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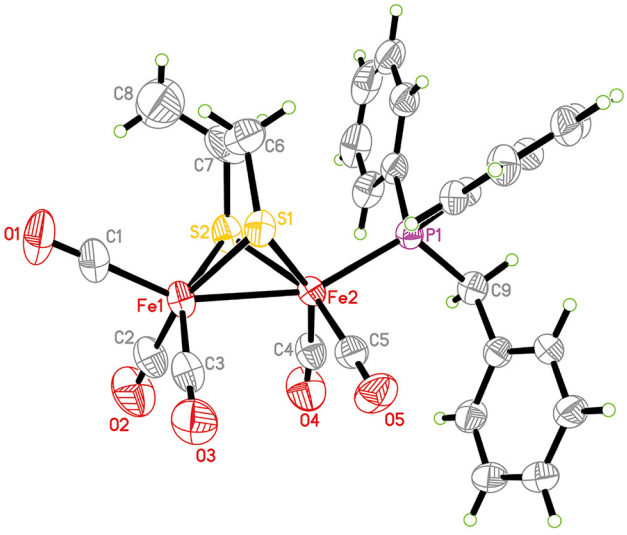
The crystal structure of tris(carbonyl)-bis(carbonyl)-[μ-propane-1,2- dithiolato]-(benzyldiphenylphosphine)diiron (*Fe—Fe*), $C_{27}H_{23}Fe_2O_5PS_2$



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Abstract

$C_{27}H_{23}Fe_2O_5PS_2$, monoclinic, $P2_1/c$ (no. 14), a = 11.3170(5) Å, b = 16.1774(8) Å, c = 15.2170(7) Å, β = 92.933(1)°, V = 2782.3(2) Å^3, Z = 4, $R_{gt}(F)$ = 0.0537, $wR_{ref}(F^2)$ = 0.1636, T = 296(2) K.

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The molecular structure is shown in the figure. Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

Table 1: Data collection and handling.

Crystal:	Red block
Size:	0.28 × 0.26 × 0.24 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ:	1.29 mm^{-1}
Diffractometer, scan mode:	Bruker D8 QUEST, ω
θ_{max}, completeness:	25.1°, 99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	55006, 4918, 0.043
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 3952
$N(param)_{refined}$:	335
Programs:	Bruker [1], Olex2 [2], SHELX [3, 4]

Source of material

To a solution of complex $[Fe_2(CO)_6\{\mu$–$SCH_2CH(CH_3)S\}]$ (1 mmol) and benzyldiphenylphosphine (1 mmol) in CH_2Cl_2 (10 mL) was added a solution of $Me_3NO{\cdot}2H_2O$ (1 mmol) in CH_3CN (10 mL). The solution was stirred for 1 h and the solvent was reduced by rotary evaporator. The title complex was obtained by TLC separation and the single crystals were collected from slow evaporation of CH_2Cl_2/isopropanol solution at 4°.

Experimental details

The structure was solved by Direct Methods with the SHELXS program. Hydrogen atoms were positioned geometrically (C–H = 0.93–0.98 Å). Their U_{iso} values were set to 1.2U_{eq} or 1.5U_{eq} of the parent atoms.

Comment

Over the past two decades, diiron propane-1,3-dithiolate complexes have been received much attention [5], nevertheless, diiron propane-1,2-dithiolate complexes have been received little attention [6–8].

The asymmetric unit of the title complex contains a diiron core ligated by a bridging propane-1,2-dithiolate, five carbonyls and a benzyldiphenylphosphine ligands. The orientation of the phosphine ligand is in an apical

