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Crystal structure of (E)-1,2-diferrocenyl-1,2-bis(furan-2-yl)ethene

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The title compound, [Fe₂(C₅H₅)₂(C₁₀H₁₄O₂)], is the product of a new synthetic route towards tetraaryl/hetaryl-substituted ethenes that reduces the occurrence of side-products. In the crystal, the molecule is centrosymmetric and the cyclopentadienyl (Cp) rings are nearly coplanar and aligned slightly closer to a staggered conformation than to an eclipsed one. The ethene plane is tilted by 32.40 (18)° with respect to that of the substituted Cp ring and by 63.19 (19)° with respect to that of the furan ring. C—H···π interactions link the molecules into a three-dimensional supramolecular framework.

1. Chemical context

Tetrasubstituted ethenes bearing aryl, hetaryl or ferrocenyl groups are of current interest, as many of them find applications as novel materials for photooptics, electronics, crystal engineering and as new medications (Astruc, 2017). Ethene derivatives with a ferrocenyl unit on one or both C atoms of the alkene deserve special attention. Prominent representatives of the first type are ferrocifene {1-[4-(2-dimethylaminoethoxy)phenyl]-1-phenyl-2-ferrocenylbut-1-ene} and its di-OH analogue, which are known as potent, organometallic antitumor drugs (Jaouen et al., 2015; Resnier et al., 2017). On the other hand, dimethyl (Z)-2,3-diferrocenylbut-2-enedioate displays interesting redox and solvatochromic properties (Solntsev et al., 2011). As typical procedures for the preparation of tetrasubstituted ethenes containing a ferrocenyl substituent, conversions of the corresponding ketones under the McMurry reaction conditions (Top et al., 1997) or reductive coupling using low-valent titanium agents are recommended (Dang et al., 1990). In both cases, the reported yields are satisfactory to good, but a serious disadvantage is the formation of side-products. Recently, we reported a new approach to tetraaryl/hetaryl-substituted ethenes via desilylation of 2-(trimethylsilyl)-4,4,5,5-tetraaryl/hetaryl-1,3-dithiolanes, obtained from diaryl/hetaryl thioketones by treatment with (trimethylsilyl)diazomethane (TMS-CHN₂) at low temperature (Młoston et al., 2017). The mechanism of this unusual conversion was explained by the assumption that the in situ-generated 1,3-dithiolane anion undergoes a spontaneous cycloelimination ([3 + 2]-cycloversion) to give the dithioformate anion and the corresponding tetrasubstituted ethene derivative. The same method was applied for the preparation of some ferrocenyl/hetaryl-substituted ethenes (Młoston et al., 2018).
Here we report the analogous synthesis and crystal structure of the known title compound, \((E)-1\), with m.p. \(485–487 \, \text{K}\). For the previously described synthesis of this product (Dang et al., 1990), a m.p. of \(489–491 \, \text{K}\) and a yield of 17% were reported and the authors tentatively assigned the \((E)\)-configuration to the obtained compound. In our case, single crystals of \((E)-1\) were grown from hexane/\(\text{CH}_2\text{Cl}_2\) and used for an X-ray diffraction analysis, from which the previous tentatively postulated structure of the obtained isomer could be confirmed.

2. Structural commentary

The molecule of \((E)-1\) sits across a crystallographic centre of inversion and is shown in Fig. 1. Within the asymmetric unit, the Fe atom sits very well centred between the cyclopentadienyl (Cp) rings with all Fe—C distances in the range \(2.0352 (17)–2.0712 (16) \, \text{Å}\). The Cp C—C bond lengths [mean 1.435 (2) Å] involving the substituted C atom, C6, are very slightly elongated compared with the other C—C distances [mean 1.418 (3) Å]. Other bond lengths and angles are unremarkable. The two Cp rings are aligned slightly closer to a staggered conformation than to an eclipsed one, with the ring rotation from perfectly eclipsed being \(20.6 (2) ^\circ\) \(\left(18^\circ\right)\) is the halfway point between eclipsed and staggered). The dihedral angle between the planes of the two Cp rings in the ferrocenyl entity is only \(4.08 (11) ^\circ\) and ethene atom C1 is coplanar with the Cp ring to which it is bonded. However, the ferrocenyl entity is tilted with respect to the ethene plane, with a dihedral angle between the substituted Cp ring and the adjacent furan ring is \(53.46 (11) ^\circ\), while that between the plane of the furan ring and the ethene plane is \(63.19 (19) ^\circ\). The planes of the two furan rings are necessarily parallel because of the centre of inversion.

