# metal-organic compounds

# ($R_C$,$S_{Fe}$)-1-[3,5-Bis(trifluoromethyl)phenyl]-3-{1-[2-(diphenylphosphanyl)ferrocenyl]ethyl}thiourea (unknown solvate)

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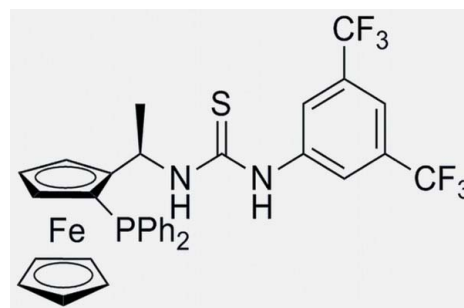
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In the molecule of the the title compound, [Fe(C$_5$H$_5$)(C$_{28}$H$_{22}$F$_6$N$_2$PS)], the absolute configuration is $R_C$,$S_{Fe}$. The dihedral angle between the trifluoromethyl-substituted phenyl ring and the thiourea plane is 41.8 (9)°. The iron atom is bound to the cyclopentadienyl rings in the typical $\eta^5$-manner in a close to eclipsed conformation. The crystal structure features N—H···S hydrogen bonds, with the S atom as an acceptor for both N—H groups, forming a layered arrangement parallel to (1$\bar{1}$0). The two –CF$_3$ groups are each disordered over two positions with refined occupancy rates for the major components of 0.66 (7) and 0.55 (5). The crystal was grown from mixed solvents (*n*-hexane and ethyl acetate). These solvents are disordered in the crystal and the resulting electron density was found to be uninterpretable. The solvent contribution to the structure factors was taken into account by back-Fourier transformation of all density found in the disordered solvent area using the SQUEEZE routine in *PLATON* [Spek (2009). *Acta Cryst.* D**65**, 148–155]. The formula mass and density do not take account of the solvent.

## Related literature

For an introduction to the Morita–Baylis–Hillman reaction, see: Basavaiah *et al.* (2010). For the synthesis of ($R_C$,$S_{Fe}$)-1-(2-(diphenylphosphanyl)ferrocenyl)ethanamine and structures related to the title compound, see: Chen *et al.* (2006). For the synthesis of the title compound, see: Sohtome *et al.* (2004). For refinement details concerning the use of SQUEEZE, see: Spek (2009).



## Experimental

### Crystal data

| | |
|---|---|
| [Fe(C$_5$H$_5$)(C$_{28}$H$_{22}$F$_6$N$_2$PS)] | $Z = 8$ |
| $M_r = 684.45$ | Mo $K\alpha$ radiation |
| Tetragonal, $P4_32_12$ | $\mu = 0.57$ mm$^{-1}$ |
| $a = 20.0898$ (16) Å | $T = 296$ K |
| $c = 18.012$ (2) Å | $0.37 \times 0.31 \times 0.25$ mm |
| $V = 7269.6$ (12) Å$^3$ | |

### Data collection

| | |
|---|---|
| Bruker APEXII CCD area-detector diffractometer | 36567 measured reflections |
| Absorption correction: multi-scan (*SADABS*; Bruker, 2008) | 6472 independent reflections |
| $T_{min} = 0.818$, $T_{max} = 0.869$ | 3487 reflections with $I > 2\sigma(I)$ |
| | $R_{int} = 0.116$ |

### Refinement

| | |
|---|---|
| $R[F^2 > 2\sigma(F^2)] = 0.054$ | $\Delta\rho_{max} = 0.21$ e Å$^{-3}$ |
| $wR(F^2) = 0.119$ | $\Delta\rho_{min} = -0.19$ e Å$^{-3}$ |
| $S = 0.95$ | Absolute structure: Flack (1983), 3680 Friedel pairs |
| 6472 reflections | Flack parameter: 0.01 (2) |
| 455 parameters | |
| H-atom parameters constrained | |

**Table 1**
Hydrogen-bond geometry (Å, °).

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| N1—H1···S1$^i$ | 0.86 | 2.46 | 3.302 (5) | 168 |
| N2—H2···S1$^i$ | 0.86 | 2.64 | 3.445 (4) | 157 |

Symmetry code: (i) $-y + \frac{3}{2}, x - \frac{1}{2}, z - \frac{1}{4}$.

Data collection: *APEX2* (Bruker, 2010); cell refinement: *SAINT* (Bruker, 2008); data reduction: *SAINT*; 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*.

## References

Basavaiah, D., Reddy, B. S. & Badsara, S. S. (2010). *Chem. Rev.* **110**, 5447–5674.

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Chen, W. P., Mbafor, W., Roberts, S. M. & Whittall, J. (2006). *Tetrahedron Asymmetry*, **17**, 1161–1164.

Flack, H. D. (1983). *Acta Cryst.* A**39**, 876–881.

Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

Sohtome, Y., Tanatani, A., Hashimoto, Y. & Nagasawa, K. (2004). *Tetrahedron Lett.* **45**, 5589–5592.

Spek, A. L. (2009). *Acta Cryst.* D**65**, 148–155.

# (*R*C,*S*Fe)-1-[3,5-Bis(trifluoromethyl)phenyl]-3-{1-[2-(diphenyl-phosphanyl)ferrocenyl]ethyl}thiourea (unknown solvate)

**Peng-Fei Ma, Xiao-Rui Zhang, Jiang-Wei Ma, Hui Chen and Ru Jiang**

## Comment

The compound, synthesized by reaction of 1-isothiocyanato-3,5-bis (trifluoromethyl)benzene with (*R*C,*S*Fe)-1-(2-(diphenylphosphanyl)ferrocenyl)ethanamine (Chen *et al.*, 2006), is part of our work towards the synthesis of new phosphanylthiourea ferrocenyl derivatives and their applications in the Morita–Baylis–Hillman (MBH) reaction. The MBH reaction is an atom-economic carbon-carbon bond-forming reaction between the α–position of activated alkenes (alkynes) with carbon electrophiles under the influence of tertiary amines or alkyl (aryl) phosphines (Basavaiah *et al.*, 2010).

The absolute configuration of the title molecule is *R*C,*S*Fe (Chen *et al.*, 2006). The dihedral angle between the trifluoro-methyl substituted phenyl ring C2 → C7 and the plane (S1, C9, N2, N1, H2, H1) comprising the thiourea moiety is 41.8 (9) °. The structure is stabilized by intermolecular N—H···S hydrogen bonds (Fig. 2), with the S atom an acceptor for both N—H groups with an H1···S1 distance of 2.46 Å and an H2···S1 distance of 2.64 Å. The N—H···S angles are 168.4 ° and 165.5 ° (Table 1).

The iron center is bound to the cyclopentadienyl rings in the typical $\eta^5$ manner. The ferrocene group deviates ca. 5 ° from an ideal eclipsed conformation. The angle between the planes of the two cyclopentadienyl rings is 2.8 (9) °.

There are solvent acessible voids in the crystal structure that accomodate solvent molecules in a very disordered way which were corrected for by back-Fourier transformation of all density found in the disordered solvent area using the Squeeze algorithm as implemented in Platon (Spek, 2009). These solvent molecules were not included in the calculation of the overall formula weight, density and absorption coefficient (see refinement section for details).

## Experimental

The title compound was prepared as follows (Sohtome *et al.*, 2004): To a solution of (*R*C,*S*Fe)-1-(2-(diphenyl-phosphanyl)ferrocenyl) ethanamine (2.19 g, 5.30 mmol) in 20 ml THF was added 1-isothiocyanato-3,5-bis (trifluoro-methyl)benzene (1.58 g, 5.8 mmol) at room temperature, and the reaction mixture was stirred for 4 h. The solvent was removed under reduced pressure. The residue was purified by column chromatography (silica gel 60–120 mesh, EtOAc/*n*-hexane 1/5) to provide the desired pure title product as a yellow/solid (3.44 g, 95%). mp = 404 K; IR (KBr, cm⁻¹): ν 3488, 3420, 3235, 2982, 2930, 2028, 1617, 1528, 1381, 1277, 1178, 1135, 886, 748, 698; ¹H NMR (CDCl₃, 500 MHz): δ 7.73 (s, 3H), 7.37–7.30 (m, 5H), 7.22 (s, 5H), 7.10 (s, 2H), 5.59–5.58 (m, 1H), 4.51 (s, 1H), 4.33 (s, 1H), 3.96 (s, 5H), 3.78 (s, 1H), 1.46 (s, 3H); ¹³C NMR (CDCl₃, 126 MHz): δ 138.92, 135.82, 134.84, 134.67, 132.34, 129.53, 128.30, 128.21 (d, *J* = 7.5 Hz), 124.83, 124.01, 121.83, 119.45, 95.04 (d, *J* = 24.6 Hz), 73.21, 72.13, 71.27, 69.85, 69.70, 65.86, 22.26, 15.28; ³¹P NMR (CDCl₃, 202 MHz): δ -24.79; HRMS calcd for C₃₃H₂₇FeN₂PS([*M*+1]⁺): 684.0886, found: 685.0931.

