The Crystal Structure of $H[MoO_2(OCH_2CH_2)_3N]$

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Summary In $H[MoO_2(OCH_2CH_2)_3N]$ the molybdenum atom has a distorted octahedral co-ordination and the MoO_2 group has the *cis*-configuration.

IN order to clarify the cause of the lengthening of the bond in the *trans*-position to the multiple bond in an octahedral complex, an X-ray study of $H[MoO_2(OCH_2CH_2)_3N]$ has been carried out.

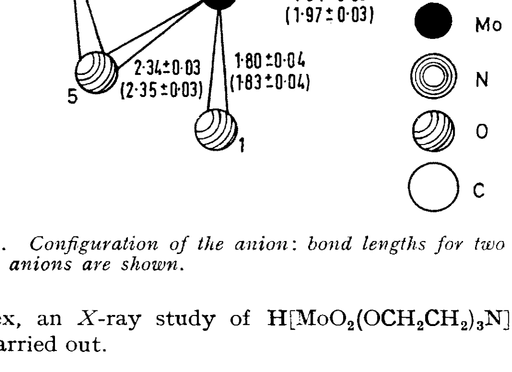

FIGURE. *Configuration of the anion: bond lengths for two independent anions are shown.*

Crystal data: $H[MoO_2(OCH_2CH_2)_3N]$, $a = 11{\cdot}88(4)$, $b = 7{\cdot}86(3)$, $c = 11{\cdot}52\ (4)$ Å, $\beta = 92° \pm 0{\cdot}5°$; space group $P2$ $M = 275$, $U = 1075$ Å^3, $D_o = 1{\cdot}73$, $D_c = 1{\cdot}71$ g cm^{-3}, $Z = 4$. The intensities of 1210 independent nonzero reflections were taken with an X-ray goniometer using unfiltered Mo radiation and were estimated visually (layers $h0l$—$h6l$, $0kl$, $1kl$). Absorption corrections were deemed to be unnecessary ($\mu = 11{\cdot}8$ cm^{-1}). The crystal structure was refined by the isotropic full-matrix least-squares method to $R = 0{\cdot}125$.

There are two independent $[MoO_2(OCH_2CH_2)_3N]^-$ anions in the unit cell; the configuration and some significant bond lengths are shown in the Figure.

The short Mo–O(1) and Mo–O(2) bond lengths apparently exclude the possibility of one being an Mo–OH bond. Moreover, the Mo–N and Mo-O(5) bonds in *trans*-positions to Mo–O(1) and Mo–O(2), respectively, are significantly longer than normal Mo–O(N) single bonds, and this also indicates a high degree of multiplicity in Mo–O(1) and Mo–O(2).[1]

Mo–N bond lengths close to ours have been found in $Na_2[O_3Mo(edta)MoO_3],8H_2O$ (2·41 Å)[2] and in $MoO_2(C_{10}H_8N_2)Br_2$ (2·26 and 2·45 Å).[3]

Thus an analysis of the bond lengths within the molecule reveals the presence of the *cis*-dioxo-group MoO_2 and apparently eliminates the formula $MoO(OH)(OCH_2CH_2)_3N$ originally suggested.[4]

We assume that the long Mo–N and Mo–O(5) bonds are mainly the result of non-bonding repulsion interactions in the metal's co-ordination sphere. Apparently non-bonding interactions are among the principal causes of high bond lengths in other "atran" complexes $EtGe(OCH_2CH_2)_3N$[5] and $PhSi(OCH_2CH_2)_3N$.[6]

We thank Dr. M. G. Voronkov and Dr. A. F. Lapsyn for supplying the crystals.

(*Received, March* **10*th*, 1970**; *Com.* 354.)

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