Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Bis[1,2-bis(ethoxycarbonyl)ethene-1,2-dithiolato-κ^2S,S']bis(η^5-pentamethylcyclopentadienyl)tetra-μ_3-sulfido-diiron(IV)diiron(III)(3 *Fe*—*Fe*)

Shohei Ito,[a] Nozomu Hisamichi,[a] Tsugiko Takase[b] and Shinji Inomata[a]*

[a]Faculty of Symbiotic Systems Science, Fukushima University, 1 Kanayagawa, Fukushima 960-1296, Japan, and [b]Center for Practical and Project-Based Learning, Cluster of Science and Technology, Fukushima University, 1 Kanayagawa, Fukushima 960-1296, Japan
Correspondence e-mail: inomata@sss.fukushima-u.ac.jp

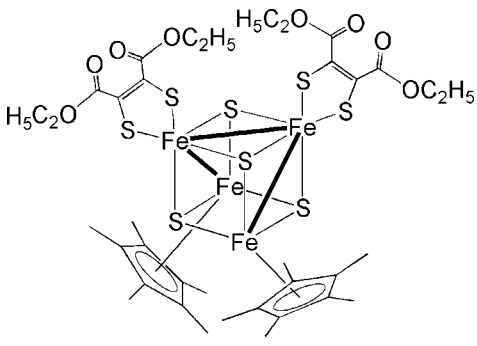
Received 12 February 2013; accepted 26 February 2013

Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.004 Å; R factor = 0.030; wR factor = 0.080; data-to-parameter ratio = 20.1.

The title compound, $[Fe_4(C_{10}H_{15})_2(C_8H_{10}O_4S_2)_2S_4]$, contains a twisted Fe_4S_4 cubane-like core. A twofold rotation axis passes through the Fe_4S_4 core, completing the coordination of the four Fe atoms with two pentamethylcyclopentadienyl ligands and two chelating dithiolate ligands. There are three short Fe—Fe and three long Fe···Fe contacts in the Fe_4S_4 core, suggesting bonding and non-bonding interactions, respectively. The Fe—S bonds in the Fe_4S_4 core range from 2.1523 (5) to 2.2667 (6) Å and are somewhat longer than the Fe—S bonds involving the dithiolate ligand.

Related literature

For details of the synthesis, see: Inomata *et al.* (1995). For related structures, see: Inomata *et al.* (1990, 1994). For general background to compounds with iron–sulfur cubane-type clusters, see: Holm (1977); Holm *et al.* (1990).



Experimental

Crystal data

$[Fe_4(C_{10}H_{15})_2(C_8H_{10}O_4S_2)_2S_4]$
M_r = 1090.65
Monoclinic, $C2/c$
a = 23.4532 (5) Å
b = 10.4466 (2) Å
c = 18.3113 (3) Å
β = 90.6186 (7)°
V = 4486.14 (15) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 1.69 mm^{-1}
T = 296 K
0.30 × 0.20 × 0.20 mm

Data collection

Rigaku R-AXIS RAPID diffractometer
Absorption correction: multi-scan (*REQAB*; Jacobson, 1998)
T_{min} = 0.560, T_{max} = 0.714
21429 measured reflections
5111 independent reflections
4694 reflections with $I > 2\sigma(I)$
R_{int} = 0.044

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.030
$wR(F^2)$ = 0.080
S = 1.06
5111 reflections
254 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.50 e Å^{-3}
$\Delta\rho_{min}$ = −0.33 e Å^{-3}

Table 1
Selected bond lengths (Å).

Fe1—Fe1^{i}	3.3743 (3)	Fe2—Fe2^{i}	2.7619 (3)
Fe1—Fe2	2.7253 (4)	Fe2—S1	2.2736 (5)
Fe1—Fe2^{i}	3.2683 (3)	Fe2—S2	2.1523 (5)
Fe1—S1	2.1956 (5)	Fe2—S2^{i}	2.2667 (6)
Fe1—S1^{i}	2.2551 (5)	Fe2—S3	2.1541 (5)
Fe1—S2	2.1749 (5)	Fe2—S4	2.1934 (6)

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

Data collection: *RAPID-AUTO* (Rigaku, 2006); cell refinement: *RAPID-AUTO*; data reduction: *RAPID-AUTO*; program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 2012); software used to prepare material for publication: *CrystalStructure* (Rigaku, 2006).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: WM2725).

