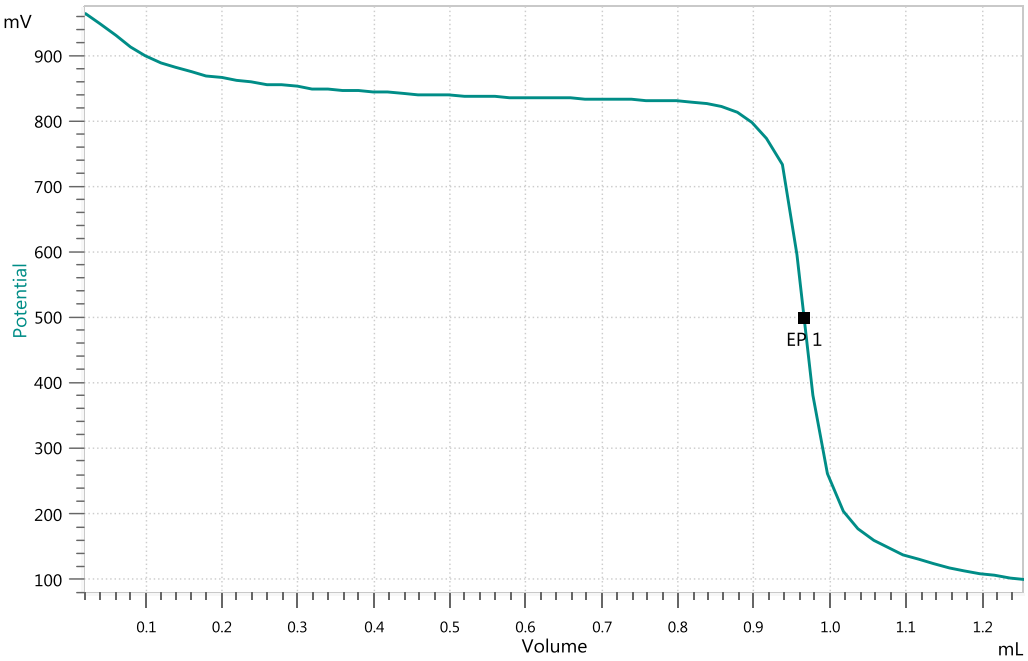
Titration Application Note T-207

Determination of bromine index of aromatic hydrocarbons according to ASTM D5776 and SH/T 1767



The bromine index is an important quality control parameter for the determination of aliphatic C=C double bonds in aromatic hydrocarbons and is thus a measure for the presence of aliphatic unsaturation in these materials.

In situ generated bromine reacts with the aliphatic double bonds. When the titration is finished an excess of free bromine causes a sudden change in the measured potential thus indicating the equivalence point.

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Method description

Sample

Xylene, mixture of isomers

Sample preparation

No sample preparation is required.

Configuration

OMNIS Advanced Titrator with magnetic stirrer	2.1001.0220
OMNIS Dosing module	2.1003.0010
Titration vessel with thermostat jacket / 50-150 mL	6.1418.250
Titration vessel lid with 5 openings	6.1414.010
Lauda RE 304 circulation thermostat bath	-
Lauda E 300 immersion thermostat	-
OMNIS 5 mL cylinder unit	6.03001.150
OMNIS 50 mL cylinder unit	6.03001.250
Analog measuring module	6.02101.010
Cable MDL PL/SO 1 m	6.02102.020
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
Stirring propeller 30 mm ETFE	6.01900.010
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	$c(Br_2)$ = 0.05 mol/L $w(KBr)$ = 1.02% and $w(KBrO_3)$ = 0.28% in deionized water, if possible this solution should be bought from a supplier
Titration solvent	714 mL glacial acetic acid, 134 mL 1-methyl-2-pyrrolidinone, 134 mL methanol and 18 mL $w(H_2SO_4)$ = 16.7% are added into a 1000 mL brown-glass flask and mixed well.

Analysis

Blank

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

Sample

110 mL titration solvent and an appropriate amount of sample (see table below) are added into a titration vessel. While stirring, the solution is cooled down to 0 – 5 °C. The solution is then titrated with $c(Br_2)$ = 0.05 mol/L until after the equivalence point.

Expected bromine index in mg bromine/100 g sample	Sample weight in g
0 – 20	50
20 – 100	30 to 40
100 – 200	20 to 30
200 – 500	8 to 10

Parameters

Mode	MET Ipol
Pause	60 s
Start volume	0 mL
Stirring rate	15
Signal drift	Off
Min. waiting time	30 s
Max. waiting time	30 s
Volume increment	0.020 mL
I(pol)	1.0 µA
Stop volume	Off
Stop measured value	100 mV
Stop EP	Off
EP criterion	30 mV
EP recognition	Greatest

Results

Sample (n = 6)	Bromine index in mg bromine/100 g sample	s(rel) /%
Xylene	23.0	0.7

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