

**BUREAU OF INDIAN STANDARDS**

DRAFT FOR COMMENTS ONLY

*(Not to be reproduced without permission of BIS  
or used as an Indian Standard)*

भारतीय मानक मसौदा

पेट्रोलियम और इसके उत्पाद — परीक्षण विधियाँ  
भाग 113 विमान टरबाइन ईंधन में कुल अम्लता का निर्धारण

(IS 1448 भाग 113 का पहला पुनरीक्षण)

*Draft Indian Standards*

**PETROLEUM AND ITS PRODUCTS — TEST METHODS**  
**PART: 113 DETERMINATIONS OF TOTAL ACIDITY IN AVIATION**  
**TURBINE FUEL**

*(First Revision of IS 1448 Part 113)*

(ICS No. 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  
19 January 2025

---

**FOREWORD**

*(Formal clause will be added later)*

This standard was published in 1983 and assistance was taken from ASTM D 3242-79 and IP 354/81. The first revision has been taken up to keep pace with the latest technological developments and international practices. In this revision following major changes have been made:

1. The procedures for the standardization of Potassium Hydroxide solution and Quality Control checks have been incorporated.
2. The procedures for the preparation of the electrode system and various solutions have been incorporated.

This standard is formulated based on experience of petroleum industry laboratories and considerable assistance has been derived from ASTM D 3242-2023 'Standard Test Method for Acidity in Aviation Turbine Fuel'.

In reporting the result of the test 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 113) describes the determination of the total acidity in aviation turbine fuel in the range from 0.000 mg KOH/g to 0.100 mg KOH/g.

## **2. OUTLINE OF THE METHOD**

**2.1** The sample is dissolved in a mixture of toluene and isopropyl alcohol containing a small amount of water. The resulting single-phase solution is blanketed by a stream of nitrogen bubbling through it and is titrated with standard alcoholic potassium hydroxide to the end point indicated by the colour change (orange in acid and green in base) of the added *p*-Naphtholbenzein solution.

## **3. APPARATUS**

### **3.1 Burette**

A 25 ml burette graduated in 0.1 ml subdivisions, or a 10 ml burette graduated in 0.05 ml subdivisions.

## **4. REAGENTS**

### **4.1 *p*-Naphtholbenzein Indicator Solution**

The *p*-Naphtholbenzein shall meet the specifications given in Annex A. Prepare a solution containing 10 g of *p*-Naphtholbenzein per litre of titration solvent (*see* 4.5).

**4.2 Nitrogen** — Dry type, carbon dioxide-free.

CAUTION — Compressed gas under high pressure. Gas reduces oxygen available for breathing.

### **4.3 Standard Alcoholic Potassium Hydroxide Solution (0.01 N)**

Add 0.6 g of solid KOH to approximately 1 litre of anhydrous isopropyl alcohol (containing less than 0.9 percent water) in a 2 L Erlenmeyer flask. Boil the mixture gently for 10 min to 15 min, stirring to prevent the solids from forming a cake on the bottom. Add at least, 0.2 g of barium hydroxide Ba (OH)<sub>2</sub> and again boil gently for 5 min to 10 min. Cool to room temperature, allow to stand for several hours, and filter the supernatant liquid through a fine sintered-glass or porcelain filtering funnel: avoid unnecessary exposure to carbon dioxide (CO<sub>2</sub>) during filtration. Store the solution in a chemically resistant dispensing bottle out of contact with cork, rubber, or saponifiable stop-cock lubricant and protected by a guard tube containing soda lime or soda-asbestos. Standardize frequently enough to detect changes of 0.000 2 N, preferably against pure potassium acid phthalate in about 100 ml of CO<sub>2</sub> - free water, using phenolphthalein to detect the end point.

NOTE — Because of the relatively large coefficient of cubic expansion of organic liquids, such as isopropyl alcohol, the standard alcoholic solutions should be standardized at temperatures close to those employed in the titrations of samples.