References

Altomare, A., Burla, M. C., Camalli, M., Cascarano, G. L., Giacovazzo, C., Guagliardi, A., Moliterni, A. G. G., Polidori, G. & Spagna, R. (1999). *J. Appl. Cryst.* **32**, 115–119.
Farrugia, L. J. (2012). *J. Appl. Cryst.* **45**, 849–854.
Holm, R. H. (1977). *Acc. Chem. Res.* **12**, 427–434.
Holm, R. H., Ciurli, S. & Weigel, J. A. (1990). *Prog. Inorg. Chem.* **38**, 1–74.
Inomata, S., Hiyama, K., Tobita, H. & Ogino, H. (1994). *Inorg. Chem.* **33**, 5337–5342.
Inomata, S., Takano, H., Hiyama, K., Tobita, H. & Ogino, H. (1995). *Organometallics*, **14**, 2112–2114.
Inomata, S., Tobita, H. & Ogino, H. (1990). *J. Am. Chem. Soc.* **112**, 6145–6146.
Jacobson, R. (1998). *REQAB*. Private communication to the Rigaku Corporation, Tokyo, Japan.
Rigaku (2006). *CrystalStructure* and *RAPID-AUTO*. Rigaku Corporation, Tokyo, Japan.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

supporting information

Acta Cryst. (2013). E**69**, m181 [doi:10.1107/S1600536813005564]

Bis[1,2-bis(ethoxycarbonyl)ethene-1,2-dithiolato-κ^2S,S']bis(η^5-pentamethylcyclopentadienyl)tetra-μ_3-sulfido-diiron(IV)diiron(III)(3 *Fe*—*Fe*)

Shohei Ito, Nozomu Hisamichi, Tsugiko Takase and Shinji Inomata

S1. Comment

Iron-sulfur cubane-type clusters have extensively been investigated as model systems of metal-containing proteins (Holm, 1977; Holm *et al.*, 1990). Among these compounds, the Fe_4S_4 core is usually surrounded by the same supporting ligand (*L*) yielding a moiety $Fe_4S_4L_4$. However, mixed-ligand-type clusters are rather rare. Previously, we succeeded to prepare this type of iron-sulfur cluster from the reaction of $(C_5Me_5)_2Fe_2(CO)_4$ ($(Cp^*)_2Fe_2(CO)_4$) with S_8 and diphenylacetylene (Inomata *et al.*, 1990; 1994). One of the products was $[Fe_4(Cp^*)_2(Ph_2C_2S_2)_2(\mu_3\text{-}S)_4]$, in which two Cp* ligands and two diphenyldithiolate ligands are additionally bonded to the Fe_4S_4 core. In order to expand our research on this subject, we prepared a cluster containg bis(ethoxycarbonyl)dithiolate ligands instead of diphenyldithiolate ligands. Here we report the structural details of the title compound $[Fe_4(C_{10}H_{15})_2(C_8H_{10}O_4S_2)_2S_4]$ or $[Fe_4(Cp^*)_2\{(EtO_2C)_2C_2S_2)\}_2(\mu_3\text{-}S)_4]$, (I).

Compound (I) contains a twisted Fe_4S_4 cubane-like core surrounded by two Cp* ligands and two dithiolato ligands $\{(EtO_2C)_2C_2S_2\}$ (Fig. 1). A crystallographic twofold rotation axis passes through the Fe_4S_4 core and completes the coordination environment of all iron atoms. There are three iron—iron bonds of 2.7253 (4) and 2.7619 (3) Å (Table 1). The remaining three Fe···Fe distances are very long (3.2683 (3) and 3.3743 (3) Å), indicating no bonding interactions (Table 1). The iron—sulfur distances in the Fe_4S_4 core range from 2.1523 (5) to 2.2667 (6) Å and are normal values (Table 1). On the other hand, the distances between iron and sulfur in the dithiolato ligand are somewhat short (2.1541 (5) and 2.1934 (6) Å) (Table 1).

