Designation: D 268 – 96

AMERICAN SOCIETY FOR TESTING AND MATERIALS
100 Barr Harbor Dr., West Conshohocken, PA 19428
Reprinted from the Annual Book of ASTM Standards. Copyright ASTM

Standard Guide for Sampling and Testing Volatile Solvents and Chemical Intermediates for Use in Paint and Related Coatings and Material[1]

This standard is issued under the fixed designation D 268; 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.

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

1. Scope

1.1 This guide covers procedures for the sampling and testing of volatile solvents used in the manufacture of paint, lacquer, varnish, and related products. The test methods are listed in Table 1.

1.2 *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 consult and establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*

D 13 Specification for Spirits of Turpentine[2]
D 56 Test Method for Flash Point by Tag Closed Tester[3]
D 86 Test Method for Distillation of Petroleum Products[3]
D 93 Test Methods for Flash Point by Pensky-Martens Closed Cup Tester[3]
D 156 Test Method for Saybolt Color of Petroleum Products (Saybolt Chromometer Method)[3]
D 233 Methods of Sampling and Testing Turpentine[2]
D 235 Specification for Mineral Spirits (Petroleum Spirits) (Hydrocarbon Dry Cleaning Solvent)[4]
D 329 Specification for Acetone[4]
D 611 Test Method for Aniline Point and Mixed Aniline Point of Petroleum Products and Hydrocarbon Solvents[3]
D 847 Test Method for Acidity of Benzene, Toluene, Xylenes, Solvent Naphthas, and Similar Industrial Aromatic Hydrocarbons[4]
D 848 Test Method for Acid Wash Color of Industrial Aromatic Hydrocarbons[4]
D 849 Test Method for Copper Strip Corrosion of Industrial Aromatic Hydrocarbons[4]
D 850 Test Method for Distillation of Industrial Aromatic

TABLE 1 List of Test Methods

Test Method	Section	ASTM Method
Acidity in:		
Aromatic hydrocarbons	11	D 847
Volatile solvents	11	D 1613
Acid wash color of aromatics	23	D 848
Alcohols in ketones	18	D 2804, D 3329
Alkalinity in acetone	12	D 1614
Aromatics in mineral spirits	25	D 3257
Color, platinum cobalt scale	6	D 1209
Copper corrosion test:		
Aromatic hydrocarbons	14	D 849
Mineral spirits	14	D 1616
Distillation range:		
Aromatic hydrocarbons	7	D 850
Mineral spirits, turpentine	7	D 86
Volatile organic liquids	7	D 1078
Ester value	13	D 1617
Esters, purity	13	D 3545
Flash point:		
Pensky-Martens closed cup	17	D 93
Tag closed cup	17	D 56
Tag open cup	17	D 1310
Setaflash tester	17	D 3278
Method surveys:		
Ethylene and propylene glycols	22	E 202
Methanol	21	E 346
Nonaromatics in aromatics	24	D 2360
Nonvolatile matter	8	D 1353
Odor	9	D 1296
Paraffins in aromatics	24	D 2360
Permanganate time for acetone and methanol	16	D 1363
Purity of ketones	18	D 2192, D 2804, D 3329, D 3893
Sampling	4	E 300
Solvent power evaluation:		
Aniline point and mixed aniline point of petroleum products and hydrocarbon solvents	19	D 611
Kauri-butanol value of hydrocarbon solvents	19	D 1133
Dilution ratio in cellulose nitrate solution for active solvents, hydrocarbon diluents, and cellulose nitrates	19	D 1720
Specific gravity	5	D 891, D 2935, D 3505, D 1555
Sulfur as hydrogen sulfide and sulfur dioxide	15	D 853
Water:		
Fischer reagent titration method	10	D 1364, E 203
Turbidity method	10	D 1476
Water miscibility of water-soluble solvents	20	D 1722

[1] This guide is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings, Materials, and Applications and is the direct responsibility of Subcommittee D01.35 on Solvents, Plasticizers, and Chemical Intermediates.

