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Ferrocene carboxylic anhydride: identification of a new polymorph

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Synopsis A new polymorph of ferrocenecarboxylic anhydride was obtained, characterized and its structural features were compared to the other cyclopentadienyl carboxylic anhydrides.

Abstract A new polymorph of ferrocenecarboxylic anhydride which crystallise in the monoclinic crystalline system was obtained. Three molecules are present in the asymmetric unit, two of them nearly identical (RMSD 0.1726) with the iron atoms on the same side of the anhydride functional group. In the third molecule, the two iron atoms are located at opposite sides of the functional group (ferrocene/ferrocene torsion angle 146.2 °), a very unusual feature in metallocene anhydrides. An network of intermolecular weak hydrogen bonds was also disclosed.

Keywords: crystal structure; ferrocene; acid anhydride; hydrogen bonds; polymorph

1. Introduction

Anhydrides are a well-known class of acylation reagents widely used in organic synthesis. While they are usually prepared intentionally for synthetic use, serendipity sometimes joins the game and leads to unexpected new compounds, as it is the case for ferrocene carboxylic anhydride (**1**, Scheme 1). It was first reported in 1959 by Lau and Hart during studies to access ferrocene peroxide (Lau & Hart, 1959). By refluxing ferrocene carboxylic chloride (FcCOCl) with sodium peroxide, they isolated **1**, which was characterized by its melting point, IR spectrum and elemental analysis. The same year, Acton and Silverstein obtained **1** during the synthesis of ferrocene carboxamides from ferrocene carboxylic acid (FcCO₂H) in the presence of dicyclohexylcarbodiimide (Acton & Silverstein, 1959). Reference samples

were further prepared by the reaction of FcCOCl with water in the presence of pyridine. In 1966, Kupchik and Kiesel reported the unexpected formation of **1** during the reaction of FcCOCl with tributyl-tin hydride, supposed to deliver the corresponding ferrocenecarboxaldehyde or ester (Kupchik & Kiesel, 1966). A reference sample was also prepared, that time by reacting FcCOCl with FcCO₂Na. It is almost 40 years later that Runqiu and Qingmin reported the formation of **1** (up to 47%) during the synthesis of FcCOCl in the conditions described by Lau and Hart (Runqiu & Qingmin, 2001). In addition to the previously reported melting point, IR spectrum and elemental analysis, the ¹H NMR spectrum and mass analysis of ferrocene carboxylic anhydride were described. In 2004, Schetter and Speiser also reported the formation of **1** during the reaction of FcCO₂H with various carbodiimides (Schetter & Speiser, 2004). Finally, in 2016, McAdam and Simpson reported that **1** was isolated during the reaction of FcCO₂H with triphosgene as a by-product (McAdam & Simpson, 2016). In this case the crystals suitable for X-ray analysis were obtained and the solid-state structure has been determined.

Apart from these synthesis reports, **1** was sporadically used as a reagent. Wenzler was the first one to use it for the esterification of cholesterol in the presence of pyridine (Hoffmann *et al.*, 1980). In 1990, Eckert reported the amidation of H-Phe-O*t*Bu in the presence of trimethylamine and the reaction of **1** with bovine serum albumin in buffered solution (Eckert & Koller, 1990). In 2004, Kihara and Takata used **1** in the synthesis of a redox-active rotaxane (Kihara *et al.*, 2004). A ferrocene stopper was introduced by the esterification of an alcohol in the presence of catalytic amounts of *n*Bu₃P. The same year, Schetter and Speiser reported the amidation of 3-(triethoxysilyl)propylamine with **1** in the presence of a carbodiimide in 37% yield (Schetter & Speiser, 2004). Finally, in 2012, Burrows and Marken reported the post-functionalization of metal-organic frameworks with **1** to access new redox-active materials (Halls *et al.*, 2012). It is to note that, apart from the work of Kihara and Tanaka, the yields of ester and amide formations remain surprisingly low (20 to 47%). This lack of reactivity was also noticed by Runqiu and Qingmin who failed to react **1** with primary or secondary amines and with hydrazines (Runqiu & Qingmin, 2001).

It appears that **1** is usually obtained as a by-product in the synthesis of FcCOCl with various chlorinating reagents or during the formation of amides in the presence of carbodiimides. In the course of a program dedicated to the synthesis of new ferrocene carboxamides (Palabindela *et al.*, 2016), we reacted FcCO₂H with thionyl chloride to access FcCOCl. During the purification steps, column chromatography afforded an unknown product, and ¹H and ¹³C NMR studies revealed the presence of a monosubstituted ferrocene with a carboxyl group, which was not an acid, an acyl chloride, nor an ester. While the possibility of a stable anhydride raised in our minds, slow crystallization of the pure product occurred, and the solid-state structure obtained by X-ray diffraction confirmed our hypothesis. However, instead of the known crystal structure already described by McAdam and Simpson (McAdam & Simpson, 2016), we managed to obtain a previously unknown polymorph of **1**. Its spectroscopic data and crystal structure will be described here and compared to other cyclopentadienyl (Cp) carboxylic anhydrides.

2. Experimental

2.1. Synthesis and crystallization

Dimethylformamide (0.1 ml) and SOCl_2 (6.4 ml, 88 mmol, 2.2 eq.) were added dropwise to a suspension of FcCO_2H (9.2 g, 40 mmol, 1.0 eq.) in dry dichloromethane (110 ml) at room temperature (Scheme 2). After 15 min, the reaction mixture was concentrated under vacuum and the residue was dissolved in dry dichloromethane (150 ml) before diisopropylamine (17 ml, 120 mmol, 3.0 eq.) was added dropwise. The reaction mixture was then stirred at room temperature until reaction completion. HCl (1M) was added until pH 1 and the reaction mixture was extracted with EtOAc. The combined organic layers were dried over MgSO_4 , filtrated and concentrated under vacuum to give the crude product. Purification by column chromatography, eluting with heptane/EtOAc (80:20) afforded the desired FcCOCl as the main product and the new ferrocene carboxylic anhydride polymorph **1B** by crystallization upon standing (0.18 g, 2%). Mp 113–116 °C.

2.2. Spectroscopic analyses

IR spectra were taken on a Perkin-Elmer Spectrum 100 spectrometer. ^1H and ^{13}C Nuclear Magnetic Resonance (NMR) spectra were recorded on a Bruker Avance III at respectively 500 MHz and 125 MHz. ^1H chemical shifts (δ) are given in ppm relative to the solvent residual peak and ^{13}C chemical shifts are relative to the central peak of the solvent signal. Peak assignments were based on two-dimensional experiments. ν_{max} (film)/ cm^{-1} 2919, 1768 (C=O, *sym*), 1713 (C=O, *asym*), 1444, 1243, 1067, 1044, 1007. ^1H NMR (500.0 MHz, CDCl_3): δ (ppm) 4.88 (t, $J = 1.9$ Hz, 4H, 4 x Fc CH), 4.54 (t, $J = 1.9$ Hz, 4H, 4 x Fc CH), 4.35 (s, 10H, 10 x Fc CH). ^{13}C NMR (125.7 MHz, CDCl_3): δ (ppm) 167.9 (2 x C=O), 72.9 (4 x Fc CH), 71.0 (4 x Fc CH), 70.4 (10 x Fc CH), 69.6 (2 x Fc C).

2.3. Refinement

The crystal structure was solved in the non-centrosymmetric Cc space group by dual-space algorithm using the *SHELXT* program (Sheldrick, 2015), and then refined with full-matrix least-square methods based on F^2 (*SHELXL*) (Sheldrick, 2015). The use of combined TWIN and BASF instructions led to a Flack parameter value of 0.21(3), corresponding to a mixture of ~80% of a particular enantiomer and ~20% of the opposite one. All non-hydrogen atoms were refined with anisotropic atomic displacement parameters. H atoms were included in calculated positions. In the absence of restraints or constraints on ADP's of C51-C55 ring atoms, some B and C alerts appeared in the CHECKCIF procedure, due to large values of ADP max/min ratios, especially on C52 (ratio = 4.2) and C55 (ratio=3.5). Introduction of restraints by RIGU instructions applied on these C51-C55 atoms in SHELXL refinement led to decrease these ADP max/min ratios (4.0 and 3.2 respectively) and provided more realistic anisotropic atomic displacements on this Cp ring. No constraints were used to refine the other rings. CCDC 1553140.

3. Results and discussion

The molecular structures of previously described by Simpson orthorhombic polymorph (**1A**, Figure 1, top) obtained and the monoclinic polymorph (**1B**, Figure 1, bottom) feature various differences and the crystallographic data of both polymorphs are disclosed in Table 1. The crystal packing of **1B** is shown in Figure 2. Only one molecule is present in the asymmetric unit of **1A**, while three independent molecules crystallized in the unit of **1B**. It can be seen that the anhydride molecules comprising the Fe1/Fe2 and Fe5/Fe6 atoms appear nearly identical by a 180 ° rotation around the b axis. This is confirmed by their molecular overlay (Mercury automatic overlay module was used), with the metal atom (Fe1/Fe2 and Fe5/Fe6) located on the same side of the anhydride (Figure 3, bottom, RMSD of 0.1726). However, much larger difference can be seen with the remaining anhydride molecule in which the two metal atoms (namely Fe3 and Fe4) are at the opposite sides on the anhydride, as confirmed by the overlay of molecules containing Fe1-Fe2 and Fe3-Fe4 (Figure 4, top right, RMSD 2.0862) and molecules containing Fe3-Fe4 and Fe5-Fe6 (Figure 4, top left, RMSD 2.1374). A packing similarity search (Mercury Solid Form module used, 20% distance tolerance, 20 ° angle tolerance, H-position and bound type ignored, structure inversion allowed) revealed one hit between the known orthorhombic polymorph **1A** and the anhydride molecules containing the Fe1 and Fe2 atoms of the new monoclinic polymorph **1B**. Indeed, as shown in figure 4, a moderate overlap can be seen with a RMSD of 0.624.

Figure 4 clearly shows why an intramolecular hydrogen bond occurred on **1A** (C206-H206...O1) and not on **1B** (C2-H2...O15): both H...A distances (**1A**: 2.56 Å, **1B**: 3.34) and D-H...A angles (**1A**: 148.9°, **1B**: 140.9) are more favorable to develop these weak interactions in **1A**. In addition to this intramolecular hydrogen bond, three intermolecular (C205-H205...O2, C204-H204...O1, C207-H207...O2) contacts, supporting the zig-zag chains of molecules along the a axis, were observed by the authors in **1A**. In **1B**, although no traditional H-bond donors are present, each carbonyl group can act as a weak hydrogen bond acceptor (Steiner, 2002; Desiraju, 2011). Although no zig-zag chains as present in **1A** could be seen in **1B**, we here identified a set of weak hydrogen bonds (Table 2). Along the b and c axes, four hydrogen bonds structure the packing (C85-H85...O42, C49-H49...O72, C31-H31...O12, C5-H5...O45) whereas three other ones (C6-H6...O15, C39-H39...O45 and C69-H69...O75) link molecules along the a axis [symmetry codes: (i) $x-1/2, -y+1/2, z+1/2$; (ii) $x, -y+1, z-1/2$; (iii) $x-1, y, z$; (iv) $x+1, y, z$.] (Figure 5).

It is difficult to assess if the large Fc-Fc torsion angle (146.2°), placing the two ferrocene moieties at opposite sides of the anhydride group in one of the molecules of **1B**, is it the result or the origin of the C31-H31...O12 hydrogen bond. Therefore, to get more information on this large angle, we also compared the new polymorph **1B** to the five other examples of Cp-based carboxylic anhydrides for which a solid-state structure was reported (Scheme 3): trovacencarboxylic anhydride (**2**, Elschenbroich *et al.*, 1997, NEWFIE, CCDC 1219005), 1,1'-((*tert*-butoxycarbonyl)amino)ferrocenecarboxylic

anhydride (**3**, Siebler *et al.*, 2010, KAJBUU, CCDC 769441), ruthenocenecarboxylic anhydride (**4**, Micallef *et al.*, 2011, IVOYOI, CCDC 794550) and the ferrocenecyclophane **5** (Liu *et al.*, 2015, HOVYEV, CCDC 1038498). Our goal was therefore to see if the structure of **1B** was closer to the free rotating anhydrides **2** and **4** or to geometrically restrained ones such as **3** (due to the presence of a hydrogen bond between the two *tert*-butoxycarbonyl groups) and **5** (cyclophane structure).

In **1B**, the torsion angles measured for the three anhydride molecules (O12-C11...C14-O15, O42-C41...C44-O45 and O72-C71...C74-O75) are lower than the those observed for the free rotating anhydrides **1A**, **2** and **4** (Table 3). Furthermore, the torsion angles between the two ferrocenyl cores of each anhydride molecule of **1B** are outside the usual range of values. This suggests that the polymorph **1B** is closer to geometrically restrained metallocene anhydrides than to the polymorph **1A**.

Intramolecular dihedral angles between the Cp rings are in good agreement with those observed for the other derivatives. However, two Cp rings of one ferrocenyl core of **1B** are clearly eclipsed with a C40-Cg3b...Cg3a-C35 torsion angle of 17.1°. This results from the hydrogen bond C31-H31...O12, which mimics the interactions observed in the 1,1'-disubstituted ferrocene-based anhydride **3**. For all six ferrocenyl cores, the angles between Cp ring planes are within the expected range of values, indicating the almost coplanarity of the Cp rings.

