



Designation: D268 – 22

# Standard Guide for Sampling and Testing Volatile Solvents and Chemical Intermediates for Use in Paint and Related Coatings and Material<sup>1</sup>

This standard is issued under the fixed designation D268; 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 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 For specific hazard information and guidance, see Suppliers' Material Safety Data Sheet for materials listed in this guide.

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

1.4 *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.*

1.5 *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>

- [D13 Specification for Spirits of Turpentine](#)
- [D56 Test Method for Flash Point by Tag Closed Cup Tester](#)
- [D86 Test Method for Distillation of Petroleum Products and](#)

- [Liquid Fuels at Atmospheric Pressure](#)
- [D93 Test Methods for Flash Point by Pensky-Martens Closed Cup Tester](#)
- [D130 Test Method for Corrosiveness to Copper from Petroleum Products by Copper Strip Test](#)
- [D156 Test Method for Saybolt Color of Petroleum Products \(Saybolt Chromometer Method\)](#)
- [D233 Test Methods of Sampling and Testing Turpentine](#)
- [D235 Specification for Mineral Spirits \(Petroleum Spirits\) \(Hydrocarbon Dry Cleaning Solvent\) \(Withdrawn 2021\)<sup>3</sup>](#)
- [D329 Specification for Acetone](#)
- [D611 Test Methods for Aniline Point and Mixed Aniline Point of Petroleum Products and Hydrocarbon Solvents](#)
- [D847 Test Method for Acidity of Benzene, Toluene, Xylenes, Solvent Naphthas, and Similar Industrial Aromatic Hydrocarbons](#)
- [D848 Test Method for Acid Wash Color of Industrial Aromatic Hydrocarbons](#)
- [D849 Test Method for Copper Strip Corrosion by Industrial Aromatic Hydrocarbons](#)
- [D850 Test Method for Distillation of Industrial Aromatic Hydrocarbons and Related Materials](#)
- [D853 Test Method for Hydrogen Sulfide and Sulfur Dioxide Content \(Qualitative\) of Industrial Aromatic Hydrocarbons \(Withdrawn 2013\)<sup>3</sup>](#)
- [D891 Test Methods for Specific Gravity, Apparent, of Liquid Industrial Chemicals](#)
- [D1078 Test Method for Distillation Range of Volatile Organic Liquids](#)
- [D1133 Test Method for Kauri-Butanol Value of Hydrocarbon Solvents](#)
- [D1209 Test Method for Color of Clear Liquids \(Platinum-Cobalt Scale\)](#)
- [D1296 Test Method for Odor of Volatile Solvents and Diluents \(Withdrawn 2021\)<sup>3</sup>](#)
- [D1310 Test Method for Flash Point and Fire Point of Liquids by Tag Open-Cup Apparatus](#)

<sup>1</sup> This guide is under the jurisdiction of ASTM Committee D01 on Paint and Related Coatings, Materials, and Applications and is the direct responsibility of Subcommittee D01.35 on Solvents, Plasticizers, and Chemical Intermediates.

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<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.

<sup>3</sup> The last approved version of this historical standard is referenced on [www.astm.org](http://www.astm.org).

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

**TABLE 1 List of Test Methods**

Test Method	Section	ASTM Method
Acidity in:		
Aromatic hydrocarbons	11	D847
Volatile solvents	11	D1613
Acid wash color of aromatics	23	D848
Alcohols in ketones	18	D2804, D3329
Alkalinity in acetone	12	D1614
Aromatics in mineral spirits	25	D3257
Color, platinum cobalt scale	6	D1209
Copper corrosion test:		
Aromatic hydrocarbons	14	D849
Mineral spirits	14	D130
Distillation range:		
Aromatic hydrocarbons	7	D850
Mineral spirits, turpentine	7	D86
Volatile organic liquids	7	D1078
Ester value	13	D1617
Esters, purity	13	D3545
Flash point:		
Pensky-Martens closed cup	17	D93
Tag closed cup	17	D56
Tag open cup	17	D1310
Setaflash tester	17	D3278
Method surveys:		
Ethylene and propylene glycols	22	E202
Methanol	21	E346
Nonaromatics in aromatics	24	D2360
Nonvolatile matter	8	D1353
Odor	9	D1296
Paraffins in aromatics	24	D2360
Permanganate time for acetone and methanol	16	D1363
Purity of ketones	18	D2192, D2804, D3329, D3893
Sampling	4	E300
Solvent power evaluation:		
Aniline point and mixed aniline point of petroleum products and hydrocarbon solvents	19	D611
Kauri-butanol value of hydrocarbon solvents	19	D1133
Dilution ratio in cellulose nitrate solution for active solvents, hydrocarbon diluents, and cellulose nitrates	19	D1720
Specific gravity	5	D891, D2935, D3505, D1555
Sulfur as hydrogen sulfide and sulfur dioxide	15	D853
Water:		
Fischer reagent titration method	10	D1364, E203
Turbidity method	10	D1476
Water miscibility of water-soluble solvents	20	D1722

