Application Bulletin 268/3 e

Potentiometric titration of surfactants and pharmaceutical compounds – an overview

Branch

General analytical chemistry, private laboratories; organic chemistry, chemistry; pharmaceutical industry; metals, electroplating; detergents, surfactants, cosmetics

Keywords

Two-phase titration; anionic surfactants; cationic surfactants; Epton titration; Surfactrode Resistant; Surfactrode Refill; Ionic surfactant electrode; Cationic surfactant electrode; NIO Surfactant electrode; Ionophor ; 6.0507.130; nonionic surfactants; TEGO; SDS; STPB; 6.0507.140; 6.0507.150; 6.0507.120; 6.0507.010; branch 1; branch 3; branch 4; branch 10; branch12

Summary

This bulletin provides an overview of the numerous surfactants and pharmaceutical compounds that can be determined by potentiometric titration. Metrohm offers five different surfactant electrodes for the indication of the titration endpoint: the lonic surfactant electrode, the Cationic surfactant electrode, the Surfactrode Resistant, the Surfactrode Refill and the NIO Surfactant electrode. Preparation of the titrants used and the determination of their titer are described in detail.

In addition, the bulletin also offers a tabulated listing of over 170 proven applications from the field of surfactant and pharmaceutical compound analysis. This guide will help you reach your goal: At a glance you will discover from the table which surfactant electrode and which titrant are suitable to analyze your product.

Instruments

- Titrator with DET, MET mode
- 20 mL buret
- Rod Stirrer

Electrode

The following five surfactant electrodes are used for the potentiometric titrations:

Titrations in aqueous solution

Ionic Surfactant electrode	6.0507.120
Cationic Surfactant electrode	6.0504.150
NIO electrode	6.0507.010
Two-phase titrations	
Surfactrode Resistant	6.0507.130
Surfactrode Refill	6.0507.140
Reference electrode	
Ag/AgCI Reference Electrode (KCI)	6.0726.107

When using sodium tetraphenylborate (STBP) as titrant (titration of nonionic surfactants and pharmaceutical compounds with the NIO Surfactant electrode), the bridge electrolyte has to be replaced with c(NaCl) = 3 mol/L as the STPB reacts with the potassium.

Which surfactant electrode for which product?

Sample with a simple matrix or a raw substance can be analyzed in aqueous solution. Three electrodes can be used depending on the type of surfactant.

- The lonic Surfactant electrode can be used to analyze anionic surfactants.
- The Cationic Surfactant electrode can be used to analyze cationic surfactants.
- The NIO electrode can be used to analyze nonionic surfactants based on polyoxyethylene adducts

Samples with a more complex matrix are best analyzed with the two-phase titration. There are two electrodes available which are resistant to organic solvents, the Surfactrode Resistant and the Surfactrode Refill.

The Surfactrode Resistant is maintenance-free and is best suitable for routine analysis. It is the only electrode which can be used for chlorinated solvents.



Application Bulletin 268/3

Potentiometric titration of surfactants and pharmaceutical compounds – an overview

The Surfactrode Refill. is optimal for samples with a high salt content and relative low surfactant content (e.g. electroplating baths) or measurements at pH values >10 (e.g. soaps). It should not be used with chloroform or other chlorinated solvents as the electrode's paste-like sensor material is leached out.

For all surfactants the alkyl chain must contain at least 12 carbon atoms to be measureable and for the NIO surfactants it needs at least 7 polyoxyethylene (POE) groups to react stoichiometric with BaCl₂.

Reagents

To determine anionic surfactants

- TEGO®trant A100, e.g Metrohm no. 6.2317.010 Sodium dodecyl sulfate (sodium lauryl sulfate)
- Buffer solution pH = 3.0
- Methanol, p.a.

To determine cationic surfactants

Sodium dodecyl sulfate (sodium lauryl sulfate),
 Formaldehyde solution, at least w(HCHO) = 35%

To determine nonionic surfactants

- Sodium tetraphenylborate,
- Polyvinyl alcohol protective colloid
- Papaverine hydrochloride
- Sodium hydroxide solution, c(NaOH) = 1.0 mol/L
- Boric acid, H₃BO₃, p.a.
- Hydrochloric acid, w(HCl) = 36 38%, p.a.

Solutions

Below a list can be found of recommended titrants and their concentrations, for more details see: *Preparation of titrants and titer determination*.

To determine anionic surfactants

Titrant A	c(TEGO®trant A100) = 0.005 mol/L
Titrant B	c(TEGO®trant A100) = 0.02 mol/L
Titrant C	c(TEGO®trant A100) = 0.05 mol/L

To determine cationic surfactants

Titrant D	c(SDS) = 0.005 mol/L
Titrant E	c(SDS) = 0.02 mol/L

To determine nonionic surfactants

Titrant F	c(STPB) = 0.01 mol/L
Titrant G	c(STPB) = 0.10 mol/L
Titrant H	c(STPB) = 0.002 mol/L

Precision and trueness of the results

There are no primary/secondary standards available to standardize the titrants. The cationic titrant is standardized against the anionic titrant and vice versa. The raw substances to make the titrants are also not 100% pure. Therefore the purity of substance written in the analysis certificate has to be taken into account for calculation.

Also the titration of surfactants is not linear. The linearity differs from sample to sample. It is therefore necessary to titrate different sample sizes in order to find the linear range.

Weighing, dilution and pipetting errors are common errors, which always occur but in the case of surfactants they are even more pronounced, because surfactants are surface active. They stick to all surfaces they contact, i.e. weighing boat, walls of the volumetric flask and pipettes. So with every preparation step, a small fraction of the sample is lost.