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

Atom	x	y	z	U_{iso}*/U_{eq}
Fe1	0.48758 (6)	0.19616 (5)	0.81165 (5)	0.0596 (2)
Fe2	0.30929 (5)	0.18092 (4)	0.70930 (4)	0.0476 (2)
S1	0.31644 (11)	0.14035 (9)	0.84981 (8)	0.0617 (3)
S2	0.46509 (11)	0.09407 (9)	0.71136 (10)	0.0707 (4)
P1	0.16105 (10)	0.11101 (7)	0.64175 (7)	0.0491 (3)
O1	0.6462 (5)	0.1408 (4)	0.9564 (4)	0.1243 (19)
O2	0.6613 (5)	0.2688 (4)	0.7000 (4)	0.135 (2)
O3	0.4179 (5)	0.3563 (3)	0.8819 (3)	0.1078 (16)
O4	0.4108 (4)	0.2613 (4)	0.5605 (3)	0.1104 (17)
O5	0.1726 (4)	0.3239 (3)	0.7601 (3)	0.0965 (14)
C1	0.5868 (5)	0.1603 (4)	0.9000 (4)	0.0828 (17)
C2	0.5941 (5)	0.2409 (5)	0.7446 (4)	0.0854 (18)
C3	0.4463 (5)	0.2935 (4)	0.8558 (4)	0.0745 (15)
C4	0.3666 (5)	0.2292 (4)	0.6171 (3)	0.0714 (15)
C5	0.2245 (5)	0.2677 (3)	0.7377 (3)	0.0622 (12)
C6	0.3421 (7)	0.0276 (4)	0.8472 (5)	0.104 (2)
H6A	0.3777	0.0097	0.9033	0.125*
H6B	0.2670	−0.0008	0.8378	0.125*
C7	0.4225 (7)	0.0050 (4)	0.7746 (6)	0.108 (2)
H7	0.3782	−0.0323	0.7343	0.130*
C8	0.5182 (10)	−0.0450 (7)	0.8212 (7)	0.157 (3)
H8A	0.5748	−0.0085	0.8501	0.236*
H8B	0.5571	−0.0784	0.7793	0.236*
H8C	0.4841	−0.0800	0.8641	0.236*
C9	0.0958 (5)	0.1602 (3)	0.5416 (3)	0.0617 (12)
H9A	0.1549	0.1618	0.4976	0.074*
H9B	0.0307	0.1263	0.5185	0.074*
C10	0.0507 (4)	0.2472 (3)	0.5556 (3)	0.0576 (11)
C11	−0.0546 (4)	0.2612 (3)	0.5949 (3)	0.0631 (13)
H11	−0.1013	0.2168	0.6104	0.076*
C12	−0.0911 (5)	0.3408 (3)	0.6113 (4)	0.0743 (15)
H12	−0.1627	0.3492	0.6372	0.089*
C13	−0.0244 (5)	0.4069 (4)	0.5904 (4)	0.0801 (16)
H13	−0.0488	0.4602	0.6034	0.096*
C14	0.0800 (6)	0.3940 (4)	0.5497 (4)	0.0845 (18)
H14	0.1260	0.4388	0.5343	0.101*
C15	0.1160 (5)	0.3152 (4)	0.5320 (4)	0.0704 (15)
H15	0.1858	0.3072	0.5034	0.085*
C16	0.2047 (4)	0.0104 (3)	0.5995 (3)	0.0589 (12)
C17	0.1654 (5)	−0.0640 (3)	0.6293 (4)	0.0746 (15)
H17	0.1105	−0.0649	0.6727	0.090*
C18	0.2060 (7)	−0.1379 (4)	0.5960 (5)	0.096 (2)
H18	0.1794	−0.1881	0.6174	0.115*
C19	0.2862 (8)	−0.1362 (5)	0.5308 (6)	0.112 (3)
H19	0.3113	−0.1855	0.5063	0.134*
C20	0.3281 (6)	−0.0644 (5)	0.5025 (4)	0.096 (2)
H20	0.3842	−0.0644	0.4600	0.116*
C21	0.2891 (5)	0.0102 (4)	0.5355 (4)	0.0766 (16)
H21	0.3190	0.0598	0.5152	0.092*
C22	0.0277 (4)	0.0902 (3)	0.7011 (3)	0.0527 (11)
C23	−0.0656 (5)	0.0447 (3)	0.6618 (4)	0.0695 (14)
H23	−0.0588	0.0229	0.6058	0.083*
C24	−0.1673 (5)	0.0317 (4)	0.7054 (4)	0.0798 (17)
H24	−0.2283	0.0005	0.6790	0.096*
C25	−0.1794 (5)	0.0640 (4)	0.7861 (4)	0.0796 (16)
H25	−0.2489	0.0555	0.8148	0.095*
C26	−0.0893 (4)	0.1095 (4)	0.8260 (4)	0.0722 (14)
H26	−0.0979	0.1317	0.8817	0.087*
C27	0.0135 (4)	0.1220 (3)	0.7837 (3)	0.0569 (11)
H27	0.0745	0.1524	0.8114	0.068*

position of the distorted octahedral geometry of the Fe2 atom, is analogous to phosphine-containing compounds [9–13]. The Fe1–Fe2 bond length [2.4972(9) Å] is shorter than that of the parent complex $[Fe_2(CO)_6\{\mu$–$SCH_2CH(CH_3)S\}]$ [2.5196(7) Å] [14], inconsistent with the fact that the phosphine coordination will lengthen the Fe–Fe bond due to the phosphine ligand having stronger electron-donor than CO [15, 16]. Furthermore, the Fe1–Fe2 bond length is similar to other all-carbonyl analogues [17–19], and a directly related structure [22], but much shorter than those in natural enzymed [20, 21].

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