S2. Experimental

The title cluster compound was prepared according to the literature method (Inomata *et al.*, 1995) by using diethyl acetylenedicarboxylate instead of dimethyl acetylenedicarboxylate.

S3. Refinement

All hydrogen atoms were placed in calculated positions with C—H distances of 0.96 Å for H atoms on methyl groups and 0.97 Å for those on methylene groups. The U_{iso}(H) values were fixed at 1.2 times the U_{eq}(C) values of the carbon atoms to which they are covalently bonded.

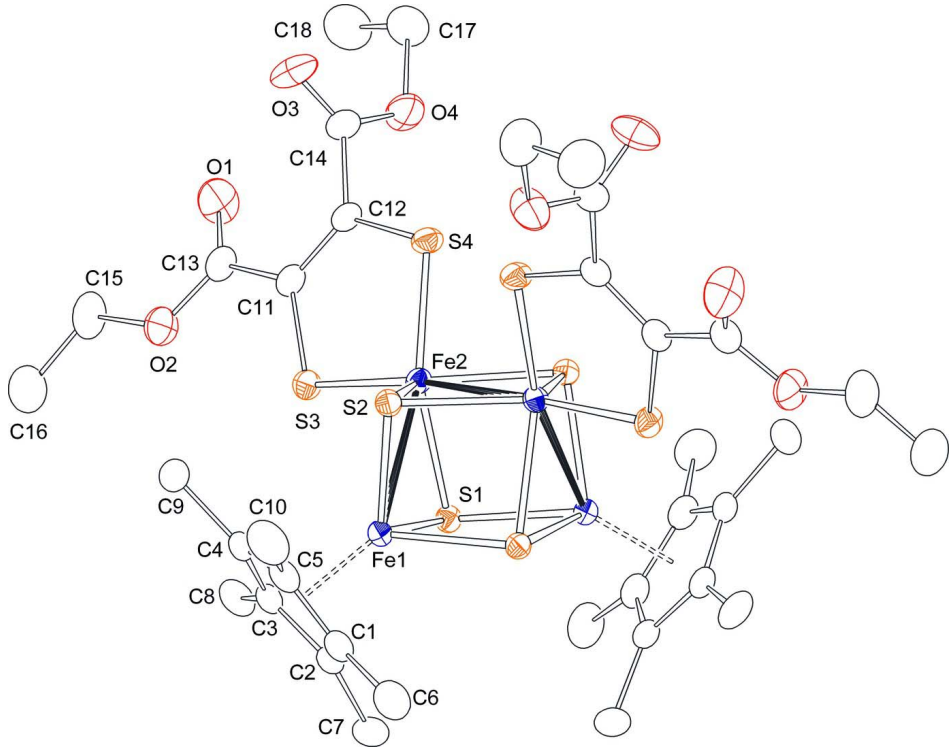


Figure 1

The molecular structure of the title compound, with atom labels and displacement ellipsoids at the 30% probability level. All hydrogen atoms were omitted for clarity. Solid lines indicate short Fe—Fe contacts.

