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Key indicators

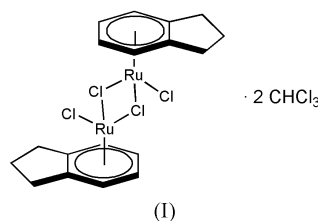
Single-crystal X-ray study
 $T = 153\text{ K}$
Mean $\sigma(\text{C}-\text{C}) = 0.006\text{ \AA}$
 R factor = 0.032
 wR factor = 0.089
Data-to-parameter ratio = 17.4For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.Bis[dichloro- η^6 -indaneruthenium(II)]
dichloroform solvate

In the chloroform solvate of the dinuclear ruthenium complex $[\text{RuCl}_2(\eta^6\text{-indane})]_2$, $\text{C}_{18}\text{H}_{20}\text{Cl}_4\text{Ru}_2 \cdot 2\text{CHCl}_3$, accessible from $\text{RuCl}_3 \cdot n\text{H}_2\text{O}$ and bicyclo[4.3.0]nona-3,6(1)-diene, the indane ligands adopt an envelope conformation toward the ruthenium atoms. The dimeric chloro-bridged molecule has crystallographic inversion symmetry.

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Comment

In the search for new building blocks for the synthesis of catalytically active arene ruthenium clusters (Süss-Fink *et al.*, 2002), the title compound, (I), has been synthesized. $\text{RuCl}_3 \cdot n\text{H}_2\text{O}$ reacts with bicyclo[4.3.0]nona-3,6(1)-diene, in refluxing ethanol, to afford $[\text{RuCl}_2(\eta^6\text{-indane})]_2$ in good yield. The two halves of the dinuclear complex are related by a crystallographic inversion centre. The bond distances and angles are similar to other $[\text{RuCl}_2(\eta^6\text{-arene})]_2$ complexes, where arene is hexamethylbenzene (McCormick & Gleason, 1988), trindane (Gupta *et al.*, 1997), ethyl benzoate (Therrien *et al.*, 1998) and 1,2,3,4-tetrahydronaphthalene (Bown & Bennett, 1999).



The complexation of the indane to the ruthenium metal generates diastereotopic methylene protons, the two faces of the arene being non-equivalent. Each benzylic methylene proton appears as a doublet of doublets of doublets, while the central methylene protons both exhibit an 18-line pattern for a doublet of triplets of triplets. A similar ^1H NMR spectrum has been observed by Gupta *et al.* (1997) for the related complex $[\text{RuCl}_2(\eta^6\text{-trindane})]_2$.

The indane adopts an envelope conformation, in which the five-membered ring is folded toward the metal. The aromatic ring of the indane is planar, with a maximum deviation from the mean plane of 0.011 (3) \AA for C5, and the Ru—indane (centroid) distance is 1.644 \AA . The metal possesses one terminal and two bridging chlorines; the average Ru—Cl (bridged) distance is 2.4525 \AA , while the Ru—Cl (terminal) distance is 2.4079 (9) \AA . In the crystal structure, there is no significant interaction between the complex and the chloroform molecules. The closest C1S—H1S \cdots Cl contacts are 3.426 and 3.523 \AA , with C—H \cdots Cl angles of 137 and 140° for Cl2ⁱ and Cl1ⁱ, respectively [symmetry code (i): $1 - x, \frac{1}{2} + y, \frac{1}{2} - z$].

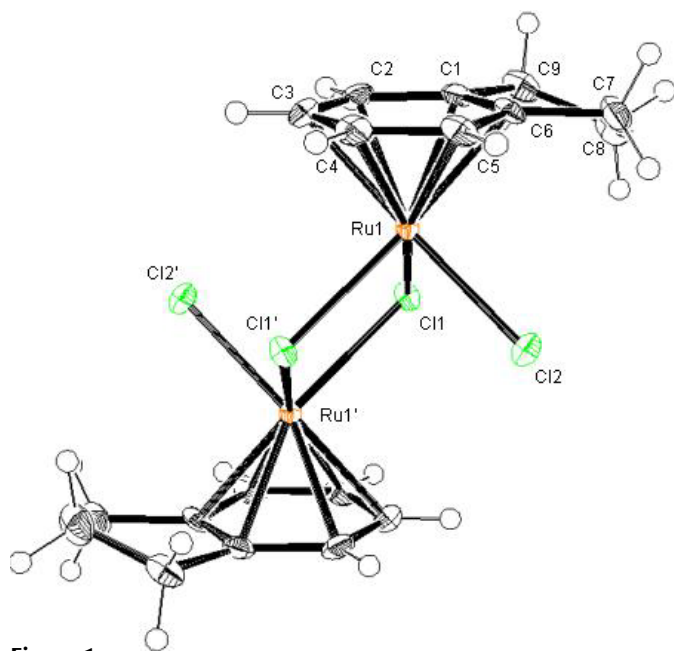


Figure 1
The molecular structure of $[\text{RuCl}_2(\eta^6\text{-indane})]_2$; CHCl_3 molecules have been omitted for clarity. Displacement ellipsoids are drawn at the 50% probability level.

