

## Application Bulletin 269/4 e

# Potentiometric determination of ionic surfactants by two-phase titration using Surfactrodes

### Branch

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

### Keywords

Two-phase titration; anionic and cationic surfactants; Epton titration; Surfactrode Resistant; Surfactrode Refill; 6.0507.130; 6.0507.140; branch 1; branch 3; branch 4; branch 10; branch 12

### Summary

By way of numerous practical examples this bulletin describes the «two-phase titration» with potentiometric indication of ionic surfactants in raw materials and a wide range of formulations.

Two surfactant electrodes – the Surfactrode Resistant and the Surfactrode Refill – allow this type of surfactant titration to be performed in a way analogous to the classical «Epton titration» and with a high level of automation. The results obtained show excellent correlation with those of the Epton titration. The toxic, carcinogenic and environmentally hazardous chloroform can be replaced by alternative solvents such as methyl isobutyl ketone or n-hexane.

### Instruments

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

### Electrodes

Surfactrode Resistant	6.0507.130
Surfactrode Refill	6.0507.140
Ag/AgCl Reference Electrode (KCl)	6.0726.107

### Reagents

- Sulphuric acid  $c(\text{H}_2\text{SO}_4) = 0.1 \text{ mol/L}$
- Sodium hydroxide  $c(\text{NaOH}) = 0.1 \text{ mol/L}$

- Methanol, p.a.
- Ethanol, denatured
- Solvents such as methyl isobutyl ketone (MIBK), cyclohexane or n-hexane
- TEGO add (order no. 6.2317.100 (50 mL) or 6.2317.110 (500 mL), an additive especially developed for surfactant titrations with the Metrohm Surfactrodes.
- TEGO<sup>®</sup>trant A100: 1,3-didecyl-2-methylimidazolium chloride (DDMICI);. The product contains approx. 8% water.
  - 6 g: order no. 6.2317.000
  - 60 g: order no. 6.2317.010
  - 500 g: order no. 6.2317.020
- Hyamine 1622: benzethonium chloride;
- Sodium dodecyl sulphate (sodium lauryl sulphate, SDS, LAS);

### Solutions

#### General

Selection of the titrant is extremely important. The better the extraction of the compounds formed into the solvent phase, the larger and steeper the potential jump of the titration curve. This effect should be made use of, particularly when formulations with a complex composition have to be titrated.

Work is preferably carried out with titrants of concentration 0.005 mol/L or 0.02 mol/L. Titrants of concentration 0.02 mol/L are primarily used if:

- the formulations contain considerable amounts of non-ionic surfactants in addition to the ionic surfactants,
- the formulations being analyzed contain ethoxylated sulphosuccinic acid monoesters (lauryleth sulphosuccinates), sarcosinates, taurides, cocoyl-isethionates or fatty alcohol ether sulphates (Polyoxyethylenegroup, POE >3 )
- pure substances or raw materials are analyzed.

## Preparation of the titrants

### General

The required titrant weight for one liter is given by the formula:

$$\text{Weight [in g]} = \frac{M_t \times c_t \times 100}{p_t}$$

$M_t$ : Molar mass of titrant

$c_t$ : Titrant concentration, here 0.005 or 0.02 mol/L

100: Conversion factor due to % in L

$p_t$ : Purity of the titrant in %

The required quantity (plus a small excess\*, ~5 – 8%) is weighed exactly and dissolved in dist. water with gentle warming if necessary. The titrant is filled up to 1 liter with dist. water at 20 °C. The titer is determined against an anionic or cationic surfactant. As long as the solution in the bottle and buret has not stabilized, the titer is also not stable. (Surfactants tend to adhere to surfaces; this applies particularly to cationic surfactants.) For preparation of a fresh titrant it is recommended to use the same bottles and burets and allow the solutions to stand for at least a day before determining the titer.

\*The substances naturally do not contain 100% active substance. The largest secondary component is usually water. The water content can be as high as 8% and is very difficult to remove. It is essential to take these circumstances into account when weighing in the sample.

### Preparation of c(TEGO®trant A100) = 0.005 mol/L

Approx. 2.12 g 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 filled 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.

Approx. 1.44 g 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.

