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BUREAU OF INDIAN STANDARDS

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भारतीय मानक मासौदा

पेट्रोलियम और उसके उत्पाद — परीक्षण पद्धतियाँ भाग 1
पेट्रोलियम उत्पादों की आधार संख्या का निर्धारण
अनुभाग 2 पोटेंशियोमेट्रिक अनुमापन
(IS 1448 भाग 1/ अनुभाग 2 का *तीसरा पुनरीक्षण*)

Draft Indian Standard

PETROLEUM AND ITS PRODUCTS — TEST METHODS
PART 1 DETERMINATION OF BASE NUMBER OF PETROLEUM PRODUCTS
SECTION 2 POTENTIOMETRIC TITRATION

[*Third Revision* of IS 1448 (Part 1/Sec 2)]

(ICS 75.080)

Methods of Sampling and Test for Petroleum and related Products of Natural or Synthetic Origin (excluding bitumen) Sectional Committee PCD 01	Last date for receipt of comment is **05 January 2025**

FOREWORD

(*Formal clause will be added later*)

The standard IS 1448 Part 1 was originally published in 1971 covering the determination of acidic or basic constituents in petroleum products and lubricants. This standard was based on joint publication of ASTM D664-58 and IP 177-64. ASTM D664 was revised in 1989 covering only determination of acid number and a separate standard was published for determination of base number, namely, ASTM D4739-92. Base number portion was deleted from ASTM D664-89 due to poor precision caused mainly by the following:

a) Poorly defined inflection points;
b) Poor solvency with non-polar samples;
c) Contamination of electrodes;
d) Poorly specified end-point selection criteria; and
e) A tedious procedure that is unsuitable for automatic titrators.

Considering the revision of ASTM D664 in 1989, and publication of ASTM D4739 in 1992, this standard was revised for second time in 2002 and the standard was divided into two sections, namely, section 1 to cover determination of acid number of petroleum products and Section 2 to cover determination of base number of petroleum products. Following major changes were made during the second revision:

a) Title was modified as ‘Determination of base number of petroleum products by potentiometric titration’;
b) Test method prescribed for determination of acid number was deleted and method for acid number determination was published as, IS 1448 (Part 1 /Section 1).
c) The standard was aligned with ASTM D4739 and precision was improved through introduction of following changes:

    i) Modification of the titration solvent by including chloroform,
    ii) Clear specification of end-point selection criteria, and
    iii) Usage of fixed increment/fixed time titration mode.

The third revision has been brought out to keep pace with the latest technological developments and international practices. In this revision, the following major change has been made:

a) Use of commercially available buffer solutions has been incorporated.

In the preparation of this standard, considerable assistance has been derived from the following standards:

ASTM D4739-17 Standard Test Method for Base Number Determination by Potentiometric Hydrochloric Acid Titration

In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS 2:2022 ‘Rules for rounding off numerical values (*second revision*)’.

1 SCOPE

1.1 This standard (Part 1/Section 2) prescribes the method of test for determination of basic constituents in petroleum products and lubricants (*see* Note). The test method resolves these constituents into groups having weak-base and strong-base ionization properties, provided the dissociation constants of the more strongly basic compounds are at least 1000 times that of the next weaker groups.

NOTE — In new and used oils, the constituents which can be considered to have basic properties are primarily organic and inorganic bases including amino compounds, although certain salts of heavy metals, salts of weak acids, basic salts of polyacidic compounds, and some additives, such as inhibitors or detergents may show basic characteristics.

1.1.1 This test method covers base numbers up to 70. While it can be extended to higher base numbers, the precision of the test method for base numbers greater than 70 has not been determined.

1.2 This test method may be used to indicate relative changes that occur in an oil during use under oxidizing or other service conditions regardless of the colour or other properties of the resulting oil. Although the analysis is made under closely specified conditions, the method is not intended to predict performance of an oil, under all service conditions; for example, no overall relationship is known between bearing corrosion or the control of corrosive wear in the engine and base number.

