ted interatomic distances are shown in Table 4. The pentagonal bipyramid is quite regular: the O—O distances corresponding to the edges shared with the octahedra vary from 2·420 (8) to 2·467 (8) Å and the remaining distances from 2·80 (3) to 2·88 (3) Å. On the other hand, the MO_6 octahedra are highly distorted with O—O distances between 2·420 (8) and 3·129 (8) Å. The average M—O distances of the three octahedrally coordinated cations are 1·957 (5), 1·969 (6) and 1·950 (5) Å, while that for the seven-coordinated cation is 2·041 (7) Å. These values agree closely with those found for Nb$_2$WO$_8$ (Lundberg, 1972).

The symmetry of the structural units and the mechanism by which they are connected to form the structure are in general different from compound to compound. In Ta$_2$WO$_8$ and Nb$_2$WO$_8$ they are linked by corner sharing and form layers like that shown in Fig. 1. In the third direction the layers are stacked on top of one another and each polyhedron shares a vertex with the corresponding one of the next layer. The array of corner-sharing octahedra creates large tunnels extending in the direction of the a(*Pbcm*) axis. The tunnels are surrounded by three- or four-octahedron rings. Probably these are the sites in which the Li ions are located in the structures of LiNb$_6$O$_{15}$F and the high-temperature form of LiTa$_3$O$_8$ (Santoro, Roth & Minor, 1976).
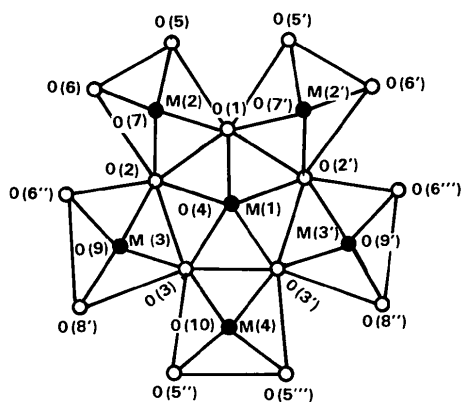

Fig. 2. Basic unit in the structure of Ta$_2$WO$_8$. A mirror perpendicular to the plane of the figure passes through the atoms O(1), M(1), O(4), M(4), and O(10).

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Dodecacarbonyltetrakis-μ_3-[(pentacarbonylrhenio)indio]-*tetrahedro*-tetrarhenium, $Re_4(CO)_{12}\{\mu_3\text{-}InRe(CO)_5\}_4$

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Abstract. In$_4$Re$_8$(CO)$_{32}$, M_r = 2845·22, tetragonal, a = 15·042 (2), b = 15·040 (2), c = 12·075 (2) Å, U = 2731·7 Å^3; Z = 2, D_c = 3·457 Mg m^{-3}; F(000) = 2488; Mo $K\alpha$ radiation, λ = 0·71069 Å, μ(Mo $K\alpha$) = 18·537 mm^{-1}. Space group $P\bar{4}2_1c$. The structure was solved by the heavy-atom method with 1586 unique reflexions (I > 3σ) and refined to a final R of 0·0435. The core of the molecule is formed by eight Re atoms and four In atoms which are bonded to each other by a total of 16 In—Re bonds [r(In—Re): 2·796 (2), 2·820 (2), 2·848 (2); 2·720 (2) Å] and four Re—Re bonds [r(Re—Re): 2·997 (1), 3·034 (1) Å]. Four Re

atoms form an inner Re tetrahedron; above each of its four planes there is one In atom bound to three Re atoms of the inner tetrahedron and to an Re atom of an outer Re tetrahedron.

Introduction. The heteronuclear metal atom cluster $Re_4(CO)_{12}\{\mu_3\text{-}InRe(CO)_5\}_4$ was obtained by a reaction of In metal with $Re_2(CO)_{10}$ (Haupt, Neumann & Preut, 1975).

