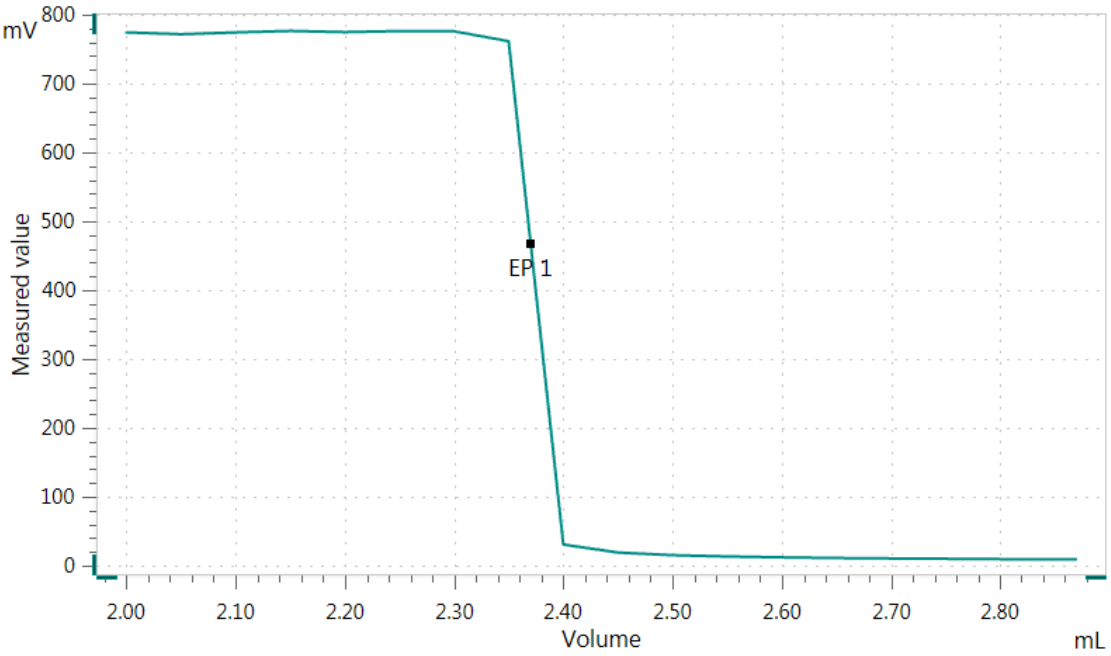
Titration Application Note T-182

Determination of the bromine number in petroleum products and aliphatic olefins according to ASTM D1159



The bromine number is an important parameter for the determination of aliphatic C=C double bonds in petroleum products. The bromine number is usually determined using electrochemical titration at 5 °C, where the bromine is generated in situ from a bromide/bromate solution. For the titration, a solvent mixture of glacial acetic acid, methanol, and chloroform is used. In this Application Note, the toxic chloroform was replaced with diethyl carbonate.

Metrohm

Method description

Samples

Cyclohexene

Transformer oil spiked with 6.6 g/L cyclohexene

Sample preparation

Based on the expected bromine number a known amount of sample is dissolved in 50 mL diethyl carbonate.

Analysis

Blank

The blank is determined the same way as the sample, just with diethyl carbonate instead of sample.

Sample

110 mL titration solvent and 5 mL sample solution are pipetted into a titration vessel. While stirring, the solution is cooled down to 0–5 °C. The solution is then titrated with $c(Br_2)$ = 0.25 mol/L until after the equivalence point.

Configuration

OMNIS Advanced Titrator with stirrer	2.1001.0220
OMNIS Dosing Module	2.1003.0010
OMNIS 50 mL cylinder unit, 1x (titration solvent)	6.03001.250
OMNIS 10 mL cylinder unit, 1x (titrant)	6.03001.210
Analog measuring module	6.02101.010
Lauda RE 304 circulation thermostat bath	-
Lauda E 300 immersion thermostat	-
Electrode cable plug-in head G (pol.) / plug P, 0.55 m	6.02104.040
Electrode cable plug-in head G (temp.) / plug P, 0.55 m	6.02104.020
Cable MDL PL/SO 1 m	6.02102.020
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
Double Pt-wire electrode for coulometry	6.0341.100
Pt1000 Temperature Sensor	6.1110.100

Solutions

Titrant	$c(Br_2)$ = 0.25 mol/L $w(KBr)$ = 5.1% and $w(KBrO_3)$ = 1.392% in deionized water, if possible this solution should be bought from a supplier
Titration solvent	Φ(diethyl carbonate) = 13.4%, (v/v) Φ(methanol) = 13.4%, (v/v) $\Phi(w(H_2SO_4) = 20\%)$ = 1.8%, (v/v) Φ(glacial acetic acid) = 71.4%, (v/v) 134 mL w(toluene) ≥ 99.3%, 134 mL w(methanol) ≥ 99.8% and 18 mL $w(H_2SO_4)$ = 20% are added into a 1000 mL volumetric flask. The flask is then filled up to the mark with w(glacial acetic acid) ≥ 99.8%.

Parameters

Mode	MET Ipol
Pause	60 s
Start volume	2 mL
Stirring rate	6
Volume increment	0.05 mL
Signal drift	30 mV/min
Min. waiting time	0 s
Max. waiting time	32 s
Dosing rate	Maximum
I(pol)	1.0 µA
Stop volume	10 mL
Stop EP	1
Volume after EP	0.5 mL
EP criterion	200 mV
EP recognition	All

Results

Sample	Result / (n = 8)	s(rel)
Cyclohexene	193.57 g bromine/100g	0.28%
Transformer oil	12.82 g bromine/100g	0.32%

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Version 1, published in December 2017