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μ-Chloro-μ-hydrido-bis[chloro(η^6-*p*-cymene)-ruthenium(II)](*Ru*—*Ru*): a second polymorph

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The title dinuclear complex, $[Ru_2(C_{10}H_{14})Cl_4H]$, has twofold symmetry. The terminal Cl atom interacts with three C—H H atoms of symmetry-related neighbouring molecules. These interactions play a key role in the packing arrangement.

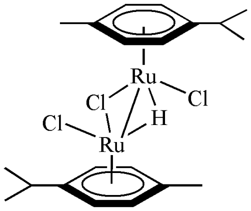
Key indicators

Single-crystal X-ray study
T = 153 K
Mean σ(C–C) = 0.004 Å
R factor = 0.022
wR factor = 0.049
Data-to-parameter ratio = 18.2

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

Comment

The title compound, (I), was first obtained by Bennett & Ennett (1984) from the reaction of $[(p\text{-Me-}C_6H_4\text{-Pr}^i)RuCl(\mu\text{-}Cl)]_2$ with molecular hydrogen; the single-crystal X-ray structure analysis of (I), crystallized from a benzene solution, has been reported by Süss-Fink *et al.* (2000). In contrast with that crystal structure in the orthorhombic space group *Pbca*, we have analysed crystals of (I), which we had obtained from a dichloromethane solution. These crystals belong to the monoclinic space group *C*2/*c*, thus giving rise to a completely different packing arrangement from that in the crystals obtained from benzene. Although the molecular structure (bond lengths and angles) of (I) is almost identical to the earlier reported structure, the intermolecular interactions are quite different.



(I)

The dinuclear title complex, in which the bridging chloride and hydride ligands lie on a twofold axis, is shown in Fig. 1. The metal atom possesses one terminal and one bridging chloride; the bridging Ru—Cl distance is 2.4185 (14) Å, while the terminal Ru—Cl distance is 2.4021 (7) Å. The bridging Ru—Cl distance is shorter than that observed in other arene–ruthenium complexes possessing μ-Cl ligands (McCormick & Gleason, 1988; Gupta *et al.*, 1997; Therrien *et al.*, 1998; Bown & Bennett, 1999; Vieille-Petit *et al.*, 2002). The Ru—Ru distance of 2.9598 (7) Å is in accordance with a metal–metal single bond, and is comparable with the isoelectronic hydrido–trichloro complexes $[(C_5Me_5)_2Rh_2Cl_2(\mu\text{-}Cl)(\mu\text{-}H)]$ (Churchill & Wei-Yang Ni, 1973) and $[(C_5Me_5)_2Ir_2Cl_2(\mu\text{-}Cl)(\mu\text{-}H)]$ (Churchill & Julis, 1977).

Interestingly, the terminal Cl atom interacts with three C—H H atoms of two symmetry-related neighbouring molecules. These interactions are shown in Fig. 2 and summarized in Table 2. In the orthorhombic polymorph (Süss–Fink *et al.*, 2000), the terminal Cl atoms also interact with three C—H H atoms, but in that case with three symmetry-related neighbouring molecules. Furthermore, in the orthorhombic form, the dinuclear complex forms one-dimensional supramolecular chains through π-stacking interactions. Similar interactions are not observed in (I). Instead, the *p*-cymene moiety is surrounded by methyl groups. The closest C atoms are the isopropyl methyl C atoms, 3.77 and 4.31 Å from the benzene ring centroid of the *p*-cymene ligand.

Experimental

The compound was prepared as described by Bennett & Ennett (1984), and this monoclinic form was obtained by crystallization from dichloromethane solution.

Crystal data

$[Ru_2(C_{10}H_{14})Cl_4H]$
M_r = 577.92
Monoclinic, $C2/c$
a = 14.782 (3) Å
b = 17.431 (3) Å
c = 10.425 (2) Å
β = 124.16 (3)°
V = 2222.7 (11) Å^3
Z = 4
D_x = 1.727 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 8000 reflections
θ = 2.0–25.9°
μ = 1.72 mm^{-1}
T = 153 (2) K
Block, orange
0.36 × 0.22 × 0.14 mm