The angles between each carbonyl group and their respective Cp ring were finally compared. A comparison of the torsion angles of **1A-5** clearly shows that, as the geometry is constrained (intramolecular hydrogen bond in **3**, cyclophane structure in **5**), high values are observed (**3**: 19.4°, **5**: 21.7°). Such a torsion might be viewed as another mechanism to relieve steric strain. The torsion angles measured on the three molecules that crystallize in **1B** are between the ones of the unconstrained anhydrides (**1A**, **2**, **4**) and those of the constrained **3** and **5**. Accordingly, this suggests that steric strain exists at a higher degree in the new monoclinic polymorph, when compared to the orthorhombic one. Therefore, this higher strain in **1B** might have an effect on the thermodynamic stability of the polymorph.

Indeed, the density rule, as introduced by Burger and Ramberger (Burger & Ramberger, 1979), states that, at absolute zero, the most stable polymorph will have the highest density. However, the density difference between **1A** and **1B** (1.661 and 1.667 g/cm³ for **1A** and **1B**, respectively) is too small to apply this rule. Therefore, it appears difficult to clearly state which polymorph is here the more stable, although the lower melting point of **1B** (113-116 °C), when compared to **1A** (140-141°C), suggests a higher stability for the orthorhombic polymorph **1A**.

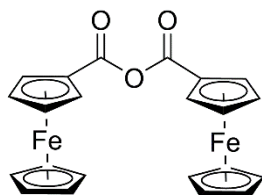
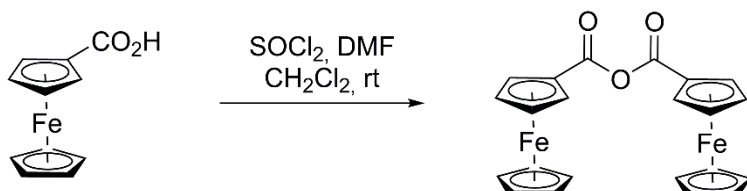
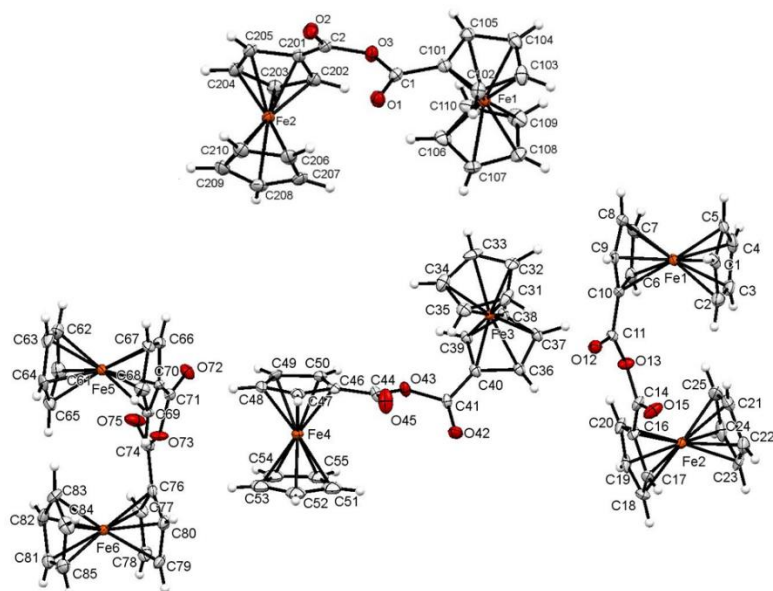
Scheme 1 Structure of ferrocenecarboxylic anhydride **1**.**Scheme 2** Reaction scheme.**Figure 1** Structures of the orthorhombic polymorph (**1A**, top) and the monoclinic polymorph (**1B**, bottom). Thermal ellipsoids shown at the 30% probability level.

Figure 2 Partial packing view of the monoclinic polymorph **1B** showing the hydrogen bonds as dashed red lines. Hydrogen not implicated in hydrogen bonds were omitted for clarity.

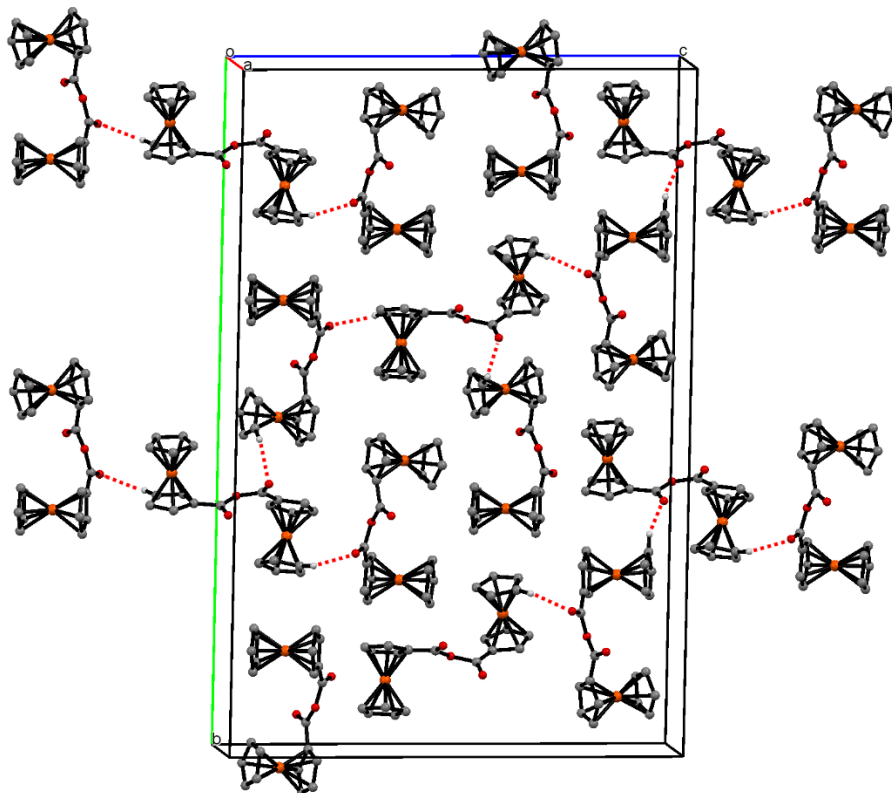


Figure 3 Overlay of molecules containing Fe3-Fe4 (light grey) and Fe5-Fe6 (light blue), top left; Overlay of molecules containing Fe1-Fe2 (light purple) and Fe3-Fe4 (light grey), top right; Overlay of molecules containing Fe1-Fe2 (light purple) and Fe5-Fe6 (light blue), bottom.

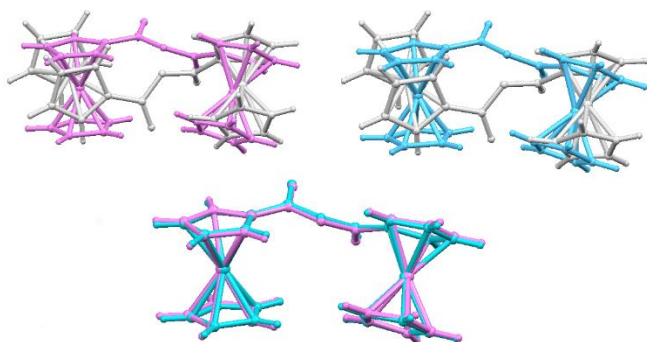


Figure 4 Overlay of the orthorhombic polymorph **1A** (light blue) and the monoclinic polymorph **1B** (black and grey). Hydrogen were omitted for clarity.

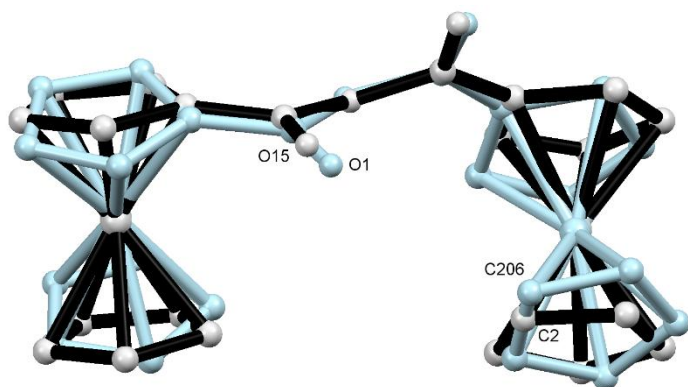
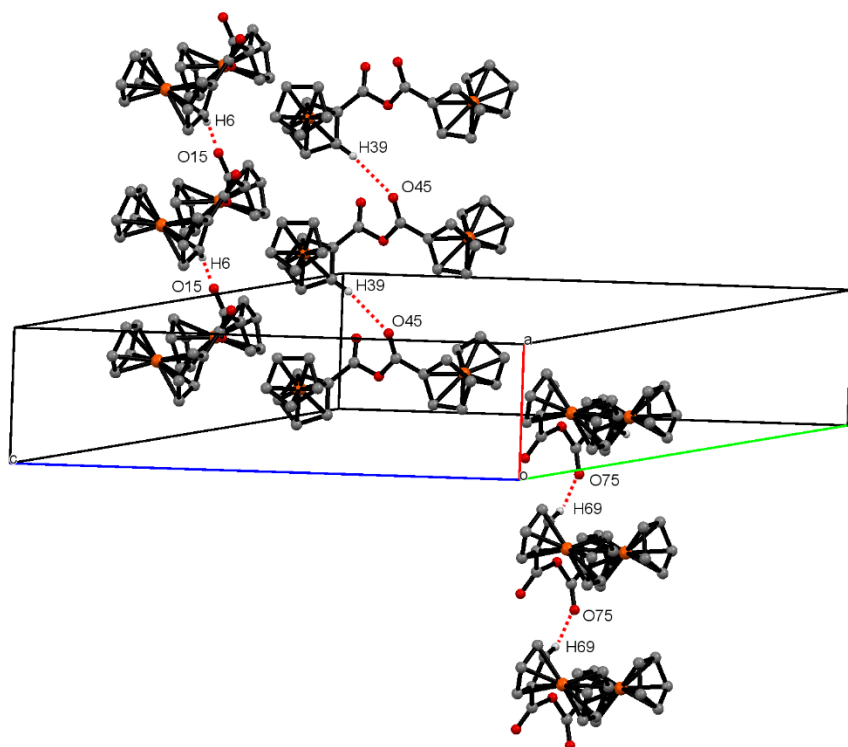


Figure 5 Chains of molecules of the monoclinic polymorph **1B** along the a axis showing the hydrogen bonds as dashed red lines. Hydrogen not implicated in hydrogen bonds were omitted for clarity.



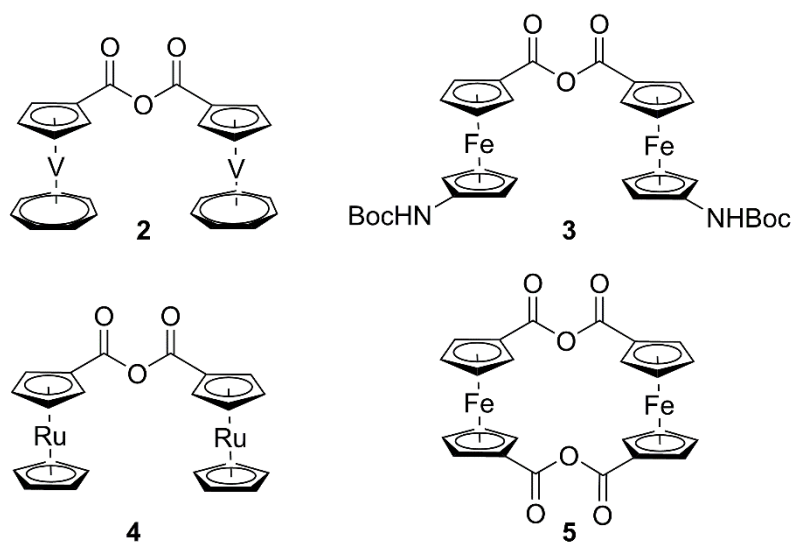
Scheme 3 Examples of Cp-based carboxylic anhydrides. Boc = CO₂tBu.

Table 1 Experimental details.

