ACTA CHEMICA SCANDINAVICA 20 (1966) 1658–1666

The Crystal Structure of V_2MoO_8

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The crystal structure of a phase observed in the molybdenum-vanadium-oxygen system — V_2MoO_8 — has been determined and refined in space group $C2$. On the basis of X-ray powder diffraction data, the monoclinic unit cell has the dimensions: $a = 19.398 \pm 0.009$ Å, $b' = 3.629 \pm 0.001$ Å, $c = 4.117 \pm 0.001$ Å, $\beta = 90.34 \pm 0.03°$. Single crystal studies revealed that the b axis is doubled. The extra reflections are continuous streaks and suggest the presence of one-dimensional disorder.

The structure which is isotypic with Nb_3O_7F consists of ReO_3-type slabs infinitely long in two dimensions but only three octahedra thick. These slabs are connected by edge sharing between component octahedra. The ideal arrangement is considerably distorted, mainly by displacement of the metal atoms from the centres of the octahedra. Vanadium and molybdenum atoms are distributed almost, but not quite, statistically over the two non-equivalent positions.

The metal-oxygen bond distances are generally intermediate between the corresponding values in V_2O_5 and MoO_3.

On the basis of the structure, a homogeneity range, $V_{2-x}Mo_{1+x}O_8$, where $x \geq 0$, is expected for this phase.

A phase observed in the V_2O_5—MoO_3 system and thought to have a narrow homogeneity range, a composition close to $V_2O_5 \cdot MoO_3$ ($V_{0.67}Mo_{0.33}O_{2.67}$), and a density of 3.73 g/cm^3, was reported in 1951 by Magnéli and Blomberg.[1] This phase has been a topic of discussion recently in many papers. According to Munch and Pierron,[2] it is slightly deficient in oxygen and should be assigned the formula $V_9Mo_6O_{40}$ ($V_{0.60}Mo_{0.40}O_{2.67}$). However, their powder diffraction data and cell dimensions do not agree with the results of others. Strupler and Morette [3] have studied the liquid/solid equilibrium in the V_2O_5—MoO_3 system. They reported a broad melting point maximum around the composition V_2MoO_8. Tridot *et al.* have prepared a phase at the composition V_2MoO_8 and have reported its d-values and relative intensities.[4] Freundlich and Pailleret have studied the system V_2MoO_{8-x} in the range $0 \leq x \leq 2$.[5] They state that this V_2MoO_8 phase has a homogeneity range extending from $x = 0$ to $x = 0.1$

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($MO_{2.67}$—$MO_{2.63}$), but do not indicate whether their conclusion results from observed variations in the lattice parameters or merely failure to detect a second phase within this composition interval. They indexed the powder diffraction pattern on the basis of a monoclinic unit cell with the dimensions $a = 19.40$, $b = 3.62$, $c = 4.13$ Å, $\beta \simeq 90°$, and reported the space group to be $C2/m$ (*cf.* below).

A detailed study of the V—Mo—O system at 600°C undertaken by one of us[6] confirmed the presence of a phase of the approximate composition V_2MoO_8. This study indicates that a homogeneity range whose limits are as yet unknown probably extends towards the molybdenum-rich side.

The details of the single crystal X-ray studies made of this V_2MoO_8 phase, a resumé of which has appeared elsewhere,[7] are reported below.

EXPERIMENTAL

The crystal chosen for this investigation was selected from a sample prepared by heating an equimolar mixture of V_2O_5 and MoO_3. The mixture, sealed into an evacuated gold tube which was subsequently sealed into evacuated silica, was heated for one day at 630°C and then another day at 580°C. The product consisted of black, prismatic crystals. X-Ray powder diffraction photographs taken with a Guinier-type focusing camera indicated the presence of three phases: V_2MoO_8, small amounts of $(V_{0.95}Mo_{0.05})O_{2.5}$, and an as yet unidentified phase. The diffraction lines belonging to the V_2MoO_8 phase (Table 1) were indexed on the basis of a monoclinic unit cell (Table 2). The density of another

Table 1. X-Ray powder diffraction data for V_2MoO_8, Cu$K\alpha_1$ radiation ($\lambda = 1.54051$ Å).

