

# ASOMA PHOENIX II with Direct Excitation

## X-Ray Fluorescence Application Report - Cosmetics

### Objective

To calibrate the ASOMA PHOENIX II analyzer for  $\text{TiO}_2$  &  $\text{Fe}_2\text{O}_3$  in makeup powders,  $\text{TiO}_2$  in tinted makeup base,  $\text{TiO}_2$  in lotion (no iron), and  $\text{ZnO}$  in lotion, and to demonstrate instrument precision.

### Background

The accurate and precise measurement of titanium, iron and zinc oxides is important during the quality control process of the final product to determine if the cosmetics product is manufactured within the product specifications. This report details the measurement capabilities of the ASOMA PHOENIX II analyzer optimized for this purpose.

### Sample Preparation

The base/lotion-like samples were mixed using a glass stir bar. Each sample was then transferred to a sample cup using the stirring rod. The sample cup was filled consistently so that only ¼ inch at the top of the cup was left unfilled.

Care was taken not to introduce air bubbles and to spread the sample evenly on the bottom of the cup. The sample cup was then tapped approximately 10 times to help remove any remaining air bubbles.



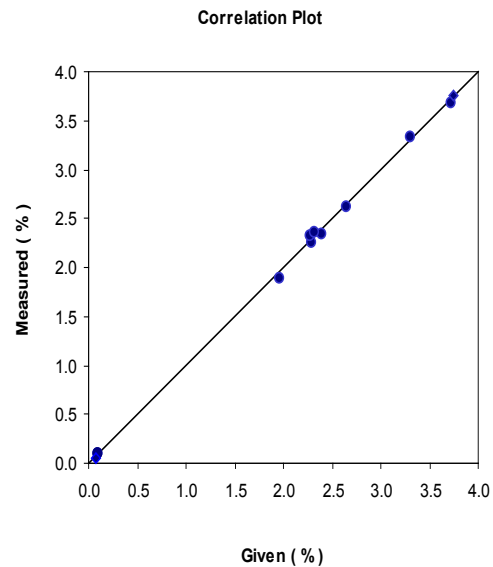
Each powdered sample was poured into a prepared samples cup and pressed using 250 in/lb of torque using a manual sample press prior to analysis.



<b>Instrument:</b>	<b>ASOMA Phoenix II Direct Excitation Optics</b>	<b>Analysis Time:</b>	100 seconds
<b>Source:</b>	X-Ray Tube	<b>Options:</b>	Tube filter, standard window ring, manual sample press
<b>Detector:</b>	Proportional Counter		

Calibration Results for TiO<sub>2</sub> in Makeup Powders

Element: Ti		
Units: %TiO <sub>2</sub> Std. Error of Estimate: 0.044		
Sample	Given	Measured
1	0.09	0.110
2	3.72	3.690
3	2.39	2.353
4	0.09	0.100
5	0.08	0.059
6	3.75	3.754
7	3.30	3.338
8	2.64	2.627
9	2.29	2.263
10	2.27	2.335
11	2.32	2.366
12	1.95	1.890

**Precision**

10 repeat analyses at 100 seconds per analysis

Element: Ti		Units: %TiO <sub>2</sub>		
Sample	Given	Mean	Std. Dev.	% Relative
2	3.72	3.714	0.011	0.3
5	0.08	0.062	0.003	5.6
12	1.95	1.903	0.009	0.5

