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Bis[dicarbonyl(η^5-cyclopentadienyl)ferrio]-(*N*,*N*-diisobutyldithiocarbamato)tin(II)

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Key indicators

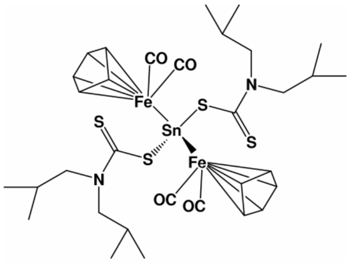
Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.015 Å
R factor = 0.054
wR factor = 0.153
Data-to-parameter ratio = 16.8

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

The title compound, $[\{CpFe(CO)_2\}_2Sn\{S_2CN(C_4H_9\text{-}i)_2\}_2]$ or $[SnFe_2(C_5H_5)_2(C_9H_{18}NS_2)_2(CO)_4]$, is obtained from the reaction of $[C_5H_5Fe(CO)_2]_2SnCl_2$ and potassium *N*,*N*-diisobutyldithiocarbamate in a mixed solvent (C_6H_6–THF 1:1). The crystal structure shows that the Sn atom is situated on a twofold rotation axis. An Fe—Sn—Fe cluster is present in the complex, the Sn and Fe atoms having coordination numbers 4 and 8, respectively. There are non-conventional intramolecular C—H···S and C—H···N hydrogen bonds in the structure.

Comment

Because of its varied uses and strong coordinating ability, the dithiocarbamate ligand has attracted the attention of many investigators. Examples of its use are as a chelating agent in the treatment of metal poisoning (Tandon *et al.*, 1996) and as a lubricant additive (Tanaka *et al.*, 1996). The dithiocarbamate group can coordinate in either monodentate or bidentate mode (Johnson *et al.*, 1969), the latter being much more common. In the present study, we have synthesized a new organotin–iron complex by the reaction of potassium *N*,*N*-diisobutyldithiocarbamate with dichlorobis[dicarbonyl(η^5-cyclopentadienyl)iron]tin and report its crystal structure here.



(I)

In the title complex, $[CpFe(CO)_2]_2Sn[S_2CN(C_4H_9\text{-}i)_2]_2$, (I), the Sn atom is situated on a twofold rotation axis. The coordination number of the Sn atom is four and that of the Fe atoms is eight. The Sn atom adopts a distorted tetrahedral configuration, the range of angles around Sn being 71.57 (9)–129.01 (6)°. The Sn—S distance is 2.577 (2) Å and the Sn—Fe distance is 2.5568 (14) Å. The C1—S1 and C1═S2 distances (Fig. 1) are 1.738 (7) and 1.682 (7) Å, respectively, indicating localization of the single and double bonds. A search of the Cambridge Structural Database (Allen, 2002) for the *N*,*N*-diisobutyldithiocarbamate ligand coordinated to any metal yielded only five hits. Of these, only one involved monodentate coordination, *viz.* bis(diisobutyldithiocarbamato)-

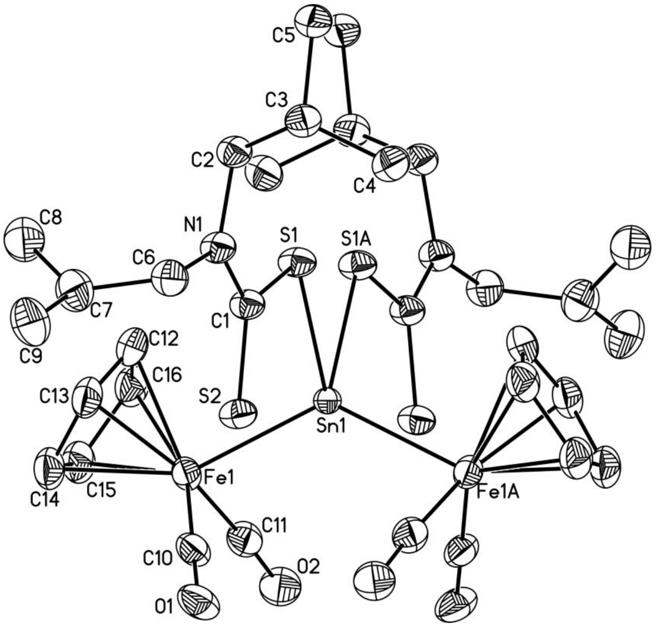


Figure 1
The molecular structure of the title compound. Ellipsoids are drawn at the 50% probability level. Atoms labelled with the suffix *A* are symmetry-related to those with no suffix.

bis(dimethylphenylphosphine)platinum(II) (Lin *et al.*, 1978). In the crystal structure, there are three non-conventional hydrogen bonds. Details are reported in Table 1.

