de symétrie binaire, pratiquement parallèle à l'axe [001] (axe de croissance du cristal) passant par le milieu de la liaison C(5)–C(6). Il en résulte entre autres que les quatre branches C–C$\overset{O}{\underset{O}{\diagdown}}$ sont situés d'un même côté. d'un plan perpendiculaire à l'axe 2 et contenant la liaison C(5)–C(6).

Le passage pour l'ion H_2Y^{2-}, d'une symétrie du type $2/m$ dans le sel birubidique à une symétrie du type 2 dans le sel bipotassique est *a priori* assez surprenante. Elle devrait toutefois pouvoir s'expliquer par le pouvoir polarisant plus élevé pour l'ion K^+ que pour l'ion Rb^+. C'est ainsi que dans le sel bipotassique, l'un des ions K^+ hexacoordonné, est lié à trois oxygènes appartenant à un même ion H_2Y^{2-}, alors que chaque ion Rb^+ est lié à des oxygènes appartenant à des molécules différentes.

Ici encore, la configuration générale de l'ion H_2Y^{2-} est compatible avec une formule de type ion bipolaire. En effet:

1. Les angles C–N–C voisins de 110° correspondent certainement à des atomes d'azote protonés NH^+.

2. Dans les branches N–C–C$\overset{O}{\underset{O}{\diagdown}}$ approximativement planes, les groupements COO^- sont repliées de façon que l'un des atomes d'oxygène soit le plus près possible de l'atome d'hydrogène d'un groupement NH^+.

Chaque groupement NH^+ est engagé dans des liaisons d'hydrogène intramoléculaires de type trifide, avec les groupements COO^- voisins puisque:

1. L'atome N(1) est proche des atomes d'oxygène O(2), O(3) et O(6).

2. L'atome N(2) est proche des atomes d'oxygène O(7), O(2) et O(6).

Ainsi, dans les deux sels alcalins bihydratés de l'EDTA, l'ion H_2Y^{2-} est un ion bipolaire, où les atomes d'azote sont protonés. L'hydrogène du groupement NH^+ est engagé, dans les deux cas, dans des liaisons hydrogène de type trifide. Dans le cas du sel de Rubidium, l'une des liaisons est intermoléculaire, alors qu'elles sont toutes trois, intramoléculaires dans le sel de potassium. L'ion H_2Y^{2-} de symétrie $2/m$ dans le sel birubidique possède une symétrie 2 dans le sel bipotassique.

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The Crystal and Molecular Structure of Molybdenum(V) Oxytribromide

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Crystals of molybdenum(V) oxytribromide are tetragonal, $a = 11\cdot360$, $c = 3\cdot948$ Å, with four formula units of $MoOBr_3$ per unit cell. There is considerable doubt as to the correct structure, but a disordered model has been refined in space group $P4_2/mnm$, using 149 non-zero reflexions, to an R value of $0\cdot072$. This structure consists of non-planar Mo_2Br_6 units joined by –Mo–O–Mo– bonds into infinite chains. The oxygen atoms are not equidistant from two molybdenum atoms, the Mo–O distances being $1\cdot65$ and $2\cdot31$ Å.

Introduction

Oxytrichlorides and oxytribromides of the heavier metals in group VA, group VIA and group VIIA are well established (Canterford, Colton & Tomkins, 1968). These compounds can be divided into two general groupings: the first with $MoOCl_3$, $TcOCl_3$ and $ReOBr_3$ have metal–oxygen stretching frequencies

in the range 1000–1020 cm^{-1} and it has been assumed that they contain a terminal M–O bond. A single-crystal X-ray structure analysis of $MoOCl_3$ by Drew & Tomkins (1970) has shown that the structure is a chlorine-bridged polymer with terminal Mo–O bonds. The second group contains such compounds as $WOCl_3$, $WOBr_3$, $MoOBr_3$, $NbOCl_3$ and $TcOBr_3$. They have metal–oxygen stretching frequencies in the range 732

