| | | | |
|---|---|---|---|
| Rh3—B4 | 2.125 (5) | N2—C17 | 1.331 (5) |
| Rh3—B7 | 2.147 (4) | N2—C21 | 1.447 (5) |
| Rh3—B8 | 2.163 (4) | N2—C27 | 1.473 (5) |
| Rh3—S1 | 2.3471 (10) | | |
| Br1—Rh3—Br1$^{\rm i}$ | 85.31 (2) | Rh3—Br1—Rh3$^{\rm i}$ | 94.69 (2) |
| Br1—Rh3—C1 | 90.14 (11) | Rh3—B8—C27 | 117.5 (3) |
| Br1—Rh3—C2 | 121.46 (11) | Rh3—S1—C17 | 107.70 (13) |
| Br1—Rh3—B4 | 91.56 (12) | C11—N1—C17 | 124.4 (3) |
| Br1—Rh3—B7 | 169.95 (12) | C17—N2—C21 | 121.1 (3) |
| Br1—Rh3—B8 | 133.53 (11) | C17—N2—C27 | 120.7 (3) |
| Br1—Rh3—S1 | 81.51 (3) | C21—N2—C27 | 118.3 (3) |
| Br1$^{\rm i}$—Rh3—C1 | 112.75 (11) | S1—C17—N1 | 118.3 (3) |
| Br1$^{\rm i}$—Rh3—C2 | 84.99 (11) | S1—C17—N2 | 122.0 (3) |
| Br1$^{\rm i}$—Rh3—B4 | 161.25 (13) | N1—C17—N2 | 119.7 (3) |
| Br1$^{\rm i}$—Rh3—B7 | 93.66 (12) | B8—C27—N2 | 114.3 (3) |
| Br1$^{\rm i}$—Rh3—B8 | 139.51 (12) | N1—H1*n*—C14$^{\rm ii}$ | 147 |
| Br1$^{\rm i}$—Rh3—S1 | 85.93 (3) | | |

Symmetry codes: (i) $1 - x, 1 - y, 1 - z$; (ii) $2 - x, 2 - y, 1 - z$.

All H atoms were visible in difference maps; they were positioned geometrically and included as riding atoms in the structure-factor calculations. Section of the difference maps showing the H atoms bonded to N1 and C27 have been deposited. Examination of the structures with *PLATON* (Spek, 1994*a*) showed that there were no solvent-accessible voids in the crystal lattice.

Data collection: *CAD*-4 *Software* (Enraf–Nonius, 1992). Cell refinement: *SET*4, *CELDIM* (Enraf–Nonius, 1992). Data reduction: *DATRD*2 in *NRCVAX*94 (Gabe, Le Page, Charland, Lee & White, 1989). Program(s) used to solve structure: *NRCVAX*94 and Patterson heavy-atom method. Program(s) used to refine structure: *NRCVAX*94. Molecular graphics: *ORTEP*II (Johnson, 1976) as implemented in *PLATON* (Spek, 1994*a*), *NRCVAX*94 and *PLUTON* (Spek, 1994*b*). Software used to prepare material for publication: *NRCVAX*94.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: AB1253). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

## References

Allen, F. H., Kennard, O. & Taylor, R. (1983). *Acc. Chem. Res.* **16**, 146–153.

Chizhevsky, I. T., Pisareva, I. V., Petrovskii, P. V., Bregadze, V. I., Yanovsky, A. I., Struchkov, Yu. T., Knobler, C. B. & Hawthorne, M. F. (1993). *Inorg. Chem.* **32**, 3393–3394.

Churchill, M. R. & Julis, S. A. (1978). *Inorg. Chem.* **17**, 3011–3019.

Enraf–Nonius (1992). *CAD-4-PC Software*. Version 1.1. Enraf–Nonius, Delft, The Netherlands.

Ferguson, G., Faridoon & Spalding, T. R. (1988). *Acta Cryst.* C**44**, 1371–1374.

