

SEMI C3.58-1011 (Reapproved 0418) SPECIFICATION FOR OCTAFLUOROCYCLOBUTANE, C₄F₈, ELECTRONIC GRADE IN CYLINDERS, 99.999% QUALITY

This Standard was technically approved by the Gases Global Technical Committee. This edition was approved for publication by the global Audits and Reviews Subcommittee on August 18, 2017. Available at www.semiviews.org and www.semi.org in April 2018; originally published June 2000; previously published October 2011.

1 Purpose

1.1 The purpose of this Document is to provide specification for octafluorocyclobutane (C₄F₈), that is used in the semiconductor industry.

2 Scope

2.1 This Document covers requirements for octafluorocyclobutane (C₄F₈) used in the semiconductor industry.

NOTICE: SEMI Standards and Safety Guidelines do not purport to address all safety issues associated with their use. It is the responsibility of the users of the Documents to establish appropriate safety and health practices, and determine the applicability of regulatory or other limitations prior to use.

3 Referenced Standards and Documents

3.1 None.

4 Description

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

4.2 The gas is a nonflammable, nontoxic, colorless and odorless gas which is stable under normal conditions.

5 Specifications

Table 1 Impurity Limits for Octafluorocyclobutane

<i>Grade</i>	99.999%
<i>Impurities</i>	<i>Maximum Acceptable Level</i>
Oxygen	1 ppmv
Moisture	3 ppmv
Other organic (PFC-1216, PFC-31-10mc, PFC-1318my, PFC-31-10my, CFC-114, CFC-114a, THF)	10 ppmv
Acidity (as HCl)	100 ppbw
Air (N ₂ , O ₂ , CO, CO ₂)	3 ppmv

6 Properties of Octafluorocyclobutane (for information only)

Table 2 Properties of Octafluorocyclobutane

Molecular Formula	C ₄ F ₈ or CF ₂ • CF ₂ • CF ₂ • CF ₂
CAS Number	115-25-3
Molecular Weight	200
Melting Point	-41.4°C
Boiling Point	-6°C
Relative density, gas	6.9 (air = 1)
Relative density, liquid	1.6 (water = 1)
Vapor pressure 20°C	270 kPa (2.7 bar)
Appearance/Color	Colorless gas
Odor	None

7 Analytical Procedures

NOTE 1: Samples of octafluorocyclobutane should be equilibrated at no less than 30°C (85°F) for at least 24 hours prior to analysis to ensure adequate vapor pressure for analysis and consistency.

7.1 *Acidity* — This procedure is for the determination of acidity in octafluorocyclobutane utilizing an aqueous conductivity measurement. Calculations are made relating sample conductivity to free acidity, calculated as hydrochloric acid, in the sample.

7.1.1 *Detection Limit* — 30 ppb as HCl.

7.1.2 *Apparatus* — A conductivity bridge and cell, heat gun, graduated cylinders, gas dispersion tube, wide-mouth polyethylene bottle, hypodermic syringes, polyethylene beaker, water purification system.

7.1.3 *Reagents*

7.1.3.1 Hydrochloric acid, 0.01 N standard in SDA-2B ethyl alcohol (store the standard in separate 50-mL serum capped vials).

7.1.3.2 CFC-11, special (analyzed to have an aqueous conductance of <0.5 µmho/cm). If CFC-11 is not available, a similar degreasing solvent can be substituted, such as HFC-43-10.

7.1.3.3 Distilled and deionized (DI) water, aqueous conductivity <0.5 µmho/cm, prepared immediately prior to use by passing distilled water through a water purification system and analyzing it with a conductivity bridge. DO NOT STORE DEIONIZED WATER FOR USE IN THIS ANALYSIS.

7.1.3.4 RBS-35 cleaner.

7.1.4 *Operating Procedure*

7.1.4.1 Attach a suitable regulator to the cylinder outlet.

7.1.4.2 *Conductivity Bridge/Cell* — Store the conductivity cell in a polyethylene beaker containing DI water when not in use.

