McPhail, Garner & Mabbs, 1976). The complex $[Cu(imH)_4(O_2CCH_2OCH_3)_2]$ has a *trans* pair of imidazole rings with a torsional angle of 26° and another pair of imidazoles with a torsional angle of $-26°$ (Prout, Allison & Rossotti, 1976).

An unusual feature of this tetrakis(imidazole)-copper(II) complex is the unequal bond lengths for the two iodide ligands. The lack of improper rotations in the point symmetry signifies chirality and the absolute configuration of the chiral complex has been determined using the η parameter (Rogers, 1981) for the selected crystal.

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Structure of Bis[*cis*-1,2-bis(diphenylphosphino)ethylene]dichlororhenium(II) Hexane Solvate

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Abstract. $[ReCl_2(C_{26}H_{22}P_2)_2].C_6H_{14}$ (I), $M_r = 1136.11$, monoclinic, $P2_1/c$, $a = 11.426\ (2)$, $b = 13.078\ (2)$, $c = 17.323\ (3)$ Å, $\beta = 95.27\ (1)°$, $V = 2577.7\ (6)$ Å^3, $Z = 2$, $D_x = 1.46$ g cm^{-3}, $\lambda(\text{Mo}\ K\alpha) = 0.71073$ Å, $\mu = 26.5$ cm^{-1}, $F(000) = 1150$, $T = 296$ K, $R = 0.034$ for 3269 $[F_o^2 > 3\sigma(F_o^2)]$ reflections. The Re atom in (I) is located on an inversion point, fixed at the origin. One Cl and a complete tertiary phosphine ligand ($Ph_2PCHCHPPh_2$) are situated around the Re atom so as to generate a pseudo-octahedral geometry with a P1—Re—P2 angle of 79.07 (5)° and P1—Re—P2′ angle of 100.93 (5)°. The Re—Cl, Re—P1 and Re—P2 distances are 2.432 (2), 2.401 (2) and 2.398 (2) Å, respectively. Bond dis-

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Table 1. *Positional and isotropic or equivalent isotropic thermal parameters* (Å^2)

$$B_{eq} = 1/3[a^2a^{*2}B_{11} + b^2b^{*2}B_{22} + c^2c^{*2}B_{33} + 2ab(\cos\gamma)a^*b^*B_{12} + 2ac(\cos\beta)a^*c^*B_{13} + 2bc(\cos\alpha)b^*c^*B_{23}].$$

