

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

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Received 22 December 2021

Accepted 23 December 2021

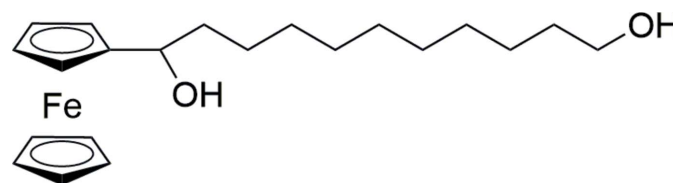
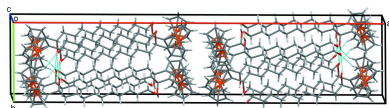
Edited by W. T. A. Harrison, University of Aberdeen, Scotland

Keywords: crystal structure; ferrocene; undecane-1,11-diol; hydrogen bonds; C—H... π contact; Hirshfeld surface analysis.**CCDC reference:** 2130725**Supporting information:** this article has supporting information at journals.iucr.org/e

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.*, 2008*a,b*). Friedel–Crafts methodology (Saji *et al.*, 1991) provided the 1-ferrocenyl-undec-10-en-1-one precursor. This was reduced to the racemic alcohol 1-ferrocenyl-undec-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 ferrocenylalkenes 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-ferrocenyl-undec-10-ene and 1-ferrocenyl-undec-11-ol. Instead, a successful synthesis of **1** was achieved using hydroboration of **2** with 9-BBN.



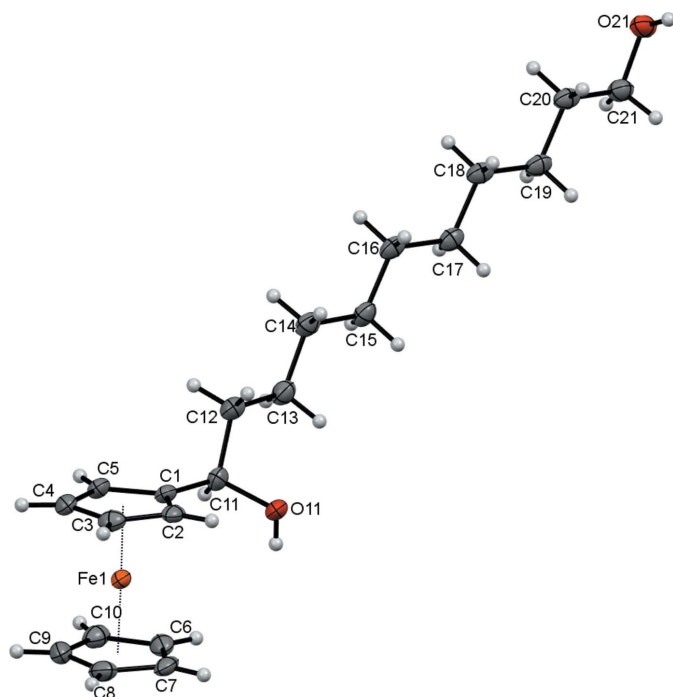


Figure 1
The molecular structure of **1** with ellipsoids drawn at the 50% probability level.

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 **1** is conformationally extended with the typical antiperiplanar (Kane & Hersh, 2000) arrangement for

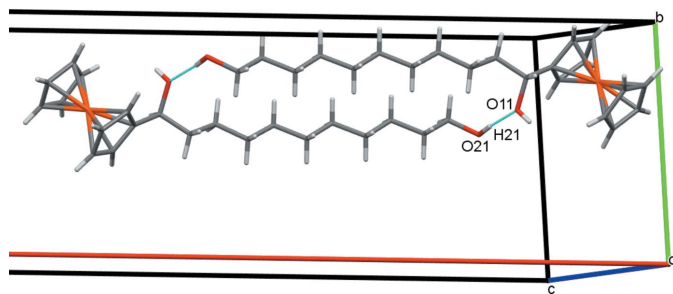


Figure 2
Inversion dimers of **1** in the *ab* plane with O–H···O hydrogen bonds shown as blue lines.

