are assigned to ν_3, ν_4, and a lattice vibration, respectively. The $S_4N_4 \cdot SbCl_5$ adduct showed absorptions at 345.5 and 177.5 cm^{-1}, and $S_4N_4 \cdot BCl_3 \cdot SbCl_5$ showed absorptions at 357 and 181 cm^{-1}. The small differences in the respective Sb–Cl frequencies in all three cases does not allow a choice between formulations B and C for the diadduct. Absorptions in the B–Cl and B–N region slightly favor formulation B, with structure II. Nöth and Lukas[14] have reported B–Cl frequencies at 849 and 842 cm^{-1} and a B–N absorption at 925 cm^{-1}, for $\{[(CH_3)_2NH]_2BCl_2\}Cl$. Possibly the peaks at 840 and 932 cm^{-1} of the diadduct correspond to the B–Cl and B–N frequencies, respectively, expected for tetravalent boron.

The formation of the mixed adduct was surprising because diadducts of BCl_3 and $SbCl_5$ were not formed even though excess BCl_3 and $SbCl_5$ were present in the preparation of the corresponding monoadducts. Our attempts to form diadducts with BCl_3 and $SbCl_5$ indicated that, if the diadducts formed at all, they were unstable under the reaction conditions.

Although we were unable to convert $S_4N_4 \cdot BCl_3$ into $S_4N_4 \cdot SbCl_5$ directly by treatment with $SbCl_5$, this conversion can be effected by the following successive reactions.

$$S_4N_4 \cdot BCl_3 + SbCl_5 \xrightarrow{CH_2Cl_2} S_4N_4 \cdot BCl_3 \cdot SbCl_5$$

$$S_4N_4 \cdot BCl_3 \cdot SbCl_5 \xrightarrow{85\text{–}90°} S_4N_4 \cdot SbCl_5 + BCl_3$$

Acknowledgment.—This work was supported by the United States Atomic Energy Commission.

(14) H. Nöth and S. Lukas, *Chem. Ber.*, **95**, 1505 (1962).

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Reactions of Boron Trifluoride with Oxides of Nitrogen

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A quantitative study of the reactions between BF_3 and the oxides of nitrogen (N_2O_3, N_2O_4, and N_2O_5) was carried out in the absence of solvents. The final products were mixtures of B_2O_3 and $NOBF_4$ in the case of N_2O_3, and $NOBF_4$ and O_2 with N_2O_4 and NO_2BF_4 in the case of N_2O_5. In the detailed study of the N_2O_4–BF_3 system some of the postulated intermediates were isolated. A mechanism capable of explaining the observations of different authors under divergent conditions is suggested.

The reactions between boron trifluoride and oxides of nitrogen have been the subject of a number of papers.[1–4] Most of these reactions were carried out in the presence of a solvent and below room temperature. The various solid products obtained in these reactions were formulated as NO^+ or NO_2^+ salts of complex boron-containing anions;[1–4] the solids $BF_3 \cdot N_2O_3$, $BF_3 \cdot N_2O_4$, and $BF_3 \cdot N_2O_5$ were regarded,[1–3] respectively, as $NO^+(BF_3NO_2)^-$, $NO_2^+(BF_3NO_2)^-$, and $NO_2^+(BF_3ONO_2)^-$. Recently[5] it has been shown that the above formulations are erroneous and in reality they are, respectively, mixtures of B_2O_3 and $NOBF_4$, B_2O_3 and various proportions of $NOBF_4$ and NO_2BF_4 (depending upon reaction conditions), and B_2O_3 and NO_2BF_4.

Other allied interesting reactions[4,6,7] are those of alkali metal nitrate and nitrite with boron trifluoride in the higher temperature range. Sprague, *et al.*,[4] have carried out the reactions between oxides of nitrogen and BF_3 under various conditions: (a) in the presence of solvents at low temperatures and (b) in the gaseous phase. They also studied the reactions of KNO_3 and $NaNO_2$ with BF_3 at elevated temperatures. In all of the above reactions where the same reactants were used they claimed to have obtained identical compounds, *i.e.*, NO^+ or NO_2^+ salts of complex boron-containing species. However, Scott and Shriver[7] have shown (at 180°) that with $NaNO_3$ the only products obtained were $NaBF_4$, $NOBF_4$, B_2O_3, and O_2, while with nitrite the products depending upon reaction conditions were nitric oxide, nitrate, B_2O_3, $NaBF_4$, and $NOBF_4$. In no case were the compounds claimed by Sprague, *et al.*, obtained.

