

Determination of OH-Value according to ASTM 1899

Description

Polyurethanes are produced by a polyaddition reaction of diisocyanates and di- or polyols. Next to the NCO value, which describes the amount of isocyanate groups, the hydroxyl- or OH number (OHV) is an important parameter in the production of polyurethanes. Also in the production of paints, varnishes and resins, the content of reactive OH groups plays an important role for the product properties.

This application note describes the determination of the OHV by titration with an alcoholic tetrabutylammonium hydroxide solution after the reaction of the OH groups with p-toluenesulfonyl isocyanate (TSI) to form an acidic carbamate. It is titrated to 2 equivalence points, from the difference, the OHV is calculated. This method only determines primary and secondary OH groups.

The result is calculated as $\text{mg}_{(\text{KOH})} / \text{g}$.

Instruments

Titration	TL 7000 or higher
Electrode	N 6480 eth
Cable	L 1 A
Stirrer	Magnetic stirrer TM 235 or similar
Lab accessory	Plastic beaker 100 ml
	Watch glass
	Magnetic stirrer bar 30 mm

Reagents

1	Tetrabutylammonium hydroxide 0,1 mol/l in Isopropyle alcohol / Methanole
2	p-Toluenesulfonyl-isocyanate (TSI)
3	Acetonitrile
4	Distilled water
5	Isopropyl alcohol
All reagents should be of analytical grade or better.	

Titration procedure

Reagents

TSI reagent

40.0 ml of TSI are dissolved in 300 ml of acetonitrile and made up to 0.5 l.

The reagent is sensitive to moisture and can only be used for about 4 weeks.

Cleaning of the electrode

The electrode is cleaned with ethanol or acetone and then rinsed with water.

Sample preparation

The sample is weighed into a 100 ml beaker and dissolved in 10 ml acetonitrile. To improve the solubility of the sample, some of the chloroform or toluene can be added.

10 ml of the TSI reagent are added; the beaker is covered with a watch glass and gently stirred on a magnetic stirrer. After 5 minutes reaction time, 0.5 ml of water is added to remove the excess of TSI.

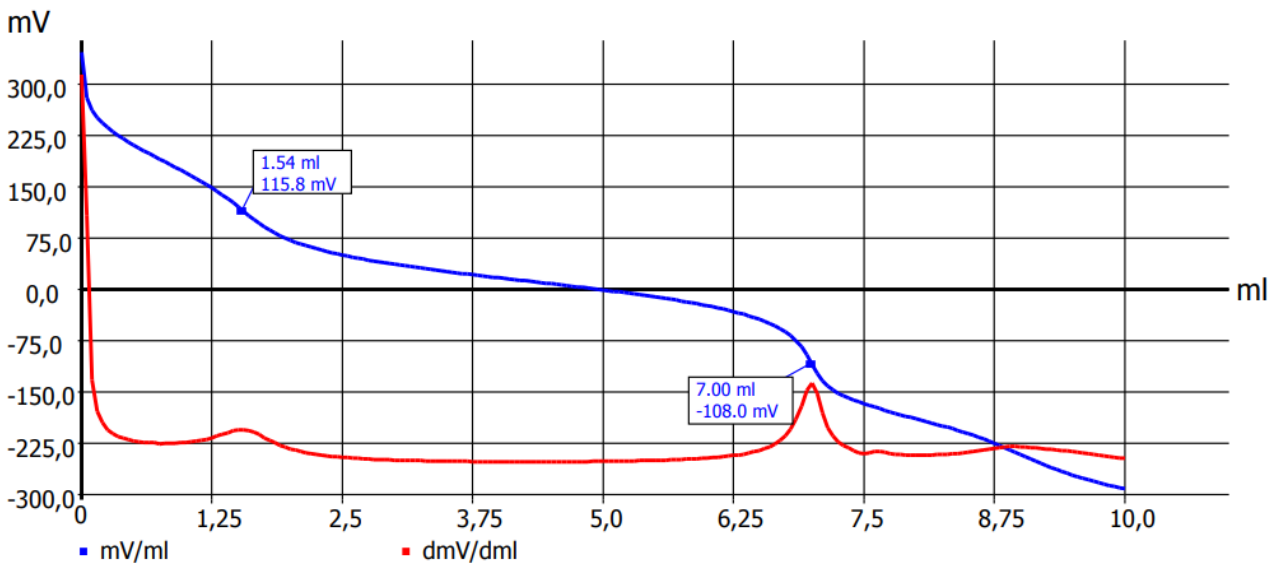
After a further minute, the solution is made up to 50-60 ml with acetonitrile and titrated with TBA-OH 0.1 mol/l to 2 equivalence points.

The required amount of sample can be estimated according to this rule of thumb:

$$W(g) = \frac{40}{\text{expected OH} - \text{Value}}$$

Titration parameter

Sample titration



Default method	--		
Method type	Automatic titration		
Modus	linear		
Measured value	mV		
Measuring speed / drift	User defined	Minimum holding time	7 s
		Maximum holding time	20 s
		Measuring time	3 s
		Drift	20 mV/min
Initial waiting time	0 s		
Linear Steps	0.05 ml		
Damping	none	Titration direction	decrease
Pretitration	off	Delay time	0 s
End value	off		
EQ	On (2)	Slope value	60
Max. titration volume	10 ml		
Dosing speed	100%	Filling speed	30 s

Calculation:

$$OHV [mg/g] = \frac{(EQ2 - EQ1) * T * M * F1}{V * F2}$$

EQ1		Consumption of titrant at first Equivalence point
EQ2		Consumption of titrant at second Equivalence point
T	WA	Actual concentration of the titrant
M	56,11	Molecular weight KOH
W	man	sample weight [g]
F1	1	Conversion factor
F2	1	Conversion factor