Table 3. *Deviations of atoms* (Å) *from specified least-squares planes*

Plane 1: C(1),C(2),B(4) B(7),B(8)		Plane 2: B(5),B(6),B(9) B(11),B(12)		Plane 3: Co,B(8),B(6), B(10)	
C(1)	0·012 (8)	B(5)	−0·010 (10)	Co	0·009 (1)
C(2)	0·009 (8)	B(6)	0·018 (10)	B(8)	−0·010 (9)
B(4)	−0·028 (8)	B(9)	−0·001 (10)	B(6)	−0·010 (11)
B(7)	−0·027 (10)	B(11)	−0·019 (8)	B(10)	0·012 (11)
B(8)	0·033 (8)	B(12)	0·012 (10)		
Co	−1·482 (1)	B(10)	0·940 (10)	C(1)	0·773 (8)
I	−0·520 (1)	I	−2·101 (1)	C(2)	−0·814 (9)
				B(4)	1·432 (9)
				B(7)	−1·436 (9)
				B(5)	1·425 (11)
				B(11)	−1·413 (11)
				B(9)	0·904 (11)
				B(12)	−0·880 (10)

Plane 4: C'(1),C'(2), B'(4),B'(7),B'(8)		Plane 5: B'(5),B'(6), B'(9),B'(11),B'(12)		Plane 6: Co,B'(8),B'(6), B'(10)	
C'(1)	−0·005 (8)	B'(5)	0·009 (8)	Co	0·005 (1)
C'(2)	−0·009 (8)	B'(6)	−0·013 (10)	B'(8)	−0·006 (10)
B'(4)	0·017 (8)	B'(9)	−0·002 (10)	B'(6)	−0·006 (11)
B'(7)	0·020 (10)	B'(11)	0·011 (10)	B'(10)	0·007 (11)
B'(8)	−0·022 (8)	B'(12)	−0·005 (10)		
Co	1·480 (1)	B'(10)	−0·917 (10)	C'(1)	0·793 (9)
				C'(2)	−0·822 (8)
				B'(4)	1·434 (9)
				B'(7)	−1·449 (10)
				B'(5)	1·433 (10)
				B'(11)	−1·429 (11)
				B'(9)	0·881 (10)
				B'(12)	−0·906 (10)

The two icosahedral cages are rotated with respect to one another through an angle of 108 (1)° from the orientation in which the C atoms would overlap (Fig. 1). In the unit cell there are two (−)- and two (+)-enantiomorphic rotational isomers. The angle B(10)—Co—B'(10) is 177·1 (4)° and thus the molecule as a whole is slightly bent.

Using the general nomenclature which has been described by Janoušek, Plešek, Heřmánek, Baše, Todd & Wright (1981) for rotation of non-bridged $(C_2B_9H_{11})_2^{4-}$ sandwiches, the staggered conformation $(s-1,4',2,1')$ is 'frozen' in the present structure. This conformation corresponds to a transition state inter-

mediate between $(s-1,8',2,4')$ *trans*-antiprismatic and $(s-1,1',2,2')$ *cis*-antiprismatic. The reason for the occurrence of this conformation may lie in the steric effect of the halide substituent and repulsion between δ^+ partially charged C atoms present in opposite planes. Thus rotation into one of the non-symmetric conformations will be prevented. A different situation was found in the crystal structure of $(C_2B_9H_{11})_2Ni$ (St Clair, Zalkin & Templeton, 1970). Here the enantiomorphic rotational isomers have cages mutally turned through an angle of 36°. The vectors B(10) to Ni of the first cage and Ni to B(10) of the second cage are parallel. In this case, the structure is 'frozen' in the $(s-1,1',2,2')$ *cis*-antiprismatic conformation.

