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[Bis-μ-iodo-bis(tricarbonylrhenio)-(*Re*—*Re*)][μ-iodo-bis(tetracarbonylrhenio)-(*Re*—*Re*)]indium(III) and Bis(pentacarbonylrhenio)(tetracarbonyl-bis-μ-iodo-rhenio)indium(III)

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Abstract. $[\{(CO)_3Re(\mu\text{-}I)_2Re(CO)_3\}In\{(CO)_4Re(\mu\text{-}I)Re(CO)_4\}]$, $M_r = 1632{\cdot}5$, monoclinic, $C2/c$, $a = 16{\cdot}176\,(3)$, $b = 11{\cdot}798\,(3)$, $c = 16{\cdot}147\,(4)$ Å, $\beta = 115{\cdot}36\,(1)°$, $U = 2785{\cdot}1$ Å^3, $Z = 4$, $D_x = 3{\cdot}892$ Mg m^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, μ(Mo $K\alpha$) = 21·72 mm^{-1}, $F(000) = 2816$, $T = 298\,(1)$ K, $R = 0{\cdot}0416$ for 1829 unique reflections. The spiro μ_4-In atom is bound to the four Re atoms of two bidentate chelate ligands $[Re_2(CO)_8(\mu\text{-}I)]^-$ and $[Re_2(CO)_6(\mu\text{-}I)_2]^{2-}$ each with a Re—Re bond. The mean values of the metal—metal bond lengths are In—Re = 2·801 (1) and 3·073 (2) Å. $[(CO)_4Re(\mu\text{-}I)_2In\{Re(CO)_5\}_2]$, $M_r = 1319{\cdot}4$, triclinic, $P\bar{1}$, $a = 7{\cdot}202\,(1)$, $b = 11{\cdot}821\,(3)$, $c = 15{\cdot}664\,(4)$ Å, $\alpha = 87{\cdot}92\,(2)$, $\beta = 83{\cdot}17\,(3)$, $\gamma = 78{\cdot}25\,(1)°$, $U = 1296{\cdot}2$ Å^3, $Z = 2$, $D_x = 3{\cdot}379$ Mg m^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, μ(Mo $K\alpha$) = 17·42 mm^{-1}, $F(000) = 1152$, $T = 298\,(1)$ K, $R = 0{\cdot}0419$ for 3416 unique reflections. The molecular structure shows as the central fragment a folded four-membered $Re(\mu\text{-}I)_2In$ ring with a dihedral angle of 23·7°. The mean values of the ring bond parameters are Re—I = 2·826 (1), In—I = 3·025 (1) Å, and bond angles subtended at (μ-I) 94·1 (1), In 79·8 (1) and Re 86·7 (1)°.

Introduction. In the course of our investigations on bonding capabilities between transition-metal carbonyl fragments (M') and main-group metals (M) in cluster compounds (Haupt & Flörke, 1988) both title compounds were synthesized and each crystal structure determined. Of special interest in this family of heterometallic compounds are the stereochemistry and other properties of those compounds with a so-called naked M atom (Herrmann, 1986). The first title compound shows a spirocyclic μ_4-In atom bound to four Re atoms of the different bidentate chelate ligands $[Re_2(CO)_8(\mu\text{-}I)]^-$ and $[Re_2(CO)_6(\mu\text{-}I)]^{2-}$. It is the first structurally determined sample with a spiro μ_4 third main-group metal attached to four transition-metal atoms. The structural analysis of the other title substance continues an earlier characterization of halogeno bridging bonds in similar compounds of the type $\{In[M'(CO)_5]_2\}_2(\mu\text{-}X)_2$ (M' = Mn, Re) (Haupt, Wolfes & Preut, 1976, 1979).

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Experimental. Red crystals of $(\mu_4\text{-In})Re_4(CO)_{14}(\mu\text{-I})_3$, (I), and yellow crystals of $In(\mu\text{-I})_2Re(CO)_4[Re[CO]_5]_2$, (II), were prepared by reaction of InI_3 and $Re_2(CO)_{10}$ in pressure-balanced Schlenk tubes at 473 K (Balsaa, 1987). Crystal size of (I) 0·10 × 0·10 × 0·13 mm, Nicolet diffractometer, graphite-monochromated Mo $K\alpha$ radiation, $\lambda = 0·71073$ Å, lattice parameters refined from 20 reflections $5 \le 2\theta \le 25°$, ω–2θ-scan, 4044 reflections recorded $2 \le 2\theta \le 45°$, $0 \le h \le 17$, $0 \le k \le 12$, $-17 \le l \le 17$, three standards recorded every 400 reflections, only random deviations, Lp correction, empirical absorption correction *via* φ scans, min./max. transmission factors 0·117/0·183, after merging ($R_{\text{int}} = 0·024$) 1829 unique reflections of which 511 were considered unobserved $[I \le 2\sigma(I)]$; structure solved by interpretation of Patterson map and subsequent ΔF maps, full-matrix least-squares refinement based on F (94 parameters); Re, In and I refined anisotropically, max. $(\Delta/\sigma) = 0·001$, max. height in final ΔF syntheses 1·3 e Å^{-3} near heavy-atom position, refinement converged to $R = 0·0416$, $wR = 0·0469$, $w = 1/[\sigma^2(F) + 0·00447F^2]$. Complex neutral-atom scattering factors corrected for anomalous dispersion (*International Tables for X-ray Crystallography,* 1974), all calculations with *SHELXTL-PLUS* (Nicolet XRD Corporation, 1988).

