Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

1,1′-Bis(ferrocenylphenylphosphino)-ferrocene

Lars S. von Chrzanowski, Martin Lutz* and Anthony L. Spek

Bijvoet Center for Biomolecular Research, Crystal and Structural Chemistry, Utrecht University, Padualaan 8, 3584 CH Utrecht, The Netherlands
Correspondence e-mail: m.lutz@chem.uu.nl

Received 25 June 2007; accepted 4 July 2007

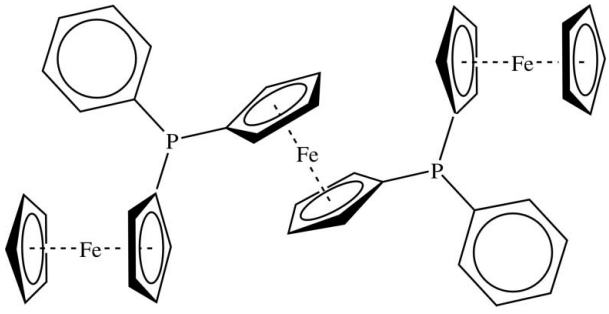
Key indicators: single-crystal X-ray study; T = 150 K; mean σ(C–C) = 0.004 Å; R factor = 0.030; wR factor = 0.076; data-to-parameter ratio = 13.5.

In the title compound, $[Fe_3(C_5H_5)_2(C_{16}H_{13}P)_2]$, two $(C_6H_5)P[(C_5H_4)Fe(C_5H_5)](C_5H_4)$ units are bridged by an Fe atom, located on an inversion center, giving a central third ferrocene unit. Each Fe atom is coordinated by two cyclopentadienyl rings in a parallel sandwich fashion, with twisted conformations. The geometry of the phosphine P atom is pseudo-tetrahedral, defined by a phenyl ring, two cyclopentadienyl rings and an electron pair.

Related literature

The synthesis of the title compound is described by Nettekoven *et al.* (2001). Related structures are reported by Houlton *et al.* (1990) and Nettekoven *et al.* (2003).

For related literature, see: Haaland (1979).



Experimental

Crystal data

$[Fe_3(C_5H_5)_2(C_{16}H_{13}P)_2]$
M_r = 770.20
Triclinic, $P\overline{1}$
a = 5.9308 (1) Å
b = 9.5875 (2) Å
c = 15.7529 (3) Å
α = 82.2873 (10)°
β = 89.4928 (9)°
γ = 72.8206 (9)°
V = 847.58 (3) Å^3
Z = 1
Mo $K\alpha$ radiation
μ = 1.39 mm^{-1}
T = 150 (2) K
0.33 × 0.27 × 0.12 mm

Data collection

Nonius KappaCCD diffractometer
Absorption correction: multi-scan (*MULABS* routine of *PLATON*; Spek, 2003)
T_{min} = 0.77, T_{max} = 0.84
19960 measured reflections
3872 independent reflections
3567 reflections with $I > 2\sigma(I)$
R_{int} = 0.038

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.030
$wR(F^2)$ = 0.077
S = 1.03
3872 reflections
286 parameters
All H-atom parameters refined
$\Delta\rho_{max}$ = 1.02 e Å^{-3}
$\Delta\rho_{min}$ = −0.37 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Fe1—C2	2.0451 (17)	Fe2—C15	2.044 (2)
Fe1—C4	2.0460 (19)	Fe2—C8	2.0502 (19)
Fe1—C3	2.0487 (18)	Fe2—C13	2.051 (2)
Fe1—C5	2.0488 (17)	Fe2—C14	2.055 (2)
Fe1—C1	2.0513 (16)	Fe2—C6	2.0556 (17)
Fe2—C11	2.028 (3)	Fe2—C9	2.058 (2)
Fe2—C12	2.032 (2)	P1—C6	1.8130 (18)
Fe2—C10	2.0433 (18)	P1—C1	1.8210 (18)
Fe2—C7	2.0435 (18)	P1—C16	1.8361 (19)
C6—P1—C1	100.46 (8)	C1—P1—C16	100.66 (9)
C6—P1—C16	103.22 (8)		

Data collection: *COLLECT* (Nonius, 1999); cell refinement: *DENZO* (Version 1.11.0; Otwinowski & Minor, 1997); data reduction: *DENZO*; program(s) used to solve structure: *DIRDIF97* (Beurskens *et al.*, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97*.

This work was supported by the Council for Chemical Sciences of the Netherlands Organization for Scientific Research (CW-NWO) (ML and ALS). Crystals were kindly supplied by Professor Piet W. N. M. van Leeuwen and Dr Ulrike Nettekoven, University of Amsterdam, The Netherlands.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: NC2045).

References

Beurskens, P. T., Admiraal, G., Beurskens, G., Bosman, W. P., Garcia-Granda, S., Gould, R. O., Smits, J. M. M. & Smykalla, C. (1997). *The DIRDIF97 Program System*. Technical Report. Crystallography Laboratory, University of Nijmegen, The Netherlands.
Haaland, A. (1979). *Acc. Chem. Res.* **12**, 415–422.
Houlton, A., Roberts, R. M. G. & Silver, J. (1990). *J. Chem. Soc. Dalton Trans.* pp. 1543–1547.
Nettekoven, U., Widhalm, M., Kalchhauser, H., Kamer, P. C. J., van Leeuwen, P. W. N. M., Lutz, M. & Spek, A. L. (2001). *J. Org. Chem.* **66**, 759–770.
Nettekoven, U., Widhalm, M. & Mereiter, K. (2003). Private communication (refcode ILUVAM). CCDC, Union Road, Cambridge, England.
Nonius (1999). *COLLECT*. Nonius BV, Delft, The Netherlands.
Otwinowski, Z. & Minor, W. (1997). *Methods in Enzymology*, Vol. 276, *Macromolecular Crystallography*, Part A, edited by C. W. Carter Jr & R. M. Sweet, pp. 307–326. New York: Academic Press.
Sheldrick, G. M. (1997). *SHELXL97*. University of Göttingen, Germany.
Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.

  doi:10.1107/S160053680703262X

supplementary materials

Acta Cryst. (2007). E**63**, m2098 [**doi:10.1107/S160053680703262X**]

1,1'-Bis(ferrocenylphenylphosphino)ferrocene

L. S. von Chrzanowski, M. Lutz and A. L. Spek

Comment

Phosphorous-chiral ligands based on ferrocene play important roles in stereoselective synthesis and asymmetric catalysis. In this context we report here the crystal structure of (I).

