

ASOMA® PHOENIX II

Measurement of Sulfur in ULSD (Ultra-Low Sulfur Diesel) for Product Screening/Identification

ASOMA® Phoenix II

All samples were analyzed using the PHOENIX II EDXRF Benchtop system. This report demonstrates the capability of the PHOENIX II to perform product screening and identification of ULSD based on elemental analysis of sulfur.

The PHOENIX II uses polarized source X-rays and a proportional counter detector, optimized for ULSD analysis. It offers unparalleled sensitivity and precision compared to traditional direct excitation XRF analyzers.

The PHOENIX II complies with:

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

Introduction

The ASOMA® PHOENIX II is a benchtop XRF analyzer designed for at-line QC analysis. The PHOENIX II is a tool ideally suited for ULSD product screening, identification and verification.

The XRF analyzers employ state-of-the-art optics. Polarization excitation offers unique benefits, eliminating 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, giving 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 touchscreen 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 network Ethernet connection.

Calibrations are readily carried out using assayed standards. This ensures easy traceability of results for quality purposes. 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.

Experimental Portion

Equipment

All measurements were conducted using a PHOENIX II XRF analyzer. The total analysis time per sample was 300 seconds.

Sample Preparation

Shake the bottle gently and allow bubbles to settle. Then simply place a measured amount of sample into an XRF sample cup and place the sample cup in the analysis chamber.

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).



To measure a sample, simply press the ANALYZE button on the main screen.

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: Helium purge

Options: HOPG for polarized X-rays; Detector filter; Polypropylene 4 µm film

Results for Sulfur in ULSD

Calibration

Element: S		
Units: ppm		Std. Error of Estimate: 0.35
Sample	Given	Measured
A	5.0	4.9
A	5.0	4.8
B	10.0	9.8
B	10.0	10.3
C	15.0	14.8
C	15.0	15.1
D	25.0	24.6
D	25.0	25.0
E	50.0	50.2
E	50.0	49.8

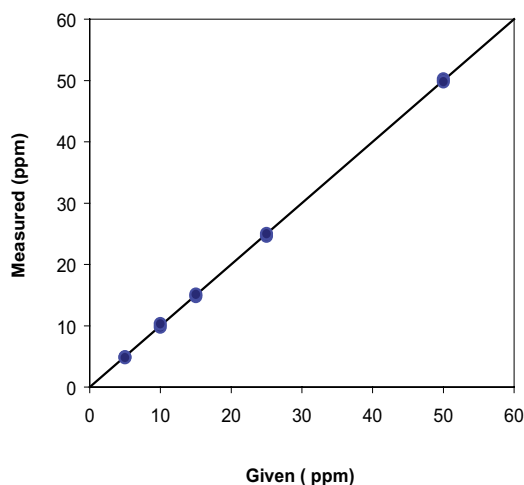
XRF Sample Cup



Easy assembly with film window



Correlation Plot



Precision

10 repeat analyses at 300 seconds per analysis

Element: Sulfur		Units: ppm		
Sample	Given	Mean	Std. Dev.	% Rel.
B	10.0	10.43	0.34	3.2
C	15.0	15.05	0.33	2.2
D	25.0	25.41	0.35	1.4
E	50.0	50.22	0.65	1.3

Reproducibility

One analysis per day for 20 consecutive days, using a calibration verification protocol before each analysis

Element: Sulfur		Units: ppm		
Sample	Given	Mean	Std. Dev.	% Rel.
Certified Sample	10.0	10.24	0.48	4.7

Minimum Detection Limit (MDL)

The Minimum Detection Limit (MDL) for an element is determined as three times the standard deviation of ten analyses of the blank sample. The following MDL was derived using this empirical method. The Lower Limit of Quantification is defined as 3 times the MDL.

Element	MDL
S	1.5 ppm

Conclusion

As can be seen from the above data, the use of the PHOENIX II XRF system gives excellent performance when applied to the determination of ultra-low sulfur in diesel for screening, product identification and verification uses. Results are rapid, precise and analysis is easily carried out, even by non-laboratory personnel. Because no consumable chemicals are used for sample preparation (only plastic sample cups and window film), the relative "cost of ownership" is much lower than other analytical techniques.



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