

JIS

JAPANESE INDUSTRIAL STANDARD

Petroleum products and lubricants — Determination of neutralization number

JIS K 2501—1992

Translated and Published

by

Japanese Standards Association

Printed in Japan

29 S

In the event of any doubt arising,
the original Standard in Japanese is to be final authority.

JAPANESE INDUSTRIAL STANDARD

J I S

Petroleum products and lubricants
- Determination of neutralization number

K 2501-1992

1. Scope

This Japanese Industrial Standard specifies methods for determination for neutralization number of petroleum products and lubricants according to indicator titration methods and potentiometric titration methods.

Remarks 1. The neutralization number is the general term of acid number and base number.

2. The applicable standards to this standard are given in Attached Table 1.

3. The corresponding International Standards of this standard are the following:

ISO 3771: 1977 Petroleum products - Total base number - Perchloric acid potentiometric titration method

ISO 6618: 1987 Petroleum products and lubricants - Neutralization number - Colour-indicator titration method

ISO 6619: 1988 Petroleum products and lubricants - Neutralization number - Potentiometric titration method

4. The reference foreign standards to this standard are given in Attached Table 2.

2. Terms and definitions

For the purpose of this standard, main terms and definitions shall be as follows:

- (1) total acid number The quantity of potassium hydroxide, expressed in milligrams, required for neutralization of all acidic constituents present in 1 g of sample.
- (2) strong acid number The quantity of potassium hydroxide, expressed in milligrams, required for neutralization of strong acid constituents present in 1 g of sample.

- (3) total base number The quantity of potassium hydroxide equivalent to hydrochloric acid or perchloric acid, expressed in milligrams, required for neutralization of all basic constituents present in 1 g of sample.

Remarks: It may be expressed as milliequivalent of hydroxide per gram.

- (4) strong base number The quantity of potassium hydroxide, expressed in milligrams, equivalent to hydrochloric acid or perchloric acid required for neutralization of strong base constituents present in 1 g of sample.

3. Classification of test methods

The classification of test methods shall be in accordance with Table 1.

Table 1. Classification of test methods

Test method	Test item	(Informative reference) Specific feature	Clause No.
Indicator titration method	Total acid number and strong acid number	1. Since the determination is made according to the change of colour of indicator, the method is suitable to apply to light-coloured oils and such oils that contain comparatively few additives. 2. The determination is made with simple apparatus in a short time. (Corresponds to ISO 6618)	4.
	Strong base number		
Potentiometric titration method (acid number)	Total acid number and strong acid number	1. This method is applicable widely to such kinds of oils as new oils, used oils, light-coloured oils and dark-coloured oils. 2. Since this method is a potentiometric titration method with non-aqueous solution, the response is slow so that it takes more time for determination. 3. Since this method uses a potentiometer, automatic system is readily applicable (manual and automatic titration methods are specified). (Corresponds to ISO 6619)	5.

Table 1. (Continued)

Test method	Test item	(Informative reference) Specific feature	Clause No.
Potentiometric titration method (base number)	Total base number and strong base number	1. This method is applicable widely to such kinds of oils as new oils, used oils, light-coloured oils and dark-coloured oils. 2. Since this method is a potentiometric titration method with non-aqueous solution, the response is slow so that it takes more time for determination. 3. In comparison with the perchloric acid method, lower values are frequently obtained. 4. Since this method uses a potentiometer, automatic system is readily applicable (manual and automatic titration methods are specified).	6.
Potentiometric titration method (base number, perchloric acid method)	Total base number	1. This method is suitable to determine the total base number for new oils with basic additives and used oils. 2. The determination is made accurately in a short time. 3. Since this method uses a potentiometer, automatic system is readily applicable (manual and automatic titration methods are specified). However, toxic and/or stimulative solvent is used so that special care is necessary. (Corresponds to ISO 3711)	7.

Informative reference: In addition to this standard, the following ones are specified for the total acid number test methods.

- (1) JIS C 2101 for electrical insulating oil
- (2) JIS K 2276 for fuel oil for aviation turbine
- (3) JIS K 2514 for oxidation stability test for turbine oil

4. Indicator titration method

4.1 Principle of test method For testing the total acid number or strong base number, dissolve the sample in a mixture solvent of toluene, propan-2-ol containing a small amount of water, and titrate with potassium hydroxide standard propan-2-ol solution or hydrochloric acid standard propan-2-ol solution at room temperature using *p*-naphtol-benzen as indicator. For testing the strong acid number, extract the strong acid constituents in the sample with hot water, and titrate with potassium hydroxide standard propan-2-ol solution with methyl orange as indicator.