The title compound, (I), consists of two $(C_6H_5)P[(C_5H_4)Fe(C_5H_4)](C_5H_4)$ units, which are connected *via* Fe1 into a substituted ferrocene (Fig. 1). Fe1 is located on an inversion center. Each Fe atom is coordinated in a sandwich fashion by two cyclopentadienyl (Cp) rings. Fe1—C(Cp) distances range from 2.0451 (17) to 2.0513 (16) Å, the variation of the Fe2—C(Cp) bond length is with 2.028 (3) to 2.058 (2) Å wider. This ranges compares well to the related $(Ph)P[(Cp)Fe(Cp)]_2$ complex [Fe—C = 1.999 (12) – 2.060 (11)] reported by Houlton *et al.* (1990).

The planes of the Cp rings are standing parallel on top of each other, with the largest deviation from parallelity of 2.24 (15) ° for Cp2 (C6–C10) with respect to Cp3 (C11—C15). The conformation of the Cp rings are twisted, the Cp–C atoms do not cover each other as can be seen in plots viewed perpendicular to the Cp rings in Fig. 2. This rotation is expressed by variation of the C—*Cg*—*Cg*—C (*Cg* is the centroid of the Cp rings) torsions angles. The rotation of Cp2 with respect to Cp3 is with 23.13 [C6—*Cg*3—*Cg*1—C15] to 23.61 ° [C7—*Cg*2—*Cg*3—C11] smaller compared to the rotation of *Cg*1 to $Cg1^{i}$ [symmetry code: (i) $1-x, 1-y, 1-z$] with 35.48 ° [C2—*Cg*1—$Cg1^{i}$—$C4^{i}$] to 36.01 ° [C1—*Cg*1—$Cg1^{i}$—$C4^{i}$]. Since the first structure determinations on metallocenes there have been discussions, whether the Cp–H atoms are bent away from or towards to the metal (Haaland, 1979). In (I) all Cp–H atoms are found in the planes of the corresponding Cp–C atoms within standard uncertainties.

The phosphine P is in a tetrahedral environment, defined by a phenyl (Ph) ring, two cyclopentadienyl rings and an electron pair. C—P—C angles are less than the ideal value of 109°, they vary between 100.46 (8) and 103.22 (8)°. P—C(Cp) distances are with 1.8130 (18) and 1.8210 (18) Å shorter than the P—C(Ph) distance [1.8361 (19) Å]. Again the P geometry is similar to the related $(Ph)P[(Cp)Fe(Cp)]_2$ complex, where bond lengths and angles agree well with those in (I), the longest P—C distance [1.843 (11) Å] corresponds again to P—C(Ph).

In a similar structure, where the phosphine P is protected by a BH_3 group (Nettekoven *et al.*, 2003), the C—P—C angles range from 102.29 (12) to 108.10 (11)° and the P geometry is therefore closer to tetrahedral. P—C(Cp) and P—C(Ph) bond distances are shorter than in (I) [P—C(Cp) = 1.786 (3), 1.798 (3) Å; P—C(Ph) = 1.820 (2) Å].

Experimental

The synthesis of the title compound, (I), is described by Nettekoven *et al.* (2001). Orange crystals for data collection were obtained after recrystallization from CH_2Cl_2/hexane (ratio 1:1).

Refinement

All hydrogen atoms were located in the difference Fourier map and where refined freely with isotropic displacement parameters.

The final residual density of 1.021 e $Å^{-3}$ is located 1.77 Å from C20.

Atoms C11–C13 belong to the unsubstituted, only to Fe2 coordinated Cp ring C11–C15 and show anomalous anisotropic displacement parameters along their chemical bonds. This is due to the higher ability of rotation compared to Cp rings C1–C5 and C6–C10.

Figures

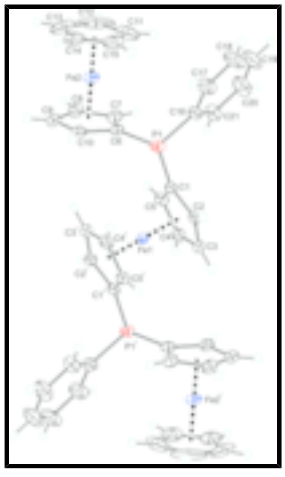


Fig. 1. : Molecular structure of (I) with the atomic numbering scheme. Displacement ellipsoids are drawn at the 50% probability level. [Symmetry code: (i) 1 − *x*, 1 − *y*, 1 − *z*]

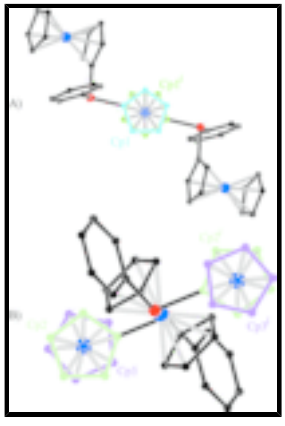


Fig. 2. : The conformation of the cyclopentadienyl (Cp) rings in (I). Hydrogen atoms have been omitted for clarity. A) View perpendicular to the Cp1-ring-plane. Color scheme: light blue Cp1, green $Cp1^{i}$ (Cp1 = C1–C5). B) View perpendicular to the Cp2- and Cp3-ring-plane. Color scheme: light green Cp2 and $Cp2^{i}$, pink Cp3 and $Cp3^{i}$ (Cp2 = C6–C10 and Cp3 = C11–C15). [Symmetry code: (i) 1 − *x*, 1 − *y*, 1 − *z*]

1,1'-Bis(ferrocenylphenylphosphino)ferrocene

Crystal data

$[Fe_3(C_5H_5)_2(C_{16}H_{13}P)_2]$	$Z = 1$
$M_r = 770.20$	$F_{000} = 396$

Triclinic, $P\bar{1}$	D_x = 1.509 Mg m^{-3}
Hall symbol: -P 1	Mo $K\alpha$ radiation λ = 0.71073 Å
a = 5.9308 (1) Å	Cell parameters from 33761 reflections
b = 9.5875 (2) Å	θ = 1.0–27.5º
c = 15.7529 (3) Å	μ = 1.39 mm^{-1}
α = 82.2873 (10)º	T = 150 (2) K
β = 89.4928 (9)º	Block, orange
γ = 72.8206 (9)º	0.33 × 0.27 × 0.12 mm
V = 847.58 (3) Å^3	

Data collection

Nonius KappaCCD diffractometer	3872 independent reflections
Radiation source: rotating anode	3567 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.038
T = 150(2) K	θ_{max} = 27.4º
φ and ω scans	θ_{min} = 2.2º
Absorption correction: multi-scan (MULABS routine of PLATON; Spek, 2003)	h = −7→7
T_{min} = 0.77, T_{max} = 0.84	k = −12→12
19960 measured reflections	l = −20→20

