

1 4,5-diferrocenyl-1,2-dithiol-3-thione

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5 Abstract

6 The structure of 4,5-diferrocenyl-1,2-dithiol-3-thione, $C_{23}H_{18}Fe_2S_3$, at 130 K has monoclinic ($P2_1/c$) symmetry. The com-
7 pound has two ferrocenyl units bridged by a 3-dithiol-3-thione moiety. It is of interest with respect to the ferrocenyl into
8 biological molecules offers the potential to develop better more efficacious therapeutic drugs. The crystal the packing is
9 assumed to be mainly dictated by van der Waals forces.

10 Structure description

11 Ferrocene is known for its stable sandwich structure. The incorporation of ferrocenyl into biological molecules offers the
12 potential to develop better and more efficacious therapeutic drugs. 1,2-dithiole-3-thiones show significant biological ac-
13 tivity, which include amongst others antitumor, antioxidant, chemotherapeutic, antithrombotic and radio protective prop-
14 erties (Rakitin *et al.*, 2021). The 1,2-dithiole-3-thione moiety can be found in commercial drugs such as Oltipraz
15 (Maxuitenko 15 *et al.*, 1998) anethole dithiolethione ADT (Chen *et al.*, 2010), S-Danshensu (Bian *et al.*, 2012) and
16 NOSH-1 (Jia *et al.*, 16 2013). The synthons can be useful for many sulfurs heterocycles (Konstantinova *et al.*, 2007), and
17 their optical properties have been employed for the creation of organic electronic conductors (Yamashita *et al.*, 1998),
18 photoconductive materials (Perepichka *et al.*, 2001), and semiconducting polymers (Hou *et al.*, 2011).

19 The asymmetric unit of the title compound is constituted by a pair of ferrocenyl units bridged by a dithiol-3-thione ring
20 (Fig. 1). The cyclopentadienyl (Cp) rings are almost parallel, with angle of 4.06 (2) and 4.24 (2) $^\circ$ between Cp planes for
21 ferrocenyl groups with Fe1 and Fe2 respectively. The cyclopentadienyl rings of the ferrocenyl moiety adopt eclipsed
22 conformation. The dithiol-3-thione ring is planarity with r.m.s. of 0.0295 for $-3.79(2)x + 9.17(1)y + 10.04(1)z = 4.21$
23 (1) equation plane. The dithiol-3-thione ring, form a angle of 34.91 (2) and 49.20 (8) $^\circ$, with C4/C8 and C14/C18 rings re-
24 spectively. The dihedral angles between the dithiol-3-thione ring and the substituted cyclopentadienyl rings are 36.42 (3)
25 and 49.28 (2) $^\circ$. The crystalline array, it is stabilized by the weak intermolecular interactions C—H \cdots S, S—S \cdots π mainly.
26 The intermolecular C21—H21 \cdots S3 contact is found at 2.883 Å, while the interaction S—S \cdots π it is set between the di-
27 thiol-3-thione group and the cyclopentadienyl (Cp) ring of the ferrocenyl group. The figure 2 shows the [010] projection
28 of the unit cell. In summary, the packing of the molecules is assumed to be mainly dictated by van der Waals forces.

29 Synthesis and crystallization

30 Mixture of sodium sulfide (10 mmol) and S₈ (10 mmol) in ethanol (80 ml) is added the 1,2-diferrocenylcyclopropenone
31 (5 mmol) was stirred at 353 K for 8 h. The solvents were removed *in vacuo*, the residues was purified by column
32 chromatography with alumina, and hexane: diethyl ether (ratio 1:1 *v/v*). crystals of 4,5-diferrocenyl-1,2-dithiol-3-thione,
33 suitable for single-crystal diffraction analysis, were obtained by slow evaporation of saturated dichloromethane/hexane
34 (ratio 1:1 *v/v*) solution, yield (50%), black crystals, mp. 498–500 K.

35 ¹H NMR(Fig. 3) (400 MHz, CDCl₃) δ : 4.12 (5 H, s, C₅H₅), δ : 4.18 (5 H, s, C₅H₅), δ : 4.19 (2 H, m, C₅H₄), δ : 4.35 (2 H,
36 m, C₅H₄), δ : 4.38 (2 H, m, C₅H₄), δ : 4.40 (2 H, m, C₅H₄) p.p.m., ¹³C NMR (Fig. 4) (75 MHz, CDCl₃) δ : 69.71, 70.92
37 (C₅H₅), 67.45, 69.74, 70.14, 70.92 (2C₅H₄) 79.60, 80.05 (Cipso Fc), 141.37, 169.18 (C), 214.0 (C=S) p.p.m., MS: m/z
38 502 [M]⁺ 40. Anal. Calcd for C₂₃H₁₈Fe₂S₃: C, 55.02, H, 3.61, S, 19.15, Found C, 55.10, H, 3.71, S, 19.22%.