to 810 cm^{-1}, suggestive of oxygen bridging. The powder patterns of $WOCl_3$, $WOBr_3$, $MoOBr_3$ and $NbOCl_3$ are similar and it has been suggested by Crouch, Fowles, Frost, Marshall & Walton (1968) that they are isomorphous. A single-crystal X-ray study of $NbOCl_3$ by Sands, Zalkin & Elson (1959) shows planar Nb_2Cl_6 units joined by $-Nb-O-Nb-$ bonds into infinite chains. We have now undertaken a single-crystal X-ray structure analysis of molybdenum(V) oxytribromide: the structure is almost certainly not isomorphous with the published structure for $NbOCl_3$. We have also repeated the refinement of $NbOCl_3$, using the published data, and show that the validity of that structure determination is debatable.

Experimental

Crystals were prepared from the reaction of MoO_2Br_2 with PBr_5 using the method of Colton & Tomkins (1965). $MoOBr_3$ is tetragonal with $a = 11\cdot360(8)$, $c = 3\cdot948(4)$ Å. M.W. $351\cdot64$, $V = 509\cdot48$ Å^3 $d_c = 3\cdot508$ g. cm^{-3}, $\mu = 207\cdot9$cm^{-1}, for Mo $K\alpha$ radiation. Laue symmetry $4/mmm$. Systematic absences are, for $0kl$, $k+l=2n+1$. Possible space groups are $P4_2nm$, $P\bar{4}n2$ and $P4_2/mnm$ (respectively Nos. 102, 118, 136). The general positions in space group $P4_2nm$ are $8(d)$ $(x, y, z$: y, x, z: $\bar{x}, \bar{y}, z$: $\bar{y}, \bar{x}, z$: $\frac{1}{2}+x, \frac{1}{2}-y, \frac{1}{2}+z$: $\frac{1}{2}+y, \frac{1}{2}-x, \frac{1}{2}+z$: $\frac{1}{2}-x, \frac{1}{2}+y, \frac{1}{2}+z$: $\frac{1}{2}-y, \frac{1}{2}+x, \frac{1}{2}+z)$. The general positions $16(k)$ in space group $P4_2/mnm$ include these eight with the additional centrosymmetric set.

Needle-shaped crystals of $MoOBr_3$ were sealed inside thin-walled 0·3 mm diameter capillaries under an atmosphere of nitrogen. A crystal of approximate dimensions (0·03 × 0·03 × 0·5 mm) was selected for the analysis and was mounted with the c axis parallel to the instrument axis. Diffraction intensities (and cell dimensions) were measured with a General Electric XRD-5 apparatus equipped with a manual goniostat, scintillation counter and pulse-height analyser. A molybdenum tube was used at 45kV and 20mA with a zirconium filter at the receiving slit. The stationary crystal–stationary counter method was used to measure 276 independent reflexions ($2\theta < 50°$) with a 4° take-off angle. The counting time was 20 seconds. Individual backgrounds were measured for those reflexions whose counts were seriously affected by the streaking of other orders. For other reflexions, backgrounds were taken from plots of background as functions of 2θ. Of the 276 reflexions, 127 gave counts less than, or equivalent to, the estimated backgrounds

and were given zero weight in the subsequent refinement. Several standard reflexions were measured repeatedly during the course of the experiment, but no significant change in intensity was detected. The 002 reflexion was centred at $\chi = 90°$ and a graph plotted of φ against intensity. It was found that the ratio between the highest and lowest intensity was 1·2. This ratio was small enough for the crystal to be considered as a cylinder, and with a calculated value of μR of 0·3, the absorption correction was negligible up to $\theta = 25°$ (*International Tables for X-ray Crystallography*, 1962). Extinction corrections were not applied. In the calculation of structure factors, atomic scattering factors for Mo, O and Br were taken from *International Tables for X-ray Crystallography* (1962). Dispersion corrections ($\Delta f'$ and $\Delta f''$) for Mo and Br were taken from the same source and applied in all refinements. The isotropic temperature factor was defined as $\exp(-B\sin^2\theta/\lambda^2)$; the anisotropic thermal parameters used have the form: $\exp(-0\cdot25 \sum_i \sum_j h_ih_jb_ib_jB_{ij}), i, j = 1, 2, 3$, and where b_i is the ith reciprocal-cell length. Calculations were made with the ATLAS computer at Didcot, Berkshire. We used the *X-ray* 63 system of programs, compiled by J. M. Stewart & D. High and amended for that machine by J. Baldwin. Some of our own programs were used on the Elliott 4130 at this university. During the least-squares refinements, a weighting scheme was chosen which gave average values of $w\Delta^2$ for groups of reflexions which did not vary with the size of $|F_o|$ or with $\sin \theta/\lambda$. The scheme was that of Hughes (1941): with $\sqrt{w} = 1$ for $|F_o| \le 60$ and $\sqrt{w} = 60/|F_o|$ for $|F_o| > 60$.

