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John C. Gordon, Rinaldo Poli. A Tetranuclear Butterfly Cluster of Molybdenum, [PPh<sub>4</sub>]<sub>2</sub>[Mo<sub>4</sub>I<sub>11</sub>]. Acta Crystallographica Section C: Crystal Structure Communications [1968-2013], 1995, 51 (1), pp.14-18. 10.1107/S0108270194004877 . hal-03532533

**HAL Id: hal-03532533**

**<https://hal.science/hal-03532533>**

Submitted on 18 Jan 2022

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# A Tetranuclear Butterfly Cluster of Molybdenum, $[\text{PPh}_4]_2[\text{Mo}_4\text{I}_{11}]$

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*(Received 19 April 1993; accepted 4 May 1994)*

## Abstract

Bis(tetraphenylphosphonium) undeca-iodotetramolybdate,  $2[\text{PPh}_4]^+[\text{Mo}_4\text{I}_{11}]^{2-}$ , was obtained by treating  $[\text{Mo}_2(\text{MeCN})_8]^{4+}$  with  $\text{PPN}^+\text{I}^-$  in dichloromethane. The four Mo atoms of the anion adopt a 'butterfly' arrangement which can be viewed as a portion of an octahedron with two missing mutually *cis* vertices. Ten of the eleven I atoms are located at the vertices of a cube in which the ideal octahedron would be inscribed; two of them cap the two triangular faces of the  $\text{Mo}_4$  butterfly, four bridge the edges and four are terminally coordinated to the metal atoms. The eleventh I atom bridges the two 'wing-tip' metal atoms. The metal-metal distances for four edges of the butterfly wings are in the range 2.533 (3)–2.547 (3) Å, whereas the 'hinge' edge is slightly longer [2.688 (3) Å] and the separation between the two wing tips is 3.051 (3) Å.

## Comment

Octahalodimolybdate tetraanions,  $[\text{Mo}_2\text{X}_8]^{4-}$ , are known for  $X = \text{Cl}$  (Brencic & Cotton, 1969) and  $\text{Br}$  (Brencic, Dobcnik & Segedin, 1974) but have never been reported for  $X = \text{I}$ . The above complexes were prepared by interaction of the acetate complex  $\text{Mo}_2(\text{OOCCH}_3)_4$  or the sulfate complex  $(\text{NH}_4)_4\text{Mo}_2(\text{SO}_4)_4 \cdot 2\text{H}_2\text{O}$  with aqueous  $\text{HX}$  solutions. On the other hand, the analogous interaction between  $\text{Mo}_2(\text{OOCCH}_3)_4$  and  $\text{HI}$  affords, after treatment with  $[\text{nBu}_4\text{N}]\text{I}$ , the salt  $[\text{nBu}_4\text{N}]_2[\text{Mo}_4\text{I}_{11}]$  (Glicksman & Walton, 1978). There are, however, numerous dimeric complexes of the  $\text{Mo}_2\text{X}_4\text{L}_4$  type where  $X = \text{I}$  (Cotton & Walton, 1982) and the hexaiodo complexes  $[\text{Cat}]_2[\text{Mo}_2\text{I}_6(\text{H}_2\text{O})_2]$  ( $\text{Cat} = \text{pyH}^+$ ,  $\text{picH}^+$ ) are also known (Brencic & Segedin, 1978; Brenic & Golic, 1977). We attempted the synthesis of the octaiododimolybdate tetraanion by a new route, *i.e.* the interaction of the trifluoromethylsulfonate (triflate) salt of the acetonitrile complex  $[\text{Mo}_2(\text{MeCN})_8]$  (Mayer & Abbott, 1983) with tetraphenylphosphonium iodide. The product of the reaction was not a salt of the  $[\text{Mo}_2\text{I}_8]^{4-}$  anion as expected, but a new salt of the previously reported  $[\text{Mo}_4\text{I}_{11}]^{2-}$  anion. It seems therefore that the hypothetical  $[\text{Mo}_2\text{I}_8]^{4-}$  ion might be unstable since all the reactions that are expected to produce it {thermal

decomposition of  $[\text{Mo}_3(\text{CO})_4]^-$  (Stensvad, Helland, Babich, Jacobson & McCarley, 1978), treatment of  $\text{Mo}_2(\text{OOCCH}_3)_4$  with HI (Glicksman & Walton, 1978), and now reaction of  $[\text{Mo}_2(\text{MeCN})_8]^{4+}$  with  $\text{I}^-$  invariably generate the same  $[\text{Mo}_4\text{I}_{11}]^{2-}$  cluster instead.

