SI Analytics

a **xylem** brand

Application

Determination of betaine, alkyl-betaine, cocamidopropyl-betaine with perchloric acid

Application

Appliances

- Titrator: TL 7000

Electrodes

- Electrodes: N 6480 Eth

Reagents

- Titrant: Perchloric acid ($HClO_4$) = 0.1 mol/l in 1,4-Dioxane

  (in a 500 ml volumetric flask with 300 ml Dioxan add slowly 5,6 ml Perchloric acid 60 % p.a. and fill up with Dioxane to 500 ml. The solution is standardized with Kaliumhydrogenphthalate in 60 ml Acetic acid similar to HClO4 in glacial acetic acid)

- Methanol p.a.

- Methyl-glycol p.a. (Ethylene glycol monomethyl ether)

- Basification solution (4 g Sodium hydroxide p.a. and 8 g Sodium acetate p.a. dissolved and fill up to 100 ml dest. water)

Use

The method descripts the non-aqueous titration of Betaine in methanol/methyl-glycol with perchloric acid 0.1 mol/l in Dioxane. The sample is dissolved in methanol and basified with NaOH/NaAcetate solution to deprotonating all acids compounds contain the sample. After a reaction time the methyl glycol is added and the sample is titrated with $HClO_4$ in Dioxane. The complete curve give three EQs:
EQ1 is the NaOH excess,
EQ2 is the Na-Acetate and acids or amines contains the sample,
EQ 3 is the Betaine. The Betain concentration would them calculated EQ3-EQ2.
The first EQ is for the Betaine determination not important. The method incudes a pretitration of 6 ml to negate the first EQ (NaOH)! This simplifies the evaluation.
The rest of the titration curve shows two EQs, the Betaine is calculated as the difference EQ2-EQ1.

Description

For Samples with 30 % (BASF I):
Into a 150 ml baker weigh 0,6000 g Betaine sample, dissolve in 20 ml methanol, added 0,5 ml basification solution and stir for 5 to 8 minutes. Then add 20 ml methanol and 60 ml methyl-glycol. The sample is titrated with $HClO_4$ 0.1 mol/l in Dioxane. The Method includes a pretitration of 6 ml with 30 sec waiting time, it's important to overtitrate the first EQ (NaOH) for a correct calculation. The electrode is rinsed first with dest. water then with alcohol before a next and first titration.

For Samples with 1 % (BASF II):
Into a 150 ml baker weigh 5,0000 g Betaine sample, dissolve in 40 ml methanol, add 0,5 ml basification solution and stir for 5 to 8 minutes. Then add 60 ml Methyl glycol. Titrate the Sample with $HClO_4$ 0.1 mol/l in Dioxane. The Method includes a pretitration of 6 ml with 30 sec waiting time, it's important to overtitrate the first EQ (NaOH) for a correct calculation. The electrode is rinsed first with dest. water then with alcohol before a next and first titration.
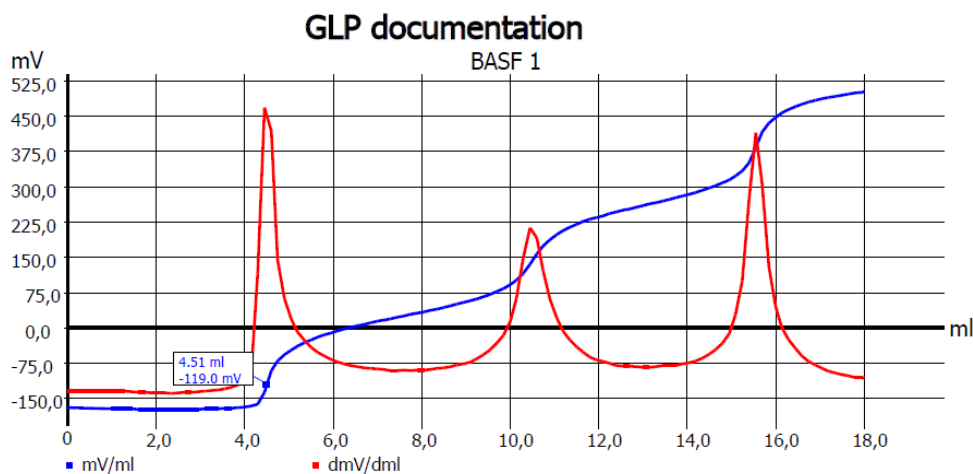
Application

Examples / Results

BASF (I)			
Weight in g	EQ1 in ml	EQ 2 in ml	Betaine in %
0,6047	10,532	15,590	29,72
0,6201	10,518	15,713	29,77
0,6069	10,542	15,615	29,70
		Mean.	**29,73**
		RSD in %	0,12

BASF (II)			
Weight in g	EQ1 in ml	EQ 2 in ml	Betaine in %
5,0301	11,294	13,1	1,28
5,0961	11,284	13,107	1,27
5,0661	11,381	13,131	1,23
		Mean.	**1,26**
		RSD in %	2,10

Application

BASF I (complete curve):

