Refinement

Refinement on F^2
Final $R(F) = 0.0269$ for
  $F > 4\sigma(F)$ data
$wR(F^2) = 0.0616$ for all data
$S = 1.057$
3565 reflections
150 parameters
Calculated weights
  $w = 1/[\sigma^2(F_o^2)+(0.0202P)^2$
  $+0.8121P]$
  where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.194$ e Å^{-3}
$\Delta\rho_{min} = -0.150$ e Å^{-3}
Atomic scattering factors
  from *International Tables
  for Crystallography* (1992,
  Vol. C, Tables 4.2.6.8 and
  6.1.1.4)
Absolute configuration according to Flack (1983)

Data collection: *DIF4* (Stoe & Cie, 1988). Cell refinement: *DIF4*. Data reduction: *REDU4* (Stoe & Cie, 1988). Program(s) used to solve structure: *SHELXS-92* (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL-92* (Sheldrick, 1992). Molecular graphics: *SHELXTL-Plus* (Sheldrick, 1991). Software used to prepare material for publication: *SHELXL-92*.

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

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

	x	y	z	U_{eq}
S1	0.07958 (3)	0.50720 (4)	0.98797 (2)	0.0327 (2)
S2	0.11554 (3)	0.63303 (3)	0.92854 (2)	0.0319 (2)
S3	0.10418 (3)	0.60140 (3)	0.82065 (2)	0.0276 (2)
Na1	0.10709 (5)	0.38329 (5)	0.83890 (3)	0.0247 (3)
N1	0.13872 (12)	0.21307 (11)	0.90150 (8)	0.0313 (8)
N2	0.29592 (11)	0.36127 (11)	0.85166 (8)	0.0289 (7)
N3	0.17224 (11)	0.36322 (12)	0.71214 (8)	0.0325 (8)
C11	0.0722 (2)	0.1363 (2)	0.87043 (13)	0.0511 (13)
C12	0.1193 (2)	0.2189 (2)	0.98012 (10)	0.0433 (12)
C13	0.24482 (14)	0.18790 (15)	0.88728 (11)	0.0347 (10)
C21	0.3395 (2)	0.4542 (2)	0.88127 (12)	0.0433 (11)
C22	0.31559 (14)	0.27586 (14)	0.90035 (10)	0.0338 (9)
C23	0.33531 (14)	0.3415 (2)	0.77781 (10)	0.0349 (9)
C31	0.1173 (2)	0.4340 (2)	0.66560 (10)	0.0431 (12)
C32	0.1640 (2)	0.2622 (2)	0.68081 (11)	0.0424 (12)
C33	0.27755 (13)	0.3961 (2)	0.71878 (10)	0.0378 (10)

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

S1—S2	2.0487 (7)	S3—Na1	2.9073 (9)
S1—S1^i	2.1158 (9)	Na1—N3	2.501 (2)
S2—S3	2.0344 (7)	Na1—N2	2.528 (2)
S3—Na1^i	2.8245 (9)	Na1—N1	2.565 (2)
S2—S1—S1^i	107.74 (3)	N3—Na1—S3^i	103.79 (4)
S3—S2—S1	109.65 (3)	N2—Na1—S3^i	177.09 (4)
S2—S3—Na1^i	86.70 (2)	N1—Na1—S3^i	106.14 (4)
S2—S3—Na1	95.21 (2)	N3—Na1—S3	90.14 (4)
Na1^i—S3—Na1	94.05 (2)	N2—Na1—S3	97.95 (4)
N3—Na1—N2	74.53 (5)	N1—Na1—S3	157.90 (4)
N3—Na1—N1	105.62 (5)	S3^i—Na1—S3	84.37 (2)
N2—Na1—N1	72.28 (5)		

Symmetry code: (i) $-x, 1 - y, z$.

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Structure of μ-[Bis(chloroacetato-*O*)-mercury(II)-κ²*Hg*]-μ-[1,8-naphthalenediamino-κ²*NN'*:κ²*NN'*]-bis[*cis*-dicarbonyl-(triisopropylphosphine-*P*)ruthenium(I)]- (*Ru—Ru*)

S. García-Granda* and J. F. Van der Maelen Uría

Departamento de Química Física y Analítica, Facultad de Química, Universidad de Oviedo, Julián Clavería s/n, 33006 Oviedo, Spain

J. A. Cabeza, J. M. Fernández-Colinas and V. Riera

Departamento de Química Orgánica e Inorgánica, Facultad de Química, Universidad de Oviedo, Julián Clavería s/n, 33006 Oviedo, Spain

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Abstract

The molecule shows a twofold symmetry through the Hg atom and the diaminonaphthalene ligand. Ru—Ru and Ru—Hg distances are 2.799 (1) and 2.795 (1) Å, respectively.

