Designation: D1045 – 14

Standard Test Methods for Sampling and Testing Plasticizers Used in Plastics[1]

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 and health 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.

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D70 Test Method for Density of Semi-Solid Bituminous Materials (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
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.

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 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

[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).

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[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.

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

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 All temperature measurements shall be made with ASTM thermometers of suitable range, accurate to within 0.1°C and conforming to the requirements prescribed in Specification E1. Thermometric devices such as RTDs, thermistors, thermocouples, and liquid-in-glass thermometers (as in Specification E2251), with appropriate temperature range and equal or better accuracy as the selected thermometer, are acceptable for use.

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:

$$\textit{Acid Number}\,(\textit{Mg KOH/g}) = \textit{Milliequivalent}\,(\textit{Meq})/100\ g \times 0.561$$
$$\%\ \textit{Acid} = \textit{Meq}/100\ g \times \textit{milliequivalent weight of acid}$$

TABLE Milliequivalent Weight of Various Acids

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

12. Ester Content

12.1 *Selection of Test Method:*

12.1.1 Ester content shall be determined using either the gas chromatographic method (Test Method D3465) or the titrimetric method (Section 13).

13. Titrimetric Method

13.1 *Reagents:*

13.1.1 *Bromthymol Blue Indicator Solution.*

13.1.2 *Hydrochloric Acid, Standard (0.5N)*Prepare and standardize a 0.5 N aqueous solution of hydrochloric acid (HCl).

13.1.3 *Potassium Hydroxide, Standard Solution (0.5 N)*—Prepare and standardize a 0.5 *N* alcoholic solution of potassium hydroxide (KOH).

13.2 *Procedure:*

13.2.1 Weigh accurately about 2 g of the sample into a 250-mL Erlenmeyer flask with ground-glass joint. By means of a constant delivery pipet or buret, add 50 mL of 0.5 *N* KOH solution. Connect to a water-cooled condenser with ground-glass joint and reflux for a period of 1 to 4 h, depending on the ester being tested, or until saponification is complete.

13.2.2 After the apparatus has cooled, wash down the condenser with water and disconnect. Add a few drops of bromthymol blue indicator solution to contents of the flask and titrate with 0.5 *N* HCl.

[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.

13.2.3 *Blank*—Run a blank, containing 50 mL of the 0.5 *N* KOH solution, along with the sample.

13.3 *Calculation:*

13.3.1 Calculate the ester content, expressed in milligrams of KOH per gram of sample, as follows:

$$\text{Ester content} = [(D - E)N \times 56.1]/G - F \quad (2)$$

where:

D = HCl required for titration of the blank, mL,
E = HCl required for titration of the sample, mL,
F = correction for acidity of sample (Section 11),
N = normality of the HCl, and
G = sample used, g.

13.3.2 If desired, it is acceptable to express the results as a percentage of the appropriate ester by weight, by using the proper factor in the equation in 13.3.1.

14. Specific Gravity

14.1 *Selection of Test Method:*

14.1.1 Specific gravity of low viscosity plasticizers shall be determined using a hydrometer (Test Method D287), Westphal balance, pycnometer (Section 15) or Digital Density Meter (Test Method D4052). Extremely viscous plasticizers shall be tested by Test Methods D792 or Test Method D70.

15. Procedure Using Pycnometer

15.1 Determine the weight capacity of the pycnometer with water at 23 ± 1°C. Fill this standardized pycnometer with a portion of the sample that has been cooled to approximately 20°C. Insert the thermometer or capillary tube, taking care to avoid introduction of air bubbles. Set the pycnometer in a constant-temperature water bath maintained at 23 ± 1°C (73.4 ± 1.8°F) for a period of at least 30 min. Remove the droplet of sample from the overflow capillary and cover with the glass cap. Clean the outside of the pycnometer and weigh.

15.2 *Calculation:*

15.2.1 Calculate the specific gravity as follows:

$$\text{Specific gravity at } 23/23°C = A/B \quad (3)$$

where:

A = grams of sample used, and
B = water capacity of pycnometer in grams.

16. Color

16.1 *Selection of Test Method:*

16.1.1 The color of plasticizers shall be determined using either Test Methods D5386 or D1209 for color less than 200 APHA and Test Method D1544 for color higher than 200 APHA.

17. Refractive Index

17.1 *Procedure:*

17.1.1 The refractive index of plasticizers shall be determined using Test Method D1218.

18. Water Content

18.1 *Procedure:*

18.1.1 The water content shall be measured using Test Method E203.

19. Report

19.1 Report the results for acid number, ester content, specific gravity, color, refractive index, and water content in a table along with the identification of the plasticizer tested and the test methods used.

20. Precision and Bias

20.1 Precision and bias statements are an integral part of referenced test procedures used in this test method. These statements are to be considered applicable to test results obtained by use of this test method.

20.2 Attempts to develop precision and bias statements for Acid Number, Ester Content-Titrimetric, Specific Gravity-Pycnometer, Color, and Refractive Index have not been successful. For this reason, data on precision and bias cannot be given. If anyone wishes to participate in the development of precision and bias data, contact the ASTM headquarters.

21. Keywords

21.1 plasticizer; plasticizer acid number; plasticizer color; plasticizer ester content; plasticizer refractive index; sampling; test methods

SUMMARY OF CHANGES

Committee D20 has identified the location of selected changes to this standard since the last issue (D1045 - 08) that may impact the use of this standard. (March 1, 2014)

(1) Updated ISO equivalency statement in Note 1.
(2) Updated Subcommittee information.
(3) Updated thermometers to include Specification E2251, as well as Section 8.
(4) Moved Section 8 to precede "Acid Number."
(5) Assigned section numbers to tests: Acid Number, Ester Content, etc.
(6) Added a Report section (Section 19).