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Poly[(μ_3-nicotinato-κ^3O:O':N)-(μ_2-nicotinato-κ^3O,O':N)iron(II)]

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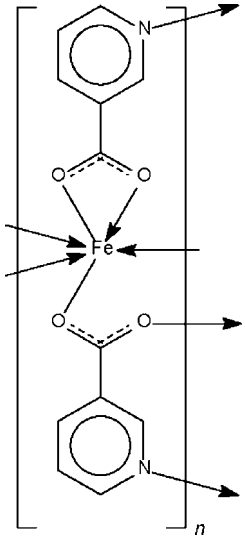
Key indicators: single-crystal X-ray study; T = 295 K; mean σ(C–C) = 0.003 Å; R factor = 0.029; wR factor = 0.079; data-to-parameter ratio = 16.1.

In the crystal structure of the title compound, $[Fe(C_6H_4NO_2)_2]_n$, one nicotinate group O,O'-chelates one Fe atom and binds through the N atom to the other Fe atom; the second nicotinate group bridges three Fe atoms through the N and two O atoms. The μ_2- and μ_3-bridging modes of the two nicotinate groups result in a polymeric three-dimensional network structure. The Fe atom shows octahedral coordination geometry but one of the Fe—O bonds is somewhat long [2.522 (2) Å].

Related literature

For zwitterionic tetraaquadi(nicotinato-κN)iron(II), see: Liang *et al.* (2005).



Experimental

Crystal data

$[Fe(C_6H_4NO_2)_2]$
M_r = 300.05
Monoclinic, $P2_1/n$
a = 10.8771 (7) Å
b = 9.6066 (6) Å
c = 12.7284 (8) Å
β = 111.619 (1)°
V = 1236.5 (1) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 1.23 mm^{-1}
T = 295 (2) K
0.41 × 0.34 × 0.25 mm

Data collection

Bruker SMART APEX diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.564, T_{max} = 0.749
7255 measured reflections
2762 independent reflections
2428 reflections with $I > 2\sigma(I)$
R_{int} = 0.018

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.028
$wR(F^2)$ = 0.078
S = 1.02
2762 reflections
172 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.24 e Å^{-3}
$\Delta\rho_{min}$ = −0.27 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Fe1—O1	2.522 (2)	Fe1—O4^{i}	2.061 (1)
Fe1—O2	2.072 (1)	Fe1—N1^{ii}	2.212 (1)
Fe1—O3	2.012 (1)	Fe1—N2^{iii}	2.224 (1)
O1—Fe1—O2	56.18 (5)	O2—Fe1—N2^{iii}	90.93 (5)
O1—Fe1—O3	96.95 (5)	O3—Fe1—O4^{i}	120.67 (6)
O1—Fe1—O4^{i}	142.30 (5)	O3—Fe1—N1^{ii}	88.50 (5)
O1—Fe1—N1^{ii}	89.36 (5)	O3—Fe1—N2^{iii}	89.17 (5)
O1—Fe1—N2^{iii}	93.18 (5)	O4^{i}—Fe1—N1^{ii}	89.39 (6)
O2—Fe1—O3	153.10 (6)	O4^{i}—Fe1—N2^{iii}	89.83 (6)
O2—Fe1—O4^{i}	86.23 (5)	N1^{ii}—Fe1—N2^{iii}	176.74 (5)
O2—Fe1—N1^{ii}	92.17 (5)		

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

Data collection: *SMART* (Bruker, 2004); cell refinement: *SAINT* (Bruker, 2004); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *X-SEED* (Barbour, 2001) and *OLEX* (Dolomanov *et al.*, 2003); software used to prepare material for publication: *publCIF* (Westrip, 2008).

I thank Mr Yan-Zhen Zheng of Sun Yat-Sen University for synthesizing the compound and measuring the crystal and the University of Malaya for supporting this study.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: XU2410).