I	d_{obs}	$\sin^2\theta_{obs} \times 10^5$	hkl	$(\sin^2\theta_{obs} - \sin^2\theta_{calc}) \times 10^5$
vw	9.67 Å	635	200	+ 4
w	4.843	2530	400	+ 7
vs	4.118	3499	001	− 1
w	3.772	4169	201	+20
s	3.565	4668	110	+ 6
w	3.235	5670	600	− 6
w	3.120	6094	401	+35
w	2.698	8149	$\bar{1}11$	− 4
m	2.650	8446	510	0
w	2.060	13984	002	−17
vw	2.011	14677	202	+10
w	1.941	15747	10,0,0	−20
m	1.814	18025	020	− 8
w	1.785	18630	$\bar{1}12$	−15
			220	−17
w	1.700	20537	420	− 2
m	1.660	21532	021	+15
w	1.587	23567	11,1,0	−16
w	1.582	23694	620	+ 1
w	1.360	32077	$\bar{2}03$	− 2
w	1.325	33783	10,2,0	− 1
vw	1.322	33931	$\bar{4}03$	+13
vw	1.263	37190	$\bar{1}0,2,1$	− 5
w	1.258	37493	313	−12

sample, demonstrated by its powder pattern to be monophasic V_2MoO_8, was found to be 3.83 ± 0.02 g/cm³. These data correspond to a cell content of 2.05, or approximately 2 formula units V_2MoO_8. Rotation and Weissenberg photographs of several crystals, taken during the preliminary studies, indicated that the true b axis was twice as long as that calculated from the powder diffraction data: $b = 2b' = 7.258$ Å. However, all reflections with k odd were just continuous streaks along the bows parallel to a^*. This phenomenon indicates that the structural details which necessitate the doubling of the b axis exhibit no periodicity in the direction of the a axis — a kind of one-dimensional disorder [8] to be discussed later.

Every crystal which was examined was twinned across (001). Three-dimensional intensity data were collected from the best crystal which had a major individual comprising 97 % of the total volume without any disturbing interference of the twin reflections. This crystal had the shape of a somewhat truncated rectangular prism with the dimensions 0.0154 mm ([100]) × 0.098 mm ([010]) × 0.039 mm ([001]).

Weissenberg photographs were taken by the multiple film technique with Cu$K\alpha$ radiation. Relative intensities of the sharp reflections $hk'l$ in the four layer lines $k' = 0-3$ were estimated by means of a calibrated scale.

All computational work was carried out on a FACIT EDB computer using the following programs (indicated by the numbers assigned to them in Ref. 9): No. 6019 for absorption correction,[10] No. 6024 for Lp correction, No. 6015 for structure factor calculations,[11] No. 6014 for Fourier summations,[11] No. 6023 for least squares refinement, No. 6016 for the calculations of interatomic distances and angles, and, in addition to these, a program for the calculation of standard deviations of interatomic distances written recently by R. Norrestam. The least squares program makes use of the block diagonal matrix approximation including individual, isotropic temperature factors.

A value of $\mu = 471.6$ cm^{-1} was calculated for the linear absorption coefficient from the data compiled in the *International Tables*.[12] Values of the atomic scattering factors for unionized atoms listed in the *International Tables*,[12] based on S.C.F. calculations for oxygen and vanadium, and on statistical methods for molybdenum (Thomas and Umeda) were used for structure factor calculations. The real part of the dispersion correction [12] was applied to these scattering curves.

Cruickshank's weighting function was used throughout the least squares calculations. The following form was applied in the last cycles: $w = (40 + F_{obs} + 0.002\,F_{obs}^2)^{-1}$. The weight analysis obtained in the last cycle of refinement is presented in Table 3.

DETERMINATION AND REFINEMENT OF THE STRUCTURE

As has been mentioned, the crystal exhibited one-dimensional disorder — a phenomenon which occurs frequently in layer structures and which can be explained in terms of stacking faults. In this case, the structural information inherent in the sharp reflections can be described in the "stacking lattice"[13] with an identity period $b' = 3.629$ Å.

The absence of reflections $hk'l$ for $h + k' = 2n+1$ indicated a C-centered (sub-)cell. Since there were no further extinctions among the $h0l$ reflections, possible space groups were $C2$ (No. 5), Cm (No. 8), and $C2/m$ (No. 12).

