



Designation: D3242 – 11 (Reapproved 2017)



Designation: 354/98

## Standard Test Method for Acidity in Aviation Turbine Fuel<sup>1</sup>

This standard is issued under the fixed designation D3242; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

*This standard has been approved for use by agencies of the U.S. Department of Defense.*

### 1. Scope

1.1 This test method covers the determination of the acidity in aviation turbine fuel in the range from 0.000 mg/g to 0.100 mg/g KOH.

1.2 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.3 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

1.4 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

### 2. Referenced Documents

2.1 *ASTM Standards:*<sup>2</sup>

D664 Test Method for Acid Number of Petroleum Products by Potentiometric Titration

D1193 Specification for Reagent Water

### 3. Terminology

3.1 *Definitions:*

3.1.1 *acid number, n*—the quantity of a specified base, expressed in milligrams of potassium hydroxide per gram of

<sup>1</sup> This test method is under the jurisdiction of ASTM Committee D02 on Petroleum Products, Liquid Fuels, and Lubricants and is the direct responsibility of Subcommittee D02.06 on Analysis of Liquid Fuels and Lubricants.

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This test method has been approved by the sponsoring committees and accepted by the cooperating societies in accordance with established procedures.

<sup>2</sup> For referenced ASTM standards, visit the ASTM website, [www.astm.org](http://www.astm.org), or contact ASTM Customer Service at [service@astm.org](mailto:service@astm.org). For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

sample, required to titrate a sample in a specified solvent to a specified endpoint using a specified detection system.

3.1.1.1 *Discussion*—*in this test method*, the solvent is a toluene-water-isopropanol mixture and the end point is determined when a green/green brown color is obtained using the specified *p*-naphtholbenzein indicator solution.

### 4. Summary of Test Method

4.1 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 color change (orange in acid and green in base) of the added *p*-naphtholbenzein solution.

### 5. Significance and Use

5.1 Some acids can be present in aviation turbine fuels due either to the acid treatment during the refining process or to naturally occurring organic acids. Significant acid contamination is not likely to be present because of the many check tests made during the various stages of refining. However, trace amounts of acid can be present and are undesirable because of the consequent tendencies of the fuel to corrode metals that it contacts or to impair the water separation characteristics of the aviation turbine fuel.

5.2 This test method is designed to measure the levels of acidity that can be present in aviation turbine fuel and is not suitable for determining significant acid contamination.

### 6. Apparatus

6.1 *Buret*—A 25 mL buret graduated in 0.1 mL subdivisions, or a 10 mL buret graduated in 0.05 mL subdivisions.

NOTE 1—An automated buret capable of delivering titrant amounts in 0.05 mL or smaller increments can be used, but the stated precision data were obtained using manual burets only.

### 7. Reagents and Materials

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.<sup>3</sup> 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.

NOTE 2—Commercially available reagents may be used in place of laboratory preparations when they are certified in accordance with 7.1.

7.2 *Purity of Water*—References to water shall be understood to mean distilled water as defined by Type III water of Specification D1193.

7.3 *p-Naphtholbenzein*<sup>4,5</sup> Indicator Solution—The *p*-naphtholbenzein must meet the specifications given in Annex A1. Prepare a solution of *p*-naphtholbenzein in titration solvent equal to 10 g/L ± 0.01 g/L.

7.4 *Nitrogen*, dry-type, carbon dioxide-free. (**Warning**—Compressed gas under high pressure. Gas reduces oxygen available for breathing.)

7.5 *Potassium Hydroxide Solution, Standard Alcoholic* (0.01 N)—Add 0.6 g of solid KOH (**Warning**—Highly corrosive to all body tissue both in solid form and in solution.) to approximately 1 L of anhydrous isopropyl alcohol (**Warning**—Flammable. Vapor harmful. Keep away from heat, sparks, and open flame.) (containing less than 0.9 % 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>) (**Warning**—Poisonous if ingested. Strongly alkaline, causes severe irritation producing dermatitis.) 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 stopcock lubricant and protected by a guard tube containing soda lime.

