



## Standard Test Method for Acid Value of Organic Coating Materials<sup>1</sup>

This standard is issued under the fixed designation D 1639; 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 Department of Defense.*

<sup>ε1</sup> NOTE—Keywords were added editorially in October 1996.

### 1. Scope

1.1 This test method covers the measurement of the free acidity present in the nonvolatile portion of varnishes, oils, certain resins, and paint vehicles, by the reaction with standard alkali solution.

1.2 If carboxylic anhydrides are present, only one half of the reactive groups will be titrated and indicated by this test method.

1.3 *This standard does not purport to address 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.*

### 2. Referenced Documents

#### 2.1 ASTM Standards:

D 362 Specification for Industrial Grade Toluene<sup>2</sup>

D 770 Specification for Isopropyl Alcohol<sup>3</sup>

D 1259 Test Methods for Nonvolatile Content of Resin Solutions<sup>4</sup>

D 1644 Test Methods for Nonvolatile Content of Varnishes<sup>4</sup>

D 1960 Test Method for Loss on Heating of Drying Oils<sup>5</sup>

### 3. Terminology

#### 3.1 Definitions:

3.1.1 *acid value*—the number of milligrams of potassium hydroxide (KOH) required to neutralize the alkali-reactive groups in 1 g of material under the conditions of test (see 6.4).

### 4. Significance and Use

4.1 This test method is used to determine the free acidity present in the nonvolatile portion of varnishes, oils, certain resins, and paint vehicles by the reaction with a standard alkali solution. Use of this test method provides a means whereby the

relative applicability of the varnish, oil, resin, or paint vehicle to the particular end use may be estimated by the buyer and the seller.

4.2 This test method also provides a convenient method of process control for the manufacture of certain resins and paint vehicles designed to meet particular requirements of the buyer and the seller.

### 5. Reagents and Solvents

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>6</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.

5.2 *Phenolphthalein Indicator Solution* (10 g/L)—Dissolve 1 g of phenolphthalein in 100 mL of methanol, ethanol, or isopropanol.

5.3 *Potassium Hydroxide, Methyl Alcohol (Methanol) Solution* (1 mL = 5.6 mg KOH)—Dissolve 6.6 g of potassium hydroxide (KOH) in 1 L of methyl alcohol. Standardize against National Institute of Standards and Technology standard potassium hydrogen phthalate Standard Reference No. 84, using phenolphthalein as the indicator (5.2). Do not adjust the concentration of the solution, but calculate the milligrams of KOH per litre of solution, *K*. (See Note.)

NOTE 1—At the discretion of the purchaser and the seller, an aqueous solution of potassium hydroxide of 0.1 *N* or 0.5 *N* may be used.

NOTE 2—Potassium hydroxide, methyl alcohol (methanol) solution may be purchased from most major laboratory chemical supplier houses. This material should be standardized as just indicated.

5.4 *Neutral Solvent Mixture*—Mix equal parts by volume of isopropyl alcohol (isopropanol) (see Specification D 770) and industrial toluene (see Specification D 362). Neutralize the

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<sup>2</sup> Discontinued; See 1989 Annual Book of ASTM Standards, Vol 06.03.

<sup>3</sup> Annual Book of ASTM Standards, Vol 06.04.

<sup>4</sup> Annual Book of ASTM Standards, Vol 06.01.

<sup>5</sup> Annual Book of ASTM Standards, Vol 06.03.

<sup>6</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 *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopoeia and National Formulary*, U.S. Pharmacopoeial Convention, Inc. (USPC), Rockville, MD.

mixture using 0.1 *N* KOH solution (4.3) and phenolphthalein indicator solution (5.2) until the pink color persists for 1 min.

## 6. Procedure

6.1 Weigh or transfer into a 250-mL Erlenmeyer flask the specimen mass, *S*, prescribed in Table 1.

6.2 Add 100 mL of neutral solvent. Mix until all material is dissolved, warming if necessary. Cool to room temperature, about 77°F (25°C), before titrating. Add 1 mL of phenolphthalein indicator solution and titrate with the 0.1 *N* KOH solution (5.3) to the end point, which is a pink color persisting for 30 s (*V*).

6.3 Repeat with a second specimen.

6.4 In the case of materials containing a solvent, determine the nonvolatile content in accordance with Test Methods D 1259, D 1644, D 1960 or other applicable method agreed upon between the purchaser and the seller.

## 7. Calculation

7.1 Calculate the acid value *A* as follows:

$$A = \frac{VK}{S \times N}$$

**TABLE 1 Specimen Size**

Expected Acid Value	Approximate Specimen Mass, g	Accuracy of weighing, plus or minus, mg
0 to 5	20	50
Over 5 to 15	10	50
Over 15 to 30	5	50
Over 30 to 100	2.5	1
Over 100	1	1

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where:

*V* = volume of KOH solution required for titration of the specimen, mL,

*K* = weight of KOH per millilitre of KOH solution, mg,

*S* = specimen weight, g, and

*N* = nonvolatile content of the material expressed as a decimal fraction.

7.2 Calculate the mean of the two runs.

## 8. Report

8.1 Report the acid value of the nonvolatile matter to the nearest decimal (0.1).

## 9. Precision

9.1 Because of the many types of material covered by this test method, the precision is not as good as might be expected of a quantitative analytical method. The following criteria should be used for judging the acceptability of results:

9.1.1 *Repeatability*—Duplicate results obtained by the same operator should be considered suspect if they differ by more than 10 % of the mean acid value.

9.1.2 *Reproducibility*—Two results obtained by operators in different laboratories should be considered suspect if they differ by more than 20 % of the mean acid value.

## 10. Keywords

10.1 free acidity; nonvolatile; oils; organic coating materials; resins; varnishes