An additional problem of surfactant titrations is the formation of foam. This should be prevented as the concentration of surfactants in foam is much higher as in the solution. The presence of foam can therefore lead to errors in the results.

A typical relative standard deviation for the standardization is higher than 2% if all the points mentioned above are considered. It is therefore easily possible to obtain a relative standard deviation of 5% and higher for a sample titration. In statistics the relative standard deviation might be lower but it does not contain the errors described above.

Potentiometric titration of surfactants and pharmaceutical compounds – an overview

Preparation of titrants and titer determination

1. Titrants based on TEGO $\ensuremath{\mathbb{R}}$ trant A100 for the determination of anionic surfactants (A, B, C)

Owing to the high surface affinity of cationic titrants, these must be added to the buret one day before use to ensure wetting of all glass parts and tubing that come into contact with the standard solution. Only if all surfaces are saturated with the Cationic surfactant the titer of the titrant is stable (at least six months).

Preparation of the titrants based on TEGO®trant A100

Required weights of TEGO®trant A100

Titrant	c(TEGO®trant	m(TEGO®trant A100)
	A100) / mol/L	/ g
А	0.005	approx. 2.12
В	0.02	approx. 8.50
С	0.05	approx. 21.25

The required quantity of TEGO®trant A100 is weighed into a glass beaker with an accuracy of 0.1 mg and dissolved in approx. 150 mL water. This solution is transferred quantitatively with dist. water to a 1 L volumetric flask and fille up to the mark. Detailed information can be found on the leaflet which is delivered with the Tego®trant.

Preparation of the comparison standard solutions from sodium dodecyl sulfate (SDS)

The raw substances contain some impurities e.g water. Therefore it is recommended to take the purity of the raw material into the calculation of the weigh in.

Required weights of sodium dodecyl sulfate
--

Titrant	c(TEGO®trant A100) / mol L ⁻¹	c(SDS) / mol L ⁻¹	m(SDS) / g
А	0.005	0.005	1.44 1.45
В	0.02	0.02	5.75 5.80
С	0.05	0.05	14.38 14.50

The required quantity of sodium dodecyl sulfate is weighed into a glass beaker with an accuracy of 0.1 mg and dissolved in approx. 200 mL water. This solution is transferred quantitatively with water to a 1 L volumetric flask, the flask is filled to the mark and its contents are carefully mixed.

The exact sample weight must be noted as it is needed for the subsequent titer calculation.

Titer determination of the titrants (titration in aqueous solution)

10.0 mL of the corresponding sodium dodecyl sulfate standard solution is pipetted into a glass beaker. Followed by adding of 5 mL methanol, 75 mL water and 10 mL buffer solution pH = 3.0. The sample solution is efficiently stirred and then titrated using the corresponding TEGO®trant A100 solution as titrant with the following instrument settings:

Pause	30 s
Signal drift	50 mV/min
Measuring point density	4
Min. increment	10.0 µL
Stop volume	20 mL
EP recognition	all

For the potentiometric two-phase titration the titer determination is carried out in an analogous way, see Application Bulletin No. 269.

The results may be used only if just one equivalence point is recognized. If this is not the case, additional titrations must be performed.

Calculation of the titer

A threefold determination is always performed. The resulting mean value is calculated to four decimal places.

$$f = \frac{m_{s} \times V_{S} \times C_{S}}{V_{EP1} \times M_{S} \times 100 \times c_{TEGOtrant}}$$

- f: Titer of the titrant
- V_{EP1}: Titrant consumption in mL
- m_S: Sample weight of SDS standard in g
- Vs: Added volume of SDS solution in mL, here 10.0
- $\label{eq:CS} C_S: \qquad \mbox{Active substance content of the SDS used in \%,} \\ & \mbox{here 99.2} \end{cases}$
- M_S: Molecular weight of reference substance; here 288.4 g/mol
- 100: Conversion factor due to %

2. Titrants based on sodium dodecyl sulfate (sodium lauryl sulfate) for the determination of cationic surfactants (D, E)

A titer determination in the usual manner cannot be performed for anionic titrants as there are no suitable

primary standards. Cationic surfactants are normally quaternary ammonium compounds that usually cannot be prepared with the purity required for a primary standard. The degree of quaternization of these compounds would have to be 100%, but this is never the case. In addition, most of these compounds are highly hygroscopic. As a result, owing to water uptake, the active substance content changes each time the container is opened.

As a titer determination in the normal sense is not possible, the standard solutions are prepared by very exact weighing in of sodium dodecyl sulfate.

Determination of the required weights of sodium dodecyl sulfate

The Na_2SO_4 can be determined by photometric titration and the H_2O by Karl Fischer titration. Dodecyl alcohol can be determined by gas chromatography.

Weight per 1 L solution in g =
$$\frac{M_t \times c_{SDS} \times 100}{C_S}$$

- Mt: Molecular weight of the titrant in g/mol; here 288.4 g/mol
- C_S: Active substance content of the SDS used in %; here 99.2 %
- 100: conversion factor due to %

Titrant	c(SDS) / mol L ⁻¹	m(SDS) / g
D	0.005	1.44 1.45
E	0.02	5.75 5.80

Preparation of the titrants based on sodium dodecyl sulfate (D, E)

The required quantity of sodium dodecyl sulfate (1.4 g or 5.8 g) is weighed exactly into a glass beaker and dissolved in approx. 250 mL dist. water. This solution is rinsed quantitatively into a 1 L volumetric flask with dist. water, 10 mL w(HCHO) = 35% is added and the flask is filled to the mark with dist. water. The addition of formaldehyde prevents bacterial decomposition of the titrant without having an adverse effect on the surfactant titration. The disinfecting action of the quantity specified is sufficient to keep the titer stable for at least three months. To guarantee thorough mixing, a magnetic stirring bar is added to the flask and the solution is stirred on a magnetic stirrer ensuring that foam formation is kept to a minimum. The titrant can then be transferred to the buret.

