



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 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 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.