Single crystals were obtained from a solution of the title compound in a mixture of *n*-hexane and EtOAc.

## Refinement

All H atoms were placed in idealized positions and allowed to ride on the respective parent atom with C—H distances of 0.98 Å (ferrocenyl), 0.93 Å (aromatic), 0.96 Å (CH$_3$), or 0.98 Å (CH) and N—H distance of 0.86 Å, and with $U_{iso}$(H) = 1.5 $U_{eq}$(C) for methyl H atoms, with $U_{iso}$(H) = 1.2 $U_{eq}$(C) for aromatic and methylene H atoms.

In the crystal molecule, the two CF$_3$ groups are both disordered over two orientations. The refined occupancy rate for the major moiety of group (F1, F2, F3) is 0.55 (5) [0.66 (7) for group (F4, F5, F6)].

Some residual electron densities were difficult to model, therefore the SQUEEZE function of *PLATON* (Spek, 2009) was used to eliminate the contribution of the electron density in the solvent region from the intensity data, and the solvent-free model was employed for the final refinement. There are two cavities of 652 Å$^3$ per unit cell. *PLATON* estimated that each cavity contains 74 electrons. PLATON estimated that each single crystal molecular contains 20 residual electrons. Because single crystals were obtained from a solution of the title compound in a mixture of n-hexane and EtOAc, so we could not be sure which solvent it was. It may be n-hexane [CH$_3$(CH$_2$)3CH$_3$], EtOAc (CH$_3$COOCH$_2$CH$_3$) or water (H$_2$O). The solvent is therefore not given in the formula, scheme, Mr etc. .

## Computing details

Data collection: *APEX2* (Bruker, 2010); cell refinement: *SAINT* (Bruker, 2008); data reduction: *SAINT* (Bruker, 2008); 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* (Sheldrick, 2008).
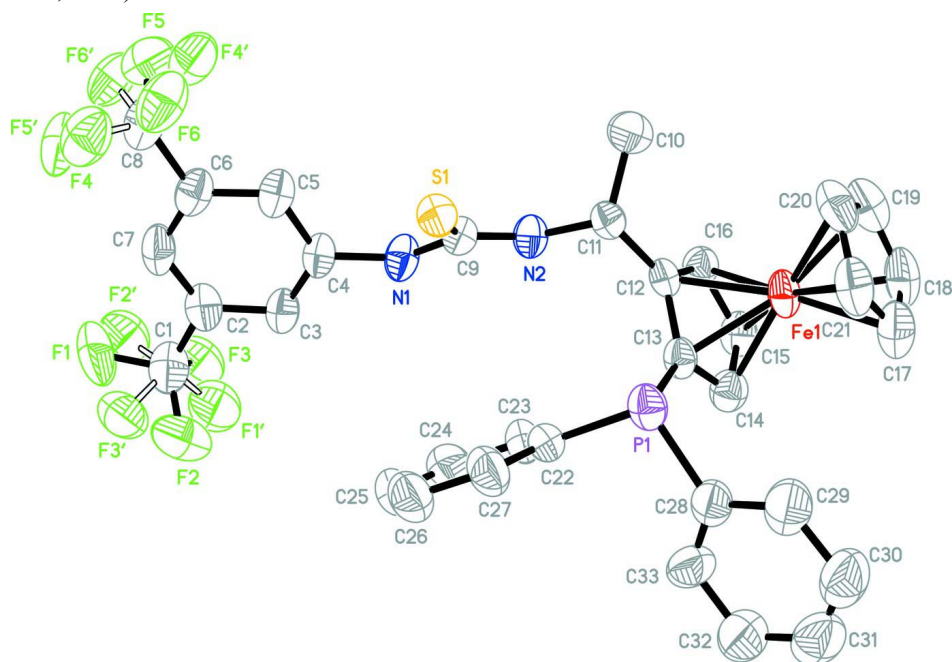


## Figure 1

The molecular structure of the title compound, with displacement ellipsoids at the 30% probability level. All H atoms have been omitted for clarity. The disorder of the CF$_3$ groups has been indicated.
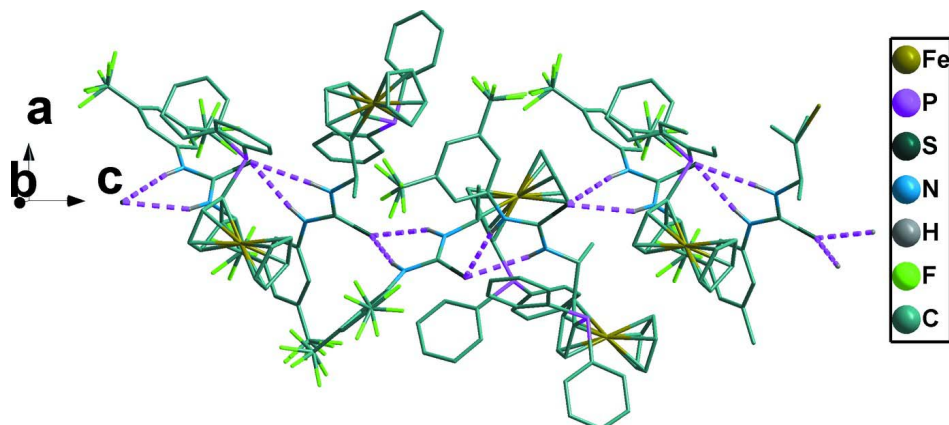
**Figure 2**

Crystal packing in the title compound where molecules are linked *via* N–H···S hydrogen bonds (dashed lines). Except for those involved in hydrogen-bonding interactions, H atoms have been omitted for clarity.

## ($R_C$,$S_{Fe}$)-1-[3,5-Bis(trifluoromethyl)phenyl]-3-{1-[2-(diphenylphosphanyl)ferrocenyl]ethyl}thiourea (unknown solvate)

*Crystal data*

[Fe(C$_5$H$_5$)(C$_{28}$H$_{22}$F$_6$N$_2$PS)]
$M_r$ = 684.45
Tetragonal, $P4_32_12$
Hall symbol: P 4nw 2abw
$a$ = 20.0898 (16) Å
$c$ = 18.012 (2) Å
$V$ = 7269.6 (12) Å$^3$
$Z$ = 8
$F(000)$ = 2800

$D_x$ = 1.251 Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda$ = 0.71073 Å
Cell parameters from 3087 reflections
$\theta$ = 2.5–15.4°
$\mu$ = 0.57 mm$^{-1}$
$T$ = 296 K
Column, yellow
0.37 × 0.31 × 0.25 mm

*Data collection*

Bruker APEXII CCD area-detector
  diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
phi and $\omega$ scans
Absorption correction: multi-scan
  (*SADABS*; Bruker, 2008)
$T_{min}$ = 0.818, $T_{max}$ = 0.869

36567 measured reflections
6472 independent reflections
3487 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.116
$\theta_{max}$ = 25.1°, $\theta_{min}$ = 1.8°
$h$ = −23→21
$k$ = −23→22
$l$ = −21→21

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.054
$wR(F^2)$ = 0.119
$S$ = 0.95
6472 reflections
455 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
$w = 1/[\sigma^2(F_o^2) + (0.0469P)^2]$
  where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.21 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.19 e Å$^{-3}$

Extinction correction: *SHELXL97* (Sheldrick, 2008), Fc*=kFc[1+0.001xFc²*λ*³/sin(2*θ*)]⁻¹ᐟ⁴

