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Studies on Peroxomolybdates

IX. The Crystal Structure of Potassium Diperoxoheptamolybdate(VI) Octahydrate, $K_6Mo_7O_{22}(O_2)_2.8H_2O$

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The crystal structure of $K_6Mo_7O_{22}(O_2)_2.8H_2O$ has been determined from visually estimated X-ray reflexion intensities, using Patterson and Fourier methods, and refined by the least-squares technique to a final R-value of 0.097, based on 1593 observed reflexions.

The yellow crystals are orthorhombic, belonging to space group $C2cm$, with $a = 8.487$ (2) Å, $b = 19.047$ (5) Å, $c = 19.974$ (5) Å, $V = 3228.8$ Å^3 and $Z = 4$.

The diperoxoheptamolybdate ion has the same general appearance as the normal heptamolybdate ion,[10] the difference being that two peroxo groups replace two oxygen atoms, one at either end of the heptamolybdate ion. The molybdenum atoms which are coordinated to a peroxo group are thus surrounded by seven oxygen atoms. The geometry is pentagonal bipyramidal, the equatorial plane of the bipyramid containing five oxygen atoms, two of which belong to a peroxo group. The apical positions are occupied by one double-bonded and one weakly bonded oxygen atom. Molybdenum is displaced 0.31 Å out of the equatorial plane towards the double-bonded oxygen atom. Bond distances: $Mo-O_{bridging}$ 1.76–2.49 Å, $Mo-O_{peroxo}$ 1.86–1.96 Å, Mo=O 1.64–1.75 Å and $(O-O)_{peroxo}$ 1.38 Å.

Baerwald [1] was, unwittingly, in 1885, probably the first to prepare crystals of peroxoheptamolybdates. He dissolved ammonium paramolybdate in an excess of hydrogen peroxide and evaporated the solution isothermally. He formulated the compound he obtained, however, $14NH_3.18MoO_3.3H_2O_2.18H_2O$.

From the short historical review of ammonium peroxoheptamolybdates given by Hansson and Lindqvist,[2] it is obvious that the earlier contributions are rather confusing, since the conditions of formation were not studied and the formula of the paramolybdate was unknown until 1937 when Sturdivant [3] showed the paramolybdate to be a heptamolybdate.

By varying the $MoO_3 : H_2O_2$ ratio or the temperature in solutions of paramolybdates, Gagliotti [4] obtained crystals of varying peroxide content. It is,

however, possible to obtain peroxoheptamolybdates with stoichiometric compositions by choosing the conditions carefully. Stomberg and Trysberg [5] have shown that the nature of the crystals formed by the isothermal evaporation of solutions, obtained by adding hydrogen peroxide and a strong acid to solutions of normal potassium molybdate, depends on the H_2O_2 : Mo ratio, the acidity and the temperature of the solution. When the H_2O_2 : Mo ratio is 0.1 – 0.8 and the pH 5.8 – 6.8, two peroxoheptamolybdates are obtained, one with orthorhombic and one with monoclinic symmetry. The structure of the orthorhombic peroxoheptamolybdate is described in this paper, while an investigation of the structure of the monoclinic compound is soon to be commenced. Some of the results of the present investigation have been published previously in a preliminary report.[6]

EXPERIMENTAL

Preparation of crystals. Crystals of $K_6Mo_7O_{22}(O_2)_2.8H_2O$ can be prepared by the method proposed by Trysberg [7] in which 1.0 ml 8 M hydrogen chloride and 1.75 ml 6 M hydrogen peroxide are added to 20.0 ml 1.3 M potassium molybdate. The acidity of the solution is adjusted to pH = 6.2. The yellow solution is set to evaporate at room temperature (below 25°C), yellow, rod-shaped crystals being eventually deposited.

Analysis. Crystals of the potassium diperoxoheptamolybdate used in this investigation were analyzed by Trysberg.[7] (Found: K 15.8; Mo 45.5; $O_{peroxide}$ 3.65; H_2O 10.8. Calc. for $K_6Mo_7O_{24-x}(O_2)_x.9H_2O$ with $x = 1.67$; K 15.9; Mo 45.4; $O_{peroxide}$ 3.65; H_2O 11.0.) The analysis indicates nine water molecules in the formula, while the X-ray analysis is more consistent with eight water molecules. It is possible to obtain crystals of the compound with $x = 2$. The X-ray data were, however, collected from a crystal of the compound with $x = 1.67$.[6] For simplicity we formulate the compound $K_6Mo_7O_{22}(O_2)_2.8H_2O$.

X-Ray methods. For the single crystal work multiple-film (5 films) equi-inclination Weissenberg photographs were taken of a crystal of approximate dimensions $0.5 \times 0.12 \times 0.12$ mm^3 with rotation about [100] (layer lines 0 – 7) and [010] (layer line 0), using Cu$K\alpha$ radiation.

The relative intensities of the reflexions were estimated visually by comparison with a standard scale (obtained by the rotating sector method) and were corrected for absorption, extinction, Lorentz and polarization effects. Errors due to anomalous dispersion were taken into consideration but the correction did not affect the result. The $|F_o|$-values were brought on to an absolute scale by comparison with the finally calculated structure factors. A total of 1593 independent reflexions was obtained.

Computing methods. Absorption, extinction, Lorentz and polarization corrections, Fourier summations, structure factor least-squares refinements, calculations of interatomic distances and angles, calculations of planes to fit sets of atoms and plotting of the crystal structure were performed on the electronic computer IBM 360/65, using the set of crystallographic programmes, *viz.* DATAP2, DRF, LALS, LINUS, DISTAN, PLANEFIT, and ORTEP, in use at this department.[8]

The atomic scattering factors for potassium, molybdenum, and oxygen used in the calculations of the structure factors were taken from Vol. III of the *International Tables for X-Ray Crystallography*, 1962.

CRYSTAL DATA

Crystal data have been reported previously by Stomberg.[9] $K_6Mo_7O_{22}(O_2)_2.8H_2O$ is orthorhombic with

$a = 8.487(2)$ Å
$b = 19.047(5)$ Å
$c = 19.974(5)$ Å
$V = 3228.8$ Å^3

$Z = 4$
$D_{obs} = 3.08$ g/cm^3
$D_{calc} = 3.01$ g/cm^3
Space group = $C2cm$

STRUCTURE DETERMINATION

Atomic positions. Owing to the relatively large number of interatomic vectors, a comparison of the vector set with that obtained for the normal heptamolybdate ion, the structure of which has been determined by Lindqvist [10] and later confirmed by Shimao [11] (Fig. 1), was thought worth-while.

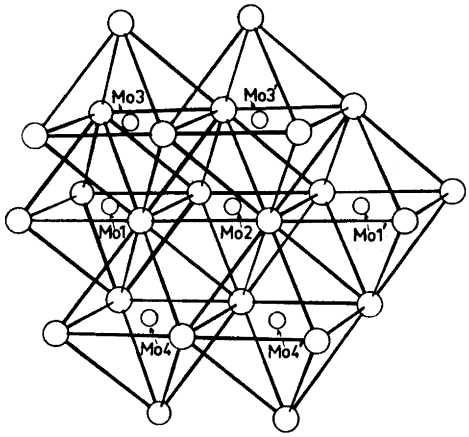

Fig. 1. Idealized structure of the heptamolybdate ion, showing the octahedral arrangement of ligands about molybdenum.

The coordinates of some dominant peaks in the Patterson synthesis are listed in Table 1a together with the observed peak heights and lengths of interatomic vectors. In Table 1b some interatomic distances within the heptamolybdate ion are given.[10] As is apparent from the two tables, there are obvious similarities in vector lengths. The relative orientation of the vectors was also found to be comparable. It therefore seemed likely that the molybdenum atoms were arranged in the same way in the peroxoheptamolybdate as in the normal heptamolybdate. Comparison between expected and observed lengths of vectors indicates that the three molybdenum atoms Mo(1) – Mo(2) – Mo(1′) are situated in a straight line parallel to the c-axis. Judging from the appearance of the heptamolybdate ion, it is obvious that the correct space group is $C2cm$, with Mo(2) occupying the four-fold position 4*b* and the remaining molybdenum atoms occupying three eight-fold positions 8*c*. At this stage the z-coordinates of all molybdenum atoms were known. The x-coordinates were obtained by identifying interatomic vectors Nos. III and V (Table 1b) with Patterson peaks Nos. *4* and *5* (Table 1a), respectively. (Owing to the symmetry of space group $C2cm$, the x-coordinate of Mo(2) is fixed at zero.) By identifying Patterson vectors with corresponding expected vectors between molybdenum atoms

Table 1a. Coordinates of some dominant peaks in the Patterson synthesis, together with observed peak heights and lengths of interatomic vectors.

Peak No.	*u*	*v*	*w*	Peak height (arbitrary units)	Length of interatomic vector (Å)
1	0	0	0	999	0
2	0	0	0.17	347	3.34
3	0.14	0.22	0	115	4.28
4	0.15	0.14	0.09	95	3.40
5	0	0	0.34	94	6.81
6	0.29	0.08	0.09	82	3.35

Table 1b. Some interatomic distances within the heptamolybdate ion. In the last column the corresponding Patterson vectors for the peroxoheptamolybdate ion are given.

