

Journal of Organometallic Chemistry, 427 (1992) 193–200
Elsevier Sequoia S.A., Lausanne
JOM 22407

Crystal structure of $[\text{Rh}(\text{SnCl}_3)(\text{norbornadiene})(\text{dppp})]$. A ^1H NMR study of $[\text{Rh}(\text{SnCl}_3)(\text{norbornadiene})(\text{diphosphine})]$ complexes

María A. Garralda

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

Elena Pinilla

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

and M. Angeles Monge

*Instituto de Ciencias de Materiales, Sede D, C.S.I.C., Laboratorio de Difracción de Rayos X,
Facultad de Ciencias Químicas, Universidad Complutense, 28040 Madrid (Spain)*

(Received June 11, 1991)

Abstract

The molecular structure of $[\text{Rh}(\text{SnCl}_3)(\text{norbornadiene})(\text{dppp})]$ (dppp = 1,3-bis(diphenylphosphino)propane) **1** has been determined by an X-ray diffraction study. The structure is best described 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. The temperature dependence of the ^1H -NMR spectra of $[\text{Rh}(\text{SnCl}_3)(\text{norbornadiene})(\text{diphosphine})]$ complexes indicates that this structure is also the most stable in solution at -30°C .

Introduction

The reactions of halogeno compounds of the transition elements with tin(II) halides have attracted considerable interest mainly due to the potential catalytic activity of the resulting complexes in organic transformations, and their chemistry has been recently reviewed [1]. Several rhodium(I) complexes, containing trichlorostannato fragments and diolefins, have been described [2–8]. Most of

Correspondence to: Dr. M.A. Garralda, Facultad de Ciencias Químicas de San Sebastian, Universidad del País Vasco, Apdo. 1072, 20080 San Sebastián, Spain, or Dr. M.A. Monge, Instituto de Ciencias de Materiales, Sede D, C.S.I.C. Laboratorio de Difracción de Rayos X, Facultad de Ciencias Químicas, Universidad Complutense, 28040 Madrid, Spain.

these are pentacoordinated and they have been assumed to have distorted trigonal bipyramidal structure, with the diolefinic ligands occupying one axial and one equatorial position and undergoing a Berry pseudorotation mechanism at low temperatures that accounts for the observed NMR spectra [2,9,10].

To our knowledge, only two structures of complexes containing the Rh–SnCl₃ fragment have been reported [11,12], and both compounds are dimers. We thus thought it of interest to determine the crystal structure of [Rh(SnCl₃)(C₇H₈)(dppp)] (dppp = 1,3-bis(diphenylphosphino)propane) in order to establish the preferred coordination around the rhodium atom. A variable temperature ¹H-NMR study of [Rh(SnCl₃)(C₇H₈)(diphosphine)] complexes in which diphosphine = dppp or dppb (1,4-bis(diphenylphosphino)butane) is also reported.

Results and discussion

Rh(SnCl₃)(C₇H₈)(dppp) **1**, was prepared as previously reported [2]. The diffraction study gave the atomic parameters of **1** in Table 1 and bond lengths and angles listed in Table 2. The geometry of the molecule is shown in Fig. 1 [13].

