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Bis(μ-{(1,3-phenylene)[*tert*-butyl(pyrazol-1-yl)borato][*tert*-butyl(dipyrazol-1-yl)borato]})dihydroxydiiron(II) tetrahydrofuran disolvate

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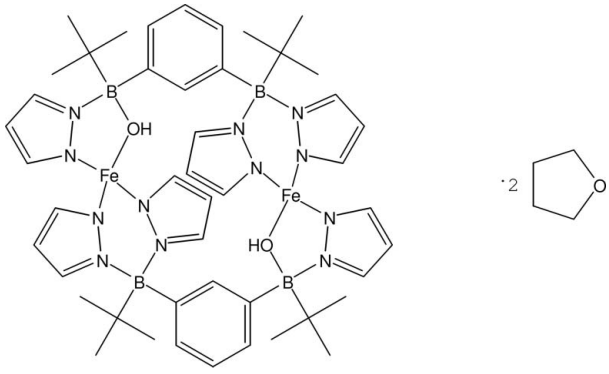
Key indicators: single-crystal X-ray study; T = 173 K; mean σ(C–C) = 0.010 Å; R factor = 0.062; wR factor = 0.155; data-to-parameter ratio = 15.7.

The title compound, $[Fe_2(C_{23}H_{32}B_2N_6O)_2]\cdot 2C_4H_8O$, features a dinuclear complex located on a centre of inversion. It crystallizes with two molecules of tetrahydrofuran in the asymmetric unit. The molecular conformation of a pseudo-polymorph of the title complex with two molecules of toluene in the asymmetric unit is almost identical. It is noteworthy that the hydroxyl H atom is shielded from any classical hydrogen-bond acceptor and there is only a short contact to the centre of an aromatic ring (centroid···H = 2.48 Å).

Related literature

For the toluene disolvate and synthesis, see: Zhang *et al.* (2007).



Experimental

Crystal data

$[Fe_2(C_{23}H_{32}B_2N_6O)_2]\cdot 2C_4H_8O$
M_r = 1116.24
Triclinic, $P\overline{1}$
a = 11.645 (2) Å
b = 12.363 (2) Å
c = 12.507 (2) Å
α = 71.619 (12)°
β = 63.139 (12)°
γ = 66.862 (13)°
V = 1456.1 (5) Å^3
Z = 1
Mo $K\alpha$ radiation
μ = 0.55 mm^{-1}
T = 173 (2) K
0.32 × 0.24 × 0.13 mm

Data collection

Stoe IPDS II two-circle diffractometer
Absorption correction: multi-scan (*MULABS*; Spek, 2003; Blessing, 1995)
T_{min} = 0.843, T_{max} = 0.932
12022 measured reflections
5438 independent reflections
3411 reflections with $I > 2\sigma(I)$
R_{int} = 0.079

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.063
$wR(F^2)$ = 0.155
S = 0.89
5438 reflections
346 parameters
3 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.71 e Å^{-3}
$\Delta\rho_{min}$ = −0.48 e Å^{-3}

Data collection: *X-AREA* (Stoe & Cie, 2001); cell refinement: *X-AREA*; data reduction: *X-AREA*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *XP* in *SHELXTL-Plus* (Sheldrick, 1991); software used to prepare material for publication: *SHELXL97* and *PLATON* (Spek, 2003).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: AT2388).

References

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Zhang, F., Morawitz, T., Bieller, S., Bolte, M., Lerner, H.-W. & Wagner, M. (2007). *J. Chem. Soc. Dalton Trans.* DOI 10.1039/b707807b.

supplementary materials

Acta Cryst. (2007). E**63**, m2505 [**doi:10.1107/S1600536807043346**]

Bis(μ-{(1,3-phenylene)[*tert*-butyl(pyrazol-1-yl)borato][*tert*-butyl(dipyrazol-1-yl)borato]})dihydroxydiiron(II) tetrahydrofuran disolvate

F. Zhang, H.-W. Lerner and M. Bolte

Comment

Careful hydrolysis of the homoditopic phenylene-bridged Li and K scorpionates [*m*-$(pz_2BtBu)_2C_6H_4]^{2-}$ leads to the corresponding heteroditopic derivative [*m*-$(pz(HO)BtBu)(pz_2BtBu)C_6H_4]^{2-}$. We report here the X-ray crystal structure analysis of the macrocyclic complex (I). Very recently we have described the synthesis of the metallomacrocycle (I) (Zhang *et al.*, 2007). X-ray quality crystals of the macrocycle (I) were grown from a tetrahydrofuran solution at ambient temperature.