It is also interesting to note that in the methods of preparation of $NOBF_4$ both N_2O_3[8,9] and N_2O_4[10] have been recommended.

We studied the reactions between BF_3 and oxides of nitrogen at room temperature and in the absence of any solvent. Particular attention was given to N_2O_4. In order to obtain stoichiometry of the reactions, various proportions of the reactants were employed.

The main purpose of this paper is to correlate the

(1) G. B. Bachman and J. Hokama, *J. Am. Chem. Soc.*, **79**, 4370 (1957).

(2) G. B. Bachman, H. Feuer, B. R. Bluestein, and C. M. Vogt, *ibid.*, **77**, 6188 (1955).

(3) G. B. Bachman and J. L. Dever, *ibid.*, **80**, 5871 (1958).

(4) R. W. Sprague, A. B. Garett, and H. H. Sisler, *ibid.*, **82**, 1059 (1960).

(5) J. C. Evans, H. W. Rinn, S. J. Kuhn, and G. A. Olah, *Inorg. Chem.*, **3**, 857 (1964).

(6) P. Baumgarten and W. Burns, *Ber.*, **72**, 1753 (1939).

(7) R. N. Scott and D. F. Shriver, *Inorg. Chem.*, **3**, 857 (1964).

(8) E. Wilke-Dörfurt and G. Balz, *Z. Anorg. Allgem. Chem.*, **159**, 197 (1927).

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(10) S. A. Voznesenskii and P. P. Kurski, *J. Gen. Chem. USSR*, **8**, 524 (1938).

observations of the various authors noted above and to postulate a reaction mechanism capable of explaining all of the observed facts.

Experimental Section

Materials.—All the gases were purchased from Matheson (reagent grade).

Nitric oxide was purified by passing through a −160° bath and condensing in a liquid nitrogen bath and pumping.

Dinitrogen tetroxide was purified by passing through a −45° bath and condensing in a −95° bath.

Dinitrogen trioxide was made by mixing calculated quantities of purified NO and N_2O_4 in a glass storage bulb and was checked by analysis.

Dinitrogen pentoxide was made by distilling KNO_3 (reagent grade, Baker) with concentrated H_2SO_4 (reagent grade, Baker) and fractionating, and then distilling under vacuum the nitric acid thus obtained with P_2O_5 in a current of ozonized oxygen (dry).

Boron trifluoride was purified by passing through a −125° bath. Infrared spectra of the reactants were checked for purity and found satisfactory.

Reaction of Boron Trifluoride and Nitric Oxide.—Various proportions of the reactants were condensed in a glass reactor (200 ml) with a 24/40 female joint and an adapter with a Teflon stopcock (Scientific Glass, JS-4880) with a 24/40 male and ending in a 12/30 male joint. The adapter was joined to the reactor with Py-seal cement. The reactor was then allowed to warm up to room temperature and was kept at this temperature or higher (up to 80°) for various periods of time, but in no case was a reaction observed. Both of the reactants were quantitatively recovered. When BF_3 and NO were condensed together at liquid nitrogen temperature, an orange color was observed, but the color disappeared at about −160° and then reappeared on cooling. This observation was similar to that of Finlay.[11]

Reaction of Boron Trifluoride and Dinitrogen Tetroxide.—In a typical experiment, boron trifluoride (8 mmoles) and N_2O_4 (10 mmoles) were mixed in the manner noted above and were allowed to warm up to room temperature and then to stand for 24 hr. Formation of a white solid was observed at about room temperature. Gaseous products were then admitted to the vacuum line, condensables were collected at −196°, and noncondensables were collected by means of a Toepler pump. After the *PVT* measurements of the fractions, condensables were characterized by infrared and noncondensables by mass spectra. Solid products were identified by taking infrared spectra of the Nujol and Kel-F mulls under proper conditions (drybox under nitrogen atmosphere) and also by chemical means (treating with HCl gas and heating with dry KCl under vacuum).

In order to follow the reaction more closely, the reactants were mixed in a gas cell fitted with NaCl optics.

