmable detector filters, offers unparalleled sensitivity and precision compared to other XRF analyzers in its price range.

In addition to ensuring product quality throughout the process, XRF petroleum applications require little sample preparation and the analysis time is typically less than four minutes. The sample is simply poured into an XRF sample cup and analyzed. These benefits work together to maximize quality and reduce operational costs.

The PHOENIX II conforms to ASTM D4294, IP 496, IP 336, ISO 8754 & ISO 20847 for the analysis of sulfur in petroleum oils.

1. Introduction

The ASOMA PHOENIX II is an excellent benchtop XRF analyzer for at-line QC analysis or the laboratory alike. The PHOENIX II offers a fast, precise, simple and non-destructive analysis technique well suited for the determination of sulfur in crude oil and fuel oils, such as bunker fuels, residual oil, diesel, kerosene, jet fuels, etc.

The ASOMA PHOENIX II employs state-of-the-art optics. Polarization excitation offers unique benefits because it eliminates most of the background scatter emerging from the x-ray tube before it arrives at the sample. This results in a dramatic improvement in peak-to-background signal, especially in highly scattering materials such as petrochemical products. This translates to vastly improved precision and lower detection limits than traditional direct excitation XRF systems can achieve.

The PHOENIX II uses an onboard Panel PC computer with a simple touch screen interface. Thus, an external computer is not required. Data handling and results storage can be printed on the built-in thermal paper printer and are also stored in the hard drive of the PHOENIX II. The data can be readily transferred to a USB thumb-drive or a via an Ethernet connection.

Calibrations are easily created using assayed standards. This ensures the traceability of results for quality control purposes. This initial calibration process is a “once only” procedure. Subsequently, the calibration can be updated through the use of a single standard.

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

2. Experimental Portion

Equipment

All measurements were conducted using a ASOMA PHOENIX II XRF analyzer. The total analysis time per sample was 300 seconds for the standard sulfur in diesel application. The total analysis time for the sulfur in crude oil and high sulfur in diesel application was 200 seconds.

Sample Preparation

Each sample was simply shaken, poured into a sample cup, and placed in the sample chamber for analysis.

Measurement Parameters

All measurement parameters are easily controlled through the touch screen on the display panel. Operators simply choose the correct method from the analysis screen (there may be more than one method stored, e.g. to deal with diesel samples or crude oil samples) 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; on an optional external printer; and in the database history within the analyzer.

Instrument Configuration

ASOMA PHOENIX II

Excitation: 48 kV 50 W Air-cooled X-ray Tube

Detection: Gas-filled Proportional Counter

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

Atmosphere: Air

Options: HOPG crystal for polarized X-rays; Detector Filter; Polypropylene 4.0um film

Note: No helium purge is required.

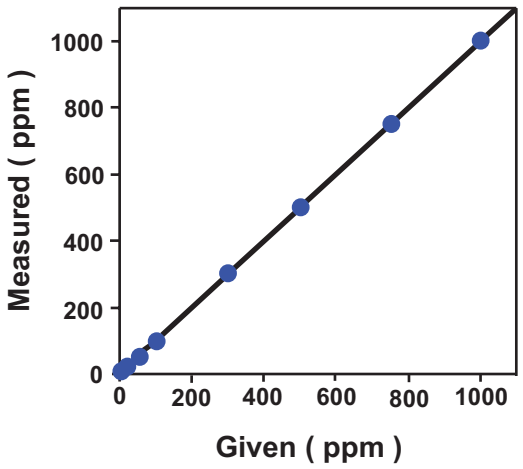
XRF Sample Cup

Easy assembly with film window

3. Calibration Results for Sulfur in Diesel Fuel

Element: S		
Units: ppm	Std. Error of Estimate: 3.3	
Sample	**Given**	**Measured**
1	15.0	15.6
2	26.0	26.2
3	50.0	49.1
4	100	95.9
5	300	302
6	500	504
7	750	745
8	1000	1002

Correlation Plot for S



Precision - Sulfur in Diesel Fuel

10 repeat analyses at 300 seconds per analysis

Element: S		Units: ppm		
Sample	**Given**	**Mean**	**Std. Dev.**	**% Rel.**
2	26.0	26.7	0.3	1.2
4	100	99.7	0.6	0.6
6	500	500.7	0.9	0.2
8	1000	1002.9	4.1	0.4

4. Calibration Results for High Sulfur in Diesel Fuel

Element: S	Std. Error of Estimate: 0.017	
Units: %	R.M.S.: 0.007	
Sample	**Given**	**Measured**
1	5.0	5.01
2	4.0	3.97
3	3.0	3.03
4	2.0	2.00
5	1.0	1.00
6	0.50	0.48
7	0.10	0.10

Precision - High Sulfur in Diesel Fuel

10 repeat analyses at 200 seconds per analysis

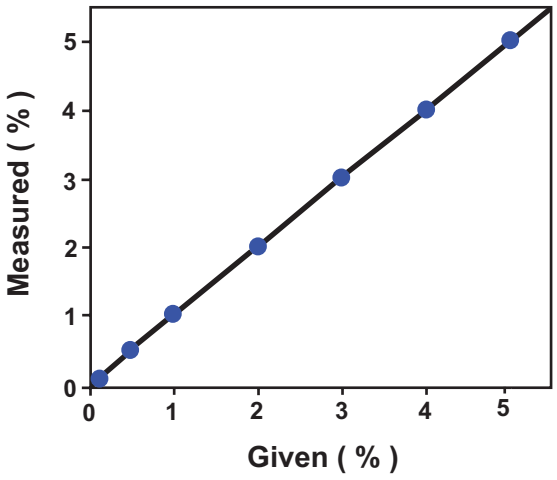
Element: S		Units: %		
Sample	**Given**	**Mean**	**Std. Dev.**	**% Rel.**
1	5.0	5.027	0.0146	0.29
4	2.0	2.053	0.0064	0.31
6	1.0	1.010	0.0046	0.46
8	0.1	0.096	0.0007	0.73

Note: Results for diesel fuel are indicative of results for kerosene and jet fuels.

5. Calibration Results for Sulfur in Crude Oil

Element: S	Std. Error of Estimate: 0.0039	
Units: %	R.M.S.: 0.0014	
Sample	**Given**	**Measured**
1	5.0	4.99
2	4.0	4.01
3	3.0	3.00
4	2.0	2.00
5	1.5	1.50
6	1.0	1.0
7	0.50	0.50
8	0.25	0.251
9	0.10	0.100
10	0.05	0.050

Correlation Plot



Precision - Sulfur in Crude Oil

10 repeat analyses at 200 seconds per analysis

Element: S		Units: %		
Sample	**Given**	**Mean**	**Std. Dev.**	**% Rel.**
1	5.0	4.970	0.0254	0.51
3	3.0	3.043	0.0105	0.35
6	1.0	1.014	0.0044	0.43
9	0.1	0.1003	0.0005	0.50

Note: Results for crude oil are indicative of results for bunker fuels and residual oil.

6. Minimum Detection Limit

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

Element	MDL
S	3 ppm

7. Conclusion

As can be seen from the above data, the use of the ASOMA PHOENIX II XRF system gives excellent performance when applied to the determination of sulfur in crude oil and fuel oils. Results are rapid, precise rapid. Because no consumable chemicals are used (only plastic sample cups and window film), the relative ”cost of ownership“ is much lower than many other analytical techniques.

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