Further work is in progress on the stereochemistry of the deoxystreptamine and neosamine B fragments.

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THE PREPARATION OF A NEW BORON HYDRIDE $B_{18}H_{22}$

Sir:

We wish to report the synthesis of a new boron hydride which probably contains more boron atoms per molecule than any boron hydride yet isolated in quantity. At the present time we prefer the formulation $B_{18}H_{22}$ for this hydride although the difficulty in obtaining precise hydrogen analyses in a molecule of this size makes the hydrogen content questionable. A complete structural analysis is currently underway in the laboratory of Prof. William N. Lipscomb.

The oxidation of the $B_{10}H_{10}^{-2}$ ion[1–5] with aqueous ferric chloride has been reported[6] to produce an ion, $B_{20}H_{18}^{-2}$ (m.p. triethylammonium salt, 173–174°, I). An ethanolic solution of I was passed through an acidic ion exchange resin and the resulting solution was concentrated to a yellow syrup at steam bath temperature in the air or under reduced pressure in a vacuum system. The addition of water to a diethyl ether solution of the concentrate results in rapid hydrolysis accompanied by the evolution of hydrogen. The hydride was obtained by evaporation of the solvent followed by recrystallization from cyclohexane and sublimation. Yields of up to 60% have been obtained. Preliminary work indicates that a least one other new hydride is also produced. The purified hydride is stable in the air and melts at 177–178.5° without decomposition. *Anal.* Calcd. for $B_{18}H_{22}$: B, 89.95; H, 10.15. Found. B, 89.11; H, 10.44. A more precise analysis for hydrogen was carried out by the thermal decomposition of a weighed sample to the elements at 900°[7] (mole H_2 calcd. for $B_{18}H_{22}$, 2.18×10^{-3}. Found. 2.21×10^{-3}). Ebullioscopic molecular weights averaged 212.7 (calcd., 216.8). Titration with aqueous hydroxide ion revealed that the presumed $B_{18}H_{22}$ was a strong monoprotic acid (equiv. wt. 219.5). Unit cell dimensions and an accurate density determination[8] fixes the molecular weight at 216 ± 1. This value confirms the B_{18} formulation. The derived anion is bright yellow in color and has been separated as the triethylammonium and tetramethylammonium salts.

The infrared spectrum of the hydride exhibits an intense terminal B–H stretching band at 2850 cm.$^{-1}$ and shows B–H–B bridge absorption at 1950 cm.$^{-1}$. Extremely complex skeletal absorptions are present at longer wave lengths, a fact which suggests low symmetry in the hydride. Solutions of the hydride in hydrocarbon solvents exhibit a purple fluorescence. Three ultraviolet absorptions are observed: $(\lambda^{m\mu}_{max})_1/\epsilon_{max}$) 332/6,560; 273.5/3,560 and 217/15,900. The anion derived from the hydride has two major absorption bands in the ultraviolet: 352/5,950 and 216/11,600.

The B^{11} nuclear magnetic resonance spectrum of the hydride is complex and has not been resolved. Further work is in progress.

Acknowledgment.—The authors are indebted to Prof. William N. Lipscomb for information regarding the $B_{20}H_{18}^{-2}$ ion received prior to publication and the X-ray molecular weight value reported herein. This investigation was supported by Army Ordnance Contract No. DA-01-021-ORD-11878 with the Rohm and Haas Company.

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(8) Private communication of results obtained by P. G. Simpson, R. Lewin and W. N. Lipscomb.

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CHEMISTRY OF THE NEOMYCINS. X.[1]

NEOMYCINS B AND C

Sir:

Structures of the fragments of neomycins B and C—the unitary deoxystreptamine,[1,2,3] neosamine C,[4,5] neosamine B (incomplete stereochemistry),[6] and D-ribose[7]; the binary neamine[1] and neobiosamine B[6,8]—have been established in earlier investigations, and the stereochemistry of neosamine

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(3) Professor R. U. Lemieux (personal communication) recently has confirmed by n.m.r. studies the all-*trans* stereochemistry[1] of deoxystreptamine.

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(8) Earlier[4] the linkage between neosamine C and D-ribose was assigned to the C-2 carbon atom of ribose, from the 2-mole periodate uptake of methyl N,N,-dibenzoylneobiosaminide C. As is demonstrated in the present communication, the C-2 assignment was in error, for reasons not yet clear; it will be discussed in the full paper.