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trans-Diaquabis[4-(4-methylphenyl)-3,5-bis(2-pyridyl)-4*H*-1,2,4-triazole]nickel(II) diperchlorate tetrahydrate

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.010 Å
H-atom completeness 91%
Disorder in solvent or counterion
R factor = 0.065
wR factor = 0.171
Data-to-parameter ratio = 12.5

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

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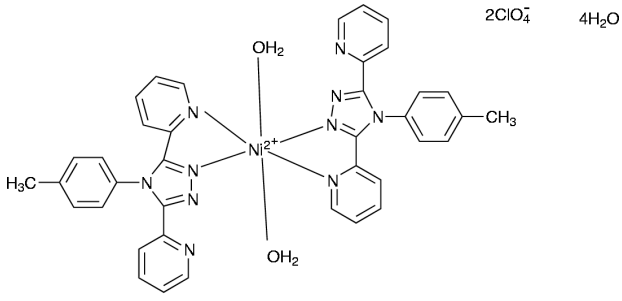
In the title centrosymmetric mononuclear nickel(II) compound, $[Ni(C_{19}H_{15}N_5)_2(H_2O)_2](ClO_4)_2\cdot 4H_2O$, the central Ni^{II} atom is coordinated by four N atoms from two 4-(4-methylphenyl)-3,5-bis(2-pyridyl)-4*H*-1,2,4-triazole ligands, and two O atoms from two water molecules. The coordination geometry is slightly distorted octahedral. In the crystal structure, the molecules are linked together by intermolecular O—H···O hydrogen bonds.

Comment

Substituted 1,2,4-triazoles have been actively studied as bridging ligands between transition metal(II) ions coordinating through atoms N1 and N2, since their complexes have interesting structures and specific magnetic properties (Antolini *et al.*, 1990, 1991; Bencini *et al.*, 1987; Lavrenova *et al.*, 1995; van Koningsbruggen *et al.*, 1997). We report here the crystal structure of a new nickel(II) complex with the ligand 4-(4-methylphenyl)-3,5-bis(2-pyridyl)-4-*H*-1,2,4-triazole (MBPT).



(I)

The title compound, (I), consists of a centrosymmtric mononuclear nickel(II) complex cation, two perchlorate counter-ions and four uncoordinated water molecules. In the cation (Fig. 1), the central Ni atom lies on a crystallographic inversion centre and is six-coordinated by four N atoms from two inversion-related MBPT ligands, and by the centrosymmetrically related O atoms from two water molecules. The substituted triazole molecule acts as a bidentate ligand. The Ni^{II} atom is in a slightly distorted octahedral environment, with the four N atoms occupying the equatorial plane (exactly planar) and the axial positions occupied by the water atoms O1*W* and O1*W*$(1-x, 1-y, 1-z)$ at 2.062 (5) Å on either side. The three *trans* angles at the Ni^{II} atom are exactly 180° by virtue of the crystallographic symmetry (Table 1), and the other angles subtended at the Ni^{II} atom are close to 90°, ranging from 77.76 (19) to 102.24 (19)°.

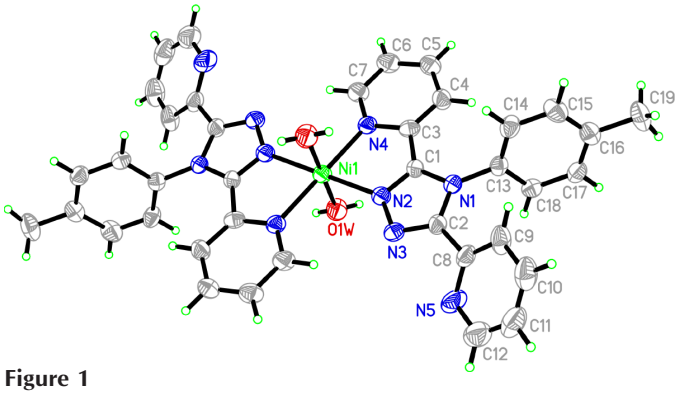


Figure 1
The structure of (I), showing 30% probability displacement ellipsoids and the atom-numbering scheme. Unlabelled atoms are related to labelled atoms by $1-x, 1-y, 1-z$. The perchlorate ion and water molecules have been omitted for clarity.