2 REFERENCES

The following Indian Standards contain provisions which, through reference in this text, constitute provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent edition of the standard indicated below:

IS No.	*Title*
IS 1070: 2023	Reagents Grade Water — Specification (*fourth revision*)

3 TERMINOLOGY

3.1 Base Numbers — The quantity of acid, expressed in milligrams of potassium hydroxide per gram of sample (*see* Note) that is required to titrate a sample to a specified end-point.

> NOTE — In this test method, the sample is titrated to a meter reading corresponding to a freshly prepared non-aqueous acidic buffer solution or appropriate inflection point.

3.2 Strong Base Number — The quantity of acid, expressed in terms of the equivalent number of milligrams of potassium hydroxide per gram of sample that is required to titrate a sample dissolved in the specified solvent from the initial meter reading to a meter reading corresponding to a freshly prepared basic buffer solution.

4 OUTLINE OF THE TEST METHOD

The sample is dissolved in a mixture of toluene, isopropyl alcohol, chloroform, and a small amount of water and titrated potentiometrically with alcoholic hydrochloric acid solution. The test results of this procedure are obtained by titration mode of fixed increment and fixed time additions of the titrant. An end-point is selected from a titration curve according to the criteria given in **12.1** and used to calculate a base number.

5 SIGNIFICANCE AND USE

New and used petroleum products can contain basic constituents that are present as additives or as degradation products formed during service. The relative amount of these materials can be determined by titrating with acids. The base number is a measure of the amount of basic substances in the oil always under the conditions of the test. The base 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 in service.

6 APPARATUS

6.1 Potentiometric Titrimeter

Automatic or manual with capability of adding fixed increments of titrant at fixed time intervals (*see* Annex A).

6.1.1 The titrimeter shall be capable of automatically or manually controlling the rate of addition of titrant as given in **6.1.2**.

6.1.2 Delivery of titrant will be incremental after delivery of precisely a 0.100 ml increment (*see* **6.1.3**), the delivery is stopped and a fixed time period of 90 s is allowed to pass before another 0.100 ml increment of titrant is delivered. This procedure is repeated until the titration is completed.

6.1.3 The precision of addition of the 0.100 ml increments of titrant should be + 0.001 ml for automatic titrators. For manual burette, it should be + 0.005 ml.

6.2 Glass Indicating Electrode — *p*H 0 to 14, general purpose.

6.3 Reference Electrode — Sleeve type, saturated calomel electrode, or Ag/AgCl electrode, with inner and outer electrolyte filling solutions of saturated lithium chloride (LiCl) in isopropyl alcohol (*see* Note).

NOTE — Combination electrodes, because of their slow response, have been found unsuitable for this test method.

6.4 Stirrer, burette, stand, titration vessel, as specified in Annex A are required. A typical cell assembly is shown in Fig. 1.

