Crystal structure of [Rh(SnCl₃)(norbornadiene)(dppb)] and [Rh(SnCl₃)(1,5-cyclooctadiene)(dppb)] (dppb = 1,4-bis(diphenylphosphino) butane)

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Abstract

The molecular structure of [Rh(SnCl₃)(norbornadiene)(dppb)] (**1**) and [Rh(SnCl₃)(1,5-cyclooctadiene)(dppb)] (**2**) (dppb = 1,4-bis(diphenylphosphino)butane) has been determined by single-crystal X-ray techniques. In both compounds the structure is best described as distorted square pyramidal with the equatorial positions occupied by the diolefin and the diphosphine respectively and the SnCl₃ fragment in the apical position. Substitution of norbornadiene by 1,5-cyclooctadiene favours slightly the trigonal bipyramidal distortion. The P–C–C and C–C–C angles of the aliphatic chain in dppb are larger than expected for sp³ atoms, specially in **2**. © 1997 Elsevier Science S.A.

1. Introduction

Five-coordinated compounds may adopt trigonal bipyramidal or square pyramidal geometry and both types of structures are well known for pentacoordinated rhodium(I) complexes [1–7]. Diolefinic compounds are trigonal bipyramidal when containing other monodentate [8] or tridentate ligands such as bis(indazol-1-yl)pyridin-2′-ylmethane [9], 2,6-diallylpyridine [10] or X(CH₂PPh₂)₃ (X = MeSi, n-BuSn) [11] though the related compound containing a ligand with lower steric bulk, i.e. MeC(CH₂PPh₂)₃ adopts a square pyramidal structure [11]. Norbornadiene compounds containing bidentate ligands such as chloro(2-(phenylazo)pyridine)(norbornadiene)rhodium(I) [12] or (trichlorostannato)(norbornadiene)(dppp)rhodium(I) (dppp = 1,3-bis(diphenylphosphino)propane) [13] are square pyramidal while an analogous complex with a less strained diolefinic ligand, i.e. (trichlorostannato)(1,5-cyclooctadiene)(dppp)rhodium(I), contains two discrete molecules per asymmetric unit, one best described as distorted trigonal bipyramidal and the other as distorted square pyramidal [14]. In view of

these previous results, we thought it interesting to solve the crystal structure of related compounds containing 1,4-bis(diphenylphosphino)butane, previously synthesised by Uson et al. [15], in order to determine the influence of the bidentate ligands in the molecular geometry.

2. Results and discussion

[Rh(SnCl₃)(NBD)(dppb)] (**1**) and [Rh(SnCl₃)(COD)(dppb)]Et₂O (**2**) were prepared as previously reported [15]. The diffraction study of these compounds gave the atomic parameters in Tables 1 and 2, and selected bond lengths (Å) and angles (°) for both complexes are listed in Table 3. Fig. 1 and Fig. 2 show ORTEP drawings including labelling schemes of the molecular structures of compounds **1** and **2**, respectively [16]. In both compounds the coordination geometry around the rhodium atom may be described as distorted square pyramidal with the equatorial positions occupied by the midpoints of the olefinic ligands, G1 and G2, of the NBD ligand in **1** or the COD ligand in **2**, and by P1 and P2 of the diphosphine, with the SnCl₃ ligand in the apical position.

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Table 1
Atomic coordinates and thermal parameters for [Rh(SnCl$_3$)(NBD)(dppb)] **1**; $U_{eq} = 1/3\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\boldsymbol{a}_i\cdot\boldsymbol{a}_j \times 10^4$

