

# 3M<sup>™</sup> 11B Enriched Boron Trifluoride for Semiconductor Manufacturing

**Note:** The purpose of this guide is to provide basic information to product users for use in evaluating, processing, and troubleshooting their use of certain 3M Technical Ceramics products. The information provided is general or summary in nature and is offered to assist the user. The information is not intended to replace the user's careful consideration of the unique circumstances and conditions involved in its use and processing of 3M Technical Ceramics products. The user is responsible for determining whether this information is suitable and appropriate for the user's particular use and intended application. The user is solely responsible for evaluating third party intellectual property rights and for ensuring that user's use and intended application of 3M Technical Ceramics product does not violate any third party intellectual rights.

# Introduction

3M Technical Ceramics has produced high purity B-11 enriched boron trifluoride ( $^{11}BF_3$ ) at the boron isotope enrichment facility located in Quapaw, Oklahoma for over a decade. As one of the largest boron isotope separation facilities in the world, 3M Technical Ceramics is committed to providing our customers with the high quality products for use in the semiconductor industry.

# Use of 3M<sup>™</sup> 11B Enriched Boron Trifluoride in the Semiconductor Industry

In contrast to electrons, protons, photons and heavy charged particles, neutrons undergo extremely weak electromagnetic interactions. With no electrical charge to cause repulsion, even low energy neutrons are able to

	High Purity Grade	lon Implant Grade
Assay		
Chemical Purity*	>99.99%	>99.9 %
Isotopic Purity (B-11 atomic %)	>99.8 %	>99.0 %
Limits of Impurities		
$N_2$	<15 ppmv	<25 ppmv
O <sub>2</sub> /Ar	<15 ppmv	<50 ppmv
CO <sub>2</sub>	<15 ppmv	<25 ppmv
SO <sub>2</sub>	<15 ppmv	<25 ppmv
HF	<15 ppmv	<25 ppmv
SiF₄	<15 ppm	-

Table 1

\*Calculated from analyzed impurities

initiate reactions with a target nucleus. The probability of reaction, or capture, of the neutron with a nucleus is controlled by several parameters including neutron absorption cross-section ( $\sigma$  in barns; 1 barn = 10<sup>-24</sup> cm<sup>2</sup>) and the speed (energy) of the neutron. Less energetic neutrons are usually more likely to react with an atomic nucleus.

Certain chemical elements are particularly efficient at absorbing or capturing incident neutrons; this characteristic is related to the specific isotope rather than simply the element. Boron has two naturally occurring stable isotopes <sup>10</sup>B (~20 atomic %) and <sup>11</sup>B (~80 atomic %).

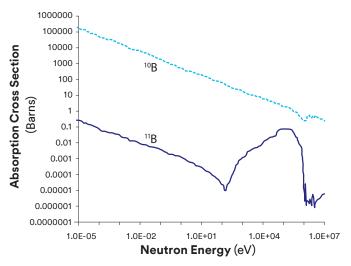
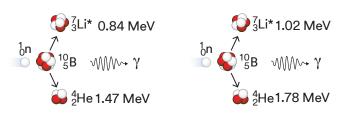


Figure 1. Neutron absorption cross sections for  $^{10}\text{B}$  (dashed light blue) and  $^{11}\text{B}$  (solid dark blue).

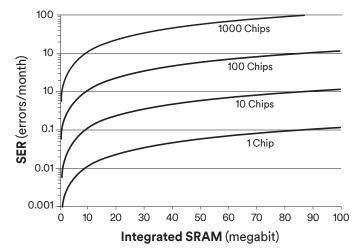


**Figure 2.** Major (left) and minor (right) reaction outcomes between <sup>10</sup>B and a neutron.

Of these two isotopes, <sup>10</sup>B nuclei are several orders of magnitude <u>more</u> likely to capture a neutron than <sup>11</sup>B nuclei for most neutron energies (Figure 1).

After capturing the neutron, <sup>10</sup>B will undergo one of two reactions (Figure 2). The major reaction (94%) generates an excited <sup>7</sup>Li atom and an alpha particle with kinetic energies of 0.84 MeV and 1.47 MeV respectively. The minor reaction (6%) results in a lithium atom (1.02 MeV kinetic energy) and an alpha particle (1.78 MeV kinetic energy).

