Designation: D 6126 – 97 (Reapproved 2003)ε1

Standard Specification for HFC-23 (Trifluoromethane, CHF_3)[1]

This standard is issued under the fixed designation D 6126; 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.

ε[1] NOTE—Editorial changes were made in December 2003.

1. Scope

1.1 This specification covers the requirements for HFC-23 as a fire fighting medium.

1.2 This specification does not address the fire fighting equipment or hardware that employs HFC-23 or the conditions of employing such equipment (for example: handshields, fixed installations, etc.).

1.3 This specification does not address the storage or transportation of HFC-23 Storage handling, and transportation issues are addressed in Practice D 6127.

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 to the test methods portion, Section 6, 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.*

2. Referenced Documents

2.1 *ASTM Standards:* [2]

D 4081 Specification for Dry-Cleaning Grade Perchloroethylene

D 6127 Practice for Handling, Transportation, and Storage of HFC-23

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. Governmental Standards:*

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

DOT-E 9491, Exception to Code of Federal Regulations (CFR) Title 49, Part 173.302 and Part 173.304[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:*

ASRE Standard 34, Designation of Refrigerants[6]

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *halogenated hydrocarbon*—the halogenated compound coding terminology system provides a convenient means to reference halogenated hydrocarbons, ASRE 34. Halogenated hydrocarbons are saturated hydrocarbons in which one or more of the hydrogen atoms have been replaced by atoms of the halogen series (fluorine, chlorine, bromine, and

[1] This specification is under the jurisdiction of ASTM Committee D26 on Halogenated Organic Solvents 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.

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.

[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).

iodine). It is convention to prefix the number with an abbreviation of the compound:

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

3.1.1.1 *Discussion*—By definitions, the right most digit of the numbering system is the number of fluorine atoms.

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

3.1.1.3 *Discussion*—The third digit from the right is one less (-1) than number of carbon atoms in the compound (when this number is zero (0) it is omitted from the number.

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

3.1.1.5 *Discussion*—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.

3.1.1.6 *Discussion—Example*—CHF_3= R-23 = HFC-23.

3.1.2 *HFC-23*[7]—the compound trifluoromethane; CHF_3.

4. Material Requirements

4.1 Nitrogen (N_2) partial pressure is not required for this product, it is shipped neat. The saturated vapor pressure of HFC-23 is 42 bars at 21°C (610 psig at 70°F). HFC-23 has a critical temperature of 25.9°C (78.6°F). The filling density should be that which will not liquid fill the container at temperatures below 25.9°C (78.6°F) or exceed 5/4 the pressure rating of the container at 130 °F. For example, the U.S. DOT 3AL cylinder with a minimum working pressure of 1800 psig shall not exceed 42 bar at 21°C (610 psig at 70°F) for a maximum filling density of 84.4 %. For this example the cylinder pressure is 141 bars at 54°C (2045 psig at 130°F).

4.1.1 HFC-23 shall conform to the requirements prescribed in Table 1 when tested by the appropriate test method(s) listed in Section 6.

4.1.2 When material analysis is required, by agreement between the purchaser and the supplier, the total pressure in the HFC-23 container, the fill density of the HFC-23 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-23 shall be reported in degrees Celsius (preferred) or in degrees Fahrenheit and shall conform to the applicable regulations for the HFC-23 container design and use.

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

4.3 *Product Requirements*—See Table 1.

5. Sampling

5.1 Samples of HFC-23, 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.

5.2 The HFC-23 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 6.1.2-6.1.5 or another acceptable laboratory technique providing equivalent results.

6.1.2 *Apparatus*—The following special apparatus is required to determine the percent HFC-23.

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 (m) length by 1/8 in. (mm) outside diameter (OD) stainless steel tubing, packed with 80 to 100 mesh PORAPAK Q or equivalent. (Column is available prepacked from any chromatographic supply vender).

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 to 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 the 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.

[7] FE - 13™ is the registered trademark for HFC-23 and is manufactured by E.I. Dupont de Nemours Co., Wilmington, DE.

TABLE 1 Requirements

Property	Requirement
HFC-23 purity, %, mol/mol, max	99.0
Acidity, ppm by mass, as HCl, max	3.0
Water content, ppm by mass, max	10
Nonvolatile residue, % by weight, max	0.08
Suspended matter or sediment	none visible

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_2O_2) should elute after about 0.4 min and HFC-23 should elute after approximately 2 min.

6.1.5 *Calculation*:

6.1.5.1 Calculate percent HFC-23 as follows:

$$\% \text{ HFC}-23 = A_{II}(100)/A_T \quad (1)$$

where:

A_{II} = area of the HFC-23 peak (peak area × attenuation), and

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

6.1.5.2 Percent HFC-23 below that specified in Table 1 shall constitute failure by this test method.

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 6.2.1.2 through 6.2.2.3, a pH indicator, or another acceptable laboratory technique providing equivalent results.

6.2.1 *Sodium Hydroxide Titration*:

6.2.1.1 *Reagents*:

6.2.1.2 *Sodium Hydroxide*, 0.01 *N* solution, standardized against reagent grade potassium acid phthalate.

6.2.1.3 *Methyl Red Indicator*, 0.1 % solution.

6.2.1.4 *Procedure*—Place 10 mL of distilled water - crushed ice (made from distilled water) slurry in a 250 mL Erlenmeyer flask. Sparge 50 g of the HFC-23 into the slurry. Loosely stopper the flask and swirl the flask gently form time to time until the ice is completely melted. Add one drop of methyl red indicator, swirl, 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 (no HFC-23) along with the sample.

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

$$\text{acid halides, ppm} = \frac{(A - B) \times N\,\text{NaOH} \times 0.03645 \times 10^6}{\text{weight of sample, grams}} \quad (2)$$

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).

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

6.3 *Water Contents*—Test HFC-23 for water content. The accuracy of the results and the standard method shall be by Karl Fisher method. The analysis may be conducted by the phosphorous pentoxide 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 6.5 (suspended matter or sediment) while performing this analysis.

6.5 *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 method.

7. Container, Packaging, and Package Marking

7.1 Containers used for shipping and storage of HFC-23 conforming to this specification shall be marked in the accordance with Code of Federal Regulations (CFR) Title 49, Part 172 Sub D. HFC-23 has a critical temperature of 25.9°C (78.6°F). Therefore, the proper shipping name (49 CFR 172.101) is “Compressed gas, n.o.s.”. The DOT exemption that contains this information is DOT-E 9491. In addition to DOT requirements containers must be marked with the following information as a minimum:

7.1.1 Supplier’s name and address,

7.1.2 HFC-23 (trifluoromethane), and

7.1.3 Statement that material conforms to ASTM Specification D 6126.

8. Keywords

8.1 CHF_3; FE-13™; fire fighting; fire fighting agent; fire protection; fire suppressant; HFC-23; hydrofluorocarbon; trifluoromethane