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Diaquadibenzimidazolebis(5-fluoro-2,4-dioxo-1,2,3,4-tetrahydropyrimidine-1-acetato)nickel(II)

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Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.003 Å
R factor = 0.029
wR factor = 0.074
Data-to-parameter ratio = 10.8

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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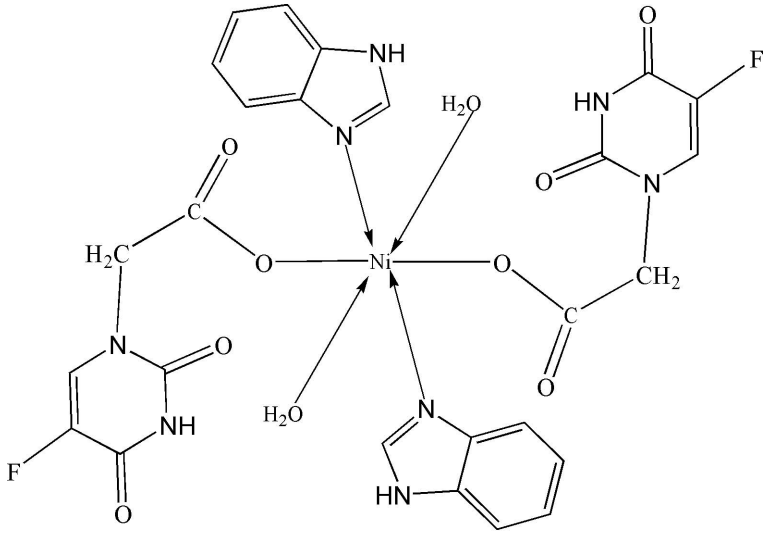
In the title centrosymmetric compound, $[Ni(C_6H_4FN_2O_4)_2(C_7H_6N_2)_2(H_2O)_2]$, each Ni^{II} ion is coordinated by two 5-fluorouracil-1-acetate (5-fluoro-2,4-dioxo-1,2,3,4-tetrahydropyrimidine-1-acetate) anions *via* carboxylate O atoms, two water molecules and two benzimidazole ligands, forming a six-coordinate octahedral environment; the N—H···F, N—H···O and O—H···O hydrogen-bonding interactions link adjacent molecules into a three-dimensional network.

Comment

5-Fluorouracil (5-FU) is an antimetabolite with good antimicrobial and antitumor activity but has toxic side effects (Ouchi *et al.*, 1997; Nichifor & Schacht, 1994; Nichifor *et al.*, 1997; Hulme *et al.*, 2005). In order to improve the topical delivery of 5-FU, as well as to reduce the side effects, many derivatives of 5-FU have been synthesized, some of which are of improved activity. 5-Fluorouracil-1-acetic acid (5-fluoro-2,4-dioxo-1,2,3,4-tetrahydropyrimidine-1-acetic acid) is a member of the family (Sloan *et al.*, 1993; Li *et al.*, 2000; Beall & Sloan, 2001, 2002). As increasing attention has been paid to the anticancer activity of 5-FU and its derivatives (Akgerman & Guney, 2000), a few of their transition metal complexes have been reported (Wang *et al.*, 1993). To extend such studies, we report the nickel derivative diaquadibenzimidazolebis(5-fluorouracil-1-acetate)nickel(II), (I).



(I)

Mononuclear (I) consists of an Ni atom, two coordinated water molecules, two 5-fluorouracil-1-acetate anions binding

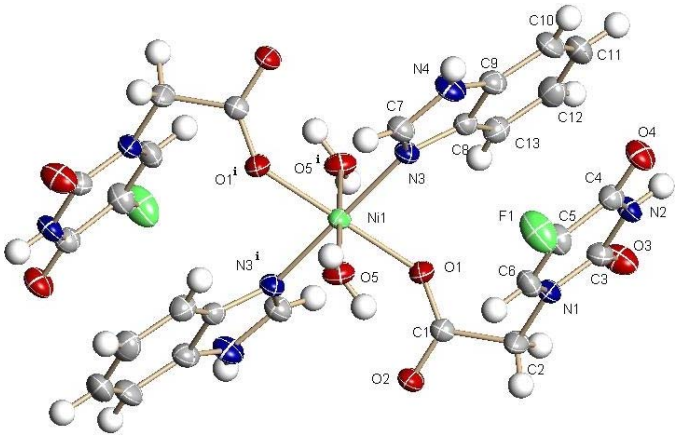


Figure 1
The coordination environment of the nickel(II) ion in (I), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

