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Octacarbonyl(5-methoxy-2,3-dihydro-1*H*-benzimidazol-2-yl)di-μ_3-sulfido-diiron(I)iron(II)(2 *Fe*—*Fe*)

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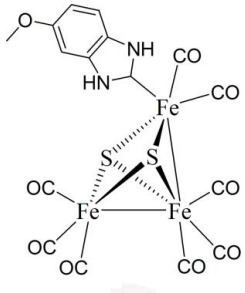
Key indicators: single-crystal X-ray study; T = 273 K; mean σ(C–C) = 0.004 Å; R factor = 0.027; wR factor = 0.070; data-to-parameter ratio = 14.7.

The title compound, $[Fe_3(C_8H_8N_2O)S_2(CO)_8]$, was prepared by the direct reaction of $Fe_3(CO)_{12}$ and 5-methoxy-1*H*-benzoimidazole-2-thiol in tetrahydrofuran. Desulfurization took place readily to form a sulfide carbonyl cluster. The molecule contains a triangle consisting of three Fe atoms capped by two S atoms above and below. There are two Fe—Fe bonds [2.6322 (5) and 2.5582 (5) Å] in the triangle; the length of the third edge [3.3987 (5) Å] is too long to represent an Fe—Fe bond.

Related literature

For related literature, see: Adams & Wang (1985); Adams & Yang (1983); Bard *et al.* (1985); Benoit *et al.* (1982); Weininger & Stermitz (1984).



Experimental

Crystal data

$[Fe_3(C_8H_8N_2O)S_2(CO)_8]$
M_r = 603.91
Triclinic, $P\overline{1}$
a = 9.0068 (4) Å
b = 11.4504 (5) Å
c = 12.5976 (6) Å
α = 112.332 (3)°
β = 108.728 (3)°
γ = 96.229 (3)°
V = 1098.0 (1) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 2.20 mm^{-1}
T = 273 (2) K
0.25 × 0.20 × 0.10 mm

Data collection

Bruker SMART APEXII CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 1997)
T_{min} = 0.610, T_{max} = 0.810
8226 measured reflections
4253 independent reflections
3584 reflections with $I > 2\sigma(I)$
R_{int} = 0.019

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.028
$wR(F^2)$ = 0.070
S = 1.05
4253 reflections
289 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.28 e Å^{-3}
$\Delta\rho_{min}$ = −0.30 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Fe2—S2	2.2505 (7)	Fe1—S1	2.2294 (6)
Fe2—S1	2.2621 (7)	Fe1—S2	2.2337 (7)
Fe2—Fe3	2.5582 (5)	Fe3—S1	2.2415 (6)
Fe2—Fe1	2.6322 (5)	Fe3—S2	2.2455 (6)
Fe1—C9	1.914 (2)		
C9—Fe1—Fe2	138.68 (7)	Fe3—S2—Fe2	69.362 (19)
C6—Fe3—Fe2	145.32 (10)	Fe1—S1—Fe3	98.96 (2)
Fe1—S2—Fe3	98.71 (2)	Fe1—S1—Fe2	71.75 (2)
Fe1—S2—Fe2	71.89 (2)	Fe3—S1—Fe2	69.23 (2)

Data collection: *APEX2* (Bruker, 2001); cell refinement: *SAINT-Plus* (Bruker, 2001); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1997); 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: HY2102).

References

Adams, R. D. & Wang, S. (1985). *Organometallics*, **5**, 1272–1274.
Adams, R. D. & Yang, L.-W. (1983). *J. Am. Chem. Soc.* **105**, 235–240.
Bard, A. J., Cowley, A. H., Leland, J. K., Thomas, G. J. N., Norman, N. C., Jutzi, P., Morley, C. P. & Schlüter, E. (1985). *J. Chem. Soc. Dalton Trans.* pp. 1303–1307.
Benoit, A., Marouille, J. L. & Patin, C. (1982). *J. Organomet. Chem.* **233**, C51–C54.
Bruker (1997). *SADABS* (Version 2.03) and *SHELXTL* (Version 5.10). Bruker AXS Inc., Madison, Wisconsin, USA.
Bruker (2001). *SAINT-Plus* and *APEX2*. Bruker AXS Inc., Madison, Wisconsin, USA.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Weininger, S. J. & Stermitz, F. R. (1984). *Organic Chemistry*. New York: Academic Press.

