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Metal Complexes of 2,4-Diamino-5-(3',4',5'-trimethoxybenzyl)pyrimidine (Trimethoprim) and 2,4-Diamino-5-(*p*-chlorophenyl)-6-ethylpyrimidine (Pyrimethamine).

Part III. Syntheses and X-ray Structures of $[Rh_2(O_2CCH_3)_4(Trimethoprim)_2]\cdot 2C_6H_6\cdot CH_3OH$ and $[Rh_2(O_2CCH_3)_4(Pyrimethamine)_2]$

MARIA ANTONIETTA ZORODDU*

Istituto C.N.R. per l'Applicazione delle Tecniche Chimiche Avanzate ai Problemi Agrobiologici, Via Vienna 2, 07100 Sassari, Italy

LUCIANA NALDINI

Istituto di Chimica Generale ed Inorganica dell'Università, Via Vienna 2, 07100 Sassari, Italy

FRANCESCO DEMARTIN* and NORBERTO MASCIOCCHI

Istituto di Chimica Strutturistica Inorganica, Via G. Venezian 21, 20133 Milan, Italy

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Abstract

The syntheses and structures of $[Rh_2(O_2CCH_3)_4Trim_2]\cdot 2C_6H_6\cdot CH_3OH$ (1) and $[Rh_2(O_2CCH_3)_4Pyr_2]$ (2) are reported. Compound 1 crystallizes in the monoclinic space group $C2/c$ with $a = 24.135(5)$, $b = 15.196(14)$, $c = 16.536(3)$ Å, $\beta = 116.85(2)^\circ$ and $Z = 4$. The Trim ligands coordinate the two axial positions of the dirhodium tetraacetato moiety through the pyrimidinic nitrogen N(1) with a Rh–N(1) bond length of 2.289(2) Å. Compound 2 is monoclinic, space group $C2/m$ with $a = 24.200(6)$, $b = 10.295(3)$, $c = 7.474(2)$ Å, $\beta = 90.61(3)^\circ$ and $Z = 2$. The coordination of the Pyr ligands occurs via the pyrimidinic nitrogen N(3) with a Rh–N(3) distance of 2.365(3) Å. In both complexes the Rh–Rh interaction is 2.409(1) Å.

Introduction

The structure of dirhodium tetracarboxylates and related complexes has been extensively investigated to date, in order to obtain insights into the intermetallic and metal–ligand bonds [1]. Following our program to study the interaction of 2,4-diaminopyrimidine-derived ligands with metal ions [2, 3], we have examined the reaction between $[Rh_2(O_2CCH_3)_4(H_2O)_2]$ and 2,4-diamino-5-(3',4',5'-trimethoxybenzyl)pyrimidine (Trim) or 2,4-diamino-5-(*p*-chlorophenyl)-6-ethylpyrimidine (Pyr) respectively, which are well known biological agents, also employed as metabolic inhibitors [4]. The $[Rh_2(O_2CCH_3)_4Trim_2]\cdot 2C_6H_6\cdot CH_3OH$ (1) and $[Rh_2(O_2CCH_3)_4Pyr_2]$ (2) complexes were obtained and characterized by single crystal X-ray diffraction.

Experimental

$[Rh_2(O_2CCH_3)_4(H_2O)_2]$ was purchased from Fluka A.G., Trimethoprim (Trim) and Pyrimethamine (Pyr) from Sigma Chemical Co. Solvents and reagents were used as received without further purification.

Nitrogen, carbon and hydrogen were analysed using a Perkin-Elmer 240 Elemental Analyser.

Preparation of $[Rh_2(O_2CCH_3)_4Trim_2]$

Dropwise addition of a methanolic solution of Trim (1 mmol, 30 ml) to a solution of $[Rh_2(O_2CCH_3)_4(H_2O)_2]$ (1 mmol, 30 ml) in the same solvent at 40 °C and under magnetic stirring resulted in precipitation of the extremely insoluble microcrystalline powder. Melting point (m.p.) (dec.) > 220 °C. *Anal.* Calc. for $C_{36}H_{48}N_8O_{14}Rh_2$: C, 42.28; H, 4.73; N, 10.95; Found: C, 42.50; H, 4.78; N, 10.99%.

