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Benzotriazol-1-yl methyl ferrocene-1,1′-dicarboxylate

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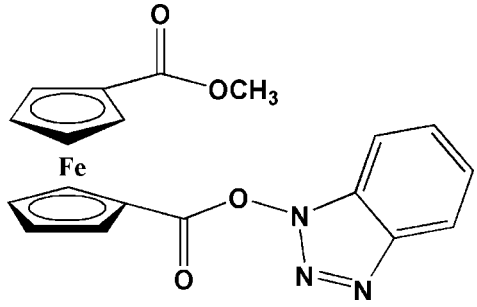
Key indicators: single-crystal X-ray study; T = 173 K; mean σ(C–C) = 0.003 Å; R factor = 0.038; wR factor = 0.096; data-to-parameter ratio = 14.1.

The title compound, CH_3O–C(O)–Fc–C(O)–OBt or $[Fe(C_7H_7O_2)(C_{12}H_8N_3O_2)]$ (Bt is benzotriazole), shows a *cis* configuration of the Bt group relative to the ferrocene core, with the benzene ring in close proximity to the methyl ester group.

Related literature

For related literature, see: Heinze & Schlenker (2004); Kraatz *et al.* (1997); Mahmoud *et al.* (2005); Taylor & Kennard (1982).



Experimental

Crystal data

$[Fe(C_7H_7O_2)(C_{12}H_8N_3O_2)]$
M_r = 405.19
Monoclinic, $P2_1/c$
a = 8.2720 (3) Å
b = 10.9180 (3) Å
c = 18.7460 (5) Å
β = 91.383 (2)°
V = 1692.53 (9) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.92 mm^{-1}
T = 173 (2) K
0.20 × 0.20 × 0.13 mm

Data collection

Bruker–Nonius KappaCCD diffractometer
Absorption correction: multi-scan (*SCALEPACK*; Otwinowski & Minor, 1997)
T_{min} = 0.837, T_{max} = 0.889
12048 measured reflections
3460 independent reflections
2801 reflections with $I > 2\sigma(I)$
R_{int} = 0.060

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.038
$wR(F^2)$ = 0.096
S = 1.05
3460 reflections
245 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.31 e Å^{-3}
$\Delta\rho_{min}$ = −0.47 e Å^{-3}

Data collection: *COLLECT* (Nonius, 1998); cell refinement: *HKL DENZO* and *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *HKL DENZO* and *SCALEPACK*; program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP* in *SHELXTL-NT* (Bruker, 2003); software used to prepare material for publication: *WinGX* (Version 1.70.01; Farrugia, 1999).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: IM2012).

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supplementary materials

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Benzotriazol-1-yl methyl ferrocene-1,1'-dicarboxylate

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Comment

The ferrocene ester of 1-hydroxybenzotriazole (HOBt) is useful in the preparation of ferrocene peptide conjugates (Kraatz *et al.*, 1997; Mahmoud *et al.*, 2005). The reactivity of Fc-OBt ester towards external nucleophiles is attributed to the increasing electrophilicity of the carbonyl carbon upon esterfication, driven by the lack of interaction between the Cp and OBt π-systems (Mahmoud *et al.*, 2005). Compound (**1**) crystalizes in the monoclinic space group $P2_1/c$. The two cyclopentadienyl groups of the ferrocence moiety are in the eclipsed conformation with a Cp—Fe—Cp angle 177.8 (6)°. The two ester groups are in the 1,2' conformation around the ferrocene center, they are almost coplanar to the Cp rings with torsion angles for C(14)—C10)—C(15)—O(12) and C(24)—C(20)—C(25)—O(22) of 1.1 (3)° and −0.3 (3)°, respectively, unlike to those found in Fc—C(O)-OBt (10.06 (10)°; Kraatz *et al.*, 1997) and Boc-N(H)—Fc—C(O)-OBt (5.0 (7)°; Mahmoud *et al.*, 2005). The benzotriazole moiety is perpendicular with respect to the Cp it is attached to [C(15)—O(12)—N(11)—C(16)] = 87.0 (0)°). This type of arrangement is also found in the related compounds with *cis*-configuration relative to ferrocene moiety in Fc—C(O)-OBt (96.56 (6)°; Kraatz *et al.*, 1997), Boc-N(H)—Fc—C(O)-OBt (80.8 (2)°); Mahmoud *et al.*, 2005) and *trans*-configuration in Ac—N(H)—Fc—C(O)-OBt (88.6°) (Heinze *et al.*, 2004). Presumably the weak hydrogen bond interaction between O(21) and C(17)(H) is responsible for the observed conformation in 1, (C=O(21)—C(17)(H) 3.233 Å; sum of the van der waals radii 3.25 Å; Taylor *et al.*,1982).