### **4.3.1 Standardization of Potassium Hydroxide Solution**

The Alcoholic Potassium Hydroxide Solution is to be Standardized frequently enough to detect changes of 0.0002 N. Weigh, to the nearest 0.1 mg, approximately 0.02 g of potassium acid phthalate, which has been dried for at least 1 h at  $(110 \pm 1)$  °C and dissolve in  $40 \text{ ml} \pm 1 \text{ ml}$  of water, free of CO<sub>2</sub>. Titrate with the Potassium hydroxide alcoholic solution to either of the following end points: (1) when the titration is electrometric, titrate to a well-defined inflection point at the voltage that corresponds to the voltage of the basic buffer solution; (2) when the titration is colorimetric, add 6 drops of phenolphthalein indicator solution and titrate to the appearance of a permanent pink color. Perform the blank titration on the water used to dissolve the potassium acid phthalate. Calculate the normality using the equation:

$$\text{Normality} = \frac{W_p}{204.23} \times \frac{1000}{V - V_b}$$

where:

$W_p$  = weight of the potassium acid phthalate, g,

204.23 = molecular weight of the potassium acid phthalate,

$V$  = volume of titrant used to titrate the salt to the specific end point, ml, and

$V_b$  = volume of titrant used to titrate the blank, ml.

### **4.4 Phenolphthalein Indicator Solution**

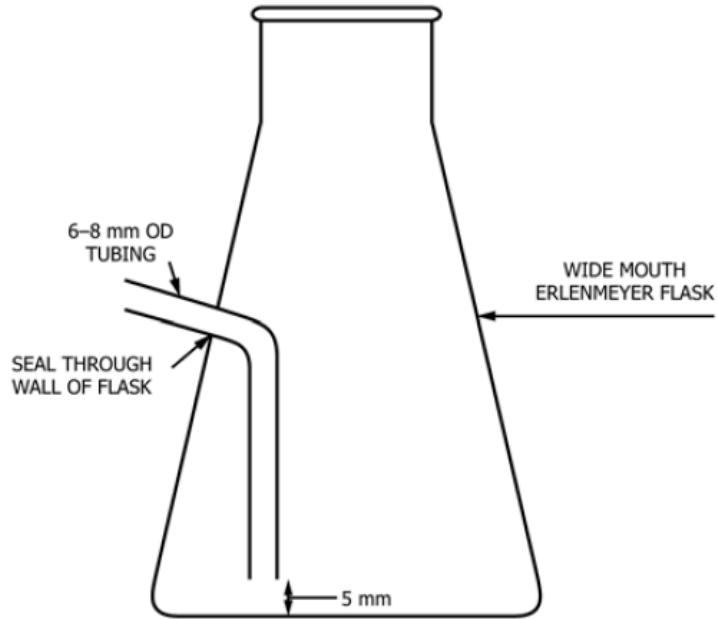
Dissolve  $(0.1 \pm 0.01)$  g of pure solid phenolphthalein in 50 ml of water, free of CO<sub>2</sub>, and 50 ml of ethanol.

### **4.5 Titration Solvent**

Add 500 ml of toluene and 5 ml of water to 495 ml of anhydrous isopropyl alcohol.

## **5. PROCEDURE**

**5.1** Introduce  $(100 \pm 5)$  g of the sample weighed to the nearest 0.5 g, into a 500 ml wide-mouth Erlenmeyer flask. (One type of suitable modified flask is shown in Fig. 1). Add 100 ml of the titration solvent and 0.1 ml of the indicator solution. Introduce nitrogen (*see 5.1.1*) through a 6 mm to 8 mm outside diameter glass tube to a point within 5 mm of the flask bottom at a rate of 600 ml/min to 800 ml/min. Bubble the solution for 3 min with occasional swirling.



**FIG 1 TITRATION FLASK**

**5.1.1 Caution** — The vapour from this treatment contains toluene and should be removed with adequate ventilation.

**5.2** Continue the nitrogen addition and titrate immediately at a temperature below 30 °C. Add 0.01 N KOH solution in increments and swirl to disperse until a green end point is reached that persists for 15 s.

### **5.3 Blank**

Make a blank titration on 100 ml of titration solvent and 0.1 ml of the indicator solution, introducing the nitrogen in the same manner and titrating to the same end point as above.

### **6.0 Quality Control Checks**

6.1 Confirm the performance of the equipment or the procedure each day it is in use, by analyzing a quality control (QC) sample. It is advisable to analyze additional QC samples as appropriate, such as at the end of a batch of samples or after a fixed number of samples to ensure the quality of the results. Analysis of result(s) from these QC samples can be carried out using control chart techniques. When the QC sample result causes the laboratory to be in an out-of-control situation, such as exceeding the laboratory's control limits, instrument recalibration may be required. An ample supply of QC sample material shall be available for the intended period of use, and shall be homogeneous and stable under the anticipated storage conditions. If possible, the QC sample shall be representative of samples typically analyzed and the average value and control limits of the QC sample shall be determined prior to monitoring the measurement process. The QC sample precision shall be checked against the method precision to ensure data quality.