$[Rh_2(O_2CCH_3)_4Trim_2]\cdot 2C_6H_6\cdot CH_3OH$ (1): garnet-red crystals suitable for X-ray structural analysis were obtained by slow diffusion of the methanolic solution of $[Rh_2(O_2CCH_3)_4(H_2O)_2]$ (1 mmol, 50 ml) into a methanolic solution of Trim (1 mmol, 50 ml) after adding benzene (50 ml) and standing for one day. m.p. (dec.) > 220 °C. *Anal.* Calc. for $C_{49}H_{64}N_8O_{15}Rh_2$: C, 48.6; H, 5.33; N, 9.25; Found: C, 49.0; H, 5.50; N, 9.39%.

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*Authors to whom correspondence should be addressed.

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TABLE I. Crystal Data and Intensity Collection Parameters

Compound	1	2
Formula	$C_{36}H_{48}N_8O_{14}Rh_2 \cdot 2C_6H_6 \cdot CH_3OH$	$C_{32}H_{38}Cl_2N_8O_8Rh_2$
Formula weight (amu)	1210.91	939.42
Crystal system	monoclinic	monoclinic
Space group	$C2/c$	$C2/m$
a (Å)	24.135(5)	24.200(6)
b (Å)	15.196(14)	10.295(3)
c (Å)	16.536(3)	7.474(2)
β (°)	116.85(2)	90.61(3)
U (Å^3)	5411(8)	1862(2)
Z	4	2
D_{calc} (g cm^{-3})	1.486	1.675
μ(Mo Kα) (cm^{-1})	6.69	10.74
Minimum transmission factor	–	0.90
Crystal dimensions (mm)	0.1 × 0.1 × 0.15	0.17 × 0.05 × 0.37
Scan mode	ω	ω
ω-scan width (°)	$1.25 + 0.35 \tan\theta$	$1.20 + 0.35 \tan\theta$
θ-range (°)	3–25	3–25
Octants of reciprocal space explored	$\pm h, +k, +l$	$\pm h, +k, +l$
Measured reflections	4923	1849
Unique observed reflections with $I > 3\sigma(I)$	3717	1507
Final R and R_w indices[a]	0.031, 0.042	0.032, 0.050
No. of variables	338	148
e.s.d.[b]	1.236	1.904

[a] $R = [\Sigma(F_o - k|F_c|)/\Sigma F_o]$, $R_w = [\Sigma w(F_o - k|F_c|)^2/\Sigma w F_o^2]^{1/2}$. [b] $\text{e.s.d.} = [\Sigma w(F_o - k|F_c|)^2/(NO - NV)]^{1/2}$.

Preparation of [$Rh_2(O_2CCH_3)_4Pyr_2$]

To a methanolic solution of [$Rh_2(O_2CCH_3)_4$-$H_2O)_2$] (1 mmol, 70 ml) a solution of Pyr in the same solvent (1 mmol, 50 ml) was added. After standing at 4 °C for two days crystals of **2**, suitable for X-ray structural analysis, separated. m.p. (dec.) > 220 °C. *Anal.* Calc. for $C_{32}H_{38}Cl_2N_8O_8Rh_2$: C, 40.9; H, 4.07; N, 11.93; Found: C, 40.7; H, 4.14; N, 11.72%.

All the reported compounds are diamagnetic and scarcely soluble in most organic solvents.

