

## Determination of hydroxyl value (DIN EN ISO 4692-2) and acid value (DIN EN ISO 2114)

### Description

The determination described in this report is based on the standards DIN EN ISO 4629-2:2016 (Hydroxyl value) and DIN EN ISO 2114:2002-06 (acid value). These standards are valid for resins, binders for coating materials, primary alcohols, glycol und fats. The results are expressed as mg KOH/g sample.

### Instruments

Titration	TL 7000 or higher
Interchangeable unit	WA 10 (acid value) und WA 50 (hydroxyl value)
Electrode	N 6480 eth (electrolyte L 5034)
Cable	L 1 A
Stirrer	Magnetic stirrer TM 235
Titration tip	Long version TZ 1643 (2 x) is required
Lab accessory	Glass beaker 150 ml, high form, without spout
	Watch glass
	Magnetic stirring rods

### Reagents

1	KOH in ethanol, 0.1 mol/L (Titration reagent acid value)
2	KOH in ethanol, 0.5 mol/L (Titration reagent hydroxyl value)
3	Toluene
4	Ethanol
5	Deionized water
6	N-Methyl-2-Pyrrolidon
7	Acetic anhydride
8	4-N-Dimethylaminopyridine
9	N-Methyl-2-pyrrolidone
All reagents should be of analytical grade or better.	

## Titration procedure

### Reagents

#### Acid value

For the solvent mix 2 parts of toluene with one part of ethanol.

#### Hydroxyl value

##### Acetylation reagent

500 ml of N-Methyl-2-pyrrolidone are given into a 1000 ml volumetric flask. 110 ml of acetic anhydride are gently added and mixed. Afterwards the flask is filled up to the mark with N-Methyl-2-pyrrolidone. The solution is stored in an amber bottle.

##### 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.

### Sample preparation

#### Acid value

The sample is weight in into a 150 ml beaker. The amount of sample weight is adjusted to the expected acid value.

Expected acid value	Sample weight [g]
0 – 5	≥16
5 – 10	8
10 – 25	4
25 – 50	2
50 – 100	1
>100	0.7

To the sample 50 ml of solvent is added. If the sample does not dissolve completely while stirring 25 ml of a solubilizing agent like acetone or chloroform might be added. After dissolving the titration can be started.

### Hydroxyl value

The sample is weight in into a 150 ml beaker. The amount of sample weight is adjusted to the expected hydroxyl value.

Expected Hydroxylzahl	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

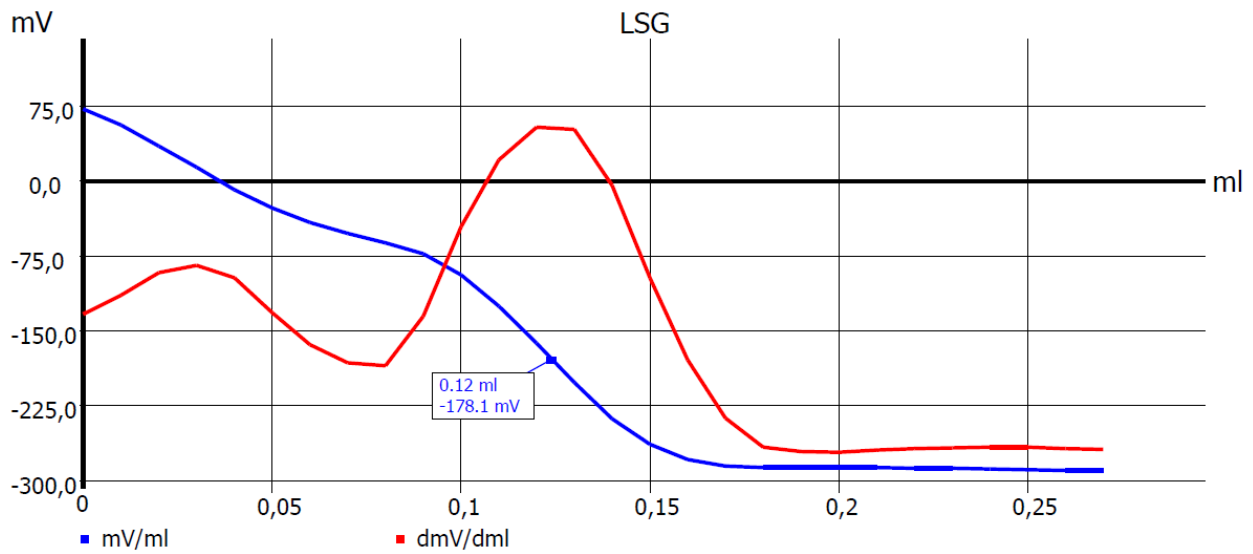
To the sample 25 ml of the catalyst solution and 10 ml of the acetylation reagent are added. The beaker is covered with a watch glass and stirred. If the sample does not dissolve completely 25 ml of a solubilizing agent like dichlormethane or chloroform might be added. As soon as the sample is dissolved a reaction time of 15 minutes starts. After these 15 minutes 3 ml deionized water is added, the beaker is covered again and stirred for another 12 minutes. After the reaction time the titration can be started.