Extinction coefficient: 0.00072 (19)

Absolute structure: Flack (1983), 3680 Friedel pairs

Flack parameter: 0.01 (2)

*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* | $U_{iso}$*/$U_{eq}$ | Occ. (<1) |
|---|---|---|---|---|---|
| Fe1 | 1.03592 (4) | 0.27046 (4) | 0.90856 (4) | 0.0791 (3) | |
| P1 | 0.87204 (7) | 0.31616 (7) | 0.91033 (8) | 0.0779 (4) | |
| S1 | 0.88996 (6) | 0.53723 (7) | 0.86099 (6) | 0.0725 (4) | |
| N1 | 0.9005 (2) | 0.5092 (2) | 0.7164 (2) | 0.0824 (13) | |
| H1 | 0.9129 | 0.4805 | 0.6838 | 0.099* | |
| N2 | 0.9655 (2) | 0.44743 (19) | 0.7912 (2) | 0.0723 (11) | |
| H2 | 0.9774 | 0.4304 | 0.7495 | 0.087* | |
| F1 | 0.7195 (16) | 0.6313 (10) | 0.4957 (19) | 0.169 (12) | 0.55 (5) |
| F2 | 0.6984 (12) | 0.533 (2) | 0.5454 (8) | 0.165 (12) | 0.55 (5) |
| F3 | 0.7835 (11) | 0.5518 (18) | 0.4735 (9) | 0.141 (9) | 0.55 (5) |
| F4 | 0.7825 (10) | 0.7839 (8) | 0.683 (2) | 0.136 (5) | 0.66 (7) |
| F5 | 0.8914 (15) | 0.7744 (15) | 0.678 (2) | 0.129 (7) | 0.66 (7) |
| F6 | 0.835 (2) | 0.7507 (6) | 0.7759 (6) | 0.127 (9) | 0.66 (7) |
| F1′ | 0.753 (2) | 0.5192 (16) | 0.500 (2) | 0.166 (16) | 0.45 (5) |
| F2′ | 0.755 (2) | 0.6185 (17) | 0.4681 (12) | 0.154 (12) | 0.45 (5) |
| F3′ | 0.6802 (10) | 0.586 (2) | 0.5446 (9) | 0.143 (11) | 0.45 (5) |
| F4′ | 0.883 (4) | 0.7539 (10) | 0.770 (2) | 0.131 (12) | 0.34 (7) |
| F5′ | 0.789 (2) | 0.774 (2) | 0.726 (4) | 0.134 (16) | 0.34 (7) |
| F6′ | 0.864 (3) | 0.786 (2) | 0.659 (2) | 0.117 (12) | 0.34 (7) |
| C1 | 0.7441 (6) | 0.5779 (6) | 0.5286 (6) | 0.127 (3) | |
| C2 | 0.7878 (3) | 0.5970 (4) | 0.5932 (3) | 0.0953 (17) | |
| C3 | 0.8220 (3) | 0.5467 (3) | 0.6282 (3) | 0.0840 (16) | |
| H3 | 0.8185 | 0.5030 | 0.6114 | 0.101* | |
| C4 | 0.8613 (3) | 0.5617 (3) | 0.6880 (3) | 0.0769 (15) | |
| C5 | 0.8671 (3) | 0.6269 (3) | 0.7140 (3) | 0.0844 (15) | |
| H5 | 0.8947 | 0.6368 | 0.7539 | 0.101* | |
| C6 | 0.8310 (3) | 0.6761 (3) | 0.6793 (3) | 0.0855 (17) | |
| C7 | 0.7920 (3) | 0.6615 (4) | 0.6185 (4) | 0.0983 (19) | |
| H7 | 0.7685 | 0.6952 | 0.5946 | 0.118* | |
| C8 | 0.8385 (6) | 0.7456 (4) | 0.7049 (5) | 0.107 (2) | |
| C9 | 0.9223 (3) | 0.4961 (3) | 0.7875 (3) | 0.0687 (13) | |
| C10 | 1.0638 (3) | 0.4510 (3) | 0.8698 (4) | 0.114 (2) | |
| H10A | 1.0577 | 0.4960 | 0.8862 | 0.171* | |

| | | | | |
|---|---|---|---|---|
| H10B | 1.0885 | 0.4508 | 0.8242 | 0.171* |
| H10C | 1.0878 | 0.4264 | 0.9068 | 0.171* |
| C11 | 0.9958 (2) | 0.4186 (2) | 0.8571 (3) | 0.0687 (13) |
| H11 | 0.9673 | 0.4275 | 0.9001 | 0.082* |
| C12 | 0.9997 (2) | 0.3449 (2) | 0.8450 (2) | 0.0636 (12) |
| C13 | 0.9480 (2) | 0.2979 (3) | 0.8596 (2) | 0.0663 (13) |
| C14 | 0.9697 (3) | 0.2346 (3) | 0.8330 (3) | 0.0825 (15) |
| H14 | 0.9451 | 0.1927 | 0.8367 | 0.099* |
| C15 | 1.0332 (3) | 0.2433 (3) | 0.7997 (3) | 0.0829 (16) |
| H15 | 1.0601 | 0.2083 | 0.7765 | 0.099* |
| C16 | 1.0515 (3) | 0.3111 (3) | 0.8070 (3) | 0.0776 (15) |
| H16 | 1.0932 | 0.3311 | 0.7896 | 0.093* |
| C17 | 1.0393 (4) | 0.2044 (4) | 0.9946 (4) | 0.115 (2) |
| H17 | 1.0116 | 0.1646 | 1.0001 | 0.138* |
| C18 | 1.1014 (4) | 0.2080 (5) | 0.9573 (4) | 0.121 (2) |
| H18 | 1.1246 | 0.1709 | 0.9332 | 0.146* |
| C19 | 1.1240 (4) | 0.2719 (5) | 0.9628 (4) | 0.118 (2) |
| H19 | 1.1661 | 0.2886 | 0.9425 | 0.141* |
| C20 | 1.0774 (4) | 0.3099 (4) | 1.0026 (4) | 0.106 (2) |
| H20 | 1.0811 | 0.3574 | 1.0146 | 0.127* |
| C21 | 1.0246 (4) | 0.2680 (5) | 1.0218 (3) | 0.108 (2) |
| H21 | 0.9846 | 0.2807 | 1.0495 | 0.130* |
| C22 | 0.8187 (3) | 0.3452 (3) | 0.8353 (3) | 0.0748 (14) |
| C23 | 0.8331 (3) | 0.3392 (3) | 0.7626 (4) | 0.0925 (18) |
| H23 | 0.8721 | 0.3177 | 0.7487 | 0.111* |
| C24 | 0.7910 (4) | 0.3642 (4) | 0.7074 (4) | 0.113 (2) |
| H24 | 0.8013 | 0.3596 | 0.6573 | 0.135* |
| C25 | 0.7343 (4) | 0.3957 (4) | 0.7299 (5) | 0.115 (2) |
| H25 | 0.7059 | 0.4133 | 0.6941 | 0.139* |
| C26 | 0.7185 (3) | 0.4020 (4) | 0.7996 (5) | 0.120 (2) |
| H26 | 0.6791 | 0.4231 | 0.8132 | 0.144* |
| C27 | 0.7606 (3) | 0.3772 (3) | 0.8526 (4) | 0.1024 (18) |
| H27 | 0.7492 | 0.3823 | 0.9023 | 0.123* |
| C28 | 0.8417 (3) | 0.2309 (3) | 0.9269 (4) | 0.0954 (19) |
| C29 | 0.8552 (3) | 0.2030 (4) | 0.9948 (4) | 0.124 (2) |
| H29 | 0.8762 | 0.2281 | 1.0313 | 0.149* |
| C30 | 0.8373 (4) | 0.1364 (5) | 1.0092 (6) | 0.144 (3) |
| H30 | 0.8475 | 0.1167 | 1.0546 | 0.173* |
| C31 | 0.8057 (5) | 0.1022 (5) | 0.9568 (7) | 0.148 (4) |
| H31 | 0.7949 | 0.0579 | 0.9660 | 0.178* |
| C32 | 0.7886 (4) | 0.1290 (5) | 0.8910 (6) | 0.147 (3) |
| H32 | 0.7641 | 0.1048 | 0.8565 | 0.177* |
| C33 | 0.8088 (3) | 0.1945 (4) | 0.8757 (4) | 0.112 (2) |
| H33 | 0.7993 | 0.2131 | 0.8296 | 0.134* |