Experimental

Solid 1-hydroxybenzotriazole (HOBt) (4.40 mmol, 0.67 g) and 1-Ethyl-3-(3-dimethylaminopropyl)carbodiimide hydrochloride (EDC·HCl) (4.40 mmol, 0.84 g) were added to a solution of 1'-(methoxycarbonyl)-ferrocene-1-carboxylic acid (3.93 mmol, 0.80 g) in CH_2Cl_2 (50 ml). The reaction mixture was stirred for 2 h at room temperature, and then washed with aqueous solutions of saturated sodium bicarbonate ($NaHCO_3$), citric acid, and water. The organic phase was separated and dried with anhydrous Na_2SO_4, then filtered and the solvent CH_2Cl_2 evaporated under reduced pressure. The crude product was purified by flash chromatography (SiO_2, R_f = 0.55, EtOAc/hexane, 1:2) to give dark orange crystals of (Benzotriazole-1-yl)-1'-(methoxycarbonyl)-ferrocene-1- carboxylate (**1**) (2.38 mmol, 0.9678 g, yield: 61%).

Suitable crystals of (**1**) for X-ray diffraction were obtained by slow diffusion (layering technique) of *n*-hexane into a solution of (**1**) in CH_2Cl_2. Dark orange crystals were obtained after 24 h. An orange squared-like crystal of (**1**) was coated with oil (Paratone 8277, Exxon), was collected onto the nylon fiber of a mounted CryoLoopTM (Hampton Research, USA) under a microscope. The crystal was then mounted onto the goniometer head, which was quickly transferred to the cold stream of the X-ray diffractometer.

Refinement

The structure was solved using direct methods (*SIR97*) and refined by full-matrix least-squares method on F^2 with *SHELXL97*–2. The non-hydrogen atoms were refined anisotropically. Hydrogen atoms were included at geometrically ideal-

ized positions (C—H bond distances 0.95/0.98/1.00) and were not refined. The isotropic thermal parameters of the hydrogen atoms were fixed at 1.2 times that of the preceding carbon atom.

Figures

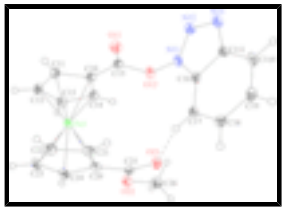


Figure 1
ORTEP diagram of the molecular structure of (**1**). Displacement ellipsoids are drawn at the 30% probability level.

Benzotriazol-1-yl methyl ferrocene-1,1'-dicarboxylate

Crystal data

$[Fe(C_7H_7O_2)(C_{12}H_8N_3O_2)]$	F_{000} = 832
M_r = 405.19	D_x = 1.590 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2ybc	Cell parameters from 3308 reflections
a = 8.2720 (3) Å	θ = 1.0–27.9º
b = 10.9180 (3) Å	μ = 0.92 mm^{-1}
c = 18.7460 (5) Å	T = 173 (2) K
β = 91.383 (2)º	Square, orange
V = 1692.53 (9) Å^3	0.20 × 0.20 × 0.13 mm
Z = 4	

Data collection

Bruker–Nonius KappaCCD diffractometer	3460 independent reflections
Radiation source: Nonius FR590D, fine-focus sealed tube	2801 reflections with $I > 2\sigma(I)$
Monochromator: horizonally mounted graphite crystal	R_{int} = 0.060
T = 173(2) K	θ_{max} = 26.4º
CCD rotation images, thick slices scans	θ_{min} = 2.9º
Absorption correction: multi-scan (HKL Scalepack; (Otwinowski & Minor, 1997)	h = −10→10
T_{min} = 0.837, T_{max} = 0.889	k = −13→13
12048 measured reflections	l = −23→22

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites

$R[F^2 > 2\sigma(F^2)] = 0.038$	H-atom parameters constrained
$wR(F^2) = 0.096$	$w = 1/[\sigma^2(F_o^2) + (0.0414P)^2 + 1.2132P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.05$	$(\Delta/\sigma)_{max} = 0.001$
3460 reflections	$\Delta\rho_{max} = 0.31$ e Å^{-3}
245 parameters	$\Delta\rho_{min} = -0.47$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

Special details

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

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

	x	*y*	*z*	U_{iso}*/U_{eq}
Fe1	0.48612 (4)	0.32889 (3)	0.651633 (18)	0.01915 (11)
O11	0.3274 (2)	0.24087 (16)	0.46478 (10)	0.0306 (4)
O12	0.1545 (2)	0.37941 (16)	0.51327 (9)	0.0269 (4)
O21	0.0387 (2)	0.30425 (16)	0.68935 (10)	0.0301 (4)
O22	0.1579 (2)	0.43584 (16)	0.76631 (9)	0.0288 (4)
N11	0.0386 (3)	0.32276 (19)	0.47099 (11)	0.0268 (5)
N12	0.0142 (3)	0.3614 (2)	0.40315 (12)	0.0331 (5)
N13	−0.0968 (3)	0.2906 (2)	0.37430 (12)	0.0313 (5)
C10	0.4275 (3)	0.3847 (2)	0.55082 (13)	0.0215 (5)
C11	0.5928 (3)	0.3463 (2)	0.55495 (14)	0.0243 (5)
H11	0.6395	0.2819	0.5283	0.029*
C12	0.6745 (3)	0.4212 (2)	0.60574 (14)	0.0278 (6)
H12	0.7858	0.4160	0.6191	0.033*
C13	0.5608 (3)	0.5061 (2)	0.63342 (14)	0.0276 (6)
H13	0.5838	0.5673	0.6683	0.033*
C14	0.4077 (3)	0.4839 (2)	0.60019 (13)	0.0241 (5)
H14	0.3102	0.5267	0.6090	0.029*
C15	0.3077 (3)	0.3240 (2)	0.50473 (13)	0.0222 (5)
C16	−0.0509 (3)	0.2232 (2)	0.48724 (13)	0.0207 (5)
C17	−0.0671 (3)	0.1509 (2)	0.54821 (13)	0.0234 (5)
H17	−0.0060	0.1659	0.5909	0.028*
C18	−0.1770 (3)	0.0568 (2)	0.54249 (13)	0.0256 (5)
H18	−0.1903	0.0033	0.5819	0.031*
C19	−0.2711 (3)	0.0377 (2)	0.47938 (14)	0.0269 (5)

