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[Bis(diphenylphosphino)methane]bis(pyridine-2-thiolato *N*-oxide)cobalt(III) perchlorate diethyl ether solvate

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Key indicators

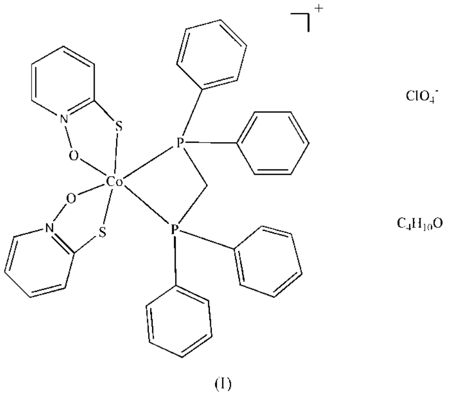
Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.013 Å
R factor = 0.081
wR factor = 0.177
Data-to-parameter ratio = 15.0

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

The reaction between the metal diphosphine $[Cu_2(\mu_2\text{-dppm})_2(MeCN)_4](ClO_4)_2$ [dppm is bis(diphenylphosphino)methane] and the metal–thiolate component $[Co(mpo)_3]$ (mpo is 2-mercaptopyridine *N*-oxide) gave the title compound, $[Co(C_5H_4NOS)_2(C_{25}H_{22}P_2)]ClO_4\cdot C_4H_{10}O$, in which one mpo ligand in $[Co(mpo)_3]$ was displaced by dppm. The Co^{III} atom is located in a distorted octahedral coordination environment. Two S atoms of two mpo ligands are in *trans* positions.

Comment

Interest in metal complexes of diphosphines has grown rapidly in recent years (Yam *et al.*, 1998). We are interested in the design of photoluminescent heterometallic cluster complexes by self-assembly between two metal components, one with potential bridging donors and the other with substitutable or vacant coordination sites (Xu *et al.*, 2002). Thus, the reaction between $[Co(mpo)_3]$ (mpo is 2-mercaptopyridine *N*-oxide), with potential bridging sulfur donors, and $[Cu_2(\mu_2\text{-dppm})_2(MeCN)_4](ClO_4)_2$ [dppm is bis(diphenylphosphino)methane], with substitutable solvent coordination sites, was expected to afford Cu—Co heteronuclear species. Nevertheless, the only isolated product of the reaction between $[Co(mpo)_3]$ and $[Cu_2(\mu_2\text{-dppm})_2(MeCN)_4](ClO_4)_2$ was the title compound, (I), in which the Co^{III} atom is coordinated by two P atoms, two O atoms and two S atoms; the Co—O distances [1.957 (4) and 1.981 (4) Å] and Co—S distances [2.2263 (19) and 2.2346 (19) Å] are similiar to those in $[Co(mpo)_3]$, and the Co—P distances are 2.196 (2) and 2.1973 (19) Å.



(I)

Experimental

$[Co(mpo)_3]$ (Manivannan *et al.*, 1993) and $[Cu_2(\mu_2\text{-dppm})_2(MeCN)_4](ClO_4)_2$ (Diez *et al.*, 1987) were prepared using reported procedures. To an acetonitrile solution of $[Co(mpo)_3]$ was added an acetonitrile solution of $[Cu_2(\mu_2\text{-dppm})_2(MeCN)_4](ClO_4)_2$, with stirring at room temperature for one day. Well-shaped dark-blue crystals

