

Designation: D6064 – 11 (Reapproved 2022)

Standard Specification for HFC-227ea, 1,1,1,2,3,3,3-Heptafluoropropane $(CF_3CHFCF_3)^1$

This standard is issued under the fixed designation D6064; 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 specification covers requirements for HFC-227ea as a fire-fighting medium.

1.2 This specification does not address the fire-fighting equipment or hardware that employs HFC-227ea or the conditions of employing such equipment (for example, handhelds, fixed installations, etc.).

1.3 This specification does not address the storage or transportation of HFC-227ea. Storage, handling, and transportation issues may be addressed in future ASTM specifications.

1.4 The values stated in both inch-pound and SI units are to be regarded separately as the standard. The values given in parentheses are for information only.

1.5 The following safety hazards caveat pertains only to the test methods portion, Section 5, of this specification: *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.* Specific hazards statements are given in Note 1.

1.6 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.

2. Referenced Documents

- 2.1 ISO Standards:²
- ISO 3363 Fluorochlorinated Hydrocarbons for Industrial Use-Determination of Acidity-Titrimetric Method
- ISO 3427 Gaseous Halogenated Hydrocarbons (Liquefied Gases)–Taking of a Sample

ISO 5789 Fluorinated Hydrocarbons for Industrial Use-Determination of Nonvolatile Residue

2.2 ASRE Standard: ASRE Standard 34³

3. Terminology

3.1 Definitions of Terms Specific to This Standard:

3.1.1 *HFC*—hydrofluorocarbon; a chemical compound in which the compound molecule is comprised exclusively of hydrogen and fluorine and carbon atoms.

3.1.2 *HFC-227ea*—the compound 1,1,1,2,3,3,3-heptafluoropropane; CF₃CHFCF₃.

3.1.2.1 Discussion—The terminology system for fluorinecontaining compounds (described in detail in ASRE Standard 34) provides a convenient means to reference the structure of individual compounds. By definition, the first digit of the numbering system represents one less than the number of carbon atoms in the compound molecule; the second digit, one more than the number of hydrogen atoms in the compound molecule; and the third digit, the number of fluorine atoms in the compound molecule. For molecules containing three carbon atoms, two appended letters are added to indicate the symmetry of the molecule. The first appended letter indicates the substitution on the central (C2) carbon; for example, the substitution CHF is assigned the designation "e." The second appended letter indicates the substitution at the C1 and C3 carbons; for example, identical substitution on the C1 and C3 carbons are assigned the designation "a." For example, the designation HFC-227ea indicates three carbon atoms (2 + 1), one hydrogen atom (2-1), and seven fluorine atoms; the designation "e" indicates that the central carbon is substituted as CHF, and the designation" a" indicates that the substitution on Carbons C1 and C3 is identical, that is, the structure is CF₃CHFCF₃.

4. Material Requirements

4.1 Type I-Mixtures of HFC-227ea and Nitrogen:

4.1.1 The nitrogen (N_2) partial pressure shall be such that the safe working pressure of the receiving vessel is not exceeded. To prevent excessive pressure, the fill density of

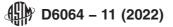
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¹This specification is under the jurisdiction of ASTM Committee D26 on Halogenated Organic Solvents and Fire Extinguishing Agents and is the direct responsibility of Subcommittee D26.09 on Fire Extinguishing Agents.

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² Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036, http://www.ansi.org.

³ American Society of Refrigeration Engineers, *Refrigeration Engineering 65*, 1957, p. 49.



HFC-227ea within a container should not exceed that needed to achieve complete filling of the container at the maximum envisaged storage temperature. For example, for the U.S. DOT 4BA500 cylinder, the nitrogen partial pressure shall not exceed 21.8 bar at 21 °C (316 psig at 70 °F) for a 1150 kg/m³ (72 lb/ft³) fill density (yielding a total pressure of 25.8 bar at 21 °C (360 psig at 70 °F). For this example, the safe working pressure of the 4BA500 cylinder is not exceeded for temperatures below 54 °C (130 °F).

4.1.2 HFC-227ea shall conform to the requirements prescribed in Table 1 when tested by the appropriate test method(s) listed in Section 6.

4.1.3 When a material analysis is required, by agreement between the purchaser and the supplier, the total pressure in the HFC-227ea container, partial pressure of the N₂, the fill density of HFC-227ea within the container, and the maximum safe storage temperature shall be part of the material analysis (certification). The pressure shall be reported in bar (preferred) or pound-force per square inch gage. The fill density shall be reported in kilograms per cubic metre at 21 °C (preferred) or pounds per cubic foot at 70 °F. The maximum safe storage temperature of the HFC-227ea container shall be reported in degrees Celsius (preferred) or in degrees Fahrenheit and shall conform to applicable regulations for the HFC-227ea container design and use.

4.2 *Type II—HFC-227ea*—HFC-227ea shall conform to the requirements of Type I, as listed in 3.1, and shall contain no more than 1.5 % by volume fixed gases in vapor phase, expressed as air when tested by the appropriate test method(s) listed in Section 6.