39 **Refinement details**

40 Crystal data, data collection and structure refinement details are summarized in Table 1.

41 **Table 1**

42 Experimental details

43	Crystal data	
44	Chemical formula	C ₂₃ H ₁₈ Fe ₂ S ₃
45	M_r	502.25
46	Crystal system, space group	Monoclinic, P2 ₁ /c
47	Temperature (K)	130
48	a, b, c (Å)	11.0149 (12), 14.0459 (12), 13.3983 (13)
49	β (°)	109.205 (12)
50	V (Å ³)	1957.5 (4)
51	Z	4
52	Radiation type	Mo $K\alpha$
53	μ (mm ⁻¹)	1.81
54	Crystal size (mm)	0.57 × 0.46 × 0.11
55	Data collection	
56	Diffractometer	Xcalibur, Atlas, Gemini
57	Absorption correction	Analytical (<i>CrysAlis RED</i> ; Agilent, 2013)
58	T_{\min}, T_{\max}	0.486, 0.852
59	No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	10068, 4559, 3445
60	R_{int}	0.039
61	$(\sin \theta/\lambda)_{\max}$ (Å ⁻¹)	0.692
62	Refinement	
63	$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.039, 0.079, 1.04
64	No. of reflections	4559
65	No. of parameters	253
66	H-atom treatment	H-atom parameters constrained
67	$\Delta\rho_{\max}, \Delta\rho_{\min}$ (e Å ⁻³)	0.43, -0.41

70 Computer programs: *CrysAlis PRO*; Agilent, 2013, *CrysAlis PRO*; Agilent, 2013, *CrysAlis RED*; Agilent, 2013, *SHELXS2018* (Sheldrick, 2015),
71 *SHELXL2018* (Sheldrick, 2015), *ORTEP-3 for Windows* (Farrugia, 2012), *Mercury* (Macrae *et al.*, 2008).72 **Acknowledgements**73 The authors thank PAPIIT-DGAPA-UNAM (**IN 217421**) for their financial support of this work.74 **Funding information**75 Funding for this research was provided by: The authors thank PAPIIT-DGAPA-UNAM (IN 217421) for their financial
76 support of this work .77 **References**

- 78 Agilent (2013).
- CrysAlis PRO*
- and
- CrysAlis RED*
- . Agilent Technologies, Yarnton Oxfordshire, England
-
- 79 Bian, J., Cai, Z. & Wu, H. (2012).
- CN Patent*
- 102417501 A, 1

- 80 Chen, P., Luo, Y., Hai, L., Qian, S. & Wu, Y. (2010). *Eur. J. Med. Chem.*, **45**, 3005–3010
- 81 Hou, Y., Long, G., Sui, D., Cai, Y., Wan, X., Yu, A. & Chen, Y. (2011). *Chem. Commun.*, **47**, 10401–10403
- 82 Jia, J., Xiao, Y., Wang, W., Wang, L., Xu, Y., Song, H., Zhen, X., Ao, G., Alksasyed, N. & Cheng, J. (2013). *Neurochem. Int.* **62**, 1072–1078
- 83 Konstantinova, L. S., Berezin, A. A., Lysov, K. A. & Rakitin, O. A. (2007). *Tetrahedron Lett.* **48**, 5851–5854
- 84 Maxuitenko, Y., Libby, A. H., Joyner, H. H., Curphey, T. J., MacMillan, D. L., Kensler, T. W. & Roebuck, B. D. (1998).
85 Carcinogenesis, **19**, 1609–1615
- 86 Perepichka, D. F., Perepichka, I. F., Bryce, M. R., Moore, A. J. & Sokolov, N. I. (2001). *Synth. Met.* **121**, 1487–1488
- 87 Rakitin, O. A. (2021). *Molecules*, **26** (12), 3595–3638
- 88 Sheldrick, G. M. (2015). *Acta Cryst. A* **71**, 3–8.
- 89 Sheldrick, G. M. (2015). *Acta Cryst. C* **71**, 3–8.
- 90 Yamashita, Y., Tomura, M. & Zaman, M. B. (1998). *Chem. Commun.* 1657–1658

92 **Figure 1**

93 The *ORTEP* diagram of the compound **4,5-diferrocenyl-1,2-dithiol-3-thione**. Displacement ellipsoids are drawn at the
94 70% probability level.

95 **Figure 2**

96 The crystal array of the title compound showing intermolecular contacts of the type C—H···S and S···p, along the base
97 vector [010].

98 **Figure 3**

99 ^1H -NMR (400 MHz, CDCl₃, TMS) spectrum of compound 4,5-diferrocenyl-1,2-dithiol-3-thione.

