useful material for the recovery of a large number of heavy metals which form insoluble sulfides.

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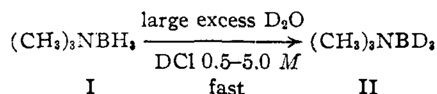
A RAPID AND QUANTITATIVE EXCHANGE OF THE BORON HYDROGENS IN TRIMETHYLAMINE BORANE WITH D_2O[1]

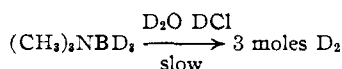
Sir:

Close examination of the slow acidic hydrolysis of trimethylamine borane[2–4] and the most noteworthy absence of a boron–hydrogen kinetic isotope effect[3] has demonstrated now that the boron hydride hydrogens are *exchanging* with the protons of the solvent.



$$\underset{\text{I}}{(CH_3)_3NBH_3} \xrightarrow[\text{DCl 0.5–5.0 } M \text{ fast}]{\text{large excess } D_2O} \underset{\text{II}}{(CH_3)_3NBD_3}$$

The homogeneous reaction of I in dilute deuteriochloric acid first produces II which can be extracted with ether or allowed to hydrolyze slowly to produce pure deuterium gas.



$$(CH_3)_3NBD_3 \xrightarrow[\text{slow}]{D_2O \text{ DCl}} 3 \text{ moles } D_2$$

In keeping with these data the hydrolysis of trimethylamine borane-d_3 in hydrochloric acid produces only hydrogen gas.

No other simple derivatives of diborane have been reported to exchange with heavy water.[5] As I has been used to reduce carbonyl compounds and hydroborate olefins,[6–8] the exchange reaction now allows convenient reductive deuteriations using heavy water as the source of deuterium. Norcamphor, benzophenone, cyclohexanone and acetone have been reduced to the α-deuterio alcohols using II and boron trifluoride etherate.[7] Deuterioborations have been performed in refluxing toluene.[8] Trimethylamine amine borane-d_3 also has been converted into sodium borodeuteride using sodium methoxide in diglyme.

Large amounts of trimethylamine-d_3 are obtained readily using this procedure: sulfuryl chloride (0.50 ml.) is vigorously stirred for twenty minutes with 20 ml. of heavy water. Trimethylamine borane (Callery Chemical Co., 1.000 g., 13.9 mmole) dissolved in fifty ml. of ether is vigorously stirred with the acidic heavy water at 25°. The exchange reaction is followed by infrared analysis. The extent of hydrolysis is determined manometrically. After six hours the deuterium content of the amine borane is 98% while less than 6% has hydrolyzed. The ether layer is dried over potassium carbonate.[9] Evaporation *in vacuo* leaves a white residue which is sublimed. The product weighs 0.997 g. (m.p. 94°, undepressed with I, 98.1% deuterium by mass spectrum).

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(6) E. C. Ashby, *ibid.*, **81**, 4791 (1959).

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(9) This solution can be used directly since the amount of II can be calculated from the amount of gas produced.

These new procedures should greatly facilitate the synthesis of compounds labeled with deuterium or tritium with known stereochemistry to aid in the elucidation of reaction mechanisms.

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ISOLATION OF ANTIBODY BY MEANS OF AN IMMUNOLOGICAL SPECIFIC ADSORBENT

Sir:

As the immunochemist strives for a better understanding of the physicochemical factors involved in antigen–antibody interactions, the need for purified reactants becomes apparent. In his search he has devised a number of non-specific and specific techniques to obtain purified preparations of antibody. The non-specific methods are based on the fractionation of serum to provide a purified preparation of γ-globulin, and include such methods as ammonium sulfate[1,2] or alcohol fractionation,[3] electrophoresis[4] and column chromatography on DEAE-cellulose.[5] Although these techniques provide fairly pure γ-globulin preparations the specific immune γ-globulin desired may be only a small percentage of the total protein in the purified preparation. Hence, one obtains a good yield but low purity with respect to antibody.

The specific methods of antibody purification involve the removal of antibody by precipitation with the specific soluble antigen or reaction of antiserum with antigen in some insoluble state, and subsequent dissociation of the antibody from the precipitate by one of several methods.[6–9] Many of these specific methods depend upon some special property (*i.e.*, insolubility of the antigen in high salt concentration, mercurial salts, etc.) of the particular antigen–antibody system and therefore limit their applicability. The more general method of alkaline dissociation of antigen–antibody precipitates often leads to denaturation of the antibody, and particularily upon prolonged contact with the antibody. The method of acid dissociation[10] of antigen–antibody precipitates at pH 3.0 has been used by many investigators, and seems to be the most applicable. However, the soluble protein antigens must be rendered insoluble by some other procedure such as

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