Larger Cyclophanes: Synthesis and Structural Characterization of [2.2.2.2]Paracyclophane Compounds with $SbBr_3$ and $BiBr_3$

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Introduction

Detailed studies of intramolecular charge-transfer (CT) interactions have enabled most important information about conformational effects in charge-transfer (CT) phenomena to be obtained.

A specific example is that of cyclophane molecules in which two (or more) benzene rings are forced to remain close to one-another by connecting -$(CH_2)_n$ chains of different lengths. In fact they present two predominant effects, namely distortion of benzene rings and transannular π-electronic interactions between couples of rings, which are most marked in smaller cyclophanes.[2]

In a previous paper, to study the effects of these properties connected with the reactivity and π-basicity of smaller cyclophanes, we reported the synthesis and the properties of the compounds of [2.2]paracyclophane with $BiBr_3$ of formula $2BiBr_3$:[2.2]paracyclophane and of [2.2]paracyclophane-pyrene with the $BiBr_3$ of formula Bi_2Br_6:[2.2]paracyclophane:pyrene.[3]

Unlike the smaller cyclophanes, the larger ones, synthetic host compounds, are normal as far as benzene ring planarity is concerned and contain an intramolecular cavity of well-defined structure.[2]

To separate the effects of the benzene rings distortion from the transannular π-electronic interactions and the effects of their molecular structure, less rigid than those of smaller cyclophanes on the CT compounds formation, we synthesized and characterized [2.2.2.2]paracyclophane, synthetic host, and its compounds with $SbBr_3$ and $BiBr_3$ of formula $SbBr_3$:[2.2.2.2]paracyclophane **1** and Bi_2Br_6:[2.2.2.2]paracyclophane:toluene **2** respectively.

Larger cyclophanes can form two types of compounds with π-acceptors:

(1) CT compounds with π-electron acceptors, organic and inorganic, coordinated to the benzene rings from the outside.[4−9] The compounds described in this paper belong to this class.

Table 1. Experimental Data for Crystallographic Analysis

compound	$(C_{32}H_{32})SbBr_3$ (**1**)	$(C_{32}H_{32})Bi_2Br_6\cdot C_7H_8$ (**2**)
empirical formula	$C_{32}H_{32}Br_3Sb$	$C_{39}H_{40}Br_6Bi_2$
M	778.1	1406.1
space group	$P2_1/n$	$P\bar{1}$
a (Å)	13.024(3)	12.513(4)
b (Å)	11.962(2)	12.957(4)
c (Å)	19.795(5)	7.165(3)
α (deg)	90	90.76(2)
β (deg)	100.58(3)	101.13(3)
γ (deg)	90	112.95(3)
V (Å^3)	3032(1)	1044(7)
Z	4	1
D_{calc} (g cm^{-3})	1.70	2.23
T (K)	273	273
λ (Å)	Mo Kα, 0.7107	Mo Kα, 0.7107
μ (cm^{-1})	48.8	141.8
conditions for obs reflcns	$F_o > 4\sigma(F_o)$	$F_o > 4\sigma(F_o)$
no. of unique reflcns	2612	2228
$R = \sum\|\Delta F\|/\sum\|F_o\|$	0.0676	0.0445
wR[a]	0.287[b]	0.1093[c]
a, b in w	0.1709, 15.13	0.0, 0.0

[a] $w = 1/[\sigma^2(F_o^2) + (aP)^2 + bP]$; $P = (\mathrm{Max}(F_o^2,0) + 2F_c^2)/3$. [b] $[\sum(w(F_o^2 - F_c^2)]^2/\sum[w(F_o^2)^2]]^{1/2}$. [c] $(\sum w(F_o - F_c)^2/\sum wF_o^2)^{1/2}$.

(2) Inclusion compounds with appropriate inorganic and organic guests lodged in the cavity.[10−12]

Larger cyclophanes have received a great deal of attention for their practical applications in bioinorganic catalysis and have an important role in the environmental technology for their capacity to sequester toxic compounds selectively.[2,13]

Experimental Section

The [2.2.2.2]paracyclophane was prepared by hydrogenation at atmospheric pressure of [2.2.2.2]paracyclophane-1,9,17,25-tetraene according to a literature method.[14] The product was characterized with ^{1}H NMR and IR spectroscopies and its spectra compared with those of the authentic samples.

All chemicals were reagent grade. The $SbBr_3$ and $BiBr_3$ were recrystallized from ethylbenzene and dried over P_4O_{10}. All solvents were dehydrated and kept on metallic sodium. All manipulations were performed in a drybox.

Synthesis of the Compounds. Compound **1** was obtained by adding to a hot para-xylene solution (80 °C) of $SbBr_3$ the solid [2.2.2.2]-paracyclophane with a metal to ligand molar-ratio ranging from 3:1 to 1:1 under vigorous stirring. Colorless crystals suitable for X-ray single-crystal diffraction analysis were separated after slow cooling to room temperature. Calcd for $C_{32}H_{32}SbBr_3$: C, 49.35; H, 4.15. Found: C, 49.20; H, 4.10.