H19	−0.3470	−0.0276	0.4778	0.032*
C20	0.3148 (3)	0.2773 (2)	0.72174 (12)	0.0220 (5)
C21	0.3525 (3)	0.1773 (2)	0.67550 (13)	0.0233 (5)
H21	0.2788	0.1359	0.6442	0.028*
C22	0.5201 (3)	0.1511 (2)	0.68493 (14)	0.0284 (6)
H22	0.5779	0.0890	0.6609	0.034*
C23	0.5864 (3)	0.2336 (3)	0.73637 (14)	0.0317 (6)
H23	0.6960	0.2359	0.7527	0.038*
C24	0.4609 (3)	0.3119 (2)	0.75923 (13)	0.0272 (6)
H24	0.4717	0.3759	0.7933	0.033*
C25	0.1567 (3)	0.3375 (2)	0.72331 (12)	0.0211 (5)
C26	0.0080 (4)	0.5040 (3)	0.76726 (15)	0.0345 (6)
H26A	−0.0236	0.5276	0.7185	0.041*
H26B	0.0229	0.5778	0.7965	0.041*
H26C	−0.0768	0.4529	0.7875	0.041*
C110	−0.2558 (3)	0.1102 (2)	0.42076 (14)	0.0270 (6)
H110	−0.3208	0.0974	0.3790	0.032*
C111	−0.1408 (3)	0.2044 (2)	0.42423 (13)	0.0233 (5)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Fe1	0.01780 (19)	0.01764 (18)	0.02191 (19)	0.00037 (13)	−0.00146 (13)	0.00061 (13)
O11	0.0315 (11)	0.0281 (10)	0.0320 (10)	−0.0011 (8)	−0.0020 (8)	−0.0040 (8)
O12	0.0203 (9)	0.0268 (9)	0.0333 (10)	−0.0017 (7)	−0.0065 (7)	−0.0018 (8)
O21	0.0234 (10)	0.0329 (10)	0.0339 (10)	−0.0006 (8)	−0.0027 (8)	−0.0069 (8)
O22	0.0311 (10)	0.0283 (9)	0.0270 (9)	0.0061 (8)	−0.0019 (7)	−0.0103 (8)
N11	0.0212 (11)	0.0313 (11)	0.0274 (11)	−0.0044 (9)	−0.0084 (9)	0.0039 (9)
N12	0.0286 (13)	0.0399 (13)	0.0306 (12)	−0.0005 (10)	−0.0040 (10)	0.0100 (10)
N13	0.0284 (12)	0.0359 (12)	0.0294 (12)	−0.0022 (10)	−0.0074 (10)	0.0063 (10)
C10	0.0206 (13)	0.0194 (11)	0.0245 (12)	−0.0008 (9)	0.0011 (10)	0.0044 (9)
C11	0.0197 (12)	0.0255 (12)	0.0276 (13)	0.0006 (10)	0.0018 (10)	0.0020 (10)
C12	0.0173 (12)	0.0329 (14)	0.0331 (14)	−0.0062 (10)	−0.0015 (10)	0.0045 (11)
C13	0.0304 (14)	0.0191 (12)	0.0333 (14)	−0.0063 (10)	−0.0022 (11)	−0.0007 (10)
C14	0.0232 (13)	0.0184 (11)	0.0309 (13)	0.0008 (10)	0.0030 (10)	0.0052 (10)
C15	0.0214 (13)	0.0216 (12)	0.0237 (12)	0.0007 (10)	0.0016 (10)	0.0068 (10)
C16	0.0159 (11)	0.0226 (12)	0.0234 (12)	0.0017 (9)	−0.0007 (9)	−0.0012 (10)
C17	0.0195 (12)	0.0296 (13)	0.0210 (12)	0.0038 (10)	−0.0019 (10)	0.0007 (10)
C18	0.0235 (13)	0.0256 (12)	0.0279 (13)	0.0052 (10)	0.0047 (10)	0.0024 (10)
C19	0.0217 (13)	0.0238 (12)	0.0352 (14)	−0.0014 (10)	0.0002 (10)	−0.0049 (11)
C20	0.0295 (14)	0.0190 (11)	0.0174 (11)	−0.0014 (10)	−0.0013 (10)	0.0007 (9)
C21	0.0272 (13)	0.0177 (11)	0.0251 (12)	−0.0021 (10)	0.0025 (10)	0.0005 (10)
C22	0.0319 (15)	0.0213 (12)	0.0322 (14)	0.0075 (10)	0.0045 (11)	0.0058 (10)
C23	0.0270 (14)	0.0379 (15)	0.0299 (14)	0.0044 (11)	−0.0054 (11)	0.0089 (12)
C24	0.0291 (14)	0.0307 (14)	0.0215 (12)	0.0015 (11)	−0.0067 (10)	−0.0012 (10)
C25	0.0257 (13)	0.0204 (11)	0.0171 (11)	−0.0029 (10)	0.0017 (10)	0.0006 (9)
C26	0.0395 (16)	0.0336 (14)	0.0307 (14)	0.0123 (12)	0.0055 (12)	−0.0017 (12)
C110	0.0219 (13)	0.0274 (13)	0.0312 (13)	0.0024 (10)	−0.0088 (11)	−0.0076 (11)