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Octacarbonyl(5-methoxy-2,3-dihydro-1*H*-benzimidazol-2-yl)di-μ_3-sulfido-diiron(I)iron(II)(2 *Fe*—*Fe*)

Tingting Zhang, Mei Wang, Ping Li and Licheng Sun

S1. Comment

The chemistry of sulfide carbonyl clusters is well developed not only on account of their fascinating structural chemistry, but also because of their ability to act as electron reservoirs and potential in catalysis (Adams & Wang, 1985; Adams & Yang, 1983). Here we report the crystal structure of the title compound.

The central Fe_3S_2 unit in the title compound is in accord with that for the well known complex [$Fe_3(\mu_3\text{-}S)_2(CO)_9$] (Bard *et al.*, 1985). The molecule contains a triangle of Fe atoms capped by two S atoms above and below. There exist two Fe—Fe bonds in the molecule, that is, Fe1—Fe2 [2.6322 (5) Å] and Fe2—Fe3 [2.5582 (5) Å] bonds, while the distance between Fe1 and Fe3 [3.3987 (5) Å] is too long to form a Fe—Fe bond. The Fe1—C9 bond length [1.914 (2) Å] is similar to that for another carbene–iron complex (Benoit *et al.*, 1982). The distances of two C9—N bonds [1.348 (3) and 1.355 (3) Å] are significantly shorter than that of the C—N single bond (1.47 Å) (Weininger & Stermitz, 1984) and longer than that of the C—N double bond (1.30 Å) (Weininger & Stermitz, 1984). The sum of the angles around the C9 atom is 359.6°, suggesting an sp^2-hybridized C atom.

S2. Experimental

$Fe_3(CO)_{12}$ (0.50 g, 1 mmol) was added to a solution of 5-methoxy-1*H*-benzoimidazole-2-thiol (0.54 g, 3 mmol) in THF (20 ml). After the solution was refluxed for 20 min, the solvent was removed under reduced pressure and the residue was chromatographed on a silica gel column with CH_2Cl_2/hexane (2:1 *v*/*v*) as the eluent to give the title compound as a brown solid (yield 60%, 0.36 g). Single crystals of the title compound were obtained by slow evaporation of the CH_2Cl_2 solution at room temperature.

S3. Refinement

H atoms attached to C and N atoms were positioned geometrically and refined as riding, with C—H = 0.93 (CH) and 0.97 Å (CH_3), and N—H = 0.86 Å, and with U_{iso}(H) = 1.5U_{eq}(C) for methyl group and U_{iso}(H) = 1.2U_{eq}(C,*N*) for the others.

