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Pentacarbonyl-1κ^2C,2κ^3C-(diphenylphosphine-1κP)(μ-2-propyl-2-azapropane-1,3-dithiolato-1κ^2S,S':2κ^2S,S')-diiron(*Fe*—*Fe*)

Zhen Wang,[a] Jian-Hui Liu[a]* and Li-Cheng Sun[a,b]

[a]State Key Laboratory of Fine Chemicals, Dalian University of Technology, Zhongshan Road 158-40, Dalian 116012, People's Republic of China, and [b]Department of Chemistry, Organic Chemistry, Royal Institute of Technology(KTH), 10044 Stockholm, Sweden
Correspondence e-mail: liujh@dlut.edu.cn

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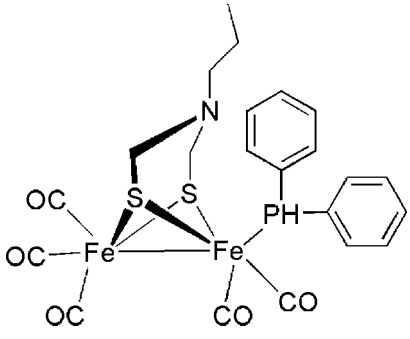
Key indicators: single-crystal X-ray study; T = 273 K; mean σ(C–C) = 0.005 Å; R factor = 0.037; wR factor = 0.087; data-to-parameter ratio = 16.2.

The title compound, $[Fe_2(C_5H_{11}NS_2)(C_{12}H_{11}P)(CO)_5]$, has been prepared and characterized as a model compound of the iron hydrogenase active site through controlled CO ligand displacement of $(\mu\text{-}SCH_2)_2N(CH_2CH_2CH_3)[Fe_2(CO)_6]$ with diphenylphosphine. The central Fe_2S_2 structure is in a butterfly conformation, and each Fe atom displays pseudo-square-pyramidal geometry. The phosphine group occupies an apical position. The propyl group on the bridging N atom is in an equatorial position and takes a zigzag form. In the crystal packing, adjacent pairs of molecules are associated by S···S (3.625 Å) intermolecular nonbonded weak interactions.

Related literature

For literature on 2Fe2S complexes, where the two Fe atoms are bridged by a dithiolate ligand and each of the Fe atoms is coordinated by carbonyl groups or tertiary phosphine ligands, see Lawrence *et al.* (2001); Liu *et al.* (2004); Wang *et al.* (2005); Gao *et al.* (2007). For related literature, see: Frey (2002); Milway *et al.* (2006); Nicolet *et al.* (2000).



Experimental

Crystal data

$[Fe_2(C_5H_{11}NS_2)(C_{12}H_{11}P)(CO)_5]$
M_r = 587.20
Monoclinic, $P2_1/c$
a = 12.4627 (15) Å
b = 12.1985 (15) Å
c = 17.2573 (2) Å
β = 90.162 (2)°
V = 2623.6 (6) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 1.36 mm^{-1}
T = 273 (2) K
0.30 × 0.20 × 0.08 mm

Data collection

Bruker SMART 1K CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.686, T_{max} = 0.899
13194 measured reflections
4885 independent reflections
3350 reflections with $I > 2\sigma(I)$
R_{int} = 0.041

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.037
$wR(F^2)$ = 0.087
S = 0.92
4885 reflections
302 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.44 e Å^{-3}
$\Delta\rho_{min}$ = −0.38 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Fe1—P	2.2021 (9)	P—H1	1.32 (2)
Fe1—Fe2	2.5143 (6)		
P—Fe1—Fe2	145.99 (3)	C7—N1—C8	113.7 (2)
C7—N1—C6	112.7 (2)	C6—N1—C8	115.3 (3)

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 2001); software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: WK2062).

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supplementary materials

Acta Cryst. (2007). E**63**, m1959-m1960 [**doi:10.1107/S1600536807029431**]

Pentacarbonyl-1κ^2C,2κ^3C-(diphenylphosphine-1κP)(μ-2-propyl-2-azapropane-1,3-dithiolato-1κ^2S,S':2κ^2S,S')diiron(*Fe-Fe*)

Z. Wang, J.-H. Liu and L.-C. Sun

Comment

Iron hydrogenases (Fe—H_2ase) are highly efficient enzymes that catalyze the reversible reduction of protons to molecular hydrogen. Crystallographic study revealed that the active site of Fe—H_2ase is comprised of one unusual low-valent diiron disulfide that is linked by a cysteine-ligand to a [Fe_4S_4] cluster, with one bridging carbonyl and an additional terminal carbonyl, cyanide and aqua ligands and the non-proteic dithiolate (Nicolet *et al.*, 2000; Frey, 2002). The molecule of the title compound, (I), (Fig. 1), exhibits some resemblance to the active site of Fe-hydrogenases.