Now we present a pseudopolymorph of this complex crystallized from tetrahydrofuran (Fig. 1). Both structures feature dinuclear complexes located on a centre of inversion. The title complex crystallizes with two molecules of tetrahydrofuran in the unit cell. It is noteworthy, that the hydroxyl H atom is shielded from any classical hydrogen bond acceptor. There is just a short contact to the centre of an aromatic ring [O1—H1 0.72 (5) Å, H1···cog($C41^{i}$—$C46^{i}$) 2.48 Å, O1—H1···cog 132.4°; symmetry operator (i): $1 - x$, $1 - y$, $1 - z$]. A least-squares of the complexes of the two pseudopolymorphs fitting all non-H atoms (r.m.s. deviation 0.091 Å) (Fig. 2) shows that the molecular conformations are almost identical. Thus, the solvent molecules in both crystal structures have no influence on the conformation of the complex.

Experimental

The title compound (I) was synthesized according to the method described by Zhang *et al.* (2007). X-ray quality crystals of (I) (0.2 mmol) were grown from a tetrahydrofuran solution (10 ml) at ambient temperature.

Refinement

H atoms were geometrically positioned and refined using a riding model with fixed individual displacement parameters [U_{iso}(H) = 1.2 U_{eq}(C,*O*) or U_{iso}(H) = 1.5 $U_{eq}(C_{methyl})$] and C—H ranging from 0.95Å to 0.99 Å. The coordinates of the hydroxyl H atom were refined. The C—C distance of the the molecules were refined with a distance restraint of 1.50 (1) Å.

Figures

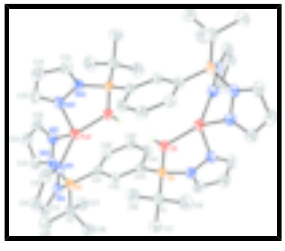


Fig. 1. Perspective view of the title compound with the atom numbering; displacement ellipsoids are at the 50% probability level. H atoms bonded to C and the solvent molecules omitted for clarity. Symmetry operator for generating equivalent atoms: $-x + 1$, $-y + 1$, $-z + 1$.

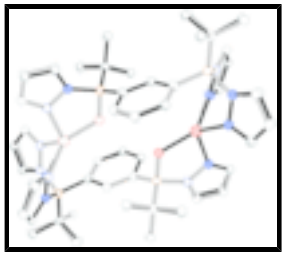


Fig. 2. Least-squares fit of the title complex (open bonds) with its pseudopolymorph crystallized with toluene (full bonds).

Crystal data

$[Fe_2(C_{23}H_{32}B_2N_6O)_2]\cdot 2C_4H_8O$	$Z = 1$
$M_r = 1116.24$	$F_{000} = 592$
Triclinic, $P\overline{1}$	$D_x = 1.273$ Mg m^{-3}
Hall symbol: -P 1	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$a = 11.645$ (2) Å	Cell parameters from 10281 reflections
$b = 12.363$ (2) Å	$\theta = 3.4$–25.8º
$c = 12.507$ (2) Å	$\mu = 0.55$ mm^{-1}
$\alpha = 71.619$ (12)º	$T = 173$ (2) K
$\beta = 63.139$ (12)º	Block, colourless
$\gamma = 66.862$ (13)º	0.32 × 0.24 × 0.13 mm
$V = 1456.1$ (5) Å^3	

Data collection

STOE IPDS II two-circle diffractometer	5438 independent reflections
Radiation source: fine-focus sealed tube	3411 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.079$
$T = 173$(2) K	$\theta_{max} = 25.7$º
ω scans	$\theta_{min} = 3.5$º
Absorption correction: multi-scan (MULABS; Spek, 2003; Blessing, 1995)	$h = -14\rightarrow14$
$T_{min} = 0.843$, $T_{max} = 0.932$	$k = -14\rightarrow15$
12022 measured reflections	$l = -15\rightarrow15$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.063$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.155$	$w = 1/[\sigma^2(F_o^2) + (0.0841P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 0.89$	$(\Delta/\sigma)_{max} = 0.006$

5438 reflections	$\Delta\rho_{max} = 0.71$ e Å^{-3}
346 parameters	$\Delta\rho_{min} = -0.48$ e Å^{-3}
3 restraints	Extinction correction: none
Primary atom site location: structure-invariant direct methods	

Special details

Experimental.