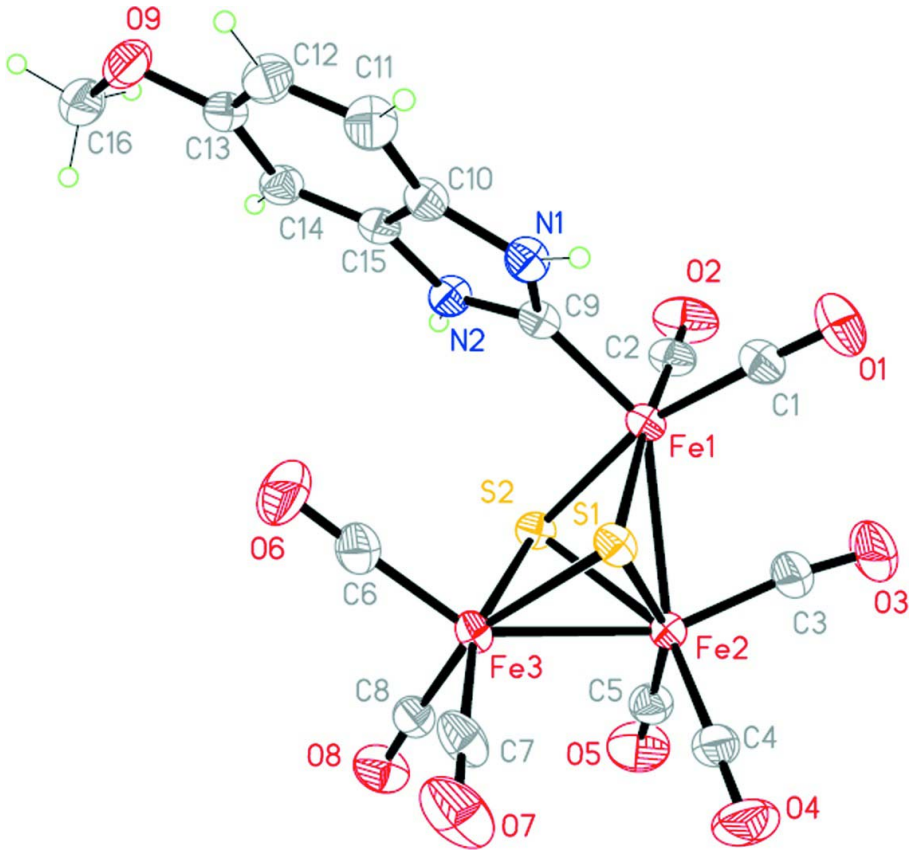

Figure 1

The molecular structure of the title compound. Displacement ellipsoids are drawn at the 30% probability level.

Octacarbonyl(5-methoxy-2,3-dihydro-1*H*-benzimidazol-2-yl)di-μ_3– sulfido-diiron(I)iron(II)(2 Fe—Fe)

Crystal data

$[Fe_3(C_8H_8N_2O)S_2(CO)_8]$
M_r = 603.91
Triclinic, $P\overline{1}$
Hall symbol: -P 1
a = 9.0068 (4) Å
b = 11.4504 (5) Å
c = 12.5976 (6) Å
α = 112.332 (3)°
β = 108.728 (3)°
γ = 96.229 (3)°
V = 1098.0 (1) Å^3
Z = 2
F(000) = 600
D_x = 1.827 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 3399 reflections
θ = 3.6–29.3°
μ = 2.20 mm^{-1}
T = 273 K
Block, brown
0.25 × 0.20 × 0.10 mm

Data collection

Bruker SMART APEXII CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 1997)
T_{min} = 0.610, T_{max} = 0.810
8226 measured reflections
4253 independent reflections
3584 reflections with $I > 2\sigma(I)$
R_{int} = 0.019
θ_{max} = 26.0°, θ_{min} = 3.6°
h = −11→10
k = −14→13
l = −15→15

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.028$
$wR(F^2) = 0.070$
$S = 1.05$
4253 reflections
289 parameters
0 restraints
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0345P)^2 + 0.1529P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.28$ e Å^{-3}
$\Delta\rho_{min} = -0.30$ e Å^{-3}

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

	x	*y*	*z*	U_{iso}*/U_{eq}
Fe2	0.76196 (4)	0.76148 (3)	0.59336 (3)	0.03973 (10)
Fe1	0.94423 (4)	1.00067 (3)	0.75013 (3)	0.03945 (10)
Fe3	0.54802 (4)	0.85600 (3)	0.66569 (3)	0.03970 (10)
S2	0.70315 (7)	0.94813 (6)	0.59519 (5)	0.03972 (14)
S1	0.79595 (7)	0.87177 (6)	0.79494 (5)	0.04141 (14)
N1	0.9696 (2)	1.2115 (2)	0.98758 (18)	0.0454 (5)
H1A	1.0240	1.1784	1.0333	0.055*
N2	0.8456 (2)	1.24400 (19)	0.83051 (17)	0.0439 (5)
H2A	0.8050	1.2356	0.7554	0.053*
C9	0.9253 (3)	1.1612 (2)	0.8629 (2)	0.0406 (5)
O8	0.2735 (2)	0.7602 (2)	0.4266 (2)	0.0715 (6)
O2	1.1025 (3)	1.1243 (2)	0.6350 (2)	0.0833 (7)
C15	0.8368 (3)	1.3437 (2)	0.9318 (2)	0.0413 (5)
C2	1.0438 (3)	1.0765 (3)	0.6799 (2)	0.0530 (6)
O1	1.2491 (3)	0.9921 (2)	0.9194 (2)	0.0819 (7)
C8	0.3784 (3)	0.7992 (2)	0.5209 (3)	0.0475 (6)
C1	1.1322 (3)	0.9977 (3)	0.8553 (2)	0.0524 (6)
C10	0.9171 (3)	1.3231 (2)	1.0340 (2)	0.0443 (5)
O5	0.5879 (3)	0.6474 (2)	0.3226 (2)	0.0884 (7)
C5	0.6572 (3)	0.6905 (3)	0.4274 (3)	0.0555 (6)
C4	0.7104 (3)	0.6124 (3)	0.6041 (3)	0.0607 (7)
O9	0.7147 (3)	1.63452 (19)	1.08295 (19)	0.0690 (5)
C14	0.7649 (3)	1.4461 (2)	0.9402 (2)	0.0479 (6)
H14A	0.7104	1.4583	0.8703	0.057*
O4	0.6759 (3)	0.5192 (2)	0.6127 (3)	0.0997 (9)
C13	0.7787 (3)	1.5292 (2)	1.0582 (2)	0.0514 (6)
O3	1.0797 (3)	0.7445 (3)	0.5882 (3)	0.1048 (9)
C3	0.9606 (4)	0.7601 (3)	0.5954 (3)	0.0638 (7)
C12	0.8608 (4)	1.5093 (3)	1.1619 (3)	0.0606 (7)
H12A	0.8691	1.5673	1.2402	0.073*
O7	0.3939 (3)	0.6703 (3)	0.7320 (3)	0.1099 (10)
O6	0.4779 (4)	1.0948 (3)	0.8090 (3)	0.1222 (11)
C16	0.6157 (4)	1.6512 (3)	0.9793 (3)	0.0694 (8)
H16A	0.5782	1.7280	1.0085	0.104*
H16B	0.5240	1.5760	0.9268	0.104*
H16C	0.6778	1.6608	0.9324	0.104*
C11	0.9296 (4)	1.4067 (3)	1.1513 (2)	0.0596 (7)

