Di-μ-chlorido-bis[chlorido(η⁶-toluene)-ruthenium(II)]

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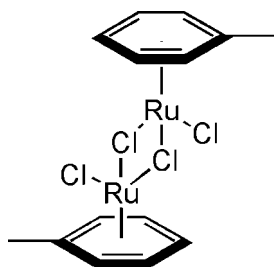
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In the centrosymmetric dinuclear title complex, [Ru₂Cl₄(η^6-C₆H₅CH₃)₂], accessible from RuCl₃·nH₂O and 1-methylcyclohexa-1,4-diene, the toluene ligand is planar with a ruthenium–centroid distance of 1.646 Å.

Related literature

For similar dinuclear chlorido-bridged arene ruthenium complexes, see: McCormick & Gleason (1988); Gupta *et al.* (1997); Therrien *et al.* (1998); Bown & Bennett (1999); Braga *et al.* (2001); Baldwin *et al.* (2002); Vieille-Petit *et al.* (2002); Allardyce *et al.* (2003); Canivet *et al.* (2005); Dinçer *et al.* (2006). For the synthesis of [RuCl₂(η^6-toluene)]₂, see: Bennett & Smith (1974).

Experimental

Crystal data

[Ru₂Cl₄(C₇H₈)₂]	a = 7.5144 (10) Å
M_r = 528.21	b = 9.1350 (10) Å
Monoclinic, $P2_1/c$	c = 11.6595 (13) Å
β = 90.691 (15)°	μ = 2.54 mm⁻¹
V = 800.30 (16) Å³	T = 173 (2) K
Z = 2	0.22 × 0.19 × 0.18 mm
Mo $K\alpha$ radiation	

Data collection

Stoe IPDS diffractometer	1567 independent reflections
Absorption correction: none	1332 reflections with $I > 2\sigma(I)$
6143 measured reflections	R_{int} = 0.165

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.039	93 parameters
$wR(F^2)$ = 0.082	H-atom parameters constrained
S = 1.07	$\Delta\rho_{max}$ = 0.91 e Å⁻³
1567 reflections	$\Delta\rho_{min}$ = −0.86 e Å⁻³

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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supplementary materials

Di-μ-chlorido-bis[chlorido(η^6-toluene)ruthenium(II)]

J. Mattsson and B. Therrien

Comment

The title compound, which is known for more then 30 years (Bennett & Smith, 1974), and regularly used as building block in organometallic chemistry, has never been characterized by X-ray structure analysis. The two halves of the dinuclear complex are related by a crystallographic inversion centre. The bond distances and angles are similar to other $[RuCl_2(\eta^6$-arene)]$_2$ complexes; arene = hexamethylbenzene (McCormick & Gleason, 1988), arene = trindane (Gupta *et al.*, 1997), arene = ethylbenzoate (Therrien *et al.*, 1998), arene = 1,2,3,4-tetrahydronaphthalene (Bown & Bennett, 1999), arene = 1,2-$C_6H_4(Me)COOMe$ (Braga *et al.*, 2001), arene = hexaethylbenzene (Baldwin *et al.*, 2002), arene = indane (Vieille-Petit *et al.*, 2002), arene = *para*-cymene (Allardyce *et al.*, 2003; Dinçer *et al.*, 2006), arene = benzene (Canivet *et al.*, 2005).

The aromatic ring of the toluene is planar and the Ru-toluene (centroid) distance is 1.648 Å. The metal possesses two bridging and one terminal chlorines, the Ru—Cl (bridged) distances are 2.4426 (10) and 2.4378 (10) Å, when the Ru—Cl (terminal) distance is 2.4037 (10) Å, see Fig. 1. No meaningful interactions between the dinuclear ruthenium complexes are observed in the crystal packing.

Experimental

$[RuCl_2(\eta^6$-toluene)]$_2$ is disolved in hot chloroform, and crystals suitable for X-ray diffraction analysis are obtained, after weeks, by slow evaporation of the diluted chloroform solution.

