



Lab Report XRF 103

S2 RANGER with XFlash LE and GEO-QUANT M Analysis of Gypsum and Carbonate Rocks

Introduction

The two major types of carbonate rocks are limestone and dolomite. Limestone is widely used as building material and raw material in cement and glass industry. The main use of dolomite is as ornamental stone, concrete aggregate and for magnesium production. Gypsum is also a sedimentary rock and is mostly used as building material and fertilizer. X-Ray fluorescence analysis (XRF) is the best suited analytical technology for grade control of industrial minerals. It is a practical and fast method due to the simple sample preparation method.

Energy dispersive X-ray fluorescence spectrometry (EDXRF) is very common for the control of mining operations and determining the final product grade. Benefits are its unique ease of use and the advanced analytical performance of modern instruments. EDXRF instruments are typically very compact in benchtop size and offering low cost of ownership which makes them attractive for dedicated analytical tasks. Up to now the performance for light elements such as sodium and magnesium was very limited. A thick detector window in conventional EDXRF spectrometers absorbed nearly all radiation of light elements. Modern detector technology combined with high power direct excitation and a thin stabilized entrance

window making the EDXRF spectrometer S2 RANGER with the XFlash LE detector far superior compared to other instruments. This lab report demonstrates the analytical performance of the S2 RANGER with XFlash LE for the analysis of carbonate rocks and gypsum with the GEO-QUANT M solution.



Picture. 1: S2 RANGER EDXRF spectrometer

Elemental analysis with EDXRF has become a very important tool in research and for process or quality control. The analytical flexibility, the low operating costs, the small size and easy operation with TouchControl make the S2 RANGER with XFlash LE an attractive tool for a wide range of applications in industry. It excites the elements in the sample directly with 50 W. Since the XFlash silicon drift detector is able to operate at very high countrates the achievable standard deviation is excellent. With a very thin stabilized entrance window the XFlash LE detector provides best sensitivity even for light elements such as sodium and magnesium. The integrated vacuum pump enables the effective analysis of carbonates without the need of helium purge. This enhanced analytical performance especially for light elements allows using the fusion method for sample preparation, which makes the achievable accuracy and precision much better.

The S2 RANGER with XFlash LE is calibrated with GEO-QUANT M. This is the Bruker solution for the analysis of major elements in geological samples. It enables the quick analysis of the major elements Na, Mg, Al, Si, P, S, K, Ca, Ti, Mn and Fe. GEO-QUANT M comes with 20 certified reference materials to cover a wide concentration range and has in addition 3 validation standards for checking the quality of the calibration. The standards are available as powders and are prepared following the recipes in the sample preparation manual using the installed fusion equipment.

Sample Preparation and Measurement Parameters

The calibration standards were prepared using 0.8 g samples with 8.0 g flux (lithiumborate mix for gypsum and lithium tetraborate for other standards) as fused beads. Eight certified reference materials carbonate rocks (GBW 07127, GBW 07128, GBW 07130, GBW 07134), limestone (IPT 44, GBW 7120), and gypsum standards (GYP-C, GYP-D) have been prepared in the same way. They are used to evaluate the quality of the GEO-QUANT M calibration. The measurements were done on the S2 RANGER with a total measurement time of 10 minutes. GEO-QUANT M provides optimized measurement parameters. Results of the accuracy test of the validation samples have been compared with the certified concentrations in table 1.

Results

To demonstrate the long time stability of the S2 RANGER in combination with GEO-QUANT M the certified reference material GBW07127 has been measured 44 times in a period of one week. The repeatability and reproducibility of the method and the long time stability of the S2 RANGER are summarized in table 2.

Figure 1 shows the stability results of MgO and CaO measurement in GBW07127 in a period of one week. Figure 2 shows the correlation of measured versus certified concentrations for Al_2O_3 and CaO.