Determination of the structure

The structure of $NbOCl_3$ has been refined by Sands, Zalkin & Elson (1959), in space group $P4_2/mnm$. There were four independent atoms: Nb in special positions $4(f)$ with coordinates $\pm(x, x, 0$: $\frac{1}{2}+x, \frac{1}{2}-x, \frac{1}{2})$: the bridging chlorine atoms and oxygen atoms in special positions $4(g)$, $\pm(x, \bar{x}, 0$: $\frac{1}{2}+x, \frac{1}{2}+x, \frac{1}{2})$, and the terminal chlorine atoms in special positions $8(i)$, $\pm(x, y, 0$: $y, x, 0$: $\frac{1}{2}+x, \frac{1}{2}-y, \frac{1}{2}$: $\frac{1}{2}+y, \frac{1}{2}-x, \frac{1}{2})$. The structure of $MoOBr_3$ was refined by full-matrix least squares using the parameters and symmetry of the above structure of $NbOCl_3$. Refinement with all atoms isotropic gave an unexpectedly high value for R, using the observed data only, of 0·186. The temperature factors of the Mo and O atoms at 3·9 and 0·1 were notably different from those

Table 1. *Final coordinates* ($\times 10^4$) *and thermal parameters* ($\times 10$) *for structure model* (a) *with* $R = 0\cdot100$

Standard deviations for the least significant figures are given in parentheses.

	x	y	z	$B_{11}(B)$	B_{22}	B_{33}	B_{12}	B_{13}	B_{23}
Mo	1201 (8)	1201 (8)	0000	10 (3)	10 (3)	142 (8)	−02 (3)	0	0
Br(1)	1074 (8)	−1074 (8)	0000	18 (3)	18 (3)	21 (3)	00 (3)	0	0
Br(2)	3300 (4)	1084 (4)	0000	19 (2)	19 (2)	28 (3)	−01 (2)	0	0
O(1)	1085 (58)	1085 (58)	5000	22 (8)					

Table 2. *Final coordinates* ($\times 10^4$) *and thermal param-*
meters ($\times 10$) *for structure model* (b) *with* $R = 0.072$

Standard deviations for the least significant figures are given
in parentheses.

	x	y	z	B
Mo	1201 (5)	1201 (5)	0846 (10)	11 (1)
Br(1)	1075 (6)	−1075 (6)	0000	18 (1)
Br(2)	3300 (3)	1088 (3)	0000	22 (1)
O(1)	1099 (39)	1099 (39)	5000	18 (5)

of the two independent bromine atoms at 2·4. The reasons for these values became apparent with the anisotropic refinement, in which the R value dropped to 0·100. The x and y parameters showed little change from the isotropic refinement. The resulting parameters, for this structure (a), are given in Table 1. In the last cycle of refinement, no shift/error ratio was greater than 0·1. Although the thermal parameters of the two bromine atoms are approximately spherical, those of the molybdenum atom are remarkable in that the thermal motion is 14 times greater in the z direction than in the xy plane. The thermal parameter of the oxygen atom increased to an acceptable value.