Crystal size of (II) 0·04 × 0·05 × 0·22 mm, lattice parameters refined from 25 reflections $5 \le 2\theta \le 25°$, ω–2θ-scan, 3566 reflections $2 \le 2\theta \le 45°$, $-7 \le h \le 7$, $-12 \le k \le 12$, $0 \le l \le 16$, three standard reflections every 400, only random deviations, corrections as above, min./max. transmission factors 0·125/0·241, after merging ($R_{\text{int}} = 0·018$) 3416 unique reflections, 801 considered unobserved $[I \le 2\sigma(I)]$, structure solution as above, full-matrix least-squares refinement on F (167 parameters); Re, In and I refined anisotropically, max. $(\Delta/\sigma) < 0·0005$, max. height in final ΔF map 0·9 e Å^{-3} near heavy atom, refinement converged to $R = 0·0419$, $wR = 0·0407$, $w = 1/[\sigma^2(F) + 0·00058F^2]$; further details of the structural analysis as above.

Discussion. The structures of the title compounds are shown in Figs. 1 and 2. Atomic positional parameters are given in Tables 1 and 2,* bond lengths and angles in Table 3.

Each molecule of the spiro compound, in the crystal, has a C_2 axis as the unique symmetry element along the direction of the I(1)—In(1) atomic positions in accordance with C_2 point group symmetry. For example, the higher C_{2v} symmetry of its $InRe_4I_3$ core is prevented by a dihedral angle of 83·9° between the intersecting Re(1)In(1)Re(1*a*) and Re(2)In(1)Re(2*a*) planes. This deviation from an orthogonal plane intersection is not caused by intramolecular repulsive forces between the 14 CO ligands. Actually, when viewed along each Re—Re bond direction the equivalent CO ligands are eclipsed and the present values of C···O and O···O [O···O ≥ 3·26 (3) Å] contact lengths do not indicate such forces. Its origin, therefore, could be seen in connection with stronger interactions between symmetry related molecules [O···O 2·96 (4) Å] including packing forces (−99 kJ mol^{-1} on the basis of nonbonding intermolecular C···C, C···O, O···O, I···C, I···O and I···I interactions) (Haupt, Flörke & Preut, 1986).

The $InRe_4I_3$ nucleus contains two Re—Re bonds, which account for the diamagnetism and give each of the Re atoms the Rn closed-shell electron configuration. Each of the atoms of the Re(1)—Re(1*a*) bond is part of a pair of edge-shared coordination octahedra with a tetravalent In atom and a bridging

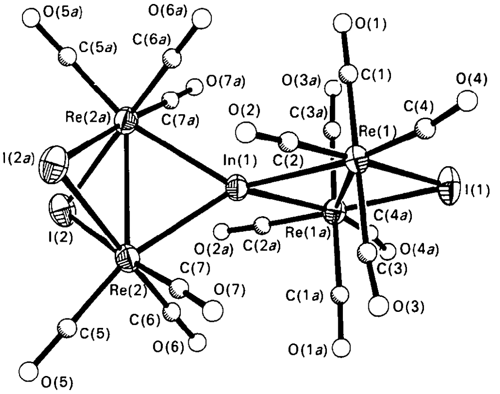

Fig. 1. General view of a molecule of $[Re_2(CO)_8(\mu\text{-I})][Re_2(CO)_6(\mu\text{-I})_2](\mu_4\text{-In})$.