Remarks 1. This standard specifies the indicator titration method for the determination of acidic or basic constituents in petroleum products and lubricants soluble in a mixture of toluene and propan-2-ol.

It is applicable for the determination of acidic constituents or weak basic ones whose dissociation constants in water are greater than 10^{-9} , but not for extremely weak acid constituents or weak basic ones whose dissociation constants are less than 10^{-9} . Salts react if their hydrolysis constants are greater than 10^{-9} .

2. In new and used oils, the constituents that may be considered to have acidic characteristics, include organic acids, inorganic acids, esters, phenolic compounds, lactones, resins, salts of heavy metals, and additives such as inhibitors and detergents. Similarly, constituents considered to have basic properties include organic and inorganic bases, amino compounds, salts of weak acids (soaps), basic salts of polyacidic bases, salts of heavy metals, and additives such as inhibitors and detergents.
3. Mostly, this method is not suitable for measuring the basic constituents of basic additive-type oils, the potentiometric titration method can be applied for this purpose.

Informative reference 1. This standard may be used to indicate relative changes that occur in an oil during use under oxidizing conditions. Although the titration is made under definite equilibrium conditions, this method does not measure an absolute acidic or basic property that can be used to predict performance of an oil under service conditions.

Further, no general relationship between corrosiveness and acid or base number is known.

2. Oils such as many cutting oils, rust proofing oils, compound oils, or excessively dark-coloured oil that cannot be analyzed by this method owing to obscurity of the colour-indicator end-point are preferably analyzed in accordance with potentiometric titration method. The numbers obtained by this indicator titration method may or may not be numerically the same as those obtained by potentiometric titration method.

4.2 Reagent

- (1) 0.1 mol/l hydrochloric acid standard propan-2-ol solution
(hereafter referred to as 0.1 mol/l HCL solution)

- (1.1) Preparation Add 9 ml of hydrochloric acid specified in JIS K 8180 to 1 l of propan-2-ol, and mix them.

(1.2) Standardization The standardization shall be carried out according to the following operations:

- (a) Weigh out correctly approximately 8 ml of propan-2-ol solution of hydrochloric acid and dilute with 125 ml of carbon-dioxide-free water.
- (b) Titrate with 0.1 mol/l of potassium hydroxide (KOH) solution with phenolphthalein as indicator, or titrate potentiometrically.
- (c) Obtain the molarity of propan-2-ol solution of hydrochloric acid down to the fourth decimal place according to the following formula:

$$c_{\text{HCL}} = \frac{c_{\text{KOH}} \times v_1}{v_2}$$

where, c_{HCL} : molarity of 0.1 mol/l HCL solution (mol/l)

c_{KOH} : molarity of 0.1 mol/l KOH solution (mol/l)

v_1 : volume of 0.1 mol/l KOH solution required for titration until the end point (ml)

v_2 : weighed out quantity of propan-2-ol solution of hydrochloric acid (ml)

- (d) Standardize frequently enough the molarity of 0.1 mol/l HCL solution so as to detect the changes of 0.0005 mol/l as the maximum.
- (2) Methyl orange indicator solution Dissolve 0.1 g of methyl orange specified in JIS K 8893 in 100 ml of water.
- (3) p-naphtholbenzein indicator solution Dissolve 1.0 g of p-naphtholbenzein specified in Annexes A and B in ISO 6618 in 100 ml of titration solvent.

Informative reference: As to p-naphtholbenzein, "CAS #145-50-6" of Kodak Company conforms to the requirements of this standard.

- (4) 0.1 mol/l Potassium hydroxide standard propan-2-ol solution (hereinafter referred to as 0.1 mol/l KOH solution)

(4.1) Preparation Take appropriate 1 l of propan-2-ol into a 2 l conical flask, add 6 g of potassium hydroxide specified in JIS K 8574, boil the mixture gently for 15 to 20 min., stirring to prevent solids from forming a cake on the bottom. Stand this solution for several days avoiding exposure to carbon dioxide, transfer the supernatant liquid into a polyethylene bottle or the like for preservation which equips a guard tube containing soda lime and desiccating agent, and store.