C111	0.0207 (12)	0.0269 (12)	0.0221 (12)	0.0038 (10)	−0.0034 (10)	−0.0017 (10)

Geometric parameters (Å, °)

Fe1—C10	2.033 (2)	C13—C14	1.419 (4)
Fe1—C20	2.035 (2)	C13—H13	0.9500
Fe1—C24	2.041 (3)	C14—H14	0.9500
Fe1—C11	2.044 (3)	C16—C111	1.396 (3)
Fe1—C14	2.046 (2)	C16—C17	1.398 (3)
Fe1—C21	2.046 (2)	C17—C18	1.375 (4)
Fe1—C22	2.056 (2)	C17—H17	0.9500
Fe1—C23	2.057 (3)	C18—C19	1.415 (4)
Fe1—C12	2.062 (3)	C18—H18	0.9500
Fe1—C13	2.062 (2)	C19—C110	1.363 (4)
O11—C15	1.191 (3)	C19—H19	0.9500
O12—N11	1.376 (3)	C20—C21	1.433 (3)
O12—C15	1.416 (3)	C20—C24	1.433 (4)
O21—C25	1.208 (3)	C20—C25	1.465 (3)
O22—C25	1.342 (3)	C21—C22	1.423 (4)
O22—C26	1.447 (3)	C21—H21	0.9500
N11—N12	1.350 (3)	C22—C23	1.420 (4)
N11—C16	1.354 (3)	C22—H22	0.9500
N12—N13	1.308 (3)	C23—C24	1.420 (4)
N13—C111	1.382 (3)	C23—H23	0.9500
C10—C11	1.431 (3)	C24—H24	0.9500
C10—C14	1.437 (3)	C26—H26A	0.9800
C10—C15	1.458 (3)	C26—H26B	0.9800
C11—C12	1.415 (4)	C26—H26C	0.9800
C11—H11	0.9500	C110—C111	1.401 (4)
C12—C13	1.427 (4)	C110—H110	0.9500
C12—H12	0.9500		
C10—Fe1—C20	121.94 (10)	C14—C13—Fe1	69.16 (13)
C10—Fe1—C24	156.62 (10)	C12—C13—Fe1	69.74 (14)
C20—Fe1—C24	41.17 (10)	C14—C13—H13	125.7
C10—Fe1—C11	41.09 (9)	C12—C13—H13	125.7
C20—Fe1—C11	157.02 (10)	Fe1—C13—H13	127.0
C24—Fe1—C11	160.29 (11)	C13—C14—C10	107.3 (2)
C10—Fe1—C14	41.25 (10)	C13—C14—Fe1	70.43 (14)
C20—Fe1—C14	108.41 (10)	C10—C14—Fe1	68.91 (13)
C24—Fe1—C14	120.14 (10)	C13—C14—H14	126.3
C11—Fe1—C14	69.14 (10)	C10—C14—H14	126.3
C10—Fe1—C21	109.12 (10)	Fe1—C14—H14	125.9
C20—Fe1—C21	41.10 (9)	O11—C15—O12	122.2 (2)
C24—Fe1—C21	69.00 (10)	O11—C15—C10	128.1 (2)
C11—Fe1—C21	121.08 (10)	O12—C15—C10	109.7 (2)
C14—Fe1—C21	127.26 (10)	N11—C16—C111	102.1 (2)
C10—Fe1—C22	126.35 (10)	N11—C16—C17	134.5 (2)
C20—Fe1—C22	68.58 (10)	C111—C16—C17	123.3 (2)
C24—Fe1—C22	68.34 (10)	C18—C17—C16	115.7 (2)

