C24—W1—C1	90.59 (14)	O7—C25—W1	178.6 (5)
C22—W1—C1	98.3 (2)	C5—C6—C19	112.1 (4)
C21—W1—C1	90.01 (15)	C5—C6—C7	111.5 (3)
C23—W1—C1	90.13 (15)	C19—C6—C7	113.9 (4)
O3—Si1—C17	102.3 (2)	C8—C9—C10	106.6 (5)
O3—Si1—C18	109.5 (2)	O5—C21—W1	179.4 (5)
C17—Si1—C18	111.8 (3)	C5—C4—C3	113.8 (3)
O3—Si1—C13	111.3 (2)	C8—C3—C4	112.4 (3)
C17—Si1—C13	110.9 (3)	C8—C3—C2	110.5 (3)
C18—Si1—C13	110.7 (3)	C4—C3—C2	110.0 (3)
O4—C1—C2	105.3 (3)	C11—C10—C9	106.7 (4)
O4—C1—W1	131.3 (2)	C15—C13—C14	109.2 (5)
C2—C1—W1	123.4 (2)	C15—C13—C16	108.3 (4)
C1—O4—C20	121.7 (3)	C14—C13—C16	108.2 (5)
C12—O3—Si1	129.2 (2)	C15—C13—Si1	111.6 (3)
C8—O2—C11	105.8 (4)	C14—C13—Si1	110.0 (3)
C1—C2—C3	110.7 (3)	C16—C13—Si1	109.4 (4)
C1—C2—C7	111.0 (3)	O9—C23—W1	174.0 (4)
C3—C2—C7	113.5 (3)	C10—C11—O2	110.6 (5)
O8—C24—W1	177.3 (5)		

Data were collected with profile analysis over all reflections (Lehman & Larsen, 1974; Grant & Gabe, 1978). Cell refinement was performed using *CRYSDA DIRDIF* (Beurskens *et al.*, 1992). The local program *DATAR* was used for data reduction. The structure was solved by Patterson interpretation using the program *SHELXS*86 (Sheldrick, 1990). Refinement was performed using *SHELX*76 (Sheldrick, 1976; Van der Maelen Uría, 1991) and *SHELXL*93 (Sheldrick, 1993). The absolute configuration was tested using the program *BIJVOET* (Beurskens, Noordik & Beurskens, 1980) giving B = 1.0000 (1) for the 200 Friedel pairs showing the largest intensity differences. The material for publication was prepared using *SHELXL*93 and *PARST* (Nardelli, 1983).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: MU1103). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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A (Phenyldithiocarbamato)rhodatelluraborane Compound: [2-{η^2-S$_2$CN(H)Ph}-2-(PPh$_3$)-*closo*-2,1-RhTeB$_{10}$H$_{10}$]

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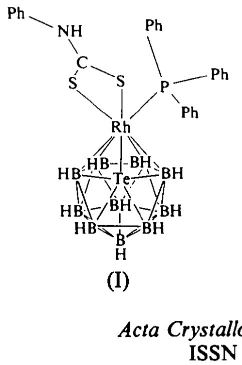
Abstract

The Rh atom in 2-(N-phenyldithiocarbamato-S,S')-2-(triphenylphosphine)-*closo*-2,1-rhodatelluradodecaborane(12), [Rh(C$_7$H$_6$NS$_2$)(C$_{18}$H$_{15}$P)(B$_{10}$H$_{10}$Te)], is bonded to triphenylphosphine [Rh—P 2.4020 (5) Å], N-phenyldithiocarbamate [Rh—S 2.4132 (5) and 2.3577 (6) Å] and the TeB$_4$ face of the TeB$_{10}$H$_{10}$ ligand [Rh—Te 2.5812 (3) Å].