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 $F^2 > \sigma(F^2)$ 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	0.67506 (6)	0.23657 (5)	0.43545 (5)	0.03806 (19)
B1	0.6272 (4)	0.6466 (4)	0.4266 (4)	0.0347 (9)
C1	0.7258 (4)	0.6458 (4)	0.4873 (4)	0.0446 (10)
C16	0.6609 (5)	0.7524 (5)	0.5551 (5)	0.0614 (13)
H16A	0.6451	0.8269	0.4982	0.092*
H16B	0.7215	0.7525	0.5898	0.092*
H16C	0.5750	0.7458	0.6206	0.092*
C17	0.8641 (5)	0.6558 (4)	0.3918 (5)	0.0584 (12)
H17A	0.8517	0.7278	0.3311	0.088*
H17B	0.9115	0.5854	0.3517	0.088*
H17C	0.9172	0.6606	0.4316	0.088*
C18	0.7467 (5)	0.5330 (4)	0.5815 (4)	0.0534 (11)
H18A	0.7884	0.4625	0.5421	0.080*
H18B	0.6594	0.5289	0.6461	0.080*
H18C	0.8055	0.5354	0.6167	0.080*
O1	0.4944 (3)	0.6315 (2)	0.5255 (3)	0.0378 (6)
H1	0.486 (5)	0.577 (4)	0.568 (4)	0.045*
B2	0.9482 (4)	0.2460 (4)	0.2463 (4)	0.0372 (10)
C2	1.0880 (4)	0.2689 (4)	0.1373 (4)	0.0451 (10)
C26	1.2155 (4)	0.1607 (4)	0.1204 (5)	0.0588 (12)
H26A	1.2004	0.0955	0.1050	0.088*
H26B	1.2358	0.1346	0.1942	0.088*
H26C	1.2916	0.1833	0.0512	0.088*
C27	1.0681 (5)	0.3157 (4)	0.0148 (4)	0.0559 (12)
H27A	1.0500	0.2550	−0.0051	0.084*
H27B	1.1498	0.3333	−0.0495	0.084*

H27C	0.9917	0.3886	0.0213	0.084*
C28	1.1163 (5)	0.3695 (4)	0.1599 (5)	0.0547 (11)
H28A	1.0363	0.4394	0.1716	0.082*
H28B	1.1924	0.3909	0.0898	0.082*
H28C	1.1382	0.3423	0.2329	0.082*
N11	0.8418 (3)	0.1957 (3)	0.4743 (3)	0.0384 (7)
N12	0.9571 (3)	0.2016 (3)	0.3765 (3)	0.0363 (7)
C13	1.0580 (4)	0.1641 (4)	0.4176 (4)	0.0451 (10)
H13	1.1492	0.1607	0.3676	0.054*
C14	1.0105 (5)	0.1319 (4)	0.5403 (4)	0.0510 (11)
H14	1.0597	0.1020	0.5921	0.061*
C15	0.8726 (5)	0.1526 (4)	0.5735 (4)	0.0472 (10)
H15	0.8104	0.1384	0.6542	0.057*
N21	0.7824 (3)	0.1335 (3)	0.3015 (3)	0.0377 (7)
N22	0.9084 (3)	0.1441 (3)	0.2278 (3)	0.0374 (7)
C23	0.9705 (5)	0.0626 (4)	0.1544 (4)	0.0466 (10)
H23	1.0594	0.0507	0.0946	0.056*
C24	0.8853 (5)	−0.0011 (4)	0.1795 (4)	0.0496 (11)
H24	0.9026	−0.0642	0.1413	0.060*
C25	0.7695 (5)	0.0458 (4)	0.2716 (4)	0.0456 (10)
H25	0.6915	0.0195	0.3086	0.055*
N31	0.5511 (3)	0.1507 (3)	0.5870 (3)	0.0409 (8)
N32	0.4261 (3)	0.2227 (3)	0.6412 (3)	0.0357 (7)
C33	0.3659 (5)	0.1651 (4)	0.7500 (4)	0.0456 (10)
H33	0.2777	0.1966	0.8057	0.055*
C34	0.4508 (5)	0.0533 (4)	0.7695 (4)	0.0548 (11)
H34	0.4346	−0.0074	0.8388	0.066*
C35	0.5645 (5)	0.0494 (4)	0.6655 (4)	0.0512 (11)
H35	0.6431	−0.0174	0.6513	0.061*
C41	0.8185 (4)	0.3640 (3)	0.2563 (3)	0.0344 (8)
C42	0.7845 (4)	0.4487 (3)	0.3267 (3)	0.0337 (8)
H42	0.8367	0.4323	0.3735	0.040*
C43	0.6796 (3)	0.5553 (3)	0.3330 (3)	0.0328 (8)
C44	0.6057 (4)	0.5772 (3)	0.2612 (4)	0.0368 (8)
H44	0.5330	0.6484	0.2627	0.044*
C45	0.6361 (4)	0.4975 (4)	0.1885 (4)	0.0394 (9)
H45	0.5851	0.5151	0.1403	0.047*
C46	0.7406 (4)	0.3923 (4)	0.1858 (3)	0.0382 (9)
H46	0.7602	0.3383	0.1358	0.046*
O51	0.2229 (15)	0.7421 (11)	0.1334 (15)	0.305 (8)
C52	0.2894 (18)	0.8316 (14)	0.0926 (15)	0.203 (7)
H52A	0.2883	0.8553	0.1617	0.244*
H52B	0.2481	0.9031	0.0436	0.244*
C53	0.4281 (17)	0.7680 (13)	0.0174 (15)	0.332 (19)
H53A	0.4875	0.7364	0.0641	0.399*
H53B	0.4665	0.8225	−0.0566	0.399*
C54	0.4151 (11)	0.6687 (18)	−0.0155 (10)	0.273 (14)
H54A	0.3975	0.6960	−0.0915	0.327*
H54B	0.4991	0.6011	−0.0277	0.327*

