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Hexakis(1*H*-imidazole-κN^3)nickel(II) benzene-1,3-dioxyacetate

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Key indicators

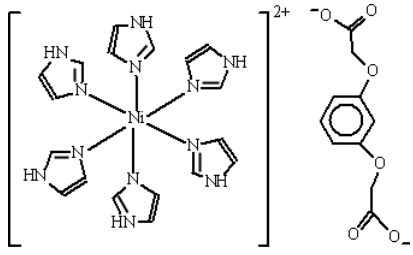
Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.005 Å
R factor = 0.044
wR factor = 0.118
Data-to-parameter ratio = 14.1

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

In the title complex, $[Ni(C_3H_4N_2)_6](C_{10}H_8O_6)$, the Ni^{II} atom shows an octahedral coordination geometry, defined by six N atoms from different imidazole ligands. The cations and anions are linked by hydrogen bonds into a three-dimensional network structure.

Comment

Phenylenedioxydiacetic acids, which have been known to show biological activities, are multidentate flexible ligands with versatile binding modes. To our knowledge, some metal derivatives of benzene-1,2-dioxyacetic acid and benzene-1,4-dioxyacetic acid ligands have been structurally characterized (McCann *et al.*, 1994; Smith *et al.*, 1987, 1991). However, the complexes of the related benzene-1,3-dioxyacetic acid are less well documented. We have recently reported the structure of a chain polymer, diaquazinc(II) benzene-1,3-dioxyacetate dihydrate (Gao *et al.*, 2004), in which the benzene-1,3-dioxyacetate acts as the bridging ligand and the zinc(II) ion displays a four-coordinate distorted tetrahedral geometry. In the present study, the structure of the title complex, (I), is reported.



(I)

As shown in Fig. 1, the crystal structure of the title ionic complex consists of $[Ni(C_3H_4N_2)_6]^{2+}$ cations and benzene-1,3-dioxyacetate dianions. The Ni^{II} atom is coordinated by six imidazole molecules in a slightly distorted octahedral geometry. The Ni—N bond lengths are in the range 2.109 (2)–2.148 (2) Å. The cations and anions are linked by extensive hydrogen bonds to form a three-dimensional network structure (Table 2 and Fig. 2).

Experimental

Benzene-1,3-dioxyacetic acid was prepared following the method described for the synthesis of benzene-1,2-dioxyacetic acid (Mirci, 1988). The title complex was prepared by the addition of benzene-1,3-dioxyacetic acid (20 mmol) to an aqueous solution of imidazole (40 mmol) and nickel diacetate tetrahydrate (20 mmol), and the pH was adjusted to 6 with 0.1 *M* sodium hydroxide. Green crystals

separated from the filtered solution after several days. Analysis calculated for $C_{28}H_{32}N_{12}NiO_6$: C 48.64, H 4.67, N 24.32%; found: C 48.81, H 4.79, N 24.15%.

Crystal data

$[Ni(C_3H_4N_2)_6](C_{10}H_8O_6)$	$Z = 2$
$M_r = 691.35$	$D_x = 1.465$ Mg m^{-3}
Triclinic, $P\overline{1}$	Mo $K\alpha$ radiation
$a = 8.998$ (2) Å	Cell parameters from 6147 reflections
$b = 11.741$ (2) Å	$\theta = 3.1$–$26.0°$
$c = 15.836$ (3) Å	$\mu = 0.68$ mm^{-1}
$\alpha = 102.28$ (3)°	$T = 293$ (2) K
$\beta = 106.11$ (3)°	Prism, green
$\gamma = 90.14$ (3)°	0.38 × 0.25 × 0.18 mm
$V = 1567.2$ (6) Å^3	

Data collection

Rigaku R-AXIS RAPID diffractometer	5968 independent reflections
ω scans	4368 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)	$R_{int} = 0.032$
$T_{min} = 0.781$, $T_{max} = 0.887$	$\theta_{max} = 26.0°$
12 850 measured reflections	$h = -11 \rightarrow 11$
	$k = -12 \rightarrow 14$
	$l = -19 \rightarrow 19$

Refinement

Refinement on F^2	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.044$	$w = 1/[\sigma^2(F_o^2) + (0.0704P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.118$	$(\Delta/\sigma)_{max} < 0.001$
$S = 1.04$	$\Delta\rho_{max} = 0.41$ e Å^{-3}
5968 reflections	$\Delta\rho_{min} = -0.24$ e Å^{-3}
424 parameters	

Table 1
Selected geometric parameters (Å, °).

