adduct is also *cis*-coordinated. The Ni—N distances are 2·046 and 2·055 Å and the S—Ni—S angles in the same dibutyldithiophosphate ligand are 79·44 and 80·47°. These are comparable with those found in Ni[$(C_2H_5O)_2PS_2$].2py (Ooi & Fernando, 1967) and Ni[$(C_4H_9O)_2PS_2$]$_2$.2py (Liu, Lin, Xu, Yu & You, 1987).

The P atoms in the dibutyldithiophosphate ligand have approximate sp^3 hybridization. The N—C and C—C distances and bond angles in imidazole are normal. Thus, this ligand interacts weakly with the metal ion.

Supported by the National Natural Science Foundation of China as an Item of Major Project.

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Acta Cryst. (1990). C**46**, 993–995

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

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(*Received* 17 *February* 1989; *accepted* 22 *August* 1989)

Abstract. [$Ni(C_6H_{14}N_2)_2$]Br_2, M_r = 478·90, monoclinic, $P2_1/n$, a = 6·1610 (2), b = 20·6379 (15), c = 6·7270 (7) Å, β = 101·329 (5)°, V = 838·7 (1) Å^3, Z = 2, D_x = 1·90 Mg m^{-3}, Mo $K\alpha$ radiation (graphite crystal monochromator, λ = 0·71073 Å), μ = 5·891 mm^{-1}, $F(000)$ = 484, T = 293 K. Final conventional R = 0·022 for 1577 observed reflections and 144 variables. The Ni atom has a square planar coordination given by two *cis*-1,2-cyclohexanediamine ligands. Distances and angles are normal. Distances Ni—N are both equal, within the experimental error, the average being 1·914 (2) Å; the N(1)—Ni—N(2) bite angle is 86·4 (1)°.

Introduction. This structural investigation was undertaken as a part of our work on nickel(II) compounds with *C*-substituted ethylenediamine ligands. In previous communications we have reported crystal and molecular structures of several octahedral and square planar complexes of this family including compounds with tetramethylethylenediamine (Alcalá-Aranda, Fernández, Gómez-Beltrán & Larena, 1977), *trans*-1,2-cyclohexanediamine (Valero-Capilla, Alcalá-Aranda & Gómez-Beltrán, 1980), 2-methyl-1,2-propanediamine (García-Granda & Gómez-Beltrán, 1984*a*), 1,2-ethanediamine (García-Granda & Gómez-Beltrán, 1984*b*), *meso*-1,2-diphenyl-1,2-ethanediamine (García-Granda & Gómez-Beltrán, 1984*c*), 2-methyl-1,2-propanediamine (García-Granda & Gómez-Beltrán, 1986). The structure of the partially oxidized compound, *catena*-(μ-bromo)bis[(1*R*,2*R*)cyclohexanediamine]-nickel 2·77-bromide, a bromide containing the *trans* form of cyclohexanediamine, has been reported (Toftlund & Simonsen, 1984). This paper describes the crystal and molecular structure of a new complex within this series.

Experimental. Yellow crystal, size 0·26 × 0·13 × 0·13 mm. Throughout the experiment Mo $K\alpha$ radiation was used with a graphite crystal monochromator on a Nonius CAD-4 single-crystal diffractometer (λ = 0·71073 Å). The unit-cell dimensions were determined from the angular settings of 25 reflections with θ between 20 and 30°. The space group was determined to be $P2_1/n$ from the systematic absences. The intensity data of 5663 reflections, in *hkl* range (−8, −29, 0) to (8, 29, 9), and θ limits ($0 < \theta < 30°$) were measured, using the ω–2θ scan technique and a variable scan rate with a maximum scan time of 60 s per reflection. The intensity of the primary beam was checked throughout the data

