京都電子工業株式会社
KYOTO ELECTRONICS MANUFACTURING CO.,LTD.

Application Note

Bromine number of cyclohexene

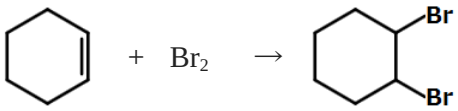
Industry	:	Petroleum
Instrument	:	Automatic potentiometric titrator
Measurement method	:	Polarization titration at constant current
Standards	:	JIS K2605

1. Scope

Bromine number of cyclohexene was measured by polarization titration at constant current based on "JIS K2605 Petroleum distillates and commercial aliphatic olefins - Determination of bromine number - Electrometric method".

Bromine number is the number of grams of bromine that is added to carbon-carbon unsatulated bond in a 100 g of sample and is expressed in "gBr_2/100g". In measurement of bromine number, bromine is released by adding potassium bromide- potassium bromate (KBr-$KBrO_3$) solution into a sample solution containing strong acid, and then the released bromine is added to carbon-carbon double bond. The chemical formulas of bromine releasing reaction and addition reaction are given in formula (1) and (2). During titration, polarization potential which needs to apply constant current between twin platinum electrodes is measured and, an equivalent point is determined by change of the polarization potential. After the equivalent point, amount of bromine in the solution is increased sharply. Polarization potential becomes smaller when amount of bromine in the solution become larger, therefore a sharp change in potential is observed at the equivalent point. The endpoint of the titration is determined by detecting the change in potential.

$$5KBr + KBrO_3 + 3H_2SO_4 \rightarrow 3Br_2 + 3K_2SO_4 + 3H_2O \quad \cdots(1)$$



$\cdots(2)$

In this measurement, toluene solution of cyclohexene was added to titration solvent whose temperature was kept in a range of 0-5°C, and then the solution was titrated with 0.25 mol/L KBr-$KBrO_3$ solution. An inflection point on the titration curve was regarded as the end point, and the bromine number of cyclohexene was calculated from the volume of KBr-$KBrO_3$ solution consumed to titrate sample to the end point.

2. Precautions

1) Handle the reagents in a well ventilated room or a draft chamber.
2) Keep a temperature of titration solvent in a range of 0-5°C during titration.
3) Discharge titrant between burette and titration nozzle once before a titration.

3. Post-measurement procedure

Wash electrode and titration nozzle with ethanol and pure water and then keep them soaked in pure water. If titration nozzle is left standing in the air, it will be clogged with precipitate of the component of titrant.

4. Apparatus

Main unit	:	Automatic potentiometric titrator (preamplifier : POT)
Electrode	:	Twin platinum electrodes
		Temperature compensation electrode

京都电子工业株式会社 - 可睦电子(上海)商贸有限公司 上海市徐汇区宜山路333号1201室
电话: 021-54488867 电邮: kemu-kem@163.com 网址: http://www.kem-china.com

5. Reagent

Titrant : 0.25 mol/L KBr-$KBrO_3$ solution
Titration solvent : Mixture of glacial acetic acid, toluene, methanol and H_2SO_4(1+5) mixture ratio of them is 714 : 134 : 134 : 18 (volumetric ratio).

6. Procedure

-Preparation of sample solution-
1) Add 10 mL of toluene into a 50 mL volumetric flask.
2) Add 0.6 - 1.0 g of cyclohexene into the flask and measure mass of it.
3) Add toluene to the mark of the flask and mix it.

-Setting of constant current value-
1) Press [Calibration] button.
2) Set Channel/Unit to "Ch3/Pol".
3) Press [Details] button and set as follows.
Calibration Mode : Current Polar Current : 5.00 μA
4) Immerse twin platinum electrode in titration solvent and perform calibration.

-Blank test-
1) Add 110 mL of titration solvent into a 200 mL tall beaker.
2) Keep temperature of the titration solvent in a range of 0-5°C by ice cooling.
3) Add 5 mL of toluene.
4) Titrate with 0.25 mol/L KBr-$KBrO_3$ solution.

-Measurement-
1) Add 110 mL of titration solvent into a 200 mL tall beaker.
2) Keep temperature of the titration solvent in a range of 0-5°C by ice cooling.
3) Add 5 mL of sample solution.
4) Titrate with 0.25 mol/L KBr-$KBrO_3$ solution.

7. Calculation

$$\text{Bromine number } (gBr_2/100g) = (EP1 - BL1) \times TF \times C1 \times K1 / (R \times S)$$

EP1 : Titer (mL)
BL1 : Titer for blank titration = 0.01207 mL
TF : Factor of titrant = 1.0065
C1 : Concentration conversion coefficient = 39.95 mg/mL
K1 : Unit conversion coefficient = 0.1
R : Dilution factor = 0.1
S : Quantity of sample in 50 mL of the sample solution (g)

8. Example

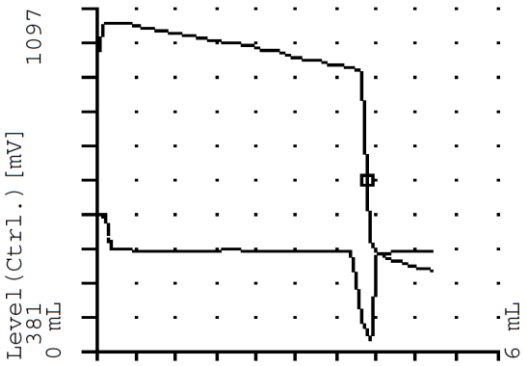
-Titration parameter-

＜Titr. Mode＞	: Auto Intermit	**＜Ctrl. Para.＞**	
＜Titr. Form＞	: EP Stop	Number of EP	: 1
		End Sense	: Auto
＜Titr. Para.＞		Gain	: 1
Max. Volume	: 0.25 (mL) (blank test)	Data Sampling	: Auto
	: 20 (mL) (sample)	Ctrl. Speed	: Standard
Channel/Unit(Ctrl.)	: Ch3, mV	Other Ctrl.	: Standard
Channel/Unit(Ref.)	: Off	Auto Int. Mode	: Standard
pH Polarity	: Standard	Stirrer Speed	: 3
Titr. Type Check	: No Check		
Direction	: Auto		
Wait Time	: 0 (s)		
Dose Mode	: None		

(The measurement parameter and the titration curve are an example of our automatic potentiometric titrator. In some titrators, parameter item may be different or another parameter item may be added.)

-Results-

-Titration curve-



-Measurement results-

	Quantity of sample in 50mL of sample solution (g)	Titer (mL)	Bromine number (gBr_2/100g)
1	0.8292	4.0185	194.28
2	0.8292	4.0146	194.09
3	0.8292	4.0235	194.52
Mean	-	-	194.30
SD	-	-	0.22
RSD (%)	-	-	0.11

9. Summary

In this measurement, the results showed a good repeatability with 0.11% RSD (relative standard deviation), and the results of bromine number was within range specified in JIS K2605 (187-199 gBr_2/100g).

In some samples, verification of the measurement capability is required. In such case, please contact us.

10. References

1) JIS K2605-1996 Petroleum distillates and commercial aliphatic olefins - Determination of bromine number - Electrometric method

京都电子工业株式会社 - 可睦电子(上海)商贸有限公司 上海市徐汇区宜山路333号1201室
电话: 021-54488867 电邮: kemu-kem@163.com 网址: http://www.kem-china.com