NOTE 3—Because of the relative 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 titration of samples.

7.5.1 *Standardization of Potassium Hydroxide Solution*—Standardize frequently enough to detect changes of 0.0002N. One way to accomplish this is as follows. 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 °C ± 1 °C and dissolve in 40 mL ± 1 mL of water, free of CO<sub>2</sub>. Titrate with

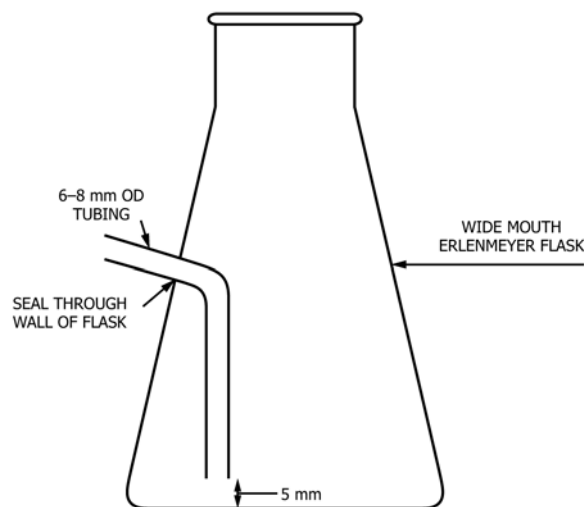


FIG. 1 Titration Flask

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:

$$\text{Normality} = \frac{W_p}{204.23} \times \frac{1000}{V - V_b} \quad (1)$$

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.

7.5.2 *Phenolphthalein Indicator Solution*—Dissolve 0.1 g ± 0.01 g of pure solid phenolphthalein in 50 mL of water, free of CO<sub>2</sub>, and 50 mL of ethanol.

7.6 *Titration Solvent*—Add 500 mL of toluene (**Warning**—Flammable. Vapor harmful. Keep away from heat, sparks, and open flame.) and 5 mL of water to 495 mL of anhydrous isopropyl alcohol.

## 8. Procedure

8.1 Introduce 100 g ± 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 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 ± 30 s with occasional mixing.

8.1.1 The vapor from this treatment contains toluene and should be removed with adequate ventilation.

<sup>3</sup> *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For Suggestions on the testing of reagents not listed by the American Chemical Society, see *Annual Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

<sup>4</sup> In a 2006 study, only Kodak, Baker (Mallinkrodt), Fluka, and Aldrich were found to meet the specifications in Annex A1. However, Kodak brand is no longer available.

<sup>5</sup> Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1626. Contact ASTM Customer Service at service@astm.org.

8.2 Continue the nitrogen addition and titrate without delay at a temperature below 30 °C. Add 0.01 N KOH solution in increments and swirl to disperse. When the solution first turns green, reduce the increment size to dropwise (manual buret) or between 0.01 mL and 0.05 mL (automated buret). Continue until a persistent green end point is reached and held for a minimum of 15 s after the addition of the last increment.

NOTE 4—The temperature can be measured by any suitable temperature measuring device.

8.3 *Blank*—Perform a blank titration on 100 mL of the 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.

## 9. Quality Control Checks

9.1 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.<sup>6</sup> 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 ASTM method precision to ensure data quality.

NOTE 5—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.

## 10. Calculations

10.1 Calculate the acid number as follows:

$$\text{Acid number, mg of KOH/g} = [(A - B)N \times 56.1] / W \quad (2)$$

where:

- A = KOH solution required for titration of the sample (8.2), mL,
- B = KOH solution required for titration of the blank (8.3), mL,

<sup>6</sup> MNL 7, *Manual on Presentation of Data Control Chart Analysis*, Section 3: Control Charts for Individuals, 6th ed., ASTM International, W. Conshohocken, 1990.