When account is taken of earlier reports [9,11,14–16], and our own results for related complexes [17], a careful analyses of the angles and bond lengths suggested that the coordination geometry is best described as distorted square pyramidal, with the equatorial positions occupied by S1 and S2 (S1 and S2 being the centres of the C1=C2 and C4=C5 bonds respectively) and by P1 and P2, with the SnCl₃ ligand in the axial position. A detailed structural study of a related iridium compound, Ir(SnCl₃)(C₇H₈)(PMe₂Ph)₂ has been reported [9], revealing it to have a distorted trigonal bipyramidal structure. Comparison of some selected angles in the two compounds shows that the Sn–Ir–P angles (95.79(4)° and 97.03(4)° respectively) are remarkably close to our Sn–Rh–P angles (96.59(6)° and 99.24(6)° respectively) and this might have been taken to indicate the same kind of structure for both compounds. However, the angles involving the diolefin indicate that the geometry of the rhodium compound is closer to distorted square pyramidal; thus, mutually *cis* groups show the angles: Sn–Rh–S1 = 99.1(3)° and Sn–Rh–S2 = 102.2(3)°, while mutually *trans* groups show the angles: P1–Rh–S2 = 156.6(6)° and P2–Rh–S1 = 159.5(3)°. In contrast, the iridium complex is closer to distorted trigonal bipyramidal: Sn–Ir–B_{ax} = 103.38(16)°, Sn–Ir–A_{ec} = 113.9(17)°, P_{ec}–Ir–A_{ec} = 146.25(18)° and P_{ax}–Ir–B_{ax} = 157.40(16)° (A and B are the centres of the olefinic double bonds). Moreover, the best least-square plane for S1S2P1P2 shows a maximum deviation of 0.093(9) Å for S1, with the Rh atom at 0.353(7) Å above this plane. The C1C2C4C5 atoms are virtually in a plane, with a maximum deviation of 0.002(8) Å for C1, and this plane forms a dihedral angle of 79.8(3)° with the S1S2P1P2 plane [18]. The plane S1S2P1P2 and the Rh–Sn bond are almost perpendicular, 87.80(2)°.

The Rh–S1 and Rh–S2 distances, 2.13(1) and 2.11(1) Å respectively, are equal within experimental error, confirming the equivalence of the positions of the olefin ligands. Similar behaviour has been observed for norbornadiene in analogous pentacoordinated complexes [15]. (When the olefinic double bonds occupy axial and equatorial positions in a trigonal bipyramid the corresponding distances normally differ significantly [9,17].) The Rh–Sn distance, 2.637(1) Å, is in accord with previously reported values [11,12], and is slightly longer than that for an

Table 1

Atomic coordinates for $[\text{Rh}(\text{SnCl}_3)(\text{C}_7\text{H}_8)(\text{dppp})]$ and thermal parameters as $U_{\text{eq}} = \frac{1}{3} \sum_i \sum_j U_{ij} a_i^* a_j^* a_i a_j 10^{-4}$

Atom	x	y	z	U_{eq}
Rh	0.73356(3)	0.38018(4)	0.55702(7)	370(2)
Sn	0.69598(3)	0.31306(3)	0.31819(6)	445(2)
P1	0.77943(10)	0.48280(11)	0.45771(22)	388(7)
P2	0.63570(10)	0.44025(12)	0.59521(22)	405(7)
Cl1	0.77789(13)	0.25468(15)	0.1700(3)	806(11)
Cl2	0.63089(14)	0.20347(15)	0.3390(3)	836(11)
Cl3	0.63278(14)	0.36740(16)	0.12122(26)	783(11)
C1	0.8296(4)	0.3438(5)	0.6581(12)	669(39)
C2	0.8141(4)	0.2968(6)	0.5480(11)	626(37)
C3	0.7740(5)	0.2363(5)	0.6188(10)	613(37)
C4	0.7102(4)	0.2763(5)	0.6600(10)	554(33)
C5	0.7264(5)	0.3238(5)	0.7686(10)	626(37)
C6	0.8009(5)	0.3131(6)	0.7941(11)	755(42)
C7	0.8071(5)	0.2322(6)	0.7672(11)	789(45)
C8	0.7234(4)	0.5381(5)	0.3490(9)	483(30)
C9	0.6615(4)	0.5645(5)	0.4303(10)	493(30)
C10	0.6108(4)	0.5041(5)	0.4546(9)	498(31)
C11	0.8199(4)	0.5483(4)	0.5767(8)	414(29)
C12	0.8433(5)	0.6135(5)	0.5208(10)	627(36)
C13	0.8781(5)	0.6614(5)	0.6093(13)	769(44)
C14	0.8900(4)	0.6457(6)	0.7527(13)	663(41)
C15	0.8680(4)	0.5824(6)	0.8056(10)	613(37)
C16	0.8334(4)	0.5326(5)	0.7199(9)	479(30)
C17	0.8465(4)	0.4636(5)	0.3332(9)	483(31)
C18	0.9110(5)	0.4598(7)	0.3748(1)	940(53)
C19	0.9624(6)	0.4453(10)	0.2897(15)	1379(79)
C20	0.9506(5)	0.4325(7)	0.1479(13)	878(50)
C21	0.8890(6)	0.4348(6)	0.0958(10)	721(42)
C22	0.8353(5)	0.4479(6)	0.1889(10)	650(38)
C23	0.6248(4)	0.4959(4)	0.7574(8)	435(29)
C24	0.6780(4)	0.5091(5)	0.8498(9)	478(31)
C25	0.6701(5)	0.5547(5)	0.9678(9)	546(35)
C26	0.6097(6)	0.5849(5)	0.9939(10)	711(42)
C27	0.5564(5)	0.5709(6)	0.9044(12)	804(45)
C28	0.5633(5)	0.5271(6)	0.7861(10)	663(38)
C29	0.5658(4)	0.3775(5)	0.6082(10)	491(30)
C30	0.5468(5)	0.3507(5)	0.7417(11)	661(39)
C31	0.4960(5)	0.3007(6)	0.7513(13)	767(44)
C32	0.4633(5)	0.2777(6)	0.6284(18)	890(56)
C33	0.4820(5)	0.3036(6)	0.4970(14)	827(48)
C34	0.5333(4)	0.3531(5)	0.4865(10)	598(35)

