Determination of bromine number according ASTM D 1159

Description

This application note describes the determination of the bromine number of petroleum distillates, olefins and similar samples with a bromine number > 1. For bromine numbers < 1 please use the test method for ASTM D 2710 or for aromatic hydrocarbons the coulometric test method ASTM D1492.

Instruments

Titrator	TL 7000 or higher
Exchangeable Head	WA 10
Electrode	Pt 1200, or KF 1100 and temperature sensor (e.g. W 5790 NN)
Cable	L 1 NN (only for Pt 1200)
Stirrer	Magnetic stirrer TM 235
Lab accessory	Glass beaker 150 ml or larger with an ice bath or double jacketed titration vessel TZ 1756 with a cryostat. The sample have to be maintained at a temperature between 0 – 5 °C. 50 ml volumetric flask with stopper, 250 ml graduated measuring cylinder 5 ml volumetric pipette or variable one
	Magnetic stirrer bar 30 mm

Reagents

1	Bromide-Bromate, Standard Solution(0.2500 M as Br_2)
2	Glacial acetic acid
3	Dichloromethane (as replacement of 1,1,1-trichloroethane)
4	Methanol
5	Sodium thiosulfate solution 0.1 M for standardization of the titrant (optional)
6	Sulfuric acid 1/5 diluted (1 part H_2SO_4 conc. + 5 parts water)
7	Potassium Iodide solution (150 g/L) for standardization of the titrant (optional)
All reagents should be of analytical grade or better.	

Titration procedure

Titration solvent

Prepare 1 L of titration solvent by mixing the following volumes of materials: 714 mL of glacial acetic acid, 134 mL of 1,1,1-trichloroethane (or better dichloromethane), 134 mL of methanol, and 18 mL of H_2SO_4(1 + 5).

Titrant solution

Dissolve 51.0 g of potassium bromide (KBr) and 13.92 g of potassium bromate ($KBrO_3$) each dried at 105 °C for 30 min in water and dilute to 1 L.

If the determination of bromine number of the standard cyclohexene exceeds the limits a standardization of the bromide-bromate titrant can be carried with $Na_2S_2O_3$ 0.1 mol/l standard solution. Please follow here the procedure in the norm ASTM D 1159.

Cleaning of the electrode

No special procedure. The double platinum electrodes can be cleaned with used solvents or e.g. an alcoholic solution and water. They can stored dry but also in the sample or solvent solution.

Sample preparation

Place 10 mL of 1,1,1-trichloroethane or better dichloromethane in a 50 mL volumetric flask and, by means of a pipet, introduce a test specimen as indicated in the table below. Either obtain the weight of specimen introduced by difference between the weight (to the nearest 1 mg) of the flask before and after addition of specimen or, if the density is known accurately, calculate the weight from the measured volume. Fill the flask to the mark with the selected solvent and mix well.

Bromine number	Sample size [g]
0 to 10	20 to 16
Over 10 to 20	10 to 8
Over 20 to 60	5 to 4
Over 60 to 100	2 to 1.5
Over 100 to 160	1.0 to 0.8
Over 160 to 200	0.8 to 0.6

Fill 110 mL of titration solvent using a measuring cylinder into the titration vessel. Add a stirrer bar and switch on the stirrer.

Blank titration

Because the reaction works only suitable when bromide ions are present in the solution the blank titration needs a small addition of solid KBr at the beginning. Otherwise the indication of the endpoint is not possible or very difficult. We used a spatula tip of the solid KBr and add it to the 110 ml solvent mixture. It has to be dissolved before you start with the cooling with the cryostat or take ice cubes in the ice bath.

Lower the titration clamp with titration tip and the electrode(s). Check the temperature using a connected Pt 1000 or NTC30 KOhm sensor or any other suitable temperature sensor. When the temperature reaches 5 – 0°C then start the blank method.

Sample titration

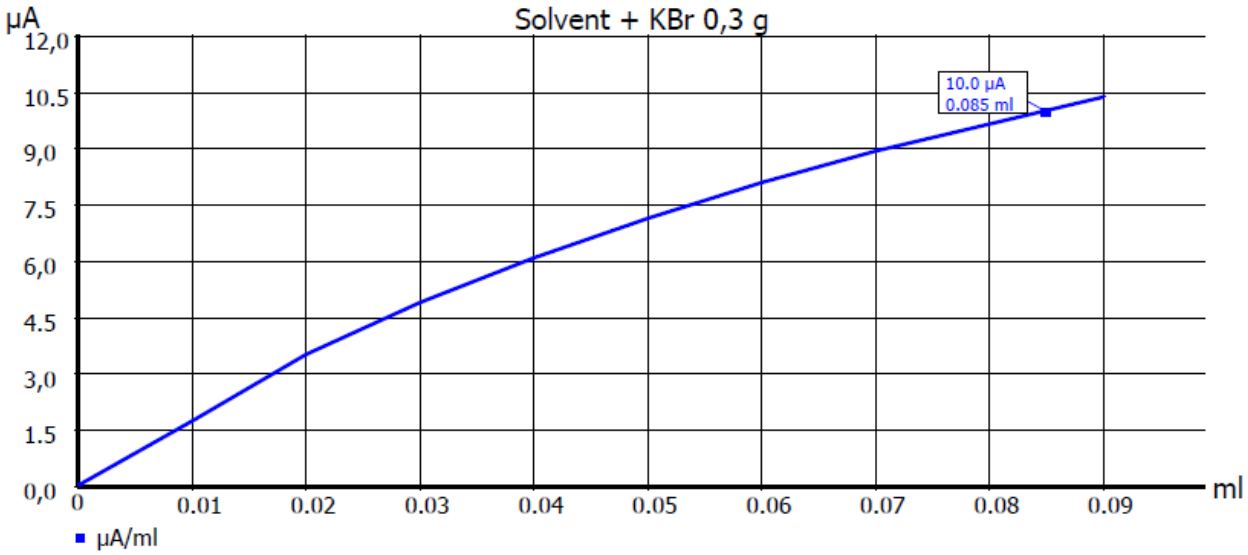
Take 5 ml aliquot from the 50 ml volumetric flask and pipette it in the 110 ml solvent mixture. Lower the titration clamp with titration tip and the electrode(s). Cool the mixture down to 5 – 0°C and start the sample titration method