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Westrip, S. P. (2008). *publCIF*. In preparation.

supplementary materials

Acta Cryst. (2008). E**64**, m728 [**doi:10.1107/S1600536808011045**]

Poly[(μ_3-nicotinato-κ^3O:O':N)(μ_2-nicotinato-κ^3O,O':N)iron(II)]

S. W. Ng

Comment

The crystal structures of a large number of divalent metal dinicotinates are known; the compounds exists as water-coordinated compounds in which the nicotinate ion binds through the aromatic N atom and not through the carboxyl group, as exemplified by tetraaquadinicotinatoiron(II). The report on this compound lists the crystal structures of tetraaquametal dinicotinates (Liang *et al.*, 2005). Tetraaquadinicotinatoiron is synthesized by reaction of the metal salt with nicotinic acid under aqueous conditions; under hydrothermal conditions, the synthesis has yielded the anhydrous compound (I). Iron dinicotinate (Fig. 1) has the nicotinate group engaged into two types of bridging interactions; one group *O*,*O*'-chelate to one Fe atom and binds through the N atom to the other Fe atom; the second nicotinate group bridges three Fe atoms through the N and two O atoms. The μ_2 and μ_3 bridging modes of the two nicotinate groups result in a polymeric three-dimensional network structure (Fig. 2). The Fe atom shows the common octahedral coordination geometry but one of the Fe–O bonds is somewhat long (Table 1).

Experimental

Iron powder (0.056 g, 1 mmol), nicotinic acid (0.218 g 2 mmol) and water (10 ml) heated in a 23-ml, Teflon-lined, Parr bomb at 423 K for 3 days. The bomb was cooled to room temperature at a rate of 10 K per min to give yellow block-shaped crystals (in 10% yield based on nicotinic acid rate of 10 ^{o}C.h-1. The yellow block crystals of iron dinicoinate were obtained (yield 8.2% based on nicotinic acid).

Refinement

Carbon-bound H-atoms were placed in calculated positions (C—H 0.93 Å) and were included in the refinement in the riding model approximation, with *U*(H) set to 1.2*U*(C).

Figures

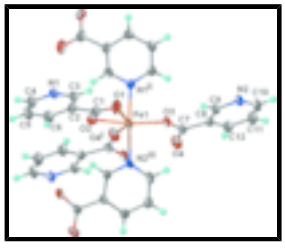

Fig. 1. 50% Probability thermal ellipsoid plot illustrating the octahedral geometry at iron.

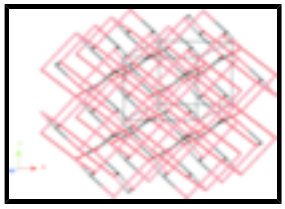

Fig. 2. *OLEX* (Dolomanov *et al.*, 2003) illustration of the three-dimensional network motif.

Poly[(μ$_3$-nicotinato-κ^3*O*:*O*':*N*)(μ$_2$-nicotinato- κ^3*O*,*O*':*N*)iron(II)]

Crystal data

$[Fe(C_6H_4NO_2)_2]$	F_{000} = 608
M_r = 300.05	D_x = 1.612 Mg m^{-3}
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2yn	Cell parameters from 6064 reflections
a = 10.8771 (7) Å	θ = 2.1–27.5º
b = 9.6066 (6) Å	μ = 1.23 mm^{-1}
c = 12.7284 (8) Å	T = 295 (2) K
β = 111.619 (1)º	Block, yellow
V = 1236.5 (1) Å^3	0.41 × 0.34 × 0.25 mm
Z = 4	

Data collection

Bruker APEX diffractometer	2762 independent reflections
Radiation source: fine-focus sealed tube	2428 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.018
T = 295(2) K	θ_{max} = 27.5º
φ and ω scans	θ_{min} = 2.1º
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	h = −14→14
T_{min} = 0.564, T_{max} = 0.749	k = −10→12
7255 measured reflections	l = −13→16

