

Structure of Bis(*cis*-1,2-cyclohexanediamine)nickel(II) Dichloride

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Abstract. $[\text{Ni}(\text{C}_6\text{H}_{14}\text{N}_2)_2]\text{Cl}_2$, $M_r = 358.00$, monoclinic, $P2_1/n$, $a = 5.9240$ (5), $b = 20.864$ (3), $c = 6.5416$ (4) Å, $\beta = 101.697$ (6)°, $V = 791.8$ (2) Å³, $Z = 2$, $D_x = 1.50$ Mg m⁻³. Mo $K\alpha$ radiation (graphite crystal monochromator, $\lambda = 0.71073$ Å), $\mu(\text{Mo } K\alpha) = 1.565$ mm⁻¹, $F(000) = 380$, $T = 293$ K. Final conventional R factor = 0.027 for 1652 'observed' reflections and 144 variables. The Ni atom has a square-planar coordination involving two *cis*-1,2-cyclohexanediamine ligands, with an average distance Ni—N of 1.912 (2) Å and an N(1)—Ni—N(2) bite angle of 86.6 (1)°.

Experimental. Yellow crystal, $0.43 \times 0.2 \times 0.1$ mm. Mo $K\alpha$ radiation, graphite-crystal monochromator, Nonius CAD-4 single-crystal diffractometer. Unit-cell dimensions determined from the angular settings of 25 reflections with θ between 20 and 30°. Space group determined to be $P2_1/n$ from the systematic absences. 5220 reflections measured, hkl range $-8, -29, 0$ to $8, 29, 9, 0 < \theta < 30^\circ$. $\omega-2\theta$ scan technique with a variable scan rate and maximum scan time of 60 s per reflection. Intensity checked by monitoring three standard reflections every 60 min. Final drift correction factors between 0.97 and 1.02. Profile analysis performed on all reflections (Lehmann & Larsen, 1974; Grant & Gabe, 1978); empirical absorption correction was applied, using ψ scans (North, Phillips & Mathews, 1968), correction-factor range 0.94 to 1.00. Symmetry-equivalent reflections averaged, $R_{\text{int}} = \sum(I - \langle I \rangle) / \sum I = 0.052$, 2317 unique reflections and 1652 observed with $I > 3\sigma(I)$. Lorentz and polarization corrections applied and data reduced to $|F_o|$ values. Structure solved by Patterson method using the program *SHELXS86* (Sheldrick, 1985) and Fourier synthesis. Isotropic least-squares refinement, using *SHELX* (Sheldrick, 1976), converged to $R = 0.084$. Empirical absorption correction using *DIFABS* (Walker & Stuart, 1983). Maximum and minimum absorption correction factors 1.10 and 0.86, respectively. Further anisotropic refinements followed by a difference Fourier synthesis allowed the location of all the H atoms.

During the final stages of the refinement the positional parameters and the anisotropic thermal

parameters of the non-H atoms were refined. All H atoms refined isotropically. The final conventional agreement factors were $R = 0.027$, $wR = 0.029$ and $S = 1.09$, for the 1652 'observed' reflections and 144 variables. Function minimized $\sum w(F_o - F_c)^2$, $w = 1/[\sigma^2(F_o) + 0.0060F_o^2]$ with $\sigma(F_o)$ from counting statistics. Maximum shift/e.s.d. in the last full-matrix least-squares cycle less than 0.03. Final difference Fourier map showed no peaks higher than $0.37 \text{ e } \text{Å}^{-3}$ nor deeper than $-0.54 \text{ e } \text{Å}^{-3}$. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV). The plot was made with program *PLUTO* (Motherwell & Clegg, 1978). Geometrical calculations were made with *PARST* (Nardelli, 1983). Final positional and thermal parameters are given in Table 1.* Molecular geometry data are collected in Table 2. Fig. 1 shows the Ni coordination and the atomic numbering scheme. A selection of angles between least-squares planes and main torsion angles have been deposited. All calculations were made on an IBM 4341 computer in the Computer Center of the University of Oviedo.

Related literature. This structural investigation was undertaken as part of our work on nickel(II) compounds with C-substituted ethylenediamine derivatives as ligands. The title compound is isostructural with the analogous bromide previously reported (García-Granda, Díaz & Gómez Beltrán, 1990). Structural parameters are very similar for both complexes, showing the same tendency for small differences in non-equivalent Ni—N distances {1.910 (2) [N(1)] versus 1.914 (2) Å [N(2)] in the present compound}. The shortest intramolecular contacts involving H atoms are four connecting Cl(1) to atoms H(1) and H(2) of N(1) and H(3) and H(4) of N(2). Two of these contacts are intramolecular,