equatorial Rh–Sn bond in a trigonal bipyramid, 2.587(3) Å [11]. The Rh–P1 and Rh–P2 distances are as expected [16,17].

The SnCl_3 ligand shows the following bond lengths: Sn–Cl1, 2.414(3); Sn–Cl2, 2.414(3) and Sn–Cl3, 2.429(3) Å. The mean Cl–Sn–Cl angle of 95.4(1) is below the 109.4 sp^3 angle, but the mean Rh–Sn–Cl angle of 121.1 is higher. This has been observed for other Rh– SnCl_3 or Ir– SnCl_3 compounds [9,11–12], and is a general feature of SnCl_3 coordination to transition metals [1].

The angles and bond lengths in the norbornadiene ligand are as expected [9,15,17].

A ^{31}P -NMR variable temperature study on both $[\text{Rh}(\text{SnCl}_3)(\text{C}_7\text{H}_8)]$ (diphos-

Table 2

Bond distances (Å) and angles (deg) with e.s.d.s in parentheses

Rh-Sn	2.637(1)	C9-C10	1.53(1)
Rh-P1	2.297(2)	C11-C12	1.39(1)
Rh-P2	2.295(2)	C11-C16	1.38(1)
Rh-C1	2.244(9)	C12-C13	1.39(1)
Rh-C2	2.236(9)	C13-C14	1.38(2)
Rh-C4	2.189(9)	C14-C15	1.34(1)
Rh-C5	2.224(9)	C15-C16	1.39(1)
Sn-C11	2.414(3)	C17-C18	1.36(1)
Sn-C12	2.414(3)	C17-C22	1.38(1)
Sn-C13	2.429(3)	C18-C19	1.36(2)
P1-C8	1.816(8)	C19-C20	1.35(2)
P1-C11	1.820(8)	C20-C21	1.33(2)
P1-C17	1.825(9)	C21-C22	1.41(1)
P2-C10	1.819(9)	C23-C24	1.39(1)
P2-C23	1.833(8)	C23-C28	1.40(1)
P2-C29	1.828(8)	C24-C25	1.39(1)
C1-C2	1.37(1)	C25-C26	1.36(1)
C1-C6	1.50(1)	C26-C27	1.38(2)
C2-C3	1.53(1)	C27-C28	1.37(1)
C3-C4	1.53(1)	C29-C30	1.39(1)
C3-C7	1.52(1)	C29-C34	1.37(1)
C4-C5	1.37(1)	C30-C31	1.38(1)
C5-C6	1.53(1)	C31-C32	1.38(2)
C6-C7	1.51(1)	C32-C33	1.37(2)
C8-C9	1.54(1)	C33-C34	1.38(1)
C4-Rh-C5	36.1(3)	C4-C3-C7	99.2(8)
C2-Rh-C5	76.6(4)	Rh-C4-C3	97.1(6)
C2-Rh-C4	65.1(3)	C3-C4-C5	107.2(8)
