Application Note

Measurement of bromine index by Karl Fischer moisture titrator (coulometric titration) - Application of one-component inner burette -

Industry	Petroleum
Instrument	Karl Fischer moisture titrator
Measurement method	Coulometric titration/Direct method
Standards	

1. Scope

The bromine indicates the amount of unsaturated bonds in petroleum product components. It is defined as the number of milligrams of bromine consumed by 100 g of sample. This Application Note introduces an example of bromine index measurement using a coulometric titration Karl Fischer moisture titrator with the one-component inner burette.
The sample is dissolved in a solvent and reacted by changing bromine ions to bromine through electrolytic oxidization. In coulometric titration (coulometry), the amount of reacted bromine is determined from the amount of electricity (coulombs) required for this electrolytic oxidization.
In this application, the one sample solution was measured using Karl Fischer moisture titrator with the two-component and one-component inner burette.
In the conclusion, the measurement results did not have much difference between two-component or one-component inner burette.

2. Precautions

In the case the reagent becomes turbid and white during measurement, replace it with the new reagent. The titration cell and inner burette used for the bromine index measurement should be sufficiently cleaned and dried when used for moisture measurement. It is recommended that the inner burette for bromine index measurement be used exclusively for the measurement.

3. Post-measurement procedure

Drain the reagent from the titration cell, then clean the titration cell, electrolysis electrode (inner burette), and twin platinum electrode with methanol.

4. Apparatus

Equipment	Karl Fischer moisture titrator for coulometric method
Option	Twin platinum electrode, Electrolytic electrode (one-component inner burette)

5. Reagents

Anolyte A solution consisting of a mixture of 600 mL of acetic acid, 260 mL of methanol, and 140 mL of 1 mol / L potassium bromide solution.
Catholyte (used for comparative test) 0.2 mol/L potassium chloride aqueous solution
Test solution 0.051 wt% solution which diluted cyclohexene with toluene as a trial sample (expected bromine index: about 99)

6. Procedure

- Preparation -

1) Fill the titration flask with approximately 100 mL of anolyte.
2) The test solution is injected 20 to 100 µL by a syringe, then perform preliminary titration.

- Measurement -

1) Collect a sample using a syringe and measure the mass.
2) Inject the sample from the syringe inlet of the titration cell and measure.
3) Measure the syringe mass after sample injection.
4) The difference in the mass of the syringe before and after injection of the sample is used as the sample collection volume.

Follow the table below for the sample injection amount.

Bromine index ($mgBr_2$/100 g)	Sample amount (g)
less than 10	10 to 15
10 - 50	5 to 10
50 - 100	3 to 5
100 - 200	1 to 3
200 or more	to 1

7. Calculation

Bromine Index ($mgBr_2$/100 g) = FA × (Data－Drift × t －Blank) / (Wt1－Wt2) × D × 0.1

FA	Collection factor (1.0)
Data	Total bromine content (µg)
Drift	Initial drift level (µg/s)
t	Measuring time (s)
Blank	Blank level (0µg)
Wt1	Sample + Syringe (g)
Wt2	Mass of empty syringe (g)
D	Dilution factor (1.0)

8. Example

— Parameter —

＜Titr.Para＞		**＜Ctrl.Para＞**	
Titration mode	Bromine index	Stable	0.5µg/min
t(stir)	0s	Control Gain	5.0
t(wait)	30s	Speed	standard
t(max)	0s	End level	300mV
Drift Stop	Rel.	Start mode	Auto
Drift(Rel.)	1.0µg/s	Data sampling time	5s
		Stirrer speed	3

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

— Example of titration curve —

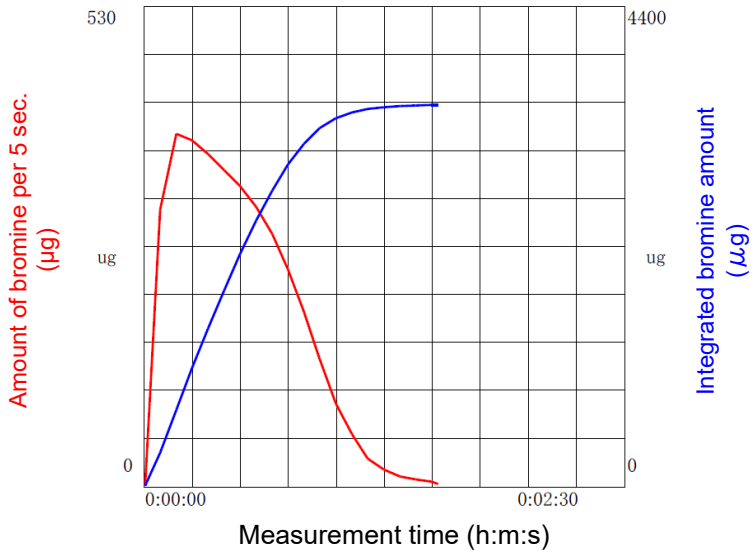

Table 1 Result of measurement of bromine index
(one-component inner burette)

	Sampling amount (g)	Bromine (µg)	Bromine index ($mgBr_2$/100 g)	Time (h:m:s)
1	3.5181	3507.2	99.69	0:01:08
2	3.3560	3353.0	99.91	0:01:09
3	3.3394	3333.1	99.81	0:01:12
Average			99.80	
SD			0.11	
RSD (%)			0.11	

Table 2 Result of measurement of bromine index
(two-component inner burette)

	Sampling amount (g)	Bromine (µg)	Bromine index ($mgBr_2$/100 g)	Time (h:m:s)
1	3.4962	3489.2	99.80	0:01:32
2	3.5591	3549.4	99.73	0:01:40
3	3.4951	3473.4	99.38	0:01:46
Average			99.64	
SD			0.23	
RSD (%)			0.23	