3. Supramolecular features

There are no significant C—H···O or π···π interactions, but some weak C—H···π interactions are present (Table 1). C8—H of the substituted Cp ring has an edge-on intermolecular interaction with the unsubstituted Cp ring at \(x + 1/2, -y + 3/2, z - 1/2\), while that between the plane of the furan ring and the ethene plane is \(63.19 (19) ^\circ\). The planes of the two furan rings are necessarily parallel because of the centre of inversion.

| D—H···A | D—H | H···A | D···A | D—H···A |
|---------|------|------|-------|---------|
| C3—H3···Cg1 \(i\) | 0.95 | 2.81 | 3.686 (3) | 153 |
| C8—H8···Cg2 \(ii\) | 0.95 | 2.85 | 3.764 (2) | 161 |
| C10—H10···Cg3 \(iii\) | 0.95 | 2.68 | 3.2097 (18) | 116 |

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

Figure 1

The molecular structure of the title compound, \((E)-1\), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level. Symmetry code: \((i)\) \(-x + 1, -y + 1, -z + 1\).

Figure 2

The sheets of molecules lying parallel to the \((101)\) plane formed by the C8—H···π interactions.
5. Synthesis and crystallization

The title compound was prepared according to the reaction sequence presented in the scheme below. A solution of thioketone \( \text{2} \) (297 mg, 1 mmol; prepared according to Młostoni et al., 2015) in THF (3 ml) was cooled to 198 K (acetone/dry ice). Then, TMS-CHN\(_2\) was added portion-wise to the mixture until the green colour of the starting thioketone disappeared. The magnetically stirred reaction mixture was allowed to warm slowly to ca 268 to 273 K and at this temperature a commercially available solution of tetrabutylammonium fluoride (TBAF, 1 ml, 1 M) was added in small portions. Stirring was continued for 20 min, and after warming to room temperature, the solvent was evaporated under vacuum. The crude product was analyzed by \(^1\)H NMR spectroscopy, which revealed the presence of two isomeric ethenes in a ratio of ca 10:1. After column chromatography (SiO\(_2\), CH\(_2\)Cl\(_2\)/hexane 3:7), the major product was isolated, contaminated with a small admixture of the minor one, as an analytically pure material, crystals suitable for the X-ray diffraction measurements were separated without additional recrystallization.

\[ \text{[Fe}_2\text{(C}_5\text{H}_5\text{)}_2\text{(C}_2\text{H}_4\text{O}_2\text{)]} \]

\(^1\)H NMR [600 MHz, CDCl\(_3\), \( \delta \) (ppm), \( J \) (Hz)]; 3.63–3.65 [m, 4CH(Fe)], 4.13–4.15 [m, 4CH(Fe)], 4.16 [s, 10CH(Fe)], 6.40 [d, \( J_{\text{HH}} = 3.0 \), 2CH(Fur)], 6.54 [dd, \( J_{\text{HH}} = 1.8, J_{\text{HH}} = 3.0 \), 2CH(Fur)], 7.58 [brs, 2CH(Fur)]. \(^{13}\)C NMR [150 MHz, CDCl\(_3\),