C1-Rh-C5	63.8(4)	Rh-C4-C5	73.4(6)
C1-Rh-C4	75.4(3)	Rh-C5-C4	70.5(5)
C1-Rh-C2	35.6(4)	C4-C5-C6	104.9(8)
P2-Rh-C5	91.3(2)	Rh-C5-C6	97.0(6)
P2-Rh-C4	99.3(2)	C1-C6-C5	102.2(8)
P2-Rh-C2	164.4(3)	C5-C6-C7	100.7(8)
P2-Rh-C1	145.1(3)	C1-C6-C7	101.3(8)
P1-Rh-C5	140.0(2)	C3-C7-C6	93.6(8)
P1-Rh-C4	168.7(2)	P1-C8-C9	114.0(6)
P1-Rh-C2	104.6(2)	C8-C9-C10	113.0(7)
P1-Rh-C1	93.6(2)	P2-C10-C9	113.2(6)
P1-Rh-P2	91.1(1)	P1-C11-C16	121.7(6)
Sn-Rh-C5	120.1(2)	P1-C11-C12	119.8(6)
Sn-Rh-C4	84.1(2)	C12-C11-C16	118.3(8)
Sn-Rh-C2	81.4(3)	C11-C12-C13	119.9(9)
Sn-Rh-C1	116.7(3)	C12-C13-C14	121.2(9)
Sn-Rh-P2	96.6(1)	C13-C14-C15	118.6(9)
Sn-Rh-P1	99.2(1)	C14-C15-C16	121.9(9)
Rh-Sn-C13	125.6(1)	C11-C16-C15	120.1(8)
Rh-Sn-C12	118.3(1)	P1-C17-C22	122.5(6)

Table 2 (continued)

Rh-Sn-Cl1	119.6(1)	P1-C17-C18	121.9(7)
Cl2-Sn-Cl3	96.9(1)	C18-C17-C22	115.5(8)
Cl1-Sn-Cl3	96.4(1)	C17-C18-C19	124 (1)
Cl1-Sn-Cl2	93.0(1)	C18-C19-C20	120 (1)
Rh-P1-C17	113.6(3)	C19-C20-C21	120 (1)
Rh-P1-C11	118.7(3)	C20-C21-C22	120.3(9)
Rh-P1-C8	115.5(3)	C17-C22-C21	120.5(9)
C11-P1-C17	100.3(4)	P2-C23-C28	120.2(6)
C8-P1-C17	102.7(4)	P2-C23-C24	120.2(6)
C8-P1-C11	103.8(4)	C24-C23-C28	119.7(8)
Rh-P2-C29	111.9(3)	C23-C24-C25	119.7(8)
Rh-P2-C23	120.5(3)	C24-C25-C26	119.8(8)
Rh-P2-C10	115.5(3)	C25-C26-C27	120.7(9)
C23-P2-C29	101.3(4)	C26-C27-C28	120.5(9)
C10-P2-C29	104.3(4)	C23-C28-C27	119.6(9)
C10-P2-C23	101.1(4)	P2-C29-C34	120.9(7)
Rh-C1-C6	96.9(6)	P2-C29-C30	120.3(7)
Rh-C1-C2	71.9(5)	C30-C29-C34	118.8(8)
C2-C1-C6	107.5(9)	C29-C30-C31	120.4(9)
Rh-C2-C1	72.5(6)	C30-C31-C32	120 (1)
C1-C2-C3	104.8(8)	C31-C32-C33	120 (1)
Rh-C2-C3	95.3(6)	C32-C33-C34	121 (1)
C2-C3-C7	101.1(8)	C29-C34-C33	120.5(9)
C2-C3-C4	102.1(7)		

