Synthesis, crystal structure and Hirshfeld surface analysis of 1-ferrocenylnundecane-1,11-diol

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The racemic title compound, [Fe(C₅H₅)(C₁₆H₂₇O₂)], comprises an α,ω-diol-substituted undecyl chain with a ferrocenyl substituent at at one terminus. The alkane chain is inclined to the substituted ring of the ferrocene grouping by 84.22 (13)°. The ferrocene rings are almost eclipsed and parallel. The crystal structure features O—H···O and C—H···O hydrogen bonds and C—H···π contacts that stack the molecules along the c-axis direction. A Hirshfeld surface analysis reveals that H···H interactions (83.2%) dominate the surface contacts.

1. Chemical context

The title compound, 1, is a rare example of a ferrocene molecule substituted with an extended, in this instance 11-membered, alkane chain. It was synthesized to provide a ferrocenyl-substituted diol for the preparation of polyesters with regular pendant electroactive groups. Similar ferrocenyl neo-pentyl diol-derived terephthalate polymers have been shown to display interesting electrochemical properties (McAdam et al., 2008a,b). Friedel–Crafts methodology (Saji et al., 1991) provided the 1-ferrocenylnundec-10-en-1-ol precursor. This was reduced to the racemic alcohol 1-ferrocenylnundec-10-en-1-ol (2) using LiAlH₄. Enantiomeric selection of the individual chiral forms should be possible using more complex synthetic methodology (Ursini et al., 2006; Schwink et al., 1998), but was deemed unnecessary for our purposes. Hydroboration of ferrocenylnalkenes has been previously reported (Lo Sterzo et al., 1984) using borane generated in situ from NaBH₄/BF₃·OEt₂. Predictably, this method was unsuitable as a means of preparing 1 from 2, the ferrocenylmethanol moiety being susceptible to attack by BF₃, and the resultant loss of OH− abetted by the formation of the stable α-ferrocenyl carbenium ion. This prediction was borne out by experiment, the Lewis acid attack resulting in synthesis of 1-ferrocenylnundec-10-ene and 1-ferrocenylnundec-11-ol. Instead, a successful synthesis of 1 was achieved using hydroboration of 2 with 9-BBN.
2. Structural commentary

The title compound, \([\text{Fe}(\text{C}_5\text{H}_5)(\text{C}_{16}\text{H}_{27}\text{O}_2)]\), comprises a ferrocene unit that carries a well-ordered undecane chain (atoms C11–C21) with hydroxyl substituents at the 1 and 11 positions along the chain (Fig. 1). The C13—C12—C11—O11 and C19—C20—C21—O21 torsion angles are 60.9 (3)° and 173.9 (2)°, respectively. Atom C11 is a stereogenic centre: in the arbitrarily chosen asymmetric molecule it has an \(R\) configuration, but crystal symmetry generates a racemic mixture. The alkane chain is almost planar with the r.m.s. deviation from the best fit plane through all 11 C atoms being 0.129 Å. This plane is nearly orthogonal to the substituted ferrocene ring with an angle of 84.22 (13)° between them. The C11 undecyl chain in \(\text{I}\) is conformationally extended with the typical antiperiplanar (Kane & Hersh, 2000) arrangement for \(\text{C}_n\text{–C}_{n+3}\) groupings and a C11•••C21 separation of 12.627 (4) Å. The C1–C5 and C6–C10 cyclopentadienyl rings of the ferrocenyl group are approximately 3° from being eclipsed and are almost coplanar with a dihedral angle of 1.7 (2)° between them; the separation of the ring centroids is is 3.298 (2) Å.

3. Supramolecular features

In the crystal of \(\text{I}\), inversion dimers form in the \(ab\) plane through pairwise classical O21•••H21•••O11 hydrogen bonds (Table 1), which generate \(R_2^2(28)\) ring motifs (Fig. 2). Additional classical O11•••H11•••O21 hydrogen bonds, supported by weaker non-classical C6•••H6•••O21 contacts, form alternating chains of molecules along the \(b\)-axis direction and O21 acts as a double acceptor (Fig. 3). A weak C7•••H7•••Cg2 (H•••π = 2.89 Å, C–H•••π = 164°) contact involving the unsubstituted ring of the ferrocene unit forms double chains of molecules propagating along the \(c\)-axis direction (Fig. 4).
where \( C_{g2} \) is the centroid of the C6–C10 cyclopentadienyl ring. Overall these various contacts combine to stack the molecules of 1 along the \( c \)-axis direction in two discrete, parallel and well-separated columns (Fig. 5).