4. Database survey

The Cambridge Structural Database (CSD, Version 5.39 with February 2018 updates; Groom et al., 2016) contains one entry for a 1,1-diferrocenylethene [1,1-bis(1’,2”3’,4”,5”-penta-methylferrocen-1’-yl)ethene, CSD refcode CIJQAN, Heigl et al., 1999] and 24 entries involving related 1,2-diferrocenylethenes, 10 of which are (E)-isomers. The archetypal structure is (E)-1,2-diferrocenylethene (REBDAD, Deniill et al., 1996), in which the Cp rings of the ferrocenyl entities adopt an almost perfectly eclipsed arrangement. With the exception of 1-(1’-benzoylferroceny)2-ferrocenylethene and 1-(1’-(4-methoxy-benzoyl)ferrocenyl)-2-ferrocenylethene (OJUWUN and OJUXAU, Roemer et al., 2016), in which the ferrocenyl Cp rings lie close to a staggered arrangement, all of the other structures of molecules with the (E)-configuration display Cp arrangements that are much closer to eclipsed than observed for (E)-1 (ACUVAV, Mata & Peris, 2001; IBAXAM, DeHope et al., 2011; IVOSER, Skibar et al., 2004; JANJAJ, Dong et al., 1989; OJUXEY, Roemer et al., 2016; QICKIWI, Chen et al., 2000; REBDAD, WIMYOH, Nagahora et al., 2007, Roemer & Lentz, 2008, Farrugia et al., 2009). The 1,1-diferrocenylethene structure also has eclipsed Cp rings. The two staggered (E)-configured examples have a bulky substituent on one of the distal Cp rings; those with a less bulky Cp substituent have the eclipsed arrangement. Interestingly, (E)-1 has no Cp substituents yet the Cp ring arrangement deviates significantly from eclipsed. The degree of eclipsing of the Cp conformations found among the molecules with the (Z)-configuration, two of which have a cyclopropene ring as the ethene bridge (AMODIP, Klimova, Berestneva, Ramirez et al., 2003; EQOMIG, Klimova, Berestneva, Cinquantini et al., 2003), is more varied (AMODOV and AMODUB, Klimova, Berestneva, Ramirez et al., 2003; BADDAM, Beletskaya et al., 2001, Solntsev et al., 2011; JAJYIF and JAJYOL, García, Flores-Alamo, Flores & Klimova, 2017; KIGQUO, Klimova et al., 2013; LUFCERW, García et al., 2014; QASPEI, QATDAT and QATDEX, García, Flores-Alamo, Ortiz-Frade & Klimova, 2017; QICKOC, Chen et al., 2000; TUIDE1, Klimova et al., 2009).

direction (Fig. 3). Finally, C10—H of the substituted Cp ring interacts intramolecularly with the \( \pi \)-system of the furan ring at \(-x + 1, -y + 1, -z + 1 \) on the opposite side of the molecule. This latter interaction is quite short, but has a sharp angle at the H atom (Table 1), so the arrangement might just be a consequence of the molecular conformation. The molecular inversion symmetry, in combination with the two types of intermolecular interactions, links the molecules into a three-dimensional supramolecular framework.
Table 2  
Experimental details.

| Crystal data |  |
| Chemical formula | [Fe₂(C₅H₅)₂(C₂₀H₁₄O₂)] |
| Mᵣ | 528.19 |
| Crystal system, space group | Monoclinic, P2₁/n |
| Temperature (K) | 160 |
| a, b, c (Å) | 5.81066 (13), 22.7138 (5), 8.38031 (18) |
| β (°) | 91.785 (2) |
| V (Å³) | 1105.40 (4) |
| Z | 2 |
| Radiation type | Mo Kα |
| μ (mm⁻¹) | 1.34 |
| Crystal size (mm) | 0.20 × 0.16 × 0.08 |
| Data collection | Oxford Diffraction SuperNova, dual-radiation diffractometer |
| Absorption correction | Multi-scan (CrysAlis PRO: Rigaku OD, 2015) |
| Tₘᵡₜ, Tₘₜₜ | 0.895, 1.000 |
| No. of measured, independent and observed [I > 2σ(I)] reflections | 13881, 3021, 2612 |
| Rᵣᵣ | 0.026 |
| (sinθ/λ)max (Å⁻¹) | 0.708 |
| Refinement | R[F² > 2σ(F²)], wR(F²), S |
| No. of reflections | 3021 |
| No. of parameters | 154 |
| H-atom treatment | H-atom parameters constrained |
| Δρmax, Δρmin (e Å⁻³) | 0.49, -0.36 |

Computer programs: CrysAlis PRO (Rigaku OD, 2015). SHELX2014 (Sheldrick, 2015b). ORTEPII (Johnson, 1976). Mercury (Macrae et al., 2008), PLATON (Spek, 2015) and pubICIF (Westrip, 2010).

δ (ppm): 68.5, 68.9 [2 signals for 8CH(Fc)], 69.6 [10CH(Fc)], 85.4 [2C(Fc)], 109.1, 111.1, 140.8 [3 signals for 6CH(Fur)], 129.1 (C=C), 153.3 [2C(Fur)], ESI–MS (mixture of isomers): 528 (100, [M⁺]), 529 (50, [M + 1⁺]). Elemental analysis calculated for C₄₀H₉₀Fe₂O₂ (528.20): C 80.62, H 4.58%; found: C 80.27, H 4.41%.

