京都電子工業株式会社
KYOTO ELECTRONICS MANUFACTURING CO.,LTD.

Application Note

Measurement of the cationic surfactants in bath detergent using surfactant electrode and particle charge detector

Industry	:	Cosmetics & soap
Instrument	:	Automatic potentiometric titrator
Measurement method	:	Potentiometric titration / Ion association titration
Standards	:	

1. Scope

Surfactant is a compound having a hydrophibic parts in the molecule. Depending on the properties of the hydrophilic part, surfactant is classified into cationic surfactant, anionic surfactant, amphoteric surfactant, and nonionic surfactant.
In these surfactants, cationic surfactants have a property which the hydrophilic group has a positive electric charge after ionic dissociation, whereas anionic surfactants have a negative charge. Both of these electric charges react and produce association formation. The produced association formation is insoluble and precipitated in water by decreasing of electric charge and increasing of hydrophobicity during the reaction. Potentiometric titration method uses this chemical reaction to directly titrate the ionic surfactant. This Application Note introduces the case in which the cationic surfactants contained in bath detergent is measured by using two kinds of detectors (a surfactant electrode and a particle charge detector).

2. Precautions

The sensing part of the surfactant electrode does not have a high resistance to organic solvents. Therefore this part should not be cleaned with organic solvents. After measurement, the electrode should be rinsed with distilled water and wiped off water with paper towel from the sensing part of electrode.
When the particle charge detector is used, immerse the probe in ethanol after each measurement and clean by driving the piston for approximately 1 minute. After that, completely remove the ethanol by rinsing it out with distilled water before starting the next measurement.

3. Post-measurement procedure

When the surfactant electrode is used, remove the internal solution and rinse the inside with distilled water several times by using dropper. After the water from inside, attach the supplied protective cap and keep it dry. In the case that the electrode is stored with distilled water, deterioration of the electrode is accelerated due to elution of the ingredients elements of ~~from~~ the sensitive membrane into the solution.
When the particle charge detector is used, immerse the probe into ethanol, and rinse by driving the piston for approximately 1 minute. Remove the ethanol by rinsing with distilled water, and wipe off away water with a paper towel from the probe and keep the probe dry.

4. Apparatus

- Automatic potentiometric titrator (preamplifier : STD)
- Combined surfactant electrode (Internal liquid: 3.3mol / L potassium chloride solution)
- Particle charge detector

5. Reagents

Titrant : 0.002nmol/L Dodecylbenzenesulfonic acid sodium
Solvent : Pure water

6. Procedure

1) Take 0.5 g of the bathwater detergent into a beaker.
2) Add 100mL of distilled water.
3) Titrate the sample solution with 0.002mol/L of Dodecylbenzenesulfonic acid sodium, and detect inflection point as end-point in titration curve.

7. Calculation

Cationic surfactant (%) = EP1×TF×C1×K1 / S

EP1・・・Titration volume of 1st end point (mL)
TF・・・Factor of titrant ＝0.9913(Surfactant electrode) *
＝1.0091(Particle charge detector) *
C1・・・Coefficient of concentration conversion ＝0.874mg/mL
K1・・・Coefficient of unit conversion ＝0.1
S・・・Sample size (g)

*The factor was measured using 0.002 mol/L benzethonium chloride standard solution.

8. Example

-Titration parameter-
*Below parameter was used when measurement by using a surfactant electrode was performed.

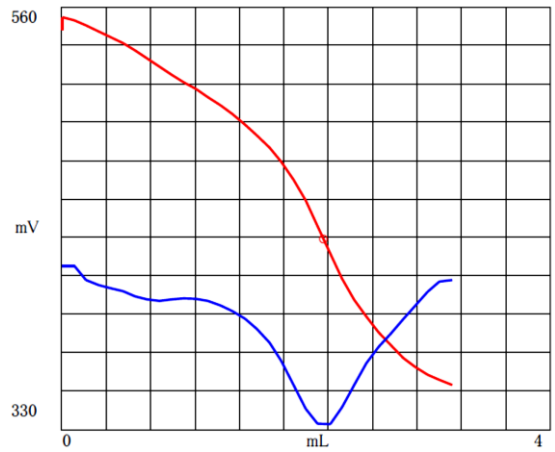
＜Titr. Mode＞	: Intermit
＜Titr. Form＞	: EP Stop
＜Titr. Para.＞	
Max. Volume	: 20 (mL)
Channel/Unit(Ctrl.)	: Ch1, mV
Channel/Unit(Ref.)	: Off
pH Polarity	: Standard
Titr. Type Check	: No Check
Wait Time	: 10 (s)
Dose Mode	: None

