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5-Ferrocenylmethyl-2,2-dimethyl-5-(prop-2-ynyl)-1,3-dioxane-4,6-dione

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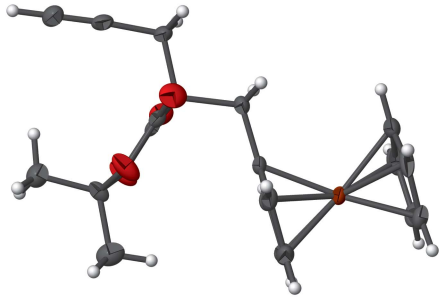
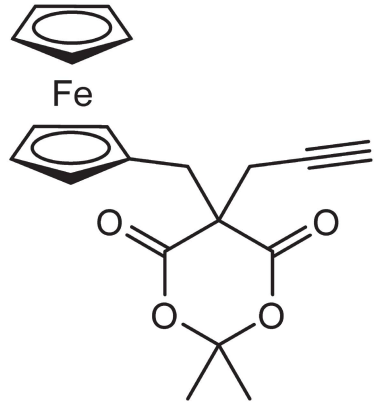
Structural data: full structural data are available from iucrdata.iucr.org

The title compound, $[Fe(C_5H_5)(C_{15}H_{15}O_4)]$, was obtained by hydrogenation and subsequent alkylation of 5-ferrocenylmethylene-2,2-dimethyl-1,3-dioxane-4,6-dione. Apart from C—H$\cdots$O=C hydrogen bonds, C≡C—H$\cdots\pi$ interactions forming crosswise chains of the molecules are observed in the crystal structure.

3D view



Chemical scheme



Structure description

The molecular structure of the title compound is shown in Fig. 1. The cyclopentadienyl groups in the ferrocene unit adopt an eclipsed conformation. The 1,3-dioxane ring is almost planar, with atom C14 displaying a maximum deviation of 0.098 (3) Å from the least-squares plane. In the crystal, weak C—H$\cdots$O=C hydrogen bonds generate a three-dimensional network (Table 1, Fig. 2). An intriguing intermolecular interaction between the acidic proton of the terminal alkyne group and the π system of the ferrocene moiety is observed. The pertinent distances are H18$\cdots$*Cg* = 2.637 Å and C18$\cdots$*Cg* = 3.533 Å, where *Cg* is the centroid of the unsubstituted cyclopentadienyl group. This interaction is directional with a C18—H18$\cdots$*Cg* angle of 157°, and the resulting chains are arranged crosswise as shown in Fig. 3.

Interactions involving cyclopentadienide anions as hydrogen-bond acceptors have been reluctantly identified as hydrogen bonds (Harder, 1999). Using the query 'C≡C—H$\cdots$*Cg*(ferrocene) non-bonded contact with H$\cdots$*Cg* distance $\leq$ 2.7 Å', several related structures have been found in the Cambridge Structure Database (Groom & Allen, 2014), see: Lin *et al.* (1996); Buchmeiser *et al.* (1998); Wong *et al.* (2001); Li *et al.* (2006); Busetto *et al.* (2012). Although not explicitly described as such by the respective authors, these structures exhibited the C≡C—H$\cdots\pi$ interactions mentioned, and the corresponding C$\cdots$*Cg* distances are $\leq$ 3.58 Å with C—H$\cdots$*Cg* angles ranging from 152 to 176°.

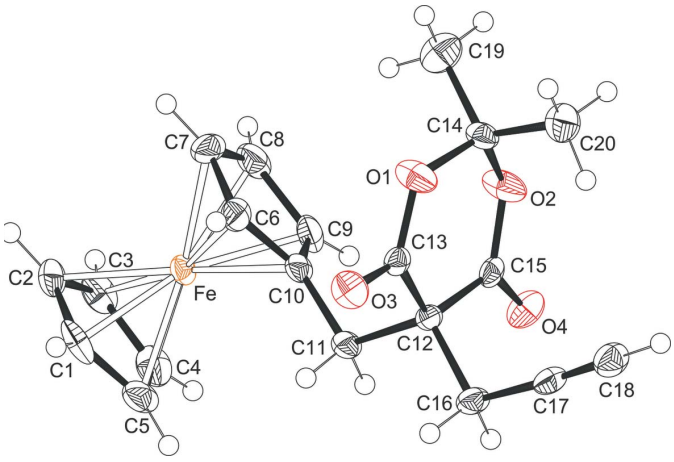


Figure 1
The asymmetric unit of the title compound, showing the atom labels and 50% probability displacement ellipsoids for non-H atoms.