### Preparation of c(sodium dodecyl sulfate) = 0.005 mol/L

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.

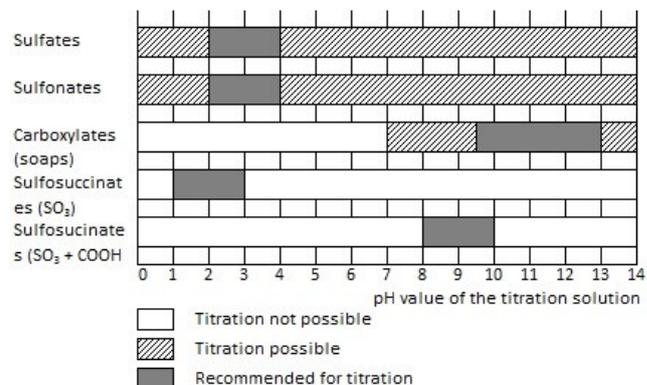
As a titer determination in the normal sense is not possible, the standard solutions are prepared by very exact weighing in of sodium dodecyl sulfate.

1.44 g of SDS 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.

## Analysis

### pH ranges for some surfactant classes

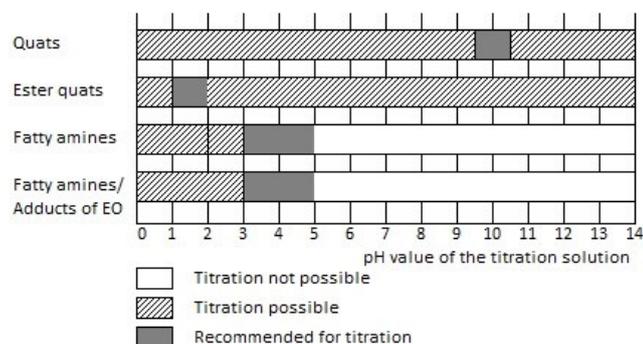
Titration recommendations for anionic surfactants



- With samples containing both sulphosuccinates and betains, the sulphosuccinates are titrated at pH = 3.0.

- For the determination of sulphosuccinates the following pH values are selected:
  - sulphonate group at pH = 2.0
  - sulphonate and carboxylate group at pH = 10.0.
- It is essential that the pH is not adjusted until shortly before the titration!
- Surfactants based on phosphoric acid esters (phosphoric surfactants), which are usually insoluble in water, can be titrated without any problems using this technique. The samples are made slightly alkaline with NaOH and after addition of ethanol/MIBK 1:1 they are titrated in the usual manner with c(TEGO®trant A100) = 0.02 mol/L. Relative standard deviations of approx. 1% were achieved.

Titration recommendations for cationic surfactants



- For the determination of the degree of quaternization of amines, an additional titration is performed at pH = 3.0 to obtain the sum of the quaternary ammonium compounds and the tertiary starting amine.
- The ester quats contained in softeners are usually titrated at pH = 2.0. However, some ester quats exhibit their greatest stability at pH = 5.0 or pH = 7.0 and hence should also be determined at these pH values. This is another case where the pH value must not be adjusted until shortly before the titration. Slightly alkaline conditions can very rapidly lead to an ester cleavage resulting in the ester losing its surfactant properties. This would lead to erroneous results.
- Betains and amphoteric surfactants cannot be titrated. However, when present in their protonated forms (pH = 0 – 2), they can interfere with the determination of other surfactants.
- If the surfactant content of the sample is more or less known one can work with a start volume, which shortens the titration time considerably.

