

HIRANUMA APPLICATION DATA	Automatic Titrator	Data No.	C11	Jun. 30, 2020
Detergents • Bath additives • Cosmetics	Standardization of benzethonium chloride standard solution			

1. Abstract

The determination method of anion surfactant uses a cation surfactant, with an opposite charge, as the titrant. The end point is the point at which the charge becomes neutral due to the reaction between the anion and the cation. Benzethonium chloride is used as the cation standard solution.

This report introduces a method for standardization of benzethonium chloride standard solution. Sodium dodecyl sulfate is used for standard solution. First, determine the purity of sodium dodecyl sulfate. Add diluted sulfuric acid to sodium dodecyl sulfate, heat to reflux, and then titrate with sodium hydroxide standard solution. Subsequently, prepare the standard solution of sodium dodecyl sulfate whose purity has been determined, and standardize the benzethonium chloride standard solution with titration against sodium dodecyl sulfate standard solution.

2. Configuration of instruments and reagents

(1) Configuration of instruments

Main unit	:	Hiranuma Automatic Titrator COM series
Electrodes	:	Glass-reference combination electrode GR-525BZ (Used to measure the purity of sodium dodecyl sulfate)
		Surfactant electrode SU-091
		Reference electrode RE-201Z (Used for the standardization of benzethonium chloride)

(2) Reagents

Titration	:	0.004 mol/L Benzethonium chloride standard solution Dissolve 0.896 g of benzethonium chloride in DI water and prepare 500 mL solution.
Standard solution	:	0.004 mol/L sodium dodecyl sulfate standard solution Dissolve 1.154 g of sodium dodecyl sulfate in DI water and prepare 1 L solution.
Additive	:	0.5 mol/L sulfuric acid
Titration	:	1 mol/L sodium hydroxide standard solution
Rinse solution	:	ethanol (purity: 99.5 %)

3. Measurement procedure

3.1. Measurement for the purity of sodium dodecyl sulfate

- (1) Take 5 g of sample into a 300 mL Erlenmeyer flask and accurately weigh it.
- (2) Add 25 mL of 0.5 mol/L sulfuric acid with a volumetric pipette.
- (3) Attach a reflux condenser to the flask and heat it with using hot plate or sand bath. Occasionally, swirl the flask gently while being careful not to foam. Reflux for an additional 2 hours after the solution becomes clear and does not foam.
- (4) Cool the flask to room temperature. Wash the inner wall of the reflux condenser with 30 mL of ethanol (99.5 %), and next with an appropriate amount of water. Detach the reflux condenser from the flask and add pure water to make it to 100 mL.
- (5) Immerse electrode (GR-525BZ) and start titration with 1 mol/L sodium hydroxide standard solution.
- (6) Perform blank measurement without sample.

3.2. Calculation for the factor of sodium dodecyl sulfate standard solution

Calculate factor for prepared 0.004 mol/L sodium dodecyl sulfate solution with the following formula:

$$F = (S \times (P / 100)) / (M \times 0.004) = (2.5 \times S \times P) / M$$

S: Weighing amount of sodium dodecyl sulfate (g)

P: Purity of sodium dodecyl sulfate (%)

M: Molecular weight of sodium dodecyl sulfate (288.38)

3.3. Standardization of benzethonium chloride standard solution

- (1) Take 10 mL of sodium dodecyl sulfate standard solution into a 100 mL beaker with a volumetric pipette.
- (2) Add 50 mL of DI water.
- (3) Immerse electrode (SU-091, RE-201Z) and start titration with 0.004 mol/L benzethonium chloride standard solution.

4. Measurement conditions and results

Example of titration condition

Titration condition for purity of sodium dodecyl sulfate (blank measurement)

Cndt No.	1	Constant No.	1	Mode No.	4
Method	Auto	Size	0 g	Pre Int	0 sec
Buret No.	1	Blank	0 mL	Del K	9
Amp No.	1	Molarity	1 mol/L	Del Sens	0 mV
D. Unit	pH	Factor	1.001	Int Time	3 sec
S-Timer	5 sec	K	0	Int Sens	3 mV
C.P. mL	22 mL	L	0	Brst Speed	2
T Timer	5 sec	Unit	mL	Pulse	40
D.P. mL	0 mL	Formula	D		
End Sens	1000	Digits	3		
Over mL	0.3 mL				
Max.Vol.	30 mL				

Titration condition for purity of sodium dodecyl sulfate (sample measurement)

Cndt No.	2	Constant No.	2	Mode No.	4
Method	Auto	Size	5.5582 g	Pre Int	0 sec
Buret No.	1	Blank	25.436 mL	Del K	9
Amp No.	1	Molarity	1 mol/L	Del Sens	0 mV
D. Unit	pH	Factor	1.001	Int Time	3 sec
S-Timer	5 sec	K	288.38	Int Sens	3 mV
C.P. mL	30 mL	L	0	Brst Speed	2
T Timer	5 sec	Unit	%	Pulse	40
D.P. mL	0 mL	Formula			
End Sens	1000				
Over mL	0.3 mL				
Max.Vol.	40 mL				
			$(D-B)*K*F*M/(S*10)$		
		Digits	3		