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Structure of Bis(5-chloro-8-quinolinolato-*N,O*)bis(pyridine)nickel(II)–Pyridine Dihydrate

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Abstract. $[Ni(C_9H_5ClNO)_2(C_5H_5N)_2].C_5H_5N.2H_2O$, $M_r = 689·2$, triclinic, $P\bar{1}$, $a = 15·729$ (5), $b = 11·065$ (5), $c = 9·497$ (7) Å, $\alpha = 95·89$ (2), $\beta = 105·20$ (2), $\gamma = 90·37$ (2)°, $V = 1586·(1)$ Å^3, $Z = 2$, $D_m = 1·44$, $D_x = 1·44$ g cm^{-3}, $\lambda(Mo\ K\alpha) = 0·71069$ Å, $\mu = 8·3$ cm^{-1}, $F(000) = 712$, $T = 298$ K, final $R =$

0·066 for 2732 observed reflections. Two crystallographically independent centrosymmetric Ni complexes without any appreciable structural difference are in unit-cell special positions (0,0,0) and ($\frac{1}{2},\frac{1}{2},\frac{1}{2}$). The Ni atom is octahedrally coordinated with two 5-chloro-8-quinolinol ligands in a plane and two pyridine ligands in *trans* positions. Distances and angles are normal [average Ni—N(quinoline) 2·074 (7), Ni—N(pyridine) 2·145 (7); Ni—O 2·045 (5) Å].

Introduction. As a continuation of our investigations on the structure of Ni complexes, the crystal structure of a complex with 5-chloro-8-quinolinol and pyridine has been determined. In previous work, we have reported several crystal structures of Ni^{II} compounds with ethylenediamine and C-substituted ethylenediamines (Garcia-Granda & Gómez-Beltrán, 1984*a,b,c*). The molecular structures of a Cu^{II} and a Zn^{II} bis(8-quinolinolato) compound, square-planar and octahedral respectively, have been reported (Palenik, 1964*a,b*). Nevertheless, as far as we know, no Ni compounds of this kind have so far been studied by X-ray diffraction.

The structures of chelate compounds of this type are of interest for their analytical applications in the solvent extraction of metal ions (Bhatki, Rane & Freiser, 1978).

Experimental. Dark-yellow crystal, 0·42 × 0·13 × 0·20 mm, density measured by flotation, unit-cell parameters and intensity data obtained on an Enraf–Nonius CAD-4F automatic diffractometer, graphite-monochromated Mo $K\alpha$ radiation, $\omega/2\theta$ scans, 0 < θ < 24°, cell dimensions refined by least-squares fitting of values of 25 reflections; no appreciable drop in intensity of three standard reflections, checked every hour; 4940 reflections measured, hkl range from (−18, −12, 0) to (18, 12, 10), only 2732 with $I > 3\sigma(I)$ used in subsequent calculations; measured intensities corrected for Lorentz and polarization effects; empirical absorption correction using *DIFABS* (Walker & Stuart, 1983) applied after isotropic refinement; scattering factors for neutral atoms and anomalous-dispersion corrections for Ni and Cl atoms from *International Tables for X-ray Crystallography* (1974); a statistical test showed a centric intensity distribution. Structure solved using *CRYSTAN* (Burzlaff, Böhme & Gomm, 1977) by Patterson and Fourier techniques. H atoms located by ΔF synthesis. Full-matrix least-squares refinement, on F, using anisotropic thermal parameters for all non-H atoms and fixed isotropic ones for H led to a final $R = 0·066$ and $wR = 0·075$; $w = 1/(a + b|F_o|)^2$ with coefficients $a = 3·2$, $b = −0·12$ if $|F_o| < 16·5$ and $a = 0·64$, $b = 0·04$ if $|F_o| > 16·5$; max. and av. Δ/σ in final cycle 0·3189 and 0·0435 respectively; a final difference synthesis had no electron density > 0·40 e Å^{-3}; least-squares weights calculated with *PESOS* (Martínez-Ripoll & Cano, 1975). Fig. 1 drawn

with *ORTEP* (Johnson, 1965); geometrical parameters calculated with *PARST* (Nardelli, 1983). Atomic parameters are listed in Table 1.*

Discussion. Fig. 1. shows the geometry of the molecule and the atom labelling. Table 2 lists bond lengths and angles.

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* Lists of structure amplitudes, anisotropic thermal parameters, H-atom parameters, bond lengths and angles involving H atoms, hydrogen-bond data, selected torsion angles and the results of least-squares-planes' calculations have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42500 (42 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. *Coordinates and equivalent isotropic thermal parameters* (Å^2 × 10^4)

$$U_{eq} = \tfrac{1}{3}\sum_i\sum_j U_{ij}a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j \cos(a_i,a_j).$$

