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

Pt—P(1)	2·07 (1)	C(11)—C(16)	1·55 (5)
Pt—P(1ⁱ)	2·10 (1)	C(12)—C(13)	1·53 (5)
Pt—Ce(1)	2·14 (1)	C(13)—C(14)	1·54 (5)
Pt—Ce(2)	2·10 (1)	C(14)—C(15)	1·54 (5)
P(1)—C(1)	1·86 (8)	C(15)—C(16)	1·54 (5)
P(1)—C(11)	1·81 (4)	C(21)—C(22)	1·55 (5)
P(1)—C(21)	1·83 (3)	C(21)—C(26)	1·56 (5)
P(1ⁱ)—C(3)	1·98 (7)	C(22)—C(23)	1·53 (5)
Ce(1)—Ce(2)	1·39 (20)	C(23)—C(24)	1·54 (6)
C(1)—C(2)	1·55 (13)	C(24)—C(25)	1·55 (6)
C(2)—C(3)	1·55 (12)	C(25)—C(26)	1·55 (5)
C(11)—C(12)	1·56 (5)		

P(1)—Pt—P(1ⁱ)	113·0 (4)	P(1ⁱ)—C(3)—C(2)	132 (5)
P(1)—Pt—Ce(1)	106 (4)	P(1)—C(11)—C(12)	110 (2)
P(1)—Pt—Ce(2)	144 (4)	P(1)—C(11)—C(16)	112 (3)
P(1ⁱ)—Pt—Ce(1)	141 (4)	C(12)—C(11)—C(16)	104 (3)
P(1ⁱ)—Pt—Ce(2)	102 (4)	C(11)—C(12)—C(13)	114 (3)
Ce(1)—Pt—Ce(2)	38 (5)	C(12)—C(13)—C(14)	110 (3)
Pt—P(1)—C(1)	112 (3)	C(13)—C(14)—C(15)	110 (3)
Pt—P(1)—C(11)	121 (1)	C(14)—C(15)—C(16)	110 (3)
Pt—P(1)—C(21)	121 (1)	C(11)—C(16)—C(15)	115 (3)
C(1)—P(1)—C(11)	102 (3)	P(1)—C(21)—C(22)	114 (2)
C(1)—P(1)—C(21)	95 (3)	P(1)—C(21)—C(26)	110 (2)
C(11)—P(1)—C(21)	101 (2)	C(22)—C(21)—C(26)	108 (3)
Pt—P(1ⁱ)—C(3)	106 (2)	C(21)—C(22)—C(23)	112 (3)
Pt—Ce(1)—Ce(2)	70 (8)	C(22)—C(23)—C(24)	109 (3)
Pt—Ce(2)—Ce(1)	72 (8)	C(23)—C(24)—C(25)	110 (3)
P(1)—C(1)—C(2)	130 (6)	C(24)—C(25)—C(26)	110 (3)
C(1)—C(2)—C(3)	108 (7)	C(21)—C(26)—C(25)	109 (3)

Symmetry code: (i) $1 - x$, $-y$, $-z$.

The structure is defined by the coordinates and dimensions given in Tables 1 and 2, and Fig. 1.*

* Lists of structure factors, anisotropic thermal parameters and mean-plane calculations have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43566 (18 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Fig. 1. View of the disordered $[Pt(C_2H_4)(C_{27}H_{50}P_2)]$ complex. Ellipsoids are at the 20% level. The asterisk is the inversion centre at (¼00). The atoms shown by small spheres and connected by line bonds are the sites of the inversion related Pt, C_2H_4 and $-(CH_2)_3-$ moieties.

Related literature. The reactivity of the complex described here is discussed by Clark & Hampden-Smith (1986). For details of the related dichloro[1,3-bis-(diphenylphosphino)propane]palladium(II) see Steffen & Palenik (1976).

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Redetermination of the Structure of Tetrakis(acetylacetonato)zirconium(IV)