There have been two structure determinations of this anion, one with the  $[\text{nBu}_4\text{N}]^+$  counterion (Stensvad, Helland, Babich, Jacobson & McCarley, 1978) and another with the  $[\text{AsPh}_4]^+$  counterion (Cotton & Poli, 1988), but neither was reported in full. We report here the full details of the structure of the tetraphenylphosphonium salt,  $[\text{PPh}_4]_2[\text{Mo}_4\text{I}_{11}]$ , which is not isomorphous with the previously described  $[\text{AsPh}_4]^+$  salt and does not have the twinning problems encountered in that case.

The compound crystallizes in the centrosymmetric space group  $P\bar{1}$  with both cations and the dianion in general positions. The two independent tetraphenylphosphonium cations are similar to each other. The average P—C bond lengths for the two ions are 1.78 (2) and 1.82 (3) Å. The values of the C—P—C angles are all very close to the expected tetrahedral value, being in the range 107–112° with averages of 109.7 (10) and 109.5 (20)° for the two ions. Other features of these cations are normal and do not merit further discussion.

A view of the  $[\text{Mo}_4\text{I}_{11}]^{2-}$  anion is shown in Fig. 1. The I atoms have been labeled according to the number of the Mo atom(s) to which they are bonded. For instance, I(23) is bonded to atoms Mo(2) and Mo(3), *etc.* I atoms with one-digit numbers are thus terminal ligands, those with two-digit numbers are doubly bridging and those with three-digit numbers are triply bridging. The best geometrical description of this ion is based on the geometry of the well known family of octahedral clusters  $[\text{Mo}_6\text{X}_8]^{4+}$ , as described for the tetra-*n*-butylammonium salt of this ion (Stensvad, Helland, Babich, Jacobson & McCarley, 1978). The most relevant example to the present discussion is  $\alpha\text{-MoI}_2$ , which can be described as  $[(\text{Mo}_6\text{I}_8)\text{I}_2\text{I}_{4/2}]_\infty$  (Aliev, Klinkova, Dubrovin & Atovmyan, 1981). Two adjacent Mo atoms of the octahedral core are removed leaving a 'butterfly' arrangement of the remaining four Mo atoms, Mo(3) and Mo(4) being located on the 'wing tips'. One I atom is also removed from the  $[\text{Mo}_6\text{I}_8]^{4+}$  core and a second I atom, *i.e.* I(34), is shifted to the middle of the cube edge to adopt a bridging position between the wing-tip atoms (Fig. 2). In this way, an  $[\text{Mo}_4\text{I}_7]^+$  fragment is obtained. Of the other six I atoms present in the parent  $[\text{Mo}_6\text{I}_8]^{4+}$  structure [all of which cap (triply bridge) a triangular face of the  $\text{Mo}_6$  octahedron] only two [*i.e.* I(123) and I(124)] remain in capping positions after this ideal transformation, the other four [I(13), I(14), I(23) and I(24)] becoming bridging atoms bonded to only two Mo atoms. The final structure is formally obtained by completing the coordination geometry around the four Mo atoms with terminal I atoms that are located radially outward from the center of the parent  $\text{Mo}_6$  octahedron and by removing one electron from the cluster.

The Mo(3) and Mo(4) atoms move toward each other as a result of the presence of the I(34) atom in a bridging position and thus distort the ideal arrangement shown in Fig. 2; the most evident result is that the Mo(3)—I(3) and Mo(4)—I(4) bonds are not collinear. Also, the Mo(4)—Mo(3)—I(3) and Mo(3)—Mo(4)—I(4) angles are 162.34 (9) and 157.75 (8)°, respectively, rather than 180°. The variations of the metal-metal distances are also a consequence of the distortion on going from the ideal  $O_h$   $[\text{Mo}_6\text{I}_8]^{4+}$  cluster (where, by symmetry, all distances between adjacent Mo atoms are equivalent) to the butterfly fragment. The Mo(1)—Mo(3), Mo(1)—Mo(4), Mo(2)—Mo(3) and Mo(2)—Mo(4) bonds remain chemically, although not crystallographically, equivalent (they are all bridged by one  $\mu_2\text{-I}$  and one  $\mu_3\text{-I}$  atom) and their distances