### **Blank value**

Both the hydroxyl and the acid value require a blank determination. Therefore the titration is carried out in the same way as explained above without addition of the sample. A double determination is recommendable (Statistic: 2).

## Titration parameter

### Blank acid value



Default method	-		
Method type	Automatic Titration		
Modus	Linear		
Measured value	mV		
Measuring speed / drift	15 s	Minimum holding time	-
		Maximum holding time	-
		Measuring time	-
		Drift	-
Initial waiting time	10 s		
Linear steps	0.01 ml		
Damping	Strong	Titration direction	decrease
Pretitration	Off	Delay time	0 s
End value	Off		
EQ	Off	Slope value	-
Max. titration volume	0.3 ml		
Dosing speed	100%	Filling speed	30 s

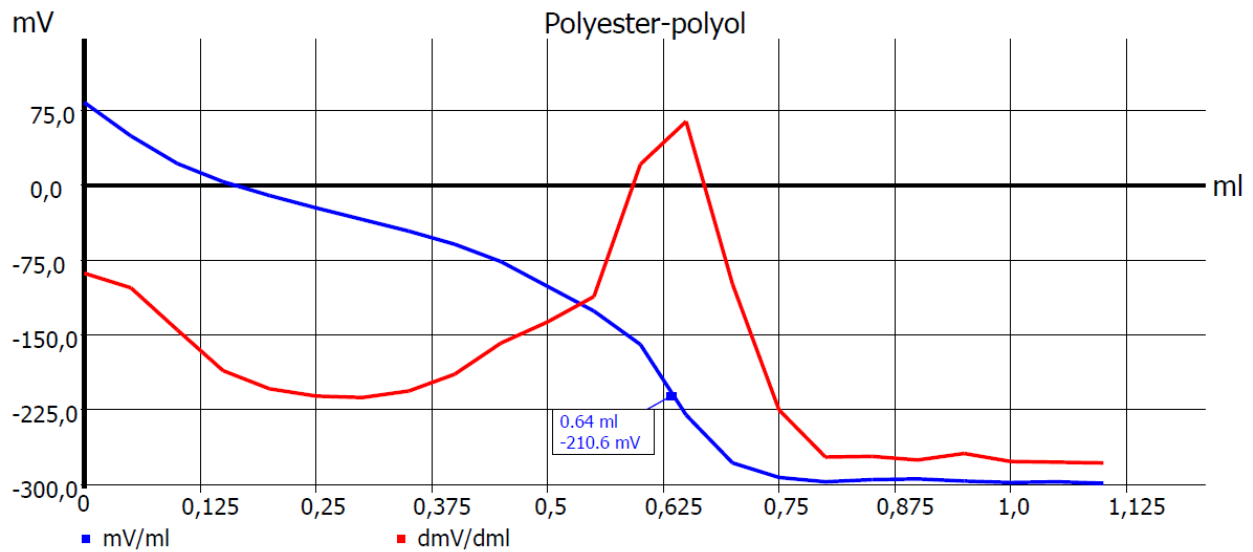
### Calculation:

$$\text{Result ml} = EQ1$$

EQ1		Consumption of titrant at the first Equivalence point
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The result is stored as global variable M01.

### Sample titration acid value



Default method	-		
Method type	Automatic Titration		
Modus	Linear		
Measured value	mV		
Measuring speed / drift	User defined	Minimum holding time	10 s
		Maximum holding time	20 s
		Measuring time	6 s
		Drift	5 mV/min
Initial waiting time	10 s		
Linear steps	0.04 ml		
Damping	Strong	Titration direction	decrease
Pretitration	Off	Delay time	0 s
End value	Off		
EQ	Off	Slope value	-
Max. titration volume	3 ml		
Dosing speed	100%	Filling speed	30 s