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Abstract. $[Zr(C_5H_7O_2)_4]$, $M_r = 487·7$, monoclinic, $C2/c$, $a = 21·662$ (2), $b = 8·360$ (1), $c = 14·107$ (1) Å, $\beta = 116·708$ (6)°, $V = 2282·1$ Å³, $Z = 4$, $D_x = 1·419$ Mg m⁻³, $\lambda(Mo\ K\alpha) = 0·71073$ Å, $\mu = 0·51$ mm⁻¹, $F(000) = 1008$, $T = 293$ K, $R = 0·020$ for 1940 unique reflections with $F > 4\sigma(F)$. Zr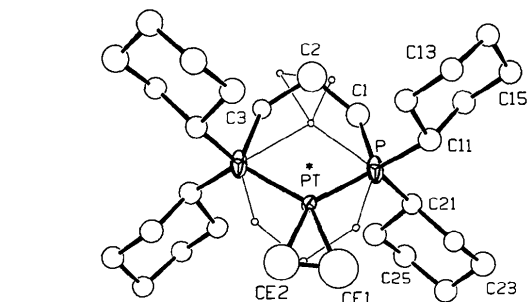, on a crystallographic twofold axis, is coordinated by four chelating ligands. The coordination polyhedron is approximately a square antiprism, with a small distortion towards dodecahedral geometry. The results confirm those of the earlier determination [Silverton & Hoard (1963). *Inorg. Chem.* **2,** 243–249]. Estimated standard deviations of final atomic coordinates and of molecular-geometry parameters are reduced in the

redetermination by factors of about five. Even with the greater precision achieved, the close agreement of chemically equivalent geometrical parameters within the chelate ligands is maintained. The greater variation of bonds and angles in the immediate coordination sphere of Zr (marginally significant in the original determination) is essentially reproduced here, but with correspondingly greater statistical significance, indicating that the susceptibility of coordination geometry to intermolecular forces is greater than that of ligand internal geometry.

Experimental. Crystal size $0.29 \times 0.46 \times 0.54$ mm, Siemens AED2 diffractometer, cell parameters from 2θ values of 32 reflections measured at $\pm\omega$ ($22 < 2\theta < 25°$). 7722 intensities measured in ω/θ scan mode, scan width $= 1.02°$ + α-doublet separation, scan time = 14–70 s, $2\theta_{max} = 50°$, limits for $h \pm25$, $k \pm9$, $l \pm16$ (whole sphere of data), no significant variation for three standard reflections, semi-empirical absorption correction, transmission 0.83–0.88, 2008 unique reflections ($R_{int} = 0.016$), 1950 with $F > 4\sigma(F)$. Structure determination by Patterson and difference syntheses, blocked-cascade refinement on F, $w^{-1} = \sigma^2(F) + 0.00002F^2$, anisotropic thermal parameters for all non-H atoms, H atoms constrained [C–H = 0.96 Å, $U(H) = 1.2U_{eq}(C)$, twofold torsional disorder of methyl groups modelled by rigid hexagons of H atoms, H–C–H = 109.5° within each methyl group, methyl H site occupation factors refined to 0.69:0.31 (2), 0.66:0.34 (2), 0.58:0.42 (2), 0.63:0.37 (2)], isotropic extinction parameter $x = 9$ (1) $\times 10^{-7}$ $[F_c' = F_c/(1 + xF_c^2/\sin2\theta)^{1/4}]$, 10 reflections omitted because of severe extinction effects, $R = 0.020$, $wR = 0.024$, max. $\Delta/\sigma = 0.039$, mean $= 0.002$, max. $\Delta\rho = 0.23$, min. $= -0.25$ e Å^{-3}, slope of normal probability plot $= 1.88$. Scattering factors from *International Tables for X-ray Crystallography* (1974), *SHELXTL* (Sheldrick, 1985) programs. The structure and atom-labelling scheme are shown in Fig. 1. Atomic coordinates are listed in Table 1,* and bond lengths and angles in Table 2.

Related literature. Zr(acac)$_4$ is a classic example of approximate square-antiprismatic eight-coordination in a M(bidentate ligand)$_4$ complex, but the original structure determintion (Silverton & Hoard, 1963) is of low precision by today's standards. Deviations from ideal square-antiprism geometry, observed here, are discussed by Hoard & Silverton (1963) and by Kepert (1978).

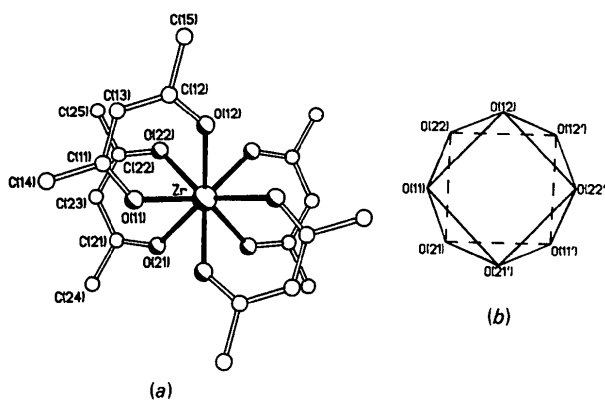Fig. 1. (a) The structure of Zr(acac)$_4$, without H atoms, showing the atom-labelling scheme; (b) the square-antiprism coordination polyhedron.

