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Poly[(μ_2-*N*,*N*-dimethylformamide-κ^2O:O)(μ_4-terephthalato-κ^4O:O':O'':O''')iron(II)]

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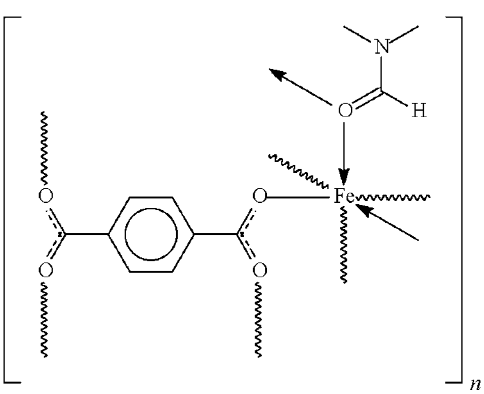
Key indicators: single-crystal X-ray study; T = 295 K; mean σ(C–C) = 0.004 Å; disorder in main residue; R factor = 0.040; wR factor = 0.113; data-to-parameter ratio = 12.6.

In the crystal structure of the title compound, $[Fe(C_8H_4O_4)(C_3H_7NO)]_n$, the Fe^{II} atom and the terephthalate group occupy special positions of $2/m$ site symmetry. The Fe^{II} atom is octahedrally coordinated: two O atoms of two dimethylformamide molecules occupy the axial positions, and the equatorial sites are occupied by the carboxylate O atoms of four different terephthalate groups. The compound adopts a polymeric three-dimensional framework structure. The C and N atoms of the dimethylformamide ligand are disordered equally over two sites each, with further disorder of the H atoms.

Related literature

This Fe^{II} compound has the same structure as the Co^{II} analogue (Fu *et al.*, 2004).



Experimental

Crystal data

$[Fe(C_8H_4O_4)(C_3H_7NO)]$
M_r = 293.06
Orthorhombic, *Imma*
a = 19.3652 (15) Å
b = 7.2856 (6) Å
c = 8.8571 (7) Å
V = 1249.62 (17) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 1.22 mm^{-1}
T = 295 (2) K
0.25 × 0.19 × 0.08 mm

Data collection

Bruker APEX area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.357, T_{max} = 0.909
3421 measured reflections
791 independent reflections
730 reflections with $I > 2\sigma(I)$
R_{int} = 0.021

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.040
$wR(F^2)$ = 0.113
S = 1.08
791 reflections
63 parameters
3 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.73 e Å^{-3}
$\Delta\rho_{min}$ = −0.66 e Å^{-3}

Data collection: *SMART* (Bruker, 2002); cell refinement: *SAINT* (Bruker, 2002); data reduction: *SAINT*; method used to solve structure: atomic coordinates taken from Fu *et al.* (2004); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *X-SEED* (Barbour, 2001); software used to prepare material for publication: *publCIF* (Westrip, 2007).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT2503).

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supplementary materials

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Poly[(μ_2-*N*,*N*-dimethylformamide-κ^2*O*:*O*)(μ_4-terephthalato-κ^4*O*:*O*':*O*'':*O*''')iron(II)]

H. Lu, Y.-L. Fu, Y. Zhang and S. W. Ng

Comment

This study continues with the study on [($C_8H_4O_4$)(C_3H_7NO)Co]$_n$ (Fu *et al.*, 2004; the present Fe(II) analog is isostructural with the Co(II) analog (Fu *et al.*, 2004).

Experimental

Ferrous chloride tetrahydrate (0.198 g, 1 mmol), terephthalic acid (0.166 g, 1 mmol) and *N*,*N*-dimethylformamide (10 ml) were sealed in a 15-ml, Teflon-lined, stainless-steel bomb, which was heated at 433 K for 2 days. Red crystals were obtained when the bomb was cooled slowly to room temperature; yield 30% based on Fe.

