

Sampling and Analysis of Boron Tribromide for Trace  
Metal Contaminants

Phil Clancy<sup>1</sup>, Dan Cowles<sup>1</sup>, Piyamit Chitrathorn<sup>1</sup>, and  
Hugh Gotts<sup>2</sup>

Air Liquide-Balazs Analytical Services  
13546 N. Central Expressway, Dallas, TX 75243<sup>1</sup>,  
46409 Landing Parkway, Fremont, CA 94538<sup>2</sup>

Boron tribromide (BBr<sub>3</sub>) is a colorless, fuming liquid widely used in the synthesis of pharmaceuticals, photovoltaic manufacturing, and as a dopant in semiconductor manufacturing. Particularly in the latter application, the purity of the compound with regard to trace metals contamination is of extreme importance since very small amounts of impurities can have a dramatic effect on the function of semiconductor circuits. Boron tribromide must be analyzed at the parts per billion level by Inductively-couple Plasma Mass-spectrometry (ICP-MS) to ensure that the material is sufficiently pure for use in semiconductor production.

The sampling and analysis of BBr<sub>3</sub> are made difficult due to the fact that the material reacts with moisture in the air, forming boron oxides and hydrogen bromide, a very corrosive acid. The vapors produced are quite toxic, and human exposure must be avoided. Consequently, BBr<sub>3</sub> must be sampled and prepared for analysis in the inert atmosphere of a glove box.

In semiconductor processing, BBr<sub>3</sub> is delivered to the wafer surface as a vapor. BBr<sub>3</sub> is commonly supplied in quartz vessels ('bubblers') that contain both liquid and vapor phases. An inert carrier-gas, such as nitrogen, bubbles through the liquid and carries dilute BBr<sub>3</sub> vapor to the process tool. Interestingly, it is *liquid-phase* BBr<sub>3</sub> that is submitted to the laboratory for analysis, primarily due to its ease of sampling. However, the concentrations of metal contaminants in the liquid and vapor phases can be quite different. Since it is usually the case that the vapor phase contains less metals contamination than the liquid phase, the analysis of the liquid phase may not accurately reflect the actual concentrations of contaminants reaching the wafer.

In our Laboratory we have explored several approaches to the sampling and analysis of BBr<sub>3</sub>. Results will be presented describing procedures for sampling both the liquid and vapor phases. Analytical results will be presented comparing the trace metals concentrations of the two phases. Sample preparation of liquid BBr<sub>3</sub> has also been investigated. Results will be presented comparing direct hydrolysis and dilution as compared to evaporation and re-suspension of the residue for analysis. Finally, analytical problems associated with the high boron and bromide matrix remaining after sample preparation will be discussed. Methods for coping with this difficult matrix will be described.