The central 2Fe2S structure in compound I is in the butterfly conformation, with a dihedral angle of 108.4 (2)° between the two Fe_2S planes, and each iron atom displays pseudo-square-pyramidal geometry. The Fe—Fe distance in (I) (2.5143 (6) Å) agrees well with those found in other similar complexes [2.49–2.51 Å; Lawrence *et al.*, 2001; Liu *et al.*, 2004; Wang *et al.*, 2005, Gao *et al.*, 2007]. The phosphine group occupies an apical position. The propyl group on the bridged nitrogen atom is in an equatorial position and the propyl chain takes the zigzag form.

As shown in the packing diagram (Fig. 2), adjacent pairs of molecules are associated by S···S(3.625 Å) intermolecular non-bonded weak interactions. The distance is close to the sum of the van der Waals radii for two sulfur atoms (3.6 Å).

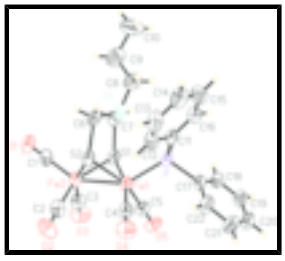
Experimental

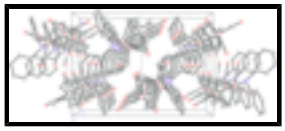
(μ-SCH_2)$_2$$NCH_2CH_2CH_3$[$Fe_2(CO)_6$] (0.10 g, 0.23 mmol) was dissolved in dry acetonitrile (15 ml) and $Me_3NO{\cdot}2H_2O$ (51 mg, 0.46 mmol) was added under an argon atmosphere. When the color of the solution turned dark red Diphenylphosphine (46 mg, 0.25 mmol) was added and the solution was stirred at 293 K for 1.5 h and the color turned to red. After the solvent was removed in vacuum, the crude product was purified by column chromatography on silica gel with CH_2Cl_2/hexane (1:1) as eluent to give I as red solid (109 mg, 81%). Single crystals of (I) for X-ray analysis were grown by slow diffusion of hexane into a solution of the complex in CH_2Cl_2 at 275 K.

Refinement

H atoms bonded to P atoms were located geometrically and allowed to ride on its parent atoms with distance restraints of P—H = 1.32 (2) Å, and with U_{iso}(H) = 1.2U_{eq}(P). Other H atoms were placed in calculated positions and refined using a riding model, with C—H = 0.93–0.97 Å and with U_{iso}(H) = 1.2 (1.5 for methyl groups) times U_{eq}(C).

Figures





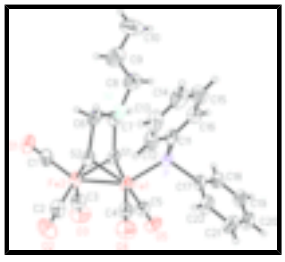


Fig. 1. The molecular structure of (I), with atom labels and 30% probability displacement ellipsoids for non-H atoms.
Fig. 2. The packing of (I), viewed down the *b* axis, showing S···S intermolecular weak interactions (dashed lines). H atoms have been omitted.

Pentacarbonyl-1κ^2*C*,2κ^3*C*-(diphenylphosphine-1κ*P*)(μ-2-propyl-2- azapropane-1,3-dithiolato-1κ^2*S*,*S*':2κ^2*S*,*S*')diiron(Fe—Fe)

Crystal data

$[Fe_2(C_5H_{11}NS_2)(C_{12}H_{11}P)(CO)_5]$	F_{000} = 1200
M_r = 587.20	D_x = 1.487 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation λ = 0.71073 Å
a = 12.4627 (15) Å	Cell parameters from 3783 reflections
b = 12.1985 (15) Å	θ = 2.4–23.4º
c = 17.2573 (2) Å	μ = 1.36 mm^{-1}
β = 90.162 (2)º	T = 273 (2) K
V = 2623.6 (6) Å^3	Block, red
Z = 4	0.30 × 0.20 × 0.08 mm

Data collection

Bruker SMART 1K CCD area-detector diffractometer	4885 independent reflections
Radiation source: fine-focus sealed tube	3350 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.041
T = 273(2) K	θ_{max} = 25.5º
φ and ω scans	θ_{min} = 1.6º
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	h = −15→8

T_{min} = 0.686, T_{max} = 0.899
13194 measured reflections

k = −13→14
l = −20→20

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)]$ = 0.037
$wR(F^2)$ = 0.087
S = 0.92
4885 reflections
302 parameters

H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0429P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.44 e Å^{-3}
$\Delta\rho_{min}$ = −0.38 e Å^{-3}
Extinction coefficient: ?