	x	y	z	B_{iso}/B_{eq}
Re	0.000	0.000	0.000	2.470 (5)
Cl	−0.1761 (2)	0.0598 (1)	−0.05490 (9)	4.10 (3)
P1	−0.0108 (1)	0.1044 (1)	−0.11446 (8)	3.11 (3)
P2	−0.1117 (1)	−0.1085 (1)	−0.09089 (8)	2.98 (3)
C11	−0.0307 (6)	0.0187 (5)	−0.1980 (3)	3.6 (1)
C21	−0.0771 (6)	−0.0725 (5)	−0.1878 (3)	3.6 (1)
C121	0.1193 (6)	0.1791 (5)	−0.1342 (3)	3.6 (1)
C122	0.2070 (6)	0.1389 (6)	−0.1739 (4)	4.8 (2)
C123	0.3066 (7)	0.1960 (7)	−0.1850 (4)	6.3 (2)
C124	0.3158 (7)	0.2946 (7)	−0.1558 (5)	6.5 (2)
C125	0.2290 (7)	0.3353 (6)	−0.1154 (4)	5.5 (2)
C126	0.1303 (6)	0.2776 (5)	−0.1048 (4)	4.5 (2)
C131	−0.1284 (6)	0.1954 (5)	−0.1465 (3)	3.6 (1)
C132	−0.1223 (7)	0.2536 (6)	−0.2140 (4)	5.1 (2)
C133	−0.2127 (7)	0.3142 (7)	−0.2412 (4)	6.3 (2)
C134	−0.3109 (8)	0.3222 (7)	−0.2031 (5)	6.8 (2)
C135	−0.3205 (8)	0.2659 (8)	−0.1360 (5)	7.5 (2)
C136	−0.2284 (7)	0.2016 (6)	−0.1084 (4)	5.2 (2)
C221	−0.2713 (5)	−0.0957 (5)	−0.0987 (3)	3.4 (1)
C222	−0.3317 (6)	−0.0453 (6)	−0.1602 (4)	4.7 (2)
C223	−0.4522 (7)	−0.0324 (7)	−0.1617 (5)	6.2 (2)
C224	−0.5123 (7)	−0.0697 (7)	−0.1025 (5)	6.8 (2)
C225	−0.4536 (7)	−0.1172 (7)	−0.0422 (4)	6.2 (2)
C226	−0.3355 (7)	−0.1293 (6)	−0.0395 (4)	5.1 (2)
C231	−0.0917 (6)	−0.2469 (5)	−0.0934 (3)	3.5 (1)
C232	−0.1816 (7)	−0.3126 (6)	−0.1164 (4)	5.5 (2)
C233	−0.1614 (9)	−0.4191 (6)	−0.1181 (5)	6.7 (2)
C234	−0.0531 (9)	−0.4561 (7)	−0.0985 (4)	6.4 (2)
C235	0.0377 (8)	−0.3914 (6)	−0.0771 (5)	6.3 (2)
C236	0.0184 (7)	−0.2883 (5)	−0.0746 (4)	5.0 (2)
C1	0.541 (2)	0.048 (2)	0.519 (1)	23.5 (6)†
C2	0.519 (2)	0.093 (2)	0.595 (1)	23.5†
C3	0.605 (2)	0.143 (2)	0.636 (1)	23.5†

† Refined isotropically.

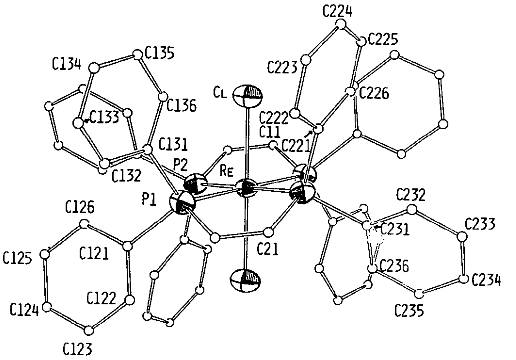

Fig. 1. An *ORTEP* drawing (Johnson, 1965) of [ReCl$_2$(Ph$_2$PCHCHPPh$_2$)$_2$] without the hexane molecule of solvation. Thermal ellipsoids have been drawn at the 50% probability level. Symmetry generated atoms are not labeled. The C atoms are shown as arbitrarily sized uniform circles.

tances and angles for the phenyl rings range from 1.34 (1) to 1.41 (1) Å and 117.4 (6) to 122.0 (7)° with averages of 1.38 (1) Å and 120.0 (1)°, respectively.

Experimental. Complex (I) was produced as a by-product in a reaction designed to form [ReCl(H$_2$)(Ph$_2$PCHCHPPh$_2$)$_2$] (II). This consisted of the reduction of ReCl$_5$ using four equivalents of Na, in tetrahydrofuran, in the presence of the phosphine ligand (Ph$_2$PCHCHPPh$_2$). Clearly, not enough reductant was added. Crystals of complex (I) were obtained when a benzene solution of a mixture of complexes (I) and (II) was layered with hexane. The approximate crystal dimensions were 0.20 × 0.20 × 0.20 mm. The crystal was mounted on a glass fiber using epoxy resin; Enraf–Nonius CAD-4 diffractometer, cell constants derived from least-squares refinement based on 21 setting-angle reflections ($23.22 \leq 2\theta \leq 38.1°$). Intensity data were collected using ω–2θ scans with a scan width of $(1.00 + 0.35\tan\theta)°$ in the range $0 \leq 2\theta \leq 50°$ ($h = 0$ to 13, $k = 0$ to 15, $l = -20$ to 20) using graphite-monochromated Mo $K\alpha$ radiation. Three standard reflections ($22\bar{8}$, $44\bar{4}$, 360) measured every 166 min indicated a modest decay of 3.6%. 4997 data were corrected for Lorentz and polarization effects, and