Synthesis and crystallization

(1) 5-Ferrocenylmethylene-2,2-dimethyl-1,3-dioxane-4,6-dione (2.50 g, 7.34 mmol) (Bai *et al.*, 2004) in MeOH (80 ml) was hydrogenated (3 atm) for 30 min at room temperature using Pd/C (0.24 g, 5 wt.% Pd) as catalyst. Crystallization from MeOH yielded a brown product (1.66 g, 66%). ^{1}H NMR (300 MHz, $CDCl_3$): δ 4.20 (*m*, 2H), 4.13 (*s*, 5H), 4.04 (*m*, 2H), 3.60 (*t*, J = 4.3 Hz, 1H), 3.23 (*d*, J = 4.3 Hz, 2H), 1.67 (*s*, 3H), 1.52 (*s*, 3H) p.p.m. ^{13}C NMR (75 MHz, $CDCl_3$): δ 165.8, 105.4, 83.4, 70.2, 69.9, 69.1, 68.9, 68.3, 48.6, 28.8, 27.8, 27.6 p.p.m. IR (neat, ATR): ν 3086 (*w*), 2992 (*w*), 1560 (*s*), 1405 (*m*), 1258 (*m*), 1199 (*m*), 1103 (*m*), 1035 (*w*), 1019 (*w*), 996 (*w*), 925 (*w*), 879 (*w*), 815 (*w*), 785 (*w*), 755 (*w*), 744 (*w*), 654 (*w*), 525 (*w*), 479 (*w*), 446 (*w*) cm^{-1}.

(2) A mixture of 5-ferrocenylmethyl-2,2-dimethyl-1,3-dioxane-4,6-dione (0.34 g, 1.0 mmol) and anhydrous K_2CO_3 (0.21 g, 1.5 mmol) in DMF (20 ml) was stirred for 30 min. A solution of 3-bromo-1-propyne (80 wt.% in toluene, 0.23 g, 1.5 mmol) was added, and stirring was continued for 48 h. After removal of the solvent, the residue was partitioned twice between CH_2Cl_2 (10 ml) and 2 *M* HCl (10 ml). The organic solution was concentrated and cooled at 278 K to yield yellow crystals (0.30 g, 76%). ^{1}H NMR (300 MHz, $CDCl_3$): δ 4.19 (*m*, 2H), 4.16 (*m*, 5H), 4.10 (*m*, 2H), 3.05 (*s*, 2H), 2.92 (*d*, J =

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

Cg is the centroid of the unsubstituted cyclopentadienyl ring.

D—H···A	D—H	H···A	D···A	D—H···A
C20—H20*C*···O3^{i}	0.98	2.50	3.419 (3)	157
C3—H3···O3^{ii}	0.95	2.50	3.431 (3)	167
C19—H19*A*···O3^{iii}	0.98	2.54	3.460 (3)	157
C16—H16*A*···O4^{iv}	0.99	2.57	3.114 (3)	114
C4—H4···O4^{v}	0.95	2.60	3.372 (3)	139
C18—H18···*Cg*^{vi}	0.95	2.64	3.533	157