Experimental

To $[C_5H_5Fe(CO)_2]_2SnCl_2$ (54 g, 1 mmol) and potassium *N,N*-diisobutyldithiocarbamate (2.1 mmol) were added 20 ml of mixed solvent (C_6H_6–THF 1:1). The mixture was stirred for *ca* 6 h under an argon atmosphere at room temperature, and the reaction was followed by thin-layer chromatography. The resulting KCl was filtered off, the remainder washed with a small amount of benzene and the filtrate was concentrated under reduced pressure at 318 K, until the volume was about 5 ml. An equivalent volume of petroleum ether was added. The solid product was collected in a Büchner funnel, washed with diethyl ether and petroleum ether, then recrystallized from benzene and dried under vacuum.

Crystal data

$[SnFe_2(C_5H_5)_2(C_9H_{18}NS_2)_2(CO)_4]$
$M_r = 881.34$
Monoclinic, $C2/c$
$a = 21.800$ (4) Å
$b = 11.587$ (2) Å
$c = 16.886$ (3) Å
$\beta = 112.17$ (3)°
$V = 3950.0$ (15) Å^3
$Z = 4$

$D_x = 1.482$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 25 reflections
$\theta = 2.0$–17.5°
$\mu = 1.60$ mm^{-1}
$T = 293$ (2) K
Block, black
0.3 × 0.2 × 0.2 mm

Data collection

Bruker *P*4 diffractometer
ω scans
Absorption correction: multi-scan (*XPREP* in *SHELXTL*; Bruker, 2000)
$T_{\min} = 0.69$, $T_{\max} = 0.72$
6972 measured reflections
3488 independent reflections
2132 reflections with $I > 2\sigma(I)$

$R_{\rm int} = 0.063$
$\theta_{\max} = 25.0°$
$h = -25 \rightarrow 24$
$k = 0 \rightarrow 13$
$l = 0 \rightarrow 20$
3 standard reflections every 97 reflections
frequency: 25 min
intensity decay: none

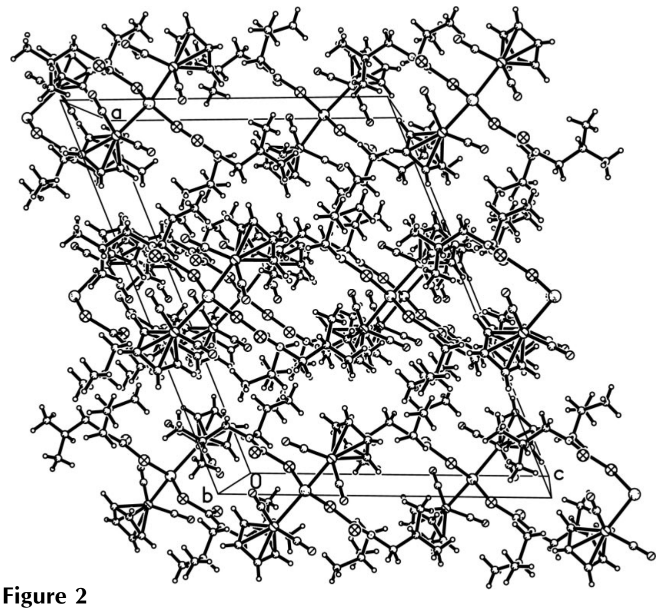


Figure 2
The crystal packing, viewed down the *b* axis.

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.054$
$wR(F^2) = 0.153$
$S = 1.05$
3488 reflections
208 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.07P)^2 + 1.66P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.78$ e Å^{-3}
$\Delta\rho_{\min} = -0.96$ e Å^{-3}

Table 1
Hydrogen-bonding geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C2—H2*B*···S1	0.97	2.49	3.009 (8)	114
C6—H6*A*···S2	0.97	2.63	3.026 (9)	105
C8—H8*C*···N1	0.96	2.50	2.960 (13)	109

All H atoms were positioned geometrically and refined isotropically, using a riding model. The C—H distances range from 0.96 to 0.98 Å.

Data collection: *XSCANS* (Bruker, 2000); cell refinement: *XSCANS*; data reduction: *SHELXTL* (Bruker, 2000); program(s) used to solve structure: *SHELXTL*; program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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