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The Crystal Structure of Silver Decamolybdate, $Ag_6Mo_{10}O_{33}$†

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A study of the fused salt system Ag_2MoO_4–MoO_3 by X-ray diffraction techniques has shown the existence of two new phases, silver dimolybdate, $Ag_2Mo_2O_7$, and silver decamolybdate, $Ag_6Mo_{10}O_{33}$. Silver decamolybdate is triclinic, space group $P\bar{1}$, with lattice parameters $a = 7.59$, $b = 8.31$, $c = 11.42$ Å, $\alpha = 82.6°$, $\beta = 102.9°$ and $\gamma = 106.4°$; there is one formula weight in the unit cell. Its structure, determined by three-dimensional Patterson and Fourier methods and refined by least-squares techniques, contains distorted MoO_6 octahedra grouped by edge-sharing into sub units of four and six; these units then link up by common corners and edges to form infinite sheets. Some of the silver ions occupy irregular five-coordinate sites within the sheets, whilst the remainder occupy irregular seven-coordinate interlayer positions. Adjacent layers have no common oxygen atoms. Similarities to the red and blue potassium molybdenum bronzes, and to the hepta- and octa-molybdate polyanions are discussed.

Introduction

As part of a general study of the crystal chemistry of both simple and complex univalent-metal molybdates, an investigation of the fused salt system Ag_2MoO_4–MoO_3 using X-ray single crystal and powder diffraction techniques has shown the existence of two new phases, silver dimolybdate, $Ag_2Mo_2O_7$, and silver decamolybdate, $Ag_6Mo_{10}O_{33}$ (*1*). The latter crystallizes over quite a wide compositional range in the phase system and this led us to initially assume the formula $Ag_2Mo_3O_{10}$ (*2*). Kohlmuller and Faurie (*3*) in a more recent study of the phase system considered this compound to be the tetramolybdate, $Ag_2Mo_4O_{13}$. However, the correct, and rather unexpected formula $Ag_6Mo_{10}O_{33}$ has been determined by a complete crystal structure analysis which is reported here.

Experimental

The Ag_2MoO_4–MoO_3 phase system was studied by fusing a series of different mole ratio mixtures of the two components,‡ and then examining the slowly cooled melts optically and by X-ray diffraction techniques. Porcelain furnace ware was used in preference to platinum as the latter produced darkening of the cooled melts. It was possible to recognize two distinct, well crystallized phases under the microscope, and a subsequent X-ray single crystal study showed both to possess triclinic symmetry. An X-ray powder diffraction investigation was also made and our results are in general agreement with those reported by Kohlmuller and Faurie (*3*).

The two compounds, corresponding to the formulae $Ag_2Mo_2O_7$ and $Ag_6Mo_{10}O_{33}$, both crystallized as bright yellow, translucent prisms elongate about the *a* axis, and were commonly twinned. The face development was sufficient to allow rapid mounting of the crystals around each of the three crystallographic axes, but accurate optical goniometry was not attempted. The lattice parameters for both compounds are summarized in Table I; an accurate density could not be determined for the decamolybdate, $Ag_6Mo_{10}O_{33}$. The recorded value was measured pycnometrically for a sample of the crystalline melt of the same composition as the compound although this would certainly have contained some of the higher density dimolybdate.

Intensity data for $Ag_6Mo_{10}O_{33}$ were collected from a small prismatic crystal ($0.10 \times 0.02 \times 0.02$ mm) oriented for rotation about the *a* axis. Intensities for the levels *0kl*–*5kl* were recorded with CuK_α radiation on multiple films using the integrating Weissenberg technique, and measured by visual comparison with a calibrated strip. The *hOl* and *hkO* levels were also recorded to provide approximate interlayer scaling factors and to enable unambiguous indexing of the reflections. Specimen absorption

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‡ Ag_2MoO_4 was prepared in the standard manner, by precipitation from aqueous solutions of $AgNO_3$ and Na_2MoO_4.

TABLE I

CRYSTALLOGRAPHIC DATA FOR SILVER DECAMOLYBDATE AND SILVER DIMOLYBDATE

	$Ag_6Mo_{10}O_{33}$	$Ag_2Mo_2O_7$
Symmetry	Triclinic	Triclinic
Space Group	$P\bar{1}$	P1 or $P\bar{1}$
Unit-cell dimensions	$a = 7.59 \pm 0.02$ Å	6.11 ± 0.02 Å
	$b = 8.31 \pm 0.02$	7.49 ± 0.02
	$c = 11.42 \pm 0.02$	7.66 ± 0.02
	$\alpha = 82.6° \pm 0.2°$	$109.8° \pm 0.2°$
	$\beta = 102.9° \pm 0.2°$	$93.5° \pm 0.2°$
	$\gamma = 106.4° \pm 0.2°$	$113.6° \pm 0.2°$
D_x	5.32 g cm^{-3}	5.86 g cm^{-3}
D_m	5.5 ± 0.1 g cm^{-3}	–
Z	1	2

was neglected. Scattering curves were those of Thomas and Umeda (*4*) for Mo^0, Ag^+ from International Tables (*5*) and O^{2-} from Suzuki (*6*). The curves for Mo^0 and Ag^+ were corrected for dispersion. A total of 1267 nonsymmetry related reflections were measured and the intensities reduced to F by application of the usual Lorentz and polarization corrections.

Structure Determination

As the formula of this triclinic compound was initially unknown, the space group was assumed to be $P1$. Then by arbitrarily placing one of the metal atoms at the origin a direct image of the remaining atom positions would be present in the three-dimensional vector set. The similarity of the scattering power of silver and molybdenum allowed no differentiation between these atoms to be made in the solution of the metal atom structure, where the independent oxygen atoms were initially neglected. The largest peak in the three-dimensional Patterson map was located at $(\frac{1}{2},0,0)$, approximately 3.8 Å from the origin, and this was considered likely to result from a number of superposed Mo–Mo vectors, produced by corner shared MoO_6 octahedra. Three-dimensional Fourier and partial difference syntheses phased on the two metal atoms at (0,0,0) and $(\frac{1}{2},0,0)$ revealed a number of further possible metal atom positions, which were consistent with the Patterson map, and these were included in the next Fourier and partial difference syntheses. By this method a total of sixteen metal atoms were eventually located which gave an agreement value of $R = 0.263$, using a Mo^0 scattering curve for all atoms, and which reduced to $R = 0.162$ in four cycles of least-squares refinement. At this stage a difference synthesis revealed thirty three oxygen atoms, resulting in an octahedral coordination around ten of the metal atoms with metal–oxygen distances in the range 1.7–2.4 Å, which is typical for molybdenum, whilst the remaining six metal atoms occupied irregular five- or seven-coordinate positions with metal–oxygen distances in the range 2.3–2.9 Å which is typical for silver. The formula of the compound was thus determined to be $Ag_6Mo_{10}O_{33}$. Because of limited computer core-storage complete least-squares refinement of the 193 variables corresponding to an isotropic temperature coordinate model was not possible, therefore a number of refinement steps were carried out. First, the scale factors and metal atom coordinates were varied, then the scale factors and oxygen atom coordinates were varied and finally all the isotropic temperature factors were varied, which resulted in an R value of 0.086. However, the temperature factors for two of the molybdenum and five of the oxygen atoms went negative, although each by less than their respective standard deviations.

An examination of this $P1$ structure revealed a centre of symmetry with one of the oxygen atoms located on this inversion centre. As a check, before the structure was refined in the centrosymmetric space group, a difference Fourier phased on the independent three silver and five molybdenum atoms in the asymmetric unit of $P\bar{1}$ was calculated. This showed sixteen independent oxygen atom positions, with a seventeenth lying, as expected, at the origin; this oxygen model was the same as that found in the non-centric space group.

All the position coordinates (except for the oxygen atom on the origin) together with individual temperature factors were then refined by full matrix least-squares to an agreement of $R = 0.120$. A difference synthesis indicated that the thermal motion of the silver atoms was anisotropic, hence a final least-squares refinement was performed with the silver atoms allowed to vibrate anisotropically, but with the molybdenum and oxygen atoms restricted to isotropic motion. R had then been reduced to 0.076 and successive shifts were now <10% of the standard errors. All the temperature factors in this $P\bar{1}$ refinement remained positive definite. Until the final least-squares cycles of refinement all reflections had been assigned unit weight, a weight analysis subsequently showed this to be satisfactory except for a small number of the strongest reflections which were then assigned individual weights. Observed and calculated structure factors are listed in Table II. The final positional and isotropic

thermal parameters are given in Table III, whilst the anisotropic temperature coordinates for the silver atoms are shown in Table IV.

A three-dimensional total-difference synthesis computed at this stage showed no maxima $> \frac{1}{5}$ nor minima $< -\frac{1}{5}$ of the mean electron density value for the observed oxygen atoms in the structure. All calculations were made on the Monash University CDC 3200 computer.

Discussion

Although refinement of the structure in each of the alternative space groups $P1$ and $P\bar{1}$ resulted in similar R values, and extremely similar atomic arrangements, the centrosymmetric space group was chosen as the temperature factors all remained positive definite after refinement in this space group.