(4.2) Standardization The standardization with potassium hydrogen phthalate shall be carried out as follows:

- (a) Dry potassium hydrogen phthalate specified in JIS K 8809 at 120°C for 2 hours, weigh out correctly its 0.1 to 0.2 g to the nearest 0.1 mg, dissolve in 125 ml of carbon-dioxide-free water, and titrate potentiometrically with propan-2-ol solution of potassium hydroxide.
- (b) Carry out a blank test on 125 ml of water according to the operation (a).
- (c) Obtain the molarity of propan-2-ol solution of potassium hydroxide down to the fourth decimal place according to the following formula:

$$c\text{KOH} = \frac{1000 W}{204.230 \times (v_1 - b)}$$

where, $c\text{KOH}$: molarity of 0.1 mol/l KOH solution (mol/l)

W : weighed out mass of potassium hydrogen phthalate (g)

v_1 : volume of propan-2-ol solution of potassium hydroxide required for titration until the end point (ml)

b : volume equivalent to v_1 in blank test (ml)

- (d) Standardize the molarity frequently enough of the 0.1 mol/l HCL solution so as to detect the change of 0.0005 mol/l as the maximum.

Remarks 1. Standardization according to amidosulfuric acid shall be carried out as follows:

- (a) Dry crystal powder of amidosulfuric acid specified in JIS K 8005 in a vacuum desiccator for approximately 48 hours, weigh out its 2.0 to 2.5 g to the nearest 0.1 mg, dissolve in carbon-dioxide-free water, transfer into a 250 ml measuring flask and add water up to the marked line.
- (b) Take its 25 ml with a transfer pipette, and add 100 ml of water. Titrate it with propan-2-ol solution of potassium hydroxide with bromothymol blue prepared according to JIS K 8001 as indicator.
- (c) Carry out a blank test with 125 ml of water.

$$c\text{KOH} = \frac{100 W}{97.093 \times (v_1 - b)}$$

where, $c\text{KOH}$: molarity of 0.1 mol/l KOH solution (mol/l)

W : weighed out mass of amido-sulfuric acid (g)

v_1 : volume of propan-2-ol solution of potassium hydroxide required for titration until the end point (ml)

b : volume equivalent to v_1 in blank test (ml)

2. Because of the relatively large coefficient of cubic expansion of organic solvent such as propan-2-ol, the 0.1 mol/l KOH solution should be standardized at temperatures close to those employed in the titration of sample.

- (5) Propan-2-ol That specified in JIS K 8839.
- (6) Phenolphthalein indicator solution Dissolve 1.0 g of phenolphthalein specified in JIS K 8799 in 100 ml of ethanol specified in JIS K 8102.
- (7) Titration solvent Add 500 ml of toluene specified in JIS K 8680 and 5 ml of water to 495 ml of propan-2-ol.

4.3 Apparatus

Burette That of capacity 50 ml, graduated in 0.1 ml subdivisions, or that of capacity 10 ml, graduated in 0.05 ml subdivisions.

Remarks: A motor-driven burette which has the equivalent performance to that mentioned above may be used. As for a burette made of glass, that shown in Fig. 99 or Fig. 100 in JIS K 2839 are suited.

4.4 Preparation of a sample of used oil Preparation of a sample of used oil shall be carried out as follows:

- (1) When sediment exists in the sample, the sediment itself may be acidic or basic, or it may have absorbed acidic or basic substance. Therefore, in weighing out samples which contain such sediment, it is essential to take mixing thoroughly by stirring so as to represent the whole test sample.

Remarks 1. A sample for test shall be taken according to the methods for sampling and preparation of secondary sample specified in JIS K 2251 or other similar methods.

2. As used oil may change in quality in storage, samples shall be tested as soon as possible after sampling.

- (2) Heat the sample to $60 \pm 5^\circ\text{C}$ to homogenize the sediment. When the sample is visibly free from sediment, the heating procedure may be omitted.
- (3) After complete suspension of all sediment, strain the sample through a $149\ \mu\text{m}$ (100-mesh) screen to remove large contaminating substances.