7.1.4.3 *Calibration*

7.1.4.3.1 Add CFC-11 to the 20 mL calibration mark of the polyethylene (PE) bottle. Immediately cap the bottle and shake contents vigorously for approximately 30 seconds. Discard the CFC-11.

7.1.4.3.2 Fill the bottle again with CFC-11 to the 20 mL mark. Immediately add 100 mL of DI water to the bottle, cap and shake vigorously for approximately 120 seconds.

7.1.4.3.3 Rinse the conductivity cell with DI water and shake it dry. Uncap the PE bottle and dip the conductivity cell into the water layer (top layer). Read and record the conductivity value. This reading will serve as the 'BLANK' value. Remove the conductivity cell from the solution, rinse with DI water and store in a beaker containing DI water. Immediately cap the PE bottle.

7.1.4.3.4 Fill and flush a clean, dry, 50 mL syringe several times with the 0.01 N standard, hydrochloric acid in SDA-2B ethanol. Fill the syringe to a volume of 30 mL. Holding the syringe vertically with needle pointing up, tap the barrel gently to permit air bubbles to rise to the top. Slowly depress the plunger until the plunger tip is aligned exactly with the 20 mL mark. Immediately uncap the PE bottle and inject the syringe contents into the bottle. Immediately cap the bottle. Shake the bottle vigorously for approximately 120 seconds. Uncap the PE bottle and dip the conductivity cell into the water layer. Read and record the conductivity value. Remove the conductivity cell from the solution, rinse with DI water and store in a beaker containing DI water.

7.1.4.3.5 Calculate the nanograms (ng) of hydrochloric acid used in the standard from the following equation:

$$ng\ HCl = \frac{V \times N \times MEQ \times 10^9}{10^3} \quad (1)$$

where:

$ng\ HCl$ = nanograms of HCl in the standard.

V = HCl volume, microliters.

N = HCl normality.

MEQ = HCl milliequivalent weight (g/meq) = 0.036461.

7.1.4.3.6 Calculate the calibration factor as follows:

$$ng\ HCl/\mu mho = \frac{A}{(C2-C1)} \quad (2)$$

where:

$ng\ HCl/\mu mho$ = calibration factor.

A = ng HCl in standard.

$C1$ = blank conductivity, μmho .

$C2$ = standard conductivity, μmho .

7.1.4.3.7 Repeat procedure at least six times. Use the average of the six calibration factors for sample calculations.

7.1.4.4 Sampling and Analysis

7.1.4.4.1 Add CFC-11 or a similar degreasing solvent to the 20 mL calibration mark of the PE bottle. Immediately cap the bottle and shake contents vigorously for approximately 30 seconds. Discard the CFC-11.

7.1.4.4.2 Fill the bottle again with CFC-11 to the 20 mL mark. Immediately add 100 mL of DI water to the bottle, cap and shake vigorously for approximately 120 seconds.

7.1.4.4.3 Rinse the conductivity cell with DI water and shake it dry. Uncap the PE bottle and dip the conductivity cell into the water layer (top layer). Read and record the conductivity value. This reading will serve as the 'BLANK' value. Remove the conductivity cell from the solution, rinse with DI water and store in a beaker containing DI water. Immediately cap the PE bottle.

7.1.4.5 Weigh the sample cylinder and record. Attach PE gas dispersion tube to the sample cylinder valve. Uncap the PE bottle and lower the gas dispersion tube into the water. Open the cylinder valve and purge sample through the bottle such that about 150 g of sample are added to the bottle over a 60 minute period. Close the cylinder valve and immediately remove the gas dispersion tube and cap the bottle. Record the final weight of the capped sample cylinder.

7.1.4.6 Shake the PE bottle vigorously for approximately 120 seconds. Read and record the conductivity value as done previously.