

Acta Crystallographica Section E

## Structure Reports

Online

ISSN 1600-5368

**(*R,S<sub>p</sub>*)-1-Diphenylphosphino-2-(1-ethoxyethyl)ferrocene**

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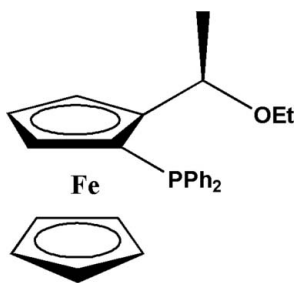
Received 12 July 2008; accepted 15 October 2008

Key indicators: single-crystal X-ray study;  $T = 113$  K; mean  $\sigma(\text{C}-\text{C}) = 0.003$  Å;  
 $R$  factor = 0.026;  $wR$  factor = 0.062; data-to-parameter ratio = 15.0.

In the crystal structure of the title compound,  $[\text{Fe}(\text{C}_5\text{H}_5)(\text{C}_{21}\text{H}_{22}\text{OP})]$ , the cyclopentadienyl (Cp) rings are almost parallel and are essentially eclipsed. The absolute configuration was determined as *S* for the planar and *R* for the central chirality.

## Related literature

For background to ferrocene derivatives applied as catalysts, see: Blaser & Schmidt (2004); Gomez Arrayas *et al.* (2006); Hayashi *et al.* (1988); Ohmura *et al.* (1995); Ojima (2000). For the structures of closely related compounds, see: Jin *et al.* (2004); Cheelama & Knochel (2007); Podlaha *et al.* (1996).



## Experimental

## Crystal data

 $[\text{Fe}(\text{C}_5\text{H}_5)(\text{C}_{21}\text{H}_{22}\text{OP})]$  $M_r = 442.30$ 

Orthorhombic,  $P2_12_12_1$   
 $a = 11.003$  (2) Å  
 $b = 12.191$  (2) Å  
 $c = 16.599$  (3) Å  
 $V = 2226.6$  (8) Å<sup>3</sup>

$Z = 4$   
Mo  $K\alpha$  radiation  
 $\mu = 0.76$  mm<sup>-1</sup>  
 $T = 113$  (2) K  
 $0.12 \times 0.10 \times 0.08$  mm

## Data collection

Rigaku Saturn diffractometer  
Absorption correction: multi-scan  
(*CrystalClear*; Rigaku, 2001)  
 $T_{\text{min}} = 0.914$ ,  $T_{\text{max}} = 0.942$

22928 measured reflections  
3929 independent reflections  
3847 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.050$

## Refinement

$R[F^2 > 2\sigma(F^2)] = 0.026$   
 $wR(F^2) = 0.062$   
 $S = 1.05$   
3929 reflections  
262 parameters  
H-atom parameters constrained

$\Delta\rho_{\text{max}} = 0.18$  e Å<sup>-3</sup>  
 $\Delta\rho_{\text{min}} = -0.28$  e Å<sup>-3</sup>  
Absolute structure: Flack (1983),  
with 1688 Friedel pairs  
Flack parameter: 0.019 (12)

Data collection: *CrystalClear* (Rigaku, 2001); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

The authors thank the Natural Science Foundation of China (grant No. 20572009) and the Basic Research Fund of Beijing Institute of Technology (grant No. 000Y05 for financial support of this work.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: NC2112).

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**supplementary materials**

*Acta Cryst.* (2008). E64, m1448 [ doi:10.1107/S1600536808033618 ]

## **(*R,S*<sub>p</sub>)-1-Diphenylphosphino-2-(1-ethoxyethyl)ferrocene**

**H. Yuan and Z.-M. Zhou**

### **Comment**

Asymmetric metal catalysis is one of the most active areas in modern organic chemistry, and considerable efforts have been made to the development of novel ligands for catalytic asymmetric transformations (Ojima, 2000). In this context, ferrocene-based ligands incorporating both chirality are very important (Gomez Arrayas *et al.*, 2006) and some of them have already been applied in industrial processes because of their stability, low price and unique structure (Blaser & Schmidt, 2004).

(*S,R*<sub>p</sub>)-1-(diphenylphosphino)-2-(1-ethoxyethyl)-ferrocene, the enantiomorph of title compound, has been used to synthesize 1,1'-binaphthyls *via* asymmetric Ni-catalysed Grignard cross-coupling with up to 68% ee, and the (*S,R*<sub>p</sub>)-1-(diphenylphosphino)-2-(1-methoxyethyl)-ferrocene provided axially chiral binaphthalenes in enantioselectivities up to 95% ee (Hayashi *et al.*, 1988). In addition, the (*R,S*<sub>p</sub>)-1-(diphenylphosphino)-2-(1-ethoxyethyl)-ferrocene was also used in asymmetric hydrosilylation (Ohmura *et al.*, 1995).

The Fe—C bond distances within the ferrocene group are in the range of 2.038 (2)–2.050 (2) Å for the unsubstituted cyclopentadienyl (Cp) ring [C1–C5] and 2.025 (2)–2.046 (2) Å for the substituted Cp ring [C6–C10]. The Cp rings are almost parallel, the dihedral angle between the Cp ring planes is 1.80 (10)°. The Cp rings are essentially eclipsed and the Fe–centroid distances are 1.654 (9) (Cg1) and 1.639 (9) Å (Cg2) with Cg1 and Cg2 are the centroids of the [C1–C5] and [C6–C10] rings. The [Cg1—Fe1—Cg2] angle is 178.60 (18)°. The C11 atom is almost in the plane of their carrier Cp ring, while the P1 atom is tilted slightly out of the plane by 0.102 (10) Å.

