Lab Report XRF 122

S2 PUMA

- Limestone: Fast, Accurate, and Precise Analysis of CaO, MgO, and Fe_2O_3 and other Main Components

Introduction

Limestone is one of the most important industrial commodities because of its numerous applications ranging from sectors like building materials to steel production to pharmaceuticals to food. Like anywhere within the industrial minerals sector, constant process control is a must to ensure consistent product quality.

Particularly sought-after is high-purity limestone with high contents of CaO and low concentrations of impurities like Fe_2O_3. But also MgO is an important natural constituent of limestone and has to be monitored. This report demonstrates the analytical performance of the S2 PUMA for the measurement of limestone.

S2 PUMA: Production Control Made Easy and Quick

The S2 PUMA is the high-performing benchtop energy-dispersive X-ray fluorescence (EDXRF) spectrometer for a wide range of applications. It is ideally suited for process control of limestone samples due to its XY Autochanger for unattended analysis of large series of process samples. Its HighSense™ beam path geometry with optional light elements (LE) configuration ensures high sample through-put in combination with best precision and accuracy even for light elements like Mg.

The instrument is perfectly equipped for industrial environments due to its ergonomic TouchControl™ interface which allows for independent routine operation in Island Mode without any PC peripherals. The dedicated instrument protection system SampleCare™, the powerful user account control of the spectrometer's software SPECTRA.ELEMENTS, and the sturdy design guarantee a high instrument uptime.

Compound	Concentration Range [wt.-%]	Calibration Standard Deviation 3σ [wt.-%]
MgO	0.39 - 17.9	0.1620
Al_2O_3	0.02 - 2.4	0.0194
SiO_2	0.05 - 8.6	0.1330
P_2O_5	0.002 - 0.12	0.0022
SO_3	0.008 - 0.1	0.0012
K_2O	0.001 - 0.4	0.0043
CaO	32.0 - 55.4	0.2491
TiO_2	0.015 - 0.14	0.0003
MnO	0.007 - 0.19	0.0032
Fe_2O_3	0.04 - 1.97	0.0163

Table 1: Details on calibration data

Calibration Data

For this report eight limestone reference samples have been used to set-up a calibration on the S2 PUMA XY Autochanger with Ag LE X-ray tube. The samples have been prepared as 40 mm pressed pellets with 9 g of milled specimen powder and 1 g of binding wax and have been pressed for 30 s applying 150 kN of pressure. The measurements took 100 s and have been performed under vacuum for optimal light element detection and lower operating costs not requiring helium flushing. Peak and background positions have been defined by fixed position, integration, and peak height or lower envelope, respectively. See Table 1 for the ranges and the standard deviations of the elements of the reference samples used for calibration.

The exemplary calibration curve of Mg (see Figure 1) shows the excellent analytical abilities of the S2 PUMA particularly for light elements with a strong linear association (R^2=0.99953) and a 3σ standard deviation of 0.1620 % for a concentration range of 17.9 %.

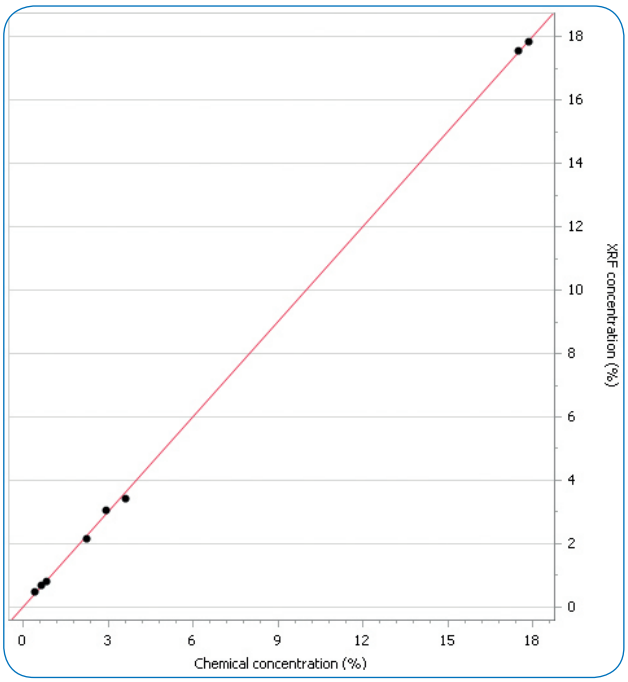

Figure 1: Calibration curve for Mg in SPECTRA.ELEMENTS

Analytical Precision

To demonstrate the impressive analytical stability of the S2 PUMA, the measurement of one sample was repeated 10 times every 17 minutes. Table 2 lists the measurement conditions. After each analysis the sample was unloaded from the sample chamber. The analytical statistics of this repetition test on the S2 PUMA and the high-precision results with low relative standard deviations are given in Table 3.