	x	y	z	U_{eq}
Ni(01)	0·0000	0·0000	0·0000	363 (6)
Ni(11)	0·5000	0·5000	0·5000	389 (6)
Cl(01)	0·0449 (2)	−0·6062 (2)	−0·2057 (3)	768 (13)
Cl(11)	0·1334 (2)	0·7792 (3)	0·6264 (4)	901 (14)
O(01)	0·0827 (3)	−0·1074 (5)	−0·1699 (6)	434 (20)
O(11)	0·4061 (4)	4262 (5)	0·5818 (6)	497 (23)
O(21)*	0·2568 (5)	−0·0772 (7)	−0·2965 (9)	794 (32)
O(31)*	0·3536 (5)	0·1856 (8)	0·5779 (8)	760 (32)
N(01)	0·0681 (4)	−0·1593 (6)	0·0256 (7)	412 (25)
N(02)	0·0701 (5)	0·0536 (6)	−0·1514 (7)	440 (26)
N(11)	0·4252 (5)	0·6530 (7)	0·5124 (8)	508 (29)
N(12)	0·5798 (5)	0·5498 (7)	0·7185 (7)	465 (27)
N(41)†	0·4778 (8)	0·1153 (12)	0·8420 (13)	1146 (59)
C(02)	0·1394 (6)	−0·1835 (8)	0·1200 (10)	539 (34)
C(03)	0·1756 (6)	−0·2981 (9)	0·1260 (12)	622 (39)
C(04)	0·1335 (7)	−0·3910 (9)	0·0200 (12)	608 (41)
C(05)	0·0071 (7)	−0·4569 (8)	−0·1931 (11)	567 (38)
C(06)	0·0670 (7)	−0·4293 (9)	−0·2879 (10)	586 (39)
C(07)	0·0991 (6)	−0·3132 (9)	−0·2837 (10)	573 (37)
C(08)	−0·0548 (5)	−0·2188 (7)	−0·1772 (9)	430 (30)
C(09)	0·0238 (5)	−0·2521 (7)	−0·0807 (9)	405 (30)
C(010)	0·0563 (6)	−0·3693 (7)	−0·0820 (10)	463 (34)
C(011)	0·0312 (6)	0·0768 (9)	−0·2851 (10)	554 (35)
C(012)	0·0735 (7)	0·1163 (9)	−0·3828 (10)	626 (41)
C(013)	0·1633 (8)	0·1246 (9)	−0·3403 (11)	635 (43)
C(014)	0·2072 (7)	0·0997 (10)	−0·2020 (12)	686 (45)
C(015)	0·1594 (6)	0·0632 (9)	−0·1085 (10)	569 (36)
C(12)	0·4360 (7)	0·7622 (9)	0·4781 (12)	629 (42)
C(13)	0·3758 (8)	0·8524 (10)	0·4856 (13)	726 (49)
C(14)	0·3011 (7)	0·8288 (9)	0·5286 (11)	653 (42)
C(15)	0·2128 (6)	0·6731 (10)	0·6114 (10)	612 (40)
C(16)	0·2050 (6)	0·5596 (10)	0·6418 (10)	606 (40)
C(17)	0·2680 (6)	0·4701 (9)	0·6332 (9)	532 (35)
C(18)	0·3449 (5)	0·5031 (8)	0·5895 (9)	469 (31)
C(19)	0·3509 (5)	0·6236 (7)	0·5554 (8)	371 (27)
C(110)	0·2871 (6)	0·7114 (8)	0·5642 (9)	518 (34)
C(111)	0·6399 (7)	0·6402 (9)	0·7447 (10)	662 (41)
C(112)	0·6991 (7)	0·6708 (10)	0·8835 (11)	746 (46)
C(113)	0·6944 (7)	0·6050 (11)	0·9957 (12)	742 (46)
C(114)	0·6310 (8)	0·5152 (11)	0·9693 (12)	809 (50)
C(115)	0·5756 (7)	0·4886 (9)	0·8294 (11)	682 (42)
C(41)†	0·5478 (10)	0·0501 (14)	0·8211 (13)	1030 (66)
C(42)†	0·6300 (9)	0·0654 (15)	0·9026 (16)	1027 (67)
C(43)†	0·6418 (12)	0·1430 (18)	1·0217 (19)	1257 (86)
C(44)†	0·5764 (15)	0·2076 (15)	1·0569 (17)	1214 (87)
C(45)†	0·4959 (12)	0·1887 (15)	0·9657 (20)	1170 (81)

* Water O.
† Atoms of uncoordinated pyridine molecule.

