Fig. 1. *ORTEP*II diagram (Johnson, 1976) showing the atom-numbering scheme of the cation. Thermal ellipsoids at 30% probability level.

(1984) and Hathaway (1982, 1983). Structures previously reported for Cu(phen)$_2$X cations are those with small X ligands which have trigonal bipyramidal geometries; these include structures in which X is

chloride (Boys, Escobar & Martinez-Carrera, 1981), cyanide (Anderson, 1975) and water (Nakai & Deguchi, 1975; Nakai & Noda, 1978).

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Bis[(N,N-diisopropyldithiocarbamato)(diphenylphosphinito)(diphenylthiophosphinito)platinato-O,S]cobalt(II)

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Abstract. [CoPt$_2$(C$_7$H$_{14}$NS$_2$)$_2$(C$_{12}$H$_{10}$OP)$_2$(C$_{12}$H$_{10}$PS)$_2$], $M_r = 1638 \cdot 6$, monoclinic, $C2/c$, $a = 29 \cdot 168$ (23), $b = 9 \cdot 693$ (15), $c = 24 \cdot 467$ (15) Å, $\beta = 101 \cdot 13$ (4)°, $V = 6788$ Å^3, $Z = 4$, $D_x = 1 \cdot 603$ Mg m^{-3}, λ(Mo Kα) = $0 \cdot 71073$ Å, $\mu = 4 \cdot 673$ mm^{-1}, $F(000) = 3236$, $T = 293$ K, $R = 0 \cdot 0648$ for 3755 unique observed reflections. The Co lies on a crystallographic twofold axis and is tetrahedrally coordinated by one S and one O donor atom from each Pt-containing ligand. At $1 \cdot 884$ (11) Å, the Co—O bond length lies significantly below the range of $1 \cdot 95$–$1 \cdot 97$ Å found in the corresponding complex where both donor atoms in each ligand are oxygen. The Co—S distance is $2 \cdot 319$ (4) Å, angles at Co range from $99 \cdot 7$ (5) to $119 \cdot 2$ (3)° and the coordination about the Pt is distorted square-planar.

Experimental. Synthesis of title compound (Anderson, Stephenson & Walkinshaw, 1988) by reaction of [Pt(S$_2$CN^{i}Pr$_2$)(Ph$_2$PO)(Ph$_2$PS)H] (Anderson, Ebsworth, Stephenson & Walkinshaw, 1981, 1982) with Co(acac)$_2$. Blue lamellar crystal $0 \cdot 04 \times 0 \cdot 24 \times 0 \cdot 52$ mm, STADI-2 diffractometer, graphite-monochromated Mo Kα radiation, cell parameters from 11 reflections with $5 \cdot 0 < \theta < 19 \cdot 0$°. For data collection, ω scans with scan width $1 \cdot 0 + 0 \cdot 5(\sin\mu/\tan\theta)$°, $\theta_{max} = 25$°, $h -34 \rightarrow 33$, $k 0 \rightarrow 11$, $l 0 \rightarrow 28$, no significant crystal movement or decay, initial absorption correction using ψ scans, 6166 reflections, 5916 unique ($R_{int} = 0 \cdot 016$), giving 2161 with $F > 6\sigma(F)$ for structure