Atom	X	Y	Z	U_{eq}
Rh	0.6974(1)	0.33068(5)	0.81140(5)	324(3)
Sn	0.6215(1)	0.25725(5)	0.91180(5)	421(3)
P1	0.5102(3)	0.4048(2)	0.7867(2)	369(11)
P2	0.8285(3)	0.4150(2)	0.8767(2)	373(11)
Cl1	0.5584(4)	0.3169(2)	1.0104(2)	601(14)
Cl2	0.7706(4)	0.1691(2)	0.9723(2)	670(15)
Cl3	0.4382(5)	0.1732(3)	0.8961(2)	828(18)
C1	0.664(1)	0.2895(7)	0.7069(6)	456(47)
C2	0.622(1)	0.2374(7)	0.7480(7)	457(46)
C3	0.746(2)	0.1880(7)	0.7692(8)	537(53)
C4	0.840(1)	0.2394(7)	0.8159(7)	469(47)
C5	0.879(1)	0.2922(8)	0.7740(7)	473(49)
C6	0.811(1)	0.2727(8)	0.7019(7)	507(51)
C7	0.808(2)	0.1891(8)	0.7054(7)	606(59)
C8	0.446(1)	0.4460(7)	0.8589(7)	472(48)
C9	0.503(1)	0.5189(8)	0.8852(7)	563(54)
C10	0.653(1)	0.5297(7)	0.8893(7)	508(50)
C11	0.745(1)	0.4720(7)	0.9307(7)	461(46)
C12	0.503(1)	0.4773(7)	0.7238(7)	424(45)
C13	0.393(2)	0.5213(8)	0.7119(8)	604(58)
C14	0.378(2)	0.5713(9)	0.6597(9)	781(72)
C15	0.474(2)	0.5773(9)	0.6208(9)	789(76)
C16	0.586(2)	0.5343(9)	0.6319(9)	806(76)
C17	0.598(2)	0.4839(8)	0.6841(7)	568(56)
C18	0.369(1)	0.3489(7)	0.7474(7)	434(45)
C19	0.335(1)	0.3434(9)	0.6782(7)	536(52)
C20	0.236(2)	0.295(1)	0.6488(8)	701(66)
C21	0.170(2)	0.2531(9)	0.6889(8)	674(62)
C22	0.203(1)	0.2579(8)	0.7584(8)	589(57)
C23	0.300(1)	0.3052(8)	0.7876(7)	500(50)
C24	0.926(1)	0.4795(7)	0.8368(7)	428(45)
C25	0.930(1)	0.4765(8)	0.7690(7)	538(53)
C26	1.009(2)	0.5244(9)	0.7382(8)	675(65)
C27	1.083(2)	0.5759(9)	0.777(1)	642(65)
C28	1.082(2)	0.5805(9)	0.845(1)	795(76)
C29	1.003(2)	0.5321(9)	0.8743(8)	731(68)
C30	0.960(1)	0.3697(7)	0.9345(6)	411(44)
C31	1.087(2)	0.360(1)	0.9185(7)	676(64)
C32	1.183(2)	0.320(1)	0.9603(9)	862(79)
C33	1.154(2)	0.289(1)	1.0180(9)	803(74)
C34	1.031(2)	0.299(1)	1.0349(8)	719(68)
C35	0.934(1)	0.3389(8)	0.9934(7)	533(51)

In compound **1** the best least-squares plane for P1, P2, G1, G2 shows a maximum deviation of 0.07(1) for the midpoint G2 with the Rh atom at 0.359(5)Å above this plane towards the Sn atom. This best least-squares plane forms a dihedral angle of 78.9(4)° with the plane containing the olefinic carbons, so that the NBD is orthogonal to the basal plane. The normal to the basal plane and the Rh,Sn line form an angle of 2.4(1)°. In compound **2** the best least-squares plane for P1, P2, G1, G2 shows a maximum deviation of 0.21(1) for the midpoint G1 with the Rh atom at 0.334(1)Å above this plane towards the Sn atom. The best least-squares plane for P1, P2, G1, G2 forms a dihedral angle of 77.9(6)° with the plane containing the olefinic carbons. The

normal to the basal plane and the Rh,Sn line form an angle of 3.4(2)° [17]. These data are indicative of both compounds, and especially the NBD complex (**1**), being closer to the square pyramidal geometry.

