

plane angle is much more acute. The summations of the absolute deviations of these dihedral angles from 90° , $\sum \Delta$, are 22.5° for molecule *A* and 30.0° for molecule *B*. The rotation of the N(1) pyridines around the Os(1)–N(1) vector is visible in Fig. 2. While electronic effects may be involved, these differences in dihedral angles can be largely attributed to packing forces. There are 28 pyridine...pyridine, pyridine...glycolate, or pyridine...osmyl intermolecular contacts with distances ranging from 3.11 (2) to 3.60 (2) Å.

Variations in the bend of the osmyl groups of the two molecules do not appear to be directly due to crystal packing effects. Both of the osmyl groups have seven intermolecular contacts and the ranges and averages of the contact distances are virtually the same. Yet the O(1)–Os–O(2) angle of $167.5 (4)^\circ$ for molecule *A* is significantly larger than the $165.2 (4)^\circ$ value of molecule *B*. This indicates that osmyl angle differences between these molecules may be attributed to electronic effects arising from the variations in pyridine-ring-to-central-plane dihedral angles.

The rationale for such an effect is based upon the ability of the pyridines to act as π acids, and the involvement of osmium d_{xz} and d_{yz} electron orbitals in Os–O bonding in the osmyl group. Rotations of the planes of the pyridine molecules which tend to facilitate overlap between these electron orbitals on the Os atom with the pyridine π -antibonding orbitals, provide the means to distort the π -electron distribution of the osmyl

group, leading to the bend. The rationale would go on to say that the larger the $\sum \Delta$ value, the more pronounced will be the osmyl bend angle. Support for this argument may be found in the osmyl oxobenzoato complex reported previously (Hinckley *et al.*, 1987). In this compound the dihedral angles of the two pyridine ligands to the central plane are 77.5 and 90.5° respectively, yielding a $\sum \Delta$ value of 13.0° , and the O(1)–Os–O(2) angle is $169.8 (3)^\circ$. In the narrow range of O–Os–O angles spanned by this group of three molecules, the relationship between $\sum \Delta$ and O–Os–O angle is linear within experimental error.

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A (Dithioformato)rhodaselenaborane Complex: [2,2-(η^2 -S₂CH)-2-(PPh₃)-1,2-SeRhB₁₀H₁₀]

BY GEORGE FERGUSON

Department of Chemistry and Biochemistry, University of Guelph, Guelph, Ontario, Canada N1G 2W1

AND FARIDOOON AND TREVOR R. SPALDING

Chemistry Department, University College, Cork, Ireland

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Abstract. 2-(η^2 -Dithioformato)-2-(triphenylphosphine)-1-selena-2-rhoda-*closo*-dodecaborane(12), C₁₉H₂₆B₁₀PRhS₂Se, $M_r = 639.5$, monoclinic, $P2_1/n$, $a = 12.874 (2)$, $b = 13.847 (2)$, $c = 14.612 (3)$ Å, $\beta = 94.16 (1)^\circ$, $V = 2598$ Å³, $Z = 4$, $D_x = 1.63$ g cm⁻³, $\lambda(\text{Mo K}\alpha) = 0.71073$ Å, $\mu = 22.6$ cm⁻¹, $F(000) = 1264$, $T = 298$ K, $R = 0.023$ for 3555 observed reflections. In the title compound the Rh atom is bonded to a bidentate S₂CH ligand asymmetrically [Rh–S 2.328 (1), 2.406 (1) Å], a PPh₃ ligand [Rh–P

2.385 (1) Å], and an SeB₄ face of an SeB₁₀H₁₀ cluster ligand [Rh–Se 2.4395 (4), Rh–B 2.223 (4)–2.293 (4) Å]. The RhSeB₁₀ cage is the basis of a 12-atom *closo* structure. The conformation of the η^2 -S₂CHRh unit above the SeB₄ face is such that the shorter Rh–S bond is directly *trans* to the Se atom and the S–Rh–S angle is $72.20 (3)^\circ$.