4. Hirshfeld surface analysis

Further details of the intermolecular interactions in 1 were obtained using Hirshfeld surface analysis (Spackman & Jayatilaka, 2009) with Hirshfeld surfaces and two-dimensional fingerprint plots generated with Crystal Explorer (Turner et al., 2017). Hirshfeld surfaces for opposite faces of 1 are shown in Fig. 6(a) and (b). Bold red areas on the Hirshfeld surfaces correspond to the classical O—H···O hydrogen bonds while the weaker C—H···O and C—H···π contacts appear as faint red circles. Fingerprint plots (Fig. 7) reveal that H···H interactions dominate the surface contacts, as would be expected for a molecule with such a predominance of H atoms, with H···C/C···H and H···O/O···H contacts also making significant contributions to the surface (Table 2).

5. Database survey

Ferrocene derivatives with pendant C\(_n\) alkyl chains (\( n \geq 11 \)) are uncommon and the majority of such structures that appear in the Cambridge Structural Database (version 5.41 Nov 2019 with updates to March 2020; Groom et al., 2016) are bis-ferrocenyl complexes. These include 1,12-bis-ferrocenyldodecane (refcodes FOHAM and FOHAM01; Bequeath et al., 2005, Wedeking et al., 2006a) and the tetradecane, octadecane and docosane derivatives (VEFXIO, VEFXOU, VEFXUA; Wedeking et al., 2006a).

### Table 1

| D—H··A | D—H | H··A | D··A | D—H··A |
|--------|-----|------|------|--------|
| O11—H11···O21\(^i\) | 0.76 | 2.06 | 2.755 (3) | 152 |
| O21—H21···O11\(^ii\) | 0.83 | 1.90 | 2.726 (3) | 175 |
| C6—H6···O21\(^i\) | 0.95 | 2.60 | 3.380 (4) | 140 |

Symmetry codes: (i) \(-x+\frac{1}{2}, y-\frac{1}{2}, z+\frac{1}{2}\); (ii) \(-x+\frac{1}{2}, y+\frac{1}{2}, z+2\).

### Table 2

| Contents | Included surface area |
|----------|-----------------------|
| H···H | 83.2 |
| H···C/C···H | 9.4 |
| H···O/O···H | 7.3 |

Figure 5
Overall packing of 1 viewed along the \( c \)-axis direction.

Figure 6
Hirshfeld surfaces for opposite faces of 1 mapped over \( d_{norm} \) in the range −0.67 to 1.35 a.u.

Figure 7
A full two-dimensional fingerprint plot for 1, (a), together with (b)–(d) separate principal contact types for the molecule: H···H, H···C/C···H and H···O/O···H, respectively.
report of the structure of 11-bromo-1-ferrocenylundecan-1-one (LICIV; McAdam et al., 2007) is the sole example of such a structure with substitution on the alkane chain. Interestingly, the structure of the related 1,11-undecanediol (HIYHAY; Nakamura et al., 1999) has also been reported. However, \(\alpha,\omega\)-dihydroxyalkane \((C_n, n \geq 10)\) structures are uncommon and often crystallize as co-crystals, see, for example, KEXZOD and KEXZJU (Loehlin et al., 2011).