C55	0.3015 (14)	0.6316 (10)	0.0861 (10)	0.158 (5)
H55A	0.3315	0.5668	0.1471	0.189*
H55B	0.2511	0.6059	0.0576	0.189*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Fe1	0.0325 (3)	0.0385 (3)	0.0401 (3)	−0.0055 (2)	−0.0123 (2)	−0.0112 (3)
B1	0.030 (2)	0.031 (2)	0.036 (2)	−0.0068 (17)	−0.0083 (17)	−0.0065 (18)
C1	0.043 (2)	0.041 (2)	0.055 (3)	−0.0087 (18)	−0.023 (2)	−0.012 (2)
C16	0.065 (3)	0.057 (3)	0.079 (3)	−0.005 (2)	−0.041 (3)	−0.028 (3)
C17	0.045 (2)	0.054 (3)	0.083 (3)	−0.015 (2)	−0.025 (2)	−0.017 (3)
C18	0.061 (3)	0.050 (3)	0.056 (3)	−0.007 (2)	−0.035 (2)	−0.009 (2)
O1	0.0384 (14)	0.0317 (15)	0.0375 (16)	−0.0121 (12)	−0.0104 (12)	−0.0022 (12)
B2	0.039 (2)	0.033 (2)	0.038 (2)	−0.0060 (18)	−0.0158 (19)	−0.0088 (19)
C2	0.041 (2)	0.038 (2)	0.046 (2)	−0.0076 (17)	−0.0095 (18)	−0.0093 (19)
C26	0.039 (2)	0.055 (3)	0.068 (3)	−0.006 (2)	−0.008 (2)	−0.022 (2)
C27	0.055 (3)	0.055 (3)	0.042 (2)	−0.016 (2)	−0.006 (2)	−0.006 (2)
C28	0.046 (2)	0.052 (3)	0.062 (3)	−0.019 (2)	−0.012 (2)	−0.010 (2)
N11	0.0416 (17)	0.0377 (18)	0.0355 (18)	−0.0098 (14)	−0.0173 (15)	−0.0038 (14)
N12	0.0354 (16)	0.0326 (16)	0.0384 (18)	−0.0056 (13)	−0.0151 (14)	−0.0067 (14)
C13	0.041 (2)	0.042 (2)	0.058 (3)	−0.0080 (18)	−0.027 (2)	−0.008 (2)
C14	0.056 (3)	0.046 (2)	0.061 (3)	−0.009 (2)	−0.039 (2)	−0.004 (2)
C15	0.058 (3)	0.040 (2)	0.044 (2)	−0.012 (2)	−0.024 (2)	−0.0037 (19)
N21	0.0368 (16)	0.0390 (18)	0.0372 (17)	−0.0112 (14)	−0.0125 (14)	−0.0085 (14)
N22	0.0375 (17)	0.0334 (17)	0.0361 (17)	−0.0052 (13)	−0.0123 (14)	−0.0083 (14)
C23	0.055 (2)	0.039 (2)	0.042 (2)	−0.0086 (19)	−0.015 (2)	−0.0126 (19)
C24	0.067 (3)	0.039 (2)	0.051 (3)	−0.015 (2)	−0.025 (2)	−0.016 (2)
C25	0.053 (2)	0.042 (2)	0.049 (2)	−0.0166 (19)	−0.025 (2)	−0.005 (2)
N31	0.0376 (17)	0.0328 (17)	0.051 (2)	−0.0016 (14)	−0.0199 (15)	−0.0112 (16)
N32	0.0355 (16)	0.0327 (17)	0.0368 (18)	−0.0100 (14)	−0.0108 (14)	−0.0074 (14)
C33	0.052 (2)	0.042 (2)	0.042 (2)	−0.0207 (19)	−0.0134 (19)	−0.0037 (19)
C34	0.070 (3)	0.041 (2)	0.054 (3)	−0.019 (2)	−0.031 (2)	0.007 (2)
C35	0.055 (3)	0.030 (2)	0.063 (3)	−0.0039 (19)	−0.028 (2)	−0.004 (2)
C41	0.0351 (19)	0.034 (2)	0.0326 (19)	−0.0124 (16)	−0.0110 (16)	−0.0041 (16)
C42	0.0333 (18)	0.0298 (19)	0.038 (2)	−0.0080 (15)	−0.0138 (16)	−0.0072 (16)
C43	0.0302 (18)	0.0296 (18)	0.0341 (19)	−0.0093 (14)	−0.0091 (15)	−0.0034 (16)
C44	0.0361 (19)	0.0302 (19)	0.041 (2)	−0.0052 (15)	−0.0169 (17)	−0.0036 (17)
C45	0.043 (2)	0.040 (2)	0.037 (2)	−0.0107 (17)	−0.0211 (17)	−0.0025 (17)
C46	0.044 (2)	0.039 (2)	0.0308 (19)	−0.0134 (17)	−0.0128 (17)	−0.0057 (17)
O51	0.276 (14)	0.186 (10)	0.390 (19)	−0.067 (10)	−0.006 (13)	−0.154 (12)
C52	0.28 (2)	0.162 (12)	0.262 (18)	−0.106 (14)	−0.177 (17)	0.019 (12)
C53	0.173 (14)	0.34 (3)	0.35 (3)	−0.134 (17)	−0.159 (18)	0.27 (2)
C54	0.086 (7)	0.60 (4)	0.111 (8)	0.005 (13)	−0.031 (6)	−0.185 (16)
C55	0.229 (14)	0.142 (9)	0.146 (9)	−0.029 (9)	−0.132 (10)	−0.020 (7)

