Acta Crystallographica Section E
Structure Reports
Online

ISSN 1600-5368

[1,2-Bis(diphenylphosphino)ethane]bis(*N*,*N*-dimethyldithiocarbamato)iron(III) tetrafluoroborate

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Received 23 May 2006
Accepted 24 May 2006

Key indicators

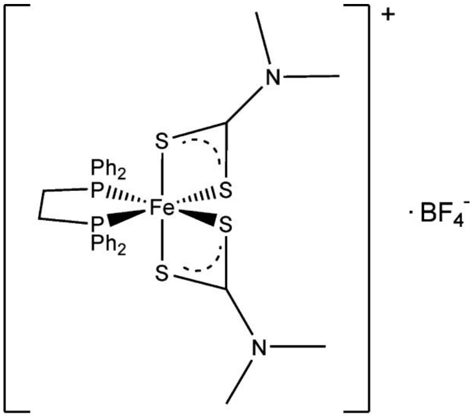
Single-crystal X-ray study
T = 203 K
Mean σ(C–C) = 0.006 Å
Disorder in solvent or counterion
R factor = 0.041
wR factor = 0.130
Data-to-parameter ratio = 17.6

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

The title compound, $[Fe(C_3H_6NS_2)_2(C_{26}H_{24}P_2)]BF_4$, contains a six-coordinate iron(III) cationic complex, with two bidentate dimethyldithiocarbamate and one 1,2-bis(diphenylphosphino)ethane ligands, and a disordered tetrafluoroborate anion. The cation possesses a crystallographic twofold rotation axis. The cations assemble in the crystal structure in two-dimensional layers *via* complementary C—H···π(phenyl) interactions with channels occupied by tetrafluorborate anions.

Comment

The ethyl analogue of the title complex, (I), was first reported in a reductive elimination reaction of an iron(IV) tris(diethyldithiocarbamate) complex with 1,2-bis(diphenylphosphino)ethane (McCleverty *et al.*, 1977). The molecular structure of (I) (Fig. 1) shows a near octahedral complex of iron(III) with four sulfur and two phosphorus ligands coordinated to the metal ion. The cation possesses a crystallographic twofold rotation axis. There are two different Fe—S bonds (Table 1) that are *trans* to either P or S ligands. Interestingly, the relative magnitude of these distances is opposite to that observed in an analogous cobalt(III) complex (Exarchos *et al.*, 2001; Pradhan *et al.*, 1999). The Fe—S bond lengths correspond to those found in low-spin tris(dialkyldithiocabamato)iron(III) complexes (Leipoldt & Coppens, 1973).



(I)

The cationic complexes assemble in the crystal structure in two-dimensional sheets (Fig. 2). Each interacts with a neighbouring complex *via* complementary phenyl C—H···π contacts [C24—H24···centroid(C22–C16) = 2.863 Å and C14—H14···centroid(C21–C26) = 3.108 Å]. There is an alternation of the complex with respect to the *b* axis within each sheet. The layers stack with phenyl rings nested between

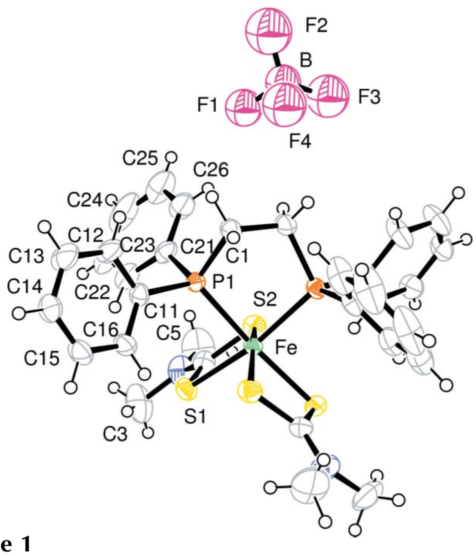


Figure 1
The structure of (I), showing 50% probability displacement ellipsoids for non-H atoms and H atoms as arbitary spheres. Only one of the disordered anion components is shown for clarity. Symmetry-related atoms are at ($1-x, y, \frac{1}{2}-z$).

