C(51)	−0.0190 (5)	0.2647 (4)	0.8117 (5)	0.059 (2)
C(52)	−0.0714 (5)	0.2895 (3)	0.9133 (4)	0.054 (2)
C(53)	−0.1062 (5)	0.2321 (4)	0.9534 (4)	0.058 (2)
C(54)	−0.0898 (4)	0.1495 (3)	0.8935 (4)	0.046 (1)

† Partial occupancy (see below).

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

Re(1)—Cl(1)	2.331 (1)	P(1)—C(1)	1.834 (4)
Re(1)—Cl(2)	2.339 (1)	P(1)—C(7)	1.832 (5)
Re(1)—Cl(3)	2.291 (3)	P(1)—C(13)	1.829 (5)
Re(1)—Cl(4)	2.338 (1)	P(2)—C(19)	1.828 (5)
Re(1)—P(1)	2.580 (1)	P(2)—C(25)	1.830 (5)
Re(1)—P(2)	2.568 (1)	P(2)—C(31)	1.822 (5)
Re(1)—O(1)	1.71 (3)	P(3)—C(37)	1.828 (5)
Re(2)—Cl(5)	2.334 (1)	P(3)—C(43)	1.822 (5)
Re(2)—Cl(6)	2.301 (1)	P(3)—C(49)	1.833 (5)
Re(2)—P(3)	2.564 (1)		
Cl(1)—Re(1)—Cl(2)	88.43 (5)	Cl(5)—Re(2)—P(3^{i})	86.50 (4)
Cl(1)—Re(1)—Cl(3)	177.69 (8)	Cl(6)—Re(2)—Cl(6^{i})	180.0
Cl(1)—Re(1)—Cl(4)	88.51 (5)	Cl(6)—Re(2)—P(3)	93.02 (4)
Cl(1)—Re(1)—P(1)	88.59 (4)	Cl(6)—Re(2)—P(3^{i})	86.98 (4)
Cl(1)—Re(1)—P(2)	92.22 (4)	P(3)—Re(2)—P(3^{i})	180.0
Cl(1)—Re(1)—O(1)	174.1 (7)	Re(1)—P(1)—C(1)	112.4 (1)
Cl(2)—Re(1)—Cl(3)	93.86 (7)	Re(1)—P(1)—C(7)	118.0 (2)
Cl(2)—Re(1)—Cl(4)	176.54 (5)	Re(1)—P(1)—C(13)	112.3 (1)
Cl(2)—Re(1)—P(1)	96.88 (4)	C(1)—P(1)—C(7)	105.2 (2)
Cl(2)—Re(1)—P(2)	84.33 (4)	C(1)—P(1)—C(13)	104.8 (2)
Cl(2)—Re(1)—O(1)	86.5 (7)	C(7)—P(1)—C(13)	103.0 (2)
Cl(3)—Re(1)—Cl(4)	89.20 (7)	Re(1)—P(2)—C(19)	111.0 (1)
Cl(3)—Re(1)—P(1)	90.90 (6)	Re(1)—P(2)—C(25)	116.1 (2)
Cl(3)—Re(1)—P(2)	88.24 (6)	Re(1)—P(2)—C(31)	114.4 (1)
Cl(4)—Re(1)—P(1)	84.67 (4)	C(19)—P(2)—C(25)	103.2 (2)
Cl(4)—Re(1)—P(2)	94.17 (4)	C(19)—P(2)—C(31)	104.7 (2)
Cl(4)—Re(1)—O(1)	96.7 (7)	C(25)—P(2)—C(31)	106.3 (2)
P(1)—Re(1)—P(2)	178.56 (4)	Re(2)—P(3)—C(37)	112.7 (1)
P(1)—Re(1)—O(1)	89.2 (6)	Re(2)—P(3)—C(43)	113.2 (2)
P(2)—Re(1)—O(1)	90.1 (6)	Re(2)—P(3)—C(49)	116.2 (1)
Cl(5)—Re(2)—Cl(5^{i})	180.0	C(37)—P(3)—C(43)	105.6 (2)
Cl(5)—Re(2)—Cl(6)	91.34 (5)	C(37)—P(3)—C(49)	104.7 (2)
Cl(5)—Re(2)—Cl(6^{i})	88.66 (5)	C(43)—P(3)—C(49)	103.3 (2)
Cl(5)—Re(2)—P(3)	93.50 (4)		