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.028	H-atom parameters constrained
$wR(F^2)$ = 0.078	$w = 1/[\sigma^2(F_o^2) + (0.0475P)^2 + 0.2265P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.02	$(\Delta/\sigma)_{max}$ = 0.001
2762 reflections	$\Delta\rho_{max}$ = 0.24 e Å^{-3}
172 parameters	$\Delta\rho_{min}$ = −0.27 e Å^{-3}

Primary atom site location: structure-invariant direct methods

Extinction correction: none

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

	x	*y*	*z*	U_{iso}*/U_{eq}
Fe1	0.40603 (2)	0.37707 (2)	0.580119 (18)	0.02707 (10)
O1	0.28044 (15)	0.19952 (16)	0.64714 (12)	0.0540 (4)
O2	0.43525 (13)	0.34442 (14)	0.74858 (11)	0.0398 (3)
O3	0.32118 (11)	0.33429 (14)	0.41409 (10)	0.0345 (3)
O4	0.45693 (13)	0.46511 (14)	0.36328 (12)	0.0477 (3)
N1	0.25364 (13)	0.02956 (15)	0.94060 (12)	0.0335 (3)
N2	0.06748 (13)	0.27149 (15)	0.09200 (12)	0.0335 (3)
C1	0.35420 (18)	0.24585 (18)	0.73955 (15)	0.0371 (4)
C2	0.35386 (17)	0.18565 (18)	0.84835 (14)	0.0336 (4)
C3	0.25803 (17)	0.09027 (18)	0.84724 (15)	0.0344 (4)
H3	0.1932	0.0671	0.7780	0.041*
C4	0.34759 (18)	0.0657 (2)	1.03903 (15)	0.0396 (4)
H4	0.3462	0.0246	1.1047	0.048*
C5	0.4460 (2)	0.1601 (2)	1.04838 (16)	0.0450 (5)
H5	0.5090	0.1824	1.1187	0.054*
C6	0.44980 (18)	0.2211 (2)	0.95150 (16)	0.0415 (4)
H6	0.5156	0.2849	0.9554	0.050*
C7	0.35595 (15)	0.39427 (16)	0.34127 (14)	0.0283 (3)
C8	0.26579 (15)	0.37708 (15)	0.22048 (14)	0.0281 (3)
C9	0.15745 (16)	0.29047 (17)	0.19585 (13)	0.0314 (3)
H9	0.1464	0.2424	0.2552	0.038*
C10	0.08654 (19)	0.3417 (2)	0.00799 (15)	0.0400 (4)
H10	0.0251	0.3306	−0.0651	0.048*
C11	0.19213 (19)	0.4291 (2)	0.02432 (15)	0.0432 (4)
H11	0.2013	0.4753	−0.0366	0.052*
C12	0.28458 (18)	0.44749 (18)	0.13239 (15)	0.0368 (4)
H12	0.3572	0.5054	0.1456	0.044*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Fe1	0.02436 (14)	0.03310 (15)	0.01968 (14)	−0.00138 (8)	0.00333 (10)	−0.00096 (8)
O1	0.0695 (10)	0.0593 (9)	0.0300 (7)	−0.0046 (8)	0.0145 (7)	0.0019 (6)
O2	0.0405 (7)	0.0448 (7)	0.0379 (7)	0.0017 (6)	0.0188 (6)	0.0113 (5)
O3	0.0304 (6)	0.0479 (7)	0.0207 (6)	−0.0014 (5)	0.0042 (5)	−0.0011 (5)
O4	0.0393 (7)	0.0507 (8)	0.0417 (8)	−0.0193 (6)	0.0014 (6)	−0.0023 (6)
N1	0.0301 (7)	0.0397 (8)	0.0282 (7)	−0.0009 (6)	0.0080 (6)	0.0040 (6)
N2	0.0311 (7)	0.0408 (8)	0.0225 (7)	−0.0059 (6)	0.0027 (6)	0.0003 (6)
C1	0.0419 (9)	0.0396 (9)	0.0332 (9)	0.0092 (8)	0.0176 (8)	0.0055 (7)
C2	0.0358 (9)	0.0359 (9)	0.0311 (9)	0.0018 (7)	0.0146 (7)	0.0027 (7)
C3	0.0331 (8)	0.0396 (9)	0.0267 (8)	0.0007 (7)	0.0065 (7)	0.0021 (7)
C4	0.0389 (9)	0.0504 (11)	0.0267 (9)	−0.0055 (8)	0.0089 (7)	0.0053 (8)
C5	0.0426 (10)	0.0566 (12)	0.0285 (9)	−0.0122 (9)	0.0045 (8)	0.0005 (8)