The two phenyl rings are oriented almost perpendicular, with a dihedral angle of 90.90 (10)°. The O1—C11 and C10—C11 bonds lengths are in agreement with those in the related complex 1-(1-Ferrocenyl-1-methoxy-3-phenyl-2-propyl)-1*H*-1,2,4-triazole (Jin *et al.*, 2004) and the geometric parameters of the PPh<sub>2</sub> group are in agreement with those in the similar structure 1-carboxy-1'-(diphenylphosphino)-ferrocene (Podlaha *et al.*, 1996).

The title compound has both central chirality and planar chirality with the configuration of C11 atom being *R*, and the configuration of planar chirality being *S*.

### **Experimental**

The title compound was prepared from (*R,S*<sub>p</sub>)-1-[1-(acetyloxy)ethyl]-2-(diphenylphosphino)-ferrocene according to literature procedures (Hayashi *et al.*, 1988). Single crystals of the title compound suitable for X-ray diffraction analysis were obtained by slow evaporation of a hexane solution.

## Refinement

All H atoms were positioned with idealized geometry with C—H = 0.93 (aromatic), 0.96 (methyl), 0.97 (methylene) or 0.98 Å (cyclopentadienyl and Cp—CH) and were refined isotropic with  $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$  or  $1.5U_{\text{eq}}(\text{methyl C})$  using a riding model. The absolute structure was determined on the basis of 1688 Friedel pairs.

## Figures

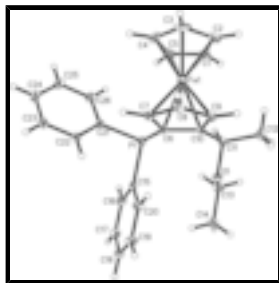


Fig. 1. A molecular view of the title compound, showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level.

## (*R,S,P*)-1-Diphenylphosphino-2-(1-ethoxyethyl)ferrocene

### Crystal data

[Fe(C<sub>5</sub>H<sub>5</sub>)(C<sub>21</sub>H<sub>22</sub>OP)]

$M_r = 442.30$

Orthorhombic,  $P2_12_12_1$

Hall symbol: P 2ac 2ab

$a = 11.003$  (2) Å

$b = 12.191$  (2) Å

$c = 16.599$  (3) Å

$V = 2226.6$  (8) Å<sup>3</sup>

$Z = 4$

$F_{000} = 928$

$D_x = 1.319$  Mg m<sup>-3</sup>

Mo  $K\alpha$  radiation

$\lambda = 0.71073$  Å

Cell parameters from 7176 reflections

$\theta = 2.2$ – $27.9^\circ$

$\mu = 0.76$  mm<sup>-1</sup>

$T = 113$  (2) K

Block, red

$0.12 \times 0.10 \times 0.08$  mm

### Data collection

Rigaku Saturn  
diffractometer

Radiation source: rotating anode

Monochromator: confocal

$T = 113$  (2) K

$\omega$  scans

Absorption correction: multi-scan  
(CrystalClear; Rigaku, 2001)

$T_{\text{min}} = 0.914$ ,  $T_{\text{max}} = 0.942$

22928 measured reflections

3929 independent reflections

3847 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.050$

$\theta_{\text{max}} = 25.0^\circ$

$\theta_{\text{min}} = 2.1^\circ$

$h = -13 \rightarrow 12$

$k = -14 \rightarrow 14$

$l = -19 \rightarrow 19$

Refinement

Refinement on $F^2$	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.026$	$w = 1/[\sigma^2(F_o^2) + (0.0301P)^2 + 0.2869P]$
$wR(F^2) = 0.062$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.05$	$(\Delta/\sigma)_{\max} = 0.002$
3929 reflections	$\Delta\rho_{\max} = 0.18 \text{ e } \text{\AA}^{-3}$
262 parameters	$\Delta\rho_{\min} = -0.28 \text{ e } \text{\AA}^{-3}$
Primary atom site location: structure-invariant direct methods	Extinction correction: none
Secondary atom site location: difference Fourier map	Absolute structure: Flack (1983), with 1688 Friedel pairs
	Flack parameter: 0.019 (12)

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 ( $\text{\AA}^2$ )

	$x$	$y$	$z$	$U_{\text{iso}}^*/U_{\text{eq}}$
Fe1	-0.03864 (3)	0.77799 (2)	0.905322 (18)	0.02169 (9)
P1	0.07707 (4)	0.98992 (4)	1.01327 (3)	0.01930 (12)
O1	0.18641 (12)	1.04499 (11)	0.82901 (9)	0.0230 (3)
C1	0.09790 (19)	0.66545 (15)	0.91286 (14)	0.0272 (5)
H1A	0.1815	0.6758	0.8941	0.033*
C2	0.0010 (2)	0.62243 (18)	0.86702 (16)	0.0358 (6)
H2A	0.0055	0.5980	0.8108	0.043*
C3	-0.1039 (2)	0.62123 (19)	0.9163 (2)	0.0513 (8)
H3A	-0.1850	0.5958	0.9004	0.062*
C4	-0.0708 (3)	0.6634 (2)	0.99241 (18)	0.0517 (8)
H4A	-0.1254	0.6724	1.0386	0.062*
C5	0.0538 (2)	0.69041 (18)	0.99089 (14)	0.0373 (6)
H5A	0.1011	0.7209	1.0356	0.045*
C6	-0.01527 (17)	0.94062 (15)	0.93016 (11)	0.0178 (4)
C7	-0.14254 (18)	0.91375 (16)	0.92785 (12)	0.0210 (4)
H7A	-0.2004	0.9224	0.9723	0.025*
C8	-0.16938 (19)	0.87040 (17)	0.84996 (13)	0.0260 (5)