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

There are 1.5 molecules in the asymmetric unit, one in a general position and one at a centre of symmetry. The oxo ligand was detectable only at one Cl site of the molecule in a general position. An anomalous displacement parameter and a short Re—Cl distance for Cl(3) indicated the presence of an oxo ligand at this site. A partial O atom, O(1), was included in the model. The population parameter of atom Cl(3) was refined with the constraint that the Cl(3) and O(1) populations summed to unity. The final values of the population parameters were 0.797 (8) for Cl(3) and 0.203 for O(1). The largest peaks and troughs on the final difference map were near the Re atom.

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1988). Cell refinement: *MSC/AFC Diffractometer Control Software*. Data reduction: *TEXSAN* (Molecular Structure Corporation, 1985, 1992). Program(s) used to solve structure: *DIRDIF* (Beurskens *et al.* 1994). Program(s) used to refine structure: *TEXSAN*. Software used to prepare material for publication: *TEXSAN*.

Financial support was provided by grants from the Natural Sciences and Engineering Research Council of Canada and the DuPont Merck Pharmaceutical Company, both of whom the authors gratefully thank.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry, including hydrogen-bonding parameters, have been deposited with the IUCr (Reference: FG1149). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

Beurskens, P. T., Admiraal, G., Beurskens, G., Bosman, W. P., de Gelder, R., Israël, R. & Smits, J. M. M. (1994). *The DIRDIF Program System*. Technical Report. Crystallography Laboratory, University of Nijmegen, The Netherlands.

Lebuis, A.-M. & Beauchamp, A. L. (1993). *Can. J. Chem.* **71**, 441–449.

Luo, H. & Orvig, C. (1996). *Can. J. Chem.* In the press.

Molecular Structure Corporation (1985, 1992). *TEXSAN. TEXRAY Structure Analysis Package*. MSC, 3200 Research Forest Drive, The Woodlands, TX 77381, USA.

Molecular Structure Corporation (1988). *MSC/AFC Diffractometer Control Software*. MSC, 3200 Research Forest Drive, The Woodlands, TX 77381, USA.

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

Rouschias, G. & Wilkinson, G. (1966). *J. Chem. Soc. A*, pp. 465–472.

Yoon, K., Parkin, G. & Rheingold, A. L. (1992). *J. Am. Chem. Soc.* **114**, 2210–2218.

Zachariasen, W. H. (1967). *Acta Cryst.* **23**, 558–564.

Acta Cryst. (1996). C**52**, 1380–1384

(Z)-2,3-Bis(4′-[2.2.2]paracyclophanyl)but-2-ene and μ-η^6:η^6-[(Z)-2,3-Bis(4′-[2.2.2]paracyclophanyl)but-2-ene]-bis[(hexafluoroantimonato-*F*)silver(I)] Toluene Dichloromethane (1/1/1)

Peter G. Jones,[a] Peter Bubenitschek,[b] Fenton Heirtzler[b]† and Henning Hopf[b]

[a]*Institut für Analytische und Anorganische Chemie, Technische Universität Braunschweig, Postfach 3329, 38023 Braunschweig, Germany, and* [b]*Institut für Organische Chemie, Technische Universität Braunschweig, Postfach 3329, 38023 Braunschweig, Germany. E-mail: p.jones@tu-bs.de*

(*Received 2 October 1995; accepted 18 December 1995*)

Abstract

The free ligand, $C_{52}H_{52}$, displays approximate twofold symmetry, with a C═C bond of 1.337 (4) Å and essentially planar olefin geometry. The (methyl C)–(olefin

---

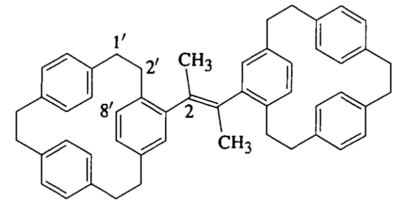
† Present address: Institut für Anorganische Chemie der Universität, Spitalstr. 51, CH-4056 Basel, Switzerland.

