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Trichlorostannato(diphosphine)rhodium(I) complexes. Crystal structure of [Rh($SnCl_3$)(1,5-cyclooctadiene)(dppp)]

Victoria García, María A. Garralda and Ricardo Hernández

Facultad de Ciencias Químicas de San Sebastián, Universidad del País Vasco, Apdo. 1072, 20080 San Sebastián (Spain)

M. Angeles Monge

Instituto de Ciencias de Materiales, Sede D, CSIC Laboratorio de Difracción de Rayos X, Facultad de Ciencias Químicas, Universidad Complutense, 28040 Madrid (Spain)

Elena Pinilla

Departamento de Química Inorgánica, Facultad de Ciencias Químicas, Universidad Complutense, 28040 Madrid (Spain)

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Abstract

The molecular structure of [Rh($SnCl_3$)(1,5-cyclooctadiene)(dppp)] [dppp = 1,3-bis(diphenylphosphino)propane] has been determined to $R_F = 3.6\%$ single-crystal X-ray techniques. The crystal contains two discrete molecules **1** and **2** per asymmetric unit. Molecule **1** is best described as distorted trigonal bipyramidal with the diolefin and the diphosphine occupying both apical and equatorial positions and the $SnCl_3$ group on an equatorial position, and molecule **2** as distorted square pyramidal with the equatorial positions occupied by the diolefin and the diphosphine, respectively, and the $SnCl_3$ fragment in the apical position. In solution at room temperature, complexes [Rh($SnCl_3$)(COD)(diphosphine)] exhibit tin dissociation and various intramolecular rearrangements.

Key words: Rhodium; X-ray diffraction; Phosphine complexes; Tin; Trichlorostannato; Diphosphine

1. Introduction

The crystal structures of several pentacoordinate rhodium(I) compounds have been reported [1–3]. Some of them adopt a trigonal bipyramid structure, while others prefer a square pyramidal arrangement around the metal atom. It has been reported that in compounds containing chelating bidentate ligands a particular coordination geometry may be adopted to make a square pyramidal structure stabler with more strained ligands [4]. Recently, we reported the crystal structure of [Rh($SnCl_3$)(NBD)(dppp)], best described as distorted square pyramidal [5], and hence we thought it interesting to solve the crystal structure of a similar compound, [Rh($SnCl_3$)(COD)(dppp)] [6] containing the less strained 1,5-cyclooctadiene instead of norbornadiene, and to study the solution behaviour of this compound and of the related [Rh($SnCl_3$)(COD)(dppb)] previously synthesized by Uson *et al.* [6].

2. Results and discussion

The crystal structure of [Rh($SnCl_3$)(COD)(dppp)] was determined. Atomic parameters are listed in Table 1. The crystal contains two discrete molecules per asymmetric unit. Table 2 contains selected bond distances and angles. The geometry of the molecules is shown separately in Fig. 1 and Fig. 2. There are slight differences. Molecule **1** is best described as distorted

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Correspondence to: Dr. M. A. Garralda, Dr. M. A. Monge.

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TABLE 1. Atomic coordinates for [Rh(SnCl$_3$)(COD)(dppp)] $U_{eq} = 1/3\Sigma_i\Sigma_j U_{ij}.a_i^*.a_j^*.a_i.a_j.10^3$

