

**Structure of Two Isomers of Bromo[(2-bromo-3,4,5,6-tetrafluorophenyl)diphenylphosphine-*Br,P*]chloro(2-diphenylphosphinoyl-3,4,5,6-tetrafluorophenyl-*O*)-rhodium(III),  $C_{36}H_{20}Br_2ClF_8OP_2Rh$  and  $C_{36}H_{20}Br_2ClF_8OP_2Rh.C_2H_6O$**

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(Received 16 July 1984; accepted 1 January 1985)

**Abstract.** (I): *trans*-[RhBrCl{(o-BrC<sub>6</sub>F<sub>4</sub>)PPh<sub>2</sub>}{(o-C<sub>6</sub>F<sub>4</sub>)OPPh<sub>2</sub>}],  $M_r = 980.67$ , hexagonal,  $P6_1$ ,  $a = 19.161$  (4),  $c = 21.054$  (6) Å,  $V = 6694$  (5) Å<sup>3</sup>,  $Z = 6$ ,  $D_x = 1.46$  Mg m<sup>-3</sup>,  $\lambda(\text{Mo } K\alpha) = 0.71069$  Å,  $\mu = 2.463$  mm<sup>-1</sup>,  $F(000) = 2868$ , room temperature. (II): *cis*-[RhBrCl{(o-BrC<sub>6</sub>F<sub>4</sub>)PPh<sub>2</sub>}{(o-C<sub>6</sub>F<sub>4</sub>)OPPh<sub>2</sub>}]·C<sub>2</sub>H<sub>6</sub>O,  $M_r = 1026.74$ , triclinic,  $P\bar{1}$ ,  $a = 17.548$  (4),  $b = 11.721$  (8),  $c = 10.440$  (7) Å,  $\alpha = 113.75$  (4),  $\beta = 92.31$  (4),  $\gamma = 95.75$  (5)°,  $V = 1948$  (3) Å<sup>3</sup>,  $Z = 2$ ,  $D_x = 1.75$  Mg m<sup>-3</sup>,  $\lambda(\text{Mo } K\alpha) = 0.71069$  Å,  $\mu = 2.826$  mm<sup>-1</sup>,  $F(000) = 1008$ , room temperature. Final  $R = 0.052$  and  $0.040$  for 1399 and 2495 observed reflections respectively. The Rh<sup>3+</sup> ions display a distorted octahedral coordination, where Cl<sup>-</sup> and Br<sup>-</sup> occupy a *trans* position in structure (I) and a *cis* position in (II). A halocarbon binding is shown in both structures with Br–Rh bond distances [average 2.63 (2) Å] longer than those obtained in the [Ir(1,5-cyclooctadiene){(o-BrC<sub>6</sub>H<sub>4</sub>)PPh<sub>2</sub>}]<sup>3+</sup> ion. The steric hindrance between the two ligands determines the distortions in coordination polyhedra and five-membered rings.

**Introduction.** The reaction of RhCl<sub>3</sub> with (o-BrC<sub>6</sub>F<sub>4</sub>)PPh<sub>2</sub> in ethanol medium and recrystallization by diffusion of ethanol into a solution of reaction products in dichloromethane led to the formation of several compounds, according to NMR and X-ray powder analyses. The first two identified compounds are now reported. The remaining compounds are under investigation.

**Experimental.** (I): yellow prism, 0.1 × 0.1 × 0.2 mm, Philips PW 1100 four-circle diffractometer, graphite-monochromatized Mo  $K\alpha$  radiation, cell parameters from 25 reflections ( $4 \leq \theta \leq 8^\circ$ ),  $\omega$ -scan technique, scan width 1°, scan speed 0.03°s<sup>-1</sup>. Three reflections every two hours as intensity control, significant differences not observed. 1501 independent reflections ( $2 \leq \theta \leq 23^\circ$ ;  $hkl$ : -15 to 0, 0 to 20, 0 to 15), 1399 with  $I \geq 2.5\sigma(I)$ . Lorentz-polarization and absorption corrections (SHELX76, Sheldrick, 1976), max. and min. transmission factors 0.75 and 0.73 respectively.