Introduction. From a study of the reactions of [2,2-(PPh₃)-2-(H)-1,2-MM'B₁₀H₁₀] ($M = \text{Se or Te}$,

$M' = \text{Rh}$ or Ir) compounds with CS_2 , we have isolated a complex ($M = \text{Se}$, $M' = \text{Rh}$) which contains the bidentate dithioformato (S_2CH) ligand (Faridoon, Ferguson, Fontaine, Kennedy & Spalding, 1988). Although complexes containing this ligand bonded to a metal are not unknown (Butler & Fenster, 1974), for example $[\text{Re}(\text{CO})_2(\text{PPh}_3)_2(\text{S}_2\text{CH})]$ (Albano, Bellon & Ciani, 1971), none has been characterized which also contains a metal-bonded heteroborane cage. A previously reported cluster compound $[\text{Co}\{B_2B'\mu\text{-S}_2\text{-}(1,2\text{-C}_2\text{B}_9\text{H}_{10})_2\}]$ (Churchill, Gold, Francis & Hawthorne, 1969) contained an S_2CH -bridged biscarbaborane system bonded to Co but direct Co to S_2CH bonding was not involved.

Experimental. Pale-yellow plate crystals were grown from dichloromethane. Accurate cell dimensions and crystal-orientation matrix determined on a CAD-4 diffractometer by a least-squares treatment of the setting angles of 25 reflections in the range $10 < \theta < 14^\circ$. Crystal dimensions $0.16 \times 0.26 \times 0.43$ mm; intensities of reflections with indices h 0 to 16, k 0 to 17, l -18 to 18, with $2 < 2\theta < 54^\circ$ measured; ω - 2θ scans; ω -scan width $(0.60 + 0.35 \tan \theta)^\circ$; graphite-monochromated Mo $K\alpha$ radiation; intensities of three reflections measured every 2 h showed no evidence of crystal decay. 6241 reflections measured, 5658 unique, 3555 with $I > 5\sigma(I)$ labelled observed and used in structure solution and refinement; $R_{\text{int}} = 0.016$. Data corrected for Lorentz, polarization and absorption effects (max. and min. transmission factors 0.717, 0.572), Gaussian integration, space group $P2_1/n$ uniquely from systematic absences $h0l$, $h + l = 2n + 1$, $0k0$, $k = 2n + 1$. The coordinates of the Rh and Se atoms were determined from analysis of the three-dimensional Patterson function and those of the remaining non-H atoms were found *via* the heavy-atom method. Refinement was by full-matrix least-squares calculations on F . Initially with isotropic and then with anisotropic thermal parameters. At an intermediate stage in the refinement, difference maps showed maxima in positions consistent with the expected locations of the H atoms; in the final rounds of calculations the H atoms were positioned on geometrical grounds (C-H 0.95, B-H 1.08 Å) and included (as riding atoms) in the structure-factor calculations. The final cycle of refinement included 308 variable parameters, $R = 0.023$, $wR = 0.034$, goodness-of-fit 1.03, $w = 1/[\sigma^2(F_o) + 0.055(F_o)^2]$. Max. shift/e.s.d. was less than 0.005; density in final difference map $0.47 \text{ e } \text{Å}^{-3}$ adjacent to Rh and Se atoms, no chemically significant features. Scattering factors and anomalous-dispersion corrections from *International Tables for X-ray Crystallography* (1974). All calculations were performed on a PDP11/73 computer using *SDP-Plus* (B. A. Frenz & Associates, Inc., 1983). Atomic coordinates and details of