Acta Crystallographica Section C
ISSN 0108-2701 © 1996

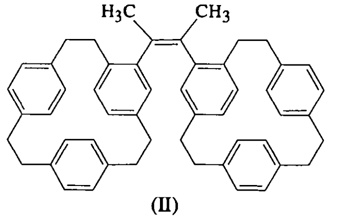
C)–(cyclophane C) angles are narrow [113.0 (2)°]. In the disilver(I) complex, [Ag$_2$(SbF$_6$)$_2$(C$_{52}$H$_{52}$)].C$_7$H$_8$.CH$_2$Cl$_2$, the olefin geometry is not greatly changed. The Ag—C distances vary from 2.498 to 2.696 (5) Å and there is a short Ag—F contact of 2.545 (4) Å. The dichloromethane molecule occupies the cleft between the primary aromatic rings.

Comment

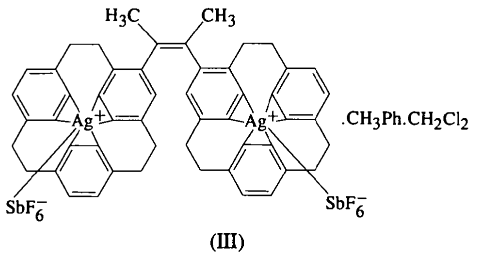
We have recently described the preparation of (*E*)- and (*Z*)-2,3-bis(4′-[2.2.2]paracyclophanyl)but-2-ene, (I) and (II), respectively (Heirtzler, Hopf, Jones & Bubenitschek, 1995*a*). [2.2.2]Paracyclophane and other π-electron-rich hydrocarbons possessing a cavity-forming topology are known to form endohedral π-complexes with silver(I) and other soft metal atoms (Cohen-Addad, Baret, Chautemps & Pierre, 1983; Heirtzler, Hopf, Jones & Bubenitschek, 1995*b*; Faust, 1995). The presence of the rigid (*E*)-2,3-but-2-endiyl spacer in (I) resulted in complicated dynamic behaviour in solution, which was influenced by various silver(I) salts. We have previously published the crystal structure analyses of (I) and its disilver(I) complex (Heirtzler *et al.*, 1995*b*) and now report the structures of the sterically congested diastereomer (II) and its bis[silver(I) hexafluoroantimonate] complex [Ag$_2$(SbF$_6$)$_2$(C$_{52}$H$_{52}$)], (III). A further topic of interest was the possibility that the primary aryl groups of the (*Z*)-configured (II) could function as an additional π-binding cleft (Gano, Subramaniam & Birnbaum, 1990).



(I)



(II)



(III)

Compound (II) (Fig. 1)‡ exhibits approximate twofold symmetry (Fig. 2), although the ring orientation in the two halves of the molecule is somewhat different. The central C═C bond length is normal [1.337 (4) Å] and its environment is planar to within 0.016 Å (r.m.s. deviation calculated for six atoms). The twist angle about the double bond is 2.8 (5)° [*cf.* torsion angle C4—C49—C50—C28 2.7 (5)°]. Major deformations from ideal 120° angles are observed, with the angles C4—C49—C51 and C28—C50—C52 very narrow at 113.0 (2)°. Similar effects were observed in (*Z*)-2,3-diphenyl-2-butene (Fronczek, Swan, Corkern & Gandour, 1984), with a twist angle of 4° and Me—C—Ph angles of 114.6 (7)°.