Comment

This work is part of a project concerning the reactivity of the Ru—Ru bonds of binuclear ruthenium(I)

complexes. The X-ray structures of some selected compounds have been published (Andreu, Cabeza, Riera, Robert & Jeannin, 1989; Cabeza, Fernández-Colinas, Riera, Pellinghelli & Tiripicchio, 1991; Cabeza, Fernández-Colinas, Riera, García-Granda & Van der Maelen Uría, 1991; Cabeza, Fernández-Colinas, García-Granda, Riera & Van der Maelen Uría, 1991). The reported compound (Fig. 1) is isotypic to the analogous trifluoroacetate (Cabeza, Fernández-Colinas, García-Granda, Riera & Van der Maelen Uría, 1992), showing the same twofold axis symmetry through Hg(1), C(6) and C(7). All structural parameters are, therefore, very similar in both structures. The Ru—Ru and Hg—Ru distances are 2.799 (1) and 2.795 (1) Å, respectively, and are slighly shorter and longer than those found in the fluoroacetate compound. The dihedral angle between the Ru—Hg—Ru and O(3)—Hg—O(3)' planes is 36.7 (2)°, against the value of 35.0 (2)° found in the structure referred to above.
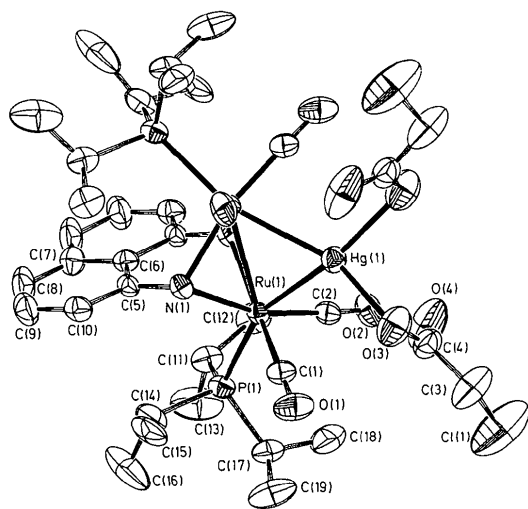

Fig. 1. An *EUCLID* (Spek, 1982) plot, showing the molecular symmetry and atomic numbering scheme.

Experimental

Crystal data

[HgRu$_2$(C$_2$H$_2$ClO$_2$)$_2$-
(C$_{10}$H$_8$N$_2$)(C$_9$H$_{21}$P)$_2$(CO)$_4$]
M_r = 1178.42
Monoclinic
C2/c
a = 23.586 (8) Å
b = 12.584 (2) Å
c = 14.540 (5) Å
β = 93.83 (3)°
V = 4306.0 (2) Å^3
Z = 4
D_x = 1.818 Mg m^{-3}

Mo $K\alpha$ radiation
λ = 0.71073 Å
Cell parameters from 25 reflections
θ = 15–17°
μ = 4.482 mm^{-1}
T = 293 K
Prismatic
0.40 × 0.33 × 0.10 mm
Yellow
Crystal source: synthesis

Data collection

Enraf-Nonius CAD-4 diffractometer
ω–2θ scans
T_{min} = 0.77, T_{max} = 1.34
4099 measured reflections
2756 independent reflections
2626 observed reflections
[I>3σ(I)]

R_{int} = 0.027
θ_{max} = 25°
h = −27 → 27
k = 0 → 14
l = 0 → 17
3 standard reflections
frequency: 60 min
intensity variation: 5%

Refinement

Final R = 0.033
wR = 0.034
2626 reflections
306 parameters
All H-atom parameters refined
w = 1/[σ^2(F)+0.0004F^2]

(Δ/σ)$_{max}$ = 0.04
$\Delta\rho_{max}$ = 0.90 e Å^{-3}
$\Delta\rho_{min}$ = −0.75 e Å^{-3}
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 1. *Fractional atomic coordinates and equivalent isotropic thermal parameters* (Å^2)