6. Synthesis and crystallization

The title compound \(1\) was prepared in two steps from 1-ferrocenyl-undec-10-en-1-one (Evans et al., 2008) via a lithium aluminium hydride reduction followed by hydroboration with 9-borabicyclo[3.3.1]nonane (9-BBN) (Arístoff et al., 1985), Fig. 8. \(\text{LiAlH}_4\) \((0.10 \text{ g}, 2.6 \text{ mmol})\) was added to 1-ferrocenyl-undec-10-en-1-one \((0.615 \text{ g}, 1.75 \text{ mmol})\) in \(\text{Et}_2\text{O}\) \((10 \text{ mL})\) at 273 K and stirred for 1 h before quenching with a few drops of water. The ether fraction was rinsed with saturated \(\text{NaCl}\) solution and dried over \(\text{MgSO}_4\). The solvent was removed under vacuum to give 0.61 g (99%) of the yellow oil as a yellow solid \((0.60 \text{ g}, 94\%)\). X-ray quality crystals were grown from the mixed solvents of \(\text{CH}_2\text{Cl}_2\) layered with hexane. Analysis calculated for \(\text{C}_{21}\text{H}_{32}\text{O}_2\text{Fe}: C, 67.74; H, 8.66. Found: C, 67.94; H, 8.92%.^{1}\text{H NMR (CDCl}_3\): 4.30 \((m, 1\text{H}, –\text{CHOH}–\)), 4.24 \((m, 1\text{H}, \text{C}_5\text{H}_4\)), 4.20 \((\delta, 5\text{H}, \text{Cp}), 4.17 \((m, 3\text{H}, \text{C}_6\text{H}_4\)), 3.64 \((m, 2\text{H}, –\text{CH}_2–\text{OH})\), 1.92 \([d (J = 4 \text{ Hz}), 1\text{H, Fe–\text{CHOH}}]\), 1.7–1.3 \([m, 18\text{H}, –(\text{CH}_2)_{17}–]\).^{13}\text{C NMR (CDCl}_3\): 94.7 \((\text{Fe ipso})\), 69.7 \((–\text{CHOH}–\), 68.3 \((\text{Cp})\), 67.9, 67.7, 67.3, 65.2 \((\text{Fe–C} \& \beta, \beta)\), 63.2 \((–\text{CH}_2\text{OH})\), 38.3, 32.9, 29.6, 29.6, 29.5, 29.5, 29.5, 26.1, 25.8 \((–\text{CH}_2–\). UV–vis \((\text{CH}_2\text{Cl}_2\): 325 \((90\%\)), 440 \((110\%\)) nm \((\epsilon)\).

7. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 3. The O-bound H atoms were located in a difference-Fourier map and their coordinates refined with \(U_{	ext{iso}}(H) = 1.5 U_{	ext{eq}}(O)\). All H-atoms bound to C and O were treated by a mixture of independent and constrained refinement with \(U_{	ext{iso}}(H) = 1.5 U_{	ext{eq}}(O)\) and \(U_{	ext{iso}}(H) = 1.2 U_{	ext{eq}}(C)\). Despite repeated attempts to grow crystals of better quality, the crystals obtained were weakly diffracting and the extent of diffraction observed is poor with sin \((\theta_{\text{max}})/\lambda = 0.544 (2\theta_{\text{max}} = 44.5^\circ)\). Despite this, the structure solved and refined adequately.

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Computing details
Data collection: APEX2 (Bruker, 2011); cell refinement: APEX2 (Bruker, 2011) and SAINT (Bruker, 2011); data reduction: SAINT (Bruker, 2011); program(s) used to solve structure: SHELXT (Sheldrick, 2015a); program(s) used to refine structure: SHELXL2018/1 (Sheldrick, 2015b) and TITAN (Hunter & Simpson, 1999); molecular graphics: Mercury (Macrae et al., 2020); software used to prepare material for publication: SHELXL2018/1 (Sheldrick, 2015b), enCIFer (Allen et al., 2004), PLATON (Spek, 2020), publCIF (Westrip 2010) and WinGX (Farrugia 2012).

1-Ferrocenylundecane-1,11-diol

Crystal data
[Fe(C5H5)(C16H27O2]
M_r = 372.31
Monoclinic, C2/c
a = 47.641 (3) Å
b = 10.1522 (7) Å
c = 7.8747 (6) Å
β = 97.091 (4)°
V = 3779.6 (5) Å³
Z = 8

F(000) = 1600
D_x = 1.309 Mg m⁻³
Mo Kα radiation, λ = 0.71073 Å
Cell parameters from 4218 reflections
θ = 2.4–22.4°
μ = 0.81 mm⁻¹
T = 92 K
Plate, yellow
0.32 × 0.14 × 0.04 mm

Data collection
CCD area detector
diffractometer
Radiation source: sealed tube
Graphite monochromator
phi and ω scans
Absorption correction: multi-scan
(SADABS; Bruker, 2011)
T_min = 0.784, T_max = 1.000

16666 measured reflections
2527 independent reflections
2150 reflections with I > 2σ(I)
R_int = 0.049
θ_max = 22.7°, θ_min = 1.7°
h = −51→51
k = −11→11
l = −8→7

Refinement
Refinement on F²
Least-squares matrix: full
R[F² > 2σ(F²)] = 0.037
wR(F²) = 0.106
S = 1.06
2527 reflections
221 parameters
0 restraints

Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement
w = 1/[σ²(F²) + (0.0649P)² + 3.6265P]
where P = (F² + 2F©²)/3
(Δ/σ)max = 0.001
Δρ_max = 0.64 e Å⁻³
Δρ_min = −0.31 e Å⁻³
Special details

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

Refinement. A reflection effected by the beamstop and two reflections with Fo >>> Fc were omitted from the final refinement cycles.