The angles P–Rh–(midpoints of the olefinic carbons)$_{trans}$ are 155.1(4) and 158.8(4)° for **1** and 155.9(4) and 167.1(4)° for **2**. Comparing these values with those found for square-planar [Rh(COD)(dppb)]$^+$, containing the ligands present in **2**, 172° and 176° [18]

Table 2
Atomic coordinates and thermal parameters for [Rh(SnCl$_3$)(COD)(dppb)]Et$_2$O **2** $U_{eq} = 1/3\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\boldsymbol{a}_i\cdot\boldsymbol{a}_j \times 10^4$

Atom	X	Y	Z	U_{eq}
Rh	0.41514(6)	0.36977(5)	0.84513(5)	238(3)
Sn	0.39530(5)	0.38633(5)	0.72931(4)	333(3)
Cl1	0.2722(2)	0.3933(3)	0.6811(2)	604(16)
Cl2	0.4469(3)	0.4797(3)	0.6758(2)	762(21)
Cl3	0.4502(3)	0.3065(3)	0.6624(2)	681(19)
P1	0.3202(2)	0.2893(2)	0.8532(2)	283(12)
P2	0.3355(2)	0.4549(2)	0.8790(2)	320(12)
C1	0.4892(9)	0.2909(8)	0.8827(7)	376(53)
C2	0.5039(8)	0.2958(7)	0.8232(7)	324(50)
C3	0.5754(9)	0.3263(8)	0.7975(8)	491(61)
C4	0.5716(9)	0.4021(8)	0.7888(7)	478(58)
C5	0.5142(9)	0.4383(8)	0.8259(7)	354(53)
C6	0.5096(8)	0.4308(7)	0.8862(8)	377(55)
C7	0.5629(8)	0.3882(8)	0.9218(8)	513(57)
C8	0.5367(10)	0.3165(8)	0.9314(8)	542(64)
C9	0.2245(9)	0.3098(9)	0.8284(7)	463(59)
C10	0.1761(11)	0.3457(12)	0.8700(11)	902(151)
C11	0.1811(13)	0.4149(14)	0.8810(11)	1203(155)
C12	0.2373(10)	0.4582(8)	0.8519(8)	537(63)
C13	0.3411(9)	0.2154(7)	0.8093(7)	382(52)
C14	0.3774(9)	0.1630(7)	0.8324(7)	419(55)
C15	0.3951(12)	0.1063(10)	0.7982(11)	812(106)
C16	0.3751(13)	0.1046(10)	0.7392(11)	914(95)
C17	0.3360(15)	0.1591(11)	0.7144(7)	852(120)
C18	0.3200(11)	0.2133(7)	0.7489(8)	517(61)
C19	0.3015(9)	0.2542(7)	0.9250(7)	397(52)
C20	0.2445(9)	0.2042(8)	0.9302(7)	429(56)
C21	0.2286(11)	0.1799(9)	0.9875(9)	612(73)
C22	0.2627(14)	0.1997(11)	1.0357(9)	727(87)
C23	0.3190(12)	0.2470(11)	1.0316(7)	646(76)
C24	0.3353(10)	0.2730(8)	0.9758(8)	481(62)
C25	0.3253(9)	0.4653(7)	0.9596(8)	413(54)
C26	0.2681(10)	0.5037(9)	0.9830(8)	512(63)
C27	0.2578(11)	0.5096(9)	1.0448(9)	555(69)
C28	0.3088(12)	0.4787(8)	1.0807(8)	570(69)
C29	0.3667(11)	0.4415(8)	1.0592(8)	549(66)
C30	0.3748(9)	0.4362(8)	0.9989(7)	458(58)
C31	0.3681(9)	0.5358(7)	0.8562(6)	373(50)
C32	0.4104(10)	0.5769(7)	0.8932(8)	474(57)
C33	0.4399(10)	0.6383(8)	0.8723(10)	682(100)
C34	0.4242(11)	0.6603(10)	0.8182(12)	790(111)
C35	0.3806(13)	0.6214(12)	0.7807(10)	895(92)
C36	0.3531(12)	0.5586(9)	0.8005(9)	670(80)
O	0.5335(10)	0.1464(8)	0.9555(8)	1011(34)
C37	0.6402(13)	0.1506(11)	0.8921(11)	885(52)
C38	0.6028(15)	0.1258(13)	0.9451(11)	1075(47)
C39	0.5021(14)	0.1184(13)	1.0078(11)	1000(47)
C40	0.4358(15)	0.0790(13)	0.9973(11)	1024(51)

Table 3

Selected bond distances (Å) and angles (°) with e.s.d. values in parentheses for [Rh(SnCl₃)(NBD)(dppb)] **1** and [Rh(SnCl₃)(COD)(dppb)]Et₂O **2**

