C17	0.8601 (3)	0.7620 (5)	0.4285 (2)	0.0426 (13)
C18	0.9107 (3)	0.6961 (5)	0.3976 (2)	0.0398 (13)
C19	0.8661 (3)	0.6426 (5)	0.3537 (2)	0.0321 (11)
C20	0.8651 (3)	0.5291 (5)	0.3486 (2)	0.0339 (11)
C21	0.8209 (3)	0.4778 (5)	0.3094 (2)	0.0326 (11)
C22	0.7758 (3)	0.5395 (4)	0.2734 (2)	0.0311 (11)
C23	0.7774 (3)	0.6532 (4)	0.2784 (2)	0.0372 (12)
C24	0.8220 (3)	0.7032 (5)	0.3178 (2)	0.0380 (13)
C25	0.5256 (3)	0.6648 (4)	0.2702 (2)	0.0274 (10)
C26	0.5561 (4)	0.7696 (4)	0.2950 (2)	0.0425 (14)
Sb	0.81721 (2)	0.17527 (3)	0.405021 (13)	0.03546 (12)
F1	0.8458 (3)	0.0363 (3)	0.38503 (15)	0.0710 (13)
F2	0.8713 (3)	0.1511 (3)	0.46660 (14)	0.0728 (13)
F3	0.9068 (3)	0.2333 (4)	0.3842 (2)	0.0739 (12)
F4	0.7642 (3)	0.2044 (4)	0.3436 (2)	0.084 (2)
F5	0.7291 (3)	0.1191 (6)	0.4257 (3)	0.129 (2)
F6	0.7918 (3)	0.3167 (3)	0.42440 (14)	0.0642 (12)
C99	1/2	0.1917 (12)	1/4	0.096 (5)
Cl	0.4462 (2)	0.0957 (3)	0.2031 (2)	0.1346 (13)
C91	0.9726 (6)	0.4899 (8)	0.0284 (3)	0.062 (4)
C92	0.9627 (5)	0.5743 (7)	−0.0059 (4)	0.060 (4)
C93	1.0063 (6)	0.5751 (7)	−0.0451 (3)	0.067 (4)
C94	1.0597 (5)	0.4916 (9)	−0.0500 (3)	0.060 (4)
C95	1.0696 (5)	0.4073 (7)	−0.0157 (3)	0.056 (3)
C96	1.0260 (5)	0.4065 (7)	0.0235 (3)	0.043 (3)
C97	0.9287 (13)	0.4965 (18)	0.0710 (8)	0.091 (6)

Table 4. *Selected geometric parameters (Å, °) for (III)*

Ag—C7	2.498 (5)	Ag—C21	2.543 (5)
Ag—C8	2.696 (5)	Ag—F6	2.545 (4)
Ag—C12	2.508 (5)	C4—C25	1.497 (7)
Ag—C13	2.593 (5)	C25—C25^i	1.332 (10)
Ag—C20	2.548 (5)	C25—C26	1.510 (7)
C25^i—C25—C4	123.8 (3)	C4—C25—C26	113.7 (4)
C25^i—C25—C26	122.5 (3)		

Symmetry code: (i) $1 - x, y, \frac{1}{2} - z$.

Solvent methyl H atoms were not included in the refinement.

For both compounds, data collection: *DIF*4 (Stoe & Cie, 1988*a*); cell refinement: *DIF*4; data reduction: *REDU*4 (Stoe & Cie, 1988*b*) (with Siemens LT-2 low-temperature attachment); program(s) used to solve structures: *SHELXS*86 (Sheldrick, 1990); program(s) used to refine structures: *SHELXL*93 (Sheldrick, 1993); molecular graphics: *XP* (Siemens, 1994); software used to prepare material for publication: *SHELXL*93.