Refinement

The high R_{int} value is not a consequence of weak high-angle diffraction, but more probably a consequence of a high ratio of collected reflections to unique reflections. The H atoms were included in calculated positions and refined using a riding model, with C—H = 0.93–0.96 Å and with $U_{iso}(H) = 1.2$ (1.5 for methyl groups) times $U_{eq}(C)$.
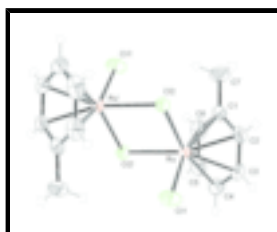
Figures

Fig. 1. The molecular structure of $[RuCl_2(\eta^6$-toluene)]$_2$ with the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level·[Symmetry code:(i) $2 - x$, $-y$, $2 - z$]

Di-µ-chlorido-bis[chlorido(η⁶-toluene)ruthenium(II)]

Crystal data

$[Ru_2Cl_4(C_7H_8)_2]$	$F_{000} = 512$
$M_r = 528.21$	$D_x = 2.192$ Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
Hall symbol: -P 2ybc	Cell parameters from 5559 reflections
$a = 7.5144$ (10) Å	$\theta = 2.2–26.1°$
$b = 9.1350$ (10) Å	$\mu = 2.54$ mm^{-1}
$c = 11.6595$ (13) Å	$T = 173$ (2) K
$\beta = 90.691$ (15)°	Block, orange
$V = 800.30$ (16) Å^3	$0.22 \times 0.19 \times 0.18$ mm
$Z = 2$	

Data collection

Stoe IPDS diffractometer	1567 independent reflections
Radiation source: fine-focus sealed tube	1332 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.165$
Detector resolution: 0.81Å pixels mm^{-1}	$\theta_{max} = 26.0°$
$T = 173(2)$ K	$\theta_{min} = 2.7°$
φ oscillation scans	$h = -9 \rightarrow 9$
Absorption correction: none	$k = -11 \rightarrow 11$
6143 measured reflections	$l = -14 \rightarrow 14$

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.039$	$w = 1/[\sigma^2(F_o^2) + (0.0382P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.082$	$(\Delta/\sigma)_{max} = 0.001$
$S = 1.07$	$\Delta\rho_{max} = 0.91$ e Å^{-3}
1567 reflections	$\Delta\rho_{min} = -0.86$ e Å^{-3}
93 parameters	Extinction correction: SHELXL97 (Sheldrick, 1997), $Fc^* = kFc[1+0.001xFc^2\lambda^3/sin(2\theta)]^{-1/4}$
Primary atom site location: structure-invariant direct methods	Extinction coefficient: 0.0116 (14)
Secondary atom site location: difference Fourier map	

Special details

Experimental. A crystal was mounted at 173 K on a Stoe Image Plate Diffraction System (Stoe & Cie, 2000) using Mo $K\alpha$ graphite monochromated radiation. Image plate distance 70 mm, φ oscillation scans $0 - 200°$, step $\Delta\varphi = 1.5°$, 3 minutes per frame.

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ($Å^2$)

	x	y	z	$U_{iso}*/U_{eq}$
C1	0.7146 (6)	−0.2107 (4)	0.8576 (4)	0.0344 (9)
C2	0.5746 (5)	−0.1062 (4)	0.8626 (4)	0.0337 (9)
H2	0.4809	−0.1215	0.9126	0.040*
C3	0.5750 (6)	0.0186 (5)	0.7941 (4)	0.0375 (10)
H3	0.4818	0.0853	0.7987	0.045*
C4	0.7162 (6)	0.0448 (6)	0.7172 (4)	0.0441 (11)
H4	0.7171	0.1279	0.6712	0.053*
C5	0.8533 (6)	−0.0569 (6)	0.7126 (4)	0.0460 (12)
H5	0.9464	−0.0412	0.6623	0.055*
C6	0.8551 (6)	−0.1834 (5)	0.7822 (4)	0.0416 (10)
H6	0.9496	−0.2488	0.7781	0.050*
C7	0.7188 (8)	−0.3395 (5)	0.9370 (5)	0.0579 (13)
H7A	0.6257	−0.3301	0.9922	0.087*
H7B	0.8319	−0.3433	0.9760	0.087*
H7C	0.7014	−0.4278	0.8936	0.087*
Cl1	0.78551 (15)	0.26134 (10)	0.94473 (10)	0.0429 (3)
Cl2	1.14333 (12)	0.04611 (11)	0.90842 (8)	0.0299 (2)
Ru	0.82237 (4)	0.01024 (3)	0.88814 (2)	0.02185 (16)