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)]$ = 0.030	All H-atom parameters refined
$wR(F^2)$ = 0.077	$w = 1/[\sigma^2(F_o^2) + (0.0331P)^2 + 0.7293P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.03	$(\Delta/\sigma)_{max}$ = 0.004
3872 reflections	$\Delta\rho_{max}$ = 1.02 e Å^{-3}
286 parameters	$\Delta\rho_{min}$ = −0.37 e Å^{-3}
Primary atom site location: heavy-atom method	Extinction correction: none

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 I>2σ(I) 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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
Fe1	0.5000	0.5000	0.5000	0.01673 (9)
Fe2	0.20709 (4)	1.07610 (3)	0.274313 (16)	0.02367 (9)
P1	0.56465 (7)	0.70824 (5)	0.31173 (3)	0.02101 (11)
C1	0.4763 (3)	0.55601 (19)	0.36935 (11)	0.0191 (3)
C2	0.6404 (3)	0.4122 (2)	0.39305 (11)	0.0228 (4)
C3	0.5199 (4)	0.3205 (2)	0.43963 (12)	0.0263 (4)
C4	0.2802 (4)	0.4057 (2)	0.44521 (12)	0.0256 (4)
C5	0.2523 (3)	0.5499 (2)	0.40199 (11)	0.0212 (3)
C6	0.3239 (3)	0.86348 (19)	0.33599 (11)	0.0203 (3)
C7	0.0769 (3)	0.9040 (2)	0.31389 (12)	0.0228 (4)
C8	−0.0417 (3)	1.0281 (2)	0.35466 (13)	0.0273 (4)
C9	0.1280 (4)	1.0664 (2)	0.40202 (13)	0.0278 (4)
C10	0.3532 (3)	0.9663 (2)	0.39040 (12)	0.0249 (4)
C11	0.2321 (7)	1.0950 (4)	0.14502 (16)	0.0615 (9)
C12	0.0377 (6)	1.2089 (5)	0.1681 (2)	0.0776 (14)
C13	0.1247 (5)	1.2925 (3)	0.2200 (2)	0.0550 (8)
C14	0.3699 (4)	1.2309 (3)	0.22715 (15)	0.0399 (5)
C15	0.4347 (5)	1.1115 (3)	0.18153 (15)	0.0432 (6)
C16	0.4953 (3)	0.6923 (2)	0.20074 (12)	0.0266 (4)
C17	0.6397 (4)	0.7335 (3)	0.13786 (15)	0.0407 (5)
C18	0.5986 (5)	0.7260 (3)	0.05218 (15)	0.0544 (7)
C19	0.4187 (5)	0.6751 (4)	0.02877 (15)	0.0565 (7)
C20	0.2766 (5)	0.6306 (3)	0.09029 (16)	0.0495 (6)
C21	0.3154 (4)	0.6396 (3)	0.17618 (14)	0.0344 (4)
H2	0.800 (4)	0.391 (2)	0.3808 (14)	0.028 (6)*
H3	0.583 (4)	0.225 (3)	0.4641 (14)	0.030 (6)*
H4	0.164 (5)	0.376 (3)	0.4752 (17)	0.044 (7)*
H5	0.113 (4)	0.623 (2)	0.3968 (14)	0.023 (5)*
H7	0.002 (4)	0.857 (3)	0.2789 (15)	0.033 (6)*
H8	−0.208 (4)	1.077 (3)	0.3502 (14)	0.031 (6)*
H9	0.095 (4)	1.144 (3)	0.4345 (16)	0.039 (7)*
H10	0.495 (4)	0.967 (3)	0.4117 (15)	0.033 (6)*
H11	0.220 (7)	1.020 (5)	0.113 (3)	0.098 (13)*
H12	−0.091 (8)	1.212 (5)	0.154 (3)	0.104 (14)*
H13	0.035 (6)	1.375 (4)	0.244 (2)	0.081 (11)*
H14	0.478 (6)	1.267 (4)	0.254 (2)	0.072 (10)*
H15	0.595 (6)	1.038 (4)	0.176 (2)	0.067 (9)*
H17	0.766 (5)	0.776 (3)	0.1540 (18)	0.052 (8)*
H18	0.695 (6)	0.755 (4)	0.011 (2)	0.067 (9)*
H19	0.400 (5)	0.668 (3)	−0.030 (2)	0.061 (8)*
H20	0.168 (5)	0.591 (3)	0.0761 (19)	0.055 (8)*
H21	0.218 (5)	0.613 (3)	0.2145 (16)	0.039 (7)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Fe1	0.01851 (16)	0.01506 (16)	0.01460 (16)	−0.00259 (12)	0.00126 (12)	−0.00050 (12)
Fe2	0.02421 (14)	0.02186 (14)	0.02401 (15)	−0.00941 (10)	−0.00277 (10)	0.00620 (10)
P1	0.0170 (2)	0.0244 (2)	0.0203 (2)	−0.00623 (17)	0.00241 (16)	0.00201 (17)
C1	0.0195 (8)	0.0214 (8)	0.0148 (7)	−0.0042 (6)	0.0009 (6)	−0.0006 (6)
C2	0.0256 (9)	0.0219 (8)	0.0170 (8)	−0.0006 (7)	0.0021 (6)	−0.0037 (6)
C3	0.0406 (10)	0.0168 (8)	0.0198 (8)	−0.0057 (7)	0.0008 (7)	−0.0036 (7)
C4	0.0331 (10)	0.0272 (9)	0.0206 (9)	−0.0155 (8)	0.0021 (7)	−0.0032 (7)
C5	0.0200 (8)	0.0247 (9)	0.0180 (8)	−0.0067 (7)	−0.0008 (6)	−0.0001 (7)
C6	0.0217 (8)	0.0196 (8)	0.0193 (8)	−0.0086 (6)	0.0009 (6)	0.0037 (6)
C7	0.0198 (8)	0.0216 (8)	0.0279 (9)	−0.0091 (7)	0.0008 (7)	0.0006 (7)
C8	0.0252 (9)	0.0220 (9)	0.0327 (10)	−0.0058 (7)	0.0063 (7)	0.0006 (7)
C9	0.0390 (11)	0.0174 (8)	0.0260 (9)	−0.0081 (8)	0.0020 (8)	0.0002 (7)
C10	0.0306 (9)	0.0233 (9)	0.0220 (9)	−0.0132 (7)	−0.0034 (7)	0.0049 (7)
C11	0.108 (3)	0.0676 (19)	0.0234 (12)	−0.056 (2)	−0.0078 (14)	0.0117 (12)
C12	0.0470 (16)	0.108 (3)	0.070 (2)	−0.0461 (19)	−0.0363 (16)	0.071 (2)
C13	0.0535 (15)	0.0282 (12)	0.0658 (18)	0.0009 (11)	0.0197 (13)	0.0238 (12)
C14	0.0469 (13)	0.0370 (12)	0.0384 (12)	−0.0247 (10)	−0.0026 (10)	0.0136 (9)
C15	0.0476 (13)	0.0394 (12)	0.0373 (12)	−0.0126 (10)	0.0127 (10)	0.0117 (10)
C16	0.0285 (9)	0.0273 (9)	0.0196 (8)	−0.0034 (7)	0.0033 (7)	0.0008 (7)
C17	0.0423 (12)	0.0495 (13)	0.0285 (11)	−0.0136 (11)	0.0112 (9)	0.0006 (9)
C18	0.0616 (17)	0.0714 (19)	0.0244 (11)	−0.0150 (14)	0.0148 (11)	0.0022 (11)
C19	0.0667 (18)	0.0726 (19)	0.0197 (11)	−0.0037 (14)	−0.0004 (11)	−0.0081 (11)
C20	0.0539 (15)	0.0633 (17)	0.0326 (12)	−0.0153 (13)	−0.0045 (11)	−0.0149 (11)
C21	0.0379 (11)	0.0407 (12)	0.0255 (10)	−0.0122 (9)	0.0043 (8)	−0.0063 (8)

