Titration of quaternary Ammonium compounds in disinfectants

Description

Titration of quaternary Ammonium compounds (surfactants) in disinfectants.
This method is used to determine quaternary ammonium compounds like Benzalkonium chlorides or other cationic surfactants. It is a precipitation titration in which the cationic quaternary ammonium salts are titrated with anionic surfactants. Described is the direct titration with SDS, as well as the back titration with Hyamin 1622 or cetylpyridinium chloride. The somewhat more complicated back-titration is used in cases where several inflection points occur during direct titration or if the jump is too flat. Even if only a burette with a standard solution is to be used for the titration of anionic and cationic surfactants, back titration is suitable.
The titration is carried out at pH 10, so that no protonated amine compounds are titrated together in one jump.
Cetylpyridinium chloride provides slightly larger potential jumps than Hyamin 1622. In most cases, Hyamin 1622A is sufficient, but sometimes, if the potential jump is very flat, Cetylpyridinium chloride should be used.
The result is calculated as % Benzalkoniumchloride.

Instruments

Titrator	TL 7000 or higher
Electrode	TEN 1100 PLH
Reference Electrode	B 2920 + filled with 3 mol/l NaCl
Cable	L 1 A
Cable	L 1 N
Stirrer	Magnetic stirrer TM 235 or similar
Lab accessoires	Glas beaker 100 ml tall form
	Magnetic stirrer bar 30 mm

Reagents

1	Sodiumdodecylsulfate (SDS) 0.004 mol/l
2	Hyamine 1622 solution 0.004 mol/l
3	Cetylpyridinium chloride solution 0.004 mol/l
4	Borate buffer solution pH10
5	Formaldehyde
6	Triton X solution
All reagents should be in analytical grade or better.	

Titration procedure

Reagents

Sodiumdodecylsulfate solution 0.004 mol/l:
1.154 g SDS are weighed into a 1 liter volumetric flask and dissolved with distilled water. 10 g of formaldehyde are added and filled up to 1 l with distilled water. 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 a set Hyamin 1622 solution (see application report “titer determination in surfactant titration”).

Hyamine 1622 solution 0.004 mol/l:
Is commercially available as a ready-to-use solution.

Cetylpyridinium chloride solution 0.004 mol/l:
1.36 g Cetylpyridiniumchlorid are weighed into a 1 liter volumetric flask and dissolved in distilled water and up to 1 l with distilled water.
The titer determination is done with Sodiumdodecylsulfate (see application report “titer determination in surfactant titration”).

Buffer solution pH10:
4,8 g sodium tetraborate decahydrate are weighted in a 1l - volumetric flask and dissolved in about 750 ml distilled water 180 ml NaOH 0,1 mol/l are added. The pH is adjusted to pH 10.0 with NaOH 0,1 mol/l and the mixture is made up to 1 liter.
Please do not use an Ammonia buffer pH 10. This can reduce the life of the electrode.

Handling the electrode

For cleaning, the electrodes are rinsed with distilled water.
Do not use organic solvents for cleaning the TEN 1100 PLH electrode!
The TEN 1100 PLH is stored dry.
To condition the electrode before the titration, the electrode is placed in a solution of 0.5 ml SDS-solution 0.004 mol/l and 0.5 ml Hyamin 1622 (or Cetylpyridinium chloride) 0.004 mol/l in water for a few minutes.

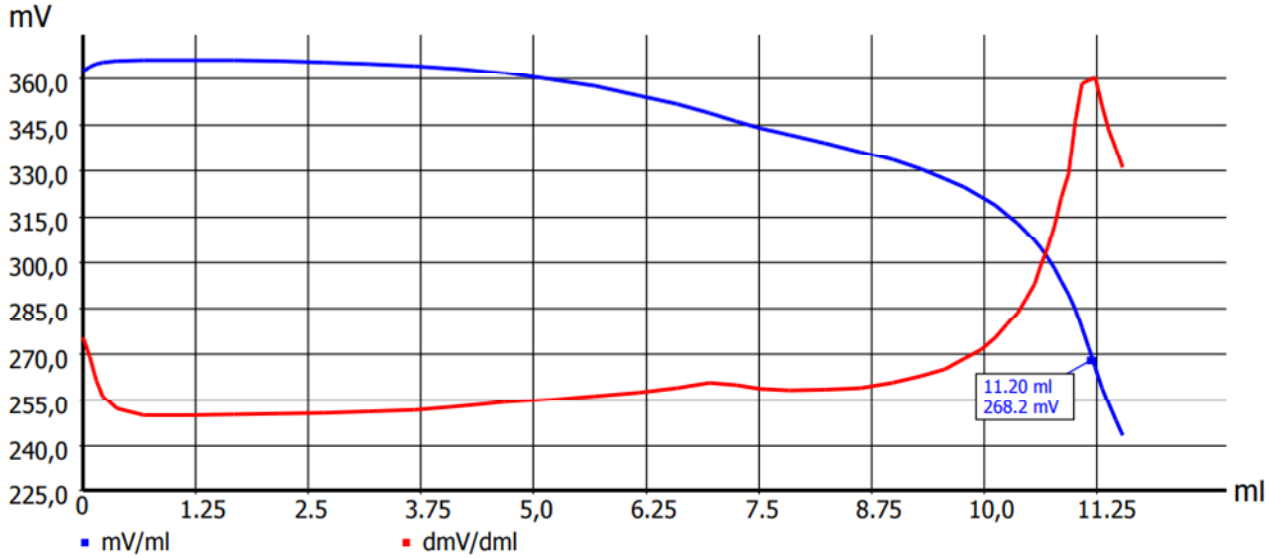
The reference electrode B 2920+ is filled with 3 mol/l NaCl solution (3 mol/l KCl is often usable as well). This electrode is stored in 3 mol/l NaCl solution (or 3 mol/l KCl)