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit *S* are based on F^2, conventional *R*-factors *R* are based on *F*, with *F* set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on *F*, and *R*- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
Fe1	0.30424 (3)	0.45814 (4)	0.12088 (2)	0.05683 (14)
Fe2	0.23171 (3)	0.36249 (4)	0.00279 (2)	0.06549 (15)
P	0.34731 (6)	0.61660 (7)	0.17360 (4)	0.0584 (2)
S2	0.13569 (6)	0.48156 (7)	0.07511 (4)	0.0643 (2)
S1	0.35457 (6)	0.49763 (7)	−0.00134 (4)	0.0620 (2)
O5	0.51096 (19)	0.3537 (2)	0.14933 (14)	0.0892 (7)
C11	0.2452 (2)	0.7071 (3)	0.21361 (15)	0.0594 (7)
C17	0.4470 (2)	0.6044 (2)	0.25035 (16)	0.0623 (8)
C5	0.4302 (3)	0.3951 (3)	0.13735 (16)	0.0657 (8)
O4	0.2164 (2)	0.3518 (2)	0.25765 (14)	0.1033 (8)
C7	0.2972 (2)	0.6299 (2)	−0.03031 (17)	0.0698 (8)
H7A	0.3553	0.6816	−0.0381	0.084*
H7B	0.2606	0.6207	−0.0796	0.084*
C22	0.4207 (3)	0.5575 (3)	0.32002 (18)	0.0893 (11)
H22A	0.3502	0.5368	0.3294	0.107*
O1	0.1227 (2)	0.3756 (2)	−0.14763 (15)	0.1042 (9)
N1	0.22289 (19)	0.6757 (2)	0.02462 (13)	0.0661 (7)
C4	0.2501 (2)	0.3940 (3)	0.20373 (19)	0.0713 (9)

O3	0.4083 (2)	0.2168 (2)	−0.04003 (16)	0.1197 (10)
C12	0.1424 (2)	0.6704 (3)	0.22953 (17)	0.0747 (9)
H12A	0.1249	0.5973	0.2206	0.090*
C18	0.5521 (3)	0.6346 (3)	0.23811 (19)	0.0735 (9)
H18A	0.5716	0.6666	0.1913	0.088*
C1	0.1643 (3)	0.3708 (3)	−0.0897 (2)	0.0764 (9)
C19	0.6285 (3)	0.6179 (3)	0.2949 (2)	0.0903 (11)
H19A	0.6991	0.6391	0.2859	0.108*
C14	0.0908 (3)	0.8476 (4)	0.2707 (2)	0.0965 (12)
H14A	0.0388	0.8952	0.2897	0.116*
C9	0.1694 (4)	0.8407 (4)	−0.0525 (2)	0.1237 (15)
H9A	0.0987	0.8103	−0.0621	0.148*
H9B	0.2157	0.8199	−0.0951	0.148*
O2	0.1204 (2)	0.1872 (2)	0.08541 (18)	0.1176 (10)
C3	0.3380 (3)	0.2727 (3)	−0.02344 (18)	0.0809 (10)
C20	0.6026 (4)	0.5713 (3)	0.3630 (3)	0.1004 (12)
H20A	0.6550	0.5599	0.4006	0.120*
C16	0.2692 (3)	0.8157 (3)	0.22726 (17)	0.0743 (9)
H16A	0.3380	0.8417	0.2174	0.089*
C2	0.1618 (3)	0.2550 (3)	0.0515 (2)	0.0834 (10)
C13	0.0657 (3)	0.7404 (4)	0.25838 (19)	0.0878 (11)
H13A	−0.0028	0.7147	0.2694	0.105*
C6	0.1233 (2)	0.6155 (3)	0.02812 (18)	0.0723 (9)
H6A	0.0964	0.6050	−0.0242	0.087*
H6B	0.0709	0.6589	0.0561	0.087*
C10	0.1619 (5)	0.9682 (4)	−0.0479 (3)	0.153 (2)
H10A	0.1334	0.9962	−0.0957	0.229*
H10B	0.2322	0.9982	−0.0392	0.229*
H10C	0.1156	0.9887	−0.0060	0.229*
C21	0.4982 (4)	0.5408 (4)	0.3766 (2)	0.1135 (14)
H21A	0.4796	0.5089	0.4237	0.136*
H1	0.3993 (18)	0.6872 (19)	0.1294 (13)	0.061 (7)*
C15	0.1918 (3)	0.8861 (3)	0.2554 (2)	0.0918 (11)
H15A	0.2083	0.9595	0.2640	0.110*
C8	0.2117 (3)	0.7970 (3)	0.0188 (2)	0.0990 (12)
H8A	0.1657	0.8212	0.0607	0.119*
H8B	0.2818	0.8292	0.0275	0.119*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Fe1	0.0436 (2)	0.0801 (3)	0.0468 (2)	−0.00482 (19)	0.00266 (17)	−0.0036 (2)
Fe2	0.0520 (3)	0.0888 (4)	0.0557 (3)	−0.0045 (2)	0.0037 (2)	−0.0147 (2)
P	0.0474 (4)	0.0806 (6)	0.0472 (4)	−0.0090 (4)	0.0007 (3)	−0.0009 (4)
S2	0.0407 (4)	0.0926 (6)	0.0597 (4)	−0.0059 (4)	0.0050 (3)	−0.0133 (4)
S1	0.0424 (4)	0.0942 (6)	0.0496 (4)	0.0001 (4)	0.0043 (3)	−0.0024 (4)
O5	0.0612 (15)	0.118 (2)	0.0883 (16)	0.0159 (13)	0.0021 (13)	0.0201 (13)
C11	0.0552 (19)	0.079 (2)	0.0445 (15)	−0.0028 (15)	−0.0037 (13)	−0.0057 (14)