Crystal Data

The crystal data for compounds **1** and **2** are summarized in Table I, together with some experimental details. The diffraction intensities were collected at room temperature on a CAD-4 diffractometer with Mo Kα radiation ($\lambda = 0.71073$ Å) and reduced to F_o values corrected for Lorentz and polarization. The absorption correction was performed for compound **2** by the empirical method described in ref. 5, while no correction was applied in the case of compound **1** because of the isotropic shape of the crystal and the low absorption coefficient. Both structures were solved by conventional Patterson and Fourier methods, and refined by full-matrix least-squares, the minimized function being $\Sigma w(F_o - k|F_c|)^2$; weights assigned to individual observations were $w = 1/\sigma^2(F_o)$, where $\sigma(F_o) = \{\sigma^2(I) + (iI)^2\}^{1/2}/2F_o\text{Lp}$, and i, the 'ignorance factor' equal to 0.05 and 0.04 for **1** and **2** respectively. The Enraf-Nonius SDP package of crystallographic programs was used with the analytical scattering factors, corrected for the real and imaginary components of the anomalous dispersion, taken from ref. 6. Most of the hydrogen atoms were detected in a difference Fourier synthesis but they were all introduced in the model in calculated positions. The structure of compound **2** could be refined either in the centrosymmetric space group $C2/m$ with a statistical disorder of the atoms C(6), C(61) and C(62) lying out of the mirror plane, or ordered in the non-centrosymmetric space group $C2$ in which the mirror is lacking. Any attempt to refine the structure model in the space group $C2$ gave difficulty of convergence, high correlation coefficients and a pattern of bond distances and angles not chemically sound, thus justifying the choice of the centrosymmetric space group $C2/m$.

Results and Discussion

Molecular Structure of [$Rh_2(O_2CCH_3)_4Trim_2$]·$2C_6H_6 \cdot CH_3OH$ (1)

The atomic positional parameters are listed in Table II. The molecular structure is depicted in Fig. 1, together with the atomic labelling scheme. Selected bond distances and angles are given in Table III. Crystals of **1** consist of binuclear [$Rh_2(O_2CCH_3)_4$-$Trim_2$] molecules located on an inversion center

TABLE II. Fractional Atomic Coordinates for Non-hydrogen Atoms of 1

Atom	*x*	*y*	*z*
Rh	0.49264(1)	0.07780(2)	0.48509(2)
O(1)	0.2923(1)	0.1242(2)	0.0479(2)
O(2)	0.2056(1)	0.0464(2)	0.0856(2)
O(3)	0.1760(1)	0.1121(2)	0.2088(2)
O(4)	0.4074(1)	0.0500(2)	0.3827(2)
O(5)	0.5312(1)	0.0621(2)	0.3989(2)
O(6)	0.4541(1)	0.0830(2)	0.5722(2)
O(7)	0.5793(1)	0.0945(2)	0.5902(2)
N(1)	0.4735(1)	0.2240(2)	0.4507(2)
N(2)	0.4945(1)	0.3778(2)	0.4731(2)
N(3)	0.5697(1)	0.2770(2)	0.5529(2)
N(4)	0.4208(1)	0.4809(2)	0.3972(2)
C(1)	0.5109(1)	0.2925(2)	0.4906(2)
C(2)	0.4369(1)	0.3962(2)	0.4106(2)
C(3)	0.3953(1)	0.3281(2)	0.3598(2)
C(4)	0.4168(2)	0.2450(2)	0.3848(2)
C(5)	0.3319(2)	0.3498(3)	0.2834(3)
C(6)	0.2974(1)	0.2701(2)	0.2291(2)
C(7)	0.3112(2)	0.2370(3)	0.1621(3)
C(8)	0.2808(2)	0.1624(3)	0.1137(2)
C(9)	0.2353(2)	0.1225(2)	0.1301(2)
C(10)	0.2215(2)	0.1562(2)	0.1970(2)
C(11)	0.2527(2)	0.2296(3)	0.2466(2)
CM(1)	0.3326(2)	0.1676(4)	0.0197(3)
CM(2)	0.1646(2)	0.0563(3)	−0.0087(3)
CM(3)	0.1638(2)	0.1389(4)	0.2815(3)
CA(1)	0.5504(2)	−0.0126(3)	0.3899(2)
CA(2)	0.5822(2)	−0.0185(3)	0.3287(3)
CA(3)	0.3892(2)	−0.0284(2)	0.3674(2)
CA(4)	0.3242(2)	−0.0450(3)	0.2943(3)

