Synthesis of Boron Phosphide and Nitride

A NOVEL technique has been found for the synthesis of boron nitride and phosphide involving thermal decomposition of halide addition compounds. Further work is intended on the reactions involved but the information thus far obtained is felt to warrant a brief, preliminary report for the benefit of other workers in this field.

The addition compound $BCl_3.PCl_5$ was prepared by refluxing phosphorus pentachloride in triethanolamine and passing boron trichloride into the reaction mass. The white product obtained was filtered off, washed with ether and dried *in vacuo* at room temperature. Samples were then sealed into Carius's tubes and heated under their own pressure development to about 300° C. Thermal dissociation occurred with the production of chlorine, some sublimation of the addition compound and deposition of a material on the tube walls. The colour of the deposit varied through the length of the tube and from experiment to experiment, from white through brown to black. On breaking the tube after cooling, chlorine gas escaped; residual addition compound was then decomposed and washed out with water and the film deposit also floated out on to water. The film was very resistant to hydrolysis and thermal decomposition, and could be heated in air to temperatures in excess of 1,000° C. without decomposition or apparent loss of integrity. X-ray and chemical analysis showed the material to be essentially cubic boron phosphide.

Further samples of the film were reheated at 800° C. in a flowing atmosphere of 5 per cent ammonia in nitrogen when even the darkest film became white in colour and phosphine was evolved. X-ray analysis of the product showed it to be cubic boron nitride.

Addition compound was prepared from phosphine and boron tribromide or trichloride. When submitted to direct heating *in vacuo*, thermal dissociation immediately occurred, yielding again a film deposit of boron phosphide, but in better yield than before. Analysis of several specimens prepared under apparently similar conditions showed the product to vary between BP and B_5P_3. A mixture of boron phosphide samples produced by the second reaction was submitted to reaction with dilute ammonia gas and again yielded boron nitride of cubic structure.

Thermal decomposition of $BCl_3.PCl_3$ or $BBr_3.PCl_3$ has not yet been examined, but it is felt that similar results would obtain in these systems.

The reactions involved would appear to be:

$$BCl_3 + PCl_5 \rightarrow BCl_3.PCl_5 \xrightarrow{\Delta} BP + 4Cl_2$$

$$BBr_3 + PH_3 \rightarrow BBr_3.PH_3 \xrightarrow{\Delta} BP + 3HBr$$

$$BP + NH_3 \rightarrow BN + PH_3$$

Addition compounds of boron and phosphorus halides have been known for several years, but the process of their thermal decomposition does not appear to have been investigated before. It would seem possible that this mode of metalloid reaction might also be applicable to other similar systems.

Fuller experimental details shortly will be submitted for publication elsewhere.

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3,4,ω-Trihydroxyacetophenone 3-methyl Ether in Adrenal Extracts

IN the course of the isolation of aldosterone from adrenal gland extracts ('Eucortone', Allen and Hanbury), the English authors detected a compound with properties similar to aldosterone in certain paper and column partition systems. This compound had ultra-violet absorption peaks at about 230 mμ and 280 mμ and gave a positive reaction with the $FeCl_3$–$K_3Fe(CN)_6$ reagent. It therefore seemed probable that it was phenolic[1]. It also gave a blue fluorescence on irradiation with ultra-violet (compound X)[2] and reduced blue tetrazolium at about the same rate as steroids having an α-ketol side chain. The compound ran as though slightly more polar than aldosterone in the Bush B_5 paper system[3] and could be completely separated from the steroid by column chromatography using the same solvent system[2].

Preliminary work[4] on a very small scale led to the conclusion that aldosterone did not absorb maximally at 240 mμ. It now seems likely that this was due to the presence of the phenol as a contaminant. The combined peaks of the phenol and aldosterone at 230, 240 and 280 mμ tend to give a flat absorption curve obscuring the single peak of the steroid. On separation of the phenol from aldosterone on the column, the steroid had maximum absorption[2] at about 240 mμ.

A fairly pure sample of the phenol, which was not crystalline, was sent to the Swiss authors who later, during large-scale isolation work on aldosterone using freshly frozen adrenal glands as source of material, isolated the compound in crystalline form and determined its structure as 3,4,ω-trihydroxyacetophenone 3-methyl ether. This has been confirmed by synthesis. The later work will be reported in detail elsewhere.[5]

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Incorporation of DL-[2-^{14} C] Mevalonic Acid Lactone into Polyisoprene

THE incorporation of DL-3-hydroxy-3-methyl-[2-^{14}C] pentano-5-lactone (DL-[2-^{14}C] mevalonic acid lactone) (MVA) into cholesterol in rat-liver homogenates was first demonstrated by Tavormina, Gibbs and Huff[1]. Subsequent work showing that this lactone is incorporated into squalene[2] and β-carotene[3] supported the view that mevalonic acid, or a derivative containing the same branched carbon atom structure, is directly involved in the biosynthesis of a wide range of polyisoprenoid compounds. Park and Bonner[4] showed that when MVA is incubated