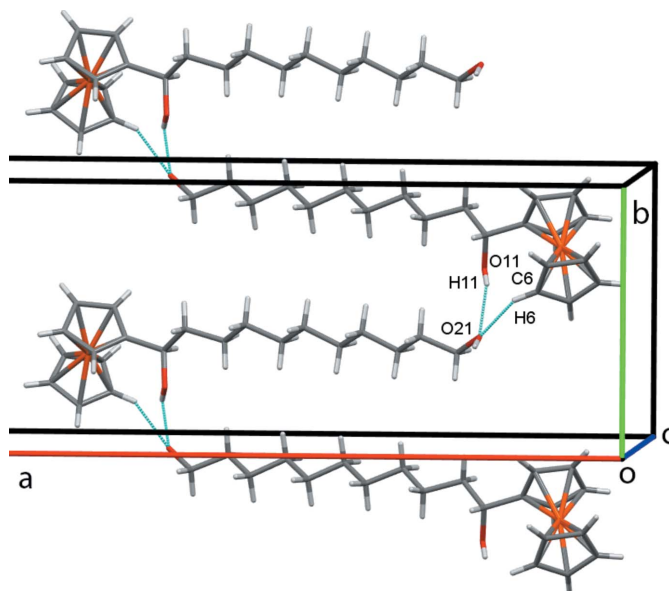


Figure 3
Chains of molecules of **1** propagating along *b* with O–H···O and C–H···O hydrogen bonds shown as blue lines.

$\text{C}_n\text{--C}_{n+3}$ 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 3.298 (2) Å.

3. Supramolecular features

In the crystal of **1**, 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)

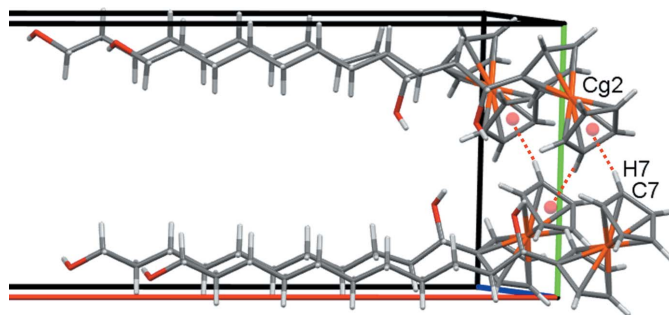


Figure 4
Double chains of molecules of **1** along *c*. Cg2 is the centroid of the C6–C10 cyclopentadienyl ring, shown here as red spheres, with the C–H··· π contacts drawn as dashed red lines.

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

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

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

Table 2
 Percentage contributions to the Hirshfeld surface of **1**.

Contents	Included surface area
H...H	83.2
H...C/C...H	9.4
H...O/O...H	7.3

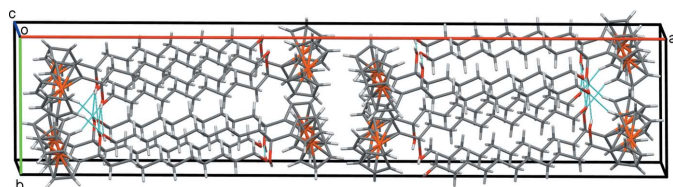
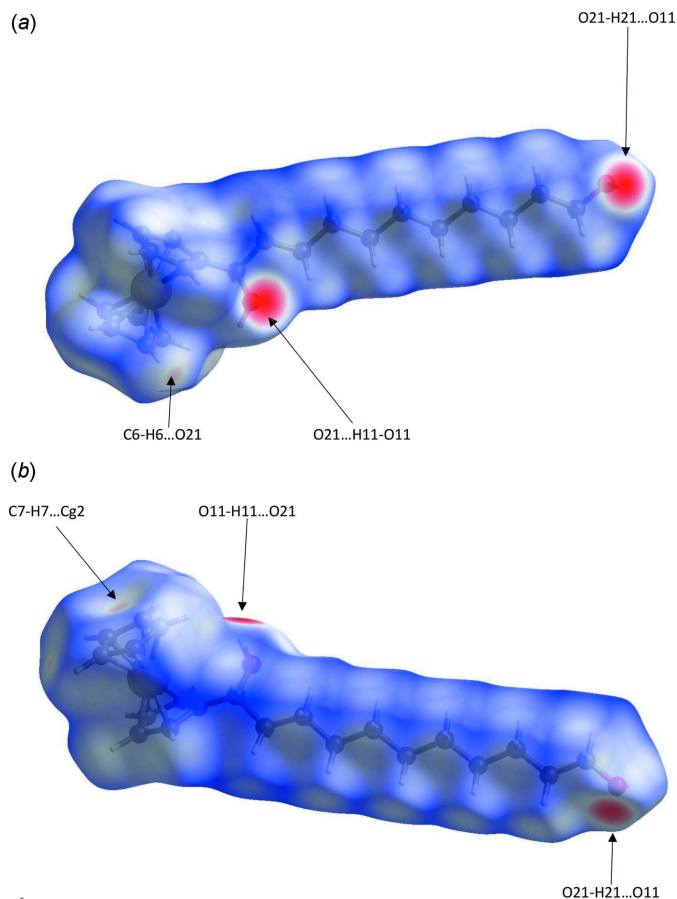
where Cg2 is the centroid of the C6–C10 cyclopentadienyl ring. Overall these various contacts combine to stack the molecules of **1** along the c -axis direction in two discrete, parallel and well-separated columns (Fig. 5).