C6	0.0403 (9)	0.0462 (10)	0.0368 (10)	−0.0110 (8)	0.0127 (8)	0.0018 (8)
C7	0.0255 (8)	0.0286 (8)	0.0262 (8)	0.0026 (6)	0.0044 (6)	−0.0032 (6)
C8	0.0270 (8)	0.0311 (8)	0.0240 (8)	−0.0006 (6)	0.0066 (6)	−0.0023 (6)
C9	0.0305 (8)	0.0383 (9)	0.0224 (8)	−0.0044 (7)	0.0061 (6)	0.0015 (6)
C10	0.0433 (10)	0.0458 (10)	0.0222 (8)	−0.0046 (8)	0.0017 (7)	0.0014 (7)
C11	0.0519 (11)	0.0481 (10)	0.0274 (9)	−0.0099 (9)	0.0121 (8)	0.0059 (8)
C12	0.0378 (9)	0.0398 (9)	0.0313 (9)	−0.0093 (7)	0.0109 (7)	0.0003 (7)

Geometric parameters (Å, °)

Fe1—O1	2.522 (2)	C2—C3	1.384 (2)
Fe1—O2	2.072 (1)	C2—C6	1.385 (3)
Fe1—O3	2.012 (1)	C3—H3	0.9300
Fe1—O4^{i}	2.061 (1)	C4—C5	1.375 (3)
Fe1—N1^{ii}	2.212 (1)	C4—H4	0.9300
Fe1—N2^{iii}	2.224 (1)	C5—C6	1.379 (3)
O1—C1	1.237 (2)	C5—H5	0.9300
O2—C1	1.270 (2)	C6—H6	0.9300
O3—C7	1.262 (2)	C7—C8	1.497 (2)
O4—C7	1.233 (2)	C8—C9	1.381 (2)
O4—Fe1^{i}	2.0611 (12)	C8—C12	1.388 (2)
N1—C4	1.338 (2)	C9—H9	0.9300
N1—C3	1.340 (2)	C10—C11	1.375 (3)
N1—Fe1^{iv}	2.2124 (14)	C10—H10	0.9300
N2—C9	1.336 (2)	C11—C12	1.384 (2)
N2—C10	1.343 (2)	C11—H11	0.9300
N2—Fe1^{v}	2.2243 (14)	C12—H12	0.9300
C1—C2	1.502 (2)		
O1—Fe1—O2	56.18 (5)	N1—C3—C2	123.49 (16)
O1—Fe1—O3	96.95 (5)	N1—C3—H3	118.3
O1—Fe1—O4^{i}	142.30 (5)	C2—C3—H3	118.3
O1—Fe1—N1^{ii}	89.36 (5)	N1—C4—C5	123.64 (17)
O1—Fe1—N2^{iii}	93.18 (5)	N1—C4—H4	118.2
O2—Fe1—O3	153.10 (6)	C5—C4—H4	118.2
O2—Fe1—O4^{i}	86.23 (5)	C4—C5—C6	118.78 (18)
O2—Fe1—N1^{ii}	92.17 (5)	C4—C5—H5	120.6
O2—Fe1—N2^{iii}	90.93 (5)	C6—C5—H5	120.6
O3—Fe1—O4^{i}	120.67 (6)	C5—C6—C2	118.87 (17)
O3—Fe1—N1^{ii}	88.50 (5)	C5—C6—H6	120.6
O3—Fe1—N2^{iii}	89.17 (5)	C2—C6—H6	120.6
O4^{i}—Fe1—N1^{ii}	89.39 (6)	O4—C7—O3	124.60 (16)
O4^{i}—Fe1—N2^{iii}	89.83 (6)	O4—C7—C8	119.06 (15)
N1^{ii}—Fe1—N2^{iii}	176.74 (5)	O3—C7—C8	116.33 (14)
C1—O1—Fe1	80.56 (11)	C9—C8—C12	118.53 (15)
C1—O2—Fe1	100.52 (11)	C9—C8—C7	118.75 (15)