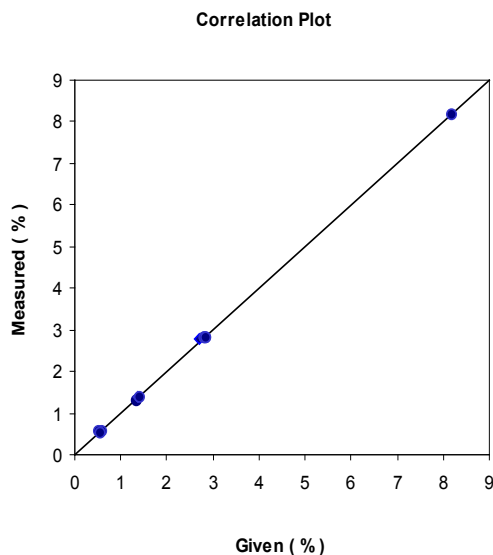
**Limit of Detection (LOD)**

The LOD (Limit of Detection) is defined as three times the standard deviation of analyzing the blank sample ten times in a static position. Based on analysis of the blank sample, Teflon, the LOD for this matrix and concentration range is:

Element	LOD
Ti	0.007 %TiO <sub>2</sub>

## Calibration Results for Fe<sub>2</sub>O<sub>3</sub> in Makeup Powders

Element: Fe		
Units: %Fe <sub>2</sub> O <sub>3</sub> Std. Error of Estimate: 0.030		
Sample	Given	Measured
1	0.57	0.555
2	0.53	0.566
3	0.54	0.550
4	1.34	1.292
5	2.72	2.767
6	1.37	1.391
7	2.80	2.826
8	2.85	2.815
9	8.19	8.188
10	2.81	2.816
11	1.41	1.381
12	2.85	2.836



### Precision

10 repeat analyses at 100 seconds per analysis

Element: Fe		Units: %Fe <sub>2</sub> O <sub>3</sub>		
Sample	Given	Mean	Std. Dev.	% Relative
2	0.53	0.572	0.002	0.4
6	1.37	1.382	0.006	0.4
9	8.19	8.200	0.026	0.3
12	2.85	2.832	0.005	0.2

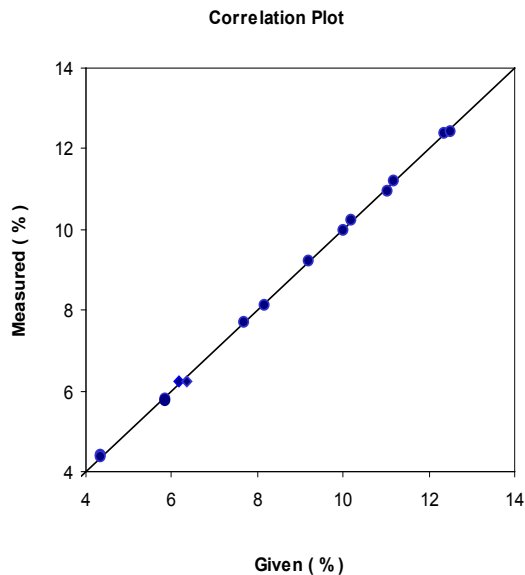
### Limit of Detection (LOD)

The LOD (Limit of Detection) is defined as three times the standard deviation of analyzing the blank sample ten times in a static position. Based on analysis of the blank sample, Teflon, the LOD for this matrix and concentration range is:

Element	LOD
Fe	0.002 %Fe <sub>2</sub> O <sub>3</sub>

Calibration Results for TiO<sub>2</sub> in Makeup Base

Element: Ti		
Units: %TiO <sub>2</sub> Std. Error of Estimate: 0.002		
Sample	Given	Measured
1	4.34	4.410
2	4.34	4.370
3	5.84	5.815
4	5.84	5.760
5	6.17	6.250
6	6.34	6.224
7	7.67	7.701
8	8.17	8.125
9	9.17	9.227
10	10.01	9.972
11	10.18	10.229
12	11.01	10.978
13	11.18	11.202
14	12.34	12.413
15	12.51	12.437



**Precision**

10 repeat analyses at 100 seconds per analysis

Element: Ti		Units: %TiO <sub>2</sub>		
Sample	Given	Mean	Std. Dev.	% Relative
2	4.34	4.321	0.008	0.2
7	7.67	7.719	0.018	0.2
10	10.01	10.037	0.024	0.2
14	12.34	12.437	0.025	0.2

