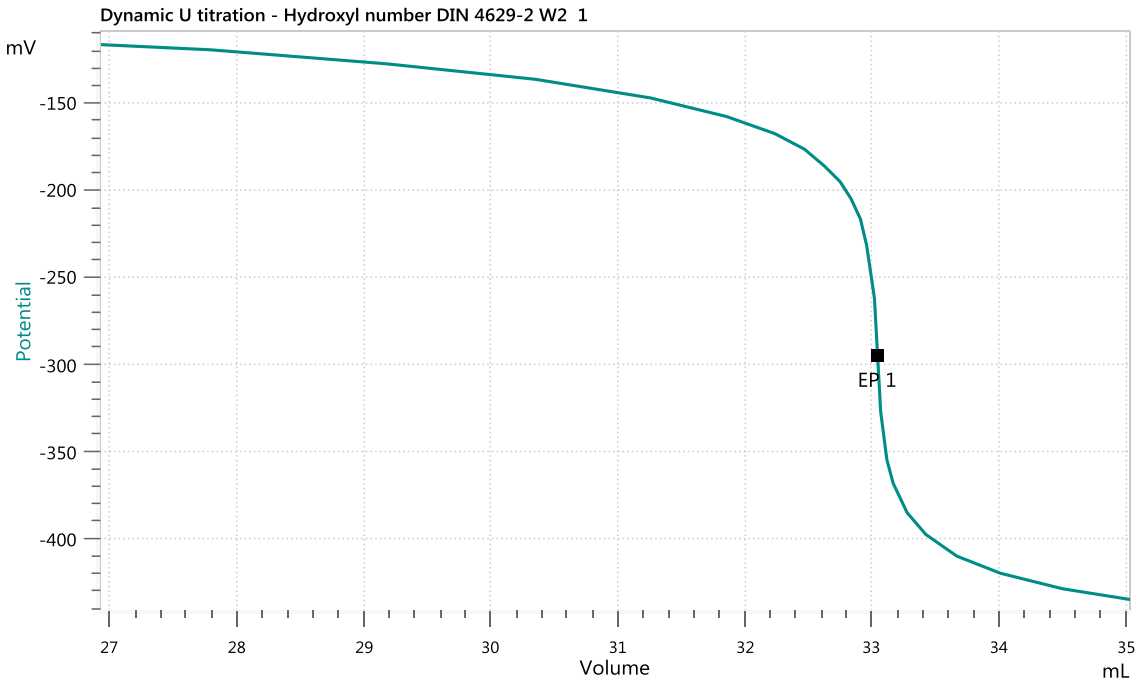
Titration Application Note T-177

Hydroxyl number in binders for paints and varnishes

Pyridine-free, fully automated determination according to EN 4629-2



The hydroxyl number is an important sum parameter for quantifying the presence of hydroxyl groups in a chemical substance. As a key quality parameter, it is regularly determined in various polymers like resins, paints, polyesterols, fats, and solvents. Unlike other standards, EN 4629-2 works pyridine-free and without refluxing at elevated temperatures for a longer time. The determination is based on the catalytic acetylation of the hydroxyl group. It is performed at room temperature, requires only a small sample volumen, and can be fully automated.

This Application Note describes the potentiometric determination of the hydroxyl number in 1-octanol and polyethylene glycol according to EN 4629-2. Using the OMNIS DIS-Cover technique, all sample preparation steps can be fully automated. Furthermore, the use of an OMNIS Sample Robot allows parallel analysis of multiple samples. The average time per analysis for one sample is thus reduced from approximately 49 min to 25 min., considerably increasing productivity in the laboratory.

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Method description

Sample

1-Octanol

Polyethylene glycol (PEG) 1000

Sample preparation

No sample preparation is required.

Configuration

OMNIS Sample Robot S	2.1010.0010
Pick&Place module with stirrer, 2x	2.1014.0110
Peristaltic (4-channel) pump module	2.1016.0110
Gripper fingers 43 – 65 mm	6.02601.010
Titration head 3xNS14 / 4x6.4 mm (P&P), 2x	6.01403.030
OMNIS Titrator Professional without stirrer	2.1001.0310
OMNIS Dosing Module without stirrer, 7x	2.1003.0010
OMNIS 50 mL cylinder unit, 4x	6.03001.250
OMNIS 10 mL cylinder unit, 2x	6.03001.210
OMNIS 5 mL cylinder unit, 2x	6.03001.150
Lid tray for OMNIS Sample Robot S	6.02007.010
Dis-Cover lid for OMNIS 120 mL sample beaker, 12 pieces	6.02710.040
Sample beakers, plastic (PP), 120 mL, 20 pieces, 2x	6.01400.200
Stirring bar / 25 mm, 40x	6.1903.030
Cable MDL PL/SO 1 m, 7x	6.02102.020
Digital measuring module, 2x	6.02100.010
Electrode cable plug-in head Q / plug P, 1.5 m, 2x	6.02104.310
OMNIS Stand-alone license	6.06003.010
OMNIS instrument license, 1x	6.06002.010
dSolvotrode electrolyte c(TEABr) = 0.4 mol/L in ethylene glycol, 2x	6.00203.300

Solutions

Acetylation solution	10% (v/v) Acetic anhydride in 1-Methyl-2-pyrrolidone (NMP) 50 mL acetic anhydride is added into a 500 mL brown-glass flask and the flask filled up to the mark with NMP.
Catalyst solution	1% (w/v) 4-(Dimethylamino)pyridine (DMAP) in NMP 10 g DMAP is weighed into a 1000 mL brown-glass flask and the flask filled up to the mark with NMP.

Analysis

Blank

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

Sample

An appropriate amount of sample is weighted into the titration beaker and 30 mL catalyst solution and 10 mL acetylation solution are added. The beaker is capped with the DIS-Cover lid and the solution is stirred for 15 min. After 3 mL deionized water is added, the beaker is capped again and the solution is stirred for 12 min. The solution is then titrated until after the second equivalence point with c(KOH) = 0.5 mol/L in methanol.

Parameters

Mode	DET U
Pause	30 s
Start volume	0 mL blank, 15 mL sample
Stirring rate	8
Signal drift	50 mV/min
Min. waiting time	15 s
Max. waiting time	30 s
Meas. point distance	4
Min. increment	50 µL
Stop EP	1
Volume after EP	2.0 mL
EP criterion	5
EP recognition	Greatest

Results

Sample	Hydroxyl number in mg KOH/g sample	s(rel) /%
1-Octanol	428.82 (n = 6)	0.4
PEG 1000 (WS2)	126.14 (n = 5)	0.6
PEG 1000 (WS3)	126.75 (n = 6)	0.3

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