Bis[1,2-bis(ethoxycarbonyl)ethene-1,2-dithiolato-κ^2S,S']bis(η^5-pentamethylcyclopentadienyl)tetra-μ_3-sulfido-diiron(IV)diiron(III)(3 *Fe*—*Fe*)

Crystal data

$[Fe_4(C_{10}H_{15})_2(C_8H_{10}O_4S_2)_2S_4]$
M_r = 1090.65
Monoclinic, *C*2/*c*
Hall symbol: -C 2yc
a = 23.4532 (5) Å
b = 10.4466 (2) Å
c = 18.3113 (3) Å
β = 90.6186 (7)°
V = 4486.14 (15) Å^3
Z = 4
F(000) = 2248.00
D_x = 1.615 Mg m^{-3}
Mo *K*α radiation, λ = 0.71075 Å
Cell parameters from 18942 reflections
θ = 3.1–27.5°
μ = 1.69 mm^{-1}
T = 296 K
Block, black
0.30 × 0.20 × 0.20 mm

Data collection

Rigaku R-AXIS RAPID diffractometer
Detector resolution: 10.00 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (*REQAB*; Jacobson, 1998)
T_{min} = 0.560, T_{max} = 0.714
21429 measured reflections
5111 independent reflections
4694 reflections with $I > 2\sigma(I)$
R_{int} = 0.044
θ_{max} = 27.5°
h = −30→30
k = −13→13
l = −23→23

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.030$
$wR(F^2) = 0.080$
$S = 1.06$
5111 reflections
254 parameters

H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.037P)^2 + 5.9377P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.002$
$\Delta\rho_{max} = 0.50$ e Å^{-3}
$\Delta\rho_{min} = -0.33$ e Å^{-3}

Special details

Refinement. Refinement was performed using all reflections. The weighted *R*-factor (*wR*) and goodness of fit (*S*) are based on F^2. *R*-factor (gt) are based on *F*. The threshold expression of $F^2 > 2.0\ \sigma(F^2)$ is used only for calculating *R*-factor (gt).

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
Fe1	0.512641 (12)	0.27575 (3)	0.159470 (14)	0.02745 (7)
Fe2	0.449295 (12)	0.07723 (3)	0.210955 (14)	0.02769 (7)
S1	0.43972 (2)	0.29022 (4)	0.23321 (3)	0.02894 (11)
S2	0.53399 (2)	0.07354 (4)	0.16691 (3)	0.03038 (11)
S3	0.38310 (2)	0.09718 (5)	0.12928 (3)	0.03767 (13)
S4	0.42061 (2)	−0.11994 (5)	0.22914 (3)	0.03867 (13)
O1	0.25875 (9)	−0.1373 (2)	0.08613 (13)	0.0745 (6)
O2	0.29459 (8)	0.02895 (19)	0.02550 (11)	0.0590 (4)
O3	0.33671 (9)	−0.35409 (19)	0.13925 (13)	0.0668 (5)
O4	0.29995 (10)	−0.2666 (2)	0.23969 (11)	0.0675 (5)
C1	0.56426 (10)	0.4192 (2)	0.10976 (12)	0.0416 (5)
C2	0.50644 (10)	0.4620 (2)	0.11373 (11)	0.0382 (4)
C3	0.47177 (10)	0.3774 (2)	0.07124 (11)	0.0388 (4)
C4	0.50767 (12)	0.2819 (2)	0.04178 (11)	0.0414 (5)
C5	0.56502 (11)	0.3076 (2)	0.06575 (12)	0.0442 (5)
C6	0.61503 (13)	0.4854 (3)	0.14245 (16)	0.0642 (7)
C7	0.48533 (15)	0.5815 (2)	0.14991 (15)	0.0594 (7)
C8	0.40936 (12)	0.3974 (2)	0.05670 (16)	0.0555 (6)
C9	0.48970 (16)	0.1760 (2)	−0.00894 (14)	0.0658 (8)
C10	0.61697 (14)	0.2362 (3)	0.04239 (17)	0.0709 (9)
C11	0.34615 (9)	−0.0459 (2)	0.12687 (12)	0.0364 (4)
C12	0.36250 (9)	−0.1409 (2)	0.17332 (12)	0.0351 (4)
C13	0.29508 (10)	−0.0580 (2)	0.07860 (13)	0.0425 (5)
C14	0.33153 (10)	−0.2667 (2)	0.18025 (14)	0.0430 (5)
C15	0.24678 (14)	0.0240 (3)	−0.02537 (17)	0.0716 (8)
C16	0.2579 (2)	0.1231 (4)	−0.0829 (2)	0.0961 (13)
C17	0.26445 (15)	−0.3807 (3)	0.25262 (19)	0.0764 (9)
C18	0.20954 (15)	−0.3640 (3)	0.2128 (2)	0.0809 (9)
H1	0.6029	0.5612	0.1675	0.077*
H2	0.6410	0.5084	0.1044	0.077*
H3	0.6338	0.4290	0.1764	0.077*
H4	0.5163	0.6223	0.1754	0.071*
H5	0.4702	0.6387	0.1136	0.071*