4.5 Procedure of test4.5.1 Total acid number

- (1) Weigh out sample into a 250 ml conical flask according to Table 2, add 100 ml of titration solvent and 0.5 ml of *p*-naphtholbenzein indication solution and mix by shaking without a stoppering until the sample is entirely dissolved in the solvent. If the mixture assumes yellow-orange colour, proceed the operation as specified in (2). If the colour of the mixture becomes green or green-dark, carry out measurement of strong base number as specified in 4.5.2.

Table 2. Mass of sample to be weighed out

Kind of sample	Total acid number or total base number mgKOH/g	Mass of sample to be weighed out g	Minimum graduation for weighing out g
New or light-coloured oils	Over 0 to 3 incl.	20 ± 2	0.05
	Over 3 to 25 incl.	2 ± 0.2	0.01
	Over 25	0.2 ± 0.02	0.001
Used or dark-coloured oils	Over 0 to 25 incl.	2 ± 0.2	0.01
	Over 25	0.2 ± 0.02	0.001

Remarks: Light-coloured sample of low acid number permit the use of 20 g test portions to obtain more precise results. The test portion size for dark-coloured oils is limited to the quantity specified to minimize possible interference by the dark colour.

- (2) Titrate immediately with 0.1 mol/l KOH solution at a temperature below 30°C. Shake sufficiently near the end-point, but avoid dissolving carbon dioxide in the solution. In the case of acidic oils, the yellow-orange colour changes to a green or green-brown as the end-point is approached. Consider the end-point definite if the colour change persists for 15 s or if it reverses on addition of 2 drops of 0.1 mol/l HCL solution.

Remarks: In the case of dark-coloured oil, observe the change of colour by titration by effervescence with vigorous shaking or under a fluorescent lamp.

- (3) Carry out a blank test on 100 ml of the titration solvent and 0.5 ml of the *p*-naphtholbenzein indicator solution, adding the 0.1 mol/l KOH solution in 0.05 to 0.1 ml increments.

4.5.2 Strong base number

- (1) If the mixed solution obtained in the operation of 4.5.1 (1) assumes a green colour or green-dark, apply the operation as specified in 4.5.1 (2), and carry out the titration until the colour of the mixed solution changes to yellow-orange with 0.1 mol/l HCL solution instead of 0.1 mol/l KOH solution.

- (2) Carry out a blank test according to 4.5.1 (3).

Remarks: Since the titration solvent usually contains weak acid impurities which react with strongly basic components of the test portion, it is thus necessary to determine the amount of weakly acidic components in the solvent for the purpose of obtaining the strong base number of the sample.

4.5.3 Strong acid number

- (1) Weigh out approximately 25 g of sample into a 250 ml separating funnel to within 0.05 g, add 100 ml of boiling water, and after shaking vigorously, separate aqueous phase, and collect it in a 500 ml beaker. Further, extract twice the acidic constituents in oil with 50 ml of boiling water, and add the separated aqueous phase to the same beaker. Add 0.1 ml of methyl orange indicator solution to this water extract, and when it assumes pink or red, titrate with 0.1 mol/l KOH solution until the colour becomes golden brown.
If the initial colour is not pink or red, take the strong acid number as zero.
- (2) For the blank test, into a 250 ml conical flask, introduce 200 ml of boiling water from the same source used in (1). Add 0.1 ml of methyl orange indicator solution, when the colour of the indicator assumes yellow-orange, titrate with 0.1 mol/l HCL solution to the same depth and shade of colour obtained in the titration of sample. If the indicator colour assumes pink or red, titrate with 0.1 mol/l KOH solution to the same end point as that used in titration of sample.

4.6 Results

4.6.1 Method of calculation Calculate the total acid number, strong acid number, and strong base number according to the following formula, and round off to the three significant figures according to JIS Z 8401. However, in the case of less than 1 mg KOH/g, round off to the two decimal place.

- (1) Total acid number

$$AN = \frac{56.1 \times c_{\text{KOH}} (V_1 - V_0)}{m}$$

where, AN : total acid number (mgKOH/g)

V_1 : volume of 0.1 mol/l KOH solution required for titration of the sample (ml)

V_0 : volume of 0.1 mol/l KOH solution required for titration of the blank solution (ml)

c_{KOH} : molarity of 0.1 mol/l KOH solution (mol/l)

m : weighed out mass of the sample (g)

(2) Strong acid number

(a) If the blank test is carried out with 0.1 mol/l HCL solution:

$$SAN = \frac{56.1(V_2 \cdot cKOH + V_3 \cdot cHCL)}{m}$$

where, SAN : strong acid number (mgKOH/g)

