Titer determination of Sodium tetraphenylborate (STPB)

Description

This application report describes the general procedure for the titer determination of sodium tetraphenylborate (STPB) for surfactant titration.
The titer is a dimensionless number of approximately 1 for correcting the specified concentration. In the software of the titration devices and the application reports from SI Analytics®, the term "titer" describes the exact concentration in mol/L and not the dimensionless factor.
A Hyamin 1622 solution with known concentration is used to determine the titer. Cetylpyridinium chloride or Papaverine hydrochloride can also be used instead.
Since the reaction in NIO surfactant titration is not always stoichiometric, the determination of a factor instead of the concentration is also described here. The factor determination differs significantly from the titer determination - here no reference material is titrated, but the surfactant to be titrated or a similar reference surfactant. It is calculated how many mg of the substance to be determined corresponds to 1 mL titration solution.

Devices

Titrator	TL 7000 or higher
Exchangeable headt	WA 10
Electrode	TEN 1100 PLH
Cable	L 1 A
Reference Electrode	B 2420+ (NaCl – electrolyte)
Cable	L 1 N
Stirrer	TM 235
Lab accessoires	Glass beakers 150 oder 250 ml

Reagents

1	Sodium tetraphenylborate (STPB) 0.004 – 0.01 mol/L
2	Barium chloride – solution 0.1 mol/L (only for factor determination)
3	Sodium chloride - solution 3 mol/L
4	Hyamin 1622 solution 0.004 mol/L
5	Gum Arabic
6	Polyvinylalkohol
7	Distilled water
All reagents should be in analytical grade or better.	

Titration procedure

Reagents

Hyamin 1622
Available as ready-to-use solution.

Barium chloride solution 0.1 mol/L
24.43 g of barium chloride dihydrate are dissolved in dist. Water and made up to 1.0 liter.

NaCl 3 mol / L
17.53 g of sodium chloride are dissolved in dist. Water and made up to 100 mL.

Polyvinyl alcohol 0.5%
0.5 g polyvinyl alcohol is placed into approx. 80 mL hot dist. water at 80-90° C and dissolved with stirring. After the solution has cooled down it is made up to 100 mL.

Gum arabic solution 5%
5 g gum arabic is placed into approx. 80 mL hot water at 80-90° C and dissolved with stirring. After the solution has cooled down, 1 ml of 30% formaldehyde is added and made up to 100 ml.

Cleaning and handling the electrodes

TEN 1100
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. The electrode is stored 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 0,2 mL Sodium tetraphenylborate (STPB) (0.01 mol/L) in 80mL Water.

B 2420+

The transport lock (the transparent silicone ring above the ground joint diaphragm) must be removed before use. The electrolyte in the electrode must be replaced with NaCl 3 mol/L. For this purpose, the refill opening is opened and the ground joint diaphragm is pushed upwards so that the electrolyte can flow out. The electrode is rinsed several times with NaCl 3 mol / L and then filled with the NaCl solution. The electrode is stored in NaCl 3 mol / L.

Titer determination with Hyamin 1622

Sample preparation

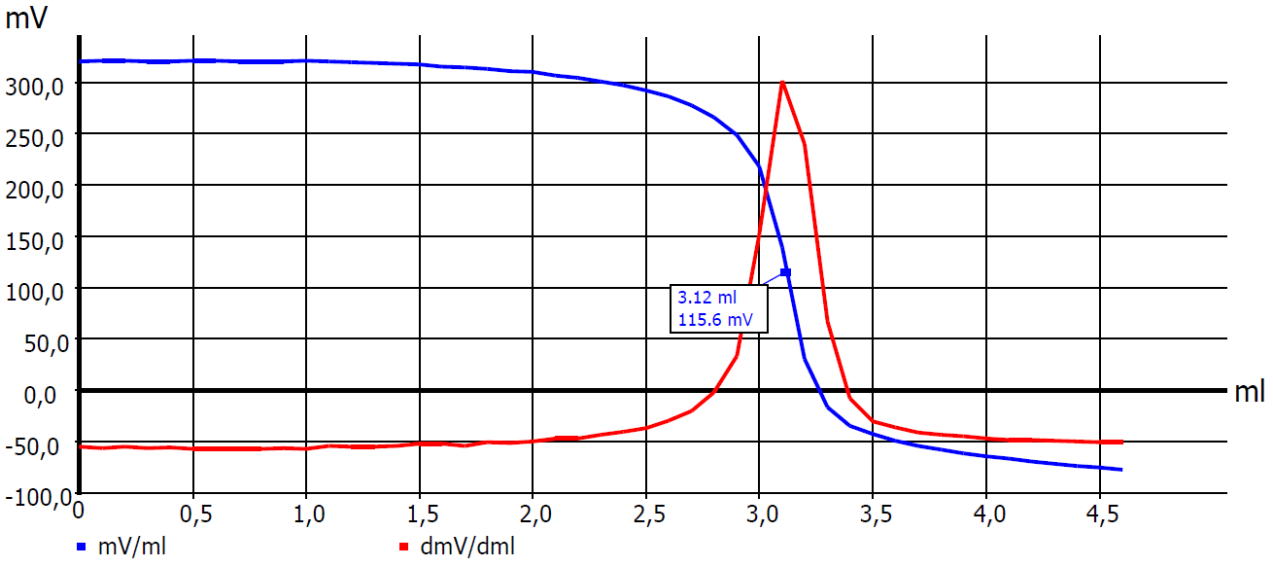
The amount of Hyamin 1622 solution 0.004 mol / L depends on the concentration of the STPB solution. The amount should be chosen so that about 5 - 10 mL are used. The required amount can be estimated using this rule of thumb:

$$V\ [mL] = 1250 * conzentration[mol/l]$$

For titer determination of a 0.01 mol/L STPB solution, 12.5 mL Hyamin 1622 0.004 mol/L are placed in a 250 mL beaker, 1 ml Polyvinyl alcohol solution 0.5% or gum arabic solution 5% are added and made up to 150 mL with dist. water. Then it is titrated to an equivalence point with sodium tetraphenylborate (STPB).