Geometric parameters (Å, °)

Fe1—C2	2.0451 (17)	C7—C8	1.423 (3)
Fe1—C4	2.0460 (19)	C7—H7	0.94 (2)
Fe1—C3	2.0487 (18)	C8—C9	1.418 (3)
Fe1—C5	2.0488 (17)	C8—H8	0.96 (2)
Fe1—C1	2.0513 (16)	C9—C10	1.422 (3)
Fe2—C11	2.028 (3)	C9—H9	0.93 (3)
Fe2—C12	2.032 (2)	C10—H10	0.91 (2)
Fe2—C10	2.0433 (18)	C11—C15	1.396 (4)
Fe2—C7	2.0435 (18)	C11—C12	1.418 (5)
Fe2—C15	2.044 (2)	C11—H11	0.95 (4)
Fe2—C8	2.0502 (19)	C12—C13	1.416 (5)
Fe2—C13	2.051 (2)	C12—H12	0.79 (4)
Fe2—C14	2.055 (2)	C13—C14	1.398 (4)
Fe2—C6	2.0556 (17)	C13—H13	0.94 (4)
Fe2—C9	2.058 (2)	C14—C15	1.389 (4)
P1—C6	1.8130 (18)	C14—H14	0.94 (3)
P1—C1	1.8210 (18)	C15—H15	1.01 (3)
P1—C16	1.8361 (19)	C16—C21	1.386 (3)

C1—C5	1.435 (2)	C16—C17	1.397 (3)
C1—C2	1.436 (2)	C17—C18	1.389 (3)
C2—C3	1.418 (3)	C17—H17	1.00 (3)
C2—H2	0.93 (2)	C18—C19	1.370 (4)
C3—C4	1.423 (3)	C18—H18	0.93 (3)
C3—H3	0.91 (2)	C19—C20	1.385 (4)
C4—C5	1.420 (3)	C19—H19	0.94 (3)
C4—H4	0.93 (3)	C20—C21	1.393 (3)
C5—H5	0.91 (2)	C20—H20	0.88 (3)
C6—C7	1.434 (2)	C21—H21	0.90 (3)
C6—C10	1.438 (3)		
$C2^{i}$—Fe1—C2	179.999 (1)	C2—C3—Fe1	69.60 (10)
$C2^{i}$—Fe1—C4	111.69 (8)	C4—C3—Fe1	69.56 (10)
C2—Fe1—C4	68.31 (8)	C2—C3—H3	127.2 (15)
C2—Fe1—$C4^{i}$	111.69 (8)	C4—C3—H3	124.8 (15)
C4—Fe1—$C4^{i}$	179.998 (1)	Fe1—C3—H3	124.4 (14)
$C2^{i}$—Fe1—C3	139.47 (8)	C5—C4—C3	108.31 (16)
C2—Fe1—C3	40.53 (8)	C5—C4—Fe1	69.82 (10)
C4—Fe1—C3	40.69 (8)	C3—C4—Fe1	69.76 (11)
$C4^{i}$—Fe1—C3	139.32 (8)	C5—C4—H4	125.1 (17)
C2—Fe1—$C3^{i}$	139.47 (8)	C3—C4—H4	126.4 (17)
C4—Fe1—$C3^{i}$	139.31 (8)	Fe1—C4—H4	122.7 (16)
C3—Fe1—$C3^{i}$	180.00 (8)	C4—C5—C1	108.37 (16)
$C2^{i}$—Fe1—C5	111.48 (7)	C4—C5—Fe1	69.60 (10)
C2—Fe1—C5	68.51 (7)	C1—C5—Fe1	69.61 (9)
C4—Fe1—C5	40.58 (7)	C4—C5—H5	123.9 (14)
$C4^{i}$—Fe1—C5	139.42 (7)	C1—C5—H5	127.7 (14)
C3—Fe1—C5	68.46 (7)	Fe1—C5—H5	126.8 (14)
$C3^{i}$—Fe1—C5	111.54 (7)	C7—C6—C10	106.61 (16)
C2—Fe1—$C5^{i}$	111.49 (7)	C7—C6—P1	130.23 (14)
C4—Fe1—$C5^{i}$	139.42 (7)	C10—C6—P1	123.00 (13)
C3—Fe1—$C5^{i}$	111.54 (7)	C7—C6—Fe2	69.07 (10)
C5—Fe1—$C5^{i}$	179.998 (2)	C10—C6—Fe2	69.00 (10)
C2—Fe1—$C1^{i}$	138.95 (7)	P1—C6—Fe2	130.26 (9)
C4—Fe1—$C1^{i}$	111.19 (7)	C8—C7—C6	108.44 (16)
C3—Fe1—$C1^{i}$	111.09 (7)	C8—C7—Fe2	69.91 (11)
C5—Fe1—$C1^{i}$	139.03 (7)	C6—C7—Fe2	69.98 (10)
$C2^{i}$—Fe1—C1	138.95 (7)	C8—C7—H7	124.7 (15)
C2—Fe1—C1	41.05 (7)	C6—C7—H7	126.8 (15)
C4—Fe1—C1	68.81 (7)	Fe2—C7—H7	126.3 (14)
$C4^{i}$—Fe1—C1	111.19 (7)	C9—C8—C7	108.42 (17)
C3—Fe1—C1	68.90 (7)	C9—C8—Fe2	70.10 (11)
$C3^{i}$—Fe1—C1	111.10 (7)	C7—C8—Fe2	69.40 (10)
C5—Fe1—C1	40.97 (7)	C9—C8—H8	126.3 (14)

