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Designation: D 2804 – 02

Standard Test Method for Purity of Methyl Ethyl Ketone By Gas Chromatography[1]

This standard is issued under the fixed designation D 2804; 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 test method covers the determination of the purity of methyl ethyl ketone by gas chromatography. Impurities including water, acidity, and nonvolatile matter are measured by appropriate ASTM procedures and the results are used to normalize the chromatographic value.

1.2 For purposes of determining conformance of an observed value or a calculated value using this test method to relevant specifications, test result(s) shall be rounded off "to the nearest unit" in the last right-hand digit used in expressing the specification limit, in accordance with the rounding-off method of Practice E 29.

1.3 For hazard information and guidance, see the supplier's Material Safety Data Sheet.

2. Referenced Documents

2.1 *ASTM Standards:*

D 1353 Test Method for Nonvolatile Matter in Volatile Solvents for Use in Paint, Varnish, Lacquer, and Related Products[2]

D 1364 Test Method for Water in Volatile Solvents (Karl Fischer Reagent Titration Method)[2]

D 1613 Test Method for Acidity in Volatile Solvents and Chemical Intermediates Used in Paint, Varnish, Lacquer and Related Products[2]

D 2593 Test Method for Butadiene Purity and Hydrocarbon Impurities by Gas Chromatography[3]

D 4626 Practice for Calculation of Gas Chromatographic Response Factor[4]

E 29 Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications[5]

E 180 Practice for Determining the Precision of ASTM Methods for Analysis and Testing of Industrial and Specialty Chemicals[6]

3. Summary of Test Method

3.1 A representative specimen is introduced into a gas-chromatographic column. The methyl ethyl ketone is separated from other impurities such as hydrocarbons, alcohols, acetone, di-*sec*-butyl ether, and ethyl acetate as the components are transported through the column by an inert carrier gas. The separated components are measured in the effluent by a detector and recorded as a chromatogram. The chromatogram is interpreted by applying component-attenuation and detector-response factors to the peak areas, and the relative concentration is determined by relating individual peak response to the total peak response. Water, acidity, and nonvolatiles are measured by the procedures listed in 3.2, and the results are used to normalize the results obtained by gas chromatography.

3.2 The appropriate ASTM test methods are:

3.2.1 *Water*—Test Method D 1364.

3.2.2 *Acidity*—Test Method D 1613.

3.2.3 *Nonvolatile Matter*—Test Method D 1353.

4. Significance and Use

4.1 This test method provides a measurement of commonly found impurities in commercially available methyl ethyl ketone. The measurement of these impurities and the results thereof can individually or when totaled and subtracted from 100 (assay) be used for specification purposes.

5. Apparatus

5.1 *Chromatograph*—Any gas chromatograph having either a thermal-conductivity or flame ionization detector provided the system has sufficient sensitivity and stability to obtain for 0.01 weight % of impurity a recorder deflection of at least 2 mm at a signal-to-noise ratio of at least 5 to 1. The specimen size to be used in judging the sensitivity must be such that the column is not overloaded.

5.2 *Column*—Any column capable of resolving methyl ethyl ketone from the impurities that may be present. Possible impurities are paraffins, acetone, methanol, ethanol, propanol, isopropanol, *tert*-butanol, *sec*-butanol, di-*sec*-butyl ether, and

[1] This test method 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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[2] *Annual Book of ASTM Standards*, Vol 06.04.

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

[4] *Annual Book of ASTM Standards*, Vol 05.02.

[5] *Annual Book of ASTM Standards*, Vol 14.02..

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

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

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ethyl acetate. The peaks should be resolved, quantitatively in proportion to concentration, within a practical elapsed time. Columns that meet the requirements of this test method are listed in Table 1. Other columns may be used, provided the user establishes that a column gives the required separation and the precision requirements of Section 13 are met.

5.3 *Specimen Introduction System*—Any specimen system capable of introducing a representative specimen into the column may be used. Systems that have been used successfully to introduce 1 to 10-μL of methyl ethyl ketone specimens include microlitre syringes, micropipets, and liquid sampling valves.

5.4 *Recorder*—An electronic integrator or a recording potentiometer with a full-scale deflection of 5 mV or less, full-scale response time of 2 s or less, and sufficient sensitivity to meet the requirements of 5.1.