Refinement

The DMF molecule (comprising the O2, N1, C4, C5 and C6 atoms) lies on the Wyckoff 4 e site (of *mm*2 symmetry) and is disordered over two positions with respect to its carbon atoms only. As the disorder refined to almost 1/2, the occupancies of these carbon atoms was fixed as 1/4. The three N—C distances were restrained to within ±0.01 Å.

Hydrogen atoms were placed at calculated positions (C—H = 0.93 Å for the sp^2 hybridized parent C atoms and 0.96 Å for the methyl C atoms) and were included in the refinements in the riding model approximation, with U(H) = 1.2U_{eq} for the aromatic H atoms and 1.5U_{eq} for the methyl H atoms. The two methyl groups were rotated so as to fit the electron density.

Figures

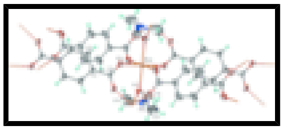

Fig. 1. Thermal ellipsoid plot (Barbour, 2001) plot of a portion of [Fe($C_8H_4O_4$)(C_3H_7NO)]$_n$. Displacement ellipsoids are drawn at the 50% probability level, and H atoms are drawn as spheres of arbitrary radii. The disorder in the DMF molecules is not shown.

Poly[(μ_2-*N*,*N*-dimethylformamide-κ^2*O*:*O*) (μ_4-terephthalato-κ^4*O*:*O*':*O*'':*O*''')iron(II)]

Crystal data

[Fe($C_8H_4O_4$)(C_3H_7NO)]	F_{000} = 600
M_r = 293.06	D_x = 1.558 Mg m^{-3}
Orthorhombic, *Imma*	Mo *K*α radiation λ = 0.71073 Å
Hall symbol: -I 2b 2	Cell parameters from 2096 reflections
a = 19.3652 (15) Å	θ = 2.5–28.5°

b = 7.2856 (6) Å	μ = 1.22 mm^{-1}
c = 8.8571 (7) Å	T = 295 (2) K
V = 1249.62 (17) Å^3	Block, red
Z = 4	0.25 × 0.19 × 0.08 mm

Data collection

Bruker APEX area-detector diffractometer	791 independent reflections
Radiation source: fine-focus sealed tube	730 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.021
T = 295(2) K	θ_{max} = 27.5°
φ and ω scans	θ_{min} = 2.1°
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	$h = -25 \rightarrow 24$
T_{min} = 0.357, T_{max} = 0.909	$k = -9 \rightarrow 7$
3421 measured reflections	$l = -8 \rightarrow 11$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.040	H-atom parameters constrained
$wR(F^2)$ = 0.113	$w = 1/[\sigma^2(F_o^2) + (0.0721P)^2 + 1.8869P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.08	$(\Delta/\sigma)_{max}$ = 0.001
791 reflections	$\Delta\rho_{max}$ = 0.73 e Å^{-3}
63 parameters	$\Delta\rho_{min}$ = −0.66 e Å^{-3}
3 restraints	Extinction correction: none
Primary atom site location: structure-invariant direct methods	

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

	x	y	z	U_{iso}*/U_{eq}	Occ. (<1)
Fe1	0.5000	0.5000	0.5000	0.0209 (3)	
O1	0.42136 (10)	0.5968 (3)	0.3639 (3)	0.0461 (6)	
O2	0.5000	0.7500	0.6390 (4)	0.0333 (8)	
N1	0.5000	0.7500	0.8974 (5)	0.0533 (15)	
C1	0.39343 (19)	0.7500	0.3459 (5)	0.0360 (8)	
C2	0.31894 (19)	0.7500	0.2960 (5)	0.0390 (9)	
C3	0.28393 (15)	0.5876 (4)	0.2733 (5)	0.0630 (11)	
H3	0.3064	0.4766	0.2896	0.076*	
C4	0.5267 (4)	0.7500	0.7594 (8)	0.0380 (16)	0.50
H4	0.5747	0.7500	0.7567	0.046*	0.50
C5	0.5296 (7)	0.7500	1.0396 (10)	0.078 (4)	0.50
H5A	0.5776	0.7814	1.0315	0.117*	0.25

