title compound. These molecules are ordered in pairs of enantiomers, between which weak hydrogen bonding (Berkovitch-Yellin & Leiserowitz, 1984) is observed: $N(1)\cdots H(1)$ $(\frac{1}{2}-x, \frac{1}{2}-y, \frac{1}{2}-z) = 2\cdot 59$ (2) and $O(1)\cdots$ $H(61)$ $(\frac{1}{2}-x, \frac{1}{2}-y, \frac{1}{2}-z) = 2\cdot 57$ (2) Å [sum of the van der Waals radii is $2\cdot 75$ and $2\cdot 72$ Å, respectively (Bondi, 1964)]. The angles $N(1)\cdots H(1)-O(1)$ and $O(1)\cdots$ $H(61)-C(6)$ are 132 (2) and 150 (2)°, respectively.

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Hexacarbonyl-tris(μ-diphenylphosphido)-bis(μ₃-hydrido)-*triangulo*-trirhenium

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Abstract. $[Re_3(CO)_6(\mu_3\text{-H})_2\{P(C_6H_5)_2\}_3].0\cdot 72CCl_4$, $M_r = 1395\cdot 0$, triclinic, $P\bar{1}$, $a = 11\cdot 058$ (2), $b = 12\cdot 505$ (3), $c = 18\cdot 122$ (6) Å, $\alpha = 99\cdot 77$ (2), $\beta = 90\cdot 13$ (2), $\gamma = 109\cdot 66$ (2)°, $U = 2321\cdot 0$ Å³, $Z = 2$, $D_x = 1\cdot 995$ Mg m⁻³, $\lambda(\text{Mo } K\alpha) = 0\cdot 71073$ Å, $\mu = 80\cdot 2$ cm⁻¹, $F(000) = 1310\cdot 6$, $T = 298$ (1) K, final $R = 0\cdot 0541$ for 3918 unique reflections. The central fragment of the molecule consists of a triangular Re ring edge-bridged by diphenylphosphido groups and triply bridged by two hydrides. The Re—Re bonds $[r(\text{Re—Re}) = 2\cdot 730$ (1) Å] indicate a multiple-bond character.

Introduction. The family of triangular M_3 cluster compounds (M = transition metal) reaches a saturated valence electron configuration with 48 electrons (CVE) in the presence of π acceptor ligands (Lauher, 1978). Numerous compounds of this and three with CVE number 46, including the recently described complex anion $[Re_3(CO)_9\{P(C_6H_5)_3\}(\mu\text{-H})_4]^-$ (Beringhelli, D'Alfonso, Freni, Ciani, Sironi & Molinari, 1986), are structurally known. The neutral title compound with different bridging of the Re_3 core is the first example with an unsaturated 44 CVE number. Its molecular determination is necessary for a molecular-orbital description to characterize the multiple Re—Re bond supported by bridging ligands.

A further important aspect of this structural determination is connected with our attempt to find the positional parameters of the two triply-bridged hydrides – earlier proved by ¹H NMR measurements – because the title substance illustrates a profitable hydrogen-bonding pattern for an unsaturated triangular M_3 metal atom arrangement at the surface of metals in heterogeneous catalytic hydrogenation reactions.

Experimental. The orange crystals were prepared by reaction of $[Re_2(CO)_8\{P(C_6H_5)_3\}_2]$ with hydrogen at 493 K (Haupt, Flörke & Balsaa, 1988). Crystal size $0\cdot 06 \times 0\cdot 08 \times 0\cdot 25$ mm, Nicolet $R3m/V$ diffractometer, ω–2θ scan, scan speed $1\cdot 5$–$9\cdot 7°$ min⁻¹, graphite-monochromated Mo $K\alpha$; lattice parameters refined