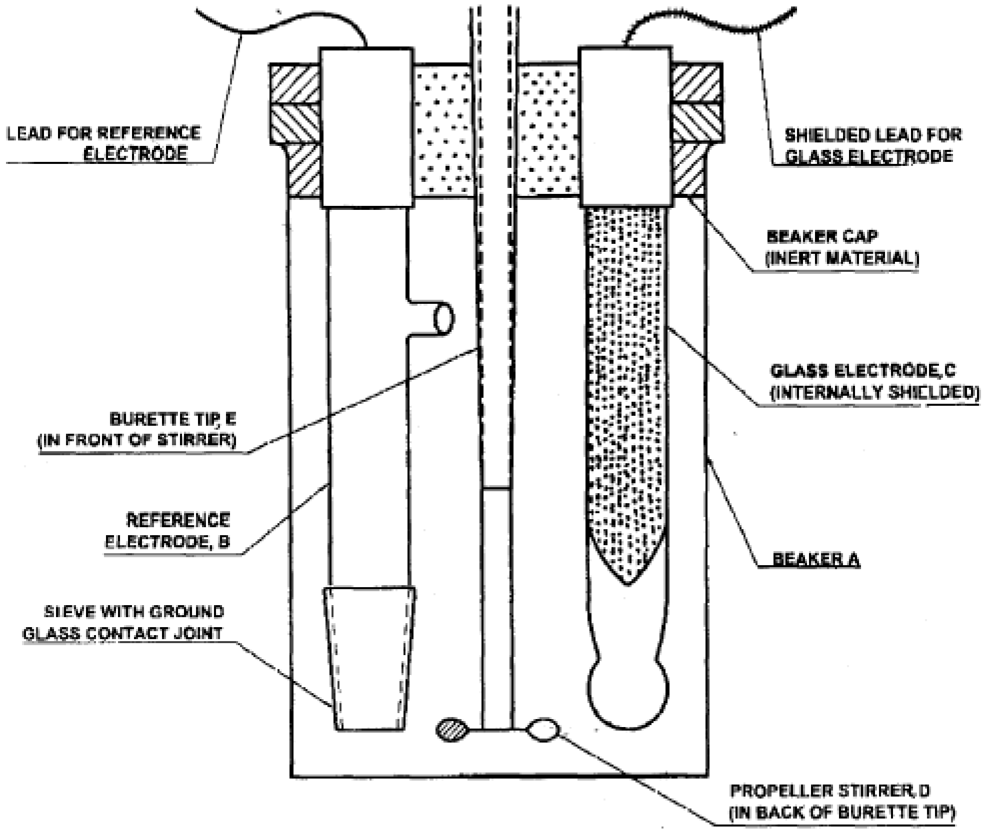

FIG. 1 CELL FOR POTENTIOMETRIC TITRATION

7 REAGENTS

7.1 Purity of Reagents

Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

7.2 Purity of Water

Unless otherwise indicated, references to water shall be understood to mean reagent water (*see* IS 1070).

7.3 Buffer, Aqueous Acid

Commercial *p*H 3 buffer solution with a tolerance of ± 0.02 *p*H units at 25 °C. This solution shall be replaced at regular intervals consistent with its stability or when contamination is suspected. Information related to the stability should be obtained from the manufacturer.

7.4 Buffer, Aqueous Basic

Commercial *p*H 10 buffer solution with a tolerance of ± 0.02 *p*H units at 25 °C. This solution shall be replaced at regular intervals consistent with its stability or when contamination is suspected. Information related to the stability should be obtained from the manufacturer.

7.5 Chloroform — Reagent grade

7.6 Hydrochloric Acid Standard Solution, Alcoholic (0.1 N)

Mix 9 ml of reagent grade hydrochloric acid (specific gravity 1.19), with 1 litre of anhydrous isopropyl alcohol. Standardize frequently enough to detect normality changes of 0.0005 by potentiometric titration with 8 ml of the 0.1 N alcoholic KOH solution (*see* **7.10**) diluted with 125 ml CO_2 free water.

7.7 Hydrochloric Acid Standard Solution, Alcoholic (0.2 N)

Prepare and standardize as described in **7.6**, but use 18 ml of HCl (specific gravity 1.19).

7.8 Isopropyl Alcohol (2-Propanol) Anhydrous (Less than 0.1 Percent H_2O)

In case of non-availability of dry reagent, dry it by distillation through a multiple plate column, discarding the first 5 percent of material distilling over and using the 95 percent remaining. Also, drying can be accomplished using molecular sieves such as Linde Type 4A, by passing the solvent upward through a molecular sieve column using 1 part of molecular sieve per 10 parts of solvent.

7.9 Lithium Chloride Electrolyte

Prepare a saturated solution of lithium chloride (LiCl) in isopropyl alcohol.

7.10 Potassium Hydroxide Solution, Standard Alcoholic (0.1 N)

Add 6 g of reagent grade potassium hydroxide (KOH) to approximately 1 litre of anhydrous isopropyl alcohol. Boil gently for 10 min to effect solution. Allow the solution to stand for 2 days and then filter the supernatant liquid through a fine sintered glass funnel. Store the solution in a chemically resistant bottle. Dispense in a manner such that the solution is protected from atmospheric carbon dioxide (CO_2) by means of a guard tube containing soda lime or soda non-fibrous silicate absorbent (Ascarite, Carbosorb, or Indicarb), and such that it does not come into contact with cork, rubber, or saponifiable stopcock grease. Standardize frequently enough to detect normality changes of 0.0005 by potentiometric titration with weighed quantities of potassium acid phthalate dissolved in CO_2 free water.