The description of the structure is made easier if the octahedral coordination of the molybdenum atoms is considered to be regular. The structure can be broken down into two fundamental groups, one of six octahedra sharing edges and the other a square of four octahedra sharing edges. Each group of six octahedra joins up with identical groups by edge-sharing to form infinite zig-zag chains running in the a axis direction. The groups of four octahedra lie between these chains linking them by corner-sharing, to form infinite two-dimensional sheets parallel to the (010) plane. As seen in Fig. 1, this

TABLE II

OBSERVED AND CALCULATED STRUCTURE FACTORS

H	K	L	F(OBS)	F(CAL)	H	K	L	F(OBS)	F(CAL)	H	K	L	F(OBS)	F(CAL)	H	K	L	F(OBS)	F(CAL)
0	0	2	1.6	1.8	0	-3	2	2.6	-2.3	0	-5	-5	3.4	-3.0	0	-9	2	2.9	2.9
0	0	4	9.0	11.2	0	-3	3	4.5	4.3	0	-5	-6	4.5	4.2	0	-9	4	2.3	1.7
0	0	5	1.3	.9	0	-3	4	5.4	-5.5	0	-5	-8	3.7	3.4	0	-9	-1	2.3	-1.9
0	0	6	3.1	3.0	0	-3	5	5.0	-5.1	0	-5	-9	1.4	-1.4	0	-9	-2	2.3	1.9
0	0	7	4.5	-4.3	0	-3	7	1.9	1.6	0	-5	-10	2.3	1.9	0	-9	-4	2.8	2.5
0	0	8	6.1	6.1	0	-3	9	2.8	-2.7	0	-5	-11	4.3	-3.6	0	-9	-5	3.5	-3.3
0	0	9	1.4	1.5	0	-3	12	3.5	3.4	0	-6	0	5.4	5.1	0	-9	-6	2.7	2.4
0	0	10	2.8	2.5	0	-3	-1	3.0	-2.8	0	-6	1	2.6	2.4	0	-9	1	1.1	-0.7
0	0	11	4.2	-3.8	0	-3	-2	4.3	-4.5	0	-6	2	5.5	-5.4	1	0	0	.3	-0.4
0	0	12	2.8	2.6	0	-3	-3	5.3	5.6	0	-6	3	3.1	2.8	1	0	1	1.1	1.0
0	-1	0	3.0	2.9	0	-3	-4	6.9	-7.4	0	-6	5	3.5	3.4	1	0	2	3.4	3.8
0	-1	1	1.3	-1.0	0	-3	-5	2.4	-2.6	0	-6	6	3.4	-3.2	1	0	3	2.0	-2.1
0	-1	2	1.7	1.5	0	-3	-6	4.0	-4.2	0	-6	8	2.8	-2.4	1	0	4	5.7	-5.6
0	-1	3	4.6	4.7	0	-3	-7	2.2	2.1	0	-6	9	5.0	4.9	1	0	6	4.1	4.0
0	-1	4	.8	-0.5	0	-3	-8	3.8	-3.2	0	-6	10	2.2	-1.9	1	0	7	4.6	-4.3
0	-1	5	3.9	-3.9	0	-3	-10	3.1	-2.9	0	-6	-1	2.6	2.4	1	0	8	4.4	-4.4
0	-1	7	4.1	4.0	0	-3	-11	2.3	2.1	0	-6	-2	4.2	-3.9	1	0	11	2.7	-2.3
0	-1	8	2.6	-2.1	0	-4	0	9.8	10.5	0	-6	-3	4.0	3.9	1	0	12	2.4	-2.6
0	-1	10	2.8	-2.9	0	-4	1	1.4	-1.3	0	-6	-5	1.7	1.5	1	0	13	3.6	-3.6
0	-1	13	2.0	1.6	0	-4	2	1.5	-1.2	0	-6	-9	1.8	1.4	1	0	-2	1.2	1.1
0	-1	-1	1.4	-1.3	0	-4	3	2.5	-2.1	0	-7	0	4.0	-3.8	1	0	-3	4.1	4.8
0	-1	-2	3.4	3.6	0	-4	4	7.9	8.4	0	-7	2	4.9	-4.7	1	0	-4	2.7	2.8
0	-1	-3	4.0	4.3	0	-4	5	1.2	-0.7	0	-7	3	1.4	1.3	1	0	-5	2.0	1.7
0	-1	-4	2.3	2.2	0	-4	6	3.2	3.1	0	-7	4	4.4	-4.2	1	0	-6	4.0	4.0
0	-1	-5	4.1	-4.1	0	-4	7	4.2	-4.1	0	-7	5	2.3	-2.4	1	0	-7	4.3	4.1
0	-1	-6	4.0	4.2	0	-4	8	3.7	3.7	0	-7	6	1.8	-1.9	1	-1	0	1.4	1.2
0	-1	-7	1.2	1.3	0	-4	10	2.9	2.7	0	-7	8	3.3	-3.1	1	-1	1	1.6	-1.6
0	-1	-8	3.4	3.4	0	-4	11	5.0	-5.1	0	-7	9	1.3	-1.4	1	-1	2	.9	.6
0	-1	-9	1.9	-2.0	0	-4	12	2.6	2.2	0	-7	-1	1.4	-1.6	1	-1	4	1.5	-1.2
0	-1	-10	2.0	1.7	0	-4	-1	2.2	-2.1	0	-7	-2	5.5	-5.5	1	-1	6	1.5	-1.6
0	-2	2	14.5	-15.7	0	-4	-2	1.7	-1.4	0	-7	-3	3.2	2.9	1	-1	7	5.1	-5.1
0	-2	3	2.3	2.2	0	-4	-4	5.7	5.7	0	-7	-4	3.5	-2.8	1	-1	9	3.1	3.0
0	-2	4	3.2	-3.1	0	-4	-5	2.4	-2.2	0	-7	-6	3.4	-3.2	1	-1	10	2.4	-2.2
0	-2	5	3.5	3.4	0	-4	-6	1.8	1.6	0	-7	-7	2.3	1.9	1	-1	11	4.5	-4.5
0	-2	6	7.7	-8.2	0	-4	-7	1.9	-1.7	0	-7	-8	3.3	-2.9	1	-1	12	1.4	1.0
0	-2	7	1.8	-1.7	0	-4	-8	2.0	1.8	0	-7	-9	3.1	2.5	1	-1	13	1.4	1.8
0	-2	8	3.8	-3.5	0	-4	-11	1.3	-1.1	0	-7	-10	2.8	-2.8	1	-1	-1	1.9	2.0
0	-2	9	4.2	4.0	0	-4	-12	2.2	2.3	0	-8	0	2.4	2.2	1	-1	-2	.8	.5
0	-2	10	3.1	-2.9	0	-5	0	7.5	8.1	0	-8	1	3.6	-3.7	1	-1	-3	4.6	4.7
0	-2	13	4.4	4.1	0	-5	2	4.9	5.1	0	-8	2	2.3	-2.5	1	-1	-4	3.5	3.5
0	-2	-2	14.5	-15.1	0	-5	3	3.9	3.8	0	-8	3	2.3	-1.8	1	-1	-6	3.8	4.1
0	-2	-3	2.2	2.3	0	-5	4	2.6	2.2	0	-8	4	1.2	1.1	1	-1	-7	7.9	8.1
0	-2	-4	.9	-1.0	0	-5	5	1.4	-1.4	0	-8	5	2.8	-2.6	1	-1	-10	3.1	2.9
0	-2	-5	1.7	1.4	0	-5	6	3.7	3.6	0	-8	7	3.6	-3.8	1	-2	0	1.7	-1.5
0	-2	-6	5.7	-6.2	0	-5	7	2.8	2.9	0	-8	-1	3.6	-3.3	1	-2	2	3.9	3.6
0	-2	-8	3.4	-3.1	0	-5	8	2.0	2.0	0	-8	-2	2.7	-2.8	1	-2	3	1.2	-1.2
0	-2	-9	3.1	2.8	0	-5	-1	1.1	-0.5	0	-8	-3	1.4	-1.3	1	-2	4	4.7	-4.6
0	-2	-10	3.2	-3.0	0	-5	-2	5.2	4.8	0	-8	-5	2.6	-2.6	1	-2	5	3.0	2.8
0	-3	0	3.3	-3.0	0	-5	-3	2.7	2.8	0	-8	-8	2.0	-1.7	1	-2	6	6.0	6.0
0	-3	1	3.4	-3.1	0	-5	-4	5.1	4.9	0	-9	0	4.5	4.4	1	-2	8	2.3	-2.2