Table 1. *Positional and thermal parameters and e.s.d.'s*

	<i>x</i>	<i>y</i>	<i>z</i>	<i>B*</i> (Å ²)
Rh	-0.00140 (2)	0.04358 (2)	0.24505 (2)	2.243 (4)
Se	-0.07565 (3)	0.10384 (3)	0.38299 (2)	3.331 (7)
S1	0.06820 (7)	-0.02473 (7)	0.11738 (7)	3.98 (2)
S2	0.16617 (7)	-0.02289 (7)	0.29407 (7)	4.15 (2)
P	0.08669 (6)	0.18792 (6)	0.20660 (5)	2.18 (1)
C1	0.1736 (3)	-0.0530 (3)	0.1855 (3)	4.51 (9)
C11	0.0336 (2)	0.2576 (2)	0.1089 (2)	2.52 (6)
C12	0.0868 (2)	0.3410 (3)	0.0845 (2)	3.09 (6)
C13	0.0557 (3)	0.3912 (2)	0.0070 (2)	3.50 (7)
C14	-0.0288 (3)	0.3595 (3)	-0.0483 (3)	4.34 (8)
C15	-0.0813 (3)	0.2771 (3)	-0.0254 (3)	4.59 (8)
C16	-0.0506 (3)	0.2270 (2)	0.0536 (2)	3.24 (7)
C21	0.0908 (2)	0.2713 (2)	0.3038 (2)	2.36 (6)
C22	0.1385 (2)	0.2432 (2)	0.3879 (2)	2.79 (6)
C23	0.1447 (3)	0.3060 (3)	0.4626 (2)	3.46 (7)
C24	0.1004 (3)	0.3964 (3)	0.4530 (2)	3.96 (8)
C25	0.0488 (3)	0.4234 (3)	0.3712 (3)	3.85 (8)
C26	0.0435 (3)	0.3616 (2)	0.2973 (2)	3.06 (6)
C31	0.2214 (2)	0.1803 (2)	0.1738 (2)	2.51 (5)
C32	0.3072 (2)	0.1980 (3)	0.2355 (2)	3.36 (7)
C33	0.4067 (3)	0.2016 (3)	0.2056 (3)	4.11 (8)
C34	0.4224 (3)	0.1871 (3)	0.1154 (3)	4.26 (8)
C35	0.3400 (3)	0.1669 (3)	0.0542 (2)	4.30 (8)
C36	0.2398 (3)	0.1625 (3)	0.0831 (2)	3.63 (7)
B1	-0.0691 (3)	-0.0529 (3)	0.3536 (3)	3.51 (8)
B2	-0.1120 (3)	-0.0820 (3)	0.2383 (3)	3.16 (8)
B3	-0.1589 (3)	0.0241 (3)	0.1746 (3)	2.85 (7)
B4	-0.1494 (3)	0.1317 (3)	0.2435 (3)	2.82 (7)
B5	-0.1946 (4)	-0.0982 (3)	0.3311 (3)	4.11 (9)
B6	-0.2467 (3)	-0.0568 (3)	0.2222 (3)	3.49 (8)
B7	-0.2690 (3)	0.0700 (3)	0.2299 (3)	3.40 (8)
B8	-0.2376 (3)	0.1076 (3)	0.3437 (3)	4.02 (9)
B9	-0.1894 (3)	-0.0019 (4)	0.4082 (3)	4.21 (9)
B10	-0.2915 (3)	-0.0082 (4)	0.3227 (3)	4.08 (9)

* Anisotropically refined atoms are given in the form of the equivalent isotropic thermal parameter defined as $\frac{1}{3}[a^2B_{11} + b^2B_{22} + c^2B_{33} + abc\cos\beta B_{12} + accos\beta B_{13} + bccos\beta B_{23}]$.

molecular geometry are given in Tables 1 and 2.* Figs. 1 and 2 are views of the molecule prepared using ORTEPII (Johnson, 1976).

Discussion. The Rh atom is bonded asymmetrically to the bidentate S_2CH ligand with the S1 atom *trans* to the Se exhibiting a stronger interaction with Rh [Rh-S1 2.328 (1) Å] than S2 [Rh-S2 2.406 (1) Å] (Figs. 1 and 2). The S-C bond lengths show a slight difference [S1-C 1.669 (4); S2-C 1.650 (5) Å]. The S-C-S angle of $114.4 (2)^\circ$ is less than 120° presumably because of the ligand bite; the RhS_2C atoms are almost coplanar (angle between the three-atom planes is 2.6°). In the (dithioformato)Re complex $[\text{Re}(\text{CO})_2(\text{PPh}_3)_2(\text{S}_2\text{CH})]$ (Albano, Bellon & Ciani, 1971), the bonding of the S_2CH group to the metal is more asymmetric [Re-S1 2.500 (3) and Re-S2 2.532 (2) Å] but the C-S distances and S-C-S angle [S1-C 1.68 (2), S2-C 1.64 (2) Å and S-C-S $116.7 (1)^\circ$] are similar to those observed in the present molecule. In the Re compound, the Re-S bonds are both *trans* to CO groups.