Current edition approved May 10, 1996. Published July 1996. Originally published as D 268 – 27 T. Last previous edition D 268 – 90 (1993)$^{\epsilon 1}$.

[2] *Annual Book of ASTM Standards*, Vol 06.03.

[3] *Annual Book of ASTM Standards*, Vol 05.01.

[4] *Annual Book of ASTM Standards*, Vol 06.04.

Hydrocarbons and Related Materials[4]
- D 853 Test Method for Hydrogen Sulfide and Sulfur Dioxide Content (Qualitative) of Industrial Aromatic Hydrocarbons[4]
- D 891 Test Methods for Specific Gravity, Apparent, of Liquid Industrial Chemicals[5]
- D 1078 Test Method for Distillation Range of Volatile Organic Liquids[4]
- D 1133 Test Method for Kauri-Butanol Value of Hydrocarbon Solvents[4]
- D 1209 Test Method for Color of Clear Liquids (Platinum-Cobalt Scale)[4]
- D 1296 Test Method for Odor of Volatile Solvents and Diluents[4]
- D 1310 Test Method for Flash Point and Fire Point of Liquids by Tag Open-Cup Apparatus[6]
- D 1353 Test Method for Nonvolatile Matter in Volatile Solvents for Use in Paint, Varnish, Lacquer, and Related Products[4]
- D 1363 Test Method for Permanganate Time of Acetone and Methanol[4]
- D 1364 Test Method for Water in Volatile Solvents (Karl Fischer Reagent Titration Method)[4]
- D 1476 Test Method for Heptane Miscibility of Lacquer Solvents[4]
- D 1555 Test Method for Calculation of Volume and Weight of Industrial Aromatic Hydrocarbons[4]
- D 1613 Test Method for Acidity in Volatile Solvents and Chemical Intermediates Used in Paint, Varnish, Lacquer, and Related Products[4]
- D 1614 Test Method for Alkalinity in Acetone[4]
- D 1616 Test Method for Copper Corrosion by Mineral Spirits[7]
- D 1617 Test Method for Ester Value of Solvents and Thinners[4]
- D 1720 Test Method for Dilution Ratio of Active Solvents in Cellulose Nitrate Solutions[4]
- D 1722 Test Method for Water Miscibility of Water-Soluble Solvents[4]
- D 2192 Test Method for Purity of Aldehydes and Ketones[4]
- D 2360 Test Method for Trace Impurities in Monocyclic Aromatic Hydrocarbons by Gas Chromatography[4]
- D 2804 Test Method for Purity of Methyl Ethyl Ketone by Gas Chromatography[4]
- D 2935 Test Method for Apparent Density of Industrial Aromatic Hydrocarbons[4]
- D 3257 Test Methods for Aromatics in Mineral Spirits by Gas Chromatography[4]
- D 3278 Test Method for Flash Point of Liquids by Setaflash Closed–Cup Apparatus[6]
- D 3329 Test Method for Purity of Methyl Isobutyl Ketone by Gas Chromatography[4]
- D 3505 Test Method for Density or Relative Density of Pure Liquid Chemicals[4]
- D 3545 Test Method for Alcohol Content and Purity of Acetate Esters by Gas Chromatography[4]
- D 3893 Test Method for Purity of Methyl Amyl Ketone and Methyl Isoamyl Ketone by Gas Chromatography[4]
- E 12 Terminology Relating to Density and Specific Gravity of Solids, Liquids, and Gases[5]
- E 201 Test Method for Calculation of Volume and Weight of Industrial Chemical Liquids[5]
- E 202 Test Methods for Analysis of Ethylene Glycols and Propylene Glycols[5]
- E 203 Test Method for Water Using Karl Fischer Reagent[5]
- E 300 Practice for Sampling Industrial Chemicals[5]
- E 346 Method for Analysis of Methanol[5]

3. Significance and Use

3.1 A brief discussion of each test method is given with the intent of helping the user in the selection of the most applicable procedure where more than one is available.

