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

Os(1)—Pt	2·720 (1)	Os(1)—Os(2)	2·758 (1)
Os(2)—Pt	2·882 (1)	Os(1)—Os(3)	3·046 (1)
Os(3)—Pt	3·837 (1)	Os(2)—Os(3)	2·882 (1)
Pt—P(1)	2·344 (5)	Os(3)—Pt(2)	2·379 (5)
Pt—C(10)	1·83 (2)	Os(1)—C(1)	1·86 (3)
Os(1)—C(2)	1·85 (2)	Os(1)—C(3)	1·85 (3)
Os(2)—C(4)	1·83 (3)	Os(2)—C(5)	1·83 (2)
Os(2)—C(6)	1·80 (3)	Os(3)—C(7)	1·83 (2)
Os(3)—C(8)	1·92 (2)	Os(3)—C(9)	1·90 (2)

Mean carbonyl C—O 1·18 (3)

Os(1)—Pt—P(1)	175·5 (2)	Os(1)—Pt—C(10)	82·2 (6)
H(1)—Pt—P(1)	84·2 (2)	H(2)—Os(1)—C(3)	164·1 (7)
Pt—Os(1)—C(1)	160·5 (6)	Os(2)—Os(1)—C(2)	166·1 (6)
Os(1)—Os(2)—C(4)	160·6 (7)	H(1)—Os(2)—C(5)	169·9 (6)
Os(3)—Os(2)—C(6)	165·9 (7)	P(2)—Os(3)—Os(2)	167·1 (2)
H(2)—Os(3)—C(7)	176·4 (6)	C(8)—Os(3)—C(9)	177·4 (8)

wider butterfly angle for the title complex, *cf.* 98·2 (1) *versus* 88·4 ($bis\text{-}PPh_3$) and 87·5° ($bis\text{-}PCy_3$). This is also seen in an increased non-bonding Os—Pt wing-tip–wing-tip distance, *cf.* Os(3)—Pt = 3·837 (1) *versus* 3·506 (1) ($bis\text{-}PPh_3$) and 3·530 (1) Å ($bis\text{-}PCy_3$). This is a reflection of the flexibility of the Os_3Pt butterfly metal skeleton. In addition the ligand sphere around Os(3) is twisted by *ca* 11·5° out of the Os_3 plane, as defined by the angles between the Os_3 plane and the mean plane Os(3), P(2), C(7), O(7), H(2). In the bis(tricyclohexylphosphine) derivative this twist is similar (11·8°), but it is much smaller in the bis(triphenylphosphine) derivative (0·4°).

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Structure of 1,4,7-Tris[(2*S*)-2-hydroxypropyl]-1,4,7-triazacyclononanenickel(II) Dibromide Monohydrate

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Abstract. $[Ni(C_{15}H_{33}N_3O_3)]Br_2.H_2O$, $M_r = 539{\cdot}96$, orthorhombic, $P2_12_12_1$, $a = 9{\cdot}7046\,(9)$, $b = 13{\cdot}742\,(3)$, $c = 16{\cdot}285\,(1)$ Å, $V = 2171{\cdot}8\,(5)$ Å^3, $Z = 4$, $D_x = 1{\cdot}65$ g cm^{-3}, $\lambda(\text{Mo } K\alpha) = 0{\cdot}71069$ Å, $\mu = 45{\cdot}65$ cm^{-1}, $F(000) = 1104$, $T = 298$ K, $R = 0{\cdot}040$ for 2823 unique observed reflections. The Ni atom is coordinated octahedrally to the *fac*-N_3O_3 donor set of the macrocyclic ligand, and a molecule of water is hydrogen bonded to one of the ligand hydroxyl groups. The twist angle φ, away from idealized octahedral coordination, is 19·0° between the N_3 donor set and the O_3 set.