C7—O3—Fe1	122.17 (11)	C12—C8—C7	122.70 (15)
C7—O4—Fe1^{i}	162.57 (13)	N2—C9—C8	124.03 (15)
C4—N1—C3	116.93 (15)	N2—C9—H9	118.0
C4—N1—Fe1^{iv}	125.34 (12)	C8—C9—H9	118.0
C3—N1—Fe1^{iv}	117.73 (11)	N2—C10—C11	123.46 (16)
C9—N2—C10	116.58 (14)	N2—C10—H10	118.3
C9—N2—Fe1^{v}	115.27 (11)	C11—C10—H10	118.3
C10—N2—Fe1^{v}	128.13 (12)	C10—C11—C12	119.27 (17)
O1—C1—O2	122.67 (17)	C10—C11—H11	120.4
O1—C1—C2	121.14 (17)	C12—C11—H11	120.4
O2—C1—C2	116.18 (16)	C11—C12—C8	118.12 (16)
C3—C2—C6	118.28 (16)	C11—C12—H12	120.9
C3—C2—C1	120.21 (16)	C8—C12—H12	120.9
C6—C2—C1	121.49 (16)		
O3—Fe1—O1—C1	177.00 (11)	C1—C2—C3—N1	178.18 (15)
O4^{i}—Fe1—O1—C1	−6.40 (16)	C3—N1—C4—C5	0.0 (3)
O2—Fe1—O1—C1	−1.54 (10)	Fe1^{iv}—N1—C4—C5	179.39 (16)
N1^{ii}—Fe1—O1—C1	−94.59 (11)	N1—C4—C5—C6	−0.4 (3)
N2^{iii}—Fe1—O1—C1	87.45 (11)	C4—C5—C6—C2	0.4 (3)
O3—Fe1—O2—C1	−1.69 (18)	C3—C2—C6—C5	0.1 (3)
O4^{i}—Fe1—O2—C1	178.53 (11)	C1—C2—C6—C5	−178.59 (17)
N1^{ii}—Fe1—O2—C1	89.28 (11)	Fe1^{i}—O4—C7—O3	−68.9 (5)
N2^{iii}—Fe1—O2—C1	−91.70 (11)	Fe1^{i}—O4—C7—C8	110.7 (4)
O1—Fe1—O2—C1	1.51 (10)	Fe1—O3—C7—O4	12.9 (2)
O4^{i}—Fe1—O3—C7	5.56 (14)	Fe1—O3—C7—C8	−166.66 (10)
O2—Fe1—O3—C7	−174.18 (11)	O4—C7—C8—C9	175.90 (15)
N1^{ii}—Fe1—O3—C7	93.97 (13)	O3—C7—C8—C9	−4.5 (2)
N2^{iii}—Fe1—O3—C7	−83.75 (13)	O4—C7—C8—C12	−5.9 (2)
O1—Fe1—O3—C7	−176.85 (12)	O3—C7—C8—C12	173.73 (15)
Fe1—O1—C1—O2	2.49 (16)	C10—N2—C9—C8	0.4 (3)
Fe1—O1—C1—C2	−176.46 (16)	Fe1^{v}—N2—C9—C8	179.22 (13)
Fe1—O2—C1—O1	−3.0 (2)	C12—C8—C9—N2	−1.1 (3)
Fe1—O2—C1—C2	175.95 (12)	C7—C8—C9—N2	177.20 (15)
O1—C1—C2—C3	−9.1 (3)	C9—N2—C10—C11	0.3 (3)
O2—C1—C2—C3	171.91 (16)	Fe1^{v}—N2—C10—C11	−178.38 (15)
O1—C1—C2—C6	169.55 (18)	N2—C10—C11—C12	−0.2 (3)
O2—C1—C2—C6	−9.5 (2)	C10—C11—C12—C8	−0.5 (3)
C4—N1—C3—C2	0.4 (3)	C9—C8—C12—C11	1.1 (3)
Fe1^{iv}—N1—C3—C2	−178.97 (13)	C7—C8—C12—C11	−177.12 (16)
C6—C2—C3—N1	−0.5 (3)		