The Patterson function projected along [010] was calculated. An analysis of this map indicated that a plausible arrangement was a structure isotypic with Nb_3O_7F.[14] The upper layer level intensity data indicated that all atoms were situated close to two y' levels separated by $\frac{1}{2}$, just as in Nb_3O_7F, further supporting the structural similarities. The Patterson gave no information about the distribution of the two kinds of metal atoms over the two different positions M_1 and M_2 (*cf.* Table 2) present in this structure. Three alternatives were therefore tried; *viz.* (*a*) ($\frac{2}{3}$ V + $\frac{1}{3}$ Mo) in both positions ("disordered"), (*b*) M_1 = Mo, M_2 = V ("ordered"), and (*c*) M_1 = V, M_2 = ($\frac{1}{2}$V + $\frac{1}{2}$Mo) ("inverse").

Table 2. The crystal structure of V_2MoO_8.

Space group: $C2$ (No. 5)
Unit cell dimensions: $a = 19.398 \pm 0.009$ Å
$b' = \frac{1}{2}b = 3.629 \pm 0.001$ Å
$c = 4.117 \pm 0.001$ Å
$\beta = 90.34 \pm 0.03°$
(Sub-) Cell content: $2V_2MoO_8$
2 M_1, 4 M_2 and 14 O in 6 × 4(c): $(x,y,z;\ \bar{x},y,\bar{z};\ \frac{1}{2}+x,\frac{1}{2}+y,z;\ \frac{1}{2}-x,\frac{1}{2}+y,\bar{z})$
2 O in 1 × 2(b): $(0,y,\frac{1}{4};\ \frac{1}{2},\frac{1}{2}+y,\frac{1}{4})$
$M_1 = (0.50\ V + 0.50\ Mo)$
$M_2 = (0.75\ V + 0.25\ Mo)$

Atom	Position	$x \pm \sigma(x)$	$y \pm \sigma(y)$	$z \pm \sigma(z)$	$B \pm \sigma(B)$
$\frac{1}{2}M_1$	4(c)	0.99986 ± 0.00015	0.9892 ± 0.0021	0.0921 ± 0.0006	1.14 ± 0.05
M_2	4(c)	0.18617 ± 0.00009	0.0123 ± 0.0020	0.0976 ± 0.0004	0.92 ± 0.025
O_1	4(c)	0.0996 ± 0.0006	0.007 ± 0.010	0.0093 ± 0.0027	2.16 ± 0.19
O_2	4(c)	0.2080 ± 0.0004	0.522 ± 0.008	0.9997 ± 0.0023	1.24 ± 0.16
O_3	4(c)	0.1877 ± 0.0005	0.029 ± 0.009	0.4967 ± 0.0025	1.86 ± 0.18
O_4	2(b)	0	0.065 ± 0.008	$\frac{1}{4}$	2.04 ± 0.30
$\frac{1}{2}O_5$	4(c)	0.0014 ± 0.0017	0.537 ± 0.011	0.0428 ± 0.0054	1.59 ± 0.31

Two-dimensional least squares calculations showed that alternative c was not refinable at all, in contrast to a and b which refined, although the refinement stopped at a rather high R value. The coordinates M_1:($x = z = 0$, nonrefinable) M_2:($x = 0.19$, $z = 0.04$) and structurally reasonable oxygen positions were used as starting parameters in these calculations. An observed steady increase of the temperature factor for M_1 during the refinement in cases a and b suggested that the coordinates of this atom might be in error. An electron density map subsequently calculated showed the M_1 peak to be considerably broadened in the z direction. A structure model in which M_1 was located, not in the origin but in two close symmetry-related general positions of half-occupancy, was tried next. Such a splitting of the atom could be justified in view of the observed doubling of the b' axis; it was then assumed that one of the equivalent positions was occupied at the level $y'+1$ ($= y+\frac{1}{2}$). This structure model refined to $R = 0.155$ and $R = 0.182$ for alternative a and b, respectively.

The refinement was continued with three-dimensional data; 323 reflections covering a quadrant of the reciprocal space. (One of the reflections, 010, was deleted in the last stages of the refinement because it was considered to suffer seriously from extinction.) Computations were made for the two alternative space groups $C2/m$ (atoms in the special positions 4i, 2c, and 2b) and $C2$ (atoms in the general position 4c and in the special positions 2b and 2a). The space group Cm was considered less probable at this stage. Differences between the results obtained for these two alternatives were negligible although $C2$ quite naturally gave a slightly lower R value because of the larger number of refinable parameters. However, in this latter case, the metal atoms had shifted a distance corresponding to 6σ (*cf.* Table 2) in opposite directions out

Table 3. Weight analysis obtained in the final cycle of the least squares refinement of V_2MoO_8. $\Delta = |F_{obs} - F_{calc}|$.