6. Refinement
Crystal data, data collection and structure refinement details are summarized in Table 2. All H atoms were placed in geometrically calculated positions and were constrained to ride on their parent atom with C—H = 0.95 Å and with Uiso(H) = 1.2Ueq(C).

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Anthony Linden, Róża Hamera-Faludyga, Grzegorz Młostóń and Heinz Heimgartner

Computing details

Data collection: CrysAlis PRO (Rigaku OD, 2015); cell refinement: CrysAlis PRO (Rigaku OD, 2015); data reduction: CrysAlis PRO (Rigaku OD, 2015); program(s) used to solve structure: SHELXT2014 (Sheldrick, 2015a); program(s) used to refine structure: SHELXL2014 (Sheldrick, 2015b); molecular graphics: ORTEPII (Johnson, 1976) and Mercury (Macrae et al., 2006); software used to prepare material for publication: SHELXL2014 (Sheldrick, 2015b), PLATON (Spek, 2015) and publCIF (Westrip, 2010).

(E)-1,2-Diferrocenyl-1,2-bis(furan-2-yl)ethene

Crystal data

\[ \text{[Fe}_2(\text{C}_5\text{H}_5)\text{C}_2\text{H}_4\text{O}_2]\]  
\( \text{Mr} = 528.19 \)  
Monoclinic, \( P2_1/n \)  
\( a = 5.81006 (13) \) Å  
\( b = 22.7138 (5) \) Å  
\( c = 8.38031 (18) \) Å  
\( \beta = 91.785 (2)° \)  
\( V = 1105.40 (4) \) Å³  
\( Z = 2 \)

\( F(000) = 544 \)
\( D_\text{x} = 1.587 \text{ Mg m}^{-3} \)
Mo \( K\alpha \) radiation, \( \lambda = 0.71073 \) Å
Cell parameters from 7365 reflections
\( \theta = 3.0–29.9° \)
\( \mu = 1.34 \text{ mm}^{-1} \)
\( T = 160 \) K
Tablet, orange
0.20 × 0.16 × 0.08 mm

Data collection

Oxford Diffraction SuperNova, dual radiation diffractometer
Radiation source: SuperNova (Mo) X-ray Source
Mirror monochromator
Detector resolution: 10.3801 pixels mm⁻¹
\( \omega \) scans
Absorption correction: multi-scan
(CrysAlisPro; Rigaku OD, 2015)

\( T_{\text{min}} = 0.895, T_{\text{max}} = 1.000 \)
13881 measured reflections
3021 independent reflections
2612 reflections with \( I > 2\sigma(I) \)
\( R_{\text{int}} = 0.026 \)
\( \theta_{\text{max}} = 30.2°, \theta_{\text{min}} = 2.6° \)
\( h = -8→7 \)
\( k = -31→31 \)
\( l = -11→11 \)

Refinement

Refinement on \( F^2 \)
Least-squares matrix: full
\( R[F^2 > 2\sigma(F^2)] = 0.032 \)
\( wR(F^2) = 0.077 \)
\( S = 1.04 \)
3021 reflections
154 parameters
0 restraints

Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
\( w = 1/[\sigma^2(F_c^2) + (0.0291P)^2 + 0.9916P] \)
where \( P = (F_c^2 + 2F_e^2)/3 \)
\( \langle \Delta/\sigma \rangle_{\text{max}} = 0.001 \)
\( \Delta\rho_{\text{max}} = 0.49 \text{ e Å}^{-3} \)
\( \Delta\rho_{\text{min}} = -0.36 \text{ e Å}^{-3} \)
**Special details**

**Experimental.** Data collection and full structure determination done by Prof. Anthony Linden:  
anthony.linden@chem.uzh.ch  
Solvent used: hexane / dichloromethane  
Cooling Device: Oxford Instruments Cryojet XL  
Crystal mount: on a glass fibre  
Frames collected: 1290  
Seconds exposure per frame: 10.0  
Degrees rotation per frame: 1602.0  
Crystal-detector distance (mm): 55.0

