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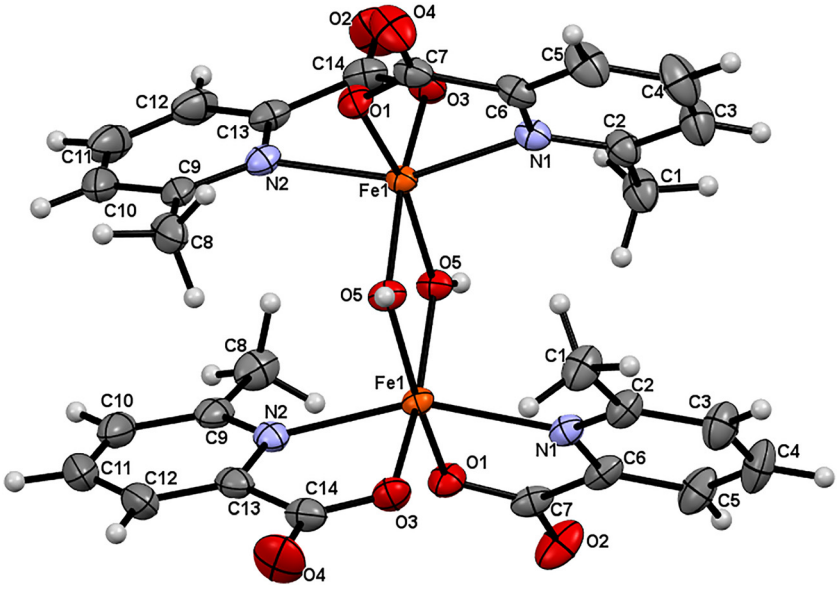
Crystal structure of di-μ_2-hydroxido-tetrakis(6-methylpyridine-2-carboxylato-k^2*N*,O) diiron(III) trihydrate $C_{28}H_{32}Fe_2N_4O_{13}$



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Abstract

$C_{28}H_{32}Fe_2N_4O_{13}$, monoclinic, $P2/n$ (no. 13), a = 8.0964(3) Å, b = 13.4889(5) Å, c = 14.0543(5) Å, β = 95.190(3)°, V = 1528.60(10) Å^3, Z = 2, $R_{gt}(F)$ = 0.0405, $wR_{ref}(F^2)$ = 0.0972, T = 173(2) K.

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The molecular structure is shown in the figure. Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

Table 1: Data collection and handling.

Crystal:	Red block
Size:	0.17 × 0.10 × 0.05 mm
Wavelength:	Mo *K*α radiation (0.71073 Å)
μ:	1.02 mm^{-1}
Diffractometer, scan mode:	Xcalibur, φ and ω
θ_{max}, completeness:	28.9°, 98%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	10,287, 3566, 0.034
Criterion for I_{obs}, $N(hkl)_{gt}$:	I_{obs} > 2 σ(I_{obs}), 2670
$N(param)_{refined}$:	226
Programs:	CrysAlis^{PRO} [1], Olex2 [2, 3], SHELX [3], Mercury [4, 5]

Source of material

The starting material 6-methyl-2-pyridinecarboxylic acid was bought from the commercial source and used without further treatment. The title compound was prepared by the solvothermal method. Firstly, 6-methyl-2-pyridinecarboxylic acid (27.4 mg, 0.2 mmol) was dissolved in 5 mL methanol with triethylamine (28 µL, 0.2 mmol). Then, the 5 mL methanolic solution of $Fe(NO_3)_3{\cdot}H_2O$ (40.4 mg, 0.1 mmol) was slowly dropped into the above solution. The mixture was stirred at room temperature for 30 min, then transferred to the Teflon autoclave and heated at 120 °C for three days. After cooling down to room temperature, red block crystals of the title compound were obtained.

