Designation: D6064 − 11 (Reapproved 2015)

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 and health practices and determine the applicability of regulatory limitations prior to use.*Specific hazards statements are given in Note 1.

2. Referenced Documents

2.1 *ISO Standards:*

ISO 3363 Fluorochlorinated Hydrocarbons for Industrial Use-Determination of Acidity-Titrimetric Method[2]

ISO 3427 Gaseous Halogenated Hydrocarbons (Liquefied Gases)–Taking of a Sample[2]

ISO 5789 Fluorinated Hydrocarbons for Industrial Use-Determination of Nonvolatile Residue[2]

2.2 *ASRE Standard:*

ASRE Standard 34[3]

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; CF_3CHFCF_3.

3.1.2.1 *Discussion*—The terminology system for fluorine-containing 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 CF_3CHFCF_3.

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 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/m^3 (72-lb/ft^3) 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).

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[1] This 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.

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 N_2, 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.

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.

TABLE 1 Requirements

Property	Requirement
HFC-227ea purity	99.0 %, mol/mol, min (exclusive of any N_2 present)
Acidity	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

6.1.2 *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 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 to 120-mesh Carbopack B[5] (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 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 mid-range 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:

$$\%HFC - 227\text{ea} = \frac{A(CF_3CHFCF_3) \times 100}{As} \tag{1}$$

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.

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.

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

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

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:

$$HCl, \text{ppm} = \left(A - B\right) \times N \times 0.03645 \times 10^6 \; \textit{weight of sample, g} \quad (3)$$

where:

A = volume of NaOH titrated in sample, mL,
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 ft^3 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 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:*

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 ft^3/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 visually for the presence of turbidity. Report the fluoride present if any turbidity develops. The appearance of any turbidity shall constitute failure of this test.

6.5 *Suspended Matter and Sediment*—Examine visually for any suspended matter or sediment. Observation of any suspended matter or sediment shall constitute failure of this test.

6.6 *Nonvolatile Residue*—Determine the nonvolatile residue in accordance with the method specified in ISO 5789, in

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[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.

accordance with 5.6.1 through 5.6.8 or another accepted laboratory technique providing equivalent results.

6.6.1 *Gravimetric Determination of Nonvolatile Residue:*

6.6.1.1 *Apparatus:*

6.6.1.2 *Analytical Balance,* 0.0001-g resolution.

6.6.1.3 *Crystallizing Dish,* 250 mL.

6.6.1.4 *Crucible Tongs.*

6.6.1.5 *Gas Sampling Cylinder.*

6.6.1.6 *Graduated Cylinder,* 100 mL.

6.6.1.7 *Drying Oven,* 105°C.

6.6.1.8 *Dessicator,* containing porcelain plate and activated drying agent.

6.6.2 *Reagents:*

6.6.2.1 *Isopropanol,* technical grade.

6.6.3 *Procedure:*

6.6.3.1 Place a clean crystallizing dish into the 105°C oven for 15 min. Remove the dish with the tongs and place in the dessicator for cooling (approximately 20 min). Weigh the dish to the nearest 0.0001 g and record the weight. Slowly add HFC-227ea from the sample cylinder to the graduated cylinder, obtaining 100 mL of sample. Wash down the sides of the graduated cylinder with isopropanol to remove condensation if necessary. Transfer the sample as quickly as possible to the weighted dish. Allow the sample to evaporate to dryness in a hood. Allow the dish to equilibrate to room temperature, reweigh, and record the weight to the nearest 0.0001 g.

6.6.3.2 *Calculation*—The difference in weight is reported as the nonvolatile residue in grams per 100 mL of sample.

6.6.4 *Gravimetric Determination of Nonvolatile Residue—Alternate Procedure:*

6.6.4.1 *Apparatus:*

6.6.4.2 *Analytical Balance,* 0.0001-g resolution.

6.6.4.3 *Crystallizing Dish,* 250 mL.

6.6.4.4 *Crucible Tongs.*

6.6.4.5 *Gas Sampling Cylinder.*

6.6.4.6 *Drying Oven,* 105°C.

6.6.4.7 *Dessicator,* containing porcelain plate and activated drying agent.

6.6.5 *Reagents:*

6.6.5.1 *Isopropanol,* technical grade.