	1B^a	1A^b
Crystal data		
Chemical formula	C ₂₂ H ₁₈ Fe ₂ O ₃	C ₂₂ H ₁₈ Fe ₂ O ₃
<i>M_r</i>	442.06	442.06
Crystal system, space group	Monoclinic, <i>Cc</i>	Orthorhombic, <i>Pbca</i>
Temperature (K)	150	100
<i>a</i> , <i>b</i> , <i>c</i> (Å)	5.9758 (5), 36.456 (3), 24.363 (2)	14.8773 (2), 12.2499 (2), 19.3288 (3)
β (°)	91.997 (3)	
<i>V</i> (Å ³)	5304.4 (8)	3522.59 (9)
<i>Z</i>	12	8
Radiation type	Mo <i>K</i> α	Cu <i>K</i> α
μ (mm ⁻¹)	1.66	13.38
Crystal size (mm)	0.56 × 0.36 × 0.30	0.16 × 0.13 × 0.11
Data collection		
Diffractometer	APEXII, Bruker-AXS	Agilent SuperNova, Dual, Cu at zero, Atlas
Absorption correction	Multi-scan [Sheldrick, G.M. (2014). <i>SADABS</i> Bruker AXS Inc., Madison, Wisconsin, USA]	Gaussian (<i>CrysAlis PRO</i> ; Agilent, 2014)
<i>T_{min}</i> , <i>T_{max}</i>	0.486, 0.608	0.267, 0.456
No. of measured, independent and observed [<i>I</i> > 2σ(<i>I</i>)] reflections	16323, 10804, 10350	13584, 3468, 3262
<i>R_{int}</i>	0.023	0.040
(sin θ/λ) _{max} (Å ⁻¹)	0.649	0.621
Refinement		
<i>R</i> [<i>F</i> ² > 2σ(<i>F</i> ²)], <i>wR</i> (<i>F</i> ²), <i>S</i>	0.049, 0.120, 1.11	0.030, 0.090, 0.84
No. of reflections	10804	3468
No. of parameters	730	244
No. of restraints	32	0
H-atom treatment	H-atom parameters constrained	H-atom parameters constrained
Weighting scheme	$w = 1/[\sigma^2(F_o^2) + (0.0346P)^2 + 44.5361P]$ where $P = (F_o^2 + 2F_c^2)/3$	$w = 1/[\sigma^2(F_o^2) + (0.089P)^2 + 0.2107P]$ where $P = (F_o^2 + 2F_c^2)/3$
Δρ _{max} , Δρ _{min} (e Å ⁻³)	0.96, -0.87	0.31, -0.68
Absolute structure	Flack <i>x</i> determined using 4113 quotients [(<i>I</i> ₊)-(<i>I</i> ₋)]/[(<i>I</i> ₊)+(<i>I</i> ₋)] (Parsons et al., 2013).	
Absolute structure parameter	0.204 (11)	

^a This work. ^b Data taken from McAdam & Simpson, 2016.

Computer programs: Bruker APEX2 (Bruker, 2014), SHELXT (Sheldrick, 2015), SHELXL2016/6 (Sheldrick, 2015), SXGRAPH (Farrugia, 1999), Mercury (CSD, 2016), CRYSCALC (T. Roisnel, local program, 2017).

Table 2 Hydrogen bonds geometry (Å, °) for **1B**.

<i>D</i> — <i>H</i> ... <i>A</i>	<i>D</i> — <i>H</i> (Å)	<i>H</i> ... <i>A</i> (Å)	<i>D</i> ... <i>A</i> (Å)	<i>D</i> — <i>H</i> ... <i>A</i> (°)
C49—H49...O72	0.95	2.64	3.517 (11)	152.9
C31—H31...O12	0.95	2.49	3.387 (10)	157.9
C5—H5...O45 ⁱ	0.95	2.57	3.368 (11)	142.3
C85—H85...O42 ⁱⁱ	0.95	2.57	3.438 (12)	151.7
C6—H6...O15 ⁱⁱⁱ	0.95	2.33	3.256 (11)	164.1
C39—H39...O45 ⁱⁱⁱ	0.95	2.65	3.595 (11)	170.4
C69—H69...O75 ^{iv}	0.95	2.35	3.273 (10)	163.9

Symmetry code(s): (i) *x*-1/2, -*y*+1/2, *z*+1/2; (ii) *x*, -*y*+1, *z*-1/2; (iii) *x*-1, *y*, *z*; (iv) *x*+1, *y*, *z*.

Table 3 Selected geometric parameters of the Cp-based carboxylic anhydrides compared (°).

Compound	Anhydride torsion angle	Fc/Fc torsion angle	Cp/Cp eclipse angle	Cp/Cp planes angle	O-C-C-C torsion angle
1A	57.4 (2)	72.2	6.3–5.3	2.84–1.75	12.1 (3)–2.1 (3)
2	62.4 (5)	75.1	n.a.	1.96–2.52	11.7 (9)–6.2 (9)
2	66.7 (5)	103.7	n.a.	2.61–2.71	9.8 (9)–6.2 (9)
3	57.0 (2)	85.9	18.9–7.0	2.31–4.55	0.7 (4)–19.4 (5)
4	62.3 (3)	72.9	12.8–3.2	2.19–2.03	3.6 (5)–4.2 (5)
5	27.5 (2)	1.5	0.6–0.4	2.51–2.51	8.3 (4)–21.7 (4)
1B	50.9 (7)	33.6	7.3–2.5	1.33–1.72	16 (1)–1 (1)
1B	46.6 (7)	146.3	17.1–0.7	0.89–2.51	14 (1)–7 (1)
1B	47.0 (7)	30.1	3.9–6.2	0.39–2.22	2 (1)–16 (1)

n.a. Not applicable.

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Supporting information

Ferrocenecarboxylic anhydride: identification of a new polymorph

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Computing details

Data collection: Bruker *APEX2* (Bruker, 2014). Cell refinement: Bruker *APEX2* (Bruker, 2014). Data reduction: Bruker *APEX2* (Bruker, 2014). Program(s) used to solve structure: *SHELXT* (Sheldrick, 2015). Program(s) used to refine structure: *SHELXL2016/6* (Sheldrick, 2015). Molecular graphics: *SXGRAPH* (Farrugia, 1999), *Mercury* (CSD, 2016). Software used to prepare material for publication: *CRYSCALC* (T. Roisnel, local program, 2017).

Ferrocenecarboxylic anhydride, monoclinic polymorph (1B)

Crystal data

$C_{22}H_{18}Fe_2O_3$	$F(000) = 2712$
$M_r = 442.06$	$D_x = 1.661 \text{ Mg m}^{-3}$
Monoclinic, <i>Cc</i>	Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$
$a = 5.9758 (5) \text{ \AA}$	Cell parameters from 9522 reflections
$b = 36.456 (3) \text{ \AA}$	$\theta = 2.2\text{--}27.5^\circ$
$c = 24.363 (2) \text{ \AA}$	$\mu = 1.66 \text{ mm}^{-1}$
$\beta = 91.997 (3)^\circ$	$T = 150 \text{ K}$
$V = 5304.4 (8) \text{ \AA}^3$	Prism, red
$Z = 12$	$0.56 \times 0.36 \times 0.30 \text{ mm}$

Data collection

APEXII, Bruker-AXS diffractometer	10804 independent reflections
Radiation source: fine-focus sealed tube	10350 reflections with $I > 2\sigma(I)$
Graphite monochromator	$R_{\text{int}} = 0.023$
CCD rotation images, thin slices scans	$\theta_{\text{max}} = 27.5^\circ$, $\theta_{\text{min}} = 3.4^\circ$
Absorption correction: multi-scan [Sheldrick, G.M. (2014). <i>SADABS</i> Bruker AXS Inc., Madison, Wisconsin, USA]	$h = -6 \rightarrow 7$
$T_{\text{min}} = 0.486$, $T_{\text{max}} = 0.608$	$k = -46 \rightarrow 41$
16323 measured reflections	$l = -31 \rightarrow 31$

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.049$	$w = 1/[\sigma^2(F_o^2) + (0.0346P)^2 + 44.5361P]$
$wR(F^2) = 0.120$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.11$	$(\Delta/\sigma)_{\text{max}} = 0.006$
10804 reflections	$\Delta_{\text{max}} = 0.96 \text{ e \AA}^{-3}$
730 parameters	$\Delta_{\text{min}} = -0.87 \text{ e \AA}^{-3}$
32 restraints	Absolute structure: Flack x determined using 4113 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ (Parsons et al., 2013).
	Absolute structure parameter: 0.204 (11)

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. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2 , conventional R-factors R are based on F , with F set to zero for negative F^2 . The threshold expression of $F^2 > 2\sigma(F^2)$ is used only for calculating R-factors(gt) etc. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F , and R-factors based on ALL data will be even larger. Crystal structure was solved in the non-centrosymmetric *Cc* space group by dual-space algorithm using the *SHELXT* program (Sheldrick, 2015), and then refined with full-

matrix least-square methods based on F^2 (SHELXL) (Sheldrick, 2015). The use of combined TWIN and BASF instructions led to a Flack parameter value of 0.21(3), corresponding to a mixture of ~80% of a particular enantiomer and ~20% of the opposite one. All non-hydrogen atoms were refined with anisotropic atomic displacement parameters. H atoms were finally included in their calculated positions. A final refinement on F^2 with 10804 unique intensities and 683 parameters converged at $\omega R(F^2) = 0.1201$ ($R(F) = 0.0500$) for 10350 observed reflections with $I > 2\sigma(I)$. In the absence of restraints or constraints on ADP's of C51-C55 ring atoms, some B and C alerts appeared in the CHECKCIF procedure, due to large values of ADP max/min ratios, especially on C52 (ratio = 4.2) and C55 (ratio=3.5). Introduction of restraints with the help of RIGU instructions applied on these C51-C55 atoms in SHELXL refinement lead to decrease these ADP max/min ratios (4.0 and 3.2 respectively) and provided more realistic anisotropic atomic displacements on this CP ring. No constraints were used to refine the other cycles. CCDC 1553140.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2) for **1B**

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
Fe1	0.66891 (17)	0.25136 (3)	0.87909 (4)	0.0139 (2)
Fe2	0.77526 (18)	0.42693 (3)	0.87935 (5)	0.0148 (2)
C1	0.9177 (15)	0.2316 (3)	0.9297 (3)	0.0214 (18)
H1	1.039949	0.217060	0.918465	0.026*
C2	0.9158 (18)	0.2706 (3)	0.9328 (4)	0.027 (2)
H2	1.035426	0.286713	0.924769	0.033*
C3	0.6988 (17)	0.2808 (3)	0.9502 (3)	0.026 (2)
H3	0.647367	0.305183	0.955349	0.031*
C4	0.5740 (17)	0.2487 (3)	0.9584 (3)	0.029 (2)
H4	0.424254	0.247739	0.970201	0.035*
C5	0.7094 (16)	0.2178 (2)	0.9460 (3)	0.0210 (18)
H5	0.667498	0.192737	0.948308	0.025*
C6	0.4812 (14)	0.2820 (2)	0.8246 (3)	0.0200 (17)
H6	0.414827	0.305149	0.831873	0.024*
C7	0.3812 (15)	0.2473 (3)	0.8312 (4)	0.0234 (19)
H7	0.235068	0.242840	0.843808	0.028*
C8	0.5414 (16)	0.2194 (2)	0.8151 (3)	0.0221 (19)
H8	0.516875	0.193697	0.814880	0.026*
C9	0.7366 (14)	0.2372 (2)	0.8001 (3)	0.0163 (16)
H9	0.869567	0.225584	0.788615	0.020*
C10	0.7017 (14)	0.2760 (2)	0.8050 (3)	0.0161 (16)
C11	0.8736 (14)	0.3030 (2)	0.7922 (3)	0.0163 (16)
O12	1.0359 (10)	0.29726 (16)	0.7668 (2)	0.0207 (12)
O13	0.8142 (10)	0.33824 (15)	0.8093 (3)	0.0217 (13)
C14	0.9835 (14)	0.3631 (2)	0.8213 (3)	0.0195 (17)
O15	1.1653 (11)	0.35399 (18)	0.8383 (3)	0.0305 (15)
C16	0.9037 (14)	0.4008 (2)	0.8148 (3)	0.0172 (16)
C17	1.0423 (15)	0.4320 (2)	0.8294 (3)	0.0195 (17)
H17	1.196249	0.431471	0.840486	0.023*
C18	0.9051 (17)	0.4633 (2)	0.8239 (4)	0.0245 (19)
H18	0.951471	0.487903	0.830795	0.029*
C19	0.6850 (17)	0.4521 (3)	0.8065 (3)	0.025 (2)
H19	0.560265	0.467821	0.799693	0.030*
C20	0.6854 (17)	0.4136 (2)	0.8010 (3)	0.0224 (19)
H20	0.560946	0.398776	0.789907	0.027*
C21	0.7724 (19)	0.3908 (3)	0.9427 (4)	0.030 (2)
H21	0.828580	0.366423	0.940968	0.036*
C22	0.8980 (19)	0.4225 (3)	0.9590 (4)	0.029 (2)
H22	1.050634	0.423020	0.971250	0.035*
C23	0.752 (2)	0.4529 (3)	0.9536 (4)	0.034 (2)
H23	0.789809	0.477738	0.961338	0.040*
C24	0.5451 (18)	0.4402 (3)	0.9352 (4)	0.032 (2)
H24	0.417746	0.455135	0.927437	0.038*
C25	0.5507 (17)	0.4018 (3)	0.9295 (4)	0.030 (2)
H25	0.428947	0.386264	0.918850	0.036*
Fe3	0.46790 (17)	0.31133 (3)	0.63680 (4)	0.0132 (2)
Fe4	0.57217 (18)	0.40600 (3)	0.37638 (5)	0.0152 (2)
C31	0.7071 (16)	0.2726 (2)	0.6569 (4)	0.0227 (18)
H31	0.828759	0.275357	0.682735	0.027*
C32	0.4892 (15)	0.2595 (2)	0.6684 (4)	0.0201 (18)
H32	0.439277	0.252079	0.703314	0.024*
C33	0.3617 (16)	0.2594 (2)	0.6191 (4)	0.028 (2)
H33	0.210478	0.251486	0.614932	0.034*

