

and its sensitivity to photographic emulsions is far less than that of Cu K . Moreover, the denser background will result from the Compton scattering and fluorescent radiations. At present, there is not any appropriate anticathode material available between copper and molybdenum. Under these circumstances, we believe that the combined use of both radiations Cu $K\alpha$ and Cu $K\beta$ in the Weissenberg technique is far superior to the use of the filtered radiation when structure analysis is attempted to carry out of crystals, particularly organic ones, whose axial parameters are of the order of 10 Å or longer.

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Unit-cell data for some anhydrous potassium borates. By J. KROGH-MOE, *Svenska Silikatforskningsinstitutet, Göteborg, Sweden*

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Phase relationships in the system potassium oxide-boron oxide have been reported by Rollet (1935, 1936). Unit-cell data have previously been given for three phases in this system, namely $K_2O \cdot B_2O_3$ (Zachariassen, 1937), $\alpha K_2O \cdot 5 B_2O_3$ (Krogh-Moe, 1959a) and $\beta K_2O \cdot 5 B_2O_3$ (Krogh-Moe, 1959b). In the following, data will be given for three other phases in the system.

1. *Potassium diborate*, $K_2O \cdot 2 B_2O_3$. Crystals of potassium diborate were prepared by fusing together stoichiometric amounts of p.a. potassium carbonate with p.a. boric acid in a platinum crucible at 1000 °C. for a short time and then annealing the resulting glass melt below 800 °C. Single crystals of sufficient quality for X-ray work could be found in the devitrified glass. Since the crystals are rather hygroscopic, they were mounted inside a protecting capillary tube. X-ray Weissenberg and oscillation photographs showed a triclinic cell. The photographs were taken (with Cu $K\alpha$ -radiation) around the b and c axes of the reduced cell. From the information thus obtained, the following unit-cell dimensions of the direct lattice were computed:

$$\begin{aligned} a &= 6.46, \quad b = 9.56, \quad c = 10.38 \text{ \AA}; \\ \alpha &= 90.7, \quad \beta = 102.6, \quad \gamma = 101.2^\circ. \end{aligned}$$

The calculated density is 2.53 g.cm.⁻³ with 4 formula units of $K_2O \cdot 2 B_2O_3$ in the cell.

2. *Potassium triborate*, $K_2O \cdot 3 B_2O_3$. Potassium-borate glasses of the triborate and the tetraborate composition were prepared in the same manner as the diborate glass described above. It was assumed that the composition was determined by the weights of the original powders, though a slight change in composition due to preferential evaporation of one compound should not be overlooked. Whereas a glass of the triborate composition refused to crystallize within a couple of weeks at a temperature between 700 and 800 °C., a glass of the tetraborate composition was readily crystallized at 780 °C. It was first believed that the resulting crystalline phase was a tetraborate. (See the phase diagram by Rollet (1935)). The observed density of the phase, however, can be reconciled with the density calculated from the X-ray data only if the phase is a triborate. This result does not disprove the existence of a potassium tetraborate, but it does indicate that a reinvestigation of the phase diagram

Finally the multiple reflections associated with the strong reflection of lower Bragg angle are so sensitive to wave length that the simultaneous use of the two radiations will be effective in detecting these (Jellinek, 1958).

References

- HIRSHFELD, F. L. & SCHMIDT, G. M. J. (1956). *Acta Cryst.* **9**, 233.
JELLINEK, F. (1958). *Acta Cryst.* **11**, 626.

in the region around 20 mol.% potassium oxide would be desirable.

Weissenberg and rotation single-crystal X-ray photographs were taken around three non-coplanar rational axes (of which two turned out to be the b and c axes of the reduced cell). From these photographs, the phase was established as triclinic with the following cell dimensions:

$$\begin{aligned} a &= 10.63, \quad b = 10.64, \quad c = 13.01 \text{ \AA}; \\ \alpha &= 90.7, \quad \beta = 95.2, \quad \gamma = 114.2^\circ. \end{aligned}$$

The calculated density is 2.26 g.cm.⁻³ with 6 formula units of $K_2O \cdot 3 B_2O_3$ in the cell. The observed density, 2.23 g.cm.⁻³, was determined by flotation of the crystalline powder in heavy liquid mixtures.

It might be pointed out that the potassium triborate cell approaches hexagonal symmetry, and has a cell content of 6 formula units.

3. *γ potassium pentaborate*, $\gamma K_2O \cdot 5 B_2O_3$. The unit cell and the space groups of the two stable modifications of potassium pentaborate are known. Rollet mentions, however, a third metastable modification, which is obtained from the α -modification below 690 °C. A crystalline phase obtained accidentally in the pentaborate region during the present investigation is conjectured to be identical with the metastable phase reported by Rollet. A single crystal of this new material was oriented along the c -axes, and from a rotation photograph and from Weissenberg zero and first layer photographs, the following monoclinic unit cell was deduced:

$$a = 11.59, \quad b = 17.80, \quad c = 12.99 \text{ \AA}; \quad \beta = 95.8^\circ.$$

The cell contains 8 formula units of $K_2O \cdot 5 B_2O_3$, corresponding to a calculated density of 2.20 g.cm.⁻³. Systematic extinctions in the general hkl reflections for $h+k=2n+1$ were observed.

References

- KROGH-MOE, J. (1959a). *Arkiv Kemi*, **14**, 439.
KROGH-MOE, J. (1959b). *Arkiv Kemi*, **14**, 567.
ROLLET, A. P. (1935). *C. R. Acad. Sci., Paris*, **200**, 1763.
ROLLET, A. P. (1936). *C. R. Acad. Sci., Paris*, **202**, 1864.
ZACHARIASEN, W. H. (1937). *J. Chem. Phys.* **5**, 919.