Crystal structure of poly-(bis(1-ethyl-6-fluoro-1,4-dihydro-4-oxo-7-(1-piperazinyl)-1,8-naphthyridine-3-carboxylato)nickel(II)) dihydrate, $Ni(C_{15}H_{15}FN_4O_3)_2 \cdot 2H_2O$

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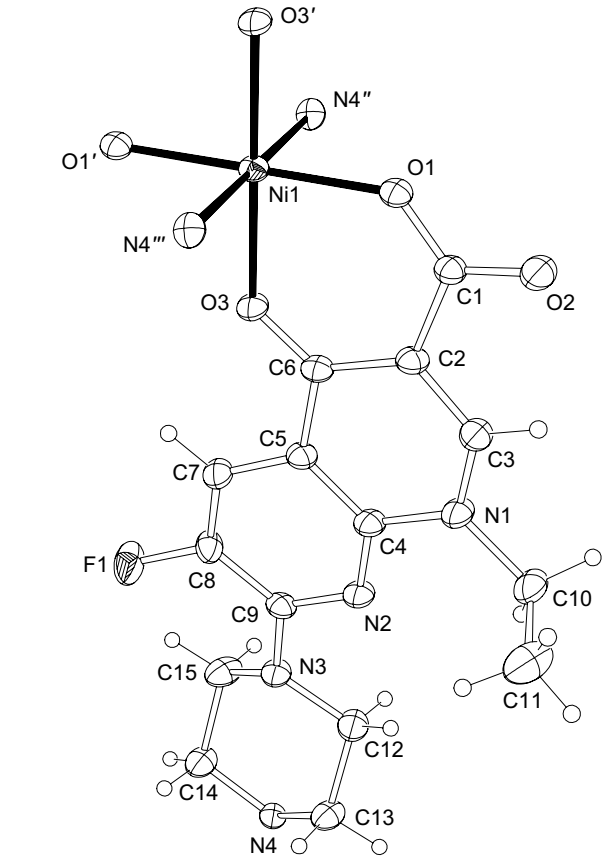
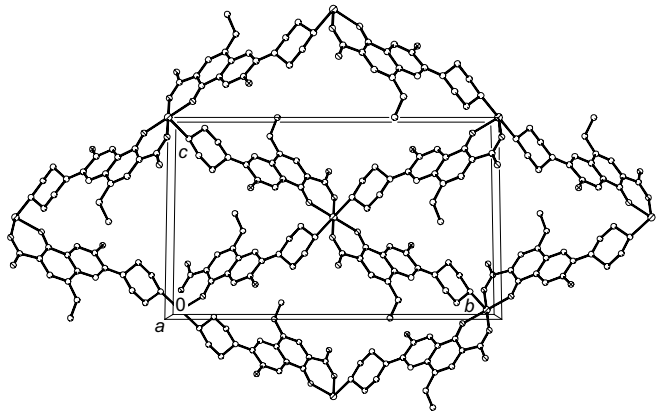
Received December 15, 2006, accepted and available on-line April 20, 2007; CCDC no. 1267/1956





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Abstract

$C_{30}H_{34}F_2N_8NiO_8$, monoclinic, $P12_1/c1$ (no. 14), a = 5.964(5) Å, b = 21.313(5) Å, c = 13.093(5) Å, β = 100.770(5)°, V = 1635.0 Å^3, Z = 2, $R_{gt}(F)$ = 0.060, $wR_{ref}(F^2)$ = 0.187, T = 273 K.

Source of material

A mixture of $Ni(NO_3)_2 \cdot 6H_2O$ (0.07 g, 0.25 mmol), H-enox (0.13 g, 0.5 mmol) and water (12 ml) was stirred for 30 min in air. The mixture was then transferred to a 23 ml Teflon-lined hydrothermal bomb. The bomb was kept at 423 K for 72 h under autogenous pressure. Green single crystals of title compound suitable for X-ray structure analysis were obtained from the reaction mixture.