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

| Atom | x      | y      | z      | Uiso*/*Ueq |
|------|--------|--------|--------|------------|
| C1   | 0.08497 (6) | 0.8323 (3) | 0.3254 (4) | 0.0242 (7) |
| C2   | 0.06289 (6) | 0.7864 (3) | 0.4173 (4) | 0.0249 (7) |
| H2   | 0.064664 | 0.719397 | 0.502125 | 0.030*      |
| C3   | 0.03794 (7) | 0.8572 (3) | 0.3612 (4) | 0.0281 (7) |
| H3   | 0.020049 | 0.845762 | 0.400751 | 0.034*      |
| C4   | 0.04438 (6) | 0.9489 (3) | 0.2348 (4) | 0.0263 (7) |
| H4   | 0.031523 | 1.009645 | 0.175642 | 0.032*      |
| C5   | 0.07318 (6) | 0.9340 (3) | 0.2122 (4) | 0.0253 (7) |
| H5   | 0.083009 | 0.982862 | 0.135301 | 0.030*      |
| Fe1  | 0.05212 (2) | 0.76105 (4) | 0.16036 (5) | 0.02107 (19) |
| C6   | 0.06708 (7) | 0.6085 (3) | 0.0268 (4) | 0.0304 (8) |
| H6   | 0.085748 | 0.573937 | 0.042214 | 0.036*      |
| C7   | 0.04426 (7) | 0.5650 (3) | 0.1116 (4) | 0.0302 (8) |
| H7   | 0.044913 | 0.496850 | 0.194681 | 0.036*      |
| C8   | 0.02022 (7) | 0.6416 (3) | 0.0500 (4) | 0.0331 (8) |
| H8   | 0.001833 | 0.633157 | 0.083682 | 0.040*     |
| C9   | 0.02838 (7) | 0.7320 (3) | −0.0694 (4) | 0.0336 (8) |
| H9   | 0.016408 | 0.795724 | −0.129627 | 0.040*      |
| C10  | 0.05726 (7) | 0.7129 (3) | −0.0855 (4) | 0.0321 (8) |
| H10  | 0.068119 | 0.760822 | −0.157882 | 0.039*      |
| C11  | 0.11527 (6) | 0.7882 (3) | 0.3455 (4) | 0.0274 (7) |
| H11A | 0.122055 | 0.786316 | 0.230314 | 0.033*      |
| O11  | 0.11807 (5) | 0.65849 (18) | 0.4193 (3) | 0.0298 (5) |
| H11  | 0.1142 (7) | 0.607 (2) | 0.350 (3) | 0.045*      |
| C12  | 0.13452 (6) | 0.8767 (3) | 0.4624 (4) | 0.0298 (7) |
| H12A | 0.128323 | 0.875560 | 0.577879 | 0.036*      |
| H12B | 0.132689 | 0.968142 | 0.418890 | 0.036*      |
| C13  | 0.16570 (6) | 0.8354 (3) | 0.4768 (4) | 0.0313 (8) |
| H13A | 0.166977 | 0.739640 | 0.500383 | 0.038*      |
| H13B | 0.172652 | 0.850703 | 0.365045 | 0.038*      |
| C14  | 0.18500 (6) | 0.9069 (3) | 0.6140 (4) | 0.0293 (7) |
| H14A | 0.176768 | 0.902415 | 0.723312 | 0.035*      |
| H14B | 0.186169 | 1.000796 | 0.581914 | 0.035*      |
| C15  | 0.21465 (6) | 0.8487 (3) | 0.6398 (4) | 0.0286 (7) |
| H15A | 0.213202 | 0.753762 | 0.665954 | 0.034*      |
| H15B | 0.222958 | 0.856302 | 0.531020 | 0.034*      |
| C16  | 0.23466 (6) | 0.9126 (3) | 0.7808 (4) | 0.0283 (7) |
supporting information