7.11 Potassium Hydroxide Solution, Standard Alcoholic (0.2 N)

Prepare, store and standardize as directed in **7.10**, but use 12 g to 13 g of KOH with approximately 1 litre of anhydrous isopropyl alcohol.

7.12 Titration Solvent

In a brown reagent bottle, add 30 ml of water to 1 litre of isopropyl alcohol and mix thoroughly. Add 1 litre each of toluene and chloroform and mix thoroughly.

7.13 Toluene — Reagent grade

7.14 Chromic Acid Solution

NOTE — Causes severe burns. Recognized carcinogen, strong oxidizer.

8 PREPARATION OF ELECTRODE SYSTEM

8.1 Maintenance of Electrodes

8.1.1 Clean the glass electrode (*see* Note) at frequent intervals (not less than once every week during continual use) by immersing in cold chromic acid cleaning solution. Drain the calomel electrode at least once in each week and refill with fresh LiCl electrolyte as far as the filling hole. Make sure that crystallized LiCl is present in the solution. Maintain the electrolyte level in the reference electrode above that of the liquid in the titration beaker at all times. When not in use, immerse the lower half of glass electrodes in water and calomel electrode in saturated solution of LiCl in isopropyl alcohol. Do not allow them to remain immersed in titration solvent for any appreciable period of time between titrations. While the electrodes are not extremely fragile, handle them carefully at all times.

NOTE — Cleaning the electrodes thoroughly, keeping the ground-glass joint free of foreign materials, and regular testing of the electrodes are important in obtaining repeatable potentials, since contamination can introduce uncertain, erratic, and unnoticeable liquid contact potentials. While this is of secondary importance, when end points are chosen from inflection points in the titration curve, it is quite serious when end points are chosen at arbitrarily fixed cell potentials (that is, the non-aqueous acidic buffer potentials).

8.1.2 When Ag/AgCl reference electrode is used for the titration and it contains an electrolyte that is not 1 m to 3 m LiCl in ethanol, replace the electrolyte. Drain the electrolyte from the electrode, wash away all the salt (if present) with water and then rinse with ethanol. Rinse several times with the LiCl electrolyte solution. Finally, replace the sleeve and fill the electrode with the LiCl electrolyte to the filling hole. When refitting the sleeve, ensure that there will be a free flow of electrolyte into the system. A combination electrode shall be prepared in the same manner. The electrolyte in a combination electrode can be removed with the aid of a vacuum suction. Prior to each titration, soak the prepared electrodes in water, *p*H 4.5 – 5.5 acidified with HCl, for at least 5 min. Rinse the electrode with propan-2-ol immediately before use, then with the titration solvent.

8.2 Testing of Electrodes

Test the meter-electrode combination (*see* Note) when it is first put into use or when new electrodes are installed and retest at intervals thereafter by dipping the electrodes into a well-stirred mixture of 100 ml of the titration solvent and 1.0 ml to 1.5 ml of 0.1 N alcoholic KOH solution (*see* **7.10**). For the meter-electrode combination to be suitable for use, the potential between the electrodes must change by more than 0.480 V from the potential between the same electrodes when dipped in the non-aqueous acidic buffer solution.

NOTE — Considerably more sensitive electrodes are now available that will show a potential change of at least 0.590 V under these conditions, and their use is recommended.

9 STANDARDIZATION OF APPARATUS

9.1 Determination of Meter Readings for the Aqueous Buffer Solution

Ensure comparable selection of end points when definite inflection points are not obtained in the titration curve. Determine daily for each electrode pair, the meter reading obtained with the aqueous acidic buffer solution to be used for the determination of base numbers, and with the aqueous basic buffer solution to be used for the determination of strong base numbers.