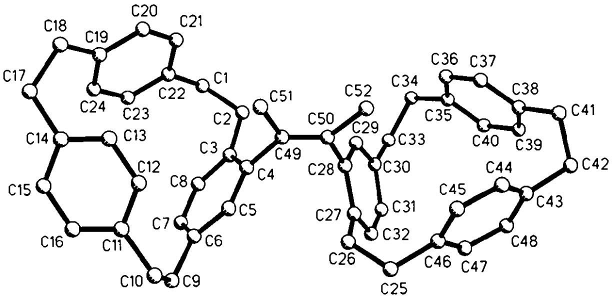


Fig. 1. The molecule of compound (II) in the crystal. Atomic radii are arbitrary. H atoms are omitted for clarity.

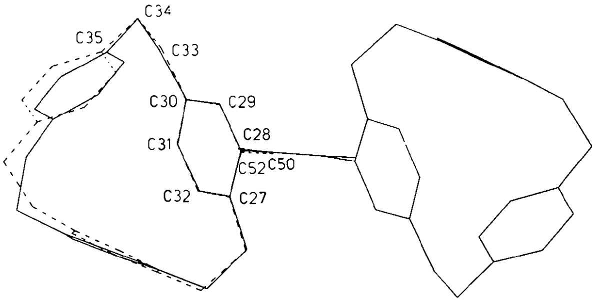


Fig. 2. Superposition of the two halves of (II) (the right-hand side of the molecule is superposed as dotted bonds on the left-hand side; only the labelled atoms were fitted).

The dihedral angles formed between the primary rings of the [2.2.2]paracyclophanyl substituents (C3–C8 and C27–C32) and the central double bond (defined by the planes C4—C49—C51 and C28—C50—C52), 73.2 (2)° and 75.6 (2)°, indicate extensive deconjugation of these groups [*cf.* individual torsion angles C3—C4—C49—C50 and C27—C28—C50—C49, both −79.6 (4)°]. The situation is intermediate between that in (*Z*)-2,3-diphenyl-2-butene (Fronczek *et al.*, 1984) and in the sterically demanding stilbene derivative (*Z*)-2,2,5,5-tetra-

---

‡ Crystallographic numbering does not correspond to the IUPAC numbering.

methyl-3,4-diphenyl-3-hexene (Gano, Park, Pinkerton & Lenoir, 1991), in which these angles are 57.6/49.3 and 86.9/85.1°, respectively. The interplanar angle between the primary rings is 69.8 (3)° in (II).

The disilver(I) complex (III) (Fig. 3) displays exact twofold symmetry. This contrasts with the inversion symmetry observed for the bis[silver(I) hexafluoroantimonate] complex of the (*E*)-configured olefin (I) (Heirtzler *et al.*, 1995*a*) and is consistent with the respective idealized olefin configurations. The structure also contains one molecule of toluene, disordered over an inversion centre, and one molecule of dichloromethane with exact twofold symmetry.

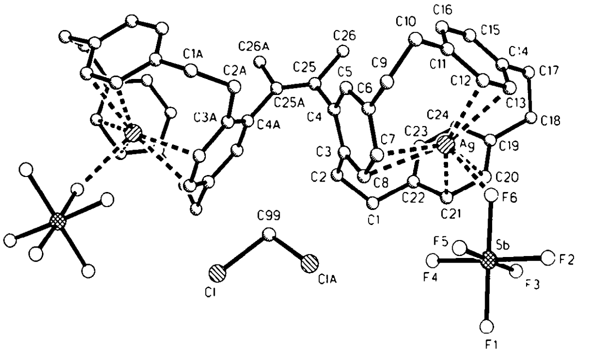


Fig. 3. The formula unit of compound (III) in the crystal. Atomic radii are arbitrary. H atoms and toluene are omitted for clarity. Atom labels appended by 'A' indicate symmetry-equivalent atoms.