Vector No.	Defining atoms	Length of vector (mean value) (Å)	Relative weight	Patterson vector No.
I	Mo(1)–Mo(2)	3.36	4	2
II	Mo(1)–Mo(3)	3.32	2	9
III	Mo(1)–Mo(4)	3.32	2	6
IV	Mo(3)–Mo(4)	4.28	2	4
V	Mo(1)–Mo(1′)	6.83	1	7

occupying positions 8*c* of space group *C*2*cm* the *y*-coordinates were obtained. The molybdenum parameters thus are:

	Position	*x*	*y*	*z*
Mo(1)	8*c*	0.0	0.135	0.08
Mo(2)	4*b*	0	0.135	1/4
Mo(3)	8*c*	0.29	0.06	0.165
Mo(4)	8*c*	0.14	0.275	0.165

A Fourier summation of the observed structure factors in which the phases of the Fourier coefficients were determined by the contributions from the molybdenum atoms alone was undertaken. This showed large peaks at the above positions and minor peaks, which were taken to be potassium and water sites. Probable coordinates for the oxygen atoms surrounding the molybdenum atoms according to the heptamolybdate model were also found. On introducing these atoms into a structure factor calculation an *R*-value of 0.25 was obtained. Since the crystal under investigation contained 1.67 peroxo groups per heptamolybdate ion instead of the stoichiometric amount 2, there were assumed to be, on average, 0.165 oxygen atoms and 0.835 peroxo groups at each peroxo group site.

Structure refinement. The structure was refined by the structure factor least squares method using 1593 observed reflexions. For each reflexion the weight w was computed according to $w=(a+|F_o|+c|F_o|^2+a|F_o|^3)^{-1}$ with $a=10.0$, $c=0.005$, and $d=0$. Anisotropic temperature parameters were included

Table 2a. Atomic coordinates (expressed as fractions of the cell edges) and isotropic thermal parameters for $K_6Mo_7O_{22}(O_2)_2.8H_2O$, with the standard deviations of the last significant figures in parentheses. Space group $C2cm$, $Z=4$.

Atom	x	y	z	B (Å^2)
Mo(1)	0.0547(6)	0.1404(1)	0.0818(1)	
Mo(2)	0[a]	0.1338(1)	1/4	
Mo(3)	0.3167(6)	0.0584(1)	0.1674(1)	
Mo(4)	0.1828(6)	0.2716(1)	0.1672(1)	
K(1)	0.6657(22)	0	0	4.7(2)
K(2)	0.8253(17)	0.3534(5)	1/4	2.9(2)
K(3)	0.5366(12)	0.4046(3)	0.1041(3)	3.4(1)
K(4)	0.5504(14)	0.2056(4)	0.0329(3)	3.8(1)
O(1)	−0.091(5)	0.084(2)	0.029(2)	4.4(6)
O(2)	−0.130(6)	0.154(2)	0.033(2)	5.4(8)
O(1,2)	−0.143(12)	0.114(5)	0.043(4)	0.7.(12)
O(3)	−0.120(4)	0.119(1)	0.180(1)	3.1(4)
O(4)	0.172(4)	0.159(1)	0.019(1)	3.6(4)
O(5)	0.187(3)	0.159(9)	0.168(1)	1.9(3)
O(6)	0.131(3)	0.047(1)	0.108(1)	2.4(3)
O(7)	0.009(4)	0.239(1)	0.108(1)	3.4(4)
O(8)	0.121(5)	0.053(2)	1/4	3.0(5)
O(9)	0.018(4)	0.235(1)	1/4	1.6(4)
O(10)	0.447(4)	0.088(2)	0.111(1)	4.1(5)
O(11)	0.409(4)	0.088(1)	1/4	2.3(4)
O(12)	0.340(4)	−0.032(1)	0.171(1)	3.7(5)
O(13)	0.328(4)	0.280(1)	0.112(1)	3.3(4)
O(14)	0.303(4)	0.273(1)	1/4	2.4(5)
O(15)	0.091(3)	0.354(1)	0.174(1)	2.5(3)
O(16)	0.508(6)	0.412(2)	1/4	4.4(7)
O(17)	0.602(7)	0.213(2)	1/4	4.7(8)
O(18)	0.266(4)	0.480(2)	0.138(1)	4.8(6)
O(19)	0.953(4)	0.405(1)	0.050(1)	3.9(5)
O(20)	0.711(4)	0.280(1)	0.133(1)	3.9(5)

[a] Arbitrarily fixed.

Table 2b. Anisotropic temperature parameters β_{ij} (with the standard deviations of the last significant figures in parentheses). The expression used is $\exp -(\beta_{11}h^2+\beta_{22}k^2+\beta_{33}l^2+\beta_{12}hk+\beta_{13}hl+\beta_{23}kl)$.

Atom	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
Mo(1)	0.0100(5)	0.00178(5)	0.00179(5)	0.0001(1)	0.0002(1)	0.00029(4)
Mo(2)	0.0057(6)	0.00128(6)	0.00174(6)	−0.0002(1)	0	0
Mo(3)	0.0104(5)	0.00143(5)	0.00181(5)	0.0007(1)	0.0003(1)	0.00009(4)
Mo(4)	0.0077(5)	0.00149(5)	0.00208(5)	0.0004(1)	0.0003(1)	0.00024(4)

Table 3. Observed and calculated structure factors for $K_6Mo_7O_{23}(O_2)_2.8H_2O$. The columns are l, $|F_o|$, $|F_c|$ and phase angle α (expressed in radians), respectively.

0 0 L			
2	262	242	3.14
4	250	267	3.14
6	119	109	0.00
8	58	61	0.00
10	251	255	3.14
12	831	869	-0.00
14	534	449	3.14
16	85	64	3.14
18	64	49	0.00
20	95	67	3.14
22	120	110	3.14
24	207	196	-0.00

0 2 L			
0	205	216	3.14
1	406	388	3.14
2	45	30	-0.00
3	171	175	3.14
4	26	33	3.14
5	251	267	3.14
6	72	67	-0.00
7	63	76	3.14
8	19	16	0.00
9	22	23	0.00
10	117	102	0.00
11	168	186	-0.00
12	24	21	-3.14
13	213	232	3.14
14	75	73	-0.00
15	118	101	3.14
16	35	35	-0.00
17	100	98	3.14
18	72	64	-0.00
19	31	29	0.00
20	42	35	-0.00
21	87	89	-0.00
22	25	26	0.00
23	119	114	-0.00
24	35	31	3.14
25	40	43	3.14

0 4 L			
0	181	200	3.14
1	300	302	3.14
2	73	97	-3.14
3	186	197	-0.00
4	170	192	3.14
5	611	502	-0.00
6	740	607	-0.00
7	157	179	3.14
8	49	41	-0.00
9	0	5	-3.14
10	59	51	3.14
11	200	201	-0.00
12	96	122	3.14
13	123	133	3.14
14	0	8	-0.00
15	85	88	-0.00
16	58	54	3.14
17	125	119	0.00
18	223	215	-0.00
19	169	151	3.14
20	55	26	-3.14
21	24	26	-3.14
23	59	50	-0.00
24	41	23	3.14
25	46	56	3.14

0 6 L			
0	269	266	3.14
1	213	198	-0.00
2	223	221	-0.00
3	79	78	0.00
4	324	283	-0.00
5	23	16	-0.00
6	126	155	3.14
7	120	127	-3.14
8	24	24	-0.00
9	118	124	3.14
10	72	76	-0.00
11	179	178	3.14
12	26	11	-3.14
13	35	30	0.00
14	194	200	-0.00
15	80	68	-0.00
16	71	58	0.00
17	152	147	-0.00
18	199	194	3.14
19	32	31	-3.14
21	56	47	3.14
22	62	55	-0.00
23	46	34	3.14
24	0	3	0.00

0 8 L			
0	196	222	-0.00
1	327	340	3.14
2	22	17	-3.14
3	263	257	3.14
4	87	83	-0.00
5	115	118	-0.00
6	277	307	3.14
7	23	15	-0.00
8	85	85	-0.00
9	141	147	-0.00
10	40	30	0.00
11	157	166	-0.00
12	150	151	-0.00
13	191	177	3.14
14	69	60	-0.00
15	65	57	3.14
16	98	98	-0.00
17	67	66	-0.00
18	130	132	3.14
19	58	58	-3.14
20	56	41	-0.00
21	0	3	0.00
22	0	5	0.00
23	58	48	-0.00
24	20	35	-0.00

0 10 L			
0	364	376	3.14
1	202	181	-0.00
2	256	245	-0.00
3	0	12	3.14
4	112	127	-0.00
5	487	453	3.14
6	36	20	-3.14
7	312	338	-0.00
8	118	141	0.00
9	52	59	0.00
10	97	112	-0.00
11	79	67	-3.14
12	349	362	3.14
13	82	82	-0.00
14	57	43	0.00
15	0	13	3.14
16	28	31	-0.00
17	163	157	3.14
18	0	5	0.00
19	145	149	-0.00
20	61	65	-0.00
21	0	6	3.14
22	51	48	-0.00
23	25	18	-3.14

0 12 L			
0	144	167	3.14
1	155	142	-0.00
2	119	104	-0.00
3	168	180	-0.00
4	0	7	0.03
5	118	127	-0.00
6	0	48	-0.00
7	139	126	3.14
8	68	60	-0.00
9	97	97	3.14
10	103	98	-0.00
11	0	11	0.65
12	93	92	3.14
13	110	87	-0.00
14	61	49	-0.00
15	62	64	-0.00
16	0	12	3.14
17	42	32	-0.00
18	48	56	-0.00
19	50	34	3.14
20	20	37	-0.00
21	25	17	3.14
22	13	18	-0.00

0 14 L			
0	391	386	-0.00
1	335	385	-0.00
2	27	29	3.14
3	33	14	-0.00
4	35	38	-0.57
5	158	148	3.14
6	36	51	3.14
7	131	134	-0.00
8	0	30	3.14
9	0	24	-0.00
10	74	70	3.14
11	204	219	3.14
12	216	220	-0.00
13	195	197	-0.00
14	58	36	3.14
15	75	54	3.14
16	0	1	-0.00
17	92	79	3.14
18	76	68	3.14
19	56	56	-0.00
20	39	48	3.14