C17	0.0560 (18)	0.075 (2)	0.0556 (17)	−0.0081 (15)	−0.0063 (14)	−0.0049 (15)
C5	0.060 (2)	0.085 (2)	0.0525 (16)	−0.0046 (17)	0.0073 (15)	0.0067 (16)
O4	0.108 (2)	0.122 (2)	0.0805 (16)	−0.0190 (15)	0.0295 (15)	0.0187 (14)
C7	0.0607 (19)	0.092 (2)	0.0566 (17)	0.0015 (16)	0.0021 (15)	0.0050 (16)
C22	0.076 (2)	0.132 (3)	0.060 (2)	−0.023 (2)	−0.0126 (18)	0.005 (2)
O1	0.101 (2)	0.140 (2)	0.0720 (16)	−0.0120 (15)	−0.0218 (15)	−0.0162 (15)
N1	0.0534 (15)	0.0848 (19)	0.0602 (15)	0.0054 (13)	0.0046 (12)	−0.0012 (13)
C4	0.061 (2)	0.087 (2)	0.066 (2)	−0.0055 (17)	0.0042 (17)	−0.0049 (18)
O3	0.114 (2)	0.118 (2)	0.128 (2)	0.0364 (18)	0.0429 (19)	0.0009 (18)
C12	0.064 (2)	0.098 (3)	0.0629 (19)	−0.0093 (18)	0.0071 (16)	−0.0216 (17)
C18	0.057 (2)	0.089 (2)	0.074 (2)	−0.0034 (16)	−0.0104 (17)	−0.0060 (17)
C1	0.060 (2)	0.097 (3)	0.073 (2)	−0.0115 (17)	0.0076 (18)	−0.0206 (19)
C19	0.064 (2)	0.096 (3)	0.111 (3)	0.0000 (19)	−0.021 (2)	−0.017 (2)
C14	0.083 (3)	0.132 (4)	0.075 (2)	0.026 (3)	−0.002 (2)	−0.027 (2)
C9	0.137 (4)	0.148 (5)	0.087 (3)	0.016 (3)	−0.010 (3)	−0.003 (3)
O2	0.118 (2)	0.103 (2)	0.131 (2)	−0.0263 (18)	0.0403 (19)	−0.0058 (18)
C3	0.081 (3)	0.093 (3)	0.069 (2)	−0.001 (2)	0.0114 (19)	−0.0051 (18)
C20	0.098 (3)	0.105 (3)	0.098 (3)	0.005 (3)	−0.046 (3)	−0.008 (2)
C16	0.070 (2)	0.086 (3)	0.067 (2)	−0.0087 (19)	−0.0003 (17)	−0.0069 (18)
C2	0.069 (2)	0.099 (3)	0.082 (2)	−0.002 (2)	0.0084 (19)	−0.025 (2)
C13	0.059 (2)	0.126 (3)	0.078 (2)	−0.001 (2)	0.0031 (17)	−0.034 (2)
C6	0.0519 (19)	0.095 (2)	0.070 (2)	0.0088 (17)	−0.0029 (15)	−0.0055 (17)
C10	0.226 (6)	0.091 (4)	0.141 (4)	0.044 (3)	0.021 (4)	0.007 (3)
C21	0.138 (4)	0.133 (4)	0.070 (2)	−0.024 (3)	−0.032 (3)	0.018 (2)
C15	0.105 (3)	0.084 (3)	0.086 (3)	0.004 (2)	0.005 (2)	−0.013 (2)
C8	0.102 (3)	0.111 (3)	0.084 (3)	0.024 (2)	0.003 (2)	0.004 (2)

Geometric parameters (Å, °)

Fe1—C4	1.766 (3)	O3—C3	1.148 (4)
Fe1—C5	1.770 (3)	C12—C13	1.376 (4)
Fe1—P	2.2021 (9)	C12—H12A	0.9300
Fe1—S1	2.2543 (8)	C18—C19	1.380 (4)
Fe1—S2	2.2603 (9)	C18—H18A	0.9300
Fe1—Fe2	2.5143 (6)	C19—C20	1.345 (5)
Fe2—C3	1.778 (4)	C19—H19A	0.9300
Fe2—C2	1.786 (4)	C14—C13	1.361 (5)
Fe2—C1	1.805 (4)	C14—C15	1.370 (5)
Fe2—S1	2.2512 (9)	C14—H14A	0.9300
Fe2—S2	2.2602 (9)	C9—C8	1.439 (5)
P—C17	1.820 (3)	C9—C10	1.561 (5)
P—C11	1.821 (3)	C9—H9A	0.9700
P—H1	1.32 (2)	C9—H9B	0.9700
S2—C6	1.831 (3)	O2—C2	1.138 (4)
S1—C7	1.833 (3)	C20—C21	1.374 (5)
O5—C5	1.145 (3)	C20—H20A	0.9300
C11—C16	1.378 (4)	C16—C15	1.381 (4)
C11—C12	1.386 (4)	C16—H16A	0.9300
C17—C22	1.372 (4)	C13—H13A	0.9300