The interplanar angle between the primary aryl residue (C3–C8) and the C=C bond is 64.5 (3)° [*cf.* individual torsion angle C3—C4—C25—C25*A* 71.2 (8)°]; between the two symmetry-related rings the angle is 64.2 (2)°. Both values are slightly less than in the free ligand (II). The length of the olefinic C=C bond is essentially unchanged at 1.332 (10) Å, and its environment remains planar to within 0.018 Å (r.m.s. deviation of six atoms), with a twist angle of 1.8 (9)° [*cf.* torsion angle C4—C25—C25*A*—C4*A* −4.9 (11)°]. The size of the cleft between the two arenes remains virtually unchanged.

The binding geometry and environment of silver resembles that in known complexes of [2.2.2]paracyclophanes (Heirtzler *et al.*, 1995*b*; Cohen-Addad *et al.*, 1983). The Ag···C interactions involve one C—C bond per ring; the shortest and longest are to C7 and C8 [2.498 (5) and 2.696 (5) Å, respectively], whereas all others are more symmetric. The Ag atom is displaced by 0.084 (2) Å from the edge of the cavity (defined as the mean plane of the six coordinating C atoms). The intramolecular Ag···Ag distance is 10.055 (2) Å.

In contrast to some other bis[silver(I) hexafluoroantimonate] complexes based on bis-[2.2.2]paracyclophanyl systems (Heirtzler *et al.*, 1995*b*), no toluene is associated *via* short contacts to the Ag^+ centres in the crystal lattice of (III). Instead, close contacts to the SbF_6^- anion are observed: Ag—F6 2.545 (4) Å. Furthermore, the dichloromethane is bound on the edge of the cleft formed by the *cis*-configured junction of the two [2.2.2]-paracyclophanyl residues. The closest nonbonding C···C contacts here are C99···C7 3.974 (10) and C99···C8 3.599 (12) Å, with corresponding Cl···C(1 − *x*, *y*, 0.5 − *z*) contacts of 3.890 (6) and 3.901 (6) Å. As noted above, the Ag···C distances involving these two atoms exhibit the greatest variation. Complexation of chloroform to the exterior of another cavity-forming π-electron-rich hydrocarbon has already been documented (Vögtle, Gross, Seel & Nieger, 1992). However, our attempts to direct silver(I) to this site, through combination of (II) with one or three equivalents of silver(I) hexafluoroantimonate, always resulted in the isolation of complex (III).

Experimental

Compound (II) was synthesized and purified as previously described (Heirtzler *et al.*, 1995*a*). Single crystals were obtained by slow evaporation of a dichoromethane–2-propanol solution. Single crystals of complex (III) formed after combining two equivalents of silver hexafluoroantimonate in toluene with (II) in dichloromethane and allowing the solution to stand undisturbed for 20 d under anhydrous conditions.

Compound (II)

Crystal data

$C_{52}H_{52}$
$M_r = 676.94$
Monoclinic
$P2_1/c$
$a = 9.692$ (3) Å
$b = 13.232$ (4) Å
$c = 29.968$ (7) Å
$\beta = 91.31$ (3)°
$V = 3842.2$ (19) Å^3
$Z = 4$
$D_x = 1.170$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 60 reflections
$\theta = 10$–11°
$\mu = 0.066$ mm^{-1}
$T = 178$ (2) K
Tablet
0.70 × 0.55 × 0.15 mm
Colourless

Data collection

Stoe Stadi-4 diffractometer
ω/θ scans
Absorption correction: none
12427 measured reflections
6795 independent reflections
4430 observed reflections [$I > 2\sigma(I)$]

$R_{\rm int} = 0.0459$
$\theta_{\rm max} = 25.05$°
$h = -11 \rightarrow 11$
$k = -14 \rightarrow 15$
$l = -35 \rightarrow 35$
3 standard reflections
frequency: 60 min
intensity decay: 6%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.0693$
$wR(F^2) = 0.1957$
$S = 1.069$
6757 reflections
471 parameters