NOTE — Because the acid number can vary while the QC sample is in storage, when an out-of-control situation arises, the stability of the QC sample can be a source of the error.

## 7. CALCULATION

7.1 Calculate the total acid number as follows:

Total acid number, mg of KOH/g

$$\frac{(A - B)N \times 56.1}{M}$$

Where

*A* = millilitres of KOH solution required for titration of the sample (5.2),

*B* = millilitres of KOH solution required for titration of the blank (5.3),

*N* = normality of the KOH solution, and

*M* = mass in g of the sample used.

## 7. REPORTING

7.1 Report the result to the nearest 0.001 mg KOH/g as total acidity.

## 8. PRECISION

8.1 Results of duplicate tests shall not differ by more than the values shown in Table 1.

**Table 1 Precision**

(Clause 8.1)

| <b>AVERAGE ACID NUMBER</b> | <b>REPEATABILITY</b> | <b>REPRODUCIBILITY</b> |
|----------------------------|----------------------|------------------------|
| 0.001                      | 0.0004               | 0.0013                 |
| 0.002                      | 0.0006               | 0.0018                 |
| 0.005                      | 0.0009               | 0.0029                 |
| 0.010                      | 0.0013               | 0.0041                 |
| 0.020                      | 0.0019               | 0.0057                 |
| 0.050                      | 0.0030               | 0.0091                 |
| 0.100                      | 0.0042               | 0.0128                 |

All values are in acid number units

These precision data were derived as follows:

$$\text{Repeatability} = 0.0132 \sqrt{a}$$

$$\text{Reproducibility} = 0.0406 \sqrt{a}, \text{ where 'a' is acid number.}$$

**ANNEX A**

(Clause 4.1)

**SPECIFICATION FOR *p*-NAPHTHOLBENZEIN**

**A-1. CONFORMITY REQUIREMENT**

**A-1.1 Appearance** — Red amorphous powder.

**A-1.2 Chlorides** — Less than 0.5 percent.

**A-1.3 Solubility** — Ten grams shall dissolve completely in 1 litre of titration solvent.

**A-1.4 Minimum Absorbance**

Exactly 0.1000 g of the sample is dissolved in 250 ml of methanol. Five millilitres of this solution is made up to 100 ml with *pH* 12 buffer. This final dilution should have a minimum absorbance of 1.20 when read at the 650-nm peak using spectrophotometer, 1 cm cells, and water as the blank.

**A-1.5 *pH* Range**

**A-1.5.1** Indicator turns to the first clear green at a relative *pH* of  $11 \pm 0.5$  when tested by the method for *pH* range of *p*-naphtholbenzein indicator as described in Annex B.

**A-1.5.2** Requires not more than 0.5 ml of 0.01 N KOH solution above that for the blank to bring the indicator solution to the first clear green.

**A-1.5.3** Requires not more than 1.0 ml of 0.01 N KOH solution above that for the blank to bring the indicator solution to a blue colour.

**A-1.5.4** Initial *pH* of the indicator solution is at least as high as that of the blank.

**A-1.5.5** Buffer is made by mixing 50 ml of 0.05 M dibasic sodium phosphate with 26.9 ml 0.1 m sodium hydroxide.

**ANNEX B**  
(Clause A-1.5.1)

**DETERMINATION OF  $pH_r$  RANGE OF  $p$ -NAPHTHOLBENZEM INDICATOR**

**B-1 OUTLINE OF THE METHOD**

**B-1.1** A prescribed amount of indicator is titrated Electrometrically through the various colour changes with alcoholic potassium hydroxide and results plotted against meter readings converted to  $pH_r$  units.

**B-2 TERMINOLOGY**

**B-2.1  $pH_r$**  — An arbitrary term which expresses the relative hydrogen ion activity in the toluene - Isopropyl alcohol medium in a manner similar to that in which the term  $pH$  expresses the actual hydrogen ion activity, in aqueous solutions. For the purpose of this method, the  $pH_r$  acidity scale is defined by two standard buffer solutions which have been designated  $pH_r$  4 and  $pH_r$  11. The exact relation between  $pH_r$  and the true  $pH$  of a toluene-isopropyl alcohol solution is not known and cannot be readily determined.

