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: *DIF*4 (Stoe & Cie, 1988). Cell refinement: *DIF*4. Data reduction: *REDU*4 (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)

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

	x	y	z	$U_{\rm eq}$
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$^{\rm i}$	2.1158 (9)	Na1—N3	2.501 (2)
S2—S3	2.0344 (7)	Na1—N2	2.528 (2)
S3—Na1$^{\rm i}$	2.8245 (9)	Na1—N1	2.565 (2)
S2—S1—S1$^{\rm i}$	107.74 (3)	N3—Na1—S3$^{\rm i}$	103.79 (4)
S3—S2—S1	109.65 (3)	N2—Na1—S3$^{\rm i}$	177.09 (4)
S2—S3—Na1$^{\rm i}$	86.70 (2)	N1—Na1—S3$^{\rm i}$	106.14 (4)
S2—S3—Na1	95.21 (2)	N3—Na1—S3	90.14 (4)
Na1$^{\rm i}$—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$^{\rm i}$—Na1—S3	84.37 (2)
N2—Na1—N1	72.28 (5)		

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

DS thanks the University of Göttingen and the DAAD (Nato scholarship) for financial support.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and bond distances have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71149 (13 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: HL1023]

References

Abrahams, S. C. & Grison, E. (1953). *Acta Cryst.* **6**, 206-213.
Banister, A. J., Barr, D., Brooker, A. T., Clegg, W., Cunnington, M. J., Doyle, M. J., Drake, S. R., Gill, W. R., Manning, K., Raithby, P. R., Snaith, R., Wade, K. & Wright, D. S. (1990). *J. Chem. Soc. Chem. Commun.* pp. 105-107.
Flack, H. D. (1983). *Acta Cryst.* A**39**, 876-881.
Sheldrick, G. M. (1990). *Acta Cryst.* A**46**, 467-473.
Sheldrick, G. M. (1991). *SHELXTL-Plus*. Siemens Analytical X-ray Instruments, Inc., Madison, Wisconsin, USA.
Sheldrick, G. M. (1992). *SHELXL*-92. Program for crystal structure refinement. Univ. of Göttingen, Germany.
Stoe & Cie (1988). *DIF*4. Version 7.08. Diffractometer control program. Stoe & Cie, Darmstadt, Germany.
Stoe & Cie (1988). *REDU*4. Data reduction program. Stoe & Cie, Darmstadt, Germany.
Tatsumi, K., Inoue, Y., Nakamura, A., Cramer, R. E., VanDoorne, W. & Gilje, J. W. (1990). *Angew. Chem.* **102**, 455-457; *Angew. Chem. Int. Ed. Engl.* **29**, 422-424.
Wells, A. F. (1984). *Structural Inorganic Chemistry*, 5th ed. Oxford Univ. Press.

Acta Cryst. (1993). C**49**, 1483–1485

Structure of μ-[Bis(chloroacetato-*O*)-mercury(II)-κ^2*Hg*]-μ-[1,8-naphthalenediamino-κ^2*NN′*:κ^2*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

(*Received* 8 *October* 1992; *accepted* 11 *February* 1993)

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.

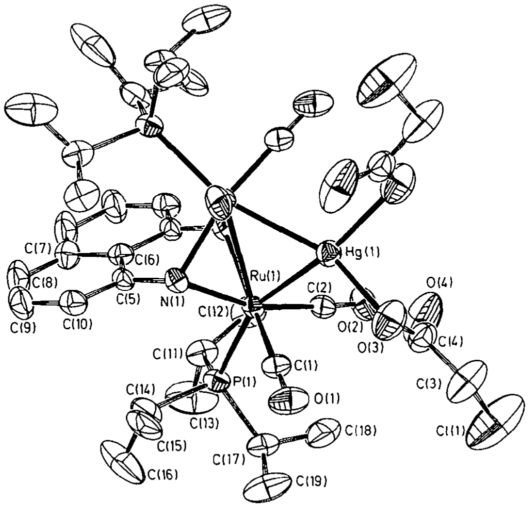

Fig. 1. An *EUCLID* (Spek, 1982) plot, showing the molecular symmetry and atomic numbering scheme.

