The Determination of Diborane \((B_2H_6)\) in Gas Mixtures

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With 1 Figure

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In manufacture of electronic devices, diborane gas mixtures are often used to produce \(p\)-doped silicon layers, by thermal decomposition of diborane-silane-hydrogen\(^1\). Diborane-argon and diborane-hydrogen mixtures are delivered in various concentrations by many suppliers.

As diborane is a very unstable compound, which decomposes in the presence of water or oxygen, (which are always present as traces in the gas container) the diborane concentration in the gas mixture has to be monitored. Existing methods for the determination of diborane are infrared spectroscopy, mass spectroscopy and gas chromatography. All these methods require expensive equipment. In the following, a simple photometric method is described, which uses the boron-carminic acid complex\(^2,3\).

**Experimental**

*Procedure:* Carefully dry a 1000-ml gas pipette (made of quartz to avoid any boron contamination) (Fig. 1) at \(110^\circ\)C and close it immediately. Flush the pipette for 5 min with a dry nitrogen stream (velocity 10—20 l/min) to expel the last traces of humidity. Add diborane-argon mixture (2—3 l/min) and stop the nitrogen stream successively. Allow the diborane-argon mixture to stream through the gas pipette for 5 min. Close valve (b) while diborane-argon is still on flowing. Close valve (a) and disconnect from the gas supply.
With the pipette in a horizontal position, open one of the valves for a short time to bring the pressure to atmospheric. The corrected volume $V$ can easily be calculated by the Boyle-Mariotte law.

Rinse the reservoir $B$ with water to remove any traces of boric acid which might have been formed on the walls from interaction of the diborane-argon mixture with traces of adherent humidity, during the flushing. Bring the pipette to the vertical position and fill the reservoir with 5 ml of demineralized water. Rinse the outside of the pipette with acetone to cool it until a slight underpressure is produced within the gas pipette. Open valve (b), allowing the water from reservoir $B$ to be sucked into the pipette. The diborane in the pipette is thereby quantitatively hydrolysed to boric acid. After 10 min transfer the solution to a 50-ml volumetric flask, rinsing the pipette 3 or 4 times with a few ml of water to remove any last traces of boric acid. Add the rinses to the solution in the 50-ml flask.

**Chemical Determination of the Boric Acid as the Carminic Acid Complex**

Transfer an aliquot part of the solution, containing 5—40 $\mu$g of boron, into a platinum dish, add 2 ml of 1% sodium hydroxide solution and evaporate the solution to dryness. Cool, add 2 ml of demineralized water, 2 drops of concentrated hydrochloric acid and 10 ml of concentrated sulphuric acid. Later, when the solution has reached room temperature again, add 10 ml of a 0.025% carminic