C34	0.4936 (16)	0.2728 (3)	0.5763 (4)	0.026 (2)
H34	0.447073	0.275965	0.538896	0.031*
C35	0.7097 (15)	0.2808 (2)	0.6001 (4)	0.0209 (17)
H35	0.834051	0.289959	0.581136	0.025*
C36	0.5523 (15)	0.3609 (2)	0.6721 (3)	0.0164 (16)
H36	0.692184	0.367483	0.689045	0.020*
C37	0.3664 (15)	0.3455 (2)	0.6989 (3)	0.0190 (17)
H37	0.360340	0.339900	0.736928	0.023*
C38	0.1902 (14)	0.3400 (2)	0.6585 (3)	0.0187 (17)
H38	0.047652	0.329672	0.665024	0.022*
C39	0.2629 (13)	0.3525 (2)	0.6072 (3)	0.0169 (16)
H39	0.176490	0.352878	0.573769	0.020*
C40	0.4902 (14)	0.3646 (2)	0.6147 (3)	0.0164 (16)
C41	0.6414 (14)	0.3784 (2)	0.5741 (3)	0.0161 (16)
O42	0.8139 (10)	0.39413 (16)	0.5815 (2)	0.0216 (12)
O43	0.5498 (9)	0.37181 (16)	0.5205 (2)	0.0190 (12)
C44	0.6921 (14)	0.3607 (2)	0.4809 (3)	0.0194 (17)
O45	0.8792 (11)	0.3501 (2)	0.4903 (3)	0.0338 (16)
C46	0.5811 (14)	0.3617 (2)	0.4260 (3)	0.0155 (16)
C47	0.6961 (15)	0.3536 (2)	0.3771 (4)	0.0193 (17)
H47	0.848487	0.346451	0.375191	0.023*
C48	0.5423 (16)	0.3582 (2)	0.3315 (3)	0.0222 (18)
H48	0.573453	0.354949	0.293882	0.027*
C49	0.3298 (15)	0.3686 (2)	0.3535 (3)	0.0210 (17)
H49	0.195783	0.373183	0.332542	0.025*
C50	0.3543 (15)	0.3708 (2)	0.4114 (3)	0.0186 (17)
H50	0.240775	0.377121	0.436083	0.022*
C51	0.728 (2)	0.4484 (3)	0.4167 (5)	0.042 (3)
H51	0.794032	0.447592	0.452742	0.050*
C52	0.8336 (18)	0.4405 (2)	0.3678 (5)	0.040 (2)
H52	0.985518	0.433183	0.365231	0.048*
C53	0.6819 (17)	0.4449 (2)	0.3231 (4)	0.030 (2)
H53	0.711781	0.441284	0.285396	0.036*
C54	0.4798 (17)	0.4557 (2)	0.3444 (4)	0.029 (2)
H54	0.345988	0.460690	0.323551	0.035*
C55	0.5059 (19)	0.4579 (2)	0.4023 (4)	0.033 (2)
H55	0.393152	0.464518	0.426996	0.039*
Fe5	0.33501 (18)	0.34823 (3)	0.12474 (5)	0.0149 (2)
Fe6	0.23840 (17)	0.51994 (3)	0.12024 (5)	0.0141 (2)
C61	0.0971 (16)	0.3689 (3)	0.0706 (4)	0.027 (2)
H61	-0.020178	0.385277	0.079383	0.032*
C62	0.0846 (17)	0.3301 (3)	0.0714 (4)	0.027 (2)
H62	-0.041745	0.315848	0.080699	0.033*
C63	0.2935 (19)	0.3164 (3)	0.0559 (4)	0.029 (2)
H63	0.331225	0.291233	0.052889	0.035*
C64	0.4396 (16)	0.3463 (3)	0.0456 (3)	0.0231 (19)
H64	0.590475	0.344795	0.034735	0.028*
C65	0.3135 (17)	0.3792 (3)	0.0547 (4)	0.025 (2)
H65	0.366353	0.403639	0.050700	0.030*
C66	0.2659 (15)	0.3323 (2)	0.2028 (3)	0.0186 (17)
H66	0.134022	0.320193	0.213841	0.022*
C67	0.4667 (17)	0.3153 (2)	0.1860 (3)	0.0232 (19)
H67	0.492770	0.289679	0.183875	0.028*
C68	0.6207 (14)	0.3431 (2)	0.1732 (4)	0.0193 (17)
H68	0.768299	0.339291	0.161127	0.023*
C69	0.5191 (14)	0.3778 (2)	0.1813 (3)	0.0165 (16)
H69	0.585792	0.401015	0.175376	0.020*
C70	0.3007 (14)	0.3715 (2)	0.1998 (3)	0.0146 (16)
C71	0.1346 (14)	0.3983 (2)	0.2152 (3)	0.0170 (16)
O72	-0.0276 (10)	0.39263 (17)	0.2416 (2)	0.0228 (13)
O73	0.1945 (10)	0.43343 (15)	0.1985 (3)	0.0214 (13)
C74	0.0267 (14)	0.4584 (2)	0.1851 (3)	0.0177 (17)
O75	-0.1611 (12)	0.45020 (19)	0.1717 (3)	0.0354 (17)
C76	0.1160 (15)	0.4959 (2)	0.1878 (3)	0.0193 (18)
C77	-0.0157 (15)	0.5279 (2)	0.1725 (4)	0.0204 (18)
H77	-0.170912	0.527990	0.162657	0.024*
C78	0.1251 (16)	0.5585 (2)	0.1748 (4)	0.0244 (19)

H78	0.082101	0.583131	0.167091	0.029*
C79	0.3463 (16)	0.5462 (2)	0.1908 (4)	0.0225 (19)
H79	0.475076	0.561301	0.195205	0.027*
C80	0.3404 (15)	0.5080 (2)	0.1988 (3)	0.0205 (18)
H80	0.463836	0.492877	0.209564	0.025*
C81	0.2522 (17)	0.5454 (3)	0.0460 (4)	0.0248 (19)
H81	0.217587	0.570332	0.038518	0.030*
C82	0.0983 (16)	0.5157 (3)	0.0433 (4)	0.028 (2)
H82	-0.056737	0.517241	0.033526	0.034*
C83	0.2178 (18)	0.4835 (3)	0.0577 (4)	0.028 (2)
H83	0.155281	0.459558	0.059418	0.034*
C84	0.4430 (17)	0.4923 (3)	0.0691 (4)	0.028 (2)
H84	0.558490	0.475534	0.079598	0.033*
C85	0.4682 (17)	0.5313 (3)	0.0620 (4)	0.026 (2)
H85	0.602518	0.545006	0.066910	0.032*

Atomic displacement parameters (Å²) for 1B

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Fe1	0.0178 (6)	0.0130 (5)	0.0110 (5)	0.0014 (5)	0.0012 (4)	0.0008 (4)
Fe2	0.0194 (6)	0.0125 (5)	0.0123 (5)	0.0006 (5)	-0.0034 (4)	-0.0006 (4)
C1	0.022 (4)	0.026 (5)	0.017 (4)	0.004 (4)	-0.002 (3)	0.007 (3)
C2	0.036 (5)	0.024 (5)	0.021 (4)	0.001 (4)	-0.007 (4)	0.000 (4)
C3	0.040 (6)	0.021 (4)	0.016 (4)	0.003 (4)	0.001 (4)	-0.007 (3)
C4	0.030 (5)	0.043 (6)	0.014 (4)	0.001 (4)	0.007 (4)	-0.002 (4)
C5	0.033 (5)	0.018 (4)	0.011 (4)	0.002 (4)	0.002 (3)	0.004 (3)
C6	0.015 (4)	0.028 (5)	0.017 (4)	0.003 (3)	-0.002 (3)	0.003 (3)
C7	0.019 (4)	0.032 (5)	0.019 (4)	-0.006 (4)	0.002 (4)	0.009 (4)
C8	0.032 (5)	0.018 (4)	0.016 (4)	-0.009 (4)	-0.002 (4)	0.002 (3)
C9	0.022 (4)	0.020 (4)	0.007 (3)	0.003 (3)	0.002 (3)	0.000 (3)
C10	0.021 (4)	0.016 (4)	0.011 (3)	-0.006 (3)	0.001 (3)	0.001 (3)
C11	0.024 (4)	0.012 (4)	0.013 (4)	0.000 (3)	0.000 (3)	0.002 (3)
O12	0.022 (3)	0.020 (3)	0.020 (3)	-0.001 (2)	0.003 (2)	-0.002 (2)
O13	0.019 (3)	0.015 (3)	0.032 (3)	0.001 (2)	0.001 (3)	-0.002 (2)
C14	0.020 (4)	0.021 (4)	0.017 (4)	0.000 (3)	-0.002 (3)	-0.009 (3)
O15	0.024 (4)	0.023 (3)	0.043 (4)	0.004 (3)	-0.013 (3)	-0.010 (3)
C16	0.023 (4)	0.014 (4)	0.015 (4)	0.002 (3)	0.002 (3)	-0.005 (3)
C17	0.020 (4)	0.022 (4)	0.017 (4)	-0.008 (3)	0.003 (3)	0.000 (3)
C18	0.036 (5)	0.017 (4)	0.021 (4)	-0.009 (4)	0.002 (4)	0.005 (3)
C19	0.037 (5)	0.027 (5)	0.011 (4)	0.011 (4)	-0.005 (4)	0.005 (3)
C20	0.033 (5)	0.017 (4)	0.016 (4)	0.007 (4)	-0.007 (4)	-0.008 (3)
C21	0.045 (6)	0.028 (5)	0.018 (4)	-0.004 (4)	0.004 (4)	0.008 (4)
C22	0.039 (6)	0.031 (5)	0.016 (4)	-0.001 (4)	-0.004 (4)	0.001 (4)
C23	0.054 (7)	0.031 (5)	0.015 (4)	-0.007 (5)	-0.004 (4)	-0.011 (4)
C24	0.038 (6)	0.044 (6)	0.013 (4)	0.014 (5)	0.005 (4)	-0.002 (4)
C25	0.029 (5)	0.039 (6)	0.022 (4)	-0.014 (4)	0.005 (4)	-0.003 (4)
Fe3	0.0149 (5)	0.0121 (5)	0.0125 (5)	0.0012 (4)	-0.0014 (4)	-0.0003 (4)
Fe4	0.0190 (6)	0.0115 (5)	0.0153 (5)	-0.0025 (4)	0.0014 (4)	0.0001 (4)
C31	0.026 (5)	0.019 (4)	0.023 (4)	0.008 (3)	-0.011 (4)	-0.007 (3)
C32	0.028 (5)	0.008 (4)	0.024 (4)	0.004 (3)	-0.006 (4)	0.003 (3)
C33	0.027 (5)	0.011 (4)	0.045 (6)	-0.002 (3)	-0.011 (4)	-0.001 (4)
C34	0.028 (5)	0.025 (5)	0.023 (4)	0.006 (4)	-0.005 (4)	-0.005 (4)
C35	0.022 (4)	0.016 (4)	0.025 (4)	0.005 (3)	0.002 (3)	0.000 (3)
C36	0.024 (4)	0.013 (4)	0.013 (3)	0.004 (3)	-0.001 (3)	-0.004 (3)
C37	0.031 (5)	0.013 (4)	0.013 (4)	0.002 (3)	0.001 (3)	0.001 (3)
C38	0.022 (4)	0.017 (4)	0.018 (4)	0.002 (3)	0.014 (3)	0.007 (3)
C39	0.009 (4)	0.022 (4)	0.020 (4)	0.001 (3)	0.003 (3)	0.009 (3)
C40	0.015 (4)	0.024 (4)	0.010 (3)	0.005 (3)	-0.001 (3)	-0.004 (3)
C41	0.018 (4)	0.019 (4)	0.011 (3)	0.005 (3)	-0.003 (3)	0.002 (3)
O42	0.019 (3)	0.023 (3)	0.023 (3)	-0.005 (2)	0.001 (2)	0.003 (2)
O43	0.016 (3)	0.029 (3)	0.012 (3)	0.001 (2)	0.001 (2)	0.003 (2)
C44	0.023 (4)	0.021 (4)	0.015 (4)	0.008 (3)	0.005 (3)	0.005 (3)
O45	0.029 (4)	0.050 (4)	0.023 (3)	0.024 (3)	0.002 (3)	-0.002 (3)
C46	0.019 (4)	0.009 (3)	0.018 (4)	0.003 (3)	0.005 (3)	0.000 (3)
C47	0.025 (4)	0.010 (3)	0.023 (4)	-0.001 (3)	0.003 (3)	0.001 (3)
C48	0.037 (5)	0.014 (4)	0.015 (4)	-0.006 (4)	0.003 (4)	0.001 (3)
C49	0.020 (4)	0.020 (4)	0.022 (4)	-0.008 (3)	-0.008 (3)	-0.001 (3)
C50	0.023 (4)	0.013 (4)	0.020 (4)	-0.007 (3)	0.003 (3)	0.000 (3)

