Technical Paper

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3M™ 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 Ceradyne 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 Ceradyne 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 Ceradyne product does not violate any third party intellectual rights.

Introduction

Ceradyne, Inc., a 3M company, 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, Ceradyne Inc. is committed to providing our customers with the high quality products for use in the semiconductor industry.

Use of 3M™ 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 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 (σ in barns; 1 barn = 10^{-24} cm^2) 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 ^{10}B (~20 atomic %) and ^{11}B (~80 atomic %). Of these two isotopes, ^{10}B nuclei are several orders of magnitude more likely to capture a neutron than ^{11}B nuclei for most neutron energies (Figure 1).

	High Purity Grade	Ion 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_2/Ar	< 15 ppmv	< 50 ppmv
CO_2	< 15 ppmv	< 25 ppmv
SO_2	< 15 ppmv	< 25 ppmv
HF	< 15 ppmv	< 25 ppmv
SiF_4	< 15 ppmv	–

Table 1

*Calculated from analyzed impurities

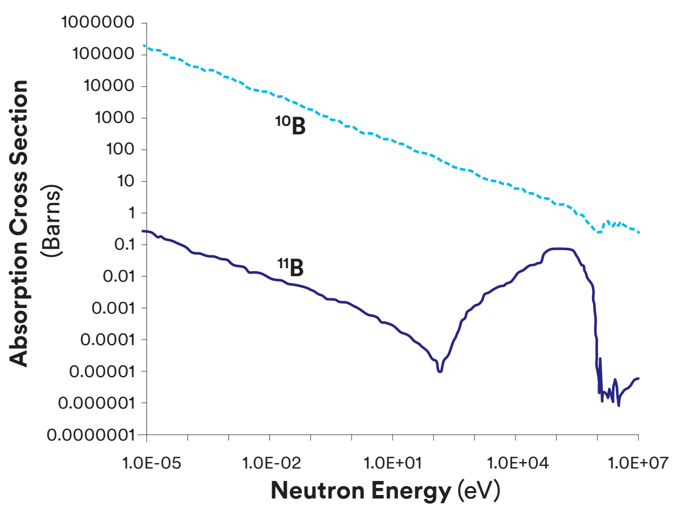

Figure 1: Neutron absorption cross sections for ^{10}B (dashed light blue) and ^{11}B (solid dark blue).

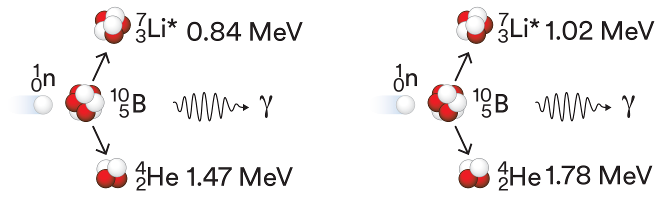

Figure 2: Major (left) and minor (right) reaction outcomes between ^{10}B and a neutron.

After capturing the neutron, ^{10}B will undergo one of two reactions (Figure 2). The major reaction (94%) generates an excited ^{7}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 ^{10}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_{coll}). If Q_{coll} exceeds the critical charge needed to trigger a change in data state (Q_{crit}), 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 10^9 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 ^{11}B isotope is a simple mitigation technique that can be used to limit soft errors due to the reaction of ^{10}B with low energy cosmic neutronic radiation.

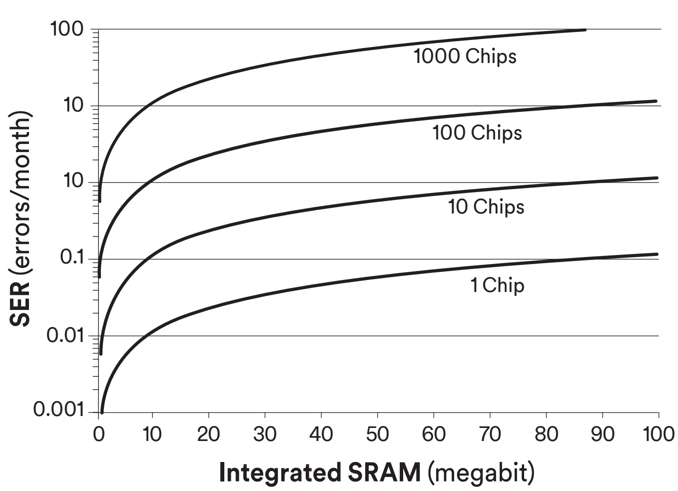

Figure 3: SER as a function of number of chips in the system and the amount of embedded SRAM per chip. From Baumann, Robert C. Radiation-Induced Soft Errors in Advanced Semiconductor Technologies. *IEEE Transactions on Device and Materials Reliability 2005, 5(3),* 305.

Basic Physical Properties of $^{11}BF_3$*

(not for specification purposes)

Formula	$^{11}BF_3$
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/cm^3)	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_3. 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

$$\text{Log } P = 5.1066 - 889.6/T \qquad \text{(Equation 1)}$$

The vapor pressure curve is provided in Figure 4.

