Refinement

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

Table 1. Selected geometric parameters (Å, °)

Ru1—C1	1.823 (7)	C1-01	1.159 (8)
Ru1—N3	2.138 (5)	N1—N2	1.360 (7)
Ru1-N1	2.181 (5)	N2—B1	1.544 (10)
Ru1—As2	2.4393 (8)	N3N4	1.359 (7)
Ru1—As1	2.4485 (7)	N4—B1	1.540 (9)
Ru1H1	1.78 (6)		
C1—Ru1—N3	177.0 (2)	C1—Ru1—H1	88 (2)
C1Ru1N1	94.1 (3)	N3—Ru1—H1	90.8 (19)
N3—Ru1—N1	87.60 (19)	N1-Ru1-H1	178 (2)
C1—Ru1—As2	88.09 (19)	As2—Ru1—H1	83 (2)
N3—Ru1—As2	94.12 (13)	As1—Ru1—H1	89 (2)
N1—Ru1—As2	96.46 (13)	01-C1-Ru1	178.9 (6)
Cl—Rul—Asl	91.13 (19)	N2-N1-Ru1	123.8 (4)
N3—Ru1—As1	86.39 (13)	N1—N2—B1	123.0 (5)
N1—Ru1—As1	92.01 (13)	N4—N3—Ru1	123.6 (4)
As2—Ru1—As1	171.53 (3)	N3—N4—B1	123.4 (5)

H atoms bonded to Ru1 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 Å from O2 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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Supplementary data for this paper are available from the IUCr electronic archives (Reference: CF1291). Services for accessing these data are described at the back of the journal.

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Acta Cryst. (1999). C55, 852-854

Di- μ -chloro-bis{chloro[(4a,5,6,7,8,8a- η^6)-1,2,3,4-tetrahydronaphthalene]ruthenium(II)}

MARK BOWN AND MARTIN A. BENNETT

Research School of Chemistry, Institute of Advanced Studies, Australian National University, Canberra, ACT 0200, Australia. E-mail: bennett@rsc.anu.edu.au

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Abstract

The title compound, $[\{RuCl(\eta^6-C_{10}H_{12})\}_2(\mu-Cl)_2]$ or $[Ru_2Cl_4(C_{10}H_{12})_2]$, has the expected edge-sharing bioctahedral geometry, in which two crystallographically related $[(4a,5,6,7,8,8a-\eta^6)-1,2,3,4-tetrahydronaphtha$ $lene]RuCl_2$ moieties are asymmetrically bridged by two symmetry-related Cl atoms, with Ru— μ -Cl bond lengths of 2.438 (1) and 2.440 (1) Å, and a terminal Ru—Cl bond length of 2.397 (1) Å.

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 [RuCl₂{($4a,5,6,7,8,8a-\eta^6$)-1,2,3,4tetrahydronaphthalene}]₂, (I). The diffraction analysis was undertaken to investigate the conformation of the ($4a,5,6,7,8,8a-\eta^6$)-1,2,3,4-tetrahydronaphthalene (η^6 -tetralin) ligand.



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 [Ru. Ru 3.6785 (9) Å]. Both Ru atoms attain a distorted octahedral coordination via the η^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 [{RuCl(η^6 -C₆Me₆)}₂(μ -Cl)₂] (McCormick & Gleason, 1988), [{RuCl(η^6 -C₆H₅-CO₂Et)}₂(μ -Cl)₂] (Therrien *et* al., 1998), [{RuCl(η^6 -trindane)}₂(μ -Cl)₂] (trindane is benzo[1,2:3,4:5,6]-1,2,3-trihydrocyclopentene; Gupta et al., 1997) and $[{OsCl(\eta^6-MeC_6H_4-4-{}^{i}Pr)}_2(\mu-Cl)_2]$ (Watkins & Fronczek, 1982). The aromatic ring of the η^6 -tetralin ligand is essentially flat [maximum deviation from the mean plane of 0.005(6) Å] and the distance of the Ru atom from the plane is 1.638 Å. However, the Ru atom is not symmetrically bonded to the η^{6} -tetralin ligand, the Ru1—C_{arene} bond lengths varying in the order C4a/8a [2.183 Å (average)] > C5/8[2.160 Å (average)] > C6/7 [2.140 Å (average)]. Similar asymmetric bonding of η^6 -tetralin also occurs in $[Mn(COMe)(CO)_2(\eta^6-tetralin)]$ (Lee *et al.*, 1995) and in η^6 -naphthalene complexes such as [Ru(η^6 -C₁₀H₈)- $(\eta^4-1, 5-C_8H_{12})$] (Crocker *et al.*, 1990). The C—C bond lengths in the aromatic ring are in the range [1.34(1)-1.426 (7) Å], the variations being unrelated to the difference in the Ru-C bond lengths or the extra-annular Data collection substitution. The uncoordinated ring of the η^6 -tetralin Rigaku AFC-6S diffractomligand is disordered over two envelope conformations, with C2—C3 and C2'—C3' 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 RuCl₃·3H₂O 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. ¹H NMR (CD₂Cl₂, 300 MHz): δ 5.48 (*dd*, $J_{5,6}$ = 4.2, $J_{5,7}$ = 2.2 Hz, $H^{6,7}$), 5.31 (dd, $H^{5,8}$), 2.83 (dt, separations = 17.3, 6.3 Hz, $H^{1,4}$), 2.26 (dt, separations = 17.5, 6.2 Hz, $H^{1,4}$), 1.95 (dt, separations = 20.2, 7.5 Hz, $H^{2,3}$), 1.64 (*dt*, separations = 17.8, 7.3 Hz, $H^{2,3}$); ${}^{13}C{}^{1}H{}$ NMR (CD₂Cl₂, 75.43 MHz): δ 94.59 (C^{8a,4a}), 82.36 (C^{5,8}), 81.16 (C^{6,7}), 26.54 (C^{1,4}), 21.49 (C^{2,3}) (assignment from single- and multiple-bond two-dimensional [¹H–¹³C]–GHMQC experiments at 500 MHz); IR (nujol): 290, $[\nu(Ru-Cl)]$; analysis calculated for $C_{20}H_{24}Cl_4Ru_2$: 262 cm^{-} C 39.49, H 3.98, Cl 23.32%; found: C 39.11, H 3.82, Cl 23.19%; MS (FAB): m/z 579 [M - Cl]⁺.