TABLE II—*continued*

H	K	L	F(OBS)	F(CAL)	H	K	L	F(OBS)	F(CAL)	H	K	L	F(OBS)	F(CAL)	H	K	L	F(OBS)	F(CAL)
1	-2	9	3,4	3,1	1	-5	7	4.2	-4.0	1	-9	0	.9	-1.3	1	3	-1	.9	.9
1	-2	10	3,1	2,9	1	-5	8	1.4	-1.3	1	-9	2	1.4	1.5	1	3	-3	1.2	.9
1	-2	11	2,6	2,6	1	-5	9	1.3	1.2	1	-9	3	1.1	1.1	1	4	1	2.1	2.0
1	-2	12	1,9	1,6	1	-5	10	3.4	-3.2	1	-9	4	2.4	-2.4	1	4	2	3.4	3.2
1	-2	-1	,8	-0,5	1	-5	11	4.0	-4.1	1	-9	-2	.9	.8	1	4	3	2.2	-2.1
1	-2	-2	2,5	-2,3	1	-5	-3	4.4	4.4	1	-9	-3	1.2	1.1	1	4	4	5.5	-5.3
1	-2	-4	1,6	-1,6	1	-5	-4	1.7	1.3	1	-9	-5	2.5	-2.8	1	4	6	2.9	2.7
1	-2	-5	2,7	-2,5	1	-5	-5	2.2	-2.0	1	-10	0	.9	1.0	1	4	7	3.6	-3.3
1	-2	-6	1,0	1,0	1	-5	-6	2.9	2.9	1	-10	1	.5	.2	1	4	8	3.7	-3.6
1	-2	-7	1,1	,9	1	-5	-7	5.7	6.0	1	-10	-1	2.3	2.4	1	4	11	1.4	-1.3
1	-2	-8	3,8	-3,9	1	-5	-8	2.0	1.4	1	-10	-2	.9	-0.9	1	4	-1	2.3	-2.0
1	-2	-10	3,6	3,5	1	-5	-9	1.4	-0.9	1	-10	-3	1.7	1.5	1	4	-3	4.0	3.9
1	-3	0	2,6	-2,4	1	-5	-10	2.3	2.2	1	-10	-4	1.7	1.8	1	4	-4	2.9	2.8
1	-3	1	,8	-1,0	1	-5	-11	2.1	2.2	1	1	0	.8	.7	1	5	0	.9	.9
1	-3	3	,9	,7	1	-6	4	2.4	-2.1	1	1	1	1.9	-2.0	1	5	1	2.7	-2.5
1	-3	4	1,5	-1,0	1	-6	5	1.4	1.0	1	1	5	1.4	1.3	1	5	2	1.2	-1.1
1	-3	5	3,8	3,7	1	-6	6	2.4	2.1	1	1	6	1.5	-1.4	1	5	4	1.9	-1.5
1	-3	6	3,9	3,7	1	-6	7	2.3	-2.4	1	1	7	3.7	-3.7	1	5	6	2.4	-2.5
1	-3	8	2,8	2,8	1	-6	8	1.3	-1.4	1	1	9	4.3	4.4	1	5	7	4.4	-4.5
1	-3	9	5,9	5,5	1	-6	9	1.1	1.0	1	1	10	2.4	-2.3	1	5	8	1.0	-0.9
1	-3	12	3,0	2,4	1	-6	-1	2.5	2.4	1	1	11	4.0	-4.0	1	5	9	.9	1.1
1	-3	-1	3,2	-2,9	1	-6	-4	2.3	2.2	1	1	12	1.4	1.2	1	5	10	3.6	-3.5
1	-3	-2	,8	,7	1	-6	-5	3.6	3.7	1	1	13	1.3	1.5	1	5	11	3.6	-4.0
1	-3	-3	2,3	2,0	1	-6	-6	2.4	2.5	1	1	-1	1.1	1.0	1	5	-1	3.2	3.0
1	-3	-4	1,3	-1,0	1	-6	-7	3.7	3.5	1	1	-2	2.1	-2.0	1	5	-3	2.2	2.1
1	-3	-5	7,2	-7,7	1	-6	-8	1.4	-1.3	1	2	0	2.7	-2.5	1	6	2	2.5	2.0
1	-3	-6	2,9	-2,7	1	-6	-9	2.3	2.4	1	2	1	1.3	1.2	1	6	4	2.8	-2.6
1	-3	-7	1,7	1,3	1	-6	-10	4.0	4.5	1	2	2	3.9	3.7	1	6	5	2.0	1.9
1	-3	-8	3,2	-3,0	1	-6	-11	1.9	1.7	1	2	3	1.5	-1.4	1	6	6	2.8	2.6
1	-3	-9	6,6	-6,5	1	-7	0	1.7	-1.4	1	2	4	5.4	-5.4	1	6	8	1.3	-1.4
1	-3	-10	1,4	-1,7	1	-7	1	2.0	-1.4	1	2	5	5.0	4.9	1	6	9	2.0	1.7
1	-4	1	1,0	-0,8	1	-7	2	2.4	2.4	1	2	6	6.9	7.3	1	6	-2	1.7	-1.3
1	-4	2	3,0	2,8	1	-7	4	1.7	-1.5	1	2	8	1.9	-1.2	1	6	-3	2.4	2.3
1	-4	3	1,1	-0,7	1	-7	6	2.3	2.1	1	2	9	3.4	3.2	1	7	0	1.4	1.1
1	-4	4	4,0	-3,8	1	-7	8	1.3	1.3	1	2	10	2.8	3.1	1	7	1	2.0	-1.9
1	-4	5	1,8	1,5	1	-7	-3	1.7	1.6	1	2	11	2.3	2.2	1	7	5	1.7	1.6
1	-4	6	3,0	2,9	1	-7	-4	1.0	-0.6	1	2	12	1.6	1.3	1	7	6	2.4	2.2
1	-4	7	2,0	-2,0	1	-7	-5	3.1	-3.3	1	2	13	1.1	1.1	1	7	8	2.2	2.5
1	-4	8	3,1	-3,0	1	-7	-7	3.0	3.0	1	2	-1	3.5	-3.5	1	7	9	3.2	3.8
1	-4	-4	1,5	1,2	1	-7	-8	1.8	-1.7	1	2	-3	3.2	3.1	1	7	-3	2.2	-1.8
1	-4	-5	2,3	-2,2	1	-7	-9	1.7	-1.6	1	3	0	.9	.8	1	8	2	2.0	2.1
1	-4	-6	1,7	1,2	1	-8	0	1.0	.4	1	3	1	2.5	-2.4	1	8	4	1.9	-1.8
1	-4	-8	3,6	-3,7	1	-8	1	2.4	-1.9	1	3	3	.7	.6	1	8	5	1.4	1.6
1	-4	-9	2,0	-2,0	1	-8	5	1.7	1.5	1	3	4	.7	.8	1	8	6	3.2	3.3
1	-4	-10	2,8	3,0	1	-8	6	3.0	3.2	1	3	5	3.0	3.0	1	8	-1	1.3	-1.4
1	-4	-11	2,6	-2,5	1	-8	-2	2.0	-2.0	1	3	6	1.9	1.9	1	8	-2	.9	-1.0
1	-4	-12	3,6	-3,6	1	-8	-3	2.4	-2.6	1	3	8	2.8	3.1	1	9	4	1.8	-1.7
1	-5	0	1,1	-0,6	1	-8	-5	3.3	-3.1	1	3	9	5.2	5.2	2	0	0	1.2	-1.2
1	-5	2	2,3	2,2	1	-8	-7	2.3	-2.6	1	3	11	1.3	-1.2	2	0	1	4.6	-5.1
1	-5	4	3,1	-2,8	1	-8	-8	3,1	-3.3	1	3	12	3.1	3.0	2	0	2	3.0	3.1