9.2 Prepare the electrodes as described in **8.2,** immerse them in the appropriate non-aqueous buffer solution, and stir for at least 2 min, maintaining the temperature of the buffer solution at a temperature within 2 ^{o}C of that at which the titrations are to be made. Read the cell voltage. The reading so obtained in the acidic buffer solution is taken as the end point for the base number if an inflection is not observed and the reading obtained in the basic buffer solution is taken as the end point for the strong base number.

10 PREPARATION OF SAMPLE OF USED OIL

10.1 Strict observation of the sampling procedure is necessary, since the sediment itself is acidic or basic or has absorbed acidic or basic material from the sample. Failure to obtain a representative sample negates a meaningful value obtained (*see* Note).

NOTE — As used oils can change appreciably in storage, samples should be tested as soon as possible after removing/removal from the lubricating system; and the dates of sampling and testing shall be noted.

10.2 Heat the sample (*see* Note) of used oil to (60 ± 5) °C in the original container and agitate the sample until all of the sediment is homogeneously suspended in the oil. If the original container is a can, or if it is glass and more than three-fourths full, transfer the entire sample to a clear-glass bottle having a capacity at least one-third greater than the volume of the sample. Transfer all traces of sediment from the original container to the bottle by vigorous agitation of portions of the sample in the original container.

NOTE — When samples are visibly free of sediment, the heating procedures described can be omitted.

10.3 After complete suspension of all sediment, strain the sample or a convenient aliquot through a 150 µm (100 mesh) screen for the removal of large contaminated particles.

11 PROCEDURE FOR BASE NUMBER AND STRONG BASE NUMBER

11.1 Calculate the quantity of sample required for its expected base number as follows:

$$A = 7/E$$

where

A = approximate mass of sample, g; and
E = expected base number.

11.1.1 Take a maximum of 5 g and a minimum of 0.1 g for analysis. The precision of weighing is as follows:

Size of sample, g	Precision of weighing, g
0.1 to 1	0.002
Above 1 to 5	0.005

11.2 Into a 250 ml titration beaker or a suitable titration vessel, introduce a weighed quantity of sample as prescribed in **11.1.1** and add 125 ml of titration solvent *(see* Note). Prepare the electrodes as directed in **8.2**. Place the beaker or titration vessel on the titration stand and adjust its position so that the electrodes are about half immersed. Start the stirrer, and stir throughout the determination at a rate sufficient to produce vigorous agitation without spattering and without stirring air into the solution.

NOTE — Some automatic titrators do not accept a beaker size that contains 125 ml of titration solvent. In such cases, a lesser amount of solvent in the range from 75 ml to 100 ml is acceptable.

11.3 Select and fill a suitable burette with the 0.1 N alcoholic HCl solution and place the burette in position on the titration assembly, taking care that the tip is immersed about 25 mm in the liquid in titration vessel. Record the initial burette and meter (cell potential) readings.

11.4 Titration

The reaction of the hydrochloric acid with the basic components is very slow with most titrations for base number. As a result, these titrations are not at equilibrium. Because of this, the titration conditions are tightly specified and must be strictly adhered to in order to achieve the precision as state (*see* Note).

NOTE — *See* Annex B for techniques for reducing the titration time of a sample.

11.4.1 Whether the titration is carried out manually or automatically, the following procedure of fixed increment, fixed time addition of titrant must be followed. Add 0.1 N HCI in increments of 0.100 ml throughout the titration with a 90 s pause between each incremental addition. Take millivolt readings at the end of each 90 s interval. Continue as above until a potential is reached which is 100 mV past the meter reading corresponding to that found for the standard acidic buffer solution (acidic buffer potential). If the volume of titrant required to reach this potential (100 mV past the acidic buffer potential) is greater than 4.0 ml, reduce the sample size by one-half and repeat the titration.

11.4.1.1 The meter readings of potential difference are plotted manually or automatically against the respective volumes of titrant, and the end point taken as described in **12.1**.