Two pyridine rings, two triazole rings and the equatorial plane of the Ni atom are nearly coplanar. The dihedral angle between the equatorial plane and the triazole ring is 4.5 (6)°. The crystal packing is stabilized by O—H···O hydrogen bonds (Table 2).

Experimental

Nickel(II) perchlorate and two molar equivalents of MBPT were dissolved in acetonitrile with stirring. After allowing the resulting clear purple solution to stand at room temperature in air for 7 d, large blue crystals were formed at the bottom of the vessel on slow evaporation of the solvent. The crystals were isolated, washed three times with acetonitrile and dried in a vacuum desiccator using anhydrous $CaCl_2$ (yield 78%). Analysis found: C 45.83, H 4.36, N 14.02%; calculated for $C_{38}H_{42}Cl_2N_{10}NiO_{14}$: C 45.99, H 4.27, N 14.11%.

Crystal data

$[Ni(C_{19}H_{15}N_5)_2(H_2O)_2](ClO_4)_2\cdot 4H_2O$
M_r = 988.39
Monoclinic, $P2_1/c$
a = 9.139 (3) Å
b = 15.480 (6) Å
c = 16.240 (6) Å
β = 95.168 (7)°
V = 2288.2 (14) Å^3
Z = 2
D_x = 1.441 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 12113 reflections
θ = 4.2–28.3°
μ = 0.62 mm^{-1}
T = 293 (2) K
Block, blue
0.20 × 0.15 × 0.09 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.887, T_{max} = 0.947
11832 measured reflections
4035 independent reflections
1637 reflections with $I > 2\sigma(I)$
R_{int} = 0.073
θ_{max} = 25.0°
$h = -10 \rightarrow 7$
$k = -18 \rightarrow 18$
$l = -19 \rightarrow 18$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.065
$wR(F^2)$ = 0.171
S = 0.84
4035 reflections
322 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.079P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.014
$\Delta\rho_{max}$ = 0.45 e Å^{-3}
$\Delta\rho_{min}$ = −0.34 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Ni1—N2	2.048 (4)	Ni1—N4	2.098 (5)
Ni1—O1*W*	2.062 (5)		
N2—Ni1—O1*W*	90.31 (19)	O1*W*—Ni1—N4	90.65 (19)
N2—Ni1—O1*W*^{i}	89.69 (19)	N2—Ni1—N4^{i}	102.24 (19)
N2—Ni1—N4	77.76 (19)	O1*W*—Ni1—N4^{i}	89.35 (19)

Symmetry code: (i) $1-x, 1-y, 1-z$.

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1*W*—H1*WA*···O2*W*^{ii}	0.77 (6)	1.89 (6)	2.657 (9)	178 (8)
O1*W*—H1*WB*···O1*A*^{iii}	0.80 (6)	1.99 (6)	2.785 (12)	169 (7)
O1*W*—H1*WB*···O4*B*^{iii}	0.80 (6)	2.16 (6)	2.933 (13)	162 (7)
O1*W*—H1*WB*···O2*C*^{iii}	0.80 (6)	2.00 (6)	2.69 (2)	144 (6)
O3*W*—H3*WA*···O2*W*	0.86 (2)	2.15 (7)	2.766 (12)	129 (7)

Symmetry codes: (ii) $1-x, y-\frac{1}{2}, \frac{1}{2}-z$; (iii) $x, \frac{1}{2}-y, \frac{1}{2}+z$.

The disordered perchlorate anion was modelled with three sets of four O atoms, each set restrained to tetrahedral geometry, and with the sum of their occupancies [0.418 (9), 0.360 (4) and 0.222 (9), respectively] strongly restrained to be equal to unity. The disordered O atoms were refined isotropically. The C-bound H atoms were positioned geometrically (C—H = 0.93 or 0.96 Å) and constrained to ride on their parent atoms, with U_{iso}(H) = 1.2U_{eq}(C) or 1.5U_{eq}(methyl C). Water atoms H1*WA*, H1*WB*, H2*WA* and H3*WA* were located in a difference map and their positional parameters were refined with a fixed U_{iso} value of 0.08 Å^2. The remaining two H atoms of the two free water molecules were not located in the difference map.

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997*a*); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997*a*); molecular graphics: *SHELXTL* (Sheldrick, 1997*b*); software used to prepare material for publication: *SHELXTL*.

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