H5B	0.5252	0.6302	1.0837	0.117*	0.25
H5C	0.5066	0.8384	1.1022	0.117*	0.25
C6	0.4266 (5)	0.7500	0.9021 (13)	0.088 (4)	0.50
H6A	0.4113	0.7010	0.9970	0.133*	0.25
H6B	0.4090	0.6756	0.8213	0.133*	0.25
H6C	0.4099	0.8734	0.8910	0.133*	0.25

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Fe1	0.0135 (4)	0.0240 (4)	0.0250 (4)	0.000	0.000	0.0017 (2)
O1	0.0312 (9)	0.0361 (11)	0.0709 (13)	0.0036 (8)	−0.0270 (9)	0.0003 (9)
O2	0.044 (2)	0.0279 (17)	0.0279 (17)	0.000	0.000	0.000
N1	0.084 (4)	0.046 (3)	0.029 (2)	0.000	0.000	0.000
C1	0.0266 (17)	0.0360 (19)	0.045 (2)	0.000	−0.0180 (15)	0.000
C2	0.0261 (17)	0.0346 (18)	0.056 (2)	0.000	−0.0204 (16)	0.000
C3	0.0374 (17)	0.0293 (14)	0.122 (3)	0.0040 (11)	−0.0381 (19)	0.0023 (17)
C4	0.052 (4)	0.029 (3)	0.033 (3)	0.000	−0.006 (3)	0.000
C5	0.136 (13)	0.068 (7)	0.030 (4)	0.000	−0.010 (6)	0.000
C6	0.125 (12)	0.094 (9)	0.047 (6)	0.000	0.026 (7)	0.000

Geometric parameters (Å, °)

Fe1—O1^{i}	2.0663 (17)	N1—C6^{iv}	1.422 (9)
Fe1—O1^{ii}	2.0663 (17)	C1—O1^{v}	1.250 (2)
Fe1—O1	2.0663 (17)	C1—C2	1.509 (5)
Fe1—O1^{iii}	2.0663 (17)	C2—C3	1.378 (3)
Fe1—O2	2.199 (2)	C2—C3^{v}	1.378 (3)
Fe1—O2^{i}	2.199 (2)	C3—C3^{vi}	1.377 (5)
O1—C1	1.250 (2)	C3—H3	0.9300
O2—C4	1.185 (8)	C4—H4	0.9300
O2—C4^{iv}	1.185 (8)	C5—H5A	0.9600
O2—Fe1^{iv}	2.1987 (19)	C5—H5B	0.9600
N1—C4^{iv}	1.327 (7)	C5—H5C	0.9600
N1—C4	1.327 (7)	C6—H6A	0.9600
N1—C5	1.384 (8)	C6—H6B	0.9600
N1—C5^{iv}	1.384 (8)	C6—H6C	0.9600
N1—C6	1.422 (9)		
O1^{i}—Fe1—O1^{ii}	85.05 (13)	C4^{iv}—N1—C6	68.7 (6)
O1^{i}—Fe1—O1	180.0	C4—N1—C6	114.6 (7)
O1^{ii}—Fe1—O1	94.95 (13)	C5—N1—C6	112.8 (8)
O1^{i}—Fe1—O1^{iii}	94.95 (13)	C5^{iv}—N1—C6	63.9 (8)
O1^{ii}—Fe1—O1^{iii}	180.00 (7)	C4^{iv}—N1—C6^{iv}	114.6 (7)
O1—Fe1—O1^{iii}	85.05 (13)	C4—N1—C6^{iv}	68.7 (6)
O1^{i}—Fe1—O2	87.49 (7)	C5—N1—C6^{iv}	63.9 (8)

