Bronze-Type Structure of KNb₂O₅F and KTa₂O₅F

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The structural type of tetragonal potassium wolfram bronze 1 has been found to be present in several oxide compounds of transition metals of the general formula $A_x Tr O_3$ (A is a mono- or divalent metal, Tr is a transition metal, $x \leq 0.6$). The structure is characterized by a framework of TrO, octahedra joined by corners in such a way as to enclose linear arrange-ments of voids. The latter, which are the sites of the A atoms, are of two kinds viz. either with a cage-like environment analogous to that of the A atoms in a perovskite structure, or in fivesided tunnels composed of oxygen atoms. The tetragonal framework which has a constant composition of TrO₃ is often deformed to adopt a lower symmetry or a superstructure. It is otherwise quite resistant towards chemical changes, as is evident from the ready substitution of A as well as of Tratoms phases leading to such

$$\begin{array}{l} K_{0.5}(W_{0.5}Ta_{0.5})O_3 \text{ and } \\ Na_{0.4}Sr_{0.1}(W_{0.4}Ta_{0.6})O_3.^2 \end{array}$$

The present investigation was undertaken in order to find out if the non-metal part of the framework can be correspondingly changed by substitution, viz. by partially replacing oxygen by fluorine. In order to maintain electroneutrality pentavalent niobium or tantalum could then be substituted for hexavalent wolfram.

The starting materials for the syntheses were potassium fluoride, niobium pentoxide and tantalum pentoxide, all of reagent grade. Stoichiometric amounts of fluoride and oxide in 1:1 proportion were intimately mixed and pressed into pellets which were sealed in thin evacuated platinum tubes and heated for 24 h at 900°C. The products thus obtained formed white crystalline powders.

X-Ray powder photographs were taken in a Guinier camera with $\operatorname{Cu}Ka_1$ radiation and with potassium chloride added to the specimens as an internal standard. The patterns thus obtained were those required by phases of the tetragonal potassium

wolfram bronze type with the unit-cell dimensions:

 ${\rm KF \cdot Nb_2O_5}$: a=12.632 Å, c=3.950 Å ${\rm KF \cdot Ta_2O_5}$: a=12.569 Å, c=3.961 Å.

The densities determined from the loss of weight in benzene are in concordance with a cell content of five formula units of KF·Nb₂O₅ $(d_{\rm obs}=4.25,\ d_{\rm calc}=4.28\ {\rm g/cm^3})$ and KF·Ta₂O₅ $(d_{\rm obs}=6.6,\ d_{\rm calc}=6.66\ {\rm g/cm^3})$.

The powder patterns gave no evidence of deviations from the tetragonal symmetry or of a superstructure. Previous experience, however, has shown that such superstructure reflections may be very weak and are preferentially looked for in single-crystal photographs.³

Further studies on bronze-type structures are in progress.

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On the Crystal Structure of a Potassium Tellurate OLIVER LINDQVIST and FREDRIK WENGELIN

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As the first member in a newly begun series of crystal structure determinations of tellurates, the structure of $K_4H_4Te_2O_{10}(H_2O)_x$, where x is probably 6, has been investigated, and a preliminary report is given in this note.

Weissenberg photographs corresponding to the reflections h0l-h4l and hk0-hk11 have been recorded, using $CuK\alpha$ radiation. From the reflections obtained, the space group was presumed to be either C2/c

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