1		**2**	
Rh–Sn	2.651(2)	Rh–Sn	2.645(2)
Rh–P1	2.316(3)	Rh–P1	2.337(4)
Rh–P2	2.301(3)	Rh–P2	2.343(4)
Rh–G1	2.09(1)	Rh–G1	2.10(1)
Rh–G2	2.10(1)	Rh–G2	2.16(1)
Rh–C1	2.21(1)	Rh–C1	2.22(2)
Rh–C2	2.20(1)	Rh–C2	2.22(1)
Rh–C4	2.21(1)	Rh–C5	2.27(2)
Rh–C5	2.22(1)	Rh–C6	2.27(1)
Sn–Cl1	2.446(4)	Sn–Cl1	2.431(4)
Sn–Cl2	2.400(4)	Sn–Cl2	2.410(5)
Sn–Cl3	2.394(5)	Sn–Cl3	2.405(5)
C1–C2	1.38(2)	C1–C2	1.36(2)
C4–C5	1.39(2)	C5–C6	1.37(2)
Sn–Rh–P2	97.1(1)	Sn–Rh–P2	98.5(1)
Sn–Rh–P1	97.6(1)	Sn–Rh–P1	94.0(1)
Sn–Rh–G1	104.0(4)	Sn–Rh–G1	105.3(4)
Sn–Rh–G2	99.5(4)	Sn–Rh–G2	98.1(4)
P1–Rh–G1	94.8(4)	P1–Rh–G1	89.0(4)
P1–Rh–G2	158.8(4)	P1–Rh–G2	167.1(4)
P1–Rh–P2	95.5(1)	P1–Rh–P2	93.0(1)
P2–Rh–G1	155.1(4)	P2–Rh–G1	155.8(4)
P2–Rh–G2	95.0(4)	P2–Rh–G2	89.7(4)
G1–Rh–G2	68.9(5)	G1–Rh–G2	83.5(5)

G1 and G2 are the midpoints between the olefinic double bonds C1=C2 and C4=C5 for **1** and C1=C2 and C5=C6 for **2**, respectively.

or in other four-coordinate square planar compounds, [Rh(diolefin)L₂]⁺ⁿ ($n = 1$ or 0), where the differences between these angles fall in the range 0–4° [18–24], we also find that compound **1** is closer to the square pyramidal geometry. The steric effects of the chelate ligands should also be taken into consideration, as can be observed in the values of the P1–Rh–P2 angles.
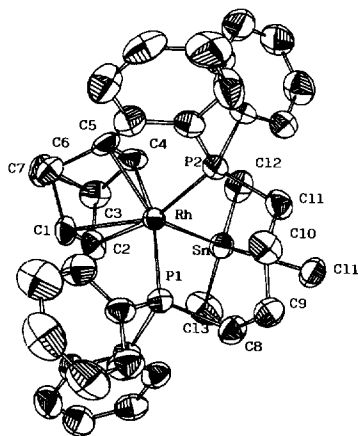

Fig. 1. ORTEP drawing of [Rh(SnCl₃)(NBD)(dppb)] showing the molecular geometry and the labelling scheme (50% probability ellipsoids). The carbon atoms of the phenyl rings are not labelled and the H atoms are omitted for clarity.
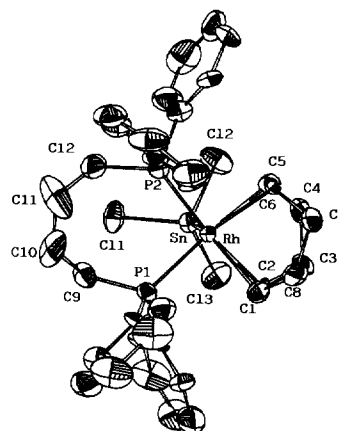

Fig. 2. ORTEP drawing of [Rh(SnCl₃)(COD)(dppb)]Et₂O showing the molecular geometry and the labelling scheme (50% probability ellipsoids). The solvent molecule and the H atoms are omitted for clarity. For this reason the carbon atoms of the phenyl rings are not labelled.