Comment

The title compound was prepared by the reaction of excess PhNCS with [2,2-(PPh$_3$)$_2$-2-H-1,2-TeRhB$_{10}$H$_{10}$] in CH$_2$Cl$_2$ solution. The X-ray analysis was undertaken to establish the nature of the sulfur-containing ligand and its mode of bonding. Our analysis shows that the Rh atom in the title compound, (I), is bonded to PPh$_3$ and η^2-S$_2$CNHPh ligands as well as to the TeB$_4$ face of the TeB$_{10}$H$_{10}$ cage. The twelve-vertex distorted *closo* dodecahedral cage can be described in terms of Wade's rules as a variation of *closo*-[B$_{12}$H$_{12}$]$^{2-}$ with the Rh(η^2-S$_2$CNHPh)(PPh$_3$) unit equivalent to BH and the Te atom to [BH]$^{2-}$ (Wade, 1976). The Rh—Te bond length of 2.5812 (3) Å is significantly shorter than that of 2.6172 (4) Å in [2,2-(PPh$_3$)$_2$-2-H-1,2-TeRhB$_{10}$H$_{10}$] (Faridoon *et al.*, 1988). The Rh—S



(I)

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distances show evidence of a *trans* effect with Rh2—S2 [2.3577 (6) Å], *trans* to Te1, considerably shorter than Rh2—S1 [2.4132 (5) Å], *trans* to the triangular face containing Rh2, B6 and B11. Within the TeB_{10} ligand the B—B distances vary from 1.747 (4) to 1.956 (3) Å and the Te—B distances range from 2.293 (3) to 2.392 (2) Å; these variations are typical of metallatelluraboranes in general (Sheehan *et al.*, 1993). The conformation of the η^2-S_2CNHPh and PPh_3 ligand group above the TeB_4 face is not unexpected (Faridoon *et al.*, 1988) and is similar to that of the η^2-S_2CH and PPh_3 ligand group in [2-{η^2-S_2CH}-2-(PPh_3)-*closo*-2,1-$RhSeB_{10}H_{10}$] (Ferguson, Faridoon & Spalding, 1988).

Other dimensions are in accord with expected values [*e.g.* phenyl C—C in the range 1.353 (5)–1.406 (3) Å, mean 1.381 (12) Å]. There are no untoward intermolecular contacts.

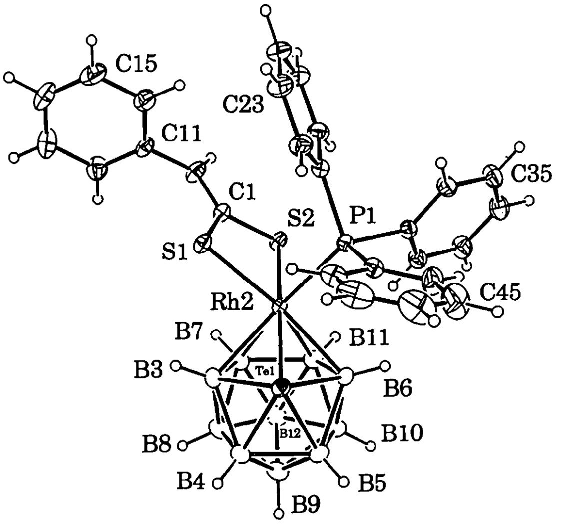

Fig. 1. A view of the title compound drawn using *ORTEP*II (Johnson, 1976). The non-H atoms are shown as displacement ellipsoids drawn at the 30% probability level. For clarity, H atoms are drawn as small spheres of arbitrary size.

Experimental

Crystal data

[$Rh(C_7H_6NS_2)(C_{18}H_{15}P)$-($B_{10}H_{10}Te$)]
$M_r = 779.22$
Triclinic
$P\bar{1}$
$a = 11.2314$ (10) Å
$b = 11.2743$ (9) Å
$c = 13.5928$ (10) Å
$\alpha = 89.322$ (6)°
$\beta = 69.114$ (7)°
$\gamma = 81.043$ (7)°
$V = 1586.7$ (2) Å^3
$Z = 2$
$D_x = 1.631$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.71069$ Å
Cell parameters from 25 reflections
$\theta = 20.00$–27.00°
$\mu = 1.63$ mm^{-1}
$T = 293$ K
Needle
0.46 × 0.15 × 0.12 mm
Orange
Crystal source: crystallized from CH_2Cl_2–hexane

Data collection

Nonius CAD-4 diffractometer
$\theta/2\theta$ scans
Absorption correction: empirical
$T_{min} = 0.624$, $T_{max} = 0.739$
6927 measured reflections
6927 independent reflections