Gabe, E. J., Le Page, Y., Charland, J.-P., Lee, F. L. & White, P. S. (1989). *J. Appl. Cryst.* **22**, 384–387.

Johnson, C. K. (1976). *ORTEP*II. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.

North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst.* A**24**, 351–359.

O'Connell, D. (1994). PhD thesis, Univ. College, Cork, Ireland.

Orpen, A. G., Brammer, L., Allen, F. H., Kennard, O., Watson, D. G. & Taylor, R. (1994). *Structure Correlation*, Vol. 2, edited by H.-B. Bürgi & J. D. Dunitz, p. 751. Weinheim: VCH.

Spek, A. L. (1994*a*). *PLATON. Molecular Geometry Program*. Univ. of Utrecht, Utrecht, Holland.

Spek, A. L. (1994*b*). *PLUTON. Molecular Graphics Program*. Univ. of Utrecht, Utrecht, Holland.

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# *exo*-2-($\eta^6$-Hexamethylbenzene)-*endo*-2-chloro-2-ruthena-*arachno*-tetraborane(8)

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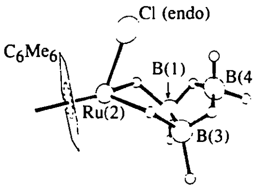
(*Received* 13 *October* 1994; *accepted* 25 *January* 1995)

## Abstract

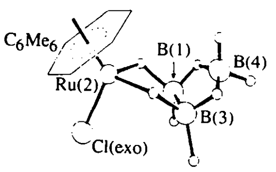
The compound *exo*-2-($\eta^6$-hexamethylbenzene)-*endo*-2-chloro-2-ruthena-*arachno*-tetraborane, $[\mathrm{RuCl(C_{12}H_{18})(B_3H_8)}]$, is based on a four-vertex butterfly structure with an Ru atom occupying a wing-tip position. The Ru atom is coordinated to the $[\mathrm{B_3H_8}]$ moiety *via* an Ru—$\mu$-H—B bridge to each of the two B atoms in the hinge positions. Additionally, the Ru atom is ligated by a hexahapto-hexamethylbenzene and a Cl atom. The compound is in a conformation with the Cl atom in the *endo* position and the $\eta^6$-arene in the *exo* position.

## Comment

Metallaoctahydrotetraboranes that maintain a halogen bound to the metal atom, $[(L_n)XM\mathrm{B_3H_8}]$, are of interest in metallaborane synthesis since they may react with borane anions to give larger and novel metallaboranes (Bown, Fontaine, Greenwood, MacKinnon, Kennedy & Thornton-Pett, 1987*a*,*b*; Bown, Greenwood & Kennedy, 1986; Kennedy, 1986). Compounds of this type were first reported several years ago [$L_n$ = $\eta^5$-$\mathrm{C_5Me_5}$, $X$ = Cl, $M$ = Ir, Rh (Kennedy, 1986); $L_n$ = $\eta^6$-$\mathrm{C_6Me_6}$, $X$ = Cl, $M$ = Ru (Kennedy, 1986; Bown, Greenwood & Kennedy, 1986; Bown, Fontaine, Greenwood, MacKinnon, Kennedy & Thornton-Pett, 1987*a*,*b*), $M$ = Os (Bown, Greenwood & Kennedy, 1986); $L_n$ = *p*-$\mathrm{MeC_6H_4{}^{\it i}Pr}$, $X$ = Cl, $M$ = Os (Bown, 1987)]. More recently, $[\mathrm{(CO)(PPh_3)_2}X\mathrm{RuB_3H_8}]$ ($X$ = Cl, Br, I) (Alcock, Burns, Claire & Hill, 1992; Burns, Hill, Thompsett, Alcock & Claire, 1992) was reported. The structure determination of the title compound was undertaken to establish which one of the two possible conformations [*endo*-chloro (I) or *exo*-chloro (II)] was adopted by the compound.