We thank the Fonds der Chemischen Industrie for financial support and Mr A. Weinkauf for technical assistance.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: AB1325). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Acta Cryst. (1996). C**52**, 1384–1388

1,4-Bis(4′-[2.2.2]paracyclophanyl)buta-1,3-diyne and its Bis[silver(I) hexafluoroantimonate] Complex

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Abstract

The free ligand, $C_{52}H_{46}$, crystallizes with imposed twofold symmetry and its disilver(I) complex, μ-η^6:η^6-[1,4-bis(4′-[2.2.2]paracyclophanyl)buta-1,3-diyne]-bis[(hexafluoroantimonato-*F*)silver(I)] bis(dichloromethane) solvate, $[Ag_2(SbF_6)_2(C_{52}H_{46})].2CH_2Cl_2$, with inversion symmetry. The planes of the arenes directly bound to the diyne spacer subtend an interplanar angle of 26° in the free ligand, whereas they are exactly coplanar in the complex. The spacer is linear, with C4···C4^i lengths of 6.65 and 6.74 Å in the free ligand and the complex, respectively. The complex crystallizes with one molecule of dichloromethane per asymmetric unit. A short Ag—F contact of 2.636 (8) Å is observed.

Comment

The preparation of polyenes disubstituted with 4′-[2.2.2]paracyclophanyl groups in the terminal positions and the investigation of their dimetallic complexes with

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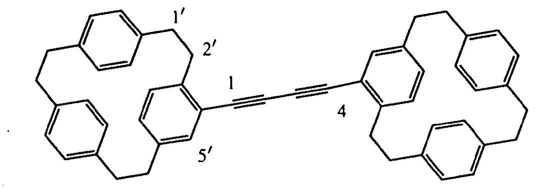
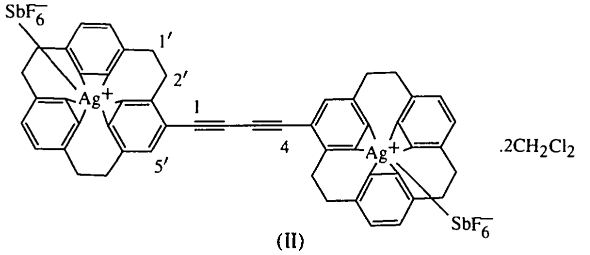
silver(I) and other soft metals are current topics of interest in our laboratories. So far, detailed structural investigations have only been possible for (*a*) disilver(I) complexes of bis-[2.2.2]paracyclophanyl derivatives spanned by unsubstituted olefinic spacers (Heirtzler, Hopf, Jones & Bubenitschek, 1995*a*) and (*b*) some ligands and disilver(I) complexes where alkyl-substituted olefinic spacers are employed (Heirtzler, Hopf, Jones & Bubenitschek, 1995*b*; Jones, Bubenitschek, Heirtzler & Hopf, 1996). In both cases, π-conjugation between the [2.2.2]paracyclophanyl units is perturbed by the non-coplanarity of their primary arenes with the conjugating spacer. Thus, there exists no X-ray crystallographic measure of the changes in ligand structure resulting from possible interactions between the complexing units where steric factors do not predominate. Consequently, we were drawn to the use of poly-yne spacers between the metal complexing units to circumvent these limitations; here we report the structures of 1,4-bis(4′-[2.2.2]paracyclophanyl)buta-1,3-diyne, (I), and its bis[silver(I) hexafluoroantimonate] complex (II).‡



(I)



(II)

Diyne (I) crystallizes with imposed twofold symmetry and the [2.2.2]paracyclophanyl groups are consequently oriented *R,R* (*S,S*)-*anti* to each other across the butadiyne spacer (see Fig. 1); for a discussion of the conformation of cyclophane systems see Heirtzler (1993). The planes of the primary arenes directly bound to this spacer (C3–C8 and its symmetry equivalent) subtend an angle of 25.9 (1)° and are essentially coplanar with it [the atoms C25–C25^{i} lie 0.045 (5)–0.215 (9) Å out of the plane of C3–C8; symmetry code: (i) $1 - x$, $1 - y$, z].

The bond lengths in the butadiyne spacer fall within normal limits (see Table 2). Its total length (C4···C4^{i}) is 6.653 (6) Å and it is almost perfectly linear [angles C(4)—C(25)—C(26) and C(25)—C(26)—C(26^{i}) are 179.0 (3) and 178.9 (3)°, respectively]. The lengths of the formal single bonds here are *ca* 0.01 Å less than reported for diphenylbutadiyne (Wiebenga, 1949), while the triple-bond length is 0.03 Å greater; we are not aware of any more recent redetermination of this structure.