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

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

	x	y	z	U_{eq} (Å^2 × 10^2)
Br(1)	0·81239 (4)	0·07516 (1)	1·54585 (4)	3·57 (1)
Ni(1)	0·50000 (0)	0·00000 (0)	1·00000 (0)	2·39 (1)
N(1)	0·3773 (4)	−0·05987 (10)	0·7916 (3)	2·86 (6)
N(2)	0·3589 (4)	−0·05087 (11)	1·1762 (3)	2·86 (6)
C(1)	0·3186 (4)	−0·12126 (11)	0·8850 (4)	2·73 (7)
C(2)	0·2103 (4)	−0·10178 (12)	1·0604 (4)	2·97 (7)
C(3)	0·1735 (5)	−0·15970 (14)	1·1908 (5)	3·98 (9)
C(4)	0·3796 (5)	−0·20134 (13)	1·2533 (5)	3·94 (9)
C(5)	0·4738 (6)	−0·22125 (14)	1·0708 (5)	4·30 (10)
C(6)	0·5248 (4)	−0·16168 (13)	0·9530 (5)	3·48 (8)

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

Ni(1)—N(1)	1·910 (2)	C(1)—C(6)	1·514 (3)
Ni(1)—N(2)	1·915 (2)	C(2)—C(3)	1·526 (4)
N(1)—C(1)	1·490 (3)	C(3)—C(4)	1·522 (4)
N(2)—C(2)	1·506 (3)	C(4)—C(5)	1·515 (5)
C(1)—C(2)	1·519 (3)	C(5)—C(6)	1·528 (4)
N(1)—Ni(1)—N(2)	86·4 (1)	N(2)—C(2)—C(3)	113·2 (2)
Ni(1)—N(1)—C(1)	109·6 (2)	C(1)—C(2)—C(3)	112·1 (2)
Ni(1)—N(2)—C(2)	111·5 (2)	C(2)—C(3)—C(4)	113·0 (2)
N(1)—C(1)—C(2)	106·4 (2)	C(3)—C(4)—C(5)	111·1 (3)
N(1)—C(1)—C(6)	109·7 (2)	C(4)—C(5)—C(6)	110·6 (2)
C(2)—C(1)—C(6)	112·4 (2)	C(1)—C(6)—C(5)	110·7 (2)
N(2)—C(2)—C(1)	105·8 (2)		

collection by monitoring three standard reflections every 60 minutes. The final drift correction factors were between 1·00 and 1·04. On all reflections profile analysis was performed (Lehmann & Larsen, 1974; Grant & Gabe, 1978); empirical absorption correction was applied, using ψ scans (North, Phillips & Mathews, 1968), μ(Mo $K\alpha$) = 5·891 mm^{-1} (correction factors were in the range 0·79 to 1·00). Symmetry equivalent reflections were averaged, $R_{int} = \sum(I - \langle I \rangle)/\sum I$ = 0·044, resulting in 2448 unique reflections of which only 1577 were observed with $I > 3\sigma(I)$. Lorentz and polarization corrections were applied and the data were reduced to $|F_o|$ values. The structure was solved by Patterson interpretation using the program *SHELXS*86 (Sheldrick, 1985) and Fourier synthesis. Isotropic least-squares refinement, using *SHELX*76 (Sheldrick, 1976), converged to R = 0·069. At this stage additional empirical absorption correction was applied (Walker & Stuart, 1983), resulting in a further decrease of R to 0·059. The maximum and minimum absorption correction factors were, respectively, 1·22 and 0·77. 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 hydrogens were refined isotropically. The final conventional agreement factors were R = 0·022 and wR = 0·023 for the 1577 observed reflections and 144 variables. The function minimized was $\sum w(F_o - F_c)^2$, $w = 1/[\sigma^2(F_o) + 0{\cdot}00020\,F_o^2]$ with $\sigma(F_o)$ from counting statistics. The maximum shift to e.s.d. ratio in the last full-matrix least-squares cycle was less than 0·04. The final difference Fourier map showed no peaks higher than 0·36 e Å^{-3} or deeper than −0·43 e Å^{-3}. Atomic scattering factors were taken from *International Tables for X-ray Crystallography* (1974). The plot was made with *PLUTO* (Motherwell, 1976). Geometrical calculations were made with *PARST* (Nardelli, 1983).

