## Refinement

Refinement on $F^{2}$
$R\left[F^{2}>2 \sigma\left(F^{2}\right)\right]=0.046$
$w R\left(F^{2}\right)=0.153$
$S=1.118$
7147 reflections
490 parameters
H atoms treated by a mixture of independent and constrained refinement
$w=1 /\left[\sigma^{2}\left(F_{o}^{2}\right)+(0.0722 P)^{2}\right.$ $+6.2199 P]$
where $P=\left(F_{o}^{2}+2 F_{c}^{2}\right) / 3$
$(\Delta / \sigma)_{\text {max }}=0.001$
$\Delta \rho_{\text {max }}=1.715 \mathrm{e}_{\AA^{-3}}$
$\Delta \rho_{\text {min }}=-0.636 \mathrm{e}^{-3}$
Extinction correction: none
Scattering factors from International Tables for Crystallography (Vol. C)

Table 1. Selected geometric parameters $\left(\AA^{\circ},^{\circ}\right)$

| Rul-Cl | 1.823 (7) | $\mathrm{Cl}-\mathrm{Ol}$ | 1.159 (8) |
| :---: | :---: | :---: | :---: |
| Rul-N3 | 2.138 (5) | $\mathrm{N} 1-\mathrm{N} 2$ | 1.360 (7) |
| Rul-N1 | 2.181 (5) | N2-B1 | 1.544 (10) |
| Ru1-As2 | 2.4393 (8) | N3-N4 | 1.359 (7) |
| Rul-Asl | 2.4485 (7) | N4-B1 | 1.540 (9) |
| Rul-H1 | 1.78 (6) |  |  |
| C1—Rul-N3 | 177.0 (2) | $\mathrm{Cl}-\mathrm{Rul}-\mathrm{H} 1$ | 88 (2) |
| $\mathrm{C} 1-\mathrm{Rul}-\mathrm{N} 1$ | 94.1 (3) | N3-Rul-H1 | 90.8 (19) |
| N3-Rul-N1 | 87.60 (19) | $\mathrm{N} 1-\mathrm{Rul}-\mathrm{H} 1$ | 178 (2) |
| C1-Rul-As2 | 88.09 (19) | As2-Rul-H1 | 83 (2) |
| N3-Ru1-As2 | 94.12 (13) | Asl-Rul-H1 | 89 (2) |
| N1-Rul-As2 | 96.46 (13) | $\mathrm{Ol}-\mathrm{Cl}-\mathrm{Rul}$ | 178.9 (6) |
| Cl-Rul-Asl | 91.13 (19) | $\mathrm{N} 2-\mathrm{N} 1-\mathrm{Ru} 1$ | 123.8 (4) |
| N3-Rul-Asl | 86.39 (13) | $\mathrm{N} 1-\mathrm{N} 2-\mathrm{B} 1$ | 123.0 (5) |
| N1-Rul-Asl | 92.01 (13) | N4-N3-Rul | 123.6 (4) |
| As2-Rul-As1 | 171.53 (3) | N3-N4-B1 | 123.4 (5) |

H atoms bonded to Rul and B1 were refined freely; others were constrained with a riding model. The H atoms of the water molecule were not located; they are presumably disordered. The largest residual peak is $1.14 \AA$ from O 2 of the water molecule.

Data collection: CAD-4-PC Software (Enraf-Nonius, 1992). Cell refinement: CAD-4-PC Software. Data reduction: XCAD4 (Harms, 1997). Program(s) used to solve structure: SHELXS97 (Sheldrick, 1997a). Program(s) used to refine structure: SHELXL97 (Sheldrick, 1997b). Molecular graphics: ORTEP-3 for Windows (Farrugia, 1997). Software used to prepare material for publication: SHELXL97.

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Di- $\mu$-chloro-bis $\left\{\right.$ chloro[(4a,5,6,7,8,8a- $\eta^{6}$ )-1,2,3,4-tetrahydronaphthalene]ruthenium(II) $\}$

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## Abstract

The title compound, $\left[\left\{\operatorname{RuCl}\left(\eta^{6}-\mathrm{C}_{10} \mathrm{H}_{12}\right)\right\}_{2}(\mu-\mathrm{Cl})_{2}\right]$ or $\left[\mathrm{Ru}_{2} \mathrm{Cl}_{4}\left(\mathrm{C}_{10} \mathrm{H}_{12}\right)_{2}\right]$, has the expected edge-sharing bioctahedral geometry, in which two crystallographically related [( $\left.4 \mathrm{a}, 5,6,7,8,8 \mathrm{a}-\eta^{6}\right)-1,2,3,4$-tetrahydronaphthalene] $\mathrm{RuCl}_{2}$ moieties are asymmetrically bridged by two symmetry-related Cl atoms, with $\mathrm{Ru}-\mu-\mathrm{Cl}$ bond lengths of $2.438(1)$ and $2.440(1) \AA$, and a terminal $\mathrm{Ru}-\mathrm{Cl}$ bond length of 2.397 (1) $\AA$.