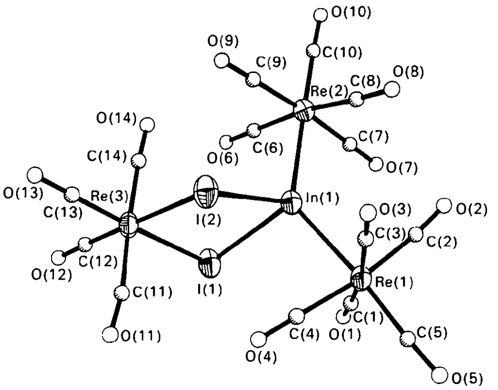

Fig. 2. General view of a molecule of $Re(CO)_4(\mu\text{-I})_2In[Re(CO)_5]_2$.

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* Lists of structure factors, anisotropic temperature factors and a complete list of bond lengths and angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51962 (25 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. *Atomic coordinates* (× 10⁴) *and equivalent isotropic displacement parameters* (Å² × 10³) *for* $C_{14}I_3InO_{14}Re_4$

Equivalent isotropic U defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U_{eq}
Re(1)	4655 (1)	2991 (1)	3276 (1)	33 (1)
Re(2)	5999 (1)	−977 (1)	3131 (1)	33 (1)
In(1)	5000	1009 (2)	2500	31 (1)
I(1)	5000	4915 (2)	2500	50 (2)
I(2)	5283 (1)	−2200 (2)	1481 (1)	44 (1)
C(1)	3405 (20)	2969 (25)	2275 (20)	52 (7)
O(1)	2634 (16)	2932 (18)	1720 (15)	67 (6)
C(2)	4454 (16)	1687 (21)	3871 (16)	34 (6)
O(2)	4363 (14)	927 (17)	4271 (14)	60 (5)
C(3)	5943 (17)	3050 (22)	4227 (17)	39 (6)
O(3)	6640 (15)	3150 (18)	4853 (15)	65 (6)
C(4)	4309 (18)	3910 (23)	4063 (18)	46 (7)
O(4)	4004 (17)	4421 (21)	4491 (17)	80 (7)
C(5)	6960 (18)	−2046 (23)	3719 (18)	43 (6)
O(5)	7549 (17)	−2708 (21)	4072 (17)	74 (6)
C(6)	6814 (20)	−169 (27)	2792 (20)	54 (7)
O(6)	7310 (17)	328 (21)	2595 (17)	81 (7)
C(7)	6396 (16)	−124 (22)	4213 (16)	36 (6)
O(7)	6669 (15)	388 (18)	4919 (15)	67 (6)

Table 2. *Atomic coordinates* (× 10⁴) *and equivalent isotropic displacement parameters* (Å² × 10³) *for* $C_{14}I_2InO_{14}Re_3$

Equivalent isotropic U defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U_{eq}
Re(1)	2237 (1)	2099 (1)	4553 (1)	43 (1)
Re(2)	2256 (1)	1577 (1)	1308 (1)	41 (1)
Re(3)	3630 (1)	6053 (1)	2045 (1)	42 (1)
I(1)	396 (2)	5244 (1)	2831 (1)	49 (1)
I(2)	5817 (2)	3914 (1)	2529 (1)	50 (1)
In(1)	2463 (2)	2731 (1)	2815 (1)	41 (1)
C(1)	−452 (29)	2783 (18)	4551 (13)	56 (5)
O(1)	−2039 (25)	3217 (15)	4527 (10)	83 (5)
C(2)	1628 (27)	651 (17)	4219 (12)	50 (5)
O(2)	1374 (24)	−196 (15)	3953 (11)	85 (5)
C(3)	4970 (32)	1438 (19)	4336 (13)	61 (6)
O(3)	6590 (28)	1048 (16)	4196 (11)	97 (5)
C(4)	2863 (29)	3626 (19)	4711 (13)	57 (5)
O(4)	3188 (25)	4510 (16)	4810 (11)	91 (5)
C(5)	2006 (32)	1803 (20)	5791 (15)	69 (6)
O(5)	1842 (24)	1559 (15)	6522 (11)	87 (5)
C(6)	614 (30)	2981 (19)	901 (13)	58 (5)
O(6)	−367 (25)	3805 (16)	668 (11)	87 (5)
C(7)	−56 (28)	1124 (17)	1894 (12)	53 (5)
O(7)	−1417 (24)	851 (14)	2266 (10)	82 (5)
C(8)	3825 (32)	317 (20)	1871 (14)	66 (6)
O(8)	4790 (25)	−508 (16)	2177 (11)	93 (5)
C(9)	4541 (29)	2116 (17)	790 (12)	53 (5)
O(9)	5901 (22)	2425 (13)	496 (9)	75 (4)
C(10)	2235 (30)	745 (19)	303 (14)	63 (6)
O(10)	2164 (25)	197 (16)	−315 (12)	94 (5)
C(11)	3888 (29)	6685 (18)	3148 (13)	59 (5)
O(11)	4244 (26)	7153 (16)	3755 (12)	95 (5)
C(12)	2036 (30)	7431 (19)	1720 (13)	58 (5)
O(12)	1102 (26)	8318 (16)	1546 (11)	92 (5)
C(13)	5784 (30)	6553 (18)	1493 (13)	56 (5)
O(13)	7091 (25)	6864 (15)	1148 (11)	86 (5)
C(14)	3299 (26)	5360 (16)	967 (12)	45 (4)
O(14)	3091 (21)	4891 (14)	353 (10)	74 (4)