0 16 L			
0	87	57	3.14
1	114	113	3.14
2	54	57	3.14
3	98	99	3.14
4	70	56	-0.00
5	117	102	3.14
6	44	32	-0.00
7	0	9	-0.00
8	0	24	3.14
9	31	38	-0.00
10	36	43	3.14
11	89	88	-0.80
12	53	62	-0.00
13	80	75	3.14
14	37	33	-0.00
15	66	63	3.14
16	36	26	-0.00
17	35	33	3.14
18	0	1	3.14
19	27	29	-0.00

0 18 L			
0	57	41	0.00
1	32	22	3.14
2	81	89	3.14
3	59	56	-0.00
4	86	95	3.14
5	0	42	3.14
6	312	324	-0.00
7	75	89	-0.00
8	79	70	3.14
9	0	0	-0.00
10	45	40	3.14
11	0	12	0.00
12	29	29	-0.00
13	0	7	-3.14
14	55	57	3.14
15	63	66	-0.00
16	43	40	3.14

0 20 L			
0	55	33	-0.00
1	46	40	3.14
2	108	82	-0.00
3	44	32	0.00
4	0	5	-3.14
5	154	166	-0.00
6	84	51	3.14
7	214	201	3.14
8	0	6	-0.00
9	37	31	3.14
10	57	52	-0.00
11	0	16	-0.00
12	0	8	3.14
13	44	43	3.14

0 22 L			
0	153	171	-0.00
1	65	48	3.14
2	63	51	3.14
3	54	50	3.14
4	21	28	3.14
5	64	60	-0.00
6	72	79	-3.14
7	35	45	-3.14
8	17	16	0.00
9	50	50	-0.00

0 24 L			
0	88	136	3.14
1	34	59	3.14
2	46	59	-0.00

1 1 L			
2	229	212	-1.29
3	73	79	-1.89
4	165	178	-1.68
5	399	348	1.86
6	551	436	3.13
7	220	206	-1.42
8	138	149	-0.76
9	188	171	0.79
10	108	117	-0.63
11	359	345	0.68
12	234	216	1.21
13	258	262	-2.18
14	95	107	-1.60
15	47	46	-2.32
16	56	53	-1.44
17	133	141	2.28
18	149	149	2.70
19	138	125	-1.44
20	52	44	-0.90
21	33	21	0.41
22	45	49	-1.80
23	89	86	0.85
24	38	51	1.54
25	51	78	-2.42

1 3 L			
0	437	396	2.92
1	64	63	-0.10
2	182	192	-1.72
3	59	63	-1.96
4	104	122	-1.12
5	496	409	3.10
6	336	327	0.74
7	74	68	2.33
8	176	176	-1.95
9	67	64	-3.07
10	93	99	-2.22
11	0	22	0.49
12	276	245	2.99
13	67	63	0.38
14	151	160	-1.86
15	58	54	-2.27
16	54	60	-1.97
17	148	151	-2.99
18	141	144	0.92
19	122	96	0.00
20	32	38	-1.38
21	54	60	0.38
22	25	25	-1.39
23	30	21	0.49
24	61	62	2.96

1 5 L			
0	692	511	-2.33
1	616	506	-0.88
2	83	68	2.54
3	147	152	0.96
4	114	110	2.58
5	336	346	0.89
6	77	92	-1.21
7	168	184	-2.08
8	0	10	-2.24
9	108	116	-2.12
10	53	44	1.62
11	276	277	2.34
12	165	180	-2.21
13	268	247	-0.86
14	102	100	0.67
15	123	110	1.14
16	51	47	0.57
17	163	175	0.88
18	80	83	-0.91
19	106	101	-2.13
20	0	12	1.06
21	62	65	-2.40
22	33	28	1.13
23	87	95	2.38
24	43	47	-2.02

1 7 L			
0	554	485	0.14
1	77	87	-0.49
2	30	24	0.08
3	93	76	-2.26
4	77	60	1.40
5	58	53	0.95
6	163	162	-2.16
7	159	171	-0.06
8	153	167	-2.94
9	44	44	-0.16
10	133	131	-1.90
11	119	130	-2.88
12	255	245	0.27
13	30	25	-0.09
14	43	40	-2.47
15	0	5	2.29
16	54	60	-2.70
17	0	7	0.65
18	112	110	-2.59
19	0	11	-0.96
20	72	68	-2.92
21	27	22	3.14
22	30	30	-2.28
23	41	44	-2.83
24	65	101	0.18

1 9 L			
0	648	562	-1.55
1	491	449	-2.98
2	257	243	2.19
3	229	211	-2.34
4	155	139	2.16
5	188	179	3.04
6	400	364	-1.73
7	195	204	-0.16
8	119	117	1.29
9	46	44	0.78
10	110	113	1.52
11	136	142	0.09
12	316	298	-1.46
13	221	243	-3.06
14	152	159	1.83
15	57	65	-1.42
16	79	72	0.99
17	60	62	2.94
18	132	148	-1.49
19	49	56	-0.08
20	61	60	1.29
21	29	24	1.24
22	53	52	1.89
23	58	82	0.15

1 11 L			
0	183	166	-2.98
1	191	185	1.44
2	108	102	-2.31
3	136	127	0.21
4	106	95	-2.83
5	194	175	-0.87
6	366	364	0.21
7	192	186	1.82
8	80	89	-2.68
9	38	38	-0.72
10	32	31	-1.80
11	117	119	-0.92
12	82	72	-2.91
13	123	126	1.35
14	82	81	-2.30
15	38	35	0.19
16	78	78	-2.98
17	62	69	-1.47
18	148	156	0.33
19	94	74	2.15
20	24	23	-3.07
21	21	22	-2.21

1 13 L			
0	108	109	-1.44
1	234	216	0.87
2	116	124	0.41
3	77	69	1.06
4	60	59	1.39
5	196	189	-0.71
6	194	191	-2.25
7	326	289	2.74
8	47	41	1.36
9	130	124	-2.30
10	47	47	0.59
11	122	140	-2.26
12	77	76	-1.95
13	144	145	0.98
14	48	46	0.87
15	0	6	0.64
16	51	42	2.48
17	125	121	-0.65
18	104	107	-2.26
19	92	92	2.28

1 15 L			
0	198	191	1.16
1	67	56	2.92
2	132	130	-2.05
3	52	50	-2.45
4	104	109	-1.37
5	0	17	0.26
6	183	166	2.44
7	63	74	3.13
8	101	108	-1.92
9	100	87	0.67
10	112	101	-2.21
11	126	99	-0.15
12	181	189	0.89
13	65	48	2.99
14	77	59	-0.75
15	69	61	-2.30
16	71	57	-0.84
17	26	19	-1.95
18	75	86	2.50
19	19	15	-2.96

1 17 L			
0	107	115	-2.85
1	103	104	2.40
2	0	12	-2.24
3	37	34	-2.16
4	101	98	-0.16
5	132	132	-2.58
6	58	56	1.57
7	150	152	0.48
8	56	60	-0.37
9	0	15	0.99
10	47	37	-2.63
11	60	58	-0.59
12	120	120	-2.90
13	77	78	2.11
14	41	37	-2.71
15	23	24	-1.84
16	39	48	-0.32
17	58	75	-2.53
18	22	26	2.02

1 19 L			
0	173	162	2.34
1	130	123	-1.25
2	89	83	-1.34
3	46	53	0.74
4	96	95	-2.34
5	112	107	1.10
6	135	140	1.01
7	127	107	-1.89
8	38	39	-2.01
9	56	56	-2.53
10	85	77	-0.86
11	120	95	2.23
12	109	101	2.27
13	89	84	-1.37
14	44	50	-1.17
15	36	46	0.72

1 21 L			
0	117	90	0.41
1	108	98	-0.02
2	23	30	0.11
3	0	10	2.92
4	37	25	-2.78
5	43	35	1.26
6	131	132	-2.66
7	45	40	-0.68
8	0	9	0.51
9	30	20	0.05
10	28	36	0.25
11	53	75	3.11
12	37	43	0.16

1 23 L			
0	58	69	-0.53
1	94	122	-1.95
2	37	45	2.63
3	27	31	-2.49
4	0	14	-0.23
5	94	104	2.21
6	42	42	-2.09
7	66	91	-1.35

2 0 L			
2	371	357	0.60
4	244	243	-0.40
6	1080	816	-3.03
8	156	139	-0.04
10	173	188	0.79
12	149	139	2.05
14	87	84	1.96
16	83	75	-1.02
18	277	237	-2.98
20	90	84	-3.35
22	0	15	1.40
24	22	26	2.40

2 2 L			
0	532	455	-1.71
1	164	195	2.66
2	199	199	2.06
3	102	112	-1.56
4	182	179	2.00
5	528	453	-2.37
6	397	373	-1.56
7	381	354	0.76
8	269	251	1.57
9	54	56	2.27
10	180	192	1.57
11	135	132	0.06
12	182	198	-1.61
13	79	82	2.41
14	93	104	1.69
15	92	96	-1.86
16	100	104	1.42
17	199	213	-2.56
18	94	87	-1.06
19	144	136	0.64
20	97	96	1.52
21	37	34	0.50
22	23	24	1.88
23	73	71	0.18
24	67	73	-1.74

2 4 L			
0	787	642	2.95
1	439	410	0.41
2	121	137	-0.76
3	123	137	0.55
4	237	238	0.76
5	54	61	1.84
6	249	260	1.06
7	65	68	0.38
8	68	69	1.46
9	27	29	1.07
10	148	143	-1.20
11	155	148	-2.91
12	296	271	-3.08
13	213	220	0.58
14	110	105	-1.22
15	31	34	2.39
16	31	32	2.03
17	70	70	-2.98
18	87	76	1.19
19	34	20	0.26
20	32	34	1.25
21	0	14	-2.21
22	38	28	-0.84
23	86	77	-2.56
24	75	76	3.04

