Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$B_{eq} = (8\pi^2/3)\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$

	Occupancy	x	y	z	B_{eq}
Cu	1.0	0.76043 (6)	0.57319 (6)	0.12616 (6)	3.47 (4)
Cl(1)	1.0	0.2207 (2)	0.6122 (2)	0.11995 (18)	7.28 (16)
Cl(2)	1.0	0.50818 (18)	0.33499 (18)	0.15953 (15)	5.26 (12)
O(11)	0.7	0.1733 (12)	0.5371 (18)	0.0924 (10)	11.5 (10)
O(12)	1.0	0.1812 (8)	0.6887 (6)	0.0923 (6)	12.2 (7)
O(13)	0.7	0.1895 (15)	0.6008 (18)	0.1894 (10)	16.7 (19)
O(14)	1.0	0.3087 (7)	0.6007 (9)	0.0975 (7)	15.3 (9)
O(21)	1.0	0.4724 (9)	0.2868 (7)	0.2133 (7)	15.9 (9)
O(22)	1.0	0.4619 (8)	0.4166 (6)	0.1594 (6)	12.8 (8)
O(23)	0.7	0.4830 (13)	0.2777 (11)	0.1073 (10)	14.5 (12)
O(24)	1.0	0.5956 (7)	0.3496 (9)	0.1710 (8)	16.2 (10)
O(11′)	0.3	0.1321 (3)	0.573 (3)	0.093 (2)	11.3 (12)
O(13′)	0.3	0.260 (4)	0.6491 (3)	0.1855 (19)	15 (4)
O(23′)	0.3	0.539 (2)	0.346 (2)	0.0928 (15)	8.6 (18)
N(1)	1.0	0.6936 (6)	0.6488 (6)	0.2066 (5)	6.7 (5)
N(2)	1.0	0.6286 (5)	0.5475 (5)	0.0969 (5)	5.8 (5)
N(3)	1.0	0.7809 (6)	0.5988 (6)	0.0196 (4)	6.0 (5)
N(4)	1.0	0.8876 (5)	0.5987 (6)	0.1561 (5)	5.7 (4)
N(5)	1.0	0.7929 (5)	0.4367 (5)	0.1594 (4)	4.7 (3)
C(1)	1.0	0.7333 (11)	0.6499 (11)	0.2773 (7)	9.8 (9)
C(2)	1.0	0.5981 (8)	0.6210 (8)	0.2056 (8)	7.4 (7)
C(3)	1.0	0.5686 (7)	0.6114 (7)	0.1299 (9)	7.4 (7)
C(4)	1.0	0.6211 (8)	0.5477 (8)	0.0180 (6)	6.3 (6)
C(5)	1.0	0.6891 (8)	0.6158 (9)	−0.0114 (6)	7.2 (6)
C(6)	1.0	0.8455 (7)	0.6677 (8)	−0.0009 (7)	6.9 (6)
C(7)	1.0	0.9369 (9)	0.6536 (9)	0.0367 (7)	7.8 (7)
C(8)	1.0	0.9381 (7)	0.6698 (8)	0.1127 (7)	7.0 (6)
C(9)	1.0	0.9378 (7)	0.5119 (8)	0.1580 (7)	7.2 (6)
C(10)	1.0	0.8791 (7)	0.4444 (7)	0.1976 (6)	6.1 (5)
C(11)	1.0	0.7958 (8)	0.3724 (6)	0.1009 (6)	6.3 (6)

Table 2. *Selected geometric parameters* (Å, °)