through their carboxylate O atoms, and two benzimidazole molecules. The Ni^{II} atom lies on an inversion center and the geometry around the Ni ion is octahedral (Fig. 1 and Table 1). A square is formed by atoms O1, $O1^{i}$, N3 and $N3^{i}$ [symmetry code: (i) $-x+1, -y+1, -z$], and is crystallographically required to be planar. N—H···F, N—H···O and O—H···O hydrogen bonds link the mononuclear units to form a three-dimensional network (Table 2).

Experimental

The compound was synthesized in a hydrothermal process from a mixture of benzimidazole (2 mmol, 0.24 g), $NiCl_2{\cdot}2H_2O$ (1 mmol, 0.16 g), 5-fluorouracil-1-acetic acid (2 mmol, 0.75 g) and water (20 ml). The reaction was carried out in a 30 ml Teflon-lined stainless-steel reactor. The reactor was heated to 418 K for 3 d. The reactor was slowly cooled to room temperature to yield green crystals that were collected and washed with water.

Crystal data

$[Ni(C_6H_4FN_2O_4)_2(C_7H_6N_2)_2(H_2O)_2]$
M_r = 705.24
Triclinic, $P\overline{1}$
a = 7.2955 (5) Å
b = 8.3616 (6) Å
c = 11.9300 (8) Å
α = 89.059 (1)°
β = 88.827 (1)°
γ = 70.549 (1)°
V = 686.04 (8) Å^3
Z = 1
D_x = 1.707 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 3096 reflections
θ = 2.6–25.2°
μ = 0.80 mm^{-1}
T = 298 (2) K
Block, green
0.28 × 0.18 × 0.13 mm

Data collection

Bruker SMART APEX area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2002)
T_{min} = 0.808, T_{max} = 0.904
5024 measured reflections
2444 independent reflections
2296 reflections with $I > 2\sigma(I)$
R_{int} = 0.015
θ_{max} = 25.3°
$h = -8 \rightarrow 8$
$k = -9 \rightarrow 10$
$l = -14 \rightarrow 14$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.029
$wR(F^2)$ = 0.074
S = 1.06
2444 reflections
227 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0386P)^2 + 0.2919P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.22 e Å^{-3}
$\Delta\rho_{min}$ = −0.31 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Ni1—O1	2.0549 (13)	O1—C1	1.250 (2)
Ni1—N3	2.0705 (14)	O2—C1	1.245 (2)
Ni1—O5	2.1168 (14)		
O1—Ni1—N3	86.21 (6)	N3—Ni1—O5	89.48 (6)
O1—Ni1—O5	89.95 (6)	O2—C1—O1	128.51 (17)

Table 2
Hydrogen-bonding geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O5—H5a···O3^{i}	0.84 (1)	2.03 (1)	2.852 (2)	170 (2)
N4—H4···O4^{ii}	0.85 (1)	2.10 (1)	2.889 (2)	155 (2)
N4—H4···F1^{ii}	0.85 (1)	2.56 (2)	3.204 (2)	134 (2)
O5—H5b···O2^{iii}	0.84 (1)	1.93 (1)	2.756 (2)	171 (2)
N2—H2···O4^{iv}	0.85 (1)	2.17 (1)	3.016 (2)	175 (2)

Symmetry codes: (i) $x, 1+y, z$; (ii) $1-x, 1-y, 2-z$; (iii) $-x, 1-y, 1-z$; (iv) $1-x, -y, 2-z$.

H atoms of the water molecule and those attached to N2 and N4 were located in difference-density maps and refined with O—H and N—H distances restrained to 0.85 (1) Å, and with U_{iso} = 1.2U_{eq}(parent atom). The other H atoms were positioned geometrically and allowed to ride on their parent atoms; Csp^2—H = 0.93 Å with U_{iso} = 1.2U_{eq}(parent atom), and Csp^3—H = 0.97 Å with U_{iso} = 1.5U_{eq}(parent atom).

Data collection: *SMART* (Bruker, 2002); cell refinement: *SAINT* (Bruker, 2002); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL*(Bruker, 2002); software used to prepare material for publication: *SHELXL97*.

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