(II): yellow-orange prism, 0.1 × 0.1 × 0.2 mm, Enraf-Nonius CAD-4 diffractometer, graphite-monochromatized Mo  $K\alpha$  radiation, cell parameters from 20 reflections ( $5 \leq \theta \leq 12^\circ$ ),  $\theta/2\theta$  scan technique, scan speed variable, according to the net count, one reflection every hour as intensity control, significant variations not observed. 6808 reflections ( $2 \leq \theta \leq 28^\circ$ ;  $hkl \pm 18, \pm 13, 12$ ), 2495 with  $I \geq 2.5\sigma(I)$ . Lorentz-polarization and absorption corrections (SHELX76), max. and min. transmission factors 0.75 and 0.73 respectively.

Both structures solved by direct methods (MULTAN80, Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980) (29 and seven weighted atoms respectively). Remaining non-hydrogen atoms from DIRDIF (Beurskens, Bosman, Doesburg, Gould, Van den Hark, Prick, Noordik, Beurskens & Parthasarathi, 1981). Hydrogen atoms not located. Full-matrix least-squares refinement, phenyl rings with constrained geometry (planar, bond distance =

1.395 Å, bond angle = 120°,  $w|F_o| - |F_c|^2$  minimized,  $w = [\sigma(F_o) + k|F_o|^2]^{-1}$ ,  $k = 0.0057$  for (I) and 0.0029 for (II),  $f, f'$  and  $f''$  from *International Tables for X-ray Crystallography* (1974). Both configurations refined in structure (I). Max. and min. peaks in final  $\Delta F$  map 0.2 e Å<sup>-3</sup> to 0.65 Å of C(22) and 0.4 e Å<sup>-3</sup> to 1.19 Å of C(4), and -0.2 and -0.3 e Å<sup>-3</sup> respectively, max. shift/e.s.d. = -2.5 for  $U_{22}$  of C(21) and 1.5 for  $U_{22}$  of C(64) respectively, final  $R(wR) = 0.052(0.050)$  and 0.040(0.044).

**Discussion.** Final atomic parameters are in Table 1, selected bond distances and angles in Table 2.\*

The structures consist of discrete molecules linked by van der Waals forces. The shortest intermolecular distances are F...F contacts [the weighted average value between distances less than 3.3 Å is 3.0 (2) Å in both structures; ranges: 2.74 (2)–3.33 (2) Å in (I) and 2.79 (1)–3.22 (1) Å in (II)].

The Rh<sup>3+</sup> ion displays a distorted octahedral coordination as shown in Figs. 1 and 2. The Br<sup>-</sup> and Cl<sup>-</sup> ions are in a *trans* position in structure (I), and a *cis* position in (II). The *o*-(BrC<sub>6</sub>F<sub>4</sub>)PPh<sub>2</sub> ligand shows a halocarbon binding in both structures which was also observed in the [Ir(cod){*o*-(BrC<sub>6</sub>H<sub>4</sub>)PPh<sub>2</sub>}]<sup>3+</sup> ion (Burk, Crabtree & Holt, 1984). The *o*-C<sub>6</sub>F<sub>4</sub>OPPh<sub>2</sub> and *o*-BrC<sub>6</sub>F<sub>4</sub>PPh<sub>2</sub> ligands show a cyclometallation of five members, the metal being coordinated to O and *o*-C and Br and P atoms, respectively.

The Rh–Br(22) bond distance [weighted average value: 2.63 (2) Å] is longer than the Rh–Br<sup>-</sup> [2.43 (1) Å] and the Ir–Br [2.473 (4) Å] bond distance observed in the previously mentioned Ir complex (Burk *et al.*, 1984). The Rh–C, Rh–O, Rh–P, P–C, P–O and C–Br bond distances are within three times the e.s.d. in the range of values observed in the literature (Barceló, Besteiro, Lahuerta, Foces-Foces, Cano & Martínez-Ripoll, 1984; *International Tables for X-ray Crystallography*, 1968; Burk *et al.*, 1984).