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

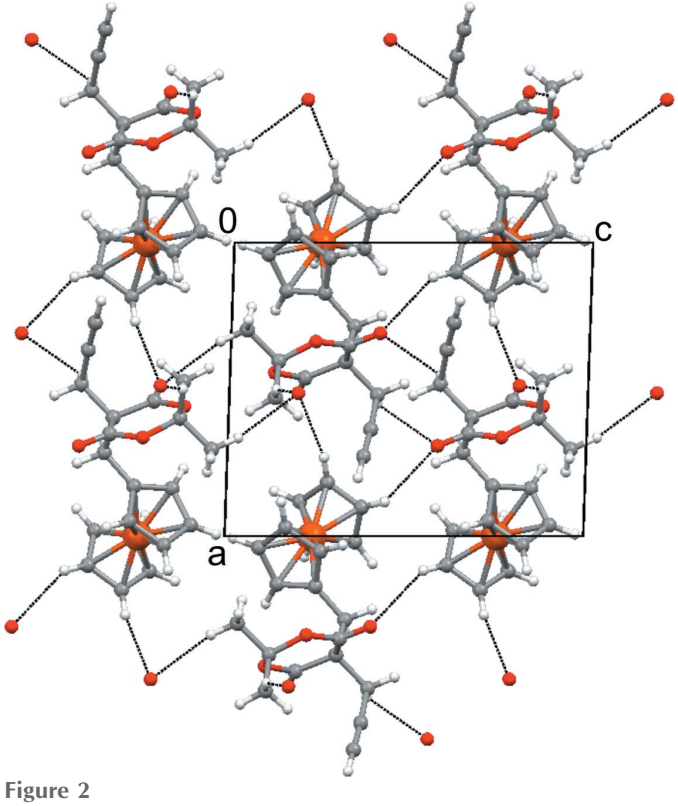


Figure 2
The crystal packing of the title compound viewed along the *b* axis. C—H···O═C hydrogen bonds are shown as dotted lines (see Table 1 for numerical details.

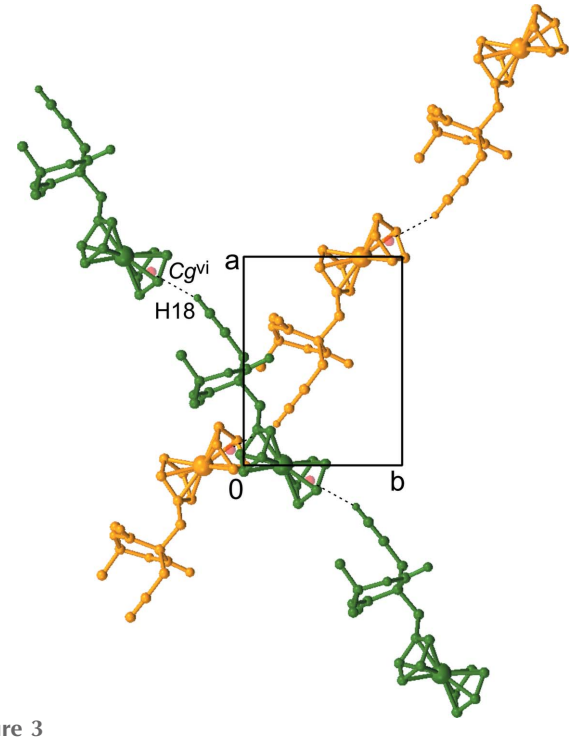


Figure 3
Chains of the title compound formed by C≡C—H···π interactions. The centroids of the rings are drawn as red spheres, and the contacts are drawn as dashed lines. The symmetry code refers to Table 1.

Table 2
Experimental details.

Crystal data	
Chemical formula	$[Fe(C_5H_5)(C_{15}H_{15}O_4)]$
M_r	380.21
Crystal system, space group	Monoclinic, $P2_1$
Temperature (K)	173
a, b, c (Å)	9.8263 (4), 7.4617 (2), 11.9888 (4)
β (°)	91.956 (1)
V (Å^3)	878.52 (5)
Z	2
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.88
Crystal size (mm)	0.18 × 0.11 × 0.07
Data collection	
Diffractometer	Bruker D8 Quest
Absorption correction	Multi-scan (*SADABS*; Bruker, 2012)
T_{min}, T_{max}	0.896, 0.942
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	17890, 3215, 3078
R_{int}	0.031
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.605
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.021, 0.052, 1.05
No. of reflections	3215
No. of parameters	227
No. of restraints	1
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.28, −0.17
Absolute structure	Flack x determined using 1346 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ (Parsons *et al.*, 2013)
Absolute structure parameter	0.009 (6)

Computer programs: *APEX2* and *SAINT* (Bruker, 2012), *SHELXT* (Sheldrick, 2015*a*), *SHELXL2014* (Sheldrick, 2015*b*), *ORTEP-3 for Windows* (Farrugia, 2012), *Mercury* (Macrae *et al.*, 2006) and *publCIF* (Westrip, 2010).

2.6 Hz, 2H), 2.11 (*t*, *J* = 2.6 Hz, 1H), 1.63 (*s*, 3H), 1.05 (*s*, 3H) p.p.m. ^{13}C NMR (75 MHz, $CDCl_3$): δ 168.1, 106.7, 80.5, 78.4, 77.4, 73.0, 70.2, 69.1, 57.0, 40.4, 30.4, 29.2, 28.4 p.p.m. IR (neat, ATR): ν 3266 (*m*), 3086 (*w*), 2934 (*w*), 1765 (*m*), 1732 (*s*), 1641 (*m*), 1431 (*w*), 1394 (*m*), 1381 (*m*), 1348 (*s*), 1269 (*s*), 1228 (*s*), 1199 (*m*), 1172 (*m*), 1054 (*s*), 1037 (*m*), 1022 (*m*), 1000 (*m*), 955 (*m*), 917 (*w*), 832 (*m*), 813 (*m*), 702 (*m*), 678 (*m*), 664 (*m*), 498 (*s*), 482 (*m*), 465 (*m*), 421 (*s*) cm^{-1}.