6. Reagents and Materials

6.1 *Carrier Gas*, appropriate to the type of detector used. Helium or hydrogen may be employed with thermal conductivity detectors, and nitrogen, helium, or argon with ionization detectors. The minimum purity of any carrier should be 99.95 mol %.

6.1.1 **Warning**—If hydrogen is used, take special safety precaution to ensure that the system is free of leaks and that the effluent is vented properly.

6.2 *Column Materials*:

6.2.1 *Liquid Phase*—The materials successfully used in cooperative work as liquid phases are listed in Table 1 (see Note 1).

6.2.2 *Solid Support*—The support for use in the packed column is usually (PTFE)-fluorocarbon, crushed firebrick, or diatomaceous earth. Table 1 lists conditions used successfully in cooperative work (see Note 1).

NOTE 1—See research report for additional information, available from ASTM International Headquarters. Request RR:D01–1107.

6.2.3 *Tubing Material*—Copper, stainless steel, nickel copper alloy, aluminum, and various plastic materials have been found to be satisfactory for column tubing. The material must be nonreactive with the substrate, sample, and carrier gas.

6.3 *Standards for Calibration and Identification*—Standard samples for all components present are needed for identification by retention time, and for calibration for quantitative measurements (Note 2).

NOTE 2—Mixtures of components may be used, provided there is no uncertainty as to the identity or concentration of compounds involved.

7. Preparation of Apparatus

7.1 *Column Preparation*—The method used to prepare the column is not critical provided that the finished column produces the required separation (Note 3). Partitioning liquids, supports, and loading levels used successfully in cooperative work are listed in Table 1. These may be obtained from most chromatography supply houses.

NOTE 3—A suitable method for column preparation is described in Test Method D 2593.

7.2 *Chromatograph*—Install the column in the chromatograph and establish the operating conditions required to give the desired separation. Relative component retention times, along with the typical retention time for methyl ethyl ketone are listed in Table 1. Allow sufficient time for the instrument to reach equilibrium as indicated by a stable recorder baseline.

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TABLE 1 Columns and Conditions Used Successfully in Cooperative Work

	Case I	Case II	Case III	Case IV	Case V	Case VI
Column:	packed	packed	packed	packed	packed	capillary
Liquid phase	polyethylene glycol 1500	polyethylene glycol 400	polyethylene glycol 300	polyethylene glycol 200	polyethylene glycol 1500	polytrifluoro-propylsiloxane
Liquid phase, weight %	10	28	20	20	20	1.2 μm film
Support type	TFE resin	Pink, diato-maceous earth	Pink, diato-maceous earth	White, diato-maceous earth	Pink, diato-maceous earth	none
Support mesh size	40/60	30/60	40/60	60/80	60/80	...
Length, ft (m)	12 (3.7)	18 (5.5)	10 (3.0)	10 (3.0)	20 (6.1)	32.8 (10.0)
Outside diameter, in. (mm)	0.25 (6.4)	0.25 (6.4)	0.25 (6.4)	0.125 (3.2)	0.25 (6.4)	0.028 (0.72)
Inside diameter, in. (mm)	0.21 (5.3)	0.21 (5.3)	0.21 (5.3)	0.085 (2.2)	0.21 (5.3)	0.021 (0.53)
Column temperature, °C	100	80	75	70	100	30
Carrier gas	helium	helium	helium	helium	helium	helium
Carrier flow rate, mL/min	60	80	35	60	50	3.7
Typical retention time, min methyl ethyl ketone	6.9	17.0	11.0	5.8	16.5	8.8
Relative retention time (methyl ethyl ketone = 1.00):						
Propyl ether	0.19	0.14	...	...	...	...
Octenes	0.54	0.21	...	0.34	...	0.65
sec-Butyl ether	0.78	0.45	...	0.47	1.21	...
Acetone	0.61	0.54	0.64	0.67	0.67	0.60
Ethyl acetate	0.81	...	0.82	0.78	0.85	0.78
Methyl ethyl ketone	1.00	1.00	1.00	1.00	1.00	1.00
tert-Butanol	1.00	1.12	1.27	1.71	1.03	0.39
Methanol	1.10	...	1.36	1.71	1.09	0.27
Isopropanol	1.20	1.32	...	2.03	2.12	0.34
Ethanol	1.30	...	1.73	2.21	1.27	0.31
sec-Butanol	2.12	2.26	2.73	3.50	1.94	0.53
n-Propanol	2.35	...	3.27	3.97	...	0.43