Titration of cationic surfactants

Description

Cationic surfactants are used in cleaning- and disinfection agents. Known representatives are e.g. Benzalkonium chloride or several Dialkyl dimethyl ammonium chlorides. The content can be determined by titration with an anionic surfactant (Sodium dodecylsulfate SDS). SDS is available as a ready-to-use solution. Since it is a precipitation titration based on the formation of hydrophobic adducts, only cationic surfactants with a sufficient hydrophobic part can be titrated. Cationic surfactants with a carbon chain <C10 are usually not fully detected. Since the hydrophobic adducts are often quite sticky and can settle on the electrodes, solutions should be diluted as possible (0.004 mol/L, sometimes up to 0.01 mol/L). The addition of non-ionic surfactants (Triton X) also helps to keep the electrodes clean.
If the sample also contains long-chained amines, the pH must be set to pH 10. Otherwise the (protonated) amines will be titrated with.

Devices

Titrator	TL 7000 or higher
Exchangeable head	WA 10
Electrode	TEN 1100 PLH
Cable	L 1 A
Reference Electrode	B 2920+ or B 3920+
Cable	L 1 N
Lab accessoires	Magnetic stirrer TM 235
	Glass beakers 150 oder 250 ml

Reagents

1	Sodium dodecylsulfate (SDS) 0,004 mol/L
2	Buffer pH 10
3	Triton X solution
4	Hyamin 1622
All reagents should be in analytical grade or better.	

Titration procedure

Reagents

Sodium dodecylsulfate (SDS) 0,004 mol/L
1.154 g Sodium dodecylsulfate (SDS) and 10g Formaldehyde are dissolved in about 0,5L dist. Water and made up to 1 L. The formaldehyde is used for preservation. If the solution is consumed quickly within a few days, it can be dispensed with.
The titer determination is done with Hyamin 1622 (see application report “titer determination in surfactant titration”).

Buffer solution pH 10
4.8 g sodium tetraborate decahydrate and 18 mL NaOH 1 mol/L are dissolved in approx. 900 mL dist. Water, adjusted to pH 10 with diluted sodium hydroxide solution and made up to 1L.
Caution: an ammonia buffer is not suitable because the life of the electrode is reduced.

Cleaning and handling the TEN 1100 electrode

The TEN 1100 electrode is rinsed with dist. water or a solution of Triton X in dist. Water. The membrane of the electrode must not be cleaned mechanically. Do not use solvents for cleaning, the membrane can be destroyed. Store the electrode clean and dry.
With a new electrode or one that has not been used for a long time, the potential jumps in the first titrations are usually quite flat. Therefore, the electrode should be conditioned before use. For this, the electrode is placed in a solution of 0.5 ml Hyamin 1622 (0.004 mol/L) and 0.5 ml sodium dodecyl sulfate (0.004 mol/L) in 80 mL water.

Sample preparation

The sample is weighed in, made up to approx. 100 mL with dist. Water, 10 mL buffer solution pH 10 and 0.5 mL Triton X solution are added. Triton X is necessary if the precipitates are sticky. If not it can be dispended with. For strong acidic samples, it could be necessary to adjust the pH to pH 10 with diluted NaOH. The sample should contain about 5 - 20 mg (0.02 – 0.06 mmol) of cationic surfactant.

In the case of poorly soluble samples, the solubility of the sample can be improved by adding a little amount Methanol or Ethanol. Attention: Methanol and Ethanol reduce the life time of the electrode, the addition should not exceed 5%.

