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REACTIONS OF BORIC ACID AND BORIC OXIDE WITH
METHYL ETHER-BORON FLUORIDE COMPLEX

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REACTIONS OF BORIC ACID AND BORIC OXIDE WITH
METHYL ETHER-BORON FLUORIDE COMPLEX

I. SUMMARY

During the isotopic exchange investigations at this laboratory, evidence was obtained for a chemical reaction between boric acid and the boron fluoride in methyl ether-boron fluoride complex, and for a similar reaction between boric oxide and the complex. In each case it appeared that a new compound had been formed which was postulated to be boron oxyfluoride with the empirical formula BOF.

Further investigations of these reactions have been carried out, the results of which are presented in this report. In the case of the boric acid reaction, definite proof of the empirical formula BOF for the reaction product has been obtained, and it is indicated that the compound probably exists as a trimer associated with one mol of methyl ether.

In the case of boric oxide, it was found that the rate of formation of (BOF)₃ was slow even at 90°C. Thus it is indicated that unreacted boric oxide may accumulate in the reboilers of the plant fractionation unit. An experiment was carried out in which the rate of reaction of boric oxide with complex was determined.

II. INTRODUCTION

During the isotopic exchange investigations (1) undertaken in connection with the fractionation unit at this laboratory (2), evidence was obtained of a chemical reaction between ortho boric acid and boron fluoride present as the methyl ether complex. A similar reaction was found to occur between boric oxide and boron fluoride in the form of the methyl ether complex.

In the case of boric acid, heating a mixture of acid and methyl ether-boron fluoride complex resulted in the liberation of methyl ether and the formation of a gel upon cooling. In a second experiment a mixture of boric acid of normal isotopic abundance and ether complex enriched in B¹⁰ isotope was contacted for a week or more at room temperature, after which the original constituents could not be recovered and the solid and liquid phases were at isotopic equilibrium. In the case of boric oxide, preliminary experiments indicated that the rate of reaction between the oxide and methyl ether-boron fluoride complex, even at 90°C., was measurably slow. Here also, the reaction product was found to be in isotopic equilibrium with the excess ether complex. The exchange aspects of these reactions are discussed in a previous report (1).

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Since the rate of chemical reaction between the oxide and the methyl ether-boron fluoride complex was indicated to be measurably slow, and since isotopic exchange between the product of the reaction and the excess ether complex has been indicated to be rapid and complete, it follows that the rate of chemical reaction effectively controls the rate at which isotopic exchange can occur. Thus, the actual point at which such exchange would occur in the unit will depend upon the rate of reaction between the oxide present and the ether complex. Further, since the boric oxide is a secondary product of thermal decomposition, it will probably always be present, at least in small quantities in the fractionation unit. For these reasons, it appeared desirable to determine the rate of reaction between the oxide and the ether complex, although it was realized that such a determination would probably be influenced by the physical form of the oxide and by the presence of traces of impurities, such as water.

The experimental procedure, discussion of results and conclusions for these studies are contained in two sections. Section III covers the reaction of boric acid with methyl ether-boron fluoride complex and Section IV covers the reaction of boric oxide with the complex.

III. REACTION OF BORIC ACID WITH METHYL ETHER-BORON FLUORIDE COMPLEX

Procedure and Results

The procedure followed in carrying out the reaction of ortho boric acid with the ether complex was simply to contact the acid and ether complex in a glass stoppered flask at room temperature. The flask was shaken occasionally. The progress of the reaction could be followed roughly by observing the appearance of the mixture; when crystalline acid was used, it lost its crystalline character and was replaced by a fine white solid. Furthermore, during the course of the reaction the solution remained cloudy and some gas was evolved. The solid was separated by vacuum filtration through a fritted glass crucible and was washed with anhydrous ethyl ether. Except in the case of the first mixture, which was dried in a stream of dry nitrogen, the recovered solids were dried and stored in a vacuum desiccator. Small samples were removed in dry weighing bottles, weighed and analyzed for elements boron and fluorine and for oxygen (hydroxyl groups) using the Karl Fischer analyses. The details of these procedures and the significance of results so obtained are discussed in a previous report (3).