**B-3 APPARATUS**

**B-3.1 Meter, Glass Electrode, Calomel Electrode, Stirrer, Beaker and Stand** — as specified in IS 1448 (Part 1/Sec 1)

**B-4 REAGENTS**

**B-4.1 Buffer Stock Solution A**

Accurately weigh  $(24.2 \pm 0.1)$  g of 2,4,6-trimethyl pyridine ( $\gamma$  -collidine), and transfer to a 1 L volumetric flask containing 100 ml of iso propyl alcohol. Using a 1 L graduated cylinder, add to the flask, while continuously stirring its contents,  $150/N_{HCl} \pm 5$  ml of 0.2 mol/l alcoholic HCl solution ( $N_{HCl}$  being the exact molarity concentration of the HCl solution found by standardization). Dilute to the 1000 ml mark

**B-4.2 Acidic Buffer Solution ( $pH_r = 4.0$ )**

Prepare a stock solution by adding 10 ml of buffer stock solution A to 100 ml of titration solvent. Use the diluted solution within 1 h.

**B-4.3 Buffer Stock Solution B**

Accurately weigh  $(27.8 \text{ g} \pm 0.1)$  g of m-nitrophenol and transfer to a 1 L volumetric flask containing 100 ml of iso propyl alcohol. Using a 250 ml graduated cylinder, add to the flask while continuously stirring its contents,  $50/N_{KOH} \pm 1$  ml of 0.2 mol/l alcoholic KOH solution ( $N_{KOH}$  being

the exact molarity concentration of the KOH solution found by standardization). Dilute to the 1000 ml mark with iso propyl alcohol, and mix thoroughly. Use within two weeks.

#### **B-4.4 Alkaline Buffer Solution ( $pH_r = 11.0$ )**

Prepare a stock solution by adding 10 ml of buffer stock solution B to 100 ml of titration solvent. Use the diluted solution within 1 h.

#### **B-4.5 Standard Alcoholic Hydrochloric Acid Solution (0.2 N)**

Mix 18 ml of Hydrochloric Acid (HCl Specific Gravity 1.19) with 1 L of anhydrous Iso propyl alcohol. Standardize frequently enough to detect normality changes of 0.0005N by potentiometric titration of 8 ml of 0.1N alcoholic KOH solution diluted to 125 ml with CO<sub>2</sub> free water.

**B-4.6 *p*-Naphtholbenzein Indicator Solution** — Prepare as described in 4.1.

**B-4.7 Potassium Chloride Electrolyte** — Prepare a saturated solution of potassium chloride (KCL) in water.

#### **B-4.8 Standard Alcoholic Potassium Hydroxide Solution (0.2 N)**

Add 12 g to 13 g of potassium hydroxide (KOH) to approximately 1 L of anhydrous isopropyl alcohol. Boil gently for 10 min to effect solution. Allow the solution to stand for two 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<sub>2</sub>) by means of a guard tube containing soda lime or soda non-fibrous silicate absorbents and such that it does not come into contact with cork, rubber, or saponifiable stopcock grease. Standardize frequently enough to detect concentration changes of 0.0005 by potentiometric titration of weighed quantities of potassium acid phthalate dissolved in CO<sub>2</sub>-free water.

#### **B-4.9 Titration Solvent**

Add 500 ml of toluene and 5 ml of water to 495 ml of anhydrous isopropyl alcohol. The titration solvent made up in large quantities.

### **B-5. PREPARATION OF ELECTRODE SYSTEM**

#### **B-5.1 Maintenance of Electrodes**

Clean the glass electrode at frequent intervals (not less than once every week during continual use) by immersing in cold chromium-free cleaning solution or in other equipment cleaning solutions. Drain the electrode at least once each week, and refill with fresh KCl electrolyte as far as the filling hole. Ascertain that crystallized KCl is present. Maintain the electrolyte level in the reference electrode above that of the liquid in the titration beaker or vessel at all times. When not in use, immerse the lower halves of the electrodes in water. 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<sup>8</sup> thoroughly, keeping the ground glass joint free of foreign materials, and regular testing of the electrodes are very important in obtaining repeatable potentials, since contamination may 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 may be quite serious when end points are chosen at arbitrarily fixed cell potentials.