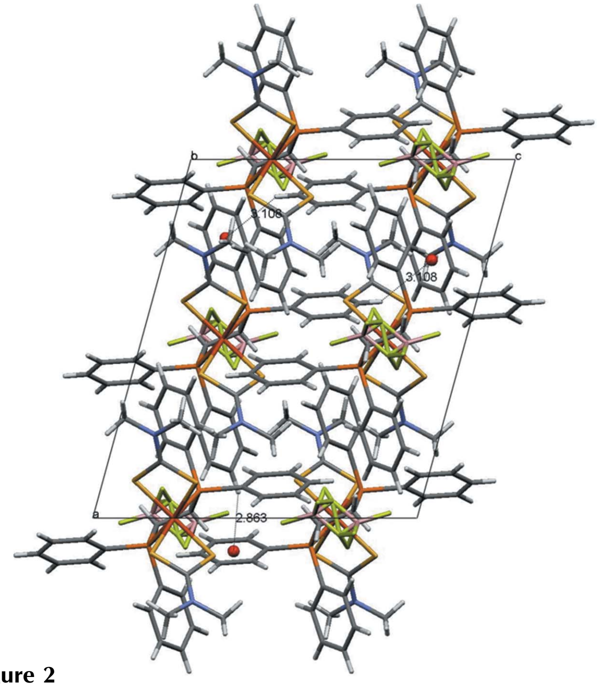


Figure 2
Stick illustration of the layer arrangement of the complex (I) in the crystal structure, with C—H$\cdots\pi$ interactions of the phenyl H atoms to phenyl rings on neighbouring complexes. C—H to phenyl-ring centroid distances are indicated. The view is along the *b* axis (Bruno *et al.*, 2002).

methyl groups of dithiocarbamate ligands. The disordered tetrafluoroborate anions occupy channels along the *c* axis formed by this assembly (Fig. 3).

Experimental

The title complex was prepared by reaction of 1,2-bis(diphenylphosphino)ethane with the iron(IV) complex tris(dimethyldithiocabamato)iron(IV) tetrafluoroborate (McCleverty *et al.*, 1977). Crystals were grown from a dichloromethane solution.

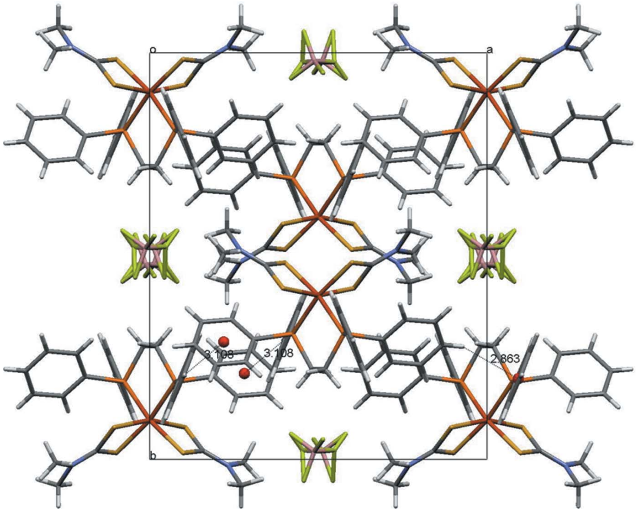


Figure 3
Disordered tetrafluoroborate anions filling channels formed between the layers. The view is along the *c* axis (Bruno *et al.*, 2002).

Crystal data

$[Fe(C_3H_6NS_2)_2(C_{26}H_{24}P_2)]BF_4$
$M_r = 781.47$
Monoclinic, $C2/c$
$a = 15.5061\ (1)$ Å
$b = 18.0353\ (2)$ Å
$c = 13.5470\ (2)$ Å
$\beta = 105.337\ (1)^\circ$
$V = 3653.59\ (7)$ Å^3

$Z = 4$
$D_x = 1.421$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 0.78$ mm^{-1}
$T = 203\ (2)$ K
Block, red
0.38 × 0.33 × 0.18 mm

Data collection

Siemens SMART CCD diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{\min} = 0.757$, $T_{\max} = 0.873$

10961 measured reflections
4038 independent reflections
3496 reflections with $I > 2\sigma(I)$
$\theta_{\max} = 27.5^\circ$
$R_{\rm int} = 0.017$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.041$
$wR(F^2) = 0.130$
$S = 0.89$
4038 reflections
230 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0678P)^2 + 17.7249P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 0.83$ e Å^{-3}
$\Delta\rho_{\min} = -0.57$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Fe—S1	2.2770 (8)	Fe—S2	2.3078 (7)
Fe—P1	2.3060 (8)		
S1—Fe—S1^i	100.38 (4)	S1^i—Fe—S2	93.54 (3)
S1—Fe—P1	87.77 (3)	P1^i—Fe—S2	96.23 (3)
P1^i—Fe—P1	85.18 (4)	P1—Fe—S2	95.56 (3)
S1—Fe—S2	76.10 (3)		

Symmetry code: (i) $-x+1, y, -z+\frac{1}{2}$.

H atoms were placed in calculated positions and refined using a riding model (C—H = 0.93–0.97 Å), with $U_{\rm iso}$(H) = 1.2 or 1.5 times

U_{eq}(C). The tetrafluoroborate anion is disordered over two sites related by ($2 - x$, y, $\frac{1}{2} - z$). Atoms B, F1, F2, F3 and F4 all have occupancies of 0.5.

Data collection: *SMART* (Siemens, 1995); cell refinement: *SAINT* (Siemens, 1995); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

This work was supported by the University of Auckland Research Committee. We thank Janet Hope for the preparation of the complex.

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