C51	0.061 (7)	0.021 (5)	0.042 (5)	-0.016 (5)	-0.015 (5)	-0.007 (4)
C52	0.031 (5)	0.013 (4)	0.076 (7)	-0.013 (4)	-0.002 (5)	0.005 (5)
C53	0.042 (5)	0.014 (4)	0.035 (5)	-0.006 (4)	0.015 (4)	0.003 (4)
C54	0.028 (5)	0.020 (4)	0.040 (5)	0.007 (4)	0.010 (4)	0.004 (4)
C55	0.053 (6)	0.008 (4)	0.039 (5)	-0.002 (4)	0.015 (4)	-0.008 (4)
Fe5	0.0181 (6)	0.0135 (5)	0.0133 (5)	-0.0010 (5)	0.0030 (4)	0.0004 (4)
Fe6	0.0159 (6)	0.0120 (5)	0.0143 (5)	-0.0006 (4)	-0.0026 (4)	-0.0012 (4)
C61	0.022 (5)	0.036 (5)	0.023 (4)	0.005 (4)	0.003 (4)	0.003 (4)
C62	0.028 (5)	0.035 (5)	0.018 (4)	-0.006 (4)	-0.004 (4)	0.001 (4)
C63	0.048 (6)	0.022 (5)	0.018 (4)	0.004 (4)	-0.003 (4)	-0.009 (4)
C64	0.027 (5)	0.033 (5)	0.010 (4)	-0.004 (4)	0.011 (3)	-0.001 (3)
C65	0.036 (5)	0.022 (5)	0.018 (4)	0.003 (4)	0.002 (4)	0.004 (3)
C66	0.025 (5)	0.014 (4)	0.017 (4)	-0.001 (3)	0.003 (3)	0.001 (3)
C67	0.037 (5)	0.016 (4)	0.016 (4)	-0.004 (4)	0.000 (4)	0.004 (3)
C68	0.015 (4)	0.019 (4)	0.024 (4)	0.003 (3)	0.004 (3)	0.002 (3)
C69	0.021 (4)	0.011 (4)	0.017 (4)	-0.006 (3)	0.003 (3)	-0.001 (3)
C70	0.020 (4)	0.011 (4)	0.013 (3)	-0.003 (3)	0.002 (3)	0.000 (3)
C71	0.021 (4)	0.015 (4)	0.014 (4)	-0.003 (3)	-0.002 (3)	0.001 (3)
O72	0.023 (3)	0.024 (3)	0.022 (3)	0.004 (2)	0.008 (3)	0.005 (2)
O73	0.019 (3)	0.010 (3)	0.035 (3)	-0.002 (2)	0.003 (3)	0.003 (2)
C74	0.016 (4)	0.018 (4)	0.020 (4)	-0.002 (3)	-0.001 (3)	0.005 (3)
O75	0.027 (4)	0.028 (4)	0.050 (4)	-0.010 (3)	-0.013 (3)	0.019 (3)
C76	0.028 (5)	0.021 (4)	0.009 (4)	0.003 (4)	0.003 (3)	0.000 (3)
C77	0.020 (4)	0.016 (4)	0.025 (4)	0.008 (3)	0.008 (4)	0.002 (3)
C78	0.034 (5)	0.018 (4)	0.021 (4)	0.005 (4)	0.002 (4)	-0.002 (3)
C79	0.029 (5)	0.020 (4)	0.018 (4)	-0.003 (4)	-0.007 (4)	-0.007 (3)
C80	0.022 (4)	0.024 (5)	0.015 (4)	0.001 (3)	-0.006 (3)	-0.001 (3)
C81	0.034 (5)	0.024 (5)	0.017 (4)	0.004 (4)	0.000 (4)	0.002 (3)
C82	0.023 (5)	0.045 (6)	0.017 (4)	-0.005 (4)	-0.005 (4)	-0.008 (4)
C83	0.040 (6)	0.022 (5)	0.023 (4)	-0.010 (4)	-0.003 (4)	-0.008 (4)
C84	0.032 (5)	0.024 (5)	0.028 (5)	0.013 (4)	0.008 (4)	0.001 (4)
C85	0.027 (5)	0.030 (5)	0.023 (4)	-0.005 (4)	0.006 (4)	-0.005 (4)

Geometric parameters (Å, °) for 1B

Fe1—C1	2.031 (9)	C36—H36	0.9500
Fe1—C10	2.031 (8)	C37—C38	1.431 (12)
Fe1—C4	2.034 (9)	C37—H37	0.9500
Fe1—C3	2.040 (8)	C38—C39	1.411 (10)
Fe1—C6	2.041 (8)	C38—H38	0.9500
Fe1—C5	2.046 (8)	C39—C40	1.433 (11)
Fe1—C9	2.047 (7)	C39—H39	0.9500
Fe1—C7	2.050 (9)	C40—C41	1.454 (11)
Fe1—C2	2.060 (10)	C41—O42	1.188 (10)
Fe1—C8	2.070 (9)	C41—O43	1.418 (9)
Fe2—C16	2.013 (8)	O43—C44	1.368 (9)
Fe2—C20	2.025 (8)	C44—O45	1.197 (10)
Fe2—C24	2.026 (10)	C44—C46	1.474 (11)
Fe2—C21	2.030 (9)	C46—C50	1.427 (12)
Fe2—C17	2.049 (9)	C46—C47	1.427 (11)
Fe2—C23	2.051 (9)	C47—C48	1.425 (12)
Fe2—C19	2.054 (8)	C47—H47	0.9500
Fe2—C22	2.058 (9)	C48—C49	1.446 (13)
Fe2—C25	2.061 (10)	C48—H48	0.9500
Fe2—C18	2.064 (9)	C49—C50	1.416 (12)
C1—C5	1.412 (13)	C49—H49	0.9500
C1—C2	1.426 (13)	C50—H50	0.9500
C1—H1	0.9500	C51—C52	1.398 (16)
C2—C3	1.427 (14)	C51—C55	1.405 (16)
C2—H2	0.9500	C51—H51	0.9500
C3—C4	1.406 (14)	C52—C53	1.403 (16)
C3—H3	0.9500	C52—H52	0.9500
C4—C5	1.425 (13)	C53—C54	1.387 (13)
C4—H4	0.9500	C53—H53	0.9500
C5—H5	0.9500	C54—C55	1.417 (14)
C6—C7	1.412 (13)	C54—H54	0.9500
C6—C10	1.435 (12)	C55—H55	0.9500
C6—H6	0.9500	Fe5—C70	2.033 (8)

C7—C8	1.458 (14)	Fe5—C69	2.040 (8)
C7—H7	0.9500	Fe5—C66	2.044 (8)
C8—C9	1.393 (12)	Fe5—C63	2.046 (9)
C8—H8	0.9500	Fe5—C65	2.047 (9)
C9—C10	1.435 (11)	Fe5—C61	2.048 (9)
C9—H9	0.9500	Fe5—C64	2.050 (8)
C10—C11	1.464 (11)	Fe5—C68	2.050 (9)
C11—O12	1.187 (10)	Fe5—C67	2.051 (9)
C11—O13	1.402 (9)	Fe5—C62	2.056 (10)
O13—C14	1.382 (10)	Fe6—C83	2.023 (9)
C14—O15	1.196 (11)	Fe6—C76	2.023 (8)
C14—C16	1.463 (11)	Fe6—C82	2.033 (9)
C16—C20	1.414 (13)	Fe6—C80	2.035 (8)
C16—C17	1.443 (11)	Fe6—C77	2.036 (8)
C17—C18	1.411 (13)	Fe6—C81	2.036 (9)
C17—H17	0.9500	Fe6—C84	2.042 (9)
C18—C19	1.428 (14)	Fe6—C79	2.051 (8)
C18—H18	0.9500	Fe6—C85	2.052 (9)
C19—C20	1.412 (12)	Fe6—C78	2.065 (9)
C19—H19	0.9500	C61—C65	1.414 (13)
C20—H20	0.9500	C61—C62	1.418 (14)
C21—C25	1.411 (15)	C61—H61	0.9500
C21—C22	1.427 (14)	C62—C63	1.407 (14)
C21—H21	0.9500	C62—H62	0.9500
C22—C23	1.414 (15)	C63—C64	1.423 (14)
C22—H22	0.9500	C63—H63	0.9500
C23—C24	1.382 (16)	C64—C65	1.439 (13)
C23—H23	0.9500	C64—H64	0.9500
C24—C25	1.408 (15)	C65—H65	0.9500
C24—H24	0.9500	C66—C67	1.423 (13)
C25—H25	0.9500	C66—C70	1.446 (11)
Fe3—C40	2.019 (8)	C66—H66	0.9500
Fe3—C33	2.040 (8)	C67—C68	1.412 (12)
Fe3—C32	2.044 (8)	C67—H67	0.9500
Fe3—C34	2.045 (9)	C68—C69	1.417 (11)
Fe3—C38	2.047 (8)	C68—H68	0.9500
Fe3—C39	2.053 (8)	C69—C70	1.414 (11)
Fe3—C35	2.054 (9)	C69—H69	0.9500
Fe3—C36	2.056 (8)	C70—C71	1.451 (11)
Fe3—C31	2.056 (9)	C71—O72	1.199 (10)
Fe3—C37	2.068 (8)	C71—O73	1.395 (9)
Fe4—C46	2.015 (8)	O73—C74	1.386 (10)
Fe4—C52	2.022 (9)	C74—O75	1.196 (10)
Fe4—C55	2.036 (8)	C74—C76	1.467 (12)
Fe4—C50	2.037 (8)	C76—C80	1.427 (13)
Fe4—C54	2.039 (9)	C76—C77	1.448 (11)
Fe4—C51	2.040 (10)	C77—C78	1.399 (13)
Fe4—C53	2.047 (9)	C77—H77	0.9500
Fe4—C47	2.048 (8)	C78—C79	1.437 (13)
Fe4—C49	2.052 (8)	C78—H78	0.9500
Fe4—C48	2.062 (8)	C79—C80	1.407 (12)
C31—C35	1.416 (12)	C79—H79	0.9500
C31—C32	1.424 (13)	C80—H80	0.9500
C31—H31	0.9500	C81—C82	1.419 (14)
C32—C33	1.401 (12)	C81—C85	1.430 (14)
C32—H32	0.9500	C81—H81	0.9500
C33—C34	1.416 (14)	C82—C83	1.413 (14)
C33—H33	0.9500	C82—H82	0.9500
C34—C35	1.427 (13)	C83—C84	1.402 (14)
C34—H34	0.9500	C83—H83	0.9500
C35—H35	0.9500	C84—C85	1.440 (13)
C36—C37	1.423 (12)	C84—H84	0.9500
C36—C40	1.442 (10)	C85—H85	0.9500
C1—Fe1—C10	127.3 (4)	C33—C34—Fe3	69.5 (5)
C1—Fe1—C4	68.2 (4)	C35—C34—Fe3	69.9 (5)
C10—Fe1—C4	154.6 (4)	C33—C34—H34	126.6