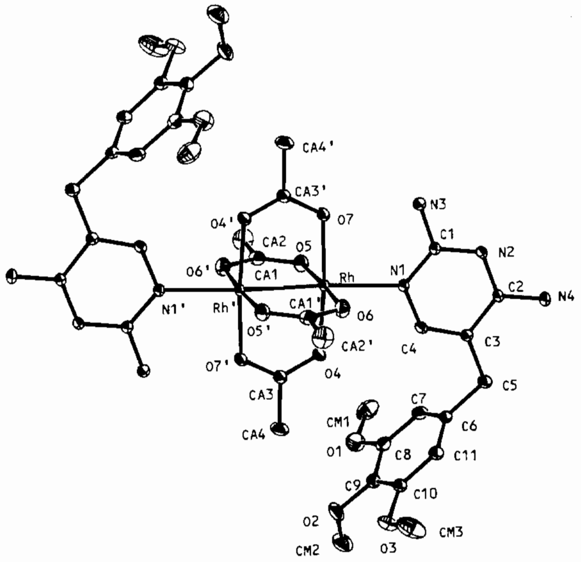

Fig. 1. ORTEP drawing of the $[Rh_2(O_2CCH_3)_4(Trim)_2]$ (1) molecule. Thermal ellipsoids are drawn with 30% probability.

TABLE III. Selected Bond Distances (Å) and Angles (°) for Compound 1

Rh–Rh′	2.409(1)	C(8)–O(1)	1.368(4)
Rh–O(4)	2.032(2)	C(9)–O(2)	1.383(4)
Rh–O(5)	2.037(2)	C(10)–O(3)	1.373(4)
Rh–O(6)	2.040(2)	O(1)–CM(1)	1.417(5)
Rh–O(7)	2.043(2)	O(2)–CM(2)	1.428(5)
Rh–N(1)	2.289(2)	O(3)–CM(3)	1.421(5)
O(4)–CA(3)	1.255(4)		
O(7)′–CA(3)	1.264(4)	Rh′–Rh–N(1)	176.96(6)
CA(3)–CA(4)	1.507(4)	O(4)–Rh–N(1)	88.88(9)
O(5)–CA(1)	1.260(4)	O(4)–Rh–Rh′	88.08(6)
O(6)′–CA(1)	1.270(4)	O(7)–Rh–N(1)	96.02(8)
CA(1)–CA(2)	1.524(5)	O(7)–Rh–Rh′	87.02(6)
N(1)–C(1)	1.339(4)	O(4)–Rh–O(5)	90.2(1)
C(1)–N(2)	1.348(4)	O(4)–Rh–O(6)	89.0(1)
N(2)–C(2)	1.335(4)	O(5)–Rh–O(7)	89.7(1)
C(2)–C(3)	1.422(4)	O(6)–Rh–O(7)	90.7(1)
C(3)–C(4)	1.355(4)	Rh–O(5)–CA(1)	119.8(2)
C(4)–N(1)	1.348(4)	O(5)–CA(1)–O(6)′	125.7(3)
C(1)–N(3)	1.347(4)	CA(1)–O(6)′–Rh′	118.9(2)
C(2)–N(4)	1.333(4)	O(5)–CA(1)–CA(2)	117.1(3)
C(3)–C(5)	1.517(4)	O(6)′–CA(1)–CA(2)	117.2(3)
C(5)–C(6)	1.515(4)	Rh′–O(7)′–CA(3)	120.1(2)
C(6)–C(7)	1.386(5)	O(7)′–CA(3)–O(4)	125.0(3)
C(7)–C(8)	1.391(5)	CA(3)–O(4)–Rh	119.8(2)
C(8)–C(9)	1.384(5)	O(7)′–CA(3)–CA(4)	117.3(3)
C(9)–C(10)	1.390(5)	O(4)–CA(3)–CA(4)	117.7(3)
C(10)–C(11)	1.388(4)	Rh–N(1)–C(1)	128.1(2)
C(11)–C(6)	1.382(5)	Rh–N(1)–C(4)	116.6(2)
		N(1)–C(1)–N(3)	118.9(3)
C(8)–C(9)–C(10)	119.4(3)	N(3)–C(1)–N(2)	116.0(3)
C(9)–C(10)–C(11)	120.3(3)	N(1)–C(1)–N(2)	125.1(3)
C(10)–C(11)–C(6)	120.1(3)	C(1)–N(2)–C(2)	118.0(3)
C(11)–C(6)–C(7)	119.9(3)	N(2)–C(2)–N(4)	117.0(3)
C(8)–O(1)–CM(1)	118.7(3)	N(2)–C(2)–C(3)	121.0(3)
C(9)–O(2)–CM(2)	115.5(3)	C(2)–C(3)–C(4)	115.3(3)
C(10)–O(3)–CM(3)	117.7(3)	C(3)–C(4)–N(1)	125.1(3)
		C(4)–N(1)–C(1)	115.2(3)
		C(2)–C(3)–C(5)	120.7(3)
		C(3)–C(5)–C(6)	113.4(3)
		C(6)–C(7)–C(8)	120.0(3)
		C(7)–C(8)–C(9)	120.3(3)

