Lab Report XRF 126

S2 PUMA

- Fast and Reliable Elemental Analysis of Barite ($BaSO_4$) as Pressed Pellets by EDXRF

The valuable benefits of fast and reliable elemental analysis

The barite mineral group is an important industrial commodity consisting mainly of barium sulfate. With its high barium concentration it is the main source for this element and has a high relevance for many Ba-containing products. A significant amount of barite is used as weighting agent for drilling fluids in exploration but due to its white color, chemical inertness, and high density there are numerous other application areas. Applications include the use as filler in paints and plastics, as compound in glasses to achieve a higher refractory index, as anti-wetting agent in the aluminum industry or as coating in automobile finishes. Barite with lower concentrations of impurities achieves higher prices on the market and therefore the importance to monitor and control associated elements is increasing. One of these elements is Fe, as lower concentrations lead to whiter and brighter end products.

Fast and reliable quality control with the S2 PUMA

The S2 PUMA, a benchtop energy-dispersive X-ray fluorescence (EDXRF) instrument is able to conduct high-performance elemental monitoring and quality control. Its HighSense™ technology with an optimized beam path geometry leads to highest count rates and ensures shortest measurement times and best precision. The S2 PUMA LE used for these measurements was equipped with a 50 W Ag X-ray tube and XFlash® LE silicon drift detector (SDD). With a single measurement, just within a few minutes after taking the sample, analytical results are available for $BaSO_4$ as well as for minor and trace compounds such as Al_2O_3, SiO_2, Fe_2O_3, and $SrSO_4$.

Simple and straight forward sample preparation as pressed pellet

For this lab report 10 g of barite sample have been grinded and mixed with 1 g binder and prepared as pressed pellet. Due to the simple and straight forward sample preparation the analytical results are available within minutes after taking the sample. Two measurement regions were defined. The tube current was optimized and fixed in order to gain maximum count rate for the various elements. Table 1 shows the detailed measurement parameters.

Elements	Tube voltage [kV]	Tube current [µA]	Filter	Measurement time [s]
Al, Si	20	Optimized for countrate of 100 000 cps	None	100
Fe, Sr, Ba	40	Optimized for countrate of 100 000 cps	500 µm Al	100

Table 1: Measurement parameters for the different elements

The samples have been measured under vacuum which avoids the use of expensive helium as purge gas. This measurement condition minimizes dramatically the costs per sample and result in lowest running costs of the instrumentation.

Calibration

Ten standards have been used to set up a calibration covering a concentration range from 87 to 98 % $BaSO_4$, the calibration curves for $BaSO_4$ and SiO_2 are shown in Figure 1 and 2, respectively. The concentration ranges for all analyzed elements are shown in Table 2.

	Minimum concentration [%]	Maximum Concentration [%]
Al_2O_3	0.02	0.23
SiO_2	0.4	11.2
Fe_2O_3	0.02	0.11
$SrSO_4$	1.4	2.8
$BaSO_4$	87	98