Reaction of Boron Trifluoride and Dinitrogen Pentoxide.—The above procedure was used, but a reaction temperature of −10° was employed.

Reaction of Boron Trifluoride and Dinitrogen Trioxide.—In a typical experiment 4.50 mmoles of NO, 4.50 mmoles of NO_2, and 7.10 mmoles of BF_3 were used. First NO was condensed in the reactor, and then N_2O_4 (required amount) was condensed on top of it. After a dark blue color developed (N_2O_3), the mixture was warmed to room temperature. Brown fumes were observed, but a blue liquid still remained inside the reactor. Then the mixture was again cooled to −196° and BF_3 was introduced into the reactor. The resulting mixture was warmed to −10° in a salt–ice bath and a white solid formed inside the reactor. The solid was treated as in the previous experiments.

Results

Dinitrogen Tetroxide.—When N_2O_4 (in excess) and BF_3 were mixed in a glass reactor, an immediate formation of a white solid was observed. The infrared spectrum of the solid in Nujol and Kel-F mulls on sodium chloride plates gave a spectrum which was consistent with that of a mixture of $NOBF_4$ and B_2O_3. In no case did we observe a band due to NO_2^+ (in the 2380-cm^{-1} region). Since it is well known[4,5] that both $NOBF_4$ and NO_2BF_4 react with NaCl plates, an independent chemical check was also made. The solid product produced only NOCl when heated under vacuum with dry KCl. With dry hydrogen chloride the solid product gave NOCl, BF_3, and HF, but no NO_2Cl was observed.

The mass spectrum of the noncondensable gas produced in the reaction showed the presence of oxygen only. *PVT* measurements showed that for each mole of BF_3 consumed 0.1875 mole of oxygen was evolved.

An infrared spectrum of the condensable gases at the end of the reaction showed only NO_2.

The over-all reaction consistent with the above observations can be represented as

$$3N_2O_4 + 8BF_3 = B_2O_3 + 6NOBF_4 + {}^3/_2O_2$$

The similarity of this equation with that proposed by Scott and Shriver[7] for $NaNO_3$ and BF_3 should be noted.

In order to see whether the reaction between N_2O_4 and BF_3 did proceed through intermediates, the two reactants were mixed in various proportions in a 10-cm gas cell (Perkin-Elmer) fitted with sodium chloride optics.

On mixing the reactants [BF_3 (3 mmoles) and N_2O_4 (0.5 mmole)] in the cell, an immediate white cloud formation was observed, but no brown gas (N_2O_4) was found. An immediate spectrum showed NO^+ and BF_4^- and, in addition, absorptions in the region of a covalent nitrate (1625, 1300, and 840–860 cm^{-1}). No NO, HNO_3, or NO_2^+ band was observed. On standing, the intensities of NO^+ and BF_4^- bands increased and traces of NO_2^+ band appeared. However, at this point evolution of a brown gas (NO_2) was also observed. A similar observation was made when N_2O_4 was in excess (N_2O_4:BF_3 was 2:1) but in this case no NO_2^+ band was observed. (After pumping off the gases from the cell, a spectrum of the cell windows showed absorption in the covalent nitrate region.[12] When the cell was warmed to 40–50°, a brown gas (NO_2) with small amounts of NOCl and NO_2Cl resulted.) The evolution of N_2O_4 in the above experiment (where BF_3 was in excess) and the absorption in the covalent nitrate region prompted the following experiment.

N_2O_4 (10 mmoles) and BF_3 (8 mmoles) were condensed in a glass reactor and allowed to stand for 0.5 hr after attaining room temperature. The condensables were fractionated in the vacuum line in the usual manner. The products observed at the various temperatures were: −45°, N_2O_5; −95°, N_2O_4; −125°, an unknown; and −196°, BF_3. The fractions were identified by infrared spectra. The unknown in the −125° fraction had absorption bands characteristic of NO^+, a covalent nitrate (1625, 1300, and 840–860

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[11] G. R. Finlay, *J. Chem. Educ.*, **24**, 149 (1947).