The most likely explanation of these results was that the z coordinate of the Mo atom had been wrongly fixed at zero. Therefore, we computed a difference Fourier synthesis, using the parameters of the two independent bromine atoms for the structure factor calculations. The Patterson function was also calculated. Although both maps contained a mirror plane at z=0, the shapes of the Mo peak in the difference Fourier synthesis and of the Mo–Br vectors in

Fig. 1. The ordered structure of molybdenum(V) oxytri-bromide.

the Patterson synthesis were consistent with a z coordinate of the molybdenum atom between 0·05 and 0·10. We next considered the possibility of a disordered structure (b) with molybdenum atoms in special positions 8(j) with half occupancy and a z coordinate of 0·075. (These special positions include x, x, z and $x, x, \bar{z}$.) This refinement, using isotropic thermal parameters gave an R value of 0·072 for the 11 parameters. The R value remained the same during anisotropic refinement – the thermal parameters of the three heavy atoms were closely isotropic. The B_{11} and B_{33} terms of the Mo atom were 1·11 and 0·56 respectively: thus the proximity (0·68 Å) of pairs of half atoms across the mirror planes at z=0 and ½ had not seriously affected the refinement. As the increase in parameters for the anisotropic refinement had not resulted in any lowering of R value, we quote the parameters from the isotropic refinement of structure (b) in Table 2.

In the final cycle, none of the final shift/parameter ratios was greater than 0·1. A difference Fourier synthesis was calculated, based on the Mo and Br positions only, but we found no evidence that the oxygen atom had been wrongly positioned at $z=\frac{1}{2}$ and that it should have been refined with an occupancy of 0·5 in special positions 8(j).

Of the two other possible space groups $P\bar{4}2n$ also contains a mirror plane at z=0. We therefore attempted to refine the structure in space group $P4_2nm$ as model (c). In this space group, the terminal bromine atoms were placed in general positions 8(d) and the z coordinate was fixed at zero. The remaining three independent atoms were placed in special positions 4(c) $(x, x, z: \bar{x}, \bar{x}, z: \frac{1}{2}+x, \frac{1}{2}-x, \frac{1}{2}+z: \frac{1}{2}-x, \frac{1}{2}+x, \frac{1}{2}+z)$. The z coordinate of the molybdenum atom was set at 0·05 while those of the Br and O atoms were left at 0·0 and 0·5. Isotropic refinement of these parameters gave an R value of 0·106 and anisotropic refinement 0·091. In both cases, none of the final shift/error ratios was greater than 0·1. The dispersion corrections for Mo and Br were then applied and two models were refined with opposite signs for z coordinates. The two R values were 0·092 and 0·093. The final parameters from the former refinement are given in Table 3.

The parameters are much more reasonable than those found in the other ordered model (a). Still, the thermal motion of the molybdenum atom is 4·5 times greater in the z direction than in the xy plane. At this stage a difference Fourier synthesis was calculated based on the Mo and Br atoms. Again, the oxygen
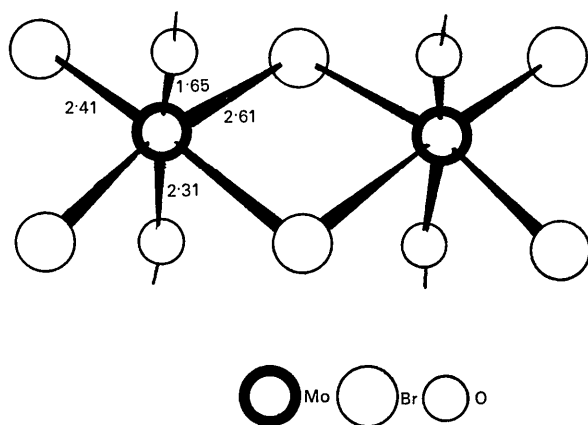
Table 3. *Final coordinates* ($\times 10^4$) *and thermal parameters* ($\times 10$) *for structure model* (c) *with* $R = 0.091$

Standard deviations for the least significant figures are given in parentheses.

