HIRANUMA APPLICATION DATA	Automatic Titrator	Data No.	O2	Feb. 03, 2022
Factor standardization	**Standardization of sodium hydroxide titrant**			

1. Abstract

The measurement method of acidic substance by neutralization titration using a sodium hydroxide standard solution is widely used in the titration analysis. Factors are indicated on the commercially available standard solution for volumetric analysis. The factor determination is required when the standard solutions are prepared in the laboratory. Also it is effective to check the repeatability by the factor measurement using a standard material to check the performance of titrator system. Japanese Industrial Standard JIS K 8001 and the Japanese Pharmacopoeia describe that amidosulfuric acid, which is a standard material for a volumetric analysis, should be used for the factor determination of sodium hydroxide standard solution. As a simpler method, it is also possible to use a hydrochloric acid standard solution, having the same molar concentration as sodium hydroxide and a known factor, as the standard substance.

In this report, the above two types of standardization methods were performed. When amidosulfuric acid is used for the standard substance, firstly a known amount of amidosulfuric acid is dissolved in pure water. And then potentiometric titration is performed with 0.1 mol/L sodium hydroxide standard solution to determine its factor. 1 mol of amidosulfuric acid and 1 mol of sodium hydroxide react quantitatively according to Eq. (1) and show an inflection point on the titration curve.

$$(NH_2)HSO_3 + NaOH \rightarrow (NH_2)NaSO_3 + H_2O \quad \cdots(1)$$

When hydrochloric acid standard solution with known factor is used for the standard substance, firstly the 0.1 mol/L hydrochloric acid standard solution is taken into a beaker with a volumetric pipette and it is diluted with pure water. And then potentiometric titration is perform with 0.1 mol/L sodium hydroxide standard solution to determine the factor. 1 mol of hydrochloric acid and 1 mol of sodium hydroxide react quantitatively according to equation (2) and show an inflection on the titration curve.

$$HCl + NaOH \rightarrow NaCl + H_2O \quad \cdots(2)$$

1) Japanese Pharmacopoeia Eighteenth Edition
2) Japanese Industrial Standard JIS K8001 General rules for test methods of reagents

2. Configuration of instruments and reagents

(1) Configuration of instruments

Main unit	: Automatic Titrator	COM Series
Electrodes	: Glass electrode	GE-101B
	Reference electrode	RE-201Z

(2) Reagents

Titrant : 0.1 mol/L (0.1 N) sodium hydroxide standard solution (Buret No. 1)

Standard material : Amidosulfuric acid, standard material for volumetric analysis
(Certified value of purity in this report: 100.00 %)
0.1 mol/L (0.1 N) hydrochloric acid standard solution (f = 1.003)

3. Measurement procedure

(1) Procedure using amidosulfuric acid as a standard substance

i) Take about 0.1 g of amidosulfuric acid into a 100 mL beaker and weigh it accurately to 0.1 mg digits.

ii) Add 50 mL of DI water and a stirrer bar to the 100 mL beaker to dissolve the sample.

iii) Immerse the electrodes and start the measurement. Titration is performed with a 0.1 mol/L sodium hydroxide standard solution, and the inflection point on the titration curve is detected as the end point.

iv) Perform the blank test with the same procedure of sample measurement.

(2) Procedure using 0.1 mol/L hydrochloric acid standard solution as a standard substance

i) Take 10 mL of 0.1 mol/L hydrochloric acid standard solution accurately into a 100 mL beaker with volumetric pipette.

ii) Add 50 mL of DI water and a stirrer bar to the 100 mL beaker.

iii) Immerse the electrodes and start the measurement. Titration is performed with a 0.1 mol/L sodium hydroxide standard solution, and the inflection point on the titration curve is detected as the end point.

iv) Perform the blank test with the same procedure of sample measurement.

4. Measurement conditions and results

Examples of titration conditions

Blank measurement

Cndt No.	1							
Method	Auto		ConstantNo.	1		Mode No.	16	*1
Buret No.	1		Size	0	g	Pre Int	0	sec
Amp No.	1		Blank	0	mL	Del K	0	
D. Unit	pH		Molarity	0.1	mol/L	Del Sens	0	mV
S-Timer	20	sec	Factor	0		Int Time	3	sec
C.P. mL	0	mL	K	0		Int Sens	3	mV
T Timer	0	sec	L	0		Brt Speed	2	
D.P. mL	0	mL				Pulse	16	
End Sens	300		Unit	mL				
Over mL	0.3	mL	Formula	D				
Max.Vol.	1	mL	Digits	3				

*1: Since the maximum change in electrode potential is shown at the first drop of this blank titration, the end point is detected in the first drop or less volume. To detect this maximum change as an end point, set Mode No. to which the blank mode function is assigned, Mode No.12-19 for COM-A19.