Ni1—N1	2.136 (2)	Ni1—N7	2.131 (2)
Ni1—N3	2.111 (2)	Ni1—N9	2.109 (2)
Ni1—N5	2.135 (2)	Ni1—N11	2.148 (2)
N1—Ni1—N11	89.56 (9)	N7—Ni1—N5	88.93 (8)
N3—Ni1—N1	88.71 (9)	N7—Ni1—N11	90.90 (9)
N3—Ni1—N5	89.42 (9)	N9—Ni1—N1	90.49 (9)
N3—Ni1—N7	89.92 (9)	N9—Ni1—N3	178.28 (8)
N3—Ni1—N11	90.3 (1)	N9—Ni1—N5	92.12 (9)
N5—Ni1—N1	90.62 (9)	N9—Ni1—N7	90.89 (9)
N5—Ni1—N11	179.68 (9)	N9—Ni1—N11	88.15 (9)
N7—Ni1—N1	178.56 (9)		

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H29···O1	0.86	1.91	2.765 (3)	176
N4—H30···O1^{i}	0.86	2.23	2.808 (4)	125
N4—H30···O3^{i}	0.86	2.28	3.109 (3)	161
N6—H31···O6^{ii}	0.86	1.97	2.731 (3)	147
N8—H32···O6^{iii}	0.86	1.95	2.775 (3)	160
N10—H33···O5^{iv}	0.86	1.90	2.751 (3)	170
N12—H34···O2^{v}	0.86	1.88	2.737 (3)	173
N12—H34···O1^{v}	0.86	2.62	3.172 (3)	123

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

H atoms were placed in calculated positions [C—H = 0.93 (aromatic) or 0.97 Å (aliphatic) and N—H = 0.86 Å (imidazole), and $U_{iso}(H) = 1.2U_{eq}(C,N)$] and refined with a riding-model approximation.

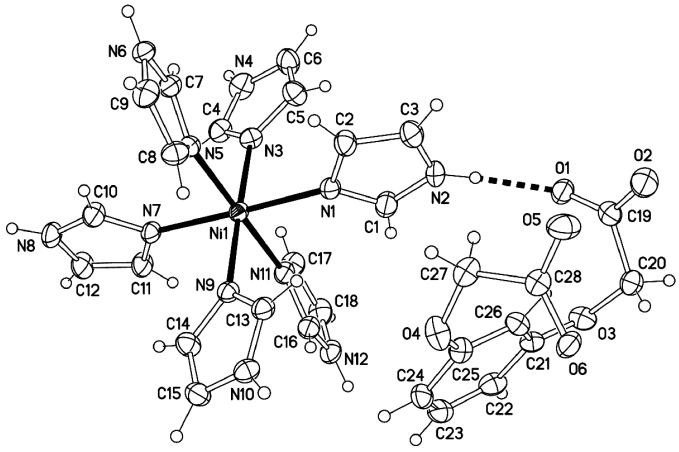


Figure 1
*ORTEP*II (Johnson, 1976) plot of (I), showing 30% probability ellipsoids. The dashed line indicates a hydrogen bond.

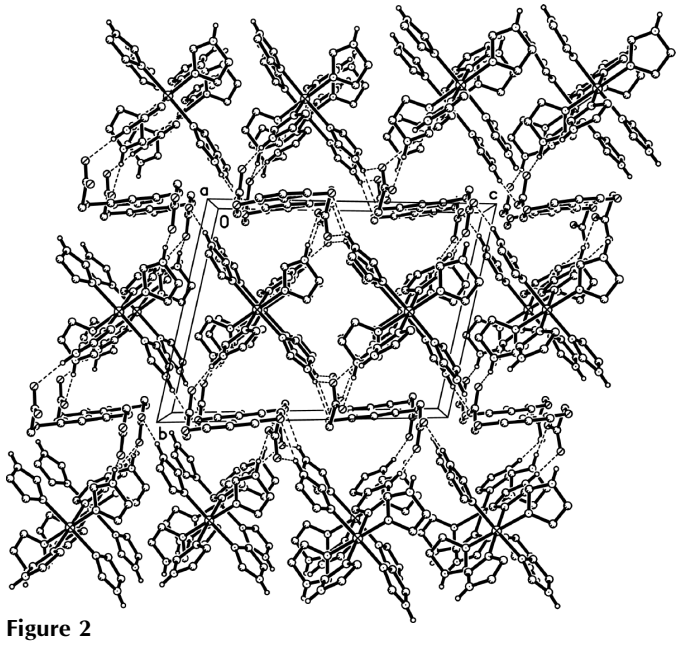


Figure 2
Packing diagram of (I).

Data collection: *RAPID-AUTO* (Rigaku, 1998); cell refinement: *RAPID-AUTO*; data reduction: *CrystalStructure* (Rigaku/MSC, 2002); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*II (Johnson, 1976); software used to prepare material for publication: *SHELXL*97.

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