C1—Fe1—C3	68.3 (4)	C35—C34—H34	126.6
C10—Fe1—C3	120.9 (4)	Fe3—C34—H34	125.6
C4—Fe1—C3	40.4 (4)	C31—C35—C34	108.3 (8)
C1—Fe1—C6	164.5 (4)	C31—C35—Fe3	69.9 (5)
C10—Fe1—C6	41.3 (3)	C34—C35—Fe3	69.3 (5)
C4—Fe1—C6	118.6 (4)	C31—C35—H35	125.8
C3—Fe1—C6	107.1 (4)	C34—C35—H35	125.8
C1—Fe1—C5	40.5 (4)	Fe3—C35—H35	126.5
C10—Fe1—C5	163.7 (4)	C37—C36—C40	107.7 (7)
C4—Fe1—C5	40.9 (4)	C37—C36—Fe3	70.3 (5)
C3—Fe1—C5	68.5 (4)	C40—C36—Fe3	67.9 (5)
C6—Fe1—C5	153.2 (4)	C37—C36—H36	126.2
C1—Fe1—C9	108.5 (4)	C40—C36—H36	126.2
C10—Fe1—C9	41.2 (3)	Fe3—C36—H36	127.2
C4—Fe1—C9	162.1 (4)	C36—C37—C38	107.9 (7)
C3—Fe1—C9	156.5 (4)	C36—C37—Fe3	69.4 (5)
C6—Fe1—C9	69.5 (3)	C38—C37—Fe3	68.9 (4)
C5—Fe1—C9	125.2 (3)	C36—C37—H37	126.1
C1—Fe1—C7	154.4 (4)	C38—C37—H37	126.1
C10—Fe1—C7	68.3 (3)	Fe3—C37—H37	127.3
C4—Fe1—C7	106.3 (4)	C39—C38—C37	108.8 (7)
C3—Fe1—C7	124.6 (4)	C39—C38—Fe3	70.1 (4)
C6—Fe1—C7	40.4 (4)	C37—C38—Fe3	70.4 (5)
C5—Fe1—C7	119.0 (4)	C39—C38—H38	125.6
C9—Fe1—C7	68.6 (4)	C37—C38—H38	125.6
C1—Fe1—C2	40.8 (4)	Fe3—C38—H38	125.5
C10—Fe1—C2	108.9 (4)	C38—C39—C40	107.9 (7)
C4—Fe1—C2	68.3 (4)	C38—C39—Fe3	69.6 (5)
C3—Fe1—C2	40.7 (4)	C40—C39—Fe3	68.2 (4)
C6—Fe1—C2	126.2 (4)	C38—C39—H39	126.1
C5—Fe1—C2	68.6 (4)	C40—C39—H39	126.1
C9—Fe1—C2	121.5 (4)	Fe3—C39—H39	127.7
C7—Fe1—C2	162.5 (4)	C39—C40—C36	107.8 (7)
C1—Fe1—C8	120.0 (4)	C39—C40—C41	129.0 (7)
C10—Fe1—C8	67.8 (3)	C36—C40—C41	123.2 (8)
C4—Fe1—C8	125.5 (4)	C39—C40—Fe3	70.6 (5)
C3—Fe1—C8	162.7 (4)	C36—C40—Fe3	70.6 (5)
C6—Fe1—C8	68.8 (4)	C41—C40—Fe3	124.3 (6)
C5—Fe1—C8	107.1 (3)	O42—C41—O43	121.8 (7)
C9—Fe1—C8	39.6 (3)	O42—C41—C40	128.3 (7)
C7—Fe1—C8	41.5 (4)	O43—C41—C40	109.8 (7)
C2—Fe1—C8	155.0 (4)	C44—O43—C41	117.9 (6)
C16—Fe2—C20	41.0 (4)	O45—C44—O43	124.0 (7)
C16—Fe2—C24	157.4 (4)	O45—C44—C46	124.7 (8)
C20—Fe2—C24	121.8 (4)	O43—C44—C46	111.2 (7)
C16—Fe2—C21	107.4 (4)	C50—C46—C47	108.7 (7)
C20—Fe2—C21	123.5 (4)	C50—C46—C44	128.7 (7)
C24—Fe2—C21	67.7 (4)	C47—C46—C44	122.6 (7)
C16—Fe2—C17	41.6 (3)	C50—C46—Fe4	70.2 (4)
C20—Fe2—C17	69.4 (4)	C47—C46—Fe4	70.7 (5)
C24—Fe2—C17	159.8 (4)	C44—C46—Fe4	124.5 (6)
C21—Fe2—C17	122.5 (4)	C48—C47—C46	108.0 (8)
C16—Fe2—C23	161.2 (4)	C48—C47—Fe4	70.3 (5)
C20—Fe2—C23	156.8 (4)	C46—C47—Fe4	68.2 (4)
C24—Fe2—C23	39.6 (4)	C48—C47—H47	126.0
C21—Fe2—C23	68.1 (4)	C46—C47—H47	126.0
C17—Fe2—C23	124.1 (4)	Fe4—C47—H47	127.1
C16—Fe2—C19	68.4 (4)	C47—C48—C49	107.1 (7)
C20—Fe2—C19	40.5 (4)	C47—C48—Fe4	69.2 (5)
C24—Fe2—C19	108.1 (4)	C49—C48—Fe4	69.1 (5)
C21—Fe2—C19	159.8 (4)	C47—C48—H48	126.5
C17—Fe2—C19	68.4 (4)	C49—C48—H48	126.5
C23—Fe2—C19	122.2 (4)	Fe4—C48—H48	126.8
C16—Fe2—C22	124.5 (4)	C50—C49—C48	108.8 (8)
C20—Fe2—C22	160.9 (4)	C50—C49—Fe4	69.2 (5)
C24—Fe2—C22	67.3 (4)	C48—C49—Fe4	69.8 (5)
C21—Fe2—C22	40.9 (4)	C50—C49—H49	125.6

C17—Fe2—C22	108.0 (4)	C48—C49—H49	125.6
C23—Fe2—C22	40.3 (4)	Fe4—C49—H49	127.0
C19—Fe2—C22	157.7 (4)	C49—C50—C46	107.4 (8)
C16—Fe2—C25	121.7 (4)	C49—C50—Fe4	70.3 (5)
C20—Fe2—C25	107.2 (4)	C46—C50—Fe4	68.6 (5)
C24—Fe2—C25	40.3 (4)	C49—C50—H50	126.3
C21—Fe2—C25	40.3 (4)	C46—C50—H50	126.3
C17—Fe2—C25	158.2 (4)	Fe4—C50—H50	126.4
C23—Fe2—C25	67.5 (4)	C52—C51—C55	106.8 (10)
C19—Fe2—C25	123.7 (4)	C52—C51—Fe4	69.2 (6)
C22—Fe2—C25	67.9 (4)	C55—C51—Fe4	69.7 (5)
C16—Fe2—C18	68.4 (4)	C52—C51—H51	126.6
C20—Fe2—C18	68.4 (4)	C55—C51—H51	126.6
C24—Fe2—C18	124.4 (4)	Fe4—C51—H51	126.1
C21—Fe2—C18	158.1 (4)	C51—C52—C53	109.9 (10)
C17—Fe2—C18	40.1 (4)	C51—C52—Fe4	70.6 (6)
C23—Fe2—C18	108.6 (4)	C53—C52—Fe4	70.8 (5)
C19—Fe2—C18	40.6 (4)	C51—C52—H52	125.1
C22—Fe2—C18	122.6 (4)	C53—C52—H52	125.1
C25—Fe2—C18	160.3 (4)	Fe4—C52—H52	125.2
C5—C1—C2	109.3 (8)	C54—C53—C52	106.8 (10)
C5—C1—Fe1	70.3 (5)	C54—C53—Fe4	69.8 (5)
C2—C1—Fe1	70.7 (5)	C52—C53—Fe4	68.9 (6)
C5—C1—H1	125.3	C54—C53—H53	126.6
C2—C1—H1	125.3	C52—C53—H53	126.6
Fe1—C1—H1	125.2	Fe4—C53—H53	126.2
C1—C2—C3	106.5 (9)	C53—C54—C55	108.8 (10)
C1—C2—Fe1	68.5 (5)	C53—C54—Fe4	70.5 (5)
C3—C2—Fe1	68.9 (5)	C55—C54—Fe4	69.5 (5)
C1—C2—H2	126.8	C53—C54—H54	125.6
C3—C2—H2	126.7	C55—C54—H54	125.6
Fe1—C2—H2	127.4	Fe4—C54—H54	126.0
C4—C3—C2	108.5 (8)	C51—C55—C54	107.8 (9)
C4—C3—Fe1	69.6 (5)	C51—C55—Fe4	70.0 (6)
C2—C3—Fe1	70.4 (5)	C54—C55—Fe4	69.8 (5)
C4—C3—H3	125.7	C51—C55—H55	126.1
C2—C3—H3	125.7	C54—C55—H55	126.1
Fe1—C3—H3	125.9	Fe4—C55—H55	125.7
C3—C4—C5	108.6 (9)	C70—Fe5—C69	40.6 (3)
C3—C4—Fe1	70.0 (5)	C70—Fe5—C66	41.6 (3)
C5—C4—Fe1	70.0 (5)	C69—Fe5—C66	69.1 (3)
C3—C4—H4	125.7	C70—Fe5—C63	163.8 (4)
C5—C4—H4	125.7	C69—Fe5—C63	154.1 (4)
Fe1—C4—H4	125.9	C66—Fe5—C63	125.4 (4)
C1—C5—C4	107.0 (8)	C70—Fe5—C65	120.9 (3)
C1—C5—Fe1	69.2 (5)	C69—Fe5—C65	106.8 (4)
C4—C5—Fe1	69.1 (5)	C66—Fe5—C65	157.4 (4)
C1—C5—H5	126.5	C63—Fe5—C65	68.1 (4)
C4—C5—H5	126.5	C70—Fe5—C61	109.6 (4)
Fe1—C5—H5	126.7	C69—Fe5—C61	125.9 (4)
C7—C6—C10	107.2 (8)	C66—Fe5—C61	123.0 (4)
C7—C6—Fe1	70.1 (5)	C63—Fe5—C61	67.7 (4)
C10—C6—Fe1	69.0 (5)	C65—Fe5—C61	40.4 (4)
C7—C6—H6	126.4	C70—Fe5—C64	154.8 (3)
C10—C6—H6	126.4	C69—Fe5—C64	118.6 (4)
Fe1—C6—H6	126.0	C66—Fe5—C64	160.7 (4)
C6—C7—C8	108.1 (8)	C63—Fe5—C64	40.7 (4)
C6—C7—Fe1	69.5 (5)	C65—Fe5—C64	41.1 (4)
C8—C7—Fe1	70.0 (5)	C61—Fe5—C64	68.6 (4)
C6—C7—H7	126.0	C70—Fe5—C68	68.1 (3)
C8—C7—H7	126.0	C69—Fe5—C68	40.5 (3)
Fe1—C7—H7	126.1	C66—Fe5—C68	68.3 (4)
C9—C8—C7	108.1 (8)	C63—Fe5—C68	119.7 (4)
C9—C8—Fe1	69.4 (5)	C65—Fe5—C68	123.8 (4)
C7—C8—Fe1	68.5 (5)	C61—Fe5—C68	161.6 (4)
C9—C8—H8	125.9	C64—Fe5—C68	105.3 (4)
C7—C8—H8	125.9	C70—Fe5—C67	68.7 (3)

Fe1—C8—H8	127.7	C69—Fe5—C67	68.4 (3)
C8—C9—C10	108.0 (8)	C66—Fe5—C67	40.7 (4)
C8—C9—Fe1	71.1 (5)	C63—Fe5—C67	107.3 (4)
C10—C9—Fe1	68.8 (4)	C65—Fe5—C67	160.3 (4)
C8—C9—H9	126.0	C61—Fe5—C67	157.5 (4)
C10—C9—H9	126.0	C64—Fe5—C67	122.9 (4)
Fe1—C9—H9	125.7	C68—Fe5—C67	40.3 (3)
C6—C10—C9	108.6 (7)	C70—Fe5—C62	127.5 (4)
C6—C10—C11	128.8 (8)	C69—Fe5—C62	163.7 (4)
C9—C10—C11	122.7 (8)	C66—Fe5—C62	109.3 (4)
C6—C10—Fe1	69.8 (5)	C63—Fe5—C62	40.1 (4)
C9—C10—Fe1	70.0 (4)	C65—Fe5—C62	68.2 (4)
C11—C10—Fe1	125.2 (6)	C61—Fe5—C62	40.4 (4)
O12—C11—O13	122.4 (7)	C64—Fe5—C62	68.5 (4)
O12—C11—C10	125.8 (7)	C68—Fe5—C62	155.2 (4)
O13—C11—C10	111.5 (7)	C67—Fe5—C62	121.6 (4)
C14—O13—C11	118.3 (7)	C83—Fe6—C76	108.3 (4)
O15—C14—O13	122.8 (8)	C83—Fe6—C82	40.8 (4)
O15—C14—C16	126.0 (8)	C76—Fe6—C82	124.6 (4)
O13—C14—C16	111.1 (7)	C83—Fe6—C80	125.2 (4)
C20—C16—C17	108.5 (7)	C76—Fe6—C80	41.2 (4)
C20—C16—C14	129.0 (8)	C82—Fe6—C80	162.0 (4)
C17—C16—C14	122.1 (8)	C83—Fe6—C77	122.5 (4)
C20—C16—Fe2	70.0 (5)	C76—Fe6—C77	41.8 (3)
C17—C16—Fe2	70.5 (5)	C82—Fe6—C77	107.2 (4)
C14—C16—Fe2	119.5 (6)	C80—Fe6—C77	69.4 (4)
C18—C17—C16	106.8 (8)	C83—Fe6—C81	68.5 (4)
C18—C17—Fe2	70.5 (5)	C76—Fe6—C81	161.1 (4)
C16—C17—Fe2	67.8 (5)	C82—Fe6—C81	40.8 (4)
C18—C17—H17	126.6	C80—Fe6—C81	155.9 (4)
C16—C17—H17	126.6	C77—Fe6—C81	123.0 (4)
Fe2—C17—H17	126.6	C83—Fe6—C84	40.3 (4)
C17—C18—C19	108.8 (8)	C76—Fe6—C84	121.6 (4)
C17—C18—Fe2	69.4 (5)	C82—Fe6—C84	68.6 (4)
C19—C18—Fe2	69.4 (5)	C80—Fe6—C84	107.6 (4)
C17—C18—H18	125.6	C77—Fe6—C84	158.0 (4)
C19—C18—H18	125.6	C81—Fe6—C84	68.8 (4)
Fe2—C18—H18	127.2	C83—Fe6—C79	161.2 (4)
C20—C19—C18	108.0 (8)	C76—Fe6—C79	68.5 (4)
C20—C19—Fe2	68.6 (5)	C82—Fe6—C79	156.3 (4)
C18—C19—Fe2	70.1 (5)	C80—Fe6—C79	40.3 (3)
C20—C19—H19	126.0	C77—Fe6—C79	68.2 (4)
C18—C19—H19	126.0	C81—Fe6—C79	120.7 (4)
Fe2—C19—H19	126.9	C84—Fe6—C79	124.3 (4)
C19—C20—C16	108.0 (8)	C83—Fe6—C85	68.6 (4)
C19—C20—Fe2	70.9 (5)	C76—Fe6—C85	156.9 (4)
C16—C20—Fe2	69.0 (5)	C82—Fe6—C85	69.0 (4)
C19—C20—H20	126.0	C80—Fe6—C85	120.5 (4)
C16—C20—H20	126.0	C77—Fe6—C85	159.4 (4)
Fe2—C20—H20	125.7	C81—Fe6—C85	41.0 (4)
C25—C21—C22	108.3 (9)	C84—Fe6—C85	41.2 (4)
C25—C21—Fe2	71.0 (6)	C79—Fe6—C85	106.7 (4)
C22—C21—Fe2	70.6 (5)	C83—Fe6—C78	156.9 (4)
C25—C21—H21	125.9	C76—Fe6—C78	68.7 (3)
C22—C21—H21	125.9	C82—Fe6—C78	120.8 (4)
Fe2—C21—H21	124.1	C80—Fe6—C78	68.6 (4)
C23—C22—C21	107.1 (10)	C77—Fe6—C78	39.9 (4)
C23—C22—Fe2	69.6 (5)	C81—Fe6—C78	106.6 (4)
C21—C22—Fe2	68.5 (5)	C84—Fe6—C78	161.0 (4)
C23—C22—H22	126.5	C79—Fe6—C78	40.9 (4)
C21—C22—H22	126.5	C85—Fe6—C78	123.4 (4)
Fe2—C22—H22	126.9	C65—C61—C62	108.6 (8)
C24—C23—C22	108.0 (9)	C65—C61—Fe5	69.8 (5)
C24—C23—Fe2	69.2 (5)	C62—C61—Fe5	70.1 (5)
C22—C23—Fe2	70.1 (5)	C65—C61—H61	125.7
C24—C23—H23	126.0	C62—C61—H61	125.7
C22—C23—H23	126.0	Fe5—C61—H61	126.0