Experimental. The title complex was synthesized as previously described (Robb, 1987), and recrystallized from water. A pale-purple prism *ca* 0·8 × 0·6 × 0·4 mm was mounted on a glass fibre; systematic absences: $h = 2n + 1$ in $h00$, $k = 2n + 1$ in $0k0$; $l = 2n + 1$ in $00l$; Enraf–Nonius CAD-4F diffractometer; graphite monochromator; $\theta/2\theta$ scan mode; cell parameters refined by least-squares methods from setting angles of 25 independent reflections with $11 < \theta < 13°$; intensities measured to $\theta = 25{\cdot}0°$ in two octants, hkl and $\overline{hkl}$ with $|h|$ 0–11, $|k|$ 0–16, $|l|$ 0–19; $2\bar{9}2$ and $\bar{1}\bar{7}1$ measured every 2 h with no significant decay noted over 69 h data collection; 4403 measured data yielded 3811 independent data, containing 1632 Friedel pairs; of these 2823 having $I > 3{\cdot}0\sigma(I)$ were considered observed and used in structure determination and refinement; R_{int} before absorption correction 0·020, after correction 0·019; corrected for Lorentz–polarization and absorption (*DIFABS*; Walker & Stuart, 1983), with max., min. applied absorption corrections of 1·10, 0·87; solved by direct methods (*MITHRIL*; Gilmore, 1984) and subsequent full-matrix least squares; anisotropic thermal parameters for all non-H atoms; C—H H atoms included at calculated positions (C—H = 1·0 Å), O—H H-atom positions obtained from difference synthesis, fixed contributions to structure factors for all H atoms, with $U = 0{\cdot}05$ Å^2; $\sum w(|F_o| - |F_c|)^2$ minimized with $w = [\sigma^2(F_o)]^{-1}$; max. $\Delta/\sigma =$

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Table 1. *Fractional coordinates with e.s.d.'s in parentheses, and isotropic thermal parameters* (Å^2) (*equivalent isotropic parameters U_{eq} for anisotropic atoms*)

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

	x	*y*	*z*	*U*
Ni	0·61228 (7)	0·24414 (6)	0·75593 (5)	0·020
Br(1)	0·69545 (10)	0·15723 (8)	0·35046 (6)	0·059
Br(2)	0·07062 (11)	0·08494 (7)	1·00001 (6)	0·069
O(1)	0·7537 (4)	0·3577 (3)	0·7654 (3)	0·030
O(2)	0·5170 (5)	0·3087 (3)	0·6558 (3)	0·032
O(3)	0·4801 (5)	0·3090 (3)	0·8390 (3)	0·029
O(4)	0·8884 (6)	0·4287 (6)	0·6398 (4)	0·106
N(1)	0·7535 (6)	0·1793 (4)	0·8328 (3)	0·029
N(2)	0·6960 (6)	0·1612 (4)	0·6628 (3)	0·030
N(3)	0·4865 (6)	0·1267 (4)	0·7804 (4)	0·034
C(10)	0·8683 (7)	0·3375 (6)	0·8187 (5)	0·038
C(11)	0·9095 (9)	0·4294 (7)	0·8635 (6)	0·066
C(12)	0·8232 (7)	0·2608 (6)	0·8756 (4)	0·035
C(13)	0·8479 (8)	0·1187 (6)	0·7826 (5)	0·036
C(14)	0·8414 (8)	0·1426 (6)	0·6894 (5)	0·040
C(20)	0·5524 (8)	0·2668 (6)	0·5762 (4)	0·040
C(21)	0·5555 (9)	0·3434 (7)	0·5127 (4)	0·059
C(22)	0·6884 (8)	0·2194 (6)	0·5869 (4)	0·042
C(23)	0·6186 (9)	0·0685 (5)	0·6577 (5)	0·048
C(24)	0·4742 (9)	0·0776 (6)	0·6973 (5)	0·051
C(30)	0·3733 (7)	0·2421 (6)	0·8701 (4)	0·037
C(31)	0·2431 (8)	0·2985 (6)	0·8856 (5)	0·048
C(32)	0·3561 (7)	0·1654 (6)	0·8087 (5)	0·038
C(33)	0·5550 (8)	0·0660 (5)	0·8419 (5)	0·048
C(34)	0·6679 (8)	0·1195 (5)	0·8901 (5)	0·039
H(1)	0·80159	0·38592	0·70373	0·050
H(2)	0·43633	0·31995	0·65313	0·050
H(3)	0·52231	0·32913	0·89052	0·050
H(4)	0·89826	0·43507	0·58977	0·050
H(5)	0·97864	0·42478	0·64216	0·050

0·057, av. $\Delta/\sigma = 0·011$; $\Delta\rho_{max} = +0·64$, $\Delta\rho_{min} = -0·88$ e Å^{-3}; final $R = 0·040$, $wR = 0·048$ using 226 parameters, $S = 2·17$; expected *S* configuration at chiral C atoms confirmed by refinement of η parameter (Rodgers, 1981) [$\eta = 0·998$ (35), $(1 + \eta)/\sigma(\eta) = 56·4$], refinement using inverted configuration converged at $R = 0·056$, $wR = 0·067$; atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV, pp. 71, 151); calculations carried out on a MicroVAX 3600 computer using the Glasgow *GX* suite of programs (Mallinson & Muir, 1985). Final positional parameters are given in Table 1, with selected bond distances and angles in Table 2.* The atomic labelling scheme and molecular structure are shown in Fig. 1.