[12] C. R. Guibert and M. D. Marshall, *J. Am. Chem. Soc.*, **88**, 189 (1966).

cm^{-1}), and B–F (1490, 1450, and 710 cm^{-1}). Qualitative chemical tests on this fraction showed the presence of boron, fluorine, and nitrogen. On standing, this fraction began to evolve NO_2. Although the evidence is inconclusive, it is postulated that the unknown is a mixture of species of the types $[F_3B\text{–}ONO_2]$[13] and $[B(ONO_2)_4]$.[12]

From the above observations a probable mechanism for the reaction between BF_3 and N_2O_4 is

$$\begin{aligned}
8N_2O_4 + 8BF_3 &= 8NO^+(F_3B\text{–}ONO_2)^- \\
8NO^+(F_3B\text{–}ONO_2)^- &= 6NOBF_4 + 2NO^+[B(ONO_2)_4]^- \\
2NO^+[B(ONO_2)_4]^- &= 2B(ONO_2)_3 + 2N_2O_4 \\
2B(ONO_2)_3 &= B_2O_3 + 3N_2O_5 \\
3N_2O_5 &= 3N_2O_4 + {}^3/_2O_2 \\
\hline
3N_2O_4 + 8BF_3 &= 6NOBF_4 + B_2O_3 + {}^3/_2O_2
\end{aligned}$$

Dinitrogen Trioxide.—In the literature[1] the product of reaction between boron trifluoride and N_2O_3 has been postulated to be $NO^+(BF_3\text{–}NO_2)^-$. However, Evans, *et al.*,[5] have shown that $NOBF_4$ and B_2O_3 are the only final products of the reaction between these two reactants. In a typical experiment as described above (where an excess of N_2O_3 was employed) the solid product contained $NOBF_4$ and B_2O_3 only. In the gaseous fraction only NO and NO_2 (N_2O_3) were found in equal amounts. No noncondensables were present. *PVT* measurements showed that 2.60 mmoles of N_2O_3 was consumed for 7.1 mmoles of BF_3; thus the reaction ratio of BF_3 to N_2O_3 was 2.73 and the following equation (identical with the one proposed by Evans[5]) is implied

$$3N_2O_3 + 8BF_3 = B_2O_3 + 6NOBF_4$$

where the BF_3:N_2O_3 ratio is 2.66.

On treating the solid with excess hydrogen chloride only NOCl, BF_3, and HF(SiF_4) were obtained.

In another experiment an excess of boron trifluoride was employed. Starting with N_2O_3 (2.96 mmoles) and BF_3 (12.10 mmoles) 4.25 mmoles of BF_3 was recovered but no NO or NO_2 was found. Thus 7.85 mmoles of BF_3 reacted with 2.96 mmoles of N_2O_3 and the ratio of BF_3 to N_2O_3 was 2.65. The solid product contained only $NOBF_4$ and B_2O_3. Thus, the above equation is implied for the reaction between BF_3 and N_2O_3.

On carrying the reaction above −10° (room temperature) oxygen was evolved showing that at this temperature N_2O_4 was the reactant.

Dinitrogen Pentoxide.—The reaction[3] between BF_3 and N_2O_5 produced a solid which has been shown by Evans, Rinn, Kuhn, and Olah[5] to be a mixture of NO_2BF_4 and B_2O_3. In a typical experiment 2.1 mmoles of N_2O_5 and 6 mmoles of BF_3 were condensed together in a glass reactor and allowed to stand in a −10° bath for 1 hr. After this period the gaseous phase contained 0.38 mmole of BF_3 and traces of oxygen. Thus the amount of BF_3 consumed was 5.62 mmoles and the ratio of BF_3 to N_2O_5 was 2.67. The solid product gave an infrared spectrum consistent with a mixture of NO_2BF_4 and B_2O_3. Small amounts of $NOBF_4$ were also observed. On treating the solid with dry KCl, NO_2Cl, and small amounts of NOCl were obtained. The presence of $NOBF_4$ and oxygen was probably due to some decomposition[5] of N_2O_5 into N_2O_4 and O_2 and subsequent reaction of N_2O_4 with BF_3. The amounts of oxygen and $NOBF_4$ increased with the rise of reaction temperature as would be expected if the source of the decomposition products were N_2O_5.