4. Sampling

4.1 Representative samples are a prerequisite for the evaluation of any product. The directions for obtaining representative samples cannot be made explicit to cover all cases and must be supplemented by judgment, skill, and sampling experience. It is recommended that Practice E 300 be employed in sampling liquid solvents.

5. Specific Gravity

5.1 Specific gravity of liquids is defined in Terminology E 12 as "the ratio of the mass of a unit volume of a material to the mass of the same volume of gas-free distilled water at a stated temperature." When the stated temperature of the water is 4.0°C, specific gravity and density are numerically equal.

5.2 The apparent specific gravity of liquid is defined in Terminology E 12 as "the ratio of the weight in air of a unit volume of material at a stated temperature to the weight in air of equal density of an equal volume of gas-free, distilled water at a stated temperature."

NOTE 1—Specific gravity or density is an intrinsic property of all substances and can to a degree be used to identify them. When such substances are of high purity, specific gravity may be used in support of other properties to define their degree of purity. The use of specific gravity for such purposes, however, is valid only when all components and their relative effects upon the specific gravity of the system are known.

5.3 The choice of test method for determining specific gravity is largely dependent on the degree of accuracy required. In general, when the product specification requires an accuracy to the third decimal place, the hydrometer or specific gravity balance method may be employed. When the product specification requires an accuracy to the fourth decimal place, a pycnometer method should be employed. Test Methods D 891 give procedures using all three techniques.

5.4 With specific reference to the determination of density or specific gravity of a number of aromatic and cyclic hydrocarbon solvents, Test Method D 3505 describes a simplified procedure for this measurement.

5.5 Methods for converting specific gravity data to weight and volume data at various temperatures are given in Method E 201 for oxygenated and chlorinated compounds, and for

[5] *Annual Book of ASTM Standards*, Vol 15.05.

[6] *Annual Book of ASTM Standards*, Vol 06.01.

[7] Discontinued, see 1981 *Annual Book of ASTM Standards*, Part 29. Replaced by Test Method D 130.

aromatic hydrocarbons in Test Method D 1555.

5.6 The measurement of density of aromatic hydrocarbons at any convenient temperature, and the conversion of the data to an applicable specification or storage temperature are described in Test Method D 2935.

6. Color

6.1 The property of color of a solvent will vary in importance with the application for which it is intended, the amount of color that can be tolerated being dependent on the color characteristics of the material in which it is used. The paint, varnish, and lacquer solvents, or diluents commercially available on today's market normally have little or no color. The presence or absence of color in such material is an indication of the degree of refinement to which the solvent has been subjected or of the cleanliness of the shipping or storage container in which it is handled, or both (see Test Method D 1209).

NOTE 2—For a number of years the term "water-white" was considered sufficient as a measurement of solvent color. Several expressions for defining" water-white" gradually appeared and it became evident that a more precise color standard was needed. This was accomplished in 1952 with the adoption of Test Method D 1209 using the platinum cobalt scale. This method is similar to the description given in the *Standard Methods for the Examination of Water and Waste Water* of the American Public Health Assn., 14th Ed., p. 65 and is referred to by many as "APHA Color." The preparation of these platinum-cobalt color standards was originally described by Hazen, A., *American Chemical Journal,* Vol. XIV, 1892, p. 300, in which he assigned the number 5 (parts per ten thousand) to his platinum-cobalt stock solution. Subsequently, in their first edition (1905) of *Standard Methods for the Examination of Water*, the American Public Health Assn., using exactly the same concentration of reagents, assigned to color designation 500 (parts per million) which is the same ratio. The parts per million nomenclature is not used since color is not referred directly to a weight relationship. It is therefore recommended that the incorrect term "Hazen Color" should not be used. Also, because it refers primarily to water, the term "APHA Color" is undesirable. The recommended nomenclature for referring to the color of organic liquids is "Platinum-Cobalt Color, Test Method D 1209."