2 6 L			
0	182	179	1.33
1	137	147	1.16
2	179	180	2.33
3	107	103	2.65
4	88	81	2.74
5	226	224	-0.74
6	110	122	0.26
7	193	210	2.49
8	198	199	-2.89
9	152	151	-2.30
10	105	104	2.61
11	315	315	-2.26
12	186	186	0.34
13	112	114	0.86
14	41	37	0.90
15	105	107	1.20
16	49	44	-3.08
17	96	103	-0.23
18	0	14	-1.89
19	77	74	2.14
20	89	82	-2.69
21	45	35	-1.48
22	21	19	1.72
23	75	78	-2.29
24	57	73	0.35

2 8 L			
0	324	364	0.84
1	252	247	-1.33
2	110	122	-2.96
3	111	109	-2.15
4	157	161	-1.91
5	234	242	2.74
6	227	226	2.33
7	247	274	0.08
8	126	137	-1.82
9	127	129	0.61
10	42	38	-2.70
11	106	103	1.42
12	268	294	0.84
13	128	141	-1.26
14	38	46	2.71
15	38	47	-2.33
16	64	62	-1.25
17	90	88	3.06
18	85	78	1.99
19	95	97	-0.25
20	69	61	-1.73
21	0	8	2.37
22	31	34	-2.77
23	30	38	2.76

2 10 L			
0	139	117	-0.14
1	399	395	-3.10
2	74	61	-2.15
3	96	84	-1.90
4	92	88	2.00
5	133	134	-1.35
6	281	278	0.58
7	131	132	1.63
8	60	53	2.32
9	53	51	2.86
10	92	95	-2.41
11	182	203	-0.11
12	47	33	-2.88
13	172	186	-3.10
14	121	132	-2.73
15	34	46	-0.44
16	62	67	3.13
17	36	39	-1.73
18	141	129	0.41
19	29	35	1.97
20	26	15	1.93
21	0	5	2.29
22	31	35	-2.71

2 12 L			
0	56	39	-2.65
1	211	231	-0.61
2	64	59	-1.99
3	111	104	0.80
4	170	154	-2.24
5	336	316	1.07
6	151	146	1.08
7	339	370	-2.11
8	0	19	-2.58
9	58	59	-2.23
10	61	58	0.37
11	121	139	-2.20
12	61	63	2.46
13	103	109	-0.66
14	0	21	3.07
15	42	32	1.75
16	30	23	2.98
17	139	133	1.15
18	118	103	0.60
19	124	121	-2.06

2 14 L			
0	134	108	1.11
1	112	101	2.97
2	88	83	0.95
3	73	54	1.47
4	74	61	-0.20
5	227	234	0.14
6	163	164	-3.09
7	142	143	-2.87
8	86	77	-1.77
9	0	16	0.32
10	38	30	1.89
11	99	77	0.27
12	86	73	0.89
13	68	46	3.02
14	62	57	0.89
15	0	11	2.23
16	0	15	-1.78
17	80	78	-0.05
18	103	112	-2.81
19	70	74	-2.97
20	27	37	-1.78

2 16 L			
0	139	130	-1.56

Table 3. Continued.