|          | $U_{11}$    | $U_{22}$    | $U_{33}$    | $U_{12}$    | $U_{13}$    | $U_{23}$    |
|----------|-------------|-------------|-------------|-------------|-------------|-------------|
| C1       | 0.0313 (17) | 0.0143 (15) | 0.0256 (17) | -0.0044 (12)| -0.0020 (13)| -0.0012 (13)|
| C2       | 0.0363 (18) | 0.0187 (15) | 0.0187 (16) | -0.0042 (13)| -0.0007 (13)| -0.0049 (13)|
| C3       | 0.0297 (17) | 0.0256 (17) | 0.0296 (17) | -0.0016 (13)| 0.0061 (14) | -0.0079 (14)|
| C4       | 0.0295 (17) | 0.0178 (16) | 0.0300 (17) | 0.0025 (12) | -0.0024 (13)| -0.0033 (13)|
| C5       | 0.0306 (17) | 0.0154 (15) | 0.0290 (17) | -0.0028 (12)| 0.0000 (13) | 0.0001 (13)|
| Fe1      | 0.0263 (3)  | 0.0149 (3)  | 0.0213 (3)  | -0.00056 (17)| -0.00010 (19)| -0.00066 (17)|
| C6       | 0.0358 (19) | 0.0259 (17) | 0.0280 (18) | 0.0068 (14) | -0.0018 (14)| -0.0080 (14)|
| C7       | 0.048 (2)   | 0.0149 (15) | 0.0263 (17) | -0.0020 (14)| 0.0010 (15) | -0.0038 (13)|
| C8       | 0.0325 (18) | 0.0266 (17) | 0.039 (2)   | -0.0059 (14)| -0.0010 (15)| -0.0088 (15)|
| C9       | 0.042 (2)   | 0.0236 (17) | 0.0314 (19) | 0.0036 (14) | -0.0105 (15)| -0.0024 (14)|
| C10      | 0.047 (2)   | 0.0248 (17) | 0.0244 (18) | -0.0047 (15)| 0.0044 (15) | -0.0020 (14)|
| C11      | 0.0305 (17) | 0.0197 (16) | 0.0306 (18) | -0.0005 (13)| -0.0014 (14)| 0.0052 (14)|
| O11      | 0.0372 (13) | 0.0157 (11) | 0.0339 (13) | 0.0004 (9)  | -0.0064 (10)| -0.0017 (9)|
| C12      | 0.0339 (18) | 0.0202 (16) | 0.0345 (19) | -0.0016 (13)| 0.0017 (14) | -0.0006 (14)|
| C13      | 0.0307 (18) | 0.0221 (17) | 0.041 (2)   | 0.0001 (13) | 0.0043 (15) | 0.0014 (14)|
| C14      | 0.0302 (18) | 0.0221 (16) | 0.0361 (19) | -0.0037 (13)| 0.0059 (14) | 0.0018 (14)|
| C15      | 0.0317 (18) | 0.0193 (16) | 0.0355 (19) | -0.0017 (13)| 0.0063 (14) | 0.0031 (13)|
| C16      | 0.0351 (18) | 0.0187 (16) | 0.0325 (18) | -0.0032 (13)| 0.0092 (14) | 0.0017 (13)|
| C17      | 0.0324 (18) | 0.0211 (16) | 0.0304 (18) | -0.0026 (13)| 0.0071 (14) | 0.0017 (13)|
| C18      | 0.0337 (18) | 0.0216 (16) | 0.0307 (18) | -0.0056 (13)| 0.0078 (14) | -0.0019 (14)|
| C19      | 0.0339 (18) | 0.0199 (16) | 0.0326 (18) | -0.0067 (13)| 0.0061 (14) | 0.0001 (13)|
| C20      | 0.0356 (18) | 0.0214 (16) | 0.0261 (17) | -0.0065 (13)| 0.0060 (14) | -0.0011 (13)|
| C21      | 0.0349 (19) | 0.0244 (17) | 0.0321 (18) | -0.0067 (13)| 0.0018 (15) | -0.0015 (14)|
| O21      | 0.0407 (13) | 0.0284 (12) | 0.0299 (12) | -0.0113 (10)| -0.0041 (10)| 0.0033 (10)|
### Geometric parameters (Å, °)

