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Supplementary data for this paper are available from the IUCr electronic archives (Reference: BM1295). Services for accessing these data are described at the back of the journal.

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A degraded glycol-substituted *iso-closo* ten-vertex ruthenaborane: [(PPh$_3$)HClRuB$_9$H$_5$(OCH$_2$CH$_2$OH)$_2$-(PPh$_3$)$_2$]·1.2CHCl$_3$

HAI-JUN YAO,[a] CHUN-HUA HU,[b] JIAN-MIN DOU,[b] JIE SUN,[c] JING-DE WEI,[a] RUO-SHUI JIN,[a] ZU-EN HUANG[a] AND PEI-JU ZHENG[b]

[a]*Department of Chemistry, Fudan University, Shanghai 200433, People's Republic of China,* [b]*Research Centre of Analysis and Measurement, Fudan University, Shanghai 200433, People's Republic of China, and* [c]*Shanghai Institute of Organic Chemistry, Chinese Academy of Science, Shanghai 200032, People's Republic of China. E-mail: pjzheng@fudan.edu.cn*

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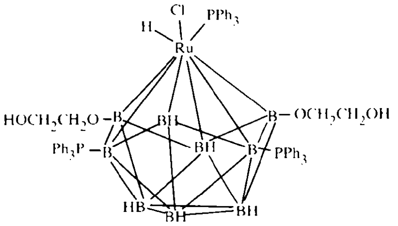
Abstract

1-Chloro-1-hydrido-2,6-bis(2-hydroxyethoxy)-1,3,5-tris(triphenylphosphine)-*iso-closo*-1-ruthenadecaborane–chloroform (1/1.2), [RuHCl(C$_{40}$H$_{45}$B$_9$O$_4$P$_2$)(C$_{18}$H$_{15}$P)]·1.2CHCl$_3$, is a ruthenaborane based on an *iso-closo* C_{3v} stack, with the nine-coordinate metal occupying the six-connected apical position. The {RuB$_9$} cage has two B-terminal –OCH$_2$CH$_2$OH groups and two PPh$_3$ substituents.

Comment

RuCl$_2$(PPh$_3$)$_3$ is a versatile metalloborane synthon. A series of ruthenaboranes have been synthesized by the reaction of RuCl$_2$(PPh$_3$)$_3$ with the *closo* ten-vertex borane anion B$_{10}$H$_{10}^{2-}$ under various conditions (Crook *et al.*, 1984, 1985; Fontaine *et al.*, 1987; Yao *et al.*, 1995). We now report the crystal structure of a degraded glycol-substituted *iso-closo* ten-vertex ruthenaborane, [(PPh$_3$)HClRuB$_9$H$_5$(OCH$_2$CH$_2$OH)$_2$(PPh$_3$)$_2$], (I), synthesized in glycol at about 373 K.



(I)

A drawing of the molecular structure of (I) is shown in Fig. 1. The ten-vertex *iso-closo* structure adopts the closed 1:3:3:3 RuB$_3$B$_3$B$_3$ cluster structure, which is similar to that in [(PPh$_3$)HClRuB$_9$H$_7$(PPh$_3$)$_2$], (II) (Crook *et al.*, 1985). The Ru atom is bound to three *exo*-polyhedral ligands (H, Cl and PPh$_3$) and six B atoms. The distances of Ru to H1, P1 and Cl1 [1.69 (8), 2.363 (2) and 2.482 (2) Å, respectively] are somewhat longer than the corresponding distances in compound (II) [1.53 (3), 2.354 (4) and 2.456 (4) Å, respectively]. Thus, the corresponding *trans*-Ru—B distances [Ru1—B3 2.482 (9), Ru1—B5 2.392 (10) and Ru1—B7 2.341 (9) Å] are shorter than those in compound (II) [2.517 (9), 2.402 (10) and 2.353 (9) Å, respectively]. The upper belt of three Ru-bound B atoms (B2, B4 and B6) is closer to the metal atom [2.057 (9)–2.205 (9) Å] than is the middle belt (B3, B5 and B7), and the Ru1—B3 distance (*trans* to Ru1—H1) is significantly longer than the distances to B5 and B7, which are *trans* to Ru1—P1 and Ru1—Cl1, respectively. Each B atom in the upper belt has a cluster connectivity of four, whereas each in the middle belt has a cluster connectivity of five.

