

Application News

Spectrophotometric Analysis

Measurement of Sodium and Potassium in Portland Cement by Flame Atomic Absorption Method

No.A441

Introduction

Portland cement may be classified into 6 types according to the intended use, including ordinary, highearly-strength, ultra-early-strength, moderate-heat, low-heat, and sulfate-resistant, all of which are available in low-alkali types¹⁾. The low-alkali types contain concentrations of Na and K that are suppressed to low levels, as they are considered to be causative agents of the alkali-aggregate reaction. Low alkalinity is defined based on total alkalinity, and is classified as such when its content is 0.6 % or less.

Total alkalinity (Na₂Oeq) = Na₂O + 0.658 × K₂O

where.

Na₂O:Percent concentration (%) of sodium oxide in Portland cement

K₂O: Percent concentration (%) of potassium oxide in Portland cement

Here, using flame atomic absorption method, we introduce an example of analysis of Na and K in a standard sample of Portland cement (211R) obtained from the Japan Cement Association to determine their respective concentrations.

Sample Preparation

Sample preparation was conducted according to the procedure specified in JIS R 5202-2010 "Methods for chemical analysis of cements." The sample decomposition flow chart is shown in Fig. 1.

Accurately weigh 1 g of sample into beaker

↓ ← Add 10 mL perchloric acid (60 %)

Stir with glass rod to dissolve

↓

Heat on hot plate until white fumes evolve

↓

Heat for about 5 minutes covered with watch glass

↓

After cooling, wash watch glass with water and remove

↓ ← Add 5 mL HCl (1 + 1) and about 20 mL warm water

Dissolve the soluble salts

↓

Filter with 5B filter paper and wash filter paper with warm water

↓

After cooling, adjust volume to 100 mL with distilled water (stock solution)

Fig. 1 Sample Decomposition Flow Chart

Analytical Method and Conditions

A 200-to-1 dilution of the stock solution was used as the measurement sample, and measurement was conducted using the calibration curve method. Standard solutions were prepared at the concentrations shown in Table 1 by diluting a commercially available standard solution for atomic absorption measurement. If interference occurs due to calcium as the principle constituent, prepare standard solutions that include calcium. The principal measurement conditions are shown in Table 2.

Table 1 Standard Solutions

	Std-1	Std-2	Std-3	Std-4	Std-5
Na, K	0	0.05	0.10	0.20	0.50

Hydrochloric acid concentration: Approx. 0.12 mol/L Unit: mg/L

Table 2 Principal Measurement Conditions

Instrument	AA-7000			
Element	Na	K		
Analysis Wavelength	589.0 nm	766.5 nm		
Slit Width	0.2 nm	0.7 nm		
Current Value	12 mA	10 mA		
Lamp Mode	NON-BGC			
Flame Type	Air-C ₂ H ₂			

Results

Fig. 2 and 3 show the calibration curves for Na and K, respectively. Fig. 4 and 5 show the respective peak profiles. Table 3 shows the measurement results. The oxide concentrations (%) in the solid substance and the total amount of alkali (%) were calculated from the Na and K concentrations in the measurement solution.

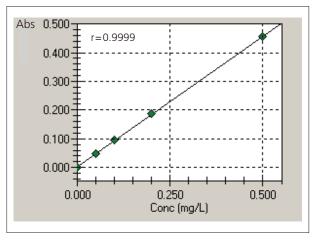


Fig. 2 Calibration Curve for Sodium

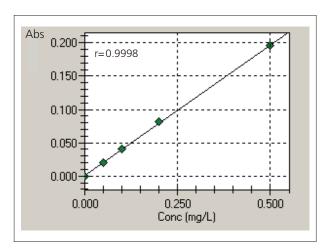


Fig. 3 Calibration Curve for Potassium

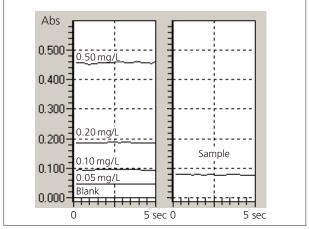


Fig. 4 Peak Profiles of Standards and Sample for Sodium

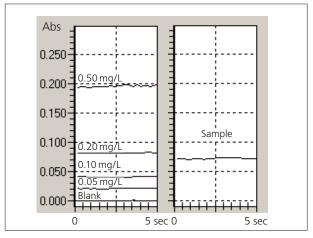


Fig. 5 Peak Profiles of Standards and Sample for Potassium

Table 3 Measurement Results

	Na ₂ O Concentration	K ₂ O Concentration	Total Alkalinity Content
	Concentration	Concentration	Content
Cement standard sample 211R	0.226 % (0.221 %)	0.436 % (0.438 %)	0.65 %

 Na_2O concentration = 1.348 × Na concentration K_2O concentration = 1.205 × K concentration

Total alkalinity content = Na_2O concentration + 0.658 x K_2O concentration Values in parentheses are the values listed on the certificate.

[Reference]

1) Japanese Industrial Standards JIS R 5210-2009 "Portland cement"