H11A	0.9830	1.3937	1.2208	0.072*
C6	0.5036 (4)	1.0008 (3)	0.7545 (3)	0.0670 (8)
C7	0.4532 (3)	0.7432 (3)	0.7076 (3)	0.0630 (8)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Fe2	0.03531 (18)	0.03891 (18)	0.0433 (2)	0.00916 (14)	0.01431 (15)	0.01758 (15)
Fe1	0.03450 (18)	0.04229 (19)	0.03923 (19)	0.00558 (14)	0.01320 (14)	0.01792 (15)
Fe3	0.03299 (17)	0.0493 (2)	0.04277 (19)	0.01194 (14)	0.01478 (15)	0.02601 (16)
S2	0.0417 (3)	0.0408 (3)	0.0379 (3)	0.0078 (2)	0.0127 (2)	0.0218 (2)
S1	0.0376 (3)	0.0511 (3)	0.0393 (3)	0.0103 (2)	0.0115 (2)	0.0271 (3)
N1	0.0463 (11)	0.0522 (12)	0.0390 (11)	0.0147 (9)	0.0118 (9)	0.0249 (9)
N2	0.0498 (12)	0.0459 (11)	0.0312 (10)	0.0135 (9)	0.0098 (9)	0.0171 (9)
C9	0.0366 (12)	0.0446 (13)	0.0386 (12)	0.0043 (10)	0.0107 (10)	0.0213 (10)
O8	0.0584 (12)	0.0816 (14)	0.0628 (13)	0.0070 (10)	−0.0016 (11)	0.0442 (11)
O2	0.0897 (16)	0.0868 (16)	0.0683 (14)	−0.0140 (12)	0.0409 (13)	0.0309 (12)
C15	0.0377 (12)	0.0419 (12)	0.0349 (12)	0.0033 (10)	0.0092 (10)	0.0135 (10)
C2	0.0492 (15)	0.0533 (15)	0.0424 (14)	−0.0003 (12)	0.0158 (12)	0.0124 (12)
O1	0.0495 (12)	0.1139 (19)	0.0762 (15)	0.0262 (12)	0.0098 (11)	0.0468 (14)
C8	0.0446 (14)	0.0504 (14)	0.0560 (16)	0.0122 (11)	0.0184 (13)	0.0333 (13)
C1	0.0425 (15)	0.0593 (16)	0.0506 (15)	0.0078 (12)	0.0174 (13)	0.0217 (13)
C10	0.0428 (13)	0.0506 (14)	0.0372 (12)	0.0095 (11)	0.0126 (10)	0.0203 (11)
O5	0.0985 (18)	0.0868 (16)	0.0450 (13)	0.0029 (13)	0.0138 (12)	0.0118 (11)
C5	0.0543 (16)	0.0519 (15)	0.0525 (17)	0.0100 (12)	0.0205 (14)	0.0167 (13)
C4	0.0521 (16)	0.0481 (15)	0.0701 (19)	0.0088 (12)	0.0095 (14)	0.0274 (14)
O9	0.0762 (14)	0.0546 (11)	0.0598 (12)	0.0263 (10)	0.0217 (11)	0.0107 (9)
C14	0.0471 (14)	0.0452 (13)	0.0421 (13)	0.0096 (11)	0.0092 (11)	0.0176 (11)
O4	0.0993 (18)	0.0560 (14)	0.126 (2)	0.0023 (13)	0.0133 (16)	0.0536 (14)
C13	0.0447 (14)	0.0470 (14)	0.0486 (15)	0.0058 (11)	0.0152 (12)	0.0115 (11)
O3	0.0574 (14)	0.115 (2)	0.134 (2)	0.0314 (14)	0.0518 (15)	0.0326 (18)
C3	0.0474 (16)	0.0646 (18)	0.074 (2)	0.0155 (13)	0.0261 (15)	0.0226 (15)
C12	0.0632 (18)	0.0668 (18)	0.0396 (14)	0.0169 (14)	0.0187 (13)	0.0125 (13)
O7	0.0737 (15)	0.156 (3)	0.152 (3)	0.0164 (16)	0.0421 (17)	0.125 (2)
O6	0.127 (2)	0.0838 (19)	0.156 (3)	0.0416 (17)	0.096 (2)	0.0157 (18)
C16	0.0660 (19)	0.0555 (17)	0.076 (2)	0.0228 (14)	0.0210 (17)	0.0227 (15)
C11	0.0653 (18)	0.0754 (19)	0.0356 (13)	0.0216 (15)	0.0157 (13)	0.0245 (13)
C6	0.0574 (17)	0.070 (2)	0.075 (2)	0.0180 (15)	0.0374 (16)	0.0234 (16)
C7	0.0397 (14)	0.095 (2)	0.0701 (19)	0.0154 (14)	0.0162 (13)	0.0568 (18)