$w = 1/[\sigma^2(F_o^2) + (0.0626P)^2 + 4.0927P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} = 0.003$
$\Delta\rho_{\rm max} = 0.863$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.511$ e Å^{-3}

Methyl groups treated as rigid, other H atoms riding

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

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

	x	y	z	U_{eq}
C1	0.5404 (3)	0.3111 (3)	0.09287 (11)	0.0421 (8)
C2	0.5646 (3)	0.3573 (3)	0.13975 (10)	0.0407 (8)
C3	0.6799 (3)	0.4335 (2)	0.14178 (9)	0.0324 (7)
C4	0.7879 (3)	0.4278 (2)	0.17327 (9)	0.0305 (6)
C5	0.8996 (3)	0.4942 (2)	0.16989 (9)	0.0339 (7)
C6	0.9084 (3)	0.5662 (2)	0.13657 (9)	0.0339 (7)
C7	0.7973 (3)	0.5739 (2)	0.10702 (10)	0.0367 (7)
C8	0.6856 (3)	0.5094 (2)	0.10968 (10)	0.0373 (7)
C9	1.0325 (3)	0.6331 (3)	0.13198 (11)	0.0439 (8)
C10	1.1721 (3)	0.5795 (3)	0.13569 (11)	0.0438 (8)
C11	1.1823 (3)	0.4898 (2)	0.10472 (10)	0.0366 (7)
C12	1.1703 (3)	0.3914 (3)	0.11986 (10)	0.0387 (7)
C13	1.1661 (3)	0.3099 (3)	0.09108 (10)	0.0392 (7)
C14	1.1736 (3)	0.3237 (3)	0.04524 (10)	0.0404 (8)
C15	1.1904 (4)	0.4219 (3)	0.03001 (11)	0.0485 (9)
C16	1.1949 (4)	0.5028 (3)	0.05897 (10)	0.0478 (9)
C17	1.1620 (4)	0.2357 (3)	0.01304 (11)	0.0494 (9)
C18	1.0591 (4)	0.1535 (3)	0.02565 (11)	0.0465 (8)
C19	0.9210 (3)	0.1916 (2)	0.04126 (10)	0.0369 (7)
C20	0.8487 (3)	0.1400 (2)	0.07369 (10)	0.0401 (7)
C21	0.7259 (3)	0.1771 (3)	0.08968 (10)	0.0408 (8)
C22	0.6701 (3)	0.2670 (2)	0.07377 (10)	0.0354 (7)
C23	0.7403 (3)	0.3172 (2)	0.04083 (10)	0.0366 (7)
C24	0.8632 (3)	0.2805 (2)	0.02483 (10)	0.0391 (7)
C25	0.8604 (5)	0.5826 (4)	0.32562 (14)	0.087 (2)
C26	0.8193 (4)	0.5510 (3)	0.28031 (12)	0.0628 (11)
C27	0.6681 (4)	0.5288 (3)	0.27103 (10)	0.0419 (8)
C28	0.6242 (3)	0.4339 (2)	0.25549 (9)	0.0313 (6)
C29	0.4840 (3)	0.4120 (3)	0.25291 (9)	0.0364 (7)
C30	0.3832 (3)	0.4807 (3)	0.26531 (10)	0.0439 (8)
C31	0.4281 (4)	0.5760 (3)	0.27878 (11)	0.0511 (9)
C32	0.5661 (4)	0.5988 (3)	0.28081 (11)	0.0526 (9)
C33	0.2333 (4)	0.4523 (3)	0.26643 (12)	0.0596 (11)
C34	0.2029 (3)	0.3472 (3)	0.28540 (12)	0.0589 (10)
C35	0.2762 (3)	0.3259 (3)	0.32919 (11)	0.0436 (8)
C36	0.3779 (4)	0.2527 (3)	0.33227 (12)	0.0487 (9)
C37	0.4529 (4)	0.2378 (3)	0.37124 (12)	0.0481 (9)
C38	0.4283 (3)	0.2939 (2)	0.40928 (11)	0.0410 (8)
C39	0.3239 (3)	0.3655 (3)	0.40689 (11)	0.0411 (8)
C40	0.2501 (3)	0.3819 (3)	0.36738 (10)	0.0407 (8)
C41	0.5147 (4)	0.2780 (3)	0.45120 (12)	0.0524 (9)
C42	0.5823 (4)	0.3741 (3)	0.46942 (10)	0.0481 (9)
C43	0.6624 (3)	0.4288 (3)	0.43430 (9)	0.0368 (7)
C44	0.7813 (3)	0.3884 (3)	0.41661 (11)	0.0442 (8)
C45	0.8463 (3)	0.4353 (3)	0.38166 (11)	0.0510 (9)
C46	0.7951 (4)	0.5250 (3)	0.36359 (11)	0.0562 (10)
C47	0.6791 (4)	0.5656 (3)	0.38212 (12)	0.0524 (9)
C48	0.6148 (3)	0.5184 (2)	0.41658 (10)	0.0404 (7)
C49	0.7969 (3)	0.3476 (2)	0.20856 (9)	0.0317 (7)
C50	0.7250 (3)	0.3514 (2)	0.24607 (9)	0.0325 (7)
C51	0.8992 (3)	0.2657 (3)	0.19818 (11)	0.0404 (8)
C52	0.7366 (3)	0.2745 (2)	0.28323 (10)	0.0380 (7)