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

$$U_{eq} = \tfrac{1}{3}(\text{trace of the orthogonalized } U_{ij} \text{ matrix}).$$

	x	y	z	U_{eq}
Zr	5000	655 (1)	2500	321 (1)
O(11)	4487 (1)	1550 (1)	880 (1)	411 (4)
O(12)	4917 (1)	−1418 (1)	1505 (1)	421 (5)
C(11)	4102 (1)	908 (2)	−12 (1)	395 (6)
C(12)	4488 (1)	−1803 (2)	566 (1)	414 (6)
C(13)	4055 (1)	−724 (2)	−185 (1)	478 (7)
C(14)	3707 (1)	2028 (2)	−908 (1)	541 (8)
C(15)	4482 (1)	−3523 (2)	271 (2)	609 (9)
O(21)	4416 (1)	2743 (1)	2608 (1)	467 (5)
O(22)	3949 (1)	−221 (1)	2029 (1)	420 (4)
C(21)	3789 (1)	3145 (2)	2105 (1)	497 (7)
C(22)	3360 (1)	444 (2)	1577 (1)	471 (7)
C(23)	3256 (1)	2073 (2)	1550 (1)	567 (8)
C(24)	3630 (1)	4882 (3)	2156 (2)	734 (10)
C(25)	2754 (1)	−675 (3)	1073 (2)	707 (10)

Table 2. *Bond lengths* (Å) *and angles* (°)

Zr–O(11)	2·176 (1)	Zr–O(12)	2·186 (1)
Zr–O(21)	2·201 (1)	Zr–O(22)	2·190 (1)
O(11)–C(11)	1·274 (2)	O(12)–C(12)	1·270 (2)
C(11)–C(13)	1·381 (2)	C(11)–C(14)	1·495 (2)
C(12)–C(13)	1·387 (2)	C(12)–C(15)	1·495 (3)
O(21)–C(21)	1·262 (2)	O(22)–C(22)	1·270 (2)
C(21)–C(23)	1·391 (2)	C(21)–C(24)	1·501 (3)
C(22)–C(23)	1·378 (3)	C(22)–C(25)	1·505 (3)
O(11)–Zr–O(12)	75·1 (1)	O(11)–Zr–O(21)	76·2 (1)
O(12)–Zr–O(21)	142·1 (1)	O(11)–Zr–O(22)	80·0 (1)
O(12)–Zr–O(22)	76·5 (1)	O(21)–Zr–O(22)	74·6 (1)
O(11)–Zr–O(11')	139·8 (1)	O(12)–Zr–O(11')	143·4 (1)
O(21)–Zr–O(11')	72·1 (1)	O(22)–Zr–O(11')	113·8 (1)
O(12)–Zr–O(12')	75·1 (1)	O(21)–Zr–O(12')	117·9 (1)
O(22)–Zr–O(12')	72·8 (1)	O(21)–Zr–O(21')	75·0 (1)
O(22)–Zr–O(21')	142·7 (1)	O(22)–Zr–O(22')	140·9 (1)
Zr–O(11)–C(11)	133·7 (1)	Zr–O(12)–C(12)	132·9 (1)
O(11)–C(11)–C(13)	123·7 (1)	O(11)–C(11)–C(14)	116·3 (1)
C(13)–C(11)–C(14)	120·0 (1)	O(12)–C(12)–C(13)	123·9 (2)
O(12)–C(12)–C(15)	116·4 (1)	C(13)–C(12)–C(15)	119·6 (1)
C(11)–C(13)–C(12)	122·5 (1)	Zr–O(21)–C(21)	132·7 (1)
Zr–O(22)–C(22)	132·8 (1)	O(21)–C(21)–C(23)	123·7 (2)
O(21)–C(21)–C(24)	116·2 (2)	C(23)–C(21)–C(24)	120·0 (2)
O(22)–C(22)–C(23)	124·1 (2)	O(22)–C(22)–C(25)	115·6 (2)
C(23)–C(22)–C(25)	120·3 (2)	C(21)–C(23)–C(22)	122·5 (2)

The prime denotes an atom generated by the twofold rotation axis through Zr (symmetry operation $1 - x, y, \tfrac{1}{2} - z$).

I thank SERC for a research grant towards crystallographic equipment.

