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PREPARATION OF $H_3B^{10}O_3$ FROM B^{10}

PAUL C. TOMPKINS

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PREPARATION OF $H_3B^{10}O_3$ FROM B^{10}

Paul C. Tompkins

It became necessary to convert a sample of pure B^{10} (Lot # 41 - 891A) obtained from the Physics Division, Oak Ridge National Laboratory to boric acid in connection with one of the programs of the Biology Division. The B^{10} sample had been prepared in the blue crystalline form which is resistant to attack by many of the reagents used to dissolve the amorphous brown form in which elementary boron is often prepared. Since others may wish to use B^{10} in the form of $H_3B^{10}O_3$, it seems worth-while to record the procedure used.

Principle of the Method:

This sample proved to be resistant to solution by concentrated HNO_3, fuming HNO_3, Br_2, and concentrated $H_2SO_4 + K_2S_2O_8$ but was slowly dissolved by long refluxing with aqua regia. The reflux was used to prevent losses of H_3BO_3 by volatilization. The acid was diluted with water and any solid particles removed by filtration. The excess acids were then distilled off and volatilized H_3BO_3 recovered by redistillation. The second distillate was evaporated over a steam bath since any loss of $H_3B^{10}O_3$ by volatility from this solution was considered to represent a negligible fraction of the total material.

It was found that when aqua regia was added to a small quantity of the boron (100 mg.), the oxidation was very slow and the material was refluxed 5 hours in the presence of excess aqua regia (30 ml.) to obtain complete solution. However, when 30 ml. of aqua regia was added to the main sample (10 gms.), at room temperature, a slight reaction started at once. In a period of 5 minutes, the reaction became so violent that it was necessary

to cool the reaction vessel. Apparently, local superheating occurred in the interior of the sample. When this stage was passed, it did not recur on the application of heat to the reaction vessel. The details of the procedure as it was done are given in the flow sheet. Many improvements will be obvious to one who wishes to repeat the preparation.

Flow Sheet:

Step 1. - To 10.7 gm. B^{10} in a 100-ml. round-bottom flask were added 30 ml. of freshly prepared aqua regia (20 cc. conc. HCl + 10 cc. conc. HNO_3). The flask was then connected to a reflux condenser. Reaction started slowly with the evolution of Cl_2 gas which soon came out the top of the condenser. Within 5 minutes the reaction had become so vigorous that the sample had frothed half way up the condenser. The reaction was stopped by running a stream of cold water over the flask for three minutes.

Very little reaction occurred after cooling, so water was poured through the top of the condenser tube until the flask was practically full (ca. 90 ml. vol.). The reaction started again and had to be cooled. However, once it had stopped, no further action was noted even when the solution was brought to a boil. Only a small part of the boron was dissolved; suspension filtered.

Step 2. - Residue-B^{10}: Washed with boiling H_2O; washings combined with the filtrate. Residue and contents of the 100 ml-flask were washed into a 1-litter	Filtrate $H_3B^{10}O_3$ + acid. (By-product) Saved and combined with other

	round-bottom flask. Water distilled off and saved. Condenser set to reflux. Ran 100 ml. freshly prepared aqua regia into the flask in 30-40-ml portions. The violent reaction at each addition was controlled by running a stream of cold water over the flask. When reaction ceased, the mixture was refluxed for 4 hours. By then a heavy sediment of H_3BO_3 crystals caused bumping, so heat was removed and the solution allowed to cool. There was still a residue of B^{10} and a heavy brown powder, presumably an amorphous form of B. 800 ml. H_2O were poured through the condenser tube, and the solution brought to a boil to dissolve the H_3BO_3. The reflux was disconnected and the hot solution was filtered through a medium porosity sintered glass filter.	<u>Filtrate</u> solutions at step 6.
Step 3. -	Residue - Washed back into the 1-liter round-bottom flask with boiling water. About 500 cc. were required to wash the filter and the suction flask. Excess water distilled down to a dry residue. Distillate added to distillate and filtrate from step 2.	**<u>Filtrate</u>** **(Main Product)** **Filtrate was clear but colored yellow.**

60 ml. freshly prepared aqua regia were added and condenser set at reflux. When initial reaction had stopped the mixture was refluxed for 6 hours. All of the heavy blue crystals of B^{10} were dissolved, but a small residue of a fine brown powder remained. 500 ml. of water were poured through the condenser and the solution heated to dissolve the H_3BO_3. The brown residue was filtered onto the same filter as that used at step 2. Filter was not washed.

Step 4. - Filtrate: Combined with filtrate from step 3 and returned to 1-liter round-bottom flask. Acid distilled until H_3BO_3 crystallized in the flask.	Residue discarded.
Step 5. - Crystals ($H_3B^{10}O_3$): The hot solution was poured into a beaker, cooled to room temperature, then stored in the ice box overnight. The $H_3B^{10}O_3$ product was filtered onto a clean, medium porosity sintered glass filter. The vacuum was broken, the crystals suspended 30 seconds in cold water, then sucked dry. Washing repeated and crystals dried by drawing air through them for one-half hour. They were then transferred to a large petri dish and dried 2 days over NaOH.	Distillate. Combined with distillate and filtrate from step 1.

Product # 1A

Step 6. - Filter (step 5) was washed with boiling water. Washings combined with filtrate from Product # 1A (step 5), filtrate from step 1. Excess acid and water distilled off until H_3BO_3 crystals formed in the distilling flask. $H_3B^{10}O_3$ crystallized and washed by same procedure as that used at step 5.

Product # 1B - Added to Product # 1A: Total yield 54.5 gms.

Step 7. - Filtrate and washings from step 6 returned to the flask along with enough of the combined distillates from steps 1 and 5 to make a volume of 800 cc. Acid and water distilled off, with occasional addition of more solution from combined distillates of preceding steps until the entire lot of distillates had been cycled through for the second time. Heating continued until $H_3B^{10}O_3$ started crystallizing and the product removed as before, yielding product # 2A. These crystals were washed once with water and then with 50 ml. of acetone to dry them before being placed in the desiccator over NaOH.

Step 8. - Filtrate, plus washings from step 7: Evaporated to saturated H_3BO_3 over steam bath and a second product removed. (Product 2B)	Crystals: Product 2A
Step 9. - Filtrate and washings from product 2 B were combined with distillates from second cycle and all fractions resulting from preliminary tests and the pilot run on 100 mg. B^{10}. The solutions were evaporated to dryness over the steam bath leaving	Product 2B Total: 8.5 gms.

a dark yellow residue which was redissolved in 75 ml. boiling H_2O and boiled with 100 mg. decolorizing charcoal. The charcoal was removed by filtration, and the excess water evaporated to a volume of 10 ml. The product was crystallized as before, and washed with acetone. The filtrate and washings were discarded.

Product # 3, (3.2 gms.) was a dark yellow color, possibly from the decomposition of the acetone during evaporation with the acid distillates. It was not purified further.

Discussion:

The final yield was apparently greater than 100% due to the addition of fractions from early tests which were not included in the initial weight. It was necessary for the yield to be greater than 95% for this to occur.

About 20% of the product was volatilized during the process of removing the excess acid. Also the solubility of H_3BO_3 in water is appreciable. However, by recycling both distillates and filtrates it was possible to recover practically all of the product.

The preparation is very easy and requires relatively little close attention. However, the extensive refluxing, distillation, evaporation and crystallization steps are time consuming. This preparation took about five days to complete.