Interval sin θ	No. of reflections	$\overline{w\,\Delta^2}$	Interval F_{obs}	No. of reflections	$\overline{w\,\Delta^2}$
0.00–0.46	46	1.05	0–10	1	0.10
0.46–0.58	38	1.08	10–20	60	1.02
0.58–0.67	40	1.02	20–30	86	1.02
0.67–0.74	30	0.90	30–40	67	0.85
0.74–0.79	37	1.00	40–50	44	0.81
0.79–0.84	27	0.68	50–60	20	0.98
0.84–0.89	34	1.09	60–70	13	0.94
0.89–0.93	27	0.73	70–80	8	1.87
0.93–0.97	24	0.78	80–90	5	0.77
0.97–1.00	19	1.76	>90	18	1.65

of the plane $y = 0$ (at which they are fixed in $C2/m$). We think this shift is statistically significant and believe $C2$ to be the correct space group for this structure.

The refinements were also carried out both for the disordered (*a*) and ordered (*b*) alternatives. Since (*a*) gave the lowest R-value and most reasonable temperature factors, it was considered to be the most correct. However, the "temperature factors" obtained for the metal atoms in this case; *viz.*, $B_1 = 0.53 \pm 0.08$, $B_2 = 1.12 \pm 0.04$ Å^2, seemed to indicate the presence of some degree of ordering. The refinement was therefore continued using scattering curves corresponding to the following two partially ordered distributions; namely, $M_1 = (0.50\ V + 0.50\ Mo)$, $M_2 = (0.75\ V + 0.25\ Mo)$, and $M_1 = (0.33\ V + 0.67\ Mo)$, $M_2 = (0.83\ V + 0.17\ Mo)$. The first of these resulted in the lowest R-value and temperature factors which were most satisfactory (see Table 2). The temperature factors obtained for the second alternative were $B_1 = 1.64 \pm 0.08$, $B_2 = 0.66 \pm 0.04$ Å^2. Thus the metal atoms are assumed to be distributed over these two positions according to the first of these partially ordered alternatives.

The atomic parameters obtained by the least squares procedure were fairly satisfactory at this stage with one exception; namely, the temperature factor calculated for the oxygen atom O_5 which was rather high ($B = 2.7 \pm 0.3$ Å^2). One could suspect that this atom was subjected to the same type of displacement from the special position as is M_1 to which it is bonded. Therefore, this atom was released by splitting it into two symmetry-related halves. The temperature factor decreased markedly (see Table 2), and the position was simultaneously shifted by a statistically significant amount $-8\sigma_z$ in the z direction.

The ultimate discrepancy index (defined in the usual way and including observed reflections only) was $R = 0.064$. The corresponding atomic parameters are given in Table 2. A three-dimensional difference synthesis was calculated in 5000 points within the unique part of the unit cell on the basis of these parameters. This function was found to be completely featureless,

showing maxima and minima with a maximum magnitude of only 10 % and 15 %, respectively, of the lowest oxygen peak height in the corresponding ϱ_{obs}-function.

Observed and calculated structure factors have been listed in a separate document [15] which may be obtained from the secretary of this institute.

DESCRIPTION AND DISCUSSION OF THE STRUCTURE

The structure of V_2MoO_8 is a distorted variant of that reported recently for Nb_3O_7F by Andersson.[14] It can be described as a shear structure composed of octahedra forming ReO_3-type slabs which are infinitely long in the *b* and *c* directions, but only three octahedra thick. Such slabs are joined together by component octahedra having common edges across the planes $x = \frac{1}{4}$ and $x = \frac{3}{4}$ which can be described as shear planes. The structure is visualized in Fig. 1.

As demonstrated by Andersson,[14,16] this structure can be considered as the second member ($n = 3$) of a homologous series of structures with the general formula M_nO_{3n-1}. The number of octahedra, which is designated by the value of n and indicates the thickness of the ReO_3-type slabs, is the only difference among the idealized members of the series. The first member of this series ($n = 2$) is the idealized V_2O_5 structure.[17] Thus, the structural

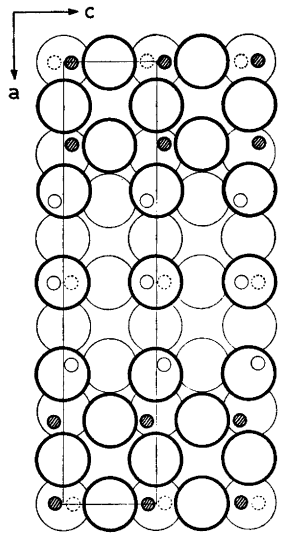

Fig. 1 a. The structure of V_2MoO_8 viewed along the *b* axis. Small circles = metal atoms, large circles = oxygen atoms. The positions indicated for the metal atoms at $x = 0, \frac{1}{2}, 1$ are chosen arbitrarily between the two alternatives (see Fig. 2).