11.4.2 On completion of the titration, remove the titration vessel and rinse the electrodes and burette tip with the titration solvent, then with water, then again with titration solvent. (Soak electrodes in distilled water for at least 5 min before using for another titration). (Store the glass electrode in deionized or distilled water and the reference electrode with a saturated solution of LiCl in isopropyl alcohol when not in use) (*see* **8.1**).

11.4.3 Blanks

For each set of samples, make a blank titration of the same volume of titration solvent used for the sample. For the base number blank, add 0.1 N alcoholic HCl solution in 0.05 ml increments, waiting 90 s between each addition, until a potential which is 100 mV past the buffer potential (*see* **11.4**) is reached. For the strong base number blank, add titrant under the same conditions until the potential corresponding to the basic buffer solution is reached.

12 CALCULATION

12.1 If an inflection (*see* Note) occurs in the potential region between the acidic buffer potential (*see* **11.4**) and a point 100 mV past this potential, mark this inflection as the end point. If no inflection occurs in the above mentioned potential region, mark as the end point the point on the curve that corresponds to the acidic buffer potential. *See* Fig. 2 for examples of end points.

NOTE — An inflection point is generally recognizable by inspection whenever at least five successive cell potential changes, caused by the addition of the corresponding five, increments of titrant, exhibit a maximum as illustrated by an example in the following table:

Titrant, ml	*mV*
1.8	8.3
1.9	10.7
2.0	11.3
2.1	10.0
2.2	7.9

The difference at the maximum shall be at least 5 mV, and the difference in between the maximum and both the first and last shall be at least 2 mV.

12.2 Calculate the base number and strong base number as follows:

$$\text{Base number, mg KOH/g} = [(A–B)\times M \times 56.1]/W$$

$$\text{Strong Base number, mg KOH/g} = [(CM + Dm) \times 56.1]/W$$

where

A = alcoholic HCl solution, ml, used to titrate the sample to the end point (aqueous acidic *p*H 3 buffer or inflection;
B = alcoholic HCl solution, ml, used to titrate the solvent blank to the same potential at which the sample end point occurs;
M = molarity of the alcoholic HC1solution;
W = mass of sample, g;
C = alcoholic HCl solution, ml, used to titrate the sample to an end point that occurs at a meter reading corresponding to the aqueous basic *p*H 10 buffer;
D = alcoholic KOH solution, ml, used to titrate the solvent blank to the potential corresponding to C; and
m = molarity of the alcoholic KOH solution.