* Lists of structure factors, all bond angles, thermal parameters, calculated H-atom coordinates and mean-plane data have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44901 (44 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 2. Bond lengths (Å) and selected bond angles (°)

Rh	Se	2.4395 (4)	C25	C26	1.376 (5)		
Rh	S1	2.328 (1)	C31	C32	1.396 (4)		
Rh	S2	2.406 (1)	C31	C36	1.385 (4)		
Rh	P	2.385 (1)	C32	C33	1.384 (5)		
Rh	B1	2.293 (4)	C33	C34	1.362 (6)		
Rh	B2	2.245 (4)	C34	C35	1.366 (5)		
Rh	B3	2.223 (4)	C35	C36	1.388 (5)		
Rh	B4	2.260 (4)	B1	B2	1.781 (6)		
Se	B1	2.215 (4)	B1	B5	1.743 (6)		
Se	B4	2.219 (4)	B1	B9	1.928 (6)		
Se	B8	2.123 (4)	B2	B3	1.819 (5)		
Se	B9	2.122 (5)	B2	B5	1.797 (6)		
S1	C1	1.669 (4)	B2	B6	1.769 (5)		
S2	C1	1.650 (5)	B3	B4	1.797 (5)		
P	C11	1.816 (3)	B3	B6	1.769 (6)		
P	C21	1.828 (3)	B3	B7	1.798 (5)		
P	C31	1.836 (3)	B4	B7	1.759 (5)		
C11	C12	1.401 (5)	B4	B8	1.946 (6)		
C11	C16	1.371 (4)	B5	B6	1.776 (6)		
C12	C13	1.364 (5)	B5	B9	1.745 (6)		
C13	C14	1.379 (5)	B5	B10	1.761 (6)		
C14	C15	1.380 (6)	B6	B7	1.784 (6)		
C15	C16	1.380 (5)	B6	B10	1.750 (6)		
C21	C22	1.389 (4)	B7	B8	1.760 (6)		
C21	C26	1.391 (4)	B7	B10	1.775 (6)		
C22	C23	1.393 (5)	B8	B9	1.867 (7)		
C23	C24	1.378 (5)	B8	B10	1.764 (7)		
C24	C25	1.376 (5)	B9	B10	1.748 (6)		
Se	Rh	S1	176.02 (3)	B1	Rh	B4	87.7 (1)
Se	Rh	S2	106.35 (3)	B2	Rh	B3	48.0 (1)
Se	Rh	P	97.44 (2)	B2	Rh	B4	83.5 (1)
Se	Rh	B1	55.7 (1)	B3	Rh	B4	47.2 (1)
Se	Rh	B2	90.9 (1)	Rh	Se	B1	58.8 (1)
Se	Rh	B3	91.4 (1)	Rh	Se	B4	57.8 (1)
Se	Rh	B4	56.2 (1)	Rh	Se	B8	102.7 (1)
S1	Rh	S2	72.20 (3)	Rh	Se	B9	103.0 (1)
S1	Rh	P	86.24 (3)	B1	Se	B4	90.7 (1)
S1	Rh	B1	120.3 (1)	B1	Se	B8	91.3 (2)
S1	Rh	B2	86.0 (1)	B1	Se	B9	52.7 (2)
S1	Rh	B3	88.3 (1)	B4	Se	B8	53.2 (2)
S1	Rh	B4	125.7 (1)	B4	Se	B9	91.4 (2)
S2	Rh	P	87.67 (3)	B8	Se	B9	52.2 (2)
S2	Rh	B1	87.1 (1)	Rh	S1	C1	87.8 (2)
S2	Rh	B2	105.6 (1)	Rh	S2	C1	85.6 (1)
S2	Rh	B3	149.2 (1)	Rh	P	C11	118.2 (1)
S2	Rh	B4	161.1 (1)	Rh	P	C21	109.5 (1)
P	Rh	B1	149.5 (1)	Rh	P	C31	119.2 (1)
P	Rh	B2	161.7 (1)	C11	P	C21	105.3 (1)
P	Rh	B3	115.2 (1)	C11	P	C31	97.7 (1)
P	Rh	B4	87.7 (1)	C21	P	C31	105.4 (1)
B1	Rh	B2	46.2 (1)	S1	C1	S2	114.4 (2)
B1	Rh	B3	82.5 (1)				