*Atomic displacement parameters (Å$^2$)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| Fe1 | 0.0709 (5) | 0.0911 (6) | 0.0755 (5) | 0.0309 (4) | 0.0026 (4) | 0.0142 (4) |
| P1 | 0.0686 (9) | 0.0906 (11) | 0.0745 (8) | 0.0174 (8) | 0.0099 (8) | 0.0051 (8) |

| | | | | | |
|---|---|---|---|---|---|
| S1 | 0.0756 (9) | 0.0757 (9) | 0.0663 (7) | 0.0083 (7) | −0.0027 (7) | −0.0090 (7) |
| N1 | 0.109 (3) | 0.067 (3) | 0.071 (3) | 0.025 (3) | −0.001 (3) | 0.000 (2) |
| N2 | 0.079 (3) | 0.071 (3) | 0.067 (3) | 0.017 (2) | 0.004 (2) | 0.003 (2) |
| F1 | 0.15 (2) | 0.190 (13) | 0.16 (2) | 0.070 (14) | −0.058 (17) | 0.021 (14) |
| F2 | 0.117 (12) | 0.20 (3) | 0.175 (10) | −0.037 (17) | −0.021 (9) | −0.004 (12) |
| F3 | 0.130 (11) | 0.18 (2) | 0.109 (8) | −0.002 (11) | −0.027 (7) | −0.005 (9) |
| F4 | 0.168 (10) | 0.092 (5) | 0.150 (14) | 0.053 (5) | −0.010 (10) | 0.022 (8) |
| F5 | 0.127 (12) | 0.101 (11) | 0.158 (19) | −0.013 (8) | 0.028 (10) | −0.013 (10) |
| F6 | 0.19 (3) | 0.090 (5) | 0.097 (6) | 0.021 (8) | 0.012 (9) | −0.006 (4) |
| F1′ | 0.18 (3) | 0.165 (19) | 0.16 (2) | −0.008 (18) | −0.06 (2) | −0.016 (16) |
| F2′ | 0.15 (2) | 0.18 (3) | 0.131 (12) | 0.003 (19) | −0.026 (12) | 0.004 (12) |
| F3′ | 0.101 (11) | 0.16 (3) | 0.164 (11) | −0.012 (11) | −0.023 (8) | 0.024 (12) |
| F4′ | 0.16 (4) | 0.083 (9) | 0.153 (18) | 0.006 (12) | −0.011 (17) | 0.001 (8) |
| F5′ | 0.14 (2) | 0.107 (17) | 0.16 (3) | 0.021 (13) | 0.04 (2) | −0.01 (2) |
| F6′ | 0.17 (4) | 0.082 (11) | 0.104 (13) | −0.005 (18) | 0.027 (19) | 0.025 (9) |
| C1 | 0.135 (10) | 0.114 (9) | 0.134 (9) | 0.025 (8) | −0.011 (8) | 0.013 (7) |
| C2 | 0.098 (5) | 0.100 (5) | 0.088 (4) | 0.022 (4) | −0.006 (4) | 0.006 (4) |
| C3 | 0.094 (4) | 0.079 (4) | 0.079 (4) | 0.018 (3) | −0.001 (3) | 0.018 (3) |
| C4 | 0.091 (4) | 0.070 (4) | 0.070 (3) | 0.019 (3) | 0.001 (3) | 0.012 (3) |
| C5 | 0.101 (4) | 0.074 (4) | 0.078 (4) | 0.022 (3) | 0.011 (3) | 0.008 (3) |
| C6 | 0.100 (5) | 0.071 (4) | 0.086 (4) | 0.017 (4) | 0.022 (3) | 0.009 (3) |
| C7 | 0.096 (5) | 0.097 (5) | 0.102 (5) | 0.031 (4) | 0.003 (4) | 0.033 (4) |
| C8 | 0.125 (8) | 0.088 (7) | 0.110 (7) | 0.023 (6) | 0.023 (6) | 0.014 (5) |
| C9 | 0.074 (4) | 0.063 (3) | 0.069 (3) | 0.003 (3) | 0.001 (3) | 0.002 (3) |
| C10 | 0.096 (5) | 0.092 (5) | 0.155 (6) | 0.001 (4) | −0.028 (4) | −0.011 (4) |
| C11 | 0.069 (3) | 0.073 (4) | 0.064 (3) | 0.002 (3) | −0.010 (3) | 0.003 (3) |
| C12 | 0.056 (3) | 0.071 (3) | 0.064 (3) | 0.013 (3) | −0.007 (3) | 0.005 (3) |
| C13 | 0.059 (3) | 0.077 (4) | 0.062 (3) | 0.015 (3) | 0.009 (2) | 0.011 (3) |
| C14 | 0.083 (4) | 0.082 (4) | 0.083 (4) | 0.009 (3) | 0.006 (3) | 0.006 (3) |
| C15 | 0.088 (4) | 0.085 (4) | 0.075 (3) | 0.023 (3) | 0.013 (3) | 0.000 (3) |
| C16 | 0.072 (4) | 0.079 (4) | 0.082 (4) | 0.006 (3) | 0.006 (3) | 0.010 (3) |
| C17 | 0.119 (7) | 0.125 (7) | 0.102 (5) | 0.031 (5) | −0.004 (5) | 0.037 (4) |
| C18 | 0.113 (6) | 0.129 (7) | 0.122 (6) | 0.055 (5) | −0.014 (5) | 0.028 (5) |
| C19 | 0.092 (5) | 0.155 (7) | 0.105 (5) | 0.023 (6) | −0.024 (4) | 0.009 (6) |
| C20 | 0.104 (5) | 0.126 (6) | 0.087 (4) | 0.025 (5) | −0.035 (4) | −0.009 (4) |
| C21 | 0.112 (6) | 0.131 (6) | 0.080 (4) | 0.034 (5) | −0.011 (4) | 0.019 (4) |
| C22 | 0.061 (3) | 0.071 (3) | 0.092 (4) | 0.002 (3) | −0.006 (3) | 0.007 (3) |
| C23 | 0.081 (4) | 0.095 (4) | 0.102 (5) | 0.013 (3) | −0.016 (4) | 0.017 (4) |
| C24 | 0.105 (6) | 0.119 (6) | 0.114 (5) | −0.014 (5) | −0.017 (5) | 0.020 (5) |
| C25 | 0.091 (5) | 0.116 (6) | 0.140 (7) | 0.008 (5) | −0.031 (5) | 0.023 (5) |
| C26 | 0.090 (5) | 0.114 (6) | 0.156 (7) | 0.021 (4) | −0.021 (6) | −0.009 (6) |
| C27 | 0.080 (4) | 0.105 (5) | 0.121 (5) | 0.023 (4) | −0.013 (4) | −0.006 (4) |
| C28 | 0.068 (4) | 0.105 (5) | 0.114 (5) | 0.012 (4) | 0.017 (4) | 0.032 (5) |
| C29 | 0.096 (5) | 0.135 (7) | 0.142 (7) | 0.018 (5) | 0.024 (5) | 0.043 (5) |
| C30 | 0.119 (7) | 0.145 (9) | 0.168 (9) | 0.018 (6) | 0.032 (6) | 0.063 (7) |
| C31 | 0.108 (7) | 0.136 (8) | 0.201 (11) | 0.005 (6) | 0.041 (7) | 0.047 (8) |
| C32 | 0.117 (6) | 0.135 (8) | 0.190 (9) | 0.002 (6) | 0.007 (6) | 0.041 (7) |
| C33 | 0.096 (5) | 0.099 (5) | 0.140 (6) | −0.017 (4) | 0.016 (5) | 0.023 (5) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| Fe1—C18 | 2.018 (6) | C10—H10B | 0.9600 |
| Fe1—C12 | 2.019 (4) | C10—H10C | 0.9600 |
| Fe1—C19 | 2.021 (6) | C11—C12 | 1.497 (6) |
| Fe1—C16 | 2.028 (5) | C11—H11 | 0.9800 |
| Fe1—C14 | 2.034 (5) | C12—C16 | 1.419 (6) |
| Fe1—C15 | 2.036 (5) | C12—C13 | 1.428 (6) |
| Fe1—C17 | 2.040 (6) | C13—C14 | 1.426 (6) |
| Fe1—C20 | 2.048 (6) | C14—C15 | 1.420 (7) |
| Fe1—C13 | 2.049 (4) | C14—H14 | 0.9800 |
| Fe1—C21 | 2.053 (6) | C15—C16 | 1.416 (7) |
| P1—C13 | 1.817 (5) | C15—H15 | 0.9800 |
| P1—C22 | 1.820 (5) | C16—H16 | 0.9800 |
| P1—C28 | 1.843 (7) | C17—C21 | 1.399 (8) |
| S1—C9 | 1.690 (5) | C17—C18 | 1.419 (9) |
| N1—C9 | 1.378 (6) | C17—H17 | 0.9800 |
| N1—C4 | 1.413 (6) | C18—C19 | 1.366 (9) |
| N1—H1 | 0.8600 | C18—H18 | 0.9800 |
| N2—C9 | 1.309 (5) | C19—C20 | 1.405 (9) |
| N2—C11 | 1.455 (5) | C19—H19 | 0.9800 |
| N2—H2 | 0.8600 | C20—C21 | 1.398 (8) |
| F1—C1 | 1.32 (2) | C20—H20 | 0.9800 |
| F2—C1 | 1.32 (2) | C21—H21 | 0.9800 |
| F3—C1 | 1.37 (2) | C22—C23 | 1.346 (7) |
| F4—C8 | 1.42 (2) | C22—C27 | 1.370 (7) |
| F5—C8 | 1.30 (2) | C23—C24 | 1.399 (7) |
| F6—C8 | 1.286 (13) | C23—H23 | 0.9300 |
| F1′—C1 | 1.30 (2) | C24—C25 | 1.365 (9) |
| F2′—C1 | 1.38 (2) | C24—H24 | 0.9300 |
| F3′—C1 | 1.33 (3) | C25—C26 | 1.302 (8) |
| F4′—C8 | 1.49 (3) | C25—H25 | 0.9300 |
| F5′—C8 | 1.22 (4) | C26—C27 | 1.369 (8) |
| F6′—C8 | 1.27 (4) | C26—H26 | 0.9300 |
| C1—C2 | 1.508 (10) | C27—H27 | 0.9300 |
| C2—C3 | 1.375 (7) | C28—C33 | 1.349 (8) |
| C2—C7 | 1.377 (8) | C28—C29 | 1.373 (8) |
| C3—C4 | 1.369 (7) | C29—C30 | 1.410 (10) |
| C3—H3 | 0.9300 | C29—H29 | 0.9300 |
| C4—C5 | 1.396 (7) | C30—C31 | 1.330 (10) |
| C5—C6 | 1.377 (7) | C30—H30 | 0.9300 |
| C5—H5 | 0.9300 | C31—C32 | 1.348 (10) |
| C6—C7 | 1.378 (7) | C31—H31 | 0.9300 |
| C6—C8 | 1.478 (9) | C32—C33 | 1.403 (9) |
| C7—H7 | 0.9300 | C32—H32 | 0.9300 |
| C10—C11 | 1.529 (7) | C33—H33 | 0.9300 |
| C10—H10A | 0.9600 | | |
| | | | |
| C18—Fe1—C12 | 160.3 (3) | N2—C9—S1 | 125.4 (4) |
| C18—Fe1—C19 | 39.5 (3) | N1—C9—S1 | 120.8 (4) |

