HIRANUMA APPLICATION DATA	Automatic Titrator	Data No.	N2	Feb. 10,2021

Cement Concrete	Determination of chloride ion in concrete

1.Abstract

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

This measurement method is described in “Potentiometric titration method using chloride ion-selective electrode” of “JIS A 1154 Method of test for chloride ion content in hardened concrete”. Nitric acid is added to adjust the pH to 3 or less, and extract chloride ion with boiling. Cool to room temperature, filtrate to separate undissolved component and collect filtrated solution as sample. Chloride ion is determined by precipitation titration using a silver nitrate standard solution.

$$Cl^- + AgNO_3 \rightarrow AgCl + NO_3^-$$

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.
Filter paper	:	For quantitative analysis

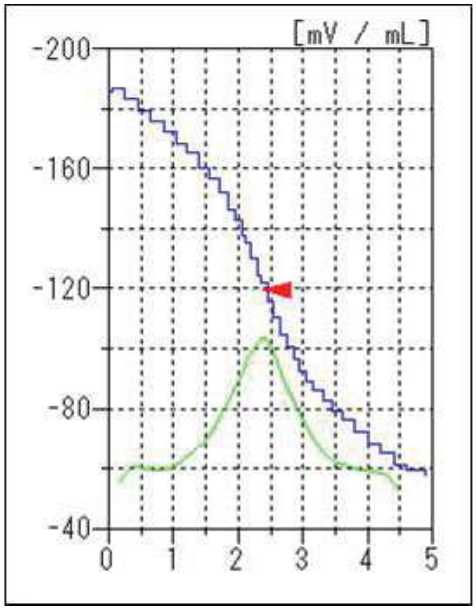
(2)Reagents

Titrant	:	0.005 mol/L Silver nitrate standard solution
Additive	:	Diluted nitric acid
		Prepared by mixing nitric acid (60 %) in a volume ratio of 1 to 6 in DI water

3. Measurement procedure

(1) Take 10 g of sample into a 200 mL beaker and accurately weigh it.
(2) Add 70 mL of diluted nitric acid slowly and stirrer it. Then measure the pH to confirm the pH is 3 or less.
(3) Cover the beaker with watch glass and heat to boil for 5 minute. Then cool it to room temperature.
(4) Filtrate the solution using filter paper with aspirating and wash the filter paper with water.
(5) Collect filtrated solution into a volumetric flask to make it 200 mL with DI water.
(6) Take the solution prepared at (5) with a 50 mL volumetric pipet into a 100 mL beaker.
(7) Immerse electrodes and titrate with 0.005 mol/L silver nitrate standard solution.

Cndt No.	1							
Method	Auto		Constant No.	1		Mode No.	8	
Buret No.	1		Size	10.0314	g	Pre Int	0	sec
Amp No.	2		Blank	0	mL	Del K	5	
D. Unit	mV		Molarity	0.005	mol/L	Del Sens	0	mV
S-Timer	5	sec	Factor	1		Int Time	5	sec
C.P. mL	0	mL	K	35.45		Int Sens	3	mV
T Timer	0	sec	L	50		Brt Speed	2	
D.P. mL	0.1	mL	Unit	%		Pulse	40	
End Sens	30		Formula					
Over mL	0.5	mL		(D-B)*K*F*M/(S*10)*200/L				
Max Vol.	20	mL	Decimal Places	4				



Example of titration curve

Measurement results

Number of measurement	Sample size(g)	Titration volume(mL)	Chloride ion (%)
1		2.326	0.0164
2	10.0314	2.340	0.0165
3		2.296	0.0162
		Avg.	0.0164 %
		SD	0.0002 %
		RSD	0.93 %

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, Concrete, JIS A1154

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