Cu—N(1)	2.121 (9)	N(3)—C(6)	1.45 (1)
Cu—N(2)	2.037 (8)	N(4)—C(8)	1.53 (1)
Cu—N(3)	2.056 (8)	N(4)—C(9)	1.49 (1)
Cu—N(4)	1.977 (8)	N(5)—C(10)	1.45 (1)
Cu—N(5)	2.182 (7)	N(5)—C(11)	1.46 (1)
N(1)—C(1)	1.45 (2)	C(2)—C(3)	1.49 (2)
N(1)—C(2)	1.45 (2)	C(4)—C(5)	1.52 (2)
N(2)—C(3)	1.44 (1)	C(6)—C(7)	1.52 (2)
N(2)—C(4)	1.48 (1)	C(7)—C(8)	1.45 (2)
N(3)—C(5)	1.48 (1)	C(9)—C(10)	1.52 (2)
N(1)—Cu—N(2)	81.8 (4)	Cu—N(3)—C(6)	119.0 (7)
N(1)—Cu—N(3)	131.2 (4)	C(5)—N(3)—C(6)	111.2 (9)
N(1)—Cu—N(4)	97.3 (4)	Cu—N(4)—C(8)	115.8 (6)
N(1)—Cu—N(5)	113.1 (3)	Cu—N(4)—C(9)	107.6 (6)
N(2)—Cu—N(3)	84.8 (4)	C(8)—N(4)—C(9)	112.3 (8)
N(2)—Cu—N(4)	179.1 (4)	Cu—N(5)—C(10)	104.8 (5)
N(2)—Cu—N(5)	96.1 (3)	Cu—N(5)—C(11)	113.8 (6)
N(3)—Cu—N(4)	96.0 (4)	C(10)—N(5)—C(11)	113.5 (8)
N(3)—Cu—N(5)	114.8 (3)	N(1)—C(2)—C(3)	108.4 (9)
N(4)—Cu—N(5)	83.9 (3)	N(2)—C(3)—C(2)	107 (1)
Cu—N(1)—C(1)	118.2 (8)	N(2)—C(4)—C(5)	108.3 (8)
Cu—N(1)—C(2)	106.3 (7)	N(3)—C(5)—C(4)	109.4 (9)
C(1)—N(1)—C(2)	114 (1)	N(3)—C(6)—C(7)	110.4 (9)
Cu—N(2)—C(3)	109.6 (7)	C(6)—C(7)—C(8)	116 (1)
Cu—N(2)—C(4)	109.8 (6)	N(4)—C(8)—C(7)	113.8 (9)
C(3)—N(2)—C(4)	113 (1)	N(4)—C(9)—C(10)	108.1 (9)
Cu—N(3)—C(5)	106.4 (7)	N(5)—C(10)—C(9)	107.5 (8)

The structure was solved by direct and Fourier methods and refined by full-matrix least-squares techniques. H atoms were located by difference Fourier methods and theoretical calculation. The high value of $(\Delta/\sigma)_{max}$ is a result of the disordered perchlorate groups. *NRCVAX* (Gabe, Le Page, White & Lee, 1987) was used for all calculations.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and hydrogen-bonding geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71636 (10 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: AS1059]

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Acta Cryst. (1994). C**50**, 516–518

[(4*RS*,7*SR*,11*RS*)-4,12-Dimethyl-4,7,11-triazatridecylamine-*N*,*N*′,*N*″,*N*‴]nickel(II) Diperchlorate

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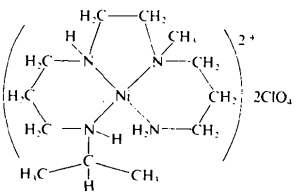
Abstract

The Ni^{II} ion of the title complex, $[Ni(C_{12}H_{30}N_4)](ClO_4)_2$, is four-coordinate with the four N atoms in equatorial positions. The Ni^{II} coordination is planar

with slight tetrahedral distortion. Each terminal six-membered ring is in a chair form and the central five-membered ring is in a skew form. Hydrogen bonds between the amino groups and the perchlorate O atoms help stabilize the crystal structure.

Comment

The thermodynamic properties of Ni^{II} complexes of aliphatic tetraamines have been studied extensively (Hinz & Margerum, 1974). Complexes containing one primary, two secondary and one tertiary amine group, however, have received very little attention. In order to study the steric effect of *N*-alkyl groups on the structure of the nickel(II) complex, the structure determination of the title complex (I) was undertaken.