Table 2. *Bond distances* (Å) *and angles* (°) *with e.s.d.'s in parentheses*

Re—Cl	2.432 (2)	C131—C136	1.37 (1)
Re—P1	2.401 (2)	C132—C133	1.35 (1)
Re—P2	2.398 (2)	C133—C134	1.36 (1)
P1—C11	1.828 (6)	C134—C135	1.39 (1)
P1—C121	1.838 (7)	C135—C136	1.40 (1)
P1—C131	1.842 (6)	C221—C222	1.382 (9)
P2—C21	1.821 (6)	C221—C226	1.39 (1)
P2—C221	1.824 (6)	C222—C223	1.39 (1)
P2—C231	1.826 (6)	C223—C224	1.37 (1)
C11—C21	1.324 (9)	C224—C225	1.34 (1)
C121—C122	1.37 (1)	C225—C226	1.36 (1)
C121—C126	1.39 (1)	C231—C232	1.37 (1)
C122—C123	1.39 (1)	C231—C236	1.38 (1)
C123—C124	1.39 (1)	C232—C233	1.41 (1)
C124—C125	1.37 (1)	C233—C234	1.34 (1)
C125—C126	1.38 (1)	C234—C235	1.36 (1)
C131—C132	1.403 (9)	C235—C236	1.37 (1)
C1—C1′	1.66 (4)	C2—C3	1.33 (3)
C1—C2	1.48 (3)		

Cl—Re—P1	99.13 (6)	C124—C125—C126	119.5 (7)
Cl—Re—P1′	80.87 (6)	C121—C126—C125	120.3 (7)
Cl—Re—P2	92.15 (5)	P1—C131—C132	120.7 (5)
Cl—Re—P2′	87.85 (5)	P1—C131—C136	120.7 (5)
P1—Re—P2	79.07 (5)	C132—C131—C136	118.4 (6)
P1—Re—P2′	100.93 (5)	C131—C132—C133	120.9 (7)
Re—P1—C11	107.3 (2)	C132—C133—C134	121.0 (7)
Re—P1—C121	118.4 (2)	C133—C134—C135	119.9 (8)
Re—P1—C131	126.6 (2)	C134—C135—C136	119.4 (8)
C11—P1—C121	102.9 (3)	C131—C136—C135	120.3 (7)
C11—P1—C131	97.1 (3)	P2—C21—C11	118.6 (4)
C121—P1—C131	100.4 (3)	P2—C221—C222	121.7 (5)
Re—P2—C21	107.8 (2)	P2—C221—C226	120.6 (5)
Re—P2—C221	117.5 (2)	C222—C221—C226	117.4 (6)
Re—P2—C231	122.8 (2)	C221—C222—C223	119.9 (7)
C21—P2—C221	102.0 (3)	C222—C223—C224	120.4 (7)
C21—P2—C231	101.3 (3)	C223—C224—C225	119.8 (7)
C221—P2—C231	102.4 (3)	C224—C225—C226	120.4 (8)
P1—C11—C21	118.0 (5)	C221—C226—C225	122.0 (7)
P1—C121—C122	122.0 (5)	P2—C231—C232	122.5 (5)
P1—C121—C126	118.2 (5)	P2—C231—C236	119.8 (5)
C122—C121—C126	119.7 (6)	C232—C231—C236	117.7 (6)
C121—C122—C123	120.6 (7)	C231—C232—C233	120.3 (8)
C122—C123—C124	119.1 (8)	C232—C233—C234	119.9 (8)
C123—C124—C125	120.8 (8)	C233—C234—C235	120.3 (8)
C234—C235—C236	120.0 (8)	C1′—C1—C2	122. (2)
C231—C236—C235	121.7 (7)	C1—C2—C3	120. (2)