$C5^{i}$—Fe1—C1	139.03 (7)	C7—C8—H8	125.3 (14)
$C1^{i}$—Fe1—C1	180.00 (10)	Fe2—C8—H8	125.9 (14)
C11—Fe2—C12	40.86 (16)	C8—C9—C10	107.81 (17)
C11—Fe2—C10	146.78 (13)	C8—C9—Fe2	69.52 (11)
C12—Fe2—C10	171.90 (15)	C10—C9—Fe2	69.16 (11)
C11—Fe2—C7	108.50 (10)	C8—C9—H9	125.1 (15)
C12—Fe2—C7	113.95 (11)	C10—C9—H9	127.1 (16)
C10—Fe2—C7	68.58 (7)	Fe2—C9—H9	126.5 (15)
C11—Fe2—C15	40.11 (12)	C9—C10—C6	108.72 (17)
C12—Fe2—C15	67.42 (12)	C9—C10—Fe2	70.26 (11)
C10—Fe2—C15	116.96 (9)	C6—C10—Fe2	69.93 (10)
C7—Fe2—C15	133.60 (9)	C9—C10—H10	126.9 (15)
C11—Fe2—C8	132.21 (11)	C6—C10—H10	124.3 (15)
C12—Fe2—C8	108.31 (11)	Fe2—C10—H10	124.2 (15)
C10—Fe2—C8	68.20 (8)	C15—C11—C12	107.0 (3)
C7—Fe2—C8	40.68 (7)	C15—C11—Fe2	70.54 (14)
C15—Fe2—C8	171.86 (10)	C12—C11—Fe2	69.72 (17)
C11—Fe2—C13	68.42 (14)	C15—C11—H11	128 (3)
C12—Fe2—C13	40.56 (15)	C12—C11—H11	125 (3)
C10—Fe2—C13	133.29 (12)	Fe2—C11—H11	122 (2)
C7—Fe2—C13	145.35 (10)	C13—C12—C11	108.1 (2)
C15—Fe2—C13	67.20 (10)	C13—C12—Fe2	70.44 (14)
C8—Fe2—C13	114.51 (9)	C11—C12—Fe2	69.42 (15)
C11—Fe2—C14	67.45 (11)	C13—C12—H12	133 (3)
C12—Fe2—C14	67.22 (11)	C11—C12—H12	119 (3)
C10—Fe2—C14	111.30 (9)	Fe2—C12—H12	123 (3)
C7—Fe2—C14	172.87 (9)	C14—C13—C12	107.1 (3)
C15—Fe2—C14	39.63 (10)	C14—C13—Fe2	70.26 (13)
C8—Fe2—C14	146.35 (10)	C12—C13—Fe2	69.01 (15)
C13—Fe2—C14	39.80 (10)	C14—C13—H13	126 (2)
C11—Fe2—C6	114.47 (11)	C12—C13—H13	127 (2)
C12—Fe2—C6	145.58 (14)	Fe2—C13—H13	127 (2)
C10—Fe2—C6	41.07 (7)	C15—C14—C13	108.8 (2)
C7—Fe2—C6	40.94 (7)	C15—C14—Fe2	69.74 (13)
C15—Fe2—C6	110.53 (9)	C13—C14—Fe2	69.94 (13)
C8—Fe2—C6	68.73 (7)	C15—C14—H14	124 (2)
C13—Fe2—C6	173.03 (11)	C13—C14—H14	127 (2)
C14—Fe2—C6	134.39 (8)	Fe2—C14—H14	130 (2)
C11—Fe2—C9	171.42 (13)	C14—C15—C11	109.0 (3)
C12—Fe2—C9	132.17 (14)	C14—C15—Fe2	70.64 (13)
C10—Fe2—C9	40.59 (8)	C11—C15—Fe2	69.35 (15)
C7—Fe2—C9	68.37 (8)	C14—C15—H15	130.1 (19)
C15—Fe2—C9	147.56 (10)	C11—C15—H15	120.8 (19)
C8—Fe2—C9	40.37 (8)	Fe2—C15—H15	122.5 (18)
C13—Fe2—C9	109.29 (10)	C21—C16—C17	118.7 (2)
C14—Fe2—C9	116.51 (9)	C21—C16—P1	124.65 (15)
C6—Fe2—C9	68.82 (7)	C17—C16—P1	116.63 (17)
C6—P1—C1	100.46 (8)	C18—C17—C16	120.4 (2)

C6—P1—C16	103.22 (8)	C18—C17—H17	119.5 (16)
C1—P1—C16	100.66 (9)	C16—C17—H17	119.9 (16)
C5—C1—C2	106.76 (15)	C19—C18—C17	120.2 (2)
C5—C1—P1	130.84 (13)	C19—C18—H18	120 (2)
C2—C1—P1	122.37 (13)	C17—C18—H18	119 (2)
C5—C1—Fe1	69.42 (9)	C18—C19—C20	120.3 (2)
C2—C1—Fe1	69.24 (10)	C18—C19—H19	117.6 (19)
P1—C1—Fe1	124.65 (9)	C20—C19—H19	122.0 (19)
C3—C2—C1	108.69 (16)	C19—C20—C21	119.7 (3)
C3—C2—Fe1	69.87 (10)	C19—C20—H20	121 (2)
C1—C2—Fe1	69.71 (10)	C21—C20—H20	120 (2)
C3—C2—H2	129.3 (14)	C16—C21—C20	120.6 (2)
C1—C2—H2	121.9 (14)	C16—C21—H21	121.5 (16)
Fe1—C2—H2	123.4 (14)	C20—C21—H21	117.8 (16)
C2—C3—C4	107.86 (16)		
C6—P1—C1—C5	17.63 (18)	C6—C7—C8—Fe2	59.60 (12)
C16—P1—C1—C5	−88.12 (17)	C11—Fe2—C8—C9	−173.79 (16)
C6—P1—C1—C2	−160.12 (15)	C12—Fe2—C8—C9	−134.62 (18)
C16—P1—C1—C2	94.14 (15)	C10—Fe2—C8—C9	37.60 (11)
C6—P1—C1—Fe1	−74.25 (11)	C7—Fe2—C8—C9	119.67 (16)
C16—P1—C1—Fe1	−179.99 (10)	C13—Fe2—C8—C9	−91.39 (15)
C2^{i}—Fe1—C1—C5	−61.85 (15)	C14—Fe2—C8—C9	−58.43 (19)
C2—Fe1—C1—C5	118.15 (15)	C6—Fe2—C8—C9	81.91 (12)
C4—Fe1—C1—C5	37.26 (11)	C11—Fe2—C8—C7	66.55 (19)
C4^{i}—Fe1—C1—C5	−142.74 (11)	C12—Fe2—C8—C7	105.71 (18)
C3—Fe1—C1—C5	81.03 (11)	C10—Fe2—C8—C7	−82.07 (12)
C3^{i}—Fe1—C1—C5	−98.97 (11)	C13—Fe2—C8—C7	148.94 (15)
C5^{i}—Fe1—C1—C5	180.001 (1)	C14—Fe2—C8—C7	−178.10 (14)
C2^{i}—Fe1—C1—C2	180.0	C6—Fe2—C8—C7	−37.76 (11)
C4—Fe1—C1—C2	−80.89 (12)	C9—Fe2—C8—C7	−119.67 (16)
C4^{i}—Fe1—C1—C2	99.11 (12)	C7—C8—C9—C10	0.3 (2)
C3—Fe1—C1—C2	−37.12 (11)	Fe2—C8—C9—C10	−58.74 (13)
C3^{i}—Fe1—C1—C2	142.88 (11)	C7—C8—C9—Fe2	59.01 (13)
C5—Fe1—C1—C2	−118.15 (15)	C12—Fe2—C9—C8	65.74 (18)
C5^{i}—Fe1—C1—C2	61.85 (15)	C10—Fe2—C9—C8	−119.45 (16)
C2^{i}—Fe1—C1—P1	64.28 (16)	C7—Fe2—C9—C8	−37.54 (11)
C2—Fe1—C1—P1	−115.72 (16)	C15—Fe2—C9—C8	−176.92 (15)
C4—Fe1—C1—P1	163.39 (13)	C13—Fe2—C9—C8	105.48 (14)
C4^{i}—Fe1—C1—P1	−16.61 (13)	C14—Fe2—C9—C8	148.16 (12)
C3—Fe1—C1—P1	−152.84 (13)	C6—Fe2—C9—C8	−81.67 (11)
C3^{i}—Fe1—C1—P1	27.16 (13)	C12—Fe2—C9—C10	−174.81 (16)
C5—Fe1—C1—P1	126.13 (16)	C7—Fe2—C9—C10	81.90 (11)
C5^{i}—Fe1—C1—P1	−53.87 (16)	C15—Fe2—C9—C10	−57.5 (2)
C5—C1—C2—C3	−0.4 (2)	C8—Fe2—C9—C10	119.45 (16)
P1—C1—C2—C3	177.83 (13)	C13—Fe2—C9—C10	−135.08 (13)
Fe1—C1—C2—C3	59.17 (13)	C14—Fe2—C9—C10	−92.39 (13)