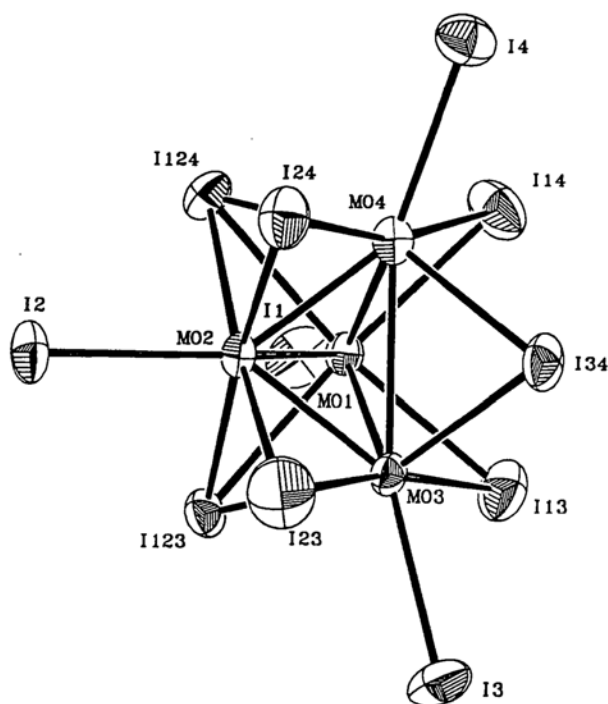


Fig. 1. Molecular configuration and atomic labelling scheme for the  $[\text{Mo}_4\text{I}_{11}]^{2-}$  ion. Ellipsoids are drawn at the 40% probability level.

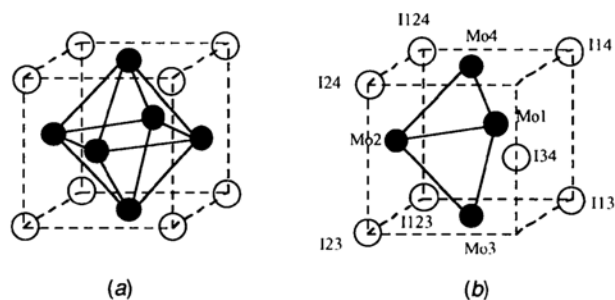


Fig. 2. Structures of (a) the  $[\text{Mo}_4\text{I}_8]^{4+}$  core and (b) the derived  $[\text{Mo}_2\text{I}_7]^+$  core.

are rather similar, *i.e.* in the range 2.533–2.547 Å with an average of 2.541 Å and a standard deviation of 0.006 Å. However, the Mo(1)—Mo(2) bond is different since it is bridged by two  $\mu_3$ -I atoms and its distance is slightly longer [2.688 (3) Å]. This latter distance is close to that reported for  $\alpha$ -MoI<sub>2</sub> (2.672 Å; Aliev, Klinkova, Dubrovin & Atovmyan, 1981) which exhibits a similar coordination environment for the Mo—Mo bonds. The Mo(3)—Mo(4) distance [3.051 (3) Å] is much shorter than the non-bonding interaction (3.779 Å) between two opposite Mo atoms in the [Mo<sub>6</sub>I<sub>8</sub>]<sup>4+</sup> cluster (Aliev, Klinkova, Dubrovin & Atovmyan, 1981). The effect of the distortion induced by the presence of the I(34) bridging atom is therefore quite substantial. Although Mo(3)—Mo(4) is considerably longer than the other Mo—Mo distances in the [Mo<sub>4</sub>I<sub>11</sub>]<sup>2-</sup> cluster, it can still be considered to represent a bonding interaction; for example, the [Mo<sub>2</sub>I<sub>7</sub>(PMe<sub>3</sub>)<sub>2</sub>]<sup>-</sup> anion has a well established Mo—Mo bonding interaction of 3.022 (1) Å (Cotton & Poli, 1987).

