

SEMI C3.27-94 STANDARD FOR BORON TRIFLUORIDE (BF₃) IN CYLINDERS, 99.0% QUALITY

1 Description

Boron trifluoride is a colorless gas. It fumes in moist air and has a pungent odor. Boron trifluoride is nonflammable and does not support combustion.

2 Specifications

QUALITY: 99.0%

<i>Impurities</i>	<i>Maximum Acceptable Level (ppm)*</i>
Gases not soluble in water	0.94%
Particles	**
Silicon Tetrafluoride (SiF ₄)	200
Sulfur Dioxide (SO ₂)	21
Total Sulfates (SO ₄ ⁻²)	7
TOTAL IMPURITIES	9628

* An analysis of significant figures has not been considered. The number of significant figures will be based on analytical accuracy and the precision of the provided procedure.

** To be determined between supplier and user.

3 Physical Constants (for information only)

	<i>Metric Units</i>	<i>US Units</i>
Molecular weight	67.81	67.81
Boiling point at 1 atm	-100.4°C	-149.0°F
Density of gas at 70°C (21.1°F) and 1 atm	3.076 g/L	0.192 lb/ft ³
Specific gravity of gas	2.7	2.7
Critical pressure	49.2 atm	723 psia
Critical Temperature	-12.25°C	9.95°F

4 Analytical Procedures

4.1 *Sampling Procedures* — Boron trifluoride is sampled separately for water-soluble gases and water-insoluble gases. The water-soluble components are collected in a plastic bottle which contains chopped ice. After the ice melts, weighed aliquots of the solution are taken for individual determinations. The water-insoluble components are collected in a modified gas collection tube.

4.1.1 Apparatus

4.1.1.1 Balance, 1000-g capacity, capable of weighing to 0.01 g.

4.1.1.2 Polyethylene bottles (wide mouth, 1-L capacity) with caps.

4.1.1.3 Tygon^R tubing, 6 mm (1/4 in).

4.1.1.4 PTFE tubing, 6 mm (1/4 in).

4.1.1.5 Valve assembly consisting of a needle valve with a stainless steel needle, a steel bushing and steel coupling with a steel gas cylinder connection; a gas take-off consisting of a steel sleeve from the valve with steel pipe leading to a 61 cm Tygon delivery tube. The delivery tube is a temporary connection and is replaced as needed (see Figure 1).

4.1.1.6 Trap: 1-liter polyethylene bottle fitted with a 2-hole Neoprene stopper and two 4-inch pieces of 1/4" PTFE tubing which do not extend below the stopper by more than 1 inch. The Tygon delivery tube is attached to one of the PTFE tubes in the stopper (Inlet to Trap). A 61 cm length of Tygon tubing is attached to the second piece of PTFE tubing in the stopper (Outlet from Trap). The trap is supported with a clamp and ring stand.

4.1.1.7 Modified gas collecting tube (see Figure 2); 250-mL Pyrex sampling tube (modified with a graduated scale on one end).

4.1.2 Reagents

4.1.2.1 Distilled-water ice, cracked.

4.1.2.2 Nitrogen, dry; 35 ppm water, or less.

4.1.3 Operating Procedure — Water-Insoluble Components

CAUTION: Perform all work in a hood.

4.1.3.1 Prepare the gas-collecting tube by washing with distilled water, rinsing with acetone, then drying in an air oven at 100–125°C. Flush with dry nitrogen for about one minute. Leave filled with dry nitrogen; close the stopcocks.

4.1.3.2 Connect the needle valve assembly apparatus to the cylinder to be tested.

4.1.3.3 Attach the gas-collecting tube to the valve nipple with a short piece of dry Tygon tubing.

4.1.3.4 Open the cylinder valve, open the gas collecting tube stopcocks, and very carefully crack open the needle valve.

4.1.3.5 Purge the tube for 5 minutes with a slow stream of BF₃ gas.

4.1.3.6 Close the needle valve, close the gas-collecting tube stopcocks, and then close the cylinder valve.

4.1.3.7 Proceed with the analysis, after removing the gas-collecting tube from the valve nipple.

4.1.4 *Operating Procedure* — Water Soluble Components

CAUTION: Perform all work in a hood.