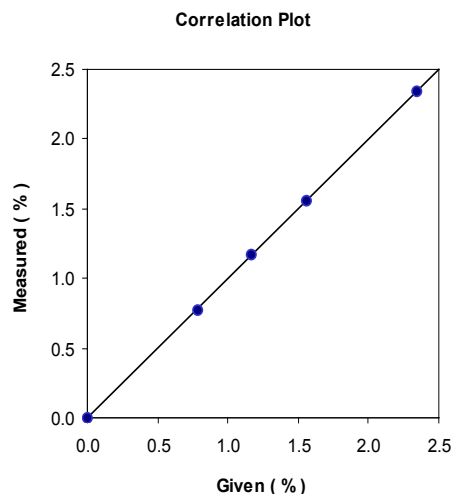
**Limit of Detection (LOD)**

The LOD (Limit of Detection) is defined as three times the standard deviation of analyzing the blank sample ten times in a static position. Based on analysis of the blank sample, Teflon, the LOD for this matrix and concentration range is:

Element	LOD
Ti	0.006 %TiO <sub>2</sub>

## Calibration Results for TiO<sub>2</sub> (no iron) in Lotion

Element: Ti		
Units: %TiO <sub>2</sub> Std. Error of Estimate: 0.002		
Sample	Given	Measured
1	2.34	2.341
2	1.56	1.559
3	1.17	1.171
4	0.78	0.778
5	0.00	0.001



### Precision

10 repeat analyses at 100 seconds per analysis

Element: Ti		Units: %TiO <sub>2</sub>		
Sample	Given	Mean	Std. Dev.	% Relative
1	2.34	2.366	0.004	0.2
2	1.56	1.562	0.003	0.2
4	0.78	0.785	0.003	0.4

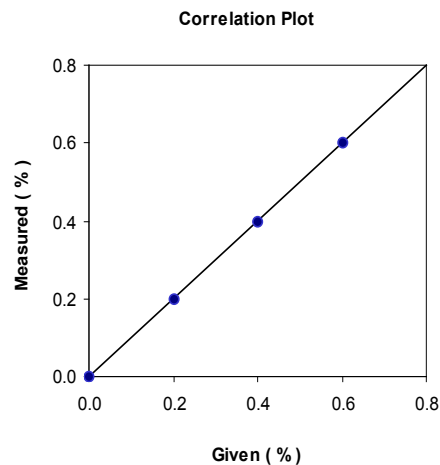
### Limit of Detection (LOD)

The LOD (Limit of Detection) is defined as three times the standard deviation of analyzing the blank sample ten times in a static position. Based on analysis of the blank sample, Teflon, the LOD for this matrix and concentration range is:

Element	LOD
Ti	0.006 %TiO <sub>2</sub>

### Calibration Results for ZnO in Lotion (Low Concentration)

Element: Zn		
Units: %ZnO      Std. Error of Estimate: 0.002		
Sample	Given	Measured
1	0.600	0.601
2	0.400	0.398
3	0.200	0.201
4	0.000	0.000



#### Precision

10 repeat analyses at 100 seconds per analysis

Element: Zn		Units: %ZnO		
Sample	Given	Mean	Std. Dev.	% Relative
1	0.60	0.608	0.003	0.2
2	0.40	0.400	0.001	0.3
3	0.20	0.202	0.0004	0.2

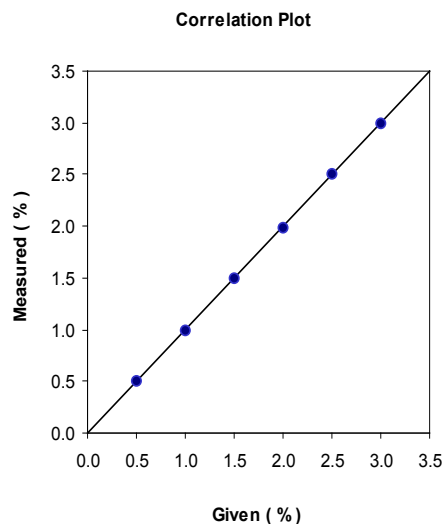
#### Limit of Detection (LOD)