4. Hirshfeld surface analysis

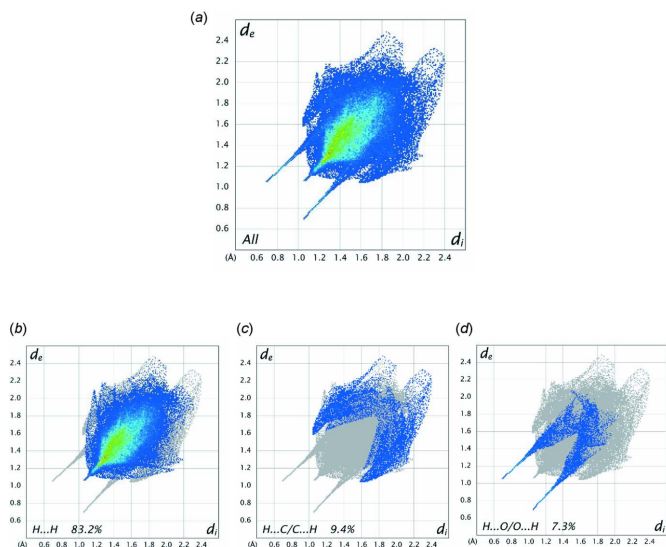
Further details of the intermolecular interactions in **1** were obtained using Hirshfeld surface analysis (Spackman & Jayatilaka, 2009) with Hirshfeld surfaces and two-dimensional fingerprint plots generated with *Crystal Explorer* (Turner *et al.*, 2017). Hirshfeld surfaces for opposite faces of **1** 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 FOHHAM and FOHHAM01; Bequeath *et al.*, 2005, Wedeking *et al.*, 2006a) and the tetradecane, octadecane and docosane derivatives (VEFXIO, VEFXOU,


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.

VEFXUA; Wedeking *et al.*, 2006a). n -Tetradecylferrocene (MEFRUL; Wedeking *et al.*, 2006b) is the only mono-ferrocene with an unsubstituted alkane chain, while our earlier


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.

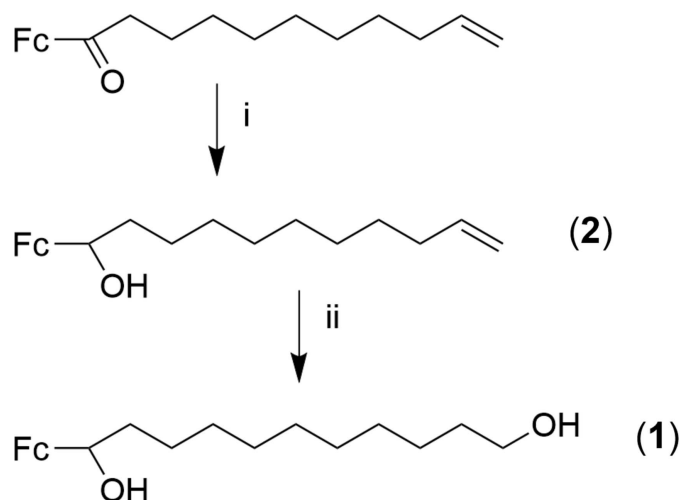


Figure 8
Preparation scheme for **1**; (i) LiAlH_4 , Et_2O ; (ii) 9-BBN, THF.