Discussion

Enoxacin (H-enox, $C_{15}H_{17}FN_4O_3$, 1-ethyl-6-fluoro-1,4-dihydro-4-oxo-7-piperazine-1,8-naphthyridine-3-carboxylic acid) is member of the class of quinolones that is used to treat infections. The manganese(II) [1], cadmium(II) [2] and cobalt(II) [3] derivatives of enox have been reported.

In the title nickel(II) derivative, a two-dimensional coordination polymer, the anion acts in a bridging mode (figure, top). The Ni(II) atom is coordinated by four oxygen atoms and two N atoms from four enoxacin ligands (two monodentate-*N* and two *O*,*O*-bidentate) to form a square grid propagating in [001] (figure, bottom). The disordered, uncoordinated, water molecules occupy the cavities. An N–H···O hydrogen bond to the carboxylate O atom not linked to Ni completes the structure.

Table 1. Data collection and handling.

Crystal:	green block, size 0.19 × 0.27 × 0.35 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	6.67 cm^{-1}
Diffractometer, scan mode:	Bruker SMART CCD, φ/ω
$2\theta_{max}$:	56.46°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	9887, 3946
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 2819
$N(param)_{refined}$:	234
Programs:	SHELXS-97 [4], SHELXL-97 [5], SHELXTL [6]

Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	Occ.	x	y	z	U_{iso}
H(12A)	4*e*		–0.3931	0.3276	0.0715	0.056
H(12B)	4*e*		–0.3317	0.3854	0.1457	0.056
H(3)	4*e*		0.2076	0.1031	0.1661	0.047

Table 2. Continued.

Atom	Site	Occ.	*x*	*y*	*z*	U_{iso}
H(7)	4*e*		–0.4615	0.1583	0.3998	0.045
H(10A)	4*e*		0.0047	0.2466	0.0789	0.063
H(10B)	4*e*		0.1927	0.1964	0.0705	0.063
H(11A)	4*e*		–0.0652	0.1345	–0.0314	0.137
H(11B)	4*e*		–0.0867	0.2022	–0.0794	0.137
H(11C)	4*e*		–0.2663	0.1784	–0.0148	0.137
H(13A)	4*e*		–0.5717	0.4177	–0.0054	0.051
H(13B)	4*e*		–0.7536	0.3675	0.0106	0.051

Table 2. Continued.

Atom	Site	Occ.	*x*	*y*	*z*	U_{iso}
H(14A)	4*e*		–0.9527	0.3815	0.1589	0.054
H(14B)	4*e*		–0.8891	0.4400	0.2307	0.054
H(15A)	4*e*		–0.5254	0.3983	0.2942	0.057
H(15B)	4*e*		–0.7108	0.3489	0.3110	0.057
H(1A)	4*e*	0.56	0.3545	0.0245	0.0502	0.186
H(1B)	4*e*	0.56	0.1132	–0.0124	0.0350	0.186
H(2A)	4*e*	0.44	0.3624	0.0270	0.0493	0.214
H(2B)	4*e*	0.44	0.5828	0.0698	0.0982	0.214

Table 3. Atomic coordinates and displacement parameters (in $Å^2$).