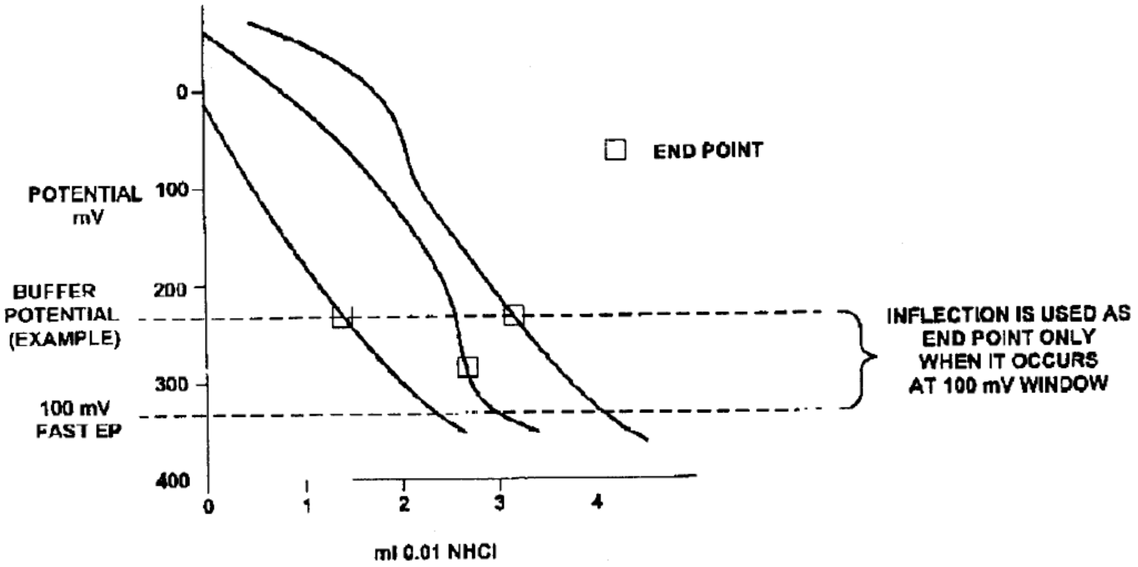

FIG. 2 EXAMPLE OF TITRATION CURVES TO ILLUSTRATE SELECTION OF END POINTS

13 REPORT

Report the results as base number and strong base number.

14 PRECISION AND BIAS

14.1 Precision

The precision of this test method as determined by statistical examination of results on nine samples of new and used oils run in duplicate by twelve different laboratories is given below.

14.1.1 *Base Number*

14.1.1.1 *Repeatability*

The difference between two test results, obtained by the same operator with the same apparatus under constant operating conditions on identical test material, would, in the long run, in the normal and correct operation of the test method, exceed the following value only in one case in twenty:

10.4 percent of the mean of the two test results.

14.1.1.2 *Reproducibility*

The difference between two single and independent results obtained by different operators working in different laboratories on identical test material, would, in the long run, in the normal and correct operation of the test method, exceed the following value only in one case in twenty (*see* Note):

21.1 percent of the mean of the two test results.

NOTE — The range of base number values for which the precision values were established was 0.5 to 70.

14.1.2 *Strong Base Number*

Precision data have not been developed for strong base number because of its rare occurrence.

14.2 Bias

A statement of bias is not applicable since a standard reference material for this property is not available.

ANNEX A
(*Clauses* 6.1 *and* 6.4)
APPARATUS

A-1 APPARATUS FOR MANUAL TITRATION

It shall consist of the following:

A-1.1 Meter

A voltmeter or potentiometer that will operate with an accuracy of ± 0.005 V and a sensitivity of ± 0.002 V, over a range of at least ± 0.5 V, when the meter is used with the electrodes specified in **A-1.2** and **A-1.3**, and when the resistance between the electrodes falls within the range from 0.2 MΩ to 20 MΩ. The meter shall be protected from stray electrostatic fields so that no permanent change in the meter readings over the entire operating range is produced by touching with a grounded lead (*see* Note), any part of the exposed surface of the glass electrode, the glass electrode leads, the titration stands, or the meter. A desirable apparatus may consist of a continuous-reading electronic voltmeter with specified range, accuracy, and sensitivity, that is designed to operate on an input of less than 5×10^{-12}A (when an electrode system having

1000 MΩ) resistance is connected across the meter terminals, and that is provided with a satisfactory terminal to connect the shielded connection wire from the glass electrode to the meter without interferences from the presence of external electrostatic field.

> NOTE — Grounded or connected to the ground means connected through a resistance of not more than 100 to a standard ground potential such as that of a water-service pipe.

A-1.2 Glass Electrode

A pencil-type glass electrode, C (*see* Fig. 1), 125 mm to 180 mm in length and 8 mm to 14 mm in diameter. The body of the electrode shall be made of a chemically resistant glass tube with a wall thickness of 1 mm to 3 mm. The end dipping into the solution shall be closed with a hemisphere of corning 015 glass or equivalent sealed on to the electrode tube, and the radius of this hemisphere shall be about 7 mm. The thickness of the glass in the hemisphere shall be great enough so that the resistance of the hemisphere is 100 MΩ to 1000 MΩ at 25 °C. The electrode shall contain a reproducible, permanently sealed liquid cell for making electrical connection with the inner surface of the hemisphere. The entire electrical connection from the sealed contact cell to the meter terminal shall be surrounded by an electrical shield that will prevent electrostatic interferences when the shield is grounded. The shield shall be insulated from the electrical connection by insulating material of the highest quality, such as rubber and glass, so that the resistance between the shield and the entire length of the electrical connection is greater than 50 000 M.

