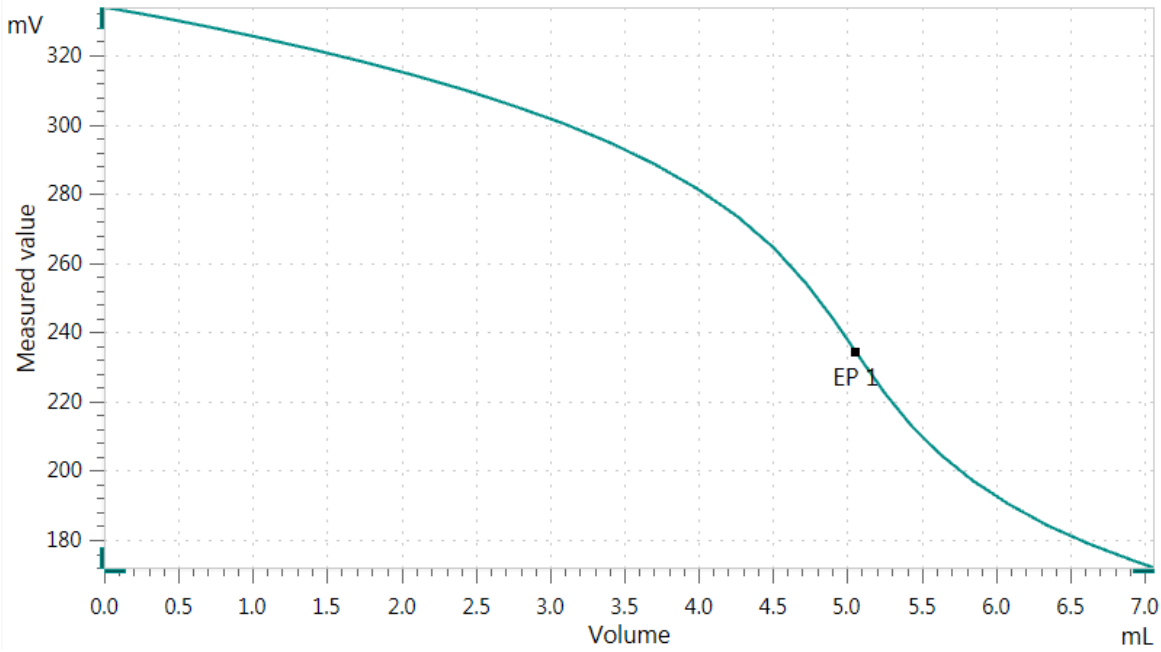
Titration Application Note T-189

Determination of water-soluble carbonyl compounds in cyclic and acyclic solvents by potentiometric titration



Compounds with carbonyl groups are particularly prone to nucleophilic additions of oxygen for which reason their stability often suffers during storage or processing. The method presented here is suitable for the determination of aldehydes and ketones sparingly soluble in water.

Samples are dissolved in deionized water. After a reaction with the hydroxylamine hydrochloride at 50 °C, carbonyl groups are quickly and accurately determined by potentiometric titration using the dUnitrode and sodium hydroxide as titrant.

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Method description

Samples

Cyclohexanone and 2-butanone

Sample preparation

5 g sample is solved in 100 mL deionized water, respectively.

Configuration

OMNIS Basic Titrator with stirrer	2.1001.0120
OMNIS Dosing Module, 2x	2.1003.0010
OMNIS 50 mL cylinder unit, 2x (deionized water, reaction solution)	6.03001.250
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
Cable MDL PL/SO 0.5 m, 2x	6.02102.010
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
dUnitrode with Pt1000	6.00200.300

Solutions

Titrant	c(NaOH) = 0.1 mol/L, if possible this solution should be bought from a supplier.
Reaction solution	w(hydroxylamine hydrochloride) = 0.7% 7 g $H_2NOH \cdot HCl$, is weighed into a 1000 mL volumetric flask and dissolved in approximately 500 mL deionized water. 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 with 90 mL deionized water, instead.

Sample

40 mL deionized water, 50 mL reaction solution and 10 mL sample solution are pipetted 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(NaOH) = 0.1 mol/L until after the equivalence point using the dUnitrode.

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	5
EP recognition	Greatest

Results

Sample	Result carbonyl compound (n = 5)	Recovery / %	s(rel) / %
Cyclohexanone	9.96 mmol/g	98.2	0.2
2-butanone	13.17 mmol/g	96.0	2.5

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