There are two similar complex molecules in the unit cell with Ni atoms in special positions (0,0,0) and ($\frac{1}{2}$,$\frac{1}{2}$,$\frac{1}{2}$). As was expected the Ni environment is pseudo-octahedral, with the pyridine ligands in *trans* positions. Ni—N average distances are 2·145 (7) Å for pyridine, and 2·074 (7) Å for 5-chloro-8-quinolinol, and Ni—O is 2·045 (5) Å; the average N—Ni—O angle is 81·9 (2)°.

Water molecules contribute to the packing through hydrogen bonds.* The N(41)—O(31) distance, 2·92 (1) Å, indicates a pyridine–water interaction, but apparently not involving hydrogen bonding.

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* The geometrical features of the hydrogen bonds have been deposited. See deposition footnote.
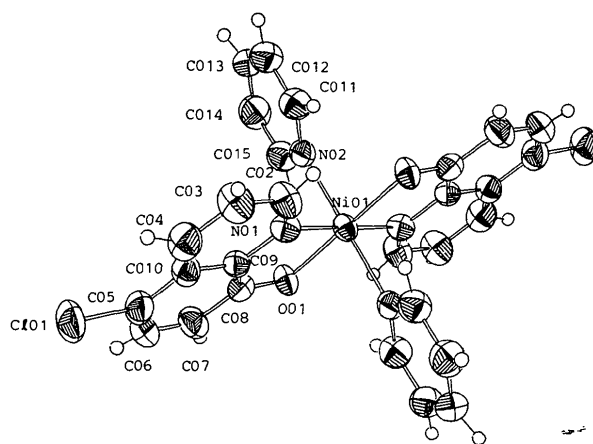

Fig. 1. View of the complex molecule centred at (0,0,0) showing the atom numbering. Atoms of the molecule centred at ($\frac{1}{2}$,$\frac{1}{2}$,$\frac{1}{2}$), are numbered in a similar manner, but with the initial '0' replaced by a '1'.

Large anisotropic thermal parameters for the atoms of the non-coordinated pyridine molecule |max.: U_{11} of C(44) = 0·167 Å²] suggests a possible slight disorder.

Most of the calculations were carried out on the HP-1000/F minicomputer of the Computing Center of the University of Oviedo.

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Table 2. *Bond lengths (Å) and bond angles (°) with e.s.d.'s in parentheses*

Ni(01)—O(01)	2·048 (5)	C(06)—C(07)	1·384 (13)
Ni(01)—N(01)	2·071 (7)	C(07)—C(08)	1·415 (11)
Ni(01)—N(02)	2·152 (8)	C(08)—C(09)	1·409 (10)
Ni(11)—O(11)	2·042 (6)	C(09)—C(010)	1·397 (11)
Ni(11)—N(11)	2·079 (7)	C(011)—C(012)	1·377 (16)
Ni(11)—N(12)	2·139 (6)	C(012)—C(013)	1·363 (15)
Cl(01)—C(05)	1·762 (9)	C(013)—C(014)	1·371 (14)
Cl(11)—C(15)	1·742 (11)	C(014)—C(015)	1·392 (16)
O(01)—C(08)	1·312 (10)	C(12)—C(13)	1·389 (15)
O(11)—C(18)	1·300 (10)	C(13)—C(14)	1·373 (18)
N(01)—C(02)	1·288 (10)	C(14)—C(110)	1·406 (14)
N(01)—C(09)	1·403 (9)	C(15)—C(16)	1·329 (16)
N(02)—C(011)	1·309 (11)	C(15)—C(110)	1·433 (15)
N(02)—C(015)	1·356 (11)	C(16)—C(17)	1·417 (14)
N(11)—C(12)	1·305 (12)	C(17)—C(18)	1·434 (13)
N(11)—C(19)	1·382 (12)	C(18)—C(19)	1·412 (12)
N(12)—C(111)	1·330 (12)	C(19)—C(110)	1·413 (12)
N(12)—C(115)	1·325 (13)	C(111)—C(112)	1·407 (13)
N(41)—C(41)	1·367 (20)	C(112)—C(113)	1·368 (17)
N(41)—C(45)	1·322 (20)	C(113)—C(114)	1·360 (17)
C(02)—C(03)	1·394 (13)	C(114)—C(115)	1·386 (13)
C(03)—C(04)	1·392 (13)	C(41)—C(42)	1·321 (19)
C(04)—C(010)	1·380 (12)	C(42)—C(43)	1·319 (23)
C(05)—C(06)	1·332 (13)	C(43)—C(44)	1·350 (29)
C(05)—C(010)	1·416 (11)	C(44)—C(45)	1·335 (25)