	x	y	z	U_{eq}
Sn1	0.25877(9)	0.28380(4)	0.47071(6)	38(1)
Rh1	0.31903(9)	0.28822(5)	0.33759(7)	31(1)
Sn2	0.25609(8)	0.22810(4)	0.93972(6)	36(1)
Rh2	0.01349(8)	0.17454(5)	0.87985(7)	28(1)
P11	0.3718(3)	0.4136(2)	0.3388(2)	36(2)
P12	0.5554(3)	0.2795(2)	0.3436(2)	37(2)
P21	−0.0188(3)	0.2689(2)	0.8031(2)	32(2)
P22	0.0973(3)	0.1159(2)	0.7867(2)	34(2)
Cl11	0.2699(4)	0.1695(2)	0.5354(2)	57(2)
Cl12	0.0252(4)	0.2995(2)	0.4976(3)	74(2)
Cl13	0.3622(4)	0.3611(2)	0.5662(3)	70(2)
Cl21	0.4689(3)	0.2596(2)	0.8806(2)	62(2)
Cl22	0.3643(3)	0.1524(2)	1.0269(2)	59(2)
Cl23	0.2795(4)	0.3365(2)	1.0126(3)	70(2)
C11	0.4000(13)	0.1770(7)	0.2820(10)	47(4)
C12	0.2438(12)	0.1681(6)	0.3484(9)	40(7)
C13	0.0885(12)	0.1593(6)	0.3628(9)	51(7)
C14	0.0079(12)	0.2176(7)	0.3242(8)	46(7)
C15	0.0957(13)	0.2909(7)	0.3133(9)	36(7)
C16	0.1662(13)	0.3065(7)	0.2529(10)	43(8)
C17	0.1733(13)	0.2540(7)	0.1924(9)	57(4)
C18	0.2091(13)	0.1763(7)	0.2150(9)	58(4)
C19	0.5033(12)	0.4472(6)	0.4076(8)	36(6)
C110	0.6473(12)	0.4247(7)	0.3900(9)	50(7)
C111	0.6584(12)	0.3414(7)	0.4070(8)	43(7)
C112	0.4352(11)	0.4594(6)	0.2604(9)	33(3)
C113	0.4751(14)	0.5364(8)	0.2649(10)	50(8)
C114	0.5332(15)	0.5697(10)	0.2043(14)	58(11)
C115	0.5452(14)	0.5324(10)	0.1422(12)	56(10)
C116	0.5047(14)	0.4574(9)	0.1402(10)	52(9)
C117	0.4487(12)	0.4217(7)	0.2004(10)	40(8)
C118	0.2249(13)	0.4662(6)	0.3574(11)	34(8)
C119	0.1325(13)	0.4790(7)	0.2999(10)	53(8)
C120	0.0112(14)	0.5154(7)	0.3142(11)	62(8)
C121	−0.0001(14)	0.5391(8)	0.3831(12)	55(4)
C122	0.0952(17)	0.5301(9)	0.4372(11)	82(10)
C123	0.2098(14)	0.4897(8)	0.4244(12)	57(9)
C124	0.6432(12)	0.2926(6)	0.2617(8)	34(7)
C125	0.5766(14)	0.2648(7)	0.1999(11)	46(4)
C126	0.6357(15)	0.2766(7)	0.1352(10)	52(8)
C127	0.7656(15)	0.3184(7)	0.1325(10)	57(8)
C128	0.8300(15)	0.3459(8)	0.1944(12)	62(4)
C129	0.7732(13)	0.3330(7)	0.2595(10)	51(4)
C130	0.6026(12)	0.1906(7)	0.3760(11)	50(8)
C131	0.6439(14)	0.1394(7)	0.3284(9)	56(7)
C132	0.6722(17)	0.0694(9)	0.3572(13)	89(0)
C133	0.6549(17)	0.0532(9)	0.4261(13)	89(0)
C134	0.6129(19)	0.1057(10)	0.4727(12)	102(11)
C135	0.5839(16)	0.1746(9)	0.4467(13)	71(6)
C21	0.0272(12)	0.0869(6)	0.9612(9)	37(7)
C22	−0.0771(11)	0.0629(6)	0.9104(8)	35(6)
C23	−0.2297(12)	0.0604(7)	0.9230(9)	61(7)
C24	−0.2743(13)	0.1333(7)	0.9489(9)	57(7)
C25	−0.1792(12)	0.2016(6)	0.9288(8)	36(6)
C26	−0.0678(13)	0.2281(7)	0.9719(9)	50(4)
C27	−0.0214(13)	0.1929(7)	1.0403(9)	57(4)
C28	−0.0014(13)	0.1110(7)	1.0343(10)	50(4)
C29	0.1276(12)	0.2969(6)	0.7488(8)	42(3)
C210	0.1637(12)	0.2367(7)	0.6964(9)	49(4)
C211	0.2252(12)	0.1716(6)	0.7376(8)	38(6)
C212	−0.0418(12)	0.3568(6)	0.8434(8)	34(6)
C213	0.0746(12)	0.4007(6)	0.8698(8)	39(7)
C214	0.0609(16)	0.4678(7)	0.9023(9)	60(8)
C215	−0.0689(18)	0.4905(8)	0.9101(10)	65(8)
C216	−0.1837(16)	0.4467(9)	0.8819(9)	63(8)
C217	−0.1727(13)	0.3789(7)	0.8491(9)	48(7)
C218	−0.1677(12)	0.2542(7)	0.7421(9)	43(7)
C219	−0.1701(19)	0.2966(11)	0.6794(13)	99(11)
C220	−0.2914(22)	0.2876(11)	0.6326(12)	112(12)
C221	−0.3997(16)	0.2352(10)	0.6498(12)	72(10)
C222	−0.3911(14)	0.1935(10)	0.7085(11)	58(10)
C223	−0.2754(13)	0.2026(7)	0.7565(9)	48(7)
C224	0.1956(11)	0.0392(6)	0.8139(8)	31(6)
C225	0.1438(12)	−0.0329(6)	0.8004(8)	38(7)
C226	0.2217(12)	−0.0899(6)	0.8247(8)	37(7)
C227	0.3475(13)	−0.0751(7)	0.8607(8)	40(7)
C228	0.3971(13)	−0.0021(7)	0.8749(8)	48(7)
C229	0.3222(12)	0.0556(7)	0.8501(8)	47(7)
C230	−0.0236(13)	0.0742(6)	0.7164(11)	40(4)
C231	−0.1568(14)	0.0479(8)	0.7376(9)	61(8)
C232	−0.2559(18)	0.0157(9)	0.6825(12)	89(0)
C233	−0.2117(18)	0.0147(9)	0.6158(12)	89(0)
C234	−0.0839(20)	0.0387(10)	0.5964(12)	99(11)
C235	0.0175(18)	0.0689(8)	0.6476(13)	75(9)