**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     | \(U_{eq}^*/U_{eq}\) |
|----|-------|-------|-------|-----------------|
| Fe1| 0.46176 (4) | 0.65950 (2) | 0.41357 (3) | 0.01767 (8) |
| O1 | 0.5980 (2) | 0.54191 (6) | 0.79034 (16) | 0.0295 (3) |
| C1 | 0.5597 (3) | 0.52572 (7) | 0.5053 (2) | 0.0159 (3) |
| C2 | 0.7063 (3) | 0.53842 (7) | 0.6485 (2) | 0.0187 (3) |
| C3 | 0.7668 (5) | 0.55207 (10) | 0.9050 (3) | 0.0408 (6) |
| H3 | 0.7408 | 0.5562 | 1.0158 | 0.049* |
| C4 | 0.9717 (4) | 0.55529 (10) | 0.8395 (3) | 0.0418 (6) |
| H4 | 1.1150 | 0.5624 | 0.8938 | 0.050* |
| C5 | 0.9354 (3) | 0.54589 (8) | 0.6699 (2) | 0.0256 (4) |
| H5 | 1.0487 | 0.5451 | 0.5906 | 0.031* |
| C6 | 0.5501 (3) | 0.57204 (7) | 0.38168 (19) | 0.0168 (3) |
| C7 | 0.7351 (3) | 0.61094 (7) | 0.3435 (2) | 0.0212 (3) |
| H7 | 0.8841 | 0.6111 | 0.3937 | 0.025* |
| C8 | 0.6590 (4) | 0.64909 (8) | 0.2187 (2) | 0.0264 (4) |
| H8 | 0.7483 | 0.6788 | 0.1701 | 0.032* |
| C9 | 0.4265 (4) | 0.63519 (8) | 0.1791 (2) | 0.0262 (4) |
| H9 | 0.3324 | 0.6541 | 0.0998 | 0.031* |
| C10 | 0.3583 (3) | 0.58793 (7) | 0.2787 (2) | 0.0200 (3) |
| H10 | 0.2106 | 0.5700 | 0.2772 | 0.024* |
| C11 | 0.4528 (4) | 0.68024 (9) | 0.6520 (2) | 0.0295 (4) |
| H11 | 0.5075 | 0.6563 | 0.7382 | 0.035* |
| C12 | 0.5838 (3) | 0.72260 (9) | 0.5685 (2) | 0.0310 (4) |
| H12 | 0.7412 | 0.7320 | 0.5891 | 0.037* |
| C13 | 0.4377 (4) | 0.74812 (8) | 0.4493 (2) | 0.0286 (4) |
| H13 | 0.4798 | 0.7778 | 0.3758 | 0.034* |
| C14 | 0.2176 (3) | 0.72175 (8) | 0.4587 (2) | 0.0267 (4) |
| H14 | 0.0866 | 0.7305 | 0.3922 | 0.032* |
| C15 | 0.2268 (3) | 0.68009 (8) | 0.5840 (2) | 0.0268 (4) |
| H15 | 0.1028 | 0.6562 | 0.6169 | 0.032* |

**Atomic displacement parameters (Å²)**

|     | \(U_{11}\) | \(U_{22}\) | \(U_{33}\) | \(U_{12}\) | \(U_{13}\) | \(U_{23}\) |
|-----|--------|--------|--------|--------|--------|--------|
| Fe1 | 0.02204 (13) | 0.01261 (12) | 0.01843 (13) | 0.00067 (9) | 0.00192 (9) | -0.00138 (9) |
| O1  | 0.0359 (8) | 0.0287 (7) | 0.0239 (7) | 0.0074 (6) | 0.0035 (6) | -0.0018 (6) |
### Geometric parameters (Å, °)

