

Application Note

Chloride ion in sodium hydroxide

Industry	:	Chemicals
Instrument	:	Automatic potentiometric titrator
Measurement method	:	Potentiometric titration / Precipitation titration
Standards	:	ASTM E291

1. Scope

A test sample (Set concentration of Cl⁻ : 0.57%) was prepared by adding sodium chloride (NaCl) to 52% sodium hydroxide (NaOH) solution, and chloride ion (Cl⁻) concentration of the sample was measured based on “ASTM E291 Standard Test Method for Chemical Analysis of Caustic Soda and Caustic Potash (Sodium Hydroxide and Potassium Hydroxide)”.

A sample was diluted by pure water and neutralized by concentrated nitric acid (HNO₃). Then, the sample solution was potentiometrically titrated with 0.1mol/L silver nitrate (AgNO₃) solution. An inflection point on the titration curve was regarded as the endpoint, and the Cl⁻ concentration was calculated from the volume of AgNO₃ solution consumed to titrate sample to the endpoint.

2. Precautions

- 1) Samples are strong base and might cause loss of eyesight if they get into the eyes, so wear safety glasses when handling samples.
- 2) When a sample is neutralized by concentrated HNO₃, temperature of the sample solution rises by neutralization heat. If precipitation titration is performed at high temperature, measurement error might occur, so the titration should be performed after the temperature of the sample solution cooled to a room temperature.

3. Post-measurement procedure

- 1) Samples are strong base and corrode glass-made electrodes. To remove samples, soak electrodes into pure water with stirring after each measurement.
- 2) When precipitation of silver chloride (AgCl) adheres to the surface of silver electrode, remove the precipitation by polishing paper.

4. Apparatus

Main unit	:	Automatic potentiometric titrator (preamplifier : STD)
Electrode	:	Combined silver electrode (inner solution : 1mol/L KNO ₃ solution) pH glass electrode Temperature compensation electrode

5. Reagents

Titrant	:	0.1mol/L AgNO ₃ aqueous solution
pH adjusting reagent	:	Concentrated HNO ₃
Dispersion	:	1% Polyoxyethylene sorbitan monolaurate (Tween20) aqueous solution

6. Procedure

-Measurement-

- 1) Weigh 10g of a sample into a 200mL beaker, and then add pure water to make the total volume of the solution about 100mL.
- 2) Add concentrated HNO₃ until pH of the solution become 8.2 and further add 0.4mL of it.
- 3) Cool the solution to room temperature.
- 4) Add 1.5mL of 1% Tween20 solution.*
- 5) Titrate with 0.1mol/L AgNO₃ solution to measure Cl concentration.

-Blank test-

- 1) Add 100mL of pure water into a 200mL beaker.
- 2) Add concentrated HNO₃ of the same amount as that of the concentrated HNO₃ used to neutralize the sample.
- 3) Add 1.5mL of 1% Tween20 solution.*
- 4) Titrate with 0.1mol/L AgNO₃ solution to measure blank level.

*Though this operation is not described in the standard, addition of Tween20 improves dispersibility of AgCl and suppresses contamination of electrodes and burette tip.

7. Calculation

$$\text{Cl concentration (\%)} = (\text{EP1} - \text{BL1}) \times \text{TF} \times \text{C1} \times \text{K1} / \text{S}$$

EP1	:	Titer (mL)
BL1	:	Blank level = 0.0156mL
TF	:	Factor of titrant = 0.9939
C1	:	Concentration conversion coefficient = 3.5453mg/mL
K1	:	Unit conversion coefficient = 0.1
S	:	Sample (g)

8. Example

-Titration parameter-

Neutralization by concentrated HNO₃

< **Titr. Mode** > : Auto Int.
 < **Titr. Form** > : Level Stop

< **Titr. Para.** >

Max. Volume : 20 (mL)
 Channel/Unit(Ctrl.) : Ch1, pH
 Channel/Unit(Ref.) : Off
 pH Polarity : Standard
 Titr. Type Check : No Check
 Direction : Auto
 Wait Time : 0 (s)
 Dose Mode : None

< **Ctrl. Para.** >

Number of EP : 1
 1st End Level : 8.20 pH
 Gain : 1
 Data Sampling : Auto
 Ctrl. Speed : Standard
 Other Ctrl. : Set
 Titr. Over Vol. : 0.40 (mL)
 End Time : 0 (s)
 Auto Int. Mode : Standard
 Stirrer Speed : 3

Measurement of NaCl concentration

< Titr. Mode > : Auto Int.
 < Titr. Form > : EP Stop

< Ctrl. Para. >

Number of EP : 1
 End Sense : Auto
 Gain : 1
 Data Sampling : Auto
 Ctrl. Speed : Standard
 Other Ctrl. : Standard
 Auto Int. Mode : Standard (Sample)
 : Blank (Blank test)
 Stirrer Speed : 3

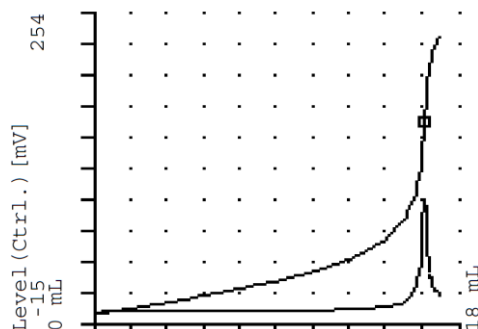
< Titr. Para. >

Max. Volume : 20 (mL) (Sample)
 : 1 (mL) (Blank test)
 Channel/Unit(Ctrl.) : Ch2, mV
 Channel/Unit(Ref.) : Off
 pH Polarity : Standard
 Titr. Type Check : No Check
 Direction : Auto
 Wait Time : 5 (s)
 Dose Mode : None

(The measurement parameter and the titration curve are an example of our automatic potentiometric titrator. In some titrators, parameter item may be different or another parameter item may be added.)

Sample (set concentration : 0.57%)

-Titration curve-



-Measurement results-

	Sample	HNO ₃ Amt.	Titer	Cl ⁻ Conc.
	(g)	(mL)	(mL)	(%)
1	9.9278	10.0110	16.1285	0.572
2	9.9656	10.0560	16.2562	0.574
3	9.9450	10.0280	16.1899	0.573
Mean	-	-	-	0.573
SD	-	-	-	0.001
RSD (%)	-	-	-	0.18

9. Summary

In this measurement, the results showed a good repeatability with 0.18% RSD (Relative standard deviation).

In some samples, verification of the measurement capability is required. In such case, please contact us.

10. References

- 1) ASTM E291-09 Standard Test Method for Chemical Analysis of Caustic Soda and Caustic Potash (Sodium Hydroxide and Potassium Hydroxide)