## supplementary materials

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H8A	-0.2489	0.8442	0.8315	0.031*
C9	-0.06047 (19)	0.87097 (17)	0.80413 (12)	0.0230 (4)
H9A	-0.0519	0.8449	0.7486	0.028*
C10	0.03465 (19)	0.91267 (15)	0.85299 (11)	0.0198 (4)
C11	0.16655 (19)	0.92877 (16)	0.83152 (12)	0.0204 (4)
H11A	0.2167	0.8975	0.8746	0.024*
C12	0.2039 (2)	0.87699 (19)	0.75210 (13)	0.0306 (5)
H12A	0.2887	0.8903	0.7428	0.046*
H12B	0.1572	0.9086	0.7091	0.046*
H12C	0.1894	0.7994	0.7542	0.046*
C13	0.3051 (2)	1.07659 (18)	0.85426 (15)	0.0291 (5)
H13A	0.3658	1.0419	0.8204	0.035*
H13B	0.3188	1.0538	0.9095	0.035*
C14	0.3145 (2)	1.19937 (17)	0.84759 (15)	0.0334 (5)
H14A	0.3939	1.2225	0.8645	0.050*
H14B	0.2541	1.2329	0.8813	0.050*
H14C	0.3014	1.2210	0.7927	0.050*
C15	0.06768 (17)	1.14024 (16)	1.00256 (11)	0.0189 (4)
C16	0.1436 (2)	1.20172 (18)	1.05214 (12)	0.0253 (5)
H16A	0.1945	1.1661	1.0885	0.030*
C17	0.1443 (2)	1.31563 (18)	1.04809 (13)	0.0308 (5)
H17A	0.1950	1.3558	1.0819	0.037*
C18	0.0702 (2)	1.36905 (18)	0.99405 (13)	0.0300 (5)
H18A	0.0702	1.4452	0.9913	0.036*
C19	-0.0040 (2)	1.30890 (18)	0.94408 (14)	0.0305 (5)
H19A	-0.0540	1.3448	0.9074	0.037*
C20	-0.00485 (19)	1.19497 (17)	0.94788 (13)	0.0263 (5)
H20A	-0.0547	1.1553	0.9133	0.032*
C21	-0.02654 (17)	0.96925 (15)	1.09878 (11)	0.0203 (4)
C22	-0.13214 (18)	1.03066 (16)	1.10886 (12)	0.0231 (5)
H22A	-0.1524	1.0840	1.0711	0.028*
C23	-0.2073 (2)	1.01308 (18)	1.17447 (13)	0.0277 (5)
H23A	-0.2772	1.0551	1.1809	0.033*
C24	-0.1788 (2)	0.93330 (19)	1.23050 (13)	0.0303 (5)
H24A	-0.2297	0.9215	1.2744	0.036*
C25	-0.0752 (2)	0.8714 (2)	1.22134 (13)	0.0319 (5)
H25A	-0.0562	0.8173	1.2588	0.038*
C26	0.0012 (2)	0.88959 (18)	1.15610 (12)	0.0281 (5)
H26A	0.0716	0.8481	1.1506	0.034*

### Atomic displacement parameters ( $\text{\AA}^2$ )

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
Fe1	0.02261 (15)	0.01571 (14)	0.02675 (16)	-0.00025 (12)	0.00374 (13)	0.00058 (13)
P1	0.0201 (3)	0.0192 (3)	0.0186 (3)	0.0020 (2)	-0.0007 (2)	0.0016 (2)
O1	0.0219 (7)	0.0174 (7)	0.0297 (8)	-0.0010 (6)	0.0016 (6)	0.0015 (6)
C1	0.0283 (11)	0.0154 (10)	0.0378 (12)	0.0046 (9)	0.0035 (11)	0.0019 (10)
C2	0.0387 (14)	0.0162 (11)	0.0525 (15)	0.0022 (10)	0.0012 (12)	-0.0069 (10)