C17—C18	1.377 (4)	C6—H6A	0.9700
O4—C4	1.144 (3)	C6—H6B	0.9700
C7—N1	1.440 (3)	C10—H10A	0.9600
C7—H7A	0.9700	C10—H10B	0.9600
C7—H7B	0.9700	C10—H10C	0.9600
C22—C21	1.387 (5)	C21—H21A	0.9300
C22—H22A	0.9300	C15—H15A	0.9300
O1—C1	1.126 (4)	C8—H8A	0.9700
N1—C6	1.444 (4)	C8—H8B	0.9700
N1—C8	1.490 (4)		
C4—Fe1—C5	91.03 (13)	C7—N1—C6	112.7 (2)
C4—Fe1—P	98.50 (11)	C7—N1—C8	113.7 (2)
C5—Fe1—P	95.76 (11)	C6—N1—C8	115.3 (3)
C4—Fe1—S1	163.81 (11)	O4—C4—Fe1	179.0 (3)
C5—Fe1—S1	89.67 (9)	C13—C12—C11	121.0 (3)
P—Fe1—S1	97.52 (3)	C13—C12—H12A	119.5
C4—Fe1—S2	88.97 (10)	C11—C12—H12A	119.5
C5—Fe1—S2	159.03 (11)	C17—C18—C19	120.4 (3)
P—Fe1—S2	104.99 (3)	C17—C18—H18A	119.8
S1—Fe1—S2	84.65 (3)	C19—C18—H18A	119.8
C4—Fe1—Fe2	108.26 (10)	O1—C1—Fe2	179.5 (3)
C5—Fe1—Fe2	104.18 (10)	C20—C19—C18	121.1 (4)
P—Fe1—Fe2	145.99 (3)	C20—C19—H19A	119.5
S1—Fe1—Fe2	56.02 (2)	C18—C19—H19A	119.5
S2—Fe1—Fe2	56.20 (2)	C13—C14—C15	120.7 (3)
C3—Fe2—C2	91.87 (16)	C13—C14—H14A	119.7
C3—Fe2—C1	98.92 (15)	C15—C14—H14A	119.7
C2—Fe2—C1	103.33 (15)	C8—C9—C10	110.4 (4)
C3—Fe2—S1	86.31 (12)	C8—C9—H9A	109.6
C2—Fe2—S1	152.32 (11)	C10—C9—H9A	109.6
C1—Fe2—S1	104.24 (11)	C8—C9—H9B	109.6
C3—Fe2—S2	158.79 (11)	C10—C9—H9B	109.6
C2—Fe2—S2	87.26 (11)	H9A—C9—H9B	108.1
C1—Fe2—S2	101.90 (11)	O3—C3—Fe2	178.4 (4)
S1—Fe2—S2	84.73 (3)	C19—C20—C21	119.5 (4)
C3—Fe2—Fe1	103.04 (11)	C19—C20—H20A	120.2
C2—Fe2—Fe1	97.72 (11)	C21—C20—H20A	120.2
C1—Fe2—Fe1	148.90 (11)	C11—C16—C15	120.5 (3)
S1—Fe2—Fe1	56.14 (2)	C11—C16—H16A	119.8
S2—Fe2—Fe1	56.21 (2)	C15—C16—H16A	119.8
C17—P—C11	104.48 (13)	O2—C2—Fe2	176.7 (4)
C17—P—Fe1	113.19 (10)	C14—C13—C12	119.6 (3)
C11—P—Fe1	121.25 (10)	C14—C13—H13A	120.2
C17—P—H1	98.0 (10)	C12—C13—H13A	120.2
C11—P—H1	99.6 (10)	N1—C6—S2	113.6 (2)
Fe1—P—H1	117.0 (10)	N1—C6—H6A	108.8
C6—S2—Fe2	111.91 (10)	S2—C6—H6A	108.8
C6—S2—Fe1	110.18 (10)	N1—C6—H6B	108.8
Fe2—S2—Fe1	67.59 (3)	S2—C6—H6B	108.8