An equation consistent with the facts is

$$3N_2O_5 + 8BF_3 = 6NO_2BF_4 + B_2O_3$$

Discussion

Except for the reaction with N_2O_4 our results are substantially in agreement with those of Olah and co-workers.[5] The result of the N_2O_4 reaction agreed with that proposed for $NaNO_3$ and BF_3 by Scott and Shriver.[7] The apparent discrepancy in the N_2O_4 reaction can be explained by the fact that N_2O_5 is one of the intermediates in the reaction and the products obtained will depend upon its behavior. In the method employed by Olah and co-workers it is evident that the formation of NO_2BF_4 resulted from the reaction of N_2O_5 with BF_3. Because of the fact that N_2O_5 decomposes[14] appreciably above 0° and in the presence of light and organic impurities, the formation of NO_2BF_4 will be a function not only of temperature but also of the purity of the reactants and the manner in which the reaction is performed.

Note that when BF_3 was in excess in our experiments with N_2O_4, evidence for the presence of NO_2^+ band was found. It is obvious from the mechanism proposed for N_2O_4 and BF_3 that the formation of NO_2BF_4 will take place along with $NOBF_4$ if proper conditions such as lower temperature, solvent to prevent decomposition of N_2O_5, and an excess of BF_3 are employed. Evans' finding that NO^+ and NO_2^+ tetrafluoroborates were formed in different ratios depending upon reaction conditions can be explained by the different amounts of N_2O_5 decomposing under the prevalent conditions.

On the basis of the above proposed mechanism the reaction between BF_3 and $NaNO_3$ proposed by Scott and Shriver[7] can be explained as

$$\begin{aligned}
8NaNO_3 + 8BF_3 &\longrightarrow 8Na[F_3BONO_2] \\
8Na[F_3BONO_2] &\longrightarrow 6NaBF_4 + 2Na[B(ONO_2)_4] \\
2Na[B(ONO_2)_4] &\longrightarrow 2NaNO_3 + 2B(ONO_2)_3 \\
2B(ONO_2)_3 &\longrightarrow B_2O_3 + 3N_2O_5 \\
3N_2O_5 &\longrightarrow 3N_2O_4 + {}^3/_2O_2 \\
\hline
6NaNO_3 + 8BF_3 &= 6NaBF_4 + B_2O_3 + 3N_2O_4 + {}^3/_2O_2
\end{aligned}$$

and

$$3N_2O_4 + 8BF_3 = 6NOBF_4 + B_2O_3 + {}^3/_2O_2$$

adding and dividing we have

$$3NaNO_3 + 8BF_3 = 3NaBF_4 + 3NOBF_4 + B_2O_3 + {}^3/_2O_2$$

The above equation is identical with the one proposed by Scott and Shriver.

In the above scheme some of the intermediates,

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(13) J. Weidlein and K. Dehnicke, *Chem. Ber.*, **98**, 3053 (1965).

(14) N. V. Sidgwick in "Chemical Elements and their Compounds," Vol. 1, Oxford University Press, London, 1950, p. 691.

such as $[B(ONO_2)_4]^-$, should be isolable in view of recent work.[12] We did find that when $(CH_3)_4NNO_3$ is treated with BF_3 at room temperature for 0.5 hr and then the excess BF_3 is pumped off, the solid gave a spectrum (Irtran plates) which had absorptions in the covalent nitrate region[12] (1582–1626 and 1297–1311 cm^{-1}).

Similarly, the reaction between $NaNO_2$ and BF_3, where the amount of BF_3 is less than the stoichiometric quantity, can be visualized as taking place in the following manner

$$\begin{array}{rcl}
8NaNO_2 + 8BF_3 & \longrightarrow & 8Na[F_3BNO_2] \\
8Na[F_3BNO_2] & \longrightarrow & 6NaBF_4 + 2Na[B(NO_2)_4] \\
2Na[B(NO_2)_4] & \longrightarrow & 2NaNO_2 + 2B(NO_2)_3 \\
2B(NO_2)_3 & \longrightarrow & B_2O_3 + 3N_2O_3 \\
3N_2O_3 & \longrightarrow & 3NO + 3NO_2 \\
3NO_2 + 3NaNO_2 & \longrightarrow & 3NaNO_3 + 3NO \\
\hline
9NaNO_2 + 8BF_3 & = & 6NaBF_4 + 3NaNO_3 + 6NO + B_2O_3
\end{array}$$

The above equation is identical with the one proposed by Scott and Shriver for the above reaction conditions.

