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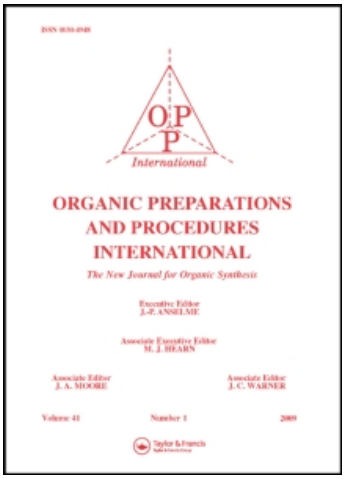

Organic Preparations and Procedures International
Publication details, including instructions for authors and subscription information:
http://www.informaworld.com/smpp/title~content=t902189982

TRIMETHYLBORON

J. Casanova Jr.[a]; H. R. Kiefer[a]; R. E. Williams[a]
[a] Department of Chemistry, California State College, Los Angeles, Los Angeles, California

To cite this Article Casanova Jr., J. , Kiefer, H. R. and Williams, R. E.(1969) 'TRIMETHYLBORON', Organic Preparations and Procedures International, 1: 1, 57 — 59
To link to this Article: DOI: 10.1080/00304946909458349
URL: http://dx.doi.org/10.1080/00304946909458349

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ORGANIC PREPARATIONS AND PROCEDURES 1(1), 57-59 (1969)

TRIMETHYLBORON

J. Casanova, Jr., H. R. Kiefer and R. E. Williams

Department of Chemistry

California State College, Los Angeles

Los Angeles, California 90032

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$$nAl(CH_3)_3 + nB(OCH_3)_3 \longrightarrow nB(CH_3)_3 + [Al(OCH_3)_3]_n \ (?)$$

While trimethylboron has been widely reported and employed in the literature there appears to be only one satisfactory method of synthesis to which frequent reference is made. This is the reaction between methyl magnesium bromide or iodide and boron triflouride etherate.[1] A disadvantage of this Grignard method of synthesis of trimethylboron is the difficulty is separating the product from diethyl ether. An alternate procedure for its preparation which appears not to have been developed extensively depends on the reaction of trimethylaluminum and boron trichloride.[2] To our knowledge trimethylboron is commercially available from only one source.[3]

The procedure described below is a convenient method for the preparation of trimethylboron in yields of 50-55%, starting with readily available materials. It is carried out in an apparatus which permits preparation of large or small quantities of trimethylboron in a short time. Without extensive purification the product is better than 99.9%

pure. This method of preparation is based upon a report by R. Köster[4] which describes the reactions of some alkyl aluminum and alkoxyboron compounds.

Experimental

Trimethylboron - An apparatus which is very convenient for the synthesis is shown in Figure 1. In the description of the experiment reference is made to the figure. Trimethylaluminum[5] (3.0 ml., 30 millimoles) (A) was added dropwise from a dropping funnel (B) to a solution of freshly distilled methyl borate (3.42 ml., 30 millimoles) in 10 ml. of dry diglyme (D) while the temperature was maintained at -18°. The mixture was stirred with a magnetic stirrer and was maintained under an atmosphere of dry nitrogen. A vigorous reaction occurred when the addition

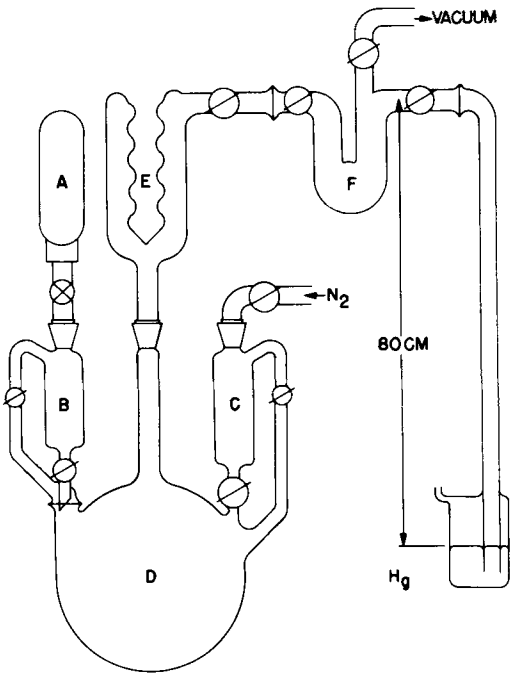

FIG. 1
Apparatus for the Preparation of Trimethylboron

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was complete and the solution was allowed to warm to room temperature. During the warming trimethylboron was noted to reflux from the dry ice condenser (E). The mixture became solid slightly above room temperature. Trimethylboron was trapped at -200° in Trap F. After three hours no further evolution of product was noted. The trap containing product was isolated and evacuated and the material transferred to a high vacuum line. The product was pumped through a trap at -90° and collected at -200°.

In three experiments the yield was 51.7%, 55.5%, and 54.2%. The yield is unaffected by the elimination of diglyme solvent, but the mixture solidifies at a much lower temperature when the solvent is not used. Addition of a second equivalent of trimethylaluminum raises the yield of trimethylboron to 65%.

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An infrared spectrum of this material was identical in all respects with that prepared by a Grignard reaction of methyl magnesium iodide and boron triflouride etherate. A vapor phase chromatogram (retention volume, 310 ml. of He (flow 50 ml./min., 61°, 20' x 3/8" SE-30 silicone gum rubber on 45/60 chromosorb) showed the purity to be in excess of 99.9%.

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(Received August 30, 1968)