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ARGONNE NATIONAL LABORATORY
P. O. Box 299
Lemont, Illinois

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PREPARATION OF METHYL BORATE FROM BORON TRIFLUORIDE

by

Raymond F. Barnes, Herbert Diamond, and Paul R. Fields

Chemistry Division

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A liquid scintillation neutron counter based on observing the scintillations produced by the alpha particles from the nuclear reaction, $B^{10}(n,\alpha)Li^7$, has been reported by Muehlhause and Thomas.¹ Since only boron-10 (18.8% of natural boron) can undergo the (n,α) reaction with low energy neutrons, an enrichment of this isotope would increase the efficiency of this neutron counter. The enriched boron compounds which can be used must be miscible with the fluorescent medium without quenching the scintillations. From a group of such compounds, methyl borate was chosen because of its high boron density and the low neutron capture cross sections of the other elements in the compound.

It was found that the methyl borate had to be prepared from organic compounds which were old with respect to the carbon-14 half-life, since the carbon-14 beta particles could cause an annoying background in the counter. The reagents were tested before using, and several sources of methyl alcohol were found to contain sufficient carbon-14 to be troublesome.

Although several procedures have been reported for the preparation of methyl borate,² none of them utilize boron trifluoride, the available source of enriched boron, as the starting material. The procedure developed here obtained methyl borate by direct reaction of boron trifluoride (as the boron trifluoride-ether complex) with sodium methylate in anhydrous methyl alcohol.

¹C. O. Muehlhause and G. E. Thomas, Jr., *Nucleonics*, 11, 44 (1953).

²(a) Ebelman and Bouquet, *Ann.*, 60, 252 (1846); (b) V. Gasselin, *Ann. chim. phys.*, (7) 3, 22 (1894); (c) T. H. Vaughan, U. S. Patent 2,088,935; C. A. 31, 6675 (1937); (d) H. Schiff, *Ann. Supl.*, 5, 183 (1867); (e) J. J. Etridge and S. Sugden, *J. Chem. Soc.*, 989 (1928); (f) R. P. Calvert and O. L. Thomas, U. S. Patent 1,308,576; C. A., 13, 2262 (1919); (g) S. H. Webster and L. M. Dennis, *J. Am. Chem. Soc.*, 55, 3233 (1933); (h) F. J. Appel, U. S. Patent 2,217,354; C. A., 35, 1071 (1941); (i) E. Schulek and G. Vastagh, *Z. anal. Chem.*, 84, 167 (1931); (j) H. I. Schlesinger, H. C. Brown, D. L. Mayfield, and J. R. Gilbreath, *J. Am. Chem. Soc.*, 75, 213 (1953).

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The methyl borate was separated from the reaction products by distillation as an azeotropic mixture with methyl alcohol. The methyl borate was separated from the methyl alcohol by the lithium chloride salting method.^{2(j)} This method has the advantage of simplicity and freedom from possible scintillation quenching substances.

The sodium methylate and methyl alcohol used were both readily obtainable commercial products. However, to insure the absence of any carbon-14, sodium methylate was made by reacting sodium with anhydrous methyl alcohol. The boron trifluoride-ether complex contained 86 % boron-10.

Three moles (162 gms) of sodium methylate were added with stirring to twelve moles (384 gms) of anhydrous methyl alcohol in a 3-necked liter flask equipped with a Stedmann fractionating column and a partial-condensation, partial-takeoff head. Stirring was effected by means of a magnetic stirrer. One mole (141 gms) of boron trifluoride-ether complex was added with stirring to the mixture in the flask. The mixture was refluxed for several hours and the ether distilled off at 37°C. The methyl borate-methyl alcohol azeotrope was collected from 54-62°C. The azeotrope was assayed for boron content by titration with dilute sodium hydroxide to the phenolphthalein end-point in the presence of mannitol.³ The yield of methyl borate was 65.8 % of the theoretical, of which 1.7 % was found in the ether fraction.

The methyl borate was separated from the azeotrope by the lithium chloride salting method.^{2(j)} The carbon disulfide method^{2(j)} was avoided since an extremely small amount of carbon disulfide in the methyl borate was found to act as a scintillation quenching agent in the counter.

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The advice and information from Anderson Laboratories of Adrian, Michigan is gratefully acknowledged.

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³J. A. M. van Liempt, Rec. trav. chim., 39, 358 (1920).