TABLE II—*continued*

H	K	L	F(OBS)	F(CAL)	H	K	L	F(OBS)	F(CAL)	H	K	L	F(OBS)	F(CAL)	H	K	L	F(OBS)	F(CAL)
2	0	3	3.0	-2.9	2	-2	-6	4.6	-4.5	2	-5	10	1.5	-1.4	2	-8	2	3.0	3.1
2	0	4	2.5	-2.3	2	-2	-8	3.0	2.9	2	-5	-1	1.3	1.2	2	-8	5	3.9	3.7
2	0	5	2.7	-2.8	2	-3	1	1.0	.7	2	-5	-2	3.6	3.3	2	-8	6	3.3	3.2
2	0	6	2.0	1.9	2	-3	2	2.6	2.4	2	-5	-3	8.1	-8.6	2	-8	-1	3.4	3.1
2	0	7	5.1	-4.8	2	-3	3	9.0	10.4	2	-5	-4	2.1	1.8	2	-8	-3	1.9	1.7
2	0	8	1.9	1.7	2	-3	5	4.4	4.1	2	-5	-6	3.1	2.7	2	-8	-4	1.4	1.2
2	0	9	5.4	-5.2	2	-3	7	4.6	4.6	2	-5	-7	3.8	-3.8	2	-8	-5	2.7	2.5
2	0	11	.9	-0.8	2	-3	8	2.8	2.6	2	-5	-8	1.4	1.2	2	-8	-6	3.2	-3.0
2	0	12	2.6	2.1	2	-3	9	2.4	1.9	2	-5	-9	2.4	-1.9	2	-8	-7	2.1	1.9
2	0	-1	3.5	-3.5	2	-3	10	1.8	-1.4	2	-5	-10	3.8	3.4	2	-8	-9	1.2	1.1
2	0	-2	.9	-0.6	2	-3	11	4.3	4.0	2	-5	-11	1.3	-1.2	2	-8	-10	1.8	-1.7
2	0	-3	3.4	-3.6	2	-3	-1	11.0	11.9	2	-6	0	3.0	-3.0	2	-9	0	2.5	2.5
2	0	-4	3.7	3.5	2	-3	-2	1.7	1.4	2	-6	1	2.0	-1.7	2	-9	1	3.2	-3.0
2	0	-5	1.1	-1.0	2	-3	-3	1.8	-1.5	2	-6	3	2.3	-2.3	2	-9	2	3.9	3.9
2	0	-6	3.6	-3.5	2	-3	-4	3.7	-3.7	2	-6	4	4.7	-4.7	2	-9	4	2.8	3.0
2	0	-7	1.1	-1.0	2	-3	-5	7.1	8.1	2	-6	5	1.0	.9	2	-9	5	1.4	-1.4
2	0	-8	3.5	2.8	2	-3	-6	1.5	-1.3	2	-6	6	1.0	-1.0	2	-9	-2	3.0	3.0
2	-1	0	2.9	-2.8	2	-3	-7	2.3	2.3	2	-6	7	2.3	-2.1	2	-9	-3	3.2	-3.2
2	-1	1	12.1	-13.2	2	-3	-8	3.8	-3.9	2	-6	8	2.7	-2.8	2	-9	-4	2.7	2.2
2	-1	2	1.5	1.4	2	-3	-9	3.0	2.3	2	-6	10	2.4	-1.7	2	-9	-6	1.1	.8
2	-1	3	1.2	.9	2	-3	-11	1.4	1.4	2	-6	-1	3.3	-3.1	2	-9	-7	.7	-0.8
2	-1	4	.9	-0.9	2	-4	0	3.5	3.2	2	-6	-2	2.8	-2.6	2	-9	-8	1.7	1.6
2	-1	5	5.3	-5.6	2	-4	1	3.0	2.8	2	-6	-3	3.1	-3.0	2	-10	1	2.1	-1.9
2	-1	6	1.1	-1.2	2	-4	2	5.9	5.7	2	-6	-4	1.3	1.3	2	-10	-1	2.1	-2.0
2	-1	7	3.3	-3.1	2	-4	3	1.5	-1.4	2	-6	-5	1.3	-1.1	2	-10	-3	2.6	-2.6
2	-1	8	.9	.8	2	-4	5	2.2	2.0	2	-6	-6	2.7	-2.5	2	-10	-4	2.5	2.4
2	-1	9	3.7	-3.7	2	-4	6	4.6	4.6	2	-6	-7	1.4	-1.4	2	1	0	2.7	-2.6
2	-1	10	1.7	-1.3	2	-4	7	2.2	-1.8	2	-6	-8	2.8	2.7	2	1	2	1.8	1.8
2	-1	-2	3.1	3.1	2	-4	8	1.7	1.3	2	-6	-9	2.1	-1.7	2	1	3	4.9	5.0
2	-1	-3	11.1	-12.1	2	-4	9	1.6	-1.6	2	-6	-10	2.2	2.0	2	1	4	1.1	-1.1
2	-1	-4	2.5	-2.6	2	-4	10	2.1	1.8	2	-6	-11	1.1	-1.0	2	1	5	1.8	1.9
2	-1	-5	1.8	1.5	2	-4	-3	1.5	1.1	2	-6	-12	2.0	2.0	2	1	7	2.5	2.3
2	-1	-6	3.8	4.0	2	-4	-4	3.9	3.9	2	-7	0	3.6	-3.3	2	1	11	2.0	1.7
2	-1	-7	4.6	-4.3	2	-4	-5	2.1	2.1	2	-7	2	1.7	-1.4	2	1	-1	10.3	11.9
2	-1	-9	3.8	-3.3	2	-4	-6	3.5	-3.5	2	-7	3	5.9	5.7	2	1	-2	1.3	1.1
2	-1	-10	6.7	6.5	2	-4	-7	1.8	1.7	2	-7	4	1.9	-1.5	2	1	-3	1.6	-1.3
2	-2	0	4.1	-3.6	2	-4	-8	3.5	3.4	2	-7	5	2.3	1.7	2	2	0	1.5	1.4
2	-2	1	2.7	2.4	2	-4	-9	1.4	1.1	2	-7	6	3.3	-3.1	2	2	1	5.9	6.1
2	-2	2	1.0	.7	2	-4	-10	2.0	-1.9	2	-7	7	3.5	3.1	2	2	2	3.6	4.0
2	-2	4	4.4	-4.5	2	-4	-12	2.9	-2.8	2	-7	8	1.0	1.4	2	2	3	6.9	7.0
2	-2	5	4.1	4.0	2	-5	0	1.1	-0.5	2	-7	-1	6.6	6.7	2	2	4	3.0	-2.8
2	-2	7	2.6	2.3	2	-5	1	9.4	-10.0	2	-7	-2	1.9	-1.8	2	2	5	5.8	6.2
2	-2	8	1.4	-1.4	2	-5	2	2.0	1.6	2	-7	-4	3.6	-3.2	2	2	6	1.2	1.2
2	-2	9	2.0	1.8	2	-5	3	1.7	-1.4	2	-7	-5	4.4	4.5	2	2	7	4.4	4.5
2	-2	10	1.9	-1.7	2	-5	4	1.3	.9	2	-7	-6	2.0	-1.7	2	2	9	3.1	2.7
2	-2	11	4.1	3.7	2	-5	5	5.7	-5.6	2	-7	-7	1.4	1.4	2	2	10	2.6	-2.1
2	-2	12	2.4	-2.0	2	-5	6	1.4	-1.3	2	-7	-8	1.9	-1.4	2	2	11	5.0	4.6
2	-2	-1	1.9	1.9	2	-5	7	3.4	-3.0	2	-7	-9	1.2	1.2	2	2	12	1.4	-1.3
2	-2	-2	3.2	-3.2	2	-5	8	3.3	2.8	2	-7	-11	1.9	1.7	2	2	-1	4.2	4.2
2	-2	-4	3.0	2.8	2	-5	9	4.7	-4.2	2	-8	1	3.0	3.2	2	2	-3	2.8	2.7

TABLE II—*continued*

H	K	L	F(OBS)	F(CAL)	H	K	L	F(OBS)	F(CAL)	H	K	L	F(OBS)	F(CAL)	H	K	L	F(OBS)	F(CAL)
2	2	-4	1.9	1.6	2	8	5	1.4	-1.2	3	-2	-10	4.2	-3.9	3	-5	10	.9	.9
2	3	1	5.0	-4.7	2	8	6	2.2	2.3	3	-3	0	3.7	3.4	3	-5	-1	1.5	-1.1
2	3	2	3.3	3.1	2	8	-1	2.3	-2.2	3	-3	1	4.7	4.9	3	-5	-2	1.0	-1.0
2	3	6	1.8	1.7	3	0	0	2.5	-2.7	3	-3	2	1.5	-1.4	3	-5	-4	1.4	1.2
2	3	8	1.4	1.0	3	0	2	1.3	1.2	3	-3	3	4.1	-4.2	3	-5	-8	2.8	2.7
2	3	11	2.7	2.5	3	0	4	1.0	.8	3	-3	4	3.8	3.8	3	-5	-9	1.3	-1.2
2	3	-1	1.9	1.8	3	0	6	2.4	-2.7	3	-3	5	3.5	3.3	3	-5	-11	3.6	3.2
2	3	-2	2.8	2.5	3	0	7	3.4	3.4	3	-3	6	3.1	-2.9	3	-5	-12	1.9	1.7
2	3	-3	6.8	-6.8	3	0	8	3.0	2.7	3	-3	7	4.2	-4.1	3	-6	0	3.2	-3.1
2	3	-4	1.5	1.5	3	0	9	1.3	-1.4	3	-3	11	1.9	-1.9	3	-6	2	1.8	-1.6
2	4	0	2.7	-2.9	3	0	10	2.0	-1.9	3	-3	-2	1.2	-1.1	3	-6	6	1.9	-1.5
2	4	1	7.0	-7.2	3	0	11	3.5	3.1	3	-3	-3	1.3	1.2	3	-6	7	2.8	2.8
2	4	2	2.8	2.6	3	0	12	2.2	2.5	3	-3	-4	3.2	3.2	3	-6	8	3.1	2.7
2	4	3	5.1	-5.0	3	0	-2	1.5	1.4	3	-3	-5	3.0	2.9	3	-6	9	.5	-0.7
2	4	4	1.3	-1.5	3	-1	0	.8	.8	3	-3	-6	1.3	-1.0	3	-6	-1	1.7	-1.6
2	4	5	5.1	-4.9	3	-1	1	3.0	3.1	3	-3	-7	1.6	1.2	3	-6	-2	1.7	-1.6
2	4	6	1.4	1.1	3	-1	2	3.9	-4.8	3	-3	-8	3.7	3.6	3	-6	-3	2.7	-2.6
2	4	7	3.7	-3.5	3	-1	3	4.3	-4.8	3	-3	-10	1.9	-1.8	3	-6	-4	1.8	-1.6
2	4	8	1.7	1.3	3	-1	4	2.8	2.7	3	-3	-11	3.8	3.3	3	-6	-5	1.8	-1.5
2	4	9	4.4	-4.3	3	-1	5	4.2	4.1	3	-4	0	2.0	1.7	3	-6	-6	5.0	-5.0
2	4	-1	4.3	-4.0	3	-1	6	2.1	-2.0	3	-4	1	3.3	3.0	3	-6	-7	3.3	-3.0
2	4	-3	5.9	-5.3	3	-1	7	.9	-1.2	3	-4	2	1.8	1.6	3	-6	-9	2.3	-1.9
2	4	-4	1.7	1.6	3	-1	8	3.5	3.1	3	-4	4	2.0	1.6	3	-6	-10	3.9	-3.8
2	5	0	2.5	-2.6	3	-1	9	2.7	2.6	3	-4	5	1.8	-1.7	3	-7	0	1.6	-1.1
2	5	1	2.5	-2.8	3	-1	10	1.5	1.2	3	-4	6	2.6	-2.5	3	-7	1	1.9	1.8
2	5	4	2.7	-2.6	3	-1	11	1.9	1.6	3	-4	7	1.3	1.0	3	-7	3	2.7	-2.2
2	5	7	3.0	-3.0	3	-1	-2	2.0	-2.1	3	-4	8	2.6	2.5	3	-7	5	2.4	2.4
2	5	9	2.2	-2.3	3	-1	-3	3.0	-3.3	3	-4	9	3.2	-3.2	3	-7	6	2.1	-1.8
2	5	-1	2.5	2.4	3	-1	-5	2.5	-2.4	3	-4	10	2.6	-2.6	3	-7	7	1.1	-1.1
2	5	-2	1.6	-1.3	3	-1	-6	5.8	-6.3	3	-4	11	1.4	1.2	3	-7	8	.9	1.0
2	5	-3	2.6	-2.7	3	-1	-7	3.2	-3.3	3	-4	-1	2.8	2.6	3	-7	-3	1.3	-1.3
2	5	-4	2.8	-2.7	3	-1	-9	4.0	-3.7	3	-4	-2	4.0	4.0	3	-7	-4	1.6	-1.5
2	6	0	2.7	-2.7	3	-2	0	2.8	-3.0	3	-4	-3	1.3	1.1	3	-7	-6	3.0	-3.1
2	6	1	3.4	3.1	3	-2	1	1.6	-1.5	3	-4	-4	4.9	4.6	3	-7	-9	3.5	-3.3
2	6	3	5.1	4.7	3	-2	2	1.2	1.0	3	-4	-5	4.1	4.2	3	-7	-10	2.4	-2.3
2	6	4	3.1	-2.8	3	-2	4	.7	.6	3	-4	-7	2.7	2.5	3	-7	-11	1.8	1.7
2	6	5	3.3	3.2	3	-2	6	3.0	-2.8	3	-4	-8	5.4	5.1	3	-8	1	1.6	1.5
2	6	7	2.8	3.1	3	-2	7	2.6	2.6	3	-4	-9	2.3	2.2	3	-8	2	1.8	1.7
2	6	9	1.4	1.3	3	-2	8	3.3	3.0	3	-4	-11	2.9	2.4	3	-8	4	1.4	1.3
2	6	-1	6.3	5.9	3	-2	9	3.2	-3.2	3	-4	-12	2.9	2.6	3	-8	6	1.9	-1.7
2	6	-3	3.1	2.9	3	-2	10	2.1	-2.1	3	-5	0	1.1	1.1	3	-8	-2	1.3	1.1
2	7	0	1.7	1.8	3	-2	11	2.1	1.9	3	-5	1	1.9	1.7	3	-8	-3	.9	.9
2	7	2	2.7	2.8	3	-2	-1	.7	-0.6	3	-5	2	1.1	-1.2	3	-8	-4	2.6	2.5
2	7	3	1.9	2.0	3	-2	-2	.4	.4	3	-5	3	4.2	-4.1	3	-8	-5	2.3	2.0
2	7	5	3.7	3.5	3	-2	-3	1.4	-1.4	3	-5	4	2.2	2.2	3	-8	-8	3.6	3.6
2	7	7	1.5	1.7	3	-2	-4	2.0	-1.9	3	-5	5	4.0	3.9	3	-8	-10	.9	-0.9
2	7	-1	3.3	3.4	3	-2	-5	2.3	-2.2	3	-5	6	2.3	-2.2	3	-9	1	2.0	2.2
2	7	-2	1.6	1.7	3	-2	-6	4.1	-4.2	3	-5	7	1.9	-2.0	3	-9	3	1.4	-1.4
2	8	1	3.4	-3.0	3	-2	-7	2.4	-2.2	3	-5	8	1.2	1.1	3	-9	4	1.6	1.7
2	8	2	3.9	3.8	3	-2	-9	2.9	-2.8	3	-5	9	1.8	1.5	3	-9	-2	3.2	2.9