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2.

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full crystallographic data

IUCrData (2016). **1**, x160569 [doi:10.1107/S2414314616005691]

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Crystal data

$[Fe(C_5H_5)(C_{15}H_{15}O_4)]$
M_r = 380.21
Monoclinic, $P2_1$
a = 9.8263 (4) Å
b = 7.4617 (2) Å
c = 11.9888 (4) Å
β = 91.956 (1)°
V = 878.52 (5) Å^3
Z = 2
$F(000)$ = 396
D_x = 1.437 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 9930 reflections
θ = 5.2–50.6°
μ = 0.88 mm^{-1}
T = 173 K
Plate, yellow
0.18 × 0.11 × 0.07 mm

Data collection

Bruker D8 Quest diffractometer
Radiation source: Incoatec Microfocus
Detector resolution: 10.4 pixels mm^{-1}
phi– and ω–scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2012)
T_{min} = 0.896, T_{max} = 0.942
17890 measured reflections
3215 independent reflections
3078 reflections with $I > 2\sigma(I)$
R_{int} = 0.031
θ_{max} = 25.5°, θ_{min} = 2.6°
h = −11→11
k = −9→9
l = −14→14

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.021
$wR(F^2)$ = 0.052
S = 1.05
3215 reflections
227 parameters
1 restraint
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0281P)^2 + 0.1144P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.28 e Å^{-3}
$\Delta\rho_{min}$ = −0.16 e Å^{-3}
Extinction correction: *SHELXL2014* (Sheldrick, 2015*b*), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.019 (2)
Absolute structure: Flack x determined using 1346 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ (Parsons *et al.*, 2013)
Absolute structure parameter: 0.009 (6)