Atomic displacement parameters ($Å^2$)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.039 (2)	0.0254 (16)	0.038 (2)	−0.0065 (16)	−0.0093 (17)	−0.0074 (17)
C2	0.028 (2)	0.042 (2)	0.031 (2)	−0.0087 (16)	−0.0015 (16)	−0.0012 (18)
C3	0.028 (2)	0.044 (2)	0.040 (3)	0.0026 (16)	−0.0095 (19)	0.0028 (19)
C4	0.051 (3)	0.054 (2)	0.028 (2)	−0.013 (2)	−0.0102 (19)	0.010 (2)
C5	0.046 (3)	0.071 (3)	0.021 (2)	−0.016 (2)	0.0109 (19)	−0.018 (2)
C6	0.041 (2)	0.040 (2)	0.044 (2)	0.0051 (18)	0.0007 (19)	−0.028 (2)
C7	0.077 (3)	0.027 (2)	0.069 (3)	−0.006 (2)	−0.021 (3)	0.009 (2)
Cl1	0.0576 (7)	0.0247 (5)	0.0467 (6)	0.0059 (4)	0.0130 (5)	−0.0036 (4)

Cl2	0.0270 (5)	0.0408 (5)	0.0218 (5)	−0.0052 (4)	0.0017 (3)	0.0048 (4)
Ru	0.0244 (2)	0.0235 (2)	0.0176 (2)	0.00096 (10)	0.00095 (13)	−0.00096 (10)

Geometric parameters (Å, °)

C1—C6	1.404 (6)	C5—C6	1.412 (7)
C1—C2	1.422 (5)	C5—Ru	2.152 (4)
C1—C7	1.497 (6)	C5—H5	0.9300
C1—Ru	2.202 (4)	C6—Ru	2.173 (4)
C2—C3	1.392 (6)	C6—H6	0.9300
C2—Ru	2.162 (4)	C7—H7A	0.9600
C2—H2	0.9300	C7—H7B	0.9600
C3—C4	1.418 (6)	C7—H7C	0.9600
C3—Ru	2.149 (5)	Cl1—Ru	2.4037 (10)
C3—H3	0.9300	Cl2—Ru^i	2.4378 (10)
C4—C5	1.388 (7)	Cl2—Ru	2.4426 (10)
C4—Ru	2.161 (5)	Ru—Cl2^i	2.4378 (10)
C4—H4	0.9300		
C6—C1—C2	117.9 (4)	H7A—C7—H7C	109.5
C6—C1—C7	121.1 (4)	H7B—C7—H7C	109.5
C2—C1—C7	120.9 (4)	Ru^i—Cl2—Ru	99.12 (3)
C6—C1—Ru	70.1 (2)	C3—Ru—C5	68.10 (19)
C2—C1—Ru	69.5 (2)	C3—Ru—C4	38.42 (17)
C7—C1—Ru	128.0 (3)	C5—Ru—C4	37.5 (2)
C3—C2—C1	121.3 (4)	C3—Ru—C2	37.67 (16)
C3—C2—Ru	70.7 (2)	C5—Ru—C2	80.28 (17)
C1—C2—Ru	72.5 (2)	C4—Ru—C2	68.71 (17)
C3—C2—H2	119.3	C3—Ru—C6	80.89 (18)
C1—C2—H2	119.3	C5—Ru—C6	38.12 (19)
Ru—C2—H2	130.1	C4—Ru—C6	68.8 (2)
C2—C3—C4	120.5 (4)	C2—Ru—C6	67.93 (16)
C2—C3—Ru	71.7 (3)	C3—Ru—C1	68.63 (16)
C4—C3—Ru	71.2 (3)	C5—Ru—C1	68.22 (18)
C2—C3—H3	119.8	C4—Ru—C1	81.60 (18)
C4—C3—H3	119.8	C2—Ru—C1	38.02 (15)
Ru—C3—H3	129.8	C6—Ru—C1	37.44 (16)
C5—C4—C3	118.2 (4)	C3—Ru—Cl1	90.25 (12)
C5—C4—Ru	70.9 (3)	C5—Ru—Cl1	123.16 (15)
C3—C4—Ru	70.3 (3)	C4—Ru—Cl1	94.09 (15)
C5—C4—H4	120.9	C2—Ru—Cl1	113.92 (11)
C3—C4—H4	120.9	C6—Ru—Cl1	161.26 (14)
Ru—C4—H4	130.3	C1—Ru—Cl1	151.11 (12)
C4—C5—C6	121.8 (4)	C3—Ru—Cl2^i	125.80 (13)
C4—C5—Ru	71.6 (2)	C5—Ru—Cl2^i	148.65 (16)
C6—C5—Ru	71.7 (2)	C4—Ru—Cl2^i	164.10 (13)
C4—C5—H5	119.1	C2—Ru—Cl2^i	96.38 (11)
C6—C5—H5	119.1	C6—Ru—Cl2^i	111.70 (14)