 V_2 : volume of 0.1 mol/l KOH solution required for titration of the water extract obtained by the operation given in 4.5.3 (2) (ml) $cKOH$: molarity of 0.1 mol/l KOH solution (mol/l) V_3 : volume of 0.1 mol/l HCL solution required for titration of the blank solution (ml) $cHCL$: molarity of 0.1 mol/l HCL solution (mol/l) m : weighed out mass of the sample (g)

(b) If the blank test is carried out with 0.1 mol/l KOH solution:

$$SAN = \frac{56.1 \times cKOH (V_2 - V_4)}{m}$$

where, SAN : strong acid number (mgKOH/g)

 V_2 : volume of 0.1 mol/l KOH solution required for titration of the water extract obtained by the operation given in 4.5.3 (2) (ml) V_4 : volume of 0.1 mol/l KOH solution required for titration of the blank solution (ml) $cKOH$: molarity of 0.1 mol/l KOH solution (mol/l) m : weighed out mass of the sample (g)(3) Strong base number

$$SBN = \frac{56.1(V_5 \cdot cHCL + V_6 \cdot cKOH)}{m}$$

where, SBN : strong base number (mgKOH/g)

 V_5 : volume of 0.1 mol/l HCL solution required for titration of the sample (ml) $cHCL$: molarity of 0.1 mol/l HCL solution (mol/l) V_6 : volume of 0.1 mol/l KOH solution required for titration of the blank solution (ml) $cKOH$: molarity of 0.1 mol/l KOH solution (mol/l) m : weighed out quantity of the sample (g)

4.6.2 Precision The tolerance (probability is 0.95) on test results obtained by the indicator titration method shall be as follows:

- Remarks 1. The precision defined below is not applicable to oils that are too much coloured or obscure concerning the change of colour of indicator.
2. When the results fall outside the tolerance range, deal with by following JIS Z 8402.

- (1) Repeatability The tolerance on difference between two test results obtained by the same operator with the same apparatus on identical test material in different date or time shall be as given in Table 3.

Table 3. Repeatability

Kind of sample	Neutralization number mgKOH/g	Tolerance on repeatability mgKOH/g
New or light-colored oil	Over 0.0 to 0.1 incl.	0.03
	Over 0.1 to 0.5 incl.	0.05
	Over 0.5 to 1.0 incl.	0.08
	Over 1.0 to 2.0 incl.	0.12

- (2) Reproducibility The tolerance on difference between both single and independent test results, obtained by different operators working in different laboratories with respective apparatuses on identical sample, shall be as given in Table 4.

Table 4. Reproducibility

Kind of sample	Neutralization number mgKOH/g	Tolerance on reproducibility mgKOH/g
New or light-colored oil	Over 0.0 to 0.1 incl.	0.04
	Over 0.1 to 0.5 incl.	0.08
	Over 0.5 to 2.0 incl.	15 % of average value

4.7 Report of test results The test report shall enter the following details:

- (1) Name of sample, and location and date of sampling
- (2) A reference to JIS number
- (3) Test items for total acid number, strong acid number, or strong base number, and test results obtained by 4.6.1.
- (4) Specific description

5. Potentiometric titration method (acid number)

5.1 Principle of test The sample is dissolved in a mixture of toluene and propan-2-ol containing a small amount of water, and titrated potentiometrically with potassium hydroxide standard propan-2-ol solution using a glass electrode and a reference electrode. The meter readings (of potentiometer or pH meter) are plotted against the respective volumes of titrating standard solution and the end points are taken at inflection point(s) obtained in the resulting titration curve.

When no definite inflections are obtained, end points are taken at meter readings corresponding to those found for non-aqueous acidic or basic buffer solutions.

- Remarks 1. This standard is applicable for the determination by potentiometric method of acidic constituents in petroleum products and lubricants soluble or nearly soluble in mixtures of toluene and propan-2-ol. It is applicable for the determination of acidic constituents whose dissociation constants in water are greater than 10^{-9} , but not for extremely weak acidic ones whose dissociation constants are less than 10^{-9} . Salts react if their hydrolysis constants are greater than 10^{-9} .
2. In new and used oils, the constituents that may be considered to have acidic characteristics, include organic acids, inorganic acids, esters, phenolic compounds, lactons, resins, salts of heavy metals, salts of ammonia and other weakly acid constituents, acid salts of polybasic acids, and additives such as inhibitors and detergents.

