



Designation: D1045 – 19

Standard Test Methods for Sampling and Testing Plasticizers Used in Plastics¹

This standard is issued under the fixed designation D1045; 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 (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope*

1.1 These test methods cover sampling and testing of liquid plasticizers used in compounding of plastics. Acid number, ester content, specific gravity, color, refractive index, and water content are determined.

1.2 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.* Specific hazards information is given in Section 5.

NOTE 1—There is no known ISO equivalent to this standard.

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:*²

- D70 Test Method for Density of Semi-Solid Asphalt Binder (Pycnometer Method)
- D287 Test Method for API Gravity of Crude Petroleum and Petroleum Products (Hydrometer Method)
- D792 Test Methods for Density and Specific Gravity (Relative Density) of Plastics by Displacement
- D883 Terminology Relating to Plastics
- D1193 Specification for Reagent Water

¹ These methods are under the jurisdiction of ASTM Committee D20 on Plastics and are the direct responsibility of Subcommittee D20.15 on Thermoplastic Materials (Section D20.15.07 on Vinyl Chloride Polymers).

Current edition approved Aug. 1, 2019. Published August 2019. Originally approved in 1949. Last previous edition approved in 2014 as D1045 - 14. DOI: 10.1520/D1045-19.

² For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

- D1209 Test Method for Color of Clear Liquids (Platinum-Cobalt Scale)
- D1218 Test Method for Refractive Index and Refractive Dispersion of Hydrocarbon Liquids
- D1544 Test Method for Color of Transparent Liquids (Gardner Color Scale)
- D1600 Terminology for Abbreviated Terms Relating to Plastics
- D3465 Test Method for Purity of Monomeric Plasticizers by Gas Chromatography
- D4052 Test Method for Density, Relative Density, and API Gravity of Liquids by Digital Density Meter
- D5386 Test Method for Color of Liquids Using Tristimulus Colorimetry
- E1 Specification for ASTM Liquid-in-Glass Thermometers
- E203 Test Method for Water Using Volumetric Karl Fischer Titration
- E2251 Specification for Liquid-in-Glass ASTM Thermometers with Low-Hazard Precision Liquids

3. Terminology

3.1 *Definitions*—Definitions are in accordance with Terminology D883 and Terminology D1600, unless otherwise indicated.

4. Significance and Use

4.1 These test methods shall be used in establishing and confirming quality control standards for liquid plasticizers used in the compounding of plastics.

5. Hazards

5.1 *Chemical Hazard of Reagents*—It is possible that some of the chemicals used in this test method are hazardous. Accepted laboratory safety procedures must be followed. See suppliers' material safety data sheets for further information.

5.2 *Hazards with Mercury Thermometers*—Hazards associated with mercury thermometers are discussed in 8.2.

6. Sampling

6.1 The method of sampling specified in 6.2 or 6.3 shall be used, according to the special conditions that exist.

6.2 *From Loaded Tank Car or Other Large Vessel*—The composite sample taken shall be not less than 2 L (½ gal). It is

*A Summary of Changes section appears at the end of this standard

important that the composite sample consists of small samples of not more than 1 L (1 qt) each, taken from near the top and bottom by means of a metal or glass container with removable stopper or top. This device, attached to a suitable pole, shall be lowered to the desired depth, when the stopper or top shall be removed and the container allowed to fill. A bomb sampler attached to a chain is convenient to use; adjust the opening so that the bomb will fill on the way down.

6.3 *From Barrels and Drums*—At least 5 % of the packages in any shipment shall be represented in the sample. It is permissible for the purchaser to increase the percentage of packages to be sampled at his discretion; in the case of plasticizers that are purchased in small quantity, it is permissible to sample and analyze each package, if desired. A portion shall be withdrawn from near the center of each package sampled by means of a “thief” or other sampling device and composited. The composite sample thus obtained shall be not less than 1 L (1 qt) and shall consist of equal portions of not less than 250 mL (½ pt) from each package sampled.