The LOD (Limit of Detection) is defined as three times the standard deviation of analyzing the blank sample ten times in a static position. Based on analysis of the blank lotion sample, the LOD for this matrix and concentration range is:

Element	LOD
Zn	3 ppm ZnO

### Calibration Results for ZnO in Lotion (High Concentration)

Element: Zn		
Units: %ZnO      Std. Error of Estimate: 0.005		
Sample	Given	Measured
1	0.50	0.499
2	1.00	1.001
3	1.50	1.502
4	2.00	1.993
5	2.50	2.509
6	3.00	2.997



#### Precision

10 repeat analyses at 100 seconds per analysis

Element: Zn		Units: %ZnO		
Sample	Given	Mean	Std. Dev.	% Relative
2	1.0	1.019	0.004	0.4
4	2.0	2.061	0.006	0.3

#### Limit of Detection (LOD)

The LOD (Limit of Detection) is defined as three times the standard deviation of analyzing the blank sample ten times in a static position. Based on analysis of the blank lotion sample, the LOD for this matrix and concentration range is:

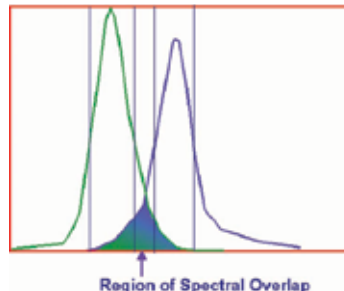
Element	LOD
Zn	6 ppm

As can be seen from the calibration and precision analyses, the results using the ASOMA PHOENIX II show excellent performance for this application.

All the calibrations required a simple background correction; however, the makeup powder and base samples calibrations also required spectral overlap corrections and alpha corrections to correct for absorption/enhancement effects of titanium and iron within the matrix.

**Spectral Overlap:**

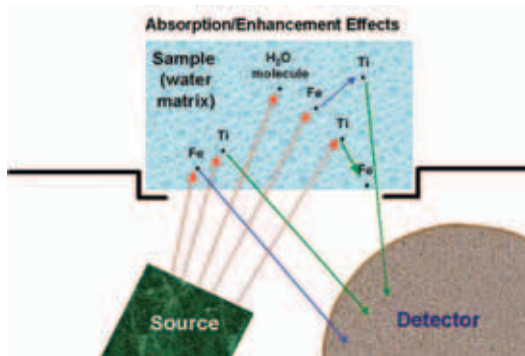
To compensate for peak overlaps, the PHOENIX II uses mathematical cross-corrections based on analyzing factory-supplied Single Element Samples. The procedure is simple and the first part of the calibration process. Spectral overlap corrections were used for titanium and iron when they were present in the same matrix.



**Alpha Corrections:**

If the sample contains a combination of many elements (like tinted make-ups), the x-rays of each element will interact. All elements within a matrix can absorb the x-rays of other elements within a matrix. However, the x-rays of elements with higher atomic numbers may also enhance the elements with lower atomic numbers, resulting in a decreased count rate for the element with the higher atomic number and an increased count rate for the element with the lower atomic number. These interactions between the elements are most obvious if the concentrations of the matrix elements vary over a great range and are collectively referred to as matrix effects.

The PHOENIX II compensates for these matrix effects by using mathematical "alpha" corrections. During calibration simply make sure the concentrations of sulfur, calcium, and iron vary independently of each other. Then when the alpha corrections are enabled in the software, the proper correction factors are determined and used automatically during day-to-day analyses.



**Conclusion**

The results shown here indicate that excellent calibration and precision is achieved using a ASOMA PHOENIX II XRF analyzer for the analysis of titanium, iron and zinc oxides in makeups and lotions. The results of this study indicate that the ASOMA PHOENIX II Analyzer will provide superior performance for these applications.



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