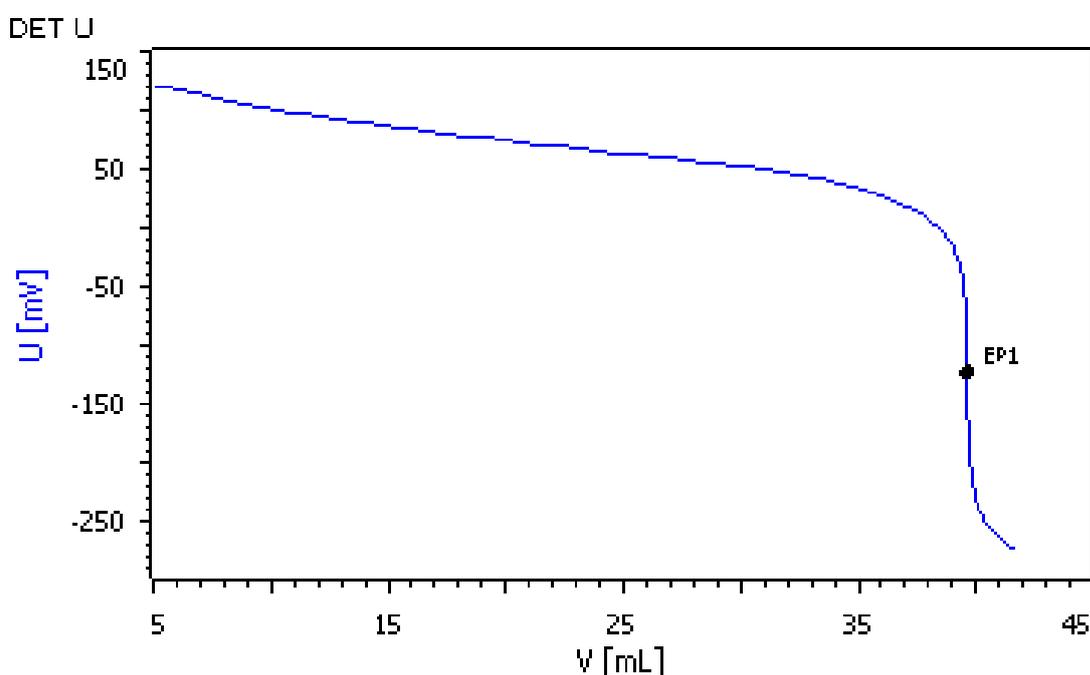


Hydroxyl number in unsaturated polyester resin according to EN ISO 2554



The hydroxyl number indicates the amount of potassium hydroxide in milligrams required to neutralize the acetic acid taken up on acetylation of 1 g of an unsaturated polyester resin (UPR) containing free hydroxyl groups. In this Application Note the determination of the hydroxyl number by automated, potentiometric titration according to EN ISO 2554 using KOH in methanol as titrant is described.

Method description

Sample

Unsaturated polyester resin (UPR)

Sample preparation

No sample preparation is required.

Configuration

907 Titrand	2.907.0010
800 Dosino, 5x	2.800.0010
814 USB Sample Processor (2T/0P)	2.814.0130
Dosing unit 50 mL, 2x	6.3032.250
Dosing unit 10 mL, 2x	6.3032.210
Dosing unit 2 mL	6.3032.120
741 Magnetic stirrer, 2x	2.741.0010
843 Pump station (peristaltic)	2.843.0150
Sample rack, PP, 22 x 120 mL	6.2041.470
Titration head, 3x NS 14	6.1458.040
Sample beaker, PP, 22 x 120 mL	6.1459.300
Titration head with DIS-Cover	6.9914.158
DIS-Cover lids for 120 mL PP beakers	6.9914.164
Solvotrode easyClean, c(TEABr) = 0.4 mol/L in ethylene glycol	6.0229.020

Solutions

Titration	c(KOH) = 0.5 mol/L in methanol, if possible this solution should be bought from a supplier.
Acetylating solution	1.4 g dry toluene-4-sulfonic acid is dissolved in 111 mL anhydrous ethyl acetate. After dissolution 12 mL acetic anhydride are added. The solution is stored under dry atmosphere
Solvent mixture	1-Butanol / Toluene, $\Phi(1\text{-Butanol}) = 66.6\%$ (v/v)
Pyridine/water mixture	Pyridine / Water, $\Phi(\text{pyridine}) = 60.0\%$ (v/v)

Analysis

Approx. 5.6 g UPR is weighed into the sample beaker and placed on the rack. 10 mL acetylating solution is automatically added to the sample. After a reaction time of 45 minutes in a water bath with $50\text{ }^{\circ}\text{C} \pm 1\text{ }^{\circ}\text{C}$, 2 mL deionized water and 10 mL pyridine/water mixture are automatically added. Just before the titration 20 mL solvent mixture is added and the solution is titrated with c(KOH) = 0.5 mol/L until after the equivalence point.

The blank is determined the same way as the sample, just without UPR.

Parameters

Mode	DET U
Start volume	5 mL
Signal drift	50 mV/min
Stirrer speed	8
Max. waiting time	26 s
Meas. point density	4
Min. increment	10 μL
Max. increment	off
EP criterion	5
EP recognition	Greatest, evaluation with window
Lower limit mL	2
Upper limit mL	50
Stop volume	50 mL
Volume after EP	2 mL

Results

Hydroxyl number / (n = 5)	s(rel)
42.60 mg KOH / g	0.5 %

Remarks

The hydroxyl number is finally calculated by taking into account the total acid number (TAN), determined separately by EN ISO 2114. For its determination have a look at AN-T-165.

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