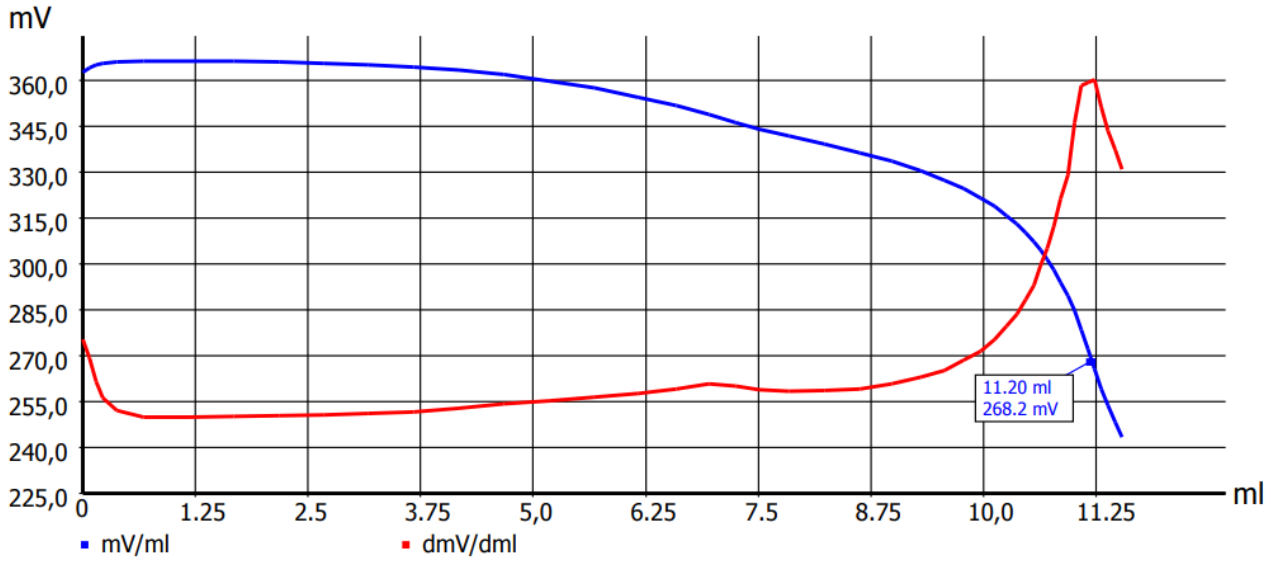
Then it is titrated to an equivalence point with Sodium dodecylsulfate (SDS). For some samples, the potential jump on the EQ is only weak. Here it is often better to use an end volume or the potential of a titrated sample as stop criteria.

If the content of surfactants or surfactant concentrates that contain large amounts of cationic components should be determined, the required amount of sample is very small and difficult to weigh. Then the following method can be used: a larger amount of sample (W_{Sample}) is weighed into a flask. For this, 50 - 200 times the amount of distilled water (W_{H2O}) is weighed and the sample is dissolved in it. From this solution an aliquot A is taken for the titration. The amount of sample contained in the aliquot A is calculated using the following formula:

$$W\ [g] = \frac{W_{Sample}\ [g]}{(W_{Sample}\ [g] + W_{H2O}[g])} * A\ [g]$$

Titration parameter

For surfactant titration a slow, linear titration with relatively large steps is well suited.
Since it is a slow precipitation titration, slow measuring speeds are necessary.
Such a titration can last 10 to 20 minutes.
In some cases also a dynamic titration can be carried out.



Default method	-		
Method type	Automatic titration		
Modus	Linear		
Measured value	mV		
Measuring speed / drift	User defined	Minimum holding time	8 s
		Maximum holding time	25 s
		Measuring time	4 s
		drift	3 mV/min
Initial waiting time	5 s		
Linear steps	0.1 mL	Max step size	0,5 mL
Damping	average	Titration direction	decrease
Pretitration	off	Delay time	0 s
End value	off		
EQ	on	Slope value	80
Max. titration volume	10 mL		
Dosing speed	100%	Filling speed	30 s

For difficult samples with weak potential jumps, it can be advantageous to use a fixed waiting time of 20s and more instead of the measurement speed / drift.

Titration can also be performed dynamically for samples that deliver a strong potential jump.

Default method	-		
Method type	Automatic titration		
Modus	Dynamic		
Measured value	mV		
Measuring speed / drift	User defined	Minimum holding time	8 s
		Maximum holding time	25 s
		Measuring time	4 s
		drift	3 mV/min
Initial waiting time	5 s		
Dynamic	User-defined	Max step size	0,5 ml
		Slope max ml	7
		Min. step size	0,075 ml
		Slope min. ml	50
Damping	average	Titration direction	decrease
Pretitration	off	Delay time	0 s
End value	off		
EQ	on	Slope value	80
Max. titration volume	10 ml		
Dosing speed	100%	Filling speed	30 s

Calculation:

$$DDAC - C10\ [\%] = \frac{(EQ1 - B) * T * M * F1}{W * F2}$$

B	0	Blank value
EQ1		Consumption at first EQ
T	WA	Exact concentration of the titrant, readed from the Exchange Unit
M	362,08	Molar mass of Didecyl dimethyl ammonium chloride
W	man	Sample weight [g]
F1	0.1	Conversion factor
F2	1	Conversion factor

In this example the result is calculated as Didecyl dimethyl ammonium chloride. For other surfactants, the corresponding molar mass must be used for the calculation.

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