

Application Note

Sodium chloride in 48% sodium hydroxide

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

1. Scope

Samples (Set concentration of NaCl : 10, 50, 100ppm) were prepared by adding each amount of NaCl solution to 48% sodium hydroxide (NaOH) solution, and NaCl concentrations of them were measured.

After a fixed amount of NaCl solution having a known concentration was added to a sample, the solution was diluted by pure water and 8mol/L HNO₃ solution was dropped to the solution until its pH become 4 or below. Then, the sample solution was potentiometrically titrated with 0.005mol/L silver nitrate (AgNO₃) solution. An inflection point on the titration curve was regarded as the endpoint, and the NaCl concentration was calculated from the volume of AgNO₃ solution consumed to titrate sample to the endpoint.

In JIS K1200-3-1, 2 standards which specifies measurements of chloride content in NaOH for industrial use, mercury(II) thiocyanate photometry or ion chromatography is specified for measurements of low NaCl content samples like the present samples, but the former method needs a mercury compound which requires strict handling managements and the latter method needs complicated pretreatments. The method described in this application note enables measurements of low NaCl content samples without use of a mercury compound or complicated pretreatments.

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 8mol/L HNO₃ solution, 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	:	0.005mol/L AgNO ₃ aqueous solution
pH adjusting reagent	:	8mol/L HNO ₃ aqueous solution
Addition reagent	:	0.005mol/L NaCl aqueous solution

6. Procedure

-Blank test-

- 1) Add 5mL of 0.005mol/L NaCl solution into a 200mL beaker, and then add pure water to make the total volume of the solution about 100mL.
- 2) Titrate with 0.005mol/L AgNO₃ solution to measure blank level.

-Measurement-

- 1) Weigh 10g of a sample into a 200mL beaker.
- 2) Add 5mL of 0.005mol/L NaCl solution into the beaker, and then add pure water to make the total volume of the solution about 100mL.
- 3) Add 8mol/L HNO₃ solution until pH of the solution become 4 or below.
- 4) Cool the solution to a room temperature.
- 5) Titrate with 0.005mol/L AgNO₃ solution to measure NaCl concentration.

7. Calculation

NaCl concentration (ppm) = (EP1 - BL1) × TF × C1 × K1 / S

EP1	:	Titer (mL)
BL1	:	Blank level = 5.0550mL (Sample①) 5.0223mL (Sample②,③)
TF	:	Factor of titrant = 0.9892 (Sample①) 0.9956 (Sample②,③)
C1	:	Concentration conversion coefficient = 0.2922mg/mL
K1	:	Unit conversion coefficient = 1000
S	:	Sample (g)

8. Example

-Titration parameter-

Neutralization by 8mol/L HNO₃ solution

< **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 : 4.00 pH
 Gain : 1
 Data Sampling : Auto
 Ctrl. Speed : Standard
 Other Ctrl. : Standard
 Auto Int. Mode : Standard
 Stirrer Speed : 3

Measurement of NaCl concentration

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

< Titr. Para. >

Max. Volume : 20 (mL)
 Channel/Unit(Ctrl.) : Ch2, mV
 Channel/Unit(Ref.) : Off
 pH Polarity : Standard
 Titr. Type Check : No Check
 Direction : Auto
 Wait Time : 10 (s)
 Dose Mode : None

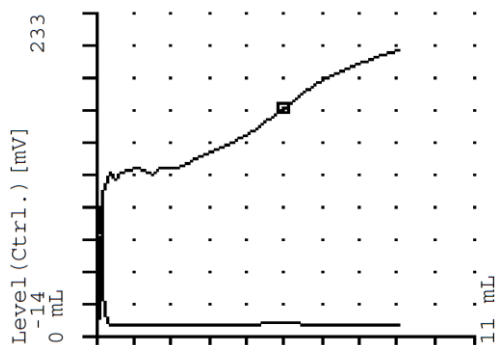
< Ctrl. Para. >

Number of EP : 1
 End Sense : Set
 dE : 50.0 dE
 dE/dmL : 50.0 dE/dmL
 Gain : 5
 Data Sampling : Set
 Data Samp. Pot. : 20.0 mV
 Data Samp. Vol. : 0.500 mL
 Ctrl. Speed : Standard
 Other Ctrl. : Standard
 Auto Int. Mode : Standard
 Stirrer Speed : 3

(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.)

Sample① (set concentration : 10ppm)

-Titration curve-

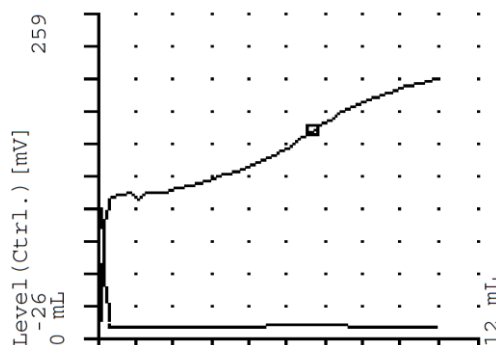


-Measurement results-

	Sample (g)	HNO ₃ Amt. (mL)	Titer (mL)	NaCl Conc. (ppm)
1	9.9953	14.7151	5.3613	8.86
2	9.9506	14.6955	5.3857	9.61
3	10.0402	14.7893	5.3535	8.59
Mean	-	-	-	9.02
SD	-	-	-	0.53
RSD (%)	-	-	-	5.86

Sample② (set concentration : 50ppm)

-Titration curve-

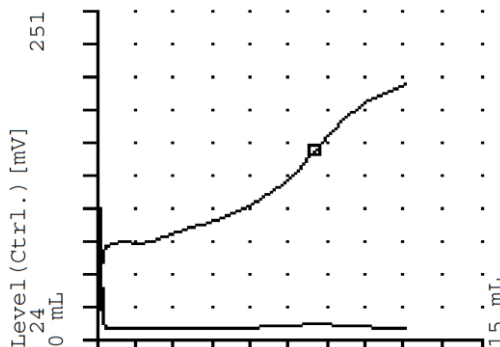


-Measurement results-

	Sample (g)	HNO ₃ Amt. (mL)	Titer (mL)	NaCl Conc. (ppm)
1	9.9777	14.9662	6.7312	49.82
2	10.0272	15.0131	6.6894	48.37
3	10.0153	15.0074	6.6841	48.27
Mean	-	-	-	48.82
SD	-	-	-	0.87
RSD (%)	-	-	-	1.78

Sample③ (set concentration : 100ppm)

-Titration curve-



-Measurement results-

	Sample (g)	HNO ₃ Amt. (mL)	Titer (mL)	NaCl Conc. (ppm)
1	9.9539	14.8170	8.4310	99.62
2	9.9718	14.8428	8.4738	100.69
3	10.0168	14.9152	8.4860	100.59
Mean	-	-	-	100.30
SD	-	-	-	0.59
RSD (%)	-	-	-	0.59

9. Summary

Though NaCl concentration of samples were as low as 100ppm or below, stable measurements were enabled by adding a fixed amount of NaCl solution having a known concentration.

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

10. References

- 1) JIS K1200-3-1 : 2000 Sodium hydroxide for industrial use - Part3 : Determination of chlorides content - Section1 : Mercury(II) thiocyanate photometry
- 2) JIS K1200-3-2 : 2000 Sodium hydroxide for industrial use - Part3 : Determination of chlorides content - Section2 : Modified Volhard method, Ion chromatographic analysis