Related literature. The Ni atom is coordinated to the *fac*-N_3O_3 donor set of the macrocyclic ligand in a distorted octahedral fashion. A molecule of water is hydrogen bonded to one of the ligand hydroxyl groups, while the two bromide ions are separated by normal van der Waals distances. The twist angle φ between the N_3 donor set and the O_3 donor set, away from idealized octahedral geometry, is 19·0°. φ is defined as (180° − *TA*), where *TA* is the *mean* of the three torsion angles O—O(CT)—N(CT)—N which are closest to 180°, and O(CT), N(CT) are the centroids of the O_3 and N_3 donor atoms respectively. The Co^{III} complex of the ligand, which crystallizes as a hydrogen-bridged dimer (Belal, Farrugia & Peacock, 1989), has φ angles of 10·2 and 11·2°.

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

Ni—O(1)	2·083 (5)	Ni—O(2)	2·073 (5)
Ni—O(3)	2·066 (5)	Ni—N(1)	2·059 (6)
Ni—N(2)	2·065 (6)	Ni—N(3)	2·062 (6)
O(1)—C(10)	1·438 (9)	O(2)—C(20)	1·459 (9)
O(3)—C(30)	1·476 (9)	N(1)—C(12)	1·483 (10)
N(1)—C(13)	1·483 (10)	N(1)—C(34)	1·495 (10)
N(2)—C(14)	1·498 (10)	N(2)—C(22)	1·473 (10)
N(2)—C(23)	1·480 (10)	N(3)—C(24)	1·516 (11)
N(3)—C(32)	1·448 (10)	N(3)—C(33)	1·463 (10)
C(10)—C(11)	1·512 (13)	C(10)—C(12)	1·471 (11)
C(13)—C(14)	1·555 (11)	C(20)—C(21)	1·476 (12)
C(20)—C(22)	1·483 (11)	C(23)—C(24)	1·548 (13)
C(30)—C(31)	1·504 (11)	C(30)—C(32)	1·462 (12)
C(33)—C(34)	1·535 (12)		

N(1)—Ni—N(2)	86·9 (3)	N(1)—Ni—N(3)	86·4 (3)
N(2)—Ni—N(3)	86·7 (3)	O(1)—Ni—O(2)	91·8 (2)
O(1)—Ni—O(3)	92·1 (2)	O(2)—Ni—O(3)	93·1 (2)
O(1)—Ni—N(1)	80·8 (2)	O(1)—Ni—N(2)	102·0 (3)
O(1)—Ni—N(3)	164·1 (3)	O(2)—Ni—N(1)	163·7 (3)
O(2)—Ni—N(2)	80·4 (3)	O(2)—Ni—N(3)	102·9 (3)
O(3)—Ni—N(1)	101·7 (2)	O(3)—Ni—N(2)	164·5 (3)
O(3)—Ni—N(3)	81·0 (3)		

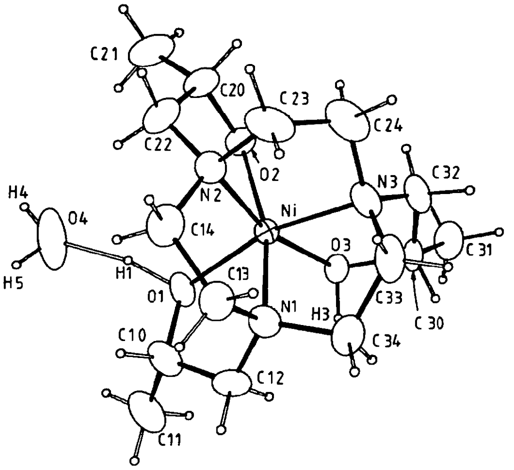

Fig. 1. Molecular structure and atomic labelling scheme, showing the Ni^{II} complex cation, and the hydrogen-bonded water molecule. Thermal parameters for anisotropic atoms are drawn at the 50% probability level, while H atoms are shown as spheres of arbitrary radius.

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* Lists of structure factors, anisotropic thermal parameters, calculated hydrogen positional parameters, and complete bond lengths, bond angles and torsion angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53848 (25 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.