

## Refinement

Refinement on  $F^2$   
 $R[F^2 > 2\sigma(F^2)] = 0.046$   
 $wR(F^2) = 0.153$   
 $S = 1.118$   
 7147 reflections  
 490 parameters  
 H atoms treated by a  
 mixture of independent  
 and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0722P)^2 + 6.2199P]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\max} = 0.001$   
 $\Delta\rho_{\max} = 1.715 \text{ e } \text{Å}^{-3}$   
 $\Delta\rho_{\min} = -0.636 \text{ e } \text{Å}^{-3}$   
 Extinction correction: none  
 Scattering factors from  
*International Tables for  
 Crystallography* (Vol. C)

Table 1. Selected geometric parameters (Å, °)

Ru1—C1	1.823 (7)	C1—O1	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)	N3—N4	1.359 (7)
Ru1—As1	2.4485 (7)	N4—B1	1.540 (9)
Ru1—H1	1.78 (6)		
C1—Ru1—N3	177.0 (2)	C1—Ru1—H1	88 (2)
C1—Ru1—N1	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)	O1—C1—Ru1	178.9 (6)
C1—Ru1—As1	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.

## References

- Cartwright, J., Harman, A. & Hill, A. F. (1990). *J. Organomet. Chem.* **396**, C31–34.  
 Enraf–Nonius (1992). *CAD-4-PC Software*. Version 1.1. Enraf–Nonius, Delft, The Netherlands.  
 Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.  
 Harms, K. (1997). *XCAD4. Program for the Reduction of CAD-4 Diffractometer Data*. University of Marburg, Germany.  
 Huh, S., Kim, Y., Park, Y. J., Youm, K.-T., Choi, M.-G. & Jun, M.-J. (1999). *Inorg. Chim. Acta*. Submitted.  
 Odulota, J. A., Viswanathan, R. & Dyke, T. R. (1979). *J. Am. Chem. Soc.* **101**, 4787–4792.

- Sánchez-Delgado, R. A., Lee, W. Y., Cho, Y., Jun, M.-J. & Choi, S. R. (1990). *Transition Met. Chem.* **16**, 241–244.  
 Sheldrick, G. M. (1997a). *SHELXS97. Program for the Solution of Crystal Structures*. University of Göttingen, Germany.  
 Sheldrick, G. M. (1997b). *SHELXL97. Program for the Refinement of Crystal Structures*. University of Göttingen, Germany.  
 Trofimenko, S. (1970). *Inorg. Synth.* **12**, 100–101.  
 Trofimenko, S. (1993). *Chem. Rev.* **93**, 943–980.

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

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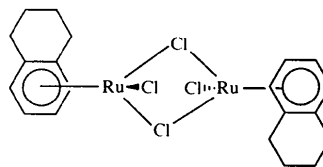
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## Abstract

The title compound,  $[\{\text{RuCl}(\eta^6\text{-C}_{10}\text{H}_{12})\}_2(\mu\text{-Cl})_2]$  or  $[\text{Ru}_2\text{Cl}_4(\text{C}_{10}\text{H}_{12})_2]$ , has the expected edge-sharing bi-octahedral geometry, in which two crystallographically related [(4a,5,6,7,8,8a- $\eta^6$ )-1,2,3,4-tetrahydronaphthalene]RuCl<sub>2</sub> moieties are asymmetrically bridged by two symmetry-related Cl atoms, with Ru— $\mu$ -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 arene-ruthenium dimer  $[\text{RuCl}_2\{(4a,5,6,7,8,8a\text{-}\eta^6)\text{-1,2,3,4-tetrahydronaphthalene}\}]_2$ , (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 ( $\eta^6$ -tetralin) ligand.



(I)



The uncoordinated ring of the  $\eta^6$ -tetralin ligand 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). 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: *MSCI/AFD Diffractometer Control Software* (Molecular Structure Corporation, 1994). Cell refinement: *MSCI/AFD Diffractometer Control Software*. Data reduction: *TEXSAN* (Molecular Structure Corporation, 1995). Program(s) used to solve structure: *PATY* 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.

## References

- Bennett, M. A., Bown, M. & Byrnes, M. J. (1998). *J. Organomet. Chem.* **571**, 139–144.
- Bennett, M. A., Bown, M., Goh, L. Y., Hockless, D. C. R. & Mitchell, T. R. B. (1995). *Organometallics*, **14**, 1000–1007.
- Bennett, M. A., Bown, M., Hockless, D. C. R., Schranz, H., Willis, A. C., McGrady, J. E. & Stranger, R. (1998). *Organometallics*, **17**, 3784–3797.
- Bennett, M. A., Goh, L. Y., McMahon, I. J., Mitchell, T. R. B., Robertson, G. B., Turney, T. W. & Wickramasinghe, W. A. (1992). *Organometallics*, **11**, 3069–3085.
- Beurskens, P. T., Admiraal, G., Beurskens, G., Bosman, W. P., Garcia-Granda, S., Gould, R. O., Smits, J. M. M. & Smykalla, C. (1992). *The DIRDIF Program System*. Technical Report. Crystallography Laboratory, University of Nijmegen, The Netherlands.
- Crocker, M., Green, M., Howard, J. A. K., Norman, N. C. & Thomas, D. M. (1990). *J. Chem. Soc. Dalton Trans.* pp. 2299–2301.
- Gupta, H. K., Lock, P. E., Hughes, D. W. & McGlinchey, M. J. (1997). *Organometallics*, **16**, 4355–4361.
- Lee, T.-Y., Lee, S. S., Chung, Y. K. & Lee, S. W. (1995). *J. Organomet. Chem.* **486**, 141–145.
- McCormick, F. B. & Gleason, W. B. (1988). *Acta Cryst.* **C44**, 603–605.
- McGrady, J. E., Stranger, R., Bown, M. & Bennett, M. A. (1996). *Organometallics*, **15**, 3109–3114.
- Molecular Structure Corporation (1994). *MSCI/AFD Diffractometer Control Software*. MSC, 3200 Research Forest Drive, The Woodlands, TX 77381, USA.
- Molecular Structure Corporation (1995). *TEXSAN. Single Crystal Structure Analysis Software*. Version 1.7. MSC, 3200 Research Forest Drive, The Woodlands, TX 77381, USA.
- North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst.* **A24**, 351–359.
- Therrien, B., Ward, T. R., Pilkington, M., Hoffmann, C., Gilardoni, F. & Weber, J. (1998). *Organometallics*, **17**, 330–337.
- Watkins, S. F. & Fronczek, F. R. (1982). *Acta Cryst.* **B38**, 270–271.

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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 H<sub>2</sub>O hemisolvate, (C<sub>24</sub>H<sub>20</sub>P)[Au(C<sub>2</sub>H<sub>3</sub>N<sub>4</sub>S)<sub>4</sub>].0.5H<sub>2</sub>O, 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)° 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)<sub>2</sub>]<sup>−</sup> is obtained when the reaction is performed in methanol. Considerable amounts of the [Au<sup>III</sup>(Smetetraz)<sub>4</sub>]<sup>−</sup> 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 Au<sup>I</sup> 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)<sub>4</sub>]<sup>−</sup> (Abram *et al.*, 1998). A pure sample of the title compound, (I), was obtained *via* a two-phase reaction, where solid NaSmetetraz.xH<sub>2</sub>O was mixed with a solution of (Ph<sub>4</sub>P)[AuCl<sub>4</sub>] in CH<sub>2</sub>Cl<sub>2</sub>. The insolubility of the sodium salt avoids an excess of the