3. Titrants based on sodium tetraphenylborate (STPB) for the determination of nonionic surfactants and pharmaceutical compounds (F, G, H)

The Titrants contain additives that significantly reduce deposition of precipitates formed during the titration on the electrode and hence allow titrations free from interferences. If titration is performed with sodium tetraphenylborate solutions prepared in a different manner, the electrodes must be cleaned after every titration. With the solution described, the electrode must be cleaned after around 10 titrations. The precipitates adhere on the electrode and reduced the response time. A mechanical cleaning with a tissue is necessary to remove the white precipitate on the electrode.

Preparation of the titrants based on sodium tetraphenylborate

Required weights of sodium tetraphenylborate

Titrant	c(STPB) / mol L ^{−1}	m(STPB) / g
F	0.01	3.4223
G	0.1	34.223
Н	0.002	0.6845

Buffer pH 10: 1.24 g H_3BO_3 is dissolved in dist. water, 10 mL c(NaOH) = 1 mol/L is added to the solution and filled up to 100 mL with dist. water.

Approx. 200 mL water is heated in a glass beaker. 10 g polyvinyl alcohol (PVA) is now added slowly while stirring and the solution is stirred until it is almost clear. The solution is allowed to cool down to room temperature, rinsed into a 1 L volumetric flask with water and 10 mL Buffer pH 10 is added.

The required quantity of sodium tetraphenylborate is weighed into a second glass beaker with an accuracy of 0.1 mg and dissolved in water. This solution is rinsed into the volumetric flask (which already contains PVA and Buffer) with water, the flask is filled to the 1 L mark and the solution is thoroughly mixed. The solution is then transferred to the buret.

Comment

 The solution of the protective colloid (PVA) must be cooled down to room temperature before addition of the sodium tetraphenylborate. If not, the titrant cannot be used.

Application Bulletin 268/3

Potentiometric titration of surfactants and pharmaceutical compounds – an overview

Titer determination of the titrants

A. Determination of nonionic surfactants based on polyoxyethylene adducts

As the precipitation with STPB does not follow stoichiometric rules and the nonionic surfactants are never uniform substances, work is performed with a so-called calibration factor f (mg NIO surfactant / mL STPB). This is described in detail in Application Bulletin No. 230.

B. Determination of pharmaceutical compounds

0.04 g (if titrant F, c(STPB) = 0.01 mol/L is used) or 0.4 g (for titrant G, c(STPB) = 0.10 mol/L) papaverine hydrochloride is weighed into a glass beaker to the nearest 0.1 mg. The substance is dissolved in 100 mL water, 3 - 4 drops of conc. HCl are added and titration is performed with efficient stirring using the corresponding sodium tetraphenylborate solution and, e.g., the following instrument settings:

Pause	60 s
Signal drift	10 mV/min
Measuring point density	4
Min. increment	150 µL
Stop volume	20 mL
EP recognition	greatest

Calculation of the titer

A threefold determination is always performed. The resulting mean value is calculated to four decimal places.

 $f = \frac{m_s \times 1000}{V_{EP1} \times M_S \times c_{STPB}}$

f: Titer of the titrant

- V_{EP1}: Titrant consumption in mL
- ms: Weight of papaverine hydrochloride in g
- 1000: Conversion factor mL to L
- M_S: Molecular weight of papaverine hydrochloride; here 375.9 g/mol
- c_{STPB} : Theoretical concentration of the titrant in mol/L; here 0.01 or 0.1

Comment

• Papaverine hydrochloride is toxic. The appropriate precautionary measures must therefore be taken when this substance is used.

Which surfactant electrode and which titrant for which product?

Surfactant electrodes from Metrohm

IS	6.0507.120 Ionic surfactant electrode or
	6.0504.150 Cationic surfactant electrode

- SR 6.0507.130 Surfactrode Resistant and6.0507.140 Surfactrode Refill
- NIO 6.0507.010 NIO Surfactant electrode

Incompatibilities of the surfactant electrodes with certain sample matrices

1. Ionic surfactant electrode, Cationic surfactant electrode and NIO Surfactant electrode

They must not be used with organic solvents, especially chlorinated hydrocarbons (e.g. chloroform), hydrocarbons (e.g. benzene, toluene) and oils (e.g. cooling lubricants, oil containing formulations).

2. Surfactrode Resistant

This electrode should not be used for samples with a high salt content and relatively low surfactant content (e.g. electroplating baths); pH values >10.

3. Surfactrode Refill

Should not be used with chloroform in two-phase titrations (the electrode's paste-like sensor material is leached out); therefore preferably use methyl isobutyl ketone (MIBK) as solvent.

Titrants used

- A c(TEGO®trant A100) = 0.005 mol/L
- **B** c(TEGO®trant A100) = 0.02 mol/L
- C c(TEGO®trant A100) = 0.05 mol/L
- **D** c(SDS) = 0.005 mol/L
- **E** c(SDS) = 0.02 mol/L
- **F** c(STPB) = 0.01 mol/L
- **G** c(STPB) = 0.10 mol/L
- H c(STPB) = 0.002 mol/L

Application Bulletin 268/3

Potentiometric titration of surfactants and pharmaceutical compounds – an overview

Metrohm Application Bulletins (AB)

- 230 Titrimetric/potentiometric determination of nonionic surfactants based on polyoxyethylene adducts using the NIO electrode
- **233** Titrimetric/potentiometric determination of anionic and cationic surfactants
- 263 Titrimetric determination of pharmaceutical compounds with the NIO electrode
- **269** Titrimetric/potentiometric determination of ionic surfactants by two-phase titration using the Metrosensor Surfactrodes
- 275 Potentiometric two-phase titration of anionic surfactants in washing powders and liquid washing agents