	x	y	z	$B_{11}(B)$	B_{22}	B_{33}	B_{12}	B_{13}	B_{23}
Mo	1195 (8)	1195 (8)	0754 (33)	12 (3)	12 (3)	57 (6)	−1 (3)	3 (3)	3 (3)
Br(1)	1075 (9)	−1075 (9)	−0062 (42)	18 (3)	21 (3)	21 (3)	2 (2)	−3 (3)	−3 (3)
Br(2)	3299 (4)	1084 (4)	0000	18 (2)	18 (2)	29 (3)	−1 (3)	2 (3)	2 (3)
O(1)	1121 (57)	1121 (57)	5058 (185)	17 (7)					

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atom appeared to be centred at $z=\frac{1}{2}$. As the ratio of observed data/variables was 6·8, it was decided not to refine the oxygen atom anisotropically. We also considered the possibility that the true Laue group of the reciprocal lattice was $4/m$ and that merohedral twinning of the lattice had occurred. However, the systematic absences $(0kl, k+l=2n+1)$ are not consistent with any space group in this group.

Discussion of the refinements

Three possible structures for molybdenum(V) oxytribromide (a), (b), (c) have been refined. In each of them, the symmetry and parameters of the bromine atoms and oxygen atoms are approximately the same. The models contain Mo_2Br_6 entities, in which the bromine atoms are planar with z coordinates equivalent to, or close to, 0. The oxygen atoms, which bridge the molybdenum atoms, have z coordinates of 0·5, or approximately 0·5, in all refinements. The variations, introduced by the three models, attempt to determine the parameters of the molybdenum atoms. In the first structure (a), where 15 parameters are refined to an R value of 0·100, the z coordinate is restricted to 0·0 in space group $P4_2/mnm$. In the structure (b) where 10 parameters are refined to an R value of 0·072, the z coordinate is $+$ and -0.085. In structure (c), where 22 parameters are refined to an R value of 0·091, the z coordinate is 0·075 in space group $P4_2nm$. From the consideration of R value alone, the most probable model is the disordered model (b). Despite its being

derived from fewer parameters than either of the ordered models (a) and (c), the R value is significantly lower. In addition, the anisotropic refinement of the molybdenum atom was not successful in the ordered structures (a) and (c), as the B_{33} term of the Mo atom increased to an unrealistic value, suggesting that the z coordinate of the molybdenum atom could not be fixed at one specific value. In structure (b), the molybdenum atoms were refined with half occupancy at $z=0.08$ and -0.08: the anisotropic thermal parameters of the molybdenum atom were reasonable, although as might have been expected, the B_{33} term was less than the B_{11} term, thus lessening any possible overlap between the half atoms. As the three models seem equally likely chemically, the crystallographic evidence, which strongly favours the disordered model structure (b), is decisive. The bond distances and angles for this structure are given in Table 4, together with the standard deviations. Table 5 is a final list of the observed and calculated structure factors.

Chemical significance of structure model (b)

In model (b), the bromine and oxygen atoms are ordered throughout the crystal. From the refinement, each individual molybdenum atom is equally likely to be above $(+z)$ or below $(-z)$ the plane of the bromine atoms. However, there are two principal restrictions to such a haphazard arrangement of molybdenum atoms that appear logical. First, it is probable that the $-Mo-O-Mo-$ chains are ordered,

Table 4. *Bond distances and angles for* $MoOBr_3$

Standard deviations for the least significant figures are given in parentheses.*

Mo–O(1)	1·65 (1) Å	O(1)–Mo—Br(1)	93·1 (16)°
Mo–O(1')	2·31 (1)	O(1)–Mo—Br(2)	101·7 (16)
Mo–Br(1)	2·61 (1)	O(1)–Mo—O(1')	170·2 (19)
Mo–Br(2)	2·41 (1)	O(1')–Mo—Br(1)	79·6 (11)
		O(1')–Mo—Br(2)	84·8 (11)
		Br(1)–Mo—Br(2)	163·5 (2)
		Br(1)–Mo—Br(2'')	89·1 (3)
		Br(1)–Mo—Br(1'')	82·8 (3)
		Br(2)–Mo—Br(2')	94·9 (3)
		Mo—Br(1)–Mo'''	95·3 (3)

* Coordinates of atoms marked ' '' ''' '''' are obtained from the coordinates in Table 2 by the symmetry operations $(x,y,z-1$: y,x,z: $\bar{x},\bar{y},z)$.

