

Application Note
Detecting Boron Trifluoride

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1. Introduction

Boron Trifluoride (BF₃) is used in the manufacture of semiconductors. Regardless of concentration, BF₃ is a difficult and dangerous gas, requiring careful attention to safety precautions. In contact with water vapor it reacts to form either hydrogen fluoride (HF) or various BF₃ hydrides. (Sources differ on what the reactions products are.) These eventually degrade to boric acid and fluoboric acid.

The Sensor Electronics SEC3000 and SEC3300 electrochemical gas detectors are both capable of detecting low concentrations of BF₃ (or possibly its reaction products), provided that special steps are taken to insure that the gas actually reaches the point of detection. This application note tells you how to do this.

The SEC3000 (or SEC3300) must be ordered with an HF cell. Because BF₃ is difficult to work with, chlorine (CL₂) is used as a surrogate gas when setting up and calibrating BF₃ units.

2. Adsorption

BF₃ and many other gases stick to various surfaces that they contact. This is called adsorption. When a fresh surface is exposed to the gas, the gas concentration above the surface remains low until the surface becomes coated (also called conditioned). Then the concentration above the surface begins to rise. If the flow of gas is stopped, the reverse happens as the gas is desorbed. The surface slowly gives off the adsorbed gas.

If a gas is passed through a tube and is adsorbed to the inside of the tube, the tube acts as a low-pass filter, slowing down any attempted rapid rise or fall in concentration.

BF₃ seems to stick to just about everything, including pressure regulators, flow meters, tubing, and the surfaces of the sensing instrument, itself. This, and the fact that the BF₃ also degrades in moist air, mean that BF₃ is particularly difficult to detect.

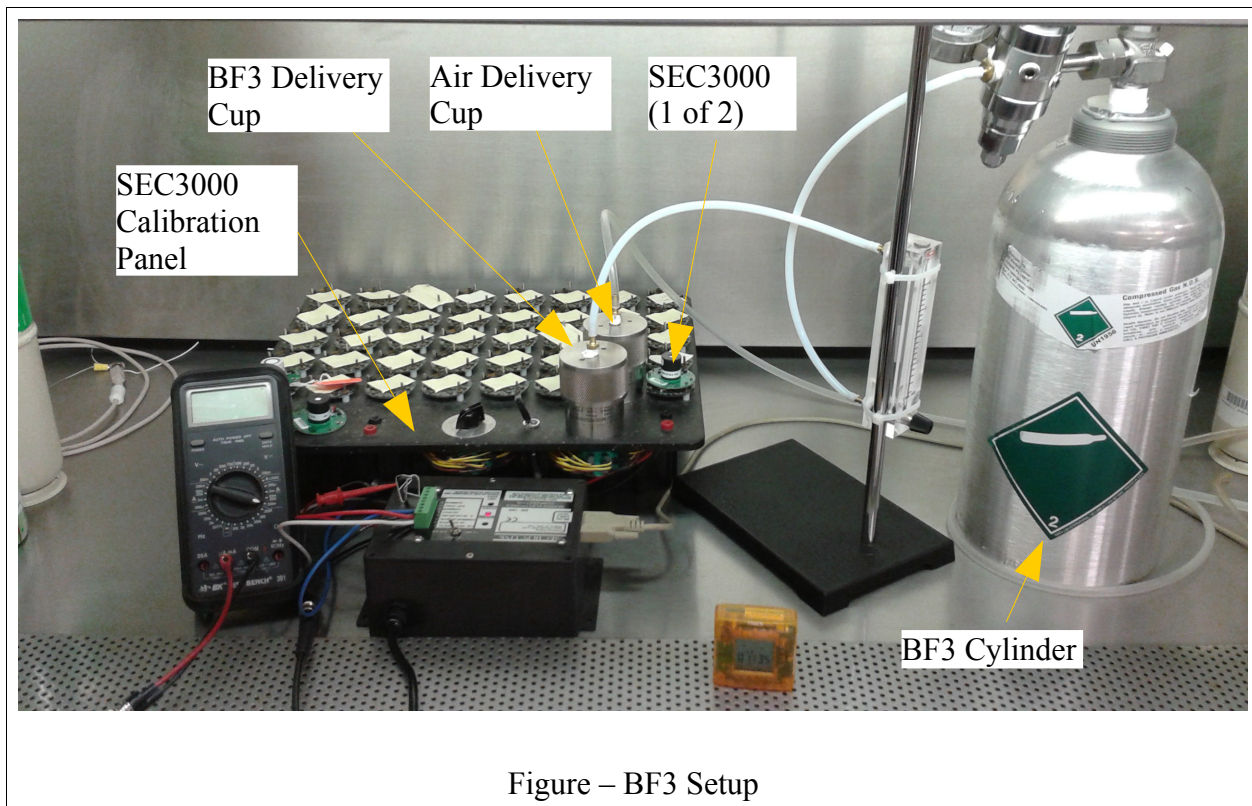
3. BF₃ Measurement Setup

The Sensor Electronics BF₃ measurement setup is shown below. A relatively large BF₃ cylinder is used because of the large amount of gas that is typically used. (A lecture bottle of BF₃ is probably not adequate.) The concentration in this case is 20 ppm with a balance of nitrogen (N₂). The cylinder was ordered from SpecGas [SPEC GAS Inc., 86 Vincent Circle, Warminster, PA 18974, USA].

