

paper, the carbohydrates being shown as dark spots on a similarly stained background. This background stain can be removed<sup>1</sup>, but the process is both time-consuming and cumbersome.

For many years Benedict's solution has been used to show the presence of reducing sugars in urine, a positive reaction being obtained in solutions containing less than 0.1 per cent glucose. This solution has been used in this laboratory to develop the spots of reducing sugars in chromatograms run to differentiate galactose and glucose in urines containing sugar. The sugars show up as bright orange spots on a pale blue to white background. The intensity of the spot is directly proportional to the concentration of the carbohydrate, and can be used to give a rough estimate of concentration. Two solvents have been used to prepare the chromatograms. These are butanol/acetic acid/water and water-saturated phenol. This last is used in an atmosphere of ammonia. The solutions are allowed to run in descending chromatography for 24 hr.; 0.01 ml. of standard solutions of 1 per cent glucose and galactose are spotted on the base line. The urines are shaken for 20 min. with 'Amberlite IR-120' (H) resin and for a further 20 min. with 'Amberlite IRA-400' (OH) resin to remove cations and anions which cause tailing of the spots. 0.02 ml. of urine is spotted on the base line and the chromatogram is prepared as described above. The papers are removed from the developing tanks and dried at 80° C. and dipped into or sprayed with Benedict's solution. I find dipping more effective as it gives a more even distribution of the solution. The chromatograms are then developed by drying at 105° C. for 30 min. The spots are fairly permanent if the papers are kept in the dark after evaluation.

This technique has been used successfully for a wide variety of reducing sugars.

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### A New Silicon Boride, SiB<sub>4</sub>

THE existence of compounds in the silicon-boron system has been a subject of conjecture for more than fifty years, since the many studies that have been made have produced negative, inconclusive, or contradicting results. Moissan and Stock<sup>1</sup>, in the original work on the system, reported that by fusion of the elements they were able to prepare two compounds, SiB<sub>3</sub> and SiB<sub>6</sub>. The SiB<sub>3</sub> was obtained as black rhombic plates with a density of 2.52 gm./cm.<sup>3</sup>, and the SiB<sub>6</sub> as black irregular crystals with a density of 2.47 gm./cm.<sup>3</sup>. The borides were reported to have high melting points, high hardness, and to be conductors of electricity, properties which prompted future workers to conduct further investigations.

Attempts by Tone<sup>2</sup> and Brewer<sup>3</sup> were unsuccessful. Studies of ternary systems involving silicon and boron<sup>4</sup> were uncertain regarding the silicon-boron binary, although a possible compound was indicated at about 90 per cent boron. Recently, Samsonov and Latysheva<sup>5</sup> have reported the properties of a tetragonal SiB<sub>3</sub> prepared by a method similar to that of Moissan. Their conclusions regarding the identification of the compound are doubtful, however, as Ormont *et al.*<sup>6</sup> have pointed out. Samsonov and

Latysheva also prepared a compound SiB<sub>6</sub>, which was studied by Zhuravlev<sup>7</sup>, who reported a structure isomorphous with that of CaB<sub>6</sub>.

Concurrent with the Russian work, Cline<sup>8</sup> prepared single crystals of SiB<sub>6</sub> and measured many of the physical properties<sup>9</sup>. The crystallographic data<sup>10</sup> indicate that the SiB<sub>6</sub> is orthorhombic and the measured density of 2.43 gm./cm.<sup>3</sup> is much higher than the value of 2.15 gm./cm.<sup>3</sup> reported by Zhuravlev for the cubic phase. Further experiments in the system involving the fusion of SiB<sub>6</sub> and silicon yielded a new phase which occurred as rhombs and hexagonal plates. The crystals are reddish-brown under reflected light and have a measured density (sink-float) of 2.44 gm./cm.<sup>3</sup>. Chemical and spectrographic analyses indicate the formula SiB<sub>4</sub>.

Rotation and Weissenberg zero- and upper-level photographs of single crystals of various forms of the SiB<sub>4</sub> indicate a rhombohedral lattice with  $a = 5.52$  Å and  $\alpha = 69.1^\circ$ . The corresponding hexagonal cell constants are  $A = 6.35$  Å, and  $C = 12.69$  Å. There are three molecules of SiB<sub>4</sub> in the rhombohedral unit cell. The probable space groups are  $R\bar{3}2$ ,  $R\bar{3}m$ , and  $R3m$ . The density calculated from these data is 2.41 gm./cm.<sup>3</sup>.

The physical properties of SiB<sub>4</sub> are under investigation, and these properties, as well as the details of the preparation, will be reported at a later date. Intensities are being measured, and a detailed examination of the structure will be made. SiB<sub>4</sub> appears to have some structural features in common with B<sub>12</sub>C (ref. 11), and with various forms of elemental boron<sup>12,13</sup>, and a comparison of the structures should prove extremely interesting.

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### Products of Decomposition of Lead Azide

PREVIOUS work on the decomposition of alpha-lead azide have been performed at temperatures close to its ignition temperature<sup>1,2</sup>. The assumption has been made that the products of such decompositions have been only lead metal and nitrogen gas. The present communication describes the results of a study on the solid material obtained from the low-temperature thermal decomposition of alpha-lead azide.

The material used was a finely divided form of lead azide, prepared in as pure a form as possible. Crystal