* Lists of structure factors, anisotropic thermal parameters, H-atom parameters, distances and angles involving H atoms, H-bond distances and angles, least-squares planes data and principal torsion angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53332 (17 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. Fractional positional and thermal parameters, with e.s.d.'s in parentheses

$$U_{eq} = (1/3) \sum_i \sum_j U_{ij} a_i^* a_j^* a_i \cdot a_j \cdot$$

	x	y	z	$U_{eq} (\times 10^3)$
Cl(1)	0.31055 (8)	0.07025 (2)	-0.44825 (8)	3.45 (1)
Ni(1)	0.00000 (0)	0.00000 (0)	0.00000 (0)	2.16 (1)
N(1)	0.1290 (3)	-0.06040 (7)	0.2110 (3)	2.59 (4)
N(2)	0.1424 (3)	-0.04934 (7)	-0.1857 (3)	2.55 (4)
C(1)	0.1868 (3)	-0.12054 (8)	0.1105 (3)	2.53 (5)
C(2)	0.2939 (3)	-0.10046 (9)	-0.0718 (3)	2.57 (5)
C(3)	0.3281 (3)	-0.1570 (1)	-0.2110 (4)	3.30 (6)
C(4)	0.1115 (4)	-0.19701 (9)	-0.2761 (4)	3.40 (6)
C(5)	0.0153 (4)	-0.21815 (9)	-0.0881 (4)	3.62 (6)
C(6)	-0.0309 (3)	-0.1602 (1)	0.0400 (3)	3.11 (6)

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

Ni(1)—N(1)	1.910 (2)	C(1)—C(6)	1.523 (3)
Ni(1)—N(2)	1.914 (2)	C(2)—C(3)	1.529 (3)
N(1)—C(1)	1.488 (2)	C(3)—C(4)	1.518 (3)
N(2)—C(2)	1.492 (2)	C(4)—C(5)	1.522 (3)
C(1)—C(2)	1.518 (2)	C(5)—C(6)	1.527 (3)
N(1)—Ni(1)—N(2)	86.6 (1)	N(2)—C(2)—C(3)	113.1 (2)
Ni(1)—N(1)—C(1)	109.3 (1)	C(1)—C(2)—C(3)	112.4 (2)
Ni(1)—N(2)—C(2)	111.4 (1)	C(2)—C(3)—C(4)	112.6 (1)
N(1)—C(1)—C(2)	106.5 (1)	C(3)—C(4)—C(5)	111.5 (2)
N(1)—C(1)—C(6)	109.6 (2)	C(4)—C(5)—C(6)	110.6 (2)
C(2)—C(1)—C(6)	112.0 (2)	C(1)—C(6)—C(5)	111.2 (2)
N(2)—C(2)—C(1)	106.5 (1)		

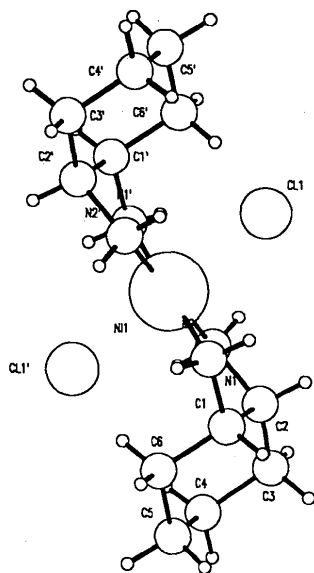


Fig. 1. PLUTO (Motherwell & Clegg, 1976) drawing showing the coordination of the central Ni atom and the atomic numbering.

Cl(1)···H(1) and Cl(1)···H(4), and the other two intermolecular. The analogous bromide compound, cited above, shows only two, one intramolecular and one intermolecular, contacts in the same range of distances.

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Natrium-Bis-(15-Krone-5)-Tetrachlorooxonibat-Acetonitril

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Abstract. Na(O₅C₁₀H₂₀)₂[NbOCl₄(CH₃CN)], $M_r = 755.3$, monoclinic, $P2_1/c$, $a = 9.053$ (2), $b = 12.046$ (1), $c = 31.383$ (2) Å, $\beta = 92.25$ (2)°, $V = 3419.6$ (6) Å³, $Z = 4$, $D_x = 1.47$ g cm⁻³, $\lambda(\text{Mo } K\alpha) = 0.7107$ Å, $\mu = 6.48$ cm⁻¹, $F(000) = 1560$, $T = 292$ K, $R = 0.076$ for 4640 unique observed reflexions. The compound consists of [NbOCl₄(CH₃CN)]⁻ ions having the acetonitrile ligand in a *trans* position to the rather short Nb=O bond [1.682 (7) Å], and of

Na⁺ ions that are coordinated to two crown ether molecules, one is linked *via* five O atoms, the other only through two, thus resulting in a seven-coordinate Na⁺ ion. The latter crown ether exhibits large thermal motion and is possibly somewhat disordered.

Experimentelles. Im Rahmen von Untersuchungen der Reaktionen von Metallhalogeniden mit Poly-