Table 1. *Fractional coordinates of atoms with e.s.d.'s in parentheses*

$$U_{eq} = \tfrac{1}{3}\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$U_{eq}(\text{Å}^2)$
Co	0.5	0.6224 (3)	0.75	0.0413 (15)
Pt	0.38907 (2)	0.64955 (6)	0.61101 (2)	0.0424 (3)
S(1)	0.38324 (16)	0.6802 (5)	0.51499 (15)	0.069 (3)
S(2)	0.32191 (15)	0.5238 (5)	0.56746 (18)	0.071 (3)
C(1)	0.3320 (6)	0.5813 (19)	0.5044 (6)	0.063 (4)
N(1)	0.3075 (6)	0.5601 (23)	0.4560 (5)	0.097 (12)
C(11)	0.3172 (14)	0.605 (14)	0.4049 (15)	0.178 (14)
C(12)	0.3345 (15)	0.504 (5)	0.3727 (17)	0.29 (3)
C(13)	0.2948 (15)	0.729 (5)	0.3865 (17)	0.261 (21)
C(21)	0.2665 (11)	0.470 (4)	0.4490 (12)	0.142 (10)
C(22)	0.2767 (18)	0.332 (6)	0.4480 (21)	0.40 (4)
C(23)	0.2313 (15)	0.540 (5)	0.4647 (17)	0.270 (23)
P(1)	0.45525 (13)	0.7720 (4)	0.63356 (14)	0.0451 (21)
C(111)	0.4908 (4)	0.7336 (11)	0.5827 (4)	0.049 (3)
C(112)	0.5143 (4)	0.6074 (11)	0.5888 (4)	0.068 (5)
C(113)	0.5425 (4)	0.5693 (11)	0.5513 (4)	0.090 (6)
C(114)	0.5472 (4)	0.6574 (11)	0.5077 (4)	0.092 (6)
C(115)	0.5238 (4)	0.7836 (11)	0.5015 (4)	0.103 (7)
C(116)	0.4956 (4)	0.8216 (11)	0.5390 (4)	0.093 (6)
C(121)	0.4459 (3)	0.9576 (8)	0.6293 (4)	0.047 (3)
C(122)	0.4771 (3)	1.0432 (8)	0.6641 (4)	0.062 (4)
C(123)	0.4700 (3)	1.1855 (8)	0.6632 (4)	0.082 (5)
C(124)	0.4315 (3)	1.2422 (8)	0.6275 (4)	0.078 (5)
C(125)	0.4003 (3)	1.1566 (8)	0.5926 (4)	0.070 (4)
C(126)	0.4074 (3)	1.0143 (8)	0.5936 (4)	0.060 (4)
P(2)	0.38544 (11)	0.6065 (4)	0.70104 (14)	0.0396 (19)
C(211)	0.3317 (3)	0.5184 (11)	0.7056 (4)	0.049 (4)
C(212)	0.3283 (3)	0.3765 (11)	0.6962 (4)	0.080 (5)
C(213)	0.2861 (3)	0.3087 (11)	0.6957 (4)	0.103 (7)
C(214)	0.2472 (3)	0.3828 (11)	0.7047 (4)	0.086 (6)
C(215)	0.2505 (3)	0.5247 (11)	0.7141 (4)	0.084 (5)
C(216)	0.2928 (3)	0.5925 (11)	0.7146 (4)	0.062 (4)
C(221)	0.3834 (4)	0.7599 (10)	0.7421 (4)	0.052 (4)
C(222)	0.3966 (4)	0.7568 (10)	0.8001 (4)	0.075 (5)
C(223)	0.3976 (4)	0.8785 (10)	0.8307 (4)	0.099 (7)
C(224)	0.3855 (4)	1.0034 (10)	0.8033 (4)	0.106 (7)
C(225)	0.3723 (4)	1.0066 (10)	0.7454 (4)	0.105 (7)
C(226)	0.3713 (4)	0.8848 (10)	0.7148 (4)	0.077 (5)
S(3)	0.43571 (12)	0.4785 (4)	0.74402 (15)	0.0490 (21)
O(1)	0.4860 (4)	0.7478 (12)	0.6903 (4)	0.064 (7)