- A great advantage of the Surfactrodes is their unproblematic and universal use. Over 90% of the samples analysed to date could be titrated using a single standard method, e.g. anionic surfactants in ca. 100 different shampoo and shower preparations at pH = 3.0. Whereas with earlier titrations the analyst had always to know the other matrix components of the formulations, this was not necessary with these analyses.
- These facts lead to considerable advantages, particularly for quality assurance laboratories. The preparation time needed for the surfactant titrations can be shortened considerably. After entry of the method in the titrator, the titrations can be performed without any problems, even by semi-skilled personnel.
- The titrant consumption in the surfactant titration should always be 10 ... 15 mL. An appropriate amount of sample is weighed into a titration beaker and dissolved in ca. 50 mL dist. water. The corresponding pH value is adjusted with diluted sodium hydroxide or sulphuric acid, 20 mL of a 1 : 1 mixture of ethanol and MIBK (or n-hexane or chloroform) as well as 0.2 mL TEGO add are added, the solution is made up to ca. 100 mL with dist. water and the titration is started. In the case of samples that are poorly soluble or even insoluble in water, the solvent mixture can also be used for dissolution. For accuracy reasons, it is advisable first to prepare an intermediate dilution of raw materials and concentrates and then to weigh in an aliquot of this solution.
- Thorough mixing of the sample during the titration is particularly important in the two-phase titration. It is extremely important that the two phases are well mixed and an emulsion is formed without the incorporation of air bubbles through stirring or the formation of a stirring vortex. A magnetic stirrer does not suffice for thorough mixing of the solution; it is essential to use the 722 Propeller Rod Stirrer.

Excessive amounts of salts, particularly NaCl and KCl, interfere with the surfactant titration with the Surfactrode Resistant. Accordingly, e.g. low surfactant contents in electroplating baths cannot be determined using this method. On the other hand, salt concentrations of the type that occur in the titration of formulations are harmless and have no influence on the titration results.

### Titration parameters

Parameter	Surfactrode Resistant / Refill
Titration mode	DET U
Pause	60 s
Signal drift	10 mV/min
Max. waiting time	120 s
Meas.pt.density	4
Min. increment	100 µL
Dosing rate	max.

### Determination of anionic surfactants

- Anionic surfactants are normally titrated at pH = 3.0. A few special cases have been discussed above. The corresponding diagram can be used as the basis to determine the correct pH value.
- True soluble soaps (sodium or potassium salts of higher fatty acids) must be titrated with TEGO trant A100 at pH values  $\geq 10$ . Other titrants produce poor titration curves that can usually not be evaluated. In mixtures, anionic surfactants and soaps are determined as a sum. The pH value for the sum titration depends on the sample used and should be determined by preliminary titrations (pH = 10 ... 12). Of the two potential jumps, the second is used for the determination of the sum of soaps and anionic surfactants. With decreasing pH value, the fraction of the soaps determined by the titration becomes increasingly smaller. A complete separation is achieved by acidifying the sample to pH = 2.0 (allow the sample to stand 15 ... 30 min for the reaction to run to completion). Only the anionic surfactants are then determined in the titration.

### Determination of cationic surfactants

- Cationic surfactants are normally titrated at pH = 10.0. A few special cases have been discussed above. The corresponding diagram can be used as the basis to determine the correct pH value.
- Samples containing amine hydrochlorides are titrated at pH = 3.0 if this class of substances has to be determined. However, if the content of cationic surfactants without the amine hydrochlorides is required, work is «normally» performed at pH = 10.0.

### Titer determination of the TEGO®trant A100

10.0 mL of the corresponding sodium dodecyl sulfate standard solution is pipetted into a glass beaker. Followed by adding of 80 mL dist. water and 20 mL ethanol/MIBk 1 : 1 as well as 0.2 mL TEGO add and titrate until the first equivalence point with  $c(\text{TEGO}^{\circ}\text{trant A100})$  0.005 mol/L while vigorous stirring.

### Calculation

#### Titer

$$f = \frac{m_s \times V_S \times C_S}{V_{EP1} \times M_S \times 100 \times c_{\text{Titrant}}}$$

- f: Titer of the titrant  
 $V_{EP1}$ : Titrant consumption in mL  
 $m_s$ : Sample weight of SDS standard in g  
 $V_S$ : Added volume of SDS solution in mL, here 10.0  
 $C_S$ : Active substance content of the SDS used in %, here 99.2  
 $M_S$ : Molecular weight of reference substance; here 288.4 g/mol  
 100: Conversion factor due to %  
 $c_{\text{Titrant}}$ : Theoretical concentration of the titrant in mol/L; here 0.005 or 0.02 or 0.05

#### Sample

$$w_{\text{Surfactant}} = \frac{V_{EP1} \times M_A \times f \times c_t \times 100}{m_s}$$