The Rh—Se bond distances of 2.4395 (4) Å may be compared to values of 2.494 (1), 2.531 (1) Å observed in the diselenium-bridged compound [(triphos)Rh(μ -Se₂)Rh(triphos)]²⁺ (Bianchini, Mealli, Meli & Sabat, 1985) and 2.514 (4) Å found in [RhCl(CSe₂)(triphos)] (Bianchini, Mealli, Meli & Sabat, 1984) [triphos is (Ph₂PCH₂)₃CMe]. The short Rh—Se distance in the title compound is taken as evidence for an essentially two-centre two-electron bond in this part of the cage. The dimensions of the triphenylphosphine moiety are similar to those observed in other rhodium—phosphine complexes (Faridoon, Ferguson, Fontaine, Kennedy & Spalding, 1988). There are no unusual intermolecular contacts.

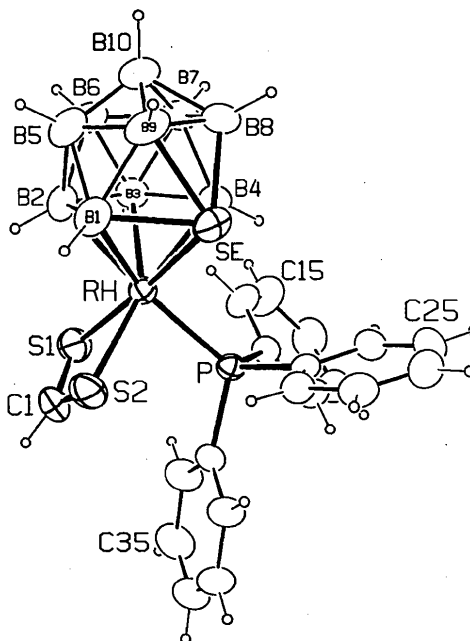


Fig. 1. A general view of the complex showing the crystallographic numbering scheme. Phenyl-ring C atoms are numbered C_i1–C_i6, *i* = 1, 2, 3.

Certain angles around the Rh atom are close to 180/90°, implying an 'octahedral' bonding environment [Se—Rh—S1 176.02 (3), S1—Rh—P 86.24 (3), S2—Rh—P 87.67 (3), P—Rh—B4 87.7 (1)°]; however, the bonding of the Rh atom to the cage can be described most simply in terms of two three-centre interactions (Rh—B1—B2 and Rh—B3—B4 opposite P and S2 atoms, respectively) and a two-centre Rh—Se interaction opposite the S1 atom. The Rh—B bonds adjacent to the Se atom [Rh—B1 2.293 (4) and Rh—B4 2.260 (4) Å] are longer than those remote from the Se atom [Rh—B2 2.245 (4) and Rh—B3 2.223 (4) Å]. The Se—B bonds similarly fall into two groups, with Se—B adjacent to the Rh atom [Se—B1 2.215 (4) and Se—B2 2.219 (4) Å] longer than those remote from the Rh atom [Se—B8 2.123 (4) and Se—B9 2.122 (4) Å].

B—B bond lengths [1.743 (6)–1.946 (6) Å] are within the normal range of values observed in boranes and their metallo derivatives (Kennedy, 1986). The Rh—P bond length of 2.385 (1) Å is typical for such bonds.

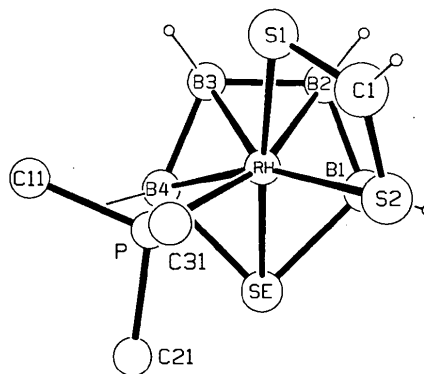


Fig. 2. A view normal to the SeB₁ plane showing the orientation of the η^2 -S₂CH and phosphine ligands on the Rh atom.

