Designation: D7090 – 04 (Reapproved 2010)

Standard Test Method for Purity of Isophorone by Capillary Gas Chromatography[1]

This standard is issued under the fixed designation D7090; 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.

1. Scope

1.1 This test method covers the determination of the purity of isophorone. This method also determines the impurities of the material in concentration level less than 0.5 mass %, which may include mesityl oxide (MSO), mesityl oxide-isomer, mesitylene, trimethyl cyclohexenone (TMCH), phorone, phorone-isomer, xylitone, and tetralone.

1.2 Water cannot be determined by this test method and shall be measured by other appropriate ASTM procedure. The result is used to normalize the chromatographic data determined by this test method.

1.3 For purposes of determining conformance of an observed 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 E29.

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 and health practices and to determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D1364 Test Method for Water in Volatile Solvents (Karl Fischer Reagent Titration Method)

E29 Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications

E300 Practice for Sampling Industrial Chemicals

3. Summary of Test Method

3.1 A representative specimen is introduced into a gas chromatograph with a bonded polyethylene glycol capillary column using temperature programming and a flame ionization detector. The concentrations of the sample components are calculated from the integrated component peaks using internal standardization technique with response factors. Water is measured in accordance with Test Method D1364 and the result is used to normalize the values obtained by gas chromatography.

4. Significance and Use

4.1 This test method determines the purity of isophorone, as well as the concentration of various potential impurities, several of which are critical in the application of these solvents.

5. Apparatus

5.1 *Chromatograph*—Any gas chromatograph utilizing a capillary column and has the following characteristics (see Table 1 for typical GC parameters):

5.1.1 *Detector*—A flame ionization detector (FID) capable of continuous operation at a temperature equivalent to the maximum column temperature employed. The detector shall have sufficient sensitivity to detect 0.001 mass % of impurity in the specimen at a peak height 3 times the noise level.

5.1.2 *Column*—fused silica capillary column with bonded polyethylene (see Table 1 for details).

5.1.3 *Column Temperature Programming*—The chromatograph shall be capable of reproducible linear temperature programming.

5.1.4 *Sample Inlet System*—The sample inlet system shall be capable of split injection, typically at a 100:1 split ratio.

NOTE 1—An autoinjector is recommended. Manual injection with a syringe is acceptable, however the observed precision may not apply.

5.1.5 *Integrator*—Means shall be provided for determining the area of the observed chromatographic peaks. This can be done by means of an electronic integrator or a computer based chromatography data system. The integrator/computer system shall have standard chromatographic software for determining the retention times and quantification of eluting peaks.

5.1.6 *Flow Controller*—The chromatograph shall be equipped with a constant flow device capable of maintaining the carrier gas at a constant flow rate throughout the temperature program.

5.1.7 *Microsyringe*—A microsyringe of appropriate capacity is required for injection of the specimen into the chromatograph. Typically, a 5 µL syringe is used.

[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] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

TABLE 1 Typical GC Parameters

Parameters	Values
Column	30 m × 0.32 mm fused silica capillary column with 0.5 micron bonded phase polyethylene glycol
Column Temperature	50°C for 5 min., programmed to 210°C at 10°C/min. Hold for 10 min.
Injector Temperature	230°C
Sample size	1 µL
Split ratio	100:1
Detector	Flame Ionization
Detector Temperature	250°C
Carrier Gas (Helium)	30 cm/s
Hydrogen Gas	30 mL/min.
Air	300 mL/min.

6. Reagents and Materials

6.1 *Purity of Reagents*—Reagent grade chemicals shall be used in the preparation of the calibration mixture.

6.2 *Calibration Mixture Components:*

6.2.1 *Isophorone*—solvent used in the preparation of the calibration mixture, and shall be free of the components of interest. If pure isophorone is not available, then isophorone containing relatively low concentration of the components of interest can be used, and the composition of the calibration mixture corrected for components already present. (**Warning**—Isophorone is a cancer suspect agent.)

6.2.2 *Mesitylene Oxide (MSO), 4-methyl-3-pentene-2-one*—calibration component.

NOTE 2—This chemical is commercially available typically as a mixture of two isomers with an MSO to MSO-isomer ratio of 10:1.

6.2.3 *Mesitylene Oxide Isomer (MSO-isomer), 4-methyl-4-pentene-2-one*—calibration component.

6.2.4 *Mesitylene, 1,3,5-trimethyl benzene*—calibration component.

6.2.5 *Trimethyl cyclohexenone (TMCH), 3,5,5-trimethyl-3-cyclohexen-1-one*— calibration component.

6.2.6 *Phorone, 2,6-dimethyl-hepta-3,5-diene-2-one*— calibration component.

6.2.7 *Phorone - Isomer, 4-6-dimethyl-hepta-3,5-diene-2-one*— calibration component.

NOTE 3—This chemical is not commercially available but comes as an impurity of phorone.

6.2.8 *Tetralone, 3,4-dihydro-1-napthalenone* —calibration component.

6.2.9 *Xylitone(s)*—optional calibration component.

6.2.10 *Decane*—internal standard.

6.3 *Carrier Gases*—Helium or hydrogen (minimum 99.95 % purity). (**Warning**—Helium and hydrogen are compressed gases under high pressure. Hydrogen is highly flammable.)

7. Sampling

7.1 Take samples of the material to be tested using procedures described in Practice E300.

8. Conditioning of Capillary Column

8.1 Condition the gas chromatographic capillary column following the column supplier recommendation.

9. Calibration and Standardization

9.1 Prepare a calibration mixture containing approximately 0.1 mass % of each of the components of interest and the decane internal standard in pure isophorone. The total weight of the calibration mixture solution should be 100 g. If pure isophorone is not available, then isophorone containing relatively low concentration of the components of interest can be used, and the composition of the calibration mixture corrected for components already present. Typical components suitable for the calibration mixture are: MSO, MSO-isomer, mesitylene, TMCH, phorone, phorone isomer, xylitone, and tetralone (see 6.2).

9.2 Record the actual weight of each added component, the internal standard, and the total weight of the calibration mixture.

9.3 Determine the detector response factor of the various components of interest, by injecting 1 µL of the calibration mixture into a GC using the typical chromatographic parameters given in Table 1, and using the equation:

$$F_i = \frac{(W_i \times A_{is})}{(W_{is} \times A_i)} \quad (1)$$

where:

F_i = detector response factor for the component of interest,
W_i = weight of the component of interest in the calibration mixture, in grams,
W_{is} = weight of the internal standard in the calibration mixture, in grams,
A_i = peak area of the component of interest in the calibration mixture, and
A_{is} = peak area of the internal standard in the calibration mixture.

NOTE 4—Most chromatographic data systems are capable of determining the detector response factors automatically by inputting the weight or concentration of the components of interest and the internal standard.

10. Procedure

10.1 *Sample Preparation*—Tare an 8-oz bottle or suitable container with a cap. Using a syringe or an appropriate dispensing device, add approximately 0.1g (130 µL) of the internal standard (decane). Record the exact weight (W_{isx}) of the added internal standard. Add the material to be tested to give a total weight of the prepared sample of 100.0 ± 0.1 g. Record the exact weight of the prepared sample (W_{isx}). Cap the container, and mix the solution thoroughly.

TABLE 2 Typical Retention Times of Chromatographic Components

Component	Approximate Retention Times (min.)
Decane (Internal Standard)	8.03
MSO-isomer	9.96
MSO	11.67
Mesitylene	13.88
TMCH	16.57
Phorone	18.46
Phorone-isomer	18.62
Isophorone	19.83
Xylitone	20.86
Tetralone	28.35