The Mo—I distances within sets of chemically equivalent bonds are quite similar with averages of 2.838 (9) for terminal bonds, 2.744 (29) for bridging bonds and 2.821 (46) Å for capping bonds. The Mo—( $\mu_2$ -I) bonds do not show particular trends for chemically non-equivalent distances [*i.e.* Mo(wedge)—I—Mo(wing tip), Mo(wedge)—I—Mo(wing tip) and Mo(wing tip)—I—Mo(wing tip) bonds], whereas the Mo—( $\mu_3$ -I) bonds involving the Mo(1) and Mo(2) 'wedge' atoms [average 2.793 (13) Å] are significantly shorter than those involving the Mo(3) and Mo(4) wing-tip atoms [average 2.877 (18) Å]. As expected, longer bonds are observed for the triply-bridging atoms compared to the doubly bridging atoms. However, the terminal Mo—I bonds would be expected to be shorter than the Mo—( $\mu$ -I) bonds which is contrary to what is found. The same phenomenon was observed for  $\alpha$ -MoI<sub>2</sub> and was attributed to the compression of the Mo<sub>6</sub> octahedron inside the I<sub>8</sub> cube because of strong Mo—Mo bonding (Aliev, Klinkova, Dubrovin & Atovmyan, 1981). A similar rationalization can be invoked here since Mo(1) is displaced from the least-square plane through I(13), I(14), I(123) and I(124), and Mo(2) is displaced from that through I(23), I(24), I(123) and I(124) both towards the center of the Mo<sub>4</sub> core, and a similar distortion has occurred for Mo(3) and Mo(4) (as discussed earlier). Evidence for this is provided by the *trans* angles at the Mo atoms which deviate significantly from 180° [I(13)—Mo(1)—I(124) 171.8 (1), I(14)—Mo(1)—I(123) 174.92 (9), I(23)—Mo(2)—I(124) 173.0 (1), I(24)—Mo(2)—I(123) 172.58 (8), I(13)—Mo(3)—I(23) 170.6 (1) and I(14)—Mo(4)—I(24) 165.5 (1)°]. This is consistent with the observation that in the family of compounds Mo<sub>3</sub>HI<sub>7</sub>L<sub>3</sub>, (L = thf, MeCN, PhCN) which can also be viewed as derived from a fragment of [Mo<sub>6</sub>I<sub>8</sub>]<sup>4+</sup> and where the terminal Mo—I bonds are no longer subject to Mo displacement effects, the Mo—I distances are in the expected order (capping > bridging > terminal) (Cotton & Poli, 1988).

## Experimental

### Crystal data

(C<sub>24</sub>H<sub>20</sub>P)<sub>2</sub>[Mo<sub>4</sub>I<sub>11</sub>]  
*M<sub>r</sub>* = 2458.5  
 Triclinic  
 $P\bar{1}$   
*a* = 10.474 (6) Å  
*b* = 12.862 (5) Å  
*c* = 23.882 (8) Å  
 $\alpha$  = 94.72 (3)°  
 $\beta$  = 91.80 (5)°  
 $\gamma$  = 107.98 (5)°  
*V* = 3044 (5) Å<sup>3</sup>  
*Z* = 2  
*D<sub>x</sub>* = 2.68 Mg m<sup>-3</sup>

Mo *K*α radiation  
 $\lambda$  = 0.71073 Å  
 Cell parameters from 25 reflections  
 $\theta$  = 8.5–13.5°  
 $\mu$  = 6.391 mm<sup>-1</sup>  
*T* = 293 K  
 Plate  
 0.5 × 0.4 × 0.1 mm  
 Black

### Data collection

Enraf–Nonius CAD-4 diffractometer  
 $\omega/2\theta$  scans  
 Absorption correction: empirical  
 $T_{\min}$  = 0.262,  $T_{\max}$  = 1.000  
 8368 measured reflections  
 7938 independent reflections  
 5900 observed reflections  
 [ $F_o > 6\sigma(F_o)$ ]

$R_{\text{int}}$  = 0.003  
 $\theta_{\text{max}}$  = 22.5°  
 $h$  = -11 → 11  
 $k$  = 0 → 13  
 $l$  = -25 → 25  
 3 standard reflections monitored every 97 reflections  
 intensity variation: -0.2%

### Refinement

Refinement on *F*  
*R* = 0.060  
 $wR$  = 0.078  
*S* = 2.97  
 5900 reflections  
 586 parameters  
 H-atom parameters not refined