Titration condition for standardization of benzethonium chloride standard solution

Cndt No.	3	Constant No.	3	Mode No.	8
Method	Auto	Size	10 mL	Pre Int	0 sec
Buret No.	2	Blank	0 mL	Del K	5
Amp No.	2	Molarity	0.004 mol/L	Del Sens	0 mV
D. Unit	mV	Factor ※1	0.9879	Int Time	5 sec
S-Timer	20 sec	K	0	Int Sens	3 mV
C.P. mL	0 mL	L	0	Brst Speed	2
T Timer	0 sec	Unit	Fact1	Pulse	40
D.P. mL	0 mL	Formula	$S/(D-B)*F$		
End Sens	100	Digits	3		
Over mL	0.3 mL				
Max.Vol.	20 mL				

※1 Set calculated result of factor for sodium dodecyl sulfate in Section 4.2.

Measurement results

4.1. Measurement results for purity of sodium dodecyl sulfate

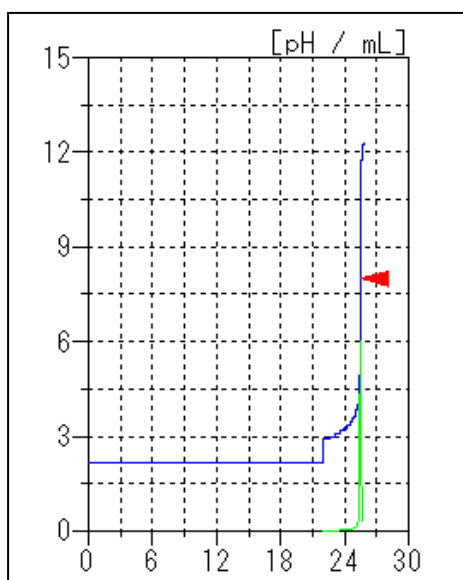
Item	Meas. No.	Size (g)	Titrant volume (mL)	Purity (%)	Average
Blank	1	—	25.444	—	25.436 mL
	2	—	25.427	—	
Purity	1	5.5582	44.462	98.813	98.780 %
	2	5.2842	43.512	98.747	

4.2. Calculated result for factor of sodium dodecyl sulfate standard solution

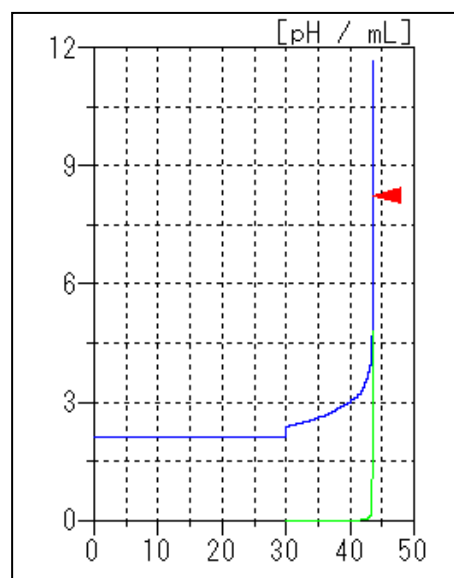
$$F = (2.5 \times 1.1536 \times 98.780) / 288.38 = \mathbf{0.9879}$$

4.3. Measurement results for standardization of benzethonium chloride standard solution

Meas. No.	Size (mL)	Titrant volume (mL)	Factor	Statistical results	
1		10.289	0.9602	Avg.:	0.9595
2	10	10.305	0.9587	SD:	0.00075
3		10.295	0.9596	RSD:	0.079 %



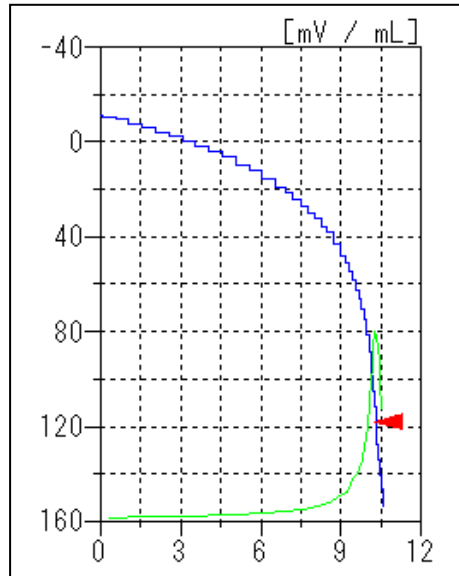
Blank



Sample

Purity of sodium dodecyl sulfate

Example of titration curve



Standardization of benzethonium chloride standard solution

Example of titration curve

5. Note

(1) Electrode

The glass reference combination electrode was used for the purity determination of sodium dodecyl sulfate, and the surfactant electrode was used for standardization of benzethonium chloride. Both measurements were able to obtain the clear inflection point on titration curve as endpoint, and it was possible to measure without problems.

Keywords: Anionic surfactant, Surfactant electrode, Sodium dodecyl sulfate, Benzethonium chloride