O(3)—Hg(1)—Ru(1)	111.6 (2)	C(1)—Ru(1)—Hg(1)	77.7 (2)
C(2)—Ru(1)—Hg(1)	74.9 (2)	C(2)—Ru(1)—C(1)	94.7 (3)
N(1)—Ru(1)—Hg(1)	95.4 (1)	N(1)—Ru(1)—C(1)	163.2 (2)
N(1)—Ru(1)—C(2)	98.4 (3)	P(1)—Ru(1)—Hg(1)	158.00 (1)
P(1)—Ru(1)—C(1)	89.4 (2)	P(1)—Ru(1)—C(2)	88.6 (2)
P(1)—Ru(1)—N(1)	101.5 (1)	Ru(1)—Hg(1)—Ru(1)*	60.10 (1)
O(1)—C(1)—Ru(1)	174.8 (6)	O(2)—C(2)—Ru(1)	177.8 (7)
C(5)—N(1)—Ru(1)	119.7 (4)	C(6)—C(5)—N(1)	119.3 (6)
C(10)—C(5)—N(1)	121.7 (6)	C(10)—C(5)—C(6)	119.0 (6)
C(7)—C(6)—C(5)	119.1 (4)	C(8)—C(7)—C(6)	118.5 (5)
C(9)—C(8)—C(7)	121.8 (8)	C(10)—C(9)—C(8)	119.4 (8)
C(9)—C(10)—C(5)	122.2 (7)	C(11)—P(1)—Ru(1)	112.8 (2)
C(14)—P(1)—Ru(1)	117.5 (3)	C(14)—P(1)—C(11)	103.1 (4)
C(17)—P(1)—Ru(1)	112.5 (3)	C(17)—P(1)—C(11)	105.0 (4)
C(17)—P(1)—C(14)	104.7 (4)	C(12)—C(11)—P(1)	113.4 (5)
C(13)—C(11)—P(1)	117.5 (6)	C(13)—C(11)—C(12)	106.9 (8)
C(15)—C(14)—P(1)	114.9 (6)	C(16)—C(14)—P(1)	116.0 (6)
C(16)—C(14)—C(15)	108.8 (8)	C(18)—C(17)—P(1)	114.0 (6)
C(19)—C(17)—P(1)	116.4 (7)	C(19)—C(17)—C(18)	109.9 (9)
C(4)—C(3)—Cl(1)	110.2 (4)	O(3)—C(4)—C(3)	112.1 (7)
O(4)—C(4)—C(3)	123.9 (8)	O(4)—C(4)—O(3)	123.7 (8)
C(4)—O(3)—Hg(1)	104.9 (5)		

Synthesis: the title compound was prepared as described previously (Cabeza *et al.*, 1992). It was recrystallized by layering diethyl ether on a concentrated solution of the complex in dichloromethane, allowing the layers to diffuse slowly at 253 K. Profile analysis was performed on all reflections (Lehmann & Larsen, 1974; Grant & Gabe, 1978). Some doubly measured reflections were averaged. Lorentz and polarization corrections were applied and data reduced to $|F_o|$ values.

Structure solved by Patterson interpretation using the program *SHELXS*86 (Sheldrick, 1985). Solution showed a C_2 axis through the Hg(1), C(5) and C(6) atoms. Isotropic least-squares refinement, using a local version of *SHELX*76 (Sheldrick, 1976; Van der Maelen Uría, 1991), converged to $R = 0.12$. Empirical absorption correction (Walker & Stuart, 1983) lowered this parameter to $R = 0.09$.

Anisotropic refinement followed by a difference Fourier synthesis allowed the location of all the H atoms. Positional parameters and anisotropic thermal parameters of the non-H atoms were refined. All H atoms were refined isotropically, including positional parameters, except for C(13), C(15), C(16) and C(19) methyl groups, H(31) and (H32), which were left riding, constraining the distances to their parent atoms.

Geometrical calculations were made with *PARST* (Nardelli, 1983). All calculations were carried out on a MicroVAX-3400 at the Scientific Computer Center, University of Oviedo, Spain.

Lists of structure factors, anisotropic thermal parameters, H-atom coordinates and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71088 (29 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: HU1035]

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Structure of Diaqua(4,7-diazadecanediamide-N^4,N^7,*O*,*O′*)nickel(II) Perchlorate

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Abstract

The Ni^{II} ion is six-coordinated in a distorted octahedral geometry, through two equatorial amino N atoms and two equatorial amide O atoms of the tetradentate ligand, and two axial water O atoms.