Fe2—C23—H23	126.3	C63—C62—C61	107.5 (9)
C23—C24—C25	110.0 (10)	C63—C62—Fe5	69.5 (5)
C23—C24—Fe2	71.2 (6)	C61—C62—Fe5	69.5 (5)
C25—C24—Fe2	71.2 (6)	C63—C62—H62	126.2
C23—C24—H24	125.0	C61—C62—H62	126.2
C25—C24—H24	125.0	Fe5—C62—H62	126.3
Fe2—C24—H24	124.2	C62—C63—C64	109.4 (9)
C24—C25—C21	106.6 (9)	C62—C63—Fe5	70.3 (5)
C24—C25—Fe2	68.5 (6)	C64—C63—Fe5	69.8 (5)
C21—C25—Fe2	68.6 (5)	C62—C63—H63	125.3
C24—C25—H25	126.7	C64—C63—H63	125.3
C21—C25—H25	126.7	Fe5—C63—H63	126.1
Fe2—C25—H25	127.7	C63—C64—C65	106.4 (8)
C40—Fe3—C33	149.2 (4)	C63—C64—Fe5	69.5 (5)
C40—Fe3—C32	170.3 (3)	C65—C64—Fe5	69.4 (5)
C33—Fe3—C32	40.1 (4)	C63—C64—H64	126.8
C40—Fe3—C34	117.4 (4)	C65—C64—H64	126.8
C33—Fe3—C34	40.6 (4)	Fe5—C64—H64	125.9
C32—Fe3—C34	68.4 (4)	C61—C65—C64	108.0 (8)
C40—Fe3—C38	68.8 (3)	C61—C65—Fe5	69.8 (5)
C33—Fe3—C38	106.3 (4)	C64—C65—Fe5	69.5 (5)
C32—Fe3—C38	114.6 (3)	C61—C65—H65	126.0
C34—Fe3—C38	128.2 (4)	C64—C65—H65	126.0
C40—Fe3—C39	41.2 (3)	Fe5—C65—H65	126.2
C33—Fe3—C39	115.3 (4)	C67—C66—C70	106.9 (8)
C32—Fe3—C39	146.9 (3)	C67—C66—Fe5	69.9 (5)
C34—Fe3—C39	107.9 (4)	C70—C66—Fe5	68.8 (4)
C38—Fe3—C39	40.3 (3)	C67—C66—H66	126.6
C40—Fe3—C35	110.6 (3)	C70—C66—H66	126.6
C33—Fe3—C35	67.8 (4)	Fe5—C66—H66	126.3
C32—Fe3—C35	68.1 (4)	C68—C67—C66	108.3 (8)
C34—Fe3—C35	40.8 (4)	C68—C67—Fe5	69.8 (5)
C38—Fe3—C35	168.2 (3)	C66—C67—Fe5	69.4 (5)
C39—Fe3—C35	131.7 (3)	C68—C67—H67	125.8
C40—Fe3—C36	41.4 (3)	C66—C67—H67	125.8
C33—Fe3—C36	167.1 (4)	Fe5—C67—H67	126.5
C32—Fe3—C36	130.1 (3)	C67—C68—C69	108.8 (8)
C34—Fe3—C36	151.8 (4)	C67—C68—Fe5	69.9 (5)
C38—Fe3—C36	68.4 (3)	C69—C68—Fe5	69.4 (5)
C39—Fe3—C36	68.8 (3)	C67—C68—H68	125.6
C35—Fe3—C36	119.5 (4)	C69—C68—H68	125.6
C40—Fe3—C31	132.2 (4)	Fe5—C68—H68	126.7
C33—Fe3—C31	67.7 (4)	C70—C69—C68	107.8 (7)
C32—Fe3—C31	40.6 (4)	C70—C69—Fe5	69.4 (5)
C34—Fe3—C31	68.4 (3)	C68—C69—Fe5	70.1 (5)
C38—Fe3—C31	148.5 (3)	C70—C69—H69	126.1
C39—Fe3—C31	170.9 (3)	C68—C69—H69	126.1
C35—Fe3—C31	40.3 (3)	Fe5—C69—H69	126.0
C36—Fe3—C31	110.2 (3)	C69—C70—C66	108.2 (7)
C40—Fe3—C37	68.9 (3)	C69—C70—C71	128.4 (7)
C33—Fe3—C37	128.2 (4)	C66—C70—C71	123.4 (8)
C32—Fe3—C37	107.3 (3)	C69—C70—Fe5	70.0 (5)
C34—Fe3—C37	166.5 (4)	C66—C70—Fe5	69.7 (5)
C38—Fe3—C37	40.7 (3)	C71—C70—Fe5	127.2 (6)
C39—Fe3—C37	68.2 (3)	O72—C71—O73	122.3 (7)
C35—Fe3—C37	151.0 (4)	O72—C71—C70	126.6 (8)
C36—Fe3—C37	40.4 (3)	O73—C71—C70	111.0 (7)
C31—Fe3—C37	117.3 (3)	C74—O73—C71	118.8 (6)
C46—Fe4—C52	123.7 (4)	O75—C74—O73	124.3 (8)
C46—Fe4—C55	124.0 (4)	O75—C74—C76	125.6 (8)
C52—Fe4—C55	67.3 (4)	O73—C74—C76	110.0 (7)
C46—Fe4—C50	41.2 (3)	C80—C76—C77	107.4 (8)
C52—Fe4—C50	160.5 (4)	C80—C76—C74	129.3 (8)
C55—Fe4—C50	108.7 (4)	C77—C76—C74	123.0 (8)
C46—Fe4—C54	160.2 (4)	C80—C76—Fe6	69.8 (5)
C52—Fe4—C54	66.9 (4)	C77—C76—Fe6	69.6 (5)
C55—Fe4—C54	40.7 (4)	C74—C76—Fe6	120.5 (6)

C50—Fe4—C54	123.5 (4)	C78—C77—C76	108.2 (8)
C46—Fe4—C51	108.4 (4)	C78—C77—Fe6	71.2 (5)
C52—Fe4—C51	40.3 (5)	C76—C77—Fe6	68.6 (4)
C55—Fe4—C51	40.3 (5)	C78—C77—H77	125.9
C50—Fe4—C51	124.3 (4)	C76—C77—H77	125.9
C54—Fe4—C51	67.9 (4)	Fe6—C77—H77	125.8
C46—Fe4—C53	158.9 (4)	C77—C78—C79	107.8 (8)
C52—Fe4—C53	40.3 (4)	C77—C78—Fe6	68.9 (5)
C55—Fe4—C53	67.9 (4)	C79—C78—Fe6	69.0 (5)
C50—Fe4—C53	157.9 (4)	C77—C78—H78	126.1
C54—Fe4—C53	39.7 (4)	C79—C78—H78	126.1
C51—Fe4—C53	68.2 (4)	Fe6—C78—H78	127.5
C46—Fe4—C47	41.1 (3)	C80—C79—C78	108.7 (8)
C52—Fe4—C47	107.5 (4)	C80—C79—Fe6	69.2 (5)
C55—Fe4—C47	159.6 (4)	C78—C79—Fe6	70.1 (5)
C50—Fe4—C47	69.2 (3)	C80—C79—H79	125.7
C54—Fe4—C47	157.5 (4)	C78—C79—H79	125.7
C51—Fe4—C47	122.9 (4)	Fe6—C79—H79	126.6
C53—Fe4—C47	121.9 (4)	C79—C80—C76	107.9 (8)
C46—Fe4—C49	68.6 (3)	C79—C80—Fe6	70.5 (5)
C52—Fe4—C49	157.8 (4)	C76—C80—Fe6	69.0 (5)
C55—Fe4—C49	123.9 (4)	C79—C80—H80	126.1
C50—Fe4—C49	40.5 (3)	C76—C80—H80	126.1
C54—Fe4—C49	107.9 (4)	Fe6—C80—H80	126.1
C51—Fe4—C49	160.2 (4)	C82—C81—C85	108.5 (8)
C53—Fe4—C49	121.8 (4)	C82—C81—Fe6	69.4 (5)
C47—Fe4—C49	68.5 (4)	C85—C81—Fe6	70.1 (5)
C46—Fe4—C48	68.9 (3)	C82—C81—H81	125.8
C52—Fe4—C48	121.6 (4)	C85—C81—H81	125.8
C55—Fe4—C48	159.2 (4)	Fe6—C81—H81	126.3
C50—Fe4—C48	69.2 (3)	C83—C82—C81	107.5 (9)
C54—Fe4—C48	122.1 (4)	C83—C82—Fe6	69.2 (5)
C51—Fe4—C48	157.8 (5)	C81—C82—Fe6	69.7 (5)
C53—Fe4—C48	105.9 (4)	C83—C82—H82	126.3
C47—Fe4—C48	40.6 (3)	C81—C82—H82	126.3
C49—Fe4—C48	41.1 (4)	Fe6—C82—H82	126.4
C35—C31—C32	107.7 (8)	C84—C83—C82	109.4 (8)
C35—C31—Fe3	69.7 (5)	C84—C83—Fe6	70.6 (5)
C32—C31—Fe3	69.2 (5)	C82—C83—Fe6	70.0 (5)
C35—C31—H31	126.1	C84—C83—H83	125.3
C32—C31—H31	126.1	C82—C83—H83	125.3
Fe3—C31—H31	126.5	Fe6—C83—H83	125.7
C33—C32—C31	107.8 (8)	C83—C84—C85	107.8 (9)
C33—C32—Fe3	69.8 (5)	C83—C84—Fe6	69.1 (5)
C31—C32—Fe3	70.2 (5)	C85—C84—Fe6	69.7 (5)
C33—C32—H32	126.1	C83—C84—H84	126.1
C31—C32—H32	126.1	C85—C84—H84	126.1
Fe3—C32—H32	125.6	Fe6—C84—H84	126.7
C32—C33—C34	109.3 (8)	C81—C85—C84	106.8 (9)
C32—C33—Fe3	70.1 (5)	C81—C85—Fe6	69.0 (5)
C34—C33—Fe3	69.9 (5)	C84—C85—Fe6	69.1 (5)
C32—C33—H33	125.3	C81—C85—H85	126.6
C34—C33—H33	125.4	C84—C85—H85	126.6
Fe3—C33—H33	126.2	Fe6—C85—H85	127.0
C33—C34—C35	106.9 (8)		
C5—C1—C2—C3	-1.3 (10)	C41—O43—C44—O45	13.6 (12)
Fe1—C1—C2—C3	58.6 (6)	C41—O43—C44—C46	-169.4 (6)
C5—C1—C2—Fe1	-59.9 (6)	O45—C44—C46—C50	173.9 (9)
C1—C2—C3—C4	1.0 (10)	O43—C44—C46—C50	-3.1 (12)
Fe1—C2—C3—C4	59.4 (6)	O45—C44—C46—C47	-6.8 (13)
C1—C2—C3—Fe1	-58.4 (6)	O43—C44—C46—C47	176.1 (7)
C2—C3—C4—C5	-0.3 (10)	O45—C44—C46—Fe4	-94.7 (10)
Fe1—C3—C4—C5	59.5 (6)	O43—C44—C46—Fe4	88.3 (8)
C2—C3—C4—Fe1	-59.9 (6)	C50—C46—C47—C48	1.0 (9)
C2—C1—C5—C4	1.1 (10)	C44—C46—C47—C48	-178.4 (7)
Fe1—C1—C5—C4	-59.0 (6)	Fe4—C46—C47—C48	-59.2 (6)