TABLE II—*continued*

H	K	L	F(OBS)	F(CAL)	H	K	L	F(OBS)	F(CAL)	H	K	L	F(OBS)	F(CAL)	H	K	L	F(OBS)	F(CAL)
3	-9	-4	2.2	2.0	3	5	2	1.3	-1.3	4	-2	-4	1.2	-1.1	4	-5	9	2.7	-2.4
3	-9	-5	2.1	2.1	3	5	3	1.6	-1.6	4	-2	-5	3.8	-3.7	4	-5	-1	3.3	-3.0
3	-9	-6	1.9	2.1	3	5	5	2.1	2.2	4	-2	-6	5.2	5.4	4	-5	-2	1.4	1.6
3	-9	-7	1.7	1.9	3	5	6	2.3	-2.3	4	-2	-8	2.3	2.4	4	-5	-3	3.0	-2.8
3	-9	-8	2.7	3.0	3	5	8	1.1	1.4	4	-2	-9	5.7	-5.9	4	-5	-5	3.3	3.2
3	-10	1	1.3	1.5	3	5	-4	1.9	1.9	4	-3	0	3.3	-3.2	4	-5	-6	2.4	2.6
3	-10	-5	1.1	1.1	3	6	2	1.3	1.3	4	-3	1	4.4	4.4	4	-5	-7	3.7	-3.6
3	1	0	5.0	5.2	3	6	6	3.1	-2.9	4	-3	2	5.4	-5.3	4	-5	-9	2.6	2.6
3	1	1	4.1	4.3	3	6	-2	2.7	2.7	4	-3	4	3.5	-3.4	4	-5	-10	1.8	2.2
3	1	2	1.6	-1.7	3	7	0	1.8	1.8	4	-3	5	2.3	2.0	4	-6	0	1.2	.8
3	1	3	3.5	-3.4	3	7	3	2.0	-2.2	4	-3	6	4.4	-4.0	4	-6	1	2.1	1.8
3	1	4	3.0	3.0	3	7	4	1.4	1.3	4	-3	7	1.8	1.6	4	-6	2	6.4	6.1
3	1	5	3.4	3.5	3	7	6	1.3	-1.5	4	-3	8	4.7	-4.6	4	-6	4	2.6	2.1
3	1	6	4.2	-4.3	4	0	0	3.9	-4.5	4	-3	10	1.7	-1.3	4	-6	6	4.7	4.6
3	1	7	3.5	-3.1	4	0	1	1.6	1.7	4	-3	-1	1.2	1.0	4	-6	7	2.5	2.2
3	1	10	1.7	-1.6	4	0	2	.8	-0.8	4	-3	-2	1.8	-1.5	4	-6	8	2.5	2.1
3	1	-1	1.3	1.4	4	0	3	2.3	-2.4	4	-3	-3	1.3	1.2	4	-6	-1	4.8	-4.6
3	1	-2	1.4	-1.1	4	0	10	2.6	2.4	4	-3	-4	4.0	-4.3	4	-6	-2	8.0	8.2
3	1	-3	1.3	1.3	4	0	11	2.0	-2.0	4	-3	-5	4.2	4.3	4	-6	-4	1.5	-1.3
3	1	-4	5.6	6.2	4	0	-1	2.5	-2.7	4	-3	-7	1.9	-1.7	4	-6	-5	4.7	-4.6
3	2	1	1.8	1.7	4	0	-2	2.0	1.9	4	-3	-8	2.1	-2.3	4	-6	-6	4.7	4.5
3	2	2	1.9	1.9	4	0	-3	2.7	2.8	4	-3	-9	3.6	3.3	4	-6	-7	1.6	-1.3
3	2	4	1.9	1.8	4	-1	0	4.5	5.5	4	-4	0	8.2	-8.6	4	-6	-8	1.8	1.8
3	2	5	1.7	-1.8	4	-1	1	1.7	1.8	4	-4	1	2.1	2.0	4	-6	-9	4.3	-4.1
3	2	6	2.8	-2.8	4	-1	2	4.8	5.3	4	-4	2	4.0	-3.7	4	-6	-10	1.7	1.4
3	2	8	2.3	2.3	4	-1	3	2.4	-2.4	4	-4	3	.8	-1.2	4	-6	-11	1.5	-1.2
3	2	9	2.9	-2.9	4	-1	4	5.3	5.2	4	-4	4	4.5	-4.3	4	-6	-12	2.1	2.0
3	2	10	2.6	-2.5	4	-1	6	6.7	7.4	4	-4	5	1.3	-0.9	4	-7	0	1.8	1.6
3	2	-1	2.1	1.9	4	-1	8	3.8	3.5	4	-4	6	2.6	-2.3	4	-7	1	4.1	3.9
3	2	-2	3.7	3.6	4	-1	9	1.5	-1.0	4	-4	8	3.7	-3.4	4	-7	2	1.8	-1.7
3	2	-3	.9	.9	4	-1	10	6.1	5.7	4	-4	9	1.8	-1.3	4	-7	3	2.2	2.0
3	2	-4	1.6	1.5	4	-1	-1	5.2	-5.6	4	-4	-1	3.7	-3.5	4	-7	4	2.1	2.2
3	3	0	1.8	1.5	4	-1	-2	6.1	7.8	4	-4	-2	1.2	.9	4	-7	5	3.7	3.7
3	3	1	1.5	.9	4	-1	-3	2.1	-2.2	4	-4	-3	5.1	5.1	4	-7	7	3.1	3.3
3	3	2	2.2	-2.1	4	-1	-4	1.7	1.7	4	-4	-4	7.0	-7.4	4	-7	-1	1.3	1.1
3	3	3	3.9	-3.8	4	-1	-6	3.7	3.9	4	-4	-5	2.3	1.9	4	-7	-2	1.3	1.0
3	3	4	3.6	3.4	4	-1	-7	4.4	-4.3	4	-4	-7	5.4	5.1	4	-7	-5	3.1	2.9
3	3	5	2.8	2.7	4	-2	0	1.0	1.0	4	-4	-8	3.3	-2.9	4	-7	-7	3.1	-2.8
3	3	6	1.9	-1.9	4	-2	1	3.6	4.0	4	-4	-9	1.8	1.5	4	-8	0	5.8	-5.5
3	3	8	2.6	2.2	4	-2	2	4.7	4.9	4	-4	-10	1.8	-1.8	4	-8	1	3.6	3.4
3	3	-1	1.4	-1.6	4	-2	4	2.6	2.6	4	-4	-11	5.6	5.2	4	-8	2	1.2	-1.0
3	3	-2	3.6	-3.5	4	-2	5	1.4	1.5	4	-5	0	2.4	2.1	4	-8	3	1.2	1.1
3	3	-3	3.0	-3.0	4	-2	6	2.8	2.8	4	-5	1	1.1	-1.2	4	-8	4	4.1	-4.1
3	4	0	3.1	-3.1	4	-2	7	2.6	2.0	4	-5	2	1.1	-1.0	4	-8	6	1.8	-1.6
3	4	6	1.9	-1.7	4	-2	8	.9	.8	4	-5	3	3.7	-3.6	4	-8	-2	1.6	1.6
3	4	7	2.3	2.2	4	-2	9	1.7	2.1	4	-5	4	3.9	3.4	4	-8	-3	4.8	4.6
3	4	8	3.3	3.4	4	-2	10	2.5	2.1	4	-5	5	2.6	-2.3	4	-8	-4	5.8	-5.5
3	4	-3	1.7	-1.6	4	-2	-1	3.8	-4.3	4	-5	6	1.6	1.3	4	-8	-5	2.1	1.4
3	4	-4	2.8	-2.8	4	-2	-2	8.9	9.7	4	-5	7	2.4	-2.2	4	-8	-7	3.4	3.1
3	5	1	2.3	2.3	4	-2	-3	2.3	2.3	4	-5	8	1.9	1.7	4	-8	-8	1.9	-1.9