Comparing the corresponding values in **1**, **2**, and related [Rh(SnCl₃)(diolefin)(dppp)] compounds [13,14], we find that for a given diphosphine, COD compounds show smaller P1–Rh–P2 angles than the analogous NBD complexes. However the values of the G1–Rh–G2 angles are equal irrespective of the diphosphine ligand. Also, the small differences in the values of the Rh–P distances, slightly longer for our COD complexes, may be due to these steric effects (see Table 4).

According to published data for pentacoordinated compounds containing diolefinic ligands spanning axial and equatorial positions in a trigonal bipyramidal geometry the distances M–(midpoints of the olefinic carbons) are slightly longer for M–(midpoints of the olefinic carbons)$_{axial}$ than for M–(midpoints of the olefinic carbons)$_{equatorial}$ [9,11,25–27]. These distances are almost equal when the geometry is a square pyramid with both diolefinic groups occupying the basal plane [11,13,14,26]. From all these data we may conclude that in rhodium(I) pentacoordinated complexes, chelating ligands such as diolefins and diphosphines favour the square pyramidal distortion and this tendency is enhanced by norbornadiene when compared to cyclooctadiene. Table 4 collects selected data for the different [Rh(SnCl₃)(diolefin)(diphosphine)] compounds.

It has been suggested that the rhodium–olefin bond may be stronger in five- than in four-coordinate complexes [12]. Comparing the bond distances in **2** and in [Rh(COD)(dppb)]⁺ [18], we find that in **2** the average for Rh–C5 and –C6 and for Rh–C1 and –C2, 2.27(2) and 2.23(2)Å respectively, are identical to the corresponding values in [Rh(COD)(dppb)]⁺.

Most of the features of the diphosphine in compounds **1** and **2** are as expected [18,28,29] however the P–C–C and C–C–C angles of the aliphatic chain of

Table 4

Selected interatomic distances (Å) and bond angles (°) in [Rh(SnCl₃)(diolefin)(diphosphine)] complexes

Diolefin/diphosphine	NBD/dppp[a]	COD/dppp[b1]	COD/dppp[b2]	NBD/dppb[c]	COD/dppb[c]
P2–Rh–P1	91.1(1)	87.0(2)	86.0(2)	95.3(1)	93.0(1)
G1–Rh–G2	68.2(4)	84.5(6)	83.9(5)	68.9(5)	83.5(5)
G2–Rh–P1	156.6(6)	169.1(6)	160.5(5)	158.8(4)	167.1(4)
G1–Rh–P2	159.5(3)	152.8(6)	162.0(5)	155.1(4)	155.8(4)
P–C–C(aliphatic)	114.0(6)	110.2(9)	114.9(9)	111.7(9)	117(1)
	113.2(6)	112.2(9)	113.3(8)	118.3(10)	118(1)
C–C–C(aliphatic)	113.0(7)	110.9(9)	110(1)	117(1)	124(1)
				116(1)	123(2)
Rh–G1	2.13(1)	2.09(1)	2.13(1)	2.09(1)	2.10(1)
Rh–G2	2.11(1)	2.16(1)	2.13(1)	2.10(1)	2.16(1)
Rh–P1	2.297(2)	2.303(3)	2.314(5)	2.316(3)	2.337(4)
Rh–P2	2.295(2)	2.325(3)	2.314(5)	2.301(3)	2.343(4)

[a] In Ref. [13]; [b1] distorted trigonal bipyramid in Ref. [14]; [b2] distorted square pyramid in Ref. [14]; [c] this work.

dppb are larger than expected for sp³ atoms, average 116(1)° for 1 and 121(2)° for 2 (see Table 4) and the largest deviations are observed for the C–C–C angles. This suggests that the four-member aliphatic chain leads to a rather tense steric situation, specially in compound 2, thus accounting for the tendency of this ligand to behave as bridge [18,29,30].

The SnCl₃ features are as expected ([13,14]; Table 3), with average for Sn–Cl of 2.413(4) and 2.415(5) Å in 1 and 2, respectively. The average for Rh–Sn–Cl and Cl–Sn–Cl are 125.2(1) and 94.7(2)° in 1 or 121.5(1) and 94.7(2)° in 2, respectively.

3. Experimental section

The preparation of the metal complexes was carried out as previously reported [15].

