Determination of hydroxyl value (DIN EN ISO 4629-2)

Description

The determination described in this report is based on the standard DIN EN ISO 4629-2:2016. This standard is valid for resins, binders for coating materials, primary alcohols, glycol und fats. For samples with a high acid number, it is necessary to take this into account when calculating the result. The acid number can be determined, for example, according to DIN ISO 2114 (see application "Acid number in polyester DIN ISO 2114"). The results are expressed as mg KOH/g sample.

Instruments

Titrator	TL 7000 or higher
Interchangeable unit	WA 50
Electrode	N 6480 eth (electrolyte L 5034)
Cable	L 1 A
Stirrer	Magnetic stirrer TM 235
Titration tip	TZ 1643
Lab accessory	Glass beaker 150 ml, high form, without spout
	Watch glass
	Magnetic stirring rods

Reagents

1	KOH in ethanol, 0.5 mol/L
2	Deionized water
3	N-Methyl-2-Pyrrolidon
4	Acetic anhydride
5	4-N-Dimethylaminopyridine
6	N-Methyl-2-pyrrolidone
All reagents should be of analytical grade or better.	

Titration procedure

Reagents

KOH in ethanol 0.5 mol/L
0.5 mol/L KOH in ethanol is available as a ready-to-use solution.
The solution must be protected against CO_2 with a CO_2 absorbent like soda lime.
The titer determination is done as described in the application note “Titer KOH”.

Acetylation reagent
500 mL of N-methyl-2-pyrrolidone are placed in a 1000 mL volumetric flask, 110 mL of acetic anhydride are added slowly and mixed. Then the mixture is made up to 1000 mL with N-methyl-2-pyrrolidone. The solution is stored in amber glass bottles.

Catalyst solution
25 g of 4-N-Dimethylaminopyridine are dissolved in 2.5 L of N-Methyl-2-pyrrolidone. The solution is stored in an amber bottle.

Cleaning of the electrode

The electrode can be cleaned with water or alcohol.
The electrode is stored in a solution of 1.5 mol/L LiCl in Ethanol (or, if another electrolyte is used, in this electrolyte solution).

Blank value

For the blank determination, 25 mL catalyst solution and 10 mL acetylation reagent are added to a beaker and covered with a watch glass. If solubilizers are added to dissolve the sample, they must also be added in the same amount for the blank determination. After stirring for 15 minutes, 3 ml dist. Water are added, the beaker is covered again and the solution is stirred for further 12 minutes. After the reaction time the titration to an equivalence point is started.

Sample preparation

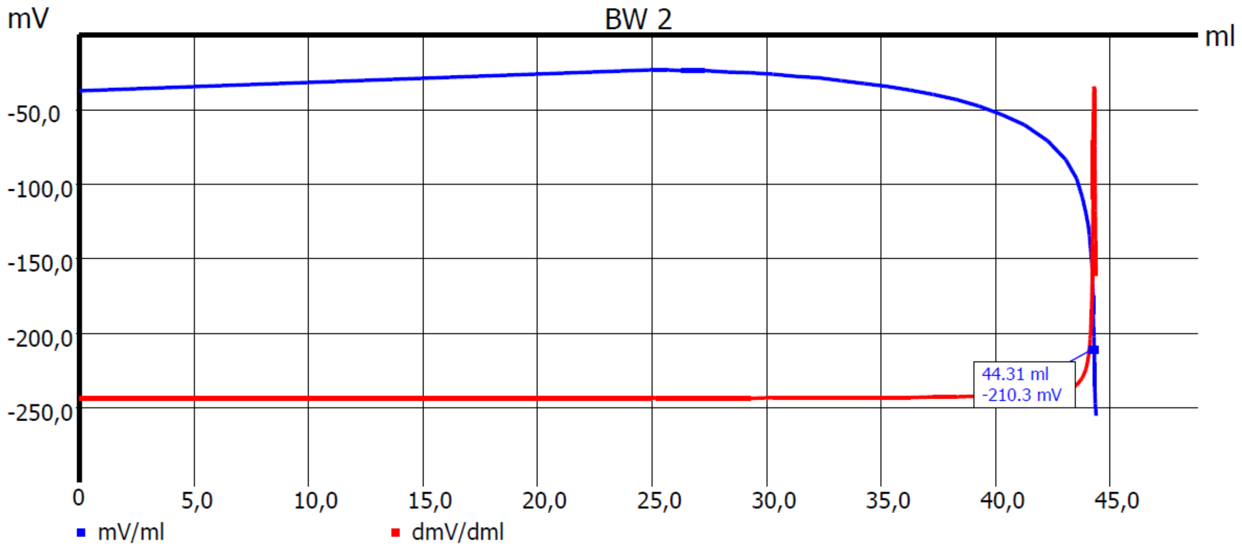
The sample is weight in into a beaker. The amount of sample weight is adjusted to the expected hydroxyl value.