- $w_{\text{Surfactant}}$ : Content of surfactant in %  
 $V_{EP1}$ : mL titrant consumed to reach the first EP  
 $M_A$ : Molecular weight of analyte in g/mol  
 1000: Conversion factor from mol to mmol  
 f: Titer of the TEGO®trant  
 $c_t$ : Concentration of the titrant, here 0.005 mol/L  
 100: Conversion factor due to %  
 $m_s$ : Sample weight in mg

If the molar mass of the surfactant under investigation is unknown or if the total surfactant content needs to be determined (without using an average molar mass), the analysis result can also be specified in mmol surfactant / 100 g sample:

$$\frac{\text{mmol surfactant}}{100 \text{ g sample}} = \frac{V_{EP1} \times 1000 \times f \times c_t \times 100}{m_S}$$

$V_{EP1}$ : mL TEGO®trant consumed to reach the first EP

1000: Conversion factor from mol to mmol

f: Titer of the TEGO®trant

$c_t$ : Concentration of the titrant, here 0.005 mol/L

100: Conversion factor due to 100 g

$m_S$ : Sample weight in mg

### Equivalence calculation

1 mL anionic surfactant 0.005 mol/L

$$= 1.9985 \text{ mg Tego®trant}$$

1 mL cationic surfactant 0.005 mol/L

$$= 1.4419 \text{ mg SDS}$$

### Comments

- The service life of the Surfactrodes can be prolonged if they are stored dry when not in use (overnight, over the weekend).
- It is always advisable to place the electrodes in the sample solution for 20 – 40 s before each titration to assure their adaptation to the sample matrix.
- If sufficient sample is available, the sample weight should be chosen to produce a titrant consumption of at least 10 mL at the equivalence point. This is the only way to ensure that the total amount of surfactants is determined. While lower sample weights produce better titration curves, they sometimes lead to results that are too low.
- For anionic surfactants, TEGO®trant A100 provides steeper and larger potential jumps than other titrants. This advantage is particularly apparent in the case of surfactants and soaps that, e.g. with Hyamine®1622, produce very weak, poorly defined potential jumps. With TEGO®trant A100, more hydrophilic surfactants (surfactants with shorter alkyl chains or those with hydrophilic groups such as esters, amides, POE compounds) can thus still be titrated.

TEGO add has a positive influence on the entire course of the titration and also keeps the electrodes clean. The addition of TEGO add is highly recommended with samples that contain abrasives (e.g. toothpastes, scouring dispersions), with washing powders and surfactant raw materials.

TEGO add produces a highly dispersed and uniform distribution of the two phases during the titration and ensures a smooth curve profile, especially in the vicinity of the inflection point. Experimental investigations in the field of the analysis of washing powders have shown clearly that, if TEGO add is added, the quality of the titration curves does not diminish as the number of titrations increases: Even after 160 titrations, the same good titration curves and results were obtained. On the other hand, without the addition of TEGO add the curves clearly became worse after just a few titrations. The builders contained in the washing powders coated the electrode surface and were virtually impossible to remove later.

## Practical examples

### 1. Analysis of raw materials

#### 1.1 Determination of anionic surfactants

##### 1.1.1 Fatty alcohol ether sulphates, fatty alcohol sulphates, $\alpha$ -olefin sulphonates, linear alkylbenzene-sulphonates, secondary alkanesulphonates

The sample is dissolved in approx. 80 mL dist. water and the pH is adjusted to 3.0 using  $c(\text{H}_2\text{SO}_4) = 0.1 \text{ mol/L}$ . Then 20 mL ethanol/MIBK 1:1 as well as 0.2 mL TEGO add are added and the sample solution is titrated with  $c(\text{TEGO}@\text{trant A100}) = 0.005 \text{ mol/L}$  (see Fig. 1).

