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{1,3,5,7-Tetraazabicyclo[3.3.1]nonane-3,7-bis(acetato)-N^3,N^7,O,O''}nickel(II)

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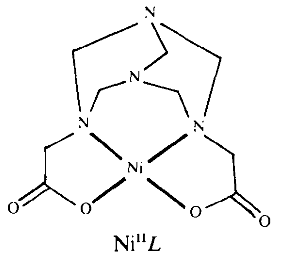
Abstract

The structure of the title compound, $[Ni(C_9H_{14}N_4O_4)]$ has been redetermined in the centrosymmetric space group *Pnma*. The Ni^{II} atom is coordinated by two N and two O atoms of the ligand in an approximately square-planar arrangement.

Comment

The structure has been solved by Teo, Teoh & Snow (1984) in the non-centrosymmetric space group $Pna2_1$; the ligating atoms and Ni were found to lie in a pseudo-mirror plane of this space group. We performed the usual statistical test based on normalized structure factors, *E* (Stout & Jensen, 1989), on our data and found that the title compound is centrosymmetric ($|E^2 - 1| = 0.917$). Therefore, it was possible to solve the structure of this compound in the space group *Pnma*.



$Ni^{II}L$

The structure is composed of isolated molecules of the $Ni^{II}L$ complex; no evidence of hydrogen bonding was found. A perspective view of the molecule is given in Fig. 1. The Ni^{II} atom is coordinated by two N atoms and two O atoms of the ligand molecule in a slightly deformed square-planar arrangement, in a mirror plane of the space group *Pnma*.

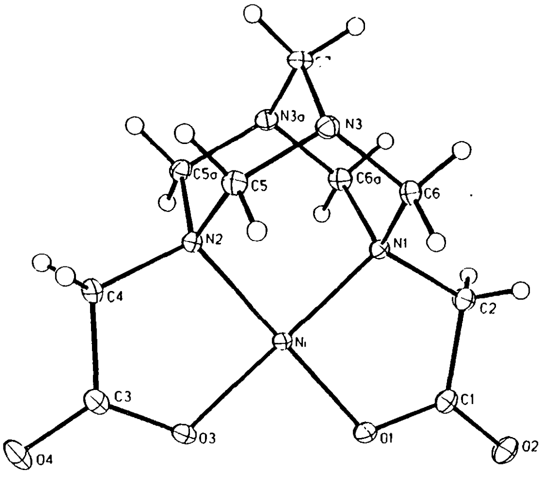


Fig. 1. View of a molecule of the $Ni^{II}L$ complex. The non-H atoms are represented by displacement ellipsoids at the 50% probability level. The H atoms have been assigned arbitrary displacement parameters.

Experimental

The new complex of general formula $Ni^{II}L$ (where L^{2-} is the macrocyclic ligand $C_9H_{14}N_4O_4$) was prepared by template condensation of $[Ni(H_2O)_2(gly)_2]$ (where gly is glycine) with formaldehyde and ammonia. The preparation and physicochemical study of the compound $Ni^{II}L$ have been described previously (Trávníček, Březina, Krausová & Šindelář, 1993). The compound was recrystallized from water.

Crystal data

$[Ni(C_9H_{14}N_4O_4)]$
$M_r = 300.9$
Orthorhombic
Pnma
$a = 10.618$ (2) Å
$b = 6.735$ (1) Å
$c = 14.683$ (3) Å
$V = 1050.0$ (3) Å^3
$Z = 4$
$D_x = 1.904$ Mg m^{-3}
$D_m = 1.87$ Mg m^{-3}
D_m measured by flotation in $CHCl_3/CH_3I$ at 293 K

Cu $K\alpha$ radiation
$\lambda = 1.54178$ Å
Cell parameters from 25 reflections
$\theta = 27.5$–$43.5°$
$\mu = 2.804$ mm^{-1}
$T = 293$ K
Needle
0.65 × 0.1 × 0.1 mm
Orange

Data collection

Kuma KM-4 diffractometer
ω–2θ scans
Absorption correction: none
1571 measured reflections
1199 independent reflections
1029 observed reflections [$I > 5\sigma(I)$]
$R_{int} = 0.0171$

$\theta_{max} = 83°$
$h = 0 \rightarrow 13$
$k = 0 \rightarrow 8$
$l = 0 \rightarrow 18$
2 standard reflections monitored every 50 reflections
intensity variation: 10%

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Refinement

Refinement on F
$R = 0.0361$
$wR = 0.0584$
$S = 1.67$
1029 reflections
131 parameters
All H-atom parameters refined

$w = 1/[\sigma^2(F) + 0.03F^2]$
$(\Delta/\sigma)_{max} = 0.0001$
$\Delta\rho_{max} = 2.15$ e Å^{-3}
$\Delta\rho_{min} = -3.20$ e Å^{-3}
Extinction correction: none
Atomic scattering factors from *The SDS System* (Petříček & Malý, 1992)

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

$B_{eq} = (1/3)\Sigma_i\Sigma_j B_{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j$.