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|---|---|---|---|
| C12—Fe1—C19 | 125.3 (3) | C11—C10—H10A | 109.5 |
| C18—Fe1—C16 | 122.8 (3) | C11—C10—H10B | 109.5 |
| C12—Fe1—C16 | 41.04 (18) | H10A—C10—H10B | 109.5 |
| C19—Fe1—C16 | 107.2 (3) | C11—C10—H10C | 109.5 |
| C18—Fe1—C14 | 119.8 (3) | H10A—C10—H10C | 109.5 |
| C12—Fe1—C14 | 69.4 (2) | H10B—C10—H10C | 109.5 |
| C19—Fe1—C14 | 154.3 (3) | N2—C11—C12 | 107.3 (4) |
| C16—Fe1—C14 | 68.9 (2) | N2—C11—C10 | 109.0 (4) |
| C18—Fe1—C15 | 105.6 (3) | C12—C11—C10 | 113.3 (4) |
| C12—Fe1—C15 | 69.09 (19) | N2—C11—H11 | 109.0 |
| C19—Fe1—C15 | 119.5 (3) | C12—C11—H11 | 109.0 |
| C16—Fe1—C15 | 40.79 (18) | C10—C11—H11 | 109.0 |
| C14—Fe1—C15 | 40.85 (19) | C16—C12—C13 | 107.7 (4) |
| C18—Fe1—C17 | 40.9 (2) | C16—C12—C11 | 125.6 (5) |
| C12—Fe1—C17 | 157.5 (3) | C13—C12—C11 | 126.1 (4) |
| C19—Fe1—C17 | 67.3 (3) | C16—C12—Fe1 | 69.8 (3) |
| C16—Fe1—C17 | 160.3 (3) | C13—C12—Fe1 | 70.6 (3) |
| C14—Fe1—C17 | 107.4 (3) | C11—C12—Fe1 | 132.0 (3) |
| C15—Fe1—C17 | 123.9 (3) | C14—C13—C12 | 107.8 (4) |
| C18—Fe1—C20 | 67.4 (3) | C14—C13—P1 | 127.3 (4) |
| C12—Fe1—C20 | 109.2 (3) | C12—C13—P1 | 124.7 (4) |
| C19—Fe1—C20 | 40.4 (2) | C14—C13—Fe1 | 69.0 (3) |
| C16—Fe1—C20 | 121.9 (3) | C12—C13—Fe1 | 68.3 (3) |
| C14—Fe1—C20 | 163.0 (3) | P1—C13—Fe1 | 124.2 (2) |
| C15—Fe1—C20 | 155.7 (3) | C15—C14—C13 | 107.9 (5) |
| C17—Fe1—C20 | 67.1 (3) | C15—C14—Fe1 | 69.7 (3) |
| C18—Fe1—C13 | 156.3 (4) | C13—C14—Fe1 | 70.1 (3) |
| C12—Fe1—C13 | 41.10 (17) | C15—C14—H14 | 126.1 |
| C19—Fe1—C13 | 163.4 (3) | C13—C14—H14 | 126.1 |
| C16—Fe1—C13 | 68.64 (19) | Fe1—C14—H14 | 126.1 |
| C14—Fe1—C13 | 40.87 (18) | C16—C15—C14 | 108.1 (5) |
| C15—Fe1—C13 | 68.5 (2) | C16—C15—Fe1 | 69.3 (3) |
| C17—Fe1—C13 | 122.0 (3) | C14—C15—Fe1 | 69.5 (3) |
| C20—Fe1—C13 | 127.1 (3) | C16—C15—H15 | 125.9 |
| C18—Fe1—C21 | 68.0 (3) | C14—C15—H15 | 125.9 |
| C12—Fe1—C21 | 122.8 (3) | Fe1—C15—H15 | 125.9 |
| C19—Fe1—C21 | 67.5 (3) | C15—C16—C12 | 108.4 (5) |
| C16—Fe1—C21 | 157.4 (3) | C15—C16—Fe1 | 69.9 (3) |
| C14—Fe1—C21 | 125.7 (3) | C12—C16—Fe1 | 69.2 (3) |
| C15—Fe1—C21 | 161.3 (3) | C15—C16—H16 | 125.8 |
| C17—Fe1—C21 | 40.0 (2) | C12—C16—H16 | 125.8 |
| C20—Fe1—C21 | 39.9 (2) | Fe1—C16—H16 | 125.8 |
| C13—Fe1—C21 | 109.8 (2) | C21—C17—C18 | 107.8 (7) |
| C13—P1—C22 | 100.7 (2) | C21—C17—Fe1 | 70.5 (4) |
| C13—P1—C28 | 99.9 (3) | C18—C17—Fe1 | 68.7 (4) |
| C22—P1—C28 | 102.9 (3) | C21—C17—H17 | 126.1 |
| C9—N1—C4 | 131.0 (4) | C18—C17—H17 | 126.1 |
| C9—N1—H1 | 114.5 | Fe1—C17—H17 | 126.1 |
| C4—N1—H1 | 114.5 | C19—C18—C17 | 107.8 (7) |

