

Application Note

Saponification number of petroleum products

Industry	Petroleum
Instrument	Automatic potentiometric titrator
Measurement method	Potentiometric titration / Neutralization titration
Standards	ASTM D94

1. Scope

The saponification number represents the number of mg of potassium hydroxide required to saponify 1 g of sample.

In this report, we describe an example of how to measure sample to determine its saponification number using an automatic potentiometric titrator (based on ASTM D94). Ester compounds contained in the sample were saponified by adding an excess amount of potassium hydroxide, and the remaining potassium hydroxide was titrated with hydrochloric acid to obtain the saponification number.

2. Precautions

- 1) Prior to the first titration, prepare the burette by dispensing and aspirating the titrant between it and the reagent bottle several times. Once done, purge approximately 10mL of the titrant from the burette and out of the titration nozzle.
- 2) Hazardous substance are used in this titration, so it must be performed in a fume hood. Operators must wear appropriate personal protective equipment.

3. Apparatus

Main unit	Automatic potentiometric titrator (Preamplifier STD)
Electrode	Glass electrode Double junction reference electrode (Internal solution:3.3 mol/L potassium chloride solution)

4. Reagents

Titrant	0.5 mol/L Hydrochloric acid standard solution
Additive reagents	0.5 mol/L Alcoholic potassium hydroxide solution, 2-butanone, hexanes

5. Procedure

- 1) Transfer the sample into an Erlenmeyer flask, and weigh precisely.
- 2) Add 25 ± 1 mL of 2-butanone, and make sure the sample appears homogenous.※¹
- 3) Add 25 ± 0.03 mL of 0.5 mol/L alcoholic potassium hydroxide solution.
- 4) Connect the condenser to the flask, then heat for 30 minutes after refluxing begins.※²
- 5) At the end of the saponification, immediately pour 50mL of the hexanes down the condenser. This not only rinses the condenser but also cools the reaction mixture.
- 6) Transfer the reaction mixture to a beaker, rinse the inside of the Erlenmeyer flask twice with 10 mL of hexanes, and combine the washing solution with the solution.
- 7) Titrate the solution while hot (without reheating) with 0.5 mol/L hydrochloric acid standard solution.※³
- 8) Perform blank test by steps 1) ~ 8) without adding sample.

※¹ Beforehand dissolve the sample with 15~25mL xylene and add 2-butanone when sample is difficult to dissolve.

※² Adjust the reflux time depending on the sample.

※³ It is especially important to perform the analysis of reaction mixtures with a high melting point as quickly as (safely) possible, as they tend to solidify.

6. Calculation

$$\text{Saponification number (mgKOH/g)} = (\text{BL1} - \text{EP1}) \times \text{TF} \times \text{K1} \times \text{C1} / \text{S}$$

BL1	Titration amount of blank test (mL)	
EP1	Titration amount (mL)	
TF	Factor of titrant	= 0.9925
K1	Unit conversion coefficient (mol/L)	= 0.5
C1	Concentration conversion coefficient (g/mol)	= 56.1
S	Sample size (g)	

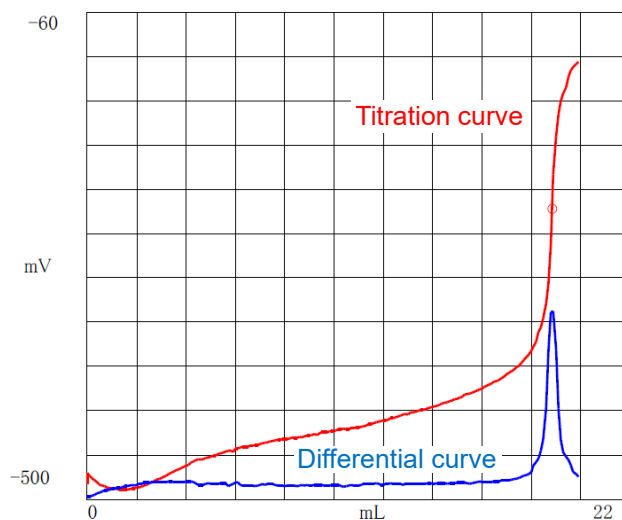
7. Example

—Titration parameters—

<u><Titr. Mode></u>	Intermit	<u><Ctrl. Para.></u>	Number of EP	1
<u><Titr. Form></u>	EP Stop		End Sense	Auto
			Gain	1
<u><Titr. Para.></u>			Data sampling	Set
Max Volume (mL)	50		Data Samp. Pot. (mV)	999.0
Channel / Unit (Ctrl.)	Ch1, mV		Data Samp. Vol. (mL)	0.1000
Wait time (s)	5		Ctl. speed Mode	Set
Dose Mode	Off		Cut-off time (s)	3
			Unit volume (mL)	0.1000
			Disp. Speed (s/mL)	1
			Other control	Standard
			Stirrer speed	4

(Listed above are example settings. Availability of settings may vary by instrument model.)

—Example of titration curve—



— Measurement results —

Table 1. Blank test

Titration (mL)	
1	26.4617
2	26.2286
3	26.3662
Mean	26.3522
SD	0.1172
RSD (%)	0.44

Table 2. Sample measurement result

	Sample (g)	Titration (mL)	Saponification number (mgKOH/g)
1	1.0060	18.8511	207.58
2	1.0196	18.7254	208.25
3	1.0021	18.8448	208.57
Mean	-	-	208.13
SD	-	-	0.51
RSD (%)	-	-	0.25

8. Reference

ASTM D94-07 Standard Test Methods for Saponification Number of Petroleum Products