and solvent molecules packed with normal van der Waals interactions. The four bidentate acetate groups bridge the Rh–Rh bond in the usual fashion giving approximate D_{4h} symmetry to the central moiety of the molecule with the exclusion of the Trim ligands. No unusual features are shown by the dirhodium tetraacetato fragment; average bond distances and angles are: Rh–O 2.038 Å, C–O 1.262 Å, C–C 1.515 Å and O–C–O 125.4°. The Rh–Rh bond length, 2.409(1) Å, is within the range found for previously studied dirhodium carboxylates.

The Trim ligands coordinate to the two axial positions of the tetraacetato dirhodium cage through the pyrimidinic nitrogen N(1). The Rh′–Rh–N(1) linkage only slightly deviates from linearity (176.96-

TABLE IV. Fractional Atomic Coordinates for Non-hydrogen Atoms of 2

Atom	*x*	*y*	*z*
Rh	−0.04362(2)	0.000	0.07615(5)
Cl	−0.43554(9)	0.000	−0.2579(4)
OA(1)	−0.0121(1)	−0.1431(3)	0.2388(4)
OA(2)	0.0691(1)	−0.1401(3)	0.0970(4)
N(1)	−0.1830(3)	0.000	0.4801(9)
N(2)	−0.0912(2)	0.000	0.4923(7)
N(3)	−0.1315(2)	0.000	0.2115(6)
N(4)	−0.1759(2)	0.000	−0.0604(7)
CA(1)	0.0365(2)	−0.1844(5)	0.2124(5)
C(2)	−0.1367(2)	0.000	0.3915(8)
CA(2)	0.0576(2)	−0.2949(6)	0.3257(7)
C(4)	−0.1792(2)	0.000	0.1172(7)
C(5)	−0.2300(3)	0.000	0.1993(9)
C(6)	−0.2306(3)	−0.0412(8)	0.382(1)
C(7)	−0.2830(3)	0.000	0.0879(8)
C(8)	−0.3071(2)	0.1142(5)	0.0341(7)
C(9)	−0.3546(2)	0.1156(5)	−0.0696(7)
C(10)	−0.3767(3)	0.000	−0.1203(9)
C(61)	−0.2813(4)	−0.067(1)	0.486(1)
C(62)	−0.2846(5)	−0.217(1)	0.521(2)