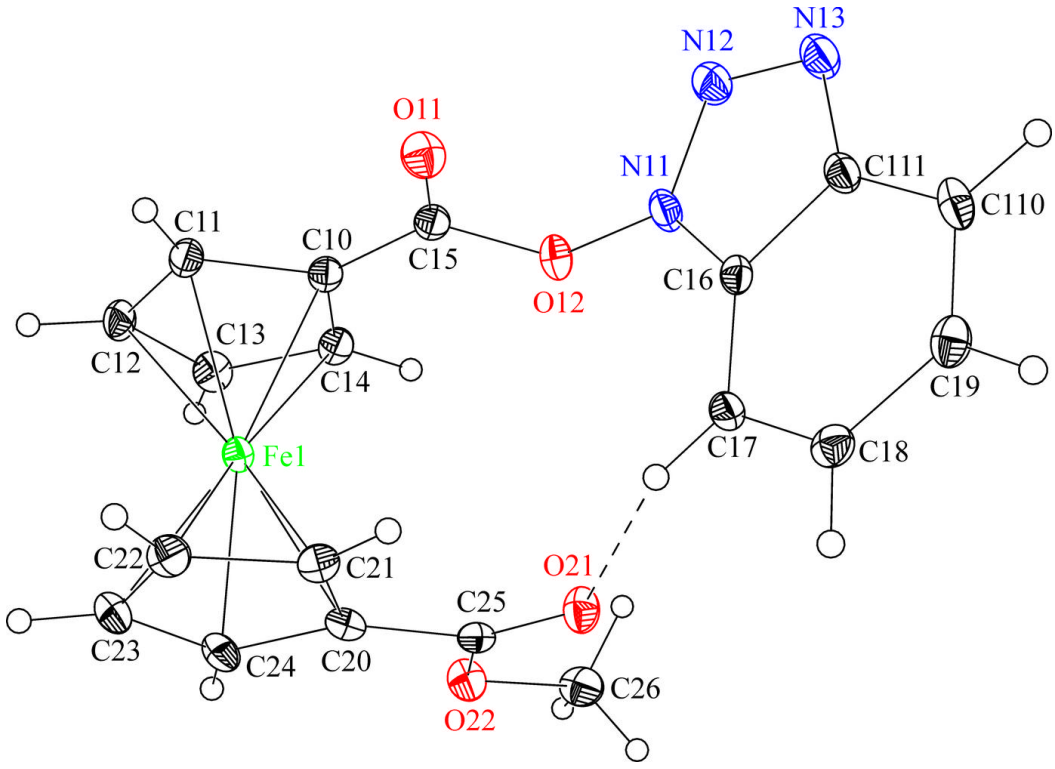
C11—Fe1—C22	107.34 (10)	C18—C17—H17	122.1
C14—Fe1—C22	164.48 (11)	C16—C17—H17	122.1
C21—Fe1—C22	40.59 (10)	C17—C18—C19	121.6 (2)
C10—Fe1—C23	162.20 (10)	C17—C18—H18	119.2
C20—Fe1—C23	68.61 (10)	C19—C18—H18	119.2
C24—Fe1—C23	40.54 (11)	C110—C19—C18	121.9 (2)
C11—Fe1—C23	123.85 (11)	C110—C19—H19	119.0
C14—Fe1—C23	154.17 (11)	C18—C19—H19	119.0
C21—Fe1—C23	68.40 (11)	C21—C20—C24	107.8 (2)
C22—Fe1—C23	40.38 (11)	C21—C20—C25	124.1 (2)
C10—Fe1—C12	68.37 (10)	C24—C20—C25	127.9 (2)
C20—Fe1—C12	161.79 (10)	C21—C20—Fe1	69.86 (14)
C24—Fe1—C12	123.59 (10)	C24—C20—Fe1	69.64 (14)
C11—Fe1—C12	40.32 (10)	C25—C20—Fe1	121.63 (16)
C14—Fe1—C12	68.47 (10)	C22—C21—C20	107.6 (2)
C21—Fe1—C12	154.80 (10)	C22—C21—Fe1	70.09 (14)
C22—Fe1—C12	119.28 (11)	C20—C21—Fe1	69.04 (13)
C23—Fe1—C12	105.95 (11)	C22—C21—H21	126.2
C10—Fe1—C13	68.33 (10)	C20—C21—H21	126.2
C20—Fe1—C13	125.65 (10)	Fe1—C21—H21	126.3
C24—Fe1—C13	106.68 (11)	C23—C22—C21	108.4 (2)
C11—Fe1—C13	68.19 (10)	C23—C22—Fe1	69.83 (15)
C14—Fe1—C13	40.41 (10)	C21—C22—Fe1	69.32 (13)
C21—Fe1—C13	163.93 (10)	C23—C22—H22	125.8
C22—Fe1—C13	153.73 (11)	C21—C22—H22	125.8
C23—Fe1—C13	119.06 (11)	Fe1—C22—H22	126.6
C12—Fe1—C13	40.49 (11)	C22—C23—C24	108.3 (2)
N11—O12—C15	110.85 (18)	C22—C23—Fe1	69.78 (15)
C25—O22—C26	115.1 (2)	C24—C23—Fe1	69.13 (14)
N12—N11—C16	113.1 (2)	C22—C23—H23	125.9
N12—N11—O12	119.2 (2)	C24—C23—H23	125.9
C16—N11—O12	127.5 (2)	Fe1—C23—H23	126.8
N13—N12—N11	107.0 (2)	C23—C24—C20	107.9 (2)
N12—N13—C111	108.4 (2)	C23—C24—Fe1	70.33 (14)
C11—C10—C14	108.0 (2)	C20—C24—Fe1	69.19 (14)
C11—C10—C15	122.3 (2)	C23—C24—H24	126.1
C14—C10—C15	129.6 (2)	C20—C24—H24	126.1
C11—C10—Fe1	69.86 (14)	Fe1—C24—H24	126.0
C14—C10—Fe1	69.84 (14)	O21—C25—O22	123.5 (2)
C15—C10—Fe1	123.94 (16)	O21—C25—C20	124.4 (2)
C12—C11—C10	107.9 (2)	O22—C25—C20	112.1 (2)
C12—C11—Fe1	70.52 (15)	O22—C26—H26A	109.5
C10—C11—Fe1	69.05 (14)	O22—C26—H26B	109.5
C12—C11—H11	126.0	H26A—C26—H26B	109.5
C10—C11—H11	126.0	O22—C26—H26C	109.5
Fe1—C11—H11	126.0	H26A—C26—H26C	109.5
C11—C12—C13	108.2 (2)	H26B—C26—H26C	109.5
C11—C12—Fe1	69.16 (14)	C19—C110—C111	117.7 (2)
C13—C12—Fe1	69.77 (14)	C19—C110—H110	121.1