When the recovered solid from experiment A was dissolved in water for analysis, effervescence was observed and an ethereal odor was detected. Since this could have been due to absorption of the ethyl ether employed in the washing step, a second experiment, B, was carried out in which the solid was dried in a vacuum desiccator. This precaution was also followed in experiments C and D. Even though the recovered solid from experiment B had been pumped on for 45 minutes in a vacuum desiccator, effervescence was again observed on mixing with water and an ethereal odor was again detected. This indicated that the ether observed was present as ether of constitution. Therefore, another portion of the solid was analyzed for methyl ether content by the evolution method (3) in which the ether is salted out of an aqueous solution by means of sodium fluoride and the loss in weight determined. This loss in weight is calculated as per cent methyl ether,

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TABLE II
Analytical Results

<u>Sample</u>	<u>Boron millimols per gram</u>	<u>Fluorine millimols per gram</u>	<u>Oxygen millimols per gram</u>	<u>Ether Evolution</u>	<u>Wt. of Solid (grams)</u>
Solid from A	10.5	10.4	13.3	-	4.62
Solid from B	16.9	17.0	18.0	0.165 g/g	-
Solid from C	17.0	17.4	18.2	-	-
Solid from D	-	-	18.3	-	2.43*
Filtrate from A	-	-	3.78**	-	
Filtrate from D	-	-	3.57**	-	
Complex for C	8.70	26.10	-	-	
Filtrate from C	8.85	26.24	-	-	

*Some solid apparently was lost during filtering and washing.
**millimols/cc.

TABLE III
Composition of Solid

Basis: one gram of solid

<u>Sample</u>	<u>W t . i n g r a m s</u>				<u>Total Weight</u>	<u>Per Cent Accounted For</u>
	<u>Boron</u>	<u>Fluorine</u>	<u>Oxygen</u>	<u>Methyl Ether</u>		
Solid A	0.110	0.198	0.213	-	0.521	52
Solid B	0.183	0.323	0.288	0.165	0.959	96
Solid C	0.185	0.331	0.292	0.165*	0.973	97

*Assumed the same as in B

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although it is possible that part of the associated ether could have been ethyl rather than methyl ether. However, the short period of contact at room temperature during washing would appear to allow little chance for substitution, if it could occur.

Since it was possible that the recovered solid might be an equilibrium mixture of several compounds rather than a single compound, a further experiment C was carried out in which the solid product obtained from mixture B was contacted with fresh methyl ether-boron fluoride complex. It appeared certain that under these conditions a change would be observed in the chemical constitution unless a pure compound had been obtained from the original mixture. A fourth and final experiment, D, was carried out in order to obtain further data on the oxygen distribution in this reaction. The data for the various experiments are given in Table I.

TABLE I

Experiment	Quantities (m o l s)		D e s c r i p t i o n		Time of Contact, Days
	Acid	Complex	Acid	Complex	
A	0.081	0.541	Crystalline U.S.P.	Distilled, 50.4% B ¹⁰	25
B	0.404	2.710	Merck, Reagent Powder	Freshly prepared in plant equipment 18.8% B ¹⁰	75
C	(3-4 grams)	1.0	(Recovered Solid from B)	" "	18
D	0.081	0.541	Merck, Reagent Powder	" "	25

The analytical results for the recovered solids are given in Table II. Included also are oxygen determinations on the liquid phase recovered from experiments A and D which are used in making an oxygen balance, and element analyses for the starting methyl ether-boron fluoride complex and the recovered liquid phase from experiment C, from which the solubility of the product of the reaction is calculated. Weight balances from which the constitution of the product of the reaction is determined are presented in Table III. These were obtained from the analyses in Table II.

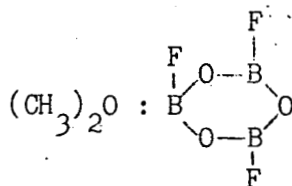
Discussion

From Table II it may be seen that the elements B, F and O are present in equimolar amounts in all of the solids. A weight balance for the solid from A, shown in Table III, however, indicates that these elements account for only 52% of the total composition. In the case of the solid recovered from B, 96 per cent is accounted for when the result of the methyl ether analysis is taken into consideration.

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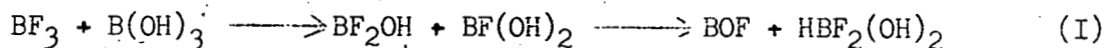
Since the same ratio of elements was obtained for the solids from experiment C as was obtained for experiments A and B, it is reasonably certain that the observed ratio of the elements is correct. Thus, it may be concluded that a compound of empirical formula BOF has been obtained and that it is associated with methyl ether as obtained from a medium of methyl ether-boron fluoride complex.