TABLE V. Selected Bond Distances (Å) and Angles (°) for Compound 2

Rh–Rh′	2.409(1)	Rh′–Rh–OA(1)	87.58(6)
Rh–OA(1)	2.052(2)	Rh′–Rh–OA(2)″	87.75(6)
Rh–OA(2)″	2.030(2)	OA(1)–Rh–N(3)	94.41(8)
Rh–N(3)	2.365(3)	OA(2)″–Rh–N(3)	90.24(8)
OA(1)–CA(1)	1.268(4)	Rh′–Rh–N(3)	177.14(8)
OA(2)–CA(2)	1.261(4)	OA(1)–Rh–OA(2)″	175.29(7)
CA(1)–CA(2)	1.504(5)	OA(1)–CA(1)–CA(2)	118.5(3)
N(3)–C(4)	1.346(5)	OA(2)–CA(1)–CA(2)	116.5(3)
C(4)–C(5)	1.381(7)	N(3)–C(4)–C(5)	122.0(4)
C(5)–C(6)	1.433(7)	C(4)–C(5)–C(6)	116.3(4)
C(6)–N(1)	1.421(8)	C(5)–C(6)–N(1)	112.7(5)
N(1)–C(2)	1.308(7)	C(6)–N(1)–C(2)	115.8(5)
C(2)–N(3)	1.352(5)	N(1)–C(2)–N(3)	126.3(4)
C(4)–N(4)	1.330(6)	C(2)–N(3)–C(4)	115.7(4)
C(2)–N(2)	1.328(6)	Rh–N(3)–C(2)	121.3(3)
C(6)–C(61)	1.480(9)	Rh–N(3)–C(4)	123.1(3)
C(61)–C(62)	1.569(13)	N(3)–C(2)–N(2)	118.6(4)
C(5)–C(7)	1.522(6)	N(1)–C(2)–N(2)	115.1(4)
C(7)–C(8)	1.370(5)	N(3)–C(4)–N(4)	117.5(4)
C(8)–C(9)	1.379(5)	C(5)–C(4)–N(4)	120.5(4)
C(9)–C(10)	1.357(4)	N(1)–C(6)–C(61)	117.2(5)
C(10)–Cl	1.747(5)	C(5)–C(6)–C(61)	124.7(6)
		C(6)–C(61)–C(62)	108.1(7)
		C(4)–C(5)–C(7)	120.4(4)
		C(6)–C(5)–C(7)	120.4(4)
		C(5)–C(7)–C(8)	121.0(2)
		C(7)–C(8)–C(9)	121.6(4)
		C(8)–C(9)–C(10)	118.1(4)
		C(9)–C(10)–Cl	118.7(2)
		C(8)–C(7)–C(8)′	118.1(5)
		C(9)–C(10)–C(9)′	122.5(5)

(6)°). The aminic nitrogen N(3) participates in an intramolecular hydrogen bond with O(7) (O(7)... H(31) 2.095(2) Å, O(7)...H(31)–N(3) 155.8(2)°, O(7)...N(3) 2.828(2) Å), thus driving the pyrimidinic ring nearly in one of the dirhodium–diacetato planes, with a dihedral angle between the 'best' plane of atoms Rh–O(4)–CA(3)–O(7′)–Rh′ and the pyrimidinic ring of 6.7°. The latter only slightly deviates from planarity while the substituted phenyl ring C(6), C(11) is planar within three e.s.d.s. Within the Trim ligand equivalent molecular parameters show in general good agreement. As in the free ligand, the C(2)–C(3) bond, involving the carbon atom C(2) with two nitrogen substituents, is significantly longer than the C(3)–C(4) one. The free rotation around the C(3)–C(5) and the C(5)–C(6) bonds gives rise to a conformation for the Trim ligand which can be described by the torsional angles C(3)–C(5)–C(6)–C(11) = 98.4° and C(2)–C(3)–C(5)–C(6) = 172.7°; these values are different from