C11—C12—H12	125.9	C111—C110—H110	121.1
C13—C12—H12	125.9	N13—C111—C16	109.3 (2)
Fe1—C12—H12	126.7	N13—C111—C110	131.1 (2)
C14—C13—C12	108.6 (2)	C16—C111—C110	119.6 (2)
C15—O12—N11—N12	−87.7 (3)	C16—C17—C18—C19	−2.0 (4)
C15—O12—N11—C16	87.0 (3)	C17—C18—C19—C110	1.0 (4)
C16—N11—N12—N13	2.4 (3)	C10—Fe1—C20—C21	82.68 (17)
O12—N11—N12—N13	177.9 (2)	C24—Fe1—C20—C21	−118.9 (2)
N11—N12—N13—C111	−1.2 (3)	C11—Fe1—C20—C21	46.3 (3)
C20—Fe1—C10—C11	−159.35 (14)	C14—Fe1—C20—C21	126.10 (15)
C24—Fe1—C10—C11	163.1 (2)	C22—Fe1—C20—C21	−37.71 (15)
C14—Fe1—C10—C11	119.1 (2)	C23—Fe1—C20—C21	−81.23 (16)
C21—Fe1—C10—C11	−115.71 (15)	C12—Fe1—C20—C21	−157.0 (3)
C22—Fe1—C10—C11	−73.68 (18)	C13—Fe1—C20—C21	167.61 (15)
C23—Fe1—C10—C11	−37.0 (4)	C10—Fe1—C20—C24	−158.47 (14)
C12—Fe1—C10—C11	37.51 (14)	C11—Fe1—C20—C24	165.1 (2)
C13—Fe1—C10—C11	81.22 (15)	C14—Fe1—C20—C24	−115.04 (15)
C20—Fe1—C10—C14	81.57 (16)	C21—Fe1—C20—C24	118.9 (2)
C24—Fe1—C10—C14	44.1 (3)	C22—Fe1—C20—C24	81.15 (16)
C11—Fe1—C10—C14	−119.1 (2)	C23—Fe1—C20—C24	37.63 (15)
C21—Fe1—C10—C14	125.21 (14)	C12—Fe1—C20—C24	−38.1 (4)
C22—Fe1—C10—C14	167.24 (15)	C13—Fe1—C20—C24	−73.54 (18)
C23—Fe1—C10—C14	−156.0 (3)	C10—Fe1—C20—C25	−35.8 (2)
C12—Fe1—C10—C14	−81.57 (15)	C24—Fe1—C20—C25	122.7 (2)
C13—Fe1—C10—C14	−37.86 (14)	C11—Fe1—C20—C25	−72.2 (3)
C20—Fe1—C10—C15	−43.3 (2)	C14—Fe1—C20—C25	7.7 (2)
C24—Fe1—C10—C15	−80.8 (3)	C21—Fe1—C20—C25	−118.4 (3)
C11—Fe1—C10—C15	116.0 (3)	C22—Fe1—C20—C25	−156.1 (2)
C14—Fe1—C10—C15	−124.9 (3)	C23—Fe1—C20—C25	160.3 (2)
C21—Fe1—C10—C15	0.3 (2)	C12—Fe1—C20—C25	84.6 (4)
C22—Fe1—C10—C15	42.4 (3)	C13—Fe1—C20—C25	49.2 (2)
C23—Fe1—C10—C15	79.1 (4)	C24—C20—C21—C22	0.1 (3)
C12—Fe1—C10—C15	153.6 (2)	C25—C20—C21—C22	174.9 (2)
C13—Fe1—C10—C15	−162.7 (2)	Fe1—C20—C21—C22	59.70 (17)
C14—C10—C11—C12	−0.4 (3)	C24—C20—C21—Fe1	−59.56 (17)
C15—C10—C11—C12	−178.2 (2)	C25—C20—C21—Fe1	115.2 (2)
Fe1—C10—C11—C12	−60.06 (17)	C10—Fe1—C21—C22	124.03 (15)
C14—C10—C11—Fe1	59.64 (16)	C20—Fe1—C21—C22	−118.9 (2)
C15—C10—C11—Fe1	−118.2 (2)	C24—Fe1—C21—C22	−80.81 (16)
C10—Fe1—C11—C12	119.0 (2)	C11—Fe1—C21—C22	80.28 (17)
C20—Fe1—C11—C12	169.0 (2)	C14—Fe1—C21—C22	166.62 (15)
C24—Fe1—C11—C12	−41.1 (4)	C23—Fe1—C21—C22	−37.16 (15)
C14—Fe1—C11—C12	80.92 (16)	C12—Fe1—C21—C22	44.4 (3)
C21—Fe1—C11—C12	−157.29 (15)	C13—Fe1—C21—C22	−158.0 (3)
C22—Fe1—C11—C12	−115.08 (16)	C10—Fe1—C21—C20	−117.03 (15)
C23—Fe1—C11—C12	−73.80 (18)	C24—Fe1—C21—C20	38.14 (15)
C13—Fe1—C11—C12	37.40 (15)	C11—Fe1—C21—C20	−160.77 (14)
C20—Fe1—C11—C10	50.0 (3)	C14—Fe1—C21—C20	−74.43 (17)
C24—Fe1—C11—C10	−160.0 (3)	C22—Fe1—C21—C20	118.9 (2)