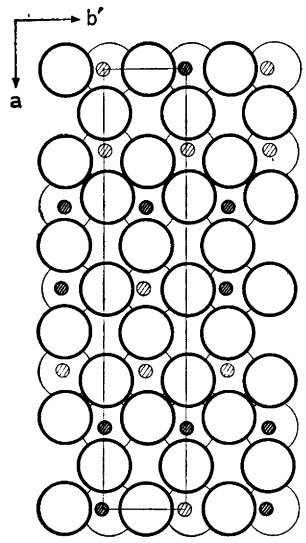

Fig. 1 b. The same viewed along the *c* axis.

Table 4. Interatomic distances (in Å) and bond angles in V_2MoO_8. Standard deviations in the last decimal places are given within parentheses. Corresponding distances in V_2O_5[19] and MoO_3[20] are also given.

Metal-oxygen distances in V_2MoO_8

M_1-O_5	1.653 (39)	1.732 (38)*	M_2-O_3	1.644 (11)	
$-O_4$	1.701 (5)		$-O_1$	1.715 (11)	
$-O_1$	1.969 (11)		$-O_2'$	1.873 (29)	
$-O_1'$	1.972 (11)		$-O_2$	1.941 (29)	
$-O_5'$	2.065 (38)	2.000 (39)*	$-O_2''$	2.096 (9)	
$-O_4'$	2.453 (4)		$-O_3'$	2.475 (11)	

		Bond angle at M_1		Distances between the oxygen atoms			Bond angle at M_2	Distances between the oxygen atoms
$O_1-M_1-O_1'$		157.5°				$O_1-M_2-O_2$	100.5°	2.81 (3)
O_1	O_4	99.9		2.82 (1)		O_1	O_2'	99.5	2.74 (3)
O_1	O_4'	79.3		2.85 (1)		O_1	O_2''	156.7	
O_1	O_5	89.5	89.2*	2.56 (4)	2.60 (4)*	O_1	O_3	102.8	2.63 (2)
O_1	O_5'	86.1	86.2*	2.76 (4)	2.71 (4)*	O_1	O_3'	79.0	2.73 (2)
O_1'	O_4	101.4		2.85 (1)		O_2	O_2'	144.2	
O_1'	O_4'	78.2		2.82 (1)		O_2	O_2''	74.3	2.44 (4)
O_1'	O_5	91.4	87.2*	2.60 (4)	2.56 (4)*	O_2	O_3	99.8	2.75 (3)
O_1'	O_5'	84.4	87.8*	2.71 (4)	2.76 (4)*	O_2	O_3'	76.4	2.76 (3)
O_4	O_4'	164.3				O_2'	O_2''	75.6	2.44 (4)
O_4	O_5	106.3	118.0*	2.68 (4)	2.94 (4)*	O_2'	O_3	104.3	2.78 (3)
O_4	O_5'	96.3	86.6*	2.82 (3)	2.55 (4)*	O_2'	O_3'	78.6	2.79 (3)
O_4'	O_5	89.4	77.7*	2.94 (4)	2.69 (4)*	O_2''	O_3	100.4	2.89 (1)
O_4'	O_5'	68.0	77.7*	2.55 (4)	2.82 (3)*	O_2''	O_3'	77.8	2.88 (1)
O_5	O_5'	157.3	155.5*			O_3	O_3'	176.1	

Metal-oxygen distances in V_2O_5		Metal-oxygen distances in MoO_3	
V—O	1.585 (4)	Mo—O	1.671 (7)
	1.780 (2)		1.734 (7)
	1.878 (2)		1.948 (2)
	1.878 (2)		1.948 (2)
	2.021 (3)		2.251 (7)
	2.785 (4)		2.332 (7)

* Refer to the 1st and 2nd alternative position of O_5, respectively. (See text and Fig. 2.)