1 146 143 -2.12	20 68 52 2.66	6 78 72 -0.40	8 183 190 -1.76	21 28 42 2.41	10 118 114 1.59	3 71 64 1.26
2 106 96 2.24	21 62 54 0.53	7 136 114 -1.91	9 160 160 1.79		11 136 142 0.36	4 196 171 2.79
3 97 89 -2.32	22 38 30 -0.17	8 71 60 -2.42	10 113 122 -1.70	4 12 L	12 97 71 2.92	5 142 121 1.33
4 100 111 1.06	23 93 100 0.38	9 108 99 -1.73	11 219 222 0.99	0 211 183 -1.76	13 117 118 2.76	6 194 189 0.25
5 137 139 2.89	24 42 49 -2.22	10 38 30 2.96	12 198 208 1.67	1 314 292 1.23	14 68 64 1.49	7 105 99 -2.24
6 141 152 -1.81		11 145 136 -3.04	13 68 56 -2.30	2 71 67 -1.38	15 97 78 -0.86	8 180 175 2.55
7 98 105 -0.77	3 5 L	12 146 141 0.44	14 88 77 -1.10	3 167 150 1.63	16 66 64 -0.03	9 0 9 0.13
8 93 97 1.17	0 165 166 -2.13	13 127 131 -0.09	15 127 126 -1.45	4 114 106 -2.92	17 102 113 -1.58	10 0 6 2.30
9 42 44 1.48	1 182 186 1.47	14 57 49 -3.00	16 65 74 -1.83	5 218 202 -1.35	18 103 90 -2.65	11 96 78 0.79
10 69 57 2.66	2 129 133 -1.61	15 54 53 1.62	17 80 70 3.04	6 49 37 -1.16	19 63 56 1.60	12 123 125 -0.72
11 146 111 1.18	3 135 166 1.39	16 47 31 -2.32	18 162 162 1.53	7 164 148 2.13	20 70 54 0.25	13 37 44 -2.86
12 99 91 -1.37	4 26 13 -2.50	17 51 44 1.65	19 42 42 -0.51	8 60 50 2.35	21 65 55 2.47	14 93 79 -1.90
13 94 81 -2.11	5 134 130 -0.04	18 40 38 -0.37	20 70 67 -1.65	9 138 132 -1.44	22 35 25 1.75	15 0 11 1.44
14 68 58 1.55	6 268 291 0.67	19 26 30 -0.93	21 121 97 1.71	10 70 74 0.45		16 90 91 2.79
15 63 53 -2.09	7 146 158 2.74		22 36 30 -1.39	11 185 185 -1.84	5 3 L	17 31 31 0.77
16 60 68 0.79	8 130 127 -2.80	3 15 L	23 65 74 0.95	12 77 70 -2.21	0 236 262 -2.19	18 102 113 0.53
17 55 61 2.84	9 150 156 -2.07	0 115 96 -0.54		13 160 170 1.28	1 194 234 -0.23	19 45 53 -1.95
18 56 75 -2.17	10 47 30 -1.26	1 131 119 -0.34	4 4 L	14 0 6 2.34	2 75 90 0.11	
	11 165 169 -2.01	2 46 54 2.05	0 289 333 -1.87	15 77 75 1.96	3 98 108 -0.54	5 13 L
2 18 L	12 57 44 0.84	3 59 56 -2.50	1 140 146 -0.09	16 33 34 2.21	4 205 230 1.82	0 169 142 1.95
0 289 291 -3.00	13 105 103 1.42	4 103 89 -0.08	2 52 64 0.11	17 78 82 -1.31	5 234 254 -3.06	1 39 26 -2.70
1 114 117 2.25	14 40 52 0.32	5 101 101 2.99	3 151 164 1.39	18 48 47 0.17	6 96 106 1.52	2 46 35 1.09
2 65 61 -0.46	15 104 102 1.25	6 150 137 -2.33	4 165 175 1.92	19 69 78 1.92	7 233 248 -0.19	3 143 119 2.31
3 0 21 0.41	16 0 18 1.59	7 139 132 0.11	5 123 132 1.27	20 25 30 1.75	8 158 145 2.22	4 71 61 -1.31
4 81 74 0.86	17 63 59 0.13	8 47 51 -0.59	6 145 157 0.30		9 86 78 2.95	5 192 166 0.64
5 56 47 -1.22	18 87 88 0.44	9 90 82 1.10	7 128 129 -2.09	4 14 L	10 75 65 -0.62	6 66 71 -3.11
6 48 38 1.73	19 70 59 2.94	10 59 54 2.34	8 140 135 2.15	0 125 135 1.34	11 111 108 2.69	7 179 157 -2.40
7 77 81 1.16	20 27 25 -2.58	11 97 87 2.50	9 40 35 -0.80	1 86 67 1.26	12 142 141 -2.09	8 37 47 -0.37
8 81 74 1.03	21 69 63 -1.71	12 88 81 0.30	10 54 55 -1.10	2 55 48 2.25	13 160 152 -0.06	9 135 116 -1.02
9 0 23 2.77	22 28 27 0.16	13 94 99 -0.23	11 119 101 1.76	3 66 51 1.96	14 71 72 -0.39	10 35 35 0.50
10 53 52 -0.37	23 53 51 -1.93	14 94 86 1.46	12 276 264 -2.00	4 55 38 -0.57	15 56 55 -1.28	11 34 21 -1.11
11 71 65 -0.94		15 47 40 -2.23	13 129 78 -0.24	5 96 89 0.37	16 59 53 2.37	12 86 77 2.43
12 150 166 -2.94	3 7 L	16 65 58 0.16	14 55 51 -1.04	6 136 129 -2.06	17 134 145 -2.86	13 0 13 -2.22
13 68 72 2.10	0 121 133 1.93	17 60 63 -2.86	15 59 59 2.38	7 53 51 -2.91	18 0 16 2.13	14 0 6 -0.91
14 59 59 -0.33	1 92 86 -3.06	18 60 77 2.34	16 56 50 2.24	8 60 58 -1.31	19 107 106 0.09	15 71 68 2.19
15 28 31 0.03	2 228 228 0.87		17 50 35 1.85	9 59 56 0.15	20 60 48 2.05	16 43 43 -0.87
16 23 31 1.06	3 41 33 -2.95	3 17 L	18 38 18 0.49	10 44 35 -2.98	21 65 52 1.99	17 69 78 0.66
	4 256 253 -0.15	0 156 144 -0.44	19 62 64 -1.77	11 9 14 2.71	22 35 35 -0.32	
2 20 L	5 235 253 -0.03	1 164 157 -2.76	20 67 58 2.34	12 83 78 1.27		5 15 L
0 75 59 -0.38	6 511 504 -2.91	2 34 44 -2.77	21 39 36 -1.04	13 51 51 1.41	5 5 L	0 89 65 -1.26
1 152 144 0.29	7 141 162 3.03	3 65 53 -1.44	22 40 35 -0.31	14 0 13 2.20	0 119 126 -2.07	1 129 105 -2.76
2 88 76 1.07	8 238 248 -0.26	4 112 98 2.29	23 17 19 2.66	15 0 6 -3.11	1 244 260 1.11	2 114 90 2.34
3 31 33 1.78	9 79 85 0.42	5 33 20 -2.34		16 37 31 -0.95	2 120 131 -1.05	3 68 52 -0.60
4 80 69 2.88	10 48 46 0.35	Δr 0 10 40.92	4 6 L	17 38 34 0.05	3 161 189 2.16	4 58 49 0.37
5 0 10 -0.33	11 79 77 -0.37	6 78 75 2.75	0 102 109 1.83	18 55 72 -2.15	4 32 37 2.46	5 88 70 -0.29
6 80 79 -1.42	12 105 95 2.63	7 70 54 1.67	1 248 260 -1.13		5 85 66 0.07	6 122 105 -1.66
7 0 13 2.36	13 84 63 -2.80	8 74 74 -2.57	2 60 63 3.10	4 16 L	6 136 134 1.58	7 92 95 2.63
8 60 49 2.35	14 123 124 0.96	9 108 97 0.53	3 164 168 2.18	0 145 131 1.48	7 82 80 2.90	8 0 18 -0.48
9 57 55 -1.73	15 48 42 2.27	10 90 84 -0.68	4 46 44 -1.30	1 129 95 2.12	8 0 17 2.45	9 35 44 1.74
10 72 70 1.12	16 135 139 -0.47	11 123 113 -2.78	5 269 270 1.22	2 92 82 -2.04	9 164 169 -0.71	10 86 81 2.36
11 118 129 -2.77	17 106 111 0.23	12 83 72 -2.56	6 98 92 -1.48	3 127 118 -1.41	10 37 39 -0.84	11 91 94 0.50
12 42 42 -1.68	18 232 237 -2.69	13 36 31 -0.83	7 315 337 -2.06	4 100 85 -0.88	11 172 156 -2.04	12 52 31 0.42
13 64 94 0.22	19 90 72 3.13	14 47 53 2.93	8 81 87 -1.93	5 171 174 -1.96	12 131 99 2.49	13 77 77 -2.85
	20 103 96 -0.56		9 164 180 -1.19	6 149 164 1.81	13 145 144 1.11	14 105 97 1.83
2 22 L	21 40 22 -0.81	3 19 L	10 87 82 -2.44	7 120 116 1.16	14 40 49 0.60	15 36 35 -0.96
0 113 113 1.09	22 30 21 1.15	0 158 127 -2.33	11 144 131 2.46	8 106 104 -1.29	15 80 72 2.40	16 27 31 0.89
1 58 57 0.75	23 19 23 -1.17	1 82 75 1.52	12 62 56 0.93	9 81 76 1.75	16 34 29 1.82	
2 24 18 -1.86		2 33 25 -0.01	13 167 168 -1.31	10 81 88 -2.04	17 35 34 -0.36	5 17 L
3 23 22 -2.84	3 9 L	3 32 35 1.23	14 33 29 1.05	11 74 70 -1.21	18 57 53 1.94	0 160 144 -2.91
4 58 64 -1.28	0 115 119 1.02	4 46 46 2.72	15 100 100 1.93	12 104 96 1.33	19 43 25 2.62	1 99 71 -1.78
5 73 88 -2.09	1 102 97 -2.05	5 0 17 -1.14	16 0 13 -1.38	13 80 60 2.20	20 0 19 -2.95	2 90 80 -0.30
6 106 122 2.89	2 82 69 -1.37	6 94 75 -0.20	17 159 159 1.09	14 38 35 -2.05	21 62 64 -0.76	3 102 84 -1.10
7 70 81 0.99	3 245 212 -1.89	7 29 28 0.07	18 77 70 -1.97	15 56 68 -1.19		4 106 93 0.85
8 55 56 -1.01	4 85 84 -0.96	8 49 50 2.49	19 93 97 -1.97	16 33 33 -0.95	5 7 L	5 82 77 -2.10
9 14 22 0.35	5 258 264 -2.54	9 74 70 -2.15	20 30 34 -2.08		0 155 253 0.48	6 155 158 2.63
	6 96 95 1.71	10 50 52 0.54	21 41 39 -0.83	4 18 L	1 147 152 -0.11	7 100 112 0.20
3 1 L	7 267 247 0.12	11 78 75 -2.14	22 0 12 2.07	0 145 124 -2.35	2 239 228 2.41	8 73 63 0.51
0 428 447 0.35	8 78 83 -0.58	12 61 57 -2.15	23 25 48 2.21	1 121 136 -1.93	3 34 37 -2.80	9 76 66 2.49
1 38 48 0.56	9 102 108 1.54	13 47 48 1.55		2 67 55 -1.04	4 56 42 2.85	10 70 68 -0.79
2 179 242 2.81	10 59 52 -0.79	14 24 31 -0.01	4 8 L	3 0 20 1.10	5 105 91 2.61	11 44 24 1.59
3 90 93 -1.54	11 34 25 2.06		0 182 180 -0.79	4 131 133 2.46	6 236 237 -0.31	12 97 100 -2.78
4 117 147 -1.77	12 92 95 1.01	3 21 L	1 124 125 2.30	5 102 100 1.95	7 57 50 0.11	13 28 33 -1.62
5 159 197 -2.96	13 73 71 -1.95	0 159 148 3.56	2 156 148 2.22	6 90 75 0.88	8 158 149 -2.31	
6 281 312 -1.42	14 51 36 -1.86	1 74 61 2.92	3 166 161 -1.23	7 78 74 -0.52	9 3 34 0.06	5 19 L
7 412 427 0.58	15 79 69 -1.63	2 33 41 1.93	4 54 48 0.75	8 50 47 2.30	10 139 142 2.79	0 120 107 -2.15
8 70 64 1.89	16 0 19 -0.96	3 48 38 2.29	5 149 139 -2.00	9 28 27 3.12	11 117 114 3.08	1 79 76 1.58
9 169 171 1.37	17 92 86 -2.87	4 32 31 -2.01	6 399 435 -1.97	10 41 38 -1.01	12 183 190 0.41	2 71 59 0.12
10 120 118 2.31	18 73 69 1.39	5 170 174 0.09	7 249 283 -1.38	11 67 60 1.45	13 99 86 0.04	3 45 41 2.47
11 61 63 1.06	19 99 88 0.33	6 70 75 -1.88	8 111 114 0.53	12 63 71 -2.23	14 120 123 2.50	4 60 49 2.69
12 306 331 0.10	20 23 30 -0.32	7 128 143 3.13	9 144 152 1.54	13 57 71 -1.93	15 34 24 2.98	5 39 40 -0.75
13 88 88 0.54	21 61 49 1.68	8 22 17 -0.66	10 129 120 2.08	14 31 44 -0.84	16 56 57 -2.17	6 51 39 -0.26
14 94 97 2.60	22 18 29 -2.21	9 45 51 -0.00	11 95 101 -0.85		17 53 48 2.52	7 26 35 2.61
15 81 79 -1.45		10 28 31 2.05	12 99 94 -0.79	4 20 L	18 101 109 -0.72	8 61 56 2.45
16 0 16 1.16	3 11 L		13 67 63 2.40	0 164 147 1.84	19 43 27 0.03	9 56 56 -0.96
17 149 159 -2.48	0 381 365 -2.96	3 23 L	14 115 112 2.07	1 85 72 -0.20	20 61 67 -2.21	10 45 48 0.54
18 72 65 -0.93	1 100 94 0.57	0 30 30 1.82	15 63 71 -1.13	2 71 49 -1.05	21 32 22 -0.66	
19 83 81 0.36	2 232 224 -0.30	1 28 23 3.05	16 80 65 0.90	3 76 66 2.11		5 21 L
20 0 23 1.88	3 84 82 0.75	2 0 3 -0.97	17 64 57 -2.11	4 72 73 -2.07	5 9 L	0 81 84 1.78
21 63 33 1.88	4 45 41 0.50	3 36 41 -1.82	18 159 153 -1.86	5 54 49 1.08	0 274 247 1.27	1 99 118 0.11
22 56 40 2.96	5 132 112 2.54	4 36 56 -0.10	19 54 59 1.19	6 70 68 1.21	1 191 178 -2.39	2 37 38 1.80
23 0 5 0.27	6 124 129 2.27		20 36 37 0.61	7 45 45 -1.99	2 107 97 -2.08	3 41 43 2.66
24 67 84 0.03	7 122 112 -0.26	4 0 L		8 58 63 -2.16	3 196 184 -1.11	4 36 43 -0.99
	8 144 145 0.52	2 42 49 2.52	4 10 L	9 55 58 -1.15	4 108 85 -1.54	5 36 54 2.86
3 3 L	9 74 70 -3.03	4 91 95 -1.54	0 210 210 -1.41	10 38 35 -0.92	5 137 122 -1.88	6 29 40 -2.38
0 45 48 -2.40	10 199 196 -0.18	6 399 404 -2.30	1 174 166 -2.37		6 156 159 2.36	
1 146 218 -2.92	11 38 37 2.54	8 153 152 -0.44	2 73 60 0.99	4 22 L	7 68 56 0.64	6 0 L
2 192 204 -0.30	12 271 293 -2.82	10 67 66 2.50	3 137 120 -1.14	0 51 58 -0.59	8 93 92 -0.15	2 223 279 2.71
3 71 76 -0.02	13 68 66 0.35	12 135 135 1.50	4 99 99 2.42	1 56 64 -1.54	9 119 119 2.46	4 63 69 -1.36
4 106 114 1.63	14 134 111 -0.60	14 39 41 -1.76	5 100 90 3.12	2 49 54 2.24	10 37 37 -2.63	6 181 195 -0.33
5 217 216 -0.52	15 0 3 -3.69	16 66 65 -0.98	6 70 65 0.49	3 31 34 -1.76	11 91 84 0.53	8 99 132 -1.00
6 429 438 0.44	16 33 32 0.93	18 119 132 -2.24	7 75 68 -0.58	4 26 27 0.01	12 114 116 1.51	10 159 159 2.76
7 667 635 2.99	17 58 63 2.87	20 74 60 -0.53	8 98 92 1.84	5 32 50 1.41	13 105 107 -2.56	12 214 230 0.31
8 132 130 2.22	18 69 62 1.77	22 29 26 -3.00	9 72 68 2.48		14 75 83 -2.05	14 170 172 3.13
9 39 41 1.32	19 50 47 -0.78		10 33 29 0.74	5 1 L	15 70 73 -0.72	16 62 62 -1.19
10 101 101 -0.97	20 61 59 0.73	4 2 L	11 98 95 0.67	0 90 126 2.60	16 0 19 0.11	18 125 126 -0.50
11 330 335 0.18	21 33 40 -2.58	0 176 227 1.97	12 175 192 -1.79	1 109 156 2.78	17 48 36 -1.75	20 63 53 -1.17
12 212 196 -2.25		1 108 120 -1.94	13 106 114 -2.54	2 109 157 1.70	18 76 93 1.82	
13 167 162 -2.84	3 13 L	2 221 277 -1.61	14 38 41 -2.39	3 114 142 -0.18	19 41 39 1.22	6 7 L
14 87 87 -1.34	0 235 221 0.44	3 191 230 -1.01	15 72 64 -0.95	4 92 105 -0.36	20 34 32 -0.21	0 207 56 -1.80
15 60 63 -2.61	1 223 218 0.14	4 236 280 -1.71	16 47 39 -2.80	5 142 143 -1.70		1 42 60 3.08
16 71 67 2.60	2 59 61 3.08	5 78 88 3.11	17 26 19 3.08	6 230 283 -2.37	5 11 L	2 52 62 0.29
17 96 87 -0.53	3 150 135 1.46	6 384 396 1.50	18 45 41 0.29	7 170 180 1.59	0 154 139 -0.47	3 122 149 -0.62
18 118 109 0.24	4 62 56 -2.53	7 194 187 -1.05	19 33 23 -0.56	8 136 138 -0.07	1 67 57 -2.51	4 86 90 1.16
19 99 89 2.96	5 72 68 2.10		20 36 37 1.87	9 156 158 2.13	2 70 43 -2.48	5 157 176 -1.32