A semiconductor device that uses natural abundance boron (p-type) dopant is susceptible to reaction of <sup>10</sup>B nuclei with incident neutrons. Particles produced in the neutron capture reaction will then lose kinetic energy as they travel through the silicon substrate. This can produce electron hole pairs, and result in charge collection (Q<sub>coll</sub>). If Q<sub>coll</sub> exceeds the critical charge needed to trigger a change in data state (Q<sub>crit</sub>), a soft error results; otherwise the node will continue normal function. The probability of a soft error increases close to a sensitive node. Without mitigation, soft error rates can exceed 50,000 FIT/chip, where one FIT is one failure in 10<sup>9</sup> device hours; for multiple chip systems this can lead to failures every few days of use (Figure 3). Enrichment of the boron dopant in the <sup>11</sup>B isotope is a simple mitigation technique that can be used to limit soft errors due to the reaction of <sup>10</sup>B with low energy cosmic neutronic radiation.



**Figure 3.** SER as a function of number of chips in the system and the amount of embedded SRAM per chip. From Baumann, Robert C. Radiation-Induced Soft Errors in Advanced Semiconductor Technologies. *IEEE Transactions on Device and Materials Reliability 2005, 5(3),* 305.

# **Basic Physical Properties of 11BF3\***

(not for specification purposes)

Formula	<sup>11</sup> BF <sub>3</sub>
Molecular Weight (g/mol)	68.00
Melting Point (°C @ 1 atm)	-128
Boiling Point (°C @ 1 atm)	-100
Density of Gas at STP (g/L)	3.08
Density of Liquid at Boiling Point (g/mL)	1.6
Density of Liquid at Melting Point (g/mL)	1.7
Critical Temperature (°C)	-12.2
Critical Pressure (kPa)	4984
Critical Density (g/cm³)	0.59
Heat of Fusion (kJ/mol)	4.24
Heat of Vaporization (kJ/mol)	18.56
Entropy @ 298.15 K (kJ/mol·°C)	253.81
Heat of Reaction with Excess Water (kJ/mol)	102.6

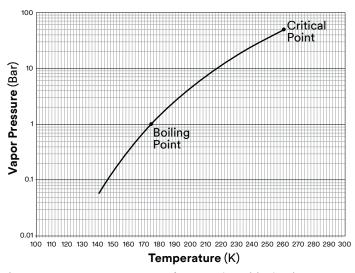
#### Table 2

\*Other than Molecular Weight, all physical properties are those of natural abundance BF<sub>3</sub>. Significant deviations from these values are not expected due to changes in isotopic enrichment.

#### Vapor Pressure

The vapor pressure of liquid  $BF_3$  is given by Equation 1, where P is the pressure in bar and T is the temperature in degrees Kelvin

The vapor pressure curve is provided in Figure 4.



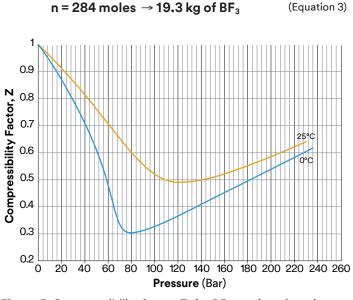
**Figure 4.** Vapor pressure curve for BF<sub>3</sub>. The critical point (-12.3°C, 49.85 bar) and boiling point (-100.4°C) are shown.

#### Compressibility

As with most compressed gases,  $BF_3$  does not follow the ideal gas law and as a result a compressibility factor, Z, must be used in order to correct calculations for  $BF_3$ . Figure 4 shows how the compressibility factor changes with pressure at 0°C and 25°C. This information can be used to calculate the mass of  $BF_3$  present in a container of known volume and pressure, where P is pressure, V is the container volume, n is the number of moles of gas, R is the ideal gas constant and T is temperature,

(Equation 2)

A 43 L cylinder of BF<sub>3</sub> at 25°C (298 K) and 90 bar (~1300 psi) would have a compressibility factor of  $Z \approx 0.55$  from Figure 5. The number of moles can be found as shown in Equation 3.