6.6.6 *Procedure:*

6.6.6.1 Place a clean crystallizing dish into the 105°C oven for 15 min. Remove the dish with the tongs and place in the dessicator for cooling (approximately 20 min). Weigh the dish to the nearest 0.0001 g and record the weight. Fill a suitable gas sampling cylinder with liquid HFC-227ea and weigh the cylinder. Slowly add HFC-227ea from the sample cylinder to the weighed dish, and then reweigh the sample cylinder. Allow the sample to evaporate to dryness in a hood. Allow the dish to equilibrate to room temperature, reweigh, and record the weight to the nearest 0.0001 g.

6.6.6.2 *Calculation*—Calculate grams of nonvolatile residue per 100 mL of sample as follows:

$$\text{nonvolatile residue (grams per 100 m}L\text{)} = (\Delta W/M) \times 1.41 \times 100 \quad (4)$$

where:

ΔW = difference in weight of weighing dish, g,
M = weight of HFC-227ea sample, g,
1.41 = density of HFC-227ea, g/mL, and
100 = factor to convert result to grams per 100 mL of sample.

6.7 *Fixed Gases in the Vapor Phase*—Test HFC-227ea for air in the vapor phase by the determination of gas not absorbable in perchloroethylene, by gas chromatography, or another accepted laboratory technique providing equivalent results.

6.7.1 *Perchloroethylene Absorption*—HFC-227ea may be tested for air in the vapor phase of the container by the determination of gases not absorbable in perchloroethylene using water as a sealant. The test may be conducted by the determination of gases not absorbable in perchloroethylene using mercury as a sealant or by the determination of gases remaining when the extinguishant is frozen. The determination of gases not absorbable in perchloroethylene using water as a sealant shall be the standard.

6.7.2 *Apparatus:*

6.7.2.1 *Absorption Vessel,* Lab Glass Co., No. LG-11119[8] or equivalent.

6.7.2.2 *Leveling Bulb and Connecting Tubing.*

6.7.2.3 *Perchloroethylene Reagent*—Specific gravity at 590/399°F (204°C), 1.628 to 1.632; no free halogens; 0.001 maximum free acid as HCl; 0.0007 maximum residue on evaporation; distill completely between 248 and 252°F (120 and 122°C) at 760 mm Hg; 31°F (– 0.5°C) maximum cloud point.

6.7.2.4 *Silicone Solution*—One part Organo Silane Ester A-1230 or Amino Silane A-1106,[9] or equivalent, in 100 parts of water.

6.7.3 *Procedure*—Wash the absorption vessel thoroughly and rinse in distilled water. Coat the inside with the silicone solution, let stand 5 min, and discard the solution. Rinse again with distilled water and dry at 212°F (100°C) for 10 min. When cool, add 20 mL of distilled water and fill with perchloroethylene. Add sufficient water to the leveling bulb to give a water seal. Collect a 100-m^3 sample from the vapor space of the HFC-227ea cylinder directly into the absorption vessel. A slight positive pressure is needed to prevent entry of extraneous air. Tilt the vessel in such a way as to remove all the water lock that separates the perchloroethylene from the gas. Allow the perchloroethylene to flow into the upper chamber of the vessel and gently agitate to aid in the absorption of the gas. Return the vessel to an upright position and allow the water layer to flow back into the burette section. Adjust the leveling bulb so that the liquid layers are at the same level. Read percent nonabsorbable gas from the burette.

6.7.3.1 *Calculation*—Make a blank run with a sample from the liquid phase of the cylinder and subtract the result from the analysis of the vapor phase. The presence of air in the vapor phase in excess of 1.5 % by volume shall constitute failure of this test.

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[8] Available from Lab Glass Co., P.O. Box 610, Dept. G., Vineland, NJ 08360.

[9] Available from QSI Specialties, Inc., P.O. Box 38002, South Charleston, WV 25303-3802.

6.7.4 *Gas Chromatography*—HFC-227ea may be tested for the concentration of air in the vapor phase by gas chromatography. A concentration of air in excess of 1.5 % by volume shall constitute failure of this test.

7. Container, Packaging, and Package Marking

7.1 Industrial or commercial quantities shall be packaged and labeled in accordance with DOT regulations as found in 40 CFR 100 to 199 and state and local regulations.

7.2 Department of Defense procurements shall be packaged and labeled in accordance with the applicable paragraphs of Fed. Spec. PPP-C-2020.

8. Keywords

8.1 CF_3CHFCF_3; fire fighting; fire-fighting agent; fire protection; fire suppressant; fluorinated hydrocarbon; FM-200®; heptafluoropropane; HFC; HFC-227ea; hydrofluorocarbon

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