

Application News

No. X254

X-ray Analysis

Quantitative Analysis of Cement by EDX-8000

High accuracy quality control analysis*1 of cement is typically conducted using a wavelength dispersive X-ray fluorescence spectrometer. EDX instruments have become capable in recent years of analysis accuracy comparable to that of low-output wavelength dispersive type instruments. Not only do they offer such conveniences as the ability to analyze powders as they are, their range of applicability has greatly widened to include the analysis of such substances as cement. Here, we introduce the analysis accuracy obtained with pressure-formed cement standard powder samples using the new model EDX-8000.

*1 ISO 29581-2, JIS R 5204

Sample Preparation

Pressure forming was conducted using a vinyl chloride ring (inner diameter 35 mm ϕ), with a total pressure of 250 kN for 60 seconds. A photograph of the sample is shown in Fig. 1.

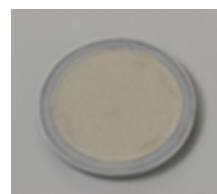


Fig. 1 Pressure-Formed Briquette of Cement

Sample

NIST Certificate of Analysis Standard Reference Materials® Portland Cement

SRM 1880b, 1881a, 1884b, 1886a, 1887b, 1888b, 1889a
Table 1 shows the standard values.

Table 1 Standard Values [mass%]

	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	CaO	MgO	SO ₃	K ₂ O	Na ₂ O
1880b	20.42	5.183	3.681	64.16	1.176	2.710	0.646	0.091
1881a	22.26	7.060	3.090	57.58	2.981	3.366	1.228	0.199
1884b	19.30	4.851	2.937	61.31	4.740	4.034	0.957	0.278
1886a	22.38	3.875	0.152	67.87	1.932	2.086	0.093	0.021
1887b	19.59	4.911	2.471	61.15	3.624	4.599	0.961	0.288
1888b	20.42	4.277	3.062	63.13	3.562	2.634	0.658	0.136
1889a	20.66	3.89	1.937	65.34	0.814	2.690	0.605	0.195

Calibration Curves

The calibration curve and accuracy (1 σ) for each element are shown in Fig. 2.

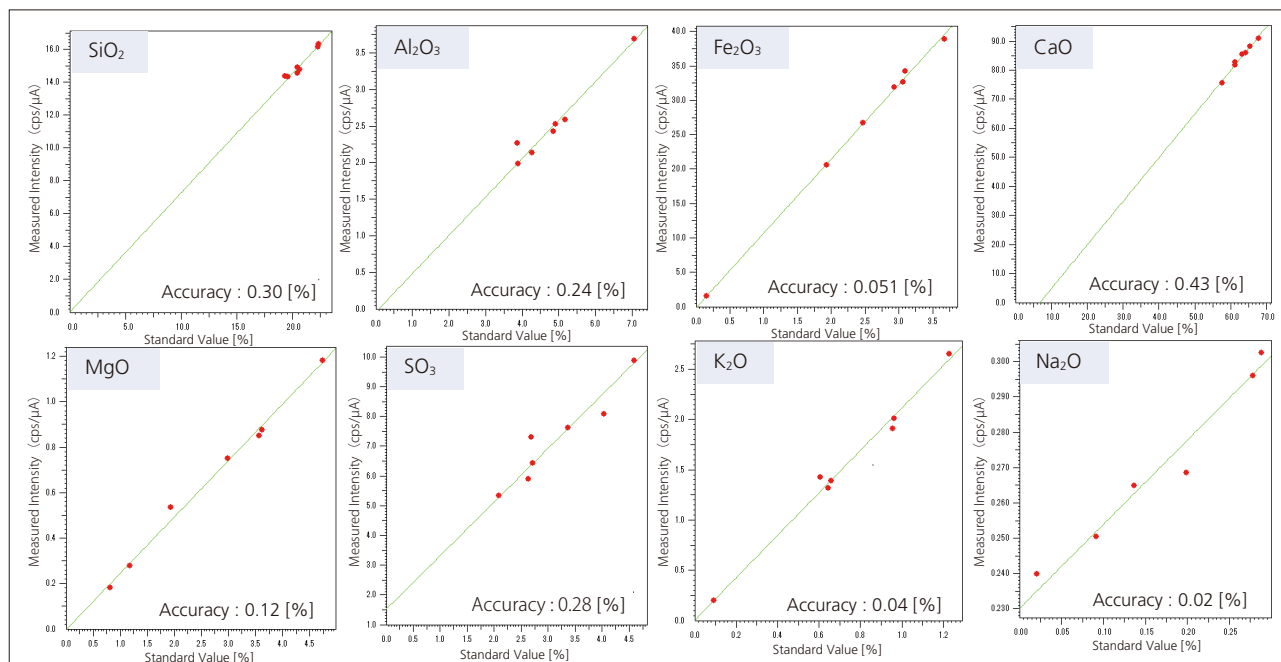


Fig. 2 Calibration Curves and Accuracy

■ Lower Limits of Detection (L.L.D.)

The lower limits of detection calculated using the above calibration curves are shown in Table 2.

Table 2 Lower Limits of Detection (300 sec, 100 sec only for Fe₂O₃)

	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	CaO	MgO	SO ₃	K ₂ O	Na ₂ O
Lower Limit of Detection (LLD)	–	0.0083	0.0022	–	0.0157	0.0066	0.0049	0.0159

■ Repeatability

Using the above calibration curve method, the repeatability test results for SRM 1880b shown in Table 3 were obtained by simply conducting 10 repeat

measurements. The X-ray fluorescence spectra for each measurement element of each sample are shown in Fig. 3.

