The Structure of a Crystalline Niobium Oxide Ethoxide, $Nb_8O_{10}(OEt)_{20}$

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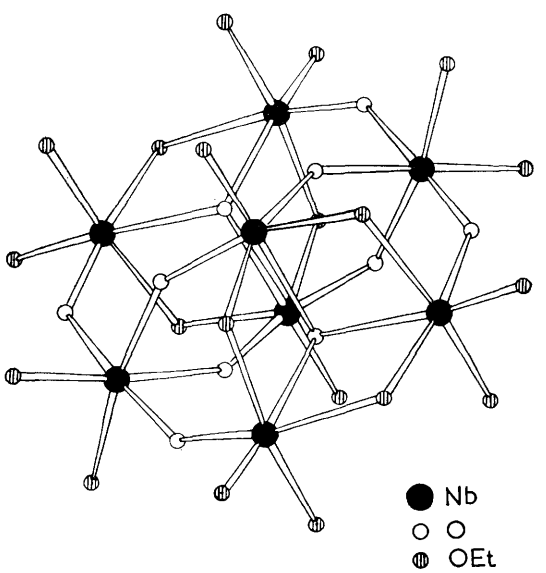
ALTHOUGH structures have been proposed for metal oxide alkoxide polymers based on molecular weight



FIGURE

studies in solution,[1] only one crystalline structure has so far been determined. This is the heptameric titanium oxide ethoxide for which the titanium and oxygen positions in the Ti_7O_{24} molecular unit were located.[2] In continuing studies on the hydrolysis of niobium pentaethoxide[3] we have isolated a crystalline product $Nb_8O_{10}(OEt)_{20}$ (satisfactory elemental analyses were obtained) and now report its structure as determined by *X*-ray diffraction.

Rod-shaped crystals (ethanol): $C_{40}H_{100}O_{30}Nb_8$, $M = 1804{\cdot}52$ monoclinic, $a = 14{\cdot}96$, $b = 14{\cdot}36$, $c = 16{\cdot}84$ Å, $\beta = 91{\cdot}0°$, $U = 3615{\cdot}45$ Å^3, $D_m = 1{\cdot}65$, $Z = 2$, $D_c = 1{\cdot}66$ g./ml.; space group $P2_1/n$ octameric molecule centrosymmetric. The structure was solved by conventional Patterson and Fourier techniques from 1513 independent reflections visually estimated from Cu-K_α Weissenberg photographs. No absorption corrections have been made. Least-squares refinement is in progress and the present *R*-factor is 0·118.

The structure is depicted in the Figure (showing the molecule viewed down the *b* axis with carbon atoms omitted for clarity). The molecule $Nb_8O_{10}(OEt)_{20}$ is comprised of eight slightly distorted NbO_6 octahedra and the arrangement may be visualized as two sets of three edge-sharing octahedra linked by two bridging octahedra through corner sharing. The presence of a vacant central octahedral hole gives a cage-like structure which is quite different from the Ti_7O_{24} unit. The structure

contains several interesting features. Thus there are no terminal Nb=O groups but eight oxide-oxygens are each bridging two niobiums and two oxide-oxygens bridge three niobiums. Also there are six bridging ethoxide-oxygens and fourteen terminal-ethoxide groups. A significant feature which might be a determining factor of this compact structure is the *cis*-configuration of all pairs of terminal-ethoxide groups. Discussion of the bond lengths (Nb–O, 2·01—2·18 Å) and bond angles is deferred until full details are published following further refinement.

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[2] K. W. Watenpaugh and C. N. Caughlan, *Chem. Comm.*, 1967, 76.
[3] D. C. Bradley and H. Holloway, results mentioned in reference 1.