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Key indicators

Single-crystal X-ray study
T = 296 K
Mean $\sigma(\text{C}-\text{C}) = 0.004 \text{ \AA}$
R factor = 0.038
wR factor = 0.094
Data-to-parameter ratio = 13.1

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

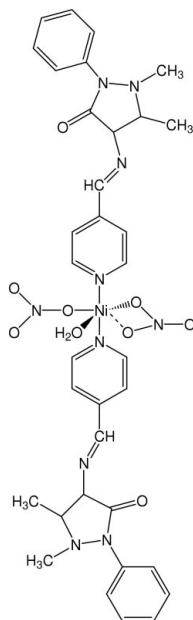
Aquabis(1,5-dimethyl-2-phenyl-4-[(*E*)-4-pyridylmethylene- κ N]amino}pyrazolidin-3-one)-dinitratonickel(II)

In the title compound, $[\text{Ni}(\text{NO}_3)_2(\text{C}_{17}\text{H}_{16}\text{N}_4\text{O})_2(\text{H}_2\text{O})]$, the six-coordinate Ni atom is bound by two *trans* 4-aminoantipyrene ligands. Such compounds possess antimicrobial properties. The crystal packing is stabilized by intermolecular hydrogen bonds and non-classical C—H...O hydrogen bonds, which generate a three-dimensional network.

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Comment

4-Aminoantipyrene Schiff base derivatives have been extensively investigated as ligands in transition metal chemistry, because these transition metal complexes possess antifungal and antimicrobial activities (Raman *et al.*, 2001). However, there are few reported crystal structures of transition metal complexes with 4-aminoantipyrene Schiff base derived ligands; examples are the copper(II) complexes reported by Liang *et al.* (2002) and Wang *et al.* (2003). We report here the crystal structure of the title compound, (I).



(I)

In (I), the central Ni^{2+} ion is six-coordinate and is bound by two N atoms from two *trans* 1,5-dimethyl-2-phenyl-4-[(*E*)-4-pyridylmethylene]amino}pyrazolidin-3-one ligands, one O atom from an aqua ligand and three O atoms from two nitrate groups; one nitrate group acts as a monodentate ligand and the other adopts a bidentate chelate mode with a bite angle of $57.67(8)^\circ$. This NiO_4N_2 centre has distorted octahedral geometry (Fig. 1).

The axial N5—Ni—N1 angle is $175.42(8)^\circ$, slightly deviating from the ideal value of 180° . Atoms N1 and N5 occupy the apical positions, and atoms O3, O4, O6 and O9

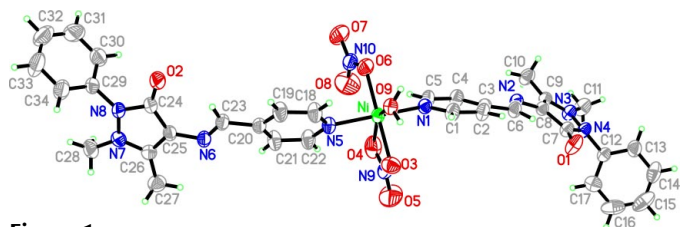


Figure 1
The structure of (I), showing 50% probability displacement ellipsoids and the atom-numbering scheme.

occupy the equatorial plane. The two Ni–N distances are equal [2.0892 (19) Å] and are shorter than in the Ni–pyridine complex (2.101 Å; Drew *et al.*, 1972). The Ni–O3 and Ni–O4 bond lengths are similar to those in other bidentate chelate nitrate groups bound to Ni²⁺ ions (Butcher *et al.*, 1981). The Ni–O6 bond length [2.0506 (18) Å] is shorter than that in a monodentate nitrate group to nickel(II) [2.101 (2) Å; Cameron *et al.*, 1972]. The bond lengths and angles for the 3-pyrazolidinone ligands are within normal ranges (Allen *et al.*, 1987).

A three-dimensional network is formed through intermolecular hydrogen bonds. The coordinated aqua ligand is hydrogen-bonded to the O atoms of 3-pyrazolidinone ligands, namely O9–H9A···O2ⁱ [2.632 (3) Å] and O9–H9B···O1ⁱⁱ [2.659 (3) Å], and there are non-classical hydrogen bonds formed by C–H···O(nitrate) (Table 2 and Fig. 2) [symmetry codes: (i) 1 – x, –y, –z; (ii) –x, 1 – y, 1 – z].

Experimental

The Schiff base ligand 1,5-dimethyl-2-phenyl-4-[(*E*)-4-pyridinylmethylidene]amino]pyrazolidin-3-one, *L*, was synthesized by the condensation of 4-aminoantipyrine with pyridine-2-carboxaldehyde in a 1:1 molar ratio in ethanol at 353 K for 2 h. Ni(NO₃)₂·6H₂O (0.4 mmol) and *L* (0.8 mmol) were placed in a thick Pyrex tube (*ca* 20 cm long). After addition of ethanol (1 ml) and *sec*-butyl alcohol (1 ml), the tube was frozen with liquid N₂, evacuated under vacuum and sealed with a torch. The tube was heated at 383 K for 1 d to yield green block-shaped crystals of (I), suitable for X-ray crystallographic analysis.