Table 3. Continued.

6 122 132 -1.49	8 124 125 -0.43	12 87 70 -2.76	2 63 38 2.38	16 42 44 -0.88	7 7 L	8 81 62 2.89
7 61 58 -2.82	9 132 139 -0.79	13 63 60 -0.08	3 100 104 -0.90	17 49 39 2.00	0 149 152 2.14	9 0 4 1.06
8 71 70 2.05	10 0 10 -0.39	14 115 127 -0.74	4 54 55 1.13	18 33 30 1.06	1 36 27 1.46	10 52 56 -0.08
9 109 104 2.54	11 142 151 -1.73	15 57 47 -0.43	5 60 43 0.20	19 60 76 -1.11	2 91 91 3.08	11 49 30 -1.40
10 75 76 0.99	12 83 83 2.79	16 0 10 -1.96	6 69 65 -1.61		3 44 45 -2.79	12 52 45 -2.12
11 62 67 0.66	13 91 96 1.13	17 77 84 -2.74	7 59 39 -2.27	7 3 L	4 99 96 -0.10	13 54 56 1.77
12 85 80 -1.67	14 50 44 0.96	18 43 57 2.07	8 0 25 0.81	0 51 37 -2.25	5 50 34 -0.20	14 74 92 -0.57
13 63 67 2.81	15 71 72 2.68		9 60 55 2.36	1 66 79 2.31	6 61 41 -1.53	15 40 23 1.75
14 67 62 1.00	16 60 63 -0.22	6 12 L	10 32 28 2.87	2 81 91 -0.73	7 64 58 2.85	
15 92 91 -0.82	17 0 9 -0.68	0 46 34 -2.60	11 63 64 0.93	3 85 87 -0.33	8 102 89 -0.46	7 13 L
16 48 48 1.62	18 63 67 -2.72	1 66 46 -1.21	12 43 57 -0.97	4 108 122 -2.80	9 30 22 0.56	0 92 41 -2.48
17 74 84 -1.45	19 36 47 1.80	2 132 96 -0.54	13 33 60 -1.92	5 43 45 -0.58	10 65 64 -2.80	1 145 113 1.26
18 38 40 -1.68	20 33 49 -0.50	3 119 104 2.25		6 131 155 1.88	11 31 24 -1.73	2 65 54 2.02
19 52 49 1.99		4 40 30 -2.39	6 18 L	7 63 66 -1.55	12 80 70 2.11	3 147 110 2.56
20 39 42 1.82	6 8 L	5 159 131 1.55	0 130 121 -0.50	8 91 78 -2.73	13 0 6 0.88	4 83 36 1.49
21 54 70 2.03	0 218 191 1.45	6 58 28 0.60	1 24 27 2.70	9 66 50 2.48	14 38 38 -2.97	5 73 72 -1.13
	1 195 173 -2.49	7 157 149 -1.52	2 37 34 -1.04	10 94 91 -0.72	15 28 25 -3.06	6 94 101 -1.30
6 4 L	2 58 57 -2.47	8 45 48 2.14	3 42 21 0.07	11 39 37 -0.13	16 42 57 -0.21	7 27 32 1.94
0 141 153 -0.60	3 110 101 -1.21	9 84 82 -1.00	4 81 91 2.92	12 41 33 -1.06	17 23 27 0.61	8 48 30 1.55
1 147 168 2.40	4 187 175 -0.66	10 62 52 0.11	5 75 56 -3.05	13 51 49 2.57		9 100 117 -0.59
2 98 99 -0.83	5 120 105 0.59	11 44 41 1.50	6 100 119 0.48	14 80 84 -0.51	7 9 L	10 24 25 1.91
3 90 105 1.97	6 87 81 3.06	12 34 28 2.64	7 90 86 0.77	15 65 71 -0.80	0 219 212 -1.46	11 75 73 -1.84
4 166 188 2.86	7 108 101 2.73	13 35 30 -0.52	8 71 85 2.92	16 46 54 -2.55	1 91 62 -2.24	12 47 37 -1.91
5 120 127 0.16	8 139 155 -0.51	14 60 55 -0.47	9 36 25 2.81	17 33 29 -1.07	2 108 78 1.79	13 53 65 1.18
6 205 216 0.85	9 115 112 2.26	15 55 62 2.43		18 70 60 1.63	3 186 160 -0.67	
7 119 111 -2.99	10 55 52 3.11	16 26 25 -2.98	6 20 L		4 72 60 1.20	7 15 L
8 144 146 2.99	11 81 66 0.79	17 55 59 1.59	0 50 57 -2.47	7 5 L	5 38 39 -2.08	0 130 106 1.55
9 43 38 -0.40	12 127 130 1.60		1 47 49 2.61	0 197 221 -1.64	6 215 210 -1.69	1 71 46 0.32
10 118 116 -0.83	13 97 104 -2.57	6 14 L	2 46 57 1.09	1 150 144 -1.77	7 33 36 0.04	2 86 92 -2.31
11 101 92 -0.02	14 44 45 -2.96	0 113 98 0.96	3 38 52 2.72	2 73 67 -0.30	8 86 85 1.69	3 42 48 -0.77
12 98 92 -0.67	15 50 56 -0.79	1 178 155 0.03		3 171 180 2.54	9 101 116 2.72	4 94 88 -0.82
13 118 114 2.21	16 63 60 -0.39	2 106 106 2.55	7 1 L	4 86 75 2.07	10 85 77 1.73	5 27 22 -2.45
14 67 53 -0.97	17 49 45 0.05	3 124 70 2.46	0 166 223 1.36	5 170 178 1.29	11 42 26 0.76	6 63 69 1.57
15 0 15 2.40	18 38 44 2.60	4 70 57 -0.50	1 136 177 -1.82	6 68 66 -1.29	12 128 143 -1.48	7 36 23 0.98
16 72 71 2.96	19 38 47 -2.78	5 94 68 3.08	2 70 98 -2.78	7 140 134 -1.58	13 35 38 -2.22	8 92 92 -0.99
17 65 70 -0.48		6 74 59 -0.80	3 104 108 -0.42	8 82 74 2.48	14 58 66 2.04	9 45 51 2.52
18 86 98 0.61	6 10 L	7 63 48 -0.02	4 92 121 -1.06	9 115 120 -0.53	15 79 89 -0.49	10 62 71 -2.45
19 55 48 -3.12	0 165 140 2.91	8 70 65 -0.78	5 130 147 1.96	10 49 35 -0.14	16 47 48 1.48	11 34 29 -2.75
20 65 62 3.01	1 97 88 -0.14	9 62 43 -0.68	6 36 32 1.54	11 114 105 1.57		
	2 156 132 -0.41	10 51 61 2.95	7 130 119 -1.13	12 74 67 -1.78	7 11 L	7 17 L
6 6 L	3 123 97 -0.63	11 116 113 2.95	8 90 95 -0.91	13 91 96 -1.59	0 62 44 3.01	0 83 59 -0.83
0 137 129 2.87	4 0 28 2.94	12 50 58 0.62	9 154 156 2.71	14 66 66 1.38	1 102 79 1.85	1 22 37 2.27
1 183 191 1.28	5 188 180 -2.71	13 88 104 3.01	10 66 71 -2.89	15 80 91 2.40	2 108 93 -0.24	2 22 13 -1.40
2 74 78 0.78	6 136 116 2.26	14 47 59 2.61	11 125 133 1.67	16 43 57 2.43	3 57 57 1.85	3 82 76 -0.51
3 120 122 2.58	7 180 182 0.36	15 41 39 2.90	12 100 103 1.52	17 68 87 1.34	4 83 73 -2.52	4 51 45 2.74
4 170 178 0.04	8 49 48 2.11		13 116 125 -1.76	18 29 47 -1.49	5 88 57 -1.90	5 67 56 -1.90
5 87 91 -1.21	9 89 89 2.71	6 16 L	14 83 96 -2.63		6 89 75 2.20	6 34 43 1.86
6 104 100 -2.73	10 123 117 -0.23	0 103 97 -1.11	15 80 83 -0.44		7 92 82 1.65	7 49 59 1.27
7 72 59 1.45	11 74 62 -3.09	1 121 99 -1.97				

Table 4. Bond distances and angles in $K_8Mo_7O_{22}(O_2)_2.8H_2O$. The numbers in parentheses are the standard deviations of the last significant figures.