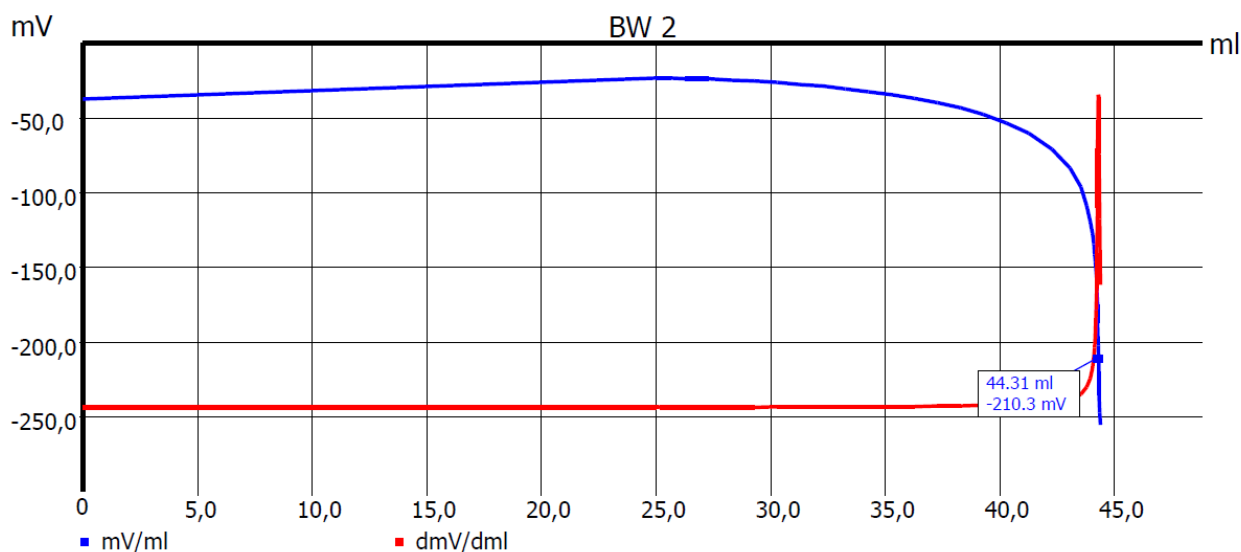
Calculation:

$$Result [mg KOH/g] = \frac{(B - EQ1) * T * M * F1}{W * F2}$$

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	0.1	Conversion factor 1
F2	1	Conversion factor 2

The result is stored as global variable M02.

### Blank hydroxyl 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

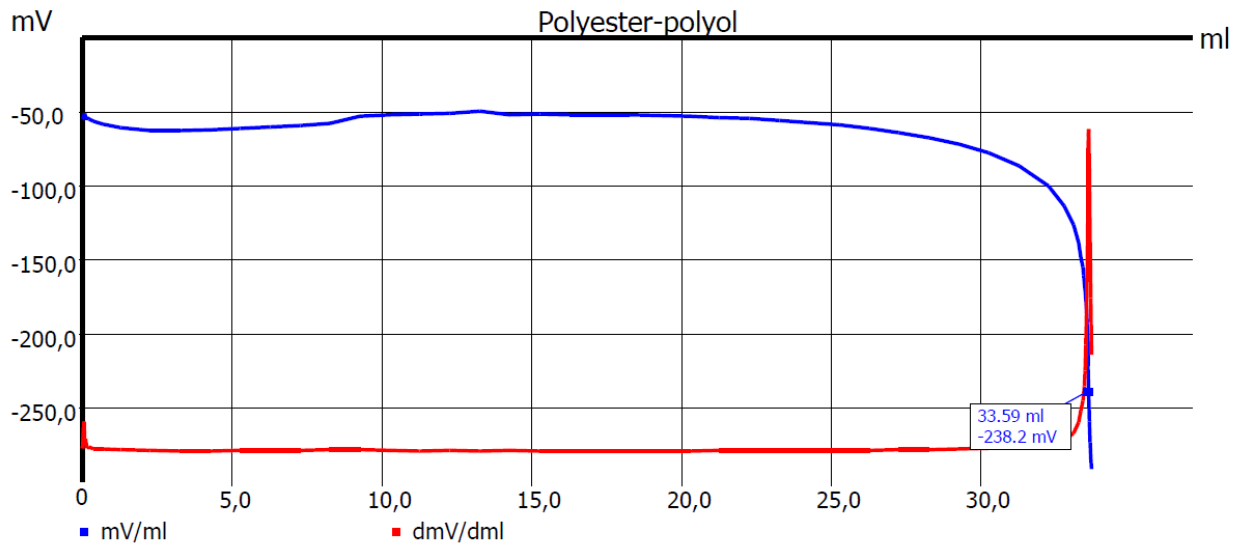
Calculation:

$$Result\ ml = EQ1$$

EQ1		Consumption of titrant at the first Equivalence point
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The result is stored as global variable M03.

### Sample titration hydroxyl 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	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:

$$\text{Result [mg KOH/g]} = \frac{(B - EQ1) * T * M}{W} + M02$$

B	M03	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
M02		Acid value

Any questions? Please contact the application team:

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