Geometric parameters (Å, °)

Fe1—O1^{i}	1.973 (3)	C14—C15	1.399 (6)
Fe1—N11	2.046 (3)	C14—H14	0.9500
Fe1—N21	2.048 (3)	C15—H15	0.9500
Fe1—N31	2.065 (4)	N21—C25	1.331 (5)
B1—O1	1.520 (5)	N21—N22	1.369 (5)
B1—N32^{i}	1.593 (5)	N22—C23	1.339 (5)
B1—C43	1.632 (5)	C23—C24	1.371 (7)
B1—C1	1.633 (6)	C23—H23	0.9500
C1—C18	1.533 (6)	C24—C25	1.372 (6)
C1—C17	1.536 (7)	C24—H24	0.9500
C1—C16	1.540 (6)	C25—H25	0.9500
C16—H16A	0.9800	N31—C35	1.329 (6)
C16—H16B	0.9800	N31—N32	1.357 (4)
C16—H16C	0.9800	N32—C33	1.330 (5)
C17—H17A	0.9800	N32—B1^{i}	1.593 (5)
C17—H17B	0.9800	C33—C34	1.370 (6)
C17—H17C	0.9800	C33—H33	0.9500
C18—H18A	0.9800	C34—C35	1.371 (7)
C18—H18B	0.9800	C34—H34	0.9500
C18—H18C	0.9800	C35—H35	0.9500
O1—Fe1^{i}	1.973 (3)	C41—C42	1.404 (5)
O1—H1	0.72 (5)	C41—C46	1.415 (5)
B2—N12	1.584 (5)	C42—C43	1.396 (5)
B2—N22	1.610 (5)	C42—H42	0.9500
B2—C41	1.625 (5)	C43—C44	1.408 (5)
B2—C2	1.644 (6)	C44—C45	1.387 (5)
C2—C28	1.537 (6)	C44—H44	0.9500
C2—C26	1.540 (6)	C45—C46	1.386 (5)
C2—C27	1.547 (6)	C45—H45	0.9500
C26—H26A	0.9800	C46—H46	0.9500
C26—H26B	0.9800	O51—C55	1.447 (13)
C26—H26C	0.9800	O51—C52	1.429 (17)
C27—H27A	0.9800	C52—C53	1.485 (9)
C27—H27B	0.9800	C52—H52A	0.9900
C27—H27C	0.9800	C52—H52B	0.9900
C28—H28A	0.9800	C53—C54	1.491 (10)
C28—H28B	0.9800	C53—H53A	0.9900
C28—H28C	0.9800	C53—H53B	0.9900
N11—C15	1.337 (5)	C54—C55	1.472 (9)
N11—N12	1.357 (5)	C54—H54A	0.9900
N12—C13	1.351 (5)	C54—H54B	0.9900
C13—C14	1.357 (7)	C55—H55A	0.9900
C13—H13	0.9500	C55—H55B	0.9900
O1^{i}—Fe1—N11	130.37 (12)	C13—C14—H14	127.8
O1^{i}—Fe1—N21	135.07 (12)	C15—C14—H14	127.8