**Figure 5.** Compressibility factor, Z, for  $BF_3$  as a function of pressure. Data from Waxman, M.; Hilsenrath, J. and Chen, W. T. *J. Chem. Phys.* 1973, 58, 3692-3701 (http://dx.doi. org/10.1063/1.1679720).

#### **Reactivity and Water Sensitivity**

In a completely dry atmosphere,  $BF_3$  is a colorless gas with distinct, pungent odor. However in the presence of even trace amounts of water, a fuming white cloud will be formed with an acidic odor similar to that of hydrochloric acid. This property makes small system leaks detectable visually.

Boron trifluoride is a strong Lewis acid (electron pair acceptor) that will readily react with organic Lewis bases (electron pair donors) to form addition complexes, often with substantial heat generation. Therefore,  $BF_3$  should be isolated from chemicals that contain oxygen, nitrogen, sulfur and other lone pair donors.

Boron trifluoride readily absorbs water (3.22 g per 1 g water at 0°C and 1 atm) and forms various hydrates including the hemihydrate ( $BF_3 \cdot 0.5H_2O$ ), monohydrate ( $BF_3 \cdot H_2O$ ), dihydrate ( $BF_3 \cdot 2H_2O$ ) and even trihydrate ( $BF_3 \cdot 3H_2O$ ) complexes. The monohydrate is most common. All these species exist as simple adducts and are liquids at room temperature. In turn these hydrate species can dissociate into highly acidic ionized species according to Reaction 1.

$$\mathbf{BF}_3 \bullet \mathbf{2H}_2 \mathbf{O} \to \mathbf{H}_3 \mathbf{O}^+ + \mathbf{BF}_3 \mathbf{OH}^- \qquad (\text{Reaction 1})$$

If excess water is present then the very strong acid fluoroboric acid, HBF<sub>4</sub>, will be formed through a suspected stepwise removal of fluorine atoms co-producing boric acid. The overall reaction is shown in Reaction 2.

$$4BF_3 + 3H_2O \rightarrow 3HBF_4 + B(OH)_3 \qquad (Reaction 2)$$

It should be noted that in the presence of boric acid no free fluoride ion,  $F^{-}$ , exists in the system and therefore hydrofluoric acid, HF, will not be present. However, in the absence of boric acid, HBF<sub>4</sub> can be further hydrolyzed to produce HF (Reaction 3).

$$HBF_4 + H_2O \rightleftharpoons HBF_3OH + HF \qquad (Reaction 3)$$

# BF<sub>3</sub> Gas Handling and Material Compatibilities

### **Compatibility and Corrosion**

The information in Tables 3–5 has been compiled from these resources:

- International Standards: Compatibility of cylinder and valve materials with gas content; Part 1: ISO 11114-1 (July 1998), Part 2: ISO 11114-2 (March 2001)
- Honeywell Boron Trifluoride Technical Information Brochure (https://www.honeywell-bf3.com/?document=bf3-technicalinformation&download=1, accessed Feb. 6, 2015)

The data contained in this section must be used with caution. No raw data can cover all possible conditions of materials usage including concentration, temperature, humidity, and impurities. This information should be used only as a starting point for material selection. Extensive investigation and testing should be carried out under the specific conditions of use. While corrosion studies on dry  $BF_3$  gas streams show many materials have a high resistance to attack even at elevated temperatures, a small amount of moisture can lead to significant corrosion. This is due to the presence of hydrogen fluoride and/or other strong acids produced under hydrolysis conditions.

## **BF**<sub>3</sub> Gas Handling

When designing BF<sub>3</sub> gas handling systems, potential corrosion and the high working pressures must be taken into account. BF<sub>3</sub> cylinders can have a nominal pressure of 1500 psi and systems need to be designed to safely handle this pressure. BF<sub>3</sub> is extremely hygroscopic and trace amounts of moisture will result in the formation of hydrates and corrosive acids which can attack most common metal. If water is present in the system, introduction of BF<sub>3</sub> will lead to the formation of white boron oxide based deposits throughout the system. This deposition can lead to pluggage of lines, valves and regulators and be a source of localized material corrosion. Corrosion products may also travel through the handling system, fouling lines, valves and regulators. As a result, whenever the system is not in use it should be purged of remaining BF<sub>3</sub> using an inert gas and protected from moisture ingress when not in use. If a system must be thoroughly cleaned using water/steam or alcohol solutions, the system must be completely dry of water/alcohol and an inert gas purge is recommended before introducing  $BF_3$  to the system.

