built up of bundles of smaller particles. The X-ray powder diffraction lines are sharpened but otherwise the pattern remains the same. A very small fraction had transformed to α-Fe_2O_3.

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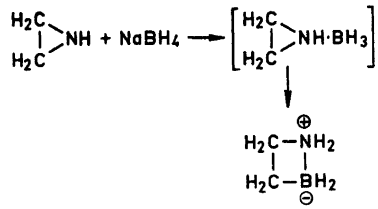
Cyclic 2-Aminoethylborane (1,2-Azaboretidine), a Product from the Reaction between Aziridine and Sodium Borohydride

STIG ÅKERFELDT
and MÄNA HELLSTRÖM

Pharmaceutical Chemistry Section, Research Institute of National Defence, Dept 1, Sundbyberg 4, Sweden

Addition compounds of the structure $RNH_2 \cdot BH_3$ or $R_2NH \cdot BH_3$ are strongly reducing and are thus of interest as potential radical scavengers and radioprotective agents. They are easily formed by reaction of alkylamine salts with borohydride. When aziridine was reacted with sodium borohydride in moist tetrahydrofuran, however, another type of substance was obtained.

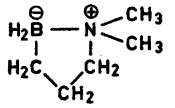
Elemental analysis showed the substance to have the composition C_2H_8BN. NMR spectra in $CDCl_3$ showed two methylene group signals at τ = 7.6 and 8.1 ppm, respectively (relative TMS as external reference). This excludes the substance from being the addition compound between aziridine and borane, which would be expected to give only one methylene signal. Thus evidence had been obtained for the following reaction



IR spectra in chloroform and KCl showed strong absorption at 1168 cm^{-1} (BH_2 scissoring [2]) and at 2330 cm^{-1} (BH stretching [2]). Furthermore CH_2-absorption at 3020 cm^{-1} indicated the cyclic nature of the isolated compound. Absorption at 1440 cm^{-1} suggested the presence of $-CH_2-N^+$.[3] The absorption at about 1630 cm^{-1} is very weak thus confirming the presence of a secondary amine.

Further investigations of this and similar compounds will be published in a forthcoming paper.

Adams and Poholsky [4] have shown that N,N-dimethylallylamine and trimethylamine borane react in toluene to give a five membered ring, similar in structure to the one isolated during this work, namely 1,1-dimethyl-1,2-azaborolidine:



This compound apparently exists as the monomer.[4]

Experiments later revealed that 1,2-azaboretidine is obtained in the absence of water when acetic acid is used to decompose the sodium borohydride. This method appears to be the best one for preparations on a larger scale.

The compound is relatively toxic, having an LD_{50} of 40 mg/kg in mice. It lacks radioprotective properties when tested in mice.

Experimental. 1) Synthesis in the presence of water. 0.8 g (20 mmole) of $NaBH_4$, 3 ml of water, 5 ml of tetrahydrofuran and 0.5 ml (10 mmole) of aziridine were refluxed for 40 min, whereafter the reaction mixture was extracted with about 50 ml of ether. The ether phase was separated, dried over sodium sulfate

and evaporated. The crystalline substance obtained was recrystallized from benzene-cyclohexane. About 0.4 g (70 %) of needle shaped crystals, m.p. 47–48°, were thus obtained. For analysis see below.

2) Synthesis in the presence of acetic acid. 2.3 ml (40 mmole) of acetic acid dissolved in 25 ml of tetrahydrofuran were slowly added under stirring during 30 min to a mixture of 1.6 g (42 mmole) of sodium borohydride and 2.0 ml (39 mmole) of aziridine in 50 ml of tetrahydrofuran. The temperature was maintained below 25°. The reaction mixture was then stirred for 3 h under dry conditions, whereafter the precipitated sodium acetate and unreacted sodium borohydride were filtered off. The filtrate was evaporated to dryness and the crystalline residue was recrystallized from benzene-cyclohexane. Yield 1.6 g (73 %), m.p. 47–48°. (Found: C 42.0; H 14.3; N 24.8; B 19.0. Calc. for C_2H_8BN: C 42.2; H 14.2; N 24.6; B 19.0).

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Chromatography of Conjugated Steroids on Lipophilic Sephadex

J. SJÖVALL and R. VIHKO*

Department of Chemisty, Karolinska Institutet, Stockholm, Sweden

Methylated Sephadex has been used in the separation of various lipid soluble substances.[1-4] In the course of gas chromatographic-mass spectrometric studies of solvolyzable 17-ketosteroids in human serum, chromatography on methylated Sephadex was found to be valuable for the purification of monosulfates of 17-ketosteroids.[5] This communication describes preliminary results on the chromatographic behaviour of conjugated steroids and bile acids on methylated Sephadex.

Methods. Methylated Sephadex G-25 with a content of methoxyl groups of about 36 % was prepared as previously described.[2] Sephadex LH-20 was kindly supplied by Dr. B. Gelotte, Pharmacia, Uppsala, Sweden. The columns were made with 25 g methylated Sephadex or 8 g Sephadex LH-20. Chloroform/methanol, 1:1, containing different electrolytes was prepared by adding one volume of chloroform to a 0.02 M solution of the electrolyte in methanol. If turbid, the solution was filtered. The samples were dissolved in 2–3 ml of the solvent. Solvent flow rate was about 0.4 ml/min. Radioactivity was determined in a Packard Tri-Carb liquid scintillation spectrometer (^{14}C) or a Frieseke-Hoepfner well counter (^{22}Na). Steroids were determined by measuring the absorbancy at the absorption maximum of the sulfuric acid chromogen after 2 h in concentrated sulfuric acid.[6] Elution volumes of the compounds were expressed relative to that of radioactive cholesterol added as a standard to all samples.

We are greatly indebted to Dr. J. F. Becker and Dr. W. Klyne for gifts of 17-ketosteroid glucuronides. Sulfates of 17-ketosteroids were prepared according to Kornel *et al.*[7] Reference samples of the sulfates of androsterone (A) and dehydroepiandrosterone (D) were kindly supplied by Dr. W. Klyne.

Results and discussion. Effect of salts on the elution of steroid conjugates. When 0.1 μg–1.5 mg of ^{14}C-labeled sodium dehydroepiandrosterone sulfate (D–S) was chromatographed on 25 g columns of methylated Sephadex in chloroform/methanol, 1:1, the conjugate appeared as a broad peak with a relative elution volume of 0.70–0.80. When added to serum which was subsequently extracted and chromatographed under the same conditions,[5] the labeled D–S appeared as a narrow peak with a relative elution volume of about 1.50. It appeared possible that serum electrolytes were responsible for the later elution of D–S added to serum (see Refs. 8, 9). Therefore steroid conjugates and bile acid were chromatographed on methylated Sephadex using chloroform/methanol containing different electrolytes as the solvent. As shown in Table 1

* Permanent address: Department of Medical Chemistry, University of Helsinki, Helsinki, Finland. This paper is a preliminary report of part of an investigation to be published by R. Vihko as a doctoral thesis.