C14—Fe1—C11—C10	−38.07 (14)	C23—Fe1—C21—C20	81.79 (16)
C21—Fe1—C11—C10	83.72 (16)	C12—Fe1—C21—C20	163.3 (2)
C22—Fe1—C11—C10	125.93 (15)	C13—Fe1—C21—C20	−39.1 (4)
C23—Fe1—C11—C10	167.21 (14)	C20—C21—C22—C23	0.0 (3)
C12—Fe1—C11—C10	−119.0 (2)	Fe1—C21—C22—C23	59.05 (18)
C13—Fe1—C11—C10	−81.59 (15)	C20—C21—C22—Fe1	−59.04 (16)
C10—C11—C12—C13	0.1 (3)	C10—Fe1—C22—C23	163.61 (15)
Fe1—C11—C12—C13	−59.05 (18)	C20—Fe1—C22—C23	−81.75 (16)
C10—C11—C12—Fe1	59.14 (17)	C24—Fe1—C22—C23	−37.33 (16)
C10—Fe1—C12—C11	−38.20 (14)	C11—Fe1—C22—C23	122.25 (16)
C20—Fe1—C12—C11	−166.2 (3)	C14—Fe1—C22—C23	−163.4 (3)
C24—Fe1—C12—C11	164.58 (14)	C21—Fe1—C22—C23	−119.9 (2)
C14—Fe1—C12—C11	−82.72 (16)	C12—Fe1—C22—C23	80.04 (18)
C21—Fe1—C12—C11	50.9 (3)	C13—Fe1—C22—C23	46.5 (3)
C22—Fe1—C12—C11	82.38 (17)	C10—Fe1—C22—C21	−76.47 (18)
C23—Fe1—C12—C11	123.96 (15)	C20—Fe1—C22—C21	38.17 (14)
C13—Fe1—C12—C11	−119.7 (2)	C24—Fe1—C22—C21	82.59 (16)
C10—Fe1—C12—C13	81.52 (16)	C11—Fe1—C22—C21	−117.83 (15)
C20—Fe1—C12—C13	−46.5 (4)	C14—Fe1—C22—C21	−43.5 (4)
C24—Fe1—C12—C13	−75.70 (18)	C23—Fe1—C22—C21	119.9 (2)
C11—Fe1—C12—C13	119.7 (2)	C12—Fe1—C22—C21	−160.04 (14)
C14—Fe1—C12—C13	37.01 (15)	C13—Fe1—C22—C21	166.5 (2)
C21—Fe1—C12—C13	170.7 (2)	C21—C22—C23—C24	−0.2 (3)
C22—Fe1—C12—C13	−157.90 (16)	Fe1—C22—C23—C24	58.57 (18)
C23—Fe1—C12—C13	−116.32 (16)	C21—C22—C23—Fe1	−58.73 (17)
C11—C12—C13—C14	0.3 (3)	C10—Fe1—C23—C22	−48.0 (4)
Fe1—C12—C13—C14	−58.39 (17)	C20—Fe1—C23—C22	81.67 (16)
C11—C12—C13—Fe1	58.68 (18)	C24—Fe1—C23—C22	119.9 (2)
C10—Fe1—C13—C14	38.62 (15)	C11—Fe1—C23—C22	−76.42 (19)
C20—Fe1—C13—C14	−75.94 (18)	C14—Fe1—C23—C22	169.9 (2)
C24—Fe1—C13—C14	−117.17 (16)	C21—Fe1—C23—C22	37.34 (15)
C11—Fe1—C13—C14	83.02 (16)	C12—Fe1—C23—C22	−116.68 (16)
C21—Fe1—C13—C14	−45.3 (4)	C13—Fe1—C23—C22	−158.43 (16)
C22—Fe1—C13—C14	168.1 (2)	C10—Fe1—C23—C24	−167.9 (3)
C23—Fe1—C13—C14	−159.34 (15)	C20—Fe1—C23—C24	−38.20 (15)
C12—Fe1—C13—C14	120.3 (2)	C11—Fe1—C23—C24	163.70 (15)
C10—Fe1—C13—C12	−81.64 (16)	C14—Fe1—C23—C24	50.0 (3)
C20—Fe1—C13—C12	163.80 (14)	C21—Fe1—C23—C24	−82.54 (17)
C24—Fe1—C13—C12	122.57 (16)	C22—Fe1—C23—C24	−119.9 (2)
C11—Fe1—C13—C12	−37.25 (15)	C12—Fe1—C23—C24	123.44 (16)
C14—Fe1—C13—C12	−120.3 (2)	C13—Fe1—C23—C24	81.69 (18)
C21—Fe1—C13—C12	−165.5 (3)	C22—C23—C24—C20	0.2 (3)
C22—Fe1—C13—C12	47.8 (3)	Fe1—C23—C24—C20	59.22 (17)
C23—Fe1—C13—C12	80.39 (17)	C22—C23—C24—Fe1	−58.97 (18)
C12—C13—C14—C10	−0.5 (3)	C21—C20—C24—C23	−0.2 (3)
Fe1—C13—C14—C10	−59.28 (16)	C25—C20—C24—C23	−174.8 (2)
C12—C13—C14—Fe1	58.74 (18)	Fe1—C20—C24—C23	−59.93 (18)
C11—C10—C14—C13	0.6 (3)	C21—C20—C24—Fe1	59.70 (17)
C15—C10—C14—C13	178.2 (2)	C25—C20—C24—Fe1	−114.8 (2)

