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Key indicators

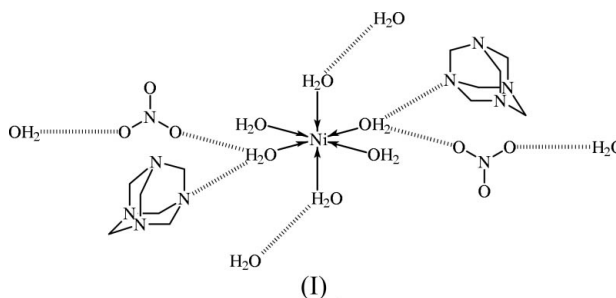
Single-crystal X-ray study
 $T = 298\text{ K}$
Mean $\sigma(\text{C-N}) = 0.004\text{ \AA}$
 R factor = 0.043
 wR factor = 0.095
Data-to-parameter ratio = 14.9For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.Hexaaquanickel(II) dinitrate bis(hexamethylene-
tetramine) tetrahydrate

In the title compound, $[\text{Ni}(\text{H}_2\text{O})_6](\text{NO}_3)_2 \cdot 2\text{C}_6\text{H}_{12}\text{N}_4 \cdot 4\text{H}_2\text{O}$, the Ni atom lies on the crystallographic inversion center and is coordinated by six water molecules in an octahedral environment. The coordinated water molecules are involved in hydrogen bonding with the hexamethylenetetramine, nitrate and lattice water molecules, thus furnishing a three-dimensional network motif. The hexamethylenetetramine entity is linked to three different $[\text{Ni}(\text{H}_2\text{O})_6]^{2+}$ octahedra, as well as to a lattice water molecule.

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Comment

Hexamethylenetetramine, a nitrogen-donor ligand, functions as a bridging entity in a number of its complexes with metal salts (Carlucci *et al.*, 1995, 1997); occasionally, it exists as a host molecule in inclusion compounds (Reddy *et al.*, 1993, 1994). Both classes have been investigated in detail (Ganesh *et al.*, 1990; Zheng *et al.*, 2001). The reaction of nickel(II) acetate with this ligand yielded a mixed crystal of dinickel tetraacetate with hexamethylenetetramine (Wang *et al.*, 2002); the crystal adopts a chain architecture.



The analogous reaction with nickel nitrate in place of nickel acetate afforded the title hexaaquanickel dinitrate bis(hexamethylenetetramine) tetrahydrate, (I); each hexamethylenetetramine entity uses three of the four nitrogen sites to interact, indirectly, with the Ni atom through the coordinated water molecules. The Ni atom, which lies at the crystallographic inversion center, shows octahedral coordination (Fig. 1). The hexamethylenetetramine, nitrate and lattice water entities lie in general positions in the crystal structure. The ligand forms four hydrogen bonds, and each water molecule forms a pair of hydrogen bonds to give rise to a tightly held three-dimensional structure.

Experimental

10 ml of an aqueous solution of nickel nitrate tetrahydrate (0.26 g, 1 mmol) was mixed with 10 ml of a water solution of hexamethylenetetramine (0.28 g, 2 mmol). The mixture was filtered, and after the solution was set aside for several weeks, blue crystals precipitated.

Crystal data

[Ni(H₂O)₆](NO₃)₂·2C₆H₁₂N₄·4H₂O
M_r = 643.28
 Triclinic, *P* $\bar{1}$
a = 9.087 (2) Å
b = 9.343 (2) Å
c = 9.682 (2) Å
 α = 87.761 (3)°
 β = 75.719 (2)°
 γ = 61.275 (2)°
V = 695.5 (2) Å³

Z = 1
D_x = 1.536 Mg m⁻³
 Mo *K*α radiation
 Cell parameters from 1562 reflections
 θ = 2.2–27.9°
 μ = 0.78 mm⁻¹
T = 298 (2) K
 Block, blue
 0.42 × 0.32 × 0.25 mm

Data collection

Siemens CCD area-detector diffractometer
 ω scans
 Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.734, *T_{max}* = 0.828
 4434 measured reflections

