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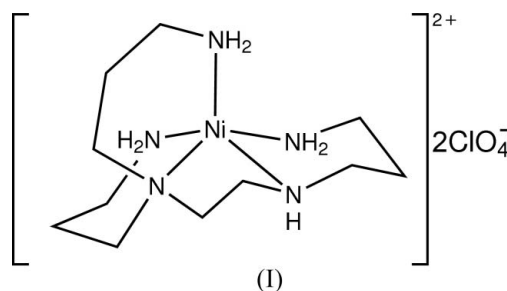
Key indicators

Single-crystal X-ray study
 $T = 298$ K
Mean $\sigma(\text{C}-\text{C}) = 0.015$ Å
Disorder in solvent or counterion
 R factor = 0.064
 wR factor = 0.131
Data-to-parameter ratio = 11.4For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.**[*N,N,N'*-Tris(3-aminopropyl)ethylenediamine- κ^5N]nickel bis(perchlorate)**The asymmetric unit of the title complex, $[\text{Ni}(\text{C}_{11}\text{H}_{29}\text{N}_5)](\text{ClO}_4)_2$, consists of an $[\text{Ni}(\text{C}_{11}\text{H}_{29}\text{N}_5)]^{2+}$ cation and two uncoordinated perchlorate anions. The Ni^{II} atom is five-coordinated in a slightly distorted square-pyramidal geometry, with four of the N atoms forming the basal plane and the fifth N atom in the apical position. The complex shows a three-dimensional network structure assembled by intermolecular hydrogen bonds.

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Comment

Research on organic polyamines is currently of great interest because of their potential applications as useful organic ligands, in which the amine N atoms have strong coordination ability to transition metal ions (Xu *et al.*, 1997). *N,N,N',N'*-Tetrakis(3-aminopropyl)ethylenediamines have been extensively studied (Mikuriya *et al.*, 1985; Micheloni *et al.*, 1986), but few new unsymmetric organic polyamine *N,N,N'*-tris(3-aminopropyl)ethylenediamines and their complexes have been reported. Here we report the synthesis and crystal structure of a new pentamine nickel(II) complex, (I). The asymmetric unit of the Ni^{II} complex consists of an [*N,N,N'*-tris(3-aminopropyl)ethylenediamine- κ^5N]nickel cation and two uncoordinated perchlorate anions.From Fig. 1, it can be seen that the Ni^{II} atom is five-coordinated by five N atoms in a slightly distorted square-pyramidal geometry. The value of the τ parameter (0.05) is close to the ideal value for a square-pyramidal coordination polyhedron (Addison *et al.*, 1984). Four N atoms (N1, N2, N3 and N4) form the basal plane, with atom N5 in the apical position. The Ni atom is displaced by 0.281 (4) Å from the basal plane.

The packing diagram (Fig. 2) shows that there is extensive hydrogen bonding in the crystal structure. The five N atoms of the pentadentate ligand form intermolecular hydrogen bonds with seven O atoms of the perchlorate anions. The perchlorate O atoms are disordered. Each perchlorate anion acts as an

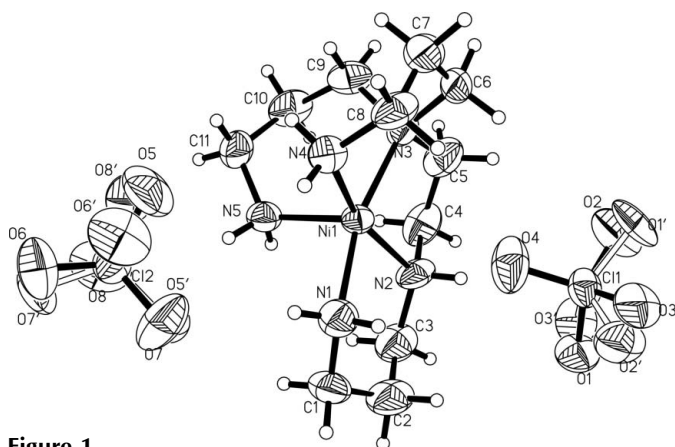


Figure 1
The asymmetric unit of the title complex, showing 30% probability displacement ellipsoids. For each anion, both disorder components are shown.

acceptor of hydrogen bonds from the amine groups of an adjacent complex cation. These intermolecular interactions form a three-dimensional network and stabilize the crystal structure.

Experimental

To a stirred solution of $\text{Ni}(\text{ClO}_4)_2 \cdot 6\text{H}_2\text{O}$ (0.5 mmol) in methanol (15 ml) was added dropwise a solution of *N,N,N'*-tris(3-amino-propyl)ethylenediamine (0.5 mmol) in methanol (10 ml) at room temperature. After stirring for 1 h at 320 K, the complex precipitated and was filtered off, washed with methanol and dried *in vacuo*. Blue-purple single crystals, in about 46% yield, suitable for X-ray structure determination were obtained by slow evaporation of the resulting filtrates for about 20 d at ambient temperature. Analysis, found: C 27.08, H 5.89, N 14.29%; calculated for $\text{C}_{11}\text{H}_{29}\text{Cl}_2\text{N}_5\text{NiO}_8$: C 27.01, H 5.93, N 14.31%.