Materials for piping, fittings, gaskets, regulators and any other equipment in direct contact with  $BF_3$  should be chosen using the data Tables 3 to 5 as guidance. Final material selection should be based on testing specific to the end use. Typically, 316 L stainless steel can be used for piping and fittings in order to provide long term corrosion resistance. Use of copper or copper based alloys is strongly discouraged. PTFE or PCTFE gaskets are preferred.

Key Compatibility Characteristics		Materials⁺ Cylinder Valve			
		×	$\checkmark$	×	
Hydrolyses to hydrogen fluoride in contact with moisture. In wet conditions see specific risk hydrogen fluoride compatibility	NS QTS SS AA		CS SS	AA B	

**Table 3.** General metal and metal alloy compatibilities for cylinder and valve;  $\checkmark$  = acceptable, X = not recommended.

<sup>+</sup> NS = Normalized or Carbon Steels, QTS = Quenched or Tempered Steels, SS = Stainless Steels, AA = Aluminum Alloys, B = Brass or Copper

	Material	Compatibility
Plastics	Polytetrafluoroethylene (PTFE)	Acceptable
	Polychlorotrifluoroethylene	Acceptable
	Polyvinylidenefluoride (PVDF)	Acceptable
	Polyamide (PA)	Not recommended (weight loss)
	Polyproylene (PP)	Acceptable
	Butyl Rubber	Not recommended
	Nitrile	Not recommended (weight loss)
mers	Chloroprene	Not recommended (weight loss)
Elastomers	Chlorofluorocarbons	Acceptable
	Silicone	Not recommended (weight loss)
	Ethylene Propylene	Not recommended (weight loss)
Lubricants	Hydrocarbon	Not recommended (weight loss)
	Fluorocarbon	Acceptable

Table 4. General Compatibility of Non-metallics with  $BF_3$  Gas.

	I	Material <sup>+</sup>	21°C (mil)	149°C (mil)	Macro Examination
		A-285 CS	0.5	6.4	Uniform Corrosion
	•	SS 304	0.2	74.7	Severe Attack
ž	Ś	SS 316	0.15	10.2	Uniform Corrosion
Metals and Alloys		SS 347	0.2	12.1	Slight Pitting
		Copper	1.0	29.8	Shallow Pitting
		Monel <sup>®</sup> 400	0.3	23.9	Severe Attack
		Inconel <sup>®</sup> 600	0.15	15.3	Uniform Corrosion
		Hastalloy® C-276	0.02	0.2	Uniform Corrosion
		Alloy 20 Cb-3	0.10	9.7	Uniform Corrosion
			Hardness	%Wt. Change	Visual
Plastics	21°C	PTFE	+7	-0.03	No change
		Polyproylene	0	+0.03	Discolored
	149°C	PTFE	+19	+0.002	No change
	149	Polypropylene	NR		

**Table 5.** Corrosion of Common Materials for a 98% BF<sub>3</sub> + 2% H<sub>2</sub>O Vapor (168 hours Exposure)

# Safety

## Detection

No detectors have been developed specifically for  $BF_3$  detection. However, leaks of  $BF_3$  are readily detectable, as under typical atmospheric conditions  $BF_3$  will undergo immediate reaction with atmospheric moisture to form  $BF_3 \cdot H_2O$ , which appears as a dense, white cloud visible at less than 1 ppm. As formation of HF is, at best, sluggish from the monohydrate, detectors calibrated for mineral acid or HF may <u>NOT</u> provide accurate quantitative numbers. Such detection systems would have to be explicitly calibrated to  $BF_3$  under constant detection conditions (temperature and humidity).

### Maintenance of BF<sub>3</sub> Handling Systems

All equipment of a BF<sub>3</sub> handling system should be inspected on routine, scheduled basis. Any pieces of equipment that show signs of corrosion or defects should be replaced. If maintenance of a process line is needed, the line should be thoroughly purged with inert gas to remove and depressurized to ambient conditions. Even though a properly executed inert gas purge will remove the bulk of BF<sub>3</sub>, there may be some observed fuming when lines are broken and exposed to the atmosphere due to trace amounts of BF<sub>3</sub> being present. If necessary, the line can be flushed of the trace BF<sub>3</sub> with solvent/water or localized exhaust directed to a scrubber can be used.