It is difficult to imagine a compound with the simple structure and low molecular weight corresponding to $F-B=O$, having the properties of the solid obtained. Baumgarten and Bruns (4) reported the conversion of BF_3 to an oxyfluoride, $(BOF)_3$, by reaction with boric anhydride and with borates at 300° and $450^\circ C.$, respectively. Thus, the trimer is more probable and also has an analogy in borazole (5). The amount of methyl ether corresponding to the evolution determination, shown in the weight balance for the solid from B, is 4.5 millimols, which is approximately one mol for every three of BOF. This strongly indicates that no appreciable contamination with ethyl ether occurred during the experiment. The suggested formula, then, is:



Since it appeared likely from this structural formula that association with additional quantities of methyl ether should be possible, the compound was investigated in a calibrated manometric glass system. It was found, however, that the compound showed no measurable vapor pressure and failed to associate with further quantities of methyl ether. It is possible that space relations will not allow further association. Alternatively, it is possible that the conditions were unfavorable, since the addition of methyl ether to the analogous borazole requires heating to $60-70^\circ C.$ for a two-week period under which conditions it ultimately associates with three mols of ether, one for each boron atom (5).

Several reactions of the boron fluoride in the methyl ether complex and boric acid may be written with BOF as a product. These are represented by the following equations, from which the methyl ether is omitted for convenience in writing:



Inspection of the three equations reveals that the equation representing the reaction can be determined by an oxygen balance over the system, recognizing the fact that the oxygen in the methyl ether is unreactive and can therefore be ignored. Determination by means of such a balance is possible because the fraction of $-O-$ added to the system as acid which is recovered in the form of

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insoluble BOF varies in the reactions as follows: (I) 33.3%, (II) 100%, and (III) 50%. Thus, using the analyses and weights of solids from mixtures A and D and the Karl Fischer analyses of the filtrates, corrected for the solubility of BOF in the methyl ether-boron fluoride complex, the distribution of oxygen may be calculated. The results of the calculations are shown in Table IV.

TABLE IV
Oxygen Balance

	A	D	Theoretical
Mols O in, as acid	0.243	0.243	0.243
Mols O recovered, as solid	0.062	0.045	0.072
Mols O recovered, in liquid*	<u>0.189</u>	<u>0.178</u>	<u>0.171</u>
Total recovery of O	0.251	0.223	0.243
Per cent Recovery	103.3	95.7	100.0
Mols O as BOF in solution**	0.009	0.009	0.009
Total mols O as BOF	0.071	0.054	0.081
Per cent of Total O in BOF			
(a) Based on added acid	29.2	22.2	33.3
(b) Based on solid recovered	28.3	24.2	

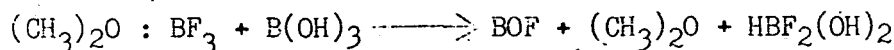
*Recovery of liquid assumed to be 50 cc.

**Calculated from the difference between the boron and fluorine analyses for the filtrate from C and the original complex for C. On the basis of 100 grams of complex, .014 mols of BOF are dissolved.

Thus, reaction (I), theoretically requiring 33.3% of the total -O- to be present in BOF, is supported in preference to those requiring 50 and 100% of the oxygen to be found in the solid product.

Conclusions

1. Methyl ether-boron fluoride complex reacts with boric acid at room temperature to form boron oxyfluoride. In an excess of the complex, the acid is completely converted to the insoluble solid oxyfluoride according to the following simplified equation:



2. Under the conditions of the experiment the product is believed to be a trimer with a cyclic structure, each mol of which is associated with one mol of methyl ether.