6126 observed reflections [$I > 3.0\sigma(I)$]
$\theta_{max} = 26.94°$
$h = -13 \rightarrow 14$
$k = 0 \rightarrow 14$
$l = -17 \rightarrow 17$
3 standard reflections
frequency: 60 min
intensity variation: 2.9%

Refinement

Refinement on F
$R = 0.0176$
$wR = 0.0347$
$S = 1.09$
6126 reflections
371 parameters
H atoms treated using a riding model with C—H, N—H 0.95, B—H 1.04–1.09 Å
$w = 1/[\sigma^2(F) + 0.0008F^2]$
$(\Delta/\sigma)_{max} = 0.001$

$\Delta\rho_{max} = 0.59$ e Å^{-3}
$\Delta\rho_{min} = -0.36$ e Å^{-3}
Extinction correction: Larson (1970)
Extinction coefficient: 1.54 (6) × 10^4
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV, Table 2.2B)

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

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

	x	y	z	U_{eq}
Te1	0.349880 (12)	0.493187 (11)	0.242278 (10)	0.03086 (9)
Rh2	0.462509 (13)	0.285254 (12)	0.150968 (10)	0.02334 (10)
B3	0.2802 (2)	0.3898 (2)	0.12615 (19)	0.0351 (12)
B4	0.1468 (2)	0.4703 (2)	0.2517 (2)	0.0391 (13)
B5	0.1850 (2)	0.4205 (2)	0.37166 (19)	0.0394 (12)
B6	0.3470 (2)	0.3068 (2)	0.32838 (17)	0.0313 (11)
B7	0.2810 (2)	0.2343 (2)	0.1436 (2)	0.0369 (13)
B8	0.1335 (2)	0.3392 (3)	0.1896 (2)	0.0440 (16)
B9	0.0785 (2)	0.3551 (3)	0.3292 (2)	0.0455 (14)
B10	0.1928 (2)	0.2641 (2)	0.3733 (2)	0.0412 (13)
B11	0.3185 (2)	0.1869 (2)	0.26008 (19)	0.0328 (11)
B12	0.1571 (2)	0.2140 (3)	0.2645 (2)	0.0447 (15)
S1	0.57132 (5)	0.31207 (4)	−0.03420 (4)	0.0327 (3)
S2	0.55877 (5)	0.09314 (4)	0.06959 (4)	0.0314 (3)
C1	0.61043 (19)	0.15999 (17)	−0.04848 (15)	0.0296 (10)
N1	0.6726 (2)	0.09758 (16)	−0.13862 (14)	0.0386 (10)
C11	0.7119 (2)	0.15016 (18)	−0.24011 (15)	0.0343 (11)
C12	0.6317 (3)	0.1611 (2)	−0.2964 (2)	0.0485 (14)
C13	0.6722 (3)	0.2073 (3)	−0.3959 (2)	0.0620 (18)
C14	0.7905 (3)	0.2396 (3)	−0.43756 (19)	0.0583 (18)
C15	0.8700 (3)	0.2309 (3)	−0.3811 (2)	0.0592 (16)
C16	0.8317 (2)	0.1850 (3)	−0.28121 (19)	0.0497 (15)
P1	0.65952 (5)	0.28265 (4)	0.18350 (4)	0.0269 (2)
C21	0.81029 (19)	0.26358 (19)	0.06740 (16)	0.0331 (10)
C22	0.8733 (2)	0.3580 (2)	0.02170 (19)	0.0441 (13)
C23	0.9847 (2)	0.3381 (3)	−0.0675 (2)	0.0570 (16)
C24	1.0343 (2)	0.2236 (3)	−0.1126 (2)	0.0573 (16)
C25	0.9721 (3)	0.1296 (3)	−0.0686 (2)	0.0537 (14)
C26	0.8610 (2)	0.1482 (2)	0.02103 (19)	0.0425 (12)
C31	0.69772 (19)	0.16087 (18)	0.26307 (15)	0.0314 (10)
C32	0.6098 (2)	0.0848 (2)	0.31133 (17)	0.0389 (12)
C33	0.6412 (3)	−0.0082 (2)	0.3697 (2)	0.0482 (14)
C34	0.7591 (3)	−0.0250 (2)	0.3826 (2)	0.0503 (14)
C35	0.8468 (2)	0.0503 (2)	0.3353 (2)	0.0474 (14)
C36	0.8177 (2)	0.1419 (2)	0.27528 (18)	0.0401 (12)
C41	0.6623 (2)	0.41887 (19)	0.25335 (17)	0.0342 (10)
C42	0.6548 (2)	0.5298 (2)	0.2058 (2)	0.0457 (13)
C43	0.6616 (3)	0.6336 (2)	0.2554 (3)	0.069 (2)