C3	0.0332 (14)	0.0176 (12)	0.103 (3)	-0.0032 (10)	0.0143 (17)	0.0083 (16)
C4	0.066 (2)	0.0258 (13)	0.0637 (19)	0.0156 (12)	0.0410 (16)	0.0200 (13)
C5	0.0570 (16)	0.0207 (11)	0.0343 (13)	0.0130 (11)	0.0039 (13)	0.0061 (10)
C6	0.0184 (10)	0.0137 (9)	0.0212 (10)	0.0026 (8)	0.0010 (8)	0.0027 (8)
C7	0.0203 (10)	0.0186 (10)	0.0240 (11)	0.0018 (8)	0.0014 (9)	0.0005 (8)
C8	0.0229 (11)	0.0258 (11)	0.0292 (12)	-0.0001 (10)	-0.0050 (10)	0.0000 (10)
C9	0.0275 (11)	0.0218 (10)	0.0196 (10)	0.0000 (9)	-0.0023 (9)	-0.0007 (8)
C10	0.0247 (11)	0.0148 (10)	0.0198 (10)	0.0011 (9)	0.0010 (9)	0.0004 (8)
C11	0.0243 (11)	0.0159 (10)	0.0209 (10)	0.0003 (9)	-0.0016 (9)	0.0005 (8)
C12	0.0309 (12)	0.0322 (12)	0.0287 (12)	0.0012 (10)	0.0077 (10)	-0.0052 (10)
C13	0.0230 (11)	0.0258 (12)	0.0386 (13)	-0.0053 (9)	0.0007 (11)	0.0006 (10)
C14	0.0289 (12)	0.0259 (12)	0.0454 (14)	-0.0053 (10)	0.0065 (11)	-0.0037 (11)
C15	0.0182 (11)	0.0198 (10)	0.0188 (10)	-0.0007 (8)	0.0048 (8)	-0.0003 (8)
C16	0.0269 (11)	0.0293 (12)	0.0198 (10)	-0.0029 (9)	-0.0046 (9)	0.0015 (9)
C17	0.0350 (13)	0.0291 (12)	0.0282 (12)	-0.0125 (10)	-0.0001 (11)	-0.0055 (10)
C18	0.0364 (14)	0.0188 (11)	0.0349 (13)	-0.0005 (9)	0.0095 (11)	0.0023 (10)
C19	0.0310 (12)	0.0240 (12)	0.0366 (12)	0.0028 (9)	-0.0037 (11)	0.0091 (10)
C20	0.0267 (11)	0.0229 (11)	0.0292 (11)	-0.0015 (9)	-0.0055 (10)	0.0005 (9)
C21	0.0235 (10)	0.0193 (10)	0.0181 (9)	-0.0017 (8)	-0.0012 (10)	0.0015 (8)
C22	0.0309 (12)	0.0192 (10)	0.0190 (10)	0.0000 (9)	-0.0003 (9)	0.0004 (8)
C23	0.0291 (12)	0.0276 (12)	0.0264 (11)	-0.0029 (10)	0.0045 (10)	-0.0071 (10)
C24	0.0346 (13)	0.0367 (13)	0.0196 (11)	-0.0114 (11)	0.0040 (10)	-0.0014 (10)
C25	0.0382 (14)	0.0332 (13)	0.0243 (12)	-0.0045 (11)	-0.0031 (10)	0.0095 (10)
C26	0.0313 (12)	0.0285 (12)	0.0245 (11)	0.0005 (10)	-0.0039 (10)	0.0038 (9)

*Geometric parameters (Å, °)*

Fe1—C10	2.025 (2)	C11—C12	1.518 (3)
Fe1—C1	2.038 (2)	C11—H11A	0.9800
Fe1—C9	2.041 (2)	C12—H12A	0.9600
Fe1—C6	2.041 (2)	C12—H12B	0.9600
Fe1—C4	2.042 (2)	C12—H12C	0.9600
Fe1—C8	2.045 (2)	C13—C14	1.505 (3)
Fe1—C7	2.046 (2)	C13—H13A	0.9700
Fe1—C2	2.047 (2)	C13—H13B	0.9700
Fe1—C5	2.047 (2)	C14—H14A	0.9600
Fe1—C3	2.050 (2)	C14—H14B	0.9600
P1—C6	1.816 (2)	C14—H14C	0.9600
P1—C21	1.838 (2)	C15—C20	1.381 (3)
P1—C15	1.844 (2)	C15—C16	1.392 (3)
O1—C13	1.424 (3)	C16—C17	1.390 (3)
O1—C11	1.434 (2)	C16—H16A	0.9300
C1—C2	1.411 (3)	C17—C18	1.376 (3)
C1—C5	1.416 (3)	C17—H17A	0.9300
C1—H1A	0.9800	C18—C19	1.375 (3)
C2—C3	1.415 (4)	C18—H18A	0.9300
C2—H2A	0.9800	C19—C20	1.390 (3)
C3—C4	1.412 (4)	C19—H19A	0.9300
C3—H3A	0.9800	C20—H20A	0.9300

