

Application Note

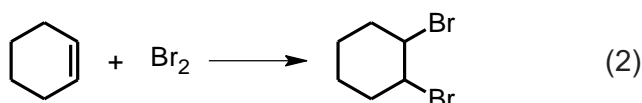
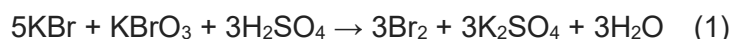
Bromine Index of Aromatic Hydrocarbons by polarization titration

Industry	:	Chemicals
Instrument	:	Automatic potentiometric titrator
Measurement method	:	Polarization titration at constant current
Standards	:	ASTM D5776-07

1. Scope

The bromine index indicates the number of unsaturated bonds in a sample component. It is defined as the number of milligrams of bromine added to unsaturated carbon-carbon bonds in a 100 g sample and is expressed as mg Br₂/100 g. This Application Note describes an example of the measurement of the bromine index by constant current polarized titration, as per ASTM D5776-07. The sample used is a solution prepared by dissolving cyclohexene in toluene.

To measure the bromine index, a potassium bromide/potassium bromate solution is titrated into a sample solution containing strong acid, and the bromine released is added to the double carbon-carbon bonds of the sample. The reactions that release and add the bromine are shown by Formulas (1) and (2).



A constant current is applied between twin platinum electrodes during titration, and the voltage between the electrodes is measured. Before the equivalent point is reached, the bromine is consumed by the cyclohexene, so the concentration of bromine in the solution is very low. After reaching the equivalent point, the concentration of bromine in the solution increases and the bromine undergoes reduction at the electrodes, so the voltage required for a constant current flow drops rapidly. By observing this voltage change, the bromine index was calculated from the titration volume required to reach the endpoint.

2. Precautions

- 1) Handle the reagents inside a fume hood.
- 2) Before starting the measurements for the day, ensure uniformity of the concentration of the titrant by purging the connection between the reagent bottle and the burette several times with titration solution. Then discharge approximately 10 mL of the titration solution between the burette and the titration nozzle.

3. Post-measurement procedure

Clean the electrodes and titration nozzle with ethanol and pure water in that order, and then immerse them in pure water. If the titration nozzle is left exposed to the air, the components in the titration solution will precipitate out, and the nozzle will clog.

4. Apparatus

Main unit : Automatic potentiometric titrator (polarization titration preamplifier :POT)

Electrode : Twin platinum electrode

5. Reagents

Titrant : 0.05 mol/L Bromide-Bromate Standard Solution (potassium bromide and potassium bromate)
 Titration solvent : 1 L of titration solvent by mixing the following volumes of Materials; 714 mL of glacial acetic acid, 134 mL of 1-Methyl-2-Pyrrolidinone, 134 mL of methanol and 18 mL of H₂SO₄ (1 + 5).

6. Procedure

-Calibration-

- 1) Press the [Calibration] button.
- 2) Set the channel/unit to “Ch3/Pol.”
- 3) Press the [Details] button, and configure the settings as follows.
 Detection mode: Constant current polarization; Constant current value: 5.00 μA
- 4) Weigh the sample solution* and mix it with 150 mL of the titration solvent. Immerse the twin platinum electrodes in the solution and perform calibration.

-Blank test-

- 1) Introduce 150 mL of the titration solvent in a beaker.
- 2) Titrate with a 0.05 mol/L potassium bromide/potassium bromate solution, and measure the blank test value.

-Measurement-

- 1) Weigh the sample solution*, and mix it with 150 mL of the titration solvent.
- 2) Titrate with a 0.05 mol/L potassium bromide/potassium bromate solution, and obtain the bromine index.

* Introduce the amount specified in ASTM D5776 corresponding to the bromine index of the sample.

7. Calculation

$$\text{Bromine index (mgBr}_2\text{/100g)} = (\text{EP1-BL1}) \times \text{TF} \times \text{C1} \times \text{K1/S}$$

EP1	:	Titration amount (mL)
BL1	:	Titration amount (mL) of Blank test = 0.08883(mL)
TF	:	Factor of Titrant = 1.0704
C1	:	Concentration conversion coefficient = 0.1
K1	:	Unit conversion factor = 7990
S	:	Sample size (g)

8. Example

— Parameter —

<u><Titr. Mode></u>	: Auto Intermit	<u><Ctrl. Para.></u>	
<u><Titr. Form></u>	: EP Stop	End Point No.	: 1
		End Sense (dE/dmL)	: Auto
<u><Titr. Para.></u>			
Max Volume	: 0.5 (mL), Blank		
	: 20 (mL), Sample		
Channel/Unit(Ctrl.)	: Ch3, μA	Gain	: 1
Channel/Unit (Ref.)	: Off	Data Sampling	: Auto
Wait Time	: 0 (s)	Ctrl. Speed	: Standard
pH Polarity	: Standard	Other Control	: Standard