Crystal data

[Ni(NO ₃) ₂ (C ₁₇ H ₁₆ N ₄ O) ₂ (NO ₃) ₂ (H ₂ O)]	<i>Z</i> = 2
<i>M_r</i> = 785.42	<i>D_x</i> = 1.443 Mg m ^{–3}
Triclinic, <i>P</i> 1̄	Mo <i>K</i> α radiation
<i>a</i> = 10.097 (2) Å	Cell parameters from 28 reflections
<i>b</i> = 12.977 (2) Å	<i>θ</i> = 2.8–14.6°
<i>c</i> = 14.860 (2) Å	<i>μ</i> = 0.61 mm ^{–1}
<i>α</i> = 88.86 (1)°	<i>T</i> = 296 (2) K
<i>β</i> = 75.41 (1)°	Block, green
<i>γ</i> = 73.86 (1)°	0.52 × 0.50 × 0.16 mm
<i>V</i> = 1807.3 (5) Å ³	

Data collection

Siemens P4 diffractometer	<i>R</i> _{int} = 0.012
<i>ω</i> scans	<i>θ</i> _{max} = 25.3°
Absorption correction: <i>ψ</i> scan (North <i>et al.</i> , 1968)	<i>h</i> = 0 → 12
<i>T</i> _{min} = 0.732, <i>T</i> _{max} = 0.908	<i>k</i> = –14 → 15
7176 measured reflections	<i>l</i> = –17 → 17
6540 independent reflections	3 standard reflections
4930 reflections with <i>I</i> > 2σ(<i>I</i>)	every 97 reflections
	intensity decay: 2.4%

Refinement

Refinement on <i>F</i> ²	<i>w</i> = 1/[σ ² (<i>F_o</i> ²) + (0.0539 <i>P</i>) ²]
<i>R</i> [<i>F</i> ² > 2σ(<i>F</i> ²)] = 0.038	where <i>P</i> = (<i>F_o</i> ² + 2 <i>F_c</i> ²)/3
<i>wR</i> (<i>F</i> ²) = 0.095	(Δ/σ) _{max} < 0.001
<i>S</i> = 0.98	Δρ _{max} = 0.42 e Å ^{–3}
6540 reflections	Δρ _{min} = –0.30 e Å ^{–3}
500 parameters	Extinction correction: <i>SHELXL97</i>
H-atom parameters constrained	Extinction coefficient: 0.0020 (5)

Table 1

Selected geometric parameters (Å, °).

Ni–O9	2.0104 (18)	Ni–O4	2.287 (2)
Ni–O6	2.0506 (18)	O1–C7	1.240 (3)
Ni–N5	2.0892 (19)	O2–C24	1.247 (3)
Ni–N1	2.0892 (19)	N2–C6	1.272 (3)
Ni–O3	2.0920 (19)	N6–C23	1.273 (3)
N5–Ni–N1	175.42 (8)	O3–Ni–O4	57.67 (8)

Table 2

Hydrogen-bonding geometry (Å, °).

<i>D</i> –H··· <i>A</i>	<i>D</i> –H	H··· <i>A</i>	<i>D</i> ··· <i>A</i>	<i>D</i> –H··· <i>A</i>
O9–H9A···O2 ⁱ	0.81	1.84	2.632 (3)	168
O9–H9B···O1 ⁱⁱ	0.82	1.87	2.659 (3)	162
C10–H10C···O5 ⁱⁱⁱ	0.94	2.57	3.053 (4)	111
C11–H11A···O3 ^{iv}	0.96	2.51	3.407 (4)	155
C14–H14···O7 ^v	0.93	2.46	3.300 (4)	149
C27–H27C···O7 ^{vi}	0.96	2.50	3.245 (4)	134
C28–H28C···O7 ^{vii}	0.96	2.60	3.179 (4)	119

Symmetry codes: (i) 1 – x, –y, –z; (ii) –x, 1 – y, 1 – z; (iii) 1 – x, 1 – y, 1 – z; (iv) x, 1 + y, z; (v) x – 1, 1 + y, 1 + z; (vi) 2 – x, –y, –z; (vii) 1 + x, y – 1, z.

All H atoms were positioned geometrically and treated as riding, with C–H = 0.93 Å and *U*_{iso}(H) = 1.2*U*_{eq}(C) or 1.5*U*_{eq}(O), except for

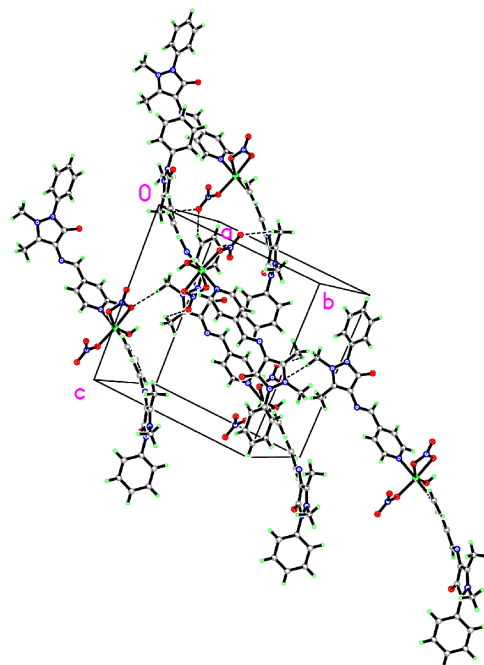


Figure 2

A packing diagram for (I), showing the three-dimensional network structure (dashed lines indicate hydrogen bonds).

the methyl H atoms, for which $C-H = 0.96 \text{ \AA}$ and $U_{\text{iso}}(H) = 1.5U_{\text{eq}}(C)$.

Data collection: *XSCANS* (Siemens, 1994); cell refinement: *XSCANS*; data reduction: *SHELXTL* (Sheldrick, 1997a); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997b); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997b); molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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