The unusual intramolecular contacts of F(53) produce the molecular distortions; thus, in structure (I), these shortest contacts are F(53)...P(1) [3.18 (1) Å] and F(53)–C(11) [2.82 (3) Å], which produce the increase in the P(1)–Rh–C(52) [105.1 (4)°] and P(1)–Rh–Br(1) [95.0 (2)°] bond angles and Rh–Br(22) bond distance, and the decrease in the Cl–Rh–P(1) [86.8 (2)°] and C(22)–Br(22)–Rh [95.0 (2)°; 102.2 (9)° in the Ir complex] bond angles. The two five-membered rings have a skew-envelope form, with Rh [0.68 (1) Å] and O [0.21 (2) Å] atoms out of the plane defined by the remaining four atoms

Table 1. Atomic coordinates ( $\times 10^4$ ) and equivalent isotropic temperature coefficients

$$B_{eq} = \frac{2}{3}\pi^2 U_{ij} a_i a_j a_k^*$$

Structure (I)	x	y	z	B <sub>eq</sub> (Å <sup>2</sup> )
Rh	4805 (1)	1696 (1)	4208	5.0 (1)
Br(22)	4926 (2)	2893 (2)	3474 (2)	6.9 (2)
Br(1)	6176 (2)	2529 (2)	4574 (2)	9.4 (2)
Cl	3411 (3)	874 (3)	3869 (3)	3.3 (3)
P(1)	5155 (4)	1354 (3)	3268 (3)	4.5 (3)
P(2)	3903 (5)	1446 (5)	5470 (4)	5.7 (4)
C(11)	4924 (12)	326 (8)	3148 (8)	5 (2)
C(12)	5413 (12)	170 (8)	2751 (8)	5 (2)
C(13)	5194 (12)	-624 (8)	2603 (8)	9 (2)
C(14)	4487 (12)	-1260 (8)	2852 (8)	7 (2)
C(15)	3999 (12)	-1104 (8)	3250 (8)	5 (2)
C(16)	4218 (12)	-311 (8)	3397 (8)	7 (2)
C(21)	4672 (9)	1628 (11)	2736 (9)	23 (3)
C(22)	4656 (9)	2345 (11)	2796 (9)	12 (3)
C(23)	4312 (9)	2581 (11)	2319 (9)	15 (3)
C(24)	3983 (9)	2100 (11)	1781 (9)	8 (2)
C(25)	3999 (9)	1383 (11)	1721 (9)	8 (3)
C(26)	4344 (9)	1147 (11)	2198 (9)	9 (3)
F(23)	4220 (10)	3205 (10)	2418 (9)	9 (2)
F(24)	3674 (14)	2406 (12)	1310 (9)	11 (3)
F(25)	3660 (10)	1017 (13)	1119 (9)	9 (2)
F(26)	4332 (10)	529 (10)	1937 (8)	8 (2)
C(31)	6215 (7)	1994 (10)	3022 (10)	8 (2)
C(32)	6457 (7)	2568 (10)	2539 (10)	10 (2)
C(33)	7272 (7)	3044 (10)	2390 (10)	7 (2)
C(34)	7846 (7)	2947 (10)	2725 (10)	11 (2)
C(35)	7605 (7)	2374 (10)	3208 (10)	11 (3)
C(36)	6789 (7)	1897 (10)	3356 (10)	14 (3)
C(41)	4187 (13)	1778 (13)	6270 (8)	7 (2)
C(42)	3644 (13)	1574 (13)	6771 (8)	12 (3)
C(43)	3906 (13)	1958 (13)	7357 (8)	12 (3)
C(44)	4713 (13)	2546 (13)	7442 (8)	17 (3)
C(45)	5256 (13)	2750 (13)	6940 (8)	24 (4)
C(46)	4994 (13)	2366 (13)	6354 (8)	16 (3)
C(51)	4210 (9)	724 (10)	5369 (9)	4 (2)
C(52)	4716 (9)	840 (10)	4855 (9)	4 (2)
C(53)	5100 (9)	386 (10)	4805 (9)	4 (2)
C(54)	4977 (9)	-183 (10)	5270 (9)	7 (2)
C(55)	4471 (9)	-299 (10)	5783 (9)	6 (2)
C(56)	4087 (9)	154 (10)	5833 (9)	7 (2)
F(53)	5638 (10)	523 (7)	4357 (8)	6 (2)
F(54)	5403 (11)	-586 (9)	5228 (8)	8 (2)
F(55)	5658 (9)	885 (9)	1214 (8)	7 (2)
F(56)	3632 (10)	-1 (9)	6351 (7)	7 (2)
C(61)	2858 (9)	1068 (15)	5465 (11)	9 (2)
C(62)	2533 (9)	1576 (15)	5439 (11)	12 (3)
C(63)	1699 (9)	1251 (15)	5433 (11)	14 (3)
C(64)	1190 (9)	418 (15)	5451 (11)	14 (3)
C(65)	1515 (9)	-91 (15)	5477 (11)	15 (3)
C(66)	2349 (9)	234 (15)	5484 (11)	11 (3)
O	4428 (15)	2010 (10)	4992 (10)	12 (3)

