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Note on the structure of dimethylamine–boron trifluoride.

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Complete structure determinations have been reported for the closely related molecular addition compounds of formulae H_3N–BF_3 (Hoard, Geller & Cashin, 1951), H_3CH_2N–BF_3 (Geller & Hoard, 1950), and $(H_3C)_3N$–BF_3 (Geller & Hoard, 1951). The purpose of this note is to report briefly on intensive but unsuccessful efforts to determine the structure of the remaining member of the series, dimethylamine–boron trifluoride, $(H_3C)_3HN$–BF_3.

The monoclinic unit of structure has

$$a = 7{\cdot}50, \quad b = 6{\cdot}06, \quad c = 12{\cdot}25\,\text{A.}, \quad \beta = 102°,$$

and contains four stoichiometric molecules. Nearly all of the data obtainable with Cu $K\alpha$ radiation were provided by complete sets of Weissenberg photographs with oscillation about b and c. Reflections of the type ($h0l$), l odd, and ($0k0$), k odd, are not observed; thus C_{2h}^5–$P2_1/c$ is uniquely indicated as the probable space group. Only a few 'forbidden' ($0k0$) spectra can be looked for, but a 26 hr. exposure on a Buerger precision camera set to bring out (010) and (030) failed to reveal either of them. The assumption of a centrosymmetric space group is further supported by the absence of a measurable pyroelectric effect, the molecule itself possessing a considerable permanent dipole moment.

The apparent wealth of information provided by the data at hand would appear to make the deduction of an approximate structure a comparatively simple problem. Presumably the effective size and shape of the molecule, $(H_3C)_3HN$–BF_3, can be estimated with some accuracy from the work on related compounds. The character of the Patterson section, $P(X, \frac{1}{2}, Z)$, and the short b translation, when considered together with the symmetry of $P2_1/c$, would seem to delimit the region in which such a molecule (the asymmetric unit) could lie. An additional clue to the orientation of the molecule is provided by $P(X, 0, Z)$, which shows one strong peak interpretable as an intramolecular distance, F–F, N–F, or just possibly C–C, between two atoms having very nearly the same y co-ordinate. Nevertheless, all possibilities suggested by these considerations and by other Patterson syntheses have led to gross inconsistencies. It seems not improbable that the true structure is based on Pc, even though the missing ($0k0$) spectra and the absence of a pyroelectric effect would still need to be explained in terms of peculiarities of the atomic arrangement.

With the hope that the method of inequalities (Harker & Kasper, 1948) might be of aid in determining some of the phases, the observed amplitudes were put on an absolute basis (Harker, 1948). As all except two of the unitary structure factors were definitely less than 0·5, the method of inequalities is not very useful in this case (cf. Hughes, 1949).

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References

Geller, S. & Hoard, J. L. (1950). *Acta Cryst.* **3**, 121.
Geller, S. & Hoard, J. L. (1951). *Acta Cryst.* **4**, 399.
Harker, D. (1948). *Amer. Min.* **33**, 762.
Harker, D. & Kasper, J. S. (1948). *Acta Cryst.* **1**, 70.
Hoard, J. L., Geller, S. & Cashin, W. M. (1951). *Acta Cryst.* **4**, 396.
Hughes, E. W. (1949). *Acta Cryst.* **2**, 34.

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A preliminary X-ray investigation of potassium- and ammonium-trioxalatochromate. III.

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The two compounds examined were (1) $K_3[Cr(C_2O_4)_3].xH_2O$ and (2) $(NH_4)_3[Cr(C_2O_4)_3].xH_2O$. Both compounds form fairly hard dark blue crystals which lose water of crystallization upon exposure to normal atmospheric conditions, decomposing into a light blue crystalline powder. During the X-ray examinations of the crystals they were coated with thin films of Perspex to prevent water loss. Single-crystal oscillation and Weissenberg photographs were taken of both compounds, using Cu $K\alpha$ and Mo $K\alpha$ radiation.

(1) $K_3[Cr(C_2O_4)_3].xH_2O$

The potassium salt crystallizes as thick prismatic needles with well-developed {031} prism faces. The crystals are monoclinic, belonging to the class $2/m$–C_{2h}, and the unit-cell dimensions are

$$a = 7{\cdot}71, \quad b = 19{\cdot}74, \quad c = 10{\cdot}40\,\text{A.}; \quad \beta = 108°0'.$$

The axial ratios

$$a:b:3c = 0{\cdot}3906:1:1{\cdot}5805$$

can be compared with those given by Jaeger (1919), namely,

$$c:b:a = 0{\cdot}3917:1:1{\cdot}5733, \quad \beta = 72°\,5\tfrac{2}{3}'.$$

The density of the crystals, determined by the method of flotation, is 2·16 g.cm.$^{-3}$ (cf. Jaeger's value 2·141 g.cm.$^{-3}$). The unit cell therefore contains four of the above formula