(I)



(II)

The X-ray study described here shows that the conformation adopted has the ruthenium-bound Cl atom lying in an *endo* position (I) with respect to the [$B_3H_8$] unit, the same as that observed in the $\eta^6$-toluene analogue (Bown & Waters, 1994). The Ru—B distances of 2.379 (8) and 2.370 (8) Å to B(1) and B(3), respectively, are slightly shorter than those found in [(CO)($PPh_3$)$_2$HRu$B_3H_8$] [2.439 (6) and 2.484 (6) Å, respectively (Alcock, Burns, Claire & Hill, 1992; Burns, Hill, Thompsett, Alcock & Claire, 1992)], and lie towards the higher end of the ranges observed for Ru—H—B linkages (2.152–2.484 Å; Kennedy, 1993) in other *arachno*-metallatetraboranes [2.273 (7)–2.566 (6) Å; Alcock, Burns, Claire & Hill, 1992]. The Ru—C distances lie in the range 2.189 (5)–2.224 (5) Å [weighted mean average = 2.207 (6) Å] and are significantly shorter than those expected for an *arachno*-ruthenaborane from a comparison with other structurally characterized ($\eta^6$-$C_6Me_6$)ruthenaboranes (Bown, Fontaine, Greenwood, Kennedy, Plešek, Štíbr & Thornton-Pett, 1990). The values observed are more commonly associated with *closo*-type metallaboranes, although the lack of more extensive data make any conclusions regarding the electronic structure of the compound which might be drawn from this observation dubious.

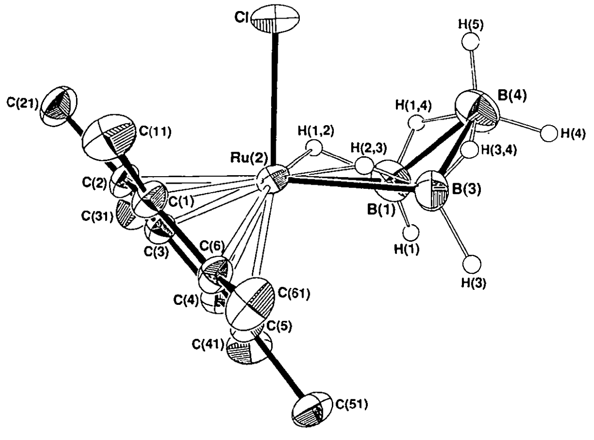


Fig. 1. *ORTEP* (Johnson, 1965) drawing of the molecular structure of the title compound. Ellipsoids are drawn at the 30% probability level. H atoms associated with the hexamethylbenzene moiety have been omitted for clarity.

## Experimental

A sample of [2-($\eta^6$-$C_6Me_6$)-2-Cl-2-Ru$B_3H_8$] was prepared from [($\eta^6$-$C_6Me_6$)Ru$Cl_2$]$_2$ and the thallium salt of *arachno*-[$B_3H_8$]$^-$ as described elsewhere (Bown, Greenwood & Kennedy, 1986; Bown, Fontaine, Greenwood, MacKinnon, Kennedy & Thornton-Pett, 1987*b*). A crystal suitable for X-ray analysis was grown by slow evaporation of a dichloromethane solution of the compound.

*Crystal data*

[RuCl($C_{12}H_{18}$)($B_3H_8$)]
$M_r = 339.28$
Orthorhombic
$P2_12_12_1$
$a = 13.401$ (2) Å
$b = 13.5794$ (10) Å
$c = 8.6950$ (10) Å
$V = 1582.4$ (3) Å$^3$
$Z = 4$
$D_x = 1.424$ Mg m$^{-3}$

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 13.50$–$15.61°$
$\mu = 1.136$ mm$^{-1}$
$T = 293$ (2) K
Hexagonal plate
0.36 × 0.16 × 0.10 mm
Red

*Data collection*

Enraf–Nonius diffractometer
$\omega/2\theta$ scans
Absorption correction: $\psi$ scans, 4 reflections
$T_{\min} = 0.972$, $T_{\max} = 0.999$
1604 measured reflections
1604 independent reflections

