Application Note

Standardization of bromide-bromate standard solution

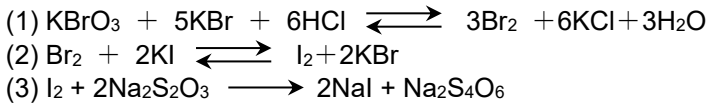
Industry	Petrochemicals
Instrument	Automatic potentiometric titrator
Measurement method	Redox titration
Standards	ASTM D1159, ASTM D5776

1. Scope

Caution

This measurement is performed using organic solvents and strong acids. When you follow this application note, wear masks, gloves, protective equipment, etc. The automatic potentiometric titrator should be installed and used in fume hoods where local exhaust ventilation is possible.

Bromide-bromate standard solution is used as a titrant to determine the bromine value and bromine index in various standards such as ASTM. This Application Note introduces the standardization of 0.25 mol/L bromide-bromate standard solution according to ASTM D1159. The reaction formula for the standardization is as follows (1) bromine formation, (2) displacement reaction with potassium iodide, (3) titration by sodium thiosulfate solution. The concentration of the adjusted bromide-bromate solution is determined by titrating with a sodium thiosulfate solution of known concentration.



2. Precautions

Before starting measurements each day, purge the titrant several times between the reagent bottle and the burette to equalize the titrant concentration, then discharge about 10 mL of the titrant between the burette and the titration nozzle.

3. Post-measurement procedure

Clean the electrodes and titration nozzle with pure water in that order, 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 (Preamplifier STD)
Electrode	Combined platinum electrode (C-272)
	Temperature compensation electrode (T-171)

5. Reagents

Titrant	0.1 mol/L Sodium thiosulfate solution
Reagents	Hydroxy chloride (35 wt%), Glacial acetic acid
Additive reagents	150 g/L Potassium iodide solution

6. Procedure

1) 50 mL of glacial acetic acid and 1 mL of hydrochloric acid (35 wt%) are added to a 100mL Erlenmeyer flask with a stopper and cooled at 0 to 5 °C for 10 minutes.
2) Drop 5 mL of 0.25mol/L bromide-bromate solution into a cooled reagent, close the stopper immediately, and shake the solution to react.
3) After the reaction solution is cooled again at 0 to 5 °C for 5 minutes, add 5 mL of 150 g/L potassium iodide solution and shake vigorously.
4) Open the reaction solution into a 200 mL tall beaker, rinse the inside of the Erlenmeyer flask and the stopper with 100 mL of pure water, and collect the reaction solution and the rinse solution.
5) Titrate with 0.1 mol / L Sodium thiosulfate solution.

7. Calculation

Factor = EP1×C1×FA/S

EP1	Titration amount (mL)
C1	Concentration conversion coefficient = 0.2
FA	Factor of 0.1 mol/L sodium thiosulfate solution = 1.0430
S	Sample size = 5 (mL)

8. Example

— Parameter —

<Titr. Mode> Auto Int.

<Titr. Form> EP Stop

<Titr. Para.>

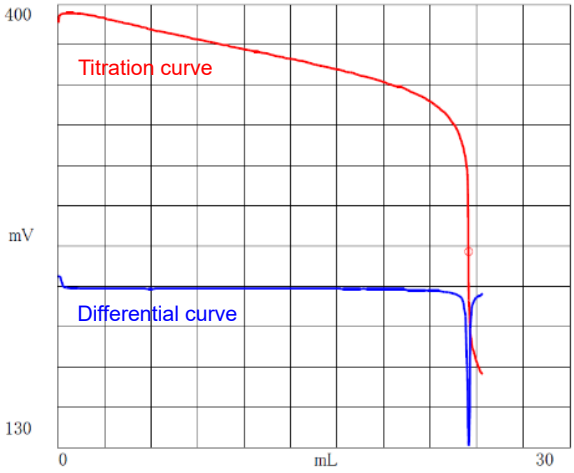
Max Volume	30(mL)
Channel/Unit(Ctrl.)	Ch1, mV
Channel/Unit(Ref.)	Off
pH Polarity	Standard
Titr. Type Check	No Check
Direction	Auto
Wait time	0(s)
Dose Mode	None

<Ctrl. Para.>

End Point No.	1
End Sense	Auto
Gain	1
Data Sampling	Standard
Ctl. Speed Mode	Standard
Other Control	Standard
Auto Int. Mode	Standard
Stir. speed	4

（The above condition is an example. The setting condition depends on the model.）

— Example of titration curve —



— Measurement results —

	Titration (mL)	Factor
1	24.1015	1.0055
2	24.0938	1.0052
3	24.0993	1.0054
Average	-	1.00537
SD	-	0.00015
RSD (%)	-	0.015

9. Notes

When the titration volume is large, as in this measurement, titration time can be shortened by setting the titrator to inject a fixed amount of titrant.

10. Reference

・ASTM D1159-07 Standard Test Method for Bromine Numbers of Petroleum Distillates and Commercial Aliphatic Olefins by Electrometric Titration
・ ASTM D5776 Standard Test Method for Bromine Index of Aromatic Hydrocarbons by Electrometric Titration