In addition to the two PPh$_3$ substituents on the middle belt of B atoms, there are two –OCH$_2$CH$_2$OH substituents on the upper belt at B2 and B6; these groups distinguish the present compound from compound (II). The effects of the two –OCH$_2$CH$_2$OH ligands are the

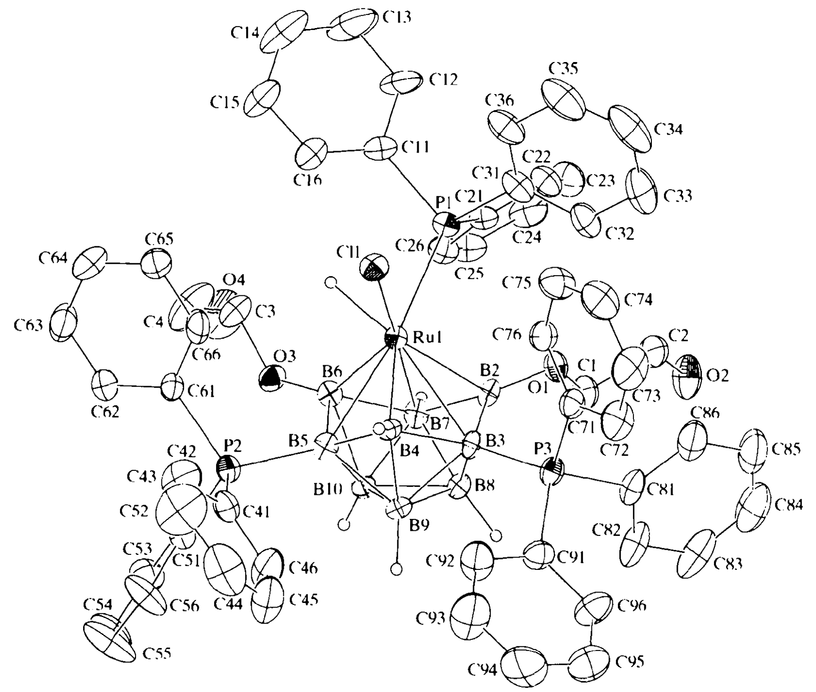


Fig. 1. *ZORTEP* (Zsolnai & Huttner, 1994) representation of (I), drawn with 30% probability displacement ellipsoids.

increased bond distances of B2—B3, B2—B7 and B2—B8 [1.755 (13)–1.789 (14) Å], and B6—B5, B6—B7 and B6—B10 [1.740 (13)–1.807 (12) Å], compared to those in compound (II) [1.742 (13)–1.774 (13) and 1.734 (14)–1.775 (10) Å, respectively]. The B—O bond distances of 1.401 (11) and 1.410 (10) Å in (I) are longer than those reported in (II) [1.361 (8)–1.368 (8) Å; Crook *et al.*, 1985].

Experimental

$RuCl_2(PPh_3)_3$ (0.4 mmol) and $(Et_4N)_2B_{10}H_{10}$ (0.4 mmol) were dissolved in $HOCH_2CH_2OH$ (*ca* 65 ml) and then heated to about 373 K under an atmosphere of dry nitrogen. After 4 h, the mixture was filtered, and the filtrate was evaporated under reduced pressure (mechanical pump, 383 K). The resulting solid was dissolved in CH_2Cl_2 and chromatographed on silica preparative TLC plates with dichloromethane/ethyl acetate (4:1) as the eluting medium, to give a yellow compound (R_f = 0.50; 30 mg). This product was recrystallized from *n*-pentane/chloroform (1:1) to give yellow crystals of (I).

Crystal data

$[RuHCl(C_{40}H_{45}B_9O_4P_2)$-
$(C_{18}H_{15}P)]\cdot 1.2CHCl_3$
M_r = 1292.03
Triclinic
$P\overline{1}$

Mo $K\alpha$ radiation
λ = 0.71069 Å
Cell parameters from 25 reflections
θ = 6.8–10.8°

a = 15.018 (5) Å
b = 18.383 (4) Å
c = 14.671 (7) Å
α = 98.91 (3)°
β = 117.43 (3)°
γ = 97.21 (2)°
V = 3461 (2) Å^3
Z = 2
D_x = 1.240 Mg m^{-3}
D_m not measured

μ = 0.514 mm^{-1}
T = 294 (2) K
Square prism
0.34 × 0.23 × 0.21 mm
Yellow

Data collection

Rigaku AFC-7*R* diffractometer
ω scans
Absorption correction: ψ-scan (North *et al.*, 1968)
T_{min} = 0.845, T_{max} = 0.900
12 079 measured reflections
11 568 independent reflections

6449 reflections with $I > 2\sigma(I)$
R_{int} = 0.066
θ_{max} = 25°
$h = 0 \rightarrow 16$
$k = -21 \rightarrow 21$
$l = -17 \rightarrow 15$
3 standard reflections every 200 reflections intensity decay: −0.42%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.070
$wR(F^2)$ = 0.242
S = 1.052

$(\Delta/\sigma)_{max}$ = 0.076
$\Delta\rho_{max}$ = 0.983 e Å^{-3}
$\Delta\rho_{min}$ = −0.548 e Å^{-3}
Extinction correction: none