## Comment

In the course of investigations into transition metal stabilized $o$-xylylene (o-quinodimethane) complexes (Bennett et al., 1992, 1995; Bennett, Bown \& Byrnes, 1998; Bennett, Bown, Hockless et al., 1998; McGrady et al., 1996), we synthesized the chloro-bridged areneruthenium dimer $\left[\mathrm{RuCl}_{2}\left\{\left(4 \mathrm{a}, 5,6,7,8,8 \mathrm{a}-\eta^{6}\right)-1,2,3,4\right.\right.$ tetrahydronaphthalene $\}]_{2}$, (I). The diffraction analysis was undertaken to investigate the conformation of the $\left(4 \mathrm{a}, 5,6,7,8,8 \mathrm{a}-\eta^{6}\right)$-1 , 2, 3,4-tetrahydronaphthalene ( $\eta^{6}$-tetralin) ligand.

(I)

The two halves of the dimeric molecule are related by a crystallographic inversion centre and are joined by two Cl atoms asymmetrically bridging the two Ru atoms (Fig. 1). There is no evidence of any metalmetal bonding in the four-atom bridge plane $[\mathrm{Ru} \cdots \mathrm{Ru}$ 3.6785 (9) Å]. Both Ru atoms attain a distorted octahedral coordination via the $\eta^{6}$-tetralin ligand, a terminal Cl atom and the bridging Cl atoms. The overall geometry is of an edge-sharing bi-octahedron. The pertinent bond lengths and angles in (I) are comparable with those in $\left[\left\{\operatorname{RuCl}\left(\eta^{6}-\mathrm{C}_{6} \mathrm{Me}_{6}\right)\right\}_{2}(\mu-\mathrm{Cl})_{2}\right]$ (McCormick \& Gleason, 1988), $\left[\left\{\mathrm{RuCl}\left(\eta^{6}-\mathrm{C}_{6} \mathrm{H}_{5}-\mathrm{CO}_{2} \mathrm{Et}\right)\right\}_{2}(\mu-\mathrm{Cl})_{2}\right]$ (Therrien et al., 1998), $\left[\left\{\mathrm{RuCl}\left(\eta^{6} \text {-trindane }\right)\right\}_{2}(\mu-\mathrm{Cl})_{2}\right]$ (trindane is benzo[1,2:3,4:5,6]-1,2,3-trihydrocyclopentene; Gupta et al., 1997) and $\left[\left\{\mathrm{OsCl}\left(\eta^{6}-\mathrm{MeC}_{6} \mathrm{H}_{4}-4{ }^{-} \mathrm{Pr}\right)\right\}_{2}(\mu-\mathrm{Cl})_{2}\right]$ (Watkins \& Fronczek, 1982). The aromatic ring of the $\eta^{6}$-tetralin ligand is essentially flat [maximum deviation from the mean plane of 0.005 (6) $\AA$ ] and the distance of the Ru atom from the plane is $1.638 \AA$. However, the Ru atom is not symmetrically bonded to the $\eta^{6}$-tetralin ligand, the Rul- $\mathrm{C}_{\text {arene }}$ bond lengths varying in the order C4a/8a [2.183 A (average)] $>\mathrm{C} 5 / 8$ [2.160 $\AA$ (average)] > C6/7 [2.140 A (average)]. Similar asymmetric bonding of $\eta^{6}$-tetralin also occurs in $\left[\mathrm{Mn}(\mathrm{COMe})(\mathrm{CO})_{2}\left(\eta^{6}\right.\right.$-tetralin)] (Lee et al., 1995) and in $\eta^{6}$-naphthalene complexes such as $\left[\mathrm{Ru}\left(\eta^{6}-\mathrm{C}_{10} \mathrm{H}_{8}\right)\right.$ ( $\eta^{4}-1,5-\mathrm{C}_{8} \mathrm{H}_{12}$ )] (Crocker et al., 1990). The C-C bond lengths in the aromatic ring are in the range [1.34 (1)1.426 (7) $\AA$ ], the variations being unrelated to the difference in the $\mathrm{Ru}-\mathrm{C}$ bond lengths or the extra-annular substitution. The uncoordinated ring of the $\eta^{6}$-tetralin ligand is disordered over two envelope conformations, with $\mathrm{C} 2-\mathrm{C} 3$ and $\mathrm{C}^{\prime}-\mathrm{C}^{\prime}$ defining the two possible twist conformations of the ring (refined to occupancies of 0.664 and 0.336 , respectively).


