



Designation: D7974 – 21

Standard Test Method for Determination of Farnesane, Saturated Hydrocarbons, and Hexahydrofarnesol Content of Synthesized Iso-Paraffins (SIP) Fuel for Blending with Jet Fuel by Gas Chromatography¹

This standard is issued under the fixed designation D7974; 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.

1. Scope*

1.1 This test method covers the determination of farnesane (2,6,10-trimethyldodecane), saturated hydrocarbons, and hexahydrofarnesol content in synthesized iso-paraffins (SIP) fuel for blending with jet fuel by gas chromatography.

1.2 Farnesane is determined from 96 % to 99.9 % by mass. Sum of saturated hydrocarbons including farnesane is determined from 97 % to 99.9 % by mass, and hexahydrofarnesol is determined from 0.02 % to 2.0 % by mass.

1.3 This test method does identify and quantify main impurities or group type of impurities but does not purport to identify all individual components that can be present in synthesized iso-paraffins (SIP) fuel for jet fuel blending.

1.4 This test method is inappropriate for impurities that boil at temperatures higher than 460 °C or for impurities that cause poor or no response in a flame ionization detector.

1.5 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.6 *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.*

1.7 *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.*

¹ This test method is under the jurisdiction of ASTM Committee D02 on Petroleum Products, Liquid Fuels, and Lubricants and is the direct responsibility of Subcommittee D02.04.0L on Gas Chromatography Methods.

Current edition approved Jan. 1, 2021. Published January 2021. Originally approved in 2015. Last previous edition approved in 2015 as D7974 – 15. DOI: 10.1520/D7974-21.

2. Referenced Documents

2.1 *ASTM Standards:*²

D4057 Practice for Manual Sampling of Petroleum and Petroleum Products

D4307 Practice for Preparation of Liquid Blends for Use as Analytical Standards

D7566 Specification for Aviation Turbine Fuel Containing Synthesized Hydrocarbons

E355 Practice for Gas Chromatography Terms and Relationships

E594 Practice for Testing Flame Ionization Detectors Used in Gas or Supercritical Fluid Chromatography

3. Terminology

3.1 *Definitions:*

3.1.1 This test method makes reference to many common gas chromatographic procedures, terms, and relationships. Detailed definitions can be found in Practices E355 and E594.

3.1.2 *saturated hydrocarbons, n*—paraffinic and naphthenic compounds.

4. Summary of Test Method

4.1 A representative aliquot of the synthesized iso-paraffins (SIP) fuel sample is introduced into a gas chromatograph equipped with a 5 % phenyl-methylpolysiloxane bonded phase capillary column. Helium or hydrogen carrier gas transports the vaporized aliquot through the column where the components are separated by the chromatographic process. Components are sensed by a flame ionization detector as they elute from the column. The detector signal is processed by an electronic data acquisition system. The farnesane and its impurities are identified by comparing their relative retention times to the ones reported in the method. Identification has been previously performed analyzing reference samples by

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.

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

mass spectrometry under identical conditions. The concentrations of all components are determined in mass percent area by normalization of the peak areas.

5. Significance and Use

5.1 Synthesized iso-paraffins (SIP) fuel are being approved for blending with jet fuel provided that they meet a purity specification of more than 97 % farnesane, more than 98 % saturated hydrocarbons, and less than 1.5 % hexahydrofarnesol in accordance with Specification **D7566**. This test method provides a method of determining the percentage of farnesane (purity) in the synthesized iso-paraffins (SIP) fuel for blending with jet fuel.

6. Apparatus

6.1 *Gas Chromatograph*, capable of operating at the conditions listed in **Table 1**. A heated flash vaporizing injector designed to provide a linear sample split injection (for example, 100:1) is required for proper sample introduction. Carrier gas controls shall be of adequate precision to provide reproducible column flows and split ratios in order to maintain analytical integrity. A hydrogen flame ionization detector with associated gas controls and electronics, designed for optimum response with open tubular columns, is required.

6.2 *Sample Introduction*—Manual or automatic liquid syringe sample injection to the splitting injector is employed. Devices capable of 1.0 μL injections are suitable.

6.3 *Column*—This test method utilizes a fused silica open tubular column with non-polar 5 % phenyl-methylpolysiloxane bonded (cross-linked) phase internal coating.

6.3.1 Open tubular column with a non-polar 5 % phenyl-methylpolysiloxane bonded (cross-linked) phase internal coating; a 30 m long by 0.25 mm internal diameter column with a 0.25 μm film thickness has been found to be suitable.

