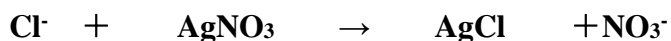


<b>HIRANUMA APPLICATION DATA</b>	Automatic Titrator	Data No.	N1	Feb. 10, 2021
<b>Cement Concrete</b>	<b>Determination of chloride ion in cement</b>			

## 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 of instruments

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.
- (7) Immerse electrodes and titrate with 0.005 mol/L silver nitrate standard solution.
- (8) As blank test, take 2 mL of 0.005 mol/L chloride ion standard solution exactly in a 200 mL beaker, add warm DI water to make 100 mL, and perform procedure (6) and (7).

### 4. Measurement conditions and results

#### Example of titration condition

#### (1) Measurement of blank

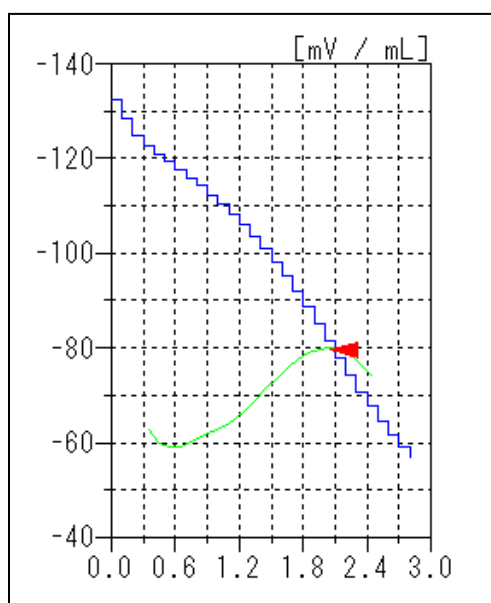
Cnd. No.	1	Constant No.	6	Mode No.	20
Method	Auto	Size	0 g	Pre Int	0 sec
Buret No.	1	Blank	0 mL	Del K	2
Amp No.	2	Morality	0.005 mol/L	Del Sens	0 mV
D.Unit	mV	Factor	1.001	Int Time	5 sec
S- Timer	5 sec	K	0	Int Sens	3 mV
C.P. mL	0 mL	L	0	BrT Speed	2
T.Timer	0 sec	Unit	mL	Pulse	80
D.P. mL	0.2 mL	Formula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

#### (2) Measurement of sample

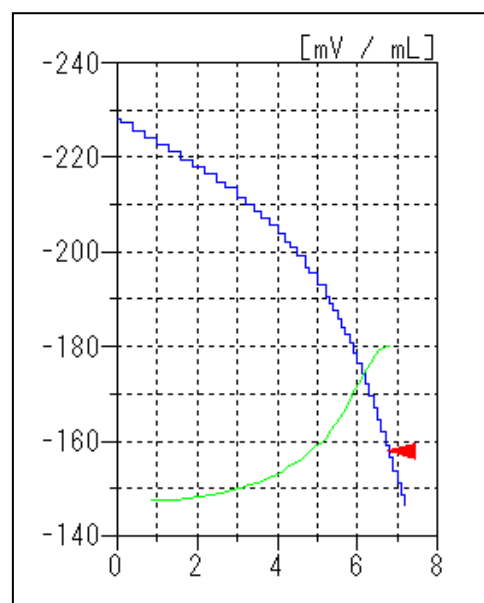
Cnd. No.	2	Constant No.	7	Mode No.	20
Method	Auto	Size	5.0233 g	Pre Int	0 sec
Buret No.	1	Blank	2.062 mL	Del K	2
Amp No.	2	Morality	0.005 mol/L	Del Sens	0 mV
D.Unit	mV	Factor	1.001	Int Time	5 sec
S- Timer	5 sec	K	0	Int Sens	3 mV
C.P. mL	0 mL	L	0	BrT Speed	2
T.Timer	0 sec	Unit	%	Pulse	80
D.P. mL	0.1 mL	Formula			
End Sens	150				
Over mL	0.5 mL		(D-B)*F*0.01773/S		
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 %
	3	5.0233	6.836	0.0169	RSD 1.03 %



Blank



Sample

### Example of titration curve

#### 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.