Titration parameter

Titer determination



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

Calculation:

$$T\ [mol/l] = \frac{V * F2}{(EQ - B) * M * F1}$$

B	0	Blank value
V	man	Amount [mL]
F2	0,004	concentration of the Hyamin 1622 solution
EQ1		Consumption at first EQ
M	1	Molar mass
F1	1	Conversion factor

It is recommended to write the exact concentration T to the Exchangable Unit (WA) automatically.

Factor determination with a surfactant

Sample preparation

The surfactant to be determined is used to determine the factor. If this surfactant is not available in sufficient purity, a similar surfactant can be used.

The factor determination should be repeated several times with different weights. The weights used should cover the entire range of the expected content of the sample so that the linearity of the determination can be checked. The amount should be selected so that the consumption is about 5 - 10 mL. The required amount can be estimated according to this rule of thumb and adjusted if necessary:

$$W\ [g] = 3{,}5 * Konzentration[mol/l]$$

Approx. 5 - 50 mg (0.02 - 0.06 mmol) non-ionic surfactant are dissolved in dist. water and 10 mL barium chloride 0.1 mol/L are added. 1 ml of polyvinyl alcohol 0.5% or gum arabic 5% are added and the mixture is made up to 150 ml with dist. Water. Then it is titrated to an equivalence point with sodium tetraphenylborate (STPB).

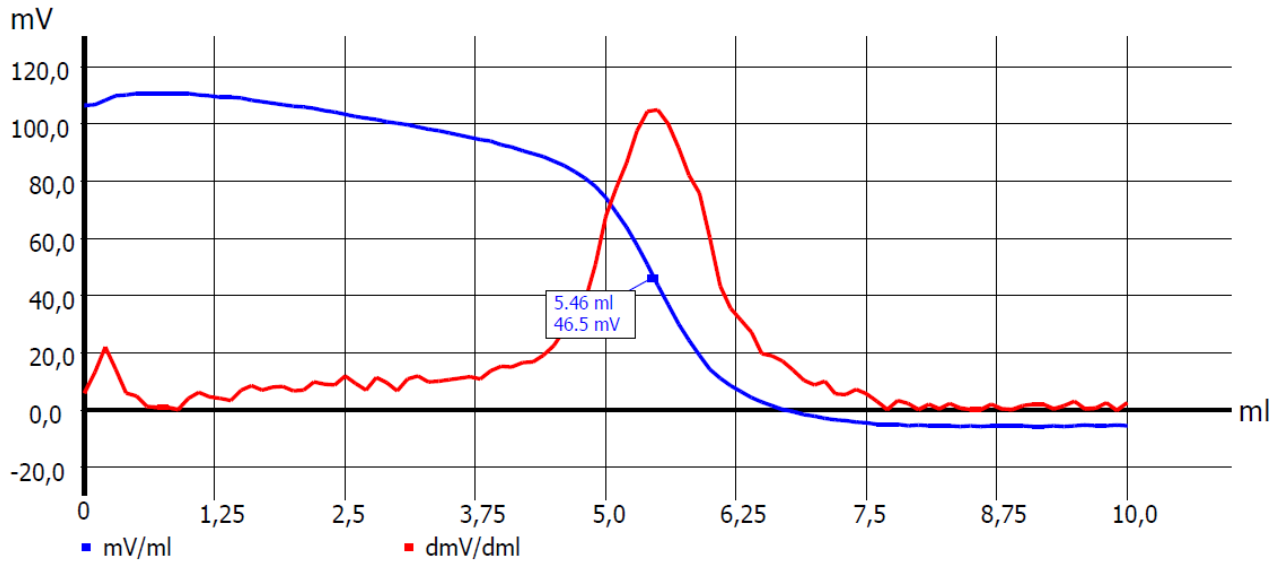
In the case of poorly soluble samples, the solubility of the sample can be improved by adding a little amount Methanol or Ethanol. Attention: Methanol and Ethanol reduce the life time of the electrode, the addition should not exceed 5%.

Due to the very small amount of reference material, it is very difficult to weigh. The following method is used here: 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. An aliquot of A is weighed out of this solution for titration. The amount of sample contained is calculated using the following formula:

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

Titration parameter

Factor determination



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

Calculation:

$$Factor\ T\ [mg/mL] = \frac{W * F2}{(EQ - B) * M * F1}$$

B	0	Blank value
W	man	Amount [mL]
F2	1000	Conversion factor
EQ1		Consumption at first EQ
M	1	Molar mass
F1	1	Conversion factor

It is recommended to write the factor T to the Exchangable Unit (WA) automatically.

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