Voltage [kV]	Current [µA]	Filter	Measurement Time [s]
20	130	none	100

Table 2: Measurement conditions

	MgO [wt.-%]	Al_2O_3 [wt.-%]	SiO_2 [wt.-%]	P_2O_5 [wt.-%]	SO_3 [wt.-%]	K_2O [wt.-%]	CaO [wt.-%]	TiO_2 [wt.-%]	MnO [wt.-%]	Fe_2O_3 [wt.-%]
Rep-1	0.855	2.27	8.60	0.119	0.096	0.442	47.28	0.129	0.074	0.833
Rep-2	0.850	2.32	8.60	0.118	0.094	0.430	47.25	0.121	0.070	0.883
Rep-3	0.893	2.29	8.61	0.116	0.096	0.447	47.23	0.126	0.069	0.860
Rep-4	0.812	2.32	8.61	0.113	0.093	0.433	47.25	0.122	0.072	0.852
Rep-5	0.874	2.39	8.60	0.116	0.093	0.444	47.30	0.120	0.078	0.848
Rep-6	0.866	2.31	8.62	0.120	0.094	0.451	47.32	0.125	0.075	0.844
Rep-7	0.879	2.26	8.62	0.111	0.095	0.452	47.28	0.121	0.078	0.822
Rep-8	0.864	2.29	8.59	0.123	0.095	0.442	47.22	0.127	0.076	0.844
Rep-9	0.810	2.27	8.61	0.110	0.095	0.447	47.34	0.121	0.073	0.865
Rep-10	0.859	2.31	8.63	0.111	0.095	0.449	47.31	0.129	0.075	0.853
Average	**0.856**	**2.30**	**8.61**	**0.116**	**0.094**	**0.444**	**47.28**	**0.124**	**0.074**	**0.850**
Abs. Std. Dev.	**0.027**	**0.04**	**0.01**	**0.004**	**0.001**	**0.007**	**0.04**	**0.004**	**0.003**	**0.017**
Rel. Std. Dev.	**3.14**	**1.66**	**0.14**	**3.79**	**1.23**	**1.61**	**0.09**	**2.97**	**4.12**	**1.97**

Table 3: Precision of the measurement (measurement time 100 s) of the main components in limestone

Measurement Accuracy

An example for the analytical accuracy of the S2 PUMA is given below. The results of the analysis, based on the same calibration as above, and the reference values are listed in Table 4.

The values listed here show the very high agreement of measured and reference concentrations of the limestone sample. Particularly the important elements for limestone such as Ca and Fe get replicated very well and even the light element Mg is very close to its reference value.

	MgO [wt.-%]	Al_2O_3 [wt.-%]	SiO_2 [wt.-%]	P_2O_5 [wt.-%]	SO_3 [wt.-%]	K_2O [wt.-%]	CaO [wt.-%]	TiO_2 [wt.-%]	MnO [wt.-%]	Fe_2O_3 [wt.-%]
Reference	17.50	1.240	4.300	0.048	T	0.430	32.00	0.060	0.042	0.650
Measured	17.52	1.275	4.302	0.046	0.103	0.430	32.13	0.063	0.037	0.635
Abs. Deviation	**0.02**	**0.035**	**0.002**	**0.002**		**0.000**	**0.13**	**0.003**	**0.006**	**0.015**

Table 4: Accuracy test of reference material measured against the calibration (measurement time 100 s)

Summary

Due to its HighSense beam path and based on the LE configuration the S2 PUMA delivers excellent light elements performance achieving optimum results within shortest measurement times. Additional benefits are the low cost of operation since the analysis was performed using the integrated vacuum mode instead of expensive helium flushing. Furthermore the simple operation due to TouchControl makes the S2 PUMA the perfect tool for quality control in industrial environments.

Links

S2 PUMA XY Autochanger

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