Designation: D 6541 – 00

Standard Specification for HFC-236fa, 1,1,1,3,3,3–Hexafluoropropane, ($CF_3CH_2CF_3$)[1]

This standard is issued under the fixed designation D 6541; 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 the requirements for HFC-236fa as a fire-fighting medium.

1.2 This specification does not address the fire-fighting equipment or hardware that employs HFC-236fa or the conditions of employing such equipment (for example, hand-helds, fixed installations, and so forth).

1.3 This specification does not address the storage or transportation of HFC-236fa. Storage, handling, and transportation issues are addressed in Practice D 6427.

1.4 The following safety hazards caveat pertains to the test methods portion, Section 6.1, 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 hazard statements are given in Note 2.

2. Referenced Documents

2.1 *ASTM Standards:*

D 6427 Practice for Handling, Transportation, and Storage of HFC-236fa, 1,1,1,3,3,3–Hexafluoropropane ($CF_3CH_2CF_3$)[2]

2.2 *ISO Standards:*

ISO 3363 Fluorinated Hydrocarbons for Industrial Use—Determination of Acidity-Titration Method[3]

ISO 3427 Gaseous Halogenated Hydrocarbons (Liquefied Gases)—Taking a Sample[3]

ISO 5789 Fluorinated Hydrocarbons for Industrial Use—Determination of Nonvolatile Residue[3]

2.3 *CGA Standards:*

No. C-4 American National Standard Method of Marking Portable Compressed Gas Containers to Identify the Material Contained[4]

No. P-1 Safe Handling of Compressed Gases in Containers[4]

2.4 *U.S. Government Standards:*

Code of Federal Regulations (CFR) Title 49, Part 172.101 Tables of Hazardous Materials and Special Provisions[5]

Code of Federal Regulations (CFR) Title 49, Part 173.302 and 173.304 Preparation and Packaging of Gases[5]

Code of Federal Regulations (CFR) Title 49, Part 172 Sub D Marking Requirements of Packaging for Transportation[5]

2.5 *American Society of Refrigeration Engineers Standard:*

ASRE Standard 34, Designation of Refrigerants[6]

3. Terminology

3.1 *Definitions of Terms Specific to This Standard*:

3.1.1 *halogenated hydrocarbon (see Note 1)*—saturated hydrocarbons in which one or more of the hydrogen atoms have been replaced by atoms of the halogen series (fluorine, chlorine, bromine, and iodine). It is convention to prefix the number with an abbreviation of the compound:

CFC	=	chlorofluorocarbon
HCFC	=	hydrochlorofluorocarbon
HFC	=	hydrofluorocarbon
FC	=	fluorocarbon
R	=	refrigerant

NOTE 1—The halogenated compound coding terminology system provides a convenient means to reference halogenated hydrocarbons (see ASRE 34).

3.1.1.1 By definition, the right-most digit of the numbering system is the number of fluorine atoms.

3.1.1.2 The second digit from the right is the number of hydrogen atoms plus one (+1).

3.1.1.3 The third digit from the right is one less (-1) than the number of carbon atoms in the compound (when this number is zero, it is omitted from the number).

3.1.1.4 Unaccounted for valance requirements are assumed to be chlorine atoms.

3.1.1.5 When the compound contains bromine or iodine, the same rules apply, except the letter B for bromine or I for iodine follows the parent compound designated number, and the number of the atoms is placed after the letter.

Example: $CF_3CH_2CF_3$ = R–236fa = HFC–236fa

[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 Halogenated Fire Extinguishants.

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[2] *Annual Book of ASTM Standards*, Vol 15.05.

[3] Available from American National Standards Institute, 11 W. 42nd St., 13th Floor, New York, NY 10036.

[4] Available from the Compressed Gas Association.

[5] Available from Superintendent of Documents, U.S. Government Printing Office, Washington, DC 20036.

[6] Available from American Society of Refrigeration Engineers, Refrigeration Engineering 65, 49 (1957).

