Short Communications

Contributions intended for publication under this heading should be expressly so marked; they should not exceed about 500 words; they should be forwarded in the usual way to the appropriate Co-editor; they will be published as speedily as possible; and proofs will not generally be submitted to authors. Publication will be quicker if the contributions are without illustrations.

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Unit cells and space groups for two etherates of sodium tridecahydrodecaborate(1-). By

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The discovery of the salt sodium tridecahydrodecaborate-(1-)($NaB_{10}H_{13}$) by Hough & Edwards (1958) has prompted further investigation of this compound with a view toward determination of its structure by single crystal X-ray methods. Attempts to prepare single crystals of the unsolvated material were unsuccessful due to its ease of hydrolysis and salt like properties. Stable macro-crystalline ethyl and methyl etherates have been prepared and preliminarily studied by single-crystal X-ray diffraction techniques.

Ethyl or methyl ether solutions of decaborane ($B_{10}H_{14}$) were reacted with slight excesses of sodium hydride at room temperature. After filtration to remove excess sodium hydride, excess ether was evaporated slowly to yield residues in which suitable single crystals were found.

By chemical analysis, the crystalline materials have been characterized as the mono-ethyl etherate,

$$NaB_{10}H_{13}\cdot(C_2H_5)_2O\ ,$$

and the bis-methyl etherate,

$$NaB_{10}H_{13}\cdot 2\,(CH_3)_2O\ .$$

Both materials are extremely hygroscopic. The bis-methyl etherate must be stored in an undiluted atmosphere of methyl ether to prevent effervesence of methyl ether.

The mono-ethyl etherate crystals were found to be triclinic, with the following unit-cell parameters:

$$a = 5{\cdot}64 \pm 0{\cdot}02,\ b = 7{\cdot}42 \pm 0{\cdot}02,\ c = 9{\cdot}05 \pm 0{\cdot}03\ \text{Å};$$
$$\alpha = 83^\circ\ 43' \pm 10',\ \beta = 86^\circ\ 39' \pm 10',\ \gamma = 74^\circ\ 0' \pm 10'\ .$$

Assuming one molecule per unit cell, the calculated density is 1·001 g.cm.$^{-3}$. The observed density is 0·983 g.cm.$^{-3}$. The space group is most certainly $P1$.

Crystals of the bis-methyl etherate were found to be monoclinic, space group $P2_1/c$, with unit-cell parameters:

$$a = 11{\cdot}49 \pm 0{\cdot}02,\ b = 29{\cdot}84 \pm 0{\cdot}02,\ c = 9{\cdot}72 \pm 0{\cdot}02\ \text{Å};$$
$$\beta = 94^\circ\ 55' \pm 5'\ .$$

Space considerations indicate that there are eight molecules in the unit cell. The density calculated on this basis is 0·946 g.cm.$^{-3}$.

Weissenberg patterns of the bis-methyl etherate show an abnormal fall-off of reflection intensity with increasing Bragg angle. Very few reflections are observed beyond 90° of 2θ. This suggests high thermal motion and/or disorder, but a Debye–Scherrer pattern obtained at -100 °C. showed no indication of a phase transition.

Further work on these compounds is not anticipated in the near future.

Reference

Hough, W. V. & Edwards, L. J. (1958). Symposium on 'Borax to Boranes,' 133rd American Chemical Society Meeting, San Francisco.

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Crystal structures of the isostructural minerals lazulite, scorzalite and barbosalite.* By

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Lazulite, $MgAl_2(PO_4)_2(OH)_2$, and scorzalite,

$$FeAl_2(PO_4)_2(OH)_2,$$

form a complete isomorphous series (Pecora & Fahey, 1949, 1950). Barbosalite, $FeFe_2(PO_4)_2(OH)_2$, the ferric analog of scorzalite (Lindberg & Pecora, 1954, 1955), is a third member of this isostructural series; no solid solution relations between barbosalite and the lazulite–scorzalite pair have been definitely established.

The present note describes the crystal structures found for the three minerals. Table 1 lists the chemical composition of the crystals used and the crystallographic data found in the current study. Visually estimated intensities obtained with Zr filtered Mo radiation were used for the structure determinations. A trial structure (common to the three minerals) was established by interpretation of the Patterson projections on the planes normal to a, b, and c, and from consideration of the known structure of the closely related tetragonal compound 'iron lazulite', $Fe_{3\cdot5}(PO_4)_2(OH)_2$, (Katz & Lipscomb, 1951). The trial structure was refined for lazulite and scorzalite by successive electron-density projections to yield the atomic parameters shown in Table 2. For

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