C44	0.6714 (4)	0.6288 (3)	0.3530 (4)	0.086 (3)
C45	0.6729 (4)	0.5229 (3)	0.4025 (3)	0.086 (3)
C46	0.6700 (3)	0.4177 (2)	0.3526 (2)	0.0538 (16)

Table 2. *Selected geometric parameters* (Å)

Te1—Rh2	2.5812 (3)	B6—B10	1.761 (3)
Te1—B3	2.382 (2)	B6—B11	1.780 (3)
Te1—B4	2.293 (3)	B7—B8	1.791 (4)
Te1—B5	2.304 (3)	B7—B11	1.832 (4)
Te1—B6	2.392 (2)	B7—B12	1.776 (4)
Rh2—B6	2.290 (2)	B8—B9	1.776 (4)
Rh2—B7	2.238 (2)	B8—B12	1.774 (4)
Rh2—B11	2.204 (2)	B9—B10	1.785 (4)
Rh2—S1	2.4132 (5)	B9—B12	1.768 (4)
Rh2—S2	2.3577 (6)	B10—B11	1.794 (4)
Rh2—P1	2.4020 (5)	B10—B12	1.783 (4)
B3—B4	1.942 (4)	B11—B12	1.770 (3)
B3—B7	1.766 (4)	S1—C1	1.698 (2)
B3—B8	1.747 (4)	S2—C1	1.710 (2)
B4—B5	1.887 (4)	C1—N1	1.320 (3)
B4—B8	1.763 (4)	N1—C11	1.441 (3)
B4—B9	1.756 (4)	P1—C21	1.842 (2)
B5—B6	1.956 (3)	P1—C31	1.831 (2)
B5—B9	1.756 (4)	P1—C41	1.826 (2)
B5—B10	1.751 (4)		

Data collection and cell refinement: *CAD*-4 *Software* (Enraf–Nonius, 1989). Data reduction, program used to solve and refine structure and software used to prepare material for publication: *NRCVAX* (Gabe, Le Page, Charland, Lee & White, 1989). The compound crystallized in the triclinic system. The space group $P\bar{1}$ was assumed and was confirmed by the successful refinement. The structure was solved by the heavy-atom method. All H atoms were clearly visible in difference maps; they were positioned geometrically and were included as riding atoms in the structure-factor calculations. Examination of the structure with *PLATON* (Spek, 1992) showed that there were no solvent-accessible voids in the crystal structure.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: AB1173). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Sels de *S*,*S*′-(Alcane-*α*,*ω*-diyle)bis(thiouronium). II. Tétrachloroplatinate(II) de *S*,*S*′-(Octane-1,8-diyle)bis(thiouronium)

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Abstract

The structure of *S*,*S*′-(1,8-octanediyl)bis(thiouronium) tetrachloroplatinate(II), $[C_{10}H_{24}N_4S_2]$-$[PtCl_4]$, consists of square-planar $PtCl_4^{2-}$ anions and $BTUO^{2+}$ cations [where $BTUO^{2+}$ is the bis-protonated form of the organic ligand 1,8-octanediylbis(thiourea)]. This cation exhibits a centre of symmetry and packs in a mixed *trans* (*t*) and *gauche* (*g*) configuration, with a *tgttttgt* zigzag sequence which is in contrast to the extended all-*trans* configuration observed in the homologous copper(II) compound $[BTUO][CuCl_4]$. The C(3) and C(4) atoms of the alkyl chain show statistical disorder. The $BTUO^{2+}$ cations are sandwiched between monomeric planar $PtCl_4^{2-}$ layers.

Commentaire

Les tétrachlorométallates(II) d'(alcane-*α*,*ω*-diyle)-bis(ammonium), $H_3N—(CH_2)_n—[NH_3][MX_4]$ ($M =$