**D1353 Test Method for Nonvolatile Matter in Volatile Solvents for Use in Paint, Varnish, Lacquer, and Related Products**

**D1363 Test Method for Permanganate Time of Acetone and Methanol**

**D1364 Test Method for Water in Volatile Solvents (Karl Fischer Reagent Titration Method) (Withdrawn 2021)<sup>3</sup>**

**D1476 Test Method for Heptane Miscibility of Lacquer Solvents (Withdrawn 2021)<sup>3</sup>**

**D1555 Test Method for Calculation of Volume and Weight of Industrial Aromatic Hydrocarbons and Cyclohexane**

**D1613 Test Method for Acidity in Volatile Solvents and Chemical Intermediates Used in Paint, Varnish, Lacquer, and Related Products**

**D1614 Test Method for Alkalinity in Acetone**

**D1617 Test Method for Ester Value of Solvents and Thinners (Withdrawn 2021)<sup>3</sup>**

**D1720 Test Method for Dilution Ratio of Active Solvents in Cellulose Nitrate Solutions (Withdrawn 2021)<sup>3</sup>**

**D1722 Test Method for Water Miscibility of Water-Soluble Solvents**

**D2192 Test Method for Purity of Aldehydes and Ketones (Withdrawn 2021)<sup>3</sup>**

**D2360 Test Method for Trace Impurities in Monocyclic Aromatic Hydrocarbons by Gas Chromatography (Withdrawn 2016)<sup>3</sup>**

**D2804 Test Method for Purity of Methyl Ethyl Ketone By Gas Chromatography (Withdrawn 2021)<sup>3</sup>**

**D2935 Test Method for Apparent Density of Industrial Aromatic Hydrocarbons (Withdrawn 2005)<sup>3</sup>**

**D3257 Test Methods for Aromatics in Mineral Spirits by Gas Chromatography (Withdrawn 2021)<sup>3</sup>**

**D3278 Test Methods for Flash Point of Liquids by Small Scale Closed-Cup Apparatus**

**D3329 Test Method for Purity of Methyl Isobutyl Ketone by Gas Chromatography**

**D3505 Test Method for Density or Relative Density of Pure Liquid Chemicals**

**D3545 Test Method for Alcohol Content and Purity of Acetate Esters by Gas Chromatography (Withdrawn 2021)<sup>3</sup>**

**D3893 Test Method for Purity of Methyl Amyl Ketone and Methyl Isoamyl Ketone by Gas Chromatography**

**E12 Terminology Relating to Density and Specific Gravity of Solids, Liquids, and Gases (Withdrawn 1996)<sup>3</sup>**

**E201 Test Method for Calculation of Volume and Weight of Industrial Chemical Liquids (Withdrawn 2001)<sup>3</sup>**

**E202 Test Methods for Analysis of Ethylene Glycols and Propylene Glycols**

**E203 Test Method for Water Using Volumetric Karl Fischer Titration**

**E300 Practice for Sampling Industrial Chemicals**

**E346 Test Methods for Analysis of Methanol (Withdrawn 2017)<sup>3</sup>**

### 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 E300 be employed in sampling liquid solvents.

### 5. Specific Gravity

5.1 Specific gravity of liquids is defined in Terminology E12 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 E12 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 **D891** 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 **D3505** 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 Test Method **E201** for oxygenated and chlorinated compounds, and for aromatic hydrocarbons in Test Method **D1555**.

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 **D2935**.

## 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 **D1209**).

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 **D1209** 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 **D1209**.”