3.1.2 *HFC-236fa*—the compound 1,1,1,3,3,3–hexafluoropropane; $CF_3CH_2CF_3$.

4. Material Requirements

4.1 *Type I—Mixtures of HFC-236fa 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 the HFC-236fa/nitrogen within the container should not exceed that needed to achieve complete filling of the container at the maximum expected storage temperature. For example, the U.S. DOT 4BA500 cylinder partial pressure shall not exceed 24.4 bar at 21°C (340 psig at 70°F) for a 1153–kg/m^3(72 lb/ft^3) fill density. 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-236fa shall conform to the requirements prescribed in Table 1 when tested by the appropriate test method(s) listed in 6.1.

4.1.3 When material analysis is required, by agreement between the purchaser and the supplier, the total pressure in the HFC-236fa container, partial pressure of the nitrogen, the fill density of the HFC-236fa 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 (psig). 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-236fa shall be reported in degrees Celsius (preferred) or in degrees Fahrenheit and shall conform to the applicable regulations for the HFC-236fa container design and use.

4.2 *Type II*—HFC-236fa shall conform to the requirements of Type I, as listed in 4.1.1, and shall contain no more than 1.5 % by volume fixed gases in the vapor phase, expressed as air when tested by the appropriate test method(s) listed in Section 6.1.

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.

NOTE 2—**Warning:** Exposure to concentrations of HFC-236fa in excess of 15 % by volume in air during periods of elevated adrenaline could produce cardiac arrhythmia in some personnel.

4.4 Unless otherwise specified, Type I is assumed.

5. Sampling

5.1 Samples of HFC-236fa taken from the liquid phase, shall be taken from filled containers in accordance with the method specified in ISO 3427. The sampling bottle shall be capable of safely resisting the vapor pressure of the sample at the highest temperature that could be encountered.

TABLE 1 Requirements

Property	Requirement
HFC-236fa purity, %, mol/mol, min	99 (exclusive of any N_2 present)
Acidity, ppm by mass, as HCl, max	1.0
Water content, ppm by mass, max	10
Nonvolatile residue, % by weight, max	0.03
Suspended matter or sediment	none visible

5.2 The HFC-236fa 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 chromatography in accordance with the technique described in 6.1.2.1-6.1.5.1 or another acceptable laboratory technique providing equivalent results.

6.1.2 *Apparatus*—The following special apparatus is required to determine the percent HFC-236fa.

6.1.2.1 *Gas Chromatograph*, equipped with a thermal conductivity detector (TCD) and an integrator, 1–mV recorder, or other output device.

6.1.2.2 *Chromatographic Column*, 6.0–ft length by ⅛–in. outside diameter (OD) stainless steel tubing, packed with 80 to 100 mesh PORAPAK Q or equivalent (column is available prepacked from any chromatographic supply vendor).

6.1.2.3 *Gas Sampling Valve*, 1–mL volume or a volume sufficient to achieve proper separation and peak area for the specified column.

6.1.3 *Reagents*:

6.1.3.1 The carrier gas shall be a chromatographic grade of helium.

6.1.3.2 The column packing shall be 80 to 100 mesh PORAPAK Q or equivalent.

6.1.4 *Procedure*:

6.1.4.1 Install the column in the gas chromatograph and set the oven temperature to 45°C, injection port to 175°C, detector block to 200°C. The oven temperature is programmed to hold at 45°C for 2 min, then rise 10°C/min, to a maximum of 150°C.

6.1.4.2 Adjust the column helium flow to 20 mL/min.

6.1.4.3 Adjust the detector voltage to the mid-range of the thermal conductivity detector (TCD) and allow the instrument to equilibrate.

6.1.4.4 Take a vapor (flashed liquid) sample from the liquid phase (inverted cylinder). Flush the sample loop and valve for approximately 30 s.

6.1.4.5 Rotate the sample valve to transfer the sample into the chromatograph and note the time.

6.1.4.6 Close the sample cylinder valve.

6.1.4.7 Allow the sample to elute for approximately 15 min, attenuating as necessary to make the peak height a convenient size. Under proper instrument settings, air (N_2, O_2) should elute after about 0.4 min, and HFC-236fa should elute after approximately 8 min.