C7—S1—Fe2	112.84 (10)	H6A—C6—H6B	107.7
C7—S1—Fe1	109.50 (10)	C9—C10—H10A	109.5
Fe2—S1—Fe1	67.84 (3)	C9—C10—H10B	109.5
C16—C11—C12	118.5 (3)	H10A—C10—H10B	109.5
C16—C11—P	119.7 (2)	C9—C10—H10C	109.5
C12—C11—P	121.8 (2)	H10A—C10—H10C	109.5
C22—C17—C18	118.4 (3)	H10B—C10—H10C	109.5
C22—C17—P	120.5 (2)	C20—C21—C22	119.8 (4)
C18—C17—P	120.9 (2)	C20—C21—H21A	120.1
O5—C5—Fe1	178.7 (3)	C22—C21—H21A	120.1
N1—C7—S1	114.4 (2)	C14—C15—C16	119.8 (4)
N1—C7—H7A	108.7	C14—C15—H15A	120.1
S1—C7—H7A	108.7	C16—C15—H15A	120.1
N1—C7—H7B	108.7	C9—C8—N1	117.4 (3)
S1—C7—H7B	108.7	C9—C8—H8A	108.0
H7A—C7—H7B	107.6	N1—C8—H8A	108.0
C17—C22—C21	120.8 (3)	C9—C8—H8B	108.0
C17—C22—H22A	119.6	N1—C8—H8B	108.0
C21—C22—H22A	119.6	H8A—C8—H8B	107.2
C4—Fe1—Fe2—C3	−99.28 (16)	C4—Fe1—S1—Fe2	14.9 (4)
C5—Fe1—Fe2—C3	−3.37 (15)	C5—Fe1—S1—Fe2	107.42 (10)
P—Fe1—Fe2—C3	120.64 (12)	P—Fe1—S1—Fe2	−156.82 (3)
S1—Fe1—Fe2—C3	76.41 (12)	S2—Fe1—S1—Fe2	−52.37 (3)
S2—Fe1—Fe2—C3	−175.18 (12)	C17—P—C11—C16	67.9 (3)
C4—Fe1—Fe2—C2	−5.54 (16)	Fe1—P—C11—C16	−162.90 (19)
C5—Fe1—Fe2—C2	90.36 (15)	C17—P—C11—C12	−113.5 (3)
P—Fe1—Fe2—C2	−145.63 (12)	Fe1—P—C11—C12	15.8 (3)
S1—Fe1—Fe2—C2	170.14 (12)	C11—P—C17—C22	63.2 (3)
S2—Fe1—Fe2—C2	−81.44 (12)	Fe1—P—C17—C22	−70.7 (3)
C4—Fe1—Fe2—C1	126.9 (2)	C11—P—C17—C18	−121.4 (3)
C5—Fe1—Fe2—C1	−137.2 (2)	Fe1—P—C17—C18	104.7 (2)
P—Fe1—Fe2—C1	−13.2 (2)	C4—Fe1—C5—O5	−11 (15)
S1—Fe1—Fe2—C1	−57.39 (19)	P—Fe1—C5—O5	87 (15)
S2—Fe1—Fe2—C1	51.03 (19)	S1—Fe1—C5—O5	−175 (100)
C4—Fe1—Fe2—S1	−175.68 (11)	S2—Fe1—C5—O5	−101 (15)
C5—Fe1—Fe2—S1	−79.78 (10)	Fe2—Fe1—C5—O5	−120 (15)
P—Fe1—Fe2—S1	44.23 (5)	Fe2—S1—C7—N1	66.4 (2)
S2—Fe1—Fe2—S1	108.41 (4)	Fe1—S1—C7—N1	−7.0 (2)
C4—Fe1—Fe2—S2	75.90 (11)	C18—C17—C22—C21	−0.2 (5)
C5—Fe1—Fe2—S2	171.81 (10)	P—C17—C22—C21	175.3 (3)
P—Fe1—Fe2—S2	−64.18 (5)	S1—C7—N1—C6	−70.3 (3)
S1—Fe1—Fe2—S2	−108.41 (4)	S1—C7—N1—C8	156.2 (2)
C4—Fe1—P—C17	62.32 (15)	C5—Fe1—C4—O4	4(20)
C5—Fe1—P—C17	−29.59 (14)	P—Fe1—C4—O4	−92 (20)
S1—Fe1—P—C17	−120.01 (11)	S1—Fe1—C4—O4	97 (20)
S2—Fe1—P—C17	153.54 (11)	S2—Fe1—C4—O4	163 (20)
Fe2—Fe1—P—C17	−155.71 (11)	Fe2—Fe1—C4—O4	110 (20)
C4—Fe1—P—C11	−62.99 (15)	C16—C11—C12—C13	−0.1 (5)
C5—Fe1—P—C11	−154.90 (14)	P—C11—C12—C13	−178.8 (2)

