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Application News

No.J115

Inductively Coupled Plasma Atomic Emission Spectrometry

Analysis of Elements in Cement Using ICPE-9820

Introduction

Cement is used as an ingredient in concrete, and its chemical composition can affect the quality of the concrete as a product. In particular, the concentration of CaO as the principle component in cement affects the time until the concrete sets to its full hardness, and trace components such as Fe_2O_3, MgO, TiO_2, etc. will affect the appearance of concrete.
Here, using the Shimadzu ICPE-9820 simultaneous ICP atomic emission spectrometer, we conducted analysis of the elements in typical Portland cement and in the Portland cement mixed with slag and fly ash. Pretreatment of the sample was conducted using the alkali melt process based on the Japan Cement Association standard test method JCAS I-52-2000[1]. The ICPE-9820, which adopts a vertically oriented plasma torch which effectively inhibits the precipitation of salts on the torch, permits accurate analysis of high salt concentration samples such as fused alkali samples.

Samples

- Ordinary Portland cement JCA-CRM-1 (Certified reference material)
- Portland cement SRM 1881a (Certified reference material) (Mixture of slag and fly ash)

Sample Preparation

Each samples was processed according to the pretreatment method adopted in the Japan Cement Association standard test method JCAS I-52-2000[1], as follows.
The sample was weighed out to 0.2 g, and 16.0 mL of nitric acid (1 + 9) was added to dissolve the sample. After filtering, the undissolved residue was ashed, and then fused after adding 0.08 g lithium meta-borate to the residue. The fused residue was then dissolved with the addition of 4 mL water and 0.8 mL nitric acid. The dissolution and filtrate solutions were combined, 1.6 mL nitric acid was added, and the total volume was adjusted to 100 mL. The resulting solution served as the analytical sample.
The standard solution was prepared by diluting a commercially available single-element standard solution with pure water, using the same concentrations of flux and acid.
Also, for the internal standards, single element standard solutions of Y (yttrium) and In (indium) were added to all of the samples at the same concentration.

Instrument and Analytical Conditions

Measurements were conducted using the Shimadzu ICPE-9820 simultaneous ICP atomic emission spectrometer. The measurement conditions are shown in Table 1. When conducting analysis of high salt concentration samples with many ICP instruments, clogging of the plasma torch due to precipitation of salts at the plasma torch tip is a common problem. The Shimadzu ICPE-9820, however, adopts a vertically-oriented torch which suppresses such clogging of the torch to an extremely low level. This, therefore, permits stable analysis of cement and high salt concentration samples that are subjected to pretreatment by the alkali fusion method.
Also, the Shimadzu ICPE-9820 adopts a vacuum spectrometer, so measurement of wavelengths in the vacuum UV region, such as in the case of P, can be conducted economically and stably without having to rely on high purity gas, which is required for purge-type spectrometers.

Table 1 Analytical Conditions

Instrument	: ICPE-9820
Radio Frequency Power	: 1.20 kW
Plasma Gas Flowrate	: 14.0 L/min
Auxiliary Gas Flowrate	: 1.20 L/min
Carrier Gas Flowrate	: 0.70 L/min
Sample Introduction	: Nebulizer, 10UES
Misting Chamber	: Cyclone chamber
Plasma Torch	: Torch for high-concentration salt solution samples
Observation	: Axial (AX)/Radial (RD)

Analysis

Using the calibration curve method–internal standard method, analysis was conducted for the certified elements, including JCA-CRM-1: Fe, Ca, Mg, Na, K, Ti, P, Mn, Sr, SRM1881a: Fe, Ca, Mg, Na, K, Ti, P, Mn, Sr, Cr, Zn.

Analytical Results

Table 2 shows the analytical results and the certification values. The analytical results were consistent with the certification values. Fig. 1 shows spectral profiles for Ca, Ti, and Mn. Fig. 2 shows the calibration curves for Fe, Mg, and P.

Conclusion

The multiple elements in the cement samples subjected to pretreatment by the alkali fusion method can be accurately analyzed using the ICPE-9820.