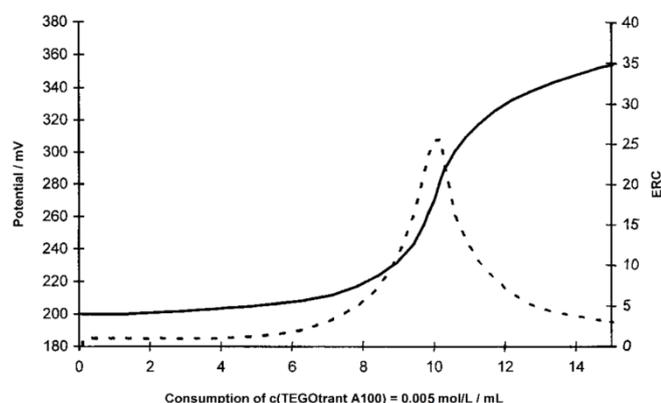


Fig. 1 Titration curve for the determination of sodium lauryl ether sulphate

##### 1.1.2 Sulphosuccinate monoesters

The sample is dissolved in approx. 50 mL dist. water. The pH is adjusted to 2.0 using  $c(\text{H}_2\text{SO}_4) = 0.1 \text{ mol/L}$  (if only the sulphonate group needs to be determined) or to pH 10.0 using  $c(\text{NaOH}) = 0.1 \text{ mol/L}$  (if both the sulphonate and the carboxylate group need to be determined). Then 20 mL ethanol/MIBK 1:1 and 0.2 mL TEGO add are added, followed by the addition of approx. 30 mL dist. water. The sample solution is then immediately titrated with  $c(\text{TEGO}@\text{trant A100}) = 0.005 \text{ mol/L}$  (see Fig. 2). Take into consideration the tendency of the ester group to hydrolyze!

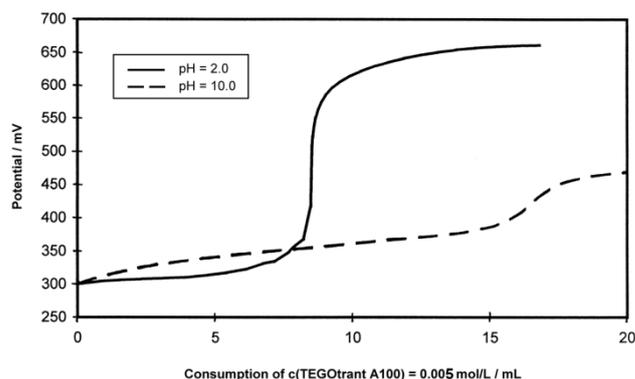


Fig. 2 Titration curves for the determination of a sulphosuccinate monoester at pH = 2.0 and pH = 10.0 (Caution: The values have been corrected to 300 mV start potential).

##### 1.1.3 Sulphosuccinate diesters

The sample is dissolved in approx. 80 mL dist. water and the pH is adjusted to 3.0 using  $c(\text{H}_2\text{SO}_4) = 0.1 \text{ mol/L}$ . Then 20 mL ethanol/MIBK 1:1 as well as 0.2 mL TEGO add are added and the solution is immediately titrated with  $c(\text{TEGO}@\text{trant A100}) = 0.005 \text{ mol/L}$ . Take into consideration that ester groups tend to hydrolysis. tendency of the ester groups to hydrolyse!

#### 1.2 Determination of cationic surfactants

##### 1.2.1 Dialkyldimethylammonium chloride, benzalkonium chloride, quaternary imidazolium compounds, fatty amines

The sample is dissolved in approx. 80 mL dist. water and the pH is adjusted to 10.0 using  $c(\text{NaOH}) = 0.1 \text{ mol/L}$ . Then 20 mL ethanol/MIBK 1:1 as well as 0.2 mL TEGO add are added and titrated with  $c(\text{SDS}) = 0.005 \text{ mol/L}$  (see Fig. 3).

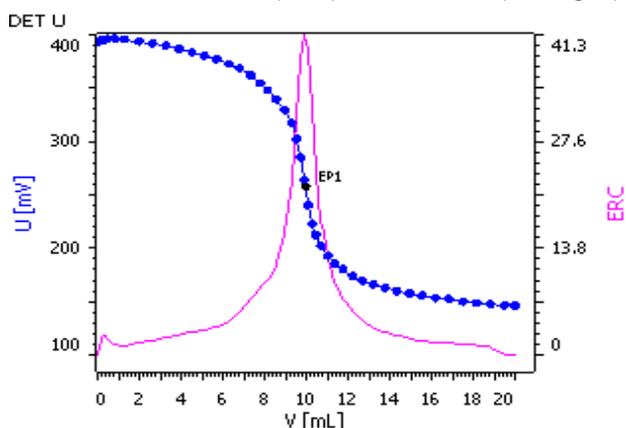


Fig. 3 Titration curve for the determination of a quaternary imidazolium compound in an antiseptic solution.