TABLE 1 Precision<sup>A</sup>

NOTE 1—All values are in acid number units.

| Average Acid Number | Repeatability | Reproducibility |
|---------------------|---------------|-----------------|
| 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          |

<sup>A</sup> These precision data were derived as follows:

$$\begin{aligned} \text{Repeatability} &= 0.0132 \sqrt{a} \\ \text{Reproducibility} &= 0.0406 \sqrt{a} \end{aligned}$$

where: *a* = acid number

*N* = normality of the KOH solution, and

*W* = sample used, g.

## 11. Report

11.1 Report the result to the nearest 0.001 mg KOH/g as Acid Number (Test Method D3242) = (Result).

## 12. Precision and Bias<sup>7</sup>

12.1 *Precision*—The precision of this test method as determined by statistical examination of interlaboratory results is as follows:

12.1.1 *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 values only in one case in twenty (see Table 1).

12.1.2 *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 values only in one case in twenty (see Table 1).

NOTE 6—The precision statements were based on the use of manual burets only. The user is cautioned that the precision statements may or may not be applicable to titrations performed with the use of automated burets, since no interlaboratory study has been conducted to date to statistically evaluate results determined by both techniques.

12.2 *Bias*—The procedure in this test method has no bias because the value of the acid can be defined only in terms of the test method.

## 13. Keywords

13.1 acidity; aviation turbine fuel

<sup>7</sup> Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1010. Contact ASTM Customer Service at service@astm.org.

**ANNEXES**
**(Mandatory Information)**
**A1. SPECIFICATIONS FOR *p*-NAPHTHOLBENZEIN**
**A1.1 Conformity Requirements**

A1.1.1 *Appearance*—Red amorphous powder.

A1.1.2 *Chlorides*—Less than 0.5 %.

A1.1.3 *Solubility*—Ten grams shall dissolve completely in 1 L of titration solvent.

A1.1.4 *Minimum Absorbance*—Exactly 0.1000 g of the sample is dissolve in 250 mL of methanol. (**Warning**—Flammable.) 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 a Beckman DU or alternative type spectrophotometer, 1 cm cells, and water as the blank.

A1.1.5 *pH Range*:

A1.1.5.1 Indicator turns to the first clear green at a relative pH of  $11 \pm 0.5$  when tested by the method for pHr range of *p*-naphtholbenzein indicator as described in **Annex A2**.

A1.1.5.2 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.

A1.1.5.3 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 color.

A1.1.5.4 Initial pHr of the indicator solution is at least as high as that of the blank.

A1.1.5.5 Buffer is made by mixing 50 mL of 0.05 *M* dibasic sodium phosphate with 26.9 mL 0.1 *M* sodium hydroxide.

**A2. TEST METHOD FOR DETERMINING pHr RANGE OF *p*-NAPHTHOLBENZEIN INDICATOR**
**A2.1. Scope**

A2.1.1 This test method is intended for determining the acceptability of *p*-naphtholbenzein indicator for use in Test Method D3242 with regard to color change over a pHr range.

**A2.2. Terminology**

A2.2.1 *Definitions of Terms Specific to This Standard*:

A2.2.1.1 *pHr*—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 test method, the pHr acidity scale is defined by two standard buffer solutions which have been designated pHr 4 and pHr 11. The exact relation between pHr and the true pH of a toluene-isopropyl alcohol solution is not known and cannot be readily determined.

**A2.3. Summary of Test Method**

A2.3.1 A prescribed amount of indicator is titrated electrometrically through the various color changes with alcoholic potassium hydroxide and results plotted against meter readings converted to pHr units.

**A2.4. Apparatus**

A2.4.1 *Meter, Reference and Glass Electrodes or Combination Electrode, Stirrer, Beaker, and Stand*, as specified in Test Method **D664** – IP 177.

**A2.5. Reagents**

A2.5.1 *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.<sup>3</sup> 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.

A2.5.2 *Purity of Water*—References to water shall be understood to mean distilled water.