| Bond/Angle | Distance/Angle | Distance/Angle |
|------------|----------------|----------------|
| Fe1—C7     | 2.0352 (17)    | C5—H5          | 0.9500         |
| Fe1—C8     | 2.0380 (18)    | C6—C10         | 1.434 (2)      |
| Fe1—C13    | 2.0404 (18)    | C6—C7          | 1.435 (2)      |
| Fe1—C9     | 2.0456 (18)    | C7—C8          | 1.419 (3)      |
| Fe1—C12    | 2.0457 (19)    | C7—H7          | 0.9500         |
| Fe1—C14    | 2.0465 (18)    | C8—C9          | 1.417 (3)      |
| Fe1—C11    | 2.0555 (19)    | C8—H8          | 0.9500         |
| Fe1—C10    | 2.0591 (17)    | C9—C10         | 1.424 (3)      |
| Fe1—C15    | 2.0605 (18)    | C9—H9          | 0.9500         |
| Fe1—C6     | 2.0712 (16)    | C10—H10        | 0.9500         |
| O1—C2      | 1.365 (2)      | C11—C15        | 1.415 (3)      |
| O1—C3      | 1.371 (3)      | C11—C12        | 1.424 (3)      |
| C1—C1i     | 1.360 (3)      | C11—H11        | 0.9500         |
| C1—C6      | 1.476 (2)      | C12—C13        | 1.415 (3)      |
| C1—C2      | 1.478 (2)      | C12—H12        | 0.9500         |
| C2—C5      | 1.349 (2)      | C13—C14        | 1.416 (3)      |
| C3—C4      | 1.329 (4)      | C13—H13        | 0.9500         |
| C3—H3      | 0.9500         | C14—C15        | 1.414 (3)      |
| C4—C5      | 1.446 (3)      | C14—H14        | 0.9500         |
| C4—H4      | 0.9500         | C15—H15        | 0.9500         |
| C7—Fe1—C8  | 40.77 (7)      | C10—C6—C7     | 106.52 (15)    |
| C7—Fe1—C13 | 129.40 (8)     | C10—C6—C1     | 127.83 (15)    |
| C8—Fe1—C13 | 105.96 (8)     | C7—C6—C1      | 125.64 (15)    |
| C7—Fe1—C9  | 68.43 (8)      | C10—C6—Fe1    | 69.23 (9)      |
| C8—Fe1—C9  | 40.59 (8)      | C7—C6—Fe1     | 68.20 (9)      |
| C13—Fe1—C9 | 113.70 (8)     | C1—C6—Fe1     | 126.65 (11)    |
| C7—Fe1—C12 | 107.73 (8)     | C8—C7—C6     | 108.76 (16)    |
| C8—Fe1—C12 | 113.42 (8)     | C8—C7—Fe1    | 69.73 (10)     |
| C13—Fe1—C12 | 40.51 (8)    | C6—C7—Fe1    | 70.89 (9)      |
| Bond                  | Bond Length (Å) | Bond Angle (°)   |
|----------------------|-----------------|-----------------|
| C9—Fe1—C12          | 145.21 (8)      |                 |
| C7—Fe1—C14          | 168.36 (8)      |                 |
| C8—Fe1—C14          | 129.68 (8)      |                 |
| C13—Fe1—C14         | 40.56 (8)       |                 |
| C9—Fe1—C14          | 108.34 (8)      |                 |
| C12—Fe1—C14         | 68.12 (8)       |                 |
| C7—Fe1—C11          | 116.63 (8)      |                 |
| C8—Fe1—C11          | 146.90 (9)      |                 |
| C13—Fe1—C11         | 68.12 (8)       |                 |
| C9—Fe1—C11          | 172.39 (9)      |                 |
| C12—Fe1—C11         | 40.64 (8)       |                 |
| C14—Fe1—C11         | 67.88 (8)       |                 |
| C7—Fe1—C10          | 68.35 (7)       |                 |
| C8—Fe1—C10          | 68.30 (7)       |                 |
| C13—Fe1—C10         | 147.05 (8)      |                 |
| C9—Fe1—C10          | 40.58 (7)       |                 |
| C12—Fe1—C10         | 172.33 (8)      |                 |
| C14—Fe1—C10         | 117.03 (7)      |                 |
| C11—Fe1—C10         | 134.35 (8)      |                 |
| C7—Fe1—C15          | 149.65 (8)      |                 |
| C8—Fe1—C15          | 169.52 (8)      |                 |
| C13—Fe1—C15         | 67.94 (8)       |                 |
| C9—Fe1—C15          | 132.74 (8)      |                 |
| C12—Fe1—C15         | 67.98 (8)       |                 |
| C14—Fe1—C15         | 40.27 (8)       |                 |
| C11—Fe1—C15         | 40.22 (8)       |                 |
| C10—Fe1—C15         | 111.80 (8)      |                 |
| C7—Fe1—C6           | 40.91 (7)       |                 |
| C8—Fe1—C6           | 68.75 (7)       |                 |
| C13—Fe1—C6          | 169.58 (8)      |                 |
| C9—Fe1—C6           | 68.58 (7)       |                 |
| C12—Fe1—C6          | 132.19 (8)      |                 |
| C14—Fe1—C6          | 149.60 (7)      |                 |
| C11—Fe1—C6          | 111.05 (7)      |                 |
| C10—Fe1—C6          | 40.64 (6)       |                 |
| C15—Fe1—C6          | 118.64 (7)      |                 |
| C2—O1—C3            | 106.35 (17)     |                 |
| C1—C1—C6            | 123.93 (19)     |                 |
| C1—C1—C2            | 120.04 (19)     |                 |
| C6—C1—C2            | 116.02 (14)     |                 |
| C5—C2—O1            | 110.90 (16)     |                 |
| C5—C2—C1            | 132.40 (16)     |                 |
| O1—C2—C1            | 116.67 (15)     |                 |
| C4—C3—O1            | 110.49 (19)     |                 |
| C4—C3—H3            | 124.8           |                 |
| O1—C3—H3            | 124.8           |                 |
| C3—C4—C5            | 107.03 (18)     |                 |
| C3—C4—H4            | 126.5           |                 |