(I)

The ligand was synthesized according to the method of Lu, Shan, Chao & Chung (1987). A solution of $Ni(ClO_4)_2.6H_2O$ (7.3 g) in methanol (80 ml) was added dropwise to a solution of the ligand (3.76 g) in ethanol (80 ml). The solution changed colour rapidly from violet to brownish and was stirred for 3 h in a steam bath. The product was evaporated to dryness. The brownish solid was dissolved in water and $Ni(OH)_2$ was filtered off. Single crystals were obtained from aqueous solution by slow evaporation.

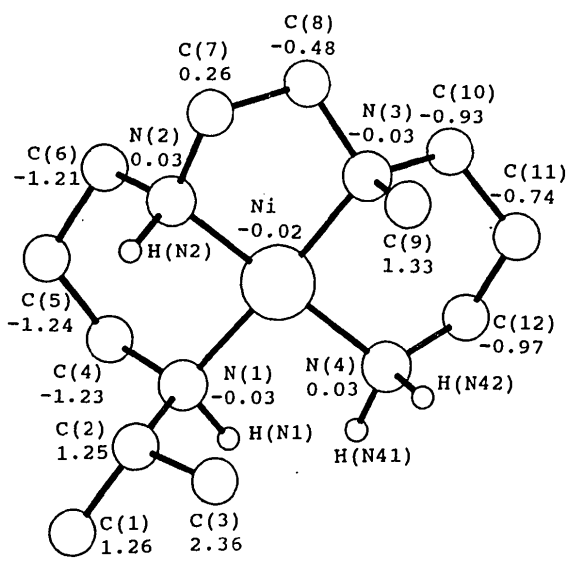


Fig. 1. A perspective view of the molecule with the atom-numbering scheme, excluding the perchlorate groups and H atoms attached to the C atoms. The displacements from the best plane formed by the four N atoms coordinated to the Ni^{II} ion are indicated. E.s.d.'s for the Ni, N and C atoms are 0.004, 0.01 and 0.02 Å, respectively.

The coordination geometry about Ni^{II} is planar with slight tetrahedral distortion. The C(9) and H(N2) atoms are on the same side of the plane defined by the four N atoms and this prevents further coordination of Ni^{II}. The Ni—N distances span a narrow range, 1.915 (7) to 1.951 (6) Å, and are comparable with the average Ni—N distance of 1.95 (4) Å for four-coordinate Ni^{II}–tetraamine complexes (Lu, Chung & Ashida, 1991). The Ni—N(1) and Ni—(3) distances are longer than the Ni—N(2) and Ni—N(4) distances. The complex has a nearly planar NiN_4 unit in which alternate six-, five- and six-membered chelate rings have stable chair, skew and chair conformations, respectively. Hydrogen bonds between amino groups and perchlorate O atoms help stabilize the crystal structure.

Experimental

Crystal data

$[Ni(C_{12}H_{30}N_4)](ClO_4)_2$
M_r = 488.0
Orthorhombic
$P2_12_12_1$
a = 8.491 (4) Å
b = 14.679 (4) Å
c = 16.827 (3) Å
V = 2097 (1) Å^3
Z = 4
D_x = 1.546 Mg m^{-3}
Mo $K\alpha$ radiation
λ = 0.7107 Å
Cell parameters from 25 reflections
θ = 6.07–16°
μ = 1.23 mm^{-1}
T = 298 (3) K
Parallelepiped
0.31 × 0.16 × 0.13 mm
Yellow

Data collection

Nonius CAD-4 diffractometer
$\theta/2\theta$ scans
Absorption correction: empirical (North, Phillips & Mathews, 1968)
T_{min} = 0.8715, T_{max} = 0.8722
3430 measured reflections
3430 independent reflections
1924 observed reflections [$I \geq 2.5\sigma(I)$]
θ_{max} = 29.9°
h = 0 → 11
k = 0 → 20
l = 0 → 23
3 standard reflections
frequency: 60 min
intensity variation: ±6%