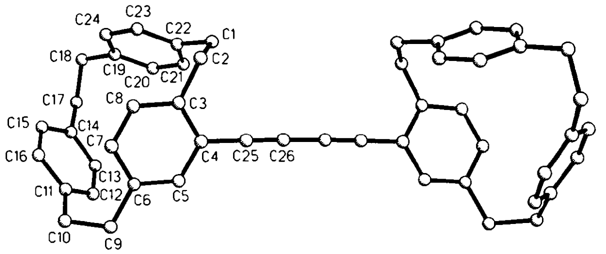


Fig. 1. The molecule of compound (I) in the crystal. Atomic radii are arbitrary. H atoms are omitted for clarity. Only the asymmetric unit is labelled.

Like the other known disilver complexes of sterically unhindered bis-[2.2.2]paracyclophanyl derivatives, (Heirtzler *et al.*, 1995*a*,*b*; Jones *et al.*, 1996), complex (II) exhibits crystallographic symmetry (inversion symmetry in this case) in the solid state (Fig. 2). The cyclophane conformation is *S,R* (*R,S*) (Heirtzler, 1993). The primary [2.2.2]paracyclophanyl arenes are exactly coplanar. The complex crystallizes as a bis(dichloromethane) solvate, but unlike the other previously mentioned disilver(I) complexes, which all crystallize with solvent molecules, the solvent molecules here are not associated with any single particular structural element; they do not form contacts to the metal atom, neither do they occupy grooves of the ligand (Jones *et al.*, 1996, and references therein). Short contacts Cl1···Cl1($1 - x$, $-y$, $2 - z$) of 3.80 (1) Å are observed.

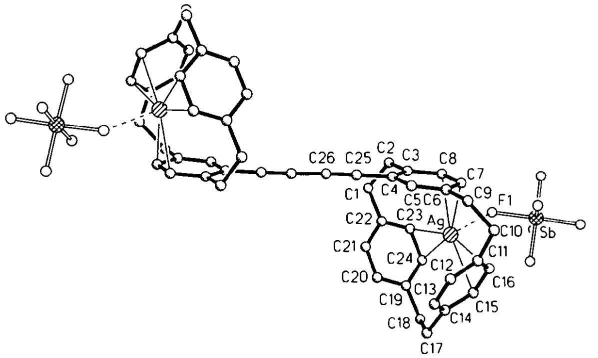


Fig. 2. The formula unit of compound (II) in the crystal. Atomic radii are arbitrary. H atoms and toluene are omitted for clarity. Only the asymmetric unit is numbered.

The individual bond lengths of the butadiyne spacer of (II) are *ca* 0.01–0.02 Å longer than in ligand (I), although the lower precision makes these differences insignificant, and combine to make a net C(4)···C(4^{i}) length of 6.74 (2) Å [symmetry code: (i) $-x$, $-y$, 1

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‡ The formula diagram of (I) shows the IUPAC numbering, which was not used in the structure determination.

$- z$; see Table 4]. This value can be contrasted with those found for two other diarylbutadiyne derivatives from which slightly elongated spacers might be expected: bis(*o*-ethoxyphenyl)butadiyne (Taga, Masaki, Osaki & Watanabe, 1971) and bis(*p*-nitrophenyl)butadiyne (Mayerle, Clarke & Bredfeldt, 1979) (6.64 and 6.62 Å). The bond angles in the linear spacer deviate slightly from the ideal 180°: C26—C25—C4 177.6 (13) and C25—C26—C26$^{\rm i}$ 175 (2)°. The spacer atoms lie from 0.06 (2) to 0.31 (3) Å out of the plane C3–C8 (for atoms C25–C25$^{\rm i}$).

