Lab Report XRF 179

S2 PUMA Series 2 with HighSense™XP

- Analysis of Geological Materials with GEO-QUANT Basic

Natural resources in the form of rocks and other geo-materials are one of the most widely used commodities. The building materials industry is a major consumer of raw materials, processing resources such as limestone, gypsum, and clay. These materials get manufactured into products like cement and concrete, screed, bricks, plaster, and dry wall panels (see Figure 1).

The refractories and raw materials suppliers are other relevant industries with a high demand for geological resources. Magnesite is used as source material for linings in blast furnaces and kilns. And as suppliers to the chemical industry, high-purity limestone producers have to apply strict product quality control to meet the demands of their customers. Thus, the analysis of raw materials is for many industries one of the most important analytical tasks. Controlling the grade and quality of the incoming materials before they enter a process gives much higher confidence that the final product will be of the required specification and helps to prolong the life of the equipment used during the process.

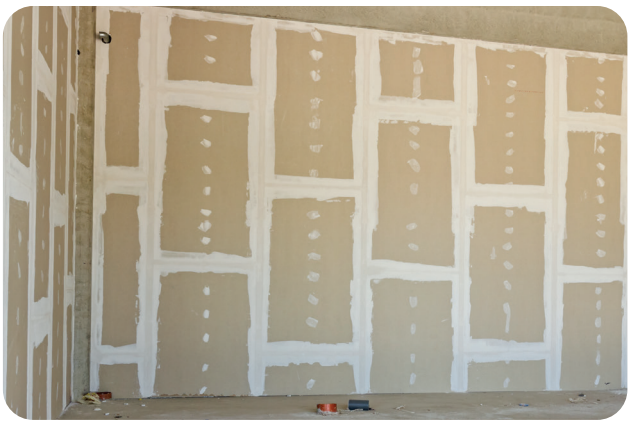

Figure 1: Dry wall panels made of gypsum

Fast and Reliable When It Counts

X-ray fluorescence (XRF) spectroscopy is an excellent method for the analysis of geological raw materials. The technique is highly sensitive to small changes in concentrations. The S2 PUMA is the high-performing benchtop energy-dispersive X-ray fluorescence (EDXRF) equipped with state-of-the-art hardware technology and running on the latest version of Bruker's powerful and intuitive spectrometer platform, SPECTRA.ELEMENTS.
The 22-position XY Autochanger provides highest flexibility and ensures unmatched throughput in the class of benchtop EDXRF. You can add, remove, and prioritize any samples (solid, liquid, powder, ...) at any time. Bruker's unique SampleCare™ technology protects the vital system components, ensuring high uptime and easy maintenance.

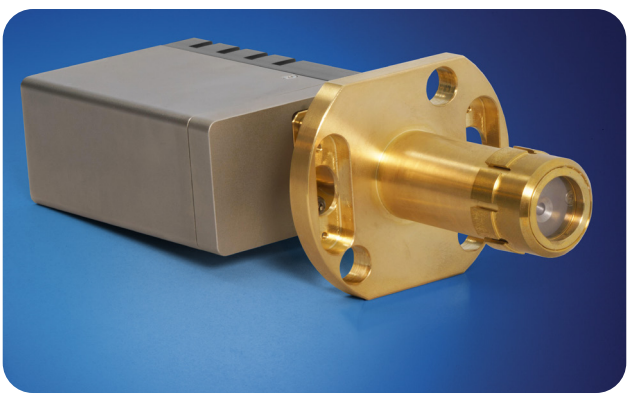

Figure 2: HighSense XP - The new premium detector of the S2 PUMA Series 2

For this lab report we used the S2 PUMA Series 2 equipped our powerful 50 W X-ray tube and the HighSense XP silicon drift detector (SDD). The HighSense XP offers best-in-class sensitivity for all elements from Carbon (C) to Americium (Am) and is equipped with a robust, high transmission graphene window (Figure 2).

The S2 PUMA is the only EDXRF benchtop instrument,, which is ris ready for integrationn into fully automated laboratory environments. This powerful feature enables autonomous process control 24/7.

GEO-QUANT Basic – The Wide-Range Calibration with Carefully Selected Certified Reference Materials

For accurate XRF analysis, standards are required to calibrate the instrument. Typically, this is done on a material-by-material basis to eliminate matrix effects between sample types, which can lead to inaccurate final results. These matrix effects can be largely overcome by the use of lithium borate fusion as a sample preparation technique. The sample is melted and digested into a lithium borate flux and then cast into a glass disk, known as a fusion bead (Figure 3).

Figure 3: Fused bead samples for XRF analysis

GEO-QUANT Basic takes advantage of the fact that with this sample preparation technique, it is not necessary to perfectly match standards to material matrices one wishes to analyze. The standards in this calibration set have been carefully selected to give a wide concentration range for a suite of elements commonly found in the raw materials required by typical industrial processes such as cement and steel production (Table 1).

