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HIRANUMA APPLICATION DATA		Automatic Titrator	Data No.	N1	Feb. 10,2021
Cement Concrete	Determination of chloride ion in cement				

1. Abstract

This report introduces an example of the determination of chloride ion in cement.

This measurement method is described in “Method for measuring chloride” of “Japanese industrial standard JIS R 5202 Method for chemical analysis of cements”. The sample is dissolved in nitric acid, a chloride ion standard solution and a hydrogen peroxide solution are added, and the sample is heat-treated, and then the measurement is performed by precipitation titration using a silver nitrate standard solution. The measurements are made by potentiometric titration using a chloride ion-selective electrode as the electrode for end point detection.



2. Configuration of instruments and Reagents

(1) Configuration

Main unit : Automatic Titrator COM series

Electrode : Chloride ion-selective electrode CLi-081 (Connect to IE-2)

Reference electrode MS-231Z (Connect to RE-2)

*Remark

The general reference electrode (RE-201Z) cannot be used for this titration because KCl inner solution might come out to sample solution and it causes measurement error.

The inner electrodes of MS-231Z use mercury (I) sulfate. When these electrodes are disposed, please ask the specialized industrial waste disposal operator.

(2) Reagents

Titrant : 0.005 mol/L Silver nitrate standard solution

Additive : Nitric acid (60 %, density 1.38 g/mL)

Hydrogen peroxide (Approx. 30 %)

0.005 mol/L Chloride ion standard solution

3. Measurement procedure

- (1) Take 5 g of sample into a 200 mL beaker and accurately weigh it.
- (2) Add about 20 mL of DI water. Then add 12 mL of nitric acid with stirring to dissolve the sample.
- (3) Add warm DI water to make 100 mL sample solution.
- (4) Add 2 mL of chloride ion standard solution with volumetric pipette.
- (5) Add 2 mL of 30 % hydrogen peroxide.
- (6) Cover the beaker with watch glass and heat to boil for 1-2 minute. Then cool it to room temperature.

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pages. beaker, add warm DI water to make 100

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4. Measurement conditions and results

Examples of titration conditions

(1) Measurement of blank

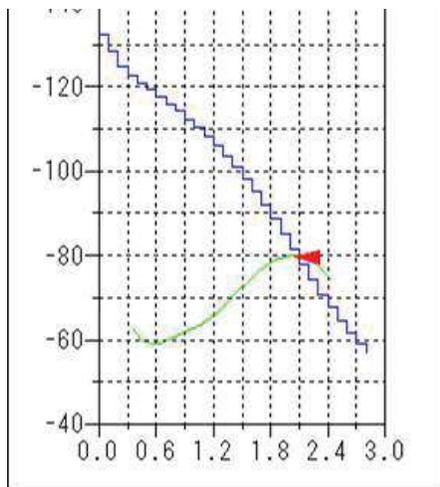
Cndt No.	1	Constant No.	6	Mode No.	20
Method	Auto	Size	0 g	Pre Int	0 sec
Buret No.	1	Blank	0 mL	Del K	2
Amp No.	2	Molarity	0.005 mol/L	Del Sens	0 mV
D. Unit	mV	Factor	1.001	Int Time	5 sec
S-Timer	5 sec	K	0	Int Sens	3 mV
C.P. mL	0 mL	L	0	BrT Speed	2
T Timer	0 sec	Unit	mL	Pulse	80
D.P. mL	0.2 mL	Formula			
End Sens	200				
Over mL	0.5 mL		D		
Max Vol.	20 mL	Decimal Places	3		

(2) Measurement of sample

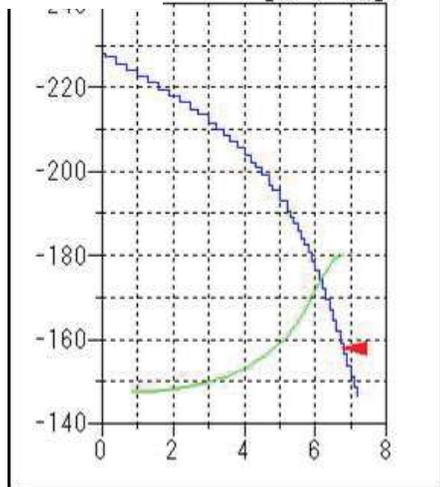
Cndt No.	2	Constant No.	7	Mode No.	20
Method	Auto	Size	5.0233 g	Pre Int	0 sec
Buret No.	1	Blank	2.062 mL	Del K	2
Amp No.	2	Molarity	0.005 mol/L	Del Sens	0 mV
D. Unit	mV	Factor	1.001	Int Time	5 sec
S-Timer	5 sec	K	0	Int Sens	3 mV
C.P. mL	0 mL	L	0	BrT Speed	2
T Timer	0 sec	Unit	%	Pulse	80
D.P. mL	0.1 mL	Formula			
End Sens	150		(D-B)*F*0.01773/S		
Over mL	0.5 mL				
Max Vol.	20 mL	Decimal Places	4		

Measurement results

Sample	Number of measurement	Sample Size(g)	Titration volume (mL)	Chloride ion(%)	Statistical Calculation
BLANK	1		2.055		Avg. 2.062 mL
	2	—	2.078	—	SD 0.014 mL
	3		2.052		RSD 0.69 %
SAMPLE	1	5.0098	6.749	0.0166	Avg. 0.0168 %
	2	5.0152	6.825	0.0169	SD 0.0002 %



Measurement of blank



Measurement of sample

Examples of measurement curves

5.Note

· Indicator electrode

Chloride ion-selective electrode was used as an indicator electrode for this measurement.

In addition to the above electrode, a silver electrode coated with silver chloride (model: AG-311A) can also be used for this measurement. However, since the silver electrode coated with silver chloride deteriorates after long-term use, the electrode potential change near the end point becomes small and unclear. On the other hand, the chloride ion-selective electrode has the advantage that when the sensitivity deteriorates, the sensitivity can be easily restored by lightly polishing the sensitive membrane with a sandpaper.

Keywords: Chloride ion, Precipitation titration, Cement, JIS R5202

*Some measurement would not be possible depending on optional configuration of system.