C5—C1—C2—Fe1	−59.56 (12)	C6—Fe2—C9—C10	37.78 (11)
P1—C1—C2—Fe1	118.66 (13)	C8—C9—C10—C6	−0.6 (2)
C4—Fe1—C2—C3	−37.76 (11)	Fe2—C9—C10—C6	−59.56 (12)
C4^{i}—Fe1—C2—C3	142.24 (11)	C8—C9—C10—Fe2	58.96 (13)
C3^{i}—Fe1—C2—C3	180.0	C7—C6—C10—C9	0.7 (2)
C5—Fe1—C2—C3	−81.56 (12)	P1—C6—C10—C9	−175.06 (13)
C5^{i}—Fe1—C2—C3	98.44 (12)	Fe2—C6—C10—C9	59.77 (13)
C1^{i}—Fe1—C2—C3	60.03 (16)	C7—C6—C10—Fe2	−59.09 (12)
C1—Fe1—C2—C3	−119.97 (16)	P1—C6—C10—Fe2	125.17 (13)
C4—Fe1—C2—C1	82.20 (11)	C11—Fe2—C10—C9	−172.65 (18)
C4^{i}—Fe1—C2—C1	−97.80 (11)	C7—Fe2—C10—C9	−81.32 (12)
C3—Fe1—C2—C1	119.97 (16)	C15—Fe2—C10—C9	149.51 (13)
C3^{i}—Fe1—C2—C1	−60.03 (16)	C8—Fe2—C10—C9	−37.41 (11)
C5—Fe1—C2—C1	38.41 (10)	C13—Fe2—C10—C9	66.30 (16)
C5^{i}—Fe1—C2—C1	−141.59 (10)	C14—Fe2—C10—C9	106.33 (13)
C1^{i}—Fe1—C2—C1	179.999 (2)	C6—Fe2—C10—C9	−119.61 (15)
C1—C2—C3—C4	0.2 (2)	C11—Fe2—C10—C6	−53.0 (2)
Fe1—C2—C3—C4	59.24 (13)	C7—Fe2—C10—C6	38.29 (10)
C1—C2—C3—Fe1	−59.07 (12)	C15—Fe2—C10—C6	−90.88 (13)
C2^{i}—Fe1—C3—C2	179.999 (1)	C8—Fe2—C10—C6	82.20 (11)
C4—Fe1—C3—C2	119.20 (15)	C13—Fe2—C10—C6	−174.09 (13)
C4^{i}—Fe1—C3—C2	−60.80 (15)	C14—Fe2—C10—C6	−134.06 (12)
C5—Fe1—C3—C2	81.70 (11)	C9—Fe2—C10—C6	119.61 (15)
C5^{i}—Fe1—C3—C2	−98.30 (11)	C12—Fe2—C11—C15	117.5 (3)
C1^{i}—Fe1—C3—C2	−142.42 (10)	C10—Fe2—C11—C15	−58.1 (3)
C1—Fe1—C3—C2	37.58 (10)	C7—Fe2—C11—C15	−137.02 (16)
C2^{i}—Fe1—C3—C4	60.80 (15)	C8—Fe2—C11—C15	−176.11 (13)
C2—Fe1—C3—C4	−119.20 (15)	C13—Fe2—C11—C15	79.80 (18)
C4^{i}—Fe1—C3—C4	180.0	C14—Fe2—C11—C15	36.72 (16)
C5—Fe1—C3—C4	−37.50 (11)	C6—Fe2—C11—C15	−93.31 (17)
C5^{i}—Fe1—C3—C4	142.50 (11)	C10—Fe2—C11—C12	−175.56 (17)
C1^{i}—Fe1—C3—C4	98.37 (11)	C7—Fe2—C11—C12	105.51 (18)
C1—Fe1—C3—C4	−81.63 (11)	C15—Fe2—C11—C12	−117.5 (2)
C2—C3—C4—C5	0.1 (2)	C8—Fe2—C11—C12	66.4 (2)
Fe1—C3—C4—C5	59.38 (13)	C13—Fe2—C11—C12	−37.67 (17)
C2—C3—C4—Fe1	−59.27 (13)	C14—Fe2—C11—C12	−80.75 (19)
C2^{i}—Fe1—C4—C5	98.14 (11)	C6—Fe2—C11—C12	149.22 (17)
C2—Fe1—C4—C5	−81.86 (11)	C15—C11—C12—C13	−1.0 (3)
C3—Fe1—C4—C5	−119.49 (15)	Fe2—C11—C12—C13	60.05 (18)
C3^{i}—Fe1—C4—C5	60.51 (15)	C15—C11—C12—Fe2	−61.02 (16)
C5^{i}—Fe1—C4—C5	180.0	C11—Fe2—C12—C13	−119.1 (2)
C1^{i}—Fe1—C4—C5	142.39 (10)	C7—Fe2—C12—C13	149.94 (15)
C1—Fe1—C4—C5	−37.61 (10)	C15—Fe2—C12—C13	−80.83 (17)
C2^{i}—Fe1—C4—C3	−142.37 (11)	C8—Fe2—C12—C13	106.57 (17)
C2—Fe1—C4—C3	37.63 (11)	C14—Fe2—C12—C13	−37.72 (16)