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

Co–S(3)	2.319 (4)	C(11)–C(12)	1.41 (6)
Co–O(1)	1.884 (11)	C(11)–C(13)	1.40 (6)
Pt–S(1)	2.342 (5)	C(21)–C(22)	1.37 (6)
Pt–S(2)	2.378 (5)	C(21)–C(23)	1.35 (6)
Pt–P(1)	2.242 (4)	P(1)–C(111)	1.806 (11)
Pt–P(2)	2.264 (4)	P(1)–C(121)	1.820 (10)
S(1)–C(1)	1.752 (18)	P(1)–O(1)	1.519 (11)
S(2)–C(1)	1.718 (18)	P(2)–C(211)	1.807 (10)
C(1)–N(1)	1.277 (25)	P(2)–C(221)	1.802 (11)
N(1)–C(11)	1.40 (4)	P(2)–S(3)	2.050 (5)
N(1)–C(21)	1.46 (4)		
S(3)–Co–O(1)	106.7 (3)	N(1)–C(21)–C(22)	114.2 (32)
S(3)–Co–O(1')	119.2 (3)	N(1)–C(21)–C(23)	108.5 (30)
S(3)–Co–S(3')	106.06 (14)	C(22)–C(21)–C(23)	132.5 (39)
O(1)–Co–O(1')	99.7 (5)	Pt–P(1)–C(111)	107.6 (4)
P(2)–S(3)–Co	99.11 (18)	Pt–P(1)–C(121)	113.4 (3)
P(1)–O(1)–Co	144.1 (7)	Pt–P(1)–O(1)	118.2 (4)
S(1)–Pt–S(2)	73.81 (16)	C(111)–P(1)–C(121)	105.3 (5)
S(1)–Pt–P(1)	94.15 (15)	C(111)–P(1)–O(1)	106.3 (6)
S(1)–Pt–P(2)	172.51 (14)	C(121)–P(1)–O(1)	105.1 (5)
S(2)–Pt–P(1)	167.77 (15)	P(1)–C(111)–C(112)	116.2 (8)
S(2)–Pt–P(2)	98.78 (14)	P(1)–C(111)–C(116)	123.8 (8)
P(1)–Pt–P(2)	93.30 (13)	P(1)–C(121)–C(122)	118.7 (7)
Pt–S(1)–C(1)	88.5 (6)	P(1)–C(121)–C(126)	121.3 (7)
Pt–S(2)–C(1)	88.1 (6)	Pt–P(2)–C(211)	110.5 (3)
S(1)–C(1)–S(2)	109.5 (9)	Pt–P(2)–C(221)	113.8 (4)
S(1)–C(1)–N(1)	122.3 (14)	Pt–P(2)–S(3)	116.59 (19)
S(2)–C(1)–N(1)	128.2 (14)	C(211)–P(2)–C(221)	103.5 (5)
C(1)–N(1)–C(11)	127.1 (22)	C(211)–P(2)–S(3)	103.0 (4)
C(1)–N(1)–C(21)	120.2 (20)	C(221)–P(2)–S(3)	108.1 (4)
C(11)–N(1)–C(21)	112.4 (24)	P(2)–C(211)–C(212)	119.4 (7)
N(1)–C(11)–C(12)	116.3 (34)	P(2)–C(211)–C(216)	120.5 (7)
N(1)–C(11)–C(13)	112.8 (33)	P(2)–C(221)–C(222)	121.1 (8)
C(12)–C(11)–C(13)	127.6 (39)	P(2)–C(221)–C(226)	118.8 (8)

Primed and unprimed atoms are related by the twofold axis passing through Co.

solution [from a Patterson synthesis (Pt) followed by iterative cycles of least-squares refinement and difference Fourier synthesis] and refinement [using full-matrix least squares on F (Sheldrick, 1976)]. At isotropic convergence, final absorption correction applied empirically using *DIFABS* (Walker & Stuart, 1983). Anisotropic thermal parameters for Pt, Co, S, P, N and O, phenyl rings refined as rigid groups and H atoms in fixed, calculated positions. At convergence, $R = 0.0648$, $wR = 0.0805$, $S = 1.060$ for 154 parameters, max. shift/e.s.d. in final cycle $= 0.007$, max. and min. residues in final difference Fourier synthesis 1.63 and -2.05 e Å^{-3} respectively. Scattering factors were inlaid (Sheldrick, 1976) except for Pt and Co (Cromer & Mann, 1968). Atom coordinates and equivalent isotropic thermal parameters are given in Table 1 while selected bond lengths and angles appear in Table 2.* The atom-numbering scheme for the complex is shown in Fig. 1, which was generated using *ORTEP* (Mallin-

* Lists of structure factors, anisotropic thermal parameters, torsion angles and H-atom coordinates have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44821 (25 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.
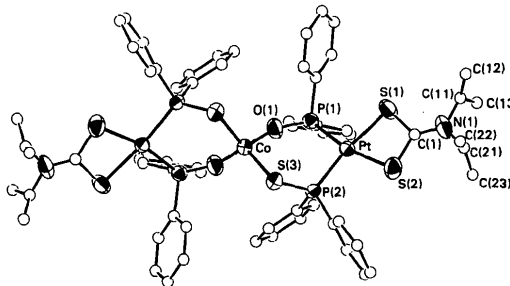

Fig. 1. View of the molecule showing atom-numbering scheme. Thermal ellipsoids are drawn at the 50% probability level, except those of C which have artificial radii of 0.15 Å. H atoms are omitted for clarity.

son & Muir, 1985). Molecular geometry calculations were performed using *CALC* (Gould & Taylor, 1985).

Related literature. The analogous bis[(*N,N*-diethyldithiocarbamato)bis(diphenylphospinito)platinato-*O,O'*]cobalt(II) complex shows a similar distorted tetrahedral Co environment (Allan, Milburn, Sawyer, Shah, Stephenson & Veitch, 1985; Marsh, 1985). We recently reported the corresponding compound with two palladium-containing ligands (Allan, Halfpenny, Milburn, Stephenson & Veitch, 1986) and, as part of the study of the complexes of both the Pt- and Pd-containing phosphinito ligands with lanthanide and

actinide elements, the structure of the uranyl complex $\{UO_2(OH_2)[(OPPh_2)_2Pd(S_2CNEt_2)]_2\}$ has been determined (Veitch, Blake, Allan & Schröder, 1987).