trigonal bipyramidal and molecule **2** as distorted square pyramidal.

In **1** (trigonal bipyramidal), Sn1, P12 and C1516 (midpoint of the C15=C16 bond) occupy the equatorial positions while P11 and C1112 (midpoint of the C11=C12 bond) occupy the axial positions. The difference between Rh1–C1112 (2.16(1) Å) and Rh1–C1516 (2.09(1) Å) is 0.07(1) Å, consistent with the midpoints of the olefin bonds occupying axial and equatorial positions, respectively [7,8]. The slight difference between Rh1–P11 and Rh1–P12 (0.022(3) Å) also confirms the different positions for the P-atoms. The best least-squares plane for P12, Sn1, Rh1, C1516 shows a maximum deviation of 0.01(1) Å for C1516 and this plane forms a dihedral angle of 92.8(3)° with the P11, Rh1, C1112 plane [9].

In **2** (square pyramidal), P21, P22, C2122 (midpoint of C21=C22) and C2526 (midpoint of C25=C26) occupy the basal positions and the Sn2 atom the axial position. Both Rh2–C2122 and Rh2–C2526 are 2.13(1) Å, and both Rh2–P21 and Rh2–P22 are also equal, 2.31(4) Å, supporting the proposed geometry. The best least-squares plane for P21, P22, C2122, C2526 shows a maximum deviation of 0.02(1) for C2526 with the Rh2 atom 0.0362(5) Å above this plane towards the Sn2 atom. The normal to this plane and the Rh2Sn2 line form an angle of 178.8(2)°.

The suggested geometries are consistent with the distances and angles (Table 2) and show that substitu-

TABLE 2. Selected bond distances (Å) and angles (°) with e.s.d.'s in parentheses for $[Rh(SnCl_3)(COD)(dppp)]$