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

C4—C49	1.499 (4)	C49—C51	1.506 (4)
C28—C50	1.497 (4)	C50—C52	1.510 (4)
C49—C50	1.337 (4)		
C50—C49—C4	122.9 (3)	C49—C50—C28	122.7 (3)
C50—C49—C51	124.0 (3)	C49—C50—C52	124.3 (3)
C4—C49—C51	113.0 (2)	C28—C50—C52	113.0 (2)

Compound (III)

Crystal data

$[Ag_2(SbF_6)_2(C_{52}H_{52})].C_7H_8.-CH_2Cl_2$
$M_r = 1541.24$
Monoclinic
$C2/c$
$a = 17.288$ (3) Å
$b = 12.159$ (2) Å
$c = 27.527$ (4) Å
$\beta = 97.06$ (2)°
$V = 5742.4$ (16) Å^3
$Z = 4$
$D_x = 1.783$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 52 reflections
$\theta = 10–11.5°$
$\mu = 1.774$ mm^{-1}
$T = 163$ (2) K
Prism
0.60 × 0.45 × 0.20 mm
Colourless

Data collection

Stoe Stadi-4 diffractometer
ω/θ scans
Absorption correction: ψ scans (*XEMP*; Nicolet, 1987)
$T_{min} = 0.686$, $T_{max} = 0.990$
5281 measured reflections
5084 independent reflections

4376 observed reflections [$I > 2\sigma(I)$]
$R_{int} = 0.0171$
$\theta_{max} = 25.09°$
$h = 0 \rightarrow 20$
$k = -14 \rightarrow 1$
$l = -32 \rightarrow 32$
3 standard reflections
frequency: 60 min
intensity decay: 4%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.0429$
$wR(F^2) = 0.1131$
$S = 1.052$
5068 reflections
338 parameters
Methyl groups treated as rigid, others H atoms riding

$w = 1/[\sigma^2(F_o^2) + (0.0431P)^2 + 55.1849P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = -0.009$
$\Delta\rho_{max} = 0.963$ e Å^{-3}
$\Delta\rho_{min} = -0.997$ 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 (III)*