Note

Meaning of the abbreviations used in the table:

- cannot be determined
- d_r determination restricted
- IS Ionic Surfactant electrode or Cationic Surfactant electrode
- SR Surfactrode Resistant and Surfactrode Refill
- NIO NIO Surfactant electrode
- AB Application Bulletin
- A c(TEGO®trant A100) = 0.005 mol/L
- B c(TEGO®trant A100) = 0.02 mol/L
- c c(TEGO®trant A100) = 0.05 mol/L
- **D** c(SDS) = 0.005 mol/L
- E c(SDS) = 0.02 mol/L
- **F** c(STPB) = 0.01 mol/L
- G c(STPB) = 0.10 mol/L
- H c(STPB) = 0.002 mol/L

Author

Competence Center Titration Metrohm International Headquarters

Product	IS	SR	NIO	AB
α-Olefin sulfonates		А		269
Alkanesulfonates, secondary (SAS)		A		269
Alkylbenzenesulfonates, linear (LAS)	A			233
Alkylphenol POE adducts			F	230
Alkyltrimethylammonium halides	D			233
All-purpose cleaners	А			233
All-purpose cleaners with high NIO content		В		269
Ambroxol hydrochloride			G	263
Amine fluorides	D			233
Anionic dyestuffs	А			233
Anionic pharmaceutical compounds	В			233
Baby care products, bathing products (anionic)		В		269
Balsam cure for wet hair (cationic)		D		269
Balsam formulations (anionic)		А		269
Bath cleaners	А			233
Benzalkonium halides	D			233
Bis(2-ethylhexyl) sulfosuccinate (DOS)	A			233
Bis(2-ethylhexyl) sulfosuccinic acid ester (DOS)	A			233
Bromhexidine hydrochloride			F	263
Car shampoos (anionic)		В		269
Caries prophylaxis gel	D			233
Castor oil POE adducts			F	230
Cationic dyestuffs			F	263
Cationic pharmaceutical compounds			F	263
Cetylpyridinium halides	D			233
Chlorhexidine digluconate			F	263
Chlorhexidine dihydrochloride			F	263
Chlorphenoxamine hydrochloride			F	263
Cleaning agents containing pine oil (anionics)		A		269
Cleaning agents containing pine oil (cationic)		D		269
Clobutinol			F	263
Clotrimazol			F	263
Cocoylisethionates	С			233
Codeine phosphate			G	263

Application Bulletin 268/3

Potentiometric titration of surfactants and pharmaceutical compounds – an overview

