

## ASOMA® PHOENIX II

### Determination of Phosphorus & Bromine Flame Retardants in Fabric



#### ASOMA® PHOENIX II

This report demonstrates the suitability of the PHOENIX II XRF analyzer for use in the quality control measurement of P and Br flame retardants in fabric. It will show that use of this instrument will deliver improvements in product quality by accurately and rapidly determining the important flame retardant additive elements.

It can be seen that there is another advantage in easily and quickly measuring these elements during the textile production process. Namely, by ensuring that additive package “overdosing” does not occur, significant reductions in operational costs can be achieved. In addition, “underdosing” is eliminated and proper flame retardation characteristics achieved.

#### Introduction

The PHOENIX II is an excellent benchtop XRF analyzer for at-line production 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 phosphorus (P) and bromine (Br) flame-retardants in many various textiles. Other applications ideal for the PHOENIX II that are related to the textiles include anti-microbial agents and additives in plastic fibers and foam.

The PHOENIX II employs state-of-the-art optics. Polarization excitation offers unique benefits because it eliminates most of the back-ground 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 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 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. This initial calibration process is a “once only” procedure. Subsequently, the curve can be re-standardized, 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. A sample fabric swatch is cut and placed flat 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 P or Br) 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:** P, Br

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

**Atmosphere:** Air

**Options:** HOPG target; Secondary target; Detector filter, Polypropylene 4 µm film

**Note:** No helium purge is required.

## A fabric swatch in analysis position



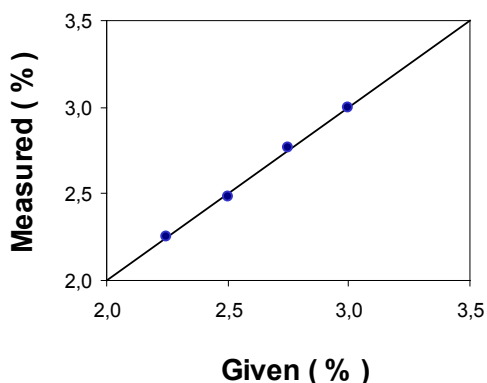
## Results for Phosphorus in Fabric

The calibration was built using an assayed suite of calibration standards of actual product provided by a current user, and are typical of results achievable for these types of applications. This phosphorus flame retardant is infused into the fabric fibers.

### Calibration for Phosphorus

Element: P		
Units: %	Std. Error of Estimate: 0.01211	
Sample	Given	Measured
8	2.25	2.256
2	2.50	2.486
3	2.75	2.762
5	3.00	2.996

**P Correlation Plot**



**Precision for P in Fabric**

10 repeat analyses at 100 seconds per measurement

Element: P		Units: %		
Sample	Given	Mean	Std. Dev.	% Rel.
8	2.25	2.234	0.002	0.1
5	3.00	3.011	0.004	0.1

**Minimum Detection Limit (MDL)**

**P in Fabric**

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

Element	Count Time	MDL
P	100 sec	0.0010 %

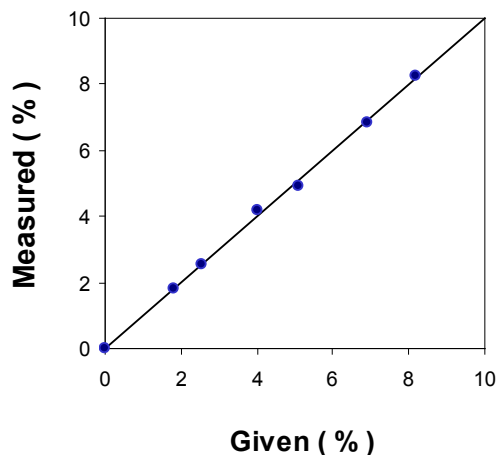
**Results for Bromine on Fabric**

The calibration was built using an assayed suite of calibration standards of actual product provided by a current user, and are typical of results achievable for these types of applications. This bromine flame retardant is coated onto the fabric. Some bromine flame retardants include the element antimony. If present, the antimony can also be measured with comparable results.

**Calibration for Bromine**

Element: Bromine		
Units: %		Std. Error of Estimate: 0.10966
Sample	Given	Measured
control	0.00	-0.005
7	1.80	1.820
2	2.57	2.534
3	4.02	4.204
4	5.08	4.914
5	6.89	6.816
6	8.18	8.258

**Br Correlation Plot**



**Precision for Br on Fabric**

10 repeat analyses at 100 seconds per measurement

Element: Br		Units: %		
Sample	Given	Mean	Std. Dev.	% Rel.
7	1.80	1.808	0.004	0.2
4	5.08	4.900	0.021	0.4
2	8.18	8.220	0.034	0.4

# Determination of Phosphorus & Bromine Flame Retardants in Fabric

## Minimum Detection Limit (MDL)

### Br on Fabric

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

Element	Count Time	MDL
Br	100 sec	0.0010 %

## 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 phosphorus and bromine flame-retardants in fabric. Antimony is often linked to bromine flame-retardants, and can also be measured with comparable performance. 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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