

Mineral Ore

Chlorine Ion in Concrete

Precipitation titration by
Automatic Potentiometric Titrator

Standard**Japan Concrete Institute**

1. Abstract

Quantification of chlorine ion (Cl^-) in hardened concrete is determined by potentiometric titration using Chlorine ion electrode and Mercury sulfate reference electrode based on the below quoted standard test methods.

After 2mol/L nitric acid and 30% hydrogen peroxide solution are added to the sample, leave it to cool to room temperature, and add 0.005mol/L sodium chloride.

Titrate with 0.005mol/L silver nitrate up to the endpoint, which is the maximum inflexion point on titration curve.

The chlorine ion is calculated from titration volume of silver nitrate.

2. Reference

- 1) Japan Concrete Institute guidance JCI-SC5 Simplified Test Method for Total Salinity in Concrete
- 2) Japan Concrete Institute proposition JCI-SC6 Test Method for Chloride Ion in Fresh Concrete by Chloride Ion Selective Electrode

3. Cautions in measurement

- 1) Handle with care when work on chemicals.
- 2) Use chlorine ion selective electrode, not silver electrode.
- 3) Since addition method using sodium chloride is applied, the blank level obtained by blank test must be deducted from titration.

4. Post-measurement care

Polish sensor element of chlorine ion electrode with supplied polishing paper (for electrode).

5. Test equipment

Main unit: Automatic potentiometric titrator (Standard preamplifier STD-)

Electrode: Chlorine ion electrode

Mercury sulfate reference electrode

6. Reagent

Titrant : 0.005mol/L silver nitrate solution (f=1.006)

Solvent : Pure water, 2mol/L nitric acid, 30% hydrogen peroxide,
0.005mol/L sodium chloride solution

7. Measurement procedure

—Measurement—

- 1) Deliver 2.0g sample to a 200mL beaker, and add 20mL pure water to stir.
- 2) Add 20.0mL of 2mol/L nitric acid.
- 3) Add 1.0mL of 30% hydrogen peroxide solution.
- 4) Add 50mL pure water.
- 5) Cover with watch glass, heat for 10 minutes, and leave it cool to room temperature.
- 6) Add 5.0mL of 0.005mol/L sodium chloride.
- 7) Titrate with 0.005mol/L silver nitrate.
- 8) Likewise, perform blank test.

8. Formula

Chlorine ion (ppm) = (EP1 - BL1) × TF × C1 × K1 / SIZE

EP1 : Titration volume (mL)

BL1 : Blank level (5.0978 mL)

FA1 : Factor of titrant (1.006)

C1 : Concentration conversion coefficient (0.1775mg/mL)
(1mL of 0.005mol/L AgNO₃ = 0.1775mg Cl⁻)

K1 : Coefficient (1000)

SIZE : Sample size (g)

9. Example of measurement

— Ambient condition —

Room temperature : 24 °C	Humidity : 63 %	Weather : Fair
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- Titration parameter -

Model : AT-510
 Method No. : 10
 Titr.mode : Intermit Titr
 Titr.form : EP Stop

[Titration parameter]

Form : EP Stop
 APB No. : 1
 Unit No. : 1
 Detector No. : 1
 Unit : mV
 Max. Volume : 20.0mL
 Wait Time : 20s
 Direction : Auto

[Control parameter]

End Point No. : 1
 End sense : Auto
 End Point Area : Off
 Separation : Off
 Over Titr.Vol. : 0mL
 Cut-off Time : 10s
 Unit Volume : 0.15mL
 Dispense Speed : 5s/mL
 Gain : 5
 Data samp.Pot. : 4.0mV
 Data samp.Vol. : 0.15mL

- Titration curve -

<Calculation>

Calc.Type : Sample
 Conc.1 : Set
 CO1=
 (EP1-BL1)*TF*C1*K1/SIZ
 E

Unit : ppm
 EP No. : 1
 Temp.Comp. : Off

<Constant>

C1(mg/mL) : 0.1775
 K1 : 1000

<Titr. Constant>

Factor : 1.006

<Blank>

Blank1 : 5.0978

*** R e s u l t ***

Sample No. : 02-01
 Date : 2000/06/20 16:19

Method No. : 10
 <Intermit Titr.>
 Method Name:
 Intermit Titr.