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
Fe1	0.86752 (4)	0.72914 (3)	0.84619 (2)	0.01856 (11)
O1	1.11457 (19)	0.71986 (12)	0.86174 (12)	0.0262 (4)
O2	1.3528 (2)	0.80230 (16)	0.86847 (15)	0.0405 (5)
O3	0.8007 (2)	0.72871 (13)	0.97914 (12)	0.0278 (4)
O4	0.6850 (3)	0.63967 (18)	1.08788 (14)	0.0510 (6)
O5	0.6330 (2)	0.73046 (13)	0.79354 (12)	0.0212 (3)
H5	0.563 (4)	0.743 (2)	0.818 (3)	0.048 (11)*
N1	0.9388 (2)	0.88338 (15)	0.86761 (14)	0.0225 (4)
N2	0.8682 (2)	0.57198 (16)	0.87877 (14)	0.0241 (4)
C1	0.6627 (3)	0.9509 (2)	0.8846 (2)	0.0343 (6)
H1A	0.609291	0.928190	0.823111	0.051*
H1B	0.613702	1.014059	0.901874	0.051*
H1C	0.646227	0.901373	0.933914	0.051*
C2	0.8442 (3)	0.96457 (19)	0.87692 (18)	0.0284 (6)
C3	0.9155 (3)	1.0585 (2)	0.8802 (2)	0.0380 (7)
H3	0.847687	1.115410	0.885441	0.046*
C4	1.0840 (4)	1.0696 (2)	0.8760 (2)	0.0427 (7)
H4	1.133033	1.133645	0.877513	0.051*
C5	1.1808 (3)	0.9851 (2)	0.8694 (2)	0.0372 (7)
H5A	1.297379	0.990375	0.867306	0.045*
C6	1.1053 (3)	0.89439 (19)	0.86614 (17)	0.0260 (5)
C7	1.2014 (3)	0.7985 (2)	0.86437 (17)	0.0261 (5)
C8	1.0186 (3)	0.5124 (2)	0.7468 (2)	0.0357 (6)
H8A	0.945174	0.533604	0.691300	0.054*
H8B	1.075693	0.451183	0.731283	0.054*
H8C	1.100660	0.564315	0.763743	0.054*
C9	0.9185 (3)	0.49403 (19)	0.82930 (19)	0.0298 (6)
C10	0.8784 (4)	0.3976 (2)	0.8568 (2)	0.0414 (7)
H10	0.911758	0.342379	0.821183	0.050*
C11	0.7918 (4)	0.3827 (2)	0.9344 (2)	0.0467 (8)
H11	0.761238	0.317477	0.951444	0.056*
C12	0.7490 (4)	0.4622 (2)	0.9877 (2)	0.0401 (7)
H12	0.692877	0.452825	1.043556	0.048*
C13	0.7890 (3)	0.5556 (2)	0.95853 (17)	0.0286 (6)
C14	0.7527 (3)	0.6475 (2)	1.01402 (18)	0.0304 (6)
O1W	0.750000	0.7497 (5)	1.250000	0.204 (5)
H1W	0.759169	0.716755	1.191366	0.244*
O2W	0.4626 (5)	0.8189 (3)	1.0859 (5)	0.1221 (15)
H2WA	0.500380	0.760399	1.081962	0.183*
H2WB	0.489898	0.798188	1.153989	0.10 (2)*

Experimental details

Hydrogen atoms were added using riding models.

Comment

Picolinic acid derivatives have been widely studied as complex ligands [6], or functional materials [7]. However, only a limited number of compounds of its 6-methyl substituted derivative (6-methyl-2-pyridinecarboxylic acid) have been reported, and most of them are mononuclear compounds [8, 9].

Fe(III) complex (see the Figure) was achieved by considering the charge balance of the whole motif. All the bond lengths are within normal ranges, similar to its dinuclear analogue with two hydroxyl bridges [10]. The Fe(III) is bridged by two OH^- and chelated by two 6-methyl-2-pyridinecarboxylato groups to form a diunclear complex. The diunclear complexes are interlinked to from a 3D supramolecular network by two intermolecular H-bonds C8–H8A···O4 (symmetry code: $x + 1/2$, $-y + 1$, $z - 1/2$) [length 2.58(3) Å, angle 140.8(2)°], O5–H1···O2 (symmetry code: $x - 1$, y, z) [length 2.06(2) Å, angle 168.4(4)°] between the dinuclear complexes, one H-bonding C1–H1B···O2W (symmetry code: $-x + 1$, $-y + 2$, $-z + 2$) [length 2.34 (7) Å, angle 165.5(8)°] between the dinuclear complex and the water (lattice solvent) molecules.

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