

ASOMA® PHOENIX II

Determination of Lead and Sulfur in Fuel Oils

ASOMA® Phoenix II

All samples were analyzed using the ASOMA® PHOENIX II EDXRF Benchtop system with optics optimized for the measurement of lead. This report demonstrates the capability of the ASOMA® PHOENIX II to analyze lead and sulfur in gasoline, diesel and other fuel oils.

The PHOENIX II with its X-ray tube source and proportional counter, complete with programmable detector filters, offers unparalleled sensitivity and precision compared to other EDXRF analyzers in its price range.

In addition to ensuring product quality throughout the process, XRF fuel applications require little sample preparation. Simply pour a sample into an XRF sample cup and analyze. Both these benefits work together to maximize quality and reduce operational costs.

The sulfur performance shown conforms to ASTM D4294-08.

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 lead and sulfur in fuel oils.

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 PC computer with a simple touch screen interface. Thus, an external computer is not required. Data handling and results storage can be obtained on a thermal paper print out 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 readily carried out using assayed standards. This ensures easy traceability of results for quality purposes. This initial calibration process is a “once only” procedure. Subsequently, the curve can be restandardized if required, by the touch of a button on the main analysis screen.

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

2. Experimental Portion

Equipment

All measurements were conducted using an ASOMA® PHOENIX II XRF analyzer with optics optimized for the measurement of lead. The total analysis time per sample was 300 seconds for the low concentration lead in gasoline and the sulfur in diesel applications. The total analysis time for the higher concentration lead in gasoline application was 100 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 gasoline 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

Analytes Optimization: Display interface control of X-ray voltage, current and X-ray filters

Atmosphere: Air

Options: HOPG crystal for polarized X-rays; Moveable secondary target; Detector filters; 5 µm polycarbonate film

Note: No helium purge is required.

XRF Sample Cup



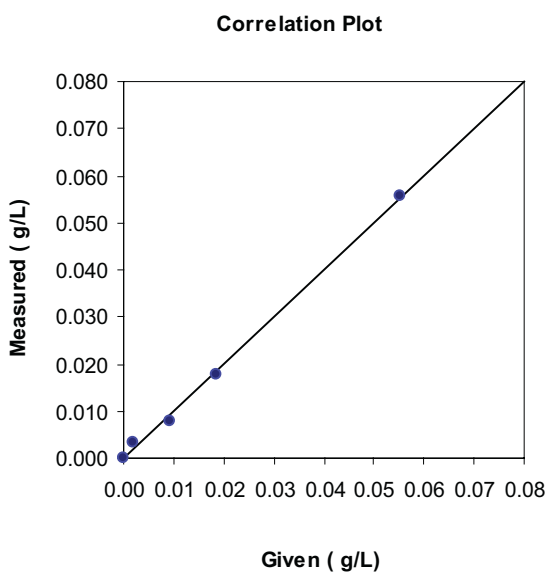
Easy assembly with film window



3. Results for Low Concentrations of Pb in Gasoline

Calibration for Lead

Element: Lead		
Units: g/L Std. Error of Estimate: 0.0011		
Sample	Given	Measured
1	0.0553	0.0555
2	0.0184	0.0179
3	0.0092	0.0080
4	0.0018	0.0031
5	0.0000	0.0001



Precision for Lead in Gasoline

10 repeat analyses at 300 seconds per measurement condition

Element: Pb		Units: g/L		
Sample	Given	Mean	Std. Dev.	% Rel.
1	0.0553	0.0545	0.0006	1.1
3	0.0092	0.0009	0.0004	4.4