Structure (II)	x	y	z	B <sub>eq</sub> (Å <sup>2</sup> )
Rh	2315 (1)	997 (1)	1413 (1)	2.41 (6)
Br(22)	2123 (1)	-1364 (1)	-359 (1)	3.9 (1)
Br(1)	3119 (2)	1497 (3)	9812 (3)	9.1 (2)
P(1)	1500 (2)	393 (3)	2745 (3)	2.3 (2)
Cl	1189 (1)	1145 (3)	167 (3)	2.1 (2)
C(11)	861 (4)	1503 (7)	3558 (9)	3.2 (8)
C(12)	111 (4)	1389 (7)	2984 (9)	3.0 (8)
C(13)	-356 (4)	2319 (7)	3636 (9)	5 (1)
C(14)	-72 (4)	3363 (7)	4860 (9)	6 (1)
C(15)	679 (4)	3476 (7)	5434 (9)	5 (1)
C(16)	1145 (4)	2547 (7)	4782 (9)	4 (1)
C(21)	943 (5)	-1123 (6)	1525 (8)	2.1 (8)
C(22)	1197 (5)	-1834 (6)	226 (8)	3.1 (8)
C(23)	792 (5)	-3001 (6)	-635 (8)	3.9 (9)
C(24)	133 (5)	-3455 (6)	-197 (8)	4 (1)
C(25)	-122 (5)	-2743 (6)	1102 (8)	5 (1)
C(26)	283 (5)	-1577 (6)	1963 (8)	4 (1)
F(23)	1070 (5)	-3703 (7)	-1834 (9)	5.0 (6)
F(24)	-259 (5)	-4568 (7)	-1063 (9)	5.3 (6)
F(25)	-753 (5)	-3173 (9)	1526 (12)	6.1 (7)
F(26)	49 (5)	-1042 (8)	3225 (9)	5.3 (6)
C(31)	1912 (5)	-67 (8)	4060 (8)	3 (1)
C(32)	2451 (5)	-915 (8)	3636 (8)	4 (1)
C(33)	2752 (5)	-1375 (8)	4564 (8)	5 (1)
C(34)	2514 (5)	-986 (8)	5918 (8)	4 (1)
C(35)	1975 (5)	-139 (8)	6343 (8)	5 (1)
C(36)	1674 (5)	321 (8)	5414 (8)	3 (1)

\* Lists of structure factors, anisotropic thermal parameters and a complete list of bond distances and angles have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42057 (24 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1 (cont.)

