

Cosmetics
Soap

Sodium Sulfate of Cosmetics

Redox titration by
Automatic Potentiometric Titrator

Standard

1. Abstract

Lead sulfate precipitates when Sodium sulfate contained in cosmetics is titrated with lead ion solution.

With Potassium ferricyan – ferrocyanide indicator is added, as Sulfate ion decreases, Fe(II) ion changes to Fe(III) ion also changing electrode potential. At the endpoint of sulfate ion precipitation, only Fe(III) ion remains with a sharp potential inflexion, which is detected by the platinum electrode, and thus concentration of Sodium sulfate is obtained from titration volume.

Depending on composition, some cosmetics may not show a sharp inflexion, however, EP can be determined by setting an appropriate rate of change (RC) for detection.

2. Reference

- 1) Experiment and Calculation for Quantitative Analysis - Vol.2 – Seiji Takagi, Kyoritsu Publication

3. Cautions in measurement

- 1) 0.05mol/L Lead nitrate used for titrant must be factor checked with Potassium sulfate standard occasionally.
- 2) Each time after measurement is over, the electrode needs to be rinsed and cleaned with water in order to remove residue of precipitated Lead sulfate and Potassium ferricyan-ferrocyanide solution. Particularly, care must be taken not to clog junction of reference electrode.
- 3) Keep junction of reference electrode filled with junction liquid.

4. Post-measurement care

After a series of measurements is over, rinse well the combination electrode with water, and keep it dipped in a beaker filled with pure water to avoid drying up.

5. Equipment

Main unit : Automatic potentiometric titrator (Standard preamplifier STD—)
Electrode : Option Combination platinum electrode
(Junction liquid 3.33M-Potassium chloride)

6. Reagent

Titration : 0.05mol/L Lead nitrate (Factor 1.008)
Solvent : Ethanol
Reagent : Potassium Ferricyan-ferrocyanide test solution
0.03mol/L Hydrochloric acid

7. Measurement procedure

—Pretreatment—

- 1) Prepare 3.2924g Potassium ferricyanide and 0.2112g Potassium ferrocyanide, and add water to make it 100mL in total as for indicator.

—Measurement—

- 1) Prepare 5.0g of sample in a 100mL beaker, and add 30mL of 0.03mol/L HCl and 30mL of Ethanol.
- 2) Add 3mL of Potassium ferricyan—ferrocyanide test solution.
- 3) Titrate with 0.05mol/L Lead nitrate up to the endpoint to obtain Sodium sulfate.

8. Formula

Sodium sulfate (%) = $(EP1 - BL1) \times TF \times C1 \times K1 / SIZE$

EP1 : Titration volume (mL)
BL1 : Blank level (0.00mL)
TF : Factor of titrant (1.008)
C1 : Concentration conversion coefficient (14.2 mg/mL)
(equivalent to 1mL of 0.05mol/L Lead nitrate)
K1 : Unit conversion coefficient (0.05)
SIZE : Sample size (g)

9. Example of measurement

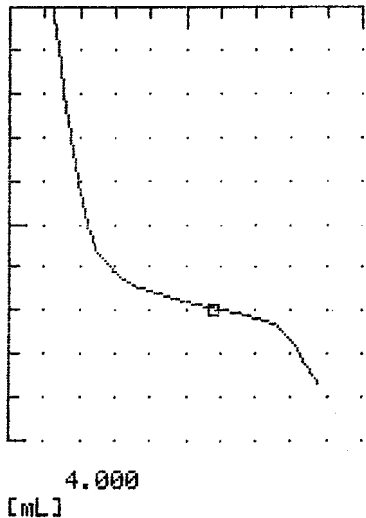
— Ambient condition —

Room temperature : 21 °C	Humidity : 24 %	Weather : Snowy
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- Titration parameter -

[Model Type] AT-510	< Calculation > Calc.Type : Sample Conc.1 : Set CO1=EP1*TF*C1*K1/SIZ E
[TITR. PARA]	
Method No 15 [A.Intermit] Method Type : Titration	Unit : % EP No. : 1 Conc2 : Off Conc3 : Off Conc4 : Off Conc5 : Off Temp.Comp. : Off
< Titration > Form : EP STOP	< Constant > C1(mg/mL) : 14.2 K1 : 0.05 TF : 1.008
APB No. : 1 Unit No. : 1 Detector No. : 1 Unit : mV Max. Volume : 20.0mL Wait Time : 0s Direction : Auto	
Titr. Form : EP STOP	
< Control > End Point No. : 1 End Sense : Auto End Point Area: Off Separation : Off Over Titr.Vol.: 0.0mL Gain : 1 Data Samp.Pot.: 4.0mV Data Samp.Vol.: 0.5mL Stability : 0.5mV/s Delay Time : 1s Limit Time : 30s	

- Titration curve -

*** Result *** Sample No. : 07-01 Date : 2000/02/15 09:24 Method No. : 15 <Auto Intermit> Method Name: Auto Intermit <Constant> C1(mg/mL) : 14.2 K1 : 0.05 Titr.Time : 00:07:47 Size : 5.0030g Conc-1 0.3993% End Point-1 Volume : 2.7913mL Potential : 444.6mV 300 [mV] 550 0.000  4.000 [mL]

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

« Titration parameter »

Form: of formula / APB No.: the burette used in titration / Unit No.: [APB Unit File number](#)/Detector No.: the detector used in titration/ Unit: potential unit in EP detection/ Max Volume: of titration /Wait Time: before titration starts / Direction: of titration

« Control parameter »

End Point No.: number of EPs / End sense: of endpoint / End Point Area: detection area / Separation: of potential Over Titr.Vol.: over-titration/Gain: sensitivity of detection signal/Data samp.Pot.: potential changes of sampling signal Data samp.Vol. titration volume for sampling detection signal / Control Speed: of dosing /Stability: [of EP sense](#) Delay Time: of stability check / Limit Time: of stability check

« Result parameter »

Calc.Type of formula/Conc.1: formula 1/Unit: of result/EP No. of calculation/Temp.Comp.: temperature compensation EP1 titration volume/BL1: blank level 1/ C1(mg/mL): concentration coefficient /TF: factor of titrant/ K1: unit conversion coefficient / Factor: of reagent

—Measurement results—

n	Sample (g)	Titration (mL)	Concentration (%)
1	<u>5.0030</u>	<u>2.7913</u>	<u>0.3993</u>
2	5.0053	2.7930	0.3994

Concentration of Sodium sulfate	
Mean	0.3994 %

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

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

10. Summary

Sodium sulfate is a substance being harmless to human contained in hot spring and believed to smooth the skin. It is used as auxiliary agent in powder shampoo. It helps to absorb dirt and stains with surface active agent.

The sample measurement shows favorable results of a good repeatability with only 1 point deviation between measurements.

Precise and reliable measurement is assured by the automated potentiometry.