

Cosmetics
Soap

Anionic Surfactant in Washing Agent

Ionic titration by
Automatic Potentiometric Titrator

Standard	JIS ISO	K 3362 2271
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1. Abstract

Here we demonstrate quantification of anionic surfactant making use of sudden change of streaming potential at neutral point during titration by AT-510 with optional PCD-500 unit.

Add cationic (normal) solution to sample liquid, and back titrate remaining cations with anionic surfactant as titrant. Thus, we obtained measurement results of a good repeatability as detailed in below.

2. Reference

- 1) JIS K 3362-1998 Test Method for Washing Agent
- 2) ISO 2271-1989 Quantification of Surfactant – Synthetic Detergent – Anionic Surfactant (Two-phase titration)
- 3) Analytical Chemistry Vol.46, No.10, pp.763-770: Quick quantification of Ionic surfactant by Streaming potential / Ionic titration

3. Cautions in measurement

- 1) Where a plural number of endpoint is anticipated with slow potential change, it is recommended to increase concentration of titrant, and thus detection sensitivity is raised so that a better repeatability is expected.
- 2) Cut-off titration at constant speed is also recommended to know titration volume in advance in order to adjust potential change at the endpoint.
- 3) After the sensor is wiped clean of residue, dip it in water for 30 to 60 minutes until potential level stabilizes.
- 4) During repeated measurements, dip the sensor in ethanol and then water each for five minutes.

4. Post-measurement care

After a series of measurement is over for the day, blot the sensor, and dip it vibrated in ethanol and water each for five minutes. Then, stop oscillation and keep the sensor dipped in water.

5. Test equipment

Main unit : Automatic potentiometric titrator (Standard preamplifier STD-)
Electrode : Option Streaming potential detection unit PCD-500

6. Reagent

Titrant : 0.004mol/L-n-Sodium Doceylbenzenesulfonate
Solvent : Distilled water
Reagent : 0.004mol/L-Benzethonium chloride
Ethanol

7. Measurement procedure

—Preparation—

- 1) Dilute 1.3949g of n-Sodium dodecylbenzenesulfonate (M.W.348.48) with distilled water to make 1L of 0.004mol/L-n- Sodium dodecylbenzenesulfonate.
- 2) Dilute 1.7936g of Benzethonium chloride (M.W.448.09) with distilled water to make total 1L of 0.004mol/L-Benzethonium chloride.
- 3) Add distilled water to 10.0g sample to make 1L of 1% sample liquid.

—Measurement—

- 1) Deliver 10.0mL of 0.004mol/L- Benzethonium chloride to a 100mL tall beaker, and add 70mL distilled water. Titrate with 0.004mol/L-n- Sodium dodecylbenzenesulfonate to obtain blank level by back titration.
- 2) Deliver 2.0mL of 1% sample liquid to a 100mL tall beaker. Add 70mL distilled water and 10.0mL of 0.004mol/L- Benzethonium chloride. Stir for 10 minutes to react.
- 3) Titrate (back titration) with 0.004mol/L-n- Sodium dodecylbenzenesulfonate. (Endpoint is automatically detected from inflexion point.)
- 4) Each time after measurement is over, dip the electrode in ethanol and water each for five minutes in preparation for the next sample test.

8. Formula

Anionic concentration (mL) = $BL1 - EP1$

EP1 : Titration volume (mL)
BL1 : Blank level (10.024mL)
SIZE : Sample size (5.0mL)