6.4 *Electronic Data Acquisition System*—Any data acquisition and integration device used for quantification of these analyses shall meet or exceed these minimum requirements:

- 6.4.1 Normalized percent calculation based on peak area,
- 6.4.2 Identification of individual components based on retention time,
- 6.4.3 Noise and spike rejection capability,
- 6.4.4 Sampling rate of 5 Hz.

7. Reagents and Materials

7.1 *Carrier Gas*, Helium or hydrogen, with a minimum purity of 99.9 % mol. Oxygen removal systems and gas purifiers should be used. (**Warning**—Helium, compressed gas under high pressure.) (**Warning**—Hydrogen, extremely flammable gas under high pressure.) The use of hydrogen sensors in the oven is strongly recommended that can shutoff the hydrogen source in case the hydrogen leaks.

7.2 *Detector Gases*, Hydrogen, air, nitrogen, and helium. The minimum purity of the gases used should be 99.9 % mol for the hydrogen, nitrogen, and helium. The air should be hydrocarbon-free grade with a minimum purity of 99.0 % mol. Gas purifiers are recommended for the detector gases. (**Warning**—Hydrogen, extremely flammable gas under high pressure.) (**Warning**—Air and helium, compressed gases under high pressure.)

7.3 *Blank of Solvent*, n-Hexane with a minimum purity of 99.0 %.

7.4 *Validation Mixture for Apparatus Performance Checks*:

7.4.1 *Solvent*, n-Octane with a minimum purity of 99.0 %.

7.4.2 *Standards*, n-decane, n-dodecane, n-tetradecane, n-hexadecane with a minimum purity of 99.0 %.

8. Sampling

8.1 See Practice **D4057** for general sampling. Transfer an aliquot of synthesized iso-paraffins (SIP) fuel sample into a septum vial and seal. Obtain the test sample for analysis directly from the sealed septum vial, for either manual or automatic syringe injection.

9. Preparation of Apparatus

9.1 Install and condition column in accordance with manufacturer's or supplier's instructions. After conditioning, attach column outlet to flame ionization detector inlet and check for leaks throughout the system. When leaks are found, tighten or replace fittings before proceeding.

9.1.1 When using hydrogen as carrier gas, column conditioning shall be performed after connecting the column to the detector.

9.2 Adjust the operating conditions of the gas chromatograph (**Table 1**) and allow the system to equilibrate.

9.3 *Performance Checks*—The apparatus shall be checked in regular intervals to make sure that it is in perfect working condition. Performance checks are realized by analyzing a calibration mixture in the analytical conditions defined in **Table 1**.

9.3.1 *Linearity Verification*—Verify linearity by analyzing a series of diluted standards or equivalent spread across the farnesane content range of the method in order to have at the minimums 15 degrees of freedom. Standard deviation of the

TABLE 1 Typical Operating Conditions

Column Temperature Program	
Column length	30 m
Column inner diameter	0.25 mm
Film thickness	0.25 μm
Initial temperature	50 °C
Initial hold time	0 min
Program rate	3 °C/min
Final temperature	270 °C
Final hold time	20 min
Injector	
Temperature	300 °C
Split ratio	100:1
Sample size	1.0 μL
Detector	
Type	Flame ionization
Temperature	300 °C
Fuel gas	Hydrogen (\approx 40 mL/min)
Oxidizing gas	Air (\approx 400 mL/min)
Make-up gases	Helium (\approx 45 mL/min) or Nitrogen (\approx 45 mL/min)
Carrier Gas	
Type	Helium or Hydrogen
Flow rate	1 mL/min (Constant)

residuals shall be less than 0.5 % which corresponds to a 95 % confidence interval of 1 %. Annex A1 gives an example of how linearity may be confirmed.

9.3.2 *Calibration Mixture Preparation*—Weigh the same amount of n-decane, n-dodecane, n-tetradecane, and n-hexadecane. Dilute the mixture in n-octane in order to obtain a concentration of 2 % mass of each component. See Practice D4307.

9.3.3 *Column Efficiency*—Calculate the number of theoretical plates on n-tetradecane peak using Eq 1:

$$n = 5.545(Rt / W_{0.5})^2 \quad (1)$$

where:

- n = the number of theoretical plates,
- Rt = the retention time of n-tetradecane, and
- $W_{0.5}$ = the peak width at half maximum of n-tetradecane, expressed in the same unit as the retention time.

9.3.3.1 The determined Number of theoretical plates shall be >500 000.

9.3.4 *Apparatus Performance Evaluation*—Percent mass of each component of the calibration mixture is determined from peak area percentages. Relative error percentage is determined from the known concentrations of the mixture using Eq 2:

$$\text{percent relative error} = \frac{100(|\text{calculated concentration} - \text{known concentration}|)}{\text{known concentration}} \quad (2)$$

9.3.4.1 Relative error shall be ≤ 5 %.

9.3.5 *Minimum Detectable Level (MDL)*—Minimum detectable level is determined by injecting the validation mixture preparation diluted in n-octane in order to obtain a concentration of 10 mg/kg of each component. The signal/noise ratio of each component shall be higher than 3. (S/N ratio = S (signal peak height)/N (noise in peak to peak)).