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

	x	y	z	U_{eq}
Hg(1)	0.00000	0.36659 (3)	0.25000	0.0381 (1)
Ru(1)	−0.05400 (2)	0.55886 (4)	0.20379 (3)	0.0264 (2)
C(1)	−0.0587 (3)	0.4860 (5)	0.0905 (5)	0.032 (2)
O(1)	−0.0591 (2)	0.4476 (4)	0.0187 (4)	0.051 (2)
C(2)	−0.1100 (3)	0.4751 (6)	0.2565 (5)	0.037 (2)
O(2)	−0.1430 (2)	0.4259 (5)	0.2909 (4)	0.061 (2)
N(1)	−0.0227 (2)	0.6411 (4)	0.3256 (4)	0.029 (2)
C(5)	−0.0214 (3)	0.7537 (5)	0.3263 (4)	0.029 (2)
C(6)	0.00000	0.8089 (7)	0.25000	0.034 (3)
C(7)	0.00000	0.9229 (8)	0.25000	0.040 (3)
C(8)	−0.0204 (4)	0.9764 (7)	0.3265 (7)	0.062 (3)
C(9)	−0.0406 (4)	0.9225 (7)	0.3993 (6)	0.053 (3)
C(10)	−0.0407 (3)	0.8117 (6)	0.3986 (5)	0.039 (2)
P(1)	−0.12884 (8)	0.6774 (1)	0.1444 (1)	0.0351 (6)
C(11)	−0.1053 (3)	0.7690 (6)	0.0526 (6)	0.045 (3)
C(12)	−0.0678 (4)	0.7150 (8)	−0.0152 (6)	0.052 (3)
C(13)	−0.1515 (4)	0.8297 (9)	−0.0051 (9)	0.093 (5)
C(14)	−0.1943 (3)	0.6159 (6)	0.0893 (6)	0.048 (3)
C(15)	−0.2332 (4)	0.5623 (9)	0.1566 (7)	0.079 (4)
C(16)	−0.1858 (4)	0.541 (1)	0.0112 (8)	0.088 (4)
C(17)	−0.1546 (4)	0.7665 (7)	0.2348 (6)	0.054 (3)
C(18)	−0.1643 (5)	0.710 (1)	0.3257 (7)	0.065 (4)
C(19)	−0.2057 (6)	0.837 (1)	0.2063 (9)	0.110 (6)
C(3)	−0.1298 (5)	0.1210 (9)	0.1822 (6)	0.100 (5)
Cl(1)	−0.1739 (5)	0.1283 (9)	0.0797 (6)	0.170 (3)
C(4)	−0.0859 (4)	0.2121 (6)	0.1857 (6)	0.050 (3)
O(3)	−0.0655 (3)	0.2293 (5)	0.2653 (4)	0.072 (2)
O(4)	−0.0701 (4)	0.2564 (6)	0.1180 (5)	0.103 (4)

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

Hg(1)—Ru(1)	2.795 (1)	P(1)—C(11)	1.876 (8)
Ru(1)—C(2)	1.892 (7)	C(11)—C(12)	1.53 (1)
Ru(1)—Ru(1)*	2.799 (1)	C(14)—C(16)	1.50 (1)
N(1)—C(5)	1.418 (8)	C(3)—Cl(1)	1.763 (1)
C(6)—C(7)	1.43 (1)	C(4)—O(4)	1.21 (1)
C(9)—C(10)	1.39 (1)	Ru(1)—C(1)	1.882 (7)
P(1)—C(17)	1.861 (8)	Ru(1)—P(1)	2.426 (2)
C(14)—C(15)	1.54 (1)	C(2)—O(2)	1.136 (8)
C(17)—C(19)	1.53 (1)	C(5)—C(10)	1.380 (9)
C(4)—O(3)	1.24 (1)	C(8)—C(9)	1.37 (1)
Hg(1)—O(3)	2.339 (6)	P(1)—C(14)	1.860 (7)
Ru(1)—N(1)	2.140 (5)	C(11)—C(13)	1.53 (1)
C(1)—O(1)	1.149 (8)	C(17)—C(18)	1.53 (1)
C(5)—C(6)	1.430 (8)	C(3)—C(4)	1.54 (1)
C(7)—C(8)	1.41 (1)		

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 SHELXS86 (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 SHELX76 (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.

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Structure of Diaqua(4,7-diazadecanediamide-N⁴,N⁷,O,O')nickel(II) Perchlorate

Tsong-Jen Lee and Arlohun Wang

Department of Physics, National Tsing Hua University, Hsinchu, 30043 Taiwan

Ta-Yung Chi and Chung-Sun Chung

Department of Chemistry, National Tsing Hua University, Hsinchu, 3004-3 Taiwan

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$_2$O$_2$ group is almost planar. The diazadiamide in