Comment

The Ni^{II} ion is six-coordinated in a distorted octahedral geometry, where the quadridentate ligand 4,7-diazadecanediamide coordinates to the Ni^{II} ion with two amino N atoms and two amide O atoms in equatorial positions (Freeman, 1967; Sigel & Martin, 1985); two water O atoms occupy the axial positions. The two asymmetric N atoms, N(2) and N(3), have the same *R* or *S* configurations. The two N(amino)—Ni—O(amide) bond angles are close to 180° and the NiN_2O_2 group is almost planar. The diazadiamide in

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the planar coordination has two terminal six-membered chelate rings in a stable chair form and the central five-membered chelate ring in a stable *gauche* form. The arrangement of 4,7-diazadecanediamide in this complex appears to be relatively free of strain. The axial Ni—O bonds [2.120 (3) and 2.133 (3) Å] are slightly longer than the equatorial Ni—O bonds [2.038 (4) and 2.067 (3) Å]. The two perchlorate anions, which act as counter ions in the lattice, are not bound to the Ni^{II} ion, as can be seen from the *ORTEP*II (Johnson, 1976) plot of Fig. 1.

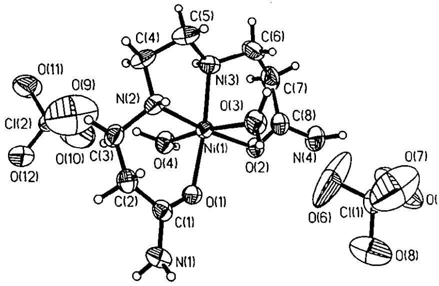

Fig. 1. *ORTEP*II drawing of a single molecule with thermal ellipsoids scaled to 50% probability. H atoms are shown by open circles but not labeled.

Experimental

Crystal data

$[Ni(C_8H_{18}N_4O_2)(H_2O)_2](ClO_4)_2$
$M_r = 495.90$
Monoclinic
$P2_1/n$
$a = 9.917$ (3) Å
$b = 10.309$ (2) Å
$c = 18.695$ (10) Å
$\beta = 99.17$ (3)°
$V = 1887.1$ (15) Å^3
$Z = 4$

$D_x = 1.745$ Mg m^{-3}
Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 9$-$15°$
$\mu = 1.3805$ mm^{-1}
$T = 300$ K
Bulk
0.4 × 0.3 × 0.3 mm
Orange

Data collection

Nonius CAD-4 diffractometer
ω-2θ scans of ω-scan rate 2-10° min^{-1}
Absorption correction: empirical
$T_{min} = 0.763$, $T_{max} = 1.000$
3452 measured reflections
3252 independent reflections

2819 observed reflections $[F > 4\sigma(F)]$
$R_{int} = 0.0661$
$\theta_{max} = 25°$
$h = 0 \rightarrow 11$
$k = -12 \rightarrow 0$
$l = -22 \rightarrow 21$
3 standard reflections
frequency: 60 min
intensity variation: 1.36%

Refinement

Refinement on F
Final $R = 0.0552$
$wR = 0.0556$
$S = 1.78$
2800 reflections
266 parameters
H-atom parameters not refined

Unit weights applied
$(\Delta/\sigma)_{max} = 0.031$
$\Delta\rho_{max} = 0.77$ e Å^{-3}
$\Delta\rho_{min} = -0.61$ e Å^{-3}
Atomic scattering factors from *SHELXTL/PC* (Siemens, 1990)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement coefficients* (Å^2)

U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U_{eq}
Ni(1)	0.8802 (1)	0.2553 (1)	0.6172 (1)	0.028 (1)
O(4)	0.9538 (4)	0.3662 (4)	0.71085 (2)	0.045 (1)
O(3)	0.8128 (4)	0.1685 (4)	0.5142 (2)	0.045 (1)
O(1)	0.7199 (3)	0.3855 (3)	0.5995 (2)	0.038 (1)
C(1)	0.6058 (5)	0.3767 (5)	0.6180 (2)	0.034 (2)
N(1)	0.5147 (5)	0.4730 (4)	0.6023 (3)	0.047 (2)
C(2)	0.5580 (5)	0.2614 (5)	0.6565 (3)	0.041 (2)
C(3)	0.6674 (5)	0.1884 (5)	0.7075 (3)	0.040 (2)
N(2)	0.7657 (4)	0.1272 (4)	0.6664 (2)	0.035 (1)
C(4)	0.8621 (6)	0.0385 (5)	0.7096 (3)	0.048 (2)
C(5)	0.9711 (6)	0.0004 (5)	0.6667 (3)	0.053 (2)
N(3)	1.0359 (4)	0.1220 (4)	0.6460 (2)	0.038 (1)
C(6)	1.1287 (6)	0.1030 (6)	0.5926 (3)	0.051 (2)
C(7)	1.2000 (6)	0.2292 (6)	0.5803 (3)	0.053 (2)
C(8)	1.1100 (5)	0.3348 (5)	0.5432 (3)	0.041 (2)
N(4)	1.1555 (6)	0.3977 (6)	0.4907 (3)	0.065 (2)
O(2)	0.9980 (3)	0.3655 (3)	0.5610 (2)	0.039 (1)
Cl(1)	0.7987 (1)	0.3625 (1)	0.3242 (1)	0.045 (1)
O(5)	0.9258 (8)	0.3884 (7)	0.3061 (6)	0.176 (5)
O(6)	0.8216 (12)	0.3278 (9)	0.3945 (3)	0.198 (6)
O(7)	0.7312 (9)	0.2672 (7)	0.2822 (4)	0.157 (4)
O(8)	0.7276 (9)	0.4826 (7)	0.3137 (4)	0.142 (4)
Cl(2)	0.9347 (1)	0.3404 (1)	0.9200 (1)	0.044 (1)
O(9)	0.8881 (11)	0.2750 (9)	0.8577 (4)	0.197 (5)
O(10)	1.0363 (9)	0.4222 (7)	0.9064 (7)	0.189 (6)
O(11)	0.9870 (8)	0.2521 (7)	0.9732 (3)	0.125 (3)
O(12)	0.8314 (6)	0.4181 (5)	0.9443 (3)	0.094 (2)