Geometric parameters (Å, º)

Fe2—C3	1.783 (3)	O8—C8	1.135 (3)
Fe2—C4	1.787 (3)	O2—C2	1.125 (3)
Fe2—C5	1.788 (3)	C15—C10	1.380 (3)
Fe2—S2	2.2505 (7)	C15—C14	1.386 (3)
Fe2—S1	2.2621 (7)	O1—C1	1.128 (3)
Fe2—Fe3	2.5582 (5)	C10—C11	1.381 (4)

Fe2—Fe1	2.6322 (5)	O5—C5	1.134 (3)
Fe1—C1	1.797 (3)	C4—O4	1.133 (3)
Fe1—C2	1.807 (3)	O9—C13	1.366 (3)
Fe1—C9	1.914 (2)	O9—C16	1.420 (3)
Fe1—S1	2.2294 (6)	C14—C13	1.383 (4)
Fe1—S2	2.2337 (7)	C14—H14A	0.9300
Fe3—C8	1.783 (3)	C13—C12	1.395 (4)
Fe3—C6	1.784 (3)	O3—C3	1.133 (3)
Fe3—C7	1.801 (3)	C12—C11	1.369 (4)
Fe3—S1	2.2415 (6)	C12—H12A	0.9300
Fe3—S2	2.2455 (6)	O7—C7	1.132 (3)
N1—C9	1.348 (3)	O6—C6	1.130 (4)
N1—C10	1.392 (3)	C16—H16A	0.9600
N1—H1A	0.8600	C16—H16B	0.9600
N2—C9	1.355 (3)	C16—H16C	0.9600
N2—C15	1.384 (3)	C11—H11A	0.9300
N2—H2A	0.8600		