from 25 reflections, $5 \le 2\theta \le 19°$; three standard reflections recorded every 400 reflections: only random deviations, 6485 reflections measured, $2 \le 2\theta \le 45°$, $0 \le h \le 11$, $-13 \le k \le 13$, $-19 \le l \le 19$, Lp correction, absorption correction *via* ψ scans, min./max. transmission $0\cdot41/0\cdot48$, after merging $(R_{int} = 0\cdot030)$ 6103 unique reflections, 2185 reflections considered unobserved, $I < 4\sigma(I)$; structure solution by direct methods and ΔF synthesis, full-matrix least-squares refinement on F with anisotropic temperature factors for Re and P, 194 parameters refined, phenyl groups treated as rigid bodies (C—C $1\cdot395$, C—H $0\cdot98$ Å), phenyl H atoms geometrically fixed, $R = 0\cdot0541$, unit weights, $(\Delta/\sigma)_{max} < 0\cdot001$, max. height in final ΔF map $0\cdot9$ e Å^{-3} near heavy-atom position, site occupation factor for enclosed CCl_4 refined to $0\cdot72$; complex neutral-atom scattering factors corrected for anomalous dispersion (Cromer & Waber, 1974); program *SHELXTL-PLUS* (Nicolet, 1987).

Discussion. The structure of the title compound is shown in Fig. 1. Atomic parameters are given in Table 1 and bond lengths and angles in Table 2.*

The rhenium cluster compound crystallizes with two molecules in the triclinic system. The central molecular fragment of this diamagnetic substance contains a triangular arrangement of the Re atoms which is edge-bridged by the diphenylphosphido groups and capped by two triply bridging hydrides. The non-crystallographic local geometry of the six non-metal atoms at one Re central atom is of idealized C_{2v}

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* Lists of structure factors, anisotropic thermal parameters, H-atom parameters and a complete list of bond lengths and angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44374 (18 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.
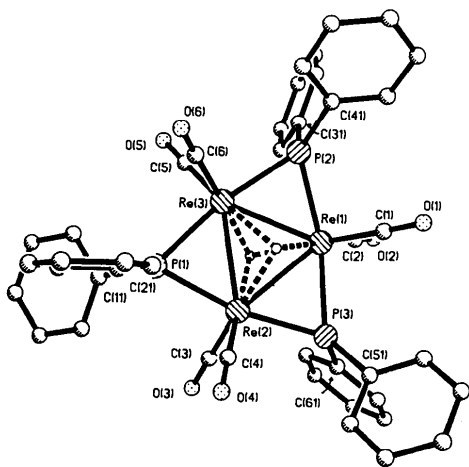

Fig. 1. General view of the molecule with atom numbering.

Table 1. *Atomic coordinates* $(\times 10^4)$ *and equivalent isotropic thermal parameters* $(\text{Å}^2 \times 10^3)$