report of the structure of 11-bromo-1-ferrocenylundecan-1-one (LICNIV; 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, α,ω -dihydroxyalkane (C_n , $n \geq 10$) structures are uncommon and often crystallize as co-crystals, see, for example, KEXZOD and KEXZUJ (Loehlin *et al.*, 2007) OTIZEX, OTIZIB, OTIZOH and OTIZUN (Martí-Rujas *et al.*, 2011).

6. Synthesis and crystallization

The title compound **1** was prepared in two steps from 1-ferrocenylundec-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) (Aristoff *et al.*, 1985), Fig. 8. LiAlH_4 (0.10 g, 2.6 mmol) was added to 1-ferrocenylundec-10-en-1-one (0.615 g, 1.75 mmol) in Et_2O (10 mL) at 273 K and stirred for 1 h before quenching with a few drops of water. The ether fraction was rinsed with saturated NaCl solution and dried over MgSO_4 . The solvent was removed under vacuum to give 0.61 g (99%) of the yellow oil 1-ferrocenylundec-10-en-1-ol. To this oil, without further purification, in THF (10 ml) was added a solution of 9-BBN (0.5 M in hexane, 3.5 mmol), the mixture stirred at room temperature for 18 h before quenching with a few drops of water. The pH was raised to 8.5 with NaOH, then hydrogen peroxide (30% in H_2O , 7 ml) was added and the mixture allowed to stir for another 2 h. The organic layer was rinsed with saturated NaCl solution and dried over MgSO_4 . Column chromatography on SiO_2 with CH_2Cl_2 eluted a trace of the unreacted alcohol. Further elution with $\text{EtOAc}/\text{CH}_2\text{Cl}_2$ gave the title compound **1** as a yellow solid (0.60 g, 94%). X-ray quality crystals were grown from the mixed solvents of CH_2Cl_2 layered with hexane. Analysis calculated for $\text{C}_{21}\text{H}_{32}\text{O}_2\text{Fe}$: C, 67.74;

Table 3
Experimental details.

Crystal data	
Chemical formula	$[\text{Fe}(\text{C}_5\text{H}_5)(\text{C}_{16}\text{H}_{27}\text{O}_2)]$
M_r	372.31
Crystal system, space group	Monoclinic, $C2/c$
Temperature (K)	92
a, b, c (Å)	47.641 (3), 10.1522 (7), 7.8747 (6)
β (°)	97.091 (4)
V (Å ³)	3779.6 (5)
Z	8
Radiation type	Mo $K\alpha$
μ (mm ⁻¹)	0.81
Crystal size (mm)	0.32 × 0.14 × 0.04
Data collection	
Diffractometer	CCD area detector
Absorption correction	Multi-scan (SADABS; Bruker, 2011)
$T_{\text{min}}, T_{\text{max}}$	0.784, 1.000
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	16666, 2527, 2150
R_{int}	0.049
θ_{max} (°)	22.7
$(\sin \theta/\lambda)_{\text{max}}$ (Å ⁻¹)	0.544
Refinement	
$R[F^2 > 2\sigma(F^2)], wR(F^2), S$	0.037, 0.106, 1.06
No. of reflections	2527
No. of parameters	221
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{\text{max}}, \Delta\rho_{\text{min}}$ (e Å ⁻³)	0.64, -0.31

Computer programs: APEX2 and SAINT (Bruker, 2011), SHELXT (Sheldrick, 2015a), SHELXL2018/1 (Sheldrick, 2015b), TITAN (Hunter & Simpson, 1999), Mercury (Macrae *et al.*, 2020), enCIFer (Allen *et al.*, 2004), PLATON (Spek, 2020), publCIF (Westrip 2010) and WinGX (Farrugia 2012).