6.1.5 *Calculation*:

6.1.5.1 Calculate percent HFC-236fa as follows:

$$\% \text{ HFC}-236\text{fa} = A_H(100)/A_T$$

where:

A_H = area of the HFC-236fa peak (peak area × attenuation), and

A_T = sum of all relevant peak areas excluding the nitrogen (air) peak (peak area × attenuation).

Percent HFC-236fa below that specified in Table 1 shall constitute failure by this test method.

6.1.5.2 Calculate the percent nitrogen (air) as follows:

$$\%\ N_2\ (\text{air}) = A_N\,(100)/(A_T + A_N)$$

where:

A_N = area of the nitrogen (air) peak, and
A_T = sum of all the relevant peak areas including the nitrogen (air) peak.

It is useful to calculate the percent nitrogen (air) to judge a safe fill density. This is the amount of nitrogen (air) in the liquid phase.

6.2 *Acidity*—Vaporize a large sample in the presence of distilled water. Determine the acidity of the solution by the appropriate method specified in ISO 3363, titration in accordance with 6.2.1.3 and 6.3, a pH indicator, or another acceptable laboratory technique providing equivalent results.

6.2.1 *Sodium Hydroxide Titration*:

6.2.1.1 *Reagents*:

(1) Sodium Hydroxide, 0.01 *N* solution, standardized against reagent grade potassium acid phthalate.

(2) Methyl Red Indicator, 0.1 % solution.

6.2.1.2 *Procedure*—Place 50 mL distilled water-crushed ice (made from distilled water) slurry in a 250–mL Erlenmeyer flask. Sparge 50 g of the HFC-236fa into the slurry. Loosely stopper the flask and swirl the flask gently from time to time until the ice is completely melted. Add 1 drop of methyl red indicator, swirl, and if a reddish color remains, titrate to a yellow end point with 0.01 *N* sodium hydroxide solution. Run a crushed ice-distilled water blank (no HFC-236fa) along with the sample.

6.2.1.3 *Calculation*— Calculate parts per million acid halides, as HCl, as follows:

$$\text{acid halides, ppm} = \frac{(A–B) \times N\ \text{NaOH} \times 0.03645 \times 10^6}{\text{weight of sample}}$$

where:

A = NaOH for sample, mL,
B = NaOH for blank, mL,
N = normality of the NaOH solution,
NaOH = sodium hydroxide, and
0.03645×10^6 = factor to express result as ppm HCl (hydrogen chloride).

Acid halides in excess of that specified in Table 1 shall constitute failure by this test method.

6.3 *Water Content*— Test HFC-236fa for water content. The standard test method shall be by the Karl Fischer method. The analysis may be conducted by the phosphorous pentoxide test method, infrared absorption, electronic moisture analysis, piezoelectric analyzer, or another acceptable laboratory technique. Water content greater than specified in Table 1 shall constitute failure by this test method.

6.4 *Nonvolatile Residue*—Determine the nonvolatile residue in accordance with the method specified in ISO 5789 or another accepted laboratory technique providing equivalent results. Determine suspended matter or sediment (see 6.6) while performing this analysis.

6.5 *Fixed Gases in the Vapor Phase*—Test HFC-236fa for the presence of air in the vapor phase by gas chromatography, or another accepted laboratory technique providing equivalent results.

6.5.1 *Gas Chromatography*—HFC-236fa 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 by this test method.

6.5.2 *Procedure I*— Follow the procedure as outlined in 6.1.1, except the sample is taken from the vapor space of the container. This will be an area, volume percent result. Air (N_2, O_2) will elute after about 0.4 min.

6.5.3 *Procedure II*:

6.5.3.1 *Apparatus*—The following special apparatus is required to determine the percent fixed gases in HFC-236fa:

(1) Gas Chromatograph, equipped with a thermal conductivity detector (TCD) and an integrator, 1–mV recorder, or other output device.