<Blank>

Blank 1 : 5.0978

Titr.Time : 00:25:20

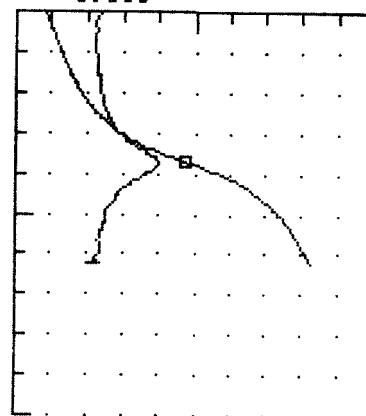
Size : 2.0167g

Conc-1 : 605.46ppm

End Point-1

Volume : 11.9358mL
 Potential :- 155.0mV

- 250 [mV] - 50
 0.000



32.000
 [mL]

(The above printout data are obtained from titration by AT-510)

«Titration parameter»

Form: of titration / APB No. the burette used in titration / Unit No.: [APB Unit File number](#)

Detector No.: the detector used in titration/ Unit of potential / Max Volume: of titration

Wait Time: before titration starts/ Direction.: of titration

«Control parameter»

End Point No.: number of EPs / End sense: of EP detection / End Point Area: EP detection area

Separation: of potential / Dispense Speed: [of dosing](#) /Over Titr.Vol. over-titrated volume / Cut-off Time: intermittent/ Unit Volume: single dose volume /Gain: sensitivity of detection signal

Data samp.Pot.: potential change of sampling signal / Data samp.Vol. : titration volume of sampling signal

«Calculation parameter»

Calc.Type: of formula / Conc.1 formula 1 / Unit: of results /EP No. of calculation

Temp.Comp.: temperature compensation / C1(mg/mL): concentration conversion coefficient

K1: unit conversion coefficient / Factor: factor of titration liquid / Blank1: blank level 1

—Measurement results—

n	Sample (g)	Titration (mL)	Chlorine ion (ppm)	Chlorine ion after batch processed	
				Mean	SD
1	<u>2.0167</u>	<u>11.9358</u>	<u>605.46</u>	607.31 ppm	2.46 ppm
2	2.0119	11.9718	610.10		0.40 %
3	2.0089	11.9196	606.37		

*The above results were obtained by 3 tests of the same sample.

* Red underline shows the data from page 3/4.

10. Summary

Salinity saturation is enhanced by concentrated salt in frozen concrete during winter time, leading to corrosion of iron and steel products. To quantify assess of salinity by freezing, it is necessary to determine the relation of concentrated salinity to time dependency factor of freezing.

Chlorine is proton number 17 element with chemical symbol Cl, which is a family member of halogens.

The test result shows a good repeatability with 0.4% relative standard deviation.

Precise and reliable measurement is assured by the automated potentiometry.

The analysis of chlorine ion in hardened concrete can be perfectly made by any of the following titration systems manufactured by Kyoto Electronics (KEM).

【AT-610】



Awarded Product of Supreme Technology from Kyoto City

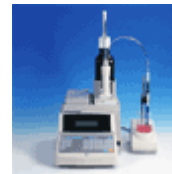
- Easy key entry by touch panel of large color LCD (8-inch wide)
- Simultaneous titration in parallel
- Both potentiometric and Karl Fischer moisture titration (coulometric·volumetric) can be performed at a time.

【AT-510】



- Compact and cost performance model
- PC card expands data memory for convenience and versatility.

【AT-500N-1】



- Low cost and high performance
- Easy view with back light LCD
- GLP/GMP conformed model

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