

## ASOMA® PHOENIX II

### Determination of S, Cl and Ca in Used Oils

#### Summary

This report demonstrates the suitability of the ASOMA® PHOENIX II XRF analyzer for determining the concentrations of common elements found in used oils. It will show that use of this instrument will deliver improvements in product quality by accurately and rapidly determining the important elements found in used oils; i.e., sulfur, chlorine and calcium. The performance shown in this report is also applicable to measuring these elements in used oils to be burned, waste oils for disposal, as well as cutting fluids.

The PHOENIX II can be used to minimize costs and maximize profitability.

#### Introduction

The 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, chlorine and calcium in used oils. Monitoring these elements is important in several applications. For example, government regulations limit the chlorine content of used oils to be burned as fuel, as well as in waste oil for proper disposal. Cutting fluids can also be monitored to ensure proper elemental levels to ensure the highest quality.

The 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 and oil-based 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 printout and are stored in the hard drive of the PHOENIX II. The data can be readily transferred to a USB thumbdrive or a network Ethernet connection.



Calibrations are readily carried out using assayed standards. This ensures 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, compact, easy-to-use design.

## Experimental Portion

### Equipment

All measurements were conducted using a PHOENIX II XRF analyzer. Performance is shown for using a total measurement time of 100 seconds.

### Sample Preparation

Sample preparation is minimal. Solution samples are 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 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 used oils or cutting fluids) 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:** S, Cl & Ca X-ray voltage, current and X-ray filters

**Atmosphere:** Air

**Options:** HOPG target ; Detector filters; Polypropylene 4 µm film

**Note:** No helium purge required.

## XRF Sample Cup



## Easy assembly with film window



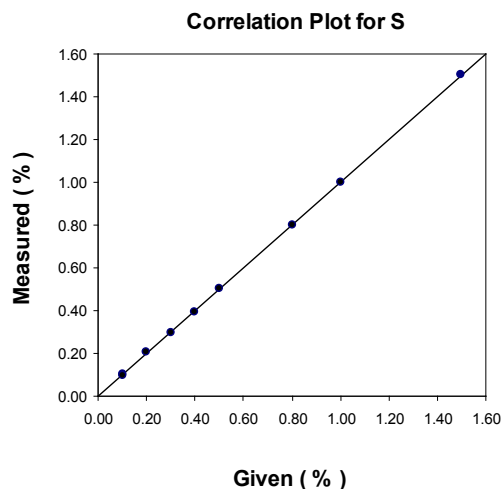
## Results for Used Oil with Constant Calcium

The results shown in this section use samples containing a constant 0.04% Ca.

### Element Sulfur

#### Calibration for Sulfur

Element: Sulfur		
Units: %      Std. Error of Estimate: 0.0047		
Sample	Given	Measured
1	0.10	0.100
2	0.10	0.096
3	0.40	0.394
4	0.30	0.298
5	0.50	0.504
6	0.80	0.801
7	1.50	1.501
8	0.20	0.209
9	1.00	0.998



#### Precision for Sulfur in Used Oil 10 repeat analyses at 100 seconds

Element: S			Units: %	
Sample	Given	Mean	Std. Dev.	%Rel.
3	0.40	0.399	0.002	0.4
5	0.50	0.494	0.002	0.4
8	0.20	0.208	0.0013	0.6

#### 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 a blank mineral oil sample. The following MDL was derived using this empirical method and applies to this matrix and concentration range.

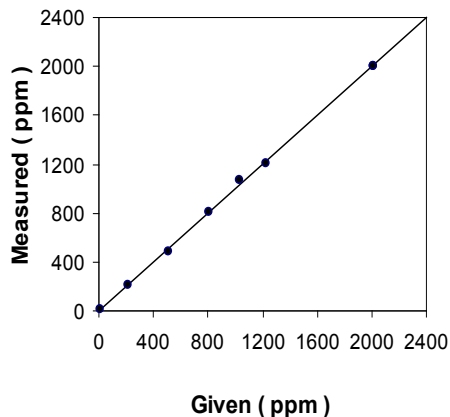
Element	Count Time	MDL
S	100 sec	0.0006 %

*Element Chlorine*

**Calibration for Chlorine**

Element: Chlorine		
Units: ppm		Std. Error of Estimate: 24.4
Sample	Given	Measured
1	10	13.7
2	210	215.1
3	510	480.2
4	810	808.3
5	1030	1073.3
6	1222	1210.1
8	2010	2004.6
9	2010	2006.6

**Correlation Plot for Cl**



**Precision for Cl in Used Oil**

**10 repeat analyses at 100 seconds**

Element: Cl			Units: ppm	
Sample	Given	Mean	Std. Dev.	% Rel.
3	510	501.1	10.1	2.0
5	1030	1065.6	12.2	1.1
8	2010	2014.6	11.0	0.6