BASF I (method with 6 ml pretitration):
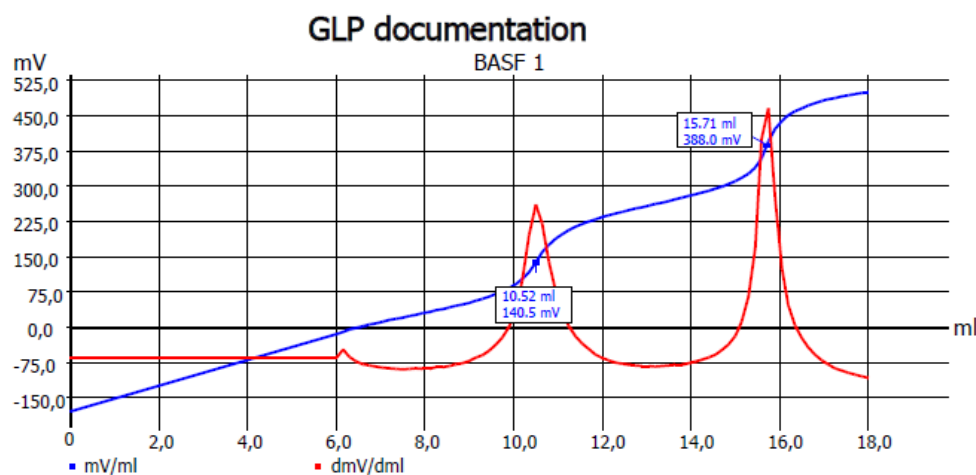
Method data

Method name:	Betaine with HClO4	Titration duration:	15 m 29 s
End date:	23.06.16	End time:	17:43:54

Titration data

Sample ID:	BASF 1	Weight:	0.62010 g
Start mV (A):	-175.5 mV	End mV (A):	499.8 mV

EQ1:	10.518 ml / 140.5 mV	EQ1:	10.518 ml
EQ2:	15.713 ml / 388.0 mV	Betaine:	29.77 %

BASF II (complete curve):
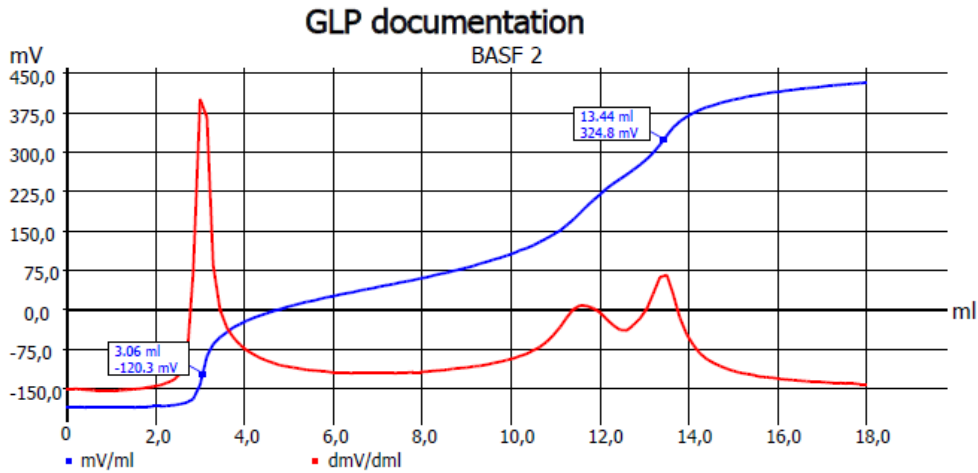
BASF II (method with 6 ml pretitration):
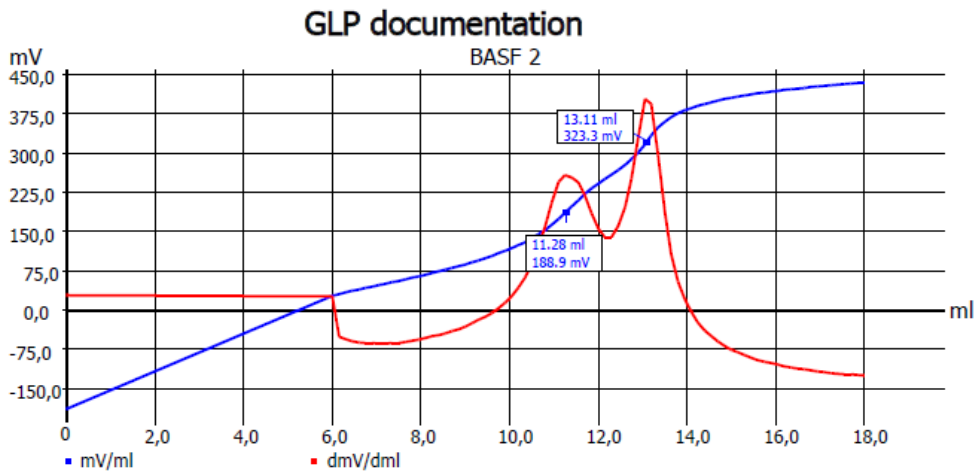
Method data

Method name:	Betaine with HClO4	Titration duration:	10 m 46 s
End date:	24.06.16	End time:	16:33:42

Titration data

Sample ID:	BASF 2	Weight:	5.09610 g
Start mV (A):	-183.5 mV	End mV (A):	434.2 mV

EQ1:	11.284 ml / 188.9 mV	EQ1:	11.284 ml
EQ2:	13.107 ml / 323.3 mV	Betaine:	1.27 %

Application

Hints

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