H6	0.4560	0.5599	0.1839	0.071*
H7	0.3950	0.3287	0.0269	0.067*
H8	0.4038	0.4772	0.0317	0.067*
H9	0.3894	0.3990	0.1022	0.067*
H10	0.5225	0.1265	−0.0222	0.079*
H11	0.4725	0.2119	−0.0521	0.079*
H12	0.4627	0.1218	0.0151	0.079*
H13	0.6502	0.2752	0.0638	0.085*
H14	0.6197	0.2389	−0.0099	0.085*
H15	0.6144	0.1488	0.0582	0.085*
H16	0.2115	0.0425	−0.0004	0.086*
H17	0.2439	−0.0604	−0.0473	0.086*
H18	0.2415	0.2033	−0.0683	0.115*
H19	0.2409	0.0963	−0.1284	0.115*
H20	0.2982	0.1331	−0.0889	0.115*
H21	0.2576	−0.3905	0.3045	0.092*
H22	0.2838	−0.4567	0.2353	0.092*
H23	0.1826	−0.3224	0.2440	0.097*
H24	0.1950	−0.4463	0.1985	0.097*
H25	0.2155	−0.3126	0.1701	0.097*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Fe1	0.03035 (15)	0.02725 (14)	0.02472 (14)	0.00053 (10)	−0.00037 (11)	0.00188 (10)
Fe2	0.02636 (14)	0.02784 (15)	0.02878 (14)	−0.00189 (10)	−0.00376 (10)	−0.00136 (10)
S1	0.0284 (2)	0.0298 (2)	0.0286 (2)	0.00303 (17)	−0.00230 (18)	−0.00054 (17)
S2	0.0330 (2)	0.0297 (2)	0.0284 (2)	0.00387 (18)	0.00056 (18)	−0.00021 (17)
S3	0.0369 (2)	0.0381 (2)	0.0377 (2)	−0.0058 (2)	−0.0122 (2)	0.0029 (2)
S4	0.0398 (2)	0.0289 (2)	0.0470 (2)	−0.0048 (2)	−0.0122 (2)	0.0007 (2)
O1	0.0465 (10)	0.0993 (16)	0.0771 (13)	−0.0314 (11)	−0.0221 (9)	0.0159 (12)
O2	0.0545 (10)	0.0647 (11)	0.0571 (10)	−0.0097 (9)	−0.0274 (8)	0.0042 (9)
O3	0.0607 (12)	0.0478 (10)	0.0923 (15)	−0.0169 (9)	0.0138 (10)	−0.0262 (10)
O4	0.0772 (14)	0.0671 (12)	0.0584 (11)	−0.0350 (11)	0.0157 (10)	−0.0072 (9)
C1	0.0466 (12)	0.0448 (12)	0.0332 (10)	−0.0098 (9)	0.0023 (9)	0.0112 (8)
C2	0.0525 (12)	0.0319 (9)	0.0299 (9)	−0.0015 (9)	−0.0011 (8)	0.0076 (7)
C3	0.0481 (12)	0.0378 (10)	0.0305 (9)	−0.0012 (9)	−0.0051 (8)	0.0094 (8)
C4	0.0636 (14)	0.0360 (10)	0.0247 (9)	−0.0021 (9)	0.0008 (9)	0.0052 (7)
C5	0.0504 (13)	0.0483 (12)	0.0341 (10)	0.0047 (10)	0.0125 (9)	0.0121 (9)
C6	0.0592 (16)	0.0761 (19)	0.0573 (15)	−0.0296 (15)	−0.0040 (13)	0.0159 (14)
C7	0.092 (2)	0.0336 (12)	0.0527 (14)	0.0044 (12)	0.0071 (14)	0.0020 (10)
C8	0.0519 (14)	0.0576 (15)	0.0567 (14)	0.0024 (12)	−0.0162 (12)	0.0211 (12)
C9	0.116 (2)	0.0464 (14)	0.0354 (12)	−0.0124 (15)	0.0032 (14)	−0.0037 (10)
C10	0.072 (2)	0.080 (2)	0.0621 (17)	0.0207 (16)	0.0324 (15)	0.0133 (15)
C11	0.0296 (9)	0.0423 (11)	0.0371 (10)	−0.0047 (8)	−0.0029 (8)	−0.0075 (8)
C12	0.0305 (9)	0.0353 (10)	0.0394 (10)	−0.0050 (8)	−0.0013 (8)	−0.0080 (8)
C13	0.0338 (11)	0.0514 (12)	0.0420 (11)	−0.0028 (9)	−0.0055 (9)	−0.0085 (9)
C14	0.0380 (11)	0.0391 (11)	0.0518 (12)	−0.0085 (9)	−0.0034 (9)	−0.0045 (9)