Experimental

Crystal data

$[HgRu_2(C_2H_2ClO_2)_2(C_{10}H_8N_2)(C_9H_{21}P)_2(CO)_4]$
$M_r = 1178.42$
Monoclinic
$C2/c$
$a = 23.586\ (8)$ Å
$b = 12.584\ (2)$ Å
$c = 14.540\ (5)$ Å
$\beta = 93.83\ (3)°$
$V = 4306.0\ (2)$ Å^3
$Z = 4$
$D_x = 1.818$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 15$-$17°$
$\mu = 4.482$ mm^{-1}
$T = 293$ K
Prismatic
$0.40 \times 0.33 \times 0.10$ 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 $[I>3\sigma(I)]$

$R_{int} = 0.027$
$\theta_{max} = 25°$
$h = -27 \rightarrow 27$
$k = 0 \rightarrow 14$
$l = 0 \rightarrow 17$
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
$w = 1/[\sigma^2(F)+0.0004F^2]$

$(\Delta/\sigma)_{max} = 0.04$
$\Delta\rho_{max} = 0.90$ 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 *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]

References

Andreu, P. L., Cabeza, J. A., Riera, V., Robert, F. & Jeannin, Y. (1989). *J. Organomet. Chem.* **372**, C15–C18.

Cabeza, J. A., Fernández-Colinas, J. M., García-Granda, S., Riera, V. & Van der Maelen Uría, J. F. (1991). *J. Chem. Soc. Chem. Commun.* pp. 168–170.

Cabeza, J. A., Fernández-Colinas, J. M., García-Granda, S., Riera, V. & Van der Maelen Uría, J. F. (1992). *Inorg. Chem.* **31**, 1233–1238.

Cabeza, J. A., Fernández-Colinas, J. M., Riera, V., García-Granda, S. & Van der Maelen Uría, J. F. (1991). *Inorg. Chim. Acta*, **185**, 187–192.

Cabeza, J. A., Fernández-Colinas, J. M., Riera, V., Pellinghelli, M. A. & Tiripicchio, A. (1991). *J. Chem. Soc. Dalton Trans.* pp. 371–377.

Grant, D. F. & Gabe, E. J. (1978). *J. Appl. Cryst.* **11**, 114–120.

Lehmann, M. S. & Larsen, F. K. (1974). *Acta Cryst.* A**30**, 580–584.

Nardelli, M. (1983). *Comput. Chem.* **7**, 95–98.

Sheldrick, G. M. (1976). *SHELX*76. Program for crystal structure determination. Univ. of Cambridge, England.

Sheldrick, G. M. (1985). *SHELXS*86. In *Crystallographic Computing* 3, edited by G. M. Sheldrick, C. Krüger & R. Goddard, pp. 175–189. Oxford: Clarendon Press.

Spek, A. L. (1982). *The EUCLID package*. In *Computational Crystallography*, edited by D. Sayre. Oxford: Clarendon Press.

Van der Maelen Uría, J. F. (1991). PhD thesis. Univ. of Oviedo, Oviedo, Spain.

Walker, N. & Stuart, D. (1983). *Acta Cryst.* A**39**, 158–166.

Acta Cryst. (1993). C**49**, 1485–1487

Structure of Diaqua(4,7-diazadecanediamide-N^4,N^7,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

(*Received* 21 *October* 1992; *accepted* 2 *February* 1993)

Abstract

The $\mathrm{Ni^{II}}$ 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 $\mathrm{Ni^{II}}$ ion is six-coordinated in a distorted octahedral geometry, where the quadridentate ligand 4,7-diazadecanediamide coordinates to the $\mathrm{Ni^{II}}$ 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 $\mathrm{NiN_2O_2}$ group is almost planar. The diazadiamide in