GEO-QUANT Basic is the dedicated, out-of-the-box solution for the analysis of geo-materials. It is an accurate method for the measurement of 14 major elements (in oxide form) in geological samples. The oxides Na_2O, MgO, Al_2O_3, SiO_2, P_2O_5, SO_3, K_2O, CaO, TiO_2, Cr_2O_3, Mn_2O_3, Fe_2O_3, ZnO, and SrO are measured.

For the optimal accuracy, 20 certified reference materials (CRM) including cement, clay, feldspar, limestone, dolomite, sedimentary rock, magnesite, refractory, bauxite, gypsum, and other different rock types are used to establish the calibration. The quality of the calibration is evaluated by by using four additional CRMs.

Figure 4: Drying oven lined with refractory bricks

Sample Preparation and Measurement Conditions

All samples were prepared as fusion beads with 1.0 g of ignited sample and 9.0 g of flux (66% Lithium Tetraborate, 34% Lithium Metaborate). If necessary, LiBr (0.2 wt%) can be used as a non-wetting agent, to aid in casting the beads.

The measurement method parameters are described in Table 2. The automatic current mode of SPECTRA.ELEMENTS is used to ensure optimal performance. The total measurement time is 6 minutes.

The four dedicated quality control (QC) samples were measured 20 times each, to check the validity and stability of the method. The results, showing the agreement with the certificates and the stability, are summarized in Table 3.

Compound	Number of Standards	Max. Conc. [wt%]
Na_2O	16	11
MgO	18	40
Al_2O_3	19	85
SiO_2	19	100
P_2O_5	16	7.5
SO_3	13	58
K_2O	18	12
CaO	18	100
TiO_2	19	4
Cr_2O_3	14	0.1
MnO	16	0.9
Fe_2O_3	20	100
ZnO	11	0.2
SrO	13	0.3

Table 1: Maximum concentrations of the 14 elements covered by GEO-QUANT.

Element	Voltage [kV]	Filter	Measurement Time [s]	Atmosphere
Na, Mg, Si, Al, P, S	20	None	180	Vacuum
K, Ca	30	Al (20 µm)	120	Vacuum
Ti, Cr, Mn, Fe, Zn, Sr	40	Al (500 µm)	60	Vacuum

Table 2: Measurement conditions for GEO-QUANT Basic on the S2 PUMA Series 2

	Cement			Iron Ore			Limestone			Gypsum		
	Mean	Cert.	SD	Mean	Cert.	SD	Mean	Cert.	SD	Mean	Cert.	SD
Na_2O	0.51	0.68	0.03	0.13	0.06	0.01				0.13	0.03	0.02
MgO	1.58	1.70	0.03	1.95	1.72	0.02	0.94	0.32	0.02	6.90	7.22	0.04
Al_2O_3	8.70	8.81	0.02	2.44	2.46	0.02	0.39	0.19	0.01	0.90	1.07	0.02
SiO_2	29.62	30.31	0.03	7.07	7.23	0.01	0.62	0.40	<0.01	4.74	4.72	0.01
P_2O_5	0.00	0.19	0.02	0.22	0.34	<0.01				0.00	0.02	<0.01
SO_3	3.18	3.16	0.01				0.66	0.04	<0.01	42.04	44.53	0.03
K_2O	2.15	2.10	0.01	0.25	0.18	<0.01	0.15	0.03	0.00	0.47	0.49	<0.01
CaO	48.24	48.24	0.06	7.71	7.98	0.01	95	98.72	0.03	41.53	41.03	0.05
TiO_2	0.45	0.47	0.01	0.04	0.16	<0.01						
Cr_2O_3	0.01	0.01	<0.01	0.03	0.03	<0.01	0.005	0.002	0.002			
Mn_2O_3	0.11	0.12	<0.01	0.65	0.66	<0.01	0.02	0.02	0.00			
Fe_2O_3	3.49	3.52	0.01	78	80.15	0.06	0.09	0.05	0.00	0.59	0.54	<0.01
ZnO	0.04	0.04	<0.01	0.01	0.01	<0.01	0.003	0.003	<0.001			
SrO	0.24	0.25	<0.01				0.02	0.03	<0.01	0.51	0.47	<0.01

Table 3: Accuracy and repeatability (20 measurements each, samples were unloaded and reloaded each time)

Conclusion

The analysis of geological raw materials is of vital importance to many industrial processes both from a quality control and process uptime point of view. The S2 PUMA Series 2 with HighSense XP and GEO-QUANT Basic is extremely well suited to fulfill the needs to these challenging analytical tasks, providing accurate and precise results for both quality control and the analysis of completely unknown geo-materials.

The use of a highly robust calibration such as GEO-QUANT Basic means that individual matrix matched standards are not required, as all material types can be analyzed using the same calibration. GEO-QUANT Basic is the quickest and most convenient way to have a fully operational calibration for geo-materials set up in no time: Get the case with the samples, prepare them, and have them run on your system. Done!
Together with industry-leading features such as the XY Autochanger, SampleCare™, and TouchControl™ the S2 PUMA is ready for quick and reliable product quality control from day one!

Figure 5: The S2 PUMA Series 2 with XY Autochanger for high throughput and ultimate flexibility.

S2 PUMA XY Autochanger

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