Conditioners for hair (cationic)DU269Cooling lubricants (cationic)DC269Cumol sulfonatesDialkyldimethylammonium halidesDU233Dihydrocodeine thiocyanateMG263Dioctylsulfosuccinate (DOS)A-233Diotylsulfosuccinate acid esterAM269Disinfectant cleaners (anionic)MC269Disinfectant cleaners (ationic)MC269Disinfectant cleaners (ationic)M233Distearyldimethylammonium choride (DSDMAC)DC233Electroplating baths (anionic)AF233Electroplating baths (cationic)DF233Electroplating baths (PEG)MF233Electroplating baths (PEG)CF233Fabric softenersDCZFat-containing formulations (anionic)AC233Fat-containing formulations (ationic)AC233Faty acid salts 2C12BDC233Fatty acid salts C3CCC233Fatty alcohol ether-2.5 sulfates (FAES)AC233Fatty alcohol ether-3 sulfates (FAES)CC233Fatty alcohol ether-4 sulfosuccinatesAC233Fatty alcohol ether-4 sulfatesAC233Fatty alcohol ether-4 sulfatesACC	Product	IS	SR	NIO	AB
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Cumous ulformation (characterity)Dialkyldimethylammonium halidesD-233Dihydrocodeine thiocyanateA233Dioctylsulfosuccinate (DOS)A-233Dioctylsulfosuccinic acid ester (DOS)AA269Disinfectant cleaners (anionic)MD269Disinfectant cleaners (ationic)MD233Distearyldimethylammonium (DSDMAC)D-233Electroplating baths (anionic)A-233Electroplating baths (cationic)DI233Electroplating baths (NIOS)-F230Electroplating baths (PEG)II233Ethacridine lactateDI233Fabric softenersDI233Fat-containing formulations (anionic)P-233Faty acid salts \geq_{12} BI233Faty acid salts C_{12} BI233Faty acid salts C_{12} BI233Faty acid salts C_{12} BIIFaty alcohol ether-2.5 sulfates (FAES)IIIFaty alcohol ether-3 sulfates (FAES)IIIFaty alcohol ether-3IIIIFaty alcohol ether-4 sulfosuccinatesIIIIFaty alcohol ether-4 sulfosuccinatesIIIIFaty alcohol ether-4 sulfosuccinatesIIII <td>Cooling lubricants (anionic)</td> <td></td> <td>А</td> <td></td> <td>269</td>	Cooling lubricants (anionic)		А		269
Dialkyldimethylammonium halidesD	Cooling lubricants (cationic)		D		269
Dihydrocodeine thiocyanateIG263Dioctylsulfosuccinic acid ester (DOS)AI233Dioctylsulfosuccinic acid ester (DOS)AI269Disinfectant cleaners (anionic)DI269Disinfectant cleaners (cationic)DI233Distearyldimethylammonium chloride (DSDMAC)DI233Electroplating baths (anionic)AI233Electroplating baths (cationic)DI233Electroplating baths (NIOS)F230Electroplating baths (PEG)II233Ethacridine lactateDI233Eye drops (quats)DI233Fat-containing formulations (anionic)AI233Fat-containing formulations (cationic)F230Fatty acid Aslts 2C12BI233Fatty acid salts C10-F230Fatty acid salts C3Fatty alcohol ether-2.5 sulfates (FAES)AIIFatty alcohol ether-3AIIIFatty alcohol ether-4 sulfosuccinatesAIIFatty alcohol ether-4 sulfosuccinatesAIIFatty alcohol ether-4 sulfosuccinatesAIIFatty alcohol ether-4 sulfosuccinatesAIIFatty alcohol ether-4 sulfosuccinatesAIIFatty alcohol ether-4 sulfosuccinatesAII	Cumol sulfonates	_	-	-	_
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Dioctylsulfosuccinic acid ester (DOS)AImage: Section 100 (Disinfectant cleaners (anionic))A269Disinfectant cleaners (cationic)DC233Distearyldimethylammonium chloride (DSDMAC)DImage: Section 233Electroplating baths (anionic)AImage: Section 233Electroplating baths (cationic)DImage: Section 233Electroplating baths (NIOS)F233Electroplating baths (PEG)Image: Section 233Electroplating baths (PEG)F233Fabric softenersDImage: Section 233Fathacridine lactateImage: Section 233Fat-containing formulationsF249(anionic)F230Fatty acid POE adductsF233Fatty acid salts C10Fatty alcohol ether-2 sulfatesAImage: Section 233Fatty alcohol ether-2 sulfatesAImage: Section 233Fatty alcohol ether-3 sulfatesAImage: Section 233Fatty alcohol ether-3AImage: Section 233Fatty alcohol ether-4AImage: Section 233Fatty alcohol ether-4AImage: Section 233 <tr< td=""><td>Dihydrocodeine thiocyanate</td><td></td><td></td><td>G</td><td>263</td></tr<>	Dihydrocodeine thiocyanate			G	263
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Disinfectant cleaners (cationic)DC269Disinfectants (quats)DC233Distearyldimethylammonium chloride (DSDMAC)DL233Electroplating baths (anionic)AC233Electroplating baths (cationic)DL233Electroplating baths (cationic)DL233Electroplating baths (NIOS)F230Electroplating baths (PEG)LF233Ethacridine lactateDL233Ethacridine lactateDL233Fat-containing formulations (anionic)A269Fat-containing formulations (cationic)F230Fatty acid salts $\geq C_{12}$ BI233Fatty acid salts C_{10} Fatty alcohol ether-2 sulfates (FAES)A233Fatty alcohol ether-3 sulfates sulfosuccinatesA233Fatty alcohol ether-4 sulfosuccinatesA233Fatty alcohol ether-4 sulfosuccinatesA233Fatty alcohol POE adductsF233Fatty alcohol POE adductsF233	•	A			233
Disinfectants (quats)DI233Distearyldimethylammonium chloride (DSDMAC)D233Electroplating baths (anionic)AI233Electroplating baths (cationic)DI233Electroplating baths (cationic)DI233Electroplating baths (NIOs)F230Electroplating baths (PEG)IF230Ester quatsDI233Ethacridine lactateII233Fabric softenersDI233Fat-containing formulations (cationic)A269Fatty acid POE adductsF230Fatty acid salts $\geq C_{12}$ BIFatty acid salts C_{10} Fatty acid salts C_{10} Fatty acid salts C_{12} BIFatty acid salts C_{10} Fatty acid salts C_{10} Fatty acid salts C_{10} Fatty alcohol ether-2 sulfates (FAES)A233Fatty alcohol ether-3 sulfates (FAES)AIFatty alcohol ether-3AI233Fatty alcohol ether-4 sulfosuccinatesAI233Fatty alcohol ether-4 sulfosuccinatesAI233Fatty alcohol POE adductsII233Fatty alcohol POE adductsII233Fatty alcohol ether-4 sulfosuccinatesAIIFatty alcohol ether-4 sulfosuccinatesA<	Disinfectant cleaners (anionic)		А		269
Distearyldimethylammonium chloride (DSDMAC)D233Electroplating baths (anionic)A	Disinfectant cleaners (cationic)		D		269
chloride (DSDMAC)IIIIIElectroplating baths (anionic)AI233Electroplating baths (NIOs)DI233Electroplating baths (PEG)IF230Ester quatsDI233Ethacridine lactateII233Eye drops (quats)DI233Fat-containing formulations (anionic)AZ69Fatty acid POE adductsF230Fatty acid salts 2C12BI233Fatty acid salts C8-F230Fatty alcohol ether-2 sulfates (FAES)AZ33Fatty alcohol ether-3AI233Fatty alcohol ether-4 sulfosuccinatesAZ33Fatty alcohol ether-4 sulfosuccinatesAZ33	Disinfectants (quats)	D			233
Electroplating baths (cationic)DII233Electroplating baths (NIOs)F230Electroplating baths (PEG)IF230Ester quatsDI233Ethacridine lactateDI233Eye drops (quats)DI233Fabric softenersDI233Fat-containing formulations (anionic)A269Fatty acid POE adductsF230Fatty acid salts \geq C12BDIFatty acid salts C3Fatty acid salts C4Fatty acid salts C4Fatty acid salts C5A233Fatty alcohol ether-2.5 sulfates (FAES)AIFatty alcohol ether-3 sulfates sulfosuccinatesAIFatty alcohol ether-3AIIFatty alcohol ether-4 sulfosuccinatesAIFatty alcohol ether-4 sulfosuccinatesAIFatty alcohol ether-4 sulfosuccinatesAIFatty alcohol ether-4 sulfosuccinatesAIFatty alcohol ether-4 sulfosuccinatesAIFatty alcohol ether-4 sulfosuccinatesAIFatty alcohol POE adductsIIFatty alcohol POE adducts <t< td=""><td>, ,</td><td>D</td><td></td><td></td><td>233</td></t<>	, ,	D			233
Electroplating baths (NIOs)F230Electroplating baths (PEG)IF230Ester quatsD233Ethacridine lactateIF263Eye drops (quats)DI233Fabric softenersDI233Fat-containing formulations (anionic)A269Fatty acid POE adductsF230Fatty acid salts $\geq C_{12}$ BIFatty acid salts C_{10} Fatty acid salts C_{8} Fatty alcohol ether-2 sulfates (FAES)A233Fatty alcohol ether-3 sulfates (FAES)A233Fatty alcohol ether-3 sulfates sulfosuccinatesA233Fatty alcohol ether-4 sulfosuccinatesA233Fatty alcohol ether-4 sulfosuccinatesA233Fatty alcohol ether-3A233Fatty alcohol ether-3A233Fatty alcohol ether-3A233Fatty alcohol ether-4 sulfosuccinatesA233Fatty alcohol ether-3AIFatty alcohol ether-4 sulfosuccinatesAIFatty alco	Electroplating baths (anionic)	А			233