Discussion. Final positional and thermal parameters are given in Table 1.* Molecular geometry data are collected in Table 2. Fig. 1 shows the nickel coordination and the atomic numbering scheme. The central nickel is square planar coordinated by two *cis*-1,2-cyclohexanediamine ligands. These complex molecules are centrosymmetric, the central nickel lying on a crystallographic symmetry center. The average Ni—N distance, 1·914 (2) Å, is the typical Ni—N distance found in square planar coordination and the N—Ni—N bite angle is 86·4 (1)°, the same as the value found for bis(*meso*-1,2-diphenyl-1,2-ethanediamine)nickel(II) dichloride dihydrate, (García-Granda & Gómez-Beltrán, 1984*c*), for which the Ni—N average distance was found to be 1·911 (4) Å. The two cyclohexane rings show a chair conformation. The shortest intramolecular contacts

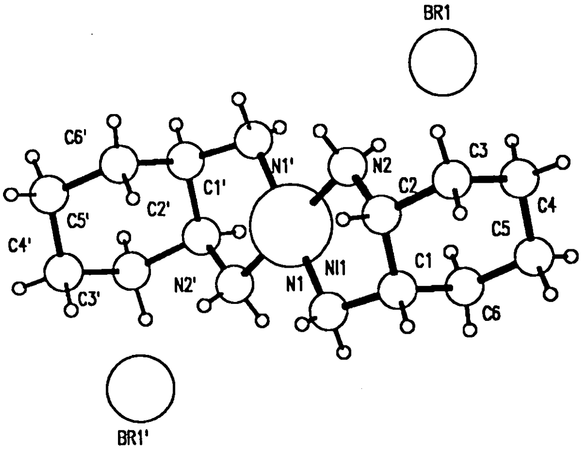

Fig. 1. A *PLUTO* (Motherwell, 1976) diagram, showing the coordination of central nickel and the atomic numbering.

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* Lists of structure amplitudes, anisotropic thermal parameters, H-atom parameters, distances and angles involving H atoms, hydrogen-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 52541 (18 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

involving H atoms are two connecting Br(1) to N(1) and N(2) respectively. A selection of angles between least-squares planes and main torsion angles has been deposited.

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Acta Cryst. (1990). C**46**, 995–998

Structure of 3-(η^6-Hexamethylbenzene)-1,2-dicarba-3-ruthena-*closo*-dodecaborane(11)

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(*Received* 20 *March* 1989; *accepted* 23 *August* 1989)

Abstract. $C_{14}H_{29}B_9Ru$, $M_r = 395·75$, orthorhombic, $Cmc2_1$, $a = 12·994\ (2)$, $b = 10·365\ (2)$, $c = 13·872\ (2)$ Å, $V = 1868·2\ (6)$ Å^3, $Z = 4$, $D_x = 1·41$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, μ = 7·40 cm^{-1}, $F(000) = 808$, room temperature, $R = 0·0177$, 890 unique reflections with $F > 3·0\sigma(F)$. The structure is based on a 12-vertex RuC_2B_9 icosahedron with the Ru and two C atoms occupying a common deltahedral face. The cluster displays true *closo* geometry and no significant slippage is evident.

Introduction. We have determined the crystal and molecular structure of the closed 12-vertex metalladicarbaborane [3-(η^6-C_6Me_6)-*closo*-3,1,2-$RuC_2B_9H_{11}$] as part of a series to compare and examine for systematic changes in the {Ru(η^6-C_6Me_6)} moiety in a number of polyhedral boron-containing compounds as the nature of the boron-containing fragment varies. The previously reported polyhedral boron-containing structures involving the Ru(η^6-C_6Me_6) centre are those compounds summarized in Table 3. The crystallographically determined molecular structure of the closely related compound [3-(η^6-C_6H_6)-3,1,2-$RuC_2B_9H_{11}$] has previously been reported (Garcia, Green, Stone, Sommerville, Welch, Briant, Cox & Mingos, 1985), but more data are available for comparing the Ru(η^6-C_6Me_6) centres among polyhedral metallaborane structures.

Experimental. A sample of [3-(η^6-C_6Me_6)-3,1,2-$RuC_2B_9H_{11}$] was prepared from [(η^6-C_6Me_6)$RuCl_2$]$_2$,

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