## supplementary materials

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C4—C5	1.410 (4)	C21—C22	1.392 (3)
C4—H4A	0.9800	C21—C26	1.393 (3)
C5—H5A	0.9800	C22—C23	1.384 (3)
C6—C10	1.435 (3)	C22—H22A	0.9300
C6—C7	1.439 (3)	C23—C24	1.382 (3)
C7—C8	1.428 (3)	C23—H23A	0.9300
C7—H7A	0.9800	C24—C25	1.376 (3)
C8—C9	1.419 (3)	C24—H24A	0.9300
C8—H8A	0.9800	C25—C26	1.388 (3)
C9—C10	1.418 (3)	C25—H25A	0.9300
C9—H9A	0.9800	C26—H26A	0.9300
C10—C11	1.507 (3)		
C10—Fe1—C1	106.17 (9)	C8—C7—C6	108.05 (18)
C10—Fe1—C9	40.83 (8)	C8—C7—Fe1	69.55 (12)
C1—Fe1—C9	120.74 (9)	C6—C7—Fe1	69.22 (11)
C10—Fe1—C6	41.32 (7)	C8—C7—H7A	126.0
C1—Fe1—C6	123.26 (8)	C6—C7—H7A	126.0
C9—Fe1—C6	68.99 (8)	Fe1—C7—H7A	126.0
C10—Fe1—C4	158.10 (11)	C9—C8—C7	107.99 (18)
C1—Fe1—C4	67.90 (9)	C9—C8—Fe1	69.49 (12)
C9—Fe1—C4	160.50 (11)	C7—C8—Fe1	69.60 (12)
C6—Fe1—C4	122.93 (10)	C9—C8—H8A	126.0
C10—Fe1—C8	68.94 (9)	C7—C8—H8A	126.0
C1—Fe1—C8	156.45 (9)	Fe1—C8—H8A	126.0
C9—Fe1—C8	40.66 (8)	C10—C9—C8	108.57 (18)
C6—Fe1—C8	69.17 (8)	C10—C9—Fe1	69.00 (11)
C4—Fe1—C8	124.97 (10)	C8—C9—Fe1	69.86 (12)
C10—Fe1—C7	69.21 (8)	C10—C9—H9A	125.7
C1—Fe1—C7	160.92 (8)	C8—C9—H9A	125.7
C9—Fe1—C7	68.61 (8)	Fe1—C9—H9A	125.7
C6—Fe1—C7	41.22 (8)	C9—C10—C6	108.25 (18)
C4—Fe1—C7	109.11 (9)	C9—C10—C11	128.47 (18)
C8—Fe1—C7	40.85 (8)	C6—C10—C11	123.28 (18)
C10—Fe1—C2	122.21 (9)	C9—C10—Fe1	70.17 (12)
C1—Fe1—C2	40.41 (9)	C6—C10—Fe1	69.95 (11)
C9—Fe1—C2	106.50 (9)	C11—C10—Fe1	126.19 (14)
C6—Fe1—C2	159.33 (9)	O1—C11—C10	106.40 (16)
C4—Fe1—C2	67.84 (10)	O1—C11—C12	110.14 (17)
C8—Fe1—C2	121.37 (10)	C10—C11—C12	114.32 (17)
C7—Fe1—C2	157.70 (9)	O1—C11—H11A	108.6
C10—Fe1—C5	121.51 (10)	C10—C11—H11A	108.6
C1—Fe1—C5	40.56 (9)	C12—C11—H11A	108.6
C9—Fe1—C5	156.82 (9)	C11—C12—H12A	109.5
C6—Fe1—C5	107.69 (9)	C11—C12—H12B	109.5
C4—Fe1—C5	40.35 (11)	H12A—C12—H12B	109.5
C8—Fe1—C5	161.50 (9)	C11—C12—H12C	109.5
C7—Fe1—C5	124.93 (9)	H12A—C12—H12C	109.5
C2—Fe1—C5	68.07 (10)	H12B—C12—H12C	109.5
C10—Fe1—C3	158.99 (11)	O1—C13—C14	108.08 (18)

C1—Fe1—C3	68.00 (9)	O1—C13—H13A	110.1
C9—Fe1—C3	123.33 (12)	C14—C13—H13A	110.1
C6—Fe1—C3	158.72 (10)	O1—C13—H13B	110.1
C4—Fe1—C3	40.39 (12)	C14—C13—H13B	110.1
C8—Fe1—C3	107.86 (10)	H13A—C13—H13B	108.4
C7—Fe1—C3	122.83 (10)	C13—C14—H14A	109.5
C2—Fe1—C3	40.40 (10)	C13—C14—H14B	109.5
C5—Fe1—C3	68.07 (11)	H14A—C14—H14B	109.5
C6—P1—C21	101.20 (9)	C13—C14—H14C	109.5
C6—P1—C15	102.97 (9)	H14A—C14—H14C	109.5
C21—P1—C15	100.13 (8)	H14B—C14—H14C	109.5
C13—O1—C11	113.47 (16)	C20—C15—C16	118.39 (19)
C2—C1—C5	108.3 (2)	C20—C15—P1	125.17 (15)
C2—C1—Fe1	70.13 (13)	C16—C15—P1	116.40 (15)
C5—C1—Fe1	70.05 (12)	C17—C16—C15	120.8 (2)
C2—C1—H1A	125.9	C17—C16—H16A	119.6
C5—C1—H1A	125.9	C15—C16—H16A	119.6
Fe1—C1—H1A	125.9	C18—C17—C16	120.0 (2)
C1—C2—C3	108.0 (2)	C18—C17—H17A	120.0
C1—C2—Fe1	69.47 (12)	C16—C17—H17A	120.0
C3—C2—Fe1	69.90 (13)	C19—C18—C17	119.5 (2)
C1—C2—H2A	126.0	C19—C18—H18A	120.2
C3—C2—H2A	126.0	C17—C18—H18A	120.2
Fe1—C2—H2A	126.0	C18—C19—C20	120.6 (2)
C4—C3—C2	107.6 (2)	C18—C19—H19A	119.7
C4—C3—Fe1	69.49 (14)	C20—C19—H19A	119.7
C2—C3—Fe1	69.70 (13)	C15—C20—C19	120.57 (19)
C4—C3—H3A	126.2	C15—C20—H20A	119.7
C2—C3—H3A	126.2	C19—C20—H20A	119.7
Fe1—C3—H3A	126.2	C22—C21—C26	118.38 (19)
C5—C4—C3	108.6 (2)	C22—C21—P1	122.46 (15)
C5—C4—Fe1	70.04 (14)	C26—C21—P1	119.16 (15)
C3—C4—Fe1	70.12 (15)	C23—C22—C21	120.66 (19)
C5—C4—H4A	125.7	C23—C22—H22A	119.7
C3—C4—H4A	125.7	C21—C22—H22A	119.7
Fe1—C4—H4A	125.7	C24—C23—C22	120.2 (2)
C4—C5—C1	107.4 (2)	C24—C23—H23A	119.9
C4—C5—Fe1	69.61 (15)	C22—C23—H23A	119.9
C1—C5—Fe1	69.39 (13)	C25—C24—C23	120.0 (2)
C4—C5—H5A	126.3	C25—C24—H24A	120.0
C1—C5—H5A	126.3	C23—C24—H24A	120.0
Fe1—C5—H5A	126.3	C24—C25—C26	120.0 (2)
C10—C6—C7	107.14 (18)	C24—C25—H25A	120.0
C10—C6—P1	122.87 (15)	C26—C25—H25A	120.0
C7—C6—P1	129.82 (15)	C25—C26—C21	120.8 (2)
C10—C6—Fe1	68.73 (11)	C25—C26—H26A	119.6
C7—C6—Fe1	69.57 (11)	C21—C26—H26A	119.6
P1—C6—Fe1	123.06 (10)		
C10—Fe1—C1—C2	-121.03 (14)	Fe1—C6—C7—C8	-58.89 (14)

