Acta Cryst. (1956). **9**, 830

The structure of meyerhofferite, $2CaO.3B_2O_3.7H_2O$, a $P\bar{1}$ crystal, determined by the direct method of Hauptman and Karle*.

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(*Received* 25 *June* 1956)

The preliminary structure of meyerhofferite,

$$2CaO.3B_2O_3.7H_2O,$$

a $P\bar{1}$ crystal, has been obtained through the use of the direct method of structure determination of Hauptman & Karle (1953).

Meyerhofferite is triclinic $P\bar{1}$,

$$a = 6{\cdot}63,\ b = 8{\cdot}35,\ c = 6{\cdot}46\ \text{Å}\ (\text{all}\ \pm 0{\cdot}015\ \text{Å}),$$
$$\alpha = 90°\ 46',\ \beta = 101°\ 59',\ \gamma = 86°\ 55'\ (\text{all}\ \pm 05');$$
$$Z = 1[2CaO.3B_2O_3.7H_2O],$$
$$d_{(\text{calc.})} = 2{\cdot}125\ \text{g.cm.}^{-3},\ d_{(\text{obs.})} = 2{\cdot}120\ \text{g.cm.}^{-3}.$$

The intensity data, estimated visually, were obtained with Mo $K\alpha$ radiation. They consist of 4342 reflections contained within the sphere of radius $s = (\sin\theta)/\lambda = 0{\cdot}9$ Å^{-1}. These data were used to construct the $K(s)$ curve (Karle & Hauptman, 1953), from which the E^2_{hkl}'s were calculated according to the relationship

$$E^2_{hkl} = \frac{K(s)F^2_{hkl}}{\sigma_2}, \quad \text{where} \quad \sigma_2 \sum_{j=1}^{N} = f_j^2(s)\ .$$

Signs of the 2678 non-zero F_{hkl}'s were determined, using all 4342 of the E^2_{hkl}'s.

Initially, 8 *ggg* $(h, k, l = 2n)F_{hkl}$'s were found with $P_+(F\mathbf{h}) \geq 0{\cdot}8$, where

$$P_+(F\mathbf{h}) = \tfrac{1}{2} + 0{\cdot}067|E_{\mathbf{h}}|(E^2-1)_{\mathbf{h}_\mu};\ \ \mathbf{h}/2 = \mathbf{h}_\mu\ .$$

These were used with Σ_3 to fix the signs of 60% of the 317 non-zero *ggg* F_{hkl}'s. The Σ_2 relationship was then applied to determine the signs of all the remaining F_{hkl}'s, as well as to recalculate the *ggg* signs. According to statistical criteria, the signs of 86% of all the non-zero F_{hkl}'s, and of 96% of all those having $|E| \geq 1{\cdot}00$, were fixed by this procedure. A detailed account of the procedure will be published at a later date.

Because of the many demands on our computing facilities, the three-dimensional electron-density function was not calculated. Instead, electron-density projections on the planes normal to the three crystallographic axes were computed. These, considered together, immediately revealed the structure of meyerhofferite in detail. The $\varrho_y(x, z)$ is shown in Fig. 1. The crystal contains polyions consisting of two $BO_2(OH)_2$ tetrahedra and a $BO_2(OH)$ triangle linked to form a ring of composition $[B_3O_3(OH)_5]^{-2}$. Each Ca^{+2} is coordinated by 6 oxygens and 1 water molecule. The formula of meyerhofferite can thus be written $CaB_3O_3(OH)_5.H_2O$. The isolated groups in meyerhofferite are the same elements which condense to form the infinite chains in colemanite, $CaB_3O_4(OH)_3.H_2O$, (Christ, Clark & Evans, 1954):

$$n[B_3O_3(OH)_5]^{-2} = [B_3O_4(OH)_3]_n^{-2n} + nH_2O\ .$$

The discrepancy factors R for this initial structure are entirely satisfactory: for the *h0l* zone $R = 0{\cdot}19$, for the *hk*0 zone $R = 0{\cdot}19$, and for the 0*kl* zone $R = 0{\cdot}21$. The structure is now being refined and will be reported in detail at a later date.

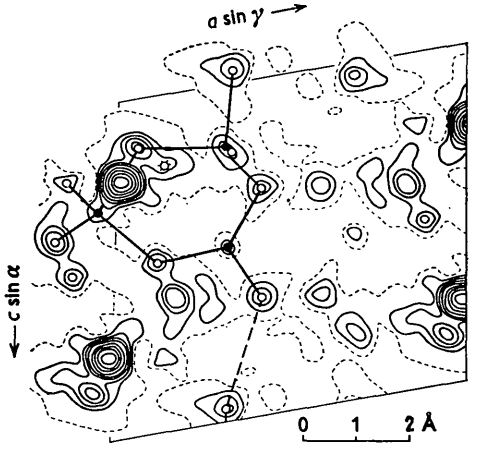

Fig. 1. Electron-density projection $\varrho_y(x, z)$ for meyerhofferite. Contour intervals are at approximately 4 e.Å^{-2}, with the 4 e.Å^{-2} contour indicated by broken lines. The Ca atom is contoured at 10 e.Å^{-2} intervals above 20 e.Å^{-2}. The small black circles indicate the boron positions, the small open circles the oxygen positions, and the spoked circle the position of the water molecule. The average B–O bond length in the tetrahedra is 1·49 Å, and in the triangle is 1·38 Å. The average Ca–O distance for nearest oxygens is 2·40 Å. The $[B_3O_3(OH)_5]^{-2n}$ rings are linked together and to the water molecule by several hydrogen bonds. The hydrogen bond linking the rings into pseudo-chains along the *c* axis is shown by the dashed line.

* Publication authorized by the Director, U.S. Geological Survey.

IBM calculations were carried out in the U.S. Geological Survey by Vincent Latorre, to whom we are very grateful. We are also indebted to Dr J. Karle and Dr H. Hauptman, of the Naval Research Laboratory, for much helpful discussion.

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