A-1.3 Calomel Electrode

A pencil-type calomel (reference electrode) B, (*see* Fig.1), 125 mm to 180 mm in length and 8 mm to 14 mm in diameter. This electrode shall be made of glass and shall be provided with an external, removable glass sleeve on the sealed end that is dipped into the titration solution. The glass sleeve shall be 8 mm to 25 mm in length, shall be slightly tapered, and shall be ground to fit the electrode so that the sealed end of the electrode protrudes 2 mm to 20 mm, beyond the sleeve. The ground surface shall be continuous and free of smooth spots. At a point midway between the extremities of the ground surface, the electrode tube shall be pierced by a hole or holes 1 mm in diameter. The electrode shall contain necessary mercury, calomel and electrical connection to mercury (calomel) or silver/silver chloride (Ag/AgCl), all arranged in a permanent manner. The electrode shall be filled almost to capacity with saturated LiCl in isopropyl alcohol electrolyte and shall be equipped with a stoppered port through which the electrolyte may be replenished. When suspended in the air and with the sleeve in place, the electrode shall not leak electrolyte at a rate greater than one drop in 10 min.

A-1.4 Stirrer

A variable-speed mechanical stirrer of any suitable type, equipped with a glass, propeller-type-stirring paddle D, (*see* Fig. 1). A propeller with blades 6 mm in radius and set at a pitch of 30 to 45° is satisfactory. A magnetic stirrer is also satisfactory. If electrical stirring apparatus is used, it must be grounded so that connecting or disconnecting the power to the motor will not produce a permanent change in meter reading during the courses of titration.

A-1.5 Burette

A 5 ml burette, E *(see* Fig. 1) graduated in 0.01 ml divisions and calibrated with an accuracy of ± 0.005 ml. The burette shall have a glass stopcock and shall have a tip that extends 100 mm to 130 mm beyond the stopcock.

A-1.6 Titration Beaker

A 250 ml beaker made of borosilicate glass, or other suitable titration beaker (*see* Fig. 1).

A-1.7 Titration Stand

A suitable stand to support the electrodes, stirrer, and burette in the position shown in Fig. 1. An arrangement that allows the removal of the beaker without disturbing the electrodes, burette, and stirrer is desirable.

A-2 APPARATUS FOR AUTOMATIC TITRATION

It shall be generally in accordance with **A-1** and provide the following technical performance characteristics of features.

A-2.1 The addition of titrant must be automatically controlled to dispense (0.100 ± 0.001) ml increments of titrant with a waiting period of 90 s between increments.

A-2.2 Interchangeable precision motor-driven burettes with volume dispensing accuracy of ± 0.001 ml.

A-2.3 A record of the complete course of a titration by continuously printing out the potential or change in potential with the addition of each increment of titrant, versus volume of titrant added.

ANNEX B

(*Clause* 11.4)

REDUCING TITRATION TIME

B-1 A long, equilibration period of 90 s increment is selected for the base number titration because the titration reaction and electrode equilibration are generally slow. This, of course, can lead to long titration time/sample, with a maximum time of 1 h based on the maximum volume of titrant of 4 ml and a rate of titrant addition of 0.1 ml/90 s. It is possible to substantially reduce the titration time by pre-dosing with rapid addition of titrant until a potential within 25 mV of the buffer potential is reached, then allowing 90 s for equilibration and completing the titration under normal conditions. This procedure is not expected to have an adverse effect on the precision of this test method, however, the precision under these conditions has not been determined.

B-2 There are many cases where the optimum in precision in the base number is not required, and in these cases the titration time can be shortened by taking a smaller sample. For example, for a base number of 2 and using a sample size calculated from the equation in **11.1,** a total titration time of 24 min would be required. By taking only one half of the prescribed sample size, the titration time would be reduced to 12 min. The effect of halving the sample size on the precision of this test method has not been determined but it is expected to be small.