## supplementary materials

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C9—Fe1—C1—C2	-79.11 (16)	C10—C6—C7—Fe1	58.67 (13)
C6—Fe1—C1—C2	-162.88 (13)	P1—C6—C7—Fe1	-116.57 (17)
C4—Fe1—C1—C2	81.32 (16)	C10—Fe1—C7—C8	81.47 (13)
C8—Fe1—C1—C2	-46.7 (3)	C1—Fe1—C7—C8	160.7 (2)
C7—Fe1—C1—C2	166.0 (2)	C9—Fe1—C7—C8	37.56 (12)
C5—Fe1—C1—C2	119.1 (2)	C6—Fe1—C7—C8	119.68 (17)
C3—Fe1—C1—C2	37.59 (16)	C4—Fe1—C7—C8	-121.79 (15)
C10—Fe1—C1—C5	119.87 (14)	C2—Fe1—C7—C8	-43.7 (3)
C9—Fe1—C1—C5	161.79 (14)	C5—Fe1—C7—C8	-163.90 (13)
C6—Fe1—C1—C5	78.03 (16)	C3—Fe1—C7—C8	-79.16 (17)
C4—Fe1—C1—C5	-37.78 (16)	C10—Fe1—C7—C6	-38.21 (11)
C8—Fe1—C1—C5	-165.8 (2)	C1—Fe1—C7—C6	41.0 (3)
C7—Fe1—C1—C5	46.9 (3)	C9—Fe1—C7—C6	-82.12 (12)
C2—Fe1—C1—C5	-119.1 (2)	C4—Fe1—C7—C6	118.53 (14)
C3—Fe1—C1—C5	-81.51 (17)	C8—Fe1—C7—C6	-119.68 (17)
C5—C1—C2—C3	0.4 (2)	C2—Fe1—C7—C6	-163.4 (2)
Fe1—C1—C2—C3	-59.49 (16)	C5—Fe1—C7—C6	76.41 (15)
C5—C1—C2—Fe1	59.89 (14)	C3—Fe1—C7—C6	161.16 (15)
C10—Fe1—C2—C1	76.58 (16)	C6—C7—C8—C9	-0.4 (2)
C9—Fe1—C2—C1	118.33 (14)	Fe1—C7—C8—C9	-59.09 (14)
C6—Fe1—C2—C1	44.2 (3)	C6—C7—C8—Fe1	58.68 (13)
C4—Fe1—C2—C1	-81.47 (16)	C10—Fe1—C8—C9	37.24 (12)
C8—Fe1—C2—C1	160.08 (13)	C1—Fe1—C8—C9	-44.9 (3)
C7—Fe1—C2—C1	-167.9 (2)	C6—Fe1—C8—C9	81.63 (13)
C5—Fe1—C2—C1	-37.77 (14)	C4—Fe1—C8—C9	-162.03 (14)
C3—Fe1—C2—C1	-119.2 (2)	C7—Fe1—C8—C9	119.40 (17)
C10—Fe1—C2—C3	-164.18 (16)	C2—Fe1—C8—C9	-78.50 (15)
C1—Fe1—C2—C3	119.2 (2)	C5—Fe1—C8—C9	165.2 (3)
C9—Fe1—C2—C3	-122.42 (17)	C3—Fe1—C8—C9	-120.72 (15)
C6—Fe1—C2—C3	163.5 (3)	C10—Fe1—C8—C7	-82.16 (12)
C4—Fe1—C2—C3	37.78 (18)	C1—Fe1—C8—C7	-164.33 (19)
C8—Fe1—C2—C3	-80.68 (19)	C9—Fe1—C8—C7	-119.40 (17)
C7—Fe1—C2—C3	-48.7 (3)	C6—Fe1—C8—C7	-37.77 (12)
C5—Fe1—C2—C3	81.48 (18)	C4—Fe1—C8—C7	78.57 (16)
C1—C2—C3—C4	-0.2 (3)	C2—Fe1—C8—C7	162.10 (12)
Fe1—C2—C3—C4	-59.38 (16)	C5—Fe1—C8—C7	45.8 (3)
C1—C2—C3—Fe1	59.22 (15)	C3—Fe1—C8—C7	119.88 (15)
C10—Fe1—C3—C4	158.9 (2)	C7—C8—C9—C10	0.9 (2)
C1—Fe1—C3—C4	81.29 (16)	Fe1—C8—C9—C10	-58.27 (14)
C9—Fe1—C3—C4	-165.50 (15)	C7—C8—C9—Fe1	59.16 (14)
C6—Fe1—C3—C4	-45.1 (4)	C1—Fe1—C9—C10	-78.90 (14)
C8—Fe1—C3—C4	-123.40 (15)	C6—Fe1—C9—C10	38.18 (11)
C7—Fe1—C3—C4	-80.96 (18)	C4—Fe1—C9—C10	169.5 (3)
C2—Fe1—C3—C4	118.9 (2)	C8—Fe1—C9—C10	120.27 (18)
C5—Fe1—C3—C4	37.39 (15)	C7—Fe1—C9—C10	82.54 (13)
C10—Fe1—C3—C2	40.1 (3)	C2—Fe1—C9—C10	-120.49 (13)
C1—Fe1—C3—C2	-37.59 (15)	C5—Fe1—C9—C10	-47.8 (3)
C9—Fe1—C3—C2	75.63 (18)	C3—Fe1—C9—C10	-161.40 (12)
C6—Fe1—C3—C2	-163.9 (2)	C10—Fe1—C9—C8	-120.27 (18)