Data were collected with a crystal of dimensions $0.12 \times 0.15 \times 0.24$ mm. Unit-cell parameters were determined by least-squares fits to the reflecting positions of 23 reflexions with a Hilger & Watts Y 290 automatic four-circle diffractometer with graphite-monochromatized Mo $K\alpha$ radiation and a scintillation counter. The intensities of 2907 reflexions ($I > 3\sigma$) with $2° \le \theta \le 26.3°$ were measured by the $\omega/2\theta$ scan technique, with a scan width $\Delta 2\theta = (1.34 + 0.34 \tan \theta)°$ from background to background and a scan speed of $0.02°$ s^{-1} in 2θ. Backgrounds were measured at either end of the scan range for 7 s. Five standard reflexions were measured every 60 reflexions and showed only random deviations from their mean intensities. Lorentz and polarization corrections were applied (no corrections for absorption) and after averaging of the equivalent reflexions, the data set contained 1586 independent reflexions. The structure was solved by Patterson and Fourier methods. The positions of the atoms Re and In were found from the Patterson synthesis. Full-matrix least-squares refinement with anisotropic thermal parameters yielded a conventional R ($= \sum \Delta / \sum F_o$, where $\Delta = |F_o - |F_c||$) of 0.074. At this stage a Fourier synthesis furnished positions for the C and O atoms. Further cycles of refinement (full-matrix) with anisotropic thermal parameters for In and Re and isotropic

thermal parameters for C and O led to a conventional R of 0.0435.* During the final stages of refinement, each reflexion was assigned a weight according to $w^{-1} = \sigma^2(F) + 0.005F^2$. The structure solution and refinement were carried out with the *SHELX* program system (Sheldrick, 1976). In addition *ORTEP* (Johnson, 1965) was used. Atomic scattering factors were taken from Cromer & Mann (1968) and Cromer & Liberman (1970). Positional parameters are shown in Table 1 and interatomic distances and angles in Table 2.

Discussion. The tetragonal unit cell contains two molecules, each having a fourfold inversion axis. Fig. 1 shows the structure of such a molecule with the numbering scheme of the atoms. The first digit indicates the symmetry operation (1: xyz, 2: $\bar{x}\bar{y}z$, 3: $\bar{y}x\bar{z}$, 4: $y\bar{x}\bar{z}$),

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Table 1. *Final atomic positional parameters* $(\times 10^4)$ *with estimated standard deviations in parentheses*

	x	y	z
In(1)	1555 (1)	−185 (1)	−1304 (2)
Re(1)	988 (1)	−129 (1)	899 (1)
Re(2)	2982 (1)	−436 (1)	−2652 (1)
C(11)	3932 (22)	−710 (26)	−3737 (29)
O(11)	4523 (18)	−873 (18)	−4330 (23)
C(12)	2517 (25)	529 (22)	−3558 (31)
O(12)	2302 (27)	1122 (24)	−4135 (34)
C(13)	3694 (23)	398 (22)	−1785 (29)
O(13)	4094 (20)	917 (20)	−1246 (27)
C(14)	3401 (21)	−1406 (19)	−1733 (25)
O(14)	3598 (21)	−1957 (19)	−1151 (26)
C(15)	2143 (20)	−1251 (20)	−3376 (27)
O(15)	1657 (19)	−1732 (18)	−3766 (24)
C(16)	1768 (19)	−1131 (18)	871 (33)
O(16)	2239 (19)	−1735 (18)	950 (25)
C(17)	2003 (19)	658 (21)	991 (32)
O(17)	2626 (16)	1082 (16)	1124 (23)
C(18)	954 (20)	−171 (20)	2479 (20)
O(18)	1037 (16)	−208 (15)	3431 (19)

Table 2. *Interatomic distances* (Å) *and selected interatomic angles* (°) *in* $Re_4(CO)_{12}\{\mu_3\text{-}InRe(CO)_5\}_4$