Fig. 1. The molecular structure of (I) showing $50 \%$ probability displacement ellipsoids. H atoms are shown as circles of arbitrary radii. The conformation of highest occupancy ( 0.664 ) is shown.

## Experimental

The title compound was synthesized in $77 \%$ yield by reaction of $\mathrm{RuCl}_{3} \cdot 3 \mathrm{H}_{2} \mathrm{O}$ with $1,2,3,4,5,8$-hexahydronaphthalene in refluxing ethanol. A crystal suitable for X-ray analysis was grown by slow evaporation of a dichloromethane solution. ${ }^{1} \mathrm{H}$ NMR ( $\left.\mathrm{CD}_{2} \mathrm{Cl}_{2}, 300 \mathrm{MHz}\right): \delta 5.48\left(\mathrm{dd}, \mathrm{J}_{5,6}=4.2, \mathrm{~J}_{5,7}=2.2 \mathrm{~Hz}\right.$, $\mathrm{H}^{6,7}$ ), $5.31\left(d d, \mathrm{H}^{5,8}\right), 2.83$ ( $d t$, separations $=17.3,6.3 \mathrm{~Hz}$, $\left.\mathrm{H}^{1,4}\right), 2.26\left(d t\right.$, separations $\left.=17.5,6.2 \mathrm{~Hz}, \mathrm{H}^{1,4}\right), 1.95(d t$, separations $=20.2,7.5 \mathrm{~Hz}, \mathrm{H}^{2,3}$ ), 1.64 (dt, separations $=17.8$, $\left.7.3 \mathrm{~Hz}, \mathrm{H}^{2,3}\right) ;{ }^{13} \mathrm{C}\left\{{ }^{1} \mathrm{H}\right\}$ NMR $\left(\mathrm{CD}_{2} \mathrm{Cl}_{2}, 75.43 \mathrm{MHz}\right): \delta 94.59$ $\left(\mathrm{C}^{8 a, 4 a}\right), 82.36\left(\mathrm{C}^{5,8}\right), 81.16\left(\mathrm{C}^{6,7}\right), 26.54\left(\mathrm{C}^{1,4}\right), 21.49\left(\mathrm{C}^{2,3}\right)$ (assignment from single- and multiple-bond two-dimensional [ ${ }^{1} \mathrm{H}^{13} \mathrm{C}$ ]-GHMQC experiments at 500 MHz ); IR (nujol): 290 , $\left.262 \mathrm{~cm}^{-1}{ }^{[ } \nu(\mathrm{Ru}-\mathrm{Cl})\right]$; analysis calculated for $\mathrm{C}_{20} \mathrm{H}_{24} \mathrm{Cl}_{4} \mathrm{Ru}_{2}$ : C 39.49 , H 3.98, Cl $23.32 \%$; found: C 39.11, H $3.82, \mathrm{Cl}$ $23.19 \%$; MS (FAB): mz $579[M-\mathrm{Cl}]^{+}$.

## Crystal data

$\left[\mathrm{Ru}_{2} \mathrm{Cl}_{4}\left(\mathrm{C}_{10} \mathrm{H}_{12}\right)_{2}\right]$
$M_{r}=608.36$
Monoclinic
$P_{1} / n$
$a=7.552$ (2) $\AA$
$b=18.013$ (3) $\AA$
$c=8.057$ (2) $\AA$
$\beta=105.02(2)^{\circ}$
$V=1058.6(5) \AA^{3}$
$Z=2$
$D_{x}=1.908 \mathrm{Mg} \mathrm{m}^{-3}$
$D_{m}$ not measured
Data collection
Rigaku AFC-6S diffractometer
$\omega-2 \theta$ scans
Absorption correction: $\psi$ scan (North et al., 1968)
$T_{\text {min }}=0.643, T_{\text {max }}=0.808$
2099 measured reflections
1947 independent reflections