	x	y	z	$B_{eq}(\text{Å}^2)$
P(2)	3857 (2)	1927 (3)	3193 (3)	2.8 (1)
C(41)	4661 (4)	2183 (7)	2272 (8)	3.3 (6)
C(42)	4805 (4)	3283 (7)	2060 (8)	4.6 (8)
C(43)	5438 (4)	3459 (7)	1363 (8)	5.3 (8)
C(44)	5926 (4)	2534 (7)	876 (8)	6.4 (9)
C(45)	5782 (4)	1433 (7)	1088 (8)	6.5 (9)
C(46)	5149 (4)	1258 (7)	1785 (8)	5.1 (8)
C(51)	3269 (3)	3131 (5)	3527 (7)	2.4 (6)
C(52)	2534 (3)	2829 (5)	2826 (7)	2.4 (5)
C(53)	2038 (3)	3745 (5)	3122 (7)	3.3 (6)
C(54)	2277 (3)	4963 (5)	4119 (7)	4.5 (8)
C(55)	3012 (3)	5265 (5)	4821 (7)	4.3 (6)
C(56)	3507 (3)	4349 (5)	4525 (7)	5.0 (7)
F(53)	1351 (4)	3593 (7)	2529 (8)	5.2 (4)
F(54)	1821 (4)	5846 (7)	4411 (10)	6.6 (5)
F(55)	3228 (5)	6445 (7)	5808 (8)	6.1 (4)
F(56)	4229 (4)	4692 (7)	5144 (8)	5.8 (4)
C(61)	4242 (4)	1945 (11)	4767 (7)	4.3 (6)
C(62)	3739 (4)	1817 (11)	5716 (7)	7 (1)
C(63)	4028 (4)	1910 (11)	7024 (7)	10 (1)
C(64)	4820 (4)	2132 (11)	7383 (7)	13 (1)
C(65)	5323 (4)	2260 (11)	6434 (7)	19 (1)
C(66)	5033 (4)	2167 (11)	5126 (7)	12 (1)
O(1)	3313 (4)	738 (7)	2367 (8)	4.0 (4)
O(2)	2565 (5)	4445 (9)	8202 (9)	5.3 (4)
C(3)	2619 (15)	4329 (26)	9759 (26)	11.3 (8)
C(4)	2629 (8)	5758 (14)	11050 (15)	5.8 (9)

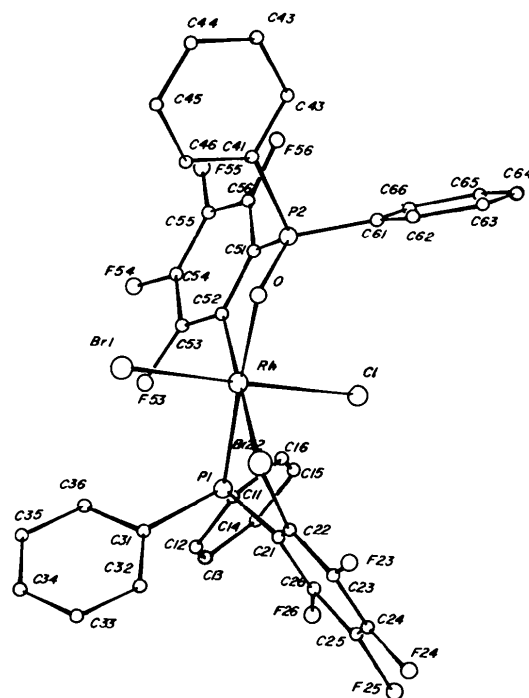
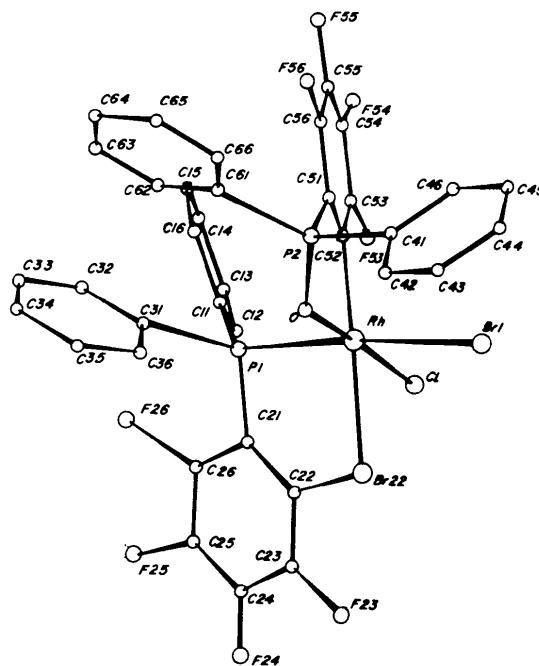
Table 2. Selected bond distances (Å) and angles (°)