Ru—C5—H5	130.3	C1—Ru—Cl2[i]	89.89 (12)
C1—C6—C5	120.2 (4)	Cl1—Ru—Cl2[i]	86.88 (4)
C1—C6—Ru	72.4 (2)	C3—Ru—Cl2	153.12 (13)
C5—C6—Ru	70.1 (2)	C5—Ru—Cl2	90.72 (13)
C1—C6—H6	119.9	C4—Ru—Cl2	115.01 (13)
C5—C6—H6	119.9	C2—Ru—Cl2	157.99 (11)
Ru—C6—H6	130.1	C6—Ru—Cl2	92.63 (12)
C1—C7—H7A	109.5	C1—Ru—Cl2	119.97 (11)
C1—C7—H7B	109.5	Cl1—Ru—Cl2	87.84 (4)
H7A—C7—H7B	109.5	Cl2[i]—Ru—Cl2	80.88 (4)
C1—C7—H7C	109.5		
C6—C1—C2—C3	0.8 (6)	C3—C4—Ru—Cl2[i]	7.8 (7)
C7—C1—C2—C3	176.1 (4)	C5—C4—Ru—Cl2	54.3 (3)
Ru—C1—C2—C3	53.3 (4)	C3—C4—Ru—Cl2	−174.8 (2)
C6—C1—C2—Ru	−52.5 (3)	C1—C2—Ru—C3	133.5 (4)
C7—C1—C2—Ru	122.8 (4)	C3—C2—Ru—C5	−66.6 (3)
C1—C2—C3—C4	−0.1 (7)	C1—C2—Ru—C5	66.9 (3)
Ru—C2—C3—C4	54.0 (4)	C3—C2—Ru—C4	−29.4 (3)
C1—C2—C3—Ru	−54.1 (4)	C1—C2—Ru—C4	104.1 (3)
C2—C3—C4—C5	−0.1 (7)	C3—C2—Ru—C6	−104.2 (3)
Ru—C3—C4—C5	54.1 (4)	C1—C2—Ru—C6	29.3 (3)
C2—C3—C4—Ru	−54.2 (4)	C3—C2—Ru—C1	−133.5 (4)
C3—C4—C5—C6	−0.3 (7)	C3—C2—Ru—Cl1	55.5 (3)
Ru—C4—C5—C6	53.5 (4)	C1—C2—Ru—Cl1	−171.0 (2)
C3—C4—C5—Ru	−53.8 (4)	C3—C2—Ru—Cl2[i]	144.9 (3)
C2—C1—C6—C5	−1.2 (6)	C1—C2—Ru—Cl2[i]	−81.6 (2)
C7—C1—C6—C5	−176.5 (4)	C3—C2—Ru—Cl2	−133.7 (3)
Ru—C1—C6—C5	−53.4 (4)	C1—C2—Ru—Cl2	−0.2 (5)
C2—C1—C6—Ru	52.2 (3)	C1—C6—Ru—C3	−66.6 (3)
C7—C1—C6—Ru	−123.1 (4)	C5—C6—Ru—C3	65.9 (3)
C4—C5—C6—C1	1.0 (7)	C1—C6—Ru—C5	−132.5 (4)
Ru—C5—C6—C1	54.4 (4)	C1—C6—Ru—C4	−104.5 (3)
C4—C5—C6—Ru	−53.4 (4)	C5—C6—Ru—C4	28.0 (3)
C2—C3—Ru—C5	102.9 (3)	C1—C6—Ru—C2	−29.7 (2)
C4—C3—Ru—C5	−29.7 (3)	C5—C6—Ru—C2	102.8 (3)
C2—C3—Ru—C4	132.6 (4)	C5—C6—Ru—C1	132.5 (4)
C4—C3—Ru—C2	−132.6 (4)	C1—C6—Ru—Cl1	−129.3 (4)
C2—C3—Ru—C6	65.5 (3)	C5—C6—Ru—Cl1	3.2 (6)
C4—C3—Ru—C6	−67.1 (3)	C1—C6—Ru—Cl2[i]	58.4 (3)
C2—C3—Ru—C1	28.7 (2)	C5—C6—Ru—Cl2[i]	−169.0 (2)
C4—C3—Ru—C1	−103.9 (3)	C1—C6—Ru—Cl2	139.7 (2)
C2—C3—Ru—Cl1	−131.1 (2)	C5—C6—Ru—Cl2	−87.8 (3)
C4—C3—Ru—Cl1	96.3 (3)	C6—C1—Ru—C3	103.4 (3)
C2—C3—Ru—Cl2[i]	−44.8 (3)	C2—C1—Ru—C3	−28.4 (3)
C4—C3—Ru—Cl2[i]	−177.4 (2)	C7—C1—Ru—C3	−142.1 (5)
C2—C3—Ru—Cl2	143.2 (2)	C6—C1—Ru—C5	29.3 (3)
C4—C3—Ru—Cl2	10.5 (4)	C2—C1—Ru—C5	−102.5 (3)

