

S4 EXPLORER

ANALYSIS OF CEMENT SAMPLES

Introduction

This lab report will describe the procedures and results of a calibration with cement samples and a reproducibility test done with the S4 EXPLORER.

Preparation

A set of eight certified reference materials (NIST) was prepared as fused beads with 1.5 g of sample and 7.5 g of flux.

Calibration

Compounds, concentration ranges and standard deviations of the calibration as well as sensitivities and lower limits of detection are given in table 1.

Reproducibility test

Two samples were measured for 9 compounds from Na_2O to Fe_2O_3 . During the measurement cycles all parameters are changed: sample, power, voltage and current of the X-ray tube, collimator, crystal, and detector.

The measurement time for one sample including loading, evacuation and unloading was five minutes, the whole period was about 12 hours without any drift correction. Within these period each sample was measured 100 times.

Results of reproducibility test

In table 2 the data for both samples are presented.

The absolute standard deviations of 0.04 % at a level of 62% CaO or 0.02% for 21% SiO_2 compound SiO_2 as clearly show that the reproducibility of the S4 matches the performance of the high-power instruments.



Table 1: Calibration data, Sensitivities and Lower Limits of Detection for cement

Compound	Concentration Range %	Calibration Std Dev %	Sensitivity Kcps / %	LLD (100s, 3σ) ppm
Na₂O	0.02 - 0.38	(0.01)	1.5	45
MgO	0.71 - 4.02	0.02	2.4	40
Al₂O₃	3.31 - 5.61	0.02	1.8	15
SiO₂	19.82 - 23.19	0.05	2.4	-
P₂O₅	0.025 - 0.29	0.01	2.3	12
SO₃	1.67 - 4.61	0.02	3.7	11
K₂O	0.16 - 1.27	0.02	6.5	9
CaO	58.68 - 67.43	0.15	6.9	-
TiO₂	0.16 - 0.29	0.001	4.7	14
Mn₂O₃	0.013 - 0.26	0.002	14	7
Fe₂O₃	0.31 - 4.68	0.015	23	5
ZnO	0.01 - 0.02	-	65	3
SrO	0.037 - 0.20	0.002	150	2

Table 2: Reproducibility Measurement of two Cement Samples

Sample	Na ₂ O	MgO	Al ₂ O ₃	SiO ₂	P ₂ O ₅	SO ₃	K ₂ O	CaO	Fe ₂ O ₃	Sum
A	%	%	%	%	%	%	%	%	%	%
B										

19:05	0,367	4,02	3,15	21,10	0,094	2,26	0,824	62,12	4,40	98,3
19:10	0,121	1,28	5,01	20,09	0,055	4,67	1,255	62,91	2,18	97,6
19:15	0,357	3,99	3,10	21,09	0,096	2,26	0,825	62,04	4,39	98,1
19:20	0,120	1,27	5,00	20,11	0,054	4,64	1,255	62,87	2,17	97,5
19:25	0,353	4,00	3,08	21,11	0,094	2,26	0,824	62,06	4,39	98,2
19:30	0,120	1,27	4,99	20,08	0,055	4,64	1,252	62,90	2,18	97,5
19:35	0,358	4,02	3,08	21,08	0,098	2,25	0,828	62,05	4,40	98,2
19:40	0,124	1,26	5,03	20,10	0,058	4,66	1,252	62,91	2,18	97,6

Each sample every 10 minutes until next morning

10:45	0,363	3,99	3,10	21,13	0,100	2,26	0,824	62,11	4,40	98,3
10:50	0,122	1,28	5,03	20,06	0,061	4,66	1,250	62,80	2,18	97,4
10:55	0,367	4,02	3,08	21,07	0,102	2,26	0,826	62,08	4,40	98,2
11:00	0,120	1,27	5,01	20,10	0,059	4,66	1,253	62,83	2,18	97,5
11:05	0,356	4,00	3,10	21,13	0,097	2,25	0,828	62,11	4,40	98,3
11:10	0,123	1,26	5,02	20,02	0,064	4,65	1,253	62,81	2,17	97,4
11:15	0,360	3,99	3,10	21,08	0,102	2,26	0,829	62,13	4,39	98,2

Results for sample A

	Na ₂ O	MgO	Al ₂ O ₃	SiO ₂	P ₂ O ₅	SO ₃	K ₂ O	CaO	Fe ₂ O ₃	Sum
	%	%	%	%	%	%	%	%	%	%
Average	0,358	4,00	3,08	21,10	0,097	2,26	0,827	62,11	4,40	98,23
StdDev	0,004	0,01	0,02	0,02	0,002	0,005	0,003	0,04	0,005	0,06
RSD	1,18	0,23	0,62	0,11	2,29	0,24	0,31	0,06	0,12	0,06

Results for sample B

	Na ₂ O	MgO	Al ₂ O ₃	SiO ₂	P ₂ O ₅	SO ₃	K ₂ O	CaO	Fe ₂ O ₃	Sum
	%	%	%	%	%	%	%	%	%	%
Average	0,121	1,27	5,00	20,08	0,059	4,65	1,253	62,85	2,17	97,45
StdDev	0,003	0,01	0,02	0,02	0,002	0,007	0,002	0,04	0,004	0,06
RSD	2,82	0,47	0,45	0,11	3,63	0,15	0,18	0,07	0,17	0,06

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