3. It is indicated that boric acid cannot exist in a medium of methyl ether-boron fluoride complex.

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IV. REACTION OF BORIC OXIDE WITH METHYL ETHER-BORON FLUORIDE COMPLEX

Procedure and Results

In the case of the reaction between boric oxide and boron fluoride, the formation of boron oxyfluoride (BOF)₃ has already been demonstrated (4). A preliminary experiment, using boric oxide and boron fluoride as the methyl ether complex, revealed that the rate of reaction was slow even at 90°C. Thus, for reasons indicated previously, it was desirable to determine a rate of reaction rather than to attempt to obtain complete reaction. It was realized that such a determination would be subject to many variables and the procedure was, therefore, arbitrary. The boric oxide and excess methyl ether-boron fluoride complex are contacted under fixed conditions and the solid present at the end of the chosen time of contact recovered for analyses. The conditions of the experiment were: (a) a temperature of 100°C.; (b) one and one-half grams of Baker and Adamson reagent boric oxide (18.8% B¹⁰); (c) ten milliliters of enriched methyl ether-boron fluoride complex (82.5 mol % B¹⁰); and (d) constant agitation of the vessel containing the reactants.

The course of the reaction was followed by starting three experiments simultaneously and terminating one after two hours, the second after four hours, and the third after six hours. In each case, the solid material was recovered by filtration and washed thoroughly with absolute ethyl ether. The solid was then dissolved in water, the solution made to volume and aliquots analyzed for elements boron and fluorine (3) and for mol per cent of B¹⁰ isotope. Analytical results are given in Table V.

TABLE V

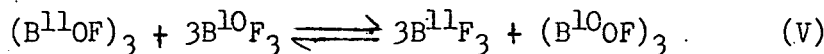
<u>Time (hrs.)</u>	<u>Ratio of F to B (millimols)^z</u>	<u>Mol % B¹⁰</u>
2	0.0335/12.11 = 0.0028	21.0
4	6.04*/10.56 = 0.57	31.3
6	2.68/10.73 = 0.25	34.3

^z The ratio is the significant value since the solid may not have been completely recovered.

* This value for F is obviously out of line.

Discussion

The results obtained may be interpreted by the following reactions:



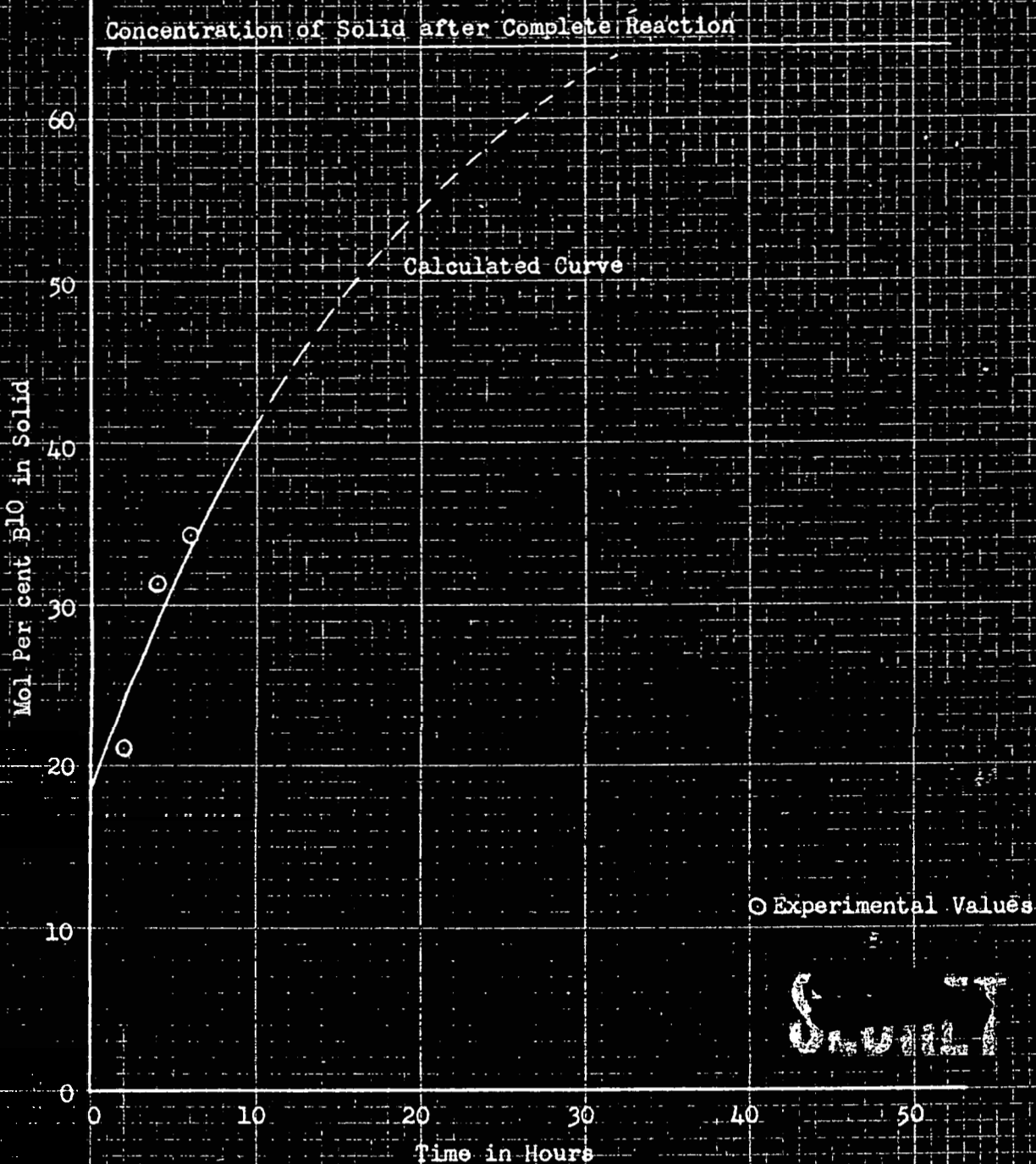
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Figure 1