NOTE 3—The petroleum industry uses the Saybolt colorimeter Test Method D 156 for measuring and defining the color of hydrocarbon solvents; however, this system of color measurement is not commonly employed outside of the petroleum industry. It has been reported by various sources that a Saybolt color of + 25 is equivalent to 25 in the platinum-cobalt system or to colors produced by masses of potassium dichromate ranging between 4.8 and 5.6 mg. dissolved in 1 L of distilled water. Because of the differences in the spectral characteristics of the several color systems being compared and the subjective manner in which the measurements are made, exact equivalencies are difficult to obtain.

7. Distillation Range

7.1 The distillation range of an organic solvent is an empirical set of data peculiar to the solvent under study and the apparatus used giving the purchaser an indication of the product quality available to him.

NOTE 4—The distillation range provides information on the initial boiling point, percent distilled at certain temperatures, and the dry point. These parameters may be affected by improper refining techniques, impurities inherent in the sample, or contamination. It is absolutely necessary that the purchaser and seller employ the same type of apparatus, including thermometers, and follow an identical procedure as agreed upon. If these factors are not followed precisely, it is quite possible disagreement will result between the parties.

7.2 Three test methods are available for determining the distillation range of solvents. The major differences among the three methods are the size of distillation flasks and type of thermometers (partial or total immersion) employed. Flask size has little to no effect on the results obtained between laboratories beyond the limits of error noted for each test method. The advantage of the larger size flask is to prevent "boil over" when high-boiling products, processing relatively high coefficients of expansion are being tested. On the other hand, differences between laboratories will be large when one laboratory employs a partial immersion thermometer and another a total immersion instrument. The spread between results will increase as the boiling range rises above 100°C. Partial immersion thermometers are preferred for narrow boiling products since they require no emergent stem temperature correction. The type of heat source may affect the distillation range of products boiling within 1 or 2°C. This is especially true for low-boiling solvents such as methyl alcohol or acetone. A large electric heater tends to distort the dry point due to the heating effect of infrared radiation on the bulb of the thermometer, while a properly adjusted gas burner minimizes this effect. The following test methods are commonly used in determining distillation ranges:

7.2.1 *Test Method D 1078*, using a 200-mL flask, high-precision partial immersion thermometers, and gas or electric heat. The latter may be used only after it has proven to give results comparable to those obtained when using gas heat. The method was designed specifically for determining the distillation range of volatile solvents used in coating compositions, but is applicable to any volatile organic liquid that boils between 30 and 300°C, and is chemically stable during the distillation process.

7.2.2 *Test Method D 850*, using a 200-mL flask, partial immersion thermometer, and electric or gas heat. This method is applicable to industrial aromatic hydrocarbons and related products. It is particularly suited to narrow boiling hydrocarbons or mixtures of hydrocarbons.

7.2.3 *Method D 86*, using a 100-mL flask for products showing an end point below 250°C, a 125-mL flask for products showing an end point above 250°C, total immersion thermometers, and electric or gas heat. This method is applicable to mineral spirits conforming to Specification D 235, and to spirits of turpentine conforming to Specification D 13, using partial immersion thermometers in accordance with Test Methods D 233, and to other hydrocarbon mixtures that have wide boiling ranges.

8. Nonvolatile Matter

8.1 The nonvolatile matter test is run usually on volatile solvents capable of evaporating in a reasonable period of time at 105°C. The finding of a residue significantly higher than 5 mg/100 mL indicates the presence of either contamination or impurities inherent in the solvent. In certain cases this may adversely affect a product or coating system into which the solvent is introduced. See Test Method D 1353.

9. Odor

9.1 The evaluation of the characteristic odor of a solvent is a quick and simple means of identifying a material as well as