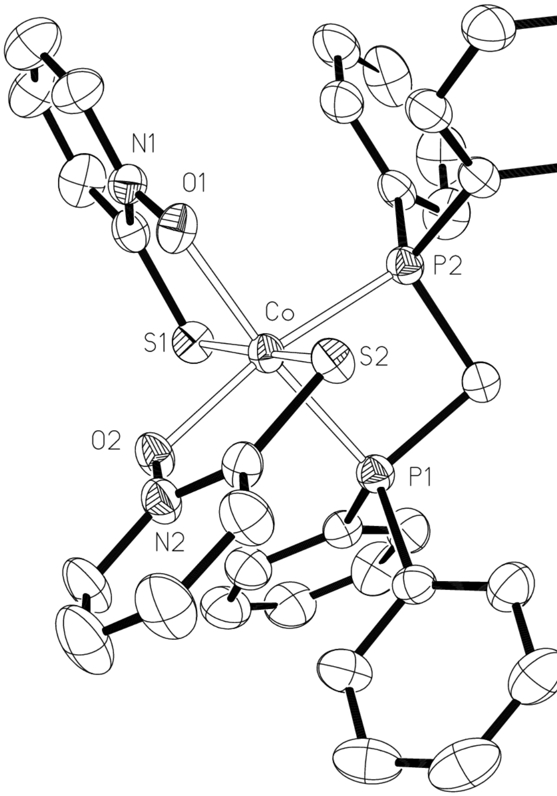


Figure 1
A view of the complex cation of (I), with the atomic numbering scheme. Displacement ellipsoids are drawn at the 30% probability level.

of (I) were grown by layering diethyl ether on to the acetonitrile solution for a period of 3 d.

Crystal data

$[Co(C_5H_4NOS)_2(C_{25}H_{22}P_2)]$-$ClO_4{\cdot}C_4H_{10}O$
$M_r = 869.17$
Monoclinic, $P2_1/c$
$a = 12.730$ (3) Å
$b = 16.873$ (4) Å
$c = 18.864$ (4) Å
$\beta = 97.572$ (7)°
$V = 4016.8$ (16) Å^3
$Z = 4$
$D_x = 1.437$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 4947 reflections
$\theta = 1.6$–25.0°
$\mu = 0.73$ mm^{-1}
$T = 293$ (2) K
Prism, blue
0.34 × 0.30 × 0.28 mm

Data collection

Siemens SMART CCD diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.612$, $T_{max} = 0.816$
12 116 measured reflections
6918 independent reflections
4927 reflections with $I > 2\sigma(I)$
$R_{int} = 0.041$
$\theta_{max} = 25.0°$
$h = -8 \rightarrow 15$
$k = -20 \rightarrow 13$
$l = -22 \rightarrow 20$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.081$
$wR(F^2) = 0.177$
$S = 1.19$
6918 reflections
462 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0203P)^2 + 18.3746P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.008$
$\Delta\rho_{max} = 0.90$ e Å^{-3}
$\Delta\rho_{min} = -0.48$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Co1—O1	1.957 (4)	Co1—P2	2.1973 (19)
Co1—O2	1.981 (4)	Co1—S1	2.2263 (19)
Co1—P1	2.196 (2)	Co1—S2	2.2346 (19)
O1—Co1—O2	91.74 (19)	P1—Co1—S1	91.92 (7)
O1—Co1—P1	94.69 (15)	P2—Co1—S1	89.99 (7)
O2—Co1—P1	173.57 (15)	O1—Co1—S2	89.82 (13)
O1—Co1—P2	170.30 (15)	O2—Co1—S2	87.49 (13)
O2—Co1—P2	97.43 (14)	P1—Co1—S2	92.74 (7)
P1—Co1—P2	76.14 (7)	P2—Co1—S2	93.74 (7)
O1—Co1—S1	87.12 (13)	S1—Co1—S2	174.62 (8)
O2—Co1—S1	88.18 (13)		

The crystal diffracted weakly during the data collection. There was high thermal motion of the diethyl ether solvent molecule, and atoms O10 and C21–C24 were refined isotropically to avoid very short bond distances. The positions of the H atoms were generated geometrically (C—H bond fixed at 0.96 Å), assigned isotropic displacement parameters and allowed to ride on their respective parent C atoms in the refinement.

Data collection: *SMART* (Siemens, 1996); cell refinement: *SMART* and *SAINT* (Siemens, 1994); data reduction: *SAINT* and *XPREP* (Siemens, 1994); program(s) used to solve structure: *SHELXTL* (Siemens, 1994); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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