VARIATION OF B¹⁰ CONCENTRATION IN SOLID PHASE



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The ratio of fluorine to boron from the six-hour test was used to calculate the extent to which reaction (IV) had taken place. In order to account for the high isotopic concentration observed in the solid formed, it was necessary for complete and rapid exchange to have taken place according to reaction (V). Thus, the equilibrium value was the average isotopic concentration for the whole system, excluding the unreacted oxide. These conclusions have already been presented in a previous report (1), and the calculations upon which they are based are given in the appendix. It is evident that reaction (IV) may proceed until all of one or the other reactants has been used up, at which time the $(BOF)_3$ produced will be in complete isotopic equilibrium due to reaction (V). Under the conditions of the experiment it was assumed that the quantity of oxide reacting (millimols/hr.) was independent of the quantity of the oxide present. Then the number of millimols reacting, or of product formed, is equal to kt , where k is the rate constant and t the time in hours.

Using the set of data for the six-hour sample, which are believed to be the most reliable, the rate constant k is calculated. The data used in this calculation are the ratio of fluorine to boron in the recovered solid, and the quantities and isotopic concentrations of the starting materials. The expression for the concentration of B^{10} in the solid phase is developed as a function of t based on the occurrence of reactor IV, followed by complete exchange according to reaction V. It is assumed further that no isotopic exchange occurs directly between the oxide and the complex. The expression obtained is in the form of the ratio of two quadratic equations, and the derivation is given in the appendix. The isotopic concentrations thus calculated are compared with the observed values in Table VI below, which also contains the calculated rate of reaction from the data at six hours contact time. It should be emphasized that the isotopic concentrations determined for the recovered solids are independent of these calculations.

TABLE VI

<u>t (hours)</u>	<u>k</u>	<u>Mol % B¹⁰ in Recovered Solid</u>	
		<u>Experimental</u>	<u>Calculated</u>
2	-	21.0	24.2
4	-	31.3	29.2
6	1.955	34.3	33.7
10	-	-	41.2
15	-	-	48.8
20	-	-	54.6
25	-	-	59.2
30	-	-	62.7

The experimental values are plotted on the curve obtained from the calculated values in Figure 1, from which it may be seen that the agreement obtained lends support to the hypothesis advanced. Thus it appears that isotopic exchange between the $(BOF)_3$ and complex is instantaneous. The theoretical rate curve crosses the line representing complete reaction at about 33 hours, thus indicating the approximate time required for complete reaction under these conditions. However, the value for the time of reaction or the rate may not be reproducible since the degree of agitation, the physical form of the oxide, and

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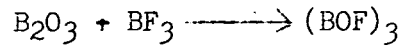
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traces of moisture may have large effects on the reaction rate. This is emphasized by the fact that the solid recovering varied greatly in hardness independently of the length of contact with polymer. Nevertheless, it is believed that an approximate measure of the rate of the reaction between boric oxide and methyl ether-boron fluoride complex has been obtained.