Table 2. *Geometric parameters* (Å, °)

Ni(1)—O(4)	2.120 (3)	Ni(1)—O(3)	2.133 (3)
Ni(1)—O(1)	2.067 (3)	Ni(1)—N(2)	2.052 (4)
Ni(1)—N(3)	2.072 (4)	Ni(1)—O(2)	2.038 (4)
O(1)—C(1)	1.237 (6)	C(1)—N(1)	1.343 (7)
C(1)—C(2)	1.506 (7)	C(2)—C(3)	1.524 (7)
C(3)—N(2)	1.476 (7)	N(2)—C(4)	1.468 (7)
C(4)—C(5)	1.497 (9)	C(5)—N(3)	1.488 (7)
N(3)—C(6)	1.475 (8)	C(6)—C(7)	1.516 (9)
C(7)—C(8)	1.506 (8)	C(8)—N(4)	1.315 (8)
C(8)—O(2)	1.249 (6)	Cl(1)—O(5)	1.382 (9)
Cl(1)—O(6)	1.346 (7)	Cl(1)—O(7)	1.364 (8)
Cl(1)—O(8)	1.422 (8)	Cl(2)—O(9)	1.361 (8)
Cl(2)—O(10)	1.368 (9)	Cl(2)—O(11)	1.387 (6)
Cl(2)—O(12)	1.430 (6)		
O(4)—Ni(1)—O(3)	171.5 (1)	O(4)—Ni(1)—O(1)	86.4 (1)
O(3)—Ni(1)—O(1)	90.3 (1)	O(4)—Ni(1)—N(2)	96.9 (2)
O(3)—Ni(1)—N(2)	91.0 (2)	O(1)—Ni(1)—N(2)	91.3 (2)
O(4)—Ni(1)—N(3)	90.0 (2)	O(3)—Ni(1)—N(3)	94.0 (2)
O(1)—Ni(1)—N(3)	173.9 (2)	N(2)—Ni(1)—N(3)	84.2 (2)
O(4)—Ni(1)—O(2)	88.6 (1)	O(3)—Ni(1)—O(2)	83.7 (1)
O(1)—Ni(1)—O(2)	92.6 (1)	N(2)—Ni(1)—O(2)	173.5 (2)
N(3)—Ni(1)—O(2)	92.3 (2)	Ni(1)—O(1)—C(1)	128.5 (3)
O(1)—C(1)—N(1)	119.9 (5)	O(1)—C(1)—C(2)	124.2 (4)
N(1)—C(1)—C(2)	115.8 (4)	C(1)—C(2)—C(3)	116.3 (4)
C(2)—C(3)—N(2)	110.4 (4)	Ni(1)—N(2)—C(3)	114.7 (3)
Ni(1)--N(2)—C(4)	106.7 (3)	C(3)—N(2)—C(4)	114.0 (4)
N(2)—C(4)—C(5)	109.4 (5)	C(4)—C(5)—N(3)	107.2 (4)
Ni(1)—N(3)—C(5)	107.0 (3)	Ni(1)—N(3)—C(6)	115.6 (3)
C(5)—N(3)—C(6)	113.9 (4)	N(3)—C(6)—C(7)	110.2 (5)
C(6)—C(7)—C(8)	115.7 (5)	C(7)—C(8)—N(4)	116.8 (5)
C(7)—C(8)—O(2)	123.2 (5)	N(4)—C(8)—O(2)	120.0 (5)
Ni(1)—O(2)—C(8)	127.1 (3)	O(5)—Cl(1)—O(6)	105.8 (7)
O(5)—Cl(1)—O(7)	112.4 (6)	O(6)—Cl(1)—O(7)	111.2 (5)
O(5)—Cl(1)—O(8)	104.4 (5)	O(6)—Cl(1)—O(8)	111.7 (6)
O(7)—Cl(1)—O(8)	111.1 (5)	O(9)—Cl(2)—O(10)	107.8 (7)