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A *cyclo*-Boronated Rhodatelluraborane Complex: [2-(PPh₃)-2-(H)-2-(Ph₂PC₆H₄)-1,2-TeRhB₁₀H₉]

BY GEORGE FERGUSON

Department of Chemistry and Biochemistry, University of Guelph, Guelph, Ontario, Canada N1G 2W1

AND FARIDOON AND TREVOR R. SPALDING

Chemistry Department, University College, Cork, Ireland

(Received 7 January 1988; accepted 24 March 1988)

Abstract. 2,7- μ -(Triphenylphosphinato-*P,C*)-2-(triphenylphosphine-*P*)-1-tellura-2-rhoda-*closo*-dodecaborane(12), C₃₆H₃₉B₁₀P₂RhTe, $M_r = 872.3$, monoclinic, $P2_1/a$, $a = 21.958$ (3), $b = 14.738$ (3), $c = 11.599$ (2) Å, $\beta = 92.80$ (2)°, $V = 3749$ Å³, $Z = 4$, $D_x = 1.55$ g cm⁻³, $\lambda(\text{Mo } K\alpha) = 0.71073$ Å, $\mu = 13.2$ cm⁻¹, $F(000) = 1728$, $T = 298$ K, $R = 0.030$ for 5212 observed reflections. The Rh atom is bonded to two phosphine ligands {PPh₃ [2.364 (1) Å] and Ph₂PC₆H₄— [2.311 (1) Å]}, an H atom (Rh—H 1.33 Å) and a TeB₄ face of the TeB₁₀ cage [Rh—Te 2.5656 (4) Å]. The cage structure of the complex can be described as a derivative of *closo*-[B₁₂H₁₂]²⁻. The formation of a Ph₂PC₆H₄ to B bond (*cyclo*-boronation) produces an Rh—P—C—C—B ring in which the Rh atom is 0.25 Å from the P—C—C—B plane. The two Rh—B distances to the B atoms which are also attached to the Te atom are distinctly longer [2.329 (4), 2.331 (4) Å] than the Rh—B distances to the two B atoms not attached to the Te atom [2.263 (5), 2.302 (5) Å]. Similarly, the two Te—B distances to B atoms also attached to the Rh atom are longer [2.379 (4), 2.439 (5) Å] than the Te—B distances to non-Rh-bonded B atoms [2.294 (5), 2.301 (5) Å].

Introduction. It is noticeable that in the chemistry of metallaboranes containing the PPh₃ ligand bonded to a Co, Rh or Ir atom, *cyclo*-boronated compounds have only been reported for Ir (Kennedy, 1986). Examples of such compounds which have been structurally characterized include [(PPh₃)(Ph₂PC₆H₄)(H)IrB₉H₈] (Bould, Greenwood, Kennedy & McDonald, 1982), [(PPh₃)(Ph₂PC₆H₄)IrB₉H₇ or ₉(PPh₃)] (Kennedy, 1986), [(PPh₃)(Ph₂PC₆H₄)(H)IrB₉H₁₂] (Bould, Crook, Greenwood & Kennedy, 1983) and [(PPh₃)(Ph₂PC₆H₄)(H)IrB₉H₁₀(PPh₃)] (Kennedy, 1986). No rhodium counterparts have been reported. In metal-lacarbaborane chemistry fewer *cyclo*-boronated compounds are known but [(PPh₃)(Ph₂PC₆H₄)IrB₈H₆(OMe)C(OH)] (Crook, Greenwood, Kennedy & McDonald, 1981) has been reported. The Rh complexes [1,3-(η^2 -3-CH₂=CHCH₂CH₂)-3-(H)-3-(PPh₃)-3,1,2-RhC₂B₉H₁₀] (Delaney, Knobler & Hawthorne, 1981) and [1,3- μ -2,3- μ -{1,2- μ -(η^2 -3,4-CH₂CH₂C(Me)=CHCH₂CH₂CH₂)-3-(H)-3-(PPh₃)-3,1,2-RhC₂B₉H₉}] (Delaney, Teller & Hawthorne, 1981) which contain carbaborane C atoms bonded to organic side chains have also been structurally characterized. The title compound was isolated in low yield from the