Experimental

To a mixture of 9.0 g. (0.506 mole) of phenanthrene in 75 ml. of glacial acetic acid was added 45 ml. of 50% hydrogen peroxide (0.792 mole). On warming slowly, the white paste went into solution. At 60–70°, the exothermic reaction commenced and the deep red solution was kept at a slow reflux for two hours. On refrigeration, 8.0 g. of yellow-white needles crystallized. These were collected and dissolved in warm caustic. After filtering the undissolved quinone, diphenic acid was precipitated as a white powder by acidification of the filtrate with concd. hydrochloric acid. Additional diphenic acid was recovered from the mother liquor by adding it slowly with stirring into warm caustic to decompose the excess peroxide and any peracetic acid formed *in situ*. The solution was treated with charcoal and filtered, and the filtrate was acidified.

The total yield of phenanthrenequinone was 0.5 g. (4%), m.p. 204–206°; of diphenic acid was 8.3 g. (68%), m.p. 229–230°.

A mixed melting point of the latter with a known sample of diphenic acid gave no depression.

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Addition Compound of Boron Tribromide and Trimethylamine

BY ROBERT C. OSTHOFF, CHARLES A. BROWN AND FRANK H. CLARKE

In view of the fact that the trimethylamine addition compounds of boron trifluoride[1] and boron trichloride[2] have been isolated and characterized, it is surprising that the similar ammine of boron tribromide, $Br_3BN(CH_3)_3$, has not been reported previously.

Johnson[3] attempted preparation of $Br_3BN(CH_3)_3$ by the introduction of gaseous trimethylamine into a solution of boron tribromide in carbon tetrachloride, but he was unable to isolate any product which could be characterized. However, Johnson did report the addition compounds of boron tribromide with triethylamine, dimethylaniline and pyridine.

The authors were able to prepare the stable white solid compound of boron tribromide and trimethylamine by direct combination. This compound further illustrates the ability of the boron halides to form stable coördinate covalent bonds with simple tertiary amines.

The following method for the study of the reaction was used. In a typical run tensiometrically pure Eastman Kodak Co. "White Label" trimethylamine (2.106×10^{-3} mole) was introduced into a previously evacuated reaction bulb by means of a compensating gas buret. The pressure of the completely vaporized sample was determined at room temperature and the trimethylamine was "frozen out" with liquid nitrogen. Successive small weighed amounts of boron tribromide[4] (vapor pressure at 23° obsd. 61 mm.; calcd. 62 mm.) were added, the reaction bulb warmed to 25° and the pressure determined following each addition.

The pressures were plotted against mole ratio, $BBr_3/N(CH_3)_3$ giving a straight line (see Fig. 1) which intersected the mole ratio axis at a value of one, corresponding to the formation of $Br_3BN(CH_3)_3$. Continued addition of boron tribromide showed formation of no other compound, and gave only the expected increase in pressure.

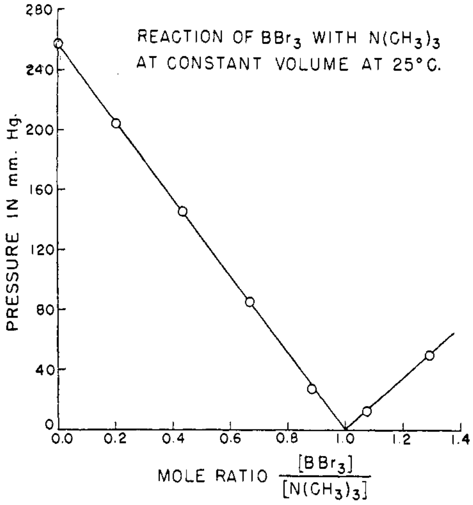

Fig. 1.

The white solid obtained from the reaction was found to be stable in air. It crystallized as long white needles from benzene, melting at 238–240° with some decomposition. The substance was soluble in benzene, chloroform and carbon tetrachloride and insoluble in water at 10°.

Anal. Calcd. for $Br_3BN(CH_3)_3$: C, 11.62; H, 2.90; N, 4.52; Br, 77.55. Found: C, 11.77; H, 2.98; N, 4.79; Br, 78.25.

Cryoscopic measurements in anhydrous benzene gave a molecular weight value of 321 (calcd. for $Br_3BN(CH_3)$ 310).

The infrared spectrum of the compound in chloroform solution was obtained with a Baird spectrophotometer. The principal absorption bands in the range of 2–16 mμ were: 3.19, 6.75, 6.87, 9.00, 10.46 and 12.22 mμ.

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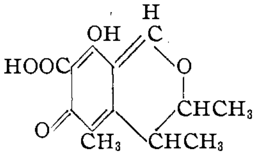
The Preparation of 6-Hydroxyisochroman[1]

BY L. SCHIELER[2] AND R. D. SPRENGER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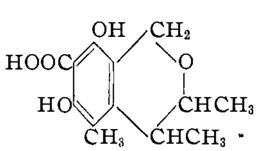
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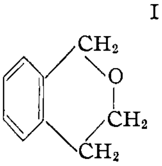
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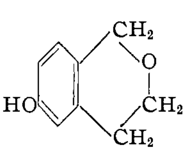
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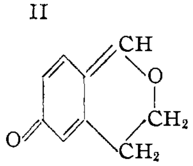
V

Recent investigations[3,4,5] have shown that the antibiotic citrinin has the structure (I). Dihydrocitrinin (II) which has been obtained by the catalytic reduction of citrinin may thus be considered a derivative of isochroman (III). The desirability of

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