Each Ag atom is bonded to three pairs of C atoms that correspond to one bond in each of three rings. The Ag—C distances are 2.45–2.58 Å and the geometry at silver parallels that found in the silver(I) complexes of other derivatives of [2.2.2]paracyclophane (Cohen-Addad, Baret, Chautemps & Pierre, 1983; Heirtzler *et al.*, 1995*a*,*b*; Jones *et al.*, 1996). Silver binds 0.079 (4) Å outside the cavity edge opposite the butadiyne spacer (as defined by the mean plane of C7, C8, C15, C16, C23 and C24); no close intermolecular contacts between Ag and the diyne moiety are evident, the shortest Ag···C contact being intramolecular: Ag···C4 3.68 (1) Å (no intermolecular contacts < 5 Å). The intramolecular distance between Ag atoms is 12.970 (5) Å, while the shortest intermolecular Ag···Ag distance is 8.463 (3) Å (operator $1 - x, 1 - y, 1 - z$). The hexafluorantimonate counterion maintains contact to silver through Ag—F1 2.636 (8) Å.

Experimental

Ligand (I) was prepared by Glaser coupling (Ciana & Haim, 1984) of 1,4′-[2.2.2]paracyclophanylethyne (Heirtzler, Hopf & Lehne, 1995). Single crystals of (I) were grown by slow vapour diffusion of *n*-pentane into a chloroform solution of (I).

Complex (II) was initially prepared analogously to our previous procedure (Heirtzler, Hopf, Jones & Bubenitschek, 1995*a*). Treatment of a dichloromethane solution of (II) with ethylbenzene and hexane and slow evaporation under atmospheric pressure (with strict exclusion of moisture) led to single crystals after *ca* 30 d.

Compound (I)

Crystal data

$C_{52}H_{46}$
$M_r = 670.89$
Orthorhombic
*Fdd*2
$a = 26.319$ (7) Å
$b = 16.313$ (4) Å
$c = 17.842$ (4) Å
$V = 7660.3$ (33) Å^3
$Z = 8$
$D_x = 1.163$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 50 reflections
$\theta = 10$–11.2°
$\mu = 0.065$ mm^{-1}
$T = 178$ (2) K
Prism
0.60 × 0.40 × 0.30 mm
Colourless

Data collection

Siemens *R*3 diffractometer
ω scans
Absorption correction: none
4552 measured reflections
2278 independent reflections
1354 observed reflections [$I > 2\sigma(I)$]
$R_{\rm int} = 0.0369$

$\theta_{\rm max} = 27.5°$
$h = -34 \rightarrow 0$
$k = -21 \rightarrow 21$
$l = -23 \rightarrow 0$
3 standard reflections monitored every 147 reflections
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.0411$
$wR(F^2) = 0.1182$
$S = 0.989$
2278 reflections
235 parameters
H atoms riding
$w = 1/[\sigma^2(F_o^2) + (0.0574P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} < 0.001$
$\Delta\rho_{\rm max} = 0.144$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.153$ 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 not determined; Flack (1983) parameter indeterminate
Origin fixing: Flack & Schwarzenbach (1988)

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

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

	x	y	z	$U_{\rm eq}$
C1	0.39071 (12)	0.5350 (2)	0.5000 (2)	0.0550 (9)
C2	0.39170 (12)	0.6216 (2)	0.5329 (2)	0.0495 (8)
C3	0.36670 (11)	0.6277 (2)	0.6086 (2)	0.0414 (7)
C4	0.38781 (10)	0.5940 (2)	0.6743 (2)	0.0418 (7)
C5	0.36094 (11)	0.5959 (2)	0.7422 (2)	0.0436 (7)
C6	0.31296 (11)	0.6303 (2)	0.7474 (2)	0.0447 (8)
C7	0.29267 (12)	0.6639 (2)	0.6822 (2)	0.0481 (8)
C8	0.31886 (12)	0.6637 (2)	0.6149 (2)	0.0472 (8)
C9	0.28383 (12)	0.6294 (2)	0.8205 (2)	0.0521 (8)
C10	0.22578 (13)	0.6233 (2)	0.8119 (2)	0.0568 (9)
C11	0.20943 (11)	0.5536 (2)	0.7618 (2)	0.0485 (8)
C12	0.22657 (12)	0.4748 (2)	0.7729 (2)	0.0509 (8)
C13	0.21541 (12)	0.4127 (2)	0.7224 (2)	0.0506 (8)
C14	0.18532 (11)	0.4262 (2)	0.6597 (2)	0.0509 (8)
C15	0.16618 (12)	0.5051 (3)	0.6500 (2)	0.0585 (9)
C16	0.17802 (12)	0.5668 (2)	0.6997 (2)	0.0562 (9)
C17	0.17647 (14)	0.3590 (2)	0.6029 (2)	0.0612 (10)
C18	0.18997 (13)	0.3838 (2)	0.5223 (2)	0.0608 (10)
C19	0.24257 (12)	0.4205 (2)	0.5147 (2)	0.0503 (8)
C20	0.28589 (13)	0.3812 (2)	0.5389 (2)	0.0558 (9)
C21	0.33351 (13)	0.4176 (2)	0.5334 (2)	0.0541 (8)
C22	0.33941 (11)	0.4944 (2)	0.5017 (2)	0.0467 (8)
C23	0.29620 (13)	0.5333 (2)	0.4761 (2)	0.0517 (8)
C24	0.24846 (14)	0.4978 (2)	0.4829 (2)	0.0529 (8)
C25	0.43629 (12)	0.5545 (2)	0.6722 (2)	0.0494 (8)
C26	0.47695 (11)	0.5200 (2)	0.6711 (2)	0.0505 (8)