7. Purity of 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.³

7.2 Unless otherwise indicated, references to water shall be understood to mean reagent water conforming to Specification D1193.

8. Thermometers

8.1 Make all temperature measurements with ASTM thermometers of suitable range, accurate to within 0.1°C and conforming to the requirements prescribed in Specifications E2251 or E1. Only use thermometers conforming to Specification E1 if a suitable thermometer conforming to Specification E2251 cannot be found. Thermometric devices such as RTDs, thermistors, and thermocouples with appropriate temperature range and equal or better accuracy as the selected thermometer, are acceptable for use.

8.2 The thermometers referenced in Specification E1 contain mercury, a mercury thallium eutectic alloy, toluene or other suitable liquid colored with a permanent red dye. **(Warning—**Mercury has been designated by EPA and many state agencies as a hazardous material that can cause central nervous system, kidney, and liver damage. Mercury or its vapor may be hazardous to health and corrosive to materials. Cautions shall be taken when handling mercury and mercury-containing products. See the applicable product Safety Data Sheets (SDS) for details and EPA’s website – <http://www.epa.gov/mercury> – for additional information. Users

should be aware that selling mercury and/or mercury-containing products in your state may be prohibited by state law.)

9. Acid Number

9.1 Reagents:

9.1.1 *Alcohol*—Denatured alcohol, Formula No. 3A of the U. S. Bureau of Alcohol, Tobacco, and Firearms.

9.1.2 *Alkali, Standard Solution (0.01 N)*—Prepare and standardize a 0.01 N aqueous solution of sodium hydroxide (NaOH) or a 0.01 N alcoholic solution of potassium hydroxide (KOH).

9.1.3 *Alkali, Standard Solution (0.1 N)*—Prepare and standardize a 0.1 N aqueous solution of sodium hydroxide (NaOH) or a 0.1 N alcoholic solution of potassium hydroxide (KOH).

9.1.4 Acetone.

9.1.5 Bromthymol Blue Indicator Solution.

10. Procedure

10.1 Weigh 25 g of the sample into a 125-mL Erlenmeyer flask and dissolve in 50 mL of alcohol. If the sample is not completely soluble in alcohol, use 50 mL of a mixture of equal parts of alcohol and acetone. With certain samples it will potentially be necessary first to add 25 mL of acetone, warm to effect solution, and then add 25 mL of alcohol.

10.2 Add a few drops of bromthymol blue indicator solution and titrate with 0.01 N NaOH or KOH solution. If the titration exceeds 10 mL, repeat the determination using 0.1 N NaOH or KOH solution.

10.3 *Blank*—Make a blank titration on 50 mL of the solvent used to dissolve the sample.

11. Calculation

11.1 Calculate the acid number, expressed as milligrams of KOH per gram of sample, as follows:

$$\text{Acid number} = [(A - B)N \times 56.1]/C \quad (1)$$

where:

A = NaOH or KOH solution required for titration of the sample, mL,

B = NaOH or KOH solution required for titration of the blank, mL,

N = normality of the NaOH or KOH solution, and

C = sample used, g.

11.2 *Conversion Factors*—In the case of esters, it is acceptable to express the results as a percentage by weight of the appropriate acid, by using the proper factor in the equation in 11.1 or by using the conversion factors below:

$$\begin{aligned} \text{Acid Number (Mg KOH/g)} &= \text{Milliequivalent (Meq)/100 g} \times 0.561 \\ \% \text{ Acid} &= \text{Meq/100 g} \times \text{milliequivalent weight of acid} \end{aligned}$$

TABLE Milliequivalent Weight of Various Acids

Maleic = 0.058
Acetic = 0.060
Phthalic = 0.083
Hydrochloric = 0.037
Phosphoric = 0.049
Adipic = 0.073

³ *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 Pharmacopeia and National Formulary*, U.S. Pharmaceutical Convention, Inc. (USPC), Rockville, MD.