$w = 1/\sigma^2(|F_o|)$   
 $(\Delta/\sigma)_{\text{max}}$  = 0.47  
 $\Delta\rho_{\text{max}}$  = 1.91 e Å<sup>-3</sup>  
 $\Delta\rho_{\text{min}}$  = -2.45 e Å<sup>-3</sup>  
 Atomic scattering factors from TEXSAN (Molecular Structure Corporation, 1985)

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters (Å<sup>2</sup>)

$$B_{\text{eq}} = (1/3)\sum_i \sum_j B_{ij} a_i^* a_j^* a_i \cdot a_j$$

	<i>x</i>	<i>y</i>	<i>z</i>	<i>B<sub>eq</sub></i>
I(1)	0.0085 (2)	-0.0171 (1)	0.71882 (8)	6.01 (8)
I(2)	0.6323 (1)	0.3583 (1)	0.75412 (8)	5.50 (7)
I(3)	0.1386 (2)	0.4661 (2)	0.59111 (8)	6.77 (9)
I(4)	0.1514 (2)	0.4197 (1)	0.93927 (7)	5.62 (8)
I(13)	-0.0786 (2)	0.2459 (1)	0.67591 (8)	5.81 (8)
I(14)	-0.0635 (2)	0.2088 (1)	0.82592 (8)	6.34 (8)
I(23)	0.4226 (2)	0.5420 (2)	0.7001 (1)	8.6 (1)
I(24)	0.4364 (2)	0.5199 (1)	0.84860 (8)	5.64 (7)
I(34)	0.0843 (2)	0.5189 (1)	0.77417 (8)	5.08 (7)
I(123)	0.2888 (1)	0.2269 (1)	0.65588 (6)	4.01 (6)
I(124)	0.3027 (1)	0.1958 (1)	0.82728 (7)	4.37 (6)
Mo(1)	0.1183 (2)	0.2145 (1)	0.74476 (8)	3.23 (7)
Mo(2)	0.3646 (2)	0.3636 (1)	0.75694 (8)	3.23 (7)
Mo(3)	0.1751 (2)	0.3918 (1)	0.69709 (8)	3.27 (7)
Mo(4)	0.1827 (2)	0.3701 (1)	0.82332 (9)	3.73 (7)
P(1)	0.7544 (5)	0.1533 (4)	0.0669 (3)	3.7 (2)
P(2)	0.3888 (6)	0.1828 (4)	0.4381 (3)	4.0 (2)
C(10)	0.698 (2)	0.229 (1)	0.0177 (9)	3.3 (8)