11 568 reflections
781 parameters
H atoms: see below
$w = 1/[\sigma^2(F_o^2) + (0.1196P)^2 + 3.8543P]$
where $P = (F_o^2 + 2F_c^2)/3$

Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. *Selected geometric parameters (Å, °)*

Ru1—B6	2.057 (9)	Ru1—B3	2.482 (9)
Ru1—B2	2.133 (9)	Ru1—H1	1.69 (8)
Ru1—B4	2.205 (9)	P2—B5	1.941 (10)
Ru1—B7	2.341 (9)	P3—B3	1.928 (9)
Ru1—P1	2.363 (2)	O1—B2	1.401 (11)
Ru1—B5	2.392 (10)	O3—B6	1.410 (10)
Ru1—Cl1	2.482 (2)		
B6—Ru1—P1	120.4 (3)	P1—Ru1—B3	123.0 (2)
B2—Ru1—P1	90.1 (3)	Cl1—Ru1—B3	105.3 (2)
B4—Ru1—P1	150.2 (2)	B6—Ru1—H1	69 (3)
B7—Ru1—P1	104.0 (3)	B2—Ru1—H1	136 (3)
P1—Ru1—B5	166.6 (2)	B4—Ru1—H1	130 (3)
B6—Ru1—Cl1	133.5 (3)	B7—Ru1—H1	100 (3)
B2—Ru1—Cl1	132.8 (2)	P1—Ru1—H1	70 (3)
B4—Ru1—Cl1	81.0 (2)	B5—Ru1—H1	98 (3)
B7—Ru1—Cl1	174.9 (3)	Cl1—Ru1—H1	84 (3)
P1—Ru1—Cl1	80.52 (8)	B3—Ru1—H1	165 (3)
B5—Ru1—Cl1	104.9 (2)		

The title structure was solved by the Patterson method and refined by full-matrix least-squares techniques. The non-H atoms were refined anisotropically to convergence. H atoms on C and O atoms were included in riding positions [phenyl C—H = 0.93 Å, other C—H = 0.97 Å, O—H = 0.82 Å and $U(\mathrm{H}) = 1.5U_{\mathrm{eq}}(\mathrm{C,O})$]. The cage H atoms and the Ru-bound H atom were located using Fourier methods and refined isotropically. The $CHCl_3$ molecule, modelled as atoms C2*S*, Cl5, Cl6 and Cl7, was refined with an occupancy of 0.20. The C—Cl distances were restrained to be approximately equal (1.68 Å).

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1994*a*). Cell refinement: *MSC/AFC Diffractometer Control Software*. Data reduction: *PROCESS* in *TEXSAN* (Molecular Structure Corporation, 1994*b*). Program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997). Molecular graphics: *ZORTEP* (Zsolnai & Huttner, 1994). Software used to prepare material for publication: *SHELXL*97.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: DA1032). Services for accessing these data are described at the back of the journal.

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$[Ni\{(CH_3)_2SO\}_6]I_4$, a redetermination and reinterpretation

De-Liang Long, Huai-Ming Hu, Jiu-Tong Chen and Jin-Shun Huang

State Key Laboratory of Structural Chemistry, Fujan Institute of Research on the Structure of Matter, Chinese Academy of Sciences, Fuzhou 350002, People's Republic of China.
E-mail: ldl@ms.fjirsm.ac.cn

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Abstract

The structure of the title polyiodide compound, hexakis(dimethyl sulfoxide-*O*)nickel(II) tetraiodide, $[Ni(C_2H_6OS)_6]I_4$, has been redetermined. The compound comprises discrete $[Ni\{(CH_3)_2SO\}_6]^{2+}$ cations and I_4^{2-} anions. The polyhedron around the six-coordinate Ni atom is a distorted octahedron with the metal atom on a $\bar{3}$ position and six equivalent Ni—O distances of 2.077 (2) Å. The linear uncoordinated centrosymmetric I_4^{2-} polyiodide ion represents a rare example of a tetraiodide ion. It exhibits a central I—I bond of 2.848 (1) Å and two terminal I—I bonds of 3.342 (2) Å.

Comment

In recent years, polyiodides have been the focus of increasing interest due to the high conductivity and non-linear optical properties of many of their salts with sulfur-rich compounds (Wanka *et al.*, 1996; Truong *et al.*, 1993), their ability to introduce partial oxidation into hydrocarbon donor molecules to stabilize mixed valence in metal complexes (Niebling *et al.*, 1996), and their rich and varied structural chemistry. In this area, I_3^-, I_5^- and I_7^- are normally used for the synthesis of these compounds. In contrast, the formation of isolated I_4^{2-} ions was indicated by theoretical calculations to be disfavoured (Sæthre *et al.*, 1988). $[Co(NH_3)_6I_3I_4]$

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