67.74; H, 8.66. Found: C, 67.94; H, 8.92%. ¹H NMR (CDCl_3): 4.30 (*m*, 1H, $-\text{CHOH}-$), 4.24 (*m*, 1H, C_5H_4), 4.20 (*s*, 5H, Cp), 4.17 (*m*, 3H, C_5H_4), 3.64 (*m*, 2H, $-\text{CH}_2-\text{OH}$), 1.92 [*d* ($J = 4$ Hz), 1H, Fc-CHOH], 1.7–1.3 [*m*, 18H, $-(\text{CH}_2)_9-$]. ¹³C NMR (CDCl_3): 94.7 (Fc *ipso*), 69.7 ($-\text{CHOH}-$), 68.3 (Cp), 67.9, 67.7, 67.3, 65.2 (Fc- $\text{C}\alpha$ & β), 63.2 ($-\text{CH}_2\text{OH}$), 38.3, 32.9, 29.6, 29.6, 29.5, 29.5, 26.1, 25.8 ($-\text{CH}_2-$). UV-vis (CH_2Cl_2): 325 (90), 440 (110) nm (ϵ).

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 $U_{\text{iso}}(\text{H}) = 1.5 U_{\text{eq}}(\text{O})$. All H-atoms bound to C were refined using a riding model with $\text{C}-\text{H} = 0.95\text{--}1.00$ Å and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$. 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.

Acknowledgements

We thank the New Zealand Ministry of Business, Innovation and Employment Science Investment Fund (grant No. UOO-

X1206) for support of this work and the University of Otago for the purchase of the diffractometer. JS also thanks the Department of Chemistry, University of Otago for support of his work.

Funding information

Funding for this research was provided by: Ministry for Business Innovation and Employment (grant No. UOO-X1206).

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supporting information

Acta Cryst. (2022). E78, 149-153 [https://doi.org/10.1107/S205698902101358X]

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

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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

$\beta = 97.091$ (4)°

$V = 3779.6$ (5) Å³

$Z = 8$

$F(000) = 1600$

$D_x = 1.309$ Mg m⁻³

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 4218 reflections

$\theta = 2.4$ – 22.4 °

$\mu = 0.81$ mm⁻¹

$T = 92$ K

Plate, yellow

$0.32 \times 0.14 \times 0.04$ 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 $I > 2\sigma(I)$

$R_{\text{int}} = 0.049$

$\theta_{\max} = 22.7$ °, $\theta_{\min} = 1.7$ °

$h = -51 \rightarrow 51$

$k = -11 \rightarrow 11$

$l = -8 \rightarrow 7$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.037$

$wR(F^2) = 0.106$

$S = 1.06$

2527 reflections

221 parameters

0 restraints

Hydrogen site location: inferred from neighbouring sites

H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0649P)^2 + 3.6265P]$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} = 0.001$

$\Delta\rho_{\max} = 0.64$ e Å⁻³

$\Delta\rho_{\min} = -0.31$ 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 $F_o \gg F_c$ were omitted from the final refinement cycles.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	x	y	z	U_{iso}^*/U_{eq}
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)

H16A	0.226058	0.908646	0.888857	0.034*
H16B	0.236941	1.006643	0.752146	0.034*
C17	0.26376 (6)	0.8485 (3)	0.8092 (4)	0.0277 (7)
H17A	0.261428	0.754213	0.836426	0.033*
H17B	0.272404	0.853228	0.701314	0.033*
C18	0.28391 (6)	0.9108 (3)	0.9514 (4)	0.0283 (7)
H18A	0.286265	1.005048	0.924393	0.034*
H18B	0.275331	0.905800	1.059485	0.034*
C19	0.31288 (6)	0.8462 (3)	0.9785 (4)	0.0286 (7)
H19A	0.310578	0.752371	1.007805	0.034*
H19B	0.321323	0.849641	0.869788	0.034*
C20	0.33312 (6)	0.9104 (3)	1.1183 (4)	0.0275 (7)
H20A	0.334116	1.005977	1.094737	0.033*
H20B	0.325615	0.899498	1.229180	0.033*
C21	0.36243 (6)	0.8533 (3)	1.1321 (4)	0.0307 (8)
H21A	0.361643	0.759959	1.167703	0.037*
H21B	0.369024	0.855208	1.017765	0.037*
O21	0.38252 (4)	0.9210 (2)	1.2505 (3)	0.0337 (6)
H21	0.3814 (5)	0.894 (3)	1.349 (4)	0.051*