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

C4—C25	1.430 (4)	C26—C26$^{\rm i}$	1.378 (6)
C25—C26	1.209 (4)		
C26—C25—C4	179.0 (3)	C25—C26—C26$^{\rm i}$	178.9 (3)

Symmetry code: (i) $1 - x, 1 - y, z$.

Compound (II)

Crystal data

$[Ag_2(SbF_6)_2(C_{52}H_{46})]$.-$2CH_2Cl_2$

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å

$M_r = 1527.98$
Monoclinic
$P2_1/n$
$a = 13.662$ (5) Å
$b = 11.267$ (5) Å
$c = 18.225$ (7) Å
$\beta = 105.83$ (3)°
$V = 2699.0$ (19) Å^3
$Z = 2$
$D_x = 1.880$ Mg m^{-3}
D_m not measured

Cell parameters from 48 reflections
$\theta = 10$–11°
$\mu = 1.982$ mm^{-1}
$T = 143$ (2) K
Prism
0.40 × 0.25 × 0.15 mm
Colourless

Data collection

Stoe Stadi-4 diffractometer
ω/θ scans
Absorption correction: none
4842 measured reflections
4714 independent reflections
3263 observed reflections [$I > 2\sigma(I)$]

$R_{\rm int} = 0.1477$
$\theta_{\rm max} = 24.94°$
$h = -16 \rightarrow 16$
$k = -13 \rightarrow 13$
$l = -13 \rightarrow 21$
3 standard reflections
frequency: 60 min
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.0662$
$wR(F^2) = 0.1824$
$S = 1.044$
4700 reflections
329 parameters
H atoms riding
$w = 1/[\sigma^2(F_o^2) + (0.0660P)^2 + 28.1746P]$
where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\rm max} < 0.001$
$\Delta\rho_{\rm max} = 2.099$ e Å^{-3}
$\Delta\rho_{\rm min} = -1.423$ 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)