4.1.4.1 Before attaching the Tygon delivery tube, purge the sampling valve system for 1 minute with a slow stream of boron trifluoride from the cylinder. Use a separate piece of tubing when purging.

4.1.4.2 Weigh an empty 1-L polyethylene bottle with cap to the nearest 0.01 g. Record this weight as A.

4.1.4.3 Fill the bottle with about 400 g of cracked, distilled-water ice. Re-weigh the bottle with cap to the nearest 0.01 g; record as weight B.

4.1.4.4 A sampling line is used to transfer BF_3 gas from the cylinder to the sample bottle.

4.1.4.5 Connect the Tygon delivery tube from the sampling valve apparatus to the trap. Support the Tygon tubing from the trap outlet; then open the needle valve and adjust until a steady flow of BF_3 is obtained. Insert this Tygon tubing (from the trap outlet) into the polyethylene sample bottle, extending down to the bottom of the bottle. Carefully introduce the BF_3 into the ice until most of the ice is melted, then close the needle valve and remove the Tygon tubing. Remove the sample bottle and cap it. Re-weigh the bottle, cap and contents to the nearest 0.01 g. Record this weight as C.

4.1.4.6 Mix thoroughly by careful inversion until all of the ice melts, being certain to keep the plastic bottle tightly capped so that none of the liquid is lost.

4.1.4.7 Proceed with the determination of water-soluble components, being sure to do the sulfur dioxide first, since opening the bottle repeatedly may result in a considerable loss of sulfur dioxide.

4.1.4.8 Calculations for sample size:

B-A = Grams of Ice (H_2O)

C-A = Weight of Solution (BF_3 Solution)

C-B = Weight of Water-soluble Material (grams of BF_3 gas)

$$\frac{\text{Grams of } \text{BF}_3 \text{ Gas}}{\text{Grams of Solution}} = \frac{C - B}{C - A} = F$$

$$\frac{\text{Grams } \text{BF}_3 \text{ needed for Method}}{F} = \frac{\text{Grams Solution}}{\text{Needed for Method}}$$

4.2 *Air* — This procedure describes the determination of insoluble gases in boron trifluoride gas. A known volume of boron trifluoride gas is absorbed in sodium chloride solution, then any undissolved gases remaining are measured and calculated as % air.

4.2.1 *Method Capabilities* — The range of this method is 0.05% to 2.3% (mole/mole).

4.2.2 *Apparatus*

4.2.2.1 The modified gas collecting tube shown in Section 4.1.1.7, Figure 2.

4.2.2.2 Separatory funnel, 2000-mL Pyrex (with 61-cm Tygon^R tubing and pinch clamp attached to stopcock end).

4.2.2.3 Tygon tubing, 1/4 inch.

4.2.2.4 Balance, capable of weighing 1000 grams to 0.01 gram.

4.2.3 *Reagents* — All reagents used are reagent grade unless otherwise indicated.

4.2.3.1 Water. All water used in the preparation of reagents and in the procedure is either distilled or deionized.

4.2.3.2 Sodium chloride solution. Dissolve 300 g of sodium chloride in 1 liter of water.

4.2.4 *Calibration* — Gas Collecting Tube

4.2.4.1 Weigh the gas collecting tube to the nearest 0.1 g; weight - A, then clamp the tube securely on a ringstand with the calibrated stem up.

4.2.4.2 Fill the tube with distilled water and allow it to stand with the top stopcock open until it has reached room temperature.

4.2.4.3 Record the room temperature to the nearest 0.01°C; temperature - °C.

4.2.4.4 Close the top stopcock. Drain and dry both of the outer stems. Weigh the water-filled collecting tube to the nearest 0.1 g; weight - B.