Atom	Site	Occ.	*x*	*y*	*z*	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Ni(1)	2*c*		0	0	½	0.0287(3)	0.0161(3)	0.0303(3)	0.0004(2)	0.0038(2)	0.0017(2)
O(1)	4*e*		0.2147(4)	–0.00284(9)	0.3969(2)	0.033(1)	0.021(1)	0.035(1)	0.0036(8)	0.0054(9)	0.0019(9)
O(2)	4*e*		0.3587(5)	0.0148(1)	0.2566(2)	0.065(2)	0.056(2)	0.053(2)	0.034(2)	0.031(2)	0.022(1)
O(3)	4*e*		–0.1531(4)	0.0767(1)	0.4236(2)	0.038(1)	0.022(1)	0.037(1)	0.0053(9)	0.010(1)	0.0086(9)
F(1)	4*e*		–0.7065(4)	0.2522(1)	0.3503(2)	0.063(2)	0.054(2)	0.104(2)	0.030(1)	0.054(2)	0.035(2)
N(1)	4*e*		–0.0146(5)	0.1705(1)	0.1684(2)	0.049(2)	0.033(2)	0.040(2)	0.014(1)	0.017(1)	0.013(1)
N(2)	4*e*		–0.2865(5)	0.2465(1)	0.1857(2)	0.041(2)	0.028(1)	0.040(2)	0.010(1)	0.010(1)	0.012(1)
N(3)	4*e*		–0.5464(5)	0.3251(1)	0.1955(2)	0.040(2)	0.025(1)	0.042(2)	0.011(1)	0.012(1)	0.010(1)
N(4)	4*e*		–0.7496(4)	0.4420(1)	0.1035(2)	0.032(1)	0.022(1)	0.035(1)	0.007(1)	0.007(1)	0.004(1)
C(1)	4*e*		0.2228(5)	0.0272(2)	0.3150(3)	0.033(2)	0.027(2)	0.032(2)	0.004(1)	0.006(1)	0.003(1)
C(2)	4*e*		0.0668(6)	0.0832(2)	0.2869(2)	0.036(2)	0.026(2)	0.034(2)	0.006(1)	0.005(1)	0.004(1)
C(3)	4*e*		0.0986(6)	0.1175(2)	0.2028(3)	0.045(2)	0.034(2)	0.041(2)	0.013(2)	0.015(2)	0.005(2)
C(4)	4*e*		–0.1854(6)	0.1918(2)	0.2190(3)	0.038(2)	0.026(2)	0.035(2)	0.007(1)	0.007(1)	0.006(1)
C(5)	4*e*		–0.2347(6)	0.1578(1)	0.3027(3)	0.033(2)	0.024(2)	0.037(2)	0.004(1)	0.005(1)	0.007(1)
C(6)	4*e*		–0.1082(5)	0.1018(1)	0.3433(2)	0.034(2)	0.018(1)	0.034(2)	0.002(1)	0.002(1)	0.002(1)
C(7)	4*e*		–0.4149(6)	0.1803(2)	0.3461(3)	0.039(2)	0.031(2)	0.044(2)	0.006(1)	0.014(2)	0.011(2)
C(8)	4*e*		–0.5217(6)	0.2337(2)	0.3106(3)	0.035(2)	0.032(2)	0.053(2)	0.007(1)	0.019(2)	0.009(2)
C(9)	4*e*		–0.4510(6)	0.2697(2)	0.2313(3)	0.032(2)	0.025(2)	0.039(2)	0.005(1)	0.003(1)	0.006(1)
C(10)	4*e*		0.0330(8)	0.2020(2)	0.0741(3)	0.061(3)	0.049(2)	0.053(2)	0.019(2)	0.027(2)	0.024(2)
C(11)	4*e*		–0.109(1)	0.1771(3)	–0.0211(4)	0.096(4)	0.125(5)	0.054(3)	–0.002(4)	0.014(3)	0.026(3)
C(12)	4*e*		–0.4548(7)	0.3579(2)	0.1141(3)	0.047(2)	0.042(2)	0.056(2)	0.019(2)	0.020(2)	0.020(2)
C(13)	4*e*		–0.6384(7)	0.3959(2)	0.0467(3)	0.056(2)	0.033(2)	0.039(2)	0.017(2)	0.014(2)	0.007(2)
C(14)	4*e*		–0.8286(7)	0.4093(2)	0.1884(3)	0.054(2)	0.038(2)	0.047(2)	0.022(2)	0.020(2)	0.013(2)
C(15)	4*e*		–0.6443(7)	0.3707(2)	0.2589(3)	0.069(3)	0.036(2)	0.040(2)	0.018(2)	0.013(2)	0.005(2)
O(1W)	4*e*	0.56(2)	0.245(3)	0.0007(6)	0.043(1)	0.187(8)	0.186(8)	0.186(8)	0.001(1)	0.035(2)	–0.001(1)
O(2W)	4*e*	0.44	0.481(3)	0.0464(9)	0.070(2)	0.21(1)	0.21(1)	0.21(1)	0.000(1)	0.040(2)	0.000(1)

Acknowledgment. The authors acknowledge the financial support of Independent Innovation Science Foundation of Harbin Medical University (grant no. 60041).

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