phine)] complexes was previously reported. The invariability of the spectra below -20°C , showing one doublet flanked by tin satellites in each case, was attributed to a Berry pseudorotation mechanism that is still fast at -90°C [2]. The crystal structure of $\text{Rh}(\text{SnCl}_3)(\text{C}_7\text{H}_8)(\text{dppp})$ described above shows the presence of two equivalent phosphorus atoms, and this could also be the structure in solution at -20°C , responsible for the reported ^{31}P -NMR spectrum. We thus thought it of interest to carry out a ^1H -NMR variable temperature study, since the olefinic and methine norbornadiene protons would not be equivalent in the limiting structure and might provide more information on the behaviour of these complexes in solution.

Above $+30^{\circ}\text{C}$ the ^1H NMR spectra of these complexes show only three signals due to the norbornadiene ligand, centred around 3.7, 3.3 and 1.4 ppm, respectively (see Table 3). At room temperature ($+18^{\circ}\text{C}$) the resonances are broad indicating the slowing down of a dynamic process. As the temperature is lowered further the signals corresponding to the olefinic and methine protons broaden and collapse at *ca.* $+10^{\circ}\text{C}$ for both complexes. By -30°C they are each split into two broad resonances. The methylene signals remain unaltered.

In the light of these results, we conclude that Berry pseudorotation may be responsible for the equilibration of the norbornadiene protons, that the coalescence temperatures are rather high owing to strain destabilization in the transition state in which the diphosphine must occupy two equatorial sites [10], and that the process is slow at -30°C .

As regards the diphosphine ligands, their ^1H -NMR signals in the aliphatic region show different temperature dependence (Table 3). Thus, *dppb* shows two

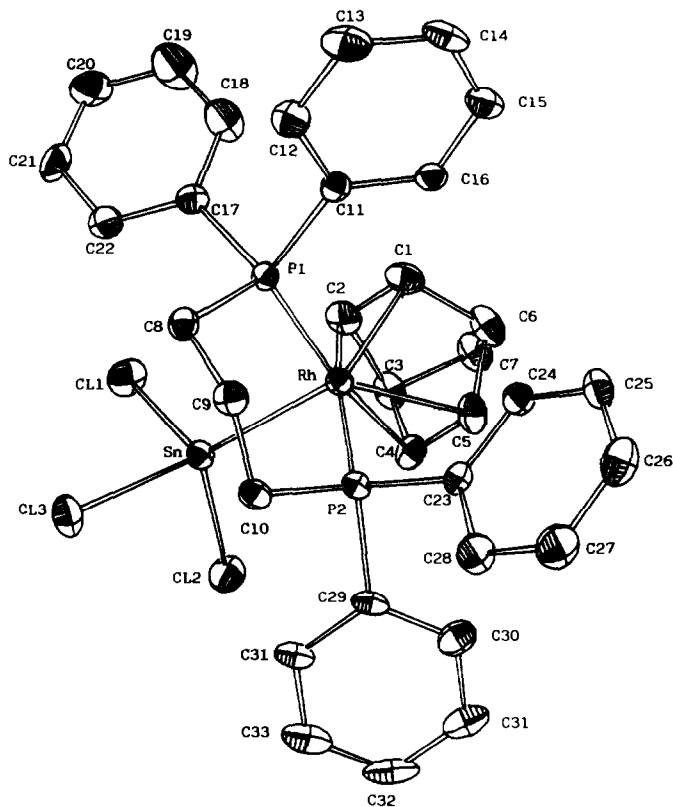


Fig. 1. View of $[\text{Rh}(\text{SnCl}_3)(\text{C}_7\text{H}_8)(\text{dppp})]$ showing the numbering schemes.