1373 observed reflections [$I > 2\sigma(I)$]
$\theta_{\max} = 24.97°$
$h = 0 \rightarrow 15$
$k = 0 \rightarrow 16$
$l = 0 \rightarrow 10$
3 standard reflections
frequency: 80 min
intensity decay: 0.2%

*Refinement*

Refinement on $F^2$
$R[F^2 > 2\sigma(F^2)] = 0.0253$
$wR(F^2) = 0.0720$
$S = 1.017$
1604 reflections
178 parameters
$w = 1/[\sigma^2(F_o^2) + (0.0567P)^2 + 0.7066P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.104$

$\Delta\rho_{\max} = 0.578$ e Å$^{-3}$
$\Delta\rho_{\min} = -0.474$ e Å$^{-3}$
Extinction correction: none
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)
Absolute configuration: $\chi = 0.19$ (9) (Flack, 1983)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å$^2$)

$$U_{\rm eq} = (1/3)\Sigma_i\Sigma_jU_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

| | $x$ | $y$ | $z$ | $U_{\rm eq}$ |
|---|---|---|---|---|
| Ru2 | 0.24633 (3) | 0.22063 (2) | 0.07956 (4) | 0.0355 (2) |
| Cl | 0.36946 (13) | 0.09575 (12) | 0.0398 (2) | 0.0632 (4) |
| C1 | 0.1749 (4) | 0.1893 (4) | −0.1456 (6) | 0.0438 (12) |
| C2 | 0.1304 (4) | 0.1269 (4) | −0.0303 (7) | 0.0407 (12) |
| C3 | 0.0917 (4) | 0.1674 (4) | 0.1067 (7) | 0.0435 (12) |
| C4 | 0.0937 (4) | 0.2718 (4) | 0.1275 (7) | 0.0426 (12) |
| C5 | 0.1360 (4) | 0.3334 (4) | 0.0152 (6) | 0.0419 (12) |
| C6 | 0.1780 (4) | 0.2922 (4) | −0.1200 (6) | 0.0427 (12) |
| C11 | 0.2205 (6) | 0.1439 (5) | −0.2869 (7) | 0.070 (2) |
| C21 | 0.1283 (6) | 0.0161 (4) | −0.0576 (9) | 0.064 (2) |
| C31 | 0.0455 (5) | 0.1022 (5) | 0.2254 (8) | 0.068 (2) |
| C41 | 0.0494 (5) | 0.3160 (5) | 0.2710 (8) | 0.064 (2) |

| | | | | |
|---|---|---|---|---|
| C51 | 0.1350 (5) | 0.4455 (4) | 0.0350 (9) | 0.062 (2) |
| C61 | 0.2241 (5) | 0.3581 (4) | −0.2420 (7) | 0.065 (2) |
| B1 | 0.3064 (6) | 0.2731 (8) | 0.3236 (9) | 0.066 (2) |
| B3 | 0.3649 (6) | 0.3346 (6) | 0.1754 (10) | 0.059 (2) |
| B4 | 0.4415 (7) | 0.2786 (9) | 0.3228 (11) | 0.078 (3) |

Table 2. *Selected geometric parameters* (Å, °)

| | | | |
|---|---|---|---|
| Ru2—C6 | 2.189 (5) | Ru2—B3 | 2.370 (8) |
| Ru2—C5 | 2.201 (5) | Ru2—B1 | 2.379 (8) |
| Ru2—C4 | 2.200 (5) | Ru2—Cl | 2.391 (2) |
| Ru2—C3 | 2.207 (5) | B1—B3 | 1.725 (12) |
| Ru2—C1 | 2.220 (6) | B1—B4 | 1.812 (12) |
| Ru2—C2 | 2.224 (5) | B3—B4 | 1.810 (12) |
| B3—Ru2—B1 | 42.6 (3) | B4—B1—Ru2 | 110.3 (5) |
| B3—Ru2—Cl | 92.9 (2) | B1—B3—B4 | 61.6 (5) |
| B1—Ru2—Cl | 96.2 (2) | B1—B3—Ru2 | 69.0 (4) |
| B3—B1—B4 | 61.5 (5) | B4—B3—Ru2 | 110.8 (5) |
| B3—B1—Ru2 | 68.4 (4) | B3—B4—B1 | 56.9 (5) |

Hexamethylbenzene H atoms were placed in calculated positions riding on the C atoms to which they are bonded. Borane H atoms were located on difference maps and their parameters refined. All H atoms were given isotropic displacement parameters of 0.1 Å$^2$.

