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Pentacarbonyl(diphenyl(*N*-propylamine)-phosphine)diiron(μ-1,3-propanedithiolate)

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The title compound, (μ-$(SCH_2)_2CH_2$)Fe(CO)$_3$Fe(CO)$_2$P-$(C_6H_5)_2NHCH_2CH_2CH_3$, has been prepared and characterized as a a model substance for a class of enzymes known as hydrogenases.

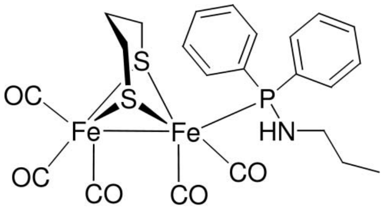
Key indicators

Single-crystal X-ray study
T = 291 K
Mean σ(C–C) = 0.006 Å
R factor = 0.035
wR factor = 0.058
Data-to-parameter ratio = 20.1

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

Comment

The title compound, (I), (Fig. 1) is a novel iron–sulfur complex containing a diphenyl(*N*-propylamine)phosphine complex. It resembles the active site in a class of enzymes called hydrogenases which catalyze hydrogen production in microorganisms by reduction of protons (Frey, 2002).



(I)

When the crystal structure of iron hydrogenase was solved (Peter *et al.*, 1998), it turned out that the active site of the enzyme consists of two Fe atoms bridged by a dithiolate ligand. At the Fe atoms, the biologically very unusual diatomic ligands CO and CN^- were coordinated. The active site resembles many well known and investigated iron–sulfur–carbonyl complexes (Reihlen *et al.*, 1928; Seyferth *et al.*, 1980). With the aim of finding a cheap and effective catalyst for hydrogen production, a variety of hydrogenase model complexes have been synthesized and studied (Evans & Pickett, 2003; Rauchfuss, 2004; Ott *et al.*, 2004), to which (I) may be now be added. Selected geometrical data for (I) are presented in Table 1.

Experimental

(μ-$(SCH_2)_2CH_2$)Fe(CO)$_3$Fe(CO)$_3$ (0.10 g, 0.26 mmol) was dissolved in dry THF (15 ml) and *n*-propylamine (5 ml) was added under an argon atmosphere. Diphenylchlorophosphine (0.2 ml, 1.1 mmol) was added and the solution was stirred at 293 K for 1.5 h and then at 348 K for 4 h. The solvent was removed by evaporation under reduced pressure and the red residue was subjected to column chromatography (silica, 20% toluene in pentane). The product was obtained as a red solid (131 mg, 83% yield). Single crystals of (I) for X-ray analysis were grown by slow diffusion of hexane into a solution of the complex in toluene at 256 K.

Crystal data

$[Fe_2(C_{15}H_{18}NP)(C_3H_6S_2)(CO)_5]$
$M_r = 601.22$
Orthorhombic, $P2_12_12_1$
$a = 8.682$ (1) Å
$b = 9.421$ (1) Å
$c = 32.108$ (3) Å
$V = 2626.2$ (5) Å^3
$Z = 4$
$D_x = 1.521$ Mg m^{-3}

Mo $K\alpha$ radiation
Cell parameters from 1371 reflections
$\theta = 1.9–28.1°$
$\mu = 1.36$ mm^{-1}
$T = 291$ (2) K
Plate, red
0.20 × 0.10 × 0.02 mm

Data collection

Stoe IPDS area-detector diffractometer
φ scans
Absorption correction: numerical (*X-RED*; Stoe, 1997*b*)
$T_{min} = 0.75$, $T_{max} = 0.97$
27690 measured reflections

6253 independent reflections
2923 reflections with $I > 2\sigma(I)$
$R_{int} = 0.099$
$\theta_{max} = 28.2°$
$h = -11 \rightarrow 11$
$k = -12 \rightarrow 12$
$l = -42 \rightarrow 42$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.035$
$wR(F^2) = 0.058$
$S = 0.89$
6253 reflections
311 parameters
H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.01P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.35$ e Å^{-3}
$\Delta\rho_{min} = -0.84$ e Å^{-3}
Absolute structure: Flack (1983); 2563 Friedel pairs
Flack parameter = −0.036 (15)

Table 1
Selected geometric parameters (Å, °).

Fe1—C4	1.742 (5)	Fe2—C2	1.787 (4)
Fe1—C5	1.758 (4)	Fe2—S4	2.2716 (11)
Fe1—P1	2.2068 (11)	Fe2—S3	2.2735 (10)
Fe1—S4	2.2625 (11)	S3—C12	1.809 (3)
Fe1—S3	2.2652 (11)	S4—C10	1.808 (4)
Fe1—Fe2	2.4936 (7)	P1—N41	1.662 (3)
Fe2—C3	1.758 (4)	P1—C21	1.819 (4)
Fe2—C1	1.774 (5)	P1—C31	1.830 (4)
P1—Fe1—S4	102.35 (4)	S4—Fe2—S3	84.17 (4)
P1—Fe1—S3	109.33 (4)	S4—Fe2—Fe1	56.46 (3)
S4—Fe1—S3	84.56 (4)	S3—Fe2—Fe1	56.51 (3)
P1—Fe1—Fe2	153.55 (4)	Fe1—S3—Fe2	66.65 (3)
S4—Fe1—Fe2	56.81 (3)	Fe1—S4—Fe2	66.73 (3)
S3—Fe1—Fe2	56.83 (3)		

The H atoms attached to C were positioned geometrically and refined as riding with displacement parameter constraints as follows: methyl $d(C—H) = 0.96$ Å, $U_{iso}(H) = 1.5U_{eq}(C)$; methylene $d(C—H) = 0.97$ Å, $U_{iso}(H) = 1.2U_{eq}(C)$; aromatic $d(C—H) = 0.93$ Å, $U_{iso}(H) = 1.2U_{eq}(C)$. The position of the H atom attached to N was freely refined with the constraint $U_{iso}(H) = 1.5U_{eq}(N)$ applied.

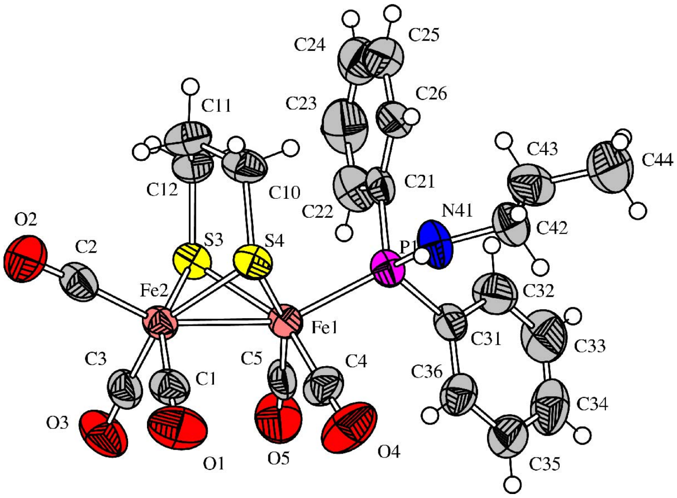


Figure 1
Molecular structure of (I), showing 50% displacement ellipsoids (arbitrary spheres for the H atoms).

Data collection: *EXPOSE* (Stoe, 1997*a*); cell refinement: *CELL* (Stoe, 1997*a*); data reduction: *INTEGRATE* (Stoe, 1997*a*); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *DIAMOND* (Bergerhoff, 1996); software used to prepare material for publication: *PLATON* (Spek, 2003).

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