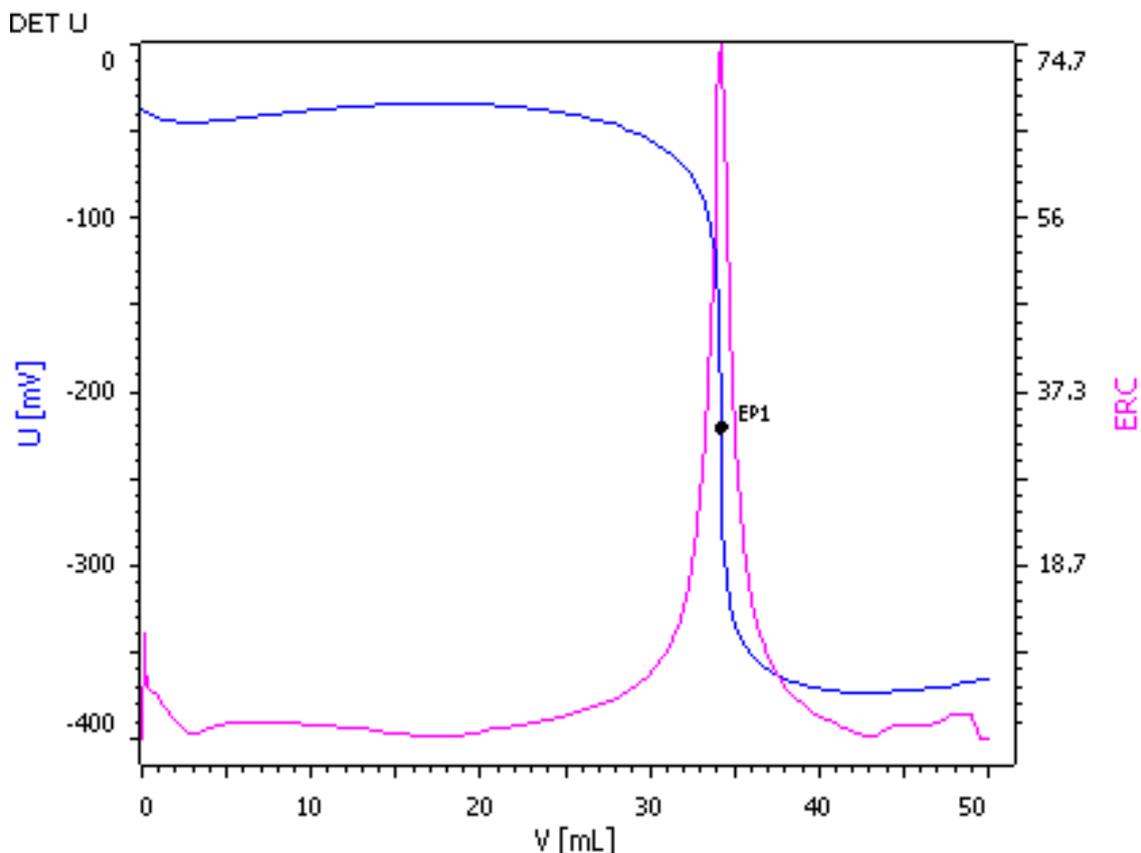


Automated determination of hydroxyl Number (HN) in polyols according to DIN53240-2



The hydroxyl group is an important functional group and knowledge of the hydroxyl number, or hydroxyl group content, is required for the production of intermediate and finished products such as polyols, resins, paint resins and lubricants (petroleum industry). Hydroxyl Number in DIN53240 is determined by the catalytic acetylation of the hydroxyl group. After hydrolysis of the intermediate product, the remaining acetic acid is titrated with alcoholic KOH solution under non-aqueous conditions. An automated system is utilized to facilitate the sample preparation and analysis.

Method description

Sample

Polyol (385 mg/100 g)

Sample preparation

No sample preparation is required.

Configuration

907 Titrando	2.907.0020
815 Sample Processor (2 tower)	2.815.0120
Dosing Unit 10 mL (2x)	6.1574.210
Dosing Unit 50 mL (2x)	6.1575.250
800 Dosino (4x)	2.800.0010
741 External Stirrer (2)	2.741.0010

Solutions

Titrant	Potassium hydroxide solution, 0.5 mol/L in methanol
Acetylation mixture	Acetic anhydride solution, 10% in NMP (store in amber glass bottle)
Catalyst solution	4-N-Dimethylaminopyridine, 1% in NMP

Analysis

Approximately 1-2 g of sample was accurately weighed into a 150 mL disposable plastic beaker and capped with DIScover lids. Next, 30 mL of 1% 4-N-dimethylaminopyridine in NMP and 10 mL of 10% acetic anhydride in NMP were added to the beaker and covered. The sample was then stirred for 15 minutes. 3 mL of water was added to the sample and stirred for 12 minutes while covered to consume the remaining reaction solution. Lastly, the sample was potentiometrically titrated with alcoholic 0.5 M KOH titrant. Between each solution addition, the plastic beakers were automatically capped using the DIScover system.

Parameters

Mode	DET U
Stirring rate	8
Signal Drift	40 mV/min
Min Waiting Time	3 s
Max Waiting Time	20 s
Measuring Point Density	4
Minimum Increment	100 µL
Maximum Increment	500 µL
Stop Volume	50 mL
ERC	20
Endpoint Recognition	greatest

Results

Mean results (n = 3)

Hydroxyl Number / (mg/100g)	385.79
s(rel) / %	1.06

Author

Joseph Head
Applications Specialist
Metrohm USA
October 2016

www.metrohm.com