| Bond/Distance | Value         |
|---------------|---------------|
| C1—C2         | 1.427 (4)     |
| C1—C5         | 1.432 (4)     |
| C1—C11        | 1.501 (4)     |
| C1—Fe1        | 2.040 (3)     |
| C2—C3         | 1.413 (4)     |
| C2—Fe1        | 2.041 (3)     |
| C2—H2         | 0.9500        |
| C3—C4         | 1.423 (4)     |
| C3—Fe1        | 2.043 (3)     |
| C3—H3         | 0.9500        |
| C4—C5         | 1.413 (4)     |
| C4—Fe1        | 2.042 (3)     |
| C4—H4         | 0.9500        |
| C5—Fe1        | 2.038 (3)     |
| Fe1—C9        | 2.033 (3)     |
| Fe1—C10       | 2.041 (3)     |
| Fe1—C6        | 2.049 (3)     |
| C6—H6         | 0.9500        |
| C7—C8         | 1.420 (4)     |
| C7—H7         | 0.9500        |
| C8—C9         | 1.402 (5)     |
| C8—H8         | 0.9500        |
| C9—C10        | 1.410 (5)     |
| C9—H9         | 0.9500        |
| C10—H10       | 0.9500        |
| C11—O11       | 1.439 (3)     |
| C11—C12       | 1.512 (4)     |
| C11—H11A      | 1.0000        |
| C2—C1—C5      | 107.1 (3)     |
| C2—C1—C11     | 127.5 (3)     |
| C5—C1—C11     | 125.4 (3)     |
| C2—C1—Fe1     | 69.60 (16)    |
| C5—C1—Fe1     | 69.37 (16)    |
| C11—C1—Fe1    | 127.9 (2)     |
| C3—C2—C1      | 108.7 (3)     |
| C3—C2—Fe1     | 69.85 (17)    |
| C1—C2—Fe1     | 69.47 (16)    |
| C3—C2—H2      | 125.7         |
| C1—C2—H2      | 125.7         |
| Fe1—C2—H2     | 126.6         |