4.3 By agreement between the purchaser and the supplier, analysis may be required and limits established for elements or compounds not specified in Table 1.

4.4 Unless otherwise specified, Type II is assumed.

Note 1—Prolonged exposure to concentrations of HFC-227ea in excess of 10.5 % by volume in air during periods of elevated adrenaline could produce cardiac arrhythmia in some personnel.

5. Sampling

5.1 Samples of HFC-227ea, taken from the liquid phase, shall be taken from filled containers in accordance with the method specified in ISO 3427. The sampling cylinder shall be capable of safely resisting the vapor pressure of the sample at the highest temperature that could be encountered.

5.2 The HFC-227ea selected in accordance with 5.1 shall be tested for quality conformance in accordance with Section 6. The presence of one or more defects shall be cause for rejection.

TABLE	1	Requirements
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Property	Requirement
HFC-227ea purity	99.0 %, mol/mol, min
Acidity	(exclusive of any N ₂ present)
	2.0 ppm by mass, as HCL, max
Water content ppm by mass, max	10 ppm by mass, max
Nonvolatile residue	0.05 g/100 mL, max
Halogen ion	passes test
Suspended matter or sediment	none visible

6. Test Methods

6.1 Purity:

6.1.1 Determine the purity by gas-liquid chromatography in accordance with the technique described in 6.1.2 - 6.1.5 or another acceptable laboratory technique providing equivalent results.

6.1.2 *Apparatus*—The following special apparatus is required to determine the percent of HFC-227ea:

6.1.2.1 *Gas Chromatograph*, capable of programmed temperature operation and equipped with a thermal conductivity detector.

6.1.2.2 *Column*, 3.1 m by 5 mm outside diameter (2.6 mm inner diameter) glass tubing, packed with 80 mesh to 120 mesh Carbopack B^4 or equivalent.

6.1.2.3 *Gas Sampling Valve*, 10 mL volume or a volume sufficient to achieve proper separation in the specified column.

6.1.2.4 Glass Syringe, 20 mL Hamilton B-D,⁵ or equivalent.

6.1.2.5 *Three-Way Purge/Isolation Valve*, Hamilton 86727 miniature inert valve with Luer Lock fittings,⁵ or equivalent.

6.1.3 *Reagents*—The carrier gas shall be a chromatographic grade of helium. The column packing shall consist of a standard solution, for example 3 % (weight/weight) methyl silicone,⁵ on 80 mesh to 120 mesh Carbopack B⁵ (or equivalent).

6.1.4 Procedure:

6.1.4.1 Install the column and adjust the temperature of the column oven to 30 °C, injection port to 100 °C, and detector block to 150 °C. The temperature should be programmed to rise 10 °C/min to 15 °C/min (from an initial temperature of 30 °C), to a maximum of 100 °C.

6.1.4.2 Adjust the helium flow to 25 mL/min.

6.1.4.3 Adjust the detector voltage to 8 V or to the midrange of the thermal conductivity detector (TCD) instrument being used and allow the instrument to stabilize.

6.1.4.4 Take the sample from the vapor phase; collect approximately 20 mL in the glass syringe.

6.1.4.5 Purge the sample loop with approximately 10 mL of sample from the syringe and transfer the sample into the chromatographic system.

6.1.4.6 Allow the sample to elute, for approximately 18 min, attenuating as necessary to make the peak heights a convenient size. Under proper instrument settings, the HFC-227ea should elute after approximately 5 min.

6.1.5 Calculation:

6.1.5.1 Calculate percent HFC-227ea as follows:

$$\% HFC - 227 \text{ea} = \frac{A(CF_3 CHFCF_3) \times 100}{As} \tag{1}$$

where:

 $A(CF_3CHFCF_3)$ = area of the HFC-227ea peak, and As = sum of the area of all peaks, excluding the nitrogen peak.

Percent HFC-227ea below that specified in Table 1 shall constitute failure of this test method.

⁴ Available from Alltech, 2051 Waukegan Road, Deerfield, IL 60015.

⁵ Available from Hamilton Co., P.O. Box 10030, Reno, NV 89520-0012.

6.1.5.2 Calculate percent nitrogen as follows:

$$\%N_2 = \frac{An \times 100}{As} \tag{2}$$

where:

An = area of nitrogen peak, and

As = sum of the area of all other peaks, including the nitrogen peak.

It is useful to calculate percent nitrogen in order to judge a safe fill density.

6.2 Acidity—Vaporize a large sample, in the presence of distilled water. Determine the acidity of the solution by the appropriate method described in ISO 3363, 6.2.1 of this standard, 6.2.2 of this standard, a pH indicator, or another acceptable laboratory technique providing equivalent results.

6.2.1 Acidity by Sodium Hydroxide Titration:

6.2.1.1 Reagents:

6.2.1.2 *Sodium Hydroxide*, 0.01 *N* solution, standardized with reagent grade potassium hydrogen phthalate, or standardized by the supplier.