9. Example of measurement

— Ambient condition —

Room temperature: 23°C Humidity : 52% Weather : Fair

— Titration parameter —

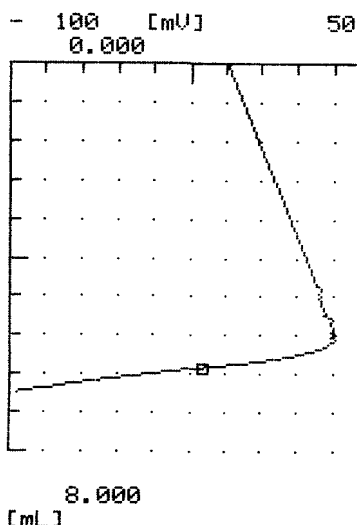
```

Model AT-510
[TITR. PARA] < Control >
                End Point No. : 1
Method No      12      End Sense   : Auto
  [Intermit Titr.]
MethodType    :      End Point Area: Off
                Separation   : Off
                Doze         Over Titr.Vol.: 0.0mL
                &Titr.      Cut-Off Time : 5s
                Unit Volume   :
< Dose >
Mode          : Vol. Stop 0.05mL
APB No.       : 1         Dispense   Speed:
Unit No.      : 1         20s/mL
Volume        : Gain      : 1
4.5mL         : Data      Samp.Pot.:
Cut-off Time  : 0s       4.0mV
Dispense      Speed:    Data      Samp.Vol.:
20s/mL        : 0.05mL
Wait Time     : 0s
                < Calculation >
< Titration >
Form          : EP Sample
STOP          Conc.1      : Set
APB No.       : 1         CO1=BL1-EP1
Unit No.      : 1
Detector No.  : 2         Unit       : mL
Unit          : mV        EP No.      : 1
Max.Volume    : Conc2     : Off
10.0mL        : Conc3     : Off
Wait Time     : 120s     Conc4     : Off
Direction     : Auto     Conc5     : Off
                Temp.Comp. : Off
Titr. Form    : EP
STOP
    
```

— Titration curve —

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*** R e s u l t ***
Sample No. : 02-05
Date : 2000/05/19 11:08
Method No. : 12
  <Intermit Titr.>
Method Name:
  Intermit Titr.
<Blank>
Blank 1 : 10.024
Titr.Time : 00:08:44
Size : 5.0007g
Conc-1 3.7266ml
End Point-1
Volume : 6.2974mL
Potential :- 19.2mV
    
```



(The above printout data are obtained from titration by AT-510 unit)

« Titration parameter »

Form: of titration / APB No. the burette used in titration / Unit No.: [APB Unit File number](#)

Detector No.: the detector used in titration / Unit of potential / Max Volume: of titration

Wait Time: before titration starts / Direction.: of titration / Titr.Form: of EP detection

« Control parameter »

End Point No.: number of EPs/End sense: of EP detection/End Point Area: EP detection area /Separation: of potential/ Over Titr.Vol. over-titrated volume /Cut-Off Time: [intermittent](#) /Unit Volume: of dose

Gain: sensitivity of detection signal/Data samp.Pot.: potential change of sampling signal / Data samp.Vol. : titration volume of sampling Stability of detection / Delay Time: before stability check /Limit Time: of stability check

« Calculation parameter »

Calc.Type: of formula / Conc.1 formula 1 / BL1: back titration volume / EP1: of back titration/ Temp .Comp.: temperature compensation /K1: unit conversion coefficient /TF: factor of titration liquid / Blank1: blank level

—Measurement results—

Run	Sample (mL)	Titration (mL)	Concentration (mL)	Anionic concentration	
				Mean	SD
1	5.0	<u>6.2974</u>	<u>3.7266</u>	3.7265mL	0.0240mL
2	5.0	6.3216	3.7024		
3	5.0	6.2736	3.7504		
				RSD	0.6440%

* The above test results were obtained by 3 tests of the same sample.

* Red underline shows the data from page 3/4.

10. Summary

The optional PCD-500 is designed by unique technology of detecting streaming potential particularly when titrating samples like surface active agent.

The ionic titration using streaming potential for EP detection shows a good repeatability of 0.6% relative standard deviation.

Precise and reliable measurement of surfactant is made possible by combination of a titration unit and PCD-500, and the analysis of anionic surfactant can be made by any of the following titration systems manufactured by Kyoto Electronics (KEM).

【AT-610】



Awarded Product of Supreme Technology from Kyoto City

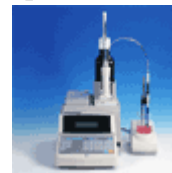
- Easy key entry by touch panel of large color LCD (8-inch wide)
- Simultaneous titration in parallel
- Both potentiometric and Karl Fischer moisture titration (coulometric + volumetric) can be performed at a time.

【AT-510】



- Compact and cost performance model
- PC card expands data memory for convenience and versatility.

【AT-500N-1】



- Low cost and high performance
- Easy view with back light LCD
- GLP/GMP conformed model

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