Table 3. *Selected bond lengths* (Å) *and angles* (°)

$C_{14}I_3InO_{14}Re_4$			
Re(1)—Re(1*a*)	3·152 (1)	Re(1)—In(1)	2·820 (2)
Re(1)—I(1)	2·763 (2)	Re(2)—Re(2*a*)	2·993 (1)
Re(2)—In(1)	2·780 (2)	Re(2)—I(2)	2·807 (2)
In(1)—Re(1)—I(1)	111·3 (1)	In(1)—Re(2)—I(2)	98·4 (1)
Re(1)—In(1)—Re(1*a*)	68·0 (1)	Re(1)—In(1)—Re(2)	136·9 (1)
Re(1)—In(1)—Re(2)	131·9 (1)	Re(2)—In(1)—Re(2*a*)	65·1 (1)
Re(1)—I(1)—Re(1*a*)	69·6 (1)	Re(2)—I(2)—Re(2*a*)	64·4 (1)
$C_{14}I_2InO_{14}Re_3$			
Re(1)—In(1)	2·791 (1)	Re(2)—In(1)	2·802 (2)
Re(3)—I(1)	2·828 (2)	Re(3)—I(2)	2·824 (2)
I(1)—In(1)	3·039 (2)	I(2)—In(1)	3·011 (2)
I(1)—Re(3)—I(2)	86·7 (1)	Re(3)—I(1)—In(1)	93·7 (1)
Re(3)—I(2)—In(1)	94·4 (1)	Re(1)—In(1)—Re(2)	133·5 (1)
Re(1)—In(1)—I(1)	103·4 (1)	Re(1)—In(1)—I(2)	104·7 (1)
Re(2)—In(1)—I(1)	113·6 (1)	Re(2)—In(1)—I(2)	108·6 (1)
I(1)—In(1)—I(2)	79·8 (1)		

I atom as edge atoms, and the homonuclear metal–metal bond corresponds to the normal to this common edge. Each of the Re atoms in the Re(2)—Re(2*a*) bond occupies the interstices of a pair of octahedra sharing a face with a tetravalent In atom and two I atoms at the corners. The Re—Re bond corresponds to the normal to this plane. The two types of octahedra pairs become cross-linked by the fourfold bridging indium(III) atom on formation of the spiro compound.

Of special interest are the bridging angles subtended at the I atoms and the μ_4-In atom to characterize the aforementioned two types of coordination octahedra pairs. The bond angle subtended at both I bridge atoms (2 and 2*a*) is 6·6 (1)° smaller than the value of 70·5° for regular octahedra sharing a face. The Re(2)—In—Re(2*a*) bridging angle, 65·1 (1)°, is closer to this value. In the case of the Re octahedra sharing an edge, the analogous Re(1)—X—Re(1*a*) (X = In, I) bond angles are reduced by about 22·0 (1) and 20·4 (1)° from the expected 90° for a regular octahedra pair.

Compared with the covalent Re—Re single bond in $Re_2(CO)_{10}$ (Churchill, Amoh & Wassermann, 1981) containing a pair of octahedra sharing a vertex, the Re(2)—Re(2*a*) bond length is reduced about 0·048 (1) Å and the Re(1)—Re(1*a*) bond is increased about 0·11 (1) Å. The shortening can be explained by a reinforcement of the metal—metal bond favoured by a triple bridge with two I atoms and an indium(III) atom. The increased Re—Re bond length points out a diminished support by the same μ_4-In and one more I bridge atom. However, the Re—Re bond length in the edge-shared octahedra pair still shows a shortening of 0·070 (1) Å compared with that of the corresponding polyhedra pair in $Re_2(CO)_8[\mu\text{-}InRe(CO)_5]_2$ (Preut & Haupt, 1975). The In—Re(2) bond length is shorter than the In—Re(1), possibly due to a stronger stabilizing chelate effect going from the $\{Re_2(CO)_8(\mu\text{-}I)\}^-$ to $\{Re_2(CO)_6(\mu\text{-}I)_2\}^{2-}$ group. The mean value of the In—Re covalent single-bond length is 2·801 (1) Å, in accordance with such bond-length values as 2·781 (1) Å in $Re_2(CO)_8[\mu\text{-}InRe(CO)_5]_2$ and 2·821 (2) Å in $Re_4(CO)_{12}[\mu_3\text{-}InRe(CO)_5]_4$ (Preut & Haupt, 1979).