3.1. X-ray crystal determinations

Single crystals of [Rh(SnCl₃)(NBD)(dppb)] (1) and [Rh(SnCl₃)(COD)(dppb)]Et₂O (2) were yellow and prismatic and were grown by layering CH₂Cl₂ solutions with diethyl ether. The data were collected on an Enraf–Nonius CAD4 diffractometer for both compounds and unit cell constants were refined by least-squares fitting of the θ values of 25 reflections, with 2θ range of 12–30° for 1 and 2θ range of 13–27° for 2. A summary of the fundamental crystal data for both crystals are given in Table 5. There was no appreciable drop in the intensities of standard reflections monitored every hour. The intensities were corrected for Lorentz and polarization effects. Scattering factors for neutral atoms and anomalous dispersion corrections for Rh, Sn, P and Cl were taken from the International Tables for X-ray Crystallography [31]. Both structures were solved by Patterson and Fourier methods. Empirical absorption corrections [32] were applied at the end of the isotropic

refinements. The maximum and minimum absorption correction factors were 1.117 and 0.830 for 1 and 1.171 and 0.806 for 2, respectively.

For 1, final full-matrix least-squares refinement using unit weights, anisotropic thermal parameters for all

Table 5

Crystal and refinement data for [Rh(SnCl₃)(NBD)(dppb)] 1 and [Rh(SnCl₃)(COD)(dppb)]Et₂O 2

	1	2
Formula	RhSnCl₃P₂C₃₅H₃₆	RhSnCl₃P₂C₄₀H₅₀
M_r	846.57	936.74
Crystal system	monoclinic	orthorhombic
Space group	P2₁/n	Pbca
a, Å	10.098(3)	17.646(4)
b, Å	18.439(2)	20.131(3)
c, Å	20.111(2)	22.459(3)
β, °	99.93(2)	–
V, Å³	3688(1)	7978(2)
Z	4	8
$F(000)$	1688	3792
ρ(calcd), g/cm⁻³	1.52	1.56
Temp., K	295	295
μ, cm⁻¹	14.5	13.4
Cryst. dimens., mm	0.2×0.3×0.15	0.15×0.2×0.2
Diffractometer	Enraf–Nonius CAD4	Enraf–Nonius CAD4
Radiation	Graphite-monochromated Mo K α- ($\lambda = 0.71069$Å)	Graphite-monochromated Mo K α- ($\lambda = 0.71069$Å)
Scan technique	ω–2θ	ω–2θ
Data collected	(−12, 0 0) to (12, 21, 23)	(0, 0, 0) to (21, 23, 26)
θ	1 < θ < 25	1 < θ < 25
Unique data	6456	7005
Unique data (I) ≥ 2σ(I)	3658	3103
R(int) (%)	3.2	–
Std. rflns	3/252	3/243
R_F, %	4.6	5.4
RW_F, %	5.6	6.2
Average shift/error	0.04	0.03

non-H atoms and fixed coordinates and isotropic thermal factors for the hydrogen atoms lead to $R = 4.8\%$. For **2**, some non-resolvable disorder has been found for the atoms of the solvent molecule, therefore these atoms have been refined only isotropically. The remaining non-hydrogen atoms have been refined anisotropically, while fixed coordinates and isotropic thermal factors were employed for the hydrogen atoms. These refinements with unit weights yield an agree factor of $R = 5.4\%$. No trend in ΔF versus (F_0) or $(\sin \theta/\lambda)$ was observed. For **1** the final difference map revealed two peaks of 3.2 eA^{-3} and 2.5 eA^{-3} at the Rh and Sn atoms positions and a third peak of 0.9 eA^{-3} close to Rh and Sn atoms. For **2** the final difference map showed peaks at 3.2 eA^{-3}, 1.9 eA^{-3} and 1.2 eA^{-3}, corresponding to the Rh and Sn atoms positions and close to Rh and Sn atoms, respectively. The remaining peaks show no significant electron density for both compounds. Most of the calculations were carried out with the XRAY80 system [33]. A full list of atomic coordinates, thermal parameters and bond lengths and angles have been deposited at the Cambridge Crystallographic Data Centre.

4. Supplementary material

Tables of observed and calculated structure factors of these compounds can be obtained from the authors.

Acknowledgements

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