If it becomes necessary to clean the lines of deposits, water, lower alcohols or steam may be used. Rinse the piping and the resulting liquid/condensate becomes clear. All lines, valves and regulators in direct contact with BF<sub>3</sub> gas must be thoroughly dried using an inert gas stream or dry air prior to the re-introduction of a BF<sub>3</sub> gas stream. To aid in this process a high volatility hydrophilic solvent may be used to rinse the lines. Following the drying process it is recommended that the system be placed under vacuum to remove moisture or solvent that may be trapped in connections or dead spots throughout the system. A final purge of inert gas will ready the system for re-introduction of BF<sub>3</sub> gas. Significant corrosion or deposit build up may indicate an uncontrolled or unknown exposure to moisture and should be rectified with a change in maintenance schedule and practices.

## Capture and Disposal of BF<sub>3</sub>

Users should check applicable environmental regulations prior to choosing the method of capture and disposal.

Water based solutions provide a good medium to capture and ultimately dispose of  $BF_3$ . In the presence of water  $BF_3$  will immediately react to form hydrates of water such that  $BF_3$  can be efficiently removed from a gas stream and is a scrubbing medium. In the presence of excess water fluorboric acid and boric acid will result, as shown in Reaction 4.

$$4BF_3 + 3H_2O \rightarrow 3HBF_4 + B(OH)_3$$

(Reaction 4)

The reaction products will result in a highly acidic solution which may need to be neutralized prior to disposal. Treatment of these solutions is often done with sodium hydroxide to give the highly water soluble sodium tetrafluoroborate salt (NaBF<sub>4</sub>, 40 wt% solubility). Alternatively, potassium hydroxide may be used to form the sparingly soluble potassium tetrafluoroborate salt (KBF<sub>4</sub>, 0.5wt% solubility).

#### **Personnel Safety**

All personnel involved with and responsible for handling 3M<sup>™</sup> 11B Enriched Boron Trifluoride should be familiar with the appropriate safety and handling precautions provided in the Safety Data Sheet. This section summarized the information found in the SDS. The SDS is available on www.3M.com and in case of emergency 1-800-364-3577 or (651) 737-6501 can be called (24 hours).

 $BF_3$  can cause severe skin burns and eye damage. Inhalation of  $BF_3$  and associated fumes formed from reaction with atmospheric moisture is highly suffocating and irritating to the respiratory system. Prolonged breathing of these fumes may be fatal. Table 6 provides exposure limits set by the U.S. Occupational Safety and Health Administration (OSHA) and the American Congress of Governmental Industrial Hygienists (ACGIH).

Ingredient	Agency	Limit
Boron trifluoride	ACGIH	CEIL: 1 ppm
Boron trifluoride	OSHA	CEIL: 3 mg/m <sup>3</sup> (1 ppm)

Table 6. Occupational exposure limits

In addition, the National Institute of Occupational Safety and Health (NIOSH) has established an immediately dangerous to life and health (IDLH) exposure level for  $BF_3 = 25$  ppm. Supplied air respiration systems should be required for exposure to concentrations greater than 25 ppm. Acid mist purifiers are not recommended.

Selection and use of gloves should follow local standards and be based on use factors such as exposure levels, concentration of the substance, frequency and duration, physical challenges such as temperature extremes and other use conditions. Consult with your glove and/or protective clothing manufacturer for selection of appropriate compatible gloves and other protection clothing. Acid resistant polymer laminate gloves are recommended.

As  $BF_3$  is extremely hygroscopic, substances which contain water will attract and readily absorb  $BF_3$ . For example, food, tobacco, and beverages will absorb and retain  $BF_3$  so their usage should be prohibited in a  $BF_3$ designated area.

