ASOMA® Application Note

ASOMA® PHOENIX II

Determination of Br, Sb & Zn in Polystyrene Using Direct Excitation

Objective

Demonstrate the capabilities of the PHOENIX II XRF analyzer using direct excitation optics for the analysis of antimony and bromine in polystyrene pellets and zinc in polystyrene pellets. Show a demonstration calibration and demonstrate instrument precision.

Background

The PHOENIX II analyzer is readily used in the quality control of the manufacture of polystyrene. The use of this instrument will deliver improvements in product quality by accurately and rapidly determining antimony, bromine and zinc concentrations, important additive elements.

Various additives in plastic affect the physical and chemical properties of the plastic. Zinc compounds are added as pigments and as a mold releasing agent in plastics; i.e., zinc on raw plastic pellets or pressed plaques. While the antimony and bromine reagents are added to give the product flame retardant capabilities.

By ensuring that the content of these elements in the manufacturing process are controlled at all times, quality and cost effectiveness are maximized; therefore, significant reductions in operational costs can be achieved.

Sample Preparation

Each sample was simply poured into a prepared sample cup; tapped 10 times to compact the pellets; and placed on the aperture for analysis.

Analysis Configuration

ASOMA® PHOENIX II

Excitation: Direct excitation optics

Source: X-Ray tube

Detection: Proportional Counter

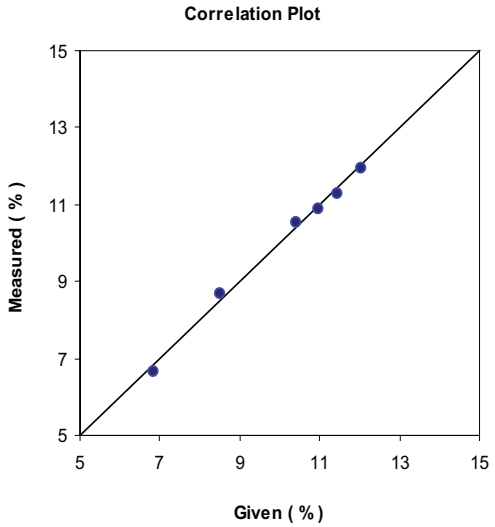
Analysis Time: 100 seconds

Options: Sample spinner, tube filters

Note: No consumable gases required.

Calibration Results for Bromine

Element: Br Std. Error of Estimate: 0.18 Units:% RMS: 0.06		
Sample	**Given**	**Measured**
1	6.82	6.659
2	8.48	8.715
3	10.38	10.551
4	10.94	10.883
5	11.44	11.304
6	12.01	11.958



Precision

10 repeat analyses at 100 seconds per analysis

Element: Br				Units: %
Sample	**Given**	**Mean**	**Std. Dev.**	**% Rel.**
1	6.82	6.521	0.039	0.60
4	10.94	10.805	0.071	0.66
6	12.01	11.838	0.079	0.67

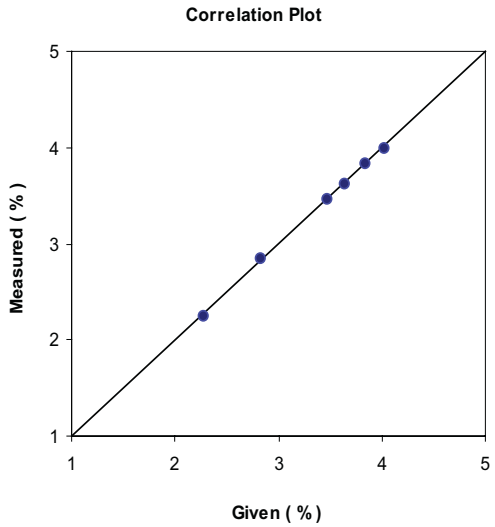
Minimum Detection Limit (MDL)

The Minimum Detection Limit (MDL) 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, the MDL for this matrix can be estimated to be:

Element	MDL
Br	0.015 %

Calibration for Antimony

Element: Sb Std. Error of Estimate: 0.018 Units:% RMS: 0.06		
Sample	**Given**	**Measured**
1	2.27	2.250
2	2.82	2.843
3	3.46	3.475
4	3.63	3.632
5	3.84	3.836
6	4.02	4.004



Precision

10 repeat analyses at 100 seconds per analysis

Element: Sb				Units: %
Sample	**Given**	**Mean**	**Std. Dev.**	**% Rel.**
1	2.27	2.217	0.014	0.63
4	3.63	3.618	0.015	0.41
6	4.02	3.990	0.027	0.68

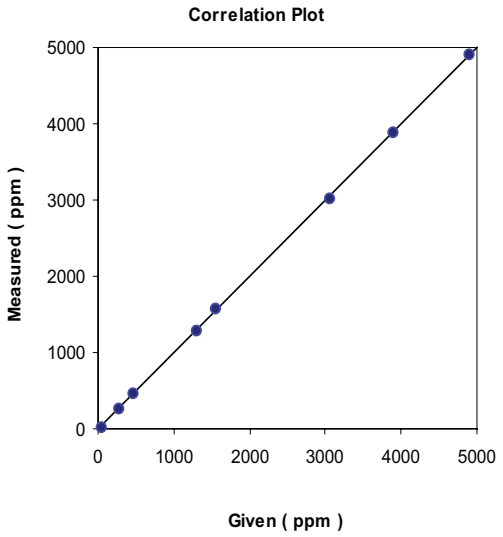
Minimum Detection Limit (MDL)

The Minimum Detection Limit (MDL) 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, the MDL for this matrix can be estimated to be:

Element	MDL
Sb	0.004 %

Calibration Results for Zinc

Element: Zn Std. Error of Estimate: 19.5 Units: ppm RMS: 6.0		
Sample	**Given**	**Measured**
1	4894	4913.9
2	3897	3884.8
3	3046	3019.7
4	1557	1567.4
5	1296	1296.9
6	453	477.6
7	263	264.1
8	44	25.7



Precision

10 repeat analyses at 100 seconds per analysis

Element: Zn				Units: %
Sample	**Given**	**Mean**	**Std. Dev.**	**% Rel.**
1	4894	4889.2	8.79	0.18
4	1557	1572.44	8.94	0.57
7	263	285.81	6.26	2.19

Minimum Detection Limit (MDL)

The Minimum Detection Limit (MDL) 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, the MDL for this matrix can be estimated to be:

Element	MDL
Zn	18 ppm

Discussion

Sample preparation was fast and simple. Each sample was simply poured in a sample cup and tapped 10 times on the tabletop to ensure the sample was evenly distributed throughout the sample cup.

Due to the nature of analyzing pellets, a sample spinner was used during analysis. The spinner rotates the sample during analysis, thus reducing the effects of X-ray scattering by the surface irregularities.

For this study two sets of standards were used. The antimony and bromine in polystyrene were present together in the same standards. The zinc standards contained only the zinc.

Conclusion

As can be seen from the calibration data, the accuracy of the measurement is very good. The statistical result table shows that the one-sigma precision values for Sb and Br in polystyrene and Zn in polystyrene are excellent.

The results of this study indicate that there is no difficulty applying XRF to these applications and that the PHOENIX II Analyzer with Direct Excitation Optics will provide superior performance for these polystyrene applications.

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