Electroplating baths (PEG)IIF230Ester quatsDI233Ethacridine lactateDI233Eye drops (quats)DI233Fabric softenersDDI233Fat-containing formulations (anionic)A269Fat-containing formulations (cationic)DI230Fatty acid POE adductsIF230Fatty acid salts ≥C12BI233Fatty acid salts C10Fatty acid salts C8-I-Fatty alcohol ether-2 sulfates (FAES)A233Fatty alcohol ether-3 sulfates (FAES)A233Fatty alcohol ether-3AI233Fatty alcohol ether-4 sulfosuccinatesA233Fatty alcohol ether-3AI233Fatty alcohol ether-4 sulfosuccinatesAI233Fatty alcohol ether-4 sulfosuccinatesAI233Fatty alcohol ether-4 sulfosuccinatesAI233Fatty alcohol POE adductsIIIIFatty alcohol POE adductsIII <td< td=""><td>Electroplating baths (cationic)</td><td>D</td><td></td><td></td><td>233</td></td<>	Electroplating baths (cationic)	D			233
Ester quatsD233Ethacridine lactateIF263Eye drops (quats)D233Fabric softenersDI233Fat-containing formulations (anionic)A269Fat-containing formulations (cationic)P269Fatty acid POE adductsF230Fatty acid salts $\geq C_{12}$ BI233Fatty acid salts $\geq C_{12}$ BI233Fatty acid salts C_{10} Fatty acid salts C_8 -I-Fatty alcohol ether-2 sulfates (FAES)A233Fatty alcohol ether-3 sulfates (FAES)A233Fatty alcohol ether-3 sulfates (FAES)A233Fatty alcohol ether-4 sulfosuccinatesA233Fatty alcohol ether-4 sulfosuccinatesA233Fatty alcohol ether-4 sulfosuccinatesA233Fatty alcohol ether-4 sulfosuccinatesA233Fatty alcohol ether-4 sulfosuccinatesA233Fatty alcohol ether-4 sulfosuccinatesAEFatty alcohol ether-4 sulfosuccinatesAEFatty alcohol ether-4 sulfosuccinatesAEFatty alcohol POE adductsIFFatty alcohol POE adductsIFatty alcoh	Electroplating baths (NIOs)			F	230
Ethacridine lactateIIF263Eye drops (quats)DII233Fabric softenersDII233Fat-containing formulations (anionic)AZ69Fat-containing formulations (cationic)DIIFat-containing formulations (cationic)BDIIFatty acid POE adductsF230Fatty acid salts $\geq C_{12}$ BI233Fatty acid salts C_{10} 233Fatty acid salts C_{10} Fatty acid salts C_8 -I-Fatty acid salts C_8 Fatty alcohol ether-2 sulfates (FAES)AIII233Fatty alcohol ether-3 sulfates (FAES)AIIII233Fatty alcohol ether-3 sulfosuccinatesAIIIIIIIIIIFatty alcohol ether-4 sulfosuccinatesAIIIIIIIIIIIIIIIIIIIIIIIIIIIIIIIII	Electroplating baths (PEG)			F	230
Eye drops (quats)D233Fabric softenersDI233Fat-containing formulations (anionic)A269Fat-containing formulations (cationic)DI269Fat-containing formulations (cationic)DI269Fatty acid POE adductsF230Fatty acid salts \geq C12BI233Fatty acid salts C10Fatty acid salts C8Fatty alcohol ether-2 sulfates (FAES)A233Fatty alcohol ether-3 sulfates (FAES)A233Fatty alcohol ether-3 sulfates sulfosuccinatesA233Fatty alcohol ether-4 sulfosuccinatesA233Fatty alcohol ether-4 sulfosuccinatesAIFatty alcohol ether-4 sulfosuccinatesAIFatty alcohol ether-4 sulfosuccinatesAIFatty alcohol ether-4 sulfosuccinatesIIFatty alcohol ether-4AIFatty alcohol ether-4 sulfosuccinatesIFatty alcohol ether-4<	Ester quats	D			233
Fabric softenersDI233Fat-containing formulations (anionic)A269Fat-containing formulations (cationic)DD269Fat-containing formulations (cationic)DF230Fatty acid POE adductsF230Fatty acid salts \geq C12BI233Fatty acid salts C10Fatty acid salts C8Fatty alcohol ether-2 sulfates (FAES)AI233Fatty alcohol ether-3 sulfates (FAES)AI233Fatty alcohol ether-3 sulfates sulfosuccinatesAI233Fatty alcohol ether-4 sulfosuccinatesAI233Fatty alcohol ether-4 sulfosuccinatesAIIFatty alcohol ether-4 sulfosuccinatesF233Fatty alcohol POE adductsIII	Ethacridine lactate			F	263
Fat-containing formulations (anionic)A269Fat-containing formulations (cationic)D269Fatty acid POE adductsF230Fatty acid salts $\geq C_{12}$ BC233Fatty acid salts $\geq C_{12}$ BC233Fatty acid salts C_{10} Fatty acid salts C_8 Fatty acid salts C_8 Fatty alcohol ether-2 sulfates (FAES)ASSFatty alcohol ether-3 sulfates (FAES)ASSFatty alcohol ether-3 sulfates sulfosuccinatesASSFatty alcohol ether-4 sulfosuccinatesASSFatty alcohol ether-4 sulfosuccinatesFSSFatty alcohol POE adductsIIISFatty alcohol POE adductsIIISFatty alcohol POE adductsIIISFatty alcohol POE adductsIIISFatty alcohol POE adducts <td>Eye drops (quats)</td> <td>D</td> <td></td> <td></td> <td>233</td>	Eye drops (quats)	D			233
(anionic)Fat-containing formulations (cationic)D269Fatty acid POE adductsF230Fatty acid salts \geq C12BI233Fatty acid salts C_{10} Fatty acid salts C3Fatty acid salts C8Fatty alcohol ether-2 sulfates (FAES)AI233Fatty alcohol ether-3 sulfates (FAES)AI233Fatty alcohol ether-3 sulfates (FAES)AI233Fatty alcohol ether-3 sulfates sulfosuccinatesAI233Fatty alcohol ether-4 sulfasesAI233Fatty alcohol ether-4 sulfosuccinatesAI233Fatty alcohol ether-4 sulfosuccinatesAIIFatty alcohol ether-4 sulfosuccinatesIIIFatty alcohol POE adductsIIIIFatty alcohol POE adductsIIIIFatty alcohol POE adductsIIIIFatty alcohol POE adductsIII </td <td>Fabric softeners</td> <td>D</td> <td></td> <td></td> <td>233</td>	Fabric softeners	D			233
(cationic)Image: Constraint of the sector of t	-		A		269
Fatty acid salts $\geq C_{12}$ BI233Fatty acid salts C_{10} Fatty acid salts C_8 Fatty alcohol ether-2 sulfates (FAES)A \sim 233Fatty alcohol ether-2.5 sulfates (FAES)A \sim 233Fatty alcohol ether-3 sulfates (FAES)A \sim 233Fatty alcohol ether-3 sulfates (FAES)A \sim 233Fatty alcohol ether-3 sulfates sulfosuccinatesA \sim 233Fatty alcohol ether-4 sulfosuccinatesA \sim 233Fatty alcohol POE adducts \sim F230	6		D		269
Fatty acid salts C10Fatty acid salts C8Fatty alcohol ether-2 sulfates (FAES)A233233Fatty alcohol ether-2.5 sulfates (FAES)AA233Fatty alcohol ether-3 sulfates (FAES)A233233Fatty alcohol ether-3 sulfates (FAES)A233233Fatty alcohol ether-3 sulfosuccinatesAS233Fatty alcohol ether-4 sulfosuccinatesAS233Fatty alcohol ether-4 sulfosuccinatesAS233Fatty alcohol ether-4 sulfosuccinatesAS233Fatty alcohol POE adductsIII230	Fatty acid POE adducts			F	230
Fatty acid salts C8Fatty alcohol ether-2 sulfates (FAES)A233Fatty alcohol ether-2.5 sulfates (FAES)AC233Fatty alcohol ether-3 sulfates (FAES)AC233Fatty alcohol ether-3 sulfates (FAES)AC233Fatty alcohol ether-3 sulfosuccinatesAC233Fatty alcohol ether-3 sulfosuccinatesAC233Fatty alcohol ether-4 sulfosuccinatesAC233Fatty alcohol ether-4 sulfosuccinatesAC233Fatty alcohol POE adductsCF230	Fatty acid salts $\ge C_{12}$	В			233
Fatty alcohol ether-2 sulfates (FAES)A233Fatty alcohol ether-2.5 sulfates (FAES)AC233Fatty alcohol ether-3 sulfates (FAES)AC233Fatty alcohol ether-3 sulfates (FAES)AC233Fatty alcohol ether-3 sulfosuccinatesAC233Fatty alcohol ether-4 sulfosuccinatesAC233Fatty alcohol ether-4 sulfosuccinatesAC233Fatty alcohol POE adductsIF230	Fatty acid salts C ₁₀	-	-	-	_
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(FAES)Fatty alcohol ether-3 sulfosuccinatesA233Fatty alcohol ether-4 sulfosuccinatesA233Fatty alcohol ether-4 sulfosuccinatesF233Fatty alcohol POE adductsF230	-	A			233
sulfosuccinatesIIIFatty alcohol ether-4 sulfosuccinatesA233Fatty alcohol POE adductsF230	-	A			233
sulfosuccinates Fatty alcohol POE adducts F 230	•	A			233
		А			233
	Fatty alcohol POE adducts			F	230
Fatty alcohol sulfates (FAS) A 233	Fatty alcohol sulfates (FAS)	А			233
Fatty amine POE adducts F 230	Fatty amine POE adducts			F	230
Fatty amine POE(1-4) adductsD233	Fatty amine POE(1-4) adducts	D			233

