Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

(η^6-Benzene)dichloro(diallylphenylphosphine)-ruthenium(II), the first structurally characterized complex with the diallylphosphine ligand

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Received 6 September 2001
Accepted 17 September 2001
Online 29 September 2001

Key indicators

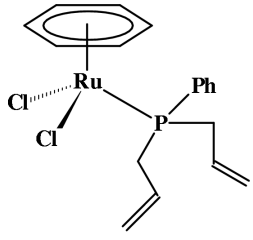
Single-crystal X-ray study
T = 123 K
Mean σ(C–C) = 0.005 Å
R factor = 0.030
wR factor = 0.079
Data-to-parameter ratio = 17.9

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

The molecule of the title complex, [(η^6-C_6H_6)-{$PhP(CH_2CH{=}CH_2)_2$}$RuCl_2$], has the classic piano-stool structure. However, it is noted that the Ru—C distances *trans* to phosphorus are longer than those *trans* to chloride. Such a manifestation of the *trans*-influence is not normally so pronounced in η^6-arene complexes of this type. This is the first structure containing the diallylphenylphosphine ligand.

Comment

Allyl and vinyl phosphines are important precursors for the preparation of more complex phosphine containing ligands (*e.g.* King & Cloyd, 1975). The title compound, (I), was prepared as part of a study on the use of allyl and vinyl phosphines in metal templated additions to secondary phosphines. This compound is an important intermediate and we undertook the X-ray study in an effort to understand aspects of the reactivity of this compound.



(I)

This is the first structural study of a complex with the diallylphosphine ligand. It reveals a classical piano-stool geometry (Fig. 1). The Ru—Cl and Ru—P distances are typical of such a species. The angles subtended at the Ru atom by P and the two Cl atoms are less than 90° [88.15 (5), 83.07 (6) and 87.96 (6)° for Cl1—Ru1—Cl2, Cl1—Ru1—P1 and Cl2—Ru1—P1, respectively], and the remaining coordination sites are occupied by the η^6-benzene ligand. The Ru—C distances reveal a distortion in the π-arene ligand. The distances *trans* to phosphorus at 2.255 (3) and 2.256 (3) Å are markedly longer than the others (2.177–2.190 Å) which are *trans* to Cl, a manifestation of the *trans*-influence. The result is a folding of the arene about the C3—C6 vector with a dihedral angle of 5.1 (4)°, the largest observed in these systems, compared with 5.0° in [(η^6-benzene)$Ru(PMePh_2)Cl_2$] (Bennett *et al.*, 1972), 2.0° in [(η^6-*p*-cymene)$Ru(PMePh_2)Cl_2$] (Bennett *et al.*, 1972), and 1.9° in [{η^6-*o*-C_6H_4(Me)(CO_2-Me)}$RuPPh_2$Neomenthyl)Cl_2] (Bennett *et al.*, 1989). In the complex [(η^6-benzene)$Ru(PPh_3)Cl_2$] (Elsegood & Tocher,

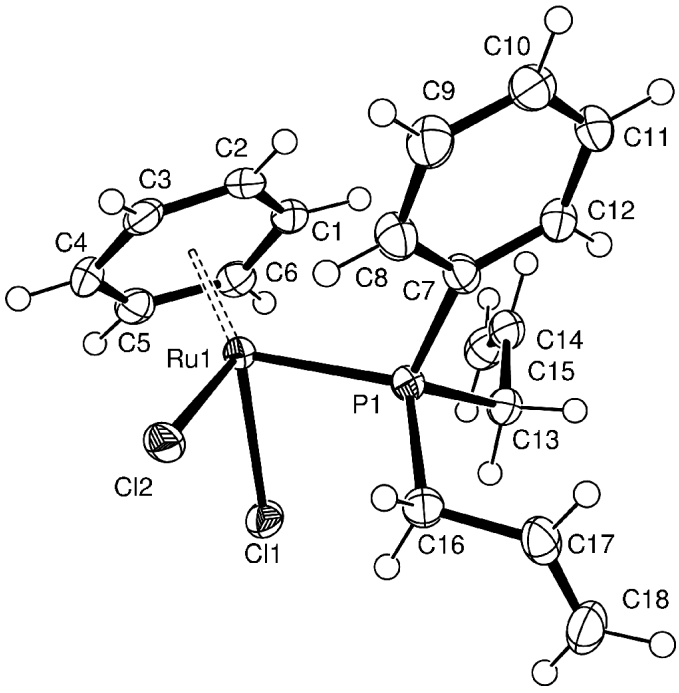


Figure 1
View of the title complex showing the atom-numbering scheme and 50% probability displacement ellipsoids.

1995), no discernible pattern of distortion in the arene can be found.

Despite the asymmetric disposition of the ligand in the solid state with regard to the metal centre, the sharp NMR spectrum, in which both allyl groups are equivalent, suggests that in solution the conformation is, on average, symmetrical, indicating that the allyl groups should both be able to react with a secondary phosphine coordinated to the metal centre, as we desired.

Experimental

$[(\eta^6\text{–}C_6H_6)RuCl_2]_2$ (0.35 g, 1.4 mmol) and excess diallylphenyl phosphine (2.26 g, 11.9 mmol) in toluene (25 ml) were refluxed under N_2 for 4 h. The resulting red solid was filtered off and washed with hexane. Yield 0.42 g, 69%. A sample suitable for X-ray diffraction was obtained as red prisms by slow diffusion of *n*-hexane into a dichloromethane solution of the product.

Crystal data

$[RuCl_2(C_6H_6)(C_{12}H_{15}P)]$
M_r = 440.29
Monoclinic, $P2_1/c$
a = 7.128 (5) Å
b = 9.995 (5) Å
c = 25.55 (2) Å
β = 94.26 (6)°
V = 1815 (2) Å^3
Z = 4
D_x = 1.611 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 25 reflections
θ = 30.5–38.3°
μ = 1.24 mm^{-1}
T = 123 (2) K
Prism, dark red
0.5 × 0.2 × 0.2 mm

Data collection

Rigaku AFC-7*S* diffractometer
ω scans with profile analysis
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.552, T_{max} = 0.780
4311 measured reflections
3991 independent reflections
3178 reflections with $I > 2\sigma(I)$
R_{int} = 0.049
θ_{max} = 27.1°
$h = 0 \rightarrow 9$
$k = 0 \rightarrow 12$
$l = -32 \rightarrow 32$
3 standard reflections
every 150 reflections
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.030
$wR(F^2)$ = 0.079
S = 1.03
3991 reflections
223 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.035P)^2 + 0.8253P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.003
$\Delta\rho_{max}$ = 0.61 e Å^{-3}
$\Delta\rho_{min}$ = −0.51 e Å^{-3}

The benzene H atoms were refined isotropically. All remaining H atoms were included in the riding motion approximation with isotropic displacement parameters equal to $1.2U_{eq}$ of the carrier atom.

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1988); cell refinement: *MSC/AFC Diffractometer Control Software*; data reduction: *TEXSAN* (Molecular Structure Corporation, 1992); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*-3 *for Windows* (Farrugia, 1997); software used to prepare material for publication: WinGX (Farrugia, 1999).

We thank Dr Alan Kennedy for the data collection.

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