10. Gas Chromatographic Analysis Procedure

10.1 Set the instrument operating variables. See Table 1 for typical operating conditions.

10.2 Inject 1.0 μL of sample into the injection port and start the analysis. Obtain a chromatogram and peak integration report. Sample chromatograms are shown in Figs. 1-6.

10.3 A blank of solvent (for example n-hexane) shall be injected in between samples to remove memory effects.

11. Identification

11.1 Before execution of the final calculations, check the chromatogram for correct peak identification by comparison with those reported in Table 2 (see also Figs. 1-6, the reference chromatogram associated to those data).

12. Calculation

12.1 Determine the relative mass percent of the individual components by using the following Eq 3:

$$RM_i = \frac{A_i}{A_t} \times 100 \quad (3)$$

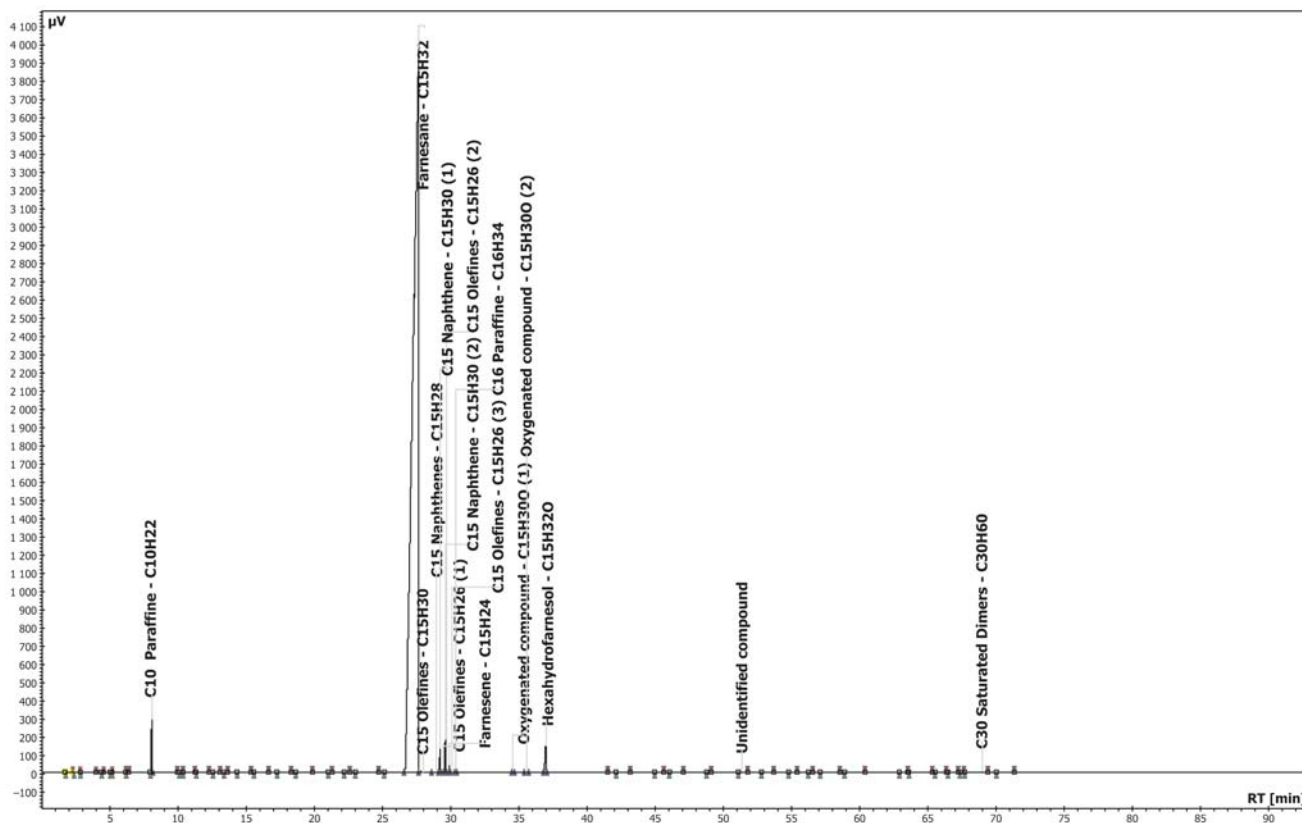


FIG. 1 Chromatogram of a Reference Distilled Farnesane