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Di-μ-chloro-bis[(η^6-benzene)chlororuthenium(II)] chloroform disolvate

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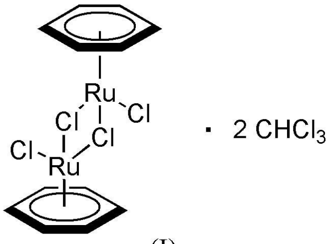
In the centrosymmetric dinuclear title complex, $[RuCl_2(\eta^6\text{-}C_6H_6)]_2\cdot 2CHCl_3$ or $[Ru_2Cl_4(C_6H_6)_2]\cdot 2CHCl_3$, accessible from $RuCl_3.nH_2O$ and 1,3-cyclohexadiene, the benzene ligands are involved in slipped-parallel π–π stacking interactions with neighbouring molecules, thus forming one-dimensional polymeric chains.

Comment

The title compound, (I), which has been known for more than 25 years (Bennett & Smith, 1974) and extensively used as a building block in organometallic chemistry, has never been characterized by X-ray structure analysis. It was crystallized here as the chloroform disolvate.



(I)

The two halves of the dinuclear complex (Fig. 1) are related by a crystallographic inversion center. The bond distances and angles (Table 1) are similar to those of other $[RuCl_2(\eta^6\text{-arene})]_2$ complexes; arene = hexamethylbenzene (McCormick & Gleason, 1988), trindane (Gupta *et al.*, 1997), ethylbenzoate (Therrien *et al.*, 1998), 1,2,3,4-tetrahydronaphthalene (Bown & Bennett, 1999), 1,2-C_6H_4(Me)COOMe (Braga *et al.*, 2001), hexaethylbenzene (Baldwin *et al.*, 2002), indane (Vieille-Petit *et al.*, 2002) and *para*-cymene (Allardyce *et al.*, 2003).

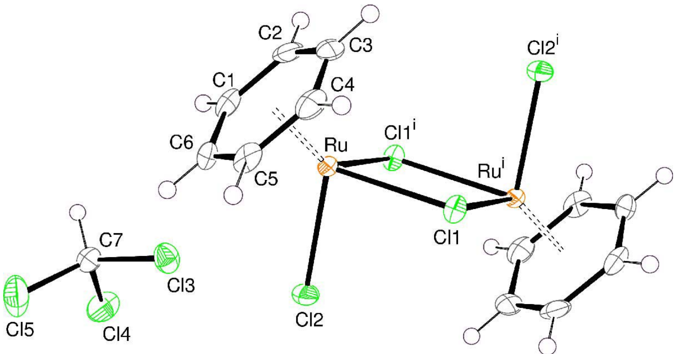


Figure 1
The molecular structure of (I), showing 50% displacement ellipsoids (arbitrary spheres for the H atoms). The symmetry code is as in Table 1. The centrosymmetrically related chloroform molecule is not shown.

In (I), the benzene ring is planar and the Ru–(benzene centroid) distance is 1.646 Å. The metal possesses two bridging and one terminal chloride ions: the bridging Ru—Cl distances are 2.4495 (11) and 2.4580 (9) Å, and the terminal Ru—Cl distance is 2.3911 (9) Å. The benzene ligands are involved in slipped-parallel π–π stacking interactions with neighbouring molecules (Fig. 2) and the shortest intermolecular C···C distance is 3.218 (5) Å. The distance observed between the centers of the interacting π systems (4.07 Å) is in good agreement with the theoretical value calculated for this π stacking mode (Tsuzuki *et al.*, 2002). No meaningful interactions between the dinuclear ruthenium complex and the chloroform molecule are observed.

Experimental

$[RuCl_2(\eta^6\text{–}C_6H_6)]_2$ was dissolved in hot chloroform and orange crystals of (I) suitable for X-ray diffraction analysis were obtained after 3 d by slow evaporation of the solvent.

Crystal data

$[Ru_2Cl_4(C_6H_6)_2]\cdot 2CHCl_3$
$M_r = 738.90$
Triclinic, $P\bar{1}$
$a = 7.9951\ (10)$ Å
$b = 8.1835\ (11)$ Å
$c = 9.6545\ (13)$ Å
$\alpha = 69.312\ (15)^\circ$
$\beta = 69.676\ (15)^\circ$
$\gamma = 86.164\ (16)^\circ$
$V = 553.03\ (15)$ Å^3

$Z = 1$
$D_x = 2.219$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 5113 reflections
$\theta = 2.4\text{–}25.9^\circ$
$\mu = 2.57$ mm^{-1}
$T = 173\ (2)$ K
Block, orange
0.35 × 0.23 × 0.12 mm

Data collection

Stoe IPDS diffractometer
φ scans
Absorption correction: refined from ΔF (*DIFABS*; Walker & Stuart, 1983)
$T_{\min} = 0.397$, $T_{\max} = 0.794$
4350 measured reflections

2014 independent reflections
1817 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.032$
$\theta_{\max} = 25.9^\circ$
$h = -9 \rightarrow 9$
$k = -10 \rightarrow 10$
$l = -11 \rightarrow 11$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.023$
$wR(F^2) = 0.079$
$S = 1.20$
2014 reflections
118 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0458P)^2 + 0.223P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.59$ e Å^{-3}
$\Delta\rho_{\min} = -0.88$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Ru—Cl1	2.4580 (9)	Ru—Cl1^i	2.4495 (11)
Ru—Cl2	2.3911 (9)		
Ru^i—Cl1—Ru	98.22 (3)	Cl1^i—Ru—Cl1	81.78 (3)
Cl2—Ru—Cl1	86.43 (3)		

Symmetry code: (i) $-x, 1-y, 1-z$.

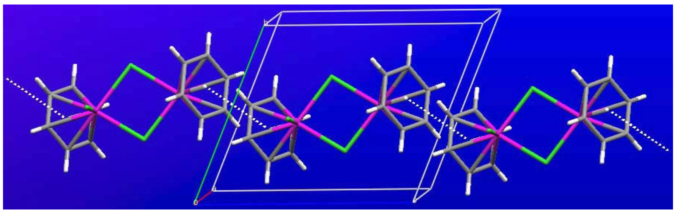


Figure 2
Part of an infinite chain of $[RuCl_2(\eta^6\text{-}C_6H_6)]_2$ molecules in (I), arising through slipped-parallel π–π stacking interactions.

H atoms were placed in calculated positions (C—H = 0.93 Å) and treated as riding atoms with the constraint $U_{iso}(H) = 1.2U_{eq}$(carrier) applied.

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: *SHELXS97* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

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