Table 2: Concentration ranges used for the barite calibration

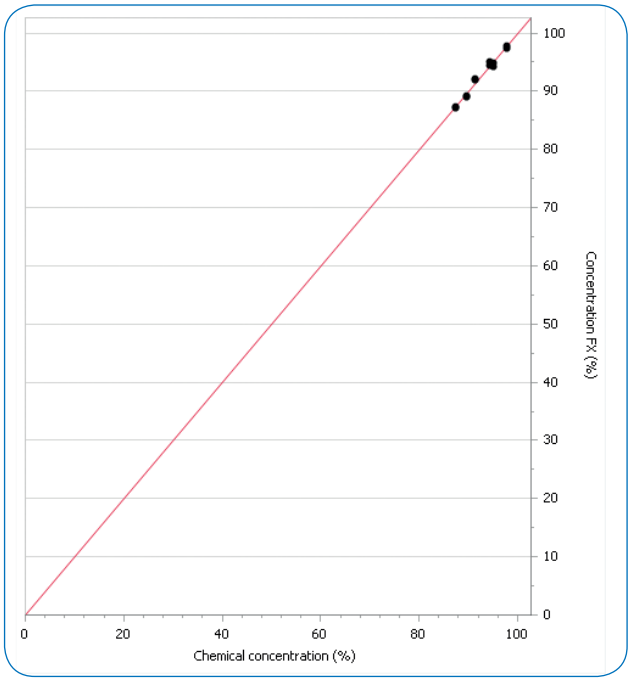

Figure 1: Calibration curve for $BaSO_4$

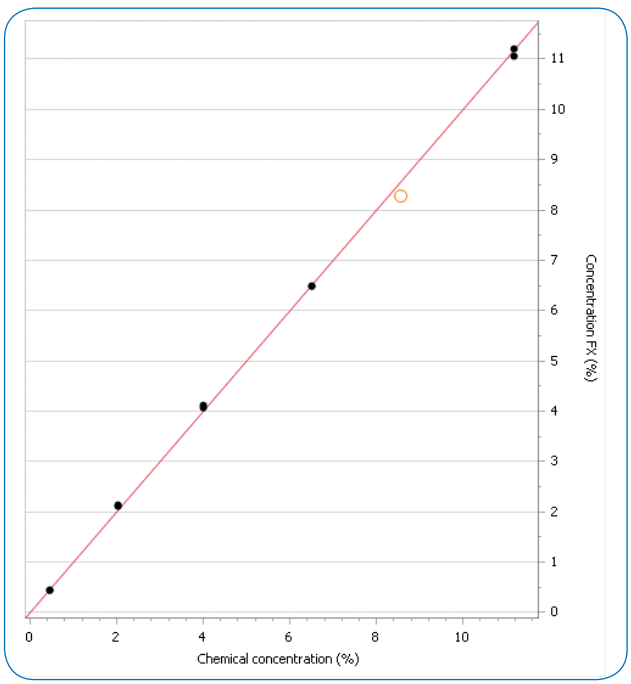

Figure 2: Calibration curve for SiO_2

Excellent precision and accuracy due to HighSense™ Technology

Typical precision and accuracy data of 10 repetitive measurements of a barite sample are shown in Table 3.

Measurement	Al_2O_3 [%]	SiO_2 [%]	Fe_2O_3 [%]	$SrSO_4$ [%]	$BaSO_4$ [%]
Rep. 01	0.02	0.47	0.02	1.65	98.5
Rep. 02	0.02	0.47	0.02	1.65	97.3
Rep. 03	0.02	0.46	0.02	1.64	98.1
Rep. 04	0.02	0.46	0.02	1.64	97.6
Rep. 05	0.03	0.47	0.02	1.64	97.9
Rep. 06	0.02	0.47	0.02	1.63	97.9
Rep. 07	0.03	0.45	0.02	1.64	98.1
Rep. 08	0.02	0.47	0.02	1.65	97.7
Rep. 09	0.01	0.47	0.02	1.65	98.3
Rep. 10	0.02	0.45	0.02	1.63	97.9
Mean value	**0.02**	**0.46**	**0.02**	**1.64**	**97.9**
Abs. standard deviation	**0.01**	**0.01**	**0.00**	**0.01**	**0.34**
Rel. standard deviation	**27.0**	**1.82**	**0.00**	**0.48**	**0.35**
Reference value	**0.02**	**0.45**	**0.02**	**1.64**	**97.8**

Table 3: Precision and accuracy test of ten repetitive measurements of a barite sample

Further precision and accuracy data for SiO_2, an impurity compound, are shown in Table 4. The detailed data for different SiO_2 concentration levels document the excellent precision as well as the good agreement between the measured concentrations in comparison to the certified values.

Measurement	SiO_2 [%]	SiO_2 [%]	SiO_2 [%]	SiO_2 [%]
	Sample A	Sample B	Sample C	Sample D
Rep. 01	0.47	2.14	4.09	6.46
Rep. 02	0.47	2.18	4.03	6.42
Rep. 03	0.46	2.15	4.13	6.44
Rep. 04	0.46	2.16	4.09	6.45
Rep. 05	0.47	2.17	4.05	6.47
Rep. 06	0.47	2.15	4.13	6.45
Rep. 07	0.45	2.19	4.12	6.42
Rep. 08	0.47	2.19	4.09	6.41
Rep. 09	0.47	2.17	4.05	6.52
Rep. 10	0.45	2.13	4.12	6.49
Mean value	**0.46**	**2.16**	**4.09**	**6.45**
Abs. standard deviation	**0.01**	**0.02**	**0.04**	**0.03**
Rel. standard deviation	**1.82**	**0.95**	**0.89**	**0.53**
Reference value	**0.45**	**2.03**	**4.00**	**6.51**

Table 4: Precision and accuracy data for SiO_2 for four different barite samples

Summary

The results achieved with the S2 PUMA demonstrate the excellent performance of the instrument. The HighSense™ technology and our powerful 50 W X-ray tube in combination with the XFlash® detector lead to an excellent precision with very low standard deviations. The high accuracy with only small variations to certified values reveals the excellent suitability of the S2 PUMA for quality control.

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