＜Ctrl. Para.＞	
Number of EP	: 1
End Sense	: Set
dE	: 50
dE/dmL	: 50
Gain	: 1
Data Sampling	: Set
Data sampling pot.	: 999mV
Data sampling vol.	: 0.1mL
Ctrl. Speed	: Set
Cut-off time	: 7s
Unit volume	: 0.1mL
Dispense speed	: 1s/mL
Other Ctrl.	: Standard
Auto Int. Mode	: Standard
Stirrer Speed	: 4

(The above condition is an example. The setting condition depends on the titrator model.)

—Titration curve—

*Below titration curve was obtained by using a surfactant electrode



-Titration parameter-

*Below parameter was used when measurement by using ~~a~~ particle charge detector was performed.

＜Titr. Mode＞ : Intermit

＜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
Wait Time	: 30 (s)
Dose Mode	: None

＜Particle charge detector＞

Piston Speed	: 7
Stirrer Speed	: 4

＜Ctrl. Para.＞

Number of EP	: 1
End Sense	: Auto
Gain	: 1
Data Sampling	: Set
Data sampling pot.	: 999mV
Data sampling vol.	: 0.1mL
Ctrl. Speed	: Set
Cut-off time	: 7s
Unit volume	: 0.1mL
Dispense speed	: 1s/mL
Other Ctrl.	: Standard
Auto Int. Mode	: Standard
Stirrer Speed	: 4

(The above condition is an example. The setting condition depends on the titrator model.)

—Titration curve—

*Below titration curve was obtained by using a particle charge detector

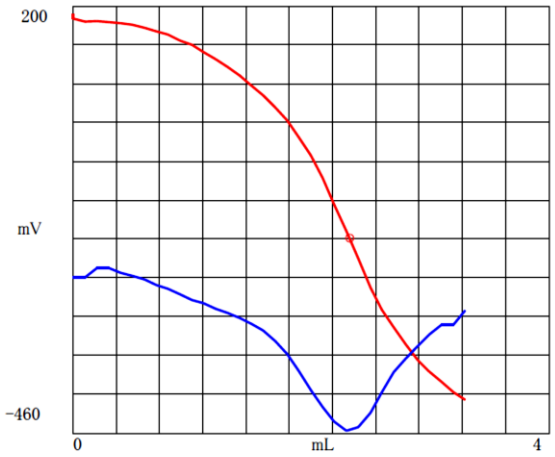


Table 1 The measurement result of cationic surfactant in bath detergent

	By using Surfactant electrode			By using Particle charge detector		
	Sample(g)	Titration amount (mL)	Conc.(%)	Sample(g)	Titration amount (mL)	Conc.(%)
1	0.5202	2.1884	0.364	0.5096	2.1613	0.374
2	0.5333	2.1768	0.354	0.5332	2.3273	0.385
3	0.5065	2.1469	0.367	0.5081	2.1433	0.372
Mean	-	-	0.362	-	-	0.377
S.D.	-	-	0.007	-	-	0.007
R.S.D(%)	-	-	1.987	-	-	1.844

9. Summary

The concentration of the anionic surfactant contained in bath detergent was determined by potentiometric titrator with using two detectors (surfactant electrode and particle charge detector).

The results by using both detectors were obtained with good repeatability which the both RSD values were less than 2%. Also, significant difference in these two measurement results were not observed.

The features of these detectors are summarized as follows: the surfactant electrode is reasonable Price compared to the particle charge detector, but the general lifetime is only from 3 to 6 months. The particle charge detector is expensive than the surfactant electrode, but probe part is chemically stable, and can be continuously used unless the main body of the unit have electrical/mechanical failure. Nevertheless, it has measurable limitation depending on some surfactant structures.

To measure surfactants by manual analysis, chloroform, which is hazardous, needs to be used. Also, the two-phase titration requires a complicated operation procedure. In automatic titration, the samples are directly titrated without using chloroform, which allows simple and quick measurement.