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.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
Fe	0.00210 (3)	0.24448 (7)	0.24251 (3)	0.01943 (12)
O1	0.4427 (2)	−0.0935 (3)	0.12458 (16)	0.0345 (5)
O2	0.3366 (2)	−0.3154 (2)	0.23702 (15)	0.0353 (5)
O3	0.51239 (19)	0.1619 (2)	0.19227 (16)	0.0319 (5)
O4	0.30812 (17)	−0.2766 (3)	0.41503 (13)	0.0308 (4)
C1	0.0061 (3)	0.5163 (4)	0.2564 (3)	0.0325 (9)
H1	0.0702	0.5942	0.2239	0.039*
C2	−0.1185 (3)	0.4599 (4)	0.2063 (2)	0.0296 (6)
H2	−0.1530	0.4933	0.1343	0.036*
C3	−0.1829 (3)	0.3452 (4)	0.2818 (3)	0.0320 (7)
H3	−0.2684	0.2878	0.2695	0.038*
C4	−0.0984 (3)	0.3308 (4)	0.3784 (2)	0.0347 (7)
H4	−0.1167	0.2618	0.4428	0.042*
C5	0.0192 (3)	0.4373 (4)	0.3630 (3)	0.0328 (7)
H5	0.0934	0.4525	0.4151	0.039*
C6	0.1633 (3)	0.1703 (4)	0.1508 (2)	0.0270 (6)
H6	0.2298	0.2475	0.1210	0.032*
C7	0.0384 (3)	0.1171 (4)	0.0963 (3)	0.0369 (7)
H7	0.0069	0.1530	0.0239	0.044*
C8	−0.0302 (3)	0.0016 (4)	0.1691 (3)	0.0366 (7)
H8	−0.1163	−0.0532	0.1543	0.044*
C9	0.0515 (3)	−0.0183 (4)	0.2677 (3)	0.0286 (7)
H9	0.0303	−0.0903	0.3301	0.034*
C10	0.1709 (3)	0.0877 (3)	0.2576 (2)	0.0220 (6)
C11	0.2850 (3)	0.1041 (4)	0.3429 (2)	0.0236 (6)
H11A	0.2495	0.0805	0.4178	0.028*
H11B	0.3201	0.2285	0.3425	0.028*
C12	0.4048 (2)	−0.0280 (3)	0.32172 (19)	0.0182 (5)
C13	0.4606 (2)	0.0193 (3)	0.2093 (2)	0.0193 (5)
C14	0.4027 (3)	−0.2778 (4)	0.13503 (18)	0.0232 (6)
C15	0.3494 (2)	−0.2168 (3)	0.32999 (19)	0.0198 (6)
C16	0.5180 (3)	0.0005 (4)	0.4130 (2)	0.0256 (6)
H16A	0.5477	0.1272	0.4119	0.031*
H16B	0.4803	−0.0239	0.4870	0.031*
C17	0.6361 (3)	−0.1157 (4)	0.3974 (2)	0.0252 (6)
C18	0.7276 (3)	−0.2127 (4)	0.3801 (2)	0.0310 (7)
H18	0.8014	−0.2909	0.3662	0.037*
C19	0.2962 (3)	−0.3082 (4)	0.0440 (2)	0.0392 (8)
H19A	0.3354	−0.2845	−0.0287	0.059*
H19B	0.2647	−0.4327	0.0465	0.059*
H19C	0.2192	−0.2273	0.0546	0.059*
C20	0.5268 (3)	−0.3920 (4)	0.1287 (3)	0.0369 (7)
H20A	0.5726	−0.3669	0.0591	0.055*
H20B	0.5888	−0.3656	0.1924	0.055*
H20C	0.5006	−0.5187	0.1306	0.055*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Fe	0.01545 (17)	0.01707 (17)	0.02571 (19)	0.00290 (14)	−0.00022 (12)	−0.00182 (17)
O1	0.0594 (14)	0.0230 (10)	0.0220 (10)	−0.0103 (10)	0.0163 (9)	−0.0017 (8)
O2	0.0580 (13)	0.0232 (10)	0.0256 (10)	−0.0160 (9)	0.0154 (9)	−0.0040 (8)
O3	0.0342 (11)	0.0232 (9)	0.0384 (12)	−0.0119 (9)	0.0015 (9)	0.0036 (9)
O4	0.0305 (9)	0.0416 (12)	0.0206 (8)	−0.0025 (10)	0.0048 (7)	0.0098 (9)
C1	0.0259 (19)	0.0139 (15)	0.059 (2)	0.0020 (11)	0.0155 (17)	−0.0054 (13)
C2	0.0258 (15)	0.0260 (15)	0.0369 (16)	0.0116 (12)	−0.0006 (12)	−0.0005 (12)
C3	0.0150 (12)	0.0284 (16)	0.0530 (19)	0.0018 (11)	0.0062 (12)	−0.0071 (14)
C4	0.0404 (17)	0.0325 (15)	0.0323 (16)	0.0081 (13)	0.0165 (13)	0.0016 (12)
C5	0.0257 (14)	0.0328 (15)	0.0397 (17)	0.0091 (12)	−0.0044 (12)	−0.0156 (13)
C6	0.0261 (14)	0.0316 (14)	0.0235 (14)	0.0125 (12)	0.0035 (11)	−0.0019 (11)
C7	0.0359 (17)	0.0468 (18)	0.0272 (16)	0.0220 (14)	−0.0113 (12)	−0.0136 (13)
C8	0.0275 (16)	0.0256 (15)	0.056 (2)	0.0052 (13)	−0.0143 (15)	−0.0158 (15)
C9	0.0273 (17)	0.0160 (14)	0.0421 (17)	0.0038 (13)	−0.0024 (13)	−0.0006 (12)
C10	0.0186 (13)	0.0209 (13)	0.0263 (14)	0.0068 (11)	−0.0015 (10)	−0.0060 (11)
C11	0.0235 (14)	0.0251 (13)	0.0221 (14)	0.0069 (11)	−0.0007 (11)	−0.0068 (11)
C12	0.0181 (12)	0.0205 (13)	0.0159 (12)	0.0025 (10)	−0.0008 (9)	−0.0013 (10)
C13	0.0152 (11)	0.0220 (13)	0.0206 (12)	0.0029 (10)	−0.0020 (10)	0.0004 (10)
C14	0.0327 (13)	0.0196 (15)	0.0177 (11)	−0.0022 (12)	0.0056 (9)	−0.0029 (11)
C15	0.0146 (11)	0.0248 (16)	0.0200 (12)	0.0063 (9)	−0.0017 (9)	0.0027 (10)
C16	0.0209 (13)	0.0332 (15)	0.0224 (13)	0.0050 (11)	−0.0059 (10)	−0.0070 (11)
C17	0.0236 (14)	0.0319 (14)	0.0195 (13)	−0.0008 (12)	−0.0063 (10)	−0.0013 (11)
C18	0.0260 (13)	0.0392 (19)	0.0276 (13)	0.0054 (12)	−0.0018 (11)	−0.0012 (12)
C19	0.0347 (16)	0.047 (2)	0.0361 (16)	−0.0046 (13)	−0.0032 (13)	0.0043 (13)
C20	0.0330 (17)	0.0367 (16)	0.0406 (19)	0.0070 (14)	−0.0037 (14)	−0.0018 (14)

