

SEMI C3.58-0600

SPECIFICATION FOR OCTAFLUOROCYCLOBUTANE, C₄F₈, ELECTRONIC GRADE IN CYLINDERS

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1 Description

1.1 Octafluorocyclobutane, also known as perfluorocyclobutane, is a gas under normal atmospheric conditions (15°C and 760 mm Hg).

1.2 The gas is a non-flammable, non-toxic, colorless and odorless gas which is stable under normal conditions.

2 Specifications

Purity: 99.99%

<i>Impurities</i>	<i>Maximum Acceptable Level</i>
Nitrogen/Oxygen	50 ppm
Moisture	5 ppm
Other halocarbons ^{#1}	40 ppm
Acidity (as HF)	1 ppm mole

#1 R13, R23, R114, R113, R11, and R116.

3 Referenced Standards

3.1 None.

4 Physical Constants (for information only)

Formula	C ₄ F ₈ or CF ₂ • CF ₂ • CF ₂ • CF ₂
Molecular weight	200
Melting point	-40.2°C
Boiling point	115°C
Relative density, gas	6.9 (air=1)
Relative density, liquid	1.6 (water=1)
Vapor pressure 20°C	2.7 bar
Appearance/Color	Colorless gas
Odor	none

5 Analytical Procedures

5.1 *Acidity* — This procedure is for the determination of acidity in octafluorocyclobutane using a classical chemical method of analysis, utilizing methyl red indicator.

5.1.1 *Detection Limit* — 1 ppm as HF.

5.1.2 *Apparatus*

5.1.2.1 Three 8 X one inch flat bottomed test tubes, flexible tubing, flowmeter, pressure regulator.

5.1.2.2 *Reagents* — 0.02% methyl red solution, 0.01 N hydrochloric acid, freshly distilled or deionized water boiled to remove carbon dioxide.

5.1.2.2.1 0.02% methyl red: methyl red is 1-carboxybenzeneazodimethylaniline. This is the acid or alcohol soluble form which shall be used. Dissolve 20 mg of methyl red (acid form) in a mixture of 7.44 mL of 0.01M sodium hydroxide and 70 mL of alcohol and dilute to 100 mL with water. The alcohol must contain not less than 95% v/v ethanol.

5.1.2.2.2 0.01 M hydrochloric acid: dilute 103.0 g of hydrochloric acid solution (SG 1.18) to 1000.0 mL with water to give 1 M solution.

5.1.2.2.3 Dilute 10.0 mL of this solution to 1000.0 mL with water to give 0.01 M solution.

5.1.2.2.4 Alternatively prepare by the dilution of a commercially available standard solution.

5.1.3 *Operating Procedure*

5.1.3.1 Attach a suitable regulator to the cylinder outlet.

5.1.3.2 Connect the flowmeter to the regulator using flexible tubing.

5.1.3.3 Open the cylinder valve and purge the flowmeter with octafluorocyclobutane.

5.1.3.4 Pour 50 mL of carbon dioxide free water into each of three 8 X one inch flat bottomed test tubes. Add 0.1 mL of 0.02% solution of methyl red in alcohol to each tube and label the tubes 'a' 'b' and 'c'. Add 0.1 mL of 0.01 M hydrochloric acid to tube 'b' and 0.2 mL of 0.01M hydrochloric acid to tube 'c'. Pass a 23 liter gaseous sample of octafluorocyclobutane through the solution in tube 'b'. Compare the color of tube 'b' after the test with the other two tubes. The intensity of the color produced in 'b' should be between those solutions 'a' and 'c'. The formation of a pink color indicates acidity. A yellow color will be produced by alkalinity.

5.1.3.4.1 The colors of the test solutions before the test should be:

'a' — pale straw

'b' — pink

'c' — pink, slightly darker than 'b'.

5.1.3.4.2 To differentiate the colors more readily after the test view the tubes by looking down through the solution rather than through the tubes. Place a sheet of white paper behind the tubes and carry out the comparisons in natural light.

6 Analytical Procedures – Instrumental Analysis

6.1 *Oxygen and Nitrogen* — This procedure is for the determination of oxygen and nitrogen in octafluorocyclobutane using a gas chromatograph with a thermal conductivity detector. Column switching is used to separate and elute the components and to backflush octafluorocyclobutane.

6.1.1 *Detection Limits* — Oxygen 1 ppm (mole/mole), nitrogen 1 ppm (mole/mole).

6.1.2 *Instrument Parameters*

6.1.2.1 *Columns*: Pre-column: Molecular sieve 13X, 60/80 mesh, 2.5 m by 2.1 mm ss or equivalent. Main column: Molecular sieve 13X, 60/80 mesh, 1.5 m by 2.1 mm ss or equivalent. Ensure that all columns are conditioned before assembly.

6.1.2.2 *Gas Flows*: Helium carrier gas 25 mL/min for each column.

6.1.2.3 *Sample Volume*: 2.5 mL

6.1.2.4 *Temperatures*: Detector 100°C, Oven 90°C isothermal.

6.1.3 *Calibration Standard*: 1–50 ppm (mole/mole) oxygen and nitrogen in helium.

6.1.4 *Chromatogram* — See Figure 1.

6.2 *Halogenated Hydrocarbons* — This procedure is for the determination of halogenated hydrocarbons in octafluorocyclobutane using a gas chromatograph with a flame ionization detector.