Böeseken, Vermaas and Küchlin[3] have also determined the equilibrium constant for this complex formation from data on the potentiometric titration of boric acid in the presence of mannitol. They do not specify the temperature at which the measurements were made but report a value of 1.7×10^{-4} for K. Neither this result nor the result obtained by us are in agreement with that reported by Deutsch and Osoling.[1] The latter workers have defined K differently, but their K can be made equivalent to ours by multiplying it by the dissociation constant for boric acid. This gives for K, 0.33×10^{-4}, a value only one-third that obtained by us. Part but certainly not all of this difference can be attributed to the fact that our measurements were made in solutions of much higher ionic strength.

In making our calculations we have assumed that both B^- and BM^- are negligible compared to H^+ and BM_2. We have chosen concentrations of both boric acid and mannitol such that the hydrogen ion concentration is in every case at least ten times what it would be in the absence of the mannitol. Moreover, by using our determined value for the dissociation constant of boric acid and the Deutsch and Osoling value for the equilibrium constant, K_1, of the reaction

$$B^- + M \rightleftarrows BM^-$$

we can calculate the concentrations of B^- and BM^- and correct our equilibrium constant accordingly. We have not reported these revised values, since we strongly question the validity of the value of K_1 as determined by Deutsch and Osoling, and since these corrections would change our values by no more than 10% which is within our known experimental uncertainty.

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[3] Böeseken, Vermaas and Küchlin, *Rec. Trav. Chim.*, **49**, 711 (1930).

RESEARCH LABORATORIES
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NORTH ADAMS, MASSACHUSETTS
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Boron Trifluoride Catalyzed Esterification of *p*-Aminosalicylic Acid

BY JOSEPH J. SCHAEFER AND LEONARD DOUB

The effectiveness of *p*-aminosalicylic acid (4-amino-2-hydroxybenzoic acid) in experimental tuberculosis chemotherapy[1,2] led us to prepare a number of its esters. These compounds have been prepared by reduction of the corresponding nitro esters,[3] but since *p*-aminosalicylic acid has become commercially available, it was desirable to investigate direct esterification. Conventional methods of esterification under various conditions led to very low yields,[4] the primary product being *m*-aminophenol.

Boron trifluoride as an esterification catalyst, following the work of Sowa and Nieuwland,[5] was tried and found to give excellent results. Approximately 70% yields were obtained with several alcohols. In general we used 4.5 moles of boron trifluoride for each mole of *p*-aminosalicyclic acid. In accord with the procedures of Sowa and Nieuwland, this provides one-half mole excess of boron trifluoride over that necessary for complex formation with the functional groups.

Experimental

Preparation of the Esters of 4-Amino-2-hydroxybenzoic Acid.—To a suspension of 153 g. (1.0 mole) of 4-amino-2-hydroxybenzoic acid in 1000 ml. of the anhydrous alcohol, 565 ml. (4.5 moles) of boron trifluoride–ethyl ether complex was added slowly, keeping below 40°. The resulting clear solution, after standing at room temperature for several days, was evaporated under reduced pressure to a thick slurry, and 500 ml. of water was introduced. Solution was effected by adding 10 *N* sodium hydroxide with cooling until alkaline to phenolphthalein. After charcoaling and filtering, solid carbon dioxide was added with agitation to precipitate the ester. This precipitate was removed by filtration and dissolved in dilute hydrochloric acid, charcoaled and filtered. The filtrate was neutralized with potassium bicarbonate. The ester precipitated, was filtered off and crystallized from ethyl alcohol.

ESTERS OF 4-AMINO-2-HYDROXYBENZOIC ACID

Ester	Reaction time, days	Yield, %	M. p., °C.	Empirical formula	Nitrogen, % Calcd.	Nitrogen, % Found
Methyl	10	74	121–122[4]	$C_8H_9NO_3$	7.73	7.88
Ethyl	10	71	114–115[3]	$C_9H_{11}NO_3$	8.38	8.27
Isopropyl	30	75	73–75[3]	$C_{10}H_{13}NO_3$	7.18	7.43 7.40

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[1] Lehmann, *Lancet*, **250**, 15 (1946).

[2] Youmans, *Quart. Bull., Northwestern Univ. Med. School*, **20**, 420 (1946).

[3] *Cf., e. g.*, Jensen, Rosdahl and Ingvorsen, *Acta Chir. Scand.*, **2**, 220 (1948).

[4] Rosdahl, *Svensk Kem. Tid.*, **60**, 12 (1948), reports the preparation of the methyl ester with sulfuric acid in methyl alcohol. In our hands this procedure gave less than 10% yield.

[5] Sowa and Nieuwland, THIS JOURNAL, **58**, 271 (1936).

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The Melting Point of Potassium Hydroxide

BY RALPH P. SEWARD AND KENNETH E. MARTIN

The melting point of potassium hydroxide has been reported as 360° by Hevesy[1] and 380° by Scarpa.[2] A determination of the melting point of potassium hydroxide was suggested by the observation that a sample which had been heated several hours to remove water was found to remain solid above 400°. The observations which are recorded below indicate the melting point to be 410 ± 1°.

The "reagent" quality potassium hydroxide employed, from titration with standard acid, was found to be 86.5% potassium hydroxide and 1.0% potassium carbonate, which agreed with the maker's analysis. On heating to constant weight at a

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[1] Hevesy, *Z. physik. Chem.*, **73**, 667 (1910).

[2] Scarpa, *Atti Acad. Lincei*, [5] **24**, 745 (1915); *C. A.*, **9**, 2828 (1915).