C3—Fe2—C4	99.51 (14)	S2—Fe3—Fe2	55.410 (18)
C3—Fe2—C5	96.64 (14)	Fe1—S2—Fe3	98.71 (2)
C4—Fe2—C5	94.66 (13)	Fe1—S2—Fe2	71.89 (2)
C3—Fe2—S2	110.82 (10)	Fe3—S2—Fe2	69.362 (19)
C4—Fe2—S2	149.33 (10)	Fe1—S1—Fe3	98.96 (2)
C5—Fe2—S2	86.53 (9)	Fe1—S1—Fe2	71.75 (2)
C3—Fe2—S1	105.97 (10)	Fe3—S1—Fe2	69.23 (2)
C4—Fe2—S1	88.38 (10)	C9—N1—C10	112.33 (19)
C5—Fe2—S1	156.38 (9)	C9—N1—H1A	123.8
S2—Fe2—S1	79.21 (2)	C10—N1—H1A	123.8
C3—Fe2—Fe3	156.00 (10)	C9—N2—C15	112.47 (19)
C4—Fe2—Fe3	94.73 (10)	C9—N2—H2A	123.8
C5—Fe2—Fe3	101.37 (9)	C15—N2—H2A	123.8
S2—Fe2—Fe3	55.228 (18)	N1—C9—N2	103.9 (2)
S1—Fe2—Fe3	55.008 (18)	N1—C9—Fe1	129.74 (17)
C3—Fe2—Fe1	74.51 (10)	N2—C9—Fe1	126.01 (17)
C4—Fe2—Fe1	135.67 (10)	C10—C15—N2	105.7 (2)
C5—Fe2—Fe1	129.47 (9)	C10—C15—C14	122.9 (2)
S2—Fe2—Fe1	53.760 (18)	N2—C15—C14	131.4 (2)
S1—Fe2—Fe1	53.551 (18)	O2—C2—Fe1	178.4 (3)
Fe3—Fe2—Fe1	81.794 (15)	O8—C8—Fe3	177.5 (3)
C1—Fe1—C2	93.34 (12)	O1—C1—Fe1	178.0 (3)
C1—Fe1—C9	96.89 (11)	C15—C10—C11	120.2 (2)
C2—Fe1—C9	94.73 (11)	C15—C10—N1	105.6 (2)
C1—Fe1—S1	92.65 (8)	C11—C10—N1	134.2 (2)
C2—Fe1—S1	167.53 (8)	O5—C5—Fe2	178.2 (3)
C9—Fe1—S1	95.40 (7)	O4—C4—Fe2	178.4 (3)
C1—Fe1—S2	165.00 (9)	C13—O9—C16	117.3 (2)
C2—Fe1—S2	91.31 (8)	C13—C14—C15	116.2 (2)
C9—Fe1—S2	96.92 (7)	C13—C14—H14A	121.9

S1—Fe1—S2	80.26 (2)	C15—C14—H14A	121.9
C1—Fe1—Fe2	110.82 (9)	O9—C13—C14	123.8 (2)
C2—Fe1—Fe2	112.86 (8)	O9—C13—C12	115.1 (2)
C9—Fe1—Fe2	138.68 (7)	C14—C13—C12	121.1 (2)
S1—Fe1—Fe2	54.701 (19)	O3—C3—Fe2	172.2 (3)
S2—Fe1—Fe2	54.352 (18)	C11—C12—C13	121.7 (2)
C8—Fe3—C6	99.45 (14)	C11—C12—H12A	119.1
C8—Fe3—C7	91.77 (12)	C13—C12—H12A	119.1
C6—Fe3—C7	99.30 (15)	O9—C16—H16A	109.5
C8—Fe3—S1	156.98 (9)	O9—C16—H16B	109.5
C6—Fe3—S1	102.43 (11)	H16A—C16—H16B	109.5
C7—Fe3—S1	91.72 (9)	O9—C16—H16C	109.5
C8—Fe3—S2	90.45 (8)	H16A—C16—H16C	109.5
C6—Fe3—S2	97.38 (10)	H16B—C16—H16C	109.5
C7—Fe3—S2	162.57 (10)	C12—C11—C10	117.8 (2)
S1—Fe3—S2	79.75 (2)	C12—C11—H11A	121.1
C8—Fe3—Fe2	101.57 (9)	C10—C11—H11A	121.1
C6—Fe3—Fe2	145.32 (10)	O6—C6—Fe3	177.5 (3)
C7—Fe3—Fe2	107.26 (10)	O7—C7—Fe3	178.6 (3)
S1—Fe3—Fe2	55.765 (19)		