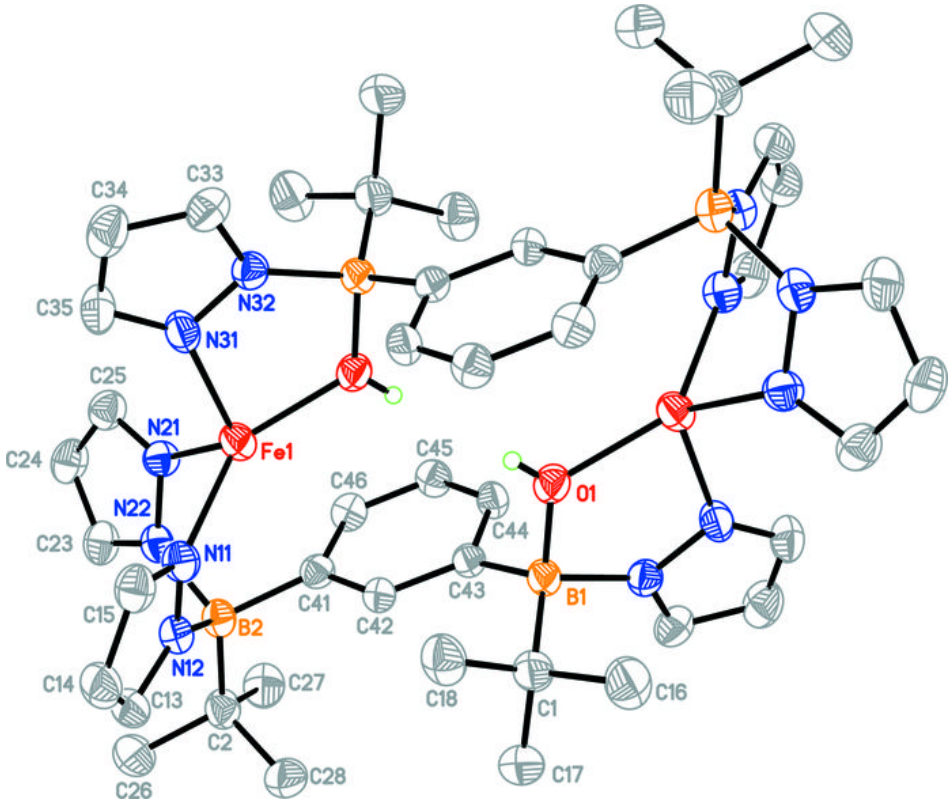
N11—Fe1—N21	91.19 (13)	N11—C15—C14	109.6 (4)
O1^{i}—Fe1—N31	79.47 (12)	N11—C15—H15	125.2
N11—Fe1—N31	103.53 (13)	C14—C15—H15	125.2
N21—Fe1—N31	110.22 (13)	C25—N21—N22	107.1 (3)
O1—B1—N32^{i}	99.5 (3)	C25—N21—Fe1	136.8 (3)
O1—B1—C43	107.7 (3)	N22—N21—Fe1	115.6 (2)
N32^{i}—B1—C43	106.7 (3)	C23—N22—N21	108.3 (3)
O1—B1—C1	110.0 (3)	C23—N22—B2	135.1 (4)
N32^{i}—B1—C1	110.4 (3)	N21—N22—B2	116.6 (3)
C43—B1—C1	120.5 (3)	N22—C23—C24	109.1 (4)
C18—C1—C17	108.9 (4)	N22—C23—H23	125.5
C18—C1—C16	106.6 (4)	C24—C23—H23	125.5
C17—C1—C16	107.4 (4)	C23—C24—C25	105.3 (4)
C18—C1—B1	111.4 (4)	C23—C24—H24	127.3
C17—C1—B1	112.4 (4)	C25—C24—H24	127.3
C16—C1—B1	109.9 (3)	N21—C25—C24	110.2 (4)
C1—C16—H16A	109.5	N21—C25—H25	124.9
C1—C16—H16B	109.5	C24—C25—H25	124.9
H16A—C16—H16B	109.5	C35—N31—N32	105.9 (3)
C1—C16—H16C	109.5	C35—N31—Fe1	137.6 (3)
H16A—C16—H16C	109.5	N32—N31—Fe1	114.5 (2)
H16B—C16—H16C	109.5	C33—N32—N31	109.1 (3)
C1—C17—H17A	109.5	C33—N32—B1^{i}	130.3 (3)
C1—C17—H17B	109.5	N31—N32—B1^{i}	120.6 (3)
H17A—C17—H17B	109.5	N32—C33—C34	109.6 (4)
C1—C17—H17C	109.5	N32—C33—H33	125.2
H17A—C17—H17C	109.5	C34—C33—H33	125.2
H17B—C17—H17C	109.5	C33—C34—C35	103.8 (4)
C1—C18—H18A	109.5	C33—C34—H34	128.1
C1—C18—H18B	109.5	C35—C34—H34	128.1
H18A—C18—H18B	109.5	N31—C35—C34	111.6 (4)
C1—C18—H18C	109.5	N31—C35—H35	124.2
H18A—C18—H18C	109.5	C34—C35—H35	124.2
H18B—C18—H18C	109.5	C42—C41—C46	116.2 (3)
B1—O1—Fe1^{i}	123.8 (2)	C42—C41—B2	122.1 (3)
B1—O1—H1	125 (4)	C46—C41—B2	121.5 (3)
Fe1^{i}—O1—H1	111 (4)	C43—C42—C41	124.7 (3)
N12—B2—N22	104.7 (3)	C43—C42—H42	117.6
N12—B2—C41	106.8 (3)	C41—C42—H42	117.6
N22—B2—C41	105.7 (3)	C42—C43—C44	116.0 (3)
N12—B2—C2	113.3 (3)	C42—C43—B1	125.2 (3)
N22—B2—C2	112.6 (3)	C44—C43—B1	118.5 (3)
C41—B2—C2	113.0 (3)	C45—C44—C43	121.8 (3)
C28—C2—C26	107.9 (4)	C45—C44—H44	119.1
C28—C2—C27	105.5 (4)	C43—C44—H44	119.1
C26—C2—C27	107.9 (4)	C46—C45—C44	120.3 (3)
C28—C2—B2	109.2 (3)	C46—C45—H45	119.8