1		2	
Sn1–Rh1	2.650(3)	Sn2–Rh2	2.67(2)
Sn1–Cl11	2.445(4)	Sn2–Cl21	2.43(4)
Sn1–Cl12	2.406(4)	Sn2–Cl22	2.44(4)
Sn1–Cl13	2.430(5)	Sn2–Cl23	2.42(4)
Rh1–P11	2.303(3)	Rh2–P21	2.314(4)
Rh1–P12	2.325(3)	Rh2–P22	2.314(5)
Rh1–C11	2.29(1)	Rh2–C21	2.25(1)
Rh1–C12	2.26(1)	Rh2–C22	2.23(1)
Rh1–C15	2.21(1)	Rh2–C25	2.24(1)
Rh1–C16	2.20(1)	Rh2–C26	2.24(2)
P11–C19	1.85(1)	P21–C29	1.84(1)
P11–C112	1.83(2)	P21–C212	1.83(1)
P11–C118	1.86(1)	P21–C218	1.82(1)
P12–C111	1.85(1)	P22–C211	1.83(1)
P12–C124	1.83(1)	P22–C224	1.84(1)
P12–C130	1.84(1)	P22–C230	1.86(2)
C11–C12	1.42(3)	C21–C22	1.41(2)
C11–C18	1.52(2)	C21–C28	1.52(3)
C12–C13	1.54(2)	C22–C23	1.52(2)
C13–C14	1.55(1)	C23–C24	1.54(2)
C14–C15	1.54(2)	C24–C25	1.54(2)
C15–C16	1.39(2)	C25–C26	1.38(2)
C16–C17	1.53(2)	C26–C27	1.53(2)
C17–C18	1.54(2)	C27–C28	1.54(2)
C19–C110	1.56(2)	C29–C210	1.57(2)
C110–C111	1.57(2)	C210–C211	1.58(2)
Cl12–Sn1–Cl13	94.5(3)	Cl22–Sn2–Cl23	94.3(3)
Cl11–Sn1–Cl13	94.4(3)	Cl21–Sn2–Cl23	95.3(2)
Cl11–Sn1–Cl12	95.5(2)	Cl21–Sn2–Cl22	93.8(2)
Rh1–Sn1–Cl13	128.4(2)	Rh2–Sn2–Cl23	122.0(2)
Rh1–Sn1–Cl12	117.4(3)	Rh2–Sn2–Cl22	117.7(2)
Rh1–Sn1–Cl11	119.3(2)	Rh2–Sn2–Cl21	126.2(3)
Sn1–Rh1–C16	122.8(4)	Sn2–Rh2–C26	82.3(4)
Sn1–Rh1–C15	86.8(4)	Sn2–Rh2–C25	118.0(4)
Sn1–Rh1–C12	79.5(4)	Sn2–Rh2–C22	118.7(4)
Sn1–Rh1–C11	115.3(4)	Sn2–Rh2–C21	83.2(4)
Sn1–Rh1–P12	102.5(3)	Sn2–Rh2–P22	97.0(2)
Sn1–Rh1–P11	93.3(2)	Sn2–Rh2–P21	98.7(2)
C12–Rh1–C16	93.9(5)	C22–Rh2–C26	92.9(6)
C12–Rh1–C15	79.5(5)	C22–Rh2–C25	79.2(5)
C11–Rh1–C16	79.3(6)	C21–Rh2–C26	78.8(6)
C11–Rh1–C15	87.4(5)	C21–Rh2–C25	87.3(5)
P12–Rh1–C16	134.5(5)	P22–Rh2–C26	178.3(4)
P12–Rh1–C15	170.3(6)	P22–Rh2–C25	144.2(4)
P12–Rh1–C12	99.1(4)	P22–Rh2–C22	85.6(4)
P12–Rh1–C11	85.9(4)	P22–Rh2–C21	99.5(4)
P11–Rh1–C16	86.2(4)	P21–Rh2–C26	95.7(4)
P11–Rh1–C15	95.6(4)	P21–Rh2–C25	86.7(4)
P11–Rh1–C12	171.4(5)	P21–Rh2–C22	142.4(4)
P11–Rh1–C11	151.4(6)	P21–Rh2–C21	173.9(4)
P11–Rh1–P12	87.0(2)	P21–Rh2–P22	86.0(2)
C1516–Rh1–P11	90.9(4)	C2526–Rh2–P21	91.3(4)
C1516–Rh1–P12	152.8(6)	C2526–Rh2–P22	162.0(5)
C1112–Rh1–P11	169.1(6)	C2122–Rh2–P12	160.5(5)
C1112–Rh1–P12	92.5(4)	C2122–Rh2–P22	92.6(4)
C1516–Rh1–Sn1	104.7(5)	C2526–Rh2–Sn2	100.2(5)
C1112–Rh1–Sn1	97.5(4)	C2122–Rh2–Sn2	100.8(4)
C1112–Rh1–C1516	84.5(6)	C2122–Rh2–C2526	83.9(5)
Rh1–P11–C118	114.4(5)	Rh2–P21–C218	116.0(5)
Rh1–P11–C112	119.3(5)	Rh2–P21–C212	115.3(6)
Rh1–P11–C19	114.0(5)	Rh2–P21–C29	115.4(5)