*Acta Cryst.* (2018). E74, 625-629
C5—C4—H4  126.5  C14—C15—H15  126.0
C2—C5—C4  105.22 (18)  C11—C15—H15  126.0
C2—C5—H5  127.4  Fe1—C15—H15  126.6
C4—C5—H5  127.4
C3—O1—C2—C5  0.1 (2)  Fe1—C8—C9—C10  −59.76 (12)
C3—O1—C2—C1  178.20 (15)  C7—C8—C9—Fe1  59.30 (12)
C1i—C1—C2—C5  115.8 (2)  C8—C9—C10—C6  0.0 (2)
C1i—C1—C2—C5  −65.4 (2)  Fe1—C9—C10—C6  −59.30 (12)
C1i—C1—C2—O1  −61.8 (2)  C8—C9—C10—Fe1  59.26 (13)
C4—C5—C4—C3  0.4 (2)  Fe1—C8—C9—C10  −59.67 (12)
C3—C4—C5—C2  0.7 (2)  C7—C6—C10—C9  0.52 (19)
C1—C1—C6—C10  32.9 (3)  Fe1—C11—C12—C13  59.52 (13)
C1—C1—C6—C10  −145.89 (16)  C15—C11—C12—C13  −0.2 (2)
C2—C1—C6—C10  123.9 (2)  C11—C12—C13—C14  −59.70 (13)
C2—C1—C6—C7  33.1 (2)  C12—C13—C14—Fe1  −59.84 (13)
C2—C1—C6—Fe1  −54.85 (19)  C12—C13—C14—C15  0.3 (2)
C10—C6—C7—C8  −0.81 (19)  Fe1—C13—C14—C15  59.79 (13)
C1—C6—C7—C8  −179.96 (15)  C12—C13—C14—Fe1  60.05 (13)
Fe1—C6—C7—C8  −58.86 (11)  C12—C13—C14—C15  −59.78 (13)
C10—C6—C7—C8  58.86 (11)  Fe1—C11—C12—C13  −59.11 (13)
C1—C6—C7—Fe1  −120.29 (16)  C13—C14—C15—Fe1  −59.48 (13)
C6—C7—C8—Fe1  0.8 (2)  C12—C11—C15—C14  0.3 (2)
Fe1—C7—C8—C9  −59.60 (13)  Fe1—C11—C15—C14  −58.88 (13)
C6—C7—C8—Fe1  60.40 (12)  C12—C11—C15—Fe1  59.22 (13)
C7—C8—C9—C10  −0.5 (2)

Symmetry code: (i) −x+1, −y+1, −z+1.

Hydrogen-bond geometry (Å, °)

Cg1, Cg2 and Cg3 are the centroids of the C6–C10, C11–C15 and C2/O1/C3–C5 rings, respectively.

|       | D—H  |   D···A  |   D···A  |   D—H···A  |
|-------|------|----------|----------|------------|
| C3—H3···Cg1  | 0.95 | 2.81     | 3.686 (3) | 153        |
| C8—H8···Cg2  | 0.95 | 2.85     | 3.764 (2) | 161        |
| C10—H10···Cg3  | 0.95 | 2.68     | 3.2097 (18) | 116      |

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