Crystal data

$[\text{Ni}(\text{C}_{11}\text{H}_{29}\text{N}_5)](\text{ClO}_4)_2$	$Z = 4$
$M_r = 489.00$	$D_x = 1.627 \text{ Mg m}^{-3}$
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation
$a = 9.454 (4) \text{ \AA}$	$\mu = 1.29 \text{ mm}^{-1}$
$b = 14.589 (7) \text{ \AA}$	$T = 298 (2) \text{ K}$
$c = 14.477 (7) \text{ \AA}$	Block, blue-purple
$\beta = 90.285 (8)^\circ$	$0.42 \times 0.30 \times 0.19 \text{ mm}$
$V = 1996.6 (16) \text{ \AA}^3$	

Data collection

Bruker SMART CCD area-detector diffractometer	10278 measured reflections
φ and ω scans	3526 independent reflections
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	1216 reflections with $I > \sigma(I)$
$T_{\min} = 0.614$, $T_{\max} = 0.792$	$R_{\text{int}} = 0.120$
	$\theta_{\text{max}} = 25.0^\circ$

Refinement

Refinement on F^2	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.064$	$w = 1/[\sigma^2(F_o^2) + (0.0268P)^2]$
$wR(F^2) = 0.131$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.00$	$(\Delta/\sigma)_{\text{max}} < 0.001$
3526 reflections	$\Delta\rho_{\text{max}} = 0.48 \text{ e \AA}^{-3}$
309 parameters	$\Delta\rho_{\text{min}} = -0.41 \text{ e \AA}^{-3}$

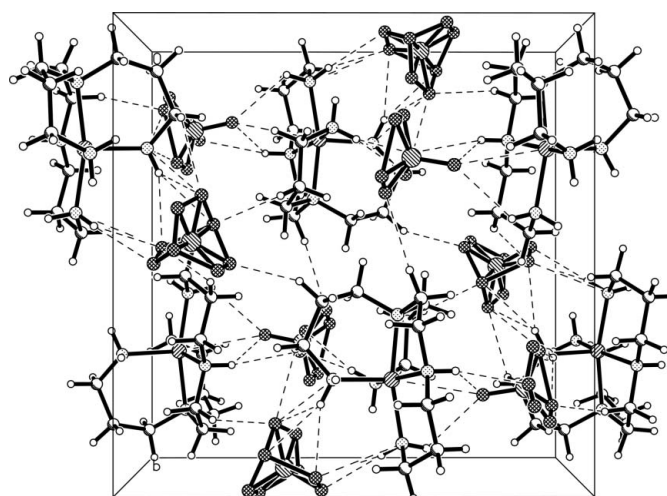


Figure 2
The crystal packing of the title complex, showing the three-dimensional network structure. Dashed lines indicate hydrogen bonds.

Table 1

Selected geometric parameters (\AA , $^\circ$).

Ni1—N5	2.015 (6)	Ni1—N2	2.058 (6)
Ni1—N4	2.026 (6)	Ni1—N3	2.084 (7)
Ni1—N1	2.049 (6)		
N5—Ni1—N4	99.5 (3)	N1—Ni1—N2	91.0 (3)
N5—Ni1—N1	97.8 (3)	N5—Ni1—N3	96.3 (3)
N4—Ni1—N1	90.3 (3)	N4—Ni1—N3	90.7 (3)
N5—Ni1—N2	97.7 (3)	N1—Ni1—N3	165.5 (3)
N4—Ni1—N2	162.4 (3)	N2—Ni1—N3	83.8 (3)

Table 2

Hydrogen-bond geometry (\AA , $^\circ$).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
N1—H1A \cdots O4	0.90	2.52	3.119 (9)	125
N1—H1B \cdots O5'	0.90	2.22	2.95 (2)	138
N1—H1B \cdots O7	0.90	2.29	3.150 (13)	160
N2—H2 \cdots O3'	0.91	2.34	3.24 (2)	166
N2—H2 \cdots O4	0.91	2.51	3.161 (10)	129
N2—H2 \cdots Cl1	0.91	2.95	3.801 (8)	157
N4—H4A \cdots O3 ⁱⁱ	0.90	2.32	3.140 (17)	151
N4—H4A \cdots O1 ⁱ	0.90	2.47	3.29 (3)	153
N4—H4B \cdots O7 ⁱⁱ	0.90	2.43	3.235 (13)	149
N4—H4B \cdots O6 ⁱⁱⁱ	0.90	2.64	3.42 (3)	145
N4—H4B \cdots O5	0.90	2.66	3.107 (12)	112
N5—H5A \cdots O1 ⁱⁱⁱ	0.90	2.31	3.14 (2)	153
N5—H5A \cdots O3 ⁱⁱⁱ	0.90	2.54	3.33 (3)	147
N5—H5B \cdots O8'	0.90	2.23	2.95 (2)	136
N5—H5B \cdots O5'	0.90	2.45	3.30 (3)	158
N5—H5B \cdots O8	0.90	2.47	3.328 (14)	160
N5—H5B \cdots Cl2	0.90	2.94	3.794 (7)	159

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

Methylene H atoms were placed in calculated positions with $C-H = 0.97 \text{ \AA}$ and torsion angles were refined to fit the electron density, $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$. Amido H atoms were placed geometrically with $N-H = 0.90$ and 0.91 \AA , and refined in riding mode with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{N})$.

Data collection: *SMART* (Siemens, 1996); cell refinement: *SMART*; data reduction: *SAINT* (Siemens, 1996); program(s) used to

solve structure: *SHELXS97* (Sheldrick, 1997a); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997a); molecular graphics: *SHELXTL* (Sheldrick, 1997b); software used to prepare material for publication: *SHELXTL*.

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