Get immediate medical attention if exposed to  $BF_3$ . If  $BF_3$  is inhaled, remove person to fresh air and keep comfortable for breathing. For skin or hair exposure, immediately take off all contaminated clothing and rinse skin with water. Contaminated clothing should be washed before reuse. If in eyes rinse cautiously with water for several minutes. Remove contact lenses if present and continue rinsing.

## Mitigation of BF<sub>3</sub> Release

When  $BF_3$  is released to the atmosphere a dense, white cloud results from immediate reaction with water, resulting in the formation of droplets of  $BF_3$  hydrates. This is true even in environments in very low humidity. The reaction is appreciably exothermic (24.51 kcal g<sup>-1</sup> mol<sup>-1</sup> or 102.7 kJ g<sup>-1</sup> mol<sup>-1</sup>) and this results in the dense cloud being buoyant even though the  $BF_3$  vapor is more dense than air. Under ambient conditions, the aerosol sized  $BF_3$ hydrate droplets may eventually precipitate as droplets but a cloud can disperse over significant volume prior to this occurring. However, as the reaction with water is rapid, water sprays are very effective in mitigating the cloud. Water sprays should be directed as close to the source of the leak as possible.

As the products (BF<sub>3</sub> hydrates, fluorboric acid, hydroxyfluorobroates) of the reaction of BF<sub>3</sub> with water are acidic, care should be taken when cleaning surfaces affected by the leak. All surfaces and exposed equipment should also be checked for signs of corrosion. Dispose of all clean up materials according to local regulations.

It should be noted that some emergency manuals incorrectly identify HF as a hazardous species present if  $BF_3$  is released to the atmosphere. This is not confirmed by the available literature.

# **Shipping and Regulatory Information**

Boron Trifluoride (UN1008) is classified by the U.S. Department of Transportation (DOT) as Hazard Class 2, Division 3 (2.3), Compressed Gas Poisonous by Inhalation. 3M Technical Ceramics has further determined that 3M<sup>™</sup> 11B Enriched Boron Trifluoride meets the DOT classification for Class 8 Corrosive and has assigned that subsidiary hazard classification for all shipments of product.

 $BF_3$  is not listed as a hazardous air pollutant under the Clean Air Act Amendments (1990) and is not a priority pollutant under the Clean Water Act (1972, 1987).

Boron Trifluoride is identified as an extremely hazardous substance in the Superfund Amendments and Reauthorization Act (1986) with a reportable release quantity (RQ) of 500 pounds.

Boron Trifluoride is listed in Appendix A of the Occupational Safety and Health, Administration (OSHA) Rule 29 CFR 1910.119 (Process Safety Management of Highly Hazardous Chemicals, Explosives, and Blasting Agents) with a threshold quantity of 250 lbs. Locations with inventory exceeding the threshold at any one time must comply with the process management requirements of this rule.  $BF_3$  is further regulated under the U.S. Environmental Protection Agency (EPA) Accidental Release Prevention Requirements: Risk Management Program Requirements Under Clean Air Act Section 112(r)(7) with a threshold quantity of 5000 lbs. Locations having more than 5000 lbs of  $BF_3$  on site must conform to the requirements of this rule.

Boron Trifluoride (CAS 7637-07-2) is listed on Appendix A to Part 27, "DHS Chemical of Interest", and is subject to the requirements of the DHS Chemical Facilities Anti-Terrorism Standards (CFATS).

Locations must familiarize themselves with and follow any local, state, or federal regulations and guidelines.

# References

- 1. Baumann, Robert C. Radiation-Induced Soft Errors in Advanced Semiconductor Technologies. *IEEE Transactions on Device and Materials Reliability 2005*, 5(3), 305.
- Honeywell Boron Trifluoride Technical Information Brochure. https://www. honeywell-bf3.com/?document=bf3-technicalinformation&download=1 accessed Feb. 6, 2015.
- 3. Jones, W. R. Incorrect Assumption of Boron Trifluoride Hydrolyzation to Hydrogen Fluoride and the Effect on Existing Monitoring Techniques. *SSA Journal*, 1998, 12, 19.
- 4. Waxman, M.; Hilsenrath, J. and Chen, W. T. Compressibility Factor and the Second Virial Coefficient of Boron Trifluoride From 0 to 225°C and for Pressures up to 250 bar. *J. Chem. Phys.* 1973, 58, 3692.

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