| | | | |
|---|---|---|---|
| Cl(2)—Cu—S(2) | 94.0 (1) | C(12)—N(5)—C(15) | 115 (1) |
| C(6)—S(1)—C(7) | 105.1 (5) | | |
| S(1)—C(6)—C(1)—N(1) | −102.3 (9) | C(1)—C(6)—S(1)—C(7) | 61.5 (9) |
| S(2)—C(11)—C(5)—N(1) | 44 (1) | C(5)—C(11)—S(2)—C(12) | 83.6 (8) |

The structure was solved by direct methods (Gilmore, 1984) and successive Fourier syntheses. Refinement was performed using full-matrix least-squares methods, with non-H atoms anisotropic and H atoms in calculated positions with fixed displacement parameters ($1.2 \times B_{eq}$ of the carrying atom). Calculations were performed with *TEXSAN* (Molecular Structure Corporation, 1989) software using a VAXstation 3520 computer. The figures were drawn with *ORTEP*II (Johnson, 1976).

RK thanks Suomen Kulttuurirahasto for a grant.

---

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and bond distances involving H atoms have been deposited with the IUCr (Reference: AB1101). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

## References

Gilmore, C. J. (1984). *J. Appl. Cryst.* **17**, 42–46.
Johnson, C. K. (1976). *ORTEP*II. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.
Molecular Structure Corporation (1989). *TEXSAN. TEXRAY Single Crystal Structure Analysis Software*. Version 5.0. MSC, 3200 Research Forest Drive, The Woodlands, TX 77381, USA.
Teixidor, F., Sánchez-Castelló, G., Lucena, N., Escriche, L., Kivekäs, R., Sundberg, M. & Casabó, J. (1991). *Inorg. Chem.* **30**, 4931–4935.
Offerman, W. & Vögtle, F. (1977). *Synthesis*, pp. 272–273.

---

*Acta Cryst.* (1994). C**50**, 1064–1065

# [$RuCl_2(PH_2Cy)(\eta^6$-*p*-cymene)]

Juan F. Van der Maelen Uría
and Santiago García-Granda

*Departamento de Química Física y Analítica,
Universidad de Oviedo, Avda. Julián Clavería 8,
33006 Oviedo, Spain*

Javier A. Cabeza and Ignacio del Río

*Departamento de Química Orgánica e Inorgánica,
Universidad de Oviedo, Avda. Julián Clavería 8,
33006 Oviedo, Spain*

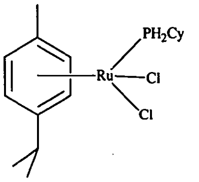
(*Received* 27 *October* 1993; *accepted* 27 *January* 1994)

## Abstract

The structure of dichloro(cyclohexylphosphine)($\eta^6$-*p*-cymene)ruthenium, [$RuCl_2(\eta^6$-$C_{10}H_{14})(C_6H_{13}P)$], is reported. The structure determination shows that the C atoms of the aromatic six-membered ring are almost equidistant from the Ru atom, at distances close to the average value of 2.21 (3) Å. The other metal-ligand distances are Ru—P 2.299 (2), Ru—Cl1 2.411 (1) and Ru—Cl2 2.408 (1) Å. The distances and angles for the Ru environment show a pseudo-octahedral coordination in which the phenyl ring occupies three of the corners of the distorted octahedron.

## Comment

This work is part of a project dealing with the reactivity of binuclear ruthenium complexes (Cabeza, Mulla & Riera, 1994), as the title compound, (I), is a derivative of [$Ru_2Cl_4(\eta^6$-*p*-cymene$)_2$]. The angles formed by the centroid (CZ) of the *p*-cymene phenyl ring and the other Ru ligands are CZ—Ru—P 131.1 (2), CZ—Ru—Cl1 128.6 (2) and CZ—Ru—Cl2 128.5 (2)°. These values and the bond distances and angles of the title compound are in good agreement with those found in similar compounds (Regragui, Dixneuf, Taylor & Carty, 1984; Cabeza, Mulla & Riera, 1994).



(I)

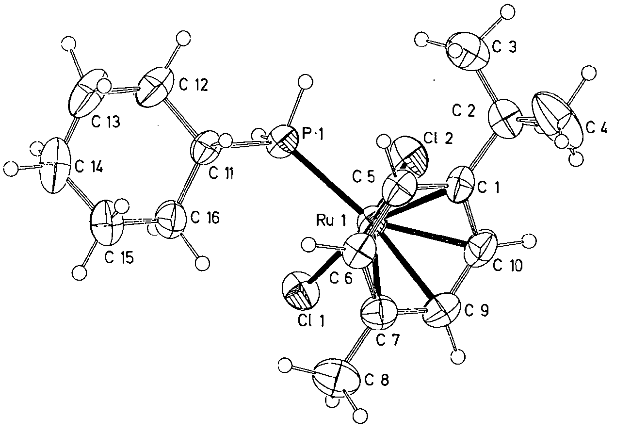


Fig. 1. *EUCLID* (Spek, 1982) plot of the title compound showing the atomic numbering scheme. Displacement ellipsoids are shown at the 50% probability level.