TABLE II—*continued*

H	K	L	F(OBS)	F(CAL)	H	K	L	F(OBS)	F(CAL)	H	K	L	F(OBS)	F(CAL)	H	K	L	F(OBS)	F(CAL)
4	-8	-9	1.2	.9	4	3	-5	2.4	-2.4	5	-1	10	1.5	1.3	5	-5	7	1.5	1.3
4	-9	1	2.1	-1.2	4	4	0	1.7	1.6	5	-1	-1	1.3	-1.2	5	-5	8	2.4	-2.3
4	-9	2	3.4	-3.5	4	4	1	2.1	2.1	5	-1	-3	2.0	2.2	5	-5	-1	3.1	-3.0
4	-9	3	3.3	-3.4	4	4	2	2.2	2.3	5	-1	-7	1.8	-1.5	5	-5	-2	1.7	-1.5
4	-9	4	1.0	.9	4	4	4	4.4	4.4	5	-2	0	3.6	4.2	5	-5	-3	1.4	-1.2
4	-9	-1	2.0	-2.1	4	4	6	3.3	3.1	5	-2	3	4.5	4.8	5	-5	-4	2.3	-2.1
4	-9	-3	1.6	-1.4	4	4	8	3.6	3.5	5	-2	4	3.3	3.2	5	-5	-5	3.0	-3.0
4	-9	-4	1.6	-1.3	4	4	-1	2.1	-1.9	5	-2	5	2.6	-2.3	5	-5	-6	1.1	-1.0
4	-9	-5	2.8	2.5	4	4	-2	5.2	5.2	5	-2	7	1.2	1.1	5	-5	-7	3.1	-2.9
4	-10	1	1.2	-1.2	4	4	-3	.9	1.0	5	-2	9	1.4	-1.1	5	-5	-8	1.7	-1.6
4	-10	2	1.0	2.3	4	4	-4	1.8	-1.8	5	-2	-1	4.0	4.3	5	-5	-11	3.3	-2.9
4	-10	-1	4.6	-4.0	4	5	0	4.4	-4.0	5	-2	-2	1.8	1.9	5	-6	0	2.9	2.7
4	-10	-2	4.7	4.1	4	5	1	4.4	4.0	5	-2	-4	1.4	1.3	5	-6	1	2.0	-2.1
4	-10	-3	1.2	-0.8	4	5	2	2.9	-2.8	5	-2	-5	4.1	4.5	5	-6	2	2.1	-1.7
4	-10	-5	2.1	-1.7	4	5	4	3.0	-2.7	5	-2	-6	4.0	4.3	5	-6	3	3.6	3.4
4	-10	-6	3.4	3.5	4	5	5	1.7	1.4	5	-2	-9	4.6	4.7	5	-6	4	2.9	2.6
4	1	0	6.8	-7.3	4	5	7	1.4	1.4	5	-2	-10	2.1	1.6	5	-6	5	2.3	-1.9
4	1	1	3.6	3.7	4	5	8	2.4	-2.3	5	-3	0	1.6	1.6	5	-6	6	1.5	-1.4
4	1	2	6.3	-7.0	4	5	-1	1.8	-1.7	5	-3	3	.7	.7	5	-6	7	1.8	1.3
4	1	3	1.4	-1.4	4	5	-2	1.8	-1.5	5	-3	4	1.6	-1.4	5	-6	-1	2.3	2.0
4	1	4	5.1	-5.6	4	5	-3	3.7	3.3	5	-3	5	1.2	-0.9	5	-6	-5	2.0	1.8
4	1	6	4.3	-4.1	4	5	-4	3.9	-3.5	5	-3	6	2.4	2.1	5	-6	-6	1.9	1.7
4	1	8	6.4	-6.3	4	6	0	3.8	-4.0	5	-3	7	2.0	1.6	5	-6	-7	2.1	-2.1
4	1	10	1.8	-1.6	4	6	2	4.3	-4.3	5	-3	8	2.6	-2.3	5	-6	-9	1.6	1.6
4	1	-1	2.0	-2.0	4	6	3	1.1	-1.5	5	-3	9	1.3	-1.1	5	-7	0	2.1	1.8
4	1	-2	2.9	-3.0	4	6	4	2.4	-2.5	5	-3	-1	1.9	-1.5	5	-7	1	3.4	3.2
4	1	-3	3.5	3.4	4	6	6	4.4	-4.9	5	-3	-3	3.1	3.4	5	-7	2	1.7	1.6
4	1	-4	5.5	-5.5	4	6	-1	.9	-1.2	5	-3	-5	1.5	1.4	5	-7	3	2.0	1.7
4	2	0	1.9	-2.1	4	7	0	1.0	.8	5	-3	-6	1.4	1.4	5	-7	6	2.6	2.6
4	2	1	2.0	1.8	4	7	3	2.6	-2.5	5	-3	-8	1.6	1.5	5	-7	-1	.9	.7
4	2	2	2.8	-2.6	4	7	4	1.1	.8	5	-3	-9	3.6	3.3	5	-7	-2	1.7	1.3
4	2	6	2.6	-2.4	4	7	-1	4.0	-4.0	5	-4	1	4.1	-4.2	5	-7	-3	3.0	3.1
4	2	8	1.7	-1.7	5	0	1	4.0	-4.9	5	-4	2	4.3	-4.2	5	-7	-4	1.7	1.4
4	2	10	1.6	-1.5	5	0	2	3.1	-3.5	5	-4	3	2.7	2.5	5	-7	-5	2.4	2.0
4	2	-1	2.1	-2.1	5	0	3	2.3	2.3	5	-4	4	2.0	1.9	5	-7	-6	2.7	2.3
4	2	-2	4.0	3.9	5	0	5	2.3	-2.1	5	-4	5	3.7	-3.5	5	-7	-7	1.6	1.5
4	2	-3	1.0	.8	5	0	7	2.0	1.9	5	-4	6	1.7	-1.7	5	-7	-9	3.3	3.2
4	2	-4	3.1	-3.1	5	0	10	.9	.6	5	-4	7	2.3	1.8	5	-8	0	2.6	2.4
4	3	0	4.1	4.3	5	0	-2	1.9	-1.9	5	-4	-1	.9	.8	5	-8	1	1.1	-0.9
4	3	1	2.8	2.8	5	0	-3	3.3	-3.3	5	-4	-2	2.1	-1.9	5	-8	2	1.5	-1.5
4	3	2	5.9	5.4	5	0	-4	4.2	-4.5	5	-4	-3	3.9	-3.9	5	-8	3	2.0	2.0
4	3	3	3.4	-3.1	5	-1	0	1.2	1.2	5	-4	-4	3.0	-3.0	5	-8	4	2.0	1.9
4	3	4	3.9	3.9	5	-1	1	1.5	1.6	5	-4	-7	4.0	-3.8	5	-8	5	1.9	-1.5
4	3	6	4.5	4.5	5	-1	2	.9	.9	5	-4	-8	2.3	-2.1	5	-8	-1	1.2	1.1
4	3	8	2.0	2.1	5	-1	3	1.4	1.4	5	-4	-9	1.5	1.2	5	-8	-5	1.6	1.6
4	3	9	1.4	-0.8	5	-1	5	1.8	-1.6	5	-4	-10	1.2	-0.7	5	-8	-6	1.1	.9
4	3	10	4.1	4.5	5	-1	6	2.4	2.3	5	-4	-11	2.8	-2.2	5	-8	-9	3.1	3.0
4	3	-1	3.9	-3.6	5	-1	7	1.7	1.4	5	-5	4	2.1	-1.8	5	-9	1	1.9	-1.6
4	3	-2	6.0	5.9	5	-1	8	2.7	-2.4	5	-5	5	1.7	-1.2	5	-9	2	1.6	-1.2
4	3	-4	2.0	2.1	5	-1	9	1.5	-1.2	5	-5	6	1.8	1.8	5	-9	3	1.0	-0.9