Factor standardization measurement with amidosulfuric acid

Cndt No.	2							
Method	Auto		ConstantNo.	2		Mode No.	4	
Buret No.	1		Size	0	g	Pre Int	0	sec
Amp No.	1		Blank	0.01	mL	Del K	9	
D. Unit	pH		Molarity	0.1	mol/L	Del Sens	0	mV
S-Timer	20	sec	Factor	1.0000	*2	Int Time	3	sec
C.P. mL	0	mL	K	97.09	*3	Int Sens	3	mV
T Timer	0	sec	L	0		Brt Speed	2	
D.P. mL	1	mL				Pulse	40	
End Sens	300		Unit	Fact2				
Over mL	0.3	mL	Formula					
Max.Vol.	20	mL	S*F*1000/(K*M*(D-B))					
			Digits	4				

*2: Purity of amidosulfuric acid / 100

*3: It is the mass (g) of amidosulfuric acid that reacts with 1 mol of sodium hydroxide by the reaction formula (1), which is a formula amount (97.09) of amidosulfuric acid.

Factor standardization measurement with 0.1 mol/L hydrochloric acid standard solution

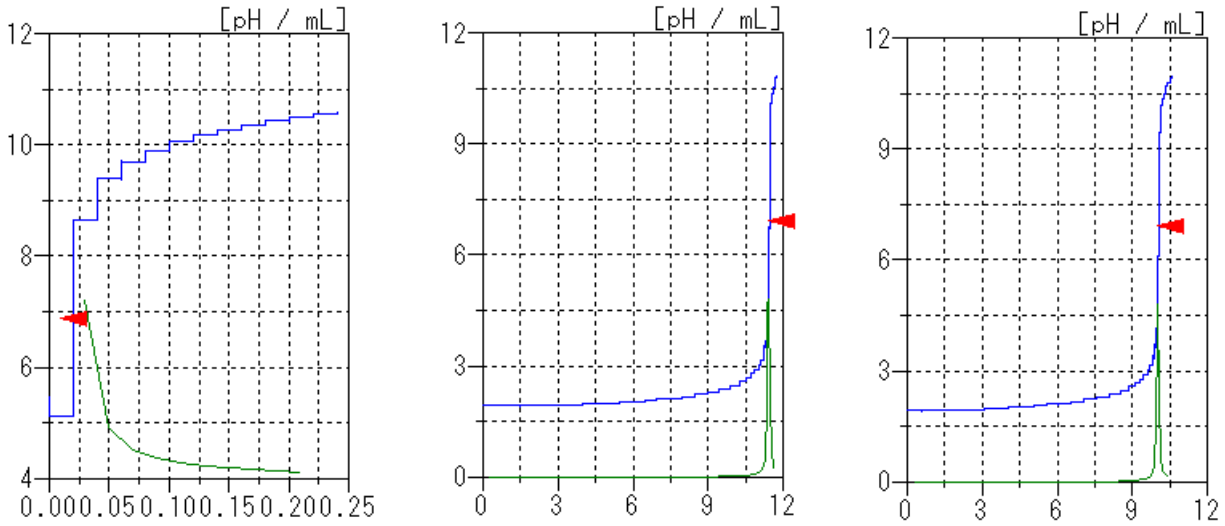
Cndt No.	3							
Method	Auto		ConstantNo.	3		Mode No.	4	
Buret No.	1		Size	10	mL	Pre Int	0	sec
Amp No.	1		Blank	0.01	mL	Del K	9	
D. Unit	pH		Molarity	0.1	mol/L	Del Sens	0	mV
S-Timer	5	sec	Factor	1.003	*4	Int Time	3	sec
C.P. mL	0	mL	K	0		Int Sens	3	mV
T Timer	0	sec	L	0		Brt Speed	2	
D.P. mL	1	mL				Pulse	40	
End Sens	500		Unit	Fact1				
Over mL	0.5	mL	Formula	S/(D-B)*F				
Max.Vol.	20	mL	Digits	4				

*4: Factor of 0.1 mol/L hydrochloric acid standard solution

Measurement results

Results of factor standardization measurement

Standard material	Measurement No.	Sample size	Titrant volume (mL)	Factor	Statistical result		
Blank	1	-	0.010	-	Avg.	0.01	mL
	2	-	0.010	-			
Amidosulfuric acid	1	0.1174 g	12.039	1.0052	Avg.	1.002	
	2	0.1190 g	12.243	1.0019	SD	0.003	
	3	0.1098 g	11.324	0.9996	RSD	0.28	%
0.1 mol/L hydrochloric acid	1	10 mL	10.009	1.0031	Avg.	1.003	
	2		10.008	1.0032	SD	0.0002	
	3		10.011	1.0029	RSD	0.02	%



Blank　　Amidosulfuric acid　　0.1 mol/L hydrochloric acid

Examples of titration curves

5. Note

(1) About the standard material

Amidosulfuric acid is used for the standardization of sodium hydroxide or potassium hydroxide standard solution in neautrarization titration. The standard material for volumetric analysis comes with a certificate value of the purity and uncertainty. If these certification and traceability are required for the management of test result, such as quality records, the standard material for volumetric analysis is used. It is necessary to prepare the standard material with pretreatment such as drying as described in its instruction before use.

For the hydrochloric acid standard solution with a known factor, either prepare it in the laboratory and standardize it according to the procedure of Application No. O1, or procure the one supplied by the reagent manufacturer as a standard solution for volumetric analysis. The measurement procedure is easier than using solid standards such as amidosulfuric acid.

Keywords：Factor standardization, Neutralization titration, Sodium hydroxide, Amidosulfuric acid