## Experimental

The compound was prepared in quantitative yield from [$Ru_2Cl_4(\eta^6$-*p*-cymene$)_2$] and $PH_2Cy$ following the procedure described for the synthesis of the analogous complex [$RuCl_2(PHPh_2)(\eta^6$-*p*-cymene)] (Regragui, Dixneuf, Taylor & Carty, 1984).

©1994 International Union of Crystallography
Printed in Great Britain – all rights reserved

*Acta Crystallographica Section C*
ISSN 0108-2701 ©1994

### *Crystal data*

$[RuCl_2(C_{10}H_{14})(C_6H_{13}P)]$
$M_r = 422.32$
Orthorhombic
$Pna2_1$
$a = 12.769 (4)$ Å
$b = 15.826 (10)$ Å
$c = 9.431 (10)$ Å
$V = 1905.9 (24)$ Å$^3$
$Z = 4$
$D_x = 1.47$ Mg m$^{-3}$

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 15$-$18°$
$\mu = 1.18$ mm$^{-1}$
$T = 293 (2)$ K
Transparent prismatic block
0.40 × 0.17 × 0.10 mm
Red

### *Data collection*

Enraf-Nonius CAD-4 diffractometer
$\omega$-$2\theta$ scans
Absorption correction: semi-empirical (Walker & Stuart, 1983)
$T_{min} = 0.97$, $T_{max} = 1.13$
3546 measured reflections
3323 independent reflections
2687 observed reflections $[I > 2\sigma(I)]$

$R_{int} = 0.017$
$\theta_{max} = 24.91°$
$h = 0 \rightarrow 15$
$k = 0 \rightarrow 18$
$l = -11 \rightarrow 11$
3 standard reflections
frequency: 60 min
intensity variation: 2%

### *Refinement*

Refinement on $F^2$
$R(F) = 0.0227$
$wR(F^2) = 0.0609$
$S = 1.101$
3323 reflections
223 parameters
Calculated weights $w = 1/[\sigma^2(F_o^2) + (0.0401P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.166$

$\Delta\rho_{max} = 0.316$ e Å$^{-3}$
$\Delta\rho_{min} = -0.341$ e Å$^{-3}$
Extinction correction: none
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)
Absolute configuration: Flack (1983)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å$^2$)

$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j$.

| | $x$ | $y$ | $z$ | $U_{eq}$ |
|---|---|---|---|---|
| Ru1 | 0.25347 (2) | 0.25530 (1) | 0.8887† | 0.04325 (8) |
| P1 | 0.32047 (7) | 0.27755 (6) | 1.1119 (2) | 0.0523 (2) |
| Cl1 | 0.12961 (7) | 0.16651 (6) | 1.0112 (2) | 0.0626 (2) |
| Cl2 | 0.14528 (7) | 0.37288 (6) | 0.9580 (2) | 0.0689 (3) |
| C1 | 0.3330 (3) | 0.3279 (2) | 0.7217 (4) | 0.0548 (8) |
| C2 | 0.3495 (3) | 0.4228 (2) | 0.7048 (5) | 0.0684 (10) |
| C3 | 0.3970 (4) | 0.4649 (3) | 0.8280 (6) | 0.102 (2) |
| C4 | 0.4146 (6) | 0.4358 (3) | 0.5686 (7) | 0.133 (3) |
| C5 | 0.4055 (3) | 0.2728 (2) | 0.7888 (4) | 0.0541 (8) |
| C6 | 0.3875 (3) | 0.1846 (2) | 0.8003 (4) | 0.0538 (8) |
| C7 | 0.2931 (3) | 0.1491 (2) | 0.7450 (4) | 0.0605 (9) |
| C8 | 0.2702 (4) | 0.0572 (3) | 0.7605 (6) | 0.0844 (14) |
| C9 | 0.2224 (4) | 0.2040 (3) | 0.6716 (5) | 0.0665 (11) |
| C10 | 0.2416 (3) | 0.2901 (3) | 0.6614 (5) | 0.0656 (11) |
| C11 | 0.4151 (3) | 0.2054 (2) | 1.1922 (4) | 0.0495 (8) |
| C12 | 0.4444 (4) | 0.2365 (3) | 1.3419 (5) | 0.0780 (13) |
| C13 | 0.5204 (4) | 0.1747 (5) | 1.4137 (5) | 0.094 (2) |
| C14 | 0.4820 (4) | 0.0853 (4) | 1.4130 (5) | 0.093 (2) |
| C15 | 0.4543 (4) | 0.0569 (3) | 1.2645 (5) | 0.0778 (12) |
| C16 | 0.3762 (3) | 0.1149 (2) | 1.1956 (4) | 0.0649 (10) |

† Coordinate fixed to define origin.