O1^{ii}—Fe1—O2	92.51 (7)	C5^{iv}—N1—C6^{iv}	112.8 (8)
O1—Fe1—O2	92.51 (7)	C6—N1—C6^{iv}	176.7 (10)
O1^{iii}—Fe1—O2	87.49 (7)	O1^{v}—C1—O1	126.4 (3)
O1^{i}—Fe1—O2^{i}	92.51 (7)	O1^{v}—C1—C2	116.81 (16)
O1^{ii}—Fe1—O2^{i}	87.49 (7)	O1—C1—C2	116.81 (16)
O1—Fe1—O2^{i}	87.49 (7)	C3—C2—C3^{v}	118.3 (3)
O1^{iii}—Fe1—O2^{i}	92.51 (7)	C3—C2—C1	120.87 (17)
O2—Fe1—O2^{i}	180.0	C3^{v}—C2—C1	120.87 (17)
C1—O1—Fe1	134.27 (19)	C3^{vi}—C3—C2	120.86 (17)
C4—O2—C4^{iv}	51.8 (8)	C3^{vi}—C3—H3	119.6
C4—O2—Fe1	120.26 (12)	C2—C3—H3	119.6
C4^{iv}—O2—Fe1	120.26 (12)	O2—C4—N1	131.1 (7)
C4—O2—Fe1^{iv}	120.26 (12)	O2—C4—H4	114.4
C4^{iv}—O2—Fe1^{iv}	120.26 (12)	N1—C4—H4	114.4
Fe1—O2—Fe1^{iv}	111.87 (15)	N1—C5—H5A	109.5
C4^{iv}—N1—C4	45.9 (7)	N1—C5—H5B	109.5
C4^{iv}—N1—C5	178.5 (7)	N1—C5—H5C	109.5
C4—N1—C5	132.6 (6)	N1—C6—H6A	109.5
C4^{iv}—N1—C5^{iv}	132.6 (6)	N1—C6—H6B	109.5
C4—N1—C5^{iv}	178.5 (7)	N1—C6—H6C	109.5
C5—N1—C5^{iv}	49.0 (12)		

O1^{ii}—Fe1—O1—C1	−80.3 (4)	Fe1—O1—C1—O1^{v}	29.3 (7)
O1^{iii}—Fe1—O1—C1	99.7 (4)	Fe1—O1—C1—C2	−151.3 (3)
O2—Fe1—O1—C1	12.5 (3)	O1^{v}—C1—C2—C3	180.0 (4)
O2^{i}—Fe1—O1—C1	−167.5 (3)	O1—C1—C2—C3	0.6 (6)
O1^{i}—Fe1—O2—C4	−17.2 (4)	O1^{v}—C1—C2—C3^{v}	−0.6 (6)
O1^{ii}—Fe1—O2—C4	−102.1 (4)	O1—C1—C2—C3^{v}	180.0 (4)
O1—Fe1—O2—C4	162.8 (4)	C3^{v}—C2—C3—C3^{vi}	1.0 (10)
O1^{iii}—Fe1—O2—C4	77.9 (4)	C1—C2—C3—C3^{vi}	−179.6 (5)
O1^{i}—Fe1—O2—C4^{iv}	−77.9 (5)	C4^{iv}—O2—C4—N1	0.0
O1^{ii}—Fe1—O2—C4^{iv}	−162.8 (4)	Fe1—O2—C4—N1	−106.5 (2)
O1—Fe1—O2—C4^{iv}	102.1 (5)	Fe1^{iv}—O2—C4—N1	106.5 (2)
O1^{iii}—Fe1—O2—C4^{iv}	17.2 (4)	C4^{iv}—N1—C4—O2	0.0
O1^{i}—Fe1—O2—Fe1^{iv}	132.46 (6)	C5^{iv}—N1—C4—O2	0.00 (5)
O1^{ii}—Fe1—O2—Fe1^{iv}	47.54 (6)	C6—N1—C4—O2	0.000 (2)
O1—Fe1—O2—Fe1^{iv}	−47.54 (6)	C6^{iv}—N1—C4—O2	180.000 (2)
O1^{iii}—Fe1—O2—Fe1^{iv}	−132.46 (6)		

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

Fig. 1