Geometric parameters (Å, º)

Fe—C10	2.032 (2)	C6—H6	0.9500
Fe—C6	2.035 (2)	C7—C8	1.414 (5)
Fe—C8	2.035 (3)	C7—H7	0.9500
Fe—C2	2.035 (3)	C8—C9	1.414 (5)
Fe—C1	2.035 (3)	C8—H8	0.9500
Fe—C7	2.036 (3)	C9—C10	1.423 (4)
Fe—C3	2.037 (3)	C9—H9	0.9500
Fe—C4	2.038 (3)	C10—C11	1.497 (4)
Fe—C9	2.040 (3)	C11—C12	1.563 (3)
Fe—C5	2.041 (3)	C11—H11A	0.9900
O1—C13	1.326 (3)	C11—H11B	0.9900
O1—C14	1.437 (3)	C12—C13	1.514 (3)
O2—C15	1.338 (3)	C12—C15	1.515 (3)
O2—C14	1.432 (3)	C12—C16	1.548 (3)
O3—C13	1.200 (3)	C14—C20	1.492 (4)
O4—C15	1.196 (3)	C14—C19	1.504 (4)
C1—C5	1.409 (4)	C16—C17	1.466 (4)

C1—C2	1.409 (4)	C16—H16A	0.9900
C1—H1	0.9500	C16—H16B	0.9900
C2—C3	1.411 (4)	C17—C18	1.178 (4)
C2—H2	0.9500	C18—H18	0.9500
C3—C4	1.406 (4)	C19—H19A	0.9800
C3—H3	0.9500	C19—H19B	0.9800
C4—C5	1.420 (4)	C19—H19C	0.9800
C4—H4	0.9500	C20—H20A	0.9800
C5—H5	0.9500	C20—H20B	0.9800
C6—C10	1.420 (4)	C20—H20C	0.9800
C6—C7	1.427 (4)		

C10—Fe—C6	40.87 (10)	Fe—C5—H5	126.3
C10—Fe—C8	68.95 (11)	C10—C6—C7	108.0 (3)
C6—Fe—C8	68.73 (12)	C10—C6—Fe	69.46 (14)
C10—Fe—C2	160.22 (11)	C7—C6—Fe	69.51 (15)
C6—Fe—C2	123.91 (11)	C10—C6—H6	126.0
C8—Fe—C2	122.02 (12)	C7—C6—H6	126.0
C10—Fe—C1	123.56 (11)	Fe—C6—H6	126.6
C6—Fe—C1	107.58 (11)	C8—C7—C6	107.9 (3)
C8—Fe—C1	157.70 (13)	C8—C7—Fe	69.65 (17)
C2—Fe—C1	40.51 (12)	C6—C7—Fe	69.45 (15)
C10—Fe—C7	69.00 (11)	C8—C7—H7	126.0
C6—Fe—C7	41.04 (12)	C6—C7—H7	126.0
C8—Fe—C7	40.64 (14)	Fe—C7—H7	126.4
C2—Fe—C7	107.64 (12)	C7—C8—C9	108.1 (3)
C1—Fe—C7	122.17 (13)	C7—C8—Fe	69.70 (16)
C10—Fe—C3	157.47 (11)	C9—C8—Fe	69.88 (15)
C6—Fe—C3	160.44 (11)	C7—C8—H8	126.0
C8—Fe—C3	107.53 (12)	C9—C8—H8	126.0
C2—Fe—C3	40.55 (11)	Fe—C8—H8	126.0
C1—Fe—C3	68.17 (11)	C8—C9—C10	108.5 (3)
C7—Fe—C3	123.72 (12)	C8—C9—Fe	69.52 (16)
C10—Fe—C4	121.80 (11)	C10—C9—Fe	69.25 (15)
C6—Fe—C4	157.64 (12)	C8—C9—H9	125.8
C8—Fe—C4	123.67 (13)	C10—C9—H9	125.8
C2—Fe—C4	68.05 (12)	Fe—C9—H9	127.1
C1—Fe—C4	68.16 (12)	C6—C10—C9	107.4 (2)
C7—Fe—C4	159.97 (13)	C6—C10—C11	126.5 (2)
C3—Fe—C4	40.37 (12)	C9—C10—C11	126.0 (3)
C10—Fe—C9	40.92 (12)	C6—C10—Fe	69.67 (14)
C6—Fe—C9	68.46 (12)	C9—C10—Fe	69.83 (15)
C8—Fe—C9	40.60 (13)	C11—C10—Fe	127.14 (17)
C2—Fe—C9	157.63 (12)	C10—C11—C12	112.8 (2)
C1—Fe—C9	160.38 (12)	C10—C11—H11A	109.0
C7—Fe—C9	68.33 (13)	C12—C11—H11A	109.0
C3—Fe—C9	122.02 (12)	C10—C11—H11B	109.0
C4—Fe—C9	107.76 (13)	C12—C11—H11B	109.0