	x	y	z	B_{eq}
Ni	0.08590 (4)	1/4	0.0146 (3)	2.0 (1)
O1	−0.0620 (2)	1/4	−0.0523 (2)	2.8 (1)
O2	−0.1363 (2)	1/4	−0.1937 (2)	3.8 (1)
O3	−0.0033 (2)	1/4	0.1233 (1)	2.9 (1)
O4	0.0214 (3)	1/4	0.2737 (2)	5.4 (1)
N1	0.1772 (2)	1/4	−0.0965 (2)	2.2 (1)
N2	0.2380 (2)	1/4	0.0835 (2)	2.2 (1)
N3	0.3585 (1)	0.0726 (3)	−0.0333 (1)	2.3 (1)
C1	−0.0478 (3)	1/4	−0.1398 (2)	2.6 (1)
C2	0.0856 (2)	1/4	−0.1740 (2)	3.3 (1)
C3	0.0640 (3)	1/4	0.1965 (2)	3.0 (1)
C4	0.2045 (3)	1/4	0.1818 (2)	3.3 (1)
C5	0.3146 (1)	0.0681 (3)	0.0600 (1)	2.4 (1)
C6	0.2595 (2)	0.0664 (3)	−0.1003 (1)	2.5 (1)
C7	0.4344 (2)	1/4	−0.0470 (2)	2.4 (1)

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

Ni—O1	1.852 (2)	N1—C6	1.517 (2)
Ni—O3	1.856 (2)	N2—C4	1.485 (4)
Ni—N1	1.899 (2)	N2—C5	1.512 (3)
Ni—N2	1.905 (2)	N3—C5	1.447 (3)
O1—C1	1.293 (4)	N3—C6	1.441 (3)
O2—C1	1.229 (4)	N3—C7	1.456 (2)
O3—C3	1.291 (4)	C1—C2	1.504 (4)
O4—C3	1.221 (4)	C3—C4	1.508 (4)
N1—C2	1.496 (4)		
O1—Ni—O3	91.3 (1)	C4—N2—C5	110.5 (1)
O1—Ni—N1	88.7 (1)	C5—N3—C6	114.3 (2)
O1—Ni—N2	180.0 (0)	C5—N3—C7	109.0 (2)
O3—Ni—N1	180.0 (0)	C6—N3—C7	109.5 (2)
O3—Ni—N2	88.6 (1)	O1—C1—O2	123.4 (3)
N1—Ni—N2	91.4 (1)	O1—C1—C2	116.2 (3)
Ni—O1—C1	115.4 (2)	O2—C1—C2	120.4 (3)
Ni—O3—C3	115.6 (2)	O3—C3—O4	124.6 (3)
Ni—N1—C2	108.8 (2)	O3—C3—C4	115.4 (3)
Ni—N1—C6	109.0 (1)	O4—C3—C4	120.0 (3)
C2—N1—C6	110.3 (1)	N2—C4—C3	112.1 (2)
Ni—N2—C4	108.2 (2)	N2—C5—N3	111.9 (2)
Ni—N2—C5	109.6 (1)	N1—C6—N3	111.8 (2)

Preliminary lattice parameters and space-group symmetry were determined from Weissenberg photographs. The structure was solved by the heavy-atom method using *SHELXS*86 (Sheldrick, 1985). All atoms, except H atoms, were refined anisotropically by block-diagonal-matrix least-squares procedures using *SDS* (Petříček & Malý, 1992). Absorption corrections were not applied. We tried to make an empirical absorption correction from diffraction data (ψ scan). The R_{int} factor before correction was 0.0171 for all data; after correction it was 0.0119. The influence of the whole correction on the final R factor and the e.s.d.'s of the refined parameters was negligible.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and bond distances involving H atoms have been deposited with the IUCr (Reference: VS1010). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Bis(tetrapropylammonium) Hexachlorodicuprate(II)

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Abstract

Bis(tetrapropylammonium) di-μ-chlorobis(dichlorocuprate)(2−), $(C_{12}H_{28}N)_2[Cu_2Cl_6]$, contains a centrosymmetric hexachlorodicuprate group in which each Cu atom shows coordination to two bridging Cl atoms [Cu—Cl 2.314 (1) Å] and two terminal Cl atoms [average Cu—Cl 2.198 (1) Å] in flattened tetrahedral geometry. There are no interactions between neighboring $[Cu_2Cl_6]^{2-}$ groups.

Comment

Structural observations for $[Cu_2Cl_6]^{2-}$ groups reported in the literature may be divided into three groups depending upon the size of the cation present. All show a pair of Cu^{II} atoms bridged by two Cl atoms, each Cu atom showing further coordination to two terminal Cl atoms. Terminal Cu—Cl distances are typically 2.20 Å and bridging Cl—Cu distances are slightly longer.

When the cation is large, *e.g.* Ph_4P^+ (Textor, Dubler & Oswald, 1974) or Ph_4As^+ (Willett & Chow, 1974), the geometry at each tetracoordinate Cu atom may be described as flattened tetrahedral and no interactions between the dianionic groups are found. When the cationic group is smaller in size, an additional 'long' bond (ap-