(2) Chromatographic Column, 9–m length by 3.175–mm outside diameter (29.5–ft by ⅛–in.) stainless steel tubing, packed with 30 to 60 mesh 13× molecular sieve. (Column is available prepacked from any chromatographic supply vendor.)

(3) Gas Sampling Valve, 1–mL volume or a volume sufficient to achieve proper separation and peak area for the specified column.

6.5.4 *Reagents*:

6.5.4.1 *Carrier Gas*, shall be a chromatographic grade of helium.

6.5.4.2 *Column Packing*, shall be 30 to 60 mesh 13× molecular sieve.

6.5.4.3 *Calibration Gas*—0.8 to 2 % N_2 and 0.2 to 1 % O_2 in helium. Available from specialty gas suppliers.

6.5.5 *Procedure*:

6.5.5.1 Install the column in the gas chromatograph and set the oven temperature to 100°C, the injection port to 200°C, and the detector block to 200°C. The oven temperature is programmed to hold at 100°C for 11 min, then rise to 35°C/min, to a maximum of 190°C, and hold for 30 min.

6.5.5.2 Adjust the column helium flow to 15 mL/min.

6.5.5.3 Adjust the detector voltage to the mid-range of the thermal conductivity detector (TCD) and allow the instrument to equlibrate.

6.5.5.4 Take the sample from the vapor phase. Flush the sample loop and valve for approximately 30 s at a flow rate of 20 mL/min.

6.5.5.5 Rotate the sample valve to transfer the sample into the chromatograph and note the time.

6.5.5.6 Close the sample cylinder valve.

6.5.5.7 Allow the sample to elute for approximately 42 min, attenuating as necessary to make the peak height a convenient size. Under proper instrument settings, the oxygen will elute at approximately 2 min and nitrogen will elute at approximately 3 min.

6.5.6 *Calculation*:

6.5.6.1 *Calibration*— Calibration factors are determined by analyzing the standard gas in accordance with 6.5.5.1 and calculating as follows:

$$F_N = \%\ N_2 \text{ in standard gas}/\ A_N$$

$$F_O = \%\ O_2 \text{ in standard gas}/\ A_O$$

where:

A_N = area of nitrogen peak,

A_O = area of oxygen peak,
F_N = nitrogen factor, and
F_O = oxygen factor.

6.5.6.2 Calculate the concentrations in the sample as follows:

$$\% N_2 = (A_N)(F_N)$$

$$\% O_2 = (A_O)(F_O)$$

$$\% NAG = \% N_2 + \% O_2$$

(NAG = nonabsorbable gas)

6.6 *Suspended Matter or Sediment*—While performing the nonvolatile residue analysis, examine visually for any suspended matter or sediment. Observation of any suspended matter or sediment shall constitute failure by this test method.

7. Container, Packaging, and Package Marking

7.1 Containers used for shipping and storage of HFC-236fa conforming to this specification shall be marked in the accordance with Code of Federal Regulations (CFR) Title 49, Part 172 Subpart D. The proper shipping name for pure 1,1,1,3,3,3–hexafluoropropane is "Liquefied Gas, n.o.s." UN3163 (49 CFR 172.101). The proper shipping name for nitrogen super-pressurized 1,1,1,3,3,3–hexafluoropropane is "Liquefied Gas, nonflammable charged with nitrogen," UN1058 (49 CFR 172.101). In addition to DOT requirements, containers shall be marked with the following information as a minimum:

7.1.1 Supplier's name and address,

7.1.2 HFC-236fa (1,1,1,3,3,3–hexafluoropropane), and

7.1.3 Statement that material conforms to Specification D 6541.

8. Keywords

8.1 $CF_3CH_2CF_3$; FE-36™; fire fighting; fire fighting agent; fire protection; fire suppressant;1,1,1,3,3,3–hexafluoropropane; HFC-236fa; hydrofluorocarbon; hydrofluorocarbon 236fa