$C3^{i}$—Fe1—C4—C3	180.000 (1)	C6—Fe2—C12—C13	−174.55 (14)
C5—Fe1—C4—C3	119.49 (15)	C9—Fe2—C12—C13	68.1 (2)
$C5^{i}$—Fe1—C4—C3	−60.51 (15)	C7—Fe2—C12—C11	−90.99 (17)
$C1^{i}$—Fe1—C4—C3	−98.12 (11)	C15—Fe2—C12—C11	38.24 (17)
C1—Fe1—C4—C3	81.89 (11)	C8—Fe2—C12—C11	−134.35 (16)
C3—C4—C5—C1	−0.4 (2)	C13—Fe2—C12—C11	119.1 (2)
Fe1—C4—C5—C1	58.99 (12)	C14—Fe2—C12—C11	81.35 (18)
C3—C4—C5—Fe1	−59.34 (13)	C6—Fe2—C12—C11	−55.5 (2)
C2—C1—C5—C4	0.5 (2)	C9—Fe2—C12—C11	−172.82 (15)
P1—C1—C5—C4	−177.56 (14)	C11—C12—C13—C14	0.8 (3)
Fe1—C1—C5—C4	−58.99 (12)	Fe2—C12—C13—C14	60.23 (17)
C2—C1—C5—Fe1	59.44 (12)	C11—C12—C13—Fe2	−59.41 (17)
P1—C1—C5—Fe1	−118.57 (15)	C11—Fe2—C13—C14	−80.24 (19)
$C2^{i}$—Fe1—C5—C4	−98.69 (12)	C12—Fe2—C13—C14	−118.2 (3)
C2—Fe1—C5—C4	81.31 (12)	C10—Fe2—C13—C14	69.4 (2)
$C4^{i}$—Fe1—C5—C4	180.0	C7—Fe2—C13—C14	−171.83 (15)
C3—Fe1—C5—C4	37.59 (11)	C15—Fe2—C13—C14	−36.79 (16)
$C3^{i}$—Fe1—C5—C4	−142.41 (11)	C8—Fe2—C13—C14	151.90 (15)
$C1^{i}$—Fe1—C5—C4	−60.20 (15)	C9—Fe2—C13—C14	108.58 (17)
C1—Fe1—C5—C4	119.80 (15)	C11—Fe2—C13—C12	37.9 (2)
$C2^{i}$—Fe1—C5—C1	141.51 (10)	C10—Fe2—C13—C12	−172.36 (18)
C2—Fe1—C5—C1	−38.49 (10)	C7—Fe2—C13—C12	−53.6 (3)
C4—Fe1—C5—C1	−119.80 (15)	C15—Fe2—C13—C12	81.4 (2)
$C4^{i}$—Fe1—C5—C1	60.20 (15)	C8—Fe2—C13—C12	−89.9 (2)
C3—Fe1—C5—C1	−82.21 (11)	C14—Fe2—C13—C12	118.2 (3)
$C3^{i}$—Fe1—C5—C1	97.79 (11)	C9—Fe2—C13—C12	−133.23 (19)
$C1^{i}$—Fe1—C5—C1	180.0	C12—C13—C14—C15	−0.3 (3)
C1—P1—C6—C7	−63.73 (17)	Fe2—C13—C14—C15	59.08 (16)
C16—P1—C6—C7	39.95 (18)	C12—C13—C14—Fe2	−59.43 (16)
C1—P1—C6—C10	110.91 (15)	C11—Fe2—C14—C15	−37.15 (18)
C16—P1—C6—C10	−145.40 (15)	C12—Fe2—C14—C15	−81.6 (2)
C1—P1—C6—Fe2	−159.66 (12)	C10—Fe2—C14—C15	106.98 (15)
C16—P1—C6—Fe2	−55.97 (14)	C8—Fe2—C14—C15	−170.70 (15)
C11—Fe2—C6—C7	−90.42 (15)	C13—Fe2—C14—C15	−120.0 (2)
C12—Fe2—C6—C7	−54.1 (2)	C6—Fe2—C14—C15	65.63 (19)
C10—Fe2—C6—C7	118.33 (15)	C9—Fe2—C14—C15	151.22 (14)
C15—Fe2—C6—C7	−133.79 (13)	C11—Fe2—C14—C13	82.9 (2)
C8—Fe2—C6—C7	37.53 (11)	C12—Fe2—C14—C13	38.4 (2)
C14—Fe2—C6—C7	−172.13 (13)	C10—Fe2—C14—C13	−132.98 (18)
C9—Fe2—C6—C7	80.98 (12)	C15—Fe2—C14—C13	120.0 (2)
C11—Fe2—C6—C10	151.25 (14)	C8—Fe2—C14—C13	−50.7 (3)
C12—Fe2—C6—C10	−172.4 (2)	C6—Fe2—C14—C13	−174.33 (17)
C7—Fe2—C6—C10	−118.33 (15)	C9—Fe2—C14—C13	−88.74 (19)
C15—Fe2—C6—C10	107.88 (13)	C13—C14—C15—C11	−0.3 (3)
C8—Fe2—C6—C10	−80.80 (12)	Fe2—C14—C15—C11	58.94 (16)
C14—Fe2—C6—C10	69.54 (16)	C13—C14—C15—Fe2	−59.21 (16)
C9—Fe2—C6—C10	−37.34 (11)	C12—C11—C15—C14	0.8 (3)

