Application Note

Measurement of anionic surfactants in kitchen detergent by 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 hydrophobic and hydrophilic 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 which the anionic surfactants contain in kitchen detergent is measured by using two kinds of detectors (surfactant electrode and 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 : 0.004mol/L benzethonium chloride
Solvent : Distilled water

6. Procedure

1) Take 2.5 g kitchen detergent precisely into a beaker.
2) Add distilled water until 500mL.
3) Take 5mL the sample solution precisely in another beaker, and add 100mL distilled water.
4) Titrate sample solution with 0.004 mol/L of benzethonium chloride solution, and detect inflection point as end-point in titration curve.

7. Calculation

Measurement result by using surfactant electrode (%) = EP1×TF×C1×K1 / (S/R)
Measurement result by using particle charge detector (%) = EP2×TF×C1×K1 / (S/R)

EP1	・・・Titration volume of 1st end point (mL)	
EP2	・・・Titration volume of 2nd end point (mL)	
TF	・・・Factor of titrant	＝0.9819*
C1	・・・Coefficient of concentration conversion	＝1.496mg/mL
K1	・・・Coefficient of unit conversion	＝0.1
S	・・・Sample size (g)	
R	・・・Purity, dilution factor	＝100

*The factor was calculated from the mass and the purity of benzethonium chloride solution when the titrant is prepared.

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

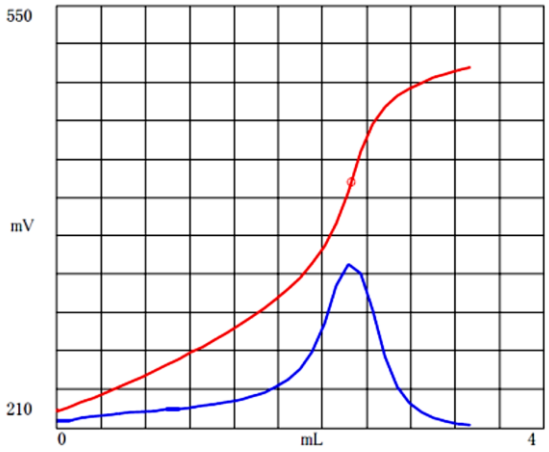
＜Ctrl. Para.＞

Number of EP	: 1
End Sense	: 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 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	: 2
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

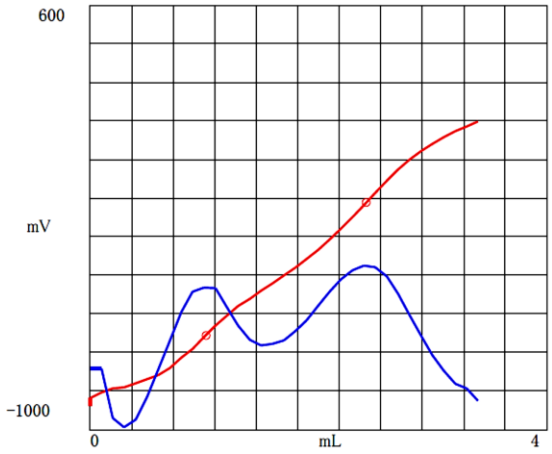


Table 1 The measurement result of anionic surfactant in kitchen detergent

	By using Surfactant electrode			By using Particle charge detector		
	Sample(g)	Titration amount (mL)	Conc.(%)	Sample(g)	Titration amount (mL)	Conc.(%)
1	2.5397	2.4231	14.01	2.5397	2.4209	14.00
2	2.5397	2.4361	14.09	2.5397	2.3877	13.81
3	2.5397	2.4418	14.12	2.5397	2.3763	13.74
Mean	-	2.4337	14.08	-	2.3950	13.85
S.D.	-	0.0096	0.06	-	0.0232	0.13
R.S.D(%)	-	0.3938	0.39	-	0.9675	0.97

9. Summary

The concentration of the anionic surfactant contained in kitchen detergent was determined by potentiometric titrator with using two detectors (surfactant electrode and particle charge detector). These results showed different of potential behaviors on the titration. When the surfactant electrode is used, single inflection point was detected in the titration curve, whereas when the particle charge detector was used, two inflection points were observed in the titration curve and the second inflection point was adopted into concentration as the end point.The above difference is caused from difference principle of these detectors.

The results by using both detectors were obtained with good repeatability which the both RSD values were less than 1%. 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 method, chloroform which is categorized in hazardous materials needs to be used. The method is the two-phase titration which requires a complicated operation procedure. In automatic titration method, the samples are directly titrated without using chloroform, which enables simple and quick measurement.