Bronze-Type Structure of KNb$_2$O$_5$F and KTa$_2$O$_5$F

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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 $A_xTrO_4$ ($A$ is a mono- or divalent metal, $Tr$ is a transition metal, $x = 0.6$). The structure is characterized by a framework of $TrO_4$ octahedra joined by corners in such a way as to enclose linear arrangements 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 $TrO_4$ 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 $Tr$ atoms leading to phases such as $K_x(W_{2-y}Ta_y)O_4$ and $Na_xSr_y(W_{2-y}Ta_y)O_4$$^2$

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 CuK$\alpha$ 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:

- KF:Nb$_2$O$_5$: $a = 12.632$ Å, $c = 3.950$ Å
- KF:Ta$_2$O$_5$: $a = 12.569$ 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:Nb$_2$O$_5$ ($d_{obs} = 4.25$, $d_{calc} = 4.28$ g/cm$^3$) and KF:Ta$_2$O$_5$ ($d_{obs} = 6.6$, $d_{calc} = 6.66$ 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

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As the first member in a newly begun series of crystal structure determinations of tellurates, the structure of $K_xH_xTeO_{16}(H_2O)_x$, 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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