## 2. Analysis of formulations

The two-phase titration of ionic surfactants is usually free from problems even in formulations with a very complex composition.

### 2.1 Anionic surfactants in washing-up liquid (threefold concentrate for manual cleaning), wool shampoo or liquid heavy-duty washing agent

The sample is dissolved in approx. 80 mL dist. water. The pH is adjusted to 3.0 using  $c(\text{H}_2\text{SO}_4) = 0.1 \text{ mol/L}$  and, after the addition of 20 mL ethanol/MIBK 1:1 and 0.2 mL TEGO add, the sample is titrated with  $c(\text{TEGO@trant A100}) = 0.005 \text{ mol/L}$ .

### 2.2 Anionic surfactants in washing-up balsam

The sample is dissolved in approx. 80 mL dist. water. The pH is adjusted to 3.0 using  $c(\text{H}_2\text{SO}_4) = 0.1 \text{ mol/L}$  and, after the addition of 20 mL ethanol/MIBK 1:1 and 0.2 mL TEGO add, the sample is titrated with  $c(\text{TEGO@trant A100}) = 0.02 \text{ mol/L}$  (see Fig. 4).

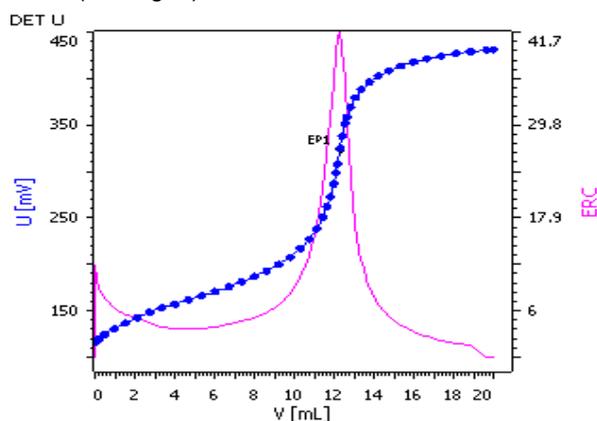


Fig. 4 Titration curve for the analysis of a washing-up balsam.

### 2.3 Anionic surfactants in cosmetic oil baths and shower oils

The sample is dissolved in 20 mL ethanol/MIBK 1:1. Then approx. 80 mL dist. water is added and the pH of the solution is adjusted to 3.0 using  $c(\text{H}_2\text{SO}_4) = 0.1 \text{ mol/L}$ . Then 0.2 mL TEGO add is added, and the sample is titrated with  $c(\text{TEGO@trant A100}) = 0.005 \text{ mol/L}$ .

### 2.4 Cooling lubricants, drilling and cutting oils, cleaning baths

The MetroSensor Surfactrodes allow determining ionic surfactants by potentiometric titration e.g. in the following complex matrices: cooling lubricants miscible or immiscible with water, drilling and cutting oils or alkaline cleaning baths contaminated with oil.

MIBK and n-hexane in particular have proved their worth as solvents. As there is no need for sample preparation, the analysis results are already available after 5 – 10 min.

### Anionic surfactants

For the determination of anionic surfactants in water-miscible cooling lubricants, relative standard deviations of 1% can be achieved with a titration time of 5 – 10 min. If higher standard deviations can be tolerated, the required analysis time can be shortened further.

### Cationic surfactants

Like the anionic surfactants, the cationic surfactants can also be titrated without any problems in most cases (titrant:  $c(\text{SDS}) = 0.005 \text{ mol/L}$ ). There is a strictly linear relationship between the titrant consumption and the content of cationic surfactants.

#### 2.4.1 Anionic surfactants in cooling lubricant

The sample is dissolved in 20 mL ethanol/MIBK 1:1 or ethanol/n-hexane 1:1. Then 50 mL dist. water is added and the pH is adjusted to 3.0 using  $c(\text{H}_2\text{SO}_4) = 0.1 \text{ mol/L}$ . After the addition of approx. 30 mL dist. water, the sample is titrated with  $c(\text{TEGO@trant A100}) = 0.005 \text{ mol/L}$  (see Fig. 5).

