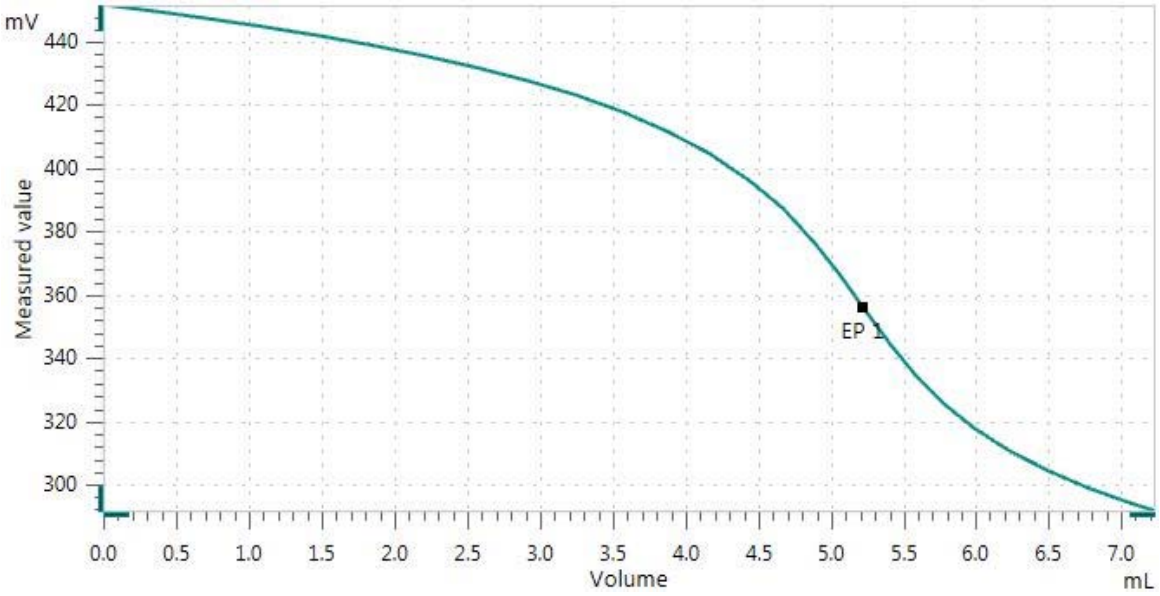
Titration Application Note T-190

Determination of carbonyl compounds in oils by potentiometric titration



Carbonyl compounds occur in many products such as bio-oils and fuels, solvents, flavors, and mineral oils. Carbonyl compounds are prone to oxidation and thus their content has an influence on stability during storage or processing. Especially for pyrolysis bio-oils stability issues are observed during storage, handling, and upgrading.

Oils are dissolved in isopropanol. After a reaction with the hydroxylamine hydrochloride at 50 °C, a fast and accurate determination by potentiometric titration using the dSolvotrode and tetra-n-butylammonium hydroxide as titrant is performed.

Metrohm

Method description

Samples

Pyrolysis bio-oil, Baby oil

Sample preparation

1 g pyrolysis bio-oil, respectively 20 g baby oil, is dissolved in 100 mL **w**(isopropanol) = 90%.

Configuration

OMNIS Advanced Titrator with stirrer	2.1001.0220
OMNIS 10 mL cylinder unit, (titrant)	6.03001.210
Digital measuring digital	6.02100.010
Lauda RE 304 circulation thermostat bath	-
Lauda E 300 immersion thermostat	-
Electrode cable plug-in head Q / plug P, 0.55 m	6.02104.300
Stirring bar / 30 mm	6.1903.060
3-way stopper with antidiffusion valve	6.1543.210
Titration vessel with thermostat jacket / 50-150 mL	6.1418.250
Titration vessel lid automation	6.1414.080
OMNIS Stand-alone license (including one instrument license)	6.06003.010
dSolvotrode, Reference electrolyte c(TEABr) = 0.4 mol/L in ethylene glycol	6.00203.300

Solutions

Titrant	c(TBAOH) = 0.1 mol/L in isopropanol/methanol, if possible this solution should be bought from a supplier.
Reaction solution	w(hydroxylamine hydrochloride) = 1.4% in w(isopropanol) = 80% 14 g $H_2NOH \cdot HCl$ is weighed into a 1000 mL volumetric flask and dissolved in 200 mL deionized water. The flask is then filled up to the mark with w(isopropanol) ≥ 99.5%.
Isopropanol	w(isopropanol) ≥ 99.5%
Isopropanol 90%	w(isopropanol) = 90% in deionized water. 900 mL w(isopropanol) ≥ 99.5% is added into a 1000 mL volumetric flask. The flask is then filled up to the mark with deionized water.

Analysis

Blank

The blank is determined the same way as the sample, just without reaction solution and 90 mL isopropanol instead.

Sample

40 mL isopropanol, 50 mL reaction solution and 10 mL sample solution are added into a titration vessel. While stirring, the solution is heated up for 5 min at 50 °C in a titration vessel with thermostat jacket. The solution is then titrated with c(TBAOH) = 0.1 mol/L until after the equivalence point. After each titration, the titration vessel, the buret tips and the electrode are rinsed with isopropanol. The glass membrane of the dSolvotrode is then reconditioned for 5 min in deionized water.

Parameters

Mode	DET U
Pause	30 s
Start volume	0 mL
Stirring rate	6
Signal drift	30 mV/min
Min. waiting time	0 s
Max. waiting time	32 s
Meas. point density	4
Min. increment	10 µL
Max. increment	Off
Dosing rate	Maximum
Stop volume	10 mL
Stop EP	1
Volume after EP	2.0 mL
EP criterion	1
EP recognition	Greatest

Results

Sample	Result carbonyl compound (n = 5)	s(rel)
Pyrolysis bio-oil	5.11 mmol/g	1.0%
Baby oil	0.14 mmol/g	2.7%

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