C15	0.0638 (18)	0.093 (2)	0.0575 (16)	0.0007 (16)	−0.0333 (14)	−0.0024 (16)
C16	0.123 (3)	0.090 (2)	0.074 (2)	0.005 (2)	−0.046 (2)	0.006 (2)
C17	0.076 (2)	0.081 (2)	0.0722 (19)	−0.0367 (18)	0.0011 (17)	0.0134 (17)
C18	0.0600 (19)	0.093 (2)	0.090 (2)	−0.0129 (18)	0.0156 (17)	0.012 (2)

Geometric parameters (Å, º)

Fe1—Fe1^{i}	3.3743 (3)	C4—C9	1.502 (3)
Fe1—Fe2	2.7253 (4)	C5—C10	1.495 (4)
Fe1—Fe2^{i}	3.2683 (3)	C11—C12	1.360 (3)
Fe1—S1	2.1956 (5)	C11—C13	1.486 (3)
Fe1—S1^{i}	2.2551 (5)	C12—C14	1.508 (3)
Fe1—S2	2.1749 (5)	C15—C16	1.503 (5)
Fe1—C1	2.136 (2)	C17—C18	1.483 (5)
Fe1—C2	2.122 (2)	C6—H1	0.960
Fe1—C3	2.150 (2)	C6—H2	0.960
Fe1—C4	2.158 (2)	C6—H3	0.960
Fe1—C5	2.147 (2)	C7—H4	0.960
Fe2—Fe2^{i}	2.7619 (3)	C7—H6	0.960
Fe2—S1	2.2736 (5)	C7—H5	0.960
Fe2—S2	2.1523 (5)	C8—H7	0.960
Fe2—S2^{i}	2.2667 (6)	C8—H8	0.960
Fe2—S3	2.1541 (5)	C8—H9	0.960
Fe2—S4	2.1934 (6)	C9—H10	0.960
S3—C11	1.728 (2)	C9—H11	0.960
S4—C12	1.709 (2)	C9—H12	0.960
O1—C13	1.198 (3)	C10—H13	0.960
O2—C13	1.330 (3)	C10—H14	0.960
O2—C15	1.451 (3)	C10—H15	0.960
O3—C14	1.189 (3)	C15—H16	0.970
O4—C14	1.323 (3)	C15—H17	0.970
O4—C17	1.474 (4)	C16—H18	0.960
C1—C2	1.431 (3)	C16—H19	0.960
C1—C5	1.417 (3)	C16—H20	0.960
C1—C6	1.496 (3)	C17—H21	0.970
C2—C3	1.426 (3)	C17—H22	0.970
C2—C7	1.500 (3)	C18—H23	0.960
C3—C4	1.416 (3)	C18—H24	0.960
C3—C8	1.500 (3)	C18—H25	0.960
C4—C5	1.435 (3)		
Fe1···Fe1^{i}	3.3743 (3)	Fe1···Fe2	2.7253 (4)
Fe2···Fe2^{i}	2.7619 (3)	Fe1···Fe2^{i}	3.2683 (3)
Fe1^{i}···Fe2^{i}	2.7253 (4)	Fe1^{i}···Fe2	3.2683 (3)
Fe2—Fe1—S1	53.736 (13)	C2—C3—C4	107.8 (2)
Fe2—Fe1—S1^{i}	90.938 (15)	C2—C3—C8	124.1 (2)
Fe2—Fe1—S2	50.595 (15)	C4—C3—C8	127.9 (2)