### **B-5.2 Preparation of Electrodes**

Before and after using, wipe the glass electrode thoroughly with a clean cloth, or a soft absorbent tissue, and rinse with water. Wipe the reference electrode with a cloth or tissue, carefully remove the ground glass sleeve, and thoroughly wipe both ground surfaces. Replace the sleeve loosely, and allow a few drops of electrolyte to drain through to flush the ground-glass joint. (*see Note*). Wet the ground surfaces thoroughly with electrolyte, set the sleeve firmly in place, and rinse the electrode with water. Prior to each titration, soak the prepared electrodes in water for at least 5 min immediately before use, and touch the tips of the electrodes with a dry cloth or tissue to remove the excess of water.

### **B-5.3 Testing of Electrodes**

Test the meter-electrode combination when 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 mol/l alcoholic KOH solution. For the meter-electrode combination to be suitable for use, the potential between the electrodes should change by more than 480 mV from the potential between the same electrodes when dipped in the nonaqueous acidic buffer solution (*see Note A2.2*).

NOTE — Considerably more sensitive electrodes are now available that will show a potential change of at least 590 mV under these conditions, and their use is recommended.

## **B-6. STANDARDIZATION OF APPARATUS**

**B-6.1** Prior to each test or series of tests, set the meter to read on the *pH* scale, insert the electrodes into a beaker containing, the acidic nonaqueous buffer solution at a temperature of  $(25 \pm 2)$  °C stir the solution vigorously. When the *pH* meter reading becomes constant adjust the asymmetry potential dial of the instrument so that the meter reads 4.0.

**B-6.2** Remove the acidic buffer, clean the electrodes, and immerse them in water for several minutes. Dry the electrodes and insert them in a beaker containing alkaline nonaqueous buffer solution at  $(25 \pm 2)$  °C. When the *pH* meter reading has become steady, record the exact value. If the reading is within 0.2 units of 11.0, the initial acidity, *pH<sub>r</sub>*, of unknown solutions may be read directly from the dial of the meter. If the reading is not within 0.2 units of 11.0 prepare a correction graph as shown in Fig. 2. Use this graph to convert *pH* meter readings to initial acidity, *pH<sub>r</sub>*.

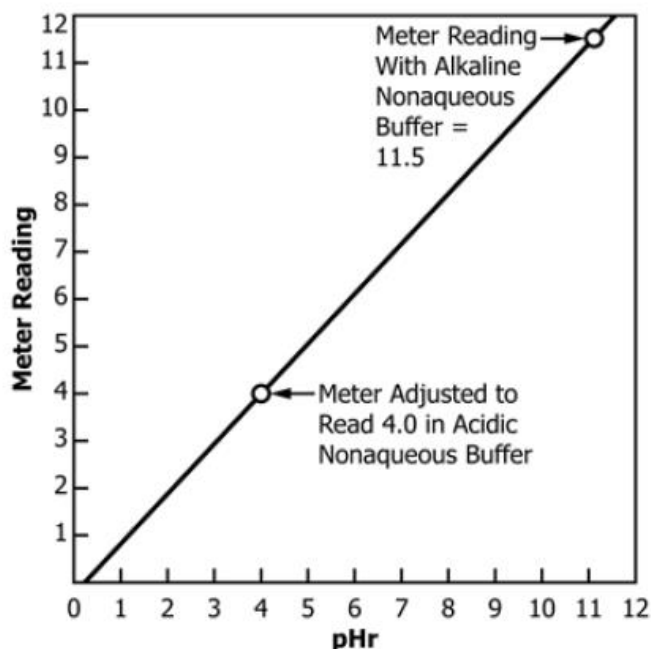


FIG 2 CALIBRATION CURVE FOR CONVERSION OF  $pH$  METER READING TO  $pHr$

## B-7. PROCEDURE

**B-7.1** Titrate 100 ml of titration solvent with 0.01 N KOH solution until the meter indicates a  $pHr$  between 13 and 14.

**B-7.2** Add 0.5 ml of indicator solution to a fresh portion of titration solvent and after cleaning the electrodes titrate with 0.01 N KOH solution until the meter indicates a  $pHr$  between 13 and 14.

**B-7.3** During the titration, plot the volume of titrant against the  $pHr$  or meter reading and note on the curve the various colour changes at the corresponding  $pHr$  values.

NOTE — The following colour changes, in order, are intended as a guide: Amber to olive green, Olive green to clear green, Clear green to bluish green, Bluish green to blue

**B-7.4** Plot the blank titration on the same paper used for the indicator.

## B-8. CALCULATION

**B-8.1** Subtract the volume of titrant used in the blank titration from that used for the indicator solution titration at the same  $pHr$  corresponding to the definite colour changes between 10 to 12  $pHr$ .