

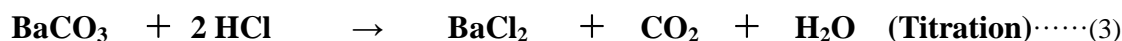
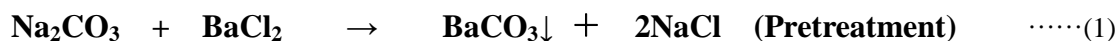
HIRANUMA APPLICATION DATA	Automatic Titrator	Data No.	H1	Apr. 19, 2018
SODA PULP INDUSTRY	Purity determination of sodium hydroxide			

1. Abstract

Sodium hydroxide is one of the most elemental reagent in chemical industrial reagents. Its production amount is large and it is used in a broad range of fields. Sodium hydroxide absorbs carbonate gas and water in atmosphere because of its properties, the purity of sodium hydroxide is gradually reduced. Japanese Industrial Standard (JIS) K8576 defines determination method for purity of sodium hydroxide (titration with indicator). Representative impure substance in sodium hydroxide includes sodium carbonate.

This report introduces an example for the quantitative determination of sodium hydroxide purity and sodium carbonate by successive and fractional potentiometric titration.

First, sodium carbonate is precipitated as barium carbonate by adding barium chloride before the titration (formula (1)). Secondly, most sodium hydroxide is titrated with highly concentrated hydrochloric acid standard solution. After that, residual sodium hydroxide is titrated with low-concentrated hydrochloric acid standard solution. It enhances the accuracy of quantitative determination for sodium hydroxide. Barium carbonate generated on pretreatment is continuously titrated with low-concentrated hydrochloric acid standard solution. The titrated volume for carbonate is doubled compared with the procedure without adding barium chloride, so it is especially available for the small amount determination of carbonate.



The reaction between sodium hydroxide and hydrochloric acid is described in formula (2). Formula (3) shows the reaction between barium carbonate and hydrochloric acid. The pHs reach 9 and 4 on the reaction (2) and (3) at each end point.

2. Configuration of instruments and reagents

(1) Configuration

Main unit : Hiranuma Automatic Titrator COM series

Option : One buret

Electrode : Platinum electrode GE-101B

: Reference electrode RE-201Z

*Instead of above electrodes, the following combination electrodes are usable.

• GR-501BZ (Fixed sleeve type)

• GR-511BZ (Flexible sleeve type)

(2) Reagents

Titrant : 1.0 mol/L Hydrochloric acid standard solution

0.1 mol/L Hydrochloric acid standard solution

Additive : 10 % Barium chloride solution

3. Measurement procedure

- (1) Take about 1.5 g of sample into 200 mL beaker and accurately weigh it.
- (2) Add 50 mL of DI water which does not contain carbon dioxide, and dissolve the sample.
- (3) Add 10mL of 10 % barium chloride solution. Flow nitrogen gas to the headspace on sample solution with 300 mL/min flow rate.
- (4) Immerse the electrodes with keeping nitrogen gas flowing, and start titration. 1.0 mol/L hydrochloric acid standard solution is dispensed first (35 mL here)*, and secondly 0.1 mol/L hydrochloric acid standard solution is titrated. *Addition amount of 1.0 mol/L hydrochloric acid is equivalent to 90 % of sodium hydroxide in sample solution.

4. Measurement conditions and results

Examples of titration conditions

- (1) Dispense 1.0 mol/L hydrochloric acid

Cndt No.	1
Method	Disp
Buret No.	1
S-Timer	5 sec
Disp Vol.	35 mL

- (2) Titration of sodium hydroxide

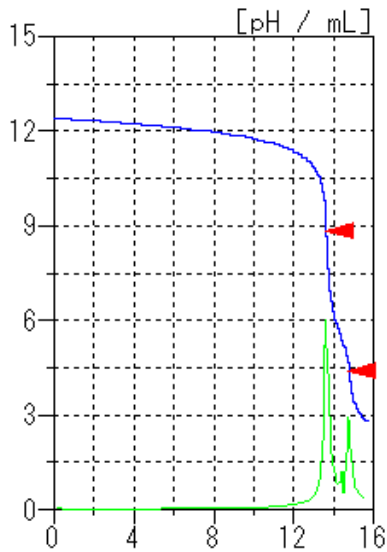
Cndt No.	2	Constant No.	2	Mode No.	5
Method	Auto	Size	1.5002 g	Pre Int	0 sec
Buret No.	2	Blank	35 mL	Del K	5
Amp No.	1	Molarity	0.10 mol/L	Del Sens	0 mV
D. Unit	pH	Factor	1.004	Int Time	3 sec
S-Timer	5 sec	K	40.00	Int Sens	3 mV
C.P. pH	0 mL	L	1.001	Brst Speed	2
Direction	0 sec	Unit	%	Pulse	40
T Timer	0 mL	Formula			
D.P. pH	1000		$(D * F * M + B * L) * K / (S * 10)$		
End Sens	0 mL	Digits	3		
Over mL	40 mL	Auto input parameter	None		

*Enter the dispensing volume and factor of 1.0 mol/L hydrochloric acid standard solution in B (Blank) and L (coefficient 2), respectively.

- (3) Titration of sodium carbonate (sodium carbonate)

Cndt No.	3	Constant No.	3	Mode No.	8
Method	Auto	Size	1.5002 g	Pre Int	0 sec
Buret No.	2	Blank	0 mL	Del K	5
Amp No.	1	Molarity	0.10 mol/L	Del Sens	0 mV
D. Unit	pH	Factor	1.004	Int Time	5 sec
S-Timer	0 sec	K	53.00	Int Sens	3 mV
C.P. pH	0 mL	L	0	Brst Speed	2
Direction	0 sec	Unit	%	Pulse	40
T Timer	0 mL	Formula			
D.P. pH	500		$(D - B) * K * F * M / (S * 10)$		
End Sens	0.2 mL	Digits	3		
Over mL	2 mL	Auto input parameter	None		

Measurement results



Example of titration curve

Measurement results of sodium hydroxide

Number of Measurement	Size (g)	Titrant Volume (mL)	Concentration (%)
1	1.5396	22.998	97.023
2	1.5002	13.590	97.052
3	1.5110	15.970	96.991
Statistic calculation			Average
			97.02 %
			Standard deviation
			0.031 %
			Coefficient of variation
			0.03 %

Measurement results of sodium carbonate

Number of Measurement	Size (g)	Titrant Volume (mL)	Concentration (%)
1	1.5396	1.236	0.429
2	1.5002	1.152	0.409
3	1.5110	1.085	0.382
Statistic calculation			Average
			0.407 %
			Standard deviation
			0.024 %
			Coefficient of variation
			5.80 %

5. Note

Atmosphere environment

Atmosphere contains about 0.03 % carbon dioxide. When carbon dioxide is absorbed into sample solution during titration for sodium hydroxide, the titrant volume consumed for sodium hydroxide is decreased, while it is increased on the titration for sodium carbonate. Flowing nitrogen gas to the headspace on sample solution serves as an effective measure against this problem. Using air passed through soda lime absorption column instead of nitrogen gas also has a positive effect.

Keywords : Sodium hydroxide, Sodium carbonate, Neutralization titration, Barium carbonate, JIS K 8576