C26—C2—B2	116.3 (4)	C44—C45—H45	119.8
C27—C2—B2	109.5 (4)	C45—C46—C41	121.0 (3)
C2—C26—H26A	109.5	C45—C46—H46	119.5
C2—C26—H26B	109.5	C41—C46—H46	119.5
H26A—C26—H26B	109.5	C55—O51—C52	116.5 (13)
C2—C26—H26C	109.5	O51—C52—C53	101.5 (13)
H26A—C26—H26C	109.5	O51—C52—H52A	111.5
H26B—C26—H26C	109.5	C53—C52—H52A	111.5
C2—C27—H27A	109.5	O51—C52—H52B	111.5
C2—C27—H27B	109.5	C53—C52—H52B	111.5
H27A—C27—H27B	109.5	H52A—C52—H52B	109.3
C2—C27—H27C	109.5	C52—C53—C54	105.8 (13)
H27A—C27—H27C	109.5	C52—C53—H53A	110.6
H27B—C27—H27C	109.5	C54—C53—H53A	110.6
C2—C28—H28A	109.5	C52—C53—H53B	110.6
C2—C28—H28B	109.5	C54—C53—H53B	110.6
H28A—C28—H28B	109.5	H53A—C53—H53B	108.7
C2—C28—H28C	109.5	C55—C54—C53	106.8 (10)
H28A—C28—H28C	109.5	C55—C54—H54A	110.4
H28B—C28—H28C	109.5	C53—C54—H54A	110.4
C15—N11—N12	107.8 (3)	C55—C54—H54B	110.4
C15—N11—Fe1	136.3 (3)	C53—C54—H54B	110.4
N12—N11—Fe1	115.5 (2)	H54A—C54—H54B	108.6
C13—N12—N11	107.7 (3)	C54—C55—O51	100.1 (10)
C13—N12—B2	134.6 (3)	C54—C55—H55A	111.8
N11—N12—B2	117.7 (3)	O51—C55—H55A	111.8
N12—C13—C14	110.4 (4)	C54—C55—H55B	111.8
N12—C13—H13	124.8	O51—C55—H55B	111.8
C14—C13—H13	124.8	H55A—C55—H55B	109.5
C13—C14—C15	104.4 (4)		
O1—B1—C1—C18	55.2 (4)	C41—B2—N22—C23	134.9 (4)
N32^{i}—B1—C1—C18	164.1 (3)	C2—B2—N22—C23	11.0 (6)
C43—B1—C1—C18	−70.9 (5)	N12—B2—N22—N21	67.6 (4)
O1—B1—C1—C17	177.7 (3)	C41—B2—N22—N21	−45.0 (4)
N32^{i}—B1—C1—C17	−73.4 (4)	C2—B2—N22—N21	−168.9 (3)
C43—B1—C1—C17	51.6 (5)	N21—N22—C23—C24	0.3 (5)
O1—B1—C1—C16	−62.7 (5)	B2—N22—C23—C24	−179.6 (4)
N32^{i}—B1—C1—C16	46.1 (5)	N22—C23—C24—C25	−0.3 (5)
C43—B1—C1—C16	171.1 (4)	N22—N21—C25—C24	0.0 (5)
N32^{i}—B1—O1—Fe1^{i}	−12.4 (4)	Fe1—N21—C25—C24	−171.5 (3)
C43—B1—O1—Fe1^{i}	−123.4 (3)	C23—C24—C25—N21	0.1 (5)
C1—B1—O1—Fe1^{i}	103.5 (3)	O1^{i}—Fe1—N31—C35	−165.3 (4)
N12—B2—C2—C28	−63.1 (4)	N11—Fe1—N31—C35	−36.0 (4)
N22—B2—C2—C28	178.4 (3)	N21—Fe1—N31—C35	60.4 (4)
C41—B2—C2—C28	58.6 (4)	O1^{i}—Fe1—N31—N32	−4.4 (2)
N12—B2—C2—C26	59.2 (5)	N11—Fe1—N31—N32	124.9 (2)
N22—B2—C2—C26	−59.3 (5)	N21—Fe1—N31—N32	−138.7 (2)