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C2—C3—C4 107.8 (3) C9—C10—H10 126.4
C2—C3—Fe1 69.69 (17) C6—C10—H10 126.4
C4—C3—Fe1 69.54 (16) Fe1—C10—H10 125.8
C2—C3—H3 126.1 O11—C11—C1 110.8 (2)
C4—C3—H3 126.1 O11—C11—C12 106.2 (2)
Fe1—C3—H3 126.2 C1—C11—C12 113.0 (2)
C5—C4—C3 108.3 (3) O11—C11—C21 79.36 (14)
C5—C4—Fe1 69.60 (16) C1—C11—C21 149.62 (17)
C3—C4—Fe1 69.67 (16) O11—C11—H11A 108.9
C5—C4—H4 125.8 C1—C11—H11A 108.9
C3—C4—H4 125.8 C12—C11—H11A 108.9
Fe1—C4—H4 126.5 C21—C11—H11A 93.4
C4—C5—C1 108.1 (3) C11—O11—H11 109.5
C4—C5—Fe1 69.87 (16) C11—C12—C13 113.0 (3)
C1—C5—Fe1 69.49 (16) C11—C12—H12A 109.0
C4—C5—H5 125.9 C13—C12—H12A 109.0
C1—C5—H5 125.9 C11—C12—H12B 109.0
Fe1—C5—H5 126.3 C13—C12—H12B 109.0
C9—Fe1—C5 120.61 (12) H12A—C12—H12B 107.8
C9—Fe1—C1 156.53 (13) C14—C13—C12 114.8 (3)
C5—Fe1—C1 41.13 (11) C14—C13—H13A 108.6
C9—Fe1—C10 40.51 (13) C12—C13—H13A 108.6
C5—Fe1—C10 106.43 (12) C14—C13—H13B 108.6
C1—Fe1—C10 121.14 (12) C12—C13—H13B 108.6
C9—Fe1—C2 160.79 (14) H13A—C13—H13B 107.6
C5—Fe1—C2 68.63 (12) C13—C14—C15 112.4 (2)
C1—Fe1—C2 40.93 (11) C13—C14—H14A 109.1
C10—Fe1—C2 157.79 (13) C15—C14—H14A 109.1
C9—Fe1—C4 106.89 (12) C13—C14—H14B 109.1
C5—Fe1—C4 40.53 (11) C15—C14—H14B 109.1
C1—Fe1—C4 68.75 (11) H14A—C14—H14B 107.9
C10—Fe1—C4 122.90 (12) C16—C15—C14 114.9 (2)
C2—Fe1—C4 68.28 (12) C16—C15—H15A 108.5
C9—Fe1—C3 123.86 (13) C14—C15—H15A 108.5
C5—Fe1—C3 68.59 (12) C16—C15—H15B 108.5
C1—Fe1—C3 68.80 (12) C14—C15—H15B 108.5
C10—Fe1—C3 159.82 (13) H15A—C15—H15B 107.5
C2—Fe1—C3 40.46 (12) C15—C16—C17 113.8 (2)
C4—Fe1—C3 40.79 (12) C15—C16—H16A 108.8
C9—Fe1—C6 67.91 (12) C17—C16—H16A 108.8
C5—Fe1—C6 124.07 (12) C15—C16—H16B 108.8
C1—Fe1—C6 107.89 (12) C17—C16—H16B 108.8
C10—Fe1—C6 40.69 (12) H16A—C16—H16B 107.7
C2—Fe1—C6 122.93 (12) C18—C17—C16 114.3 (2)
C4—Fe1—C6 160.03 (12) C18—C17—H17A 108.7
C3—Fe1—C6 158.10 (12) C16—C17—H17A 108.7
C9—Fe1—C8 40.16 (13) C18—C17—H17B 108.7
C5—Fe1—C8 156.13 (12) C16—C17—H17B 108.7
C1—Fe1—C8 161.73 (12) H17A—C17—H17B 107.6
C10—Fe1—C8 68.02 (13) C19—C18—C17 113.9 (2)
C2—Fe1—C8 125.22 (13) C19—C18—H18A 108.8
C4—Fe1—C8 121.53 (12) C17—C18—H18A 108.8
C3—Fe1—C8 108.17 (13) C19—C18—H18B 108.8
C6—Fe1—C8 67.80 (13) C17—C18—H18B 108.8
C9—Fe1—C7 67.93 (12) H18A—C18—H18B 107.7
C5—Fe1—C7 161.19 (12) C20—C19—C18 113.7 (2)
C1—Fe1—C7 124.82 (12) C20—C19—H19A 108.8
C10—Fe1—C7 68.32 (12) C18—C19—H19A 108.8
C2—Fe1—C7 109.00 (12) C20—C19—H19B 108.8
C4—Fe1—C7 157.52 (13) C18—C19—H19B 108.8
C3—Fe1—C7 122.55 (12) H19A—C19—H19B 107.7
C6—Fe1—C7 40.36 (12) C21—C20—C19 112.8 (2)
C8—Fe1—C7 40.47 (12) C21—C20—H20A 109.0
C7—C6—C10 108.3 (3) C19—C20—H20A 109.0
C7—C6—Fe1 69.98 (16) C21—C20—H20B 109.0
C10—C6—Fe1 69.35 (17) C19—C20—H20B 109.0
C7—C6—H6 125.9 H20A—C20—H20B 107.8
C10—C6—H6 125.9 O21—C21—C20 113.8 (2)
Fe1—C6—H6 126.4 O21—C21—C11 148.76 (17)
C6—C7—C8 107.6 (3) O21—C21—H21A 108.8
C6—C7—Fe1 69.66 (17) C20—C21—H21A 108.8
C8—C7—Fe1 69.73 (17) C11—C21—H21A 91.3
C6—C7—H7 126.2 O21—C21—H21B 108.8
C8—C7—H7 126.2 C20—C21—H21B 108.8
Fe1—C7—H7 126.0 C11—C21—H21B 86.3
C9—C8—C7 108.0 (3) H21A—C21—H21B 107.7
C9—C8—Fe1 69.21 (18) C21—O21—H21 109.2
C7—C8—Fe1 69.81 (17)

Hydrogen-bond geometry (Å, °)

\[D—H···A\n\[D—H\]
\[H···A\n\[D···A\n\[D—H···A\n
| D—H···A | D—H | H···A | D···A | D—H···A |
|---------|------|-------|-------|---------|
| O11—H11···O21i | 0.76 | 2.06 | 2.755 (3) | 152 |
| O21—H21···O11ii | 0.83 | 1.90 | 2.726 (3) | 175 |
| C6—H6···O21i | 0.95 | 2.60 | 3.380 (4) | 140 |

Symmetry codes: (i) −x+1/2, y−1/2, −z+3/2; (ii) −x+1/2, −y+3/2, −z+2.