Table 3. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å^2) for (II)*

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

	x	y	z	$U_{\rm eq}$
Ag	0.42504 (6)	0.26849 (7)	0.65832 (4)	0.0258 (2)
C1	0.2264 (9)	0.1772 (12)	0.4484 (6)	0.042 (3)
C2	0.1834 (9)	0.2869 (11)	0.4759 (6)	0.041 (3)
C3	0.1957 (8)	0.2932 (9)	0.5603 (6)	0.031 (2)
C4	0.1482 (8)	0.2173 (10)	0.5989 (6)	0.036 (2)
C5	0.1648 (8)	0.2244 (10)	0.6775 (7)	0.035 (2)
C6	0.2302 (8)	0.3063 (10)	0.7213 (6)	0.033 (2)
C7	0.2778 (7)	0.3854 (9)	0.6836 (6)	0.027 (2)
C8	0.2612 (8)	0.3795 (9)	0.6053 (6)	0.028 (2)
C9	0.2467 (9)	0.3110 (13)	0.8071 (7)	0.050 (3)
C10	0.3585 (9)	0.3200 (11)	0.8515 (6)	0.040 (3)
C11	0.4257 (8)	0.2303 (10)	0.8265 (5)	0.030 (2)
C12	0.4056 (8)	0.1112 (9)	0.8279 (5)	0.027 (2)
C13	0.4616 (8)	0.0299 (10)	0.7996 (6)	0.031 (2)
C14	0.5387 (8)	0.0620 (10)	0.7672 (6)	0.029 (2)
C15	0.5636 (8)	0.1844 (10)	0.7705 (5)	0.032 (2)
C16	0.5063 (7)	0.2659 (9)	0.7984 (5)	0.027 (2)
C17	0.5927 (9)	−0.0258 (11)	0.7330 (6)	0.041 (3)
C18	0.6042 (10)	0.0073 (12)	0.6541 (7)	0.046 (3)
C19	0.5078 (9)	0.0480 (10)	0.6001 (6)	0.035 (2)
C20	0.4209 (9)	−0.0198 (11)	0.5795 (6)	0.038 (2)
C21	0.3329 (9)	0.0196 (11)	0.5329 (6)	0.038 (2)
C22	0.3246 (9)	0.1353 (11)	0.5007 (5)	0.037 (2)
C23	0.4098 (8)	0.2050 (10)	0.5194 (5)	0.033 (2)
C24	0.5004 (8)	0.1638 (10)	0.5689 (6)	0.031 (2)
C25	0.0836 (10)	0.1255 (12)	0.5555 (7)	0.049 (3)
C26	0.0285 (11)	0.0461 (12)	0.5213 (8)	0.056 (4)
Sb	0.58458 (6)	0.57617 (7)	0.69089 (5)	0.0368 (2)
F1	0.5005 (6)	0.4696 (7)	0.6237 (5)	0.066 (2)
F2	0.6693 (7)	0.6780 (8)	0.7597 (5)	0.080 (3)
F3	0.4941 (6)	0.5657 (8)	0.7507 (5)	0.073 (3)
F4	0.5123 (6)	0.7020 (7)	0.6381 (5)	0.068 (2)
F5	0.6725 (6)	0.5810 (8)	0.6299 (5)	0.071 (2)
F6	0.6552 (5)	0.4465 (6)	0.7427 (5)	0.056 (2)
C100	0.3093 (14)	0.1526 (19)	1.0696 (11)	0.096 (6)
Cl1	0.4129 (4)	0.1112 (6)	1.0293 (3)	0.111 (2)
Cl2	0.1965 (3)	0.1730 (4)	0.9970 (2)	0.0769 (12)

Table 4. *Selected geometric parameters (Å, °) for (II)*

Ag—C7	2.550 (10)	Ag—C24	2.454 (10)
Ag—C8	2.516 (10)	Ag—F1	2.636 (8)
Ag—C15	2.559 (11)	C4—C25	1.45 (2)
Ag—C16	2.491 (8)	C25—C26	1.22 (2)
Ag—C23	2.584 (10)	C26—C26$^{\rm i}$	1.40 (3)
C26—C25—C4	177.6 (13)	C25—C26—C26$^{\rm i}$	175 (2)

Symmetry code: (i) $-x, -y, 1 - z$.

Data collection: *P*3 (Nicolet, 1987) (with Siemens LT-2 low-temperature attachment) for (I); *DIF*4 (Stoe & Cie, 1988*a*) (with Siemens LT-2 low-temperature attachment) for (II). Cell refinement: *P*3 for (I); *DIF*4 for (II). Data reduction: Nicolet *XDISK* (Nicolet, 1987) for (I); *REDU*4 (Stoe & Cie, 1988*b*) for (II). For both compounds, program(s) used to solve structures: *SHELXS*86 (Sheldrick, 1990); program(s) used to refine structures: *SHELXL*93 (Sheldrick, 1993); molecular graphics: *XP* (Siemens, 1994); software used to prepare material for publication: *SHELXL*93.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry, along with displacement ellipsoid diagrams have been deposited with the IUCr (Reference: AB1332). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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$[(CO)H(PPh_3)_2$-*arachno*-$OsB_3H_8]$

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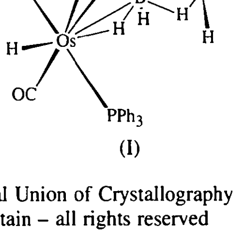
Abstract

A single-crystal X-ray diffraction study of the species 2-carbonyl-2-hydrido-*trans*-2,2-bis(triphenylphosphine)-2-osma-*arachno*-tetraborane confirms the structure as a tetraborane(10) butterfly with a *trans*-$[(CO)H(PPh_3)_2Os]$ group replacing a BH_2 group at a wing-tip or 2-position. It is compared with the previously reported ruthenaborane analogue $(CO)H(PPh_3)_2$-*arachno*-RuB_3H_8.