A2.5.3 *Acidic Buffer Solution (pHr = 4.0)*:

A2.5.3.1 *2,4,6 Trimethyl Pyridine ( $\gamma$  Collidine)*—( $(\text{CH}_3)_3\text{C}_5\text{H}_2\text{N}$ )—(*mol weight 121.18*). (**Warning**—2,4,6-trimethyl pyridine ( $\gamma$  collidine) is hazardous if swallowed, breathed, or spilled on skin or eyes.) (**Warning**—Wear chemical safety goggles, neoprene or rubber gloves and an apron. Use only in a well-ventilated hood, or wear an approved respirator for organic vapor or a supplied-air respirator. Do not take internally.) Conform to the following requirements:

|                              |                     |
|------------------------------|---------------------|
| Boiling range                | 168 °C to 170 °C    |
| Refractive index, $n_D^{20}$ | 1.4982 $\pm$ 0.0005 |
| Color                        | colorless           |

A2.5.3.2 Store the reagent over activated alumina, and keep it in a brown glass bottle.

A2.5.3.3 *Buffer Stock Solution A*—(**Warning**—Flammable.) (**Warning**—2,4,6-trimethyl pyridine ( $\gamma$  collidine) is hazardous if swallowed, breathed, or spilled on skin or eyes.) (**Warning**—Wear chemical safety goggles, neoprene or rubber gloves and an apron. Use only in a well-ventilated hood, or wear an approved respirator for organic vapor or a supplied-air respirator. Do not take internally.) Accurately weigh 24.2 g  $\pm$  0.1 g of 2,4,6-trimethyl pyridine ( $\gamma$  -collidine), and transfer to a 1 L volumetric flask containing 100 mL of propan-2-ol.

Using a 1 L graduated cylinder, add to the flask, while continuously stirring its contents, 150/C  $\pm$  5 mL of 0.2 mol/L alcoholic HCl solution (C being the exact molarity concentration of the HCl solution found by standardization). Dilute to the 1000 mL mark with propan-2-ol, and mix thoroughly. Use within two weeks.

A2.5.3.4 *Buffer, Nonaqueous Acidic*—Add 10 mL of buffer stock Solution A to 100 mL of titration solvent. Use within 1 h.

A2.5.4 *Alkaline Buffer Solution* (pHr = 11.0):

A2.5.4.1 *m*-Nitrophenol (NO<sub>2</sub>C<sub>6</sub>H<sub>4</sub>OH) (mol weight 139.11). (**Warning**—*m*-Nitrophenol can be hazardous if swallowed, breathed, or spilled on skin or eyes. Wear chemical-safety goggles, neoprene or rubber gloves, and an apron. Use only in a well-ventilated hood, or wear an approved respirator for organic vapor or a supplied-air respirator. Do not take internally.) Conform to the following requirements:

|               |                |
|---------------|----------------|
| Melting point | 96 °C to 97 °C |
| Color         | pale yellow    |

A2.5.4.2 Store the reagent in a brown glass bottle.

A2.5.4.3 *Buffer Stock Solution B*—(**Warning**—Flammable.) (**Warning**—*m*-Nitrophenol can be hazardous if swallowed, breathed, or spilled on skin or eyes. Wear chemical-safety goggles, neoprene or rubber gloves, and an apron. Use only in a well-ventilated hood, or wear an approved respirator for organic vapor or a supplied-air respirator. Do not take internally.) Accurately weigh 27.8 g  $\pm$  0.1 g of *m*-nitrophenol and transfer to a 1 L volumetric flask containing 100 mL of propan-2-ol. Using a 250 mL graduated cylinder, add to the flask while continuously stirring its contents, 50/C<sub>2</sub>  $\pm$  1 mL of 0.2 mol/L alcoholic KOH solution (C<sub>2</sub> being the exact molarity concentration of the KOH solution found by standardization). Dilute to the 1000 mL mark with propan-2-ol, and mix thoroughly. Use within two weeks.