Data collection: CAD-4 software. Cell refinement: CAD-4 software. Data reduction: *SDP-Plus* (Enraf–Nonius, 1985). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *ORTEP* (Johnson, 1965).

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: BK1106). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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## References

Alcock, N. W., Burns, I. D., Claire, K. S. & Hill, A. F. (1992). *Inorg. Chem.* **31**, 4606–4610.

Bown, M. (1987). PhD thesis, Univ. of Leeds, England.

Bown, M., Fontaine, X. L. R., Greenwood, N. N., Kennedy, J. D., Plešek, J., Štíbr, B. & Thornton-Pett, M. A. (1990). *Acta Cryst.* C**46**, 995–998.

Bown, M., Fontaine, X. L. R., Greenwood, N. N., MacKinnon, P., Kennedy, J. D. & Thornton-Pett, M. A. (1987*a*). *J. Chem. Soc. Chem. Commun.* pp. 442–443.

Bown, M., Fontaine, X. L. R., Greenwood, N. N., MacKinnon, P., Kennedy, J. D. & Thornton-Pett, M. A. (1987*b*). *J. Chem. Soc. Dalton Trans.* pp. 2781–2787.

Bown, M., Greenwood, N. N. & Kennedy, J. D. (1986). *J. Organomet. Chem.* **309**, C67–C69.

Bown, M. & Waters, J. M. (1994). Unpublished results.

Burns, I. D., Hill, A. F., Thompsett, A. R., Alcock, N. W. & Claire, K. S. (1992). *J. Organomet. Chem.* **425**, C8–C10, and references therein.

Enraf–Nonius (1985). *Structure Determination Package*. Enraf–Nonius, Delft, The Netherlands.

Flack, H. D. (1983). *Acta Cryst.* A**39**, 876–881

Johnson, C. K. (1965). *ORTEP*. Report ORNL-3794. Oak Ridge National Laboratory, Tennessee, USA, with local modifications.

Kennedy, J. D. (1986). *Prog. Inorg. Chem.* **34**, 211–434.

Kennedy, J. D. (1993). *J. Chem. Soc. Dalton Trans.* pp. 2545–2546, and references therein.

Sheldrick, G. M. (1990). *Acta Cryst.* A**46**, 467–473.

Sheldrick, G. M. (1993). *SHELXL93. Program for the Refinement of Crystal Structures.* Univ. of Göttingen, Germany.

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# Aqua(1,10-phenanthroline)(L-serinato)copper(II) Nitrate

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(*Received* 31 *October* 1994; *accepted* 10 *February* 1995)

## Abstract

The unit cell of the title complex, $[Cu(C_3H_6NO_3)(C_{12}H_8N_2)(H_2O)]NO_3$, contains two nitrate anions and two complex cations, each with two chiral centers, one in the serine molecule and the other at the Cu ion. Each Cu ion displays slightly distorted square-pyramidal coordination, with the water molecule in the apical position and the base defined by the aliphatic N atom and one of the O atoms from the aminocarboxylate ligand and the two N atoms from the phenanthroline molecule. The relative position of the apical water molecule generates the chiral center at the Cu ion. In both molecules, the five-membered chelate ring defined by atoms N1, C12, C11, N2 and Cu is roughly planar, while the ring defined by atoms N3, C13, C14, O1 and Cu has a distorted half-chair conformation.

## Comment

Our study of the anticancer properties of several mixed phenanthroline–aminocarboxylate complexes led us to prepare and crystallize the complex $[Cu(H_2O)(L\text{-ser})(phen)]NO_3$ (L-ser is L-serine and phen is 1,10-

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