C4—Fe1—C3—C2	-118.9 (2)	C1—Fe1—C9—C8	160.83 (12)
C8—Fe1—C3—C2	117.72 (16)	C6—Fe1—C9—C8	-82.09 (13)
C7—Fe1—C3—C2	160.17 (14)	C4—Fe1—C9—C8	49.2 (3)
C5—Fe1—C3—C2	-81.48 (16)	C7—Fe1—C9—C8	-37.73 (12)
C2—C3—C4—C5	-0.1 (3)	C2—Fe1—C9—C8	119.24 (13)
Fe1—C3—C4—C5	-59.65 (16)	C5—Fe1—C9—C8	-168.1 (2)
C2—C3—C4—Fe1	59.51 (16)	C3—Fe1—C9—C8	78.33 (16)
C10—Fe1—C4—C5	-40.2 (3)	C8—C9—C10—C6	-1.0 (2)
C1—Fe1—C4—C5	37.97 (14)	Fe1—C9—C10—C6	-59.82 (13)
C9—Fe1—C4—C5	158.4 (2)	C8—C9—C10—C11	179.80 (19)
C6—Fe1—C4—C5	-78.28 (16)	Fe1—C9—C10—C11	121.0 (2)
C8—Fe1—C4—C5	-164.60 (13)	C8—C9—C10—Fe1	58.80 (15)
C7—Fe1—C4—C5	-121.88 (14)	C7—C6—C10—C9	0.8 (2)
C2—Fe1—C4—C5	81.75 (15)	P1—C6—C10—C9	176.41 (14)
C3—Fe1—C4—C5	119.5 (2)	Fe1—C6—C10—C9	59.96 (14)
C10—Fe1—C4—C3	-159.8 (2)	C7—C6—C10—C11	179.99 (17)
C1—Fe1—C4—C3	-81.58 (15)	P1—C6—C10—C11	-4.4 (3)
C9—Fe1—C4—C3	38.8 (3)	Fe1—C6—C10—C11	-120.81 (18)
C6—Fe1—C4—C3	162.18 (14)	C7—C6—C10—Fe1	-59.20 (13)
C8—Fe1—C4—C3	75.85 (17)	P1—C6—C10—Fe1	116.45 (14)
C7—Fe1—C4—C3	118.57 (15)	C1—Fe1—C10—C9	118.58 (13)
C2—Fe1—C4—C3	-37.80 (15)	C6—Fe1—C10—C9	-119.07 (17)
C5—Fe1—C4—C3	-119.5 (2)	C4—Fe1—C10—C9	-170.6 (2)
C3—C4—C5—C1	0.4 (3)	C8—Fe1—C10—C9	-37.08 (12)
Fe1—C4—C5—C1	-59.31 (15)	C7—Fe1—C10—C9	-80.95 (13)
C3—C4—C5—Fe1	59.71 (17)	C2—Fe1—C10—C9	77.55 (15)
C2—C1—C5—C4	-0.5 (2)	C5—Fe1—C10—C9	159.99 (13)
Fe1—C1—C5—C4	59.45 (16)	C3—Fe1—C10—C9	48.0 (3)
C2—C1—C5—Fe1	-59.94 (15)	C1—Fe1—C10—C6	-122.34 (12)
C10—Fe1—C5—C4	163.58 (15)	C9—Fe1—C10—C6	119.07 (17)
C1—Fe1—C5—C4	-118.8 (2)	C4—Fe1—C10—C6	-51.5 (3)
C9—Fe1—C5—C4	-161.8 (2)	C8—Fe1—C10—C6	81.99 (12)
C6—Fe1—C5—C4	120.39 (16)	C7—Fe1—C10—C6	38.12 (11)
C8—Fe1—C5—C4	43.3 (4)	C2—Fe1—C10—C6	-163.37 (12)
C7—Fe1—C5—C4	78.15 (18)	C5—Fe1—C10—C6	-80.93 (14)
C2—Fe1—C5—C4	-81.13 (16)	C3—Fe1—C10—C6	167.1 (2)
C3—Fe1—C5—C4	-37.42 (16)	C1—Fe1—C10—C11	-5.16 (19)
C10—Fe1—C5—C1	-77.66 (15)	C9—Fe1—C10—C11	-123.7 (2)
C9—Fe1—C5—C1	-43.0 (3)	C6—Fe1—C10—C11	117.2 (2)
C6—Fe1—C5—C1	-120.84 (13)	C4—Fe1—C10—C11	65.6 (3)
C4—Fe1—C5—C1	118.8 (2)	C8—Fe1—C10—C11	-160.82 (19)
C8—Fe1—C5—C1	162.1 (2)	C7—Fe1—C10—C11	155.30 (19)
C7—Fe1—C5—C1	-163.08 (12)	C2—Fe1—C10—C11	-46.2 (2)
C2—Fe1—C5—C1	37.63 (13)	C5—Fe1—C10—C11	36.2 (2)
C3—Fe1—C5—C1	81.34 (15)	C3—Fe1—C10—C11	-75.7 (3)
C21—P1—C6—C10	-163.25 (16)	C13—O1—C11—C10	146.74 (17)
C15—P1—C6—C10	93.47 (17)	C13—O1—C11—C12	-88.9 (2)
C21—P1—C6—C7	11.3 (2)	C9—C10—C11—O1	111.4 (2)
C15—P1—C6—C7	-91.94 (19)	C6—C10—C11—O1	-67.6 (2)