C(11)	0.580 (2)	0.251 (2)	0.023 (1)	5 (1)	Mo(1)—I(124)—Mo(2)	57.73 (7)	Mo(1)—Mo(3)—I(13)	62.37 (8)
C(12)	0.533 (2)	0.305 (2)	-0.018 (1)	6 (1)	Mo(1)—I(124)—Mo(4)	53.00 (6)	Mo(1)—Mo(3)—I(23)	124.9 (1)
C(13)	0.611 (3)	0.339 (2)	-0.062 (1)	6 (1)	Mo(2)—I(124)—Mo(4)	53.02 (6)	Mo(1)—Mo(3)—I(34)	99.67 (9)
C(14)	0.731 (3)	0.318 (2)	-0.066 (1)	5 (1)	Mo(2)—Mo(1)—Mo(3)	58.05 (8)	Mo(1)—Mo(3)—I(123)	62.06 (7)
C(15)	0.772 (2)	0.261 (2)	-0.028 (1)	5 (1)	Mo(2)—Mo(1)—Mo(4)	57.87 (8)	Mo(2)—Mo(3)—Mo(4)	52.89 (7)
C(20)	0.629 (2)	0.112 (2)	0.116 (1)	4 (1)	Mo(2)—Mo(1)—I(1)	136.75 (9)	Mo(2)—Mo(3)—I(3)	138.1 (1)
C(21)	0.543 (2)	-0.001 (1)	0.117 (1)	4 (1)	Mo(2)—Mo(1)—I(13)	120.32 (9)	Mo(2)—Mo(3)—I(13)	126.07 (9)
C(22)	0.441 (2)	-0.026 (2)	0.153 (1)	6 (1)	Mo(2)—Mo(1)—I(14)	120.5 (1)	Mo(2)—Mo(3)—I(23)	61.32 (8)
C(23)	0.422 (2)	0.054 (2)	0.191 (1)	5 (1)	Mo(2)—Mo(1)—I(123)	61.42 (7)	Mo(2)—Mo(3)—I(34)	99.63 (9)
C(24)	0.499 (2)	0.156 (2)	0.193 (1)	5 (1)	Mo(2)—Mo(1)—I(124)	60.80 (7)	Mo(2)—Mo(3)—I(123)	62.22 (7)
C(25)	0.608 (2)	0.189 (2)	0.156 (1)	5 (1)	Mo(3)—Mo(1)—Mo(4)	73.72 (8)	Mo(4)—Mo(3)—I(3)	162.34 (9)
C(30)	0.904 (2)	0.238 (1)	0.105 (1)	3.8 (9)	Mo(3)—Mo(1)—I(1)	140.6 (1)	Mo(4)—Mo(3)—I(13)	94.71 (9)
C(31)	0.952 (2)	0.199 (2)	0.152 (1)	5 (1)	Mo(3)—Mo(1)—I(13)	62.32 (8)	Mo(4)—Mo(3)—I(23)	94.6 (1)
C(32)	1.080 (2)	0.254 (2)	0.178 (1)	5 (1)	Mo(3)—Mo(1)—I(14)	112.0 (1)	Mo(4)—Mo(3)—I(34)	56.57 (6)
C(33)	1.156 (2)	0.343 (2)	0.160 (1)	5 (1)	Mo(3)—Mo(1)—I(123)	64.45 (7)	Mo(4)—Mo(3)—I(123)	101.43 (7)
C(34)	1.116 (2)	0.388 (2)	0.114 (1)	5 (1)	Mo(3)—Mo(1)—I(124)	117.80 (9)	I(3)—Mo(3)—I(13)	86.34 (9)
C(35)	0.989 (2)	0.333 (2)	0.088 (1)	5 (1)	Mo(4)—Mo(1)—I(1)	145.0 (1)	I(3)—Mo(3)—I(23)	84.4 (1)
C(40)	0.784 (2)	0.037 (1)	0.031 (1)	3.7 (9)	Mo(4)—Mo(1)—I(13)	107.7 (1)	I(3)—Mo(3)—I(34)	105.81 (8)
C(41)	0.738 (2)	0.005 (2)	-0.022 (1)	5 (1)	Mo(4)—Mo(1)—I(14)	63.10 (8)	I(3)—Mo(3)—I(123)	96.18 (8)
C(42)	0.759 (3)	-0.090 (2)	-0.050 (1)	7 (1)	Mo(4)—Mo(1)—I(123)	117.91 (9)	I(13)—Mo(3)—I(23)	170.6 (1)
C(43)	0.827 (2)	-0.149 (2)	-0.022 (1)	5 (1)	Mo(4)—Mo(1)—I(124)	65.55 (8)	I(13)—Mo(3)—I(34)	91.45 (9)
C(44)	0.875 (2)	-0.115 (2)	0.031 (1)	5 (1)	I(1)—Mo(1)—I(13)	90.35 (9)	I(13)—Mo(3)—I(123)	90.28 (8)
C(45)	0.853 (2)	-0.023 (2)	0.058 (1)	5 (1)	I(1)—Mo(1)—I(14)	90.47 (8)	I(23)—Mo(3)—I(34)	92.8 (1)
C(50)	0.504 (2)	0.269 (2)	0.491 (1)	5 (1)	I(1)—Mo(1)—I(123)	90.52 (8)	I(23)—Mo(3)—I(123)	88.88 (9)
C(51)	0.484 (2)	0.359 (2)	0.520 (1)	5 (1)	I(1)—Mo(1)—I(124)	93.29 (8)	I(34)—Mo(3)—I(123)	158.00 (8)
C(52)	0.569 (3)	0.410 (2)	0.566 (1)	7 (1)	I(13)—Mo(1)—I(14)	83.50 (8)	Mo(1)—Mo(4)—Mo(2)	64.00 (8)
