

Determination of anionic and cationic surfactants by potentiometric two-phase titration

R. Schlegel and C. Haider



Summary

Compared to the classical Epton titration, potentiometrically indicated two-phase titrations using organic-solvent-resistant Surfactrodes can be easily automated and require no toxic and environmentally hazardous chloroform. Even challenging matrices such as fats and oils in bath oils and hair conditioners or strong oxidizing agents in washing powder and industrial cleaners do not interfere with the titration of the ionic surfactants. Results obtained show excellent agreement to those of the Epton titration. Irrespective of the matrix, relative standard deviations of threefold determinations are all below 2.1%. While the Surfactrode Resistant is mainly used for oil-containing formulations, the Surfactrode Refill is ideal for washing powders and soaps.

Both electrodes excel by their ruggedness and allow the rapid and precise determination of anionic and cationic surfactants.

Introduction

The determination of the surfactant content plays an important role in many analytical applications – from wastewater analysis up to quality control in production processes. A method that is still in widespread use is the so-called Epton titration according to ISO 2271. In this manual titration the anionic surfactant is precipitated by a cationic surfactant in a two-phase mixture of water and chloroform. The ion pair formed must be extracted into the solvent phase by vigorous shaking. The titration endpoint is determined visually using a color indicator. This complicated method suffers from the following disadvantages:

- use of chloroform, a toxic solvent
- long waiting period for phase separation
- inaccurate visual endpoint recognition.

The potentiometric two-phase titration according to DIN EN 14480 and DIN EN 14468 describes a much simpler, faster and also less hazardous determination method. The surfactant solution is pipetted into a titration vessel, to which a two-phase mixture of water and methyl isobutyl ketone/ethanol 1 : 1 and an emulsifier are added. The emulsion formed is titrated under vigorous stirring. The determination of the equivalence point (EP) takes place potentiometrically using surfactant-sensitive electrodes. In comparison with the Epton titration you profit from the following advantages:

- use of unproblematic solvents
- short determination times
- improved precision due to the computerized determination of the EP
- can easily be automated.

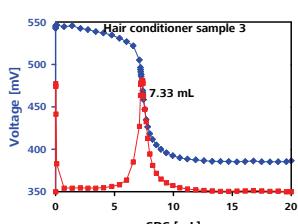
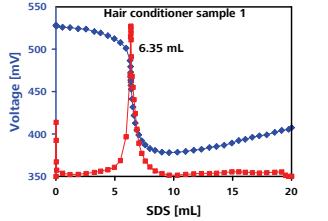
The advantage of this technology is shown in a variety of applications involving the surfactant content determination of cosmetics and household products (hair conditioner, bath oil, washing powder) as well as technical products (industrial cleaners).

Instrumentation

- 809 Titrando
- 804 Ti Stand
- 802 Stirrer
- 800 Dosino



Cationic surfactants in hair conditioner

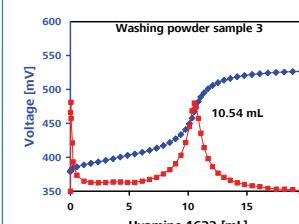
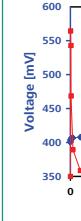


Analyte: Cationic surfactants
Sample: Hair conditioner Bellherbal, Migros, Switzerland
Solvent: Approx. 70 mL of water and 20 mL methyl-isobutyl-ketone/ethanol, (1/1, v/v)

Emulsifier: 0.2 mL TEGO add
Titrant: SDS, 0.005 mol/L Hydrochloric acid, 1.0 mol/L
pH: 2.5
Electrodes: Surfactrode Refill Reference Electrode Ag/AgCl/3 mol/L KCl
U nitrode
Titr. Mode: DET U
*1,3-didecyldimethylimidazolium chloride

Sample	EP [mL]	Surfactant content [mmol/100 g sample]	Mean value	SD [mg]	RSD [%]
1	6.35	7.45	7.40	0.05	0.68
2	4.40	7.38	7.40	0.05	0.68
3	7.33	7.36	7.36		

Anionic surfactants in washing powder

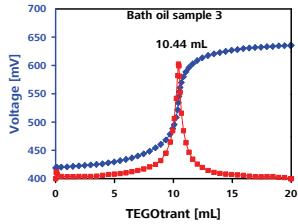
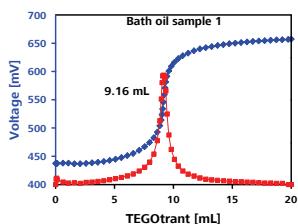


Analyte: Anionic surfactants
Sample: Total Concentré, Migros, Switzerland
Solvent: Approx. 70 mL of water and 20 mL methyl-isobutyl-ketone/ethanol, (1/1, v/v)

Emulsifier: 0.2 mL TEGO add
Titrant: Hyamine 1622*, 0.005 mol/L Hydrochloric acid, 1.0 mol/L
pH: 2
Electrodes: Surfactrode Refill Reference Electrode Ag/AgCl/3 mol/L KCl
U nitrode
Titr. Mode: DET U
*1,3-didecyldimethylimidazolium chloride

Sample	EP [mL]	Surfactant content [mmol/100 g sample]	Mean value	SD [mg]	RSD [%]
1	8.91	29.92	29.92	0.49	1.64
2	9.10	30.35	30.35	0.49	1.64
3	10.54	29.38	29.38		

Anionic surfactants in bath oil

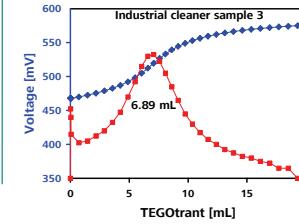
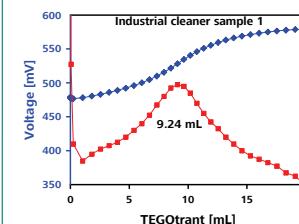


Analyte: Anionic surfactants
Sample: Bath oil (free sample) from Acapulco, Mexico
Solvent: Approx. 70 mL of water and 20 mL methyl-isobutyl-ketone/ethanol, (1/1, v/v)

Emulsifier: 0.2 mL TEGO add
Titrant: TEGOtrant A 100°, 0.005 mol/L Sulfuric acid, 0.5 mol/L
pH: 3
Electrodes: Surfactrode Resistant Reference Electrode Ag/AgCl/3 mol/L KCl
U nitrode
Titr. Mode: DET U
*1,3-didecyldimethylimidazolium chloride

Sample	EP [mL]	Surfactant content [mmol/100 g sample]	Mean value	SD [mg]	RSD [%]
1	9.16	52.32	52.32	0.20	0.38
2	8.61	52.68	52.54	0.20	0.38
3	10.44	52.63	52.63		

Anionic surfactants in industrial cleaner



Analyte: Anionic surfactants
Sample: Multi-Clean, Eurotech, Austria
Solvent: Approx. 70 mL of water and 20 mL methyl-isobutyl-ketone/ethanol, (1/1, v/v)

Emulsifier: 0.2 mL TEGO add
Titrant: TEGOtrant A 100°, 0.005 mol/L Sulfuric acid, 0.5 mol/L
pH: 3
Electrodes: Surfactrode Resistant Reference Electrode Ag/AgCl/3 mol/L KCl
U nitrode
Titr. Mode: DET U
*1,3-didecyldimethylimidazolium chloride

Sample	EP [mL]	Surfactant content [mmol/100 g sample]	Mean value	SD [mg]	RSD [%]
1	9.24	10.98	10.98	0.23	2.09
2	8.72	10.75	10.75	0.23	2.09
3	6.89	11.21	11.21		