Fe2—Fe1—C1	174.13 (6)	Fe1—C4—C3	70.52 (11)
Fe2—Fe1—C2	143.01 (6)	Fe1—C4—C5	70.12 (12)
Fe2—Fe1—C3	113.29 (6)	Fe1—C4—C9	127.37 (16)
Fe2—Fe1—C4	110.15 (6)	C3—C4—C5	108.14 (19)
Fe2—Fe1—C5	135.48 (6)	C3—C4—C9	126.2 (2)
S1—Fe1—S1^{i}	80.87 (2)	C5—C4—C9	125.6 (2)
S1—Fe1—S2	102.04 (2)	Fe1—C5—C1	70.25 (13)
S1—Fe1—C1	131.50 (6)	Fe1—C5—C4	70.93 (13)
S1—Fe1—C2	97.42 (6)	Fe1—C5—C10	128.82 (18)
S1—Fe1—C3	94.83 (6)	C1—C5—C4	108.0 (2)
S1—Fe1—C4	125.31 (7)	C1—C5—C10	126.0 (2)
S1—Fe1—C5	159.56 (6)	C4—C5—C10	125.7 (2)
S1^{i}—Fe1—S2	84.20 (2)	S3—C11—C12	118.52 (16)
S1^{i}—Fe1—C1	92.67 (6)	S3—C11—C13	119.31 (16)
S1^{i}—Fe1—C2	108.28 (5)	C12—C11—C13	122.0 (2)
S1^{i}—Fe1—C3	146.55 (6)	S4—C12—C11	119.90 (16)
S1^{i}—Fe1—C4	152.77 (7)	S4—C12—C14	116.30 (16)
S1^{i}—Fe1—C5	113.81 (6)	C11—C12—C14	123.80 (19)
S2—Fe1—C1	125.22 (6)	O1—C13—O2	123.8 (2)
S2—Fe1—C2	158.40 (6)	O1—C13—C11	124.1 (2)
S2—Fe1—C3	128.84 (6)	O2—C13—C11	112.2 (2)
S2—Fe1—C4	95.83 (6)	O3—C14—O4	125.6 (2)
S2—Fe1—C5	93.85 (6)	O3—C14—C12	124.3 (2)
C1—Fe1—C2	39.26 (8)	O4—C14—C12	110.0 (2)
C1—Fe1—C3	65.39 (8)	O2—C15—C16	106.7 (2)
C1—Fe1—C4	65.05 (8)	O4—C17—C18	108.4 (2)
C1—Fe1—C5	38.65 (8)	C1—C6—H1	109.5
C2—Fe1—C3	38.99 (8)	C1—C6—H2	109.5
C2—Fe1—C4	64.89 (8)	C1—C6—H3	109.5
C2—Fe1—C5	65.17 (8)	H1—C6—H2	109.5
C3—Fe1—C4	38.37 (8)	H1—C6—H3	109.5
C3—Fe1—C5	64.99 (8)	H2—C6—H3	109.5
C4—Fe1—C5	38.95 (9)	C2—C7—H4	109.5
Fe1—Fe2—Fe2^{i}	73.111 (11)	C2—C7—H6	109.5
Fe1—Fe2—S1	51.138 (15)	C2—C7—H5	109.5
Fe1—Fe2—S2	51.334 (14)	H4—C7—H6	109.5
Fe1—Fe2—S2^{i}	105.375 (16)	H4—C7—H5	109.5
Fe1—Fe2—S3	94.456 (17)	H6—C7—H5	109.5
Fe1—Fe2—S4	159.452 (19)	C3—C8—H7	109.5
Fe2^{i}—Fe2—S1	89.620 (16)	C3—C8—H8	109.5
Fe2^{i}—Fe2—S2	53.196 (16)	C3—C8—H9	109.5
Fe2^{i}—Fe2—S2^{i}	49.487 (14)	H7—C8—H8	109.5
Fe2^{i}—Fe2—S3	165.837 (19)	H7—C8—H9	109.5
Fe2^{i}—Fe2—S4	100.674 (17)	H8—C8—H9	109.5
S1—Fe2—S2	100.26 (2)	C4—C9—H10	109.5
S1—Fe2—S2^{i}	81.730 (19)	C4—C9—H11	109.5
S1—Fe2—S3	87.59 (2)	C4—C9—H12	109.5
S1—Fe2—S4	149.40 (2)	H10—C9—H11	109.5