TABLE 2 (continued)

1		2	
C112–P11–C118	101.4(6)	C212–P21–C218	102.9(6)
C19–P11–C118	102.3(7)	C29–P21–C218	105.0(8)
C19–P11–C112	103.2(6)	C29–P21–C212	100.2(6)
Rh1–P12–C130	113.6(5)	Rh2–P22–C230	120.1(5)
Rh1–P12–C124	116.3(5)	Rh2–P22–C224	112.8(6)
Rh1–P12–C111	115.9(5)	Rh2–P22–C211	115.4(5)
C124–P12–C130	104.3(6)	C224–P22–C230	102.8(6)
C111–P12–C130	99.7(7)	C211–P22–C230	102.4(9)
C111–P12–C124	105.1(6)	C211–P22–C224	100.9(6)
Rh1–C11–C18	112.3(9)	Rh2–C21–C28	114.3(8)
Rh1–C11–C12	70.8(8)	Rh2–C21–C22	71.1(8)
C12–C11–C18	123 (1)	C22–C21–C28	123 (1)
Rh1–C12–C13	110.0(7)	Rh2–C22–C23	112.4(8)
Rh1–C15–C16	71.2(8)	Rh2–C25–C26	72.0(8)
Rh1–C16–C17	111.4(9)	Rh2–C26–C27	111.2(9)
P11–C19–C110	110.2(9)	P21–C29–C210	114.9(9)

tion of norbornadiene (NBD) [5] by cyclooctadiene (COD) (less strained) increases the tendency to adopt the trigonal bipyramidal structure. Both bond distances and angles found for the Rh–SnCl$_3$ group and the COD are as expected [5,7,10,11]. The Rh–Sn distance in **2**, square pyramidal $[Rh(SnCl_3)(COD)(dppp)]$, is slightly longer than that of the equally square pyramidal $[Rh(SnCl_3)(NBD)(dppp)]$ [5] (2.67(2) Å compared to 2.637(1) Å). NBD is the better π-acceptor.

The solution behaviour of $[Rh(SnCl_3)(COD)(dppp)]$ and of $[Rh(SnCl_3)(COD)(dppb)]$ was studied by variable temperature NMR spectroscopy (see Table 3). The corresponding ^{1}H NMR spectra show at +40°C single singlet resonances for the olefinic protons of 1,5-cyclooctadiene. These collapse at room temperature, and are split into two broad resonances by −10°C for the dppp compound and by −30°C for the dppb complex. These spectra remain unaltered down to −60°C.

The ^{31}P-NMR spectra of each compound at +40°C consists of a doublet due to ^{103}Rh coupling and no tin–phosphorus coupling is observed. At −40°C these

TABLE 3. NMR data for complexes in CDCl$_3$ at variable temperature

	$[Rh(SnCl_3)(C_8H_{12})(dppp)]$		$[Rh(SnCl_3)(C_8H_{12})(dppb)]$	
	+40°C	−40°C	+40°C	−40°C
$\delta^{31}P$	16.1	16.1	28.7	29.1
$\Delta\delta^{31}P$	33.1		44.6	
$^1J(^{103}Rh, ^{31}P)$	117	117	122	122
$^2J(^{119}Sn, ^{31}P)$	–	88	–	104
δ^1H (=CH)	3.73	4.46 3.01	3.60	4.43 3.03

$\Delta\delta^{31}P = \delta^{31}P - \delta^{31}P$ (free).

signals are still sharp and tin satellites are observed, indicating trichlorostannate coordination to rhodium. These spectra suggest that different processes take place. The absence of tin satellites from the ^{31}P-NMR spectra at +40°C may indicate tin dissociation, via $SnCl_2$ or $SnCl_3^-$ dissociation. The reported conductivity data [6] and the evidence of halogen scrambling in related $[Rh(SnBr_2Cl)(NBD)(PR_3)_2]$ compounds [12] and of $SnCl_2$ dissociation in $[Rh(SnCl_3)$(diolefin) (diimine)] [13] makes the $SnCl_2$ dissociation most likely. The low solubility of these complexes precluded ^{119}Sn-NMR measurements.