Calculation:

Blank:

EP		Consumption of titrant at the endpoint.

The ml result of the blank is stored as global variable M01.



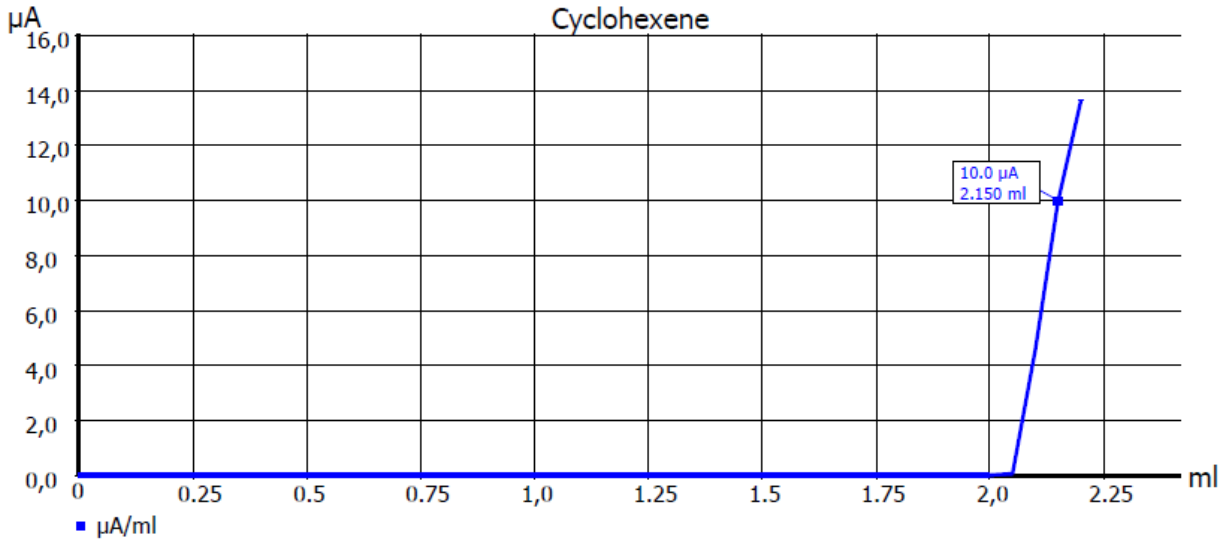
Default method	Blank Bromine no		
Method type	Automatic Titration		
Modus	D- stop		
Measured value	µA		
Measuring speed / drift	Fixed 20 seconds		
Initial waiting time	0 s		
Linear steps	0.01 ml		
Damping	n.a.	Titration direction	increase
Pretitration	Off	Delay time	0 s
Endpoint	1.0 µA	Delta Endpoint	1.0 µA
Polarization voltage	100 mV		
Max. titration volume	0.3 ml		
Dosing speed	100%	Filling speed	30 s

Sample titration

$$BN\ [\frac{g}{100g}] = \frac{(EP - B) * T * M * F1}{W * F2}$$

EP		mL of bromide-bromate solution until the endpoint for the test aliquot
B		mL of bromide-bromate solution until the endpoint for the 110 ml solvent mixture
T		Molarity of the bromide-bromate solution
M		factor for converting g of bromine per 100 g of sample and incorporating molecular weight of bromine (as Br_2) and conversion of mL to L.
F1		1
F2		1
W		Sample amount in g

The blank value B is used normally from the global variable M01



Default method	Bromine number		
Method type	Automatic Titration		
Modus	D- stop		
Measured value	µA		
Measuring speed / drift	Fixed 20 seconds		
Initial waiting time	0 s		
Linear steps	0.05 ml		
Damping	n.a.	Titration direction	increase
Pretitration	Off	Delay time	0 s
Endpoint	10.0 µA	Delta Endpoint	10.0 µA
Polarization voltage	100 mV		
Max. titration volume	10 ml		
Dosing speed	100%	Filling speed	30 s

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