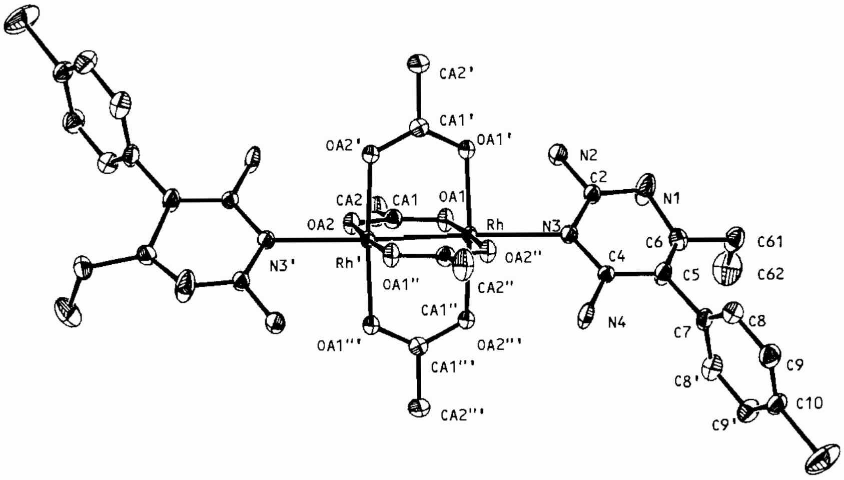

Fig. 2. ORTEP drawing of the $[Rh_2(O_2CCH_3)_4(Pyr)_2]$ (2) molecule. Thermal ellipsoids are drawn with 30% probability. Primed, double primed and triple primed atoms are related to the unprimed ones by the operations $(x, \bar{y}, z)$, $(\bar{x}, \bar{y}, \bar{z})$ and $(\bar{x}, y, \bar{z})$, respectively.

those observed for the free ligand in the solid state as a result of different packing interactions. Nevertheless, the conformation of the O–CH_3 groups is essentially the same as in the free Trim molecule and in the previously studied complexes [2–3, 7] with two of the methoxy groups approximately in the plane of the phenyl ring.

Molecular Structure of $[Rh_2(O_2CCH_3)_4Pyr_2]$ (2)

The atomic positional parameters are listed in Table IV; the molecular structure is reported in Fig. 2, together with the atomic labelling scheme. Selected bond distances and angles are given in Table V. The dirhodium tetraacetato moiety is essentially similar to that of complex **1** with the following average values for equivalent interactions: Rh–O 2.041 Å, C–O 1.264 Å, C–C 1.504(5) Å and O–C–O 125.0(3)°. The Rh–Rh′ distance is the same as that observed in **1**, 2.409(1) Å.

The apically bonded pyrimidinic ring, in contrast with the Trim ligand in **1**, coordinates through N(3) instead of N(1), and lies on a crystallographic mirror with the exception of atom C(6), which is 0.42 Å out of the plane. In the present compound this crystallographic mirror bisects the planes of the acetate groups. Such an orientation, which seems essentially determined by packing effects, prevents π back-donation from the metal to the pyrimidinic ring, and therefore the Rh–N(3) distance observed here, 2.365(3) Å, is larger than the corresponding interaction in **1**, 2.289(2) Å. The pattern of bond distances within the pyrimidinic ring in **2** is essentially comparable to that in **1**: the largest bond length is the C(5)–C(6) one, *i.e.* that between the substituted carbon atoms in the ring; the significative difference between the N(1)–C(6) and N(1)–C(2) bonds could be explained with a probable mispositioning of the N(1) atom. The refined position of N(1) on the crystallographic mirror, with its anomalous thermal ellipsoid (see Fig. 2), was determined, after all attempts of considering any possible disorder failed.

Acknowledgements

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