	(I)	(II)
Br(1)—Rh	2.417 (4)	2.437 (3)
Br(22)—Rh	2.677 (4)	2.613 (2)
P(1)—Rh	2.288 (7)	2.288 (4)
C(52)—Rh	2.07 (2)	2.043 (5)
Cl—Rh	2.433 (6)	2.381 (4)
O(1)—Rh	2.01 (2)	2.087 (8)
C(11)—P(1)	1.81 (1)	1.774 (8)
C(21)—P(1)	1.70 (1)	1.861 (7)
C(31)—P(1)	1.85 (1)	1.815 (8)
C(41)—P(2)	1.79 (2)	1.807 (8)
C(51)—P(2)	1.77 (1)	1.762 (7)
C(61)—P(2)	1.76 (2)	1.743 (9)
O(1)—P(2)	1.45 (2)	1.515 (7)
C(22)—Br(22)	1.70 (2)	1.868 (5)
Br(22)—Rh—Br(1)	91.0 (1)	89.5 (1)
Cl—Rh—Br(1)	178.1 (2)	90.5 (1)
P(1)—Rh—Br(1)	95.0 (2)	175.0 (1)
C(52)—Rh—Br(1)	86.9 (5)	89.4 (2)
C(52)—Rh—P(1)	105.1 (4)	94.9 (2)
O(1)—Rh—Br(1)	88.6 (7)	87.9 (2)
O(1)—Rh—P(1)	175.1 (9)	94.9 (3)
O(1)—Rh—C(52)	78.4 (8)	85.6 (3)
Br(22)—Rh—Cl	89.0 (2)	84.8 (1)
Br(22)—Rh—P(1)	80.4 (2)	86.3 (1)
Br(22)—Rh—C(52)	174.2 (4)	176.4 (1)
Br(22)—Rh—O(1)	96.2 (7)	91.0 (2)
Cl—Rh—P(1)	86.8 (2)	86.3 (1)
Cl—Rh—C(52)	92.9 (5)	98.6 (2)
Cl—Rh—O(1)	89.5 (7)	175.5 (2)

[P(1), C(21), C(22), Br(22) and P(2), C(51), C(52), Rh, respectively].

In structure (II) the F(53) contacts with the Cl<sup>-</sup> ion [2.911 (4) Å] produce the increase in the Cl—Rh—C(52) [98.6 (2)°] bond angle and Rh—Br(22) bond distance, and the decrease in the Cl—Rh—Br(22) [84.8 (1)°] bond angle. The five-membered rings have an envelope form in this structure, with Rh [0.80 (1) Å] and O [0.28 (2) Å] atoms out of the plane of the remaining four atoms.

This work was sponsored by grants from the University of Barcelona and CSIC.


 Fig. 1. Molecular view of the *trans* complex with the numbering of atoms.

 Fig. 2. Molecular view of the *cis* complex with the numbering of atoms.

