

Application Note

Basic nitrogen in heavy fuel oil

Industry	:	Petroleum
Instrument	:	Automatic potentiometric titrator
Measurement method	:	Potentiometric titration / Neutralization titration
Standards	:	UOP Method 269

1. Scope

Basic nitrogen in heavy fuel oil was measured based on “UOP Method 269-10 Nitrogen Bases in Hydrocarbons by Titration”.

A sample was dissolved in toluene and then acetic acid was added to the solution. The solution was titrated with 0.02 mol/L perchloric acid acetic acid solution. An inflection point on the titration curve was regarded as the end point. The basic nitrogen content of the sample was calculated from the volume of perchloric acid acetic acid solution used to titrate sample to the endpoint.

2. Precautions

- 1) When measurement is repeated, regenerating the sensitivity of the electrodes is required for each measurement. After a measurement, wash the electrodes with titration solvent and then immerse them in pure water until the indicated potential becomes stable. Before next measurement, wash the electrodes with titration solvent again.
- 2) Do not leave the electrodes to remain immersed in titration solvent.
- 3) Standardize the 0.02 mol/L perchloric acid acetic acid solution more than once a week.
- 4) Organic solvent have relatively large coefficient of thermal expansion. Therefore, difference between the temperature of titrant when standardization and the temperature of it when sample measuring should be 5°C or less.
- 5) Handle the reagents in a well ventilated room or a draft chamber.

3. Post-measurement procedure

The sensitivity of the electrodes is deteriorated after a series of measurements. Therefore, regenerate the sensitivity of them by the method described in “2. Precautions, 1)”.

4. Apparatus

Main unit	:	Automatic potentiometric titrator (preamplifier : STD)
Electrode	:	pH glass electrode Double junction reference electrode (inner solution : saturated NaClO ₄ acetic acid solution) Temperature compensation electrode

5. Reagents

- 0.02 mol/L perchloric acid acetic acid solution
- Toluene
- Glacial acetic acid

6. Procedure

-Blank test-

- 1) Add 50 mL of toluene and 75 mL of glacial acetic acid into a 200 mL tall beaker, and stir it.
- 2) Titrate with 0.02 mol/L perchloric acid acetic acid solution.

-Measurement-

- 1) Add 10 g of sample into a 200 mL tall beaker and measure mass of it.
- 2) Add 50 mL of toluene into the beaker and dissolve the sample.
- 3) Add 75 mL of glacial acetic acid into the beaker.
- 4) Titrate with 0.02 mol/L perchloric acid acetic acid solution.

7. Calculation

$$\text{Basic nitrogen (mass\%)} = (\text{EP1} - \text{BL1}) \times \text{TF} \times \text{C1} \times \text{K1} / \text{S}$$

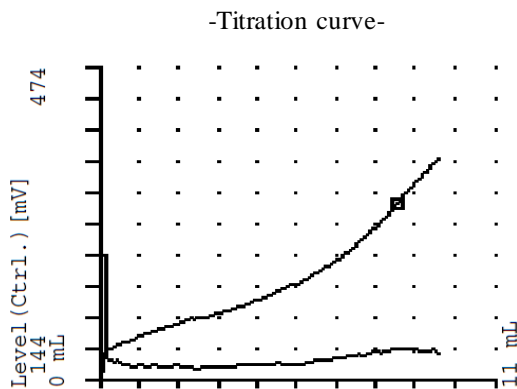
EP1	:	Titer (mL)
BL1	:	Titer for blank titration = 0.0037 mL
TF	:	Factor of titrant = 0.8993
C1	:	Concentration conversion coefficient = 0.28 mg/mL
K1	:	Unit conversion coefficient = 0.1
S	:	Quantity of sample (g)

8. Example

-Titration parameter-

<u>< Titr. Mode ></u>	: Auto Intermit	<u>< Ctrl. Para. ></u>	
<u>< Titr. Form ></u>	: EP Stop	Number of EP	: 1
		End Sense	: Auto (Blank test)
<u>< Titr. Para. ></u>			: Set (Sample)
Max. Volume	: 0.2 (mL) (Blank test)	dE	: 50.0 (dE)
	: 20 (mL) (Sample)	dE/dmL	: 30.0 (dE/dmL)
Channel/Unit(Ctrl.)	: Ch1, mV	Gain	: 1
Channel/Unit(Ref.)	: Off	Data Sampling	: Auto
pH Polarity	: Standard	Ctrl. Speed	: Standard
Titration Type Check	: No Check	Other Ctrl.	: Standard
Direction	: Auto	Auto Int. Mode	: Blank (Blank test)
Wait Time	: 0 (s)		: Standard (Sample)
Dose Mode	: None	Stirrer Speed	: 4

(The measurement parameter and the titration curve are an example of our automatic potentiometric titrator. For other models, parameter item may be different or other parameter item may be added.)



-Measurement results-

	Quantity of sample (g)	Titer (mL)	Basic nitrogen (mass%)
1	10.0094	8.2569	0.0208
2	10.0376	8.6826	0.0218
3	10.0143	8.1876	0.0206
Mean	-	-	0.0211
SD	-	-	0.0006
RSD (%)	-	-	3.0518

9. Summary

In this measurement, the results showed a good repeatability with about 3% RSD (Relative standard deviation).

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

10. References

- 1) UOP Method 269-10 Nitrogen Bases in Hydrocarbons by titration