Data collection

Stoe IPDS diffractometer
φ scans
Absorption correction: multi-scan (Blessing, 1995)
T_{min} = 0.500, T_{max} = 0.786
8650 measured reflections
2163 independent reflections
1749 reflections with $I > 2\sigma(I)$
R_{int} = 0.040
θ_{max} = 25.9°
$h = -18 \rightarrow 18$
$k = -21 \rightarrow 21$
$l = -12 \rightarrow 12$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.022
$wR(F^2)$ = 0.049
S = 0.92
2163 reflections
119 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0296P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.44 e Å^{-3}
$\Delta\rho_{min}$ = −0.55 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Cl1—Ru1	2.4185 (14)	Ru1—Ru1^i	2.9598 (7)
Cl2—Ru1	2.4021 (7)	Ru1—H1	1.711 (18)
Ru1^i—Cl2—Ru1	76.06 (3)	Cl2—Ru1—H1	82.1 (10)
Cl2—Ru1—Cl1	90.28 (2)	Cl1—Ru1—H1	87.80 (3)

Symmetry code: (i) $1 - x, y, \frac{3}{2} - z$.

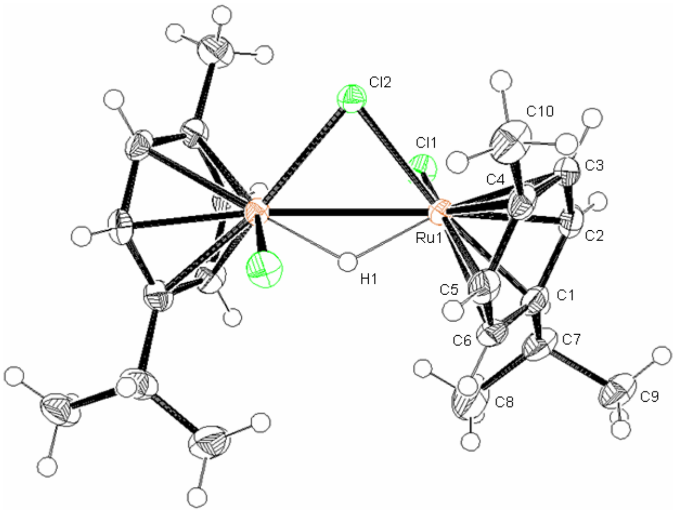


Figure 1
ORTEP-3 (Farrugia, 1997) drawing of (I), showing the atomic numbering scheme. Displacement ellipsoids are drawn at the 50% probability level. Unlabelled atoms are related to labelled atoms by $(1 - x, y, \frac{3}{2} - z)$.

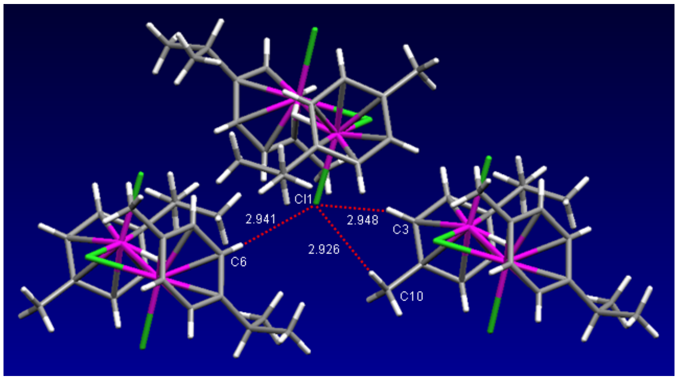


Figure 2
The intermolecular Cl···H interactions (dashed lines) observed in (I) (*MERCURY*; Bruno *et al.*, 2002).

Table 2
Hydrogen-bonding geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C3—H3···Cl1^{ii}	0.93	2.95	3.758 (3)	146
C10—H10B···Cl1^{ii}	0.96	2.93	3.770 (3)	147
C6—H6···Cl1^{iii}	0.93	2.94	3.498 (3)	120

Symmetry codes: (ii) $1 - x, 2 - y, 2 - z$; (iii) $x - \frac{1}{2}, \frac{3}{2} - y, z - \frac{1}{2}$.

The position of the hydride atom was derived from a Fourier difference map and refined, while the remaining H atoms were included in calculated positions (C—H = 0.93–0.98 Å) and refined as riding, with U_{iso}(H) = 1.2 or 1.5 times U_{eq}(parent atom).

Data collection: *EXPOSE* in *IPDS Software* (Stoe & Cie, 2000); cell refinement: *CELL* in *IPDS Software*; data reduction: *INTEGRATE* in *IPDS Software*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*-3 (Farrugia, 1997); software used to prepare material for publication: *SHELXL*97.

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