S2—Fe2—S2^{i}	102.65 (2)	H10—C9—H12	109.5
S2—Fe2—S3	113.75 (2)	H11—C9—H12	109.5
S2—Fe2—S4	109.02 (2)	C5—C10—H13	109.5
S2^{i}—Fe2—S3	143.35 (2)	C5—C10—H14	109.5
S2^{i}—Fe2—S4	83.36 (2)	C5—C10—H15	109.5
S3—Fe2—S4	88.64 (2)	H13—C10—H14	109.5
Fe1—S1—Fe1^{i}	98.59 (2)	H13—C10—H15	109.5
Fe1—S1—Fe2	75.126 (18)	H14—C10—H15	109.5
Fe1^{i}—S1—Fe2	92.386 (19)	O2—C15—H16	110.4
Fe1—S2—Fe2	78.071 (18)	O2—C15—H17	110.4
Fe1—S2—Fe2^{i}	94.73 (2)	C16—C15—H16	110.4
Fe2—S2—Fe2^{i}	77.32 (2)	C16—C15—H17	110.4
Fe2—S3—C11	106.95 (7)	H16—C15—H17	108.6
Fe2—S4—C12	105.87 (7)	C15—C16—H18	109.5
C13—O2—C15	116.4 (2)	C15—C16—H19	109.5
C14—O4—C17	117.0 (2)	C15—C16—H20	109.5
Fe1—C1—C2	69.86 (12)	H18—C16—H19	109.5
Fe1—C1—C5	71.10 (13)	H18—C16—H20	109.5
Fe1—C1—C6	127.30 (17)	H19—C16—H20	109.5
C2—C1—C5	107.7 (2)	O4—C17—H21	110.0
C2—C1—C6	125.9 (2)	O4—C17—H22	110.0
C5—C1—C6	126.4 (2)	C18—C17—H21	110.0
Fe1—C2—C1	70.88 (12)	C18—C17—H22	110.0
Fe1—C2—C3	71.55 (12)	H21—C17—H22	108.4
Fe1—C2—C7	127.60 (15)	C17—C18—H23	109.5
C1—C2—C3	108.28 (18)	C17—C18—H24	109.5
C1—C2—C7	126.9 (2)	C17—C18—H25	109.5
C3—C2—C7	124.6 (2)	H23—C18—H24	109.5
Fe1—C3—C2	69.46 (11)	H23—C18—H25	109.5
Fe1—C3—C4	71.12 (12)	H24—C18—H25	109.5
Fe1—C3—C8	128.94 (16)		

Symmetry code: (i) $-x+1, y, -z+1/2$.