Refinement

Refinement on F
R = 0.055
wR = 0.054
S = 0.59
1924 reflections
261 parameters
H-atom parameters not refined
Unit weights applied
$(\Delta/\sigma)_{max}$ = 0.78
$\Delta\rho_{max}$ = 0.54 e Å^{-3}
$\Delta\rho_{min}$ = −0.35 e Å^{-3}
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$B_{eq} = (8\pi^2/3)\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$

	Occupancy	x	y	z	B_{eq}
Ni	1.0	0.24397 (14)	0.80566 (7)	0.22878 (5)	2.82 (4)
Cl(1)	1.0	0.7905 (3)	0.65783 (19)	0.18758 (17)	4.80 (11)
Cl(2)	1.0	0.7010 (4)	0.0539 (2)	0.07441 (17)	5.17 (13)
O(1)	1.0	0.7069 (12)	0.7291 (6)	0.2195 (10)	13.8 (10)
O(2)	1.0	0.6908 (13)	0.5845 (6)	0.1929 (10)	12.5 (9)
O(3)	1.0	0.817 (2)	0.6730 (17)	0.1146 (7)	21.7 (17)
O(4)	1.0	0.9302 (11)	0.6422 (7)	0.2275 (7)	9.4 (6)
O(5)	0.85	0.743 (2)	−0.0367 (7)	0.0833 (9)	11.1 (10)
O(5′)	0.15	0.692 (6)	0.007 (3)	0.149 (3)	4.1 (9)
O(6)	0.70	0.713 (4)	0.0727 (16)	−0.0015 (9)	14.7 (18)
O(6′)	0.30	0.596 (4)	0.028 (3)	0.0201 (19)	9 (2)
O(7)	0.70	0.567 (3)	0.095 (2)	0.0970 (17)	16.0 (19)
O(7′)	0.30	0.600 (6)	0.044 (3)	0.137 (3)	8.3 (11)
O(8)	1.0	0.8190 (14)	0.1081 (8)	0.1101 (9)	11.8 (8)
N(1)	1.0	0.2128 (9)	0.8053 (5)	0.3437 (4)	3.7 (3)
N(2)	1.0	0.1431 (9)	0.9221 (5)	0.2227 (5)	3.6 (3)
N(3)	1.0	0.2691 (9)	0.8115 (6)	0.1137 (4)	3.6 (3)
N(4)	1.0	0.3555 (9)	0.6915 (5)	0.2345 (4)	4.3 (3)
C(1)	1.0	0.3018 (19)	0.8563 (10)	0.4795 (6)	7.9 (8)
C(2)	1.0	0.31396 (14)	0.8673 (8)	0.3909 (6)	4.8 (5)
C(3)	1.0	0.4803 (18)	0.8570 (12)	0.3627 (9)	7.8 (9)
C(4)	1.0	0.0387 (12)	0.8128 (8)	0.3627 (6)	4.2 (4)
C(5)	1.0	−0.0364 (12)	0.8984 (8)	0.3370 (7)	4.9 (5)
C(6)	1.0	−0.0256 (12)	0.9166 (7)	0.2476 (6)	4.3 (4)
C(7)	1.0	0.1594 (14)	0.9618 (7)	0.1409 (6)	5.0 (5)
C(8)	1.0	0.1531 (15)	0.8790 (9)	0.0846 (6)	5.6 (6)
C(9)	1.0	0.4282 (15)	0.8428 (10)	0.0963 (7)	5.9 (6)
C(10)	1.0	0.2395 (18)	0.7258 (8)	0.0700 (6)	6.1 (6)
C(11)	1.0	0.3341 (15)	0.6454 (8)	0.0947 (7)	5.8 (6)
C(12)	1.0	0.3070 (13)	0.6176 (7)	0.1765 (8)	5.3 (5)

Table 2. *Selected geometric parameters* (Å, °)