Table 2. *Selected geometric parameters* (Å, °)

| | | | |
|---|---|---|---|
| Ru1—C5 | 2.175 (4) | Ru1—C9 | 2.238 (5) |
| Ru1—C1 | 2.198 (4) | Ru1—P1 | 2.299 (2) |
| Ru1—C6 | 2.207 (3) | Ru1—Cl2 | 2.408 (1) |
| Ru1—C7 | 2.217 (4) | Ru1—Cl1 | 2.411 (1) |
| Ru1—C10 | 2.219 (5) | | |
| C5—Ru1—C1 | 37.91 (13) | C10—Ru1—C9 | 36.3 (2) |
| C5—Ru1—C6 | 37.76 (13) | P1—Ru1—Cl2 | 81.18 (5) |
| C6—Ru1—C7 | 37.65 (14) | P1—Ru1—Cl1 | 83.94 (7) |
| C1—Ru1—C10 | 37.77 (15) | Cl2—Ru1—Cl1 | 86.79 (6) |
| C7—Ru1—C9 | 37.5 (2) | | |

Data were collected using profile analysis over all reflections (Lehmann & Larsen, 1974; Grant & Gabe, 1978). The structure was solved by Patterson interpretation using the program *SHELXS*90 (Sheldrick, 1990). An isotropic least-squares refinement using a local version of *SHELX*76 (Sheldrick, 1976; Van der Maelen Uria, 1991) converged to $R = 0.09$, then a semi-empirical absorption correction was applied using *DIFABS* (Walker & Stuart, 1983) and the structure was further refined using the program *SHELXL*93 (Sheldrick, 1994). Anisotropic refinement followed by a difference Fourier synthesis allowed the location of all of the H atoms. All H atoms were refined isotropically with a common displacement parameter and were left riding with the distances to their parent atoms constrained, except for H101 and H102, which were fixed in the positions located by the difference Fourier synthesis. The final $R$ factor in the refinement on $F^2$ was 0.0227, whereas the final $R$ factor in an earlier refinement on $F$ was 0.0221. Further geometrical calculations were made with *PARST* (Nardelli, 1983). Data collection: *CAD*-4 *Software* (Enraf-Nonius, 1989). Cell refinement: *DIRDIF* (Beurskens *et al.*, 1992). Data reduction: *DATAR* (local program). Molecular graphics: *EUCLID* (Spek, 1982). Software used to prepare material for publication: *SHELXL*93.

---

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: NA1074). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

---

## References

Beurskens, P. T., Admiraal, G., Beurskens, G., Bosman, W. P., García-Granda, S., Gould, R. O., Smits, J. M. M. & Smykalla, C. (1992). *The DIRDIF Program System*. Technical Report. Crystallographic Laboratory, Univ. of Nijmegen, The Netherlands.

Cabeza, J. A., Mulla, F. & Riera, V. (1994). *J. Organomet. Chem.* In the press.

Enraf-Nonius (1989). *CAD*-4 *Software*. Version 5.0. Enraf-Nonius, Delft, The Netherlands.

Flack, H. D. (1983). *Acta Cryst.* A**39**, 876-881

Grant, D. F. & Gabe, E. J. (1978). *J. Appl. Cryst.* **11**, 114-120.

Lehmann, M. S. & Larsen, F. K. (1974). *Acta Cryst.* A**30**, 580-584.

Nardelli, M. (1983). *Comput. Chem.* **7**, 95-98.

Regragui, R., Dixneuf, P. H., Taylor, N. J. & Carty, A. J. (1984). *Organometallics*, **3**, 1020-1025.

Sheldrick, G. M. (1976). *SHELX76. Program for Crystal Structure Determination*. Univ. of Cambridge, England.

Sheldrick, G. M. (1990). *Acta Cryst.* A**46**, 467-473.

Sheldrick, G. M. (1994). *J. Appl. Cryst.* In preparation.

Spek, A. L. (1982). *The EUCLID Package. Computational Crystallography*, edited by D. Sayre, p. 528. Oxford: Clarendon Press.

Van der Maelen Uria, J. F. (1991). PhD thesis, Univ. of Oviedo, Spain.

Walker, N. & Stuart, D. (1983). *Acta Cryst.* A**39**, 158-166.