

Application News

No. X254A

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.

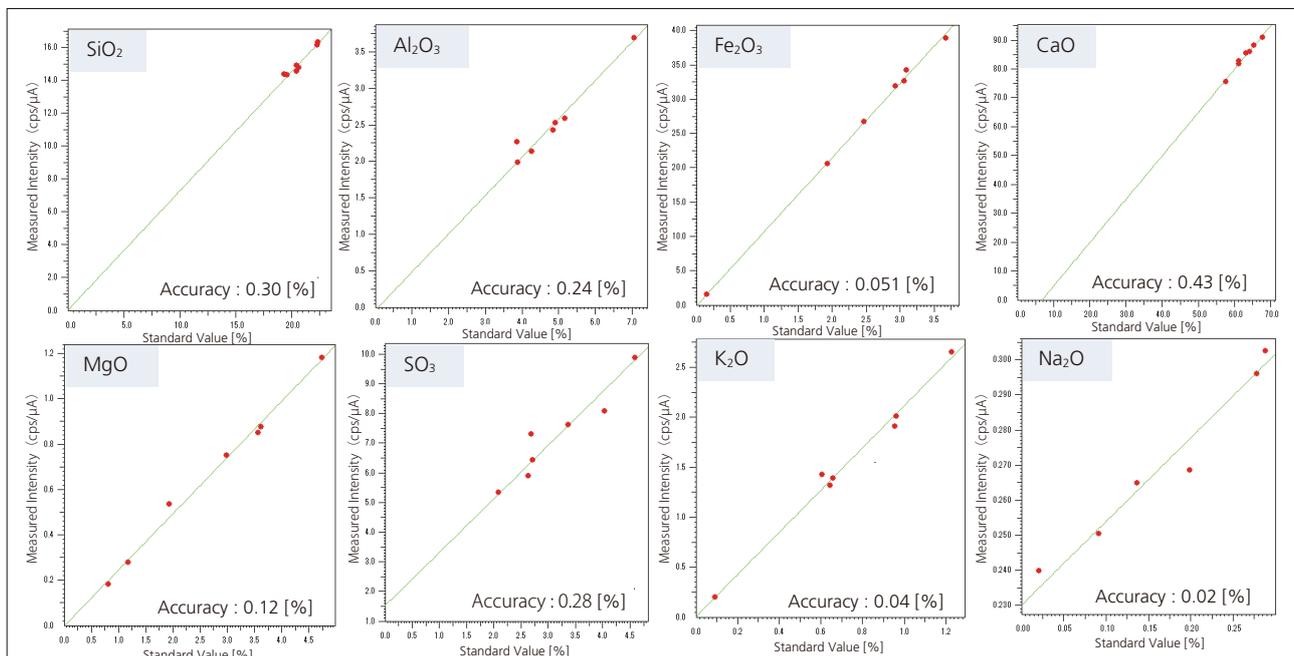


Fig. 2 Calibration Curves and Accuracy

■ 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 |

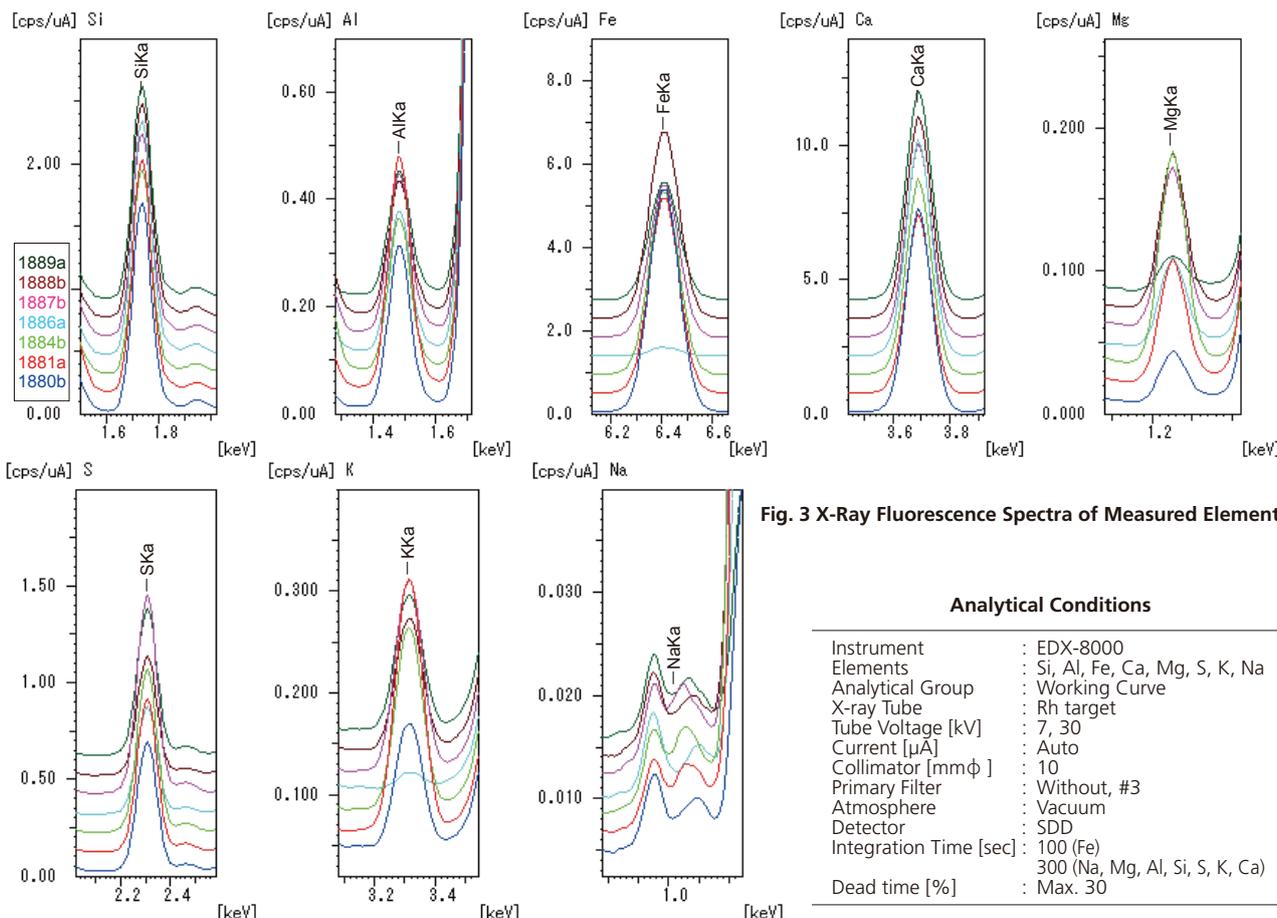


Fig. 3 X-Ray Fluorescence Spectra of Measured Elements

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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