The unsymmetrical four-membered $Re(3)I_2In$ ring in (II) is part of an edge-shared polyhedra pair with two bridging I atoms and consists of an octahedrally distorted polyhedron at the transition metal and a tetrahedrally distorted polyhedron at the main-group metal atom. The Re(3) central atom coordinated with six ligands (four CO and two μ-I) fulfills the Rn closed-shell electron configuration and the In atom with four ligands [two univalent $Re(CO)_5$ and two μ-I] the Xe closed-shell electron configuration in accordance with the diamagnetism of the compound. The unsymmetrical four-membered ring is folded with a dihedral angle of 23·7° between the planes of $Re(3)I_2$ and InI_2, whereas such rings in compounds of the types $InI_4(\mu\text{-}I)_2$ (Forrester & Zalkin, 1964), $In_2[M(CO)_5]_4(\mu\text{-}I)_2$ (M = Mn, Re) or $Re_2(CO)_8(\mu\text{-}I)_2$ (Darst, Lenhert, Lukehardt & Warfield, 1980) have planar or nearly planar shapes. It seems therefore possible that the change results from molecular packing requirements, because the shortest non-bonding O···O contact lengths are intermolecular [≤ 2·84 (2) Å]. In the sequence from $In_2[Re(CO)_5]_4(\mu\text{-}I)_2$ to the present compound it is recognizable that the subtended terminal bond angle at the In atom with the two univalent $Re(CO)_5$ groups is enlarged from 126·3 (1) to 133·5 (1)° and the ring bond angle subtended at the μ-I atom (or In atom) diminishes by about 2·4 (1)° [or 3·7 (1)°].

Intramolecular distances in both substances do not indicate interactions greater than van der Waals forces.

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5-η-Benzene-9-hydroxy-9,10-μ-triethylphosphinoaurio-5-ruthena-*nido*-decaborane(11) (Dichloromethane Solvate), $C_{12}H_{33}AuB_9OPRu$ ($0{\cdot}15CH_2Cl_2$)

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Abstract. (Crystallographic data assuming no solvate) M_r = 620·7, triclinic, $P\bar{1}$, a = 9·268 (3), b = 11·084 (5), c = 11·788 (3) Å, α = 87·87 (3), β = 86·803 (25), γ = 76·92 (3)°, V = 1177·3 (19) Å^3, Z = 2, D_x = 1·751 Mg m^{-3}, Mo $K\alpha$, λ = 0·71069 Å, μ = 6·91 mm^{-1}, $F(000)$ = 594, T = 291 K, R = 0·0578 for 3611 independent observed reflections. An r.m.s. misfit calculation shows that the cage of the title compound has a *nido* decaboranyl architecture. There is some evidence for a weak interaction between the 9,10-bridging Au atom and the 3c-2e Ru(5)—H(5,6)—B(6) unit.

Introduction. The reaction between $B_{10}H_{14}$ and $MeAuPR_3$ affords either the unique 'triple cluster' $[(B_{10}H_{12}Au)(AuPR_3)_4(AuB_{10}H_{12})]$ (1) [(1*a*) R = Et, (1*b*) R = Ph] (Wynd, Robins, Welch & Welch, 1985; Wynd, Welch & Parish, 1989), or the simple isolobally-substituted complex $[\mu\text{-}5,6\text{-}AuPR_3\text{-}B_{10}H_{13}]$ (2) [(2*a*) R = Cy, (2*b*) R = *o*-tol] (Wynd, McLennan, Reed & Welch, 1987). We are attempting to expand this chemistry to metal-containing analogues of $B_{10}H_{14}$. Several such compounds are known (Kennedy, 1986) including all four possible isomers of $[(\eta\text{-}C_6Me_6)RuB_9H_{13}]$ (Bown, Greenwood & Kennedy, 1986). Starting from a 5- or 6-metalla-B_9H_{13} species, reaction with $MeAuPR_3$ allows the possibility of the synthesis of clusters with heteronuclear metal—metal bonds. To minimize the possibility of steric control of the reaction we have synthesized $[5\text{-}(\eta\text{-}C_6H_6)\text{-}5\text{-}RuB_9H_{13}]$ (3) by an analogous route to that which afforded the appropriate C_6Me_6 species,

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