TABLE II—*continued*

H	K	L	F(OBS)	F(CAL)	H	K	L	F(OBS)	F(CAL)	H	K	L	F(OBS)	F(CAL)	H	K	L	F(OBS)	F(CAL)
5	-9	-1	2.7	-2.5	5	1	5	2.4	-2.3	5	2	-1	1.8	1.8	5	4	3	2.4	2.3
5	-9	-2	1.4	-1.2	5	1	6	1.2	1.2	5	2	-2	1.4	1.1	5	4	4	.8	1.0
5	-9	-3	1.0	-0.6	5	1	7	2.3	1.9	5	2	-5	3.1	2.9	5	4	5	1.1	-1.0
5	-9	-4	1.7	-1.7	5	1	8	1.3	-1.2	5	3	0	2.6	2.2	5	4	7	.6	.6
5	-9	-5	1.8	-1.7	5	1	10	1.4	1.3	5	3	1	1.2	1.2	5	4	-1	1.2	1.2
5	-9	-7	2.6	-2.5	5	1	-1	2.4	-2.2	5	3	2	.8	1.2	5	4	-4	1.5	-1.2
5	-9	-8	1.4	-1.2	5	1	-2	1.7	-1.5	5	3	3	2.7	2.5	5	5	1	1.6	-1.5
5	-10	-1	1.2	-1.2	5	1	-3	1.3	-1.1	5	3	5	1.7	-1.3	5	5	2	.8	-1.0
5	-10	-2	1.4	-1.5	5	1	-4	1.6	-1.8	5	3	7	1.6	1.4	5	5	5	1.6	-1.4
5	-10	-3	1.9	-1.8	5	1	-5	2.3	-2.2	5	3	8	1.5	-1.3	5	5	6	.8	1.0
5	-10	-4	1.4	-1.3	5	2	0	2.1	2.2	5	3	9	1.7	-1.7	5	5	-1	2.3	-2.3
5	-10	-5	2.0	-2.0	5	2	1	1.8	-1.7	5	3	-2	1.4	1.5	5	5	-2	1.2	-1.2
5	1	0	.9	-1.0	5	2	2	1.1	-0.9	5	3	-3	3.5	3.4	5	5	-4	2.5	-2.4
5	1	1	.9	-1.0	5	2	3	2.8	2.6	5	3	-4	1.2	1.4	5	6	1	1.9	-2.1
5	1	2	1.4	-1.3	5	2	4	1.7	1.7	5	3	-5	2.1	2.1	5	6	2	1.6	-1.7
5	1	3	1.3	1.0	5	2	5	2.1	-2.1	5	4	0	1.5	1.5	5	6	-1	1.6	-1.3
5	1	4	.8	-1.1	5	2	7	1.6	1.4	5	4	2	.8	-0.8					

TABLE III

FINAL ATOMIC PARAMETERS WITH THEIR ESTIMATED STANDARD DEVIATIONS IN PARENTHESES

	x	*y*	*z*	*B*
Ag(I)	0.6519(5)	0.0065(4)	0.2515(3)	1.29(5) Å^{2a}
Ag(II)	0.8398(6)	0.4832(4)	0.2075(3)	1.30(5)
Ag(III)	0.4438(8)	0.5227(5)	0.2388(3)	1.96(5)
Mo(I)	0.5691(5)	0.2249(3)	0.4887(3)	0.17(6)
Mo(II)	0.0731(5)	0.2334(3)	0.4909(3)	0.16(6)
Mo(III)	0.1404(5)	0.8005(3)	0.0282(3)	0.18(6)
Mo(IV)	0.3011(5)	0.2048(3)	0.0144(3)	0.17(6)
Mo(V)	0.1823(5)	0.9993(3)	0.3179(3)	0.16(6)
O(I)	0	0	0	1.19(91)
O(II)	0.8794(40)	0.6701(28)	0.0215(21)	0.19(64)
O(III)	0.3466(41)	0.9952(29)	0.0790(22)	0.36(65)
O(IV)	0.4921(43)	0.3519(31)	0.0847(24)	0.57(64)
O(V)	0.2423(40)	0.6484(28)	0.1080(22)	0.26(65)
O(VI)	0.8310(47)	0.2029(35)	0.1161(26)	1.26(64)
O(VII)	0.6694(41)	0.7948(28)	0.1334(22)	0.21(64)
O(VIII)	0.2178(41)	0.1738(29)	0.2130(22)	0.30(64)
O(IX)	0.0940(41)	0.8312(28)	0.2262(22)	0.26(64)
O(X)	0.5999(44)	0.3322(31)	0.3516(23)	0.77(64)
O(XI)	0.0427(47)	0.3725(35)	0.3709(26)	1.30(64)
O(XII)	0.9688(41)	0.0067(29)	0.3762(22)	0.26(64)
O(XIII)	0.4436(41)	0.9884(29)	0.3820(22)	0.31(64)
O(XIV)	0.8143(41)	0.6537(28)	0.3888(22)	0.22(64)
O(XV)	0.3864(42)	0.6202(29)	0.4129(22)	0.42(65)
O(XVI)	0.2982(42)	0.1742(30)	0.4625(22)	0.44(64)
O(XVII)	0.1792(42)	0.8214(30)	0.4821(22)	0.37(64)

[a] The Debye-Waller temperature factors of the silver atoms before the anisotropic refinement.

particular form of linkage of octahedra gives rise to voids in the infinite sheets, which are then filled with silver ions located in five-coordinate sites. Adjacent sheets have no common oxygen atoms, being held together by the remaining silver ions which occupy irregular seven-coordinate interlayer positions (Fig. 2).

The distortions present in the MoO_6 octahedra are shown by the Mo–O distances listed in Table V. In each case the molybdenum atom is displaced

TABLE IV

FINAL ANISOTROPIC THERMAL PARAMETERS FOR THE SILVER ATOMS WITH THEIR ESTIMATED STANDARD DEVIATIONS IN PARENTHESES[a]

	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
Ag(I)	0.0035(9)	0.0116(5)	0.0010(2)	0.0028(5)	0.0008(3)	−0.0008(3)
Ag(II)	0.0121(11)	0.0064(5)	0.0018(2)	0.0038(5)	0.0008(4)	0.0022(3)
Ag(III)	0.0396(19)	0.0208(8)	0.0015(2)	0.0275(11)	−0.0008(5)	0.0004(3)

[a] The temperature factor is of the form: $\exp[-(\beta_{11}h^2 + \beta_{22}k^2 + \beta_{33}l^2 + 2\beta_{12}hk + 2\beta_{13}hl + 2\beta_{23}kl)]$.

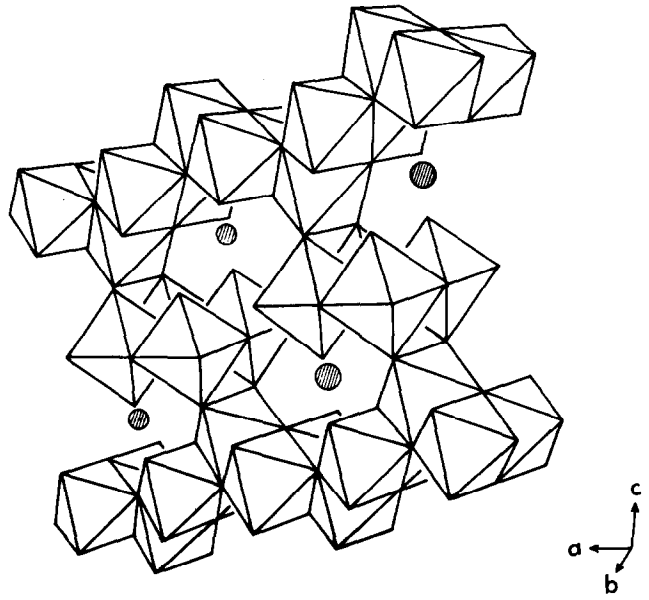

FIG. 1. A perspective view of part of an infinite sheet of idealized MoO_6 octahedra, showing how the infinite zig-zag chains are joined by the squares of four octahedra. The location of the intrasheet silver ions (hatched circles) in the voids is clearly seen.

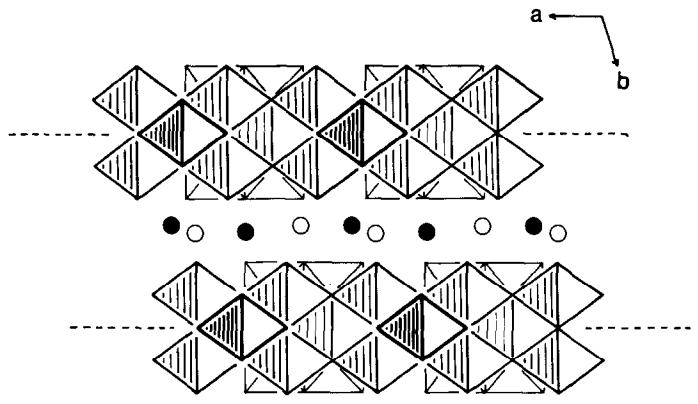

FIG. 2. Projection of the $Ag_6Mo_{10}O_{33}$ structure on (001) showing the idealized octahedral sheets linked by interlayer silver ions. The lower chains in each sheet have been omitted for clarity. The almost coplanar silver ions represented by solid circles lie ca c/2 above the almost coplanar silver ions represented by open circles. The pseudomirror plane within each sheet, parallel to (010) is indicated by a broken line.

TABLE V

SELECTED INTERATOMIC DISTANCES IN $Ag_6Mo_{10}O_{33}$

(MoO_6) Octahedra				Silver–Oxygen			
Mo(I)–O(X)	1.73(3)	Mo(IV)–O(IV)	1.72(3)	Ag(I)–O(XIII)	2.37(3)		
–O(XV)	1.73(3)	–O(VII)	1.75(3)	–O(VII)	2.40(3)		
–O(XVI)	1.94(3)	–O(II)	1.89(3)	–O(VI)	2.44(3)		
–O(XVII)	2.00(3)	–O(III)	1.90(3)	–O(XII)	2.51(3)		
–O(XIII)	2.15(3)	–O(I)	2.420(4)	–O(III)	2.67(3)		
–O(XIII)	2.30(3)	–O(VIII)	2.45(3)				
				Ag(II)–O(XI)	2.40(3)	Ag(II)–O(V)	2.27(3)
Mo(II)–O(XI)	1.69(3)	Mo(V)–O(IX)	1.74(3)	–O(II)	2.49(3)	–O(X)	2.33(3)
–O(XIV)	1.69(3)	–O(VIII)	1.76(3)	–O(VI)	2.65(3)	–O(XV)	2.41(3)
–O(XVII)	1.92(3)	–O(XII)	1.90(3)	–O(X)	2.67(3)	–O(IV)	2.54(3)
–O(XVI)	2.01(3)	–O(XIII)	1.98(3)	–O(IV)	2.69(3)	–O(VII)	2.74(3)
–O(XII)	2.29(3)	–O(XVI)	2.21(3)	–O(XIV)	2.73(3)	–O(VIII)	2.94(3)
–O(XII)	2.34(3)	–O(XVII)	2.23(3)	–O(IX)	2.99(3)	–O(XIV)	2.94(3)
Mo(III)–O(VI)	1.71(3)						
–O(V)	1.72(3)						
–O(III)	1.95(3)						
–O(II)	1.96(3)						
–O(I)	2.167(4)						
–O(IX)	2.42(3)						

Shared-Edge Oxygen–Oxygen			
(a) In the infinite chains		(b) In the units of four octahedra	
O(XII)–O(XVI)	2.54(4)	O(I)–O(II)	2.63(3)
O(XII)–O(XVII)	2.54(4)	O(I)–O(III)	2.59(3)
O(XIII)–O(XVI)	2.50(4)		
O(XIII)–O(XVII)	2.51(4)		
O(XII)–O(XII)	2.65(4)		
O(XIII)–O(XIII)	2.75(4)		

from its ideal position in the centre of the octahedron, resulting in two short, two intermediate and two long Mo–O distances; this is also found in MoO_3 (*7*), $Na_2Mo_2O_7$ (*8*), $K_2Mo_3O_{10}$ (*2*), $K_6Mo_7O_{24}\cdot 4H_2O$ (*9*) and $CoMoO_4$ (*10*).