At −40°C any equilibria should be shifted towards the trichlorostannato compound and the corresponding spectra are consistent with square pyramidal geometry with apical $SnCl_3$, as in **2**. Nevertheless, because [Rh$(SnCl_3)$(COD)(dppp)] can adopt either SP or TBP structures, the low temperature spectra also correspond to a trigonal bipyramid such as in **1** undergoing fast Berry pseudorotation, with the $SnCl_3$ group acting as pivot via a structure as in **2**, equilibrating some of the olefinic protons and the phosphine groups, giving rise to two resonances for vinylic protons and one doublet for both phosphine groups [14].

On raising the temperature, these compounds should undergo a further rearrangement to equilibrate all the vinyl resonances through a TBP intermediate containing an apical R group (R=Cl or $SnCl_3$) and with the diphosphine spanning equatorial positions [14]. This intermediate should have a low stability due to ring strain of the diphosphine and this would explain the lower barrier to this rearrangement in the less strained dppb compound. The ^1J(Rh–P) values are as expected for this type of compound, and are slightly higher for the dppb compound containing a seven-membered chelate ring than for the six-membered chelate ring dppp complex [15]. This is also observed in ^2J(Sn–P). Comparison of these values with those reported for analogous norbornadiene compounds [6], shows higher values for NBD than for COD compounds. Higher coupling constants for NBD complexes have also been observed in other trichlorostannato(diolefin) compounds [16].

3. Experimental section

The preparation of the metal complexes was carried out as previously reported [6].

NMR spectra were recorded with an XL-300 Varian spectrometer, ^{1}H (TMS internal standard) and ^{31}P (H_3PO_4 external standard) spectra were measured from $CDCl_3$ solutions at variable temperature in 5 mm tubes.

3.1. X-ray structure determinations

Table 1 shows the final atomic parameters. Crystal data and details of the procedure are given in Table 4. The cell dimensions were refined by least-squares fitting of the θ values of 25 reflections. 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 [17]. The heavy atoms were located from a three-dimensional Patterson map. The positions of the remaining atoms were obtained from the Fourier synthesis, except for C123, C133, C232 and C233 atoms which have been geometrically calculated. An empirical absorption correction [18] was applied at the end of the isotropic refinement. The maximum and minimum absorption correction factors were 1.098 and 0.996 respectively.

Final mixed refinement was undertaken with isotropic thermal parameters for a few atoms involved in some non-resolvable disorder from the thermal motion and using fixed thermal factors and coordinates for H

TABLE 4. Crystal and Refinement Data for $[Rh(SnCl_3)(C_8H_{12})$(dppp)]

formula	2 $(RhSnCl_3P_2C_{35}H_{34})$
Mr	848.6
crystal system	triclinic
space group	$P\bar{1}$
a, Å	9.753(2)
b, Å	18.327(2)
c, Å	19.20(2)
α°	89.95(4)
β°	92.86(5)
γ°	95.90(2)
V, Å^3	3409(4)
Z	2
$F(000)$	3392
ρ(calcd.), g cm^{-3}	1.65
temp,°C	21
μ, cm^{-1}	15.7
cryst. dimens, mm	0.2×0.2×0.15
diffractometer	Enraf-Nonius CAD4
radiation	graphite-monochromated Mo Kα (λ = 0.71069 Å)
scan technique	$\Omega/2\theta$
θ lim	$1 < \theta < 22$
data collected	(−10, −19, 0) to (10, 19, 20)
unique data	5289
unique data (I) ≥ 2σ(I)	3972
standard reflections	3/64 rflns
R (int), %	1.4
R_F, %	3.6
Rw_F, %	3.9
average shift/error	0.10