Crystal data

$[Ru_2Cl_4(C_{10}H_{12})_2]$
$M_r = 608.36$
Monoclinic
$P2_1/n$
a = 7.552(2) Å
b = 18.013 (3) Å
c = 8.057(2) Å
$\beta = 105.02 (2)^{\circ}$
$V = 1058.6 (5) \text{ Å}^3$
Z = 2
$D_x = 1.908 \text{ Mg m}^{-3}$
D_{m} not measured

eter ω -2 θ scans Absorption correction: ψ scan (North *et al.*, 1968) $T_{\min} = 0.643, T_{\max} = 0.808$ 2099 measured reflections

1947 independent reflections

Refinement on F R = 0.027wR = 0.035S = 1.9161528 reflections 117 parameters H atoms not refined $w = 1/[\sigma^2(F_o)$ $+ 0.00010 |F_o|^2$ Mo $K\alpha$ radiation $\lambda = 0.7107 \text{ Å}$ Cell parameters from 25 reflections $\theta = 10.4 - 13.4^{\circ}$ $\mu = 1.934 \text{ mm}^{-1}$ T = 296.2 KBlock $0.26\,\times\,0.20\,\times\,0.11$ mm Orange

1528 reflections with $I > 3\sigma(I)$ $R_{\rm int} = 0.017$ $\theta_{\rm max} = 25.05^{\circ}$ $h = 0 \rightarrow 8$ $k = 0 \rightarrow 21$ $l = -9 \rightarrow 9$ 3 standard reflections every 150 reflections intensity decay: -0.91%

Refinement

 $(\Delta/\sigma)_{\rm max} = 0.0002$ $\Delta \rho_{\rm max} = 0.69 \ {\rm e} \ {\rm \AA}^{-3}$ $\Delta \rho_{\rm min} = -0.41 \ {\rm e} \ {\rm \AA}^{-3}$ Extinction correction: none Scattering factors from International Tables for X-ray Crystallography (Vol. IV)

Table 1. Selected geometric parameters (Å, °)

Ru1···Ru1 ⁱ	3.6785 (9)	Ru1—C5	2.139 (5)
Ru1—Cl1	2.397(1)	Ru1—C6	2.145 (5)
Ru1-Cl2	2.438 (1)	Ru1—C7	2.136 (5)
Ru1-Cl2 ⁱ	2.440(1)	Ru1—C8a	2.180 (4)
Ru1-C4a	2.190 (4)	Ru1—C8	2.151 (5)
Cli—Rui—Ci2	87.37 (4)	Ru1—Cl2—Ru1 ⁱ	97.91 (4)
Cl1—Ru1—Cl2 ⁱ	87.18 (5)		
Symmetry code: (i)	-x, -y, -z		

The uncoordinated ring of the η^6 -tetralin ligand is disordered Acta Cryst. (1999). C55, 854-856 over two envelope conformations, with C2-C3 and C2'-C3' defining the two possible twist conformations of the ring (refined to occupancies of 0.664 and 0.336, respectively). The bond lengths within the C1-C2-C3-C4 and C1-C2'-C3'-C4 chains were restrained during refinement, and atoms C2, C2', C3 and C3' 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(1methyl-1,2,3,4-tetrazole-5-thiolato-S)aurate(III) hemihydrate

ERNESTO SCHULZ LANG,^a MARISA DAHMER^a AND ULRICH ABRAM^b

^aUniversidade Federal de Santa Maria, Departamento de Quimica, 97.111 Santa Maria-RS, Brazil, and ^bForschungszentrum Rossendorf, Institut für Radiochemie, c/o Technische Universität Dresden, Institut für Analytische Chemie, D-01062 Dresden, Germany. E-mail: abram@fzrossendorf.de

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Abstract

The tetraphenylphosphonium salt of tetrakis(1-methyl-1,2,3,4-tetrazole-5-thiolato)aurate(III) crystallizes as an H_2O hemisolvate, $(C_{24}H_{20}P)[Au(C_2H_3N_4S)_4] \cdot 0.5H_2O$, in the monoclinic C2/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 $[Au(Smetetraz)_2]^-$ is obtained when the reaction is performed in methanol. Considerable amounts of the [Au^{III}(Smetetraz)₄]⁻ 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 Au¹ complex which can be removed by recrystallization from common organic solvents. Following this route, we have prepared the tetraphenylarsonium and tetraethylammonium salts of $[Au(Smetetraz)_4]^-$ (Abram *et al.*, 1998). A pure sample of the title compound, (I), was obtained via a two-phase reaction, where solid NaSmetetraz $\cdot xH_2O$ was mixed with a solution of $(Ph_4P)[AuCl_4]$ in CH_2Cl_2 . The insolubility of the sodium salt avoids an excess of the



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