100 **Figure 4**

101 ^{13}C -NMR (100 MHz, CDCl₃, TMS) spectrum of compound 4,5-diferrocenyl-1,2-dithiol-3-thione

¹ full crystallographic data² 4,5-diferrocenyl-1,2-dithiol-3-thione³ (ke0822)⁴ Crystal data

⁵ C₂₃H₁₈Fe₂S₃
⁶ M_r = 502.25
⁷ Monoclinic, P2₁/c
⁸ Hall symbol: -P 2ybc
⁹ a = 11.0149 (12) Å
¹⁰ b = 14.0459 (12) Å
¹¹ c = 13.3983 (13) Å
¹² β = 109.205 (12)°
¹³ V = 1957.5 (4) Å³
¹⁴ Z = 4

F(000) = 1024
⁶ D_x = 1.704 Mg m⁻³
⁷ Mo Kα radiation, λ = 0.71073 Å
⁸ Cell parameters from 2429 reflections
⁹ θ = 3.5–29.5°
¹⁰ μ = 1.81 mm⁻¹
¹¹ T = 130 K
¹² Plate, black
¹³ 0.57 × 0.46 × 0.11 mm

¹⁵ Data collection

¹⁶ Xcalibur, Atlas, Gemini
¹⁷ diffractometer
¹⁸ Graphite monochromator
¹⁹ Detector resolution: 10.4685 pixels mm⁻¹
²⁰ ω scans
²¹ Absorption correction: analytical
²² (CrysAlis RED; Agilent, 2013)
²³ T_{min} = 0.486, T_{max} = 0.852

10068 measured reflections
¹⁶ 4559 independent reflections
¹⁷ 3445 reflections with I > 2σ(I)
¹⁸ R_{int} = 0.039
¹⁹ θ_{max} = 29.5°, θ_{min} = 3.5°
²⁰ h = -15→13
²¹ k = -19→14
²² l = -16→18

²² Refinement

²³ Refinement on F²
²⁴ Least-squares matrix: full
²⁵ R[F² > 2σ(F²)] = 0.039
²⁶ wR(F²) = 0.079
²⁷ S = 1.04
²⁸ 4559 reflections
²⁹ 253 parameters
³⁰ 0 restraints

Hydrogen site location: inferred from neighbouring
²³ sites
²⁴ H-atom parameters constrained
²⁵ w = 1/[σ²(F_o²) + (0.023P)² + 0.8521P]
²⁶ where P = (F_o² + 2F_c²)/3
²⁷ (Δ/σ)_{max} < 0.001
²⁸ Δρ_{max} = 0.43 e Å⁻³
²⁹ Δρ_{min} = -0.41 e Å⁻³

³¹ Special details

³² Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

³³ Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	y	z	U _{iso} * / U _{eq}
C1	0.4091 (3)	0.39934 (18)	0.2096 (2)	0.0128 (6)
C2	0.5328 (3)	0.38193 (18)	0.2754 (2)	0.0128 (6)
C3	0.6322 (3)	0.4379 (2)	0.2544 (2)	0.0173 (6)
C4	0.5612 (2)	0.31407 (19)	0.3639 (2)	0.0124 (6)
C5	0.6716 (3)	0.25355 (18)	0.4048 (2)	0.0144 (6)

40	H5	0.74328	0.25135	0.380255	0.017*
41	C6	0.6561 (3)	0.1977 (2)	0.4878 (2)	0.0181 (6)
42	H6	0.715344	0.151857	0.528342	0.022*
43	C7	0.5366 (3)	0.22197 (19)	0.5000 (2)	0.0179 (6)
44	H7	0.50179	0.195138	0.549818	0.021*
45	C8	0.4783 (3)	0.29337 (19)	0.4246 (2)	0.0147 (6)
46	H8	0.397726	0.322723	0.415683	0.018*
47	C9	0.7111 (3)	0.4770 (2)	0.5497 (2)	0.0194 (7)
48	H9	0.699657	0.524867	0.497316	0.023*
49	C10	0.6239 (3)	0.4545 (2)	0.6037 (2)	0.0177 (6)
50	H10	0.543627	0.484821	0.593797	0.021*
51	C11	0.6767 (3)	0.3790 (2)	0.6750 (2)	0.0213 (7)
52	H11	0.638054	0.349734	0.72102	0.026*
53	C12	0.7972 (3)	0.3551 (2)	0.6656 (2)	0.0244 (7)
54	H12	0.853732	0.306978	0.704488	0.029*
55	C13	0.8192 (3)	0.4150 (2)	0.5880 (2)	0.0238 (7)
56	H13	0.892609	0.414117	0.565643	0.029*
57	C14	0.2895 (3)	0.35130 (19)	0.2068 (2)	0.0140 (6)
58	C15	0.1742 (3)	0.3995 (2)	0.2065 (2)	0.0156 (6)
59	H15	0.162618	0.466391	0.20834	0.019*
60	C16	0.0809 (3)	0.3289 (2)	0.2032 (2)	0.0209 (7)
61	H16	-0.003988	0.340458	0.203319	0.025*
62	C17	0.1353 (3)	0.2384 (2)	0.1995 (2)	0.0209 (7)
63	H17	0.092877	0.17904	0.196154	0.025*
64	C18	0.2636 (3)	0.2511 (2)	0.2016 (2)	0.0176 (6)
65	H18	0.322191	0.201926	0.199956	0.021*
66	C19	0.1889 (3)	0.3340 (2)	-0.0603 (2)	0.0208 (7)
67	H19	0.27249	0.347447	-0.062494	0.025*
68	C20	0.1376 (3)	0.2426 (2)	-0.0567 (2)	0.0197 (7)
69	H20	0.180714	0.183946	-0.055911	0.024*
70	C21	0.0111 (3)	0.2533 (2)	-0.0543 (2)	0.0201 (7)
71	H21	-0.04585	0.203153	-0.051976	0.024*
72	C22	-0.0160 (3)	0.3527 (2)	-0.0560 (2)	0.0205 (7)
73	H22	-0.094107	0.380448	-0.054727	0.025*
74	C23	0.0941 (3)	0.4027 (2)	-0.0601 (2)	0.0201 (7)
75	H23	0.102955	0.469885	-0.062245	0.024*
76	Fe1	0.65338 (4)	0.33776 (3)	0.52362 (3)	0.01289 (11)
77	Fe2	0.13851 (4)	0.31869 (3)	0.07340 (3)	0.01267 (11)
78	S1	0.38303 (7)	0.48864 (5)	0.11639 (6)	0.01651 (16)
79	S2	0.57355 (7)	0.52584 (5)	0.15958 (6)	0.01945 (17)
80	S3	0.79134 (7)	0.43455 (6)	0.30660 (7)	0.0298 (2)

81 *Atomic displacement parameters (\AA^2)*

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}	
83	C1	0.0184 (14)	0.0122 (13)	0.0083 (13)	-0.0008 (11)	0.0049 (12)	-0.0026 (11)
84	C2	0.0153 (14)	0.0121 (13)	0.0112 (13)	-0.0026 (11)	0.0048 (12)	-0.0021 (11)
85	C3	0.0166 (14)	0.0213 (15)	0.0137 (14)	-0.0011 (12)	0.0044 (12)	-0.0015 (13)
86	C4	0.0112 (13)	0.0131 (13)	0.0116 (13)	-0.0025 (11)	0.0020 (11)	-0.0033 (12)
87	C5	0.0159 (14)	0.0118 (14)	0.0142 (14)	0.0005 (11)	0.0033 (12)	-0.0049 (12)
88	C6	0.0222 (16)	0.0124 (14)	0.0139 (14)	0.0024 (12)	-0.0019 (13)	-0.0014 (12)
89	C7	0.0227 (16)	0.0153 (14)	0.0143 (14)	-0.0074 (13)	0.0042 (13)	0.0004 (12)

90	C8	0.0121 (14)	0.0176 (15)	0.0123 (14)	-0.0036 (12)	0.0014 (12)	-0.0007 (12)
91	C9	0.0273 (16)	0.0136 (14)	0.0138 (14)	-0.0035 (13)	0.0018 (13)	-0.0043 (12)
92	C10	0.0209 (15)	0.0171 (15)	0.0135 (14)	0.0040 (12)	0.0033 (12)	-0.0076 (12)
93	C11	0.0293 (17)	0.0231 (16)	0.0090 (14)	0.0015 (14)	0.0029 (13)	-0.0036 (13)
94	C12	0.0226 (16)	0.0236 (17)	0.0182 (16)	0.0058 (14)	-0.0052 (13)	-0.0077 (14)
95	C13	0.0171 (16)	0.0279 (17)	0.0228 (16)	-0.0058 (13)	0.0016 (13)	-0.0127 (15)
96	C14	0.0145 (14)	0.0160 (14)	0.0083 (13)	0.0002 (12)	-0.0005 (11)	0.0012 (12)
97	C15	0.0150 (14)	0.0217 (15)	0.0082 (13)	-0.0019 (12)	0.0010 (12)	-0.0047 (12)
98	C16	0.0170 (15)	0.0377 (19)	0.0094 (13)	-0.0040 (14)	0.0061 (12)	-0.0037 (14)
99	C17	0.0213 (16)	0.0284 (17)	0.0104 (14)	-0.0082 (14)	0.0015 (13)	0.0049 (13)
100	C18	0.0206 (15)	0.0150 (15)	0.0137 (14)	-0.0014 (12)	0.0010 (12)	0.0012 (12)
101	C19	0.0207 (16)	0.0318 (18)	0.0088 (13)	-0.0042 (14)	0.0033 (12)	-0.0029 (13)
102	C20	0.0248 (16)	0.0210 (16)	0.0125 (14)	0.0025 (13)	0.0051 (13)	-0.0048 (13)
103	C21	0.0200 (15)	0.0247 (16)	0.0105 (14)	-0.0069 (13)	-0.0020 (12)	-0.0014 (13)
104	C22	0.0156 (15)	0.0280 (17)	0.0132 (14)	0.0013 (13)	-0.0016 (12)	-0.0006 (13)
105	C23	0.0244 (17)	0.0195 (15)	0.0112 (14)	-0.0047 (13)	-0.0011 (13)	0.0032 (13)
106	Fe1	0.0142 (2)	0.0121 (2)	0.0100 (2)	0.00071 (16)	0.00085 (17)	-0.00160 (16)
107	Fe2	0.0116 (2)	0.0164 (2)	0.00860 (19)	-0.00166 (16)	0.00152 (16)	0.00006 (17)
108	S1	0.0167 (4)	0.0158 (4)	0.0143 (3)	-0.0020 (3)	0.0014 (3)	0.0036 (3)
109	S2	0.0181 (4)	0.0207 (4)	0.0178 (4)	-0.0060 (3)	0.0035 (3)	0.0045 (3)
110	S3	0.0138 (4)	0.0402 (5)	0.0334 (5)	-0.0021 (4)	0.0051 (4)	0.0137 (4)

111 Geometric parameters (\AA , °)

112	C1—C2	1.379 (4)	C12—H12	0.95
113	C1—C14	1.470 (4)	C13—Fe1	2.055 (3)
114	C1—S1	1.726 (3)	C13—H13	0.95
115	C2—C3	1.449 (4)	C14—C18	1.433 (4)
116	C2—C4	1.472 (4)	C14—C15	1.438 (4)
117	C3—S3	1.661 (3)	C14—Fe2	2.053 (3)
118	C3—S2	1.736 (3)	C15—C16	1.418 (4)
119	C4—C5	1.436 (4)	C15—Fe2	2.040 (3)
120	C4—C8	1.439 (4)	C15—H15	0.95
121	C4—Fe1	2.072 (3)	C16—C17	1.412 (4)
122	C5—C6	1.417 (4)	C16—Fe2	2.042 (3)
123	C5—Fe1	2.046 (3)	C16—H16	0.95
124	C5—H5	0.95	C17—C18	1.416 (4)
125	C6—C7	1.420 (4)	C17—Fe2	2.041 (3)
126	C6—Fe1	2.028 (3)	C17—H17	0.95
127	C6—H6	0.95	C18—Fe2	2.048 (3)
128	C7—C8	1.418 (4)	C18—H18	0.95
129	C7—Fe1	2.033 (3)	C19—C20	1.410 (4)
130	C7—H7	0.95	C19—C23	1.422 (4)
131	C8—Fe1	2.045 (3)	C19—Fe2	2.053 (3)
132	C8—H8	0.95	C19—H19	0.95
133	C9—C10	1.415 (4)	C20—C21	1.412 (4)
134	C9—C13	1.428 (4)	C20—Fe2	2.041 (3)
135	C9—Fe1	2.051 (3)	C20—H20	0.95
136	C9—H9	0.95	C21—C22	1.426 (4)
137	C10—C11	1.418 (4)	C21—Fe2	2.040 (3)
138	C10—Fe1	2.043 (3)	C21—H21	0.95
139	C10—H10	0.95	C22—C23	1.418 (4)

140	C11—C12	1.415 (4)	C22—Fe2	2.047 (3)
141	C11—Fe1	2.043 (3)	C22—H22	0.95
142	C11—H11	0.95	C23—Fe2	2.063 (3)
143	C12—C13	1.419 (5)	C23—H23	0.95
144	C12—Fe1	2.050 (3)	S1—S2	2.0525 (10)
145				
146	C2—C1—C14	128.6 (2)	C20—C21—C22	107.8 (3)
147	C2—C1—S1	119.1 (2)	C20—C21—Fe2	69.79 (16)
148	C14—C1—S1	112.35 (19)	C22—C21—Fe2	69.84 (16)
149	C1—C2—C3	115.4 (2)	C20—C21—H21	126.1
150	C1—C2—C4	122.2 (2)	C22—C21—H21	126.1
151	C3—C2—C4	122.3 (2)	Fe2—C21—H21	125.9
152	C2—C3—S3	131.5 (2)	C23—C22—C21	108.0 (3)
153	C2—C3—S2	113.9 (2)	C23—C22—Fe2	70.40 (16)
154	S3—C3—S2	114.56 (17)	C21—C22—Fe2	69.32 (15)
155	C5—C4—C8	106.3 (2)	C23—C22—H22	126
156	C5—C4—C2	128.3 (3)	C21—C22—H22	126
157	C8—C4—C2	125.4 (2)	Fe2—C22—H22	125.8
158	C5—C4—Fe1	68.61 (15)	C22—C23—C19	107.5 (3)
159	C8—C4—Fe1	68.57 (15)	C22—C23—Fe2	69.22 (17)
160	C2—C4—Fe1	129.04 (19)	C19—C23—Fe2	69.44 (16)
161	C6—C5—C4	108.7 (3)	C22—C23—H23	126.2
162	C6—C5—Fe1	68.97 (16)	C19—C23—H23	126.2
163	C4—C5—Fe1	70.56 (15)	Fe2—C23—H23	126.7
164	C6—C5—H5	125.6	C6—Fe1—C7	40.94 (12)
165	C4—C5—H5	125.6	C6—Fe1—C11	120.22 (12)
166	Fe1—C5—H5	126.4	C7—Fe1—C11	104.35 (13)
167	C5—C6—C7	108.3 (2)	C6—Fe1—C10	156.95 (13)
168	C5—C6—Fe1	70.32 (16)	C7—Fe1—C10	121.46 (12)
169	C7—C6—Fe1	69.73 (16)	C11—Fe1—C10	40.62 (11)
170	C5—C6—H6	125.9	C6—Fe1—C8	68.57 (11)
171	C7—C6—H6	125.9	C7—Fe1—C8	40.69 (11)
172	Fe1—C6—H6	125.7	C11—Fe1—C8	120.94 (12)
173	C8—C7—C6	107.9 (3)	C10—Fe1—C8	107.83 (11)
174	C8—C7—Fe1	70.12 (15)	C6—Fe1—C5	40.71 (11)
175	C6—C7—Fe1	69.33 (16)	C7—Fe1—C5	68.62 (12)
176	C8—C7—H7	126.1	C11—Fe1—C5	157.56 (11)
177	C6—C7—H7	126.1	C10—Fe1—C5	161.08 (11)
178	Fe1—C7—H7	126.1	C8—Fe1—C5	68.41 (11)
179	C7—C8—C4	108.9 (2)	C6—Fe1—C12	105.52 (12)
180	C7—C8—Fe1	69.19 (15)	C7—Fe1—C12	119.71 (12)
181	C4—C8—Fe1	70.53 (15)	C11—Fe1—C12	40.44 (12)
182	C7—C8—H8	125.6	C10—Fe1—C12	68.02 (12)
183	C4—C8—H8	125.6	C8—Fe1—C12	156.00 (13)
184	Fe1—C8—H8	126.3	C5—Fe1—C12	123.14 (12)
185	C10—C9—C13	107.9 (3)	C6—Fe1—C9	159.73 (13)
186	C10—C9—Fe1	69.51 (16)	C7—Fe1—C9	159.11 (12)
187	C13—C9—Fe1	69.81 (16)	C11—Fe1—C9	68.25 (12)
188	C10—C9—H9	126.1	C10—Fe1—C9	40.44 (12)
189	C13—C9—H9	126.1	C8—Fe1—C9	125.06 (11)
190	Fe1—C9—H9	126.2	C5—Fe1—C9	125.45 (12)
191	C9—C10—C11	108.3 (3)	C12—Fe1—C9	68.12 (12)

192	C9—C10—Fe1	70.06 (16)	C6—Fe1—C13	122.13 (12)
193	C11—C10—Fe1	69.68 (16)	C7—Fe1—C13	156.59 (12)
194	C9—C10—H10	125.8	C11—Fe1—C13	68.25 (13)
195	C11—C10—H10	125.8	C10—Fe1—C13	68.21 (12)
196	Fe1—C10—H10	126	C8—Fe1—C13	162.09 (12)
197	C12—C11—C10	107.8 (3)	C5—Fe1—C13	109.31 (12)
198	C12—C11—Fe1	70.04 (18)	C12—Fe1—C13	40.45 (13)
199	C10—C11—Fe1	69.70 (16)	C9—Fe1—C13	40.70 (12)
200	C12—C11—H11	126.1	C6—Fe1—C4	68.90 (11)
201	C10—C11—H11	126.1	C7—Fe1—C4	68.94 (11)
202	Fe1—C11—H11	125.8	C11—Fe1—C4	158.45 (12)
203	C11—C12—C13	108.4 (3)	C10—Fe1—C4	124.24 (11)
204	C11—C12—Fe1	69.52 (16)	C8—Fe1—C4	40.90 (11)
205	C13—C12—Fe1	69.96 (17)	C5—Fe1—C4	40.83 (10)
206	C11—C12—H12	125.8	C12—Fe1—C4	160.70 (12)
207	C13—C12—H12	125.8	C9—Fe1—C4	110.39 (11)
208	Fe1—C12—H12	126.3	C13—Fe1—C4	125.76 (12)
209	C12—C13—C9	107.5 (3)	C21—Fe2—C15	149.91 (12)
210	C12—C13—Fe1	69.59 (17)	C21—Fe2—C20	40.48 (12)
211	C9—C13—Fe1	69.49 (16)	C15—Fe2—C20	169.40 (12)
212	C12—C13—H13	126.2	C21—Fe2—C17	104.53 (12)
213	C9—C13—H13	126.2	C15—Fe2—C17	68.54 (12)
214	Fe1—C13—H13	126.3	C20—Fe2—C17	114.90 (12)
215	C18—C14—C15	107.5 (2)	C21—Fe2—C16	115.13 (12)
216	C18—C14—C1	127.9 (3)	C15—Fe2—C16	40.64 (11)
217	C15—C14—C1	124.6 (2)	C20—Fe2—C16	147.95 (12)
218	C18—C14—Fe2	69.38 (15)	C17—Fe2—C16	40.47 (12)
219	C15—C14—Fe2	68.97 (15)	C21—Fe2—C22	40.84 (11)
220	C1—C14—Fe2	126.0 (2)	C15—Fe2—C22	118.59 (12)
221	C16—C15—C14	107.5 (3)	C20—Fe2—C22	68.24 (12)
222	C16—C15—Fe2	69.75 (16)	C17—Fe2—C22	126.44 (12)
223	C14—C15—Fe2	69.89 (16)	C16—Fe2—C22	107.48 (12)
224	C16—C15—H15	126.2	C21—Fe2—C18	125.60 (11)
225	C14—C15—H15	126.2	C15—Fe2—C18	68.99 (12)
226	Fe2—C15—H15	125.7	C20—Fe2—C18	106.65 (12)
227	C17—C16—C15	108.6 (3)	C17—Fe2—C18	40.51 (11)
228	C17—C16—Fe2	69.72 (17)	C16—Fe2—C18	68.30 (12)
229	C15—C16—Fe2	69.60 (16)	C22—Fe2—C18	163.99 (11)
230	C17—C16—H16	125.7	C21—Fe2—C14	165.36 (11)
231	C15—C16—H16	125.7	C15—Fe2—C14	41.13 (11)
232	Fe2—C16—H16	126.6	C20—Fe2—C14	129.44 (12)
233	C16—C17—C18	108.6 (3)	C17—Fe2—C14	68.42 (11)
234	C16—C17—Fe2	69.82 (17)	C16—Fe2—C14	68.46 (11)
235	C18—C17—Fe2	70.02 (17)	C22—Fe2—C14	153.54 (11)
236	C16—C17—H17	125.7	C18—Fe2—C14	40.92 (11)
237	C18—C17—H17	125.7	C21—Fe2—C19	67.94 (12)
238	Fe2—C17—H17	126	C15—Fe2—C19	132.43 (12)
239	C17—C18—C14	107.8 (3)	C20—Fe2—C19	40.29 (12)
240	C17—C18—Fe2	69.47 (16)	C17—Fe2—C19	149.68 (12)
241	C14—C18—Fe2	69.70 (15)	C16—Fe2—C19	169.72 (12)
242	C17—C18—H18	126.1	C22—Fe2—C19	67.94 (12)
243	C14—C18—H18	126.1	C18—Fe2—C19	118.68 (12)

244	Fe2—C18—H18	126.3	C14—Fe2—C19	111.23 (12)
245	C20—C19—C23	108.3 (3)	C21—Fe2—C23	68.23 (12)
246	C20—C19—Fe2	69.38 (17)	C15—Fe2—C23	111.25 (12)
247	C23—C19—Fe2	70.14 (17)	C20—Fe2—C23	68.04 (12)
248	C20—C19—H19	125.8	C17—Fe2—C23	165.92 (12)
249	C23—C19—H19	125.8	C16—Fe2—C23	130.29 (12)
250	Fe2—C19—H19	126.2	C22—Fe2—C23	40.38 (12)
251	C19—C20—C21	108.3 (3)	C18—Fe2—C23	153.47 (12)
252	C19—C20—Fe2	70.33 (17)	C14—Fe2—C23	121.35 (11)
253	C21—C20—Fe2	69.73 (17)	C19—Fe2—C23	40.42 (12)
254	C19—C20—H20	125.8	C1—S1—S2	93.98 (10)
255	C21—C20—H20	125.8	C3—S2—S1	97.18 (10)
256	Fe2—C20—H20	125.7		
257				
258	C14—C1—C2—C3	176.4 (3)	Fe1—C9—C13—C12	-59.4 (2)
259	S1—C1—C2—C3	-4.0 (3)	C10—C9—C13—Fe1	59.30 (19)
260	C14—C1—C2—C4	-6.1 (4)	C2—C1—C14—C18	-51.1 (4)
261	S1—C1—C2—C4	173.5 (2)	S1—C1—C14—C18	129.3 (3)
262	C1—C2—C3—S3	-174.6 (2)	C2—C1—C14—C15	130.3 (3)
263	C4—C2—C3—S3	7.9 (4)	S1—C1—C14—C15	-49.3 (3)
264	C1—C2—C3—S2	7.0 (3)	C2—C1—C14—Fe2	-141.9 (2)
265	C4—C2—C3—S2	-170.5 (2)	S1—C1—C14—Fe2	38.5 (3)
266	C1—C2—C4—C5	146.3 (3)	C18—C14—C15—C16	1.0 (3)
267	C3—C2—C4—C5	-36.4 (4)	C1—C14—C15—C16	179.8 (2)
268	C1—C2—C4—C8	-31.3 (4)	Fe2—C14—C15—C16	59.86 (19)
269	C3—C2—C4—C8	146.0 (3)	C18—C14—C15—Fe2	-58.88 (19)
270	C1—C2—C4—Fe1	-121.3 (3)	C1—C14—C15—Fe2	120.0 (3)
271	C3—C2—C4—Fe1	56.0 (4)	C14—C15—C16—C17	-1.0 (3)
272	C8—C4—C5—C6	0.1 (3)	Fe2—C15—C16—C17	59.0 (2)
273	C2—C4—C5—C6	-177.9 (3)	C14—C15—C16—Fe2	-59.95 (19)
274	Fe1—C4—C5—C6	58.56 (19)	C15—C16—C17—C18	0.6 (3)
275	C8—C4—C5—Fe1	-58.48 (18)	Fe2—C16—C17—C18	59.5 (2)
276	C2—C4—C5—Fe1	123.5 (3)	C15—C16—C17—Fe2	-58.91 (19)
277	C4—C5—C6—C7	0.1 (3)	C16—C17—C18—C14	0.0 (3)
278	Fe1—C5—C6—C7	59.64 (19)	Fe2—C17—C18—C14	59.40 (19)
279	C4—C5—C6—Fe1	-59.53 (18)	C16—C17—C18—Fe2	-59.4 (2)
280	C5—C6—C7—C8	-0.3 (3)	C15—C14—C18—C17	-0.6 (3)
281	Fe1—C6—C7—C8	59.76 (19)	C1—C14—C18—C17	-179.4 (3)
282	C5—C6—C7—Fe1	-60.01 (19)	Fe2—C14—C18—C17	-59.3 (2)
283	C6—C7—C8—C4	0.3 (3)	C15—C14—C18—Fe2	58.62 (19)
284	Fe1—C7—C8—C4	59.56 (19)	C1—C14—C18—Fe2	-120.2 (3)
285	C6—C7—C8—Fe1	-59.26 (19)	C23—C19—C20—C21	0.1 (3)
286	C5—C4—C8—C7	-0.2 (3)	Fe2—C19—C20—C21	59.6 (2)
287	C2—C4—C8—C7	177.8 (2)	C23—C19—C20—Fe2	-59.53 (19)
288	Fe1—C4—C8—C7	-58.74 (19)	C19—C20—C21—C22	-0.3 (3)
289	C5—C4—C8—Fe1	58.50 (17)	Fe2—C20—C21—C22	59.7 (2)
290	C2—C4—C8—Fe1	-123.5 (3)	C19—C20—C21—Fe2	-59.99 (19)
291	C13—C9—C10—C11	-0.1 (3)	C20—C21—C22—C23	0.3 (3)
292	Fe1—C9—C10—C11	59.39 (19)	Fe2—C21—C22—C23	60.0 (2)
293	C13—C9—C10—Fe1	-59.49 (19)	C20—C21—C22—Fe2	-59.7 (2)
294	C9—C10—C11—C12	0.3 (3)	C21—C22—C23—C19	-0.3 (3)
295	Fe1—C10—C11—C12	59.9 (2)	Fe2—C22—C23—C19	59.07 (19)

296	C9—C10—C11—Fe1	−59.62 (19)	C21—C22—C23—Fe2	−59.4 (2)
297	C10—C11—C12—C13	−0.4 (3)	C20—C19—C23—C22	0.1 (3)
298	Fe1—C11—C12—C13	59.3 (2)	Fe2—C19—C23—C22	−58.9 (2)
299	C10—C11—C12—Fe1	−59.69 (19)	C20—C19—C23—Fe2	59.06 (19)
300	C11—C12—C13—C9	0.3 (3)	C2—C1—S1—S2	−0.3 (2)
301	Fe1—C12—C13—C9	59.36 (19)	C14—C1—S1—S2	179.37 (19)
302	C11—C12—C13—Fe1	−59.1 (2)	C2—C3—S2—S1	−6.2 (2)
303	C10—C9—C13—C12	−0.1 (3)	S3—C3—S2—S1	175.17 (15)