for absorption effects based on ψ scans using the empirical method of North, Phillips & Mathews (1968); $T_{\min} = 0.6332$, $T_{\max} = 0.9988$. 3269 unique observed data $[F_o^2 > 3\sigma(F_o^2)]$ were obtained. Scattering factors, including anomalous-dispersion factors, were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV). All computations were carried out using the *MolEN* (Fair, 1990) package of programs on a VAX computer. From the systematic absences of $h0l$: $l \neq 2n$, $0k0$: $k \neq 2n$ and from subsequent least-squares refinement, the space group was determined to be $P2_1/c$. The structure was solved by the heavy-atom method based on the assumption that an Re atom must be located at the origin. The rest of the structure was revealed in successive cycles of least-squares refinement and difference Fourier maps. Three peaks located near an inversion point clearly defined a hexane molecule. All of the non-H atoms, except those belonging to the C_6H_{14} molecule, were refined with anisotropic thermal parameters. The isotropic thermal parameters for two C atoms on the hexane molecule (C2 and C3) were constrained to be the same as the third (C1) which was refined without constraint. H atoms were not included in the refinement. $wR = \{[\sum w(|F_o| - |F_c|)^2]/[\sum w(F_o)^2]\}^{1/2}$ was minimized, where $w = \sigma^2(|F_o|^2)^{-1}$ and σ is the e.s.d. in a single measurement; final $R = 0.034$, $wR = 0.051$, $S = 1.88$ for 278 variables. The largest shift/e.s.d. in the final least-squares cycle was 0.02; the maximum and minimum residual electron density in the ΔF map were 0.92 (located near Re) and -0.23 e Å^{-3}, respectively. An *ORTEP* (Johnson, 1965) representation of the molecule (without the hexane entity) and the atomic labeling scheme is depicted in Fig. 1. Final positional and equivalent isotropic thermal parameters are listed in Table 1,* with important bond distances and angles in Table 2.

Related literature. The synthesis, by a different route, and the crystal structure of $[ReCl_2(Ph_2PCHCHPPh_2)_2]$ (without the hexane molecule of solvation) has been reported (Bakir, Fanwick & Walton, 1987).

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* Lists of structure factors, anisotropic thermal parameters, H-atom parameters and a stereoscopic view of the unit-cell packing have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71073 (43 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: HH1045]

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Structure of Tetraphenylphosphonium μ-{[η^4-2,4-Bis(2-hydroxy-2-methylpropanamido)-2,4-dimethyl-3-oxopentane]cobalt(III)}tetranitratocerium(III) Dichloromethane

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Abstract. Bis(tetraphenylphosphonium) μ-{N,N'-(2,4-dimethyl-3-oxo-2,4-pentanediyl)bis[(2-hydroxy-1:2κ^2O)-2-methylpropanamido-2κN]}-tetranitrato-1κ^4O,1κ^4O'-cerate(III)cobaltate(III) dichloromethane, $[P(C_6H_5)_4]_2[Co(C_{15}H_{24}N_2O_5)\{Ce(NO_3)_4\}].CH_2Cl_2$, $M_r = 1523.2$, monoclinic, $P2_1/c$ (C_{2h}^5), $a = 22.168$ (5), $b = 13.266$ (2), $c = 24.330$ (6) Å, $\beta = 105.17$ (2)°, $V = 6905$ (3) Å^3, $Z = 4$, $D_x = 1.465$ Mg m^{-3}, λ(Mo $K\alpha$) = 0.71073 Å, $\mu = 1.09$ mm^{-1}, $T = 293$ K, $F(000) = 3108$, $R = 0.036$, $wR = 0.045$ for 8166 independent reflections having 2θ(Mo $K\alpha$) < 50.7° and $I > 3\sigma(I)$. The title compound is a dinuclear complex in which a planar cobalt(III) complex is linked *via* secondary coordination to a cerium(III) center. The Ce$^{\rm III}$ atom has four bidentate nitrato ligands and is further bridged to the

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