C2—C1—C5—Fe1	60.2 (6)	C50—C46—C47—Fe4	60.1 (5)
C3—C4—C5—C1	-0.5 (10)	C44—C46—C47—Fe4	-119.3 (8)
Fe1—C4—C5—C1	59.1 (6)	C46—C47—C48—C49	-1.0 (9)
C3—C4—C5—Fe1	-59.6 (6)	Fe4—C47—C48—C49	-58.9 (6)
C10—C6—C7—C8	-0.3 (10)	C46—C47—C48—Fe4	57.9 (5)
Fe1—C6—C7—C8	-59.6 (6)	C47—C48—C49—C50	0.7 (9)
C10—C6—C7—Fe1	59.3 (6)	Fe4—C48—C49—C50	-58.3 (6)
C6—C7—C8—C9	1.1 (10)	C47—C48—C49—Fe4	59.0 (6)
Fe1—C7—C8—C9	-58.2 (6)	C48—C49—C50—C46	-0.1 (9)
C6—C7—C8—Fe1	59.3 (6)	Fe4—C49—C50—C46	-58.8 (5)
C7—C8—C9—C10	-1.4 (9)	C48—C49—C50—Fe4	58.7 (6)
Fe1—C8—C9—C10	-59.1 (5)	C47—C46—C50—C49	-0.5 (9)
C7—C8—C9—Fe1	57.7 (6)	C44—C46—C50—C49	178.8 (8)
C7—C6—C10—C9	-0.6 (9)	Fe4—C46—C50—C49	59.9 (6)
Fe1—C6—C10—C9	59.4 (5)	C47—C46—C50—Fe4	-60.4 (6)
C7—C6—C10—C11	-179.4 (8)	C44—C46—C50—Fe4	118.9 (9)
Fe1—C6—C10—C11	-119.4 (8)	C55—C51—C52—C53	0.2 (11)
C7—C6—C10—Fe1	-60.0 (6)	Fe4—C51—C52—C53	60.1 (7)
C8—C9—C10—C6	1.2 (9)	C55—C51—C52—Fe4	-59.9 (7)
Fe1—C9—C10—C6	-59.3 (6)	C51—C52—C53—C54	-0.1 (11)
C8—C9—C10—C11	-179.8 (7)	Fe4—C52—C53—C54	59.8 (6)
Fe1—C9—C10—C11	119.7 (8)	C51—C52—C53—Fe4	-60.0 (7)
C8—C9—C10—Fe1	60.5 (6)	C52—C53—C54—C55	0.0 (10)
C6—C10—C11—O12	-165.2 (8)	Fe4—C53—C54—C55	59.2 (6)
C9—C10—C11—O12	16.1 (13)	C52—C53—C54—Fe4	-59.2 (6)
Fe1—C10—C11—O12	103.5 (9)	C52—C51—C55—C54	-0.3 (11)
C6—C10—C11—O13	9.2 (12)	Fe4—C51—C55—C54	-59.8 (6)
C9—C10—C11—O13	-169.5 (7)	C52—C51—C55—Fe4	59.5 (7)
Fe1—C10—C11—O13	-82.1 (8)	C53—C54—C55—C51	0.2 (11)
O12—C11—O13—C14	-31.4 (11)	Fe4—C54—C55—C51	59.9 (7)
C10—C11—O13—C14	153.9 (7)	C53—C54—C55—Fe4	-59.7 (7)
C11—O13—C14—O15	-29.7 (12)	C65—C61—C62—C63	0.0 (10)
C11—O13—C14—C16	155.1 (7)	Fe5—C61—C62—C63	-59.3 (6)
O15—C14—C16—C20	-172.2 (9)	C65—C61—C62—Fe5	59.4 (6)
O13—C14—C16—C20	2.9 (12)	C61—C62—C63—C64	0.3 (11)
O15—C14—C16—C17	-0.5 (14)	Fe5—C62—C63—C64	-59.0 (6)
O13—C14—C16—C17	174.6 (7)	C61—C62—C63—Fe5	59.3 (6)
O15—C14—C16—Fe2	-84.9 (11)	C62—C63—C64—C65	-0.5 (10)
O13—C14—C16—Fe2	90.1 (8)	Fe5—C63—C64—C65	-59.8 (6)
C20—C16—C17—C18	-0.1 (9)	C62—C63—C64—Fe5	59.4 (6)
C14—C16—C17—C18	-173.3 (7)	C62—C61—C65—C64	-0.3 (10)
Fe2—C16—C17—C18	-60.1 (6)	Fe5—C61—C65—C64	59.3 (6)
C20—C16—C17—Fe2	59.9 (6)	C62—C61—C65—Fe5	-59.6 (6)
C14—C16—C17—Fe2	-113.2 (8)	C63—C64—C65—C61	0.5 (10)
C16—C17—C18—C19	0.1 (10)	Fe5—C64—C65—C61	-59.5 (6)
Fe2—C17—C18—C19	-58.3 (6)	C63—C64—C65—Fe5	59.9 (6)
C16—C17—C18—Fe2	58.4 (5)	C70—C66—C67—C68	0.1 (10)
C17—C18—C19—C20	0.0 (10)	Fe5—C66—C67—C68	59.2 (6)
Fe2—C18—C19—C20	-58.3 (6)	C70—C66—C67—Fe5	-59.1 (6)
C17—C18—C19—Fe2	58.3 (6)	C66—C67—C68—C69	-0.4 (10)
C18—C19—C20—C16	-0.1 (10)	Fe5—C67—C68—C69	58.6 (6)
Fe2—C19—C20—C16	-59.3 (6)	C66—C67—C68—Fe5	-58.9 (6)
C18—C19—C20—Fe2	59.2 (6)	C67—C68—C69—C70	0.5 (10)
C17—C16—C20—C19	0.1 (10)	Fe5—C68—C69—C70	59.4 (6)
C14—C16—C20—C19	172.7 (8)	C67—C68—C69—Fe5	-58.9 (6)
Fe2—C16—C20—C19	60.4 (6)	C68—C69—C70—C66	-0.4 (9)
C17—C16—C20—Fe2	-60.3 (6)	Fe5—C69—C70—C66	59.4 (6)
C14—C16—C20—Fe2	112.3 (9)	C68—C69—C70—C71	177.9 (8)
C25—C21—C22—C23	-2.1 (10)	Fe5—C69—C70—C71	-122.2 (9)
Fe2—C21—C22—C23	59.2 (7)	C68—C69—C70—Fe5	-59.8 (6)
C25—C21—C22—Fe2	-61.4 (6)	C67—C66—C70—C69	0.2 (9)
C21—C22—C23—C24	0.5 (11)	Fe5—C66—C70—C69	-59.6 (6)
Fe2—C22—C23—C24	59.0 (7)	C67—C66—C70—C71	-178.3 (8)
C21—C22—C23—Fe2	-58.5 (6)	Fe5—C66—C70—C71	121.9 (8)
C22—C23—C24—C25	1.3 (11)	C67—C66—C70—Fe5	59.8 (6)
Fe2—C23—C24—C25	60.9 (7)	C69—C70—C71—O72	-162.7 (9)
C22—C23—C24—Fe2	-59.6 (7)	C66—C70—C71—O72	15.5 (13)

C23—C24—C25—C21	-2.6 (11)	Fe5—C70—C71—O72	104.2 (9)
Fe2—C24—C25—C21	58.3 (6)	C69—C70—C71—O73	13.2 (12)
C23—C24—C25—Fe2	-60.9 (7)	C66—C70—C71—O73	-168.6 (7)
C22—C21—C25—C24	2.9 (10)	Fe5—C70—C71—O73	-79.9 (9)
Fe2—C21—C25—C24	-58.2 (6)	O72—C71—O73—C74	-34.5 (11)
C22—C21—C25—Fe2	61.1 (6)	C70—C71—O73—C74	149.4 (7)
C35—C31—C32—C33	0.5 (10)	C71—O73—C74—O75	-21.9 (12)
Fe3—C31—C32—C33	59.9 (6)	C71—O73—C74—C76	160.0 (7)
C35—C31—C32—Fe3	-59.3 (6)	O75—C74—C76—C80	-174.3 (9)
C31—C32—C33—C34	-1.0 (10)	O73—C74—C76—C80	3.7 (12)
Fe3—C32—C33—C34	59.1 (6)	O75—C74—C76—C77	-2.0 (14)
C31—C32—C33—Fe3	-60.1 (6)	O73—C74—C76—C77	176.0 (7)
C32—C33—C34—C35	1.1 (10)	O75—C74—C76—Fe6	-86.3 (11)
Fe3—C33—C34—C35	60.3 (6)	O73—C74—C76—Fe6	91.8 (8)
C32—C33—C34—Fe3	-59.2 (6)	C80—C76—C77—C78	-0.6 (9)
C32—C31—C35—C34	0.1 (10)	C74—C76—C77—C78	-174.3 (8)
Fe3—C31—C35—C34	-58.9 (6)	Fe6—C76—C77—C78	-60.4 (6)
C32—C31—C35—Fe3	59.0 (6)	C80—C76—C77—Fe6	59.9 (6)
C33—C34—C35—C31	-0.7 (10)	C74—C76—C77—Fe6	-113.9 (7)
Fe3—C34—C35—C31	59.3 (6)	C76—C77—C78—C79	0.7 (10)
C33—C34—C35—Fe3	-60.0 (6)	Fe6—C77—C78—C79	-58.2 (6)
C40—C36—C37—C38	-0.4 (9)	C76—C77—C78—Fe6	58.9 (6)
Fe3—C36—C37—C38	-58.2 (6)	C77—C78—C79—C80	-0.5 (10)
C40—C36—C37—Fe3	57.8 (5)	Fe6—C78—C79—C80	-58.6 (6)
C36—C37—C38—C39	-1.3 (9)	C77—C78—C79—Fe6	58.1 (6)
Fe3—C37—C38—C39	-59.8 (6)	C78—C79—C80—C76	0.1 (10)
C36—C37—C38—Fe3	58.6 (6)	Fe6—C79—C80—C76	-59.0 (6)
C37—C38—C39—C40	2.5 (9)	C78—C79—C80—Fe6	59.2 (6)
Fe3—C38—C39—C40	-57.6 (6)	C77—C76—C80—C79	0.3 (9)
C37—C38—C39—Fe3	60.0 (6)	C74—C76—C80—C79	173.5 (8)
C38—C39—C40—C36	-2.7 (9)	Fe6—C76—C80—C79	60.0 (6)
Fe3—C39—C40—C36	-61.2 (6)	C77—C76—C80—Fe6	-59.7 (6)
C38—C39—C40—C41	177.4 (8)	C74—C76—C80—Fe6	113.5 (9)
Fe3—C39—C40—C41	118.9 (9)	C85—C81—C82—C83	0.3 (11)
C38—C39—C40—Fe3	58.5 (6)	Fe6—C81—C82—C83	-59.1 (6)
C37—C36—C40—C39	1.9 (9)	C85—C81—C82—Fe6	59.4 (6)
Fe3—C36—C40—C39	61.2 (6)	C81—C82—C83—C84	-0.3 (11)
C37—C36—C40—C41	-178.2 (7)	Fe6—C82—C83—C84	-59.8 (7)
Fe3—C36—C40—C41	-118.9 (8)	C81—C82—C83—Fe6	59.5 (6)
C37—C36—C40—Fe3	-59.3 (6)	C82—C83—C84—C85	0.2 (11)
C39—C40—C41—O42	165.6 (9)	Fe6—C83—C84—C85	-59.2 (6)
C36—C40—C41—O42	-14.3 (14)	C82—C83—C84—Fe6	59.4 (7)
Fe3—C40—C41—O42	-102.4 (9)	C82—C81—C85—C84	-0.1 (10)
C39—C40—C41—O43	-12.0 (12)	Fe6—C81—C85—C84	58.9 (6)
C36—C40—C41—O43	168.1 (7)	C82—C81—C85—Fe6	-59.0 (6)
Fe3—C40—C41—O43	80.0 (8)	C83—C84—C85—C81	-0.1 (10)
O42—C41—O43—C44	42.4 (11)	Fe6—C84—C85—C81	-58.8 (6)
C40—C41—O43—C44	-139.8 (7)	C83—C84—C85—Fe6	58.8 (7)

Selected hydrogen-bond parameters for **1B** (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>
C49—H49...O72	0.95	2.64	3.517 (11)	153
C31—H31...O12	0.95	2.49	3.387 (10)	158
C5—H5...O45 ⁱ	0.95	2.57	3.368 (11)	142
C85—H85...O42 ⁱⁱ	0.95	2.57	3.438 (12)	152
C6—H6...O15 ⁱⁱⁱ	0.95	2.33	3.256 (11)	164
C39—H39...O45 ⁱⁱⁱ	0.95	2.65	3.595 (11)	170
C69—H69...O75 ^{iv}	0.95	2.35	3.273 (10)	164

Symmetry code(s): (i) $x-1/2, -y+1/2, z+1/2$; (ii) $x, -y+1, z-1/2$; (iii) $x-1, y, z$; (iv) $x+1, y, z$.