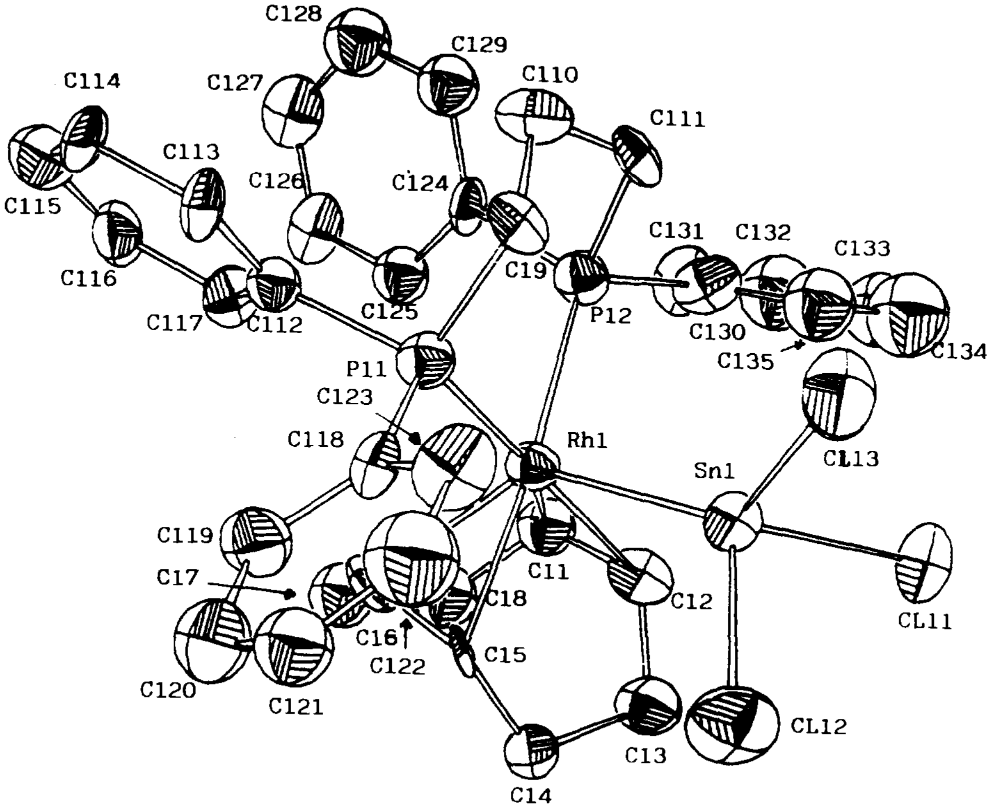

Fig. 1. View of distorted trigonal bipyramidal [Rh(SnCl$_3$)(C$_8$H$_{12}$)(dppp)] (1) showing the numbering scheme.

atoms. No trend in ΔF versus (F_0) or $(\sin \theta/\lambda)$ was observed. Most of the calculations were carried out with the XRAY80 system [19].

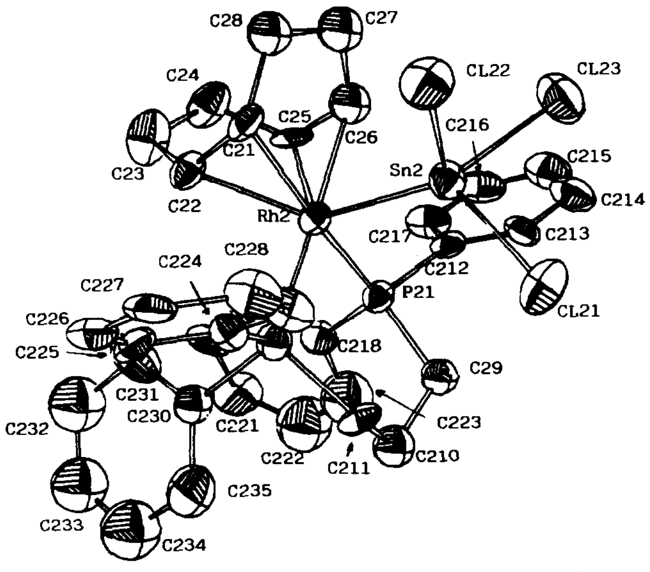

Fig. 2. View of distorted square pyramidal [Rh(SnCl$_3$)(C$_8$H$_{12}$)(dppp)] (2) showing the numbering scheme.

Supplementary material

Thermal parameters, hydrogen parameters and structure factors can be obtained from the authors on request, and all data except structure factors have been deposited with the Cambridge Crystallographic Data Centre.

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