C(53)	0.674 (3)	0.376 (2)	0.581 (1)	7 (1)	I(13)—Mo(1)—I(124)	171.8 (1)	Mo(1)—Mo(4)—Mo(3)	53.25 (7)
C(54)	0.700 (3)	0.292 (3)	0.552 (2)	9 (2)	I(13)—Mo(1)—I(123)	91.51 (9)	Mo(1)—Mo(4)—I(4)	142.51 (9)
C(55)	0.614 (3)	0.237 (2)	0.506 (1)	7 (1)	I(14)—Mo(1)—I(124)	89.17 (9)	Mo(1)—Mo(4)—I(14)	62.15 (8)
C(60)	0.477 (2)	0.131 (2)	0.384 (1)	4 (1)	I(14)—Mo(1)—I(123)	174.92 (9)	Mo(1)—Mo(4)—I(24)	126.4 (1)
C(61)	0.438 (2)	0.023 (2)	0.364 (1)	5 (1)	I(123)—Mo(1)—I(124)	95.75 (8)	Mo(1)—Mo(4)—I(34)	99.47 (9)
C(62)	0.508 (3)	-0.016 (2)	0.325 (1)	7 (1)	Mo(1)—Mo(2)—Mo(4)	58.12 (8)	Mo(1)—Mo(4)—I(124)	61.44 (7)
C(63)	0.619 (4)	0.051 (3)	0.306 (1)	8 (2)	Mo(1)—Mo(2)—Mo(3)	58.20 (7)	Mo(2)—Mo(4)—Mo(3)	53.20 (7)
C(64)	0.664 (3)	0.166 (3)	0.329 (2)	8 (2)	Mo(1)—Mo(2)—I(2)	136.02 (8)	Mo(2)—Mo(4)—I(4)	140.2 (1)
C(65)	0.589 (3)	0.198 (2)	0.368 (1)	6 (1)	Mo(1)—Mo(2)—I(23)	120.4 (1)	Mo(2)—Mo(4)—I(14)	125.6 (1)
C(70)	0.277 (2)	0.071 (1)	0.4719 (9)	3.1 (8)	Mo(1)—Mo(2)—I(24)	120.8 (1)	Mo(2)—Mo(4)—I(24)	62.61 (8)
C(71)	0.308 (2)	0.051 (2)	0.521 (1)	4 (1)	Mo(1)—Mo(2)—I(124)	61.47 (7)	Mo(2)—Mo(4)—I(34)	99.50 (9)
C(72)	0.218 (3)	-0.038 (2)	0.547 (1)	7 (2)	Mo(1)—Mo(2)—I(123)	61.25 (8)	Mo(2)—Mo(4)—I(124)	61.07 (7)
C(73)	0.106 (3)	-0.108 (2)	0.514 (2)	7 (2)	Mo(3)—Mo(2)—Mo(4)	73.91 (8)	Mo(3)—Mo(4)—I(4)	157.75 (8)
C(74)	0.080 (2)	-0.085 (2)	0.466 (1)	6 (1)	Mo(3)—Mo(2)—I(2)	142.9 (1)	Mo(3)—Mo(4)—I(14)	97.82 (9)
C(75)	0.162 (2)	0.006 (2)	0.442 (1)	5 (1)	Mo(3)—Mo(2)—I(23)	62.36 (9)	Mo(3)—Mo(4)—I(24)	96.49 (9)
C(80)	0.287 (2)	0.256 (2)	0.403 (1)	5 (1)	Mo(3)—Mo(2)—I(24)	109.99 (9)	Mo(3)—Mo(4)—I(34)	56.12 (6)
C(81)	0.293 (3)	0.277 (2)	0.352 (1)	6 (1)	Mo(3)—Mo(2)—I(124)	118.6 (9)	Mo(3)—Mo(4)—I(124)	100.54 (8)
C(82)	0.218 (3)	0.330 (2)	0.327 (1)	8 (2)	Mo(3)—Mo(2)—I(123)	64.43 (8)	I(4)—Mo(4)—I(14)	85.00 (9)
C(83)	0.130 (4)	0.362 (2)	0.357 (2)	8 (2)	Mo(4)—Mo(2)—I(2)	142.7 (1)	I(4)—Mo(4)—I(24)	82.67 (9)
C(84)	0.114 (3)	0.345 (2)	0.412 (2)	8 (2)	Mo(4)—Mo(2)—I(23)	108.8 (1)	I(4)—Mo(4)—I(34)	101.67 (8)
C(85)	0.196 (2)	0.288 (2)	0.437 (1)	6 (1)	Mo(4)—Mo(2)—I(24)	62.95 (8)	I(4)—Mo(4)—I(124)	101.65 (8)
					Mo(4)—Mo(2)—I(123)	117.98 (9)	I(14)—Mo(4)—I(24)	165.5 (1)
					Mo(4)—Mo(2)—I(124)	65.92 (7)	I(14)—Mo(4)—I(34)	96.18 (9)
					I(2)—Mo(2)—I(23)	91.6 (1)	I(14)—Mo(4)—I(124)	86.67 (8)
					I(2)—Mo(2)—I(24)	90.48 (9)	I(24)—Mo(4)—I(34)	93.80 (8)
					I(2)—Mo(2)—I(123)	91.82 (9)	I(24)—Mo(4)—I(124)	88.34 (8)
					I(2)—Mo(2)—I(124)	90.59 (8)	I(34)—Mo(4)—I(124)	156.7 (1)
					I(23)—Mo(2)—I(24)	82.31 (9)		