Informative reference 1. This standard may be used to indicate relative changes that occur in an oil during use under oxidizing conditions. Although the titration is made under definite equilibrium conditions, this method does not measure an absolute acidic or basic property that can be used to predict performance of an oil under service conditions.

Further, no general relationship between corrosiveness and acid or base number is known.

2. The values obtained by this potentiometric titration method may or may not be numerically the same as those obtained by indicator titration method.
3. New and used petroleum products contain acidic constituents that are present as additives and products formed during use, such as oxidized products. The relative amounts of these materials can be determined by titrating with bases. The acid number is a measure of this amount of acidic substances in the oil under the conditions of the test. The acid number is used as a guide in the quality control of lubricating oil formulations. It is also sometimes used as a measure of lubricant degradation by oxidation in service, but does not give the condemning limit.

As a variety of oxidation products contribute to the acid number and organic acids vary widely in corrosion property, the acid number can not be used to predict corrosiveness. No general correlation is known between acid number and the corrosive tendency of oils toward metals.

5.2 Reagents

- (1) Non-aqueous acidic buffer solution Add 10 ml of buffer stock solution A to 100 ml of titration solvent, and mix them. Use within 1 h.
- (2) Non-aqueous basic buffer solution Add 10 ml of buffer stock solution B to 100 ml of titration solvent and mix them. Use within 1 h.
- (3) Buffer stock solution A Weigh 24.2 ± 0.1 g of 2,4,6-trimethylpyridine (γ -collidine) transfer into a 1 l volumetric flask containing 100 ml of propan-2-ol, add $150/M_1 \pm 5$ ml (where M_1 is molarity of 0.5 mol/l HCL solution) of 0.2 mol/l HCL solution, dilute the whole to 1 l with propan-2-ol, store this reagent in a brown glass bottle, and use within 1 month.

The characteristics of 2,4,6-trimethylpyridine (γ -collidine) are as follows:

Boiling point: 168 to 170°C

Refractive index (n_D^{20}): 1.4982 ± 0.0005

Colour: Colourless

- (4) Buffer stock solution B Weigh 27.8 ± 0.1 g of *m*-nitrophenol, transfer into a 1 l volumetric flask containing 100 ml of propan-2-ol, add $50/M_2 \pm 1$ ml (where M_2 is molarity of 0.5 mol/l KOH solution) of 0.2 mol/l KOH solution, dilute the whole to 1 l with propan-2-ol, store this reagent in a brown glass bottle, and use within 1 month.

The characteristics of *m*-nitrophenol are as follows:

Melting point: 96 to 97°C

Colour: Pale yellow

- (5) 0.1 mol/l hydrochloric acid standard propan-2-ol solution
(hereinafter referred to as 0.1 mol/l HCL solution)
 - (5.1) Preparation Add 9 ml of hydrochloric acid specified in JIS K 8180 to 1 l of propan-2-ol, and mix them.
 - (5.2) Standardization The standardization shall be carried out according to the following operations:
 - (a) Weigh out correctly about 8 ml of propan-2-ol of hydrochloric acid, and dilute with 125 ml of carbon dioxide-free water.
 - (b) Titrate with 0.1 mol/l KOH solution with phenol-phthalein as indicator, otherwise titrate potentiometrically.

- (c) Obtain the molarity of propan-2-ol solution of hydrochloric acid down to the fourth decimal place according to the following formula:

$$c_{\text{HCL}} = \frac{c_{\text{KOH}} \times v_1}{v_2}$$

where, c_{HCL} : molarity of 0.1 mol/l HCL solution (mol/l)

c_{KOH} : molarity of 0.1 mol/l KOH solution (mol/l)

v_1 : volume of 0.1 mol/l KOH required for titration until the end point (ml)

v_2 : weighted out quantity of propan-2-ol solution of hydrochloric acid (ml)