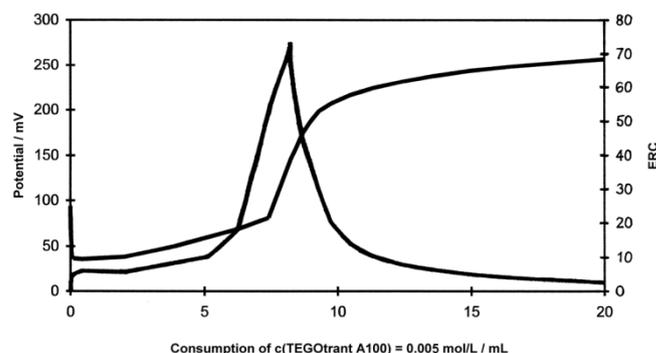


Fig. 5 Titration curve for the analysis of a cooling lubricant.

### 2.5 Anionic surfactants in household cleaner based on soap

The sample is dissolved in approx. 50 mL dist. water. Then the pH is adjusted to 10.0 using  $c(\text{NaOH}) = 0.1 \text{ mol/L}$ . Finally 20 mL ethanol/MIBK 1:1, approx. 30 mL dist. water and 0.2 mL TEGO add are added and titrated with  $c(\text{TEGO@trant A100}) = 0.005 \text{ mol/L}$ .

### 2.6 Cationic surfactants in household cleaner containing pine oil or in hair conditioner

The sample is dissolved in approx. 80 mL dist. water. The pH is adjust to 10.0 by using  $c(\text{NaOH}) = 0.1 \text{ mol/L}$ , Then 20 mL ethanol/MIBK 1:1 and 0.2 mL TEGO add are added and the sample solution is then titrated with  $c(\text{SDS}) = 0.005 \text{ mol/L}$ .

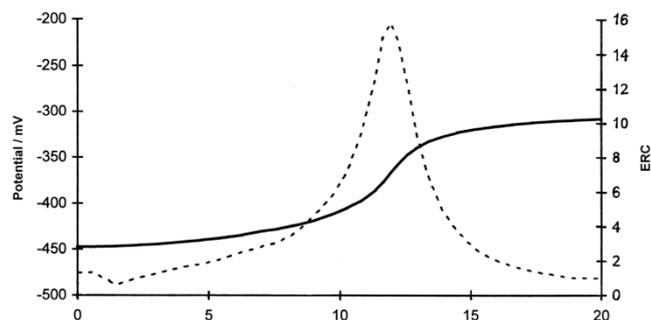


Fig. 6 Titration curve for the analysis of a household cleaner containing pine oil.

### 2.7 Anionic surfactants in scouring dispersion

The sample is suspended in approx. 50 mL dist. water and the pH is adjusted to 3.0 using  $c(\text{H}_2\text{SO}_4) = 0.1 \text{ mol/L}$ . Then 10 mL of ethanol, 10 mL chloroform and 0.2 mL TEGO add is added and the sample is titrated with  $c(\text{TEGO}@\text{trant A100}) = 0.005 \text{ mol/L}$ .

### 2.8 Washing powders

The analysis of washing powders in aqueous solutions using conventional surfactant electrodes is possible only in a few cases. Some constituents of these very complex formulations make the surfactant titration difficult. In particular, the various builder systems cause interferences. (Interactions between the anionic surfactants and the silicates lead to titration curves that can in some cases no longer be evaluated.)

With the MetroSensor Surfactrodes, however, the anionic surfactants contained in washing powders can be determined titrimetrically. Basic work on this subject was carried out in cooperation with a leading European washing agent producer.

Further information and details concerning the analysis of washing powders can be found in our Application Bulletin No. 275.

The titration curves of washing powders differ greatly, depending on the builder system used. Whereas soluble builders result in virtually ideal titration curves, in the case of insoluble builders – for instance based on Sasil® (sodium aluminosilicate; zeolite A) – flatter titration curves with smaller potential jumps are obtained (see Figs. 7 and 8).