	Distance (Å)		Distance (Å)
Mo(1) – O(1)	1.96(4)	Mo(3) – O(5)	2.20(2)
– O(2)	1.86(5)	– O(6)	1.99(2)
– O(1,2)	1.91(10)	– O(8)	2.34(3)
– O(3)	2.49(3)	– O(10)	1.68(3)
– O(4)	1.64(3)	– O(11)	1.91(3)
– O(5)	2.08(2)	– O(12)	1.73(2)
– O(6)	1.97(2)	Mo(4) – O(5)	2.16(2)
– O(7)	1.98(2)	– O(7)	1.99(3)
O(1) – O(2)	1.38(6)	– O(9)	2.28(2)
Mo(2) – O(3)	1.76(3)	– O(13)	1.66(3)
– O(5)	2.33(2)	– O(14)	1.95(2)
– O(8)	1.86(3)	– O(15)	1.75(4)
– O(9)	1.93(2)		

	Angle (°)		Angle (°)
O(1) – Mo(1) – O(2)	42(2)	O(5) – Mo(3) – O(6)	73(1)
– O(3)	88(1)	– (8)	72(1)
– O(4)	95(1)	– O(10)	92(1)

Table 4. Continued.

–O(5)	152(1)	–O(11)	87(1)
–O(6)	81(1)	–O(12)	156(1)
–O(7)	123(1)	O(6)–Mo(3)–O(8)	82(1)
O(2)–Mo(1)–O(3)	86(2)	–O(10)	99(1)
–O(4)	95(2)	–O(11)	151(1)
–O(5)	150(1)	–O(12)	90(1)
–O(6)	123(2)	O(8)–Mo(3)–O(10)	163(1)
–O(7)	81(2)	–O(11)	72(1)
O(3)–Mo(1)–O(4)	177(1)	–O(12)	90(1)
–O(5)	72(1)	O(10)–Mo(3)–O(11)	102(1)
–O(6)	81(1)	–O(12)	107(1)
–O(7)	80(1)	O(11)–Mo(3)–O(12)	102(1)
O(4)–Mo(1)–O(5)	106(1)	O(5)–Mo(4)–O(7)	73(1)
–O(6)	101(1)	–O(9)	72(1)
–O(7)	97(1)	–O(13)	95(1)
O(5)–Mo(1)–O(6)	76(1)	–O(14)	90(1)
–O(7)	76(1)	–O(15)	154(2)
O(6)–Mo(1)–O(7)	198(1)	O(7)–Mo(4)–O(9)	83(1)
O(3)–Mo(2)–O(3′)	106(2)	–O(13)	101(1)
–O(5)	82(1)	–O(14)	154(1)
–O(5′)	172(1)	–O(15)	90(2)
–O(8)	101(1)	O(9)–Mo(4)–O(13)	165(1)
–O(9)	102(1)	–O(14)	73(1)
O(5)–Mo(2)–O(5′)	90(1)	O(9)–Mo(4)–O(15)	87(1)
–O(8)	78(1)	O(13)–Mo(4)–O(14)	100(1)
–O(9)	75(1)	–O(15)	107(2)
O(8)–Mo–O(9)	141(2)	O(14)–Mo(4)–O(15)	99(1)

for all molybdenum atoms. The refinement converged after about ten cycles to the parameters given in Table 2, the ultimate R-value being 0.097. Observed and calculated structure factors are given in Table 3. Calculated bond distances and angles with their standard deviations are given in Table 4 and some packing distances in Table 5. All bond distances were found to be within the normal range. The results obtained from the refinement were checked by calculating a three-dimensional electron density difference map. This showed only small residual maxima, the largest maximum having a magnitude of about 30 % of the height of an oxygen peak in the F_o-synthesis.

DESCRIPTION AND DISCUSSION

The crystal structure of $K_6Mo_7O_{22}(O_2)_2.8H_2O$, illustrated in Fig. 2, consists of diperoxoheptamolybdate(VI) ions, potassium ions, and lattice water molecules. As can be seen from the packing distances in Table 5, there are some short distances between water oxygen atoms and other oxygen atoms indicating possible hydrogen bonds. In the structure determination no evidence for a ninth water molecule, as suggested by the chemical analysis, was found. There is, however, space enough for another water molecule and a possible explanation might be that the ninth water molecule is statistically distributed. There is no regular oxygen co-ordination around the potassium ions.

Table 5. Packing distances in $K_6Mo_7O_{22}(O_2)_2.8H_2O$. Distances less than 3.5 Å are included. The number in parentheses after the atom symbol denotes:

(1)	x	y	z	(5)	$\frac{1}{2}+x$	$\frac{1}{2}+y$	z
(2)	$1+x$	y	z	(6)	$-\frac{1}{2}+x$	$\frac{1}{2}+y$	z
(3)	$\frac{1}{2}+x$	$\frac{1}{2}-y$	$\bar{z}$	(7)	$-1+x$	y	z
(4)	$-\frac{1}{2}+x$	$\frac{1}{2}-y$	$\bar{z}$				

	Distance (Å)		Distance (Å)
K(1)(1) – O(1)(2)	2.67	K(4)(1) – O(13)(1)	2.84
– O(2)(2)	3.46	– O(19)(4)	2.81
– O(10)(1)	3.35	– O(20)(1)	2.80
– O(18)(3)	2.90	O(16)(1) – O(8)(5)	2.85
– O(19)(4)	2.74	– O(12)(5)	3.40
K(2)(1) – O(9)(2)	2.79	– O(14)(1)	3.16
– O(12)(5)	2.70	– O(18)(1)	3.31
– O(15)(2)	2.72	O(17)(1) – O(3)(2)	3.28
– O(16)(1)	2.92	– O(11)(1)	2.90
– O(17)(1)	3.27	– O(14)(1)	2.78
– O(20)(1)	2.90	– O(20)(1)	2.82
K(3)(1) – O(1)(3)	2.87	O(18)(1) – O(1)(5)	3.18
– O(2)(3)	3.28	– O(3)(5)	2.94
– O(4)(3)	2.97	– O(6)(5)	3.41
– O(6)(5)	2.82	– O(10)(6)	3.44
– O(12)(5)	3.15	– O(15)(1)	2.92
– O(13)(1)	2.96	– O(19)(7)	3.48
– O(16)(1)	2.93	O(19)(1) – O(4)(3)	3.02
– O(18)(1)	2.79	– O(7)(2)	3.41
– O(20)(1)	2.86	– O(10)(3)	3.22
K(4)(1) – O(2)(2)	2.89	– O(12)(5)	2.87
– O(2)(3)	3.36	– O(15)(2)	2.91
– O(4)(1)	3.35	O(20)(1) – O(2)(2)	3.39
– O(4)(3)	2.97	– O(4)(3)	3.27
– O(7)(3)	3.02	– O(7)(2)	2.69
– O(10)(1)	2.86	– O(13)(1)	3.28

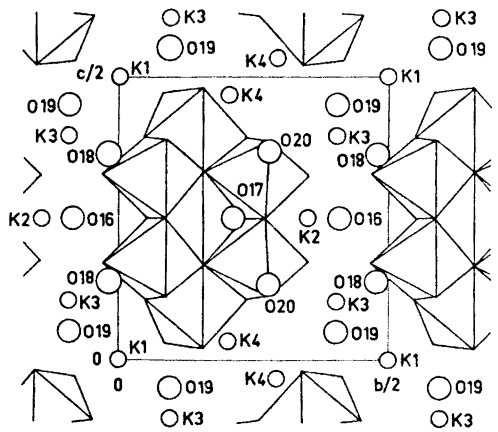

Fig. 2. The packing of the ions and water molecules in $K_6Mo_7O_{22}(O_2)_2.8H_2O$ as viewed along the x-direction. The $[Mo_7O_{22}(O_2)_2]^{6-}$ ion is shown schematically.

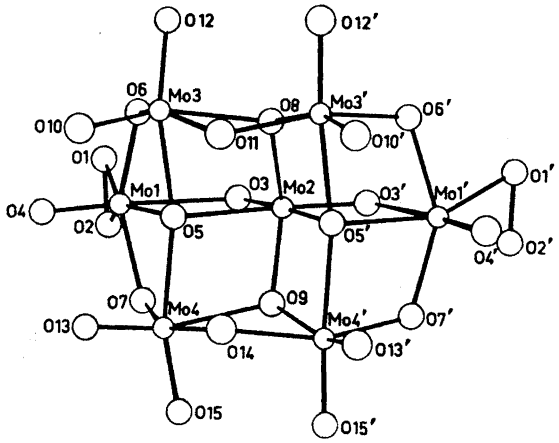

Fig. 3. The $[Mo_7O_{22}(O_2)_2]^{6-}$ ion.