| | | | |
|---|---|---|---|
| C9—N2—C11 | 128.1 (4) | C19—C18—Fe1 | 70.3 (4) |
| C9—N2—H2 | 116.0 | C17—C18—Fe1 | 70.4 (4) |
| C11—N2—H2 | 116.0 | C19—C18—H18 | 126.1 |
| F1′—C1—F2 | 64.6 (18) | C17—C18—H18 | 126.1 |
| F1′—C1—F1 | 127.5 (14) | Fe1—C18—H18 | 126.1 |
| F2—C1—F1 | 113.3 (16) | C18—C19—C20 | 109.1 (7) |
| F1′—C1—F3′ | 109.3 (19) | C18—C19—Fe1 | 70.1 (4) |
| F2—C1—F3′ | 50.1 (11) | C20—C19—Fe1 | 70.8 (4) |
| F1—C1—F3′ | 68.7 (11) | C18—C19—H19 | 125.4 |
| F2—C1—F3 | 107.8 (15) | C20—C19—H19 | 125.4 |
| F1—C1—F3 | 101.6 (16) | Fe1—C19—H19 | 125.4 |
| F3′—C1—F3 | 139.6 (12) | C21—C20—C19 | 107.6 (7) |
| F1′—C1—F2′ | 101.4 (19) | C21—C20—Fe1 | 70.2 (4) |
| F2—C1—F2′ | 134.3 (13) | C19—C20—Fe1 | 68.8 (4) |
| F3′—C1—F2′ | 105.1 (15) | C21—C20—H20 | 126.2 |
| F3—C1—F2′ | 63.8 (14) | C19—C20—H20 | 126.2 |
| F1′—C1—C2 | 117.2 (11) | Fe1—C20—H20 | 126.2 |
| F2—C1—C2 | 113.7 (12) | C20—C21—C17 | 107.7 (7) |
| F1—C1—C2 | 111.0 (12) | C20—C21—Fe1 | 69.9 (4) |
| F3′—C1—C2 | 111.5 (13) | C17—C21—Fe1 | 69.5 (4) |
| F3—C1—C2 | 108.6 (11) | C20—C21—H21 | 126.2 |
| F2′—C1—C2 | 111.3 (13) | C17—C21—H21 | 126.2 |
| C3—C2—C7 | 120.7 (6) | Fe1—C21—H21 | 126.2 |
| C3—C2—C1 | 117.2 (7) | C23—C22—C27 | 116.5 (5) |
| C7—C2—C1 | 122.0 (7) | C23—C22—P1 | 124.6 (5) |
| C4—C3—C2 | 119.1 (6) | C27—C22—P1 | 118.9 (5) |
| C4—C3—H3 | 120.4 | C22—C23—C24 | 122.0 (6) |
| C2—C3—H3 | 120.4 | C22—C23—H23 | 119.0 |
| C3—C4—C5 | 121.2 (5) | C24—C23—H23 | 119.0 |
| C3—C4—N1 | 116.2 (5) | C25—C24—C23 | 117.4 (7) |
| C5—C4—N1 | 122.2 (5) | C25—C24—H24 | 121.3 |
| C6—C5—C4 | 118.6 (5) | C23—C24—H24 | 121.3 |
| C6—C5—H5 | 120.7 | C26—C25—C24 | 122.4 (7) |
| C4—C5—H5 | 120.7 | C26—C25—H25 | 118.8 |
| C5—C6—C7 | 120.5 (6) | C24—C25—H25 | 118.8 |
| C5—C6—C8 | 118.9 (7) | C25—C26—C27 | 119.1 (7) |
| C7—C6—C8 | 120.5 (7) | C25—C26—H26 | 120.5 |
| C2—C7—C6 | 119.8 (6) | C27—C26—H26 | 120.5 |
| C2—C7—H7 | 120.1 | C26—C27—C22 | 122.7 (6) |
| C6—C7—H7 | 120.1 | C26—C27—H27 | 118.7 |
| F5′—C8—F6′ | 104 (2) | C22—C27—H27 | 118.7 |
| F5′—C8—F6 | 67 (2) | C33—C28—C29 | 119.0 (7) |
| F6′—C8—F6 | 128 (2) | C33—C28—P1 | 123.7 (6) |
| F5′—C8—F5 | 125.2 (19) | C29—C28—P1 | 117.2 (7) |
| F6—C8—F5 | 112.1 (16) | C28—C29—C30 | 120.1 (8) |
| F6′—C8—F4 | 78 (2) | C28—C29—H29 | 120.0 |
| F6—C8—F4 | 101.0 (12) | C30—C29—H29 | 120.0 |
| F5—C8—F4 | 107.7 (14) | C31—C30—C29 | 118.8 (10) |
| F5′—C8—C6 | 117.4 (17) | C31—C30—H30 | 120.6 |

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| F6′—C8—C6 | 116 (2) | C29—C30—H30 | 120.6 |
| F6—C8—C6 | 112.4 (9) | C30—C31—C32 | 122.7 (11) |
| F5—C8—C6 | 112.7 (14) | C30—C31—H31 | 118.7 |
| F4—C8—C6 | 110.3 (11) | C32—C31—H31 | 118.7 |
| F5′—C8—F4′ | 101.5 (18) | C31—C32—C33 | 118.3 (9) |
| F6′—C8—F4′ | 102 (2) | C31—C32—H32 | 120.9 |
| F5—C8—F4′ | 75.8 (18) | C33—C32—H32 | 120.9 |
| F4—C8—F4′ | 129.2 (14) | C28—C33—C32 | 121.1 (8) |
| C6—C8—F4′ | 114.5 (11) | C28—C33—H33 | 119.5 |
| N2—C9—N1 | 113.6 (4) | C32—C33—H33 | 119.5 |
| | | | |
| F1′—C1—C2—C3 | −15 (4) | C16—Fe1—C15—C14 | −119.8 (4) |
| F2—C1—C2—C3 | 57 (3) | C17—Fe1—C15—C14 | 76.9 (4) |
| F1—C1—C2—C3 | −174 (2) | C20—Fe1—C15—C14 | −172.7 (6) |
| F3′—C1—C2—C3 | 112 (2) | C13—Fe1—C15—C14 | −38.0 (3) |
| F3—C1—C2—C3 | −63 (2) | C21—Fe1—C15—C14 | 50.5 (9) |
| F2′—C1—C2—C3 | −131 (2) | C14—C15—C16—C12 | 0.2 (5) |
| F1′—C1—C2—C7 | 167 (3) | Fe1—C15—C16—C12 | −58.6 (3) |
| F2—C1—C2—C7 | −121 (2) | C14—C15—C16—Fe1 | 58.8 (3) |
| F1—C1—C2—C7 | 9 (3) | C13—C12—C16—C15 | −1.6 (5) |
| F3′—C1—C2—C7 | −66 (2) | C11—C12—C16—C15 | −173.1 (4) |
| F3—C1—C2—C7 | 119.4 (19) | Fe1—C12—C16—C15 | 59.1 (3) |
| F2′—C1—C2—C7 | 51 (3) | C13—C12—C16—Fe1 | −60.7 (3) |
| C7—C2—C3—C4 | −1.0 (9) | C11—C12—C16—Fe1 | 127.8 (5) |
| C1—C2—C3—C4 | −178.7 (8) | C18—Fe1—C16—C15 | 75.1 (5) |
| C2—C3—C4—C5 | 0.1 (8) | C12—Fe1—C16—C15 | −119.9 (4) |
| C2—C3—C4—N1 | −173.4 (5) | C19—Fe1—C16—C15 | 115.5 (4) |
| C9—N1—C4—C3 | −149.3 (5) | C14—Fe1—C16—C15 | −37.5 (3) |
| C9—N1—C4—C5 | 37.2 (8) | C17—Fe1—C16—C15 | 45.2 (9) |
| C3—C4—C5—C6 | 1.6 (8) | C20—Fe1—C16—C15 | 157.3 (4) |
| N1—C4—C5—C6 | 174.7 (5) | C13—Fe1—C16—C15 | −81.5 (3) |
| C4—C5—C6—C7 | −2.3 (8) | C21—Fe1—C16—C15 | −171.9 (6) |
| C4—C5—C6—C8 | −178.0 (6) | C18—Fe1—C16—C12 | −164.9 (4) |
| C3—C2—C7—C6 | 0.3 (9) | C19—Fe1—C16—C12 | −124.5 (4) |
| C1—C2—C7—C6 | 177.9 (8) | C14—Fe1—C16—C12 | 82.5 (3) |
| C5—C6—C7—C2 | 1.4 (9) | C15—Fe1—C16—C12 | 119.9 (4) |
| C8—C6—C7—C2 | 177.0 (7) | C17—Fe1—C16—C12 | 165.2 (7) |
| C5—C6—C8—F5′ | −122 (5) | C20—Fe1—C16—C12 | −82.8 (4) |
| C7—C6—C8—F5′ | 62 (5) | C13—Fe1—C16—C12 | 38.5 (3) |
| C5—C6—C8—F6′ | 115 (4) | C21—Fe1—C16—C12 | −52.0 (7) |
| C7—C6—C8—F6′ | −61 (4) | C18—Fe1—C17—C21 | 119.1 (7) |
| C5—C6—C8—F6 | −47 (2) | C12—Fe1—C17—C21 | −47.2 (9) |
| C7—C6—C8—F6 | 137 (2) | C19—Fe1—C17—C21 | 81.6 (5) |
| C5—C6—C8—F5 | 81 (2) | C16—Fe1—C17—C21 | 158.9 (7) |
| C7—C6—C8—F5 | −95 (2) | C14—Fe1—C17—C21 | −125.3 (5) |
| C5—C6—C8—F4 | −158.7 (16) | C15—Fe1—C17—C21 | −167.2 (4) |
| C7—C6—C8—F4 | 25.6 (19) | C20—Fe1—C17—C21 | 37.6 (4) |
| C5—C6—C8—F4′ | −3 (3) | C13—Fe1—C17—C21 | −82.9 (5) |
| C7—C6—C8—F4′ | −179 (3) | C12—Fe1—C17—C18 | −166.3 (6) |