Compound **2** was prepared by mixing a solution of $BiBr_3$ in toluene with a solution of [2.2.2.2]-paracyclophane in the same solvent at 70 °C.

The metal-to-ligand molar ratio ranging from 3:1 to 1:1, but only the 2:1 compound could be isolated.

The solution was refluxed for 4 h at 80 °C and successively was slowly cooled at room temperature. After few days, yellow crystals suitable for X-ray single-crystal diffraction analysis were separated.

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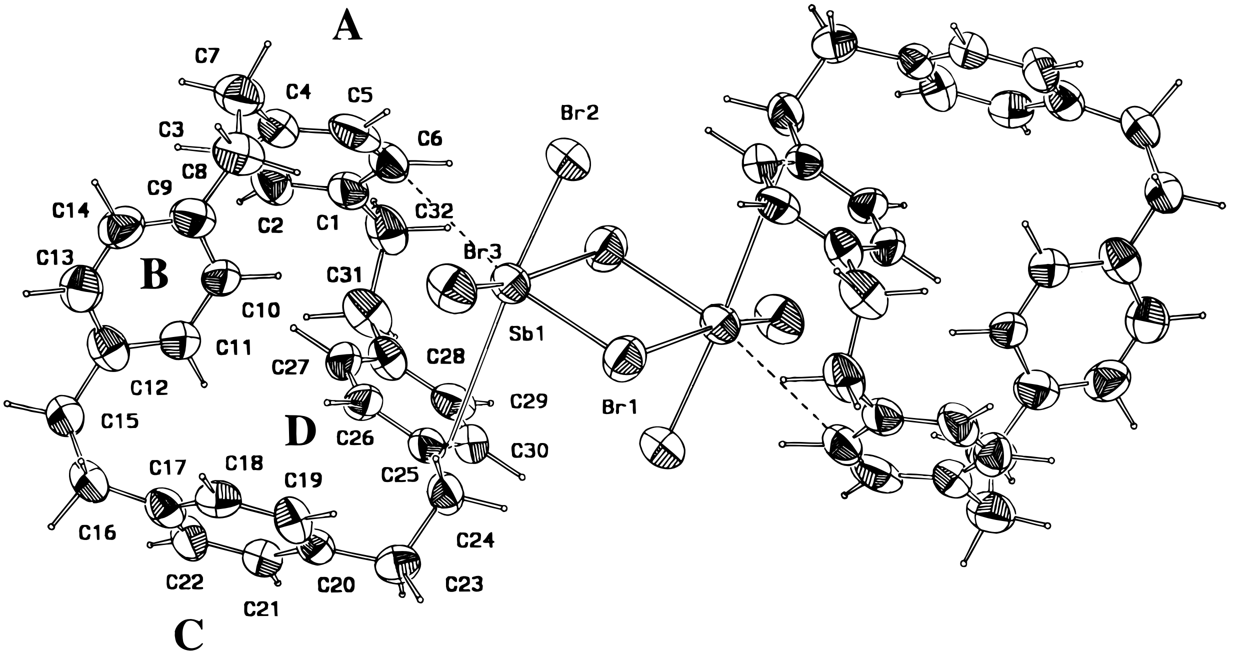

Figure 1. ORTEP drawing of compound **1** with thermal ellipsoids at 50% probability level.

Table 2. More Relevant Bond Distances (Å) and Angles (deg) with ESDs in Parentheses for **1**[a]

Sb−Br2	2.528(2)	Sb−C26	3.47(1)
Sb−Br1	2.539(2)	Sb−C27	3.67(1)
Sb−Br3	2.522(2)	Sb−C28	3.72(1)
Sb−Br1^i	3.733(2)	Sb−C29	3.56(2)
Sb−C25	3.34(1)	Sb−C30	3.36(1)
Br3−Sb−Br1^i	171.8(1)	Br2−Sb−Br1^i	78.2(1)
Br1−Sb Br1^i	84.6(1)	Br2−Sb−Br3	93.6(1)
Br1−Sb−Br3	96.9(1)	Br2−Sb−Br1	95.4(1)
C4−C7−C8−C9	−66(2)	C20−C23−C24−C25	−66(2)
C12−C15−C16−C17	−61(2)	C28−C31−C32−C1	−60(2)

[a] i: −*x*, −*y*, 1 − *z*.

The crystals were washed with hexane and dried over P_4O_{10}. Calcd for $C_{39}H_{40}Bi_2Br_6$: C, 33.28; H, 2.87. Found: C, 33.10; H, 2.67.

Physical Measurements. IR spectra were recorded with an FT-IR Bruker 113-V spectrophotometer in KBr pellets in the range 4000−400 cm^{-1} and in polyethylene pellets in the range 500−60 cm^{-1}. Carbon and hydrogen were analyzed with a Carlo-Erba model 1106 elemental analyzer.

Crystal Structure Determination. Crystal data and information relevant to data collection and structural determination are given in Table 1. The selected crystals, sealed in a glass capillary, were examined by X-ray diffraction using an automated four-circle Philips diffractometer for **1** and an Enraf Nonius CAD4 diffