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[μ-2-Methyl-5-(methylsulfanylmethyl)azaferrocene]bis(pentacarbonyltungsten)

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Key indicators

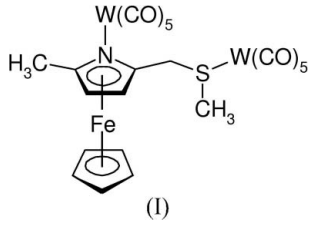
Single-crystal X-ray study
T = 173 K
Mean σ(C–C) = 0.005 Å
R factor = 0.025
wR factor = 0.063
Data-to-parameter ratio = 25.5

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

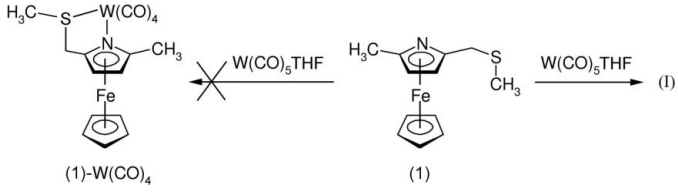
The title compound, $[W_2Fe(C_5H_5)(C_7H_{10}NS)(CO)_{10}]$, was prepared by reaction of 5-methylsulfanylmethyl-2-methyl azaferrocene with photochemically generated $[W(CO)_5]\cdot$THF. THF. The X-ray structural analysis showed the azaferrocenyl unit to have adopted a nearly eclipsed geometry. The geometries at the two tungsten centers are distorted octahedral.

Comment

Unsubstituted azaferrocene behaves as a 2-electron donor ligand towards metal centres and its coordination chemistry has been the subject of numerous studies. The first complexes (with Pd and Pt) were synthesized by Pyshnograeva *et al.* (1984) but were not then structurally characterized. However, X-ray crystallographic study of *trans*-bis(azaferrocene)-dichloropalladium(II) has been reported recently (Jerzykiewicz *et al.*, 2006). Azaferrocene has also been described as a monodendate axial ligand able to coordinate to metal centres in macrocyclic systems such as cobaltoximes and metalloporphyrins (Zakrzewski & Giannotti, 1994, 1995). Best *et al.* (1991) have synthesized and characterized unique examples of cyclometallated azaferrocene complexes. Structural and Mössbauer spectroscopic studies of $M(CO)_5$ (M = Cr, Mo, W) azaferrocene complexes indicate that azaferrocene acts as a moderate σ-donating and rather weak π-accepting ligand (Silver *et al.*, 1997). The $W(CO)_5$ group has recently been reported to enable Friedel–Crafts acylation reactions of azaferrocenes (Kowalski *et al.*, 2005*b*).



(I)



(1)-W(CO)$_4$ (1)

In contrast to the well known coordination chemistry of unsubstituted azaferrocene ligands, the coordination behaviour of its derivatives is still an underdeveloped area.

However, chelate complexes of bisazaferrocene with Ni and Pd (Salo & Guan, 2003) and azaferrocenylimines with Ni (Watanabe, 2005) have recently been described and proven to be catalysts in olefin polymerization.

In the course of our studies on the coordination behaviour of azaferrocene derivatives possessing a second donor atom (P, S or Se) in the side chain we examined the reaction of ligand (1) with photochemically generated $[W(CO)_5]$THF (see reaction scheme). Rather than the expected product (1)-$W(CO)_4$, we isolated the rather unstable title complex, (I) (Fig. 1). The X-ray structural analysis of (I) showed the azaferrocenyl unit to have adopted an eclipsed geometry, the two five-membered rings being staggered by only *ca* 60° (and inclinated by *ca* 40°). The N-bound W atom W2 is only slightly (*ca* 0.06 Å) out of the pyrrole plane. The four carbonyl groups on W2 that are *cis* to N1 adopt a staggered conformation with respect to the C_4N ring. There are no intermolecular interactions of note.

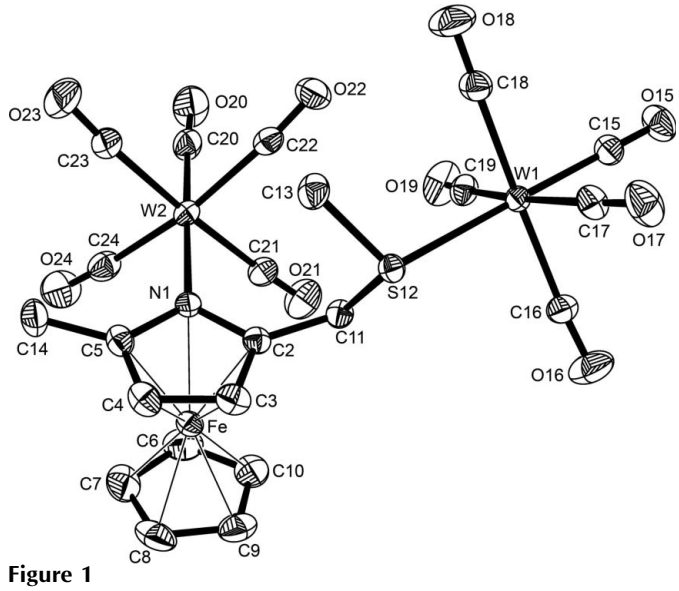


Figure 1
The molecular structure of (I) showing displacement ellipsoids at the 50% probability level. H atoms have been omitted.

Experimental

Ligand (1) was synthesized by the reaction of lithiated 2,5-dimethylazaferrocene with dimethyl disulfide according to a literature procedure (Kowalski & Zakrzewski, 2004; Kowalski *et al.*, 2005*a*). $W(CO)_6$ (200 mg, 0.56 mmol) was then dissolved in THF (50 ml) and was photolysed with a 400 W high-pressure mercury lamp for 30 min. Ligand (1) (130 mg, 0.50 mmol) was added to the photolyte and the resulting solution was stirred at 323 K for 3 h. Removal of solvent gave a red–brown oil which was subjected to column chromatography (eluent: chloroform/hexane (50:2)). Evaporation of the solvent then afforded (I) as an orange solid in 38% yield (172 mg). Crystals of (I) suitable for X-ray analysis were obtained by slow diffusion of hexane into a concentrated solution of the complex in dichloromethane.

Crystal data

$[W_2Fe(C_5H_5)(C_7H_{10}NS)(CO)_{10}]$
M_r = 908.96
Monoclinic, $P2_1/c$
a = 19.3188 (2) Å
b = 9.5893 (1) Å
c = 14.2096 (2) Å
β = 99.795 (1)°
V = 2594.01 (5) Å^3
Z = 4
D_x = 2.327 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 9.53 mm^{-1}
T = 173 (2) K
Block, orange
0.16 × 0.13 × 0.09 mm

Data collection

Oxford Diffraction Xcalibur3 CCD diffractometer
ω scans
Absorption correction: analytical [*CrysAlis RED* (Oxford Diffraction, 2006), based on Clark & Reid (1995)]
T_{min} = 0.275, T_{max} = 0.470
26034 measured reflections
8556 independent reflections
7621 reflections with $I > 2\sigma(I)$
R_{int} = 0.028
θ_{max} = 31.5°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.025
$wR(F^2)$ = 0.063
S = 1.08
8556 reflections
335 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0357P)^2 + 2.3697P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 2.98 e Å^{-3}
$\Delta\rho_{min}$ = −1.32 e Å^{-3}

H atoms were placed in geometrically idealized positions and constrained to ride on their parent atoms with C—H distances in the range 0.95–0.98 Å. For methyl H atoms, $U_{iso}(H) = 1.5U_{eq}(C)$; for all other H atoms, $U_{iso}(H) = 1.2U_{eq}(C)$. The highest peak and deepest hole in the residual electron density are located 1.06 Å and 0.43 Å, respectively, from W2.

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2006); cell refinement: *CrysAlis RED* (Oxford Diffraction, 2006); data reduction: *CrysAlis RED*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1999); software used to prepare material for publication: *SHELXTL*.

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supporting information

Acta Cryst. (2007). E**63**, m392–m393 [https://doi.org/10.1107/S1600536806056121]

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$V = 2594.01$ (5) Å^3
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$F(000) = 1696$
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Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 20170 reflections
$\theta = 3.8$–$32.5°$
$\mu = 9.53$ mm^{-1}
$T = 173$ K
Block, orange
0.16 × 0.13 × 0.09 mm

Data collection

Oxford Diffraction Xcalibur3 CCD diffractometer
Radiation source: Enhance (Mo) X-ray Source
Graphite monochromator
ω scans
Absorption correction: analytical [*CrysAlis RED* (Oxford Diffraction, 2006), based on Clark & Reid (1995)]
$T_{min} = 0.275$, $T_{max} = 0.470$
26034 measured reflections
8556 independent reflections
7621 reflections with $I > 2\sigma(I)$
$R_{int} = 0.028$
$\theta_{max} = 31.5°$, $\theta_{min} = 3.9°$
$h = -28 \rightarrow 28$
$k = -14 \rightarrow 8$
$l = -20 \rightarrow 20$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.025$
$wR(F^2) = 0.063$
$S = 1.08$
8556 reflections
335 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0357P)^2 + 2.3697P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 2.98$ e Å^{-3}
$\Delta\rho_{min} = -1.32$ e Å^{-3}

Special details

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.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit *S* are based on F^2, conventional *R*-factors *R* are based on *F*, with *F* set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on *F*, and *R*- factors based on ALL data will be even larger.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
W1	0.047570 (6)	0.286540 (12)	0.159791 (8)	0.01928 (3)
W2	0.362692 (6)	0.178735 (13)	0.174718 (8)	0.02270 (4)
Fe	0.28944 (2)	−0.17956 (4)	0.07026 (3)	0.02104 (8)
N1	0.30528 (13)	−0.0325 (3)	0.17661 (16)	0.0203 (4)
C2	0.23284 (15)	−0.0603 (3)	0.1455 (2)	0.0210 (5)
C3	0.21824 (18)	−0.2029 (3)	0.1613 (2)	0.0271 (6)
H3A	0.1738	−0.2477	0.1476	0.033*
C4	0.28288 (18)	−0.2649 (3)	0.2014 (2)	0.0278 (6)
H4A	0.2898	−0.3598	0.2197	0.033*
C5	0.33546 (17)	−0.1612 (3)	0.2096 (2)	0.0243 (6)
C6	0.3377 (2)	−0.1232 (4)	−0.0430 (2)	0.0379 (8)
H6A	0.3661	−0.0431	−0.0464	0.046*
C7	0.3612 (2)	−0.2566 (5)	−0.0074 (3)	0.0432 (9)
H7A	0.4084	−0.2818	0.0168	0.052*
C8	0.3022 (3)	−0.3451 (4)	−0.0144 (3)	0.0429 (9)
H8A	0.3027	−0.4401	0.0047	0.051*
C9	0.2426 (2)	−0.2679 (4)	−0.0544 (3)	0.0383 (8)
H9A	0.1957	−0.3017	−0.0674	0.046*
C10	0.2644 (2)	−0.1319 (4)	−0.0721 (2)	0.0362 (8)
H10A	0.2347	−0.0582	−0.0991	0.043*
C11	0.17911 (15)	0.0488 (3)	0.1152 (2)	0.0220 (5)
H11A	0.1528	0.0257	0.0511	0.026*
H11B	0.2026	0.1398	0.1110	0.026*
S12	0.11767 (4)	0.06207 (7)	0.20027 (5)	0.02046 (12)
C13	0.17877 (17)	0.0806 (4)	0.3105 (2)	0.0296 (6)
H13A	0.2050	−0.0065	0.3246	0.044*
H13B	0.1529	0.1016	0.3625	0.044*
H13C	0.2115	0.1568	0.3044	0.044*
C14	0.4108 (2)	−0.1803 (4)	0.2541 (3)	0.0350 (7)
H14A	0.4407	−0.1271	0.2180	0.053*
H14B	0.4230	−0.2794	0.2533	0.053*
H14C	0.4179	−0.1469	0.3203	0.053*
C15	−0.00474 (17)	0.4610 (3)	0.1238 (2)	0.0265 (6)
O15	−0.03425 (15)	0.5626 (3)	0.10160 (18)	0.0382 (6)
C16	0.00134 (18)	0.1998 (3)	0.0333 (2)	0.0288 (6)
O16	−0.02842 (18)	0.1549 (3)	−0.0362 (2)	0.0505 (8)
C17	−0.03686 (18)	0.2191 (3)	0.2146 (2)	0.0279 (6)
O17	−0.08778 (15)	0.1891 (3)	0.2417 (2)	0.0432 (7)
C18	0.08804 (18)	0.3645 (3)	0.2915 (2)	0.0277 (6)
O18	0.10938 (17)	0.4061 (3)	0.36625 (19)	0.0474 (7)

C19	0.12436 (17)	0.3780 (4)	0.0984 (2)	0.0284 (6)
O19	0.16367 (15)	0.4377 (3)	0.0621 (2)	0.0465 (7)
C20	0.41061 (18)	0.3591 (4)	0.1683 (3)	0.0346 (7)
O20	0.43570 (16)	0.4673 (3)	0.1640 (3)	0.0562 (8)
C21	0.31425 (18)	0.2141 (4)	0.0383 (2)	0.0295 (6)
O21	0.28939 (18)	0.2422 (4)	−0.0374 (2)	0.0510 (7)
C22	0.29270 (18)	0.3015 (3)	0.2261 (2)	0.0266 (6)
O22	0.25929 (15)	0.3848 (3)	0.25499 (19)	0.0378 (6)
C23	0.4122 (2)	0.1569 (4)	0.3142 (3)	0.0363 (8)
O23	0.4400 (2)	0.1560 (4)	0.3910 (2)	0.0642 (10)
C24	0.44666 (19)	0.0917 (4)	0.1255 (3)	0.0344 (7)
O24	0.49610 (16)	0.0557 (4)	0.0985 (2)	0.0554 (8)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
W1	0.01935 (6)	0.02064 (6)	0.01746 (5)	0.00163 (4)	0.00199 (4)	−0.00023 (4)
W2	0.02053 (6)	0.02430 (6)	0.02232 (6)	−0.00243 (4)	0.00097 (4)	−0.00156 (4)
Fe	0.0229 (2)	0.02063 (19)	0.02036 (18)	0.00142 (15)	0.00583 (15)	−0.00161 (14)
N1	0.0214 (11)	0.0214 (11)	0.0180 (10)	0.0016 (9)	0.0028 (8)	−0.0016 (8)
C2	0.0212 (12)	0.0225 (12)	0.0203 (12)	−0.0001 (10)	0.0064 (10)	−0.0009 (10)
C3	0.0286 (15)	0.0242 (14)	0.0304 (15)	−0.0041 (11)	0.0108 (12)	−0.0004 (11)
C4	0.0354 (17)	0.0237 (14)	0.0260 (14)	0.0035 (12)	0.0104 (12)	0.0070 (11)
C5	0.0278 (14)	0.0257 (14)	0.0200 (12)	0.0056 (11)	0.0059 (11)	0.0039 (10)
C6	0.047 (2)	0.045 (2)	0.0256 (15)	−0.0054 (17)	0.0195 (15)	−0.0056 (14)
C7	0.040 (2)	0.058 (2)	0.0340 (18)	0.0152 (19)	0.0127 (15)	−0.0101 (18)
C8	0.064 (3)	0.0301 (17)	0.0366 (19)	0.0085 (17)	0.0138 (18)	−0.0086 (15)
C9	0.043 (2)	0.043 (2)	0.0290 (16)	−0.0064 (16)	0.0041 (14)	−0.0121 (14)
C10	0.046 (2)	0.042 (2)	0.0198 (14)	0.0063 (16)	0.0048 (13)	−0.0015 (13)
C11	0.0217 (12)	0.0264 (13)	0.0183 (11)	0.0007 (10)	0.0044 (10)	0.0007 (10)
S12	0.0188 (3)	0.0210 (3)	0.0219 (3)	0.0008 (2)	0.0046 (2)	0.0007 (2)
C13	0.0293 (15)	0.0379 (17)	0.0210 (13)	0.0049 (13)	0.0028 (11)	0.0037 (12)
C14	0.0313 (17)	0.0352 (18)	0.0364 (18)	0.0105 (14)	−0.0003 (14)	0.0052 (14)
C15	0.0293 (15)	0.0289 (15)	0.0209 (13)	0.0055 (12)	0.0033 (11)	0.0020 (11)
O15	0.0465 (15)	0.0350 (13)	0.0328 (12)	0.0164 (11)	0.0062 (11)	0.0057 (10)
C16	0.0297 (16)	0.0283 (15)	0.0268 (14)	−0.0011 (12)	0.0002 (12)	−0.0036 (12)
O16	0.0589 (19)	0.0516 (18)	0.0350 (14)	−0.0099 (15)	−0.0093 (13)	−0.0092 (13)
C17	0.0282 (15)	0.0245 (14)	0.0320 (15)	0.0037 (11)	0.0076 (12)	0.0017 (11)
O17	0.0350 (14)	0.0375 (14)	0.0627 (19)	0.0015 (11)	0.0240 (14)	0.0013 (13)
C18	0.0331 (16)	0.0242 (14)	0.0252 (14)	0.0021 (12)	0.0031 (12)	0.0004 (11)
O18	0.0631 (19)	0.0499 (17)	0.0252 (12)	−0.0014 (14)	−0.0037 (12)	−0.0097 (11)
C19	0.0277 (15)	0.0314 (16)	0.0247 (13)	0.0021 (12)	0.0006 (11)	0.0032 (12)
O19	0.0384 (15)	0.0594 (18)	0.0425 (15)	−0.0119 (13)	0.0092 (12)	0.0184 (13)
C20	0.0254 (15)	0.0319 (17)	0.0445 (19)	−0.0039 (13)	0.0002 (13)	−0.0014 (14)
O20	0.0401 (16)	0.0370 (15)	0.088 (3)	−0.0121 (13)	−0.0002 (15)	0.0030 (16)
C21	0.0294 (16)	0.0308 (16)	0.0282 (15)	−0.0030 (12)	0.0043 (12)	0.0002 (12)
O21	0.0574 (19)	0.0617 (19)	0.0306 (14)	0.0014 (16)	−0.0016 (13)	0.0111 (14)
C22	0.0289 (15)	0.0248 (14)	0.0246 (14)	−0.0046 (12)	−0.0002 (11)	0.0005 (11)

O22	0.0434 (15)	0.0311 (12)	0.0395 (14)	0.0040 (11)	0.0085 (11)	−0.0050 (11)
C23	0.0355 (18)	0.0416 (19)	0.0296 (16)	0.0091 (15)	−0.0006 (13)	−0.0068 (14)
O23	0.062 (2)	0.089 (3)	0.0339 (15)	0.0233 (19)	−0.0127 (14)	−0.0136 (16)
C24	0.0291 (16)	0.0421 (19)	0.0323 (16)	−0.0058 (14)	0.0062 (13)	−0.0062 (14)
O24	0.0332 (15)	0.075 (2)	0.063 (2)	−0.0020 (15)	0.0202 (14)	−0.0206 (17)

Geometric parameters (Å, º)

W1—C15	1.977 (3)	C6—C10	1.408 (6)
W1—C17	2.030 (3)	C6—C7	1.421 (6)
W1—C19	2.043 (3)	C6—H6A	0.950
W1—C16	2.043 (3)	C7—C8	1.410 (6)
W1—C18	2.044 (3)	C7—H7A	0.950
W1—S12	2.5560 (7)	C8—C9	1.405 (6)
W2—C20	1.971 (4)	C8—H8A	0.950
W2—C22	2.021 (3)	C9—C10	1.406 (6)
W2—C21	2.033 (3)	C9—H9A	0.950
W2—C24	2.050 (4)	C10—H10A	0.950
W2—C23	2.060 (4)	C11—S12	1.837 (3)
W2—N1	2.312 (2)	C11—H11A	0.990
Fe—C2	2.011 (3)	C11—H11B	0.990
Fe—C8	2.032 (4)	S12—C13	1.803 (3)
Fe—C9	2.032 (4)	C13—H13A	0.980
Fe—C5	2.036 (3)	C13—H13B	0.980
Fe—C10	2.049 (3)	C13—H13C	0.980
Fe—C7	2.050 (4)	C14—H14A	0.980
Fe—N1	2.052 (2)	C14—H14B	0.980
Fe—C3	2.054 (3)	C14—H14C	0.980
Fe—C4	2.059 (3)	C15—O15	1.145 (4)
Fe—C6	2.064 (3)	C16—O16	1.139 (4)
N1—C5	1.411 (4)	C17—O17	1.152 (4)
N1—C2	1.419 (4)	C18—O18	1.143 (4)
C2—C3	1.422 (4)	C19—O19	1.142 (4)
C2—C11	1.484 (4)	C20—O20	1.152 (4)
C3—C4	1.413 (5)	C21—O21	1.134 (4)
C3—H3A	0.950	C22—O22	1.146 (4)
C4—C5	1.412 (5)	C23—O23	1.132 (5)
C4—H4A	0.950	C24—O24	1.141 (4)
C5—C14	1.496 (5)		
C15—W1—C17	87.73 (13)	C3—C2—C11	125.1 (3)
C15—W1—C19	84.40 (13)	N1—C2—Fe	71.09 (15)
C17—W1—C19	172.10 (13)	C3—C2—Fe	71.17 (17)
C15—W1—C16	89.36 (13)	C11—C2—Fe	131.6 (2)
C17—W1—C16	87.06 (14)	C4—C3—C2	106.4 (3)
C19—W1—C16	92.24 (13)	C4—C3—Fe	70.09 (18)
C15—W1—C18	91.54 (13)	C2—C3—Fe	67.90 (17)
C17—W1—C18	88.40 (14)	C4—C3—H3A	126.8

C19—W1—C18	92.41 (13)	C2—C3—H3A	126.8
C16—W1—C18	175.33 (13)	Fe—C3—H3A	126.8
C15—W1—S12	177.61 (9)	C5—C4—C3	108.1 (3)
C17—W1—S12	94.29 (9)	C5—C4—Fe	68.94 (17)
C19—W1—S12	93.57 (9)	C3—C4—Fe	69.73 (18)
C16—W1—S12	89.48 (10)	C5—C4—H4A	125.9
C18—W1—S12	89.79 (9)	C3—C4—H4A	125.9
C20—W2—C22	81.59 (14)	Fe—C4—H4A	127.0
C20—W2—C21	87.34 (14)	N1—C5—C4	109.7 (3)
C22—W2—C21	91.13 (13)	N1—C5—C14	124.4 (3)
C20—W2—C24	86.36 (15)	C4—C5—C14	125.7 (3)
C22—W2—C24	167.79 (14)	N1—C5—Fe	70.42 (15)
C21—W2—C24	90.28 (14)	C4—C5—Fe	70.72 (18)
C20—W2—C23	89.37 (16)	C14—C5—Fe	129.3 (2)
C22—W2—C23	86.63 (14)	C10—C6—C7	107.1 (4)
C21—W2—C23	176.26 (14)	C10—C6—Fe	69.42 (19)
C24—W2—C23	91.29 (15)	C7—C6—Fe	69.3 (2)
C20—W2—N1	178.05 (13)	C10—C6—H6A	126.4
C22—W2—N1	98.88 (11)	C7—C6—H6A	126.4
C21—W2—N1	90.75 (11)	Fe—C6—H6A	126.4
C24—W2—N1	93.22 (12)	C8—C7—C6	108.2 (4)
C23—W2—N1	92.55 (13)	C8—C7—Fe	69.1 (2)
C2—Fe—C8	152.97 (16)	C6—C7—Fe	70.3 (2)
C2—Fe—C9	119.93 (14)	C8—C7—H7A	125.9
C8—Fe—C9	40.46 (17)	C6—C7—H7A	125.9
C2—Fe—C5	67.71 (12)	Fe—C7—H7A	126.3
C8—Fe—C5	124.51 (15)	C9—C8—C7	107.9 (4)
C9—Fe—C5	160.08 (15)	C9—C8—Fe	69.8 (2)
C2—Fe—C10	109.85 (14)	C7—C8—Fe	70.5 (2)
C8—Fe—C10	67.79 (16)	C9—C8—H8A	126.0
C9—Fe—C10	40.29 (16)	C7—C8—H8A	126.0
C5—Fe—C10	158.64 (15)	Fe—C8—H8A	125.2
C2—Fe—C7	165.77 (16)	C8—C9—C10	108.1 (4)
C8—Fe—C7	40.42 (18)	C8—C9—Fe	69.7 (2)
C9—Fe—C7	67.77 (17)	C10—C9—Fe	70.5 (2)
C5—Fe—C7	109.37 (15)	C8—C9—H9A	125.9
C10—Fe—C7	67.43 (16)	C10—C9—H9A	125.9
C2—Fe—N1	40.88 (10)	Fe—C9—H9A	125.4
C8—Fe—N1	162.78 (15)	C9—C10—C6	108.7 (4)
C9—Fe—N1	156.53 (14)	C9—C10—Fe	69.2 (2)
C5—Fe—N1	40.38 (11)	C6—C10—Fe	70.6 (2)
C10—Fe—N1	123.66 (13)	C9—C10—H10A	125.7
C7—Fe—N1	127.91 (15)	C6—C10—H10A	125.7
C2—Fe—C3	40.93 (12)	Fe—C10—H10A	126.1
C8—Fe—C3	116.70 (16)	C2—C11—S12	110.89 (19)
C9—Fe—C3	104.90 (15)	C2—C11—H11A	109.5
C5—Fe—C3	68.00 (13)	S12—C11—H11A	109.5
C10—Fe—C3	125.10 (15)	C2—C11—H11B	109.5

C7—Fe—C3	152.36 (17)	S12—C11—H11B	109.5
N1—Fe—C3	69.09 (11)	H11A—C11—H11B	108.1
C2—Fe—C4	67.80 (12)	C13—S12—C11	100.25 (14)
C8—Fe—C4	104.93 (15)	C13—S12—W1	110.86 (12)
C9—Fe—C4	122.47 (15)	C11—S12—W1	106.69 (10)
C5—Fe—C4	40.34 (13)	S12—C13—H13A	109.5
C10—Fe—C4	160.29 (15)	S12—C13—H13B	109.5
C7—Fe—C4	119.71 (16)	H13A—C13—H13B	109.5
N1—Fe—C4	68.34 (12)	S12—C13—H13C	109.5
C3—Fe—C4	40.17 (13)	H13A—C13—H13C	109.5
C2—Fe—C6	128.59 (14)	H13B—C13—H13C	109.5
C8—Fe—C6	68.09 (17)	C5—C14—H14A	109.5
C9—Fe—C6	67.83 (16)	C5—C14—H14B	109.5
C5—Fe—C6	123.72 (15)	H14A—C14—H14B	109.5
C10—Fe—C6	40.03 (16)	C5—C14—H14C	109.5
C7—Fe—C6	40.40 (17)	H14A—C14—H14C	109.5
N1—Fe—C6	111.32 (13)	H14B—C14—H14C	109.5
C3—Fe—C6	163.52 (16)	O15—C15—W1	178.7 (3)
C4—Fe—C6	156.23 (15)	O16—C16—W1	175.6 (3)
C5—N1—C2	105.6 (2)	O17—C17—W1	174.6 (3)
C5—N1—Fe	69.21 (16)	O18—C18—W1	178.2 (3)
C2—N1—Fe	68.02 (15)	O19—C19—W1	174.5 (3)
C5—N1—W2	126.8 (2)	O20—C20—W2	176.9 (3)
C2—N1—W2	127.62 (18)	O21—C21—W2	175.4 (3)
Fe—N1—W2	127.27 (11)	O22—C22—W2	171.0 (3)
N1—C2—C3	110.0 (3)	O23—C23—W2	174.5 (4)
N1—C2—C11	124.2 (3)	O24—C24—W2	173.5 (3)

C2—Fe—N1—C5	117.6 (2)	C3—Fe—C5—N1	−83.16 (18)
C8—Fe—N1—C5	−32.8 (5)	C4—Fe—C5—N1	−120.3 (2)
C9—Fe—N1—C5	159.3 (3)	C6—Fe—C5—N1	83.7 (2)
C10—Fe—N1—C5	−160.8 (2)	C2—Fe—C5—C4	81.48 (19)
C7—Fe—N1—C5	−74.5 (2)	C8—Fe—C5—C4	−70.9 (3)
C3—Fe—N1—C5	80.23 (19)	C9—Fe—C5—C4	−35.4 (5)
C4—Fe—N1—C5	36.98 (18)	C10—Fe—C5—C4	169.2 (4)
C6—Fe—N1—C5	−117.5 (2)	C7—Fe—C5—C4	−113.4 (2)
C8—Fe—N1—C2	−150.5 (5)	N1—Fe—C5—C4	120.3 (2)
C9—Fe—N1—C2	41.7 (4)	C3—Fe—C5—C4	37.13 (18)
C5—Fe—N1—C2	−117.6 (2)	C6—Fe—C5—C4	−156.0 (2)
C10—Fe—N1—C2	81.6 (2)	C2—Fe—C5—C14	−157.7 (3)
C7—Fe—N1—C2	167.8 (2)	C8—Fe—C5—C14	49.8 (4)
C3—Fe—N1—C2	−37.41 (17)	C9—Fe—C5—C14	85.4 (5)
C4—Fe—N1—C2	−80.66 (18)	C10—Fe—C5—C14	−70.1 (5)
C6—Fe—N1—C2	124.91 (19)	C7—Fe—C5—C14	7.3 (4)
C2—Fe—N1—W2	−121.3 (2)	N1—Fe—C5—C14	−118.9 (4)
C8—Fe—N1—W2	88.2 (5)	C3—Fe—C5—C14	157.9 (3)
C9—Fe—N1—W2	−79.6 (4)	C4—Fe—C5—C14	120.8 (4)
C5—Fe—N1—W2	121.0 (2)	C6—Fe—C5—C14	−35.3 (4)

C10—Fe—N1—W2	−39.7 (2)	C2—Fe—C6—C10	−74.1 (3)
C7—Fe—N1—W2	46.5 (2)	C8—Fe—C6—C10	81.1 (3)
C3—Fe—N1—W2	−158.75 (18)	C9—Fe—C6—C10	37.3 (2)
C4—Fe—N1—W2	158.00 (18)	C5—Fe—C6—C10	−161.1 (2)
C6—Fe—N1—W2	3.6 (2)	C7—Fe—C6—C10	118.6 (3)
C22—W2—N1—C5	−135.2 (2)	N1—Fe—C6—C10	−117.4 (2)
C21—W2—N1—C5	133.6 (2)	C3—Fe—C6—C10	−29.2 (6)
C24—W2—N1—C5	43.3 (2)	C4—Fe—C6—C10	158.2 (3)
C23—W2—N1—C5	−48.2 (2)	C2—Fe—C6—C7	167.4 (2)
C22—W2—N1—C2	44.6 (2)	C8—Fe—C6—C7	−37.5 (3)
C21—W2—N1—C2	−46.7 (2)	C9—Fe—C6—C7	−81.3 (3)
C24—W2—N1—C2	−137.0 (2)	C5—Fe—C6—C7	80.3 (3)
C23—W2—N1—C2	131.6 (2)	C10—Fe—C6—C7	−118.6 (3)
C22—W2—N1—Fe	134.18 (16)	N1—Fe—C6—C7	124.0 (2)
C21—W2—N1—Fe	42.92 (16)	C3—Fe—C6—C7	−147.8 (5)
C24—W2—N1—Fe	−47.40 (17)	C4—Fe—C6—C7	39.6 (5)
C23—W2—N1—Fe	−138.84 (17)	C10—C6—C7—C8	−0.5 (4)
C5—N1—C2—C3	1.5 (3)	Fe—C6—C7—C8	58.9 (3)
Fe—N1—C2—C3	60.8 (2)	C10—C6—C7—Fe	−59.3 (2)
W2—N1—C2—C3	−178.33 (19)	C2—Fe—C7—C8	−163.5 (5)
C5—N1—C2—C11	172.7 (3)	C9—Fe—C7—C8	−38.0 (2)
Fe—N1—C2—C11	−128.0 (3)	C5—Fe—C7—C8	120.9 (2)
W2—N1—C2—C11	−7.1 (4)	C10—Fe—C7—C8	−81.8 (3)
C5—N1—C2—Fe	−59.31 (18)	N1—Fe—C7—C8	162.3 (2)
W2—N1—C2—Fe	120.89 (18)	C3—Fe—C7—C8	41.5 (4)
C8—Fe—C2—N1	161.3 (3)	C4—Fe—C7—C8	77.7 (3)
C9—Fe—C2—N1	−162.20 (18)	C6—Fe—C7—C8	−119.5 (4)
C5—Fe—C2—N1	38.33 (15)	C2—Fe—C7—C6	−44.1 (7)
C10—Fe—C2—N1	−118.91 (18)	C8—Fe—C7—C6	119.5 (4)
C7—Fe—C2—N1	−42.6 (6)	C9—Fe—C7—C6	81.4 (3)
C3—Fe—C2—N1	120.0 (2)	C5—Fe—C7—C6	−119.7 (2)
C4—Fe—C2—N1	82.08 (18)	C10—Fe—C7—C6	37.7 (2)
C6—Fe—C2—N1	−77.8 (2)	N1—Fe—C7—C6	−78.2 (3)
C8—Fe—C2—C3	41.3 (4)	C3—Fe—C7—C6	161.0 (3)
C9—Fe—C2—C3	77.8 (2)	C4—Fe—C7—C6	−162.8 (2)
C5—Fe—C2—C3	−81.6 (2)	C6—C7—C8—C9	0.4 (4)
C10—Fe—C2—C3	121.1 (2)	Fe—C7—C8—C9	60.1 (3)
C7—Fe—C2—C3	−162.5 (6)	C6—C7—C8—Fe	−59.7 (2)
N1—Fe—C2—C3	−120.0 (2)	C2—Fe—C8—C9	52.7 (4)
C4—Fe—C2—C3	−37.9 (2)	C5—Fe—C8—C9	162.2 (2)
C6—Fe—C2—C3	162.2 (2)	C10—Fe—C8—C9	−37.7 (2)
C8—Fe—C2—C11	−79.4 (4)	C7—Fe—C8—C9	−118.5 (4)
C9—Fe—C2—C11	−42.9 (3)	N1—Fe—C8—C9	−172.6 (4)
C5—Fe—C2—C11	157.7 (3)	C3—Fe—C8—C9	81.6 (3)
C10—Fe—C2—C11	0.4 (3)	C4—Fe—C8—C9	122.9 (2)
C7—Fe—C2—C11	76.8 (7)	C6—Fe—C8—C9	−81.1 (3)
N1—Fe—C2—C11	119.3 (3)	C2—Fe—C8—C7	171.2 (3)
C3—Fe—C2—C11	−120.7 (4)	C9—Fe—C8—C7	118.5 (4)