Experimental

A solution of $\text{RuCl}_3 \cdot 3\text{H}_2\text{O}$ (Johnson–Matthey Co. Ltd) (1.0 g, 3.8 mmol) and an excess of bicyclo[4.3.0]nona-3,6(1)-diene (6 ml, 46 mmol) (Aldrich) in ethanol (100 ml) was refluxed for 6 h. A nitrogen atmosphere can be used but is not strictly necessary. After cooling to room temperature, half of the volume was evaporated *in vacuo* and the solution refrigerated overnight. The red–orange microcrystalline material was filtered off, washed with ether and dried *in vacuo* to give $[\text{RuCl}_2(\eta^6\text{-indane})]_2$ in 75% yield.

^1H NMR (DMSO): 5.88 (*dd*, 2H), 5.77 (*dd*, 2H), 2.76 (*m*, 2H), 2.58 (*m*, 2H), 1.94 (*m*, 2H). ^{13}C NMR (DMSO): 107.0, 85.7, 84.6, 30.3, 23.2. IR (KBr, cm^{-1}): 3057 (*m*), 3041 (*s*), 2938 (*m*), 1444 (*m*), 1414 (*s*), 860 (*s*). MS (ESI, *m/z*): 544; $[[\text{Ru}(\eta^6\text{-indane})]_2(\mu\text{-Cl})_3]^+$. Calculated for $\text{C}_9\text{H}_{10}\text{Cl}_2\text{Ru}$: C 37.26, H 3.47%; found: C 37.67, H 3.89.

Crystals suitable for X-ray diffraction analysis were grown by slow evaporation of a chloroform/hexane solution.

Crystal data

$[\text{Ru}(\text{C}_9\text{H}_{10})\text{Cl}_2] \cdot \text{CHCl}_3$
 $M_r = 409.51$
Monoclinic, $P2_1/c$
 $a = 10.5187$ (10) Å
 $b = 11.6680$ (15) Å
 $c = 11.4020$ (10) Å
 $\beta = 99.793$ (11)°
 $V = 1379.0$ (3) Å³
 $Z = 4$

$D_x = 1.972$ Mg m⁻³
Mo $K\alpha$ radiation
Cell parameters from 8000 reflections
 $\theta = 2.0\text{--}26.0^\circ$
 $\mu = 2.08$ mm⁻¹
 $T = 153$ (2) K
Block, red
 $0.45 \times 0.45 \times 0.30$ mm

Data collection

Stoe IPDS diffractometer
 φ scans
Absorption correction:
multi-scan (Blessing, 1995)
 $T_{\min} = 0.376$, $T_{\max} = 0.566$
9749 measured reflections
2528 independent reflections

2190 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.059$
 $\theta_{\max} = 26.0^\circ$
 $h = -12 \rightarrow 12$
 $k = -14 \rightarrow 14$
 $l = -12 \rightarrow 13$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.032$
 $wR(F^2) = 0.089$
 $S = 1.11$
2528 reflections
145 parameters

H-atom parameters constrained
 $w = 1/[\sigma^2(F_o^2) + (0.0568P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 0.87$ e Å⁻³
 $\Delta\rho_{\min} = -1.00$ e Å⁻³

Table 1

Selected geometric parameters (Å, °).

Cl1–Ru1	2.170 (4)	C6–Ru1	2.181 (3)
C2–Ru1	2.170 (4)	Cl1–Ru1 ⁱ	2.4489 (9)
C3–Ru1	2.174 (4)	Cl1–Ru1	2.4561 (8)
C4–Ru1	2.165 (4)	Cl2–Ru1	2.4072 (9)
C5–Ru1	2.174 (4)		
Cl2–Ru1–Cl1 ⁱ	86.88 (3)	Cl1 ⁱ –Ru1–Cl1	81.75 (3)
Cl2–Ru1–Cl1	87.28 (3)		

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

H atoms were included in calculated positions and treated as riding atoms, using *SHELXL97* default parameters.

Data collection: *EXPOSE* (*IPDS Software*; Stoe, 2000); cell refinement: *CELL* (*IPDS Software*); data reduction: *INTEGRATE* (*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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