\[
\begin{align*}
\text{Cl(2)} & \text{C-} \text{Ca-} \text{S(2)} & 94.0 (1) \\
\text{C(6)} & \text{S(1)} & \text{C(7)} & 105.1 (5) \\
\text{S(1)} & \text{C(6)} & \text{C(1)} & \text{N(1)} & -102.3 (9) \\
\text{S(2)} & \text{C(11)} & \text{C(5)} & \text{N(1)} & 44 (1) \\
\end{align*}
\]

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 ORTEPII (Johnson, 1976).

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and bond distances involving H atoms have been deposited with the IUCr (Reference: ABI101). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References


[RuCl₂(PH₂Cy)(η⁶-p-cymene)]

JUAN F. VAN DER MAELEN URIA AND SANTIAGO GARCÍA-GRANDA

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

JAVIER A. CABEZA AND IGNACIO DEL RÍO

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

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Abstract

The structure of dichloro(cyclohexylphosphine)(η⁶-p-cymene)ruthenium, [RuCl₂(η⁶-C₁₀H₁₄)(C₆H₁₃P)], 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, Mullá & Riera, 1994), as the title compound, (I), is a derivative of [Ru₂Cl₄(η⁶-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, Mullá & Riera, 1994).

Experimental

The compound was prepared in quantitative yield from [Ru₂Cl₄(η⁶-p-cymene)₂] and PH₂Cy following the procedure described for the synthesis of the analogous complex [RuCl₂(PH₃)(η⁶-p-cymene)] (Regragui, Dixneuf, Taylor & Carty, 1984).
**Crystal data**

\[ \text{[RuCl}_2\text{(C}_6\text{H}_4\text{)(C}_5\text{H}_13\text{P})] \]

\[ M_r = 422.32 \]

Orthorhombic

\[ a = 12.769 \text{ (4) } \AA \]

\[ b = 15.826 \text{ (10) } \AA \]

\[ c = 9.431 \text{ (10) } \AA \]

\[ V = 1905.9 \text{ (24) } \AA^3 \]

\[ Z = 4 \]

\[ D_\text{t} = 1.47 \text{ Mg m}^{-3} \]

\[ \text{Data collection} \]

Enraf-Nonius CAD-4
diffractometer

\[ \omega - 2\theta \text{ scans} \]

Absorption correction:

semi-empirical (Walker & Stuart, 1983)

\[ R_{\text{min}} = 0.97, R_{\text{max}} = 1.13 \]

3546 measured reflections

3323 independent reflections

2687 observed reflections

\[ \{I > 2\sigma(I)\} \]

**Refinement**

Refinement on \( F^2 \)

\[ R(F) = 0.0227 \]

\[ wR(F^2) = 0.0609 \]

\[ S = 1.101 \]

3232 reflections

223 parameters

Calculated weights

\[ w = 1/\sigma^2(F^2) \]

\[ + (0.0401P)^2/3 \]

where \( P = (F^2 + 2F^2)/3 \)

\[ \Delta/\sigma_{\text{max}} = 0.166 \]

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

| \( x \) & \( y \) & \( z \) & \( U_{eq} \) & \( U_{eq} \) |
|-----------------------------|---------|---------|-----------------------------|
| \( \text{Ru1} \) & 0.25347 (2) & 0.25530 (1) & 0.88871 & 0.04325 (8) |
| \( \text{P1} \) & 0.32047 (7) & 0.27755 (6) & 1.1119 (2) & 0.0523 (2) |
| \( \text{C1} \) & 0.12961 (7) & 0.16651 (6) & 1.0112 (2) & 0.0626 (2) |
| \( \text{C2} \) & 0.3495 (3) & 0.4228 (2) & 0.7217 (4) & 0.0548 (8) |
| \( \text{C3} \) & 0.3970 (4) & 0.4649 (3) & 0.7820 (6) & 0.102 (2) |
| \( \text{C4} \) & 0.4166 (6) & 0.4358 (3) & 0.5868 (2) & 0.133 (3) |
| \( \text{C5} \) & 0.4055 (3) & 0.2728 (2) & 0.7888 (4) & 0.0514 (8) |
| \( \text{C6} \) & 0.3875 (4) & 0.1846 (2) & 0.8003 (4) & 0.0538 (8) |
| \( \text{C7} \) & 0.2931 (3) & 0.1491 (2) & 0.7450 (4) & 0.0605 (9) |
| \( \text{C8} \) & 0.2702 (4) & 0.0572 (3) & 0.7605 (6) & 0.0844 (14) |
| \( \text{C9} \) & 0.2224 (4) & 0.2040 (3) & 0.6716 (5) & 0.0665 (11) |
| \( \text{C10} \) & 0.2416 (3) & 0.2901 (3) & 0.6614 (5) & 0.0656 (11) |
| \( \text{C11} \) & 0.4151 (3) & 0.2054 (2) & 1.1922 (4) & 0.0495 (8) |
| \( \text{C12} \) & 0.4444 (4) & 0.3263 (3) & 1.3419 (5) & 0.0780 (13) |
| \( \text{C13} \) & 0.5204 (4) & 0.1747 (5) & 1.4137 (5) & 0.094 (2) |
| \( \text{C14} \) & 0.4820 (4) & 0.0853 (4) & 1.4130 (5) & 0.093 (2) |
| \( \text{C15} \) & 0.4543 (4) & 0.0569 (3) & 1.2645 (5) & 0.0778 (12) |
| \( \text{C16} \) & 0.3762 (3) & 0.1149 (2) & 1.1956 (4) & 0.0649 (10) |

† Coordinate fixed to define origin.

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 SHELX90 (Sheldrick, 1990). An isotropic least-squares refinement using a local version of SHELXTL (Sheldrick, 1976; Van der Maelen Uria, 1991) converged to \( R = 0.097 \), then a semi-empirical absorption correction was applied using DIFABS (Walker & Stuart, 1983) and the structure was further refined using the program SHELXL93 (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: SHELXL93.