Ni—N(1)	1.951 (6)	N(3)—C(10)	1.48 (1)
Ni—N(2)	1.915 (7)	N(4)—C(12)	1.52 (1)
Ni—N(3)	1.951 (6)	C(1)—C(2)	1.50 (2)
Ni—N(4)	1.927 (8)	C(2)—C(3)	1.50 (2)
N(1)—C(2)	1.48 (1)	C(4)—C(5)	1.47 (2)
N(1)—C(4)	1.52 (1)	C(5)—C(6)	1.53 (2)
N(2)—C(6)	1.49 (1)	C(7)—C(8)	1.54 (2)
N(2)—C(7)	1.50 (1)	C(10)—C(11)	1.49 (2)
N(3)—C(8)	1.48 (2)	C(11)—C(12)	1.45 (2)
N(3)—C(9)	1.46 (2)		
N(1)—Ni—N(2)	89.7 (3)	C(8)—N(3)—C(9)	109.9 (9)
N(1)—Ni—N(3)	177.2 (3)	C(8)—N(3)—C(10)	107.0 (8)
N(1)—Ni—N(4)	90.8 (3)	C(9)—N(3)—C(10)	109.0 (9)
N(2)—Ni—N(3)	87.5 (3)	Ni—N(4)—C(12)	117.1 (6)
N(2)—Ni—N(4)	177.1 (3)	N(1)—C(2)—C(1)	115 (1)
N(3)—Ni—N(4)	92.0 (3)	N(1)—C(2)—C(3)	108.3 (9)
Ni—N(1)—C(2)	116.8 (6)	C(1)—C(2)—C(3)	112 (1)
Ni—N(1)—C(4)	109.9 (5)	N(1)—C(4)—C(5)	114.9 (8)
C(2)—N(1)—C(4)	114.0 (8)	C(4)—C(5)—C(6)	114.3 (9)
Ni—N(2)—C(6)	111.4 (6)	N(2)—C(6)—C(5)	110.0 (8)
Ni—N(2)—C(7)	110.7 (6)	N(2)—C(7)—C(8)	104.8 (8)
C(6)—N(2)—C(7)	111.5 (7)	N(3)—C(8)—C(7)	107.5 (8)
Ni—N(3)—C(8)	106.6 (6)	N(3)—C(10)—C(11)	116.4 (9)
Ni—N(3)—C(9)	108.3 (6)	C(10)—C(11)—C(12)	113.7 (9)
N—N(3)—C(10)	115.9 (6)	N(4)—C(12)—C(11)	111.4 (8)

The structure was solved by direct and Fourier methods and refined by full-matrix least-squares techniques. H atoms were located by difference Fourier methods and theoretical calculation. *NRCVAX* (Gabe, Le Page, White & Lee, 1987) was used for all calculations.

The authors thank the National Science Council for support under grants NSC82-0208-M007-119 and NSC82-0208-M007-32. They are also indebted to Ms Shu-Fang Tung for collecting the X-ray diffraction data.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71637 (10 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: AS1060]

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Acta Cryst. (1994). C**50**, 518–520

[*N*,*N*′-Bis(3-aminopropyl)-*trans*-1,2-cyclohexanediamine-*N*,*N*′,*N*″,*N*‴]di(perchlorato-*O*)copper(II)

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Abstract

The Cu^{II} ion of the title complex, $[Cu(ClO_4)_2(C_{12}H_{28}N_4)]$, is six-coordinated with four N atoms of the tetradentate ligand on the equatorial plane and two perchlorato O atoms in axial positions. The two asymmetric donor N atoms have the same *R* or *S* configuration. The two terminal six-membered chelate rings are in stable chair forms and the central five-membered chelate ring is in a stable *gauche* form. The cyclohexane ring on the central chelate ring is in a stable chair form. The hydrogen bonds between the NH and NH_2 groups and the perchlorate O atoms help stabilize the crystal structure.

Comment

We have reported previously the crystal structure of [*N*,*N*′-bis(3-aminopropyl)-1,2-ethanediamine]per-