Conclusions

1. A reaction between boric oxide and boron fluoride in methyl ether-boron fluoride complex, believed to be:



results when the two components are contacted at 100°C.

2. The rate of the reaction under the conditions of the experiment is approximately 2.0 millimols of oxyfluoride (monomeric form) formed per hour.

3. As indicated in a previous report, it appears that isotopic exchange between the $(BOF)_3$ and excess methyl ether-boron fluoride complex is instantaneous.

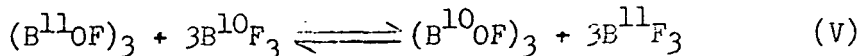
4. The slow rate of this reaction makes possible the concentration of unreacted boric oxide in the reboilers of the plant fractionation unit.

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V. APPENDIX

The following reactions are believed to occur when boric oxide is contacted with methyl ether-boron fluoride complex:



It is postulated that the rate of the reaction IV is limiting and that equilibrium according to reaction V is attained almost instantaneously (1). In the reaction mixture, the oxide and oxyfluoride form a solid phase while the unreacted complex remains as liquid. The rate of the first reaction can be followed by two methods: (1) the F to B ratio in the solid phase, determined by chemical analysis, and (2) the isotopic concentration of B^{10} in the solid phase provided the reactants are of different isotopic concentrations.

According to the first method, the F to B mol ratio is given by:

$$F/B \text{ Ratio} = \frac{kt}{X + 1/3 kt} \quad (1)$$

where k = rate of formation of $(BOF)_3$, millimols/hr. (monomeric form)
 t = time of reaction, hours.
 X = initial quantity of boron in oxide, millimols.

The conditions of the experiment were such that it was desirable to determine the rate of the reaction (IV) by means of equation (1). Then, experimentally determined B^{10} concentrations were compared with calculated concentration determined by using the experimental rate constant. The equation for the B^{10} concentration of the solid phase, C_S , is based on the assumption that the oxide does not undergo isotopic exchange, while the BF_3 and $(BOF)_3$ are in isotopic equilibrium. Then

$$C_S = \frac{\alpha C_o + \beta C_b}{\alpha + \beta} \quad (2)$$

where α = millimols of unreacted oxide
 β = millimols of oxyfluoride, BOF (monomeric form)
 C_o = mol fraction B^{10} in oxide
 C_b = mol fraction B^{10} in oxyfluoride and in complex
 C_S = mol fraction B^{10} in solid phase

The values of α , β and C_b vary as the reaction proceeds and can be determined by knowing the initial values and the reaction rate from equation (1).

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Thus, if X = millimols of boron in oxide initially
 Y = millimols of boron in complex initially
 C_p = mol fraction B^{10} in complex initially

we have

$$C = X - 2/3 kt$$

$$e = kt$$

$$C_b = \frac{Y C_p + 2/3 kt C_o}{Y + 2/3 kt}$$

Then, substituting in (2),

$$C_s = \frac{(X - 2/3 kt) C_o + kt \left(\frac{Y C_p + 2/3 kt C_o}{Y + 2/3 kt} \right)}{X - 2/3 kt + kt} \quad (3)$$

which becomes

$$C_s = \frac{C_o XY + \left[2/3 C_o (X-Y) + C_p Y \right] kt + 2/9 C_o (kt)^2}{XY + 1/3 (2X + Y) kt + 2/9 (kt)^2} \quad (4)$$

The following values of the constants are the quantities and isotopic concentrations of the boric oxide and methyl ether-boron fluoride complex used in the experiments:

- $X = 43$ millimols
- $Y = 109$ millimols
- $C_o = 0.188$
- $C_p = 0.825$

The value of k was obtained from the chemical analysis data for the six hour sample, in which the mol ratio of F/B was found to be 0.25. Thus, $0.25 = \frac{kt}{X + 1/3 kt}$, from which k is found to be 1.955.

Substituting the value for k and the values for the constants in the developed expression, the following is obtained:

$$C_s = \frac{881.2 + 159.7 t + 0.16 t^2}{4687 + 127.1 t + 0.85 t^2} \quad (5)$$

From which the predicted isotopic abundance of the recovered solid may be obtained at any time t . These values were calculated for intervals of two to thirty hours and together with the experimental values are given in Table VI and Figure 1 of this report.

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