

λ_{\max} . 250.0 $m\mu$, $\log E_M$ 4.146 and λ_{\max} . 347.5 $m\mu$, $\log E_M$ 4.014.

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Sodium Borohydride-Disodium Diborane

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We have repeated the preparation of disodium diborane as described by Stock and Laudenklos,¹ and find that the resulting product yields an X-ray powder diffraction pattern identical to that given by them, and also identical with that for sodium borohydride as described by Soldate.² The other data reported for these two compounds are similar, and it seems probable that the compounds are the same and of the composition NaBH_4 .

The disodium diborane was prepared by shaking a sodium amalgam in the presence of diborane gas and the course of the reaction was followed by observing the pressure decrease on a manometer attached to the system. As reported by Stock and Laudenklos, the reaction required several days to go to completion, and more diborane was absorbed than that required by the assumption of the simple reaction to form disodium diborane. At the end of ninety-six hours, however, the amalgam was still absorbing diborane. On plotting the logarithm of the pressure against the time a straight line was obtained for the first twenty-four hours. Mercury was distilled from the product of the reaction under vacuum, and the contents transferred in a dry box filled with nitrogen to a sublimator in which the remainder of the mercury was removed. The resulting residue was loaded into fine capillaries for the X-ray diffraction examination.

Using CuK_α radiation, the samples gave excellent patterns which checked completely the data for NaBH_4 reported by Soldate. It was evident that the same pattern (sodium chloride-type structure) was present in the photographs of Stock and Laudenklos for " $\text{Na}_2\text{B}_2\text{H}_6$ ", but, since no data were given by them for the X-ray work, it was necessary for us to measure these photographs. For " $\text{Na}_2\text{B}_2\text{H}_6$ " the measured $\sin \theta$ values for all of 16 lines were in agreement, within experimental error, with our values obtained with CuK_α .

Assuming that all photographs given by Stock and Laudenklos are for CuK_α , the following tentative conclusions can be drawn:

(1) A. Stock and H. Laudenklos, *Z. anorg. allgem. Chem.*, **233**, 178 (1936).

(2) A. M. Soldate, *This Journal*, **69**, 987 (1947).

(1) The corresponding potassium salts are isomorphous with the sodium compounds. For example, the compound reported as " $\text{K}_2(\text{B}_2\text{H}_6)$ " appears then to be KBH_4 , with an a_0 about 10% larger than that for NaBH_4 .

(2) In addition to the BH_4^- salts, there appears to be another substance present among all the various products. The pattern for this material appears especially prominent in the diffraction patterns of " $\text{K}_2(\text{B}_4\text{H}_8)$ " and " $\text{K}_2(\text{B}_5\text{H}_8)$."

(3) The sublimates of the various borane salts reported by Stock consist principally of NaBH_4 or KBH_4 with some of the unidentified substance as a separate phase.

In view of these findings, it seems to us that a reinvestigation of the reactions of alkali metals with boron hydrides and of the resulting products is called for.

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Preparation of Ethyl β -(Bromomethyl)-cinnamate^{1a}

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The Wohl-Ziegler bromination,² which has been applied successfully to several β -alkyl substituted crotonic esters,^{2,3,4} appeared to afford a method for the synthesis of β -(bromomethyl)-cinnamic esters. Since the methyl group in β -methylcinnamic ester is attached to a C-C double bond conjugated on either side with an unsaturated group, its bromination by N-bromosuccinimide would not be predicted³ to occur with ease. Such was shown to be the case. Under the usual reaction conditions,³ ethyl β -methylcinnamate did not react. Extension of the reaction period to forty-eight hours gave a 39% yield of crude brominated ester. However, addition of catalytic amounts of benzoyl peroxide⁵ caused the reaction to occur smoothly, giving a 50% yield of ethyl β -(bromomethyl)-cinnamate in less than eight hours.

Preliminary experiments have indicated that the brominated ester does not react to any significant extent in Reformatsky-type condensations.⁶

Experimental

Ethyl β -methylcinnamate was prepared according to the method of Lindenbaum.⁷

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(2) Djerassi, *Chem. Rev.*, **43**, 271 (1948).

(3) Ziegler, Späth, Schaaf, Schumann and Winkelmann, *Ann.*, **551**, 80 (1942).

(4) Campbell and Hunt, *J. Chem. Soc.*, 1176 (1947).

(5) Schmid and Karrer, *Helv. Chim. Acta*, **29**, 573 (1946).

(6) Ziegler, Schumann and Winkelmann, *Ann.*, **551**, 120 (1942).

(7) Lindenbaum, *Ber.*, **50**, 1270 (1917).