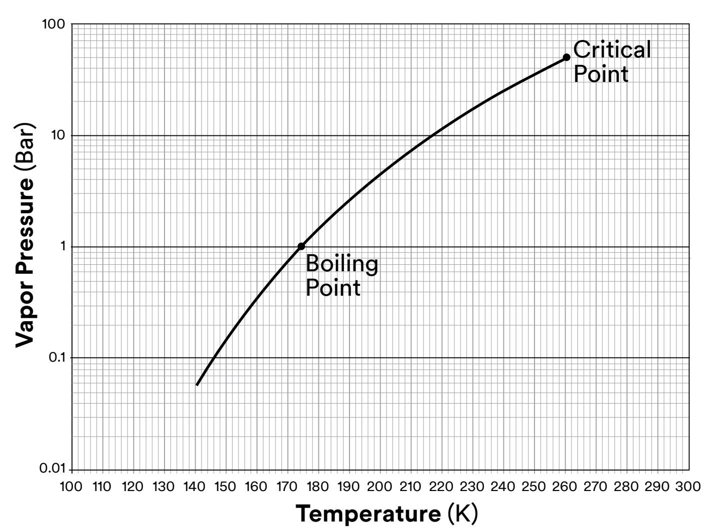

Figure 4: Vapor pressure curve for BF_3. The critical point (-12.3°C, 49.85 bar) and boiling point (-100.4°C) are shown.

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,

$$PV = ZnRT \qquad \text{(Equation 2)}$$

A 43 L cylinder of BF_3 at 25°C (298 K) and 90 bar (~1300 psi) would have a compressibility factor of Z ≈ 0.55 from Figure 5. The number of moles can be found as shown in Equation 3.

$$n = (90\ \text{bar})(43\ \text{L})/(0.55)(0.08314\ \text{L bar mol}^{-1}\ \text{K}^{-1})(298\ \text{K})$$

$$n = 284\ \text{moles} \rightarrow 19.3\ \text{kg of } BF_3 \qquad \text{(Equation 3)}$$

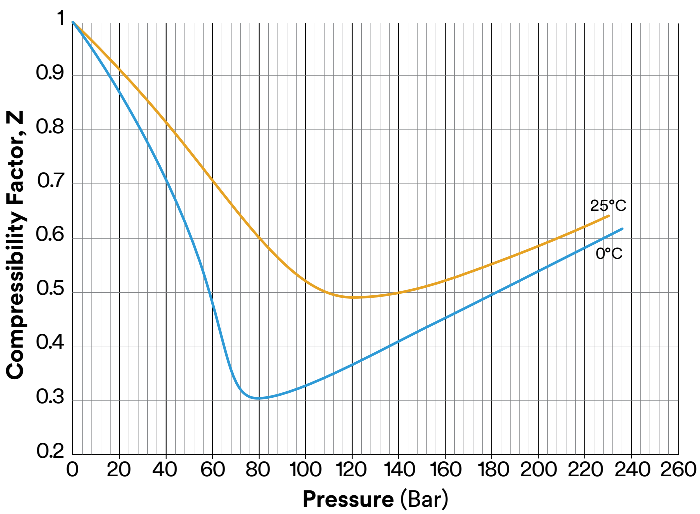

Figure 5: Compressibility factor, Z, for BF_3 as a function of pressure. Data from Waxman, M.; Hilsenrath, J. and Chen, W. T. *J. Chem. Phys.* 1973, 58, 3692-3701 (http://dx.doi.org/10.1063/1.1679720).

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.

$$BF_3{\bullet}2H_2O \rightarrow H_3O^+ + BF_3OH^- \qquad \text{(Reaction 1)}$$

If excess water is present then the very strong acid fluoroboric acid, HBF_4, will be formed through a suspected stepwise removal of fluorine atoms co-producing boric acid. The overall reaction is shown in Reaction 2.

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

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_4 can be further hydrolyzed to produce HF (Reaction 3).

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

BF_3 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-technical-information&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_3 Gas Handling

When designing BF_3 gas handling systems, potential corrosion and the high working pressures must be taken into account. BF_3 cylinders can have a nominal pressure of 1500 psi and systems need to be designed to safely handle this pressure. BF_3 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_3 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_3 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 ✓	Cylinder ✗	Valve ✓	Valve ✗
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; ✓ = acceptable, ✗ = not recommended.

† 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
Elastomers	Butyl Rubber	Not recommended
	Nitrile	Not recommended (weight loss)
	Chloroprene	Not recommended (weight loss)
	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.

		Material†	21°C (mil)	149°C (mil)	Macro Examination
Metals and Alloys		A-285 CS	0.5	6.4	Uniform Corrosion
		SS 304	0.2	74.7	Severe Attack
		SS 316	0.15	10.2	Uniform Corrosion
		SS 347	0.2	12.1	Slight Pitting
		Copper	1.0	29.8	Shallow Pitting
		Monel® 400	0.3	23.9	Severe Attack
		Inconel® 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
		Polypropylene	NR		

Table 5: Corrosion of Common Materials for a 98% BF_3 + 2% H_2O Vapor (168 h 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 NOT 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_3 Handling Systems

All equipment of a BF_3 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_3, there may be some observed fuming when lines are broken and exposed to the atmosphere due to trace amounts of BF_3 being present. If necessary, the line can be flushed of the trace BF_3 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_3 gas must be thoroughly dried using an inert gas stream or dry air prior to the re-introduction of a BF_3 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_3 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_3

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.

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

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 ($NaBF_4$, 40 wt% solubility). Alternatively, potassium hydroxide may be used to form the sparingly soluble potassium tetrafluoroborate salt (KBF_4, 0.5wt% solubility).

Personnel Safety

All personnel involved with and responsible for handling 3M™ 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^3 (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_3 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^{-1} mol^{-1} or 102.7 kJ g^{-1} mol^{-1}) 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_3 hydrates, fluorboric acid, hydroxyfluorobroates) of the reaction of BF_3 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. Ceradyne Inc., a 3M company has further determined that 3M™ 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.
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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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