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

Fig. 1

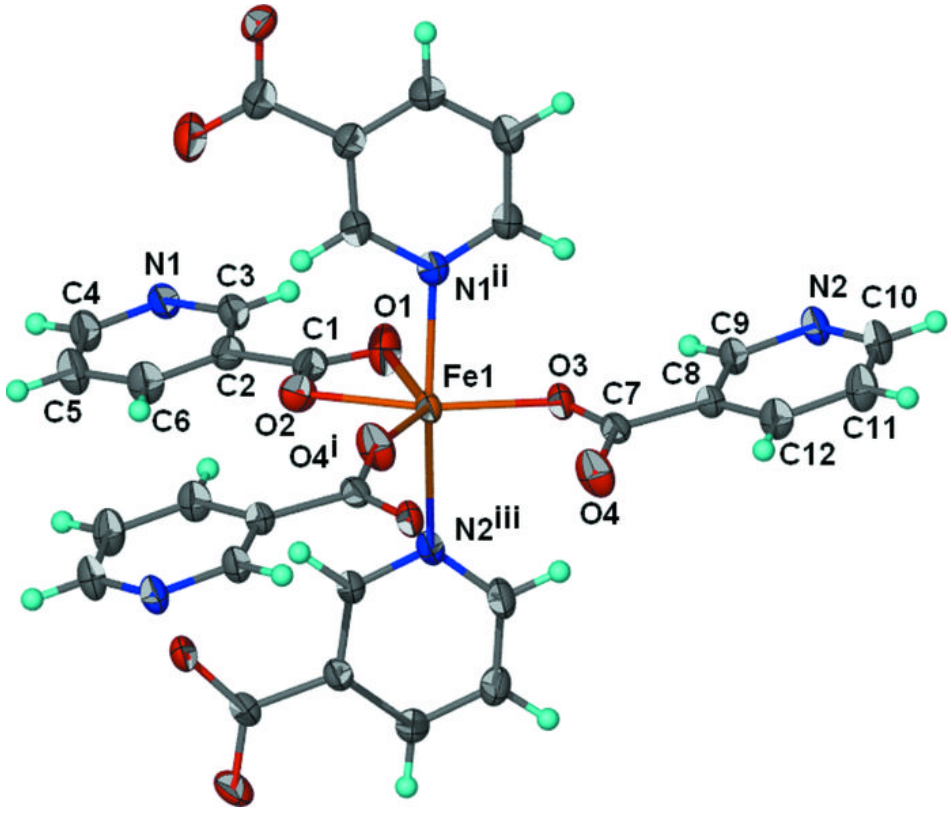
N1
C3
C4
N1ii
O1
C1
N2
C10
C9
C2
Fe1
O3
C8
C7
C5
C6
O2
O4i
C11
C12
O4
N2iii

Fig. 2