Lab Report XRF 133

S2 PUMA

- Analysis of Geological Materials using GEO-QUANT Basic with the S2 PUMA

Introduction

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 consumers of raw materials with resources processed 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. Therefore, the analysis of raw materials is for many industries one of the most important analytical tasks that they perform. 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 at building site

Fast and Reliable When It Counts: S2 PUMA

X-ray fluorescence (XRF) Spectroscopy is an excellent method for the analysis of raw materials for these purposes. The technique is highly sensitive to small changes in concentrations. The S2 PUMA is the high-performing benchtop energy-dispersive X-ray fluorescence (EDXRF) spectrometer available as Single, as Carousel, and as XY Autochanger for high sample throughput (see Figure 2).

The 20-position EasyLoad™ is accessible during analysis of samples which allows loading and changing of samples at any time.

For this lab report we used the S2 PUMA XY Autochanger

Figure 2: The S2 PUMA XY Autochanger guarantees high sample throughput

with an X-ray tube with Ag target and the XFlash® LE silicon drift detector (SDD). The LE configuration is particularly well suited for the analysis of light elements such as Na, which is an important element in many geo-materials.

No moving parts, apart from a computer controlled filter wheel to select optimum measurement conditions and sample rotation (optional), are built in. This means that no mechanical failures within the beam path can occur and this stands for high instrument uptime and low running cost.

The ergonomic TouchControl™ interface for independent routine operation without any PC peripherals and the powerful instrument software suite SPECTRA.ELEMENTS guarantee the highest analytical flexibility.

The S2 PUMA XY Autochanger is the only EDXRF benchtop instrument which can be integrated into fully automated laboratory environments. This is a very powerful feature for 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.

Figure 3: Fused bead samples for XRF analysis

The sample is melted and digested into a lithium borate flux and then cast into a glass disk, known as a fusion bead (see Figure 3).

GEO-QUANT Basic takes advantage of the fact that with this sample preparation technique, it is no longer 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.

GEO-QUANT Basic is the XRF solution for the analysis of geo-materials. It is an accurate method for 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 in wide concentration ranges (see Table 1).

Figure 4: Industrial refractory bricks

For the best 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 wide range calibration. The quality of the calibration is evaluated using four additional certified reference materials.

Measurement Conditions and Set-Up

The measurement method parameters are described below in Table 2. The four dedicated quality control samples were measured, each 20 times, by GEO-QUANT Basic to check the validity and stability of the method. The results, showing the agreement with the certificates and the stability, are summarized below (see Table 3).

For this method, the automatic current optimization mode of SPECTRA.ELEMENTS is used to ensure the optimum performance of the detector. All samples were prepared as fusion beads with 1.0 g of ignited sample and 8.0 g of flux (66% Lithium Tetraborate, 34% Lithium Metaborate).

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	0.1
SrO	13	0.1

Table 1: Maximum element concentrations and number of GEO-QUANT Basic standards for each element

Element	Voltage [kV]	Filter	Measurement Time [s]	Atmosphere
Na, Mg, P, S	20	None	60	Vacuum
Al, Si	20	Al (100 µm)	50	Vacuum
K, Ca, Ti, Cr, Mn, Fe, Zn, Sr	40	Ti (100 µm)	60	Vacuum

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

	Cement (CRM 101)			Gypsum (GYP-C)			Limestone (BCS-CRM 513)			Iron Ore (ECRM 683-1)		
	Mean	Cert.	SD	Mean	Cert.	SD	Mean	Cert.	SD	Mean	Cert.	SD
Na_2O (wt.-%)	0.55	0.68	0.04	0.04	0.03	0.04	0.21		0.04	0.13	0.06	0.04
MgO (wt.-%)	1.65	1.70	0.05	6.83	7.22	0.09	0.76	0.32	0.03	1.62	1.70	0.05
Al_2O_3 (wt.-%)	8.70	8.81	0.04	0.88	1.07	0.07	0.30	0.19	0.02	2.03	2.44	0.04
SiO_2 (wt.-%)	30.22	30.31	0.05	4.42	4.72	0.07	1.17	0.41	0.03	7.50	7.16	0.05
P_2O_5 (wt.-%)	0.18	0.19	0.01	0.24	0.02	0.04	-0.05		0.01	0.17	0.34	0.01
SO_3 (wt.-%)	3.17	3.16	0.01	44.75	44.53	0.06	0.28	0.04	0.01	0.04		0.01
K_2O (wt.-%)	1.91	2.10	0.06	0.41	0.49	0.04	0.15	0.03	0.04	0.21	0.18	0.04
CaO (wt.-%)	48.19	48.24	0.16	41.07	41.02	0.17	99.66	98.94	0.15	7.91	7.89	0.04
TiO_2 (wt.-%)	0.43	0.47	0.02	0.06	0.05	0.02	0.03		0.01	0.10	0.16	0.02
Cr_2O_3 (wt.-%)	0.01	0.01	0.00	0.01		0.00	0.01		0.01	0.06	0.03	0.01
Mn_2O_3 (wt.-%)	0.11	0.12	0.00	0.02	0.01	0.01	0.02	0.02	0.01	0.65	0.66	0.01
Fe_2O_3 (wt.-%)	3.47	3.52	0.01	0.57	0.54	0.00	0.10	0.05	0.00	78.86	79.40	0.07
ZnO (wt.-%)	0.05	0.04	0.01	0.00		0.00	0.00	0.00	0.00	0.01	0.01	0.00
SrO (wt.-%)	0.24	0.25	0.00	0.48	0.47	0.00	0.03	0.03	0.00	0.01	0.00	0.00

Table 3: Results of repeatability test with GEO-QUANT Basic

Summary

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 with 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 materials in small quantities.

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. No calibration work on your side needed.
Together with industry-leading features such as the XY Autochanger sample handler and TouchControl make product quality control with the S2 PUMA quick and reliable.

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