Fe1—C10—C14—C13	60.25 (17)	C10—Fe1—C24—C23	170.7 (2)
C11—C10—C14—Fe1	−59.65 (16)	C20—Fe1—C24—C23	119.0 (2)
C15—C10—C14—Fe1	117.9 (3)	C11—Fe1—C24—C23	−43.7 (4)
C10—Fe1—C14—C13	−118.4 (2)	C14—Fe1—C24—C23	−157.28 (16)
C20—Fe1—C14—C13	123.83 (15)	C21—Fe1—C24—C23	80.91 (17)
C24—Fe1—C14—C13	80.22 (18)	C22—Fe1—C24—C23	37.19 (17)
C11—Fe1—C14—C13	−80.46 (16)	C12—Fe1—C24—C23	−74.39 (19)
C21—Fe1—C14—C13	165.70 (15)	C13—Fe1—C24—C23	−115.46 (17)
C22—Fe1—C14—C13	−160.1 (3)	C10—Fe1—C24—C20	51.7 (3)
C23—Fe1—C14—C13	45.1 (3)	C11—Fe1—C24—C20	−162.7 (3)
C12—Fe1—C14—C13	−37.08 (15)	C14—Fe1—C24—C20	83.73 (16)
C20—Fe1—C14—C10	−117.79 (14)	C21—Fe1—C24—C20	−38.08 (14)
C24—Fe1—C14—C10	−161.39 (14)	C22—Fe1—C24—C20	−81.80 (16)
C11—Fe1—C14—C10	37.93 (14)	C23—Fe1—C24—C20	−119.0 (2)
C21—Fe1—C14—C10	−75.92 (17)	C12—Fe1—C24—C20	166.62 (14)
C22—Fe1—C14—C10	−41.7 (4)	C13—Fe1—C24—C20	125.56 (15)
C23—Fe1—C14—C10	163.4 (2)	C26—O22—C25—O21	−2.5 (3)
C12—Fe1—C14—C10	81.31 (15)	C26—O22—C25—C20	176.6 (2)
C13—Fe1—C14—C10	118.4 (2)	C21—C20—C25—O21	5.1 (4)
N11—O12—C15—O11	1.0 (3)	C24—C20—C25—O21	178.8 (2)
N11—O12—C15—C10	−179.20 (18)	Fe1—C20—C25—O21	91.0 (3)
C11—C10—C15—O11	−1.8 (4)	C21—C20—C25—O22	−174.0 (2)
C14—C10—C15—O11	−179.1 (2)	C24—C20—C25—O22	−0.3 (3)
Fe1—C10—C15—O11	−88.1 (3)	Fe1—C20—C25—O22	−88.0 (2)
C11—C10—C15—O12	178.4 (2)	C18—C19—C110—C111	1.1 (4)
C14—C10—C15—O12	1.1 (3)	N12—N13—C111—C16	−0.3 (3)
Fe1—C10—C15—O12	92.1 (2)	N12—N13—C111—C110	178.5 (3)
N12—N11—C16—C111	−2.5 (3)	N11—C16—C111—N13	1.6 (3)
O12—N11—C16—C111	−177.5 (2)	C17—C16—C111—N13	179.9 (2)
N12—N11—C16—C17	179.5 (3)	N11—C16—C111—C110	−177.3 (2)
O12—N11—C16—C17	4.5 (5)	C17—C16—C111—C110	1.0 (4)
N11—C16—C17—C18	178.7 (3)	C19—C110—C111—N13	179.3 (3)
C111—C16—C17—C18	1.0 (4)	C19—C110—C111—C16	−2.0 (4)

Fig. 1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