Table 3 Repeatability for SRM 1880b (300 sec, 100 sec only for Fe₂O₃)

	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	CaO	MgO	SO ₃	K ₂ O	Na ₂ O
1	20.09	5.032	3.615	64.12	1.088	2.729	0.622	0.100
2	20.05	5.031	3.609	64.15	1.087	2.740	0.621	0.098
3	20.04	5.043	3.615	64.18	1.087	2.736	0.612	0.107
4	20.01	5.022	3.625	64.16	1.089	2.738	0.616	0.105
5	19.96	5.038	3.618	64.18	1.148	2.744	0.620	0.100
6	20.02	5.045	3.625	64.18	1.094	2.744	0.615	0.114
7	20.11	5.052	3.630	64.18	1.157	2.743	0.616	0.110
8	20.09	5.037	3.628	64.17	1.174	2.740	0.619	0.112
9	19.98	5.032	3.631	64.17	1.101	2.741	0.616	0.109
10	20.14	5.040	3.614	64.21	1.158	2.745	0.621	0.100
Average	20.05	5.037	3.621	64.17	1.118	2.740	0.618	0.105
Standard Deviation	0.059	0.008	0.008	0.025	0.036	0.005	0.003	0.006
Coefficient of Variation [%]	0.30	0.17	0.22	0.04	3.2	0.17	0.52	5.5

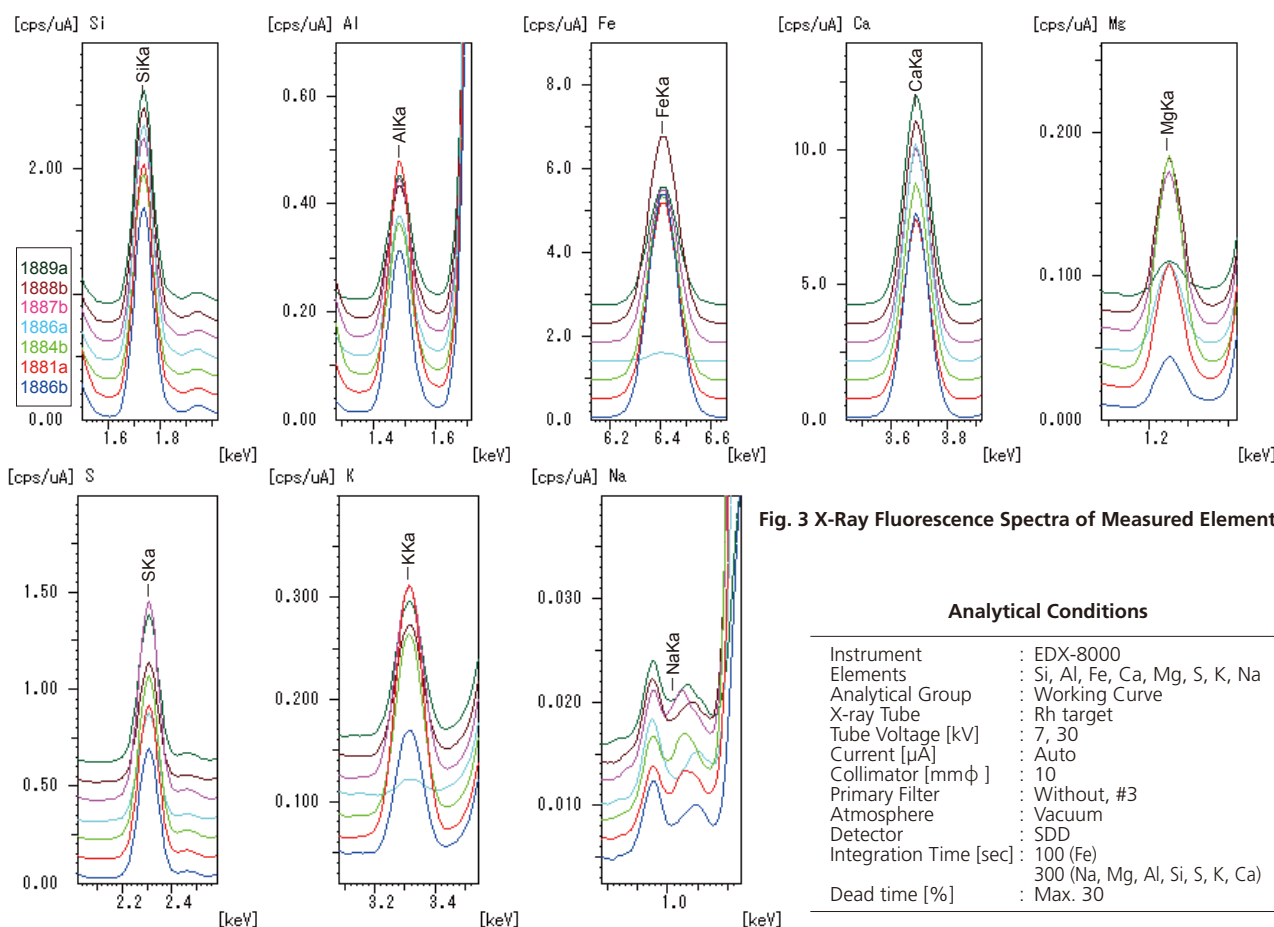


Fig. 3 X-Ray Fluorescence Spectra of Measured Elements

Analytical Conditions

Instrument	: EDX-8000
Elements	: Si, Al, Fe, Ca, Mg, S, K, Na
Analytical Group	: Working Curve
X-ray Tube	: Rh target
Tube Voltage [kV]	: 7, 30
Current [μA]	: Auto
Collimator [mmφ]	: 10
Primary Filter	: Without, #3
Atmosphere	: Vacuum
Detector	: SDD
Integration Time [sec]	: 100 (Fe) 300 (Na, Mg, Al, Si, S, K, Ca)
Dead time [%]	: Max. 30

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