Structure of Bis(tetramethylammonium) Oxopentathiocyanatomolybdate(V)

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Abstract. $[N(CH_3)_4]_2[Mo(NCS)_5O]$, $M_r = 550 \cdot 6$, orthorhombic, *Pnma*, $a = 18 \cdot 5152$ (10), $b = 14 \cdot 3794$ (8), $c = 9 \cdot 3321$ (5) Å, $V = 2484 \cdot 6$ Å^3, $Z = 4$, $D_x = 1 \cdot 472$ Mg m^{-3}, $\lambda(Mo\ K\alpha) = 0 \cdot 71073$ Å, $\mu = 0 \cdot 94$ mm^{-1}, $F(000) = 1124$, $T = 298$ K, $R = 0 \cdot 048$ for 1711 unique reflections with $F > 4\sigma(F)$. The entire anion, except for two mutually *trans* NCS$^-$ ligands, lies on a crystallographic mirror plane; the approximate symmetry is $4mm$ (C_{4v}). The NCS$^-$ ligands are coordinated to Mo through N. The axial Mo–N bond *trans* to Mo=O is considerably longer than the equatorial ones [$2 \cdot 228$ (5) Å compared with $2 \cdot 050$ (6)– $2 \cdot 063$ (6) Å; Mo=O $= 1 \cdot 646$ (5) Å]. The four equatorial ligands are bent towards the axial NCS$^-$ ligand and away from the oxo ligand, both in terms of O–Mo–N angles [in the range $96 \cdot 4$ (1)–$98 \cdot 4$ (1)°] and by significant bending at N [in the range $162 \cdot 3$ (6)– $169 \cdot 4$ (6)°]. By contrast the Mo–N–CS angle *trans* to O is $179 \cdot 4$ (5)°. The shortest NCS···C(cation) distance is $3 \cdot 43$ Å, and shortest interionic NCS···NCS contact is $3 \cdot 97$ Å, but these both involve the same NCS ligand (and its mirror-related counterpart) of the anion; in view of this marked difference in the packing surroundings of the various NCS ligands and the consistent degree and direction of their bending, this distortion is probably an intrinsic property of the anion rather than of its lattice environment.

Experimental. Crystal size $0 \cdot 8 \times 0 \cdot 3 \times 0 \cdot 08$ mm, Siemens AED2 diffractometer, cell parameters from 2θ values of 32 reflections with $20 < 2\theta < 25°$. Intensity measurements in ω/θ scan mode, scan width $= 1 \cdot 02° + \alpha$-doublet separation, scan time $= 14$–56 s, $2\theta_{max} = 50°$, $h\ 0 \rightarrow 21$, $k\ -17 \rightarrow 13$, $l\ 0 \rightarrow 11$, no significant variation for three standard reflections, semi-empirical absorption correction, transmission $0 \cdot 524$–$0 \cdot 609$, extinction negligible. 4146 reflections, 2270 unique ($R_{int} = 0 \cdot 027$), 1711 with $F > 4\sigma(F)$. Structure solved by multisolution direct methods and difference syntheses, blocked-cascade refinement on F, $w = 1/\sigma^2(F)$, anisotropic thermal parameters for non-H atoms, H atoms constrained [C–H $= 0 \cdot 96$ Å, H–C–H $= 109 \cdot 5°$ in rigid methyl groups, $U(H) = 1 \cdot 2U_{eq}(C)$]. 139 parameters, $R = 0 \cdot 048$, $wR = 0 \cdot 039$, slope of normal probability plot $= 1 \cdot 69$; max. $\Delta/\sigma = 0 \cdot 002$, mean $= 0 \cdot 001$, max. $\Delta\rho = 0 \cdot 51$, min. $= -0 \cdot 43$ e Å^{-3}. Scattering factors from *International Tables for X-ray Crystallography* (1974), *SHELXTL* (Sheldrick, 1985) programs. The structure and atom-numbering scheme are shown in Fig. 1. Atomic coordinates are listed in Table 1,* and bond lengths and angles in Table 2.

Related literature. The oxo chemistry of Mo^V is dominated by binuclear species (Cotton & Wilkinson, 1980). Known structures of mononuclear anions are mainly of the type $MoOX_5^{2-}$ ($X^- =$ halide) (*e.g.* Bino & Cotton, 1979). Bonding of thiocyanate to Mo *via* N has been demonstrated in various oxidation states (Knox & Eriks, 1968; Viossat, Rodier & Khodadad, 1979; Bino, Cotton & Fanwick, 1979); bonding *via* S is unknown.