3095 independent reflections
 2192 reflections with *I* > 2σ(*I*)
R_{int} = 0.023
 θ_{\max} = 27.9°
h = -11 → 11
k = -9 → 11
l = -12 → 12

Refinement

Refinement on *F*²
R [*F*² > 2σ(*F*²)] = 0.043
wR (*F*²) = 0.095
S = 0.88
 3095 reflections
 208 parameters

H atoms treated by a mixture of independent and constrained refinement
 $w = 1/[\sigma^2(F_o^2) + (0.044P)^2]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 0.55 \text{ e } \text{Å}^{-3}$
 $\Delta\rho_{\min} = -0.45 \text{ e } \text{Å}^{-3}$

Table 1

Selected geometric parameters (Å, °).

Ni1—O1w	2.063 (2)	Ni1—O3w	2.022 (2)
Ni1—O2w	2.059 (2)		
O1w—Ni1—O2w	92.5 (1)	O1w—Ni1—O3w ⁱ	91.2 (1)
O1w—Ni1—O2w ^j	87.5 (1)	O2w—Ni1—O3w	87.8 (1)
O1w—Ni1—O3w	88.8 (1)		

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

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>
O1w—H1w1...O1	0.84 (1)	2.03 (1)	2.872 (3)	173 (3)
O1w—H1w2...N1	0.85 (1)	2.04 (1)	2.875 (3)	167 (2)
O2w—H2w1...O2 ⁱ	0.85 (1)	2.01 (1)	2.846 (3)	169 (2)
O2w—H2w2...N4 ⁱⁱ	0.85 (1)	1.99 (1)	2.829 (3)	171 (3)
O3w—H3w1...O4w	0.85 (1)	1.84 (1)	2.662 (3)	164 (3)
O3w—H3w2...N3 ⁱⁱⁱ	0.85 (1)	1.97 (1)	2.802 (3)	170 (2)
O4w—H4w1...O5w ^{iv}	0.85 (1)	1.97 (1)	2.813 (3)	174 (3)
O4w—H4w2...N2 ^v	0.84 (1)	1.98 (1)	2.817 (3)	173 (3)
O5w—H5w1...O2 ^{vi}	0.85 (1)	2.06 (1)	2.901 (4)	168 (3)
O5w—H5w2...O3	0.85 (1)	1.96 (1)	2.799 (4)	168 (3)

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

The water-bound H atoms were located and refined, subject to O—H 0.85±0.01 Å and H...H = 1.39±0.01 Å; their displacement parameters were set to be 1.2 times those of the parent O atoms.

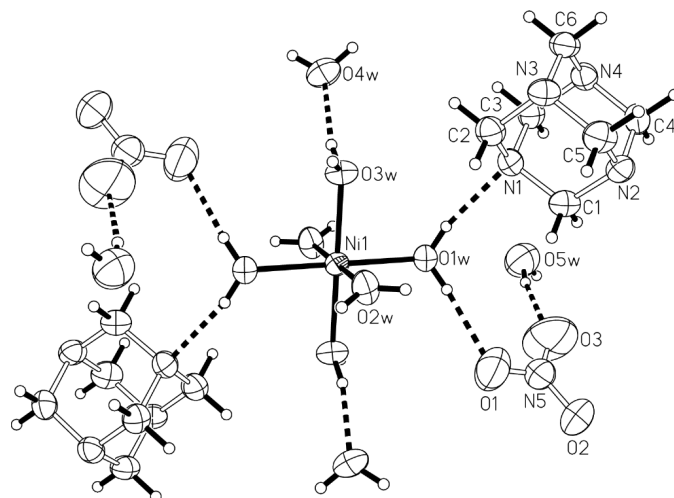


Figure 1

ORTEP II (Johnson, 1976) plot of the unit-cell contents at the 50% probability level. H atoms are drawn as spheres of arbitrary radii.

Data collection: *SMART* (Siemens, 1997); cell refinement: *SAINT* (Siemens, 1997); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP II* (Johnson, 1976); software used to prepare material for publication: *SHELXL97*.

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