The $[Mo_7O_{22}(O_2)_2]^{6-}$ complex ion is shown in Fig. 3. It is evident that the diperoxoheptamolybdate ion has the same general appearance as the normal heptamolybdate ion,[10] the difference being that two peroxo groups replace two oxygen atoms, one at either end of the heptamolybdate ion. The two molybdenum atoms coordinated to the peroxo groups are 7-coordinated by the pentagonal bipyramidal arrangement of ligand atoms frequently encountered in transition metal peroxo compounds. The structure of the diperoxoheptamolybdate ion can be described as being built up from five octahedra and two pentagonal bipyramids. As can be seen from Fig. 4, the atoms O(3) and O(4) are apically situated, while O(1), O(2), O(5), O(6), and O(7) form the equatorial plane. The two oxygen atoms O(1) and O(2) of the peroxo group are situated at 1.91 Å, on the average, from the central molybdenum atom. This implies that these Mo – O bonds are single bonds and their lengths agree well with $Mo-O_{peroxo}$ distances found in other 7-coordinated peroxomolybdates (see Table 6). The remaining three equatorial oxygen atoms are almost equally

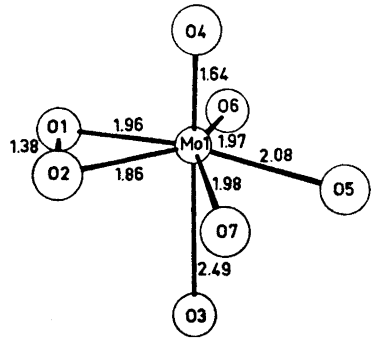

Fig. 4. The configuration about the 7-coordinated molybdenum atoms in the complex ion $[Mo_6O_{22}(O_2)_2]^{6-}$.

Table 6. Comparison between corresponding distances in $K_6Mo_7O_{22}(O_2)_2.8H_2O$ and other 7-coordinated peroxomolybdates.

Compound	Mo−$O_{bridging}$ (Å)	Mo−O_{peroxo} (Å)	M=O (Å)	(O−O)$_{peroxo}$ (Å)	Mo−equatorial plane (Å)	Ref.
$K_2[O(MoO(O_2)_2(H_2O))_2].2H_2O$	1.92−1.93	1.94−1.98	1.66	1.46−1.53	0.38−0.39	12
$(pyH)_2[O(MoO(O_2)_2(H_2O))_2]$	1.91	1.96−1.98	1.68	1.44−1.46	0.40	13, 14
$(pyH)_2[(O_2)_2OMo(OOH)_2MoO(O_2)_2]$	2.05−2.39	1.92−1.96	1.67	1.47−1.48	0.40	13, 14
$K_2[MoO(O_2)_2(C_2O_4)]$		1.93−1.96	1.68	1.44−1.47	0.35	15
$(NH_4)_3F[MoO(O_2)F_4]$		1.91	1.67	1.36	0.23	16
$K_2[MoO(O_2)F_4].H_2O$		1.94−1.95	1.64	1.44	0.18	17
$K_6Mo_7O_{22}(O_2)_2.8H_2O$	1.97−2.49	1.86−1.96	1.64	1.38	0.31	This paper

Table 7. Comparison between corresponding distances in $K_6Mo_7O_{22}(O_2)_2.8H_2O$ and other heptamolybdates.

	Distances (Å) in			
Defining atoms	$(NH_4)_6Mo_7O_{24}.4H_2O$	$(NH_4)_6Mo_7O_{24}.4H_2O$	$K_6Mo_7O_{24}.4H_2O$	$K_6Mo_7O_{22}(O_2)_2.8H_2O$
Mo(1)−Mo(2)	3.43	3.35 3.48	3.45 3.40	3.40
Mo(1)−Mo(3) Mo(1)−Mo(4)	3.21	3.21, 3.35 3.21, 3.27	3.20, 3.26 3.16, 3.19	3.21 3.22
Mo(2)−Mo(3) Mo(2)−Mo(4)	3.41	3.29, 3.39 3.36, 3.45	3.42, 3.44 3.41, 3.38	3.47 3.47
Mo(3)−Mo(3′) Mo(4)−Mo(4′)	3.26	3.27 3.28	3.25 3.24	3.30 3.31
Mo(1)−(O(1),O(2))	1.74			1.96 1.86
Mo(1)−O(3)	2.42			2.49
Mo(1)−O(4)	1.72			1.64
Mo(1)−O(5)	2.16			2.08
Mo(1)−O(6) Mo(1)−O(7)	1.92			1.97 1.98
Mo(2)−O(3)	1.75			1.76
Mo(2)−O(5)	2.26			2.33
Mo(2)−O(8) Mo(2)−O(9)	1.90			1.86 1.93
Mo(3)−O(5) Mo(4)−O(5)	—		Mo−O, distances: 1.51, 1.61−2.45, 2.62	2.20 2.16
Mo(3)−O(6) Mo(4)−O(7)	1.97			1.99 1.99
Mo(3)−O(8) Mo(4)−O(9)	2.18			2.34 2.28
Mo(3)−O(10) Mo(4)−O(13)	1.73			1.68 1.66
Mo(3)−O(11) Mo(4)−O(14)	1.95			1.91 1.95
Mo(3)−O(12) Mo(4)−O(15)	1.71			1.73 1.75
Ref.	22	10	23	This paper

remote from the central molybdenum atom (1.97 – 2.08 Å). The Mo(1) – O(4) bond, which is 1.64 Å, is significantly shorter, indicating considerable double bond character, while the Mo(1) – O(3) distance is significantly longer, *i.e.* 2.49 Å, and is thus a rather weak bond. The observed $(O-O)_{peroxo}$ distance is rather short (1.38 Å) compared with the value 1.49 Å found in hydrogen peroxide and many metal peroxides (see Table 6 in Ref. 18). It cannot, however, be claimed to be significantly shorter since the e.s.d. is rather large, (0.06 Å), but short $(O-O)_{peroxo}$ distances have been observed in several compounds, *e.g.* $[CrO(O_2)_2py]$ (1.40 Å),[19] $[CrO(O_2)_2phen]$ (1.40 Å),[20] $[CrO(O_2)_2dipy]$ (1.40 Å),[21] and $(NH_4)_3F[MoO(O_2)F_4]$ (1.36 Å).[16] Comparison between corresponding distances in $K_6Mo_7O_{22}(O_2)_2.8H_2O$ and other 7-coordinated peroxomolybdates is made in Table 6. The distances between atoms in the diperoxoheptamolybdate ion (except $Mo-O_{peroxo}$) agree well with corresponding distances in the normal heptamolybdates (see Table 7).

Within the limits of experimental error all five equatorial atoms in the pentagonal bipyramid lie in a plane. The equation of this plane, **A**, as determined by the least-squares method, is

A: $$0.6860\ X + 0.1967\ Y - 0.7005\ Z = -0.6122$$

where X, Y, and Z are the atomic coordinates expressed in Å, referred to the original axes. The distances from certain atoms to the plane are:

A – O(1)	– 0.01 Å		**A** – O(7)	0.05 Å	
O(2)	– 0.03		Mo(1)	0.31	
O(5)	– 0.05		O(3)	– 2.16	
O(6)	0.04		O(4)	1.94	

The molybdenum atom is thus displaced 0.31 Å from the equatorial plane in the direction of the double-bonded oxygen atom, O(4). A similar displacement of the metal atom from the equatorial plane has been observed in a number of 7-coordinated transition metal oxoperoxo compounds. Some examples are cited in Table 6. The atoms Mo(1), Mo(1′), Mo(2), O(3), O(3′), O(4), O(4′), O(5), and O(5′) all lie in a plane, the equation of which is

B: $$0.2840\ X - 0.9588\ Y + 0.0063\ Z = -2.4218$$

The distances of the atoms defining plane **B** from the plane **B** are:

B – Mo(1)	0.00 Å		**B** – O(4)	0.06 Å	
Mo(1′)	– 0.04		O(4′)	0.00	
Mo(2)	– 0.01		O(5)	0.00	
O(3)	0.02		O(5′)	– 0.02	
O(3′)	0.00				

The distances of selected atoms from the plane **B** and the angle between the line connecting two opposite atoms and the plane **B** are given in Table 8. It can be noticed that the differences between corresponding distances are less than 0.1 Å and the angles differ less than 5° from a right angle, which indicates

Table 8. Some distances of selected atoms from the plane **B** (see text) and the angle between the line $X-Y$ and the plane **B**. X and Y denote atoms on opposite sides of the plane **B**.

Atom $X-Y$	Distance **B**$-X$ (Å)	Distance **B**$-Y$ (Å)	Angle (°)
Mo(3)–Mo(4)	2.14	2.08	88.8
O(1)–O(2)	0.68	0.69	85.2
O(6)–O(7)	1.90	1.91	90.0
O(8)–O(9)	1.78	1.79	87.3
O(10)–O(13)	1.90	1.89	88.7
O(11)–O(14)	1.84	1.80	87.7
O(12)–O(15)	3.84	3.79	89.0

that the plane **B** is approximately a mirror plane. The angle between the two mirror planes (the plane **B** and the plane with the equation $Z = 4.9935$) in the complex ion is 89.9°. From the above evidence it can be concluded that the complex ion has approximately C_{2v} symmetry, which is the most probable symmetry of the free ion in solution. In most 7-coordinated peroxo complexes, *e.g.* $(NH_4)_3F[MoO(O_2)F_4]$,[16] $[Cr(O_2)_2(NH_3)_3]$,[24] $K_3[Cr(O_2)_2(CN)_3]$,[25] and $[CrO(O_2)_2(C_{12}H_8N_2)]$,[20] there is a mirror plane perpendicular to the equatorial plane. As the angle between the planes **A** and **B** is 89.9° this is the case here, too, within the limits of experimental error.

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