	x	y	z	U_{eq}*
Re(1)	648 (1)	2738 (1)	2201 (1)	57 (1)
Re(2)	1092 (1)	4747 (1)	3201 (1)	54 (1)
Re(3)	1015 (1)	4748 (1)	1693 (1)	58 (1)
P(1)	1409 (6)	6394 (5)	2640 (4)	61 (4)
P(2)	592 (7)	2917 (5)	919 (3)	66 (4)
P(3)	726 (6)	2911 (5)	3526 (3)	61 (4)
C(1)	1729 (28)	1851 (25)	2063 (16)	77 (8)
O(1)	2439 (22)	1342 (20)	1963 (13)	105 (7)
C(2)	−778 (30)	1463 (26)	2079 (16)	80 (8)
O(2)	−1803 (22)	649 (19)	2008 (12)	101 (7)
C(3)	−41 (28)	5111 (24)	3884 (16)	75 (8)
O(3)	−781 (22)	5330 (19)	4330 (13)	108 (7)
C(4)	2434 (31)	5490 (27)	3853 (18)	86 (9)
O(4)	3369 (26)	5987 (22)	4300 (15)	128 (9)
C(5)	−100 (29)	5061 (24)	1094 (16)	75 (8)
O(5)	−895 (22)	5217 (19)	718 (13)	101 (7)
C(6)	2337 (30)	5570 (26)	1157 (17)	81 (8)
O(6)	3306 (24)	6076 (20)	890 (13)	114 (8)
C(11)	344 (15)	7258 (13)	2791 (9)	45 (5)
C(12)	120 (15)	7692 (13)	3520 (9)	84 (9)
C(13)	−601 (15)	8424 (13)	3636 (9)	98 (10)
C(14)	−1100 (15)	8722 (13)	3025 (9)	93 (10)
C(15)	−876 (15)	8288 (13)	2297 (9)	98 (10)
C(16)	−155 (15)	7556 (13)	2180 (9)	85 (9)
C(21)	3011 (14)	7532 (14)	2731 (10)	59 (6)
C(22)	4083 (14)	7180 (14)	2720 (10)	80 (8)
C(23)	5315 (14)	7990 (14)	2732 (10)	112 (12)
C(24)	5475 (14)	9150 (14)	2755 (10)	106 (11)
C(25)	4403 (14)	9502 (14)	2767 (10)	94 (10)
C(26)	3171 (14)	8692 (14)	2755 (10)	91 (9)
C(31)	−951 (14)	2267 (15)	364 (10)	67 (7)
C(32)	−1009 (14)	1726 (15)	−381 (10)	80 (8)
C(33)	−2189 (14)	1238 (15)	−797 (10)	110 (11)
C(34)	−3311 (14)	1291 (15)	−467 (10)	92 (9)
C(35)	−3253 (14)	1832 (15)	278 (10)	82 (9)
C(36)	−2073 (14)	2320 (15)	694 (10)	76 (8)
C(41)	1719 (15)	2542 (15)	289 (9)	64 (7)
C(42)	1719 (15)	1411 (15)	188 (9)	77 (8)
C(43)	2572 (15)	1081 (15)	−287 (9)	81 (8)
C(44)	3424 (15)	1881 (15)	−661 (9)	91 (9)
C(45)	3423 (15)	3012 (15)	−560 (9)	76 (8)
C(46)	2570 (15)	3342 (15)	−85 (9)	81 (8)
C(51)	1959 (14)	2594 (14)	4045 (9)	53 (6)
C(52)	2284 (14)	1615 (14)	3790 (9)	81 (8)
C(53)	3205 (14)	1378 (14)	4198 (9)	73 (8)
C(54)	3800 (14)	2120 (14)	4861 (9)	88 (9)
C(55)	3475 (14)	3098 (14)	5116 (9)	108 (11)
C(56)	2554 (14)	3335 (14)	4708 (9)	77 (8)
C(61)	−761 (14)	2200 (14)	3959 (9)	48 (6)
C(62)	−800 (14)	1414 (14)	4428 (9)	91 (9)
C(63)	−1946 (14)	867 (14)	4741 (9)	113 (12)
C(64)	−3054 (14)	1106 (14)	4583 (9)	105 (11)
C(65)	−3016 (14)	1892 (14)	4114 (9)	80 (8)
C(66)	−1870 (14)	2439 (14)	3802 (9)	82 (8)
H(1)	2214	4196	2366	
H(2)	−538	3818	2376	
Cl(1)	5048	4137	3146	
Cl(2)	5931	2898	2343	
Cl(3)	6900	4994	2494	
Cl(4)	5043	4104	1612	
C(0)	5663	4084	2360	

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

symmetry and reaches D_{3h} symmetry in the $[Re_3(\mu_3\text{-H})_2(\mu\text{-P})_3(CO)_6]$ core. These symmetries are lost by the presence of six phenyl groups in various positions.

The multiple-bond character in the 44 CVE title compound is indicated by the average Re—Re bond length $[2\cdot730 (1) \text{ Å}]$ which differs from that of the related cluster compound $[Re_3(CO)_9\{\mu\text{-P}(C_6H_5)_2\}_3]$ [ave. Re—Re $2\cdot914 (4)$ Å] possessing 48 CVE (Balsaa, 1987). This comparison shows that the corresponding value of the unsaturated three-membered Re ring is

Table 2. *Selected bond lengths* (Å) *and angles* (°) *with e.s.d.'s in parentheses*

Re(1)—Re(2)	2·731 (1)	Re(2)—P(1)	2·371 (6)
Re(1)—Re(3)	2·726 (1)	Re(2)—P(3)	2·370 (6)
Re(2)—Re(3)	2·734 (1)	Re(3)—P(1)	2·359 (6)
Re(1)—P(2)	2·375 (6)	Re (3)—P(2)	2·369 (6)
Re(1)—P(3)	2·372 (6)		

Mean values

Re—H*	2·15	O—C	1·205 (34)
Re—C	1·836 (33)		