broad resonances at $+30^\circ\text{C}$ (markedly displaced with respect to those for the free ligand) that both broaden and collapse at $+10^\circ\text{C}$ to give two broad multiplets at *ca.* -30°C . This temperature dependence suggests the Berry mechanism is also responsible for the equilibration of the methylene protons, though conformational

Table 3

$^1\text{H-NMR}$ data for complexes in CDCl_3 at various temperatures

	$[\text{Rh}(\text{SnCl}_3)(\text{C}_7\text{H}_8)(\text{dppp})]$		$[\text{Rh}(\text{SnCl}_3)(\text{C}_7\text{H}_8)(\text{dppb})]$	
	$+30^\circ\text{C}$	-30°C	$+30^\circ\text{C}$	-30°C
<i>Norbornadiene</i>				
=CH	3.80	4.26	3.69	4.19
		3.36		3.26
$\geq\text{CH}$	3.40	4.05	3.26	3.89
		2.89		2.57
$-\text{CH}_2$	1.40	1.40	1.26	1.26
<i>Diphosphine</i>				
P- CH_2 (free)	3.00 (2.20)	3.06	2.93 (2.0)	3.09
				2.77
C- CH_2 (free)	2.60 (1.60)	2.70	1.38 (1.5)	1.60
				1.04

fluxionality of the seven-membered ring cannot be excluded. On the other hand, the dppp complex shows two broad multiplets for the methylene protons (also displaced with respect to those for the free ligand), that remain unaltered when the temperature is lowered. This is probably due to conformational fluxionality, since six-membered chelate rings may undergo a ring inversion process, still rapid at -30°C in our case, that would equilibrate the axial and equatorial methylene protons [19–20].

Experimental

The metal complexes were prepared as previously reported [2].

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

X-ray structure determinations

Crystal data and details of data collection and refinement are given in Table 4. A fibrous yellow needle-like crystal was mounted in a kappa diffractometer. Three standard reflections measured at intervals shared no significant variation. The cell dimensions refined by least-squares fitting the values of 25 reflections. The

Table 4

Crystal, collection and refinement data for $[\text{Rh}(\text{SnCl}_3)_2(\text{C}_7\text{H}_8)(\text{dppp})]$

Formula	$\text{RhSnCl}_3\text{P}_2\text{C}_{34}\text{H}_{34}$
<i>M</i>	832.54
Crystal system	monoclinic
Space group	$P2_1/n$
<i>a</i> (Å)	20.189(4)
<i>b</i> (Å)	18.373(3)
<i>c</i> (Å)	9.2683(3)
β (deg)	90.69(2)
<i>V</i> (Å ³)	3437.7(9)
<i>Z</i>	4
<i>F</i> (000)	1656
ρ (calc.) (g cm ⁻³)	1.61
Temperature (°C)	21
μ (cm ⁻¹)	15.5
Crystal dimensions (mm)	0.25 × 0.2 × 0.15
Diffractometer	Enraf-Nonius CAD4
Radiation	graphite-monochromated Mo- K_{α} ($\lambda = 0.71069$ Å)
Scan technique	$\omega/2\theta$
Data collected	($-24, 0, 0$) to ($24, 21, 11$)
Unique data	6022
Unique data $I \geq 2\sigma(I)$	3305
<i>R</i> (int) (%)	1.4
<i>R</i> _F (%)	4.0
<i>R</i> _{wF} (%)	4.8
Average shift/error	0.02

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 tables [21]. The heavy atoms were located from a three-dimensional Patterson map. The position of the remaining atoms were obtained from Fourier synthesis. An empirical absorption correction [22] was applied at the end of the isotropic refinement.