[References]
1) Japan Cement Association JCAS I-52-2000
Quantitation Method for Trace Components in Cement by ICP Emission Spectrometry and Electrical-Heating Atomic Absorption Analysis

Table 2 Analytical Results for Cement

(Unit : %)

Substance	JCA-CRM-1		SRM 1881a	
	Quantitation Value	Certification Value	Quantitation Value	Certification Value
Fe_2O_3	2.65	2.67±0.02	3.00	3.09±0.11
CaO	65.27	65.21±0.10	57.72	57.58±0.34
MgO	2.16	2.13±0.03	2.92	2.981±0.077
Na_2O	0.25	0.26±0.02	0.20	0.199±0.007
K_2O	0.56	0.56±0.02	1.24	1.228±0.029
TiO_2	0.35	0.35±0.01	0.368	0.3663±0.0030
P_2O_5	0.28	0.28±0.01	0.14	0.1459±0.0057
MnO*	0.061	0.06±0.01	-	-
Mn_2O_5*	-	-	0.105	0.1042±0.0016
SrO	0.045	0.05±0.01	0.040	0.036±0.004
Cr_2O_3	-	-	0.0586	0.0588±0.0020
ZnO	-	-	0.0509	0.0489±0.0028

* The certification value of JCA-CRM-1 pertains to MnO, and the certification value of SRM 1881a pertains to Mn_2O_5.

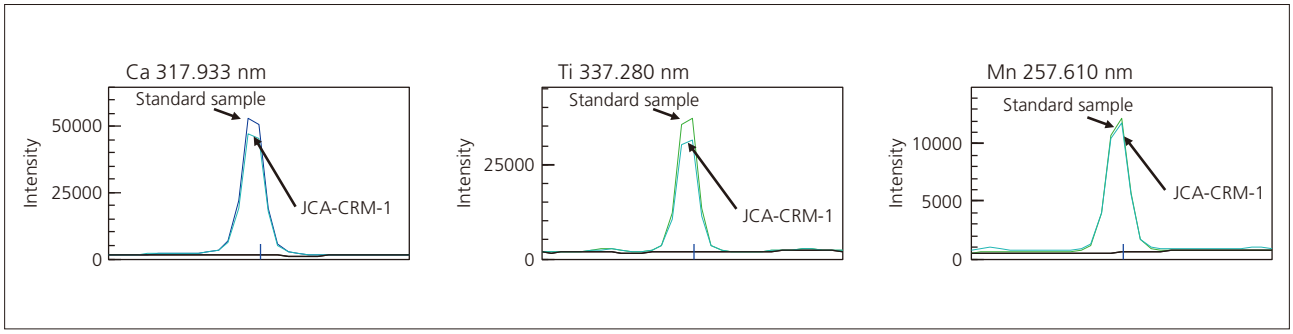

Fig. 1 Spectral Profiles of Ca, Ti, and Mn

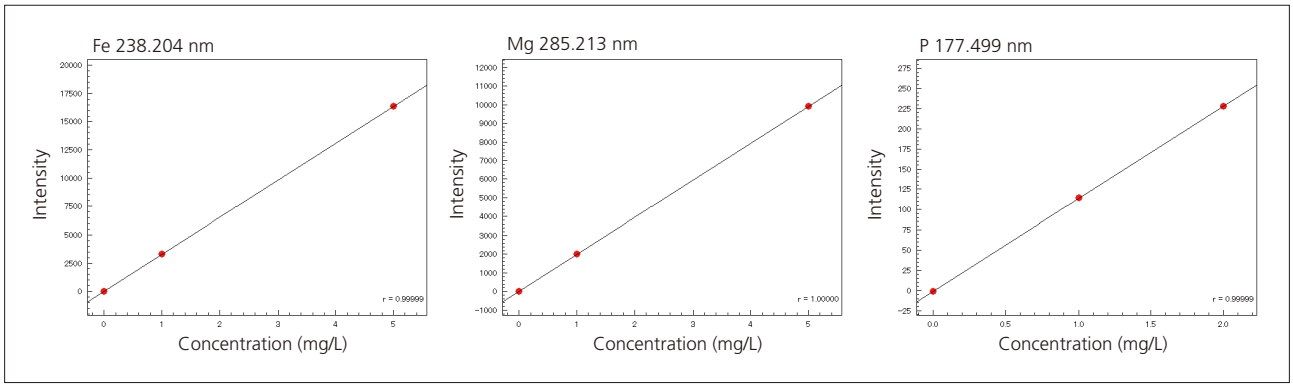

Fig. 2 Calibration Curves for Fe, Mg, and P

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