Atomic displacement parameters (\AA^2)

	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, °)

C1—C2	1.427 (4)	O11—H11	0.76 (4)
C1—C5	1.432 (4)	C12—C13	1.534 (4)
C1—C11	1.501 (4)	C12—H12A	0.9900
C1—Fe1	2.040 (3)	C12—H12B	0.9900
C2—C3	1.413 (4)	C13—C14	1.515 (4)
C2—Fe1	2.041 (3)	C13—H13A	0.9900
C2—H2	0.9500	C13—H13B	0.9900
C3—C4	1.423 (4)	C14—C15	1.521 (4)
C3—Fe1	2.043 (3)	C14—H14A	0.9900
C3—H3	0.9500	C14—H14B	0.9900
C4—C5	1.413 (4)	C15—C16	1.517 (4)
C4—Fe1	2.042 (3)	C15—H15A	0.9900
C4—H4	0.9500	C15—H15B	0.9900
C5—Fe1	2.038 (3)	C16—C17	1.523 (4)
C5—H5	0.9500	C16—H16A	0.9900
Fe1—C9	2.033 (3)	C16—H16B	0.9900
Fe1—C10	2.041 (3)	C17—C18	1.520 (4)
Fe1—C6	2.049 (3)	C17—H17A	0.9900
Fe1—C8	2.052 (3)	C17—H17B	0.9900
Fe1—C7	2.053 (3)	C18—C19	1.519 (4)
C6—C7	1.415 (4)	C18—H18A	0.9900
C6—C10	1.422 (4)	C18—H18B	0.9900
C6—H6	0.9500	C19—C20	1.518 (4)
C7—C8	1.420 (4)	C19—H19A	0.9900
C7—H7	0.9500	C19—H19B	0.9900
C8—C9	1.402 (5)	C20—C21	1.504 (4)
C8—H8	0.9500	C20—H20A	0.9900
C9—C10	1.410 (5)	C20—H20B	0.9900
C9—H9	0.9500	C21—O21	1.427 (3)
C10—H10	0.9500	C21—H21A	0.9900
C11—O11	1.439 (3)	C21—H21B	0.9900
C11—C12	1.512 (4)	O21—H21	0.83 (4)
C11—H11A	1.0000		
C2—C1—C5	107.1 (3)	C9—C8—H8	126.0
C2—C1—C11	127.5 (3)	C7—C8—H8	126.0
C5—C1—C11	125.4 (3)	Fe1—C8—H8	126.5
C2—C1—Fe1	69.60 (16)	C8—C9—C10	108.9 (3)
C5—C1—Fe1	69.37 (16)	C8—C9—Fe1	70.63 (18)
C11—C1—Fe1	127.9 (2)	C10—C9—Fe1	70.03 (18)
C3—C2—C1	108.7 (3)	C8—C9—H9	125.5
C3—C2—Fe1	69.85 (17)	C10—C9—H9	125.5
C1—C2—Fe1	69.47 (16)	Fe1—C9—H9	125.4
C3—C2—H2	125.7	C9—C10—C6	107.2 (3)
C1—C2—H2	125.7	C9—C10—Fe1	69.47 (18)
Fe1—C2—H2	126.6	C6—C10—Fe1	69.96 (17)

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 (13)	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 (13)	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	109.5
C7—C8—Fe1	69.81 (17)		

Hydrogen-bond geometry (Å, °)

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
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) $-x+1/2, y-1/2, -z+3/2$; (ii) $-x+1/2, -y+3/2, -z+2$.