A2.5.4.4 *Buffer Nonaqueous Basic*—Add 10 mL of buffer stock Solution B to 100 mL of titration solvent. Use within 1 h.

A2.5.5 *Hydrochloric Acid, Standard Alcoholic Solution* (0.2 N)—Prepare and standardize in accordance with Test Method D664 – IP 177.

A2.5.6 *p*-Naphtholbenzein Indicator Solution—Prepare as described in 7.3.

A2.5.7 *Potassium Chloride Electrolyte*—Prepare a saturated solution of potassium chloride (KCl) in water.

A2.5.8 *Potassium Hydroxide, Standard Alcoholic Solution*—(0.2 N). (**Warning**—Flammable.) (**Warning**—Causes severe burns.) Add 12 g to 13 g of potassium hydroxide (KOH) to approximately 1 L of anhydrous propan-2-ol. 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 absorbants 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.

A2.5.9 *Titration Solvent*—Add 500 mL of toluene and 5 mL of water to 495 mL of anhydrous isopropyl alcohol. The titration solvent should be made up in large quantities.

## A2.6 Preparation of Electrode System

A2.6.1 *Maintenance of Electrodes*—Clean the glass electrode (**Note A2.1**) 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. (**Warning**—Causes severe burns. Strong oxidizer. Contact with materials may cause fire. Hygroscopic.) 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 A2.1—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.

A2.6.2 *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. (**Warning**—Causes severe burns.) 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.

A2.6.3 *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 (**Note A2.2**).

NOTE A2.2—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. When combination electrodes are used, test as in 8.3.

<sup>8</sup> For a detailed discussion of the need for care in preparation of the electrodes, see Lykken, L., Porter, P., Ruliffson, H. D., and Tuemmler, F. D., "Potentiometric Determination of Acidity in Highly Colored Oils," *Industrial and Engineering Chemistry, Analytical Edition*, IENAA, Vol 16, 1944, pp. 219–234.

## A2.7 Standardization of Apparatus

A2.7.1 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  $25\text{ }^{\circ}\text{C} \pm 2\text{ }^{\circ}\text{C}$  and 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.

A2.7.2 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  $25\text{ }^{\circ}\text{C} \pm 2\text{ }^{\circ}\text{C}$ . When the pH meter reading has become steady, record the exact value. If the reading is within 0.2 pH units of 11.0, the initial acidity, pHr, 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. A2.1. Use this graph to convert pH meter readings to initial acidity, pHr.

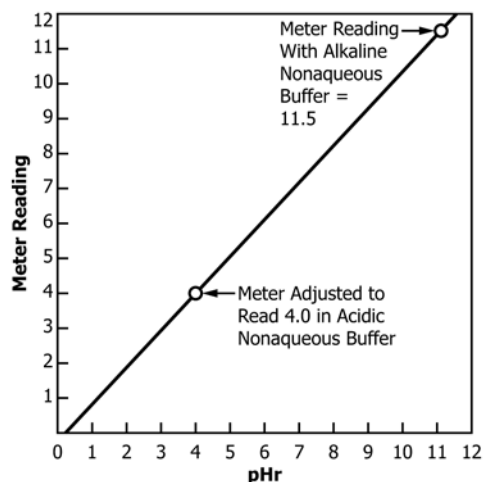


FIG. A2.1 Calibration Curve for Conversion of pH Meter Readings to pHr

## A2.8. Procedure

A2.8.1 Titrate 100 mL of titration solvent with 0.01 N KOH solution until the meter indicates a pHr between 13 and 14.

A2.8.2 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.

A2.8.3 During the titration, plot the volume of titrant against the pHr or meter reading and note on the curve the various color changes at the corresponding pHr values.

NOTE A2.3—The following color 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

A2.8.4 Plot the blank titration on the same paper used for the indicator.

## A2.9. Calculation

A2.9.1 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 color changes between 10 to 12 pHr.

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