4.2.4.5 The net weight of the water in the tube (B-A) in grams is equal to the volume of the tube in milliliters, corrected for the density of water at temperature °C. Determine this volume as follows:

$$\text{Volume in mL} = \frac{(B - A)}{D - E}$$

where D - Density (g/mL) of water at temperature °C (from Table 1).

Table 1 Density vs T°C for Water

Temp. °C	Density g/mL
20	0.99823
21	0.99802
22	780
23	756
24	732
25	0.99707
26	681
27	654
28	626
29	597
30	567

4.2.5 Procedure

4.2.5.1 Obtain the sample of BF₃ gas in the gas collecting tube. See Section 4.1.3 for sampling details.

4.2.5.2 Fill the separatory funnel with sodium chloride solution. Attach Tygon tubing and a pinch clamp to the funnel.

4.2.5.3 With a small dropper, fill the glass tubing leading to the stopcock opposite the calibrated end of the gas collecting tube with the salt solution. (Note: the gas collecting tube is inverted at this point.)

4.2.5.4 Open the separatory funnel stopcock and the pinch clamp on the Tygon tubing. Hold the tubing upright and when it completely fills with NaCl solution, slip the tubing onto the end of the gas collecting tube which was previously filled in step 4.2.5.3. (See Note 1.)

4.2.5.5 Hold the gas collecting tube vertical, with the calibrated end up and the entire collecting tube below the liquid level of the separatory funnel.

4.2.5.6 Open the lower stopcock of the gas collecting tube carefully and shake the tube to aid in dissolving of the BF₃ gas in the salt solution.

4.2.5.7 Bring the liquid level in the gas collecting tube equal to the liquid level in the separatory funnel.

4.2.5.8 Read and record the volume of undissolved gas in the upper calibrated end of the gas collecting tube and record this volume, to the nearest one-tenth of a milliliter; mL - F. Record the room temperature.

4.2.6 Calculations

4.2.6.1 F = Total milliliters of undissolved gas (from Section 4.2.5.8).

4.2.6.2 E = Total volume of gas collecting tube in mL; see Calibration — Gas Collecting Tube.

4.2.6.3

$$\frac{F}{E} \times 100 = \%air$$

Report to the nearest 0.01%.

4.3 *Sulfur Dioxide* — This procedure describes the determination of sulfur dioxide in boron trifluoride gas. A sample of the solution prepared in Section 4.1.4 is titrated with standard iodine solution to a starch end-point (blue). The sulfur dioxide is determined from the equivalents of iodine used in the titration.

4.3.1 Method Capabilities: The range of this method is 5 ppm to 1500 ppm (mole/mole).

4.3.2 Apparatus

4.3.2.1 Burette, 10 mL, graduated in units of 0.05 mL.

4.3.2.2 Beaker, 250-mL.

4.3.3 *Reagents* — All reagents used are reagent grade unless otherwise specified.

4.3.3.1 Water. All water used in the preparation of reagents and in the procedure is either distilled or deionized.

4.3.3.2 Iodine solution, 0.01 N. Prepare fresh daily and standardize.

4.3.3.3 Starch indicator solution, 10 g/L. Prepare a thin paste of 1 g of soluble starch with water. Pour the paste with constant stirring, into 100 mL of boiling water. Boil for one minute. Allow the solution to cool and add 2.5 g of potassium iodide. Keep the solution in a glass-stoppered bottle. Prepare fresh when the color obtained for end points is violet or red instead of blue. Alternatively, Thyodene dry indicator powder may be used.

4.3.4 Operating Procedure

4.3.4.1 Calculate the amount of sample (prepared in Section 4.1.4 that contains about 20 grams of BF₃ gas, rounding off to the nearest milliliter. Calculate the grams of BF₃ gas in this volume to the nearest 0.01 g, using the “F” factor in Section 4.1.4.8. Transfer this amount to a 250-mL beaker and add 5 drops of starch indicator.

4.3.4.2 Using a 10-mL burette graduated in 0.05 mL, titrate with iodine to a starch end-point (blue). Stir constantly with a Teflon stir-bar or equivalent. Record the volume of the iodine solution to the nearest 0.01 mL.