C10—Fe—C5	107.18 (11)	H11A—C11—H11B	107.8
C6—Fe—C5	121.79 (12)	C13—C12—C15	114.63 (19)
C8—Fe—C5	160.31 (13)	C13—C12—C16	108.9 (2)
C2—Fe—C5	68.12 (12)	C15—C12—C16	109.4 (2)
C1—Fe—C5	40.43 (13)	C13—C12—C11	107.2 (2)
C7—Fe—C5	157.73 (13)	C15—C12—C11	107.58 (19)
C3—Fe—C5	68.23 (12)	C16—C12—C11	108.99 (19)
C4—Fe—C5	40.74 (12)	O3—C13—O1	118.6 (2)
C9—Fe—C5	124.02 (13)	O3—C13—C12	121.8 (2)
C13—O1—C14	124.78 (19)	O1—C13—C12	119.4 (2)
C15—O2—C14	124.80 (19)	O2—C14—O1	113.30 (19)
C5—C1—C2	108.2 (3)	O2—C14—C20	109.1 (2)
C5—C1—Fe	70.02 (18)	O1—C14—C20	108.4 (2)
C2—C1—Fe	69.74 (17)	O2—C14—C19	105.4 (2)
C5—C1—H1	125.9	O1—C14—C19	105.5 (2)
C2—C1—H1	125.9	C20—C14—C19	115.3 (2)
Fe—C1—H1	125.9	O4—C15—O2	118.7 (2)
C1—C2—C3	108.1 (3)	O4—C15—C12	122.4 (2)
C1—C2—Fe	69.76 (17)	O2—C15—C12	118.7 (2)
C3—C2—Fe	69.81 (15)	C17—C16—C12	112.5 (2)
C1—C2—H2	126.0	C17—C16—H16A	109.1
C3—C2—H2	126.0	C12—C16—H16A	109.1
Fe—C2—H2	126.0	C17—C16—H16B	109.1
C4—C3—C2	108.0 (2)	C12—C16—H16B	109.1
C4—C3—Fe	69.84 (15)	H16A—C16—H16B	107.8
C2—C3—Fe	69.64 (15)	C18—C17—C16	176.5 (3)
C4—C3—H3	126.0	C17—C18—H18	180.0
C2—C3—H3	126.0	C14—C19—H19A	109.5
Fe—C3—H3	126.1	C14—C19—H19B	109.5
C3—C4—C5	108.1 (3)	H19A—C19—H19B	109.5
C3—C4—Fe	69.79 (15)	C14—C19—H19C	109.5
C5—C4—Fe	69.76 (16)	H19A—C19—H19C	109.5
C3—C4—H4	126.0	H19B—C19—H19C	109.5
C5—C4—H4	126.0	C14—C20—H20A	109.5
Fe—C4—H4	126.1	C14—C20—H20B	109.5
C1—C5—C4	107.6 (3)	H20A—C20—H20B	109.5
C1—C5—Fe	69.56 (18)	C14—C20—H20C	109.5
C4—C5—Fe	69.50 (17)	H20A—C20—H20C	109.5
C1—C5—H5	126.2	H20B—C20—H20C	109.5
C4—C5—H5	126.2		