Scott and Shriver obtained $NOBF_4$, NO, $NaBF_4$, and small amounts of a noncondensables (not identified) when BF_3 (excess) reacted with $NaNO_2$ (heated to 180°). In this reaction the quantities of the individual gaseous products were not given. This observation can also be explained by the reaction scheme for nitrite (where it was in excess). In this case after the decomposition of N_2O_3 into NO and NO_2, the NO_2 will react with BF_3 forming $NOBF_4$, B_2O_3, and O_2 according to the equation proposed for the reaction between N_2O_4 and BF_3. It can be seen easily that all of the NO formed in the decomposition of N_2O_3 will not be converted into NO_2 as sufficient oxygen is not available. Hence, a trace of oxygen together with NO (major) will be the only gaseous products of this reaction.

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Molecular and Crystal Structure of $B_7C_2H_{11}(CH_3)_2$

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A three-dimensional X-ray diffraction study has yielded a structure of C_s symmetry for $B_7C_2H_{11}(CH_3)_2$ in which the B_7C_2 unit is an icosahedral fragment. In the open face of this fragment there are two adjacent bridge H atoms and two $CH(CH_3)$ groups, as was suggested from chemical and spectroscopic studies. Molecular orbital studies of the analogous $B_7C_2H_{13}$ molecule suggest that axial methylene hydrogen atoms are more positive than equatorial methylene hydrogen atoms. There are four molecules of $B_7C_2H_{11}(CH_3)_2$ in a unit cell having parameters $a = 10.56$, $b = 17.35$, $c = 5.57$ A, and $\beta = 82.4°$. The space group is $P2_1/n$, and the value of $R = \Sigma||F_o| - |F_c||/\Sigma|F_o|$ is 0.08 for the 1146 observed reflections.

The compound $B_7C_2H_{13}$ is produced[1] by oxidation of $B_9C_2H_{11}$ by $K_2Cr_2O_7$ in acetic acid solution. Spectroscopic and chemical evidence suggests[1] that there are two bridge H atoms and two CH_2 groups. In the C,C′-dimethyl derivative, $B_7C_2H_{11}(CH_3)_2$, easily exchanged BHB bridges and CH′ units have been identified by Tebbe, Garrett, and Hawthorne, who have combined this chemical evidence with topological theory and steric information to suggest[1] that these CH′ units are axial. The molecular structure tentatively suggested by them has been confirmed for $B_7C_2H_{11}(CH_3)_2$ as described below. In addition, we have applied a new nonempirical (no experimental parameters) molecular orbital theory[2,3] to $B_7C_2H_{13}$, in which the axial hydrogen atoms are shown to be substantially more positively charged than the equatorial hydrogen atoms of the CH_2 groups in the ground electronic state. Since both CH units of a CH_2 group are terminal, this result may provide a basis for understanding the readily exchangeable axial protons in this molecular species.

Structure Determination

A needlelike crystal, about 0.2 mm in diameter and 0.7 mm in length, was grown by slow sublimation under vacuum near room temperature and was then sealed into a thin-walled glass capillary tube in a drybox. Weissenberg and precession photographs taken with the long axis (c) mounting indicated reciprocal lattice symmetry of C_{2h}, and Al-powder diffraction calibration established unit cell parameters of $a = 10.56 \pm 0.02$, $b = 17.35 \pm 0.02$, $c = 5.57 \pm 0.01$ A, and $\beta = 82.4 \pm 0.2°$. Extinctions of $0k0$ when k is odd and of $h0l$ when $h + l$ is odd indicate the space group $P2_1/n$. If four molecules are placed in the unit cell, the reasonable calculated density of 0.92 g cm^{-3} is obtained.

Relative intensities of 1823 independent reflections were measured on levels hkL for $0 \leqslant L \leqslant 6$ on the Buerger automated X-ray diffractometer. The ω scan

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[1] F. N. Tebbe, P. N. Garrett, and M. F. Hawthorne, *J. Am. Chem. Soc.*, **88**, 607 (1966).

[2] M. D. Newton, F. P. Boer, and W. N. Lipscomb, *ibid.*, **88**, 2353 (1966).

[3] F. P. Boer, M. D. Newton, and W. N. Lipscomb, *ibid.*, **88**, 2361 (1966).