Titration procedure – direct titration

Sample preparation

The amount of sample is chosen so that about 0,02 – 0,06 mmol of the cationic component are contained or the consumption is about 5 - 10 ml. An amount of sample is weighed in a 100 ml glass beaker (tall form), 10 ml of the pH10 – buffer solution is added and the mixture is filled up to 50 ml with distilled water. 0.5 ml Triton X solution is added to keep the electrodes clean.
The sample is titrated with 0,004 mol/l SDS-solution to an equivalence point or up to a maximum volume.

Titration parameter - direct titration



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	0 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	weak	Titration direction	decrease
Pretitration	off	Delay time	0 s
End value	off		
EQ	on	Slope value	80
Max. titration volume	20 ml		
Dosing speed	100%	Filling speed	30 s

Tip from practice: If the first derivative of the titration curve at the EQ is very noisy, the smallest step size can be increased to 0.1 ml. If the EQ is not recognized despite the detectable inflection point (the titrator titrates up to the maximum volume), the slope value must be reduced.

Calculation:

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

B	0	Blank value
EQ1		Consumption of titrant until first Equivalence point
T	WA	Exact concentration of the titrant, readed from the Exchange Unit
M	354	Molecular mass of Benzalkoniumchloride
W	man	Weight of the sample
F1	0.1	Conversion factor
F2	1	Conversion factor

Titration procedure – back titration

Blank value

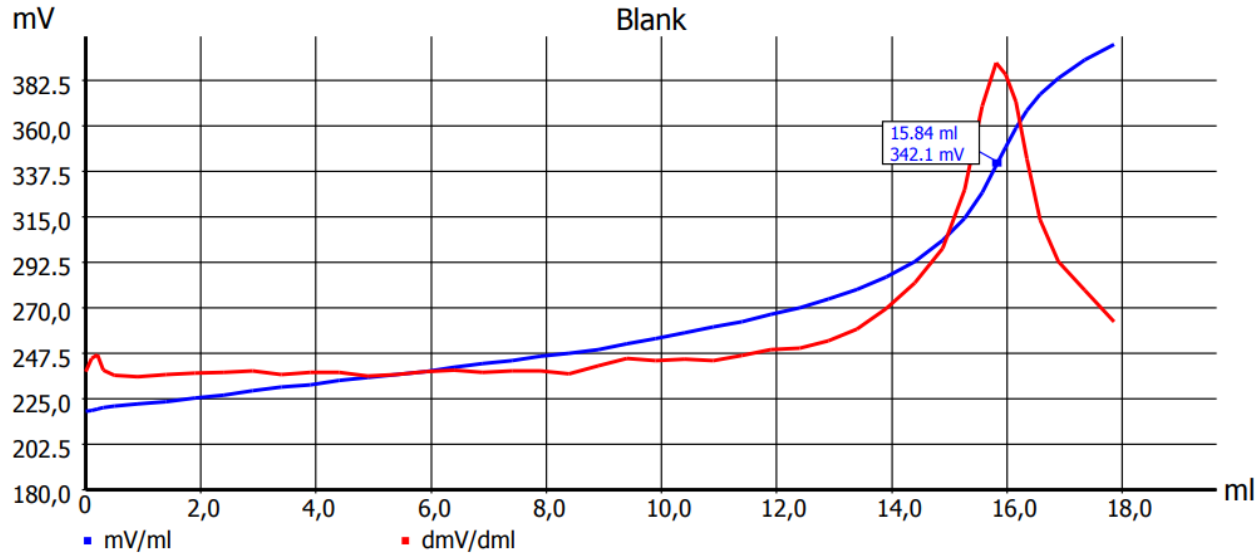
10 ml of the pH10 – buffer solution were placed in a 100 ml glass beaker (tall form), 15 ml of a 0.004 mol/l SDS – solution added and filled up to 50 ml with distilled water. 0.5 ml Triton X solution are added.
The solution is titrated with 0,004 mol/l Hyamin 1622 (or cetylpyridinium chloride) to an equivalence point or up to a maximum volume.