Comment

During our investigations of the chemistry of metallaboranes, we have prepared the osmahexaborane $(CO)(PPh_3)_2OsB_5H_9$, which is available in *ca* 80% yield from the reaction of LiB_5H_8 and $OsHCl(CO)(PPh_3)_3$ (Bould, Greenwood & Kennedy, 1983). We have found that the residues from the preparation show, from ^{11}B NMR, evidence of other metallaborane products and we have attempted to identify some of these. The products include the title compound, (I), an *arachno*-osmatetraborane cluster, and what appear to be a novel *arachno*-osmapentaborane cluster and a novel *nido*-osmadecaborane cluster. Compound (I) is formed in a very low yield (2%) and is more conveniently available in 65% yield from the reaction of TlB_3H_8 and $OsHCl(CO)(PPh_3)_3$ (Bould *et al.*, 1983).



(I)

The structure (Fig. 1) is typical of an *arachno* metallatetraborane with the metal in the 'wing-tip' position of the cluster with a distorted octahedral disposition of the ligands about the metal center (Kennedy, 1984; Alcock, Burns, Claire & Hill, 1992). The Os moiety thus replaces a BH_2 group in B_4H_{10}. This is in contrast to the 1-metallatetraboranes in which the metal moiety replaces a BH_3 group in the 1-position (Bould, Greenwood, Kennedy & McDonald, 1985; Bould, Kennedy & McDonald, 1992; Housecroft, Owen, Raithby & Shaykh, 1990). The overall dimensions of the cluster are very similar to those of the structurally characterized ruthenaborane analogue $(CO)H(PPh_3)_2$-*arachno*-RuB_3H_8 (Alcock *et al.*, 1992) (Table 3), with some small differences.

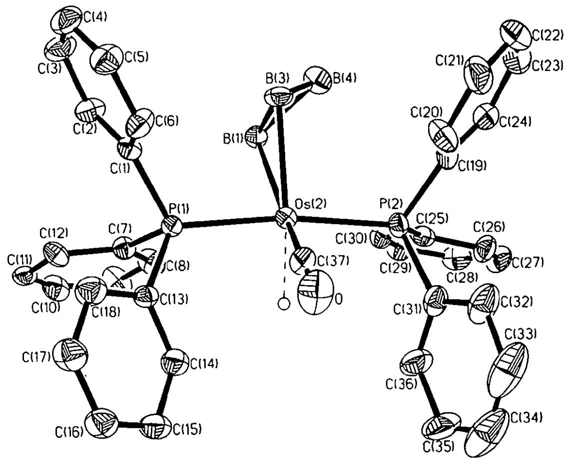


Fig. 1. The molecular structure and atomic labeling scheme for $[(CO)H(PPh_3)_2OsB_3H_8]$. Displacement ellipsoids are shown at the 30% probability level. The metal hydride position was located and refined, and is shown as a sphere of arbitrary size.

The *M*—B(1) and *M*—B(3) distances in the osmaborane are equal whereas the ruthenaborane shows a lengthening of the Ru—B vector *trans* to the metal hydride of 0.045 Å, which has been attributed by Alcock *et al.* (1992) to the *trans* effect of the hydride.

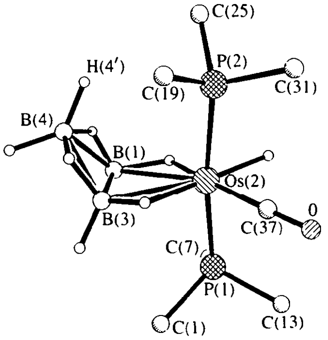


Fig. 2. An illustration of the metallaborane core of the molecule, including the H-atom positions.