NOTE 3—The petroleum industry uses the Saybolt colorimeter Test Method **D156** 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 **D1078***, 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 **D850***, 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 *Test Method D86*, 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 **D235**, and to spirits of turpentine conforming to Specification **D13**, using partial immersion thermometers in accordance with Test Methods **D233**, 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 **D1353**.

## 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 determining its suitability from an odor point of view for use in a solvent system. Note, however, that inhaling certain solvent fumes may be hazardous (see **1.2**). Several ASTM standard specifications for solvents list the odor test as an option to be agreed upon between the buyer and the seller. Residual odor may be due to improper refining techniques, impurities inherent in the solvent, or contamination. Whatever the source, a pronounced residual odor may find its way into a finished product and thus adversely affect the coating system. See Test Method **D1296**.

## 10. Water

10.1 Two test methods are available for determining the moisture or water content of a solvent:

10.1.1 *Test Method D1364*, covers the determination of moisture concentration in absolute terms. It is not only sensitive and accurate for the level of water found in commercially available solvents, but is applicable to a wide range of materials including hydrocarbon and oxygenated hydrocarbon solvents. The principles of the procedure are based on the use of the Karl Fischer reagent, Test Method **E203**.

10.1.2 *Test Method D1476*, may be used to determine whether there is sufficient moisture in a solvent to cause turbidity when the solvent is mixed with *n*-heptane. This method is limited in its usefulness in that it does not cover measurement of water in absolute terms and is subject to a wide range of sensitivity. For example, when applied to esters and higher molecular weight ketones (methyl ethyl ketone and higher) its lower limit for detecting water ranges from 0.1 to 0.3 %, depending on the particular solvent being tested. When applied to acetone and most alcohols, its lower limit of sensitivity ranges from 0.5 to 2 %, again depending on the particular material. Its main advantage is to detect the gross contamination of a solvent by water.

## 11. Acidity

11.1 Acidity in a solvent may be due to improper refining techniques, instability in storage, or contamination. Some processes are highly sensitive to acidity while others are not. Despite the fact that various acids might be involved, two calculations are commonly given for determining acidity, that is, weight percent as acetic acid, and acid number (milligrams of potassium hydroxide consumed per gram of sample). The purchaser and seller must agree as to which calculation should be used for purchase specifications. See Test Methods **D847** and **D1613**.

## 12. Alkalinity

NOTE 5—Alkalinity is so rarely encountered in commercially available solvents that among the solvents under the jurisdiction of Committee D01, only Specification **D329** for acetone contains a requirement for alkalinity.

12.1 If alkalinity is suspected as a contaminant in a solvent, determine the alkalinity in accordance with Test Method **D1614**. This method may be adapted to water-immiscible solvents by substituting isopropyl, or ethyl alcohol conforming to Formula No. 3A of the U. S. Bureau of Internal Revenue, for water in the test procedure.

## 13. Ester Value

13.1 Test Method **D1617** may be used to estimate the purity of an ester, the remaining portion of the material usually being the alcohol associated with the original reaction to produce the ester. The method also may be employed to obtain the total ester content of a lacquer thinner.

13.2 Essentially pure and urethane grade acetate esters may also be analyzed by the gas chromatographic procedure, Test Method **D3545**, which provides not only the ester content but also the concentration of the remaining parent alcohol. The alcohol content is of special interest with urethane grade solvents.

## 14. Copper Corrosion Test

14.1 The copper corrosion test normally is applied to hydrocarbon solvents (aliphatic and aromatic). However, the test also may be used in connection with oxygenated solvents. The test is a visual estimate of the presence of free and combined sulfur and is not a measure of the corrosiveness of solvent to other metals. See Test Methods **D849** and **D130**.

## 15. Sulfur

15.1 Test Method **D853** indicates the presence of sulfur dioxide or hydrogen sulfide in aromatic hydrocarbons. No absolute analysis of the sulfur content is obtained. The method is not sensitive to organic sulfur compounds. The presence in a solvent of detectable sulfur compounds using this method indicates the possibility of odor-forming bodies, as well as color-forming agents (color degradation in the final product).

## 16. Permanganate Time Test for Acetone and Methanol

16.1 The measurement of permanganate time is a sensitive means for detecting trace quantities of reducing substances such as aldehydes and unsaturates that might be present in