## Refinement

Refinement on $F$
$R=0.027$
$w R=0.035$
$S=1.916$
1528 reflections
117 parameters
H atoms not refined
$w=1 /\left[\sigma^{2}\left(F_{o}\right)\right.$
$\left.+0.00010\left|F_{o}\right|^{2}\right]$
Table 1. Selected geometric parameters ( $\left({ }^{\circ},{ }^{\circ}\right)$

| Rul $\cdot$. $\mathrm{Rul}^{\text {i }}$ | 3.6785 (9) | Ru1-C5 | 2.139 (5) |
| :---: | :---: | :---: | :---: |
| Rul-Cll | 2.397 (1) | Ru1-C6 | 2.145 (5) |
| Rul-Cl2 | 2.438 (1) | Rul-C7 | 2.136 (5) |
| $\mathrm{Rul}-\mathrm{Cl2}{ }^{\text {i }}$ | 2.440 (1) | Rul-C8a | 2.180 (4) |
| Rul-C4a | 2.190 (4) | Rul-C8 | 2.151 (5) |
| $\mathrm{Cl1}-\mathrm{Rul}-\mathrm{Cl} 2$ | 87.37 (4) | $\mathrm{Rul}-\mathrm{Cl} 2-\mathrm{Rul}{ }^{1}$ | 97.91 (4) |
| $\mathrm{Cl1}-\mathrm{Rul}-\mathrm{Cl2}^{\text {i }}$ | 87.18 (5) |  |  |

The uncoordinated ring of the $\eta^{6}$-tetralin ligand is disordered over two envelope conformations, with $\mathrm{C} 2-\mathrm{C} 3$ and $\mathrm{C} 2^{\prime}-$ $\mathrm{C} 3^{\prime}$ defining the two possible twist conformations of the ring (refined to occupancies of 0.664 and 0.336 , respectively). The bond lengths within the $\mathrm{Cl}-\mathrm{C} 2-\mathrm{C} 3-\mathrm{C} 4$ and $\mathrm{Cl}-\mathrm{C}^{\prime}-$ $\mathrm{C} 3^{\prime}-\mathrm{C} 4$ chains were restrained during refinement, and atoms $\mathrm{C} 2, \mathrm{C} 2^{\prime}, \mathrm{C} 3$ and $\mathrm{C} 3^{\prime}$ were refined isotropically.

Data collection: MSC/AFC Diffractometer Control Software (Molecular Structure Corporation, 1994). Cell refinement: MSC/AFC Diffractometer Control Software. Data reduction: TEXSAN (Molecular Structure Corporation, 1995). Program(s) used to solve structure: PATTY in DIRDIF92 (Beurskens et al., 1992). Program(s) used to refine structure: TEXSAN. Software used to prepare material for publication: TEXSAN.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: TA1223). Services for accessing these data are described at the back of the journal.

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# Tetraphenylphosphonium tetrakis(1-methyl-1,2,3,4-tetrazole-5-thiolato-S)aurate(III) hemihydrate 

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## Abstract

The tetraphenylphosphonium salt of tetrakis(1-methyl-1,2,3,4-tetrazole-5-thiolato)aurate(III) crystallizes as an $\mathrm{H}_{2} \mathrm{O}$ hemisolvate, $\left(\mathrm{C}_{24} \mathrm{H}_{20} \mathrm{P}\right)\left[\mathrm{Au}\left(\mathrm{C}_{2} \mathrm{H}_{3} \mathrm{~N}_{4} \mathrm{~S}\right)_{4}\right] \cdot 0.5 \mathrm{H}_{2} \mathrm{O}$, in the monoclinic $C 2 / c$ space group, with the Au atom situated on an inversion centre. The tetrazole rings are arranged almost orthogonally with respect to the square coordination sphere. Au-S-C angles of 106.2 (1) and $107.1(1)^{\circ}$ were found.

## Comment

As part of our systematic work on gold compounds, we studied the reaction of tetrachloroaurate(III) with sodium 1-methyl-1,2,3,4-tetrazole-5-thiolate (NaSmetetraz). The gold(I) anion $\left[\mathrm{Au}(\text { Smetetraz })_{2}\right]^{-}$is obtained when the reaction is performed in methanol. Considerable amounts of the $\left[\mathrm{Au}^{\text {III }}(\text { Smetetraz })_{4}\right]^{-}$anion can be obtained when the reaction is carried out in water and a bulky cation is added immediately after mixing the reactants. The red precipitates obtained in this way contain impurities of the $\mathrm{Au}^{1}$ complex which can be removed by recrystallization from common organic solvents. Following this route, we have prepared the tetraphenylarsonium and tetraethylammonium salts of $\left[\mathrm{Au}(\text { Smetetraz })_{4}\right]^{-}$(Abram et al., 1998). A pure sample of the title compound, (I), was obtained via a two-phase reaction, where solid $\mathrm{NaSmetetraz} \cdot \mathrm{xH}_{2} \mathrm{O}$ was mixed with a solution of $\left(\mathrm{Ph}_{4} \mathrm{P}\right)$ [ $\left.\mathrm{AuCl}_{4}\right]$ in $\mathrm{CH}_{2} \mathrm{Cl}_{2}$. The insolubility of the sodium salt avoids an excess of the

(I)