O(9)—Cl(2)—O(11)	109.0 (5)	O(10)—Cl(2)—O(11)	109.6 (5)
O(9)—Cl(2)—O(12)	112.7 (5)	O(10)—Cl(2)—O(12)	107.5 (5)
O(11)—Cl(2)—O(12)	110.1 (4)		

The ligand 4,7-diazadecanediamide was prepared from ethylenediamine (13.2 ml, 0.2 mol) and acrylamide (28.4 g, 0.4 mol) in absolute ethanol (100 ml) by heating the solution under dinitrogen gas and refluxing for 2 h. The solution was cooled and the precipitate filtered off, washed with absolute ethanol, recrystallized twice from methanol, and dried in air. Equimolar quantities of $Ni(ClO_4)_2.6H_2O$ and ligand were added to water (100 ml) and the solution allowed to react overnight at room temperature. The orange products were dried by evacuation using a rotary pump and were recrystallized twice from ethanol. The structure was solved by direct methods and refined by full-matrix least squares. *SHELXTL/PC* (Siemens, 1990) was used for all the calculations.

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Structure of μ-Pyrazine-bis[bis(hexafluoroacetylacetonato)copper(II)]

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Abstract

The title structure, μ-pyrazine-bis[bis(1,1,1,5,5,5-hexafluoro-2,4-pentanedionato)copper(II)], consists of a centrosymmetric dimer containing a bridging molecule of pyrazine. The pyrazine is essentially planar and coordinates axially to two symmetry-related molecules of $Cu(F_6acac)_2$, where acac is acetylacetone [Cu(1)—N(1) = 2.248 (6) Å]. The Cu atoms have square-pyramidal coordination geometries and are displaced by 0.235 (5) Å from the least-squares plane defined by the four O atoms of the basal F_6acac^- ligands.

Comment

Pyrazine is capable of forming a number of interesting complexes through coordination to one or more transtion-metal centers (Kaim, 1983). When reacted with Cu^{II}, complexes exhibiting three distinct structural motifs have been identified, these being discrete dimeric complexes containing a bridging molecule of pyrazine (Belford, Fenton & Truter, 1974; Haddad, Hendrickson, Cannady, Drago & Bieksza, 1979), infinite one-dimensional chain-type structures consisting of alternating molecules of pyrazine and Cu^{II} complex (Belford, Fenton & Truter 1974; Richardson & Hatfield, 1976; Santoro, Mighell & Reimann, 1970; Valentine, Silverstein & Soos, 1974), and two-dimensional networks consisting of sheets of Cu^{II} atoms bridged by bidentate pyrazine groups (Dariet, Haddad, Duesler & Hendrickson, 1979). The structures and magnetic properties of many of these systems have been investigated in some detail. Very weak antiferromagnetic couplings are observed in discrete molecular Cu^{II} complexes (Haddad, Hendrickson, Cannady, Drago & Bieksza, 1979). Magnetic data for Cu^{II}–pyrazine systems with chain-type structures can be described using a one-dimensional anisotropic Heisenberg model for antiferromagnetically coupled systems (Losee, Richardson & Hatfield, 1973; Richardson & Hatfield, 1976), whereas magnetic susceptibility data for the two-dimensional Cu^{II}–pyrazine complexes have adequately fitted to a theoretical equation for a two-dimensional Heisenberg antiferromagnet (Dariet, Haddad, Duesler & Hendrickson, 1979).

The reaction of bis(hexafluoroacetylacetonato)-copper(II)hydrate, $Cu(F_6acac)_2(H_2O)$, with pyrazine has been reported to yield two distinct products, depending on the reaction stoichiometry. The first of these is a centrosymmetric dimer where two $Cu(F_6acac)_2$ molecules are bridged symmetrically by a bidentate pyrazine ligand. The second product consists of a complex comprised of infinite chains of bridging pyrazine groups alternating with molecules of $Cu(F_6acac)_2$ (Belford, Fenton & Truter, 1974). Recently we repeated this reaction using pyrazine and anhydrous $Cu(F_6acac)_2$ and succeeded in isolating one of these products (I), in good yield. Although the structure does not differ significantly from that reported earlier (Belford, Fenton & Truter, 1974),

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