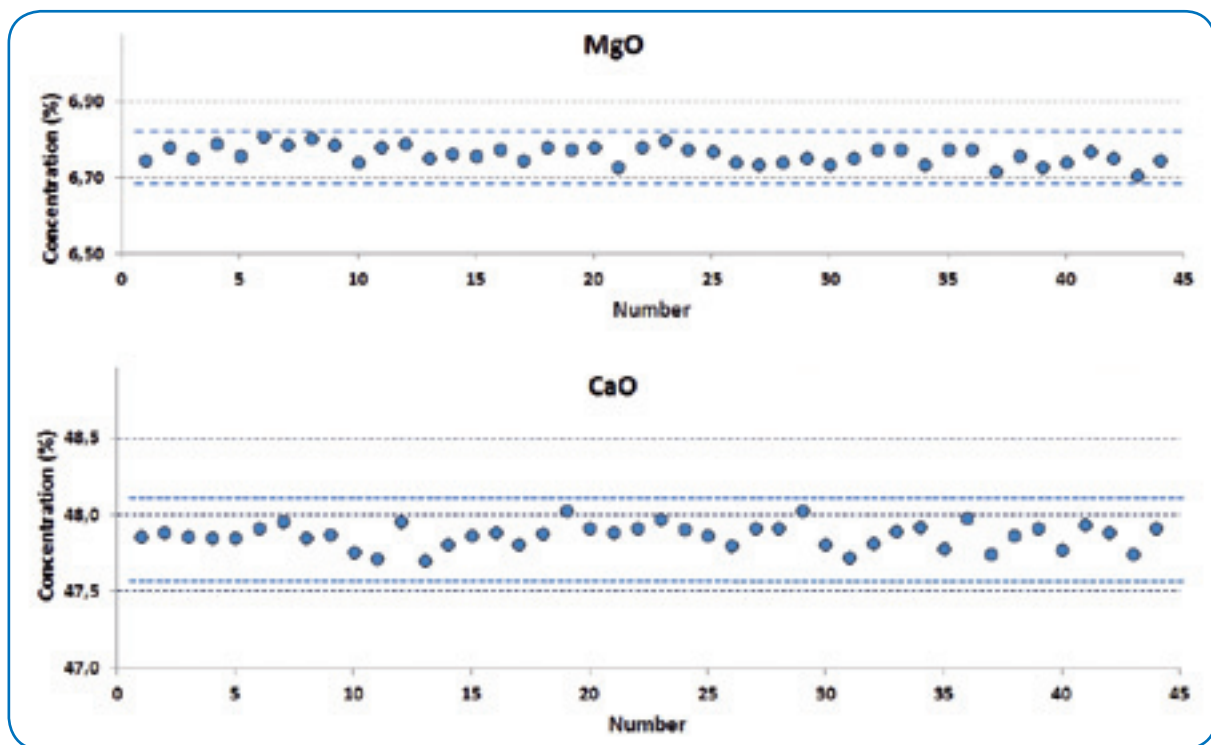


Fig. 1: The stability test of MgO and CaO in GBW07127 dashed lines show the three times of standard deviation of the measurement

Table 1: Accuracy test of GEO-QUANT M with certified materials (carbonate rocks and gypsum) standards

	Carbonate rock							
	GBW 07127		GBW 07128		GBW 07134		GBW 07130	
	Cert.	Meas.	Cert.	Meas.	Cert.	Meas.	Cert.	Meas.
Na ₂ O	0,022	0.07	0.029	0.04	0.03	0.02	0.015	0.08
MgO	6.76	6.73	11.62	11.51	14.96	14.91	1.42	1.42
Al ₂ O ₃	0.17	0.18	0.22	0.24	0.18	0.18	0.18	0.19
SiO ₂	0.55	0.53	0.72	0.71	1.17	1.14	1.08	1.19
P ₂ O ₅	0.008	0.10	0.014	0.08	0.009	0.06	0.005	0.13
SO ₃	0.017	0.08	0.013	0.09	0.041	0.02	0.014	0.09
K ₂ O	0.043	0.03	0.05	0.04	0.03	0.03	0.043	0.05
CaO	47.89	47.83	41.95	41.91	38.08	38.26	54.08	54.14
TiO ₂	0.01	0.01	0.02	0.02	0.01	0.01	0.007	0.00
Mn ₂ O ₃	0.01	0.01	0.01	0.01	0.024	0.03	0.004	0.01
Fe ₂ O ₃	0.19	0.19	0.21	0.20	0.45	0.43	0.222	0.22

	Limestone				Gypsum			
	GBW 07120		IPT 44		GYP-C		GYP-D	
	Cert.	Meas.	Cert.	Meas.	Cert.	Meas.	Cert.	Meas.
Na ₂ O	0.03	0.07	0.002	0.06	0.02	0.00	0.07	0.00
MgO	0.71	0.71	2.93	2.92	5.35	5.11	1.73	1.60
Al ₂ O ₃	0.68	0.66	0.33	0.37	0.79	0.84	2.03	2.04
SiO ₂	6.65	6.60	2.69	2.67	3.50	3.69	8.70	8.93
P ₂ O ₅	0.012	0.13	0.013	0.21	0.02	< LLD	0.03	0.00
SO ₃	0.009	0.03	n.d.	0.20	32.97	32.82	36.72	36.53
K ₂ O	0.15	0.14	n.d.	0.10	0.36	0.32	0.54	0.51
CaO	51.1	51.20	50.5	50.66	30.38	30.51	28.21	28.61
TiO ₂	0.038	0.03	0.019	0.01	Trace	0.04	Trace	0.08
Mn ₂ O ₃	0.004	0.01	0.015	0.02	Trace	0.01	Trace	0.02
Fe ₂ O ₃	0.21	0.20	0.3	0.29	0.40	0.40	1.08	1.06

Table 2: Analytical precision of carbonate rock GBW 07127 Accuracy test of GEO-QUANT M with carbonate rocks and gypsum standards
Precision test 44 times alternated measurement