C5—C1—C2—C3	−0.1 (3)	C8—C9—C10—Fe	58.59 (19)
Fe—C1—C2—C3	59.52 (18)	C6—C10—C11—C12	−83.2 (3)
C5—C1—C2—Fe	−59.7 (2)	C9—C10—C11—C12	94.8 (3)
C1—C2—C3—C4	0.0 (3)	Fe—C10—C11—C12	−174.29 (17)
Fe—C2—C3—C4	59.53 (18)	C10—C11—C12—C13	61.6 (3)
C1—C2—C3—Fe	−59.49 (19)	C10—C11—C12—C15	−62.1 (3)
C2—C3—C4—C5	0.1 (3)	C10—C11—C12—C16	179.3 (2)

Fe—C3—C4—C5	59.47 (19)	C14—O1—C13—O3	169.6 (2)
C2—C3—C4—Fe	−59.40 (18)	C14—O1—C13—C12	−15.6 (4)
C2—C1—C5—C4	0.2 (3)	C15—C12—C13—O3	−177.1 (2)
Fe—C1—C5—C4	−59.31 (19)	C16—C12—C13—O3	−54.2 (3)
C2—C1—C5—Fe	59.5 (2)	C11—C12—C13—O3	63.6 (3)
C3—C4—C5—C1	−0.1 (3)	C15—C12—C13—O1	8.3 (3)
Fe—C4—C5—C1	59.3 (2)	C16—C12—C13—O1	131.2 (2)
C3—C4—C5—Fe	−59.49 (19)	C11—C12—C13—O1	−111.1 (2)
C10—C6—C7—C8	−0.3 (3)	C15—O2—C14—O1	−22.3 (3)
Fe—C6—C7—C8	−59.23 (19)	C15—O2—C14—C20	98.5 (3)
C10—C6—C7—Fe	58.95 (18)	C15—O2—C14—C19	−137.1 (3)
C6—C7—C8—C9	−0.5 (3)	C13—O1—C14—O2	21.3 (4)
Fe—C7—C8—C9	−59.57 (19)	C13—O1—C14—C20	−99.9 (3)
C6—C7—C8—Fe	59.10 (19)	C13—O1—C14—C19	136.1 (2)
C7—C8—C9—C10	1.0 (3)	C14—O2—C15—O4	−167.7 (2)
Fe—C8—C9—C10	−58.42 (18)	C14—O2—C15—C12	17.4 (3)
C7—C8—C9—Fe	59.45 (19)	C13—C12—C15—O4	176.3 (2)
C7—C6—C10—C9	0.9 (3)	C16—C12—C15—O4	53.6 (3)
Fe—C6—C10—C9	59.89 (18)	C11—C12—C15—O4	−64.6 (3)
C7—C6—C10—C11	179.2 (2)	C13—C12—C15—O2	−9.0 (3)
Fe—C6—C10—C11	−121.8 (2)	C16—C12—C15—O2	−131.7 (2)
C7—C6—C10—Fe	−58.98 (18)	C11—C12—C15—O2	110.1 (2)
C8—C9—C10—C6	−1.2 (3)	C13—C12—C16—C17	−61.9 (3)
Fe—C9—C10—C6	−59.79 (18)	C15—C12—C16—C17	64.1 (3)
C8—C9—C10—C11	−179.5 (2)	C11—C12—C16—C17	−178.5 (2)
Fe—C9—C10—C11	121.9 (2)		

Hydrogen-bond geometry (Å, º)

Cg is the centroid of the unsubstituted cyclopentadienyl ring.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C20—H20*C*···O3^{i}	0.98	2.50	3.419 (3)	157
C3—H3···O3^{ii}	0.95	2.50	3.431 (3)	167
C19—H19*A*···O3^{iii}	0.98	2.54	3.460 (3)	157
C16—H16*A*···O4^{iv}	0.99	2.57	3.114 (3)	114
C4—H4···O4^{v}	0.95	2.60	3.372 (3)	139
C18—H18···*Cg*^{vi}	0.95	2.64	3.533	157

Symmetry codes: (i) *x*, *y*−1, *z*; (ii) *x*−1, *y*, *z*; (iii) −*x*+1, *y*−1/2, −*z*; (iv) −*x*+1, *y*+1/2, −*z*+1; (v) −*x*, *y*+1/2, −*z*+1; (vi) *x*+1, *y*−1, *z*.