Minimum Detection Limit (MDL) Pb in Gasoline

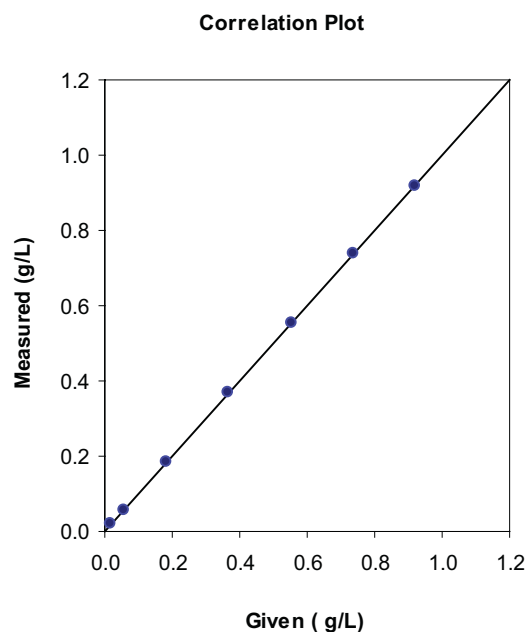
The Minimum Detection Limit (MDL) for an element is determined as three times the standard deviation of ten analyses of a blank gasoline sample. The following MDL was derived using this empirical method and applies to this matrix and concentration range.

Element	Count Time	MDL
Pb	300 sec	0.0007 g/L

4. Results for High Concentrations of Pb in Gasoline

Calibration for Lead

Element: Lead		
Units: g/L Std. Error of Estimate: 0.0018		
Sample	Given	Measured
1	0.018	0.019
2	0.055	0.056
3	0.184	0.183
4	0.368	0.368
5	0.553	0.551
6	0.737	0.740
7	0.921	0.919



Determination of Lead and Sulfur in Fuel Oils

Precision for Lead in Gasoline

10 repeat analyses at 100 seconds per measurement condition

Element: Pb		Units: g/L		
Sample	Given	Mean	Std. Dev.	% Rel.
1	0.018	0.0193	0.0007	3.6
5	0.553	0.5548	0.0011	0.2

Precision for S in Diesel

10 repeat analyses at 300 seconds per measurement condition

Element: S		Units: ppm		
Sample	Given	Mean	Std. Dev.	% Rel.
1	350	348.5	2.1	0.6
3	100	97.9	1.3	1.3
5	25	24.8	0.6	2.4

5. Results for Sulfur in Diesel

Calibration for Sulfur

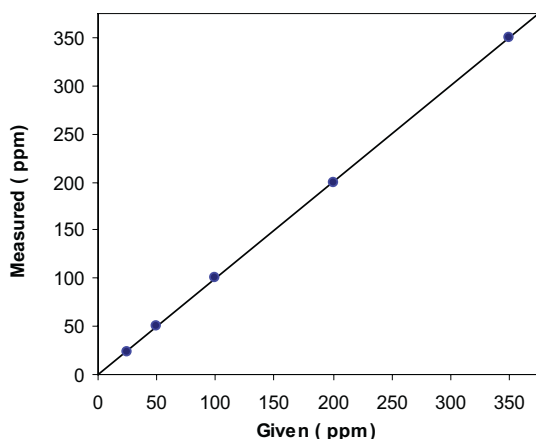
Element: Sulfur		
Units: ppm		Std. Error of Estimate: 1.14
Sample	Given	Measured
1	350	350.2
2	200	199.3
3	100	100.6
4	50	50.9
5	25	24.0

Minimum Detection Limit (MDL) S in Diesel

The Minimum Detection Limit (MDL) for an element is determined as three times the standard deviation of ten analyses of a blank diesel sample. The following MDL was derived using this empirical method and applies to this matrix and concentration range.

Element	Count Time	MDL
S	300 sec	3 ppm

Correlation Plot



6. Conclusion

As can be seen from the above data, the use of the ASOMA® PHOENIX II EDXRF system gives excellent performance when applied to the determination of lead and sulfur in fuels. Results are rapid, precise and analysis is easily carried out, even by non-laboratory personnel. Because no consumable chemicals are used (only plastic sample cups & window film), the relative "cost of ownership" is much lower than other analytical techniques.



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