S1—Fe1—P—C11	114.67 (11)	C22—C17—C18—C19	0.0 (5)
S2—Fe1—P—C11	28.22 (11)	P—C17—C18—C19	−175.5 (2)
Fe2—Fe1—P—C11	78.98 (12)	C3—Fe2—C1—O1	−5(51)
C3—Fe2—S2—C6	116.7 (3)	C2—Fe2—C1—O1	−99 (51)
C2—Fe2—S2—C6	−155.14 (16)	S1—Fe2—C1—O1	84 (51)
C1—Fe2—S2—C6	−52.10 (15)	S2—Fe2—C1—O1	171 (100)
S1—Fe2—S2—C6	51.37 (11)	Fe1—Fe2—C1—O1	130 (51)
Fe1—Fe2—S2—C6	103.67 (11)	C17—C18—C19—C20	0.4 (5)
C3—Fe2—S2—Fe1	13.1 (3)	C2—Fe2—C3—O3	−141 (12)
C2—Fe2—S2—Fe1	101.18 (12)	C1—Fe2—C3—O3	115 (12)
C1—Fe2—S2—Fe1	−155.77 (11)	S1—Fe2—C3—O3	11 (12)
S1—Fe2—S2—Fe1	−52.30 (3)	S2—Fe2—C3—O3	−54 (12)
C4—Fe1—S2—C6	140.93 (15)	Fe1—Fe2—C3—O3	−43 (12)
C5—Fe1—S2—C6	−128.9 (3)	C18—C19—C20—C21	−0.5 (6)
P—Fe1—S2—C6	42.41 (11)	C12—C11—C16—C15	−0.6 (4)
S1—Fe1—S2—C6	−53.97 (11)	P—C11—C16—C15	178.1 (2)
Fe2—Fe1—S2—C6	−106.17 (11)	C3—Fe2—C2—O2	80 (6)
C4—Fe1—S2—Fe2	−112.90 (11)	C1—Fe2—C2—O2	179 (100)
C5—Fe1—S2—Fe2	−22.7 (3)	S1—Fe2—C2—O2	−6(6)
P—Fe1—S2—Fe2	148.58 (3)	S2—Fe2—C2—O2	−79 (6)
S1—Fe1—S2—Fe2	52.21 (3)	Fe1—Fe2—C2—O2	−24 (6)
C3—Fe2—S1—C7	148.92 (15)	C15—C14—C13—C12	−0.7 (6)
C2—Fe2—S1—C7	−124.1 (3)	C11—C12—C13—C14	0.7 (5)
C1—Fe2—S1—C7	50.64 (15)	C7—N1—C6—S2	71.9 (3)
S2—Fe2—S1—C7	−50.33 (10)	C8—N1—C6—S2	−155.4 (2)
Fe1—Fe2—S1—C7	−102.69 (11)	Fe2—S2—C6—N1	−69.1 (2)
C3—Fe2—S1—Fe1	−108.40 (11)	Fe1—S2—C6—N1	4.0 (2)
C2—Fe2—S1—Fe1	−21.4 (2)	C19—C20—C21—C22	0.3 (7)
C1—Fe2—S1—Fe1	153.33 (11)	C17—C22—C21—C20	0.1 (6)
S2—Fe2—S1—Fe1	52.36 (3)	C13—C14—C15—C16	0.0 (6)
C4—Fe1—S1—C7	122.3 (4)	C11—C16—C15—C14	0.7 (5)
C5—Fe1—S1—C7	−145.10 (15)	C10—C9—C8—N1	179.3 (3)
P—Fe1—S1—C7	−49.34 (11)	C7—N1—C8—C9	63.0 (4)
S2—Fe1—S1—C7	55.12 (11)	C6—N1—C8—C9	−69.2 (4)
Fe2—Fe1—S1—C7	107.48 (11)		