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

	x	y	z	U_{eq}
Ag	0.73437 (2)	0.48430 (3)	0.377709 (14)	0.03136 (12)
C1	0.7260 (3)	0.4828 (5)	0.2319 (2)	0.0321 (11)
C2	0.6380 (3)	0.5074 (4)	0.2301 (2)	0.0299 (11)
C3	0.6064 (3)	0.4888 (4)	0.2783 (2)	0.0277 (10)
C4	0.5552 (3)	0.5626 (4)	0.2965 (2)	0.0250 (10)
C5	0.5353 (3)	0.5462 (4)	0.3438 (2)	0.0283 (10)
C6	0.5627 (3)	0.4587 (4)	0.3733 (2)	0.0282 (10)
C7	0.6103 (3)	0.3818 (4)	0.3537 (2)	0.0314 (11)
C8	0.6311 (3)	0.3973 (4)	0.3067 (2)	0.0310 (11)
C9	0.5457 (3)	0.4487 (5)	0.4258 (2)	0.0362 (12)
C10	0.5648 (3)	0.5535 (5)	0.4563 (2)	0.0354 (12)
C11	0.6418 (3)	0.6051 (4)	0.4496 (2)	0.0308 (11)
C12	0.7125 (3)	0.5495 (4)	0.4614 (2)	0.0318 (11)
C13	0.7827 (3)	0.5996 (5)	0.4547 (2)	0.0348 (12)
C14	0.7848 (3)	0.7071 (4)	0.4362 (2)	0.0343 (11)
C15	0.7150 (3)	0.7614 (4)	0.4253 (2)	0.0384 (12)
C16	0.6446 (3)	0.7112 (4)	0.4311 (2)	0.0356 (12)

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.

References

Cohen-Addad, C., Baret, P., Chautemps, P. & Pierre, J.-L. (1983). *Acta Cryst.* C**39**, 1346–1349.

Faust, R. (1995). *Angew. Chem.* **107**, 1559–1562.

Fronczek, F. R., Swan, A. M., Corkern, J. A. & Gandour, R. D. (1984). *Acta Cryst.* C**40**, 1875–1876.

Gano, J. E., Park, B.-S., Pinkerton, A. A. & Lenoir D. (1991). *Acta Cryst.* C**47**, 162–164.

Gano, J. E., Subramaniam, G. & Birnbaum, R. (1990). *J. Org. Chem.* **55**, 4760–4763.

Heirtzler, F. R., Hopf, H., Jones, P. G. & Bubenitschek, P. (1995*a*). *Tetrahedron Lett.* **36**, 1239–1242.

Heirtzler, F. R., Hopf, H., Jones, P. G. & Bubenitschek, P. (1995*b*). *Chem. Ber.* pp. 1079–1082.

Nicolet (1987). *XEMP. X-ray Program System Manuals.* Nicolet Analytical X-ray Instruments, Madison, USA.

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

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

Siemens (1994). *XP. Molecular Graphics Program.* Siemens Analytical X-ray Instruments Inc., Madison, USA.

Stoe & Cie (1988*a*). *DIF4. Diffractometer Control Program.* Version 6.2. Stoe & Cie, Darmstadt, Germany.

Stoe & Cie (1988*b*). *REDU4. Data Reduction Program.* Version 6.2. Stoe & Cie, Darmstadt, Germany.

Vögtle, F., Gross, J., Seel, C. & Nieger, M. (1992). *Angew. Chem.* **104**, 1112–1113.

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

PETER G. JONES,[a] FENTON HEIRTZLER[b]† AND HENNING HOPF[b]

[a]*Institut für Analytische und Anorganische Chemie, Technische Universität Braunschweig, Postfach 3329, 38023 Braunschweig, Germany, and* [b]*Institut für Organische Chemie, Technische Universität Braunschweig, Postfach 3329, 38023 Braunschweig, Germany. E-mail: p.jones@tu-bs.de*

(*Received 19 October 1995; accepted 18 December 1995*)

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

---

† Present address: Institut für Anorganische Chemie der Universität, Spitalstr. 51, CH-4056 Basel, Switzerland.

Acta Crystallographica Section C
ISSN 0108-2701 © 1996