C11—Fe2—C6—P1	35.19 (17)	Fe2—C11—C15—C14	−59.73 (16)
C12—Fe2—C6—P1	71.5 (2)	C12—C11—C15—Fe2	60.49 (17)
C10—Fe2—C6—P1	−116.07 (17)	C11—Fe2—C15—C14	120.0 (2)
C7—Fe2—C6—P1	125.61 (18)	C12—Fe2—C15—C14	81.1 (2)
C15—Fe2—C6—P1	−8.18 (16)	C10—Fe2—C15—C14	−91.42 (15)
C8—Fe2—C6—P1	163.13 (15)	C7—Fe2—C15—C14	−176.75 (12)
C14—Fe2—C6—P1	−46.52 (19)	C13—Fe2—C15—C14	36.94 (16)
C9—Fe2—C6—P1	−153.41 (15)	C6—Fe2—C15—C14	−135.96 (14)
C10—C6—C7—C8	−0.5 (2)	C9—Fe2—C15—C14	−53.4 (2)
P1—C6—C7—C8	174.81 (14)	C12—Fe2—C15—C11	−39.0 (2)
Fe2—C6—C7—C8	−59.55 (13)	C10—Fe2—C15—C11	148.55 (18)
C10—C6—C7—Fe2	59.04 (12)	C7—Fe2—C15—C11	63.2 (2)
P1—C6—C7—Fe2	−125.64 (15)	C13—Fe2—C15—C11	−83.1 (2)
C11—Fe2—C7—C8	−134.23 (16)	C14—Fe2—C15—C11	−120.0 (2)
C12—Fe2—C7—C8	−90.62 (19)	C6—Fe2—C15—C11	104.01 (19)
C10—Fe2—C7—C8	81.05 (12)	C9—Fe2—C15—C11	−173.47 (19)
C15—Fe2—C7—C8	−171.57 (13)	C6—P1—C16—C21	−72.89 (19)
C13—Fe2—C7—C8	−55.7 (2)	C1—P1—C16—C21	30.64 (19)
C6—Fe2—C7—C8	119.45 (16)	C6—P1—C16—C17	108.67 (17)
C9—Fe2—C7—C8	37.27 (11)	C1—P1—C16—C17	−147.80 (17)
C11—Fe2—C7—C6	106.32 (16)	C21—C16—C17—C18	2.0 (3)
C12—Fe2—C7—C6	149.93 (17)	P1—C16—C17—C18	−179.5 (2)
C10—Fe2—C7—C6	−38.41 (11)	C16—C17—C18—C19	−1.3 (4)
C15—Fe2—C7—C6	68.98 (16)	C17—C18—C19—C20	−0.1 (5)
C8—Fe2—C7—C6	−119.45 (16)	C18—C19—C20—C21	0.8 (4)
C13—Fe2—C7—C6	−175.11 (18)	C17—C16—C21—C20	−1.2 (3)
C9—Fe2—C7—C6	−82.18 (12)	P1—C16—C21—C20	−179.61 (19)
C6—C7—C8—C9	0.1 (2)	C19—C20—C21—C16	−0.2 (4)
Fe2—C7—C8—C9	−59.45 (13)		

Symmetry codes: (i) −*x*+1, −*y*+1, −*z*+1.

Fig. 1

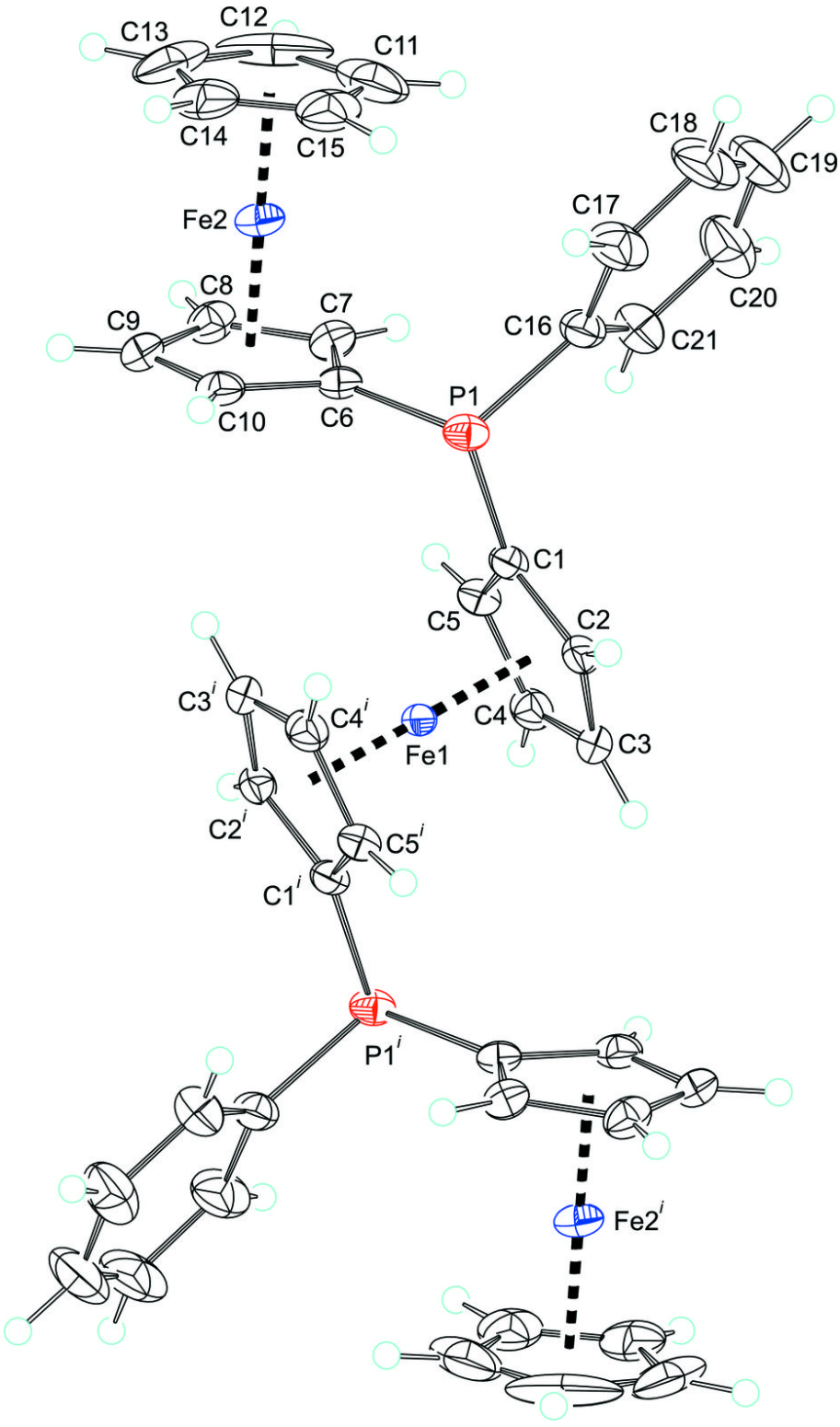

C13
C12
C11
C14
C15
Fe2
C8
C7
C9
C10
C6
P1
C16
C17
C18
C19
C20
C21
C1
C5
C2
C3^{i}
C4^{i}
Fe1
C4
C3
C2^{i}
C5^{i}
C1^{i}
P1^{i}
Fe2^{i}

Fig. 2