Product	IS	SR	NIO	AB
Fatty amines	D			233
Foam baths	A			233
Foam baths with cocoylisethionates		С		269
Foam baths with fatty alcohol PEG sulfosuccinates		В		269
Foam baths with fatty alcohol PEG->3 sulfates		В		269
Foam baths with high betain content		В		269
Foam baths with high NIO content		В		269
Formulations containing abrasives (anionic)		A		269
Formulations containing abrasives (cationic)		D		269
Gargling solution (quats)	D			233
Glass cleaning agents	А			233
Hair conditioners (cationic)		D		269
Hair cure (cationic)		D		269
Hair shampoos	А			233
Hair shampoos with cocoylisethionates		С		269
Hair shampoos with fatty alcohol PEG sulfosuccinates		В		269
Hair shampoos with fatty alcohol PEG->3 sulfates		В		269
Hair shampoos with high betain content		В		269
Hair shampoos with high NIO content		В		269
Hand disinfecting agents (quats)	D			233
Hexetidine			F	263
Household cleaners	А			233
Household cleaners (anionic)		А		269
Household cleaners (anionic) with high NIO content		В		269
Household cleaners (cationic)		D		269
Hydrotropes	-	_	_	_
Ipatropim bromide			F	263
Lauryl sarcosinates	С			233
Lidocaine			F	263
Linear alkylbenzenesulfonates (LAS)	A			233
Liquid soaps with cocoylisethionates		С		269