Re(3)—Re(1)—Re(2)	60·1 (1)	P(1)—Re(3)—Re(2)	54·9 (2)
Re(2)—Re(3)—Re(1)	60·0 (1)	P(2)—Re(3)—Re(2)	115·0 (2)
Re(3)—Re(2)—Re(1)	59·9 (1)	C(2)—Re(1)—C(1)	92·0 (13)
P(2)—Re(1)—Re(3)	54·8 (2)	C(4)—Re(2)—C(3)	91·7 (13)
P(3)—Re(1)—Re(3)	114·9 (2)	C(6)—Re(3)—C(5)	87·6 (13)
P(3)—Re(1)—Re(2)	54·8 (2)	Re(3)—P(1)—Re(2)	70·6 (2)
P(2)—Re(1)—Re(2)	115·0 (2)	Re(3)—P(2)—Re(1)	70·2 (2)
P(1)—Re(2)—Re(3)	54·5 (2)	Re(2)—P(3)—Re(1)	70·3 (2)
P(3)—Re(2)—Re(3)	114·7 (2)	P(3)—Re(2)—P(1)	169·2 (2)
P(3)—Re(2)—Re(1)	54·9 (2)	P(3)—Re(1)—P(2)	169·7 (2)
P(1)—Re(2)—Re(1)	114·3 (2)	P(2)—Re(3)—P(1)	169·9 (2)
P(2)—Re(3)—Re(1)	55·0 (2)	Re—H—Re	79*
P(1)—Re(3)—Re(1)	114·9 (2)		

* See text.

0·184 (4) Å shorter. This decided bond shortening is accompanied by a small decrease of the acute Re—P—Re bond angle by 4·1 (1)° and of the average Re—P bond length by a value of about 0·06 (3) Å. The Re—Re bond shortening in the title substance has similarities to the behaviour of such bond lengths in the formal double bond in [Re$_2$H$_2$(CO)$_8$] [2·896 (3) Å] (Bennett, Graham, Hoyano & Hutcheon, 1972), the unsaturated triangular Re$_3$ core in [Re$_3$(CO)$_8$-{P(C$_6$H$_5$)$_3$}(μ-H)$_4$]$^-$ [2·797 (1), 3·193 (1) and 3·234 (1) Å] and the unsupported bond in [Re$_2$(CO)$_{10}$] [3·104 (1) Å] (Churchill, Amoh & Wassermann, 1981) with values of 0·166 (3), 0·067 (1) and 0·374 (1) Å. All

these facts confirm the shortened Re—Re bond in the title compound and agree therefore with an assumed π-bond property for the metal—metal bonds in the Re$_3$ ring.

The positional parameters of the two hydrides [H(1) and H(2)] could be located from a ΔF map and fit well with the expected values for two H atoms positioned *trans* to the CO ligands. This is in accordance with the known extremely marked *trans* influence of a hydride. The average values of the Re—(μ$_3$-H) bond length and the Re—(μ-H)—Re bond angle are 2·15 Å and 79°. Refinement of the hydride positions yielded no improvement. The estimated Re—(μ$_3$-H) bond length is elongated relative to that of Re—(μ-H) ascertained by neutron diffraction analysis of [Re$_2$H$_8${P(C$_2$H$_5$)$_2$-(C$_6$H$_5$)}$_4$] [ave. 1·878 (7) Å] (Bau, Carroll, Teller & Koetzle, 1977).

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Structure of Lead(II) Disaccharinate Monohydrate*

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Abstract. [Pb(C$_7$H$_4$NO$_3$S)$_2$].H$_2$O, $M_r = 589.56$, monoclinic, $P2_1/c$, $a = 16·037$ (3), $b = 13·356$ (5), $c = 8·132$ (3) Å, $\beta = 93·04$ (2)°, $V = 1739·34$ Å^3, $Z = 4$, $D_m = 2·14$, $D_x = 2·251$ Mg m^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, $\mu = 9·58$ mm^{-1}, $F(000) = 1112$, room temperature, final $R = 0·050$ for 2718 observed unique

* Saccharin is 1,2-benzisothiazol-3(2*H*)-one 1,1-dioxide.