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C21—P1—C6—Fe1	-78.69 (12)	Fe1—C10—C11—O1	-156.03 (13)
C15—P1—C6—Fe1	178.04 (11)	C9—C10—C11—C12	-10.4 (3)
C1—Fe1—C6—C10	76.02 (14)	C6—C10—C11—C12	170.58 (18)
C9—Fe1—C6—C10	-37.74 (12)	Fe1—C10—C11—C12	82.2 (2)
C4—Fe1—C6—C10	159.63 (14)	C11—O1—C13—C14	179.14 (17)
C8—Fe1—C6—C10	-81.41 (12)	C6—P1—C15—C20	5.9 (2)
C7—Fe1—C6—C10	-118.85 (17)	C21—P1—C15—C20	-98.16 (18)
C2—Fe1—C6—C10	43.3 (3)	C6—P1—C15—C16	-171.67 (15)
C5—Fe1—C6—C10	117.91 (13)	C21—P1—C15—C16	84.23 (16)
C3—Fe1—C6—C10	-167.2 (3)	C20—C15—C16—C17	1.5 (3)
C10—Fe1—C6—C7	118.85 (16)	P1—C15—C16—C17	179.30 (17)
C1—Fe1—C6—C7	-165.12 (12)	C15—C16—C17—C18	-0.6 (3)
C9—Fe1—C6—C7	81.11 (12)	C16—C17—C18—C19	-0.3 (3)
C4—Fe1—C6—C7	-81.52 (15)	C17—C18—C19—C20	0.2 (3)
C8—Fe1—C6—C7	37.44 (12)	C16—C15—C20—C19	-1.6 (3)
C2—Fe1—C6—C7	162.1 (2)	P1—C15—C20—C19	-179.18 (17)
C5—Fe1—C6—C7	-123.24 (13)	C18—C19—C20—C15	0.8 (3)
C3—Fe1—C6—C7	-48.4 (3)	C6—P1—C21—C22	-68.60 (17)
C10—Fe1—C6—P1	-116.20 (17)	C15—P1—C21—C22	36.93 (18)
C1—Fe1—C6—P1	-40.17 (16)	C6—P1—C21—C26	111.64 (17)
C9—Fe1—C6—P1	-153.94 (14)	C15—P1—C21—C26	-142.83 (16)
C4—Fe1—C6—P1	43.43 (16)	C26—C21—C22—C23	0.3 (3)
C8—Fe1—C6—P1	162.39 (14)	P1—C21—C22—C23	-179.51 (16)
C7—Fe1—C6—P1	124.95 (17)	C21—C22—C23—C24	-0.6 (3)
C2—Fe1—C6—P1	-72.9 (3)	C22—C23—C24—C25	0.3 (3)
C5—Fe1—C6—P1	1.71 (14)	C23—C24—C25—C26	0.4 (3)
C3—Fe1—C6—P1	76.6 (3)	C24—C25—C26—C21	-0.8 (3)
C10—C6—C7—C8	-0.2 (2)	C22—C21—C26—C25	0.5 (3)
P1—C6—C7—C8	-175.46 (15)	P1—C21—C26—C25	-179.76 (17)

Fig. 1

