

AQUACOUNTER Application Sheet	COM series	DATA No. H1	1st edition
Paper and Pulp		Measurement of sodium hydroxide purity	

1. Measurement outline

Sodium hydroxide is the most basic chemical among industrial chemicals, and it has a wide range of fields for use with large production volume. Due to its nature, sodium hydroxide absorbs carbon dioxide gas and moisture in air and deteriorates in its purity gradually. The method for measuring sodium hydroxide purity (indicator titration) is stipulated in JIS K 8576 (reagents). Sodium carbonate is a typical impurity in sodium hydroxide.

This section introduces an example in which quantification of 48% sodium hydroxide and 0.1% sodium carbonate were successively titrated with fractionation by potentiometric titration method.

In this section, most of the sodium hydroxide was titrated first by a high-concentration hydrochloric acid titrant, followed by titration with a low-concentration hydrochloric acid titrant to increase quantification precision. In addition, a method in which barium chloride solution was added in advance to turn sodium carbonate into sedimentary barium carbonate for titration with a low-concentration hydrochloric acid was adopted in order to increase the quantification precision for sodium carbonate.

- (1) Approximately 80g of sample is weighed precisely to be diluted accurately with decarboxylated water.
- (2) 20mL of the diluted sample solution is collected using a whole pipette. 50mL decarboxylated water and 10mL 10% barium chloride are added.
- (3) While aerating the beaker with nitrogen gas at the flow rate of 300mL/min, 1mol/L hydrochloric acid is titrated until the end point for sodium hydroxide is near in advance to successive titration with 0.1mol/L hydrochloric acid.

- Titration of sodium hydroxide



- Titration of sodium carbonate



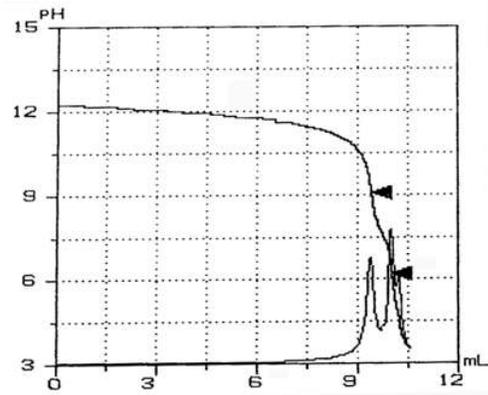
2. Reagents and Electrodes

(1) Reagents	Titrant	1. 1mol/L hydrochloric acid titrant 2. 0.1mol/L hydrochloric acid titrant
	Loading buffer	10% barium chloride solution
(2) Electrodes *standard accessories	Indicator electrode	*Glass electrode GE-101B to IE jack
	Reference electrode	*Reference electrode RE-201 to RE jack

3. Measurement conditions example (COM-1600S + 1 units of Buret B-2000-20)

Master file 1								
Condition file 1 + 2 + 3								
Parameters for condition file 1 (For addition of 1 mol/L HCl)			Parameters for condition file 2 (For NaOH determination)			Parameters for condition file 3 (For Na ₂ CO ₃ determination)		
Method	DISP		Method	AUTO		Method	AUTO	
Buret No.	1		Amp No.	1		Amp No.	1	
S Timer	10 sec		Buret No.	2		Buret No.	2	
Disp volume	38 mL		Meas Unit	pH		Meas Unit	pH	
			S Timer	10 sec		S Timer	0 sec	
			CP mL	0 mL		CP mL	0 mL	
			DP mL	0 mL		DP mL	0 mL	
			End Sens	500		End Sens	1000	
			Over mL	0 mL		Over mL	2 mL	
			Max. Vol.	40 mL		Max. Vol.	20 mL	
Mode No.	5	8	Unit	%		Unit	%	
Pre Int	0	0	Blank	38 mL		Blank	0	
Del K	5	5	Factor	Titer of the titrant		Factor	Titer of the titrant	
Del Sens	0	0	Molarity	0.1		Molarity	0.1	
Int Time	3	5	K	40		K	53	
Int Sens	3	3	L	1		L	0	
Brt Speed	2	2	Formula	$(D \times M + B \times L) \times K / (S \times 10)$		Formula	$(D - B) \times K \times F \times M / (S \times 10)$	
Pulse	40	40	Mode No.	5		Mode No.	8	

4. Measurement example



Measurement results on NaOH

Sample No.	Sample volume (g)	Titration value (mL)*	Concentration (%)
1	3.1812	9.325	48.955
2	3.1812	9.325	48.962
3	3.1812	9.358	48.959
Avg. (Average value)			48.96 %
Std. Dev. (Standard deviation)			0.004 %
C.V. (Coefficient of variation)			0.007 %

*Indicates the titration value for 0.1mol/L HCl. The dispensed volume of 1mol/L HCl was 38mL.

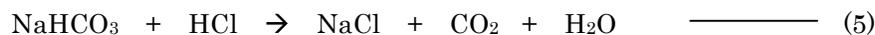
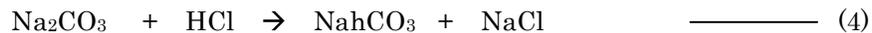
Measurement results on Na₂CO₃

Sample No.	Sample volume (g)	Titration value (mL)	Concentration (%)
1	3.1812	0.646	0.1078
2	3.1812	0.609	0.1017
3	3.1812	0.546	0.0912
Avg. (Average value)			0.100 %
Std. Dev. (Standard deviation)			0.008 %
C.V. (Coefficient of variation)			8.38 %

5. Outline

(1) About titration method for sodium carbonate

In this section, barium chloride was added to sodium carbonate to form barium carbonate for titration. In general, a method in which titration is conducted without adding the barium chloride used in this section is adopted. The feature of this method is that it is effective when the concentration of sodium carbonate is small, and it shows a titration curve with one inflection point near pH4. On the other hand, titration curve for titration method without barium chloride addition shows 2 inflection points. Their reaction formulas are provided in Formulas (4) and (5). The method without barium chloride addition shows titration curve with 2 inflection points, and the titration value to each inflection point will be 1/2 of the titration value for the barium chloride addition method in this section. Therefore, the barium chloride addition method whose titration value is twice is more advantageous in measurement of low-concentration sodium carbonate.



However, this method is not suited when the concentration of sodium carbonate is high since the precipitation of barium carbonate formed in Formula (2) becomes large and thus leads to slow titration speed in Formula (3).

(2) About titration environment

Since approximately 0.03% CO₂ is contained in air, CO₂ is absorbed and the titration value for NaOH decreases and the titration value for Na₂CO₃ increases during titration on NaOH. As a measure against this, the nitrogen gas purging method that was adopted in this section is valid. Favorable results are also possible by using air that has passed the absorption tube with soda lime instead of nitrogen gas.

Key words

Measurement of sodium hydroxide purity, titration of sodium carbonate, neutralization titration, barium chloride

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