relationship between V_2MoO_8 and V_2O_5 becomes apparent, even though both structures are considerably distorted from the ideal atomic arrangement and this distortion is quite different for V_2MoO_8 and V_2O_5. The relationships between these structures will be discussed further in a forthcoming paper in connection with the structure of the solid solutions $(V_{1-x}Mo_x)O_{2.5}$.[18]

The distortion of the octahedral coordination around the metal atoms, apparent in Fig. 1, is also demonstrated by the large variations in the metal-oxygen distances listed in Table 4. In general, the distortion is restricted

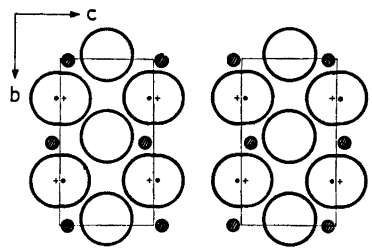

Fig. 2. The two alternative arrangements of atoms close to the planes $x = 0, \frac{1}{2}$, (displaced $\frac{1}{2}b' = \frac{1}{4}b$ at $x = \frac{1}{2}$) occurring in a random sequence in the structure. The ambiguity in the location of the oxygen atom O_5 is indicated in the figure by centres marked by crosses and dots for the two possible cases, respectively.

to the metal atoms which are displaced towards one of the octahedral edges, thereby shortening two and lengthening two other $M-O$ bonds. The coordination around M_1 and M_2 is essentially identical (see Table 4). A comparison with the corresponding bond lengths in V_2O_5 [19] and MoO_3,[20] also listed in Table 4, shows that the coordination in V_2MoO_8 is generally intermediate between that observed in these component phases.

The displacement of M_1 in the z direction is supposed to be opposite for atoms which differ in y by $\frac{1}{2}$ (of the true b axis), but have the same x coordinate. This is shown in Fig. 2.

The oxygen atom O_5 is probably displaced also in a similar way from the symmetrical position at $z = 0$, as was discussed previously. Although this displacement should also be completely ordered within the planes at $x = 0$ and $\frac{1}{2}$, it produces an ambiguity with respect to the relative positions of the atoms M_1 and O_5. Two alternative distances must therefore be given for each bond between these atoms (*cf.* Table 4). All four distances are reasonable but a comparison with those around M_2 as well as the $M-O$ distances in V_2O_5 and MoO_3 indicates that the first of the alternative positions of O_5 (marked by crosses in Fig. 2) gives rise to the most probable distances.

The observed non-periodicity in the x direction is restricted to the atoms at the planes $x = 0, \frac{1}{2}$ (see above). The two possible arrangements of these atoms are indicated in Fig. 2. The disorder can be explained by assuming that these alternatives occur in a random sequence in the direction of the a axis.

No degree of ordering along the a axis was observed in any of the 14 crystals examined. However, it is possible that an ordered structure might be obtained by using a different preparatory procedure or by extended thermal annealing.

In this connection, it should be mentioned that some of the crystals which were rejected for various reasons gave Weissenberg photographs in which also the reflections with k even were somewhat elongated. These crystals thus exhibited a tendency towards not only partial, but complete non-periodicity in the direction of the a axis.

In view of the evidently very weak ordering forces acting between the atoms at $x = 0$ and $x = \frac{1}{2}$, one could expect that twinning across (100) should occur. However, only twinning across (001) has been observed.

Because of the low degree of ordering of the two kinds of metal atoms over the two positions M_1 and M_2, a range of homogeneity, $(V_{2-x}Mo_{1+x})O_8$,

where $x \geq 0$, might be expected. Although the establishment of the true extension of the single phase region has been very difficult, the results obtained hitherto [6] seem to support such a supposition.

The oxygen-oxygen distances which range from 2.44 Å to 3.02 Å within the primary coordination spheres seem quite normal. The shortest of these distances occurs along the edge shared between two octahedra, O_2-O_2'. Such short shared edges are commonly observed when metal-metal bonding is absent and are in accordance with Pauling's rules.

Acknowledgements. The authors wish to thank Professor A. Magnéli for valuable comments on the manuscript and for all facilities put at their disposal.

This investigation has been financially supported by the *Swedish Natural Science Research Council*. The FACIT EDB computer was made available by the *Computer Division* of the *National Swedish Rationalization Agency*. H.A.E. acknowledges partial support of the *United States Atomic Energy Commission*.

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Received March 4, 1966.