Application Bulletin 268/3

Potentiometric titration of surfactants and pharmaceutical compounds – an overview

Product	IS	SR	NIO	AB
Liquid soaps with fatty alcohol PEG sulfosuccinates		В		269
Liquid soaps with fatty alcohol PEG->3 sulfates		В		269
Liquid soaps with high betain content		В		269
Liquid soaps with high NIO content		В		269
Liquid washing agents		А		269
Metal soaps	В			233
Metoclopramide hydrochloride			F	263
Mouth rinse solution (anionic)	Α			233
Mouth rinse solution (cationic)	D			233
Mouth rinse solution (quats)	D			233
Neutral cleaners	А			233
Neutral cleaners with high betain content		В		269
Neutral cleaners with high NIO content		В		269
NIO surfactants, POE <4	-	_	_	_
NIO surfactants, POE >4			F	230
Nose drops (quats)	D			233
Octenidine dihydrochloride			F	263
Oil baths (anionic)		А		269
Oil-containing formulations (anionic)		A		269
Oil-containing formulations (cationic)		D		269
Olefin sulfonates		А		269
Papaverine			F	263
Paraffin sulfonates		А		269
PEG (polyethylene glycols)			F	230
Phenyltoloxamine dihydrogen citrate			F	263
Phosphoric surfactants		В		269
POE alkylphenol adducts			F	230
POE castor oil			F	230
POE fatty acid adducts			F	230
POE fatty alcohol adducts			F	230
POE fatty amine adducts			F	230
POE glucose esters			F	230
POE glycerol fatty acid partial esters			F	230 dr
POE polyglycerol fatty acid partial			F	230

esters POE POP polymerisates POE sorbitan fatty acid partial			F	dr
				230
esters			F	230 dr
Polyethers			F	230
Polyethylene glycols (PEG)			F	230
Propafenone			F	263
Quaternary ammonium Compounds	C			233
Quaternary imidazoline E compounds	C			233
Quaternary imidazolium E compounds	C			233
Salbutamol sulfate			F	263
Samples containing abraded metal particles (anionic)		A		269
Samples containing abraded metal particles (cationic)		D		269
Samples containing active chlorine (anionic)		A		269
Samples containing active chlorine (cationic)		D		269
Samples containing active oxygen (anionic)		A		269
Samples containing active oxygen (cationic)		D		269
Scouring dispersion (anionic)	4			233
Scouring dispersion (anionic)		А		269
Scouring powder (anionic)	4			233
Scouring powder (anionic)		A		269
Secondary alkanesulfonates (SAS)		A		269
 Sensitive formulations (anionic) shower baths liquid soaps household cleaners foam baths dishwashing liquids (manual cleaning) 		В		269
Shampoos A	4			233
Shampoos with cocoylisethionates		С		269
Shampoos with fatty alcohol PEG sulfosuccinates		В		269
Shampoos with fatty alcohol PEG- >3 sulfates		В		269

Application Bulletin 268/3

Potentiometric titration of surfactants and pharmaceutical compounds – an overview

Product	IS	SR	NIO	AB
Shampoos with high betain content		В		269
Shampoos with high NIO content		В		269
Shower baths	А			233
Shower baths with cocoylisethionates		С		269
Shower baths with fatty alcohol PEG sulfosuccinates		В		269
Shower baths with fatty alcohol PEG->3 sulfates		В		269
Shower baths with high betain content		В		269
Shower baths with high NIO content		В		269
Shower oils (anionic)		А		269
Skin disinfecting agents (quats)	D			233
$Soaps \ge C_{12}$	В			233
Soaps C ₁₀	-	_	-	-
Soaps C ₈	-	-	-	-
Solvent-containing formulations (anionic)		A		269
Solvent-containing formulations (cationic)		D		269
Sulfosuccinate diesters	А			233
Sulfosuccinate monoesters	А			233
Taurides		В		269
Toluene sulfonates	-	-	-	-
Toothpaste (amine fluorides)	D			233
Toothpaste (anionic)	А			233
Toothpaste (anionic)		А		269
Verapamil hydrochloride			F	263
Washing powders (anionic and soaps)		A		269
Washing powders (anionic)		А		269
Washing-up liquids	А			233
Washing-up liquids with high NIO content		В		269
Window cleaning agents	А			233
Xylene sulfonates	-	_	-	_
NIO in waste water			Н	230