Expected Hydroxyl no.	Sample weight [g]
0 – 15	10
15 – 20	6
20 – 25	5
25 – 30	4
30 – 40	3
40 – 50	2.5
50 – 100	2
100 – 200	1.2
200 – 300	0.6
300 – 400	0.4
400 – 500	0.3
500 – 750	0.2
750 – 1000	0.15
>1000	0.1

To the sample 25 mL of catalyst solution and 10 mL of acetylation reagent are added. The beaker is covered with a watch glass and stirred. If the sample does not dissolve, additional solubilizers such as dichloromethane or chloroform can be added. Once the sample is dissolved, a reaction time of 15 minutes begins. After the reaction time, 3 mL dist. water are added, the beaker is covered again and the solution is stirred for a further 12 minutes. After the reaction time the titration to one equivalence point is started.

Titration parameter

Blank value



Default method	-		
Method type	Automatic Titration		
Modus	Dynamic		
Measured value	mV		
Measuring speed / drift	User defined	Minimum holding time	3 s
		Maximum holding time	15 s
		Measuring time	2 s
		Drift	10 mV/min
Initial waiting time	0 s		
Dynamic	Steep	Max step size	-
		Slope max ml	-
		Min. step size	-
		Slope min. ml	-
Damping	Average	Titration direction	decrease
Pretitration	25 mL	Delay time	60 s
End value	Off		
EQ	On(1)	Slope value	Steep
Max. titration volume	50 mL		
Dosing speed	100%	Filling speed	30 s

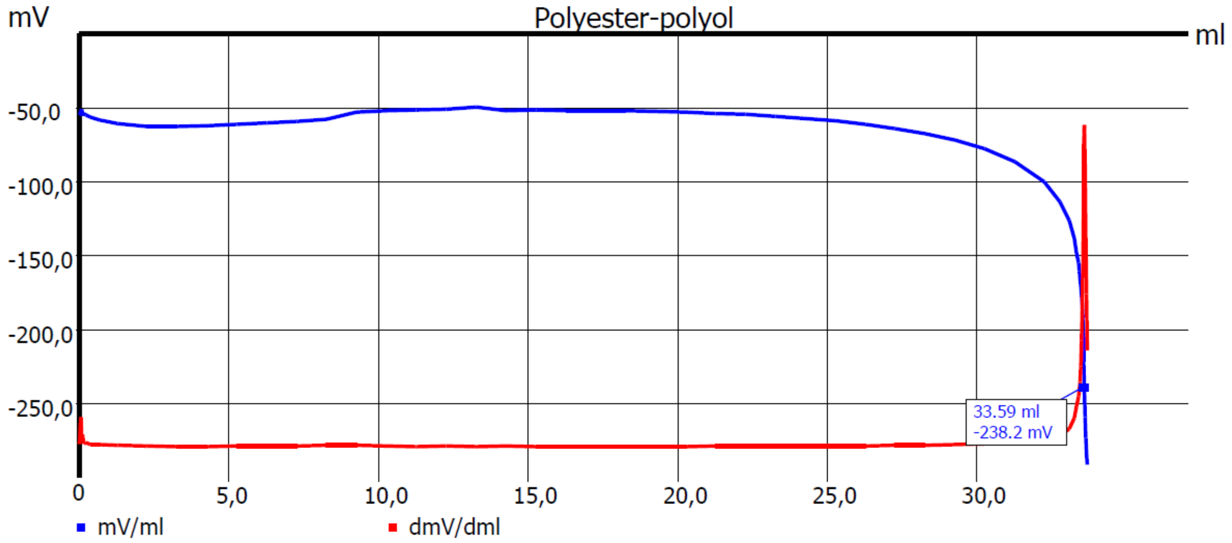
Calculation:

$$Result\ mL = EQ1$$

EQ1		Consumption of titrant at the first Equivalence point

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

Sample titration



Default method	-		
Method type	Automatic Titration		
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Measuring speed / drift	User defined	Minimum holding time	3 s
		Maximum holding time	15 s
		Measuring time	2 s
		Drift	10 mV/min
Initial waiting time	0 s		
Dynamic	Steep	Max step size	-
		Slope max ml	-
		Min. step size	-
		Slope min. ml	-
Damping	Average	Titration direction	Decrease
Pretitration	Off	Delay time	0 s
End value	Off		
EQ	On(1)	Slope value	Steep
Max. titration volume	50 mL		
Dosing speed	100%	Filling speed	30 s

Calculation:

$$Result\ [mg\ KOH/g] = \frac{(B - EQ1) * T * M}{W} + F3$$

B	M01	Blank value
EQ1		Consumption of titrant at the first Equivalence point
T	WA	Actual Concentration of the titrant
M	56,1	Molecular weight
W	Man	Sample weight in g
F1	1	Conversion factor
F2	1	Conversion factor
F3		Acid number in mg KOH/g, e.g. according to DIN ISO 2114

For F3 either a manual value, a fixed value or a global memory can be used. For samples with a very low acid number, F3 = 0 can also be used.