C41—B2—C2—C26	−179.1 (4)	C35—N31—N32—C33	−0.2 (4)
N12—B2—C2—C27	−178.2 (3)	Fe1—N31—N32—C33	−166.9 (3)
N22—B2—C2—C27	63.3 (4)	C35—N31—N32—B1^{i}	−179.6 (3)
C41—B2—C2—C27	−56.5 (4)	Fe1—N31—N32—B1^{i}	13.7 (4)
O1^{i}—Fe1—N11—C15	71.6 (4)	N31—N32—C33—C34	0.0 (5)
N21—Fe1—N11—C15	−127.1 (4)	B1^{i}—N32—C33—C34	179.3 (4)
N31—Fe1—N11—C15	−16.0 (4)	N32—C33—C34—C35	0.3 (5)
O1^{i}—Fe1—N11—N12	−116.0 (2)	N32—N31—C35—C34	0.4 (5)
N21—Fe1—N11—N12	45.2 (3)	Fe1—N31—C35—C34	162.3 (3)
N31—Fe1—N11—N12	156.4 (2)	C33—C34—C35—N31	−0.4 (5)
C15—N11—N12—C13	−1.2 (4)	N12—B2—C41—C42	37.8 (5)
Fe1—N11—N12—C13	−175.7 (3)	N22—B2—C41—C42	148.9 (4)
C15—N11—N12—B2	177.9 (3)	C2—B2—C41—C42	−87.5 (4)
Fe1—N11—N12—B2	3.4 (4)	N12—B2—C41—C46	−147.9 (4)
N22—B2—N12—C13	113.1 (5)	N22—B2—C41—C46	−36.8 (5)
C41—B2—N12—C13	−135.0 (4)	C2—B2—C41—C46	86.8 (4)
C2—B2—N12—C13	−9.9 (6)	C46—C41—C42—C43	1.2 (6)
N22—B2—N12—N11	−65.7 (4)	B2—C41—C42—C43	175.8 (4)
C41—B2—N12—N11	46.2 (4)	C41—C42—C43—C44	−0.7 (6)
C2—B2—N12—N11	171.3 (3)	C41—C42—C43—B1	172.1 (4)
N11—N12—C13—C14	1.0 (4)	O1—B1—C43—C42	−105.1 (4)
B2—N12—C13—C14	−177.9 (4)	N32^{i}—B1—C43—C42	148.9 (4)
N12—C13—C14—C15	−0.4 (5)	C1—B1—C43—C42	22.1 (6)
N12—N11—C15—C14	1.0 (5)	O1—B1—C43—C44	67.6 (4)
Fe1—N11—C15—C14	173.8 (3)	N32^{i}—B1—C43—C44	−38.5 (4)
C13—C14—C15—N11	−0.4 (5)	C1—B1—C43—C44	−165.2 (4)
O1^{i}—Fe1—N21—C25	−72.2 (4)	C42—C43—C44—C45	−0.3 (6)
N11—Fe1—N21—C25	128.0 (4)	B1—C43—C44—C45	−173.6 (4)
N31—Fe1—N21—C25	23.2 (4)	C43—C44—C45—C46	0.8 (6)
O1^{i}—Fe1—N21—N22	116.8 (3)	C44—C45—C46—C41	−0.3 (6)
N11—Fe1—N21—N22	−42.9 (3)	C42—C41—C46—C45	−0.7 (6)
N31—Fe1—N21—N22	−147.8 (2)	B2—C41—C46—C45	−175.3 (4)
C25—N21—N22—C23	−0.2 (4)	C55—O51—C52—C53	−4(2)
Fe1—N21—N22—C23	173.4 (3)	O51—C52—C53—C54	20.9 (17)
C25—N21—N22—B2	179.7 (3)	C52—C53—C54—C55	−31.5 (17)
Fe1—N21—N22—B2	−6.7 (4)	C53—C54—C55—O51	27.2 (15)
N12—B2—N22—C23	−112.5 (5)	C52—O51—C55—C54	−14.7 (17)

Symmetry codes: (i) −*x*+1, −*y*+1, −*z*+1.

Fig. 1


C34
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C26
C28
C45
C46
C44
O1
C41
C42
C43
B1
C18
C1
C16
C17

Fig. 2