Table 2. Selected geometric parameters (Å, °)

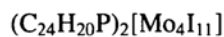
I(1)—Mo(1)	2.848 (3)	I(124)—Mo(1)	2.793 (3)
I(2)—Mo(2)	2.829 (3)	I(124)—Mo(2)	2.775 (2)
I(3)—Mo(3)	2.833 (3)	I(124)—Mo(4)	2.895 (3)
I(4)—Mo(4)	2.843 (3)	Mo(1)—Mo(2)	2.688 (3)
I(13)—Mo(1)	2.744 (3)	Mo(1)—Mo(3)	2.547 (3)
I(13)—Mo(3)	2.743 (3)	Mo(1)—Mo(4)	2.540 (3)
I(14)—Mo(1)	2.749 (3)	Mo(2)—Mo(3)	2.543 (3)
I(14)—Mo(4)	2.773 (3)	Mo(2)—Mo(4)	2.533 (3)
I(23)—Mo(2)	2.681 (3)	Mo(3)—Mo(4)	3.051 (3)
I(23)—Mo(3)	2.707 (3)	P(1)—C(10)	1.78 (2)
I(24)—Mo(2)	2.764 (3)	P(1)—C(20)	1.78 (2)
I(24)—Mo(4)	2.772 (3)	P(1)—C(30)	1.78 (2)
I(34)—Mo(3)	2.745 (3)	P(1)—C(40)	1.78 (2)
I(34)—Mo(4)	2.760 (3)	P(2)—C(50)	1.78 (2)
I(123)—Mo(1)	2.800 (3)	P(2)—C(60)	1.81 (2)
I(123)—Mo(2)	2.804 (3)	P(2)—C(70)	1.82 (2)
I(123)—Mo(3)	2.859 (2)	P(2)—C(80)	1.86 (2)
Mo(1)—I(13)—Mo(3)	55.31 (7)	I(23)—Mo(2)—I(124)	173.0 (1)
Mo(1)—I(14)—Mo(4)	54.76 (7)	I(23)—Mo(2)—I(123)	90.6 (1)
Mo(2)—I(23)—Mo(3)	56.31 (8)	I(24)—Mo(2)—I(123)	172.58 (8)
Mo(2)—I(24)—Mo(4)	54.45 (7)	I(24)—Mo(2)—I(124)	90.97 (8)
Mo(3)—I(34)—Mo(4)	67.31 (7)	I(124)—Mo(2)—I(123)	96.05 (7)
Mo(1)—I(123)—Mo(2)	57.33 (7)	Mo(1)—Mo(3)—Mo(2)	63.76 (8)
Mo(1)—I(123)—Mo(3)	53.48 (6)	Mo(1)—Mo(3)—Mo(4)	53.03 (7)
Mo(2)—I(123)—Mo(3)	53.35 (6)	Mo(1)—Mo(3)—I(3)	140.0 (1)

The largest peak and deepest hole in the final difference Fourier map and all other peaks of  $> 1.0 e \text{ \AA}^{-3}$  were closer than 1.5 Å to heavy atoms (Mo or I).

Crystallographic computing was performed with *TEXSAN* (Molecular Structure Corporation, 1985).

We gratefully acknowledge support by the NSF (CHE-9058375) and the Exxon Education Foundation. The X-ray diffractometer and MicroVAX computer system were funded in part by the NSF (CHE-84-02155).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: GR1011). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CHI 2HU, England.



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