The molybdenum–molybdenum distances also show wide variation (Fig. 3) lying in the range 3.230(5)–3.714(5) Å for edge-shared octahedra and in the fairly close range 3.791(5)–3.840(5) Å for corner-shared octahedra. Intersheet distances are much greater and the closest approach is 4.901(5) Å. It is interesting to note that in the infinite chains one molybdenum atom, Mo(V), is almost equidistant from four others, Mo(I), Mo(I′), Mo(II), Mo(II′), while in the units of four octahedra the molybdenum atoms lie on the corners of a rather regular square. These two arrangements are exactly the same as found in the heptamolybdate (*10*) and octamolybdate (*11*) anions respectively, where even the corresponding metal–metal distances are almost the same.

A careful examination of the structure reveals a number of vacant octahedral sites within the infinite sheets (Fig. 1). Two of these unfilled octahedra are centrally located one above and one below the squares of filled octahedra; and two more, occurring as an edge-shared pair, lie between and share faces with adjacent squares of four filled octahedra in the *a* axis direction, where the Mo–Mo distance across these unfilled octahedra is 4.386(5) Å (see Fig. 3).

The oxygen–oxygen distances in the MoO_6 octahedra range 2.50(4)–3.19(4) Å and the shared-edge distances (Table V) are generally shorter than the unshared-edge distances. Thus the oxygen atoms which form the octahedral framework are more or less close packed although the presence of larger silver ions within the sheets disrupts the packing somewhat. The temperature factors for the molybdenum atoms, which are among the lowest reported for molybdenum oxides, are consistent with this interpretation. The interlayer silver ions control the closeness of approach of the infinite sheets of octahedra, with the shortest intersheet oxygen–oxygen distances being 2.74(4) and 2.82(4) Å.

Although the over-all crystal symmetry is low ($P\bar{1}$), the arrangement of atoms in the structure is almost consistent with the presence of a mirror plane parallel to (010), where the atoms Mo(V), Ag(I), O(I), O(III), O(XII), and O(XIII) actually lie in the plane. This pseudomirror plane, the position of which is shown in Fig. 2, results in a marked

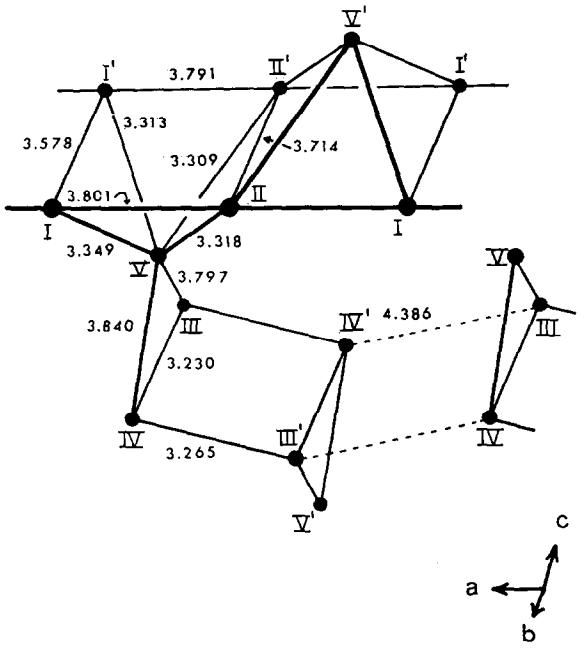

FIG. 3. Diagrammatic representation of the arrangement of molybdenum atoms within the octahedral sheets. The atoms are labelled corresponding to the assignment in Table III, and the primes denote the symmetry related atoms. The estimated standard deviation for the metal–metal distances is ±0.005 Å.

pseudosymmetry on the (O*kl*) Weissenberg photograph.

The Ag–O distances lie in the range 2.37(3)–2.67(3) Å for the five-coordinate silver ions (mean value 2.48 Å), and in the range 2.26(3)–2.99(3) Å for the seven-coordinate silver ions (mean value 2.63 Å). These may be compared with the Ag–O distances for the five-coordinate silver ions in the silver vanadium bronze, $Ag_{0.68}V_2O_5$ (*12*), which range 2.48–2.68 Å and with the mean distance of 2.53 Å for the seven-coordinate silver ions in silver dichromate, $Ag_2Cr_2O_7$ (*13*). The oxygen coordination around the intrasheet silver ion is distorted trigonal bipyramidal, while that around the intersheet silver ions can only be described as irregular sevenfold. The closest approach of any two silver ions is 3.215(5) Å† which may be compared with a distance of 2.889 Å in the metal (*5*).

The only other silver molybdate which has been investigated so far is the simple molybdate, Ag_2MoO_4, which was shown to adopt the spinel structure (*14*). Donohue and Shand (*14*) reported a Ag–O distance of 2.42(2) Å for the octahedrally coordinated silver ions in this compound, but in a recent reinvestigation of the structure we have found a value of 2.48(2) Å (*15*). Although no definite rule may be applied, it has been found that in a number of silver salts the Ag–O distance is related to colour. In the colourless salts $AgClO_3$, Ag_2SO_4 and $KAgCO_3$, Ag–O distances of 2.51, 2.50, and 2.42 Å respectively have been found (*16*), while in the yellow salts Ag_3PO_4 and Ag_2CO_3 the distances are 2.34 and 2.3 Å, respectively (*16*) and in Ag_3AsO_4 which is red, the Ag–O distance is 2.34 Å (*16*). The Ag–O distance of 2.48 Å in the colourless Ag_2MoO_4 fits nicely into this pattern, as does the compound described in the present work, $Ag_6Mo_{10}O_{13}$, which is yellow and contains a number of short Ag–O distances of the order of 2.3 Å. The Ag–O distances found in this compound are shown in Table V, where it can be seen that many are somewhat shorter than the ionic radius sum of 2.66 Å (*17*), and these bonds must have an appreciable covalent character.

Perhaps the most significant feature of the structure, is that the repeating unit of six octahedra is extremely similar to the basic structural units found in the red and blue potassium molybdenum bronzes, $K_{0.26}MoO_3$ and $K_{0.28}MoO_3$, respectively (*18, 19*), and $CoMoO_4$ (*10*), and also to the subunit of six octahedra common to both the octa- and heptamolybdates which contain discrete anions $Mo_8O_{26}^{4-}$ and $Mo_7O_{24}^{6-}$ (*20*). Further, the square units of four octahedra which are seen to link the infinite chains in this structure are also found in the octamolybdate anion where two such units are joined by common edges (*11*).

Wadsley (*21*) described how simplifications could be introduced into the crystal chemistry of lattice compounds by replacing the single polyhedron with the group as the basic unit of structure, where these groups could join by peripheral corner- or edge-sharing with the same or with different groups to form two- and three-dimensional networks which might include interstitial metal ions of another kind. Silver decamolybdate provides an excellent example of the above, where the structure is seen to be built up from two basic groups, which are very similar to, or the same as those found in other complex molybdates prepared from a melt or an aqueous solution. A mechanism for crystallization of this compound immediately suggests itself, where just prior to crystallization the two basic groups of octahedra found in the structure, corresponding to the hypothetical anions $Mo_4O_{17}^{10-}$ and $Mo_6O_{23}^{10-}$,

† These are the intersheet ions Ag(II) and Ag(III) which effectively occur in "pairs", with no oxygen atoms between them, and occupy large intersheet coordination sites or cavities. A similar situation has been described in silver nitrate (*22*).

exist in the melt. Initially the subunits of six octahedra begin to link up, forming zig-zag chains in the prism (a)-axis direction. The subunits of four octahedra then link these chains into sheets in a manner which may well be controlled by the presence of the silver ions, some of which are actually coordinated within the sheets. The remaining silver ions then find energetically favourable interlayer positions and effectively hold the infinite sheets together. It is quite conceivable that if the conditions under which the fused salt system Ag_2MoO_4–MoO_3 is allowed to crystallize, are varied, the groups of octahedra mentioned above might condense in a number of ways to give stable structures. These structures, if a direct comparison to the phase system K_2MoO_4–MoO_3 may be made, would be expected to be nonstoicheiometric with a bronze-like nature.

A single-crystal structure investigation of the second compound found to exist in the Ag_2MoO_4–MoO_3 phase system, apparently of formula $Ag_2Mo_2O_7$, is now in progress, and it will be interesting to compare its structure with the chain structure found for the analogous sodium compound, $Na_2Mo_2O_7$ (*8*).

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