6.2.1.3 Methyl Red Indicator, 0.1 % aqueous solution.

6.2.1.4 *Procedure*—Fill a suitable gas sampling cylinder with liquid HFC-227ea, and weigh the cylinder. Place 50 mL of a crushed ice-distilled water slurry in a 250 mL stoppered Erlenmeyer flask, slowly add the HFC-227ea under the slurry surface, and then reweigh the sample cylinder. Place the stopper in the flask loosely and swirl the flask gently from time to time until the ice has melted completely. Add one drop of methyl red indicator, and if a reddish color remains, titrate to a yellow endpoint with 0.01 N sodium hydroxide solution. Run a crushed ice-distilled water blank (with no HFC-227ea) along with the sample.

6.2.1.5 *Calculation*—Calculate parts per million hydrogen chloride as follows:

HCl, ppm = $(A - B) \times N \times 0.03645 \times 10^6$ weight of sample, g (3)

where:

B = volume of NaOH titrated in the blank, mL,

N =normality of NaOH,

W = weight of HFC-227ea, g, and

where:

 0.03645×10^6 = factor to express result as ppm HCl (hydrogen chloride).

Acidity in excess of the amount specified in Table 1 shall constitute failure of this test.

6.2.2 Acidity by Universal Indicator:

6.2.2.1 Apparatus.

6.2.2.2 *Fritted Glass Sparger*, of coarse porosity, contained in a 100 mL glass scrubbing bottle provided with inlet and outlet tubes.

6.2.2.3 Neoprene Connecting Tubing.

6.2.2.4 Wet Test Meter, 0.1 ft³ revolution.

6.2.2.5 Needle Valve Control.⁶

6.2.2.6 *Reagent Universal Indicator*, ⁷ with color chart, or equivalent.

6.2.2.7 Procedure-Prepare neutralized distilled water by adding 0.4 mL of universal indicator solution to 100 mL of deionized water, and titrate with 0.01 N sodium hydroxide until the water shows a pH of 7.0 when compared to the Universal Color Chart. Add 50 mL of the neutralized water to the glass scrubbing bottle fitted with the glass gas sparger. Attach a needle valve control to the sample cylinder, and connect the cylinder, inverted, to an empty safety trap. Connect the safety trap outlet to the scrubbing bottle inlet. Connect the scrubbing bottle outlet to the inlet of the wet test meter. Open the needle valve slowly and pass 20 L of sample through the scrubber at a flow rate of approximately 500 mL/min. Turn off the needle valve and disconnect the sample cylinder from the scrubbing bottle. Transfer 10 mL to 12 mL of water solution to a clean test tube. Add 0.3 mL of universal indicator solution and swirl. Read the pH of the solution by comparison with the universal color chart. Report the pH reading. No observable change in pH indicates an acidity of less than 3.0 ppm.

6.3 *Water Content*—Test HFC-227ea for water content. The analysis may be conducted by the phosphorus pentoxide method, infrared absorption, electrolytic moisture analysis, piezoelectric analyzer, or another acceptable laboratory technique. The accuracy of the results and the standard method should be by orthodox Karl Fischer method. Water content greater than specified in Table 1 shall constitute failure of this test.

6.4 *Qualitative Test for Fluoride Ion*—Test a sample for the presence of fluoride ions in accordance with 6.4.1 through 6.4.3 or by another acceptable laboratory technique providing equivalent results. Generally, a sample treated with a saturated aqueous solution of calcium chloride shall exhibit no turbidity or precipitation of calcium fluoride.

-6.4.1 Apparatus: 265271241/astm-d6064-112022

6.4.1.1 *Fritted Glass Gas Sparger*, of coarse or A porosity, contained in a 100-mL glass scrubbing bottle provided with inlet and outlet tubes.

6.4.1.2 Neoprene Tubing.

6.4.1.3 Wet Test Meter, 0 to 1 ft³/revolution.

6.4.1.4 Needle Valve Control. 7

6.4.2 *Reagents:*

6.4.2.1 Calcium Chloride, saturated solution in water.

6.4.3 *Procedure*—Add 10 mL of saturated calcium chloride solution to the scrubber assembly. Attach a needle valve control to the sample cylinder. Connect the sample cylinder in the upright position to an empty safety trap with neoprene tubing. Connect the outlet of the safety trap to the inlet of the scrubbing bottle assembly with neoprene tubing. Connect the outlet of the scrubbing bottle assembly to the inlet of the wet test meter. Open the needle valve slowly and pass approximately 2 L of sample through the scrubber at a flow rate of approximately 100 mL/min. Turn off the needle valve and disconnect the scrubber assembly from the sample cylinder and the wet test meter. Examine the contents of the scrubber

A = volume of NaOH titrated in sample, mL,

⁶ Available from Matheson Co., 430-T Caredean Road, Horsham, PA 19044.

⁷ Available from Fisher Scientific Co., Mid-Atlantic Region, 585 A Drive, Pittsburgh, PA 15238.