



Designation: D 3329 – 99

Standard Test Method for Purity of Methyl Isobutyl Ketone by Gas Chromatography^{1,2}

This standard is issued under the fixed designation D 3329; 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 reappraisal. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reappraisal.

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 isobutyl ketone (MIBK) by gas chromatography and in addition provides a means for measuring certain impurities such as methyl isobutyl carbinol which are of interest. Impurities such as water and acidity are measured by other appropriate ASTM procedures and the results are used to normalize the chromatographic value.

1.2 *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.*

1.3 For specific hazard information and guidance, see the supplier's Material Safety Data Sheet for the materials listed in this test method.

2. Referenced Documents

2.1 ASTM Standards:

- D 1364 Test Method for Water in Volatile Solvents (Karl Fischer Reagent Titration Method)³
- D 1613 Test Method for Acidity in Volatile Solvents and Chemical Intermediates Used in Paint, Varnish, Lacquer, and Related Products³
- D 2593 Test Method for Butadiene Purity and Hydrocarbon Impurities by Gas Chromatography⁴
- E 180 Practice for Determining the Precision of ASTM Methods for Analysis and Testing of Industrial Chemicals⁵
- E 260 Practice for Packed Column Gas Chromatography⁶

3. Summary of Test Method

3.1 A representative specimen is introduced onto a gas-liquid partition column. Methyl isobutyl ketone is separated

from impurities such as acetone, 2-propanol, pentanone, mesityl oxide, methyl isobutyl carbinol, and several unidentified compounds 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 concentrations are determined by relating the individual peak responses to the total peak response. Water and acidity are measured in accordance with Test Methods D 1364 and D 1613 and the results are used to normalize the values obtained by gas chromatography.

4. Significance and Use

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

5. Apparatus

5.1 *Chromatograph*—Any gas liquid chromatographic instrument having either a thermal conductivity or flame ionization detector provided the system has sufficient sensitivity and stability to obtain for 0.01 % 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 used in judging the sensitivity must be such that the column is not overloaded, which would result in peak broadening, loss of resolution, shifting retention times and formation of leading peaks.

5.2 *Column*—Any column capable of resolving methyl isobutyl ketone from the impurities that may be present. Possible impurities are hydrocarbons, acetone, 2-propanol, 2-pentanone, 3-pentanone, mesityl oxide, mesityl oxide isomer, and methyl isobutyl carbinol. Columns that meet this requirement are described in Table 1. Other columns, including capillary columns, may be used, provided the user establishes that a column gives the required separation and the peak shapes are satisfactory for measurement so that the precision requirements of Section 12 are met.

5.3 *Specimen Introduction System*—Any system capable of introducing a representative specimen onto the column. Microtitre syringes have been used successfully.

¹ This test method 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.

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² IUPAC approved name is 4-methyl-2-pentanone.

³ *Annual Book of ASTM Standards*, Vol 06.04.

⁴ *Annual Book of ASTM Standards*, Vol 05.01.

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

⁶ *Annual Book of ASTM Standards*, Vol 14.02.

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

TABLE 1 Columns and Conditions Used Successfully in Cooperative Work (See Note 2)

	Case I	Case II	Case III	Case IV	Case V	Case VI
<i>Column:</i>						
Liquid phase	polyethylene glycol, MW 1450	polyethylene glycol, MW 540	ethylene oxide/propylene oxide copolymer	polyethylene glycol, MW 20M	polyethylene glycol, MW 3350	polyethylene glycol
Liquid phase, weight %	15	20	20	25	25	1.2 μm film
Support	diatomaceous earth, acid washed	diatomaceous earth	diatomaceous earth, acid washed	diatomaceous earth	diatomaceous earth	capillary
Support mesh size	60 to 80	60 to 80	45 to 60	60 to 80	60 to 80	N/A
Length, ft (m)	20 (6.1)	18 (5.5)	20 (6.1)	10 (3.0)	12 (3.7)	33 (10)
Diameter, in. (mm)	1/8 (3.2)	1/4 (6.4)	1/4 (6.4)	1/8 (3.2)	1/4 (6.4)	0.021 (0.53)
Temperature, °C	90	100	160	75 to 200 programmed at 4°/min	145	40 to 180 at 5°/min
Carrier gas:	helium	helium	helium	helium	helium	helium
Flow rate, mL/min	30	50	60	20	80	5
Typical retention time, min:						
Methyl isobutyl ketone	9.3	27.2	8.1	11.2	7.7	1.84
Relative retention time, MIBK = 1.00:						
Acetone	0.42	0.39	0.52	0.42	0.39	0.36
2-propanol	0.66		0.57	0.65	0.56	0.59
2- and 3-pentanone	0.85	0.82	0.78	0.79
Methyl isobutyl ketone	1.00	1.00	1.00	1.00	1.00	1.00
α-Mesityl oxide	1.45	1.41	1.27
β-Mesityl oxide	1.89	1.85	1.63	1.24	1.52	...
Methyl isobutyl carbinol	2.11	2.07	1.48	1.42	1.95	2.24

5.4 *Recorder*—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 and stability to meet the requirements of 5.1.

6. Reagents and Materials

6.1 *Carrier Gas*—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 flame ionization detectors. The minimum purity of the carrier gas used should be 99.95 mol %.

NOTE 1—**Precaution:** If hydrogen is used, take special safety precautions to ensure that the system is free from leaks and that the effluent is vented properly.

6.2 Column Materials:

6.2.1 *Liquid Phase*—The materials successfully used in cooperative test work are listed in Table 1. (See Note 2.)

NOTE 2—Suppliers of liquid phases and supports can be found in Research Report RR:D01-1042, available from ASTM Headquarters.

6.2.2 *Solid Support*—The materials and their mesh sizes successfully used in cooperative work are shown in Table 1. (See Note 2.)

6.2.3 *Tubing Material*—Copper, stainless steel, and aluminum have been found satisfactory for column tubing. The tubing must be nonreactive with the substrate, sample, and carrier gas and be of uniform internal diameter.

6.3 *Standards for Calibration and Identification*—Standard samples of all components present are needed for identification by retention time and for calibration for quantitative measurements.

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.

NOTE 3—A suitable method for column preparation is described in Test Method D 2593. Additional useful information may be found in Practice E 260.

7.1.1 Prepacked columns conforming to requirements listed in Table 1 and in 5.2 and 6.2 may be obtained from any reputable chromatography supply company.

7.2 *Chromatograph*—Install the column in the chromatograph and establish the operating conditions required to give the desired separation. Allow sufficient time for the instrument to reach equilibrium as indicated by a stable recorder baseline. Control the detector temperature so that it is constant to within 1°C without thermostat cycling that causes an uneven baseline. Adjust the carrier-gas flow rate to a constant value.

8. Calibration and Standardization

8.1 *Identification*—Select the conditions of column temperature and carrier gas flow that will give the necessary resolution of the components. Determine the retention time of each component by injecting small amounts either separately or in known mixtures. Relative component retention times along with the typical retention time for methyl isobutyl ketone are given in Table 1.

8.2 *Standardization*—The area under each peak of the chromatogram is considered a quantitative measure of the corresponding compound. The relative area is proportional to concentration if the detector responds equally to all the sample components. When flame ionization detectors are used the response to different components is generally significantly different. Differences in detector response may be corrected by use of relative response factors obtained by injecting and measuring the response to pure (99 weight % minimum)