Both compressed air (tank not shown) and BF₃ are used. The compressed air clears out the BF₃ during measurement of the falling concentration. The remaining components are:

1. Two-stage stainless steel regulator.
2. Dwyer flow regulator having a range of 0 to 4 liter / minute.
3. PTFE tubing (BF₃ only), approximately 2 feet total
4. air delivery cup (brass, steel)
5. BF₃ delivery cup (brass, steel)

Measurements are done in a hood.



Only one of two SEC3000 units used in the tests can be seen in the Figure. The other is covered by the air delivery cup. Also in the Figure, the BF3 delivery cup has been moved to the front of the calibration panel, maintaining approximately the desired bend in the PTFE tubing, which is not very flexible.

A test starts by flowing both air and BF3 at 1 liter per minute. (**Note: Once these gas flows are started, they are maintained throughout the procedure.**) The air delivery cup is placed over the test unit immediately. The BF3 delivery cup is moved to the side (while BF3 flow is maintained). After time T1, the air delivery cup is removed from the test unit and replaced by the BF3 delivery cup. At time T2, the BF3 delivery cup is removed from the test unit and replaced with the air delivery cup. At time T3, the test is complete. The table below shows what is occurring.

TIME	ACTIVITY
$0 < \text{time} < T1$	Unit is being cleared with air, BF3 is flowing in BF3 delivery system.
$T1 < \text{time} < T2$	Unit is being exposed to BF3.
$T2 < \text{time} < T3$	Unit is being cleared with air.

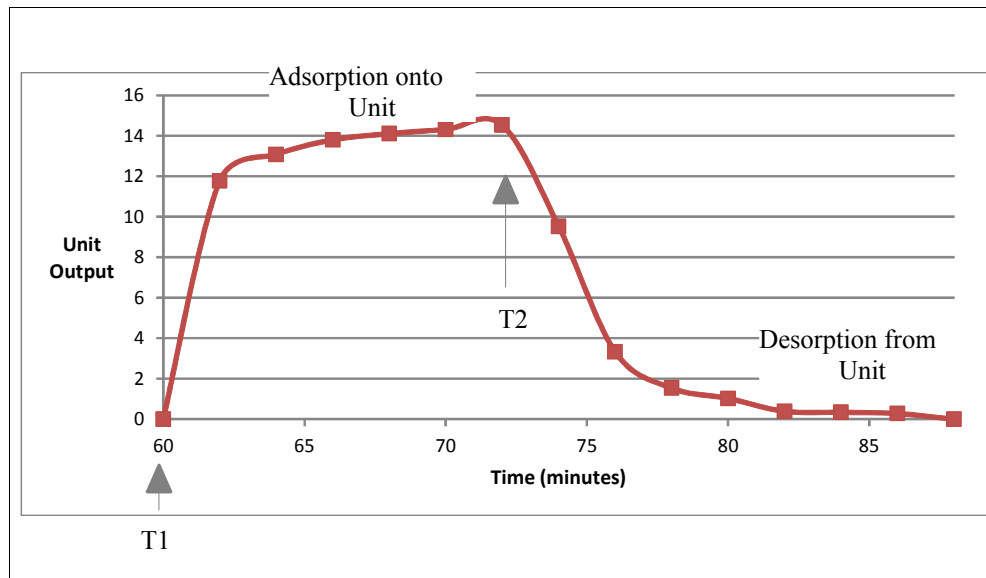
The best responses resulted using

$T1 = 60$ minutes, $T2 = 72$ minutes, $T3 = 84$ minutes.

Notice that $T1 = 60$ minutes is used to provide adequate conditioning of the BF3 delivery chain.

4 Results and Conclusion

Using the stated times for T1, T2, and T3, both test units responded to BF3 and the removal of BF3. On applying BF3 or removing it, the output of the SEC3000 unit starts to change immediately (within 10 second). This is followed by a “fast” change in output, lasting about 1 or 2 minutes. Finally, there are slow ramps indicative of further adsorption or desorption. This is illustrated below for one of the test runs.



We conclude that, although BF3 can be detected using the SEC3000 / SEC3300, the procedure for demonstrating this detection is not straightforward. The user should be aware of problems – especially adsorption – that can occur.