In order to prevent bias on ΔF vs. (F_o) or $\sin \theta/\lambda$, the last steps of the refinement were carried out by PESOS program [23] with weights $w = 1/(a + b|F_o|)^2$ and the following coefficients: $|F_o| < 37$, $a = 8.99$, $b = -0.16$; $37 < |F_o| < 458$, $a = 2.09$, $b = -0.002$. Final mixed refinement was with fixed isotropic factors and coordinates for H atoms. Final difference synthesis showed no significantly electron density. Most of the calculations were carried out with the X-RAY80 system [24].

Thermal parameters, hydrogen parameters and structure factors are available from the authors.

Acknowledgment

We thank the DGICYT (0083/87) for financial support.

References

- 1 M.S. Holt, W.L. Wilson and J.H. Nelson, *Chem. Rev.*, 89 (1989) 11.
- 2 R. Usón, L.A. Oro, M.T. Pinillos, K.A. Ostoj-Starzewski and P.S. Pregosin, *J. Organomet. Chem.*, 192 (1980) 227.
- 3 M. Garralda, V. García, M. Kretschmer, P.S. Pregosin and H. Ruegger, *Helvet. Chim. Acta*, 64 (1981) 1150.
- 4 M. Kretschmer, P.S. Pregosin and M.A. Garralda, *J. Organomet. Chem.*, 244 (1983) 175.
- 5 M. Kretschmer, P.S. Pregosin and H. Ruegger, *J. Organomet. Chem.*, 241 (1983) 87.
- 6 R. Usón, L.A. Oro, J. Reyes, D. Carmona and P. Lahuerta, *Trans. Met. Chem.*, 8 (1983) 46.
- 7 V. García, M.A. Garralda and L. Ibarlucea, *Transition Met. Chem.*, 10 (1985) 288.
- 8 V. García, M.A. Garralda and E. Zugasti, *J. Organomet. Chem.*, 332 (1987) 249.
- 9 M.R. Churchill and K.G. Lin, *J. Am. Chem. Soc.*, 96 (1974) 76.
- 10 J.R. Shapley and J.A. Osborn, *Acc. Chem. Res.*, 6 (1973) 305.
- 11 A.L. Balch, H. Hope and F.E. Wood, *J. Am. Chem. Soc.*, 107 (1985) 6936.
- 12 S.G. Bolt, J.C. Machell, M.P. Mingos and M.T. Watson, *J. Chem. Soc., Dalton Trans.*, (1991) 859.
- 13 C.K. Johnson, ORTEP, Report ORNL-3794, Oak Ridge National Laboratory, Tennessee, 1965.
- 14 D.M.T. Chan and T.B. Marder, *Angew. Chem. Ind. Ed.*, (1988) 27.
- 15 J.J. Robertson, A. Kadziola, R.A. Krause and S. Larsen, *Inorg. Chem.*, 28 (1989) 2097.
- 16 M.P. Anderson and L.H. Pignolet, *Inorg. Chem.*, 20 (1981) 4101.
- 17 M. Cano, J.V. Heras, P. Ovejero, E. Pinilla and A. Monge, *J. Organomet. Chem.*, 410 (1991) 101.
- 18 M. Nardelli, A. Musatti, P. Domiano and G.D. Andreetti, *Ric. Sci., Part 2, Sez. A*, 8 (1965) 807.
- 19 A.L. Crumbliss, M.J. Topping, J. Szewczyk, A.T. McPhail and L.D. Quin, *J. Chem. Soc., Dalton Trans.*, (1986) 1895.
- 20 P.A. McNeil, N.K. Roberts and E. Bosnich, *J. Am. Chem. Soc.*, 103 (1981) 2273.
- 21 International Tables for X-ray Crystallography, Vol. IV, Kynoch, Birmingham, 1974, p. 72-98.
- 22 N. Walker and D. Stuart, *Acta Cryst. A*, 39 (1983) 158.
- 23 M. Martinez-Ripoll and F.H. Cano, PESOS program, Instituto Rocasolano, CSIC, Serrano 119, 28006-Madrid, Spain, 1975.
- 24 J.M. Stewart, The XRAY80 System, Computer Science Center, University of Maryland, College Park, 1985.