

## ASOMA® PHOENIX II

### Measurement of Titanium in Nylon Sheets and Other Polymers

#### ASOMA® Phoenix II

This report demonstrates the capability of the PHOENIX II Benchtop XRF analyzer for measuring titanium in nylon sheets and other polymers (polystyrene, polyethylene, polypropylene, etc.).

The PHOENIX II can quickly and accurately give results for titanium at-line. Titanium dioxide is added to Nylon and polymers as a pigment and its other chemical properties. Measuring the titanium ensures quality is monitored and controlled throughout the manufacturing process. The measurement require no sample preparation; simply place the Nylon sample in the chamber or fill an XRF sample cup with pellets and analyze. These benefits work together to maximize quality and reduce operational costs.

#### Introduction

The PHOENIX II is an excellent QC benchtop analyzer that offers a fast, precise, simple and non-destructive analysis technique well suited for the analysis of titanium in nylon and other polymers.

The PHOENIX II is an exceptionally powerful tool. The PHOENIX II uses the rugged, time-proven proportional counter as its detection section. A 48kV X-ray tube and indirect excitation with a HOPG polarizing crystal yield extremely low background for exceptional detection limits. The PHOENIX II combines ruggedness and power in a small, compact design with simplicity designed for non-technical operators.

The PHOENIX II can also be equipped with a moveable secondary target along with the HOPG crystal, making the PHOENIX II a versatile tool in measuring many other elements in polymers and chemicals, whether in solid or liquid form.

Data handling and results storage can be obtained on a 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 networked using the Ethernet connection.

Calibrations are readily carried out with assayed calibration standards. This ensures easy traceability of results for quality purposes. The calibration process is a “once only” procedure in which the curve can be revalidated by using a simple standardization procedure that automatically brings the calibrations back to original status.

#### Experimental Portion

##### **Equipment**

All measurements were conducted using a PHOENIX II XRF analyzer. The total analysis time per sample was 200 seconds.

##### **Sample Preparation**

No sample preparation is required. Nylon squares or each sample cup were placed in the sample chamber for analysis.

##### **Measurement Parameters**

All measurement parameters are easily controlled by a built in touch screen PC. Operators simply choose the correct method from the analysis screen (there may be more than one method stored, e.g. to deal with nylon and other polymers) and then press 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:** Compact, air-cooled X-ray tube

**Detection:** Gas-filled Proportional Counter

**Analyte Optimization:** Display interface control of X-ray voltage and current

**Atmosphere:** Air

**Options:** HOPG Polarizing crystal; flat window ring (nylon); Sample spinner

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

Nylon sample in analysis position



Cup assembly with film window



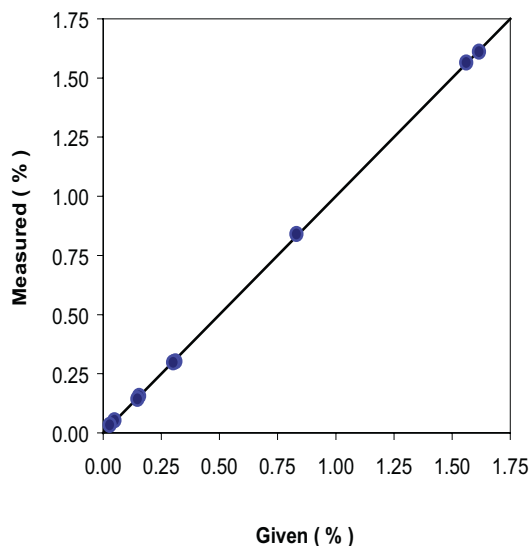
Sample Spinner



Calibration Results for the Titanium in Nylon Sheets

Element: Ti		
Units: % TiO <sub>2</sub> Std. Error of Estimate: 0.006		
Sample	Given	Measured
1	1.616	1.61
2	1.561	1.56
3	0.831	0.84
4	0.311	0.30
5	0.301	0.30
6	0.155	0.16
7	0.147	0.14
8	0.049	0.05
9	0.028	0.03

Correlation Plot



**Precision**

10 repeat analyses at 200 seconds per analysis

Element: Ti		Units: % TiO <sub>2</sub>		
Sample	Given	Mean	Std. Dev.	% Rel.
1	1.61	1.624	0.0029	0.18
5	0.301	0.298	0.0012	0.40
8	0.049	0.053	0.0003	0.57

**Minimum Detection Limit (MDL)**

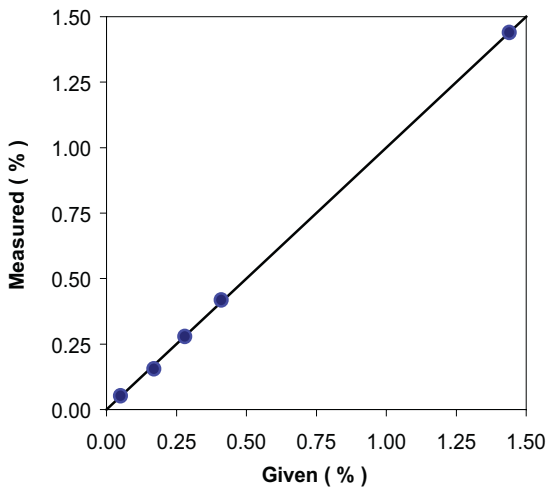
The Minimum Detection Limit (MDL) for an element is determined as three times the standard deviation of ten analyses of the blank sample. The following MDL was derived using this empirical method and applies to this matrix and concentration range.

Element	MDL
Ti	10 ppm TiO <sub>2</sub>

**Calibration Results for the Titanium in Polymer Pellets**

Element: Ti		
Units: % TiO <sub>2</sub> Std. Error of Estimate: 0.006		
Sample	Given	Measured
1	1.440	1.4395
2	0.050	0.0518
3	0.170	0.1553
4	0.280	0.2783
5	0.410	0.4182

Correlation Plot  
Correlation Plot



**Precision**

**10 repeat analyses at 200 seconds per analysis**

Element: Ti		Units: % TiO <sub>2</sub>		
Sample	Given	Mean	Std. Dev.	% Rel.
1	1.44	1.430	0.0059	0.4
2	0.05	0.052	0.0005	0.9
5	0.41	0.417	0.0012	0.3

**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 and applies to this matrix and concentration range.

Element	MDL
Ti	10 ppm TiO <sub>2</sub>

**Conclusion**

As can be seen from the above data, the use of the PHOENIX II XRF system gives excellent performance when applied to the quality control of titanium in Nylon and other polymers. Results are rapid and precise, and analysis is easily carried out by non-laboratory personnel. Because no consumable chemicals are used (only polymer sample cups and window film) the relative "cost of ownership" is much lower than other analytical techniques.



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