Sample preparation

The amount of sample is chosen so that about 0,02 – 0,04 mmol of the cationic component are contained. The amount of sample is weighed in a 100 ml glass beaker (tall form), 10 ml of the pH10 – buffer solution are added and the mixture is filled up to 40 ml with distilled water. Then, 15ml of 0.004 mol/l SDS solution are added. 0.5 ml Triton X solution are added.
This mixture is titrated with 0,004 mol/l Hyamin 1622 (or cetylpyridinium chloride) to an equivalence point or up to a maximum volume.

Titration parameter back titration

Blank value



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	0 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	weak	Titration direction	increase
Pretitration	off	Delay time	0 s
End value	off		
EQ	on	Slope value	80
Max. titration volume	20 ml		
Dosing speed	100%	Filling speed	30 s

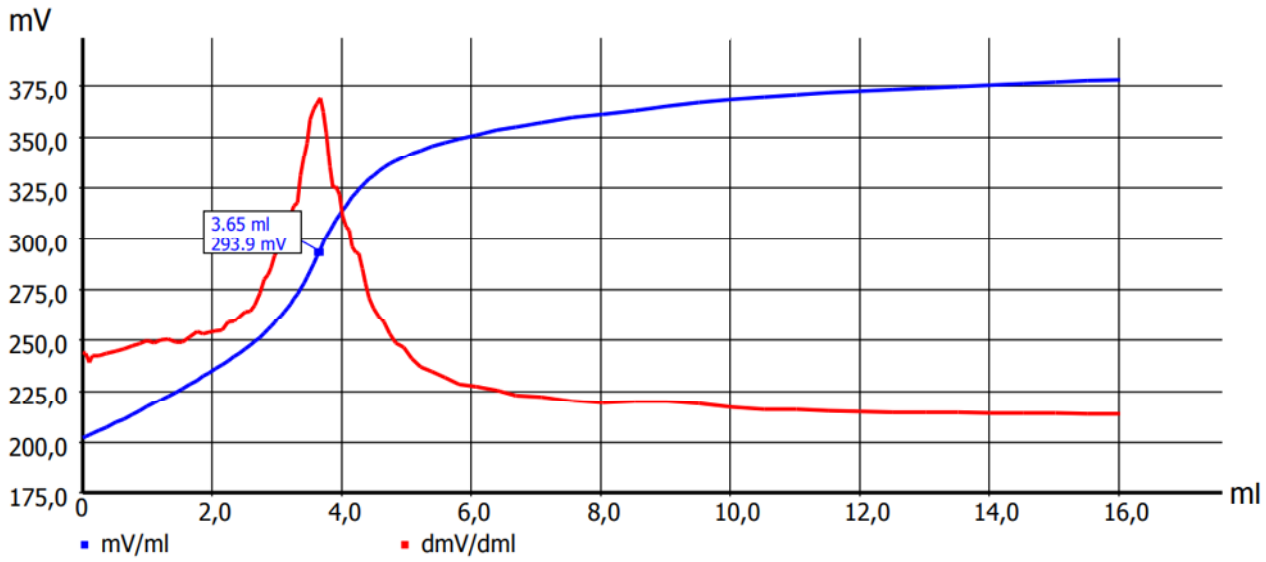
Tip from practice: If the first derivative of the titration curve at the EQ is very noisy, the smallest step size can be increased to 0.1 ml. If the EQ is not recognized despite the detectable inflection point (the titrator titrates up to the maximum volume), the slope value must be reduced.

Calculation:

$$ml = EQ1$$

The result is saved in a global memory, e.g. M01. We recommend to use statistics = 3.

Sample titration



For the sample titration, the same parameters are used as for the blank determination.

Calculation:

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

B	M01	Blank value, saved in global Memory M01
EQ1		Consumption of titrant until first Equivalence point
T	WA	Exact concentration of the titrant, readed from the Exchange Unit
M	354	Molecular mass of Benzalkoniumchloride
W	man	Weight of the sample
F1	0.1	Conversion factor
F2	1	Conversion factor

Any questions? Please contact the application team:

Xylem Analytics Germany Sales GmbH & Co. KG, SI Analytics
Hattenbergstraße 10
D-55122 Mainz, Germany
Telefon: + 49 6131 66 5126
Fax: + 49 6131 66 5101
E-Mail: titration@si-analytics.com

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