C4—Fe—C2—C11	−158.6 (3)	C5—Fe—C8—C7	−79.3 (3)
C6—Fe—C2—C11	41.5 (3)	C10—Fe—C8—C7	80.8 (3)
N1—C2—C3—C4	−0.9 (3)	N1—Fe—C8—C7	−54.0 (6)
C11—C2—C3—C4	−172.1 (3)	C3—Fe—C8—C7	−159.9 (2)
Fe—C2—C3—C4	59.8 (2)	C4—Fe—C8—C7	−118.6 (3)
N1—C2—C3—Fe	−60.74 (19)	C6—Fe—C8—C7	37.5 (3)
C11—C2—C3—Fe	128.1 (3)	C7—C8—C9—C10	−0.2 (4)
C2—Fe—C3—C4	−118.2 (3)	Fe—C8—C9—C10	60.3 (2)
C8—Fe—C3—C4	81.5 (2)	C7—C8—C9—Fe	−60.5 (3)
C9—Fe—C3—C4	123.1 (2)	C2—Fe—C9—C8	−155.4 (2)
C5—Fe—C3—C4	−37.28 (19)	C5—Fe—C9—C8	−47.7 (5)
C10—Fe—C3—C4	162.0 (2)	C10—Fe—C9—C8	118.8 (4)
C7—Fe—C3—C4	52.7 (4)	C7—Fe—C9—C8	38.0 (3)
N1—Fe—C3—C4	−80.8 (2)	N1—Fe—C9—C8	174.5 (3)
C6—Fe—C3—C4	−175.4 (5)	C3—Fe—C9—C8	−113.9 (3)
C8—Fe—C3—C2	−160.4 (2)	C4—Fe—C9—C8	−74.0 (3)
C9—Fe—C3—C2	−118.8 (2)	C6—Fe—C9—C8	81.8 (3)
C5—Fe—C3—C2	80.89 (19)	C2—Fe—C9—C10	85.8 (2)
C10—Fe—C3—C2	−79.8 (2)	C8—Fe—C9—C10	−118.8 (4)
C7—Fe—C3—C2	170.9 (3)	C5—Fe—C9—C10	−166.5 (4)
N1—Fe—C3—C2	37.37 (17)	C7—Fe—C9—C10	−80.8 (3)
C4—Fe—C3—C2	118.2 (3)	N1—Fe—C9—C10	55.6 (4)
C6—Fe—C3—C2	−57.2 (6)	C3—Fe—C9—C10	127.3 (2)
C2—C3—C4—C5	0.0 (3)	C4—Fe—C9—C10	167.2 (2)
Fe—C3—C4—C5	58.4 (2)	C6—Fe—C9—C10	−37.1 (2)
C2—C3—C4—Fe	−58.4 (2)	C8—C9—C10—C6	−0.1 (4)
C2—Fe—C4—C5	−81.24 (19)	Fe—C9—C10—C6	59.7 (2)
C8—Fe—C4—C5	126.3 (2)	C8—C9—C10—Fe	−59.8 (3)
C9—Fe—C4—C5	166.5 (2)	C7—C6—C10—C9	0.4 (4)
C10—Fe—C4—C5	−168.3 (4)	Fe—C6—C10—C9	−58.9 (2)
C7—Fe—C4—C5	85.3 (2)	C7—C6—C10—Fe	59.3 (2)
N1—Fe—C4—C5	−37.01 (17)	C2—Fe—C10—C9	−113.2 (2)
C3—Fe—C4—C5	−119.8 (3)	C8—Fe—C10—C9	37.9 (3)
C6—Fe—C4—C5	56.9 (4)	C5—Fe—C10—C9	167.4 (3)
C2—Fe—C4—C3	38.60 (18)	C7—Fe—C10—C9	81.8 (3)
C8—Fe—C4—C3	−113.9 (2)	N1—Fe—C10—C9	−156.7 (2)
C9—Fe—C4—C3	−73.7 (2)	C3—Fe—C10—C9	−69.9 (3)
C5—Fe—C4—C3	119.8 (3)	C4—Fe—C10—C9	−33.8 (5)
C10—Fe—C4—C3	−48.4 (5)	C6—Fe—C10—C9	119.8 (3)
C7—Fe—C4—C3	−154.9 (2)	C2—Fe—C10—C6	127.0 (2)
N1—Fe—C4—C3	82.83 (19)	C8—Fe—C10—C6	−81.9 (3)
C6—Fe—C4—C3	176.8 (3)	C9—Fe—C10—C6	−119.8 (3)
C2—N1—C5—C4	−1.5 (3)	C5—Fe—C10—C6	47.6 (5)
Fe—N1—C5—C4	−60.0 (2)	C7—Fe—C10—C6	−38.0 (2)
W2—N1—C5—C4	178.35 (19)	N1—Fe—C10—C6	83.5 (2)
C2—N1—C5—C14	−176.6 (3)	C3—Fe—C10—C6	170.2 (2)
Fe—N1—C5—C14	124.8 (3)	C4—Fe—C10—C6	−153.6 (4)
W2—N1—C5—C14	3.2 (4)	N1—C2—C11—S12	−112.4 (3)

C2—N1—C5—Fe	58.54 (17)	C3—C2—C11—S12	57.5 (3)
W2—N1—C5—Fe	−121.66 (18)	Fe—C2—C11—S12	153.07 (18)
C3—C4—C5—N1	0.9 (3)	C2—C11—S12—C13	51.9 (2)
Fe—C4—C5—N1	59.8 (2)	C2—C11—S12—W1	167.45 (18)
C3—C4—C5—C14	176.0 (3)	C17—W1—S12—C13	−97.47 (15)
Fe—C4—C5—C14	−125.1 (3)	C19—W1—S12—C13	83.31 (15)
C3—C4—C5—Fe	−58.9 (2)	C16—W1—S12—C13	175.52 (15)
C2—Fe—C5—N1	−38.80 (16)	C18—W1—S12—C13	−9.09 (15)
C8—Fe—C5—N1	168.8 (2)	C17—W1—S12—C11	154.29 (14)
C9—Fe—C5—N1	−155.6 (4)	C19—W1—S12—C11	−24.94 (14)
C10—Fe—C5—N1	48.9 (4)	C16—W1—S12—C11	67.28 (14)
C7—Fe—C5—N1	126.3 (2)	C18—W1—S12—C11	−117.34 (14)