In(11)−Re(11)	2.796 (2)	Re(11)−C(16)	1.91 (3)
In(11)−Re(31)	2.820 (2)	Re(11)−C(17)	1.94 (3)
In(11)−Re(41)	2.848 (2)	Re(11)−C(18)	1.91 (3)
In(11)−Re(12)	2.720 (2)	C(11)−O(11)	1.17 (4)
Re(11)−Re(21)	2.997 (1)	C(12)−O(12)	1.18 (5)
Re(11)−Re(31)	3.034 (1)	C(13)−O(13)	1.18 (5)
Re(12)−C(11)	1.98 (3)	C(14)−O(14)	1.13 (4)
Re(12)−C(12)	1.95 (3)	C(15)−O(15)	1.13 (4)
Re(12)−C(13)	1.95 (3)	C(16)−O(16)	1.16 (4)
Re(12)−C(14)	1.94 (3)	C(17)−O(17)	1.14 (4)
Re(12)−C(15)	1.97 (3)	C(18)−O(18)	1.16 (3)

Bond angles at the atom Re(11)

Re(21)−Re(11)−Re(31)	60.4 (1)	C(16)−Re(11)−Re(41)	89.5 (10)
Re(21)−Re(11)−Re(41)	60.4 (1)	C(17)−Re(11)−Re(31)	92.2 (10)
Re(31)−Re(11)−Re(41)	59.2 (1)	C(18)−Re(11)−Re(21)	88.7 (8)
In(11)−Re(11)−In(31)	108.6 (1)	C(16)−Re(11)−In(11)	76.8 (10)
In(11)−Re(11)−In(41)	109.5 (1)	C(16)−Re(11)−In(41)	77.8 (10)
In(31)−Re(11)−In(41)	112.4 (1)	C(17)−Re(11)−In(11)	80.3 (10)
In(11)−Re(11)−Re(31)	57.7 (1)	C(17)−Re(11)−In(31)	77.3 (10)
In(11)−Re(11)−Re(41)	58.3 (1)	C(18)−Re(11)−In(31)	81.2 (8)
In(11)−Re(11)−Re(21)	107.9 (1)	C(18)−Re(11)−In(41)	77.6 (8)
In(31)−Re(11)−Re(21)	57.6 (1)	C(16)−Re(11)−C(17)	89.9 (14)
		C(16)−Re(11)−C(18)	90.5 (13)
		C(17)−Re(11)−C(18)	89.1 (13)

Bond angles at the atom In(11)

Re(11)−In(11)−Re(31)	65.4 (1)	Re(12)−In(11)−Re(11)	144.5 (1)
Re(11)−In(11)−Re(41)	65.0 (1)	Re(12)−In(11)−Re(3)	142.3 (1)
Re(31)−In(11)−Re(41)	63.8 (1)	Re(12)−In(11)−Re(41)	138.3 (1)

Bond angles at the atom Re(12)