**Minimum Detection Limit (MDL)**

**Cl in Used Oil**

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

Element	Count Time	MDL
Cl	100 sec	13 ppm

## Results for Used Oil with Variable Calcium

### Element Sulfur

#### Calibration for Sulfur

Element: Sulfur		
Units: %      Std. Error of Estimate: 0.0138		
Sample	Given	Measured
1	0.10	0.108
2	0.10	0.108
3	0.40	0.395
4	0.30	0.309
5	0.50	0.506
6	0.80	0.810
7	1.50	1.499
8	0.20	0.212
9	1.00	1.004
1c	0.10	0.111
2c	0.10	0.097
3c	0.40	0.378
4c	0.30	0.307
5c	0.50	0.497
6c	0.80	0.801
7c	1.50	1.513
8c	0.20	0.210
9c	1.00	0.967
10c	0.05	0.018

### Precision for S in Used Oil

#### 10 repeat analyses at 100 seconds

Element: S      Units: %				
Sample	Given	Mean	Std. Dev.	% Rel.
3c	0.40	0.386	0.001	0.3
7c	1.50	1.525	0.008	0.6
8c	0.20	0.209	0.002	1.0
2	0.10	0.108	0.001	0.5
9	1.00	1.028	0.006	0.6

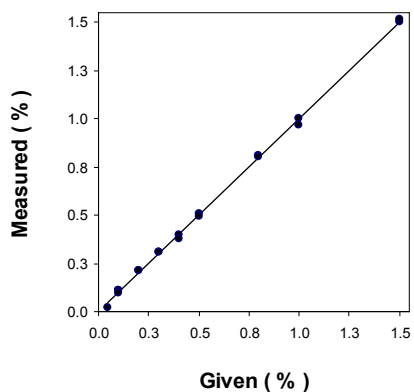
### Minimum Detection Limit (MDL)

#### 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 sample. The following MDL was derived using this empirical method and applies to this matrix and concentration range.

Element	Count Time	MDL
S	100 sec	0.0009 %

Correlation Plot for S



*Element Chlorine*

**Calibration for Chlorine**

Element: Cl		
Units: ppm		Std. Error of Estimate: 38.2
Sample	Given	Measured
1	10	14.1
2	210	212.5
3	510	486.5
4	810	822.0
5	1030	1076.1
6	1220	1245.9
7	1500	1479.6
8	2010	1974.5
1c	10	16.7
2c	210	226.6
3c	510	470.2
4c	810	818.2
5c	1000	1013.6
6c	1200	1185.4
7c	1500	1457.7
8c	2010	2017.4
9c	2000	1944.2

**Precision for Cl in Used Oil**

10 repeat analyses at 100 seconds

Element: Cl		Units: ppm		
Sample	Given	Mean	Std. Dev.	% Rel.
3c	510	454.7	10.3	2.3
7c	1510	1342.1	27.1	2.0
8c	2020	1961.5	16.1	0.8
2	210	211.8	7.0	3.3
9	2010	2117.5	66.6	3.2

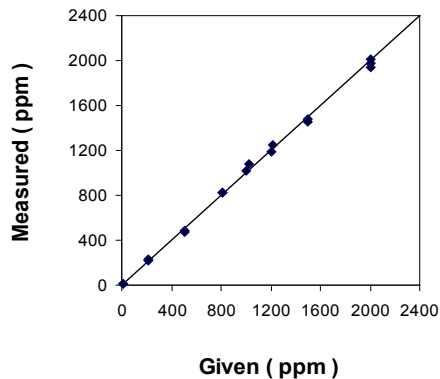
**Minimum Detection Limit (MDL)**

**Cl in Used Oil**

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

Element	Count Time	MDL
Cl	100 sec	10 ppm

**Correlation Plot for Cl**



*Element Calcium*

**Calibration for Calcium**

Element: Calcium		
Units: %		Std. Error of Estimate: 0.0085
Sample	Given	Measured
1	0.040	0.041
2	0.040	0.041
3	0.041	0.040
4	0.041	0.040
5	0.040	0.040
6	0.040	0.047
8	0.040	0.032
9	0.040	0.048
2c	0.250	0.252
3c	0.350	0.342
4c	0.040	0.037
5c	0.400	0.407
7c	0.150	0.144
8c	0.300	0.300

**Precision for Ca in Used Oil**  
10 repeat analyses at 100 seconds

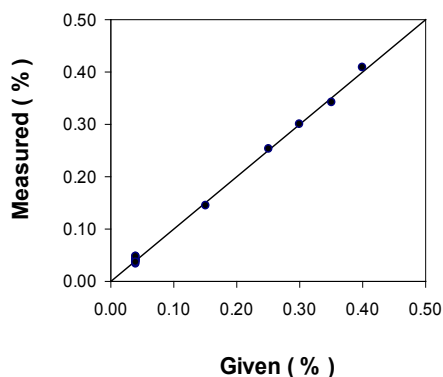
Element: Ca		Units: %		
Sample	Given	Mean	Std. Dev.	% Rel.
3c	0.350	0.358	0.005	1.4
7c	0.150	0.144	0.003	2.3
8c	0.300	0.307	0.004	1.4
2	0.040	0.040	0.002	5.8

**Minimum Detection Limit (MDL)**  
*Ca in Used Oil*

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

Element	Count Time	MDL
Ca	100 sec	0.0053 %

**Correlation Plot for Ca**



## 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 sulfur, chlorine and calcium in used oils. 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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