- (d) Standardize the molarity frequently enough of 0.1 mol/l HCL solution so as to detect the change of 0.0005 mol/l as the maximum.
- (6) 0.2 mol/l Hydrochloric acid standard propan-2-ol solution (hereafter referred to as 0.2 mol/l HCL solution) Add 18 ml of hydrochloric acid specified in JIS K 8180 to 1 l of propan-2-ol, mix for preparation, and standardize according to (5.2).
- (7) Propan-2-ol That specified in JIS K 8839.
- (8) Potassium chloride electrode solution (hereafter referred to as KCL solution) Use concentrated solution of which potassium chloride specified in JIS K 8121 is dissolved in water so as to be 3 mol/l or more in concentration.
- (9) 0.1 mol/l Potassium hydroxide standard propan-2-ol solution (hereafter referred to as 0.1 mol/l KOH solution)
- (9.1) Preparation Take approximately 1 l of propan-2-ol into a 2 l conical flask, add 6 g of potassium hydroxide specified in JIS K 8574, boil the mixture gently for 15 to 20 minutes stirring to prevent solids from forming a cake on the bottom. Let this solution stand for several days avoiding exposure to carbon dioxide, transfer the supernatant liquid into a polyethylene bottle or the like for preservation which equips a guard tube containing soda lime and desiccating agent, and then store.
- (9.2) Standardization Standardization with potassium hydrogen phthalate shall be carried out as follows:
- (a) Dry potassium hydrogen phthalate specified in JIS K 8809 at 120°C for 2 h, weigh out correctly its 0.1 to 0.2 g to the nearest 0.1 mg, dissolve in 125 ml of carbon-dioxide-free water, and titrate potentiometrically with propan-2-ol solution of potassium hydroxide.
- (b) Carry out the blank test on 125 ml of water according to the operation (a).

- (c) Obtain the molarity of propan-2-ol solution of potassium hydroxide down to the fourth decimal place according to the following formula:

$$c_{\text{KOH}} = \frac{1000 W}{204.230 \times (v_1 - b)}$$

where, c_{KOH} : molarity of 0.1 mol/l KOH solution (mol/l)

W : weighed out mass of potassium hydrogen phthalate (g)

v_1 : volume of propan-2-ol solution of potassium hydroxide required for titration until the end point (ml)

b : volume equivalent to v_1 in blank test (ml)

- (d) Standardize frequently enough the molarity of 0.1 mol/l KOH solution so as to detect the change of 0.0005 mol/l as the maximum.

Remarks 1. Standardization with amidosulfuric acid shall be carried out as follows:

- (a) Dry crystal powder of amidosulfuric acid specified in JIS K 8005 in a vacuum desiccator for approximately 48 h, read its 2.0 to 2.5 g to the nearest 0.1 mg, dissolve in carbon-dioxide-free water, transfer into a 250 ml volumetric flask, and add water up to the marked line.
- (b) Take its 25 ml by using a transfer pipette, and add 100 ml of water. Titrate it with propan-2-ol solution of potassium hydroxide with bromothymol blue prepared according to JIS K 8001 as indicator.
- (c) Carry out blank test with 125 ml of water.

$$c_{\text{KOH}} = \frac{100 W}{97.093 \times (v_1 - b)}$$

where, c_{KOH} : molarity of 0.1 mol/l KOH solution (mol/l)

W : weighed out mass of amidosulfuric acid (g)

v_1 : volume of propan-2-ol solution of potassium hydroxide required for titration until the end point (ml)

b : volume equivalent to v_1 in blank test (ml)

2. Because of the relatively large coefficient of cubic expansion of organic solvent such as propan-2-ol, the 0.1 mol/l KOH solution should be standardized at temperatures close to those employed in the titration of sample.

- (10) 0.2 mol/l Potassium hydroxide standard propan-2-ol solution (hereafter referred to as 0.2 mol/l KOH solution) Dissolve 12 g of potassium hydroxide specified in JIS K 8574 in approximately 1 l of propan-2-ol, and prepare and standardize according to (9).
- (11) Titration solvent Add 500 ml of toluene specified in JIS K 8680 and 5 ml of water to 495 ml of propan-2-ol.

Remarks: A titration solvent which contains chloroform may be used instead of toluene to dissolve completely certain heavy residues in oil or asphaltic materials.

5.3 Neutralization number apparatus An example of an apparatus used for the potentiometric titration is shown in Fig. 1.

Remarks: The insulating performance of an apparatus shall be 5 MΩ or more when the insulation resistance between the power source terminal and the container is measured by closing the electric circuit.

Fig. 1. Neutralization number apparatus (potentiometric titration method) (an example)