Table 5. *Observed and calculated structure factors* $(\times 2.0)$ *for molybdenum(V) oxytribromide*

Adjacent columns are for h, $|F_o|$ and F_c.

each molybdenum atom being strongly bonded (1·65 Å) and weakly bonded (2·31 Å) to oxygen atoms. Secondly, if both molybdenum atoms in the Mo_2Br_6 groupings are the same side of the plane of the bromine atoms, it is possible for individual unit cells to conform to space group $P4_2nm$: the alternative, with molybdenum atoms either side of the plane in the Mo_2Br_6 groupings, would conform to no tetragonal space group.

Thus, it is proposed that the individual unit cells are ordered (in $P4_2nm$) and that there is little or no disorder in the c direction. It is suggested that domains in the crystal will be ordered: in some of these domains the molybdenum atoms have $+z$ coordinate throughout, in the others $-z$. In the refinement, it has been assumed that the proportion of unit cells with $+$ and $-z$ coordinates are approximately the same.

The chemical implications of the structure can, therefore, be discussed on the basis of an ordered molecule which is shown in Fig. 1. Each molybdenum atom is six-coordinated and has a distorted octahedral environment. It is surrounded by two terminal and two bridging bromine atoms and two bridging oxygen atoms. The four bromine atoms bonded to each molybdenum atom are coplanar. The molybdenum atom is 0·34 Å above this plane and weakly bonded to one oxygen atom at 2·31(1) Å, and strongly bonded to the other at 1·65(1) Å. The two molybdenum atoms bridged by oxygen atoms are 3·95 Å apart, while those bridged by the bromine atom are 3·96(1) Å apart. The strong Mo–O bond distance at 1·65(1) Å can be usefully compared with Mo–terminal O distances obtained in $Ba[MoO(C_2O_4)(H_2O)_2O_2].3H_2O$, found by Cotton & Morehouse (1965) to be 1·70 Å and in $MoOCl_3$, found by Drew & Tomkins (1970) to be 1·63 Å. The environment of the molybdenum atom in the present compound is very similar to that observed by Scane (1967) in the $[MoOBr_4(H_2O)]^-$ anion, and by Drew & Tomkins (1970) in $MoOCl_3$. In the $[MoOBr_4(H_2O)]^-$ anion, which has $4mm$ symmetry, the Mo–O and Mo–OH_2 distances are 1·78 and 2·23 Å respectively, with the Mo atom 0·32 Å above the plane of the bromine atoms. In $MoOCl_3$, the Mo atom, which is six-coordinated, is 0·36 Å above the plane of the four chlorine atoms, cis to the terminal oxygen atom.

Refinement of $NbOCl_3$

In view of the above results for $MoOBr_3$, it was decided to re-examine the data for $NbOCl_3$ (Sands, Zalkin & Elson, 1959). 61 independent non-zero reflexions were measured by photographic methods and 4 independent scale factors were used. Because of the data/parameter ratio, we could not usefully refine $NbOCl_3$ anisotropically: we refined the data using the positional parameters in the above models (a), (b) and (c) with isotropic thermal parameters. In all three refinements, unit weights were used and the final shift/parameter ratios were less than 0·1. Structure (a) in $P4_2/mnm$, with the z coordinate of Mo fixed at zero gave an R value of 0·076 for the 13 parameters; structure (b) in $P4_2/mnm$, with the molybdenum atom in half occupancy at $z = \pm 0·08$ gave an R value of 0·079 for 14 parameters, while structure (c) in space group $P4_2nm$ with the Mo atom at $z = 0·05$ gave an R value of 0·048 for 16 parameters. In using the Hamilton (1965) test for comparing refinements, we find that the hypotheses that structure (a) or structure (b) are preferable to structure (c) can both be rejected at the 0·005 level of significance. Nevertheless, with the present data, we consider these three refinements of limited value in determining the correct structure for $NbOCl_3$: however, it can be concluded that the structure proposed by Sands, Zalkin & Elson (1959) is only one of the several possible models.

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