	Na ₂ O	MgO	Al ₂ O ₃	SiO ₂	P ₂ O ₅	SO ₃	K ₂ O	CaO	TiO ₂	Mn ₂ O ₃	Fe ₂ O ₃
Certified Concentration [%]	0.022	6.76	0.17	0.55	0.008	0.017	0.043	47.89	0.011	0.008	0.193
Average [%]	0.05	6.76	0.07	0.53	0.10	0.09	0.04	47.87	0.01	0.01	0.18
Min. [%]	0.02	6.71	0.06	0.52	0.09	0.08	0.03	47.70	0.00	0.01	0.18
Max. [%]	0.08	6.81	0.07	0.53	0.12	0.10	0.06	48.03	0.02	0.01	0.19
Abs. Std. Dev. [%]	0.02	0.02	0.00	0.00	0.01	0.00	0.01	0.08	0.01	0.00	0.00
Rel. Std. Dev.	36.84	0.34	5.05	0.69	5.30	2.31	15.79	0.16	86.12	22.73	0.80

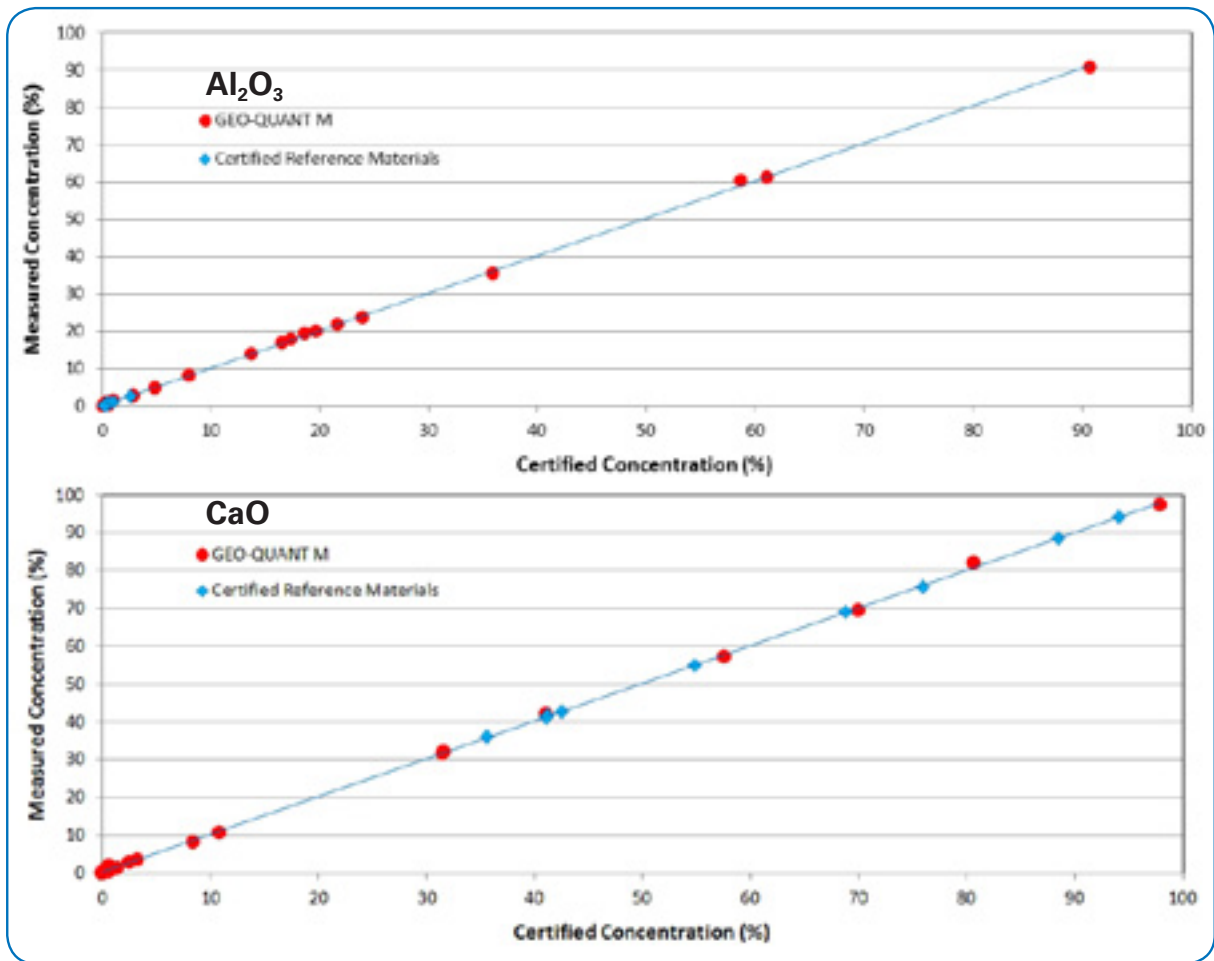


Fig. 2: Correlation of measured versus certified concentrations for Al₂O₃ and CaO for GEO-QUANT M standards (red) and certified reference materials (blue)

Conclusions

The results of the stability test show that the S2 RANGER with XFlash LE delivers in combination with GEO-QUANT M based on fused beads high analytical precision and excellent accuracy. The measurement parameters have been selected to use the optimum performance of the spectrometer. The results of repeated measurements in a period of one week also show that the S2 RANGER LE is very long term stable. The achieved precision of the wide range oxide calibration of GEO-QUANT M complies with the requirement of both industry and research. It is therefore a valuable tool for grade control in mining sites for limestone, dolomite and Gypsum.

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