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| C11—N2—C9—N1 | −176.6 (4) | C19—Fe1—C17—C18 | −37.5 (5) |
| C11—N2—C9—S1 | −1.0 (7) | C16—Fe1—C17—C18 | 39.8 (10) |
| C4—N1—C9—N2 | −171.2 (5) | C14—Fe1—C17—C18 | 115.7 (5) |
| C4—N1—C9—S1 | 13.0 (8) | C15—Fe1—C17—C18 | 73.8 (6) |
| C9—N2—C11—C12 | 140.7 (5) | C20—Fe1—C17—C18 | −81.5 (5) |
| C9—N2—C11—C10 | −96.2 (6) | C13—Fe1—C17—C18 | 158.1 (5) |
| N2—C11—C12—C16 | 84.9 (5) | C21—Fe1—C17—C18 | −119.1 (7) |
| C10—C11—C12—C16 | −35.5 (7) | C21—C17—C18—C19 | 0.8 (7) |
| N2—C11—C12—C13 | −85.0 (6) | Fe1—C17—C18—C19 | 60.7 (5) |
| C10—C11—C12—C13 | 154.6 (5) | C21—C17—C18—Fe1 | −59.9 (4) |
| N2—C11—C12—Fe1 | 179.1 (4) | C12—Fe1—C18—C19 | 46.2 (11) |
| C10—C11—C12—Fe1 | 58.6 (6) | C16—Fe1—C18—C19 | 76.7 (6) |
| C18—Fe1—C12—C16 | 40.5 (9) | C14—Fe1—C18—C19 | 159.6 (5) |
| C19—Fe1—C12—C16 | 74.8 (4) | C15—Fe1—C18—C19 | 117.7 (5) |
| C14—Fe1—C12—C16 | −81.1 (3) | C17—Fe1—C18—C19 | −118.1 (7) |
| C15—Fe1—C12—C16 | −37.3 (3) | C20—Fe1—C18—C19 | −37.6 (5) |
| C17—Fe1—C12—C16 | −167.0 (7) | C13—Fe1—C18—C19 | −170.2 (5) |
| C20—Fe1—C12—C16 | 116.9 (4) | C21—Fe1—C18—C19 | −80.8 (5) |
| C13—Fe1—C12—C16 | −118.2 (4) | C12—Fe1—C18—C17 | 164.4 (7) |
| C21—Fe1—C12—C16 | 158.9 (4) | C19—Fe1—C18—C17 | 118.1 (7) |
| C18—Fe1—C12—C13 | 158.7 (8) | C16—Fe1—C18—C17 | −165.2 (4) |
| C19—Fe1—C12—C13 | −167.0 (4) | C14—Fe1—C18—C17 | −82.3 (5) |
| C16—Fe1—C12—C13 | 118.2 (4) | C15—Fe1—C18—C17 | −124.2 (5) |
| C14—Fe1—C12—C13 | 37.1 (3) | C20—Fe1—C18—C17 | 80.5 (5) |
| C15—Fe1—C12—C13 | 80.9 (3) | C13—Fe1—C18—C17 | −52.1 (9) |
| C17—Fe1—C12—C13 | −48.8 (8) | C21—Fe1—C18—C17 | 37.3 (4) |
| C20—Fe1—C12—C13 | −124.9 (4) | C17—C18—C19—C20 | −0.4 (8) |
| C21—Fe1—C12—C13 | −82.8 (4) | Fe1—C18—C19—C20 | 60.4 (5) |
| C18—Fe1—C12—C11 | −79.7 (10) | C17—C18—C19—Fe1 | −60.7 (5) |
| C19—Fe1—C12—C11 | −45.4 (6) | C12—Fe1—C19—C18 | −162.7 (4) |
| C16—Fe1—C12—C11 | −120.2 (6) | C16—Fe1—C19—C18 | −121.1 (5) |
| C14—Fe1—C12—C11 | 158.7 (5) | C14—Fe1—C19—C18 | −44.2 (9) |
| C15—Fe1—C12—C11 | −157.5 (5) | C15—Fe1—C19—C18 | −78.5 (6) |
| C17—Fe1—C12—C11 | 72.8 (9) | C17—Fe1—C19—C18 | 38.8 (5) |
| C20—Fe1—C12—C11 | −3.3 (6) | C20—Fe1—C19—C18 | 119.6 (7) |
| C13—Fe1—C12—C11 | 121.6 (6) | C13—Fe1—C19—C18 | 166.1 (7) |
| C21—Fe1—C12—C11 | 38.7 (6) | C21—Fe1—C19—C18 | 82.2 (5) |
| C16—C12—C13—C14 | 2.5 (5) | C18—Fe1—C19—C20 | −119.6 (7) |
| C11—C12—C13—C14 | 173.9 (4) | C12—Fe1—C19—C20 | 77.7 (6) |
| Fe1—C12—C13—C14 | −57.8 (3) | C16—Fe1—C19—C20 | 119.3 (5) |
| C16—C12—C13—P1 | 177.6 (3) | C14—Fe1—C19—C20 | −163.8 (5) |
| C11—C12—C13—P1 | −11.0 (7) | C15—Fe1—C19—C20 | 161.9 (5) |
| Fe1—C12—C13—P1 | 117.4 (3) | C17—Fe1—C19—C20 | −80.8 (5) |
| C16—C12—C13—Fe1 | 60.2 (3) | C13—Fe1—C19—C20 | 46.5 (11) |
| C11—C12—C13—Fe1 | −128.4 (5) | C21—Fe1—C19—C20 | −37.4 (4) |
| C22—P1—C13—C14 | −97.6 (4) | C18—C19—C20—C21 | −0.2 (7) |
| C28—P1—C13—C14 | 7.6 (5) | Fe1—C19—C20—C21 | 59.7 (4) |
| C22—P1—C13—C12 | 88.2 (4) | C18—C19—C20—Fe1 | −59.9 (5) |
| C28—P1—C13—C12 | −166.6 (4) | C18—Fe1—C20—C21 | −82.2 (5) |