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*Acta Cryst.* (1985). **C41**, 844–846

**Structure of {Diaqua[*N,N'*-bis( $\beta$ -carbamoylethyl)-2-hydroxy-trimethylenediamine]}nickel(II) Dichloride,\* [Ni(C<sub>9</sub>H<sub>20</sub>N<sub>4</sub>O<sub>3</sub>)(H<sub>2</sub>O)<sub>2</sub>]Cl<sub>2</sub>**

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(Received 12 October 1984; accepted 2 January 1985)

**Abstract.**  $M_r = 397.89$ , monoclinic,  $P2_1/m$ ,  $a = 7.002$  (6),  $b = 17.483$  (11),  $c = 7.369$  (7) Å,  $\beta = 117.73$  (5)°,  $U = 827.74$  (1) Å<sup>3</sup>,  $Z = 2$ ,  $D_x = 1.596$ ,  $D_m = 1.60$  (5) Mg m<sup>-3</sup> (by flotation in CCl<sub>4</sub>/hexane mixture),  $Mo K\alpha$ ,  $\lambda = 0.71069$  Å,  $\mu = 1.515$  mm<sup>-1</sup>,  $F(000) = 416$ ,  $T = 296$  (4) K,  $R(F) = 0.047$  for 3153 observed reflections [ $I > 3\sigma(I)$ ]. The coordination geometry about Ni<sup>II</sup> is a pseudo-octahedron comprising two O atoms of amide groups [Ni–O 2.056 (2) Å] and two N atoms of amine groups [Ni–N 2.085 (2) Å] at the four corners of the equatorial plane and two O atoms of water molecules at the two apices [Ni–O 2.100 (3), 2.159 (2) Å]. The two H atoms of the amine groups are in *cis* configuration with respect to the equatorial plane and the two chiral amine N centers have the *RS* configuration. Besides the ionic interaction, the Cl<sup>-</sup> ions form intra- and intermolecular hydrogen bonds with the coordinated water molecules.

**Introduction.** In a previous work we have reported the crystal structure of [Cu(bchtn)](ClO<sub>4</sub>)<sub>2</sub> (Lee, Hong, Liu, Chung & Lee, 1984) [bchtn is *N,N'*-bis( $\beta$ -carbamoylethyl)-2-hydroxytrimethylenediamine]. In this work the crystal structure of [Ni(bchtn)(H<sub>2</sub>O)<sub>2</sub>]Cl<sub>2</sub> is reported.

\* [Diaqua(6-hydroxy-4,8-diazaundecanediamide)]nickel(II) dichloride.

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**Experimental.** [Ni(bchtn)(H<sub>2</sub>O)<sub>2</sub>]Cl<sub>2</sub> crystals were obtained similarly to those of [Cu(bchtn)](ClO<sub>4</sub>)<sub>2</sub>, except that NiCl<sub>2</sub> was used in place of Cu(ClO<sub>4</sub>)<sub>2</sub>. The crystals thus obtained were light blue and 0.3 ~ 0.5 mm in size.

Experimental data and structure-solution parameters together with the standard refinement procedures are listed in Table 1. The H atoms of the amine, amide and OH groups were located in the difference Fourier maps. Other H-atom parameters were treated as in our previous work (Lee *et al.*, 1984).

**Discussion.** The atomic coordinates and temperature factors are listed in Table 2.‡ A perspective view of the molecule and the numbering scheme and deviations of atoms from the least-squares plane through O(1)–N(2)–N(2')–O(1') are shown in Fig. 1 (*a*).

The Ni<sup>II</sup> ion in the title complex is surrounded pseudo-octahedrally. Two N atoms and two O atoms of bchtn are coordinated to the Ni<sup>II</sup> ion in a plane. The two axial coordination sites are occupied by two O atoms of water molecules at distances of 2.159 (2) and 2.100 (3) Å, respectively. The bond distances and bite angles about the metal ion are given in Table 3 along

‡ Lists of structure factors, anisotropic thermal parameters, H-atom parameters and details of the least-squares plane have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42005 (36 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.