C4—C5—Ru—C3	30.4 (3)	C7—C1—Ru—C5	143.8 (5)
C6—C5—Ru—C3	−103.6 (3)	C6—C1—Ru—C4	65.8 (3)
C6—C5—Ru—C4	−134.0 (4)	C2—C1—Ru—C4	−66.0 (3)
C4—C5—Ru—C2	67.6 (3)	C7—C1—Ru—C4	−179.7 (5)
C6—C5—Ru—C2	−66.5 (3)	C6—C1—Ru—C2	131.8 (4)
C4—C5—Ru—C6	134.0 (4)	C7—C1—Ru—C2	−113.7 (5)
C4—C5—Ru—C1	105.2 (3)	C2—C1—Ru—C6	−131.8 (4)
C6—C5—Ru—C1	−28.9 (3)	C7—C1—Ru—C6	114.5 (5)
C4—C5—Ru—Cl1	−44.7 (3)	C6—C1—Ru—Cl1	149.0 (3)
C6—C5—Ru—Cl1	−178.8 (2)	C2—C1—Ru—Cl1	17.2 (4)
C4—C5—Ru—Cl2^i	153.9 (2)	C7—C1—Ru—Cl1	−96.5 (5)
C6—C5—Ru—Cl2^i	19.9 (4)	C6—C1—Ru—Cl2^i	−127.7 (3)
C4—C5—Ru—Cl2	−132.6 (3)	C2—C1—Ru—Cl2^i	100.5 (2)
C6—C5—Ru—Cl2	93.3 (3)	C7—C1—Ru—Cl2^i	−13.2 (4)
C5—C4—Ru—C3	−131.0 (4)	C6—C1—Ru—Cl2	−48.3 (3)
C3—C4—Ru—C5	131.0 (4)	C2—C1—Ru—Cl2	179.9 (2)
C5—C4—Ru—C2	−102.1 (3)	C7—C1—Ru—Cl2	66.2 (5)
C3—C4—Ru—C2	28.9 (3)	Ru^i—Cl2—Ru—C3	173.5 (3)
C5—C4—Ru—C6	−28.4 (3)	Ru^i—Cl2—Ru—C5	−149.65 (15)
C3—C4—Ru—C6	102.5 (3)	Ru^i—Cl2—Ru—C4	−179.30 (16)
C5—C4—Ru—C1	−65.0 (3)	Ru^i—Cl2—Ru—C2	−84.4 (3)
C3—C4—Ru—C1	66.0 (3)	Ru^i—Cl2—Ru—C6	−111.56 (14)
C5—C4—Ru—Cl1	143.8 (3)	Ru^i—Cl2—Ru—C1	−84.55 (14)
C3—C4—Ru—Cl1	−85.2 (3)	Ru^i—Cl2—Ru—Cl1	87.19 (4)
C5—C4—Ru—Cl2^i	−123.2 (5)	Ru^i—Cl2—Ru—Cl2^i	0.0

Symmetry codes: (i) −x+2, −y, −z+2.

Fig. 1