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| C22—P1—C13—Fe1 | 174.0 (3) | C12—Fe1—C20—C21 | 118.5 (4) |
| C28—P1—C13—Fe1 | −80.8 (4) | C19—Fe1—C20—C21 | −119.0 (7) |
| C18—Fe1—C13—C14 | −41.9 (7) | C16—Fe1—C20—C21 | 162.1 (4) |
| C12—Fe1—C13—C14 | 120.4 (4) | C14—Fe1—C20—C21 | 36.5 (11) |
| C19—Fe1—C13—C14 | 160.4 (8) | C15—Fe1—C20—C21 | −160.0 (6) |
| C16—Fe1—C13—C14 | 82.0 (3) | C17—Fe1—C20—C21 | −37.7 (4) |
| C15—Fe1—C13—C14 | 38.0 (3) | C13—Fe1—C20—C21 | 76.0 (5) |
| C17—Fe1—C13—C14 | −79.5 (4) | C18—Fe1—C20—C19 | 36.8 (5) |
| C20—Fe1—C13—C14 | −163.5 (4) | C12—Fe1—C20—C19 | −122.4 (5) |
| C21—Fe1—C13—C14 | −122.1 (4) | C16—Fe1—C20—C19 | −78.8 (6) |
| C18—Fe1—C13—C12 | −162.3 (6) | C14—Fe1—C20—C19 | 155.6 (8) |
| C19—Fe1—C13—C12 | 40.0 (10) | C15—Fe1—C20—C19 | −41.0 (9) |
| C16—Fe1—C13—C12 | −38.4 (3) | C17—Fe1—C20—C19 | 81.4 (5) |
| C14—Fe1—C13—C12 | −120.4 (4) | C13—Fe1—C20—C19 | −164.9 (5) |
| C15—Fe1—C13—C12 | −82.4 (3) | C21—Fe1—C20—C19 | 119.0 (7) |
| C17—Fe1—C13—C12 | 160.2 (4) | C19—C20—C21—C17 | 0.7 (7) |
| C20—Fe1—C13—C12 | 76.1 (4) | Fe1—C20—C21—C17 | 59.5 (4) |
| C21—Fe1—C13—C12 | 117.5 (4) | C19—C20—C21—Fe1 | −58.8 (4) |
| C18—Fe1—C13—P1 | 79.6 (7) | C18—C17—C21—C20 | −0.9 (7) |
| C12—Fe1—C13—P1 | −118.1 (5) | Fe1—C17—C21—C20 | −59.7 (4) |
| C19—Fe1—C13—P1 | −78.1 (10) | C18—C17—C21—Fe1 | 58.8 (4) |
| C16—Fe1—C13—P1 | −156.5 (4) | C18—Fe1—C21—C20 | 80.7 (5) |
| C14—Fe1—C13—P1 | 121.6 (5) | C12—Fe1—C21—C20 | −80.7 (5) |
| C15—Fe1—C13—P1 | 159.6 (4) | C19—Fe1—C21—C20 | 37.8 (4) |
| C17—Fe1—C13—P1 | 42.1 (5) | C16—Fe1—C21—C20 | −42.8 (8) |
| C20—Fe1—C13—P1 | −41.9 (5) | C14—Fe1—C21—C20 | −167.6 (4) |
| C21—Fe1—C13—P1 | −0.5 (5) | C15—Fe1—C21—C20 | 153.9 (7) |
| C12—C13—C14—C15 | −2.3 (5) | C17—Fe1—C21—C20 | 118.8 (6) |
| P1—C13—C14—C15 | −177.3 (3) | C13—Fe1—C21—C20 | −124.6 (4) |
| Fe1—C13—C14—C15 | −59.7 (3) | C18—Fe1—C21—C17 | −38.1 (4) |
| C12—C13—C14—Fe1 | 57.4 (3) | C12—Fe1—C21—C17 | 160.5 (4) |
| P1—C13—C14—Fe1 | −117.6 (4) | C19—Fe1—C21—C17 | −81.0 (5) |
| C18—Fe1—C14—C15 | −79.2 (4) | C16—Fe1—C21—C17 | −161.6 (6) |
| C12—Fe1—C14—C15 | 81.5 (3) | C14—Fe1—C21—C17 | 73.5 (5) |
| C19—Fe1—C14—C15 | −48.4 (7) | C15—Fe1—C21—C17 | 35.1 (10) |
| C16—Fe1—C14—C15 | 37.4 (3) | C20—Fe1—C21—C17 | −118.8 (6) |
| C17—Fe1—C14—C15 | −122.1 (4) | C13—Fe1—C21—C17 | 116.6 (5) |
| C20—Fe1—C14—C15 | 169.7 (8) | C13—P1—C22—C23 | 11.6 (5) |
| C13—Fe1—C14—C15 | 118.8 (4) | C28—P1—C22—C23 | −91.3 (5) |
| C21—Fe1—C14—C15 | −162.3 (4) | C13—P1—C22—C27 | −166.5 (4) |
| C18—Fe1—C14—C13 | 162.0 (4) | C28—P1—C22—C27 | 90.7 (5) |
| C12—Fe1—C14—C13 | −37.3 (3) | C27—C22—C23—C24 | 0.0 (9) |
| C19—Fe1—C14—C13 | −167.2 (6) | P1—C22—C23—C24 | −178.1 (4) |
| C16—Fe1—C14—C13 | −81.4 (3) | C22—C23—C24—C25 | 0.3 (9) |
| C15—Fe1—C14—C13 | −118.8 (4) | C23—C24—C25—C26 | −0.9 (11) |
| C17—Fe1—C14—C13 | 119.1 (4) | C24—C25—C26—C27 | 1.1 (12) |
| C20—Fe1—C14—C13 | 50.9 (10) | C25—C26—C27—C22 | −0.7 (11) |
| C21—Fe1—C14—C13 | 78.9 (4) | C23—C22—C27—C26 | 0.2 (9) |
| C13—C14—C15—C16 | 1.3 (5) | P1—C22—C27—C26 | 178.4 (5) |

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| Fe1—C14—C15—C16 | −58.7 (3) | C13—P1—C28—C33 | −80.4 (5) |
| C13—C14—C15—Fe1 | 60.0 (3) | C22—P1—C28—C33 | 23.1 (6) |
| C18—Fe1—C15—C16 | −122.5 (4) | C13—P1—C28—C29 | 97.9 (5) |
| C12—Fe1—C15—C16 | 37.5 (3) | C22—P1—C28—C29 | −158.6 (5) |
| C19—Fe1—C15—C16 | −82.2 (4) | C33—C28—C29—C30 | 2.6 (10) |
| C14—Fe1—C15—C16 | 119.8 (4) | P1—C28—C29—C30 | −175.7 (5) |
| C17—Fe1—C15—C16 | −163.3 (4) | C28—C29—C30—C31 | −1.9 (11) |
| C20—Fe1—C15—C16 | −52.9 (7) | C29—C30—C31—C32 | −1.5 (14) |
| C13—Fe1—C15—C16 | 81.7 (3) | C30—C31—C32—C33 | 3.9 (13) |
| C21—Fe1—C15—C16 | 170.3 (7) | C29—C28—C33—C32 | −0.2 (10) |
| C18—Fe1—C15—C14 | 117.7 (4) | P1—C28—C33—C32 | 178.1 (5) |
| C12—Fe1—C15—C14 | −82.3 (3) | C31—C32—C33—C28 | −3.1 (11) |
| C19—Fe1—C15—C14 | 158.1 (4) | | |

*Hydrogen-bond geometry (Å, º)*

| D—H···A | D—H | H···A | D···A | D—H···A |
|---|---|---|---|---|
| N1—H1···S1[i] | 0.86 | 2.46 | 3.302 (5) | 168 |
| N2—H2···S1[i] | 0.86 | 2.64 | 3.445 (4) | 157 |

Symmetry code: (i) −*y*+3/2, *x*−1/2, *z*−1/4.