In contrast to the classical Epton titration, in the potentiometric two-phase titration there is no need to carry out the very time-consuming ethanol extraction of the surfactants under investigation contained in the washing powder. This means a considerable saving of time and costs.

A total of approx. 30 different washing powders have been analysed and the results compared with the classical two-phase titration (mixed indicator disulphine blue / dimidium

bromide). The results of all samples were in very good agreement! Even if the titration curves of the different builder systems differed greatly visually, the analysis results were affected only to a minor extent. The relative standard deviations were all around 0.5%.

#### 2.8.1 Anionic surfactants in washing powder

A representative sampling is of great importance in the analysis of washing agents. The washing powder sample can either be weighed directly into the titration beaker or an intermediate dilution can be prepared. In the latter case, the insoluble builders should be allowed to settle before the determination. The relative deviation between the results obtained with the two techniques is less than 1%.

The sample is dissolved in approx. 50 mL dist. water and the pH is adjusted to 3.0 using  $c(\text{H}_2\text{SO}_4) = 0.1 \text{ mol/L}$ . Then 20 mL ethanol/MIBK 1:1, 0.2 mL TEGO add and approx. 30 mL dist. water is added. The sample is then titrated with  $c(\text{TEGO}@\text{trant A100}) = 0.02 \text{ mol/L}$ .

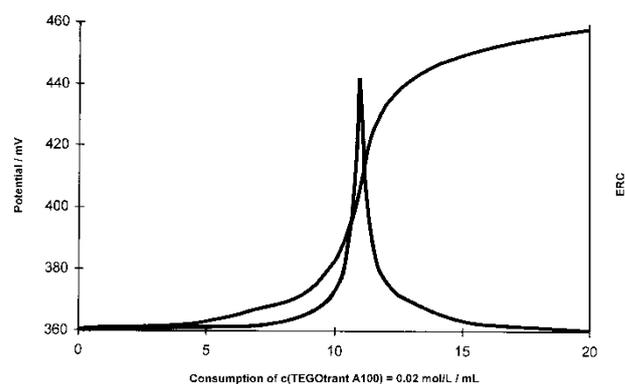


Fig. 7 Titration curve for the analysis of a washing powder with water-soluble builder.

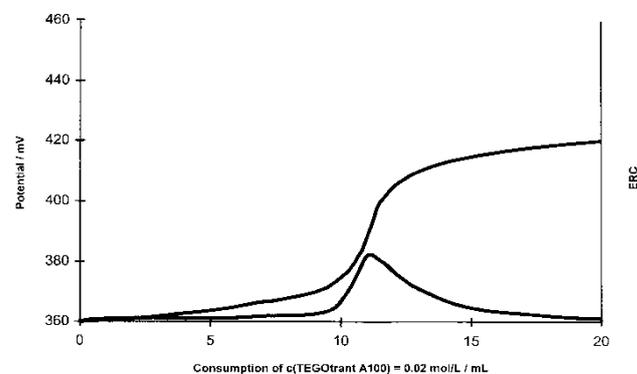


Fig. 8 Titration curve for the analysis of a washing powder with water-insoluble builder.

**References**

- R. Schulz  
Titration von Tensiden und Pharmaka – Moderne Methoden für den Praktiker  
Verlag für chemische Industrie, H. Ziolkowsky GmbH, Augsburg, 1996  
ISBN 3-87846-182-8.
- R. Schulz, R. Gerhards  
Neue Spezial-TSE für ionische Tenside  
LaborPraxis 20/10 (1996) 74–76.
- R. Schulz, J. Thiede  
Titration ionischer Tenside in Kühlschmierstoffen  
LaborPraxis 21/11 (1997) 64–68.
- R. Schulz  
Documentation on the seminars on modern surfactant analysis held jointly with Metrohm Ltd.
- Metrohm Application Bulletin No. 233  
Titrimetric/potentiometric determination of anionic and cationic surfactants
- Metrohm Application Bulletin No. 268  
Potentiometric titration of surfactants and pharmaceutical compounds – an overview  
Metrohm Application Bulletin No. 275
- Potentiometric two-phase titration of anionic surfactants in washing powders and liquid washing agents  
R. Schulz  
Determination of ionic surfactants in cosmetic products  
Metrohm Monograph No. 50233

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