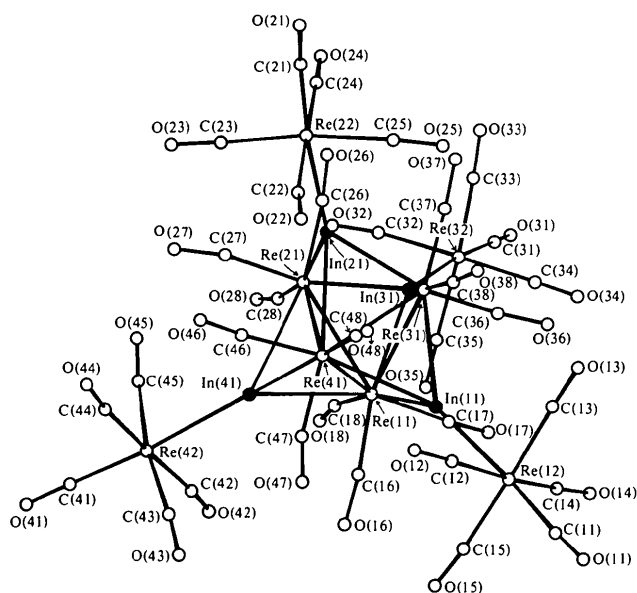|			
In(11)−Re(12)−C(11)	173.3 (10)	C(14)−Re(12)−C(15)	89.7 (13)
In(11)−Re(12)−C(12)	87.1 (11)	C(15)−Re(12)−C(12)	89.1 (14)
In(11)−Re(12)−C(13)	91.3 (10)	C(12)−Re(12)−C(11)	92.4 (15)
In(11)−Re(12)−C(14)	91.0 (9)	C(13)−Re(12)−C(11)	95.3 (14)
In(11)−Re(12)−C(15)	81.1 (9)	C(14)−Re(12)−C(11)	89.3 (14)
C(12)−Re(12)−C(13)	91.1 (15)	C(15)−Re(12)−C(11)	92.3 (14)
C(13)−Re(12)−C(14)	89.9 (13)		

Fig. 1. Drawing of the molecule Re$_4$(CO)$_{12}${μ_3-InRe(CO)$_5$}$_4$ with the numbering scheme of the atoms.

Fig. 2. Stereoscopic view of the molecule Re$_4$(CO)$_{12}${μ_3-InRe-(CO)$_5$}$_4$.

while the second digit is a consecutive number for the element concerned. The core of the molecule is formed by a metal cluster, consisting of an inner Re tetrahedron [Re(11), Re(21), Re(31) and Re(41)] with the two non-equivalent distances r(Re—Re) of 2·997 (1) and 3·034 (1) Å. Above each of the four planes of this inner Re tetrahedron there is an In atom that is 2·796 (2), 2·820 (2) and 2·848 (2) Å distant from the three neighbouring Re atoms of the inner tetrahedron. To each of these four In atoms, which form a nearly ideal tetrahedron, is bound an outer Re atom with a bond distance of r(In—Re) = 2·720 (2) Å. As a result there is a metal cluster, which consists of three dovetailed tetrahedra. A stereoview of the molecule is shown in Fig. 2. The $\bar{4}$ axis runs through the midpoint of the Re(11)—Re(21) and the Re(31)—Re(41) bonds. The Re atoms of the inner tetrahedron have coordination number nine. To each of these atoms are bonded three Re atoms, three In atoms and three C atoms. The two non-equivalent Re—Re bond distances [2·997 (1),
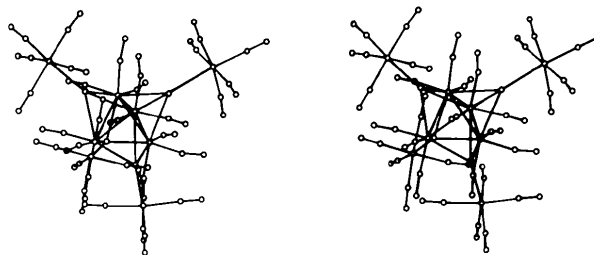
3·034 (1) Å] are significantly shorter than the distance in Re$_2$(CO)$_8${μ-InRe(CO)$_5$}$_2$ [3·232 (1) Å] (Preut & Haupt, 1975) and the three non-equivalent In—Re bond distances [2·796 (2), 2·848 (2), 2·820 (2) Å] have greater values than those in the inner metal ring of Re$_2$(CO)$_8${μ-InRe(CO)$_5$}$_2$ [2·754 (1), 2·807 (1) Å].

The three C atoms are bonded to the Re atom in such a way as to be situated in the plane that contains the neighbouring edge of the inner tetrahedron and its centre. To the In atom are bonded four Re atoms with bond distances significantly different from each other. The three bonds to the inner Re tetrahedron however are each distinctly greater than the bond to the fourth outer Re atom.

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