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Structure of 1,2,2,2,3,3,3,4,4,4-Decacarbonyl-1,2;3,4-di-μ-hydrido-1,3-bis(tricyclohexylphosphine)-*cyclo*-platinumtriosmium(2 *Pt–Os*)(3 *Os–Os*)–Toluene (1/1)

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Abstract. $[Os_3Pt(\mu\text{-}H)_2(CO)_{10}\{P(C_6H_{11})_3\}_2].C_7H_8$, $M_r = 1700 \cdot 8$, triclinic, $P\bar{1}$, $a = 11 \cdot 427$ (2), $b = 14 \cdot 842$ (3), $c = 17 \cdot 815$ (2) Å, $\alpha = 93 \cdot 32$ (1), $\beta = 99 \cdot 60$ (1), $\gamma = 104 \cdot 24$ (1)°, $V = 2872 \cdot 0$ (8) Å^3, $Z = 2$, $D_x = 1 \cdot 97$ g cm^{-3}, $\lambda(\text{Mo } K\alpha) = 0 \cdot 71069$ Å, $\mu = 91 \cdot 8$ cm^{-1}, $F(000) = 1620$, $T = 298$ K, $R = 0 \cdot 024$ for 7123 observed reflections. The metal atom skeleton is of the 'butterfly' form, with a non-bonding Pt$\cdots$Os(1) distance of $3 \cdot 506$ (1) Å, and a butterfly angle, defined by the torsion angle Os(1)–Os(2)–Os(3)–Pt, of $87 \cdot 5$ (1)°. The two hydride ligands are *cis* to the phosphine groups, lying along Os–Os and Os–Pt edges.

Experimental. Yellow prisms from toluene solution: crystal dimensions *ca* $0 \cdot 3 \times 0 \cdot 4 \times 0 \cdot 3$ mm; Enraf–Nonius CAD-4F diffractometer; graphite monochromator; $\theta/2\theta$ scan mode; cell parameters refined by least-squares method on basis of 25 independent θ values, $11 \le \theta \le 13°$; intensities measured to $\theta = 25°$ over range of *hkl* 0 to 13, −17 to +17, −21 to +21; $\bar{3}63$, $3\bar{2}7$, $6\bar{2}6$ measured every 2 h, linear decay correction applied corresponding to 8% decay over 169 h data collection, 10 643 reflections measured, 10 008 independent data with 7123 having $I \ge 3 \cdot 0\sigma(I)$ considered observed and used in structure determination and refinement; R_{int} $0 \cdot 028$ before and $0 \cdot 027$ after absorption correction; corrected for Lorentz/polarization, absorption (*DIFABS*; Stuart & Walker, 1983);

range of transmission factors on F, $0 \cdot 823$ to $1 \cdot 238$; solved by direct methods (*MITHRIL*; Gilmore, 1984) and subsequent full-matrix least squares; anisotropic thermal parameters for non-H atoms; H atoms included at fixed calculated positions [cyclohexyl C–H $= 1 \cdot 0$ Å: hydrides (*HYDEX*; Orpen, 1980) Os–H, Pt–H *ca* $1 \cdot 85$ Å] with fixed isotropic ($U = 0 \cdot 05$ Å^2) thermal parameters; solvent molecule of toluene disordered over two sites (50:50), the benzene ring treated as rigid hexagon C–C $= 1 \cdot 395$ Å: H-atom contributions from solvent not included; $w(|F_o| - |F_c|)^2$ minimized with $w = [\sigma^2(F_o)]^{-1}$; max. Δ/σ $0 \cdot 29$, ave. $0 \cdot 025$; $(\Delta\rho)_{max}$ $+0 \cdot 72$, $(\Delta\rho)_{min}$ $-0 \cdot 74$ e Å^{-3} in vicinity of heavy metal atoms; $R = 0 \cdot 024$, $wR = 0 \cdot 028$; $R(wR) = 0 \cdot 051$ ($0 \cdot 030$) for all data; $S = 1 \cdot 27$; atomic scattering factors including anomalous terms from *International Tables for X-ray Crystallography* (1974); calculations carried out on a Gould-SEL 32/27 minicomputer using the *GX* suite of programs (Mallinson & Muir, 1985). Final positional parameters are given in Table 1, with selected bond distances and angles in Table 2.* The molecular structure and atomic labelling scheme are shown in Fig.

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* Lists of structure factors, anisotropic thermal parameters, calculated hydrogen positional parameters, and a complete listing of bond lengths and angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44915 (59 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.