Fig. 1

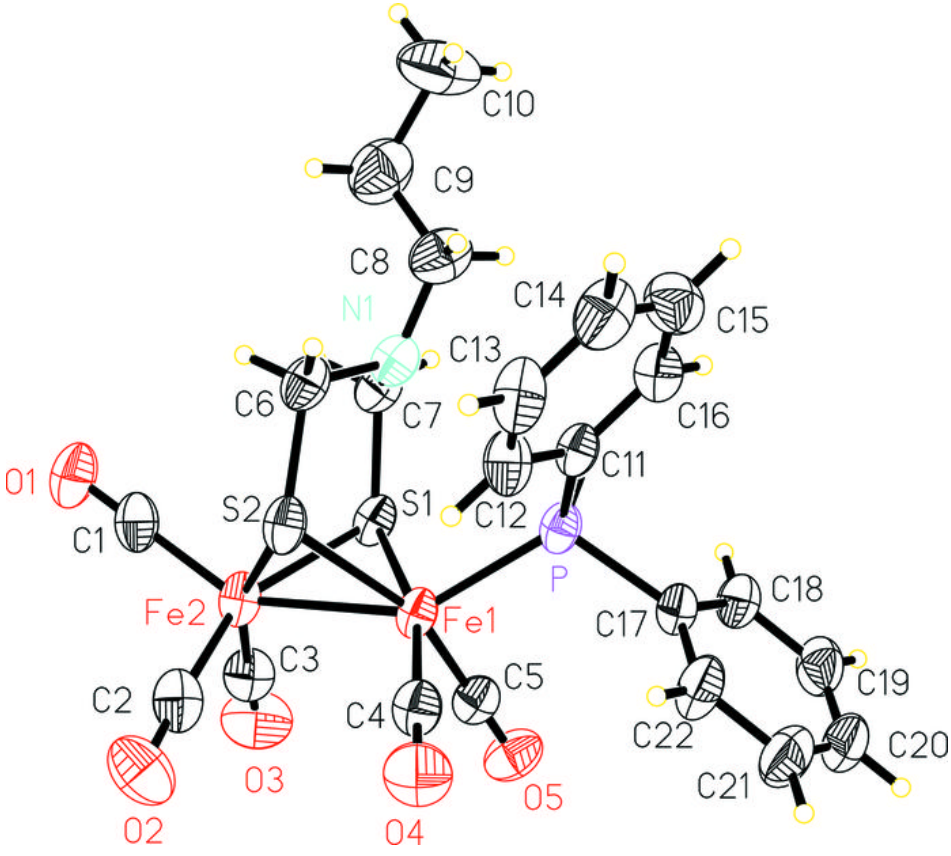

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Fig. 2

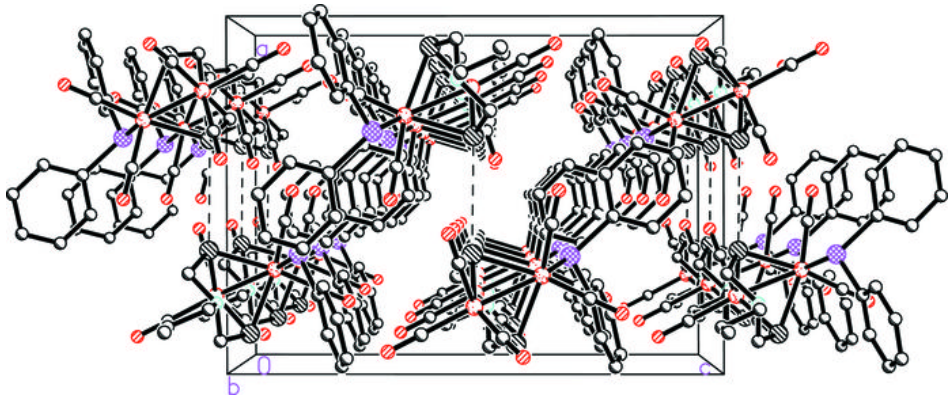
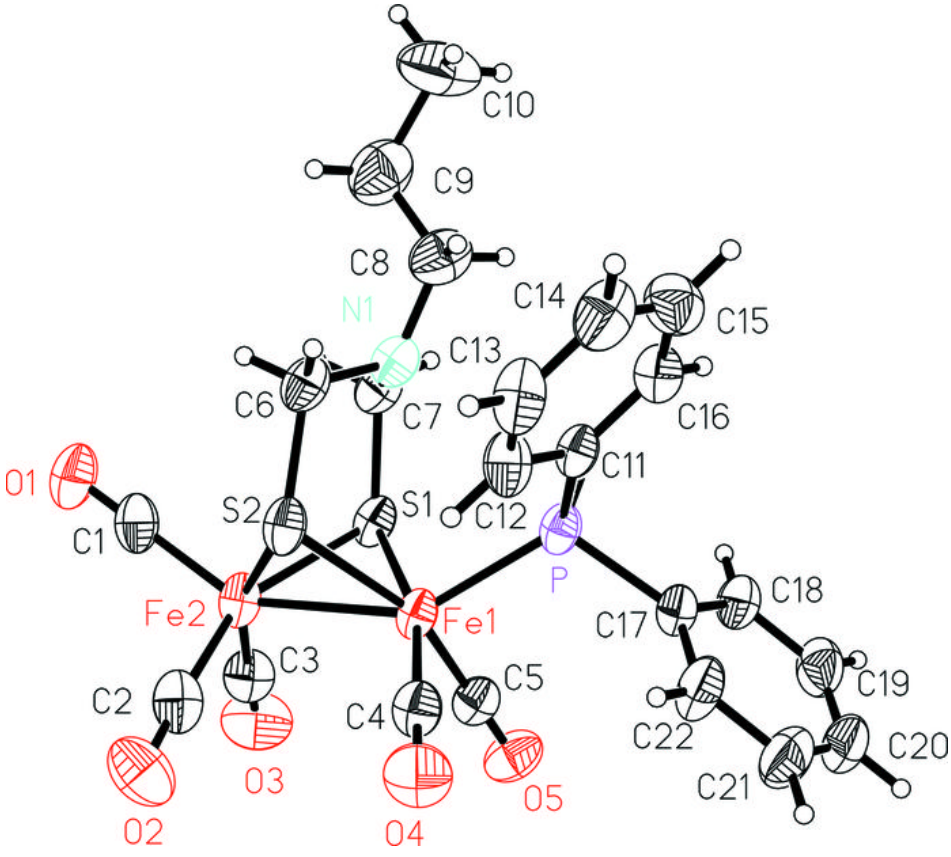


Fig. 3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