N(01)—Ni(01)—N(02)	91·9 (3)	C(05)—C(010)—C(09)	116·0 (8)
O(01)—Ni(01)—N(02)	88·8 (2)	C(04)—C(010)—C(09)	119·0 (9)
O(01)—Ni(01)—N(01)	82·2 (3)	C(04)—C(010)—C(05)	125·0 (8)
N(11)—Ni(11)—N(12)	91·0 (3)	N(02)—C(011)—C(012)	125·1 (9)
O(11)—Ni(11)—N(12)	90·0 (3)	C(011)—C(012)—C(013)	117·4 (9)
O(11)—Ni(11)—N(11)	81·6 (3)	C(012)—C(013)—C(014)	120 (1)
Ni(01)—O(01)—C(08)	110·7 (5)	C(013)—C(014)—C(015)	119 (1)
Ni(11)—O(11)—C(18)	111·0 (6)	N(02)—C(015)—C(014)	121·0 (9)
Ni(01)—N(01)—C(09)	109·9 (6)	N(11)—C(12)—C(13)	122 (1)
Ni(01)—N(01)—C(02)	131·0 (6)	C(12)—C(13)—C(14)	121 (1)
C(02)—N(01)—C(09)	119·1 (8)	C(13)—C(14)—C(110)	118 (1)
Ni(01)—N(02)—C(015)	119·2 (6)	Cl(11)—C(15)—C(110)	118·0 (8)
Ni(01)—N(02)—C(011)	123·5 (6)	Cl(11)—C(15)—C(16)	121·4 (8)
C(011)—N(02)—C(015)	117·3 (8)	C(16)—C(15)—C(110)	120·6 (9)
Ni(11)—N(11)—C(19)	109·9 (5)	C(15)—C(16)—C(17)	124 (1)
Ni(11)—N(11)—C(12)	130·2 (7)	C(16)—C(17)—C(18)	118·5 (8)
C(12)—N(11)—C(19)	119·7 (8)	O(11)—C(18)—C(17)	121·8 (8)
Ni(11)—N(12)—C(115)	122·2 (7)	C(17)—C(18)—C(19)	116·6 (8)
Ni(11)—N(12)—C(111)	119·9 (6)	O(11)—C(18)—C(19)	121·6 (8)
C(111)—N(12)—C(115)	117·8 (8)	N(11)—C(19)—C(18)	115·5 (7)
C(41)—N(41)—C(45)	113·9 (3)	C(18)—C(19)—C(110)	124·0 (8)
N(01)—C(02)—C(03)	124·1 (9)	N(11)—C(19)—C(110)	120·5 (8)
C(02)—C(03)—C(04)	117·7 (9)	C(15)—C(110)—C(19)	116·6 (8)
C(03)—C(04)—C(010)	119·9 (9)	C(14)—C(110)—C(19)	118·5 (8)
Cl(01)—C(05)—C(010)	119·1 (7)	C(14)—C(110)—C(15)	124·9 (9)
Cl(01)—C(05)—C(06)	119·3 (7)	N(12)—C(111)—C(112)	122·6 (9)
C(06)—C(05)—C(010)	121·6 (9)	C(111)—C(112)—C(113)	119 (1)
C(05)—C(06)—C(07)	121·6 (9)	C(112)—C(113)—C(114)	119 (1)
C(06)—C(07)—C(08)	121·3 (9)	C(113)—C(114)—C(115)	120 (1)
O(01)—C(08)—C(07)	123·2 (8)	N(12)—C(115)—C(114)	123 (1)
C(07)—C(08)—C(09)	115·0 (8)	N(41)—C(41)—C(42)	126 (1)
O(01)—C(08)—C(09)	121·8 (8)	C(41)—C(42)—C(43)	115 (2)
N(01)—C(09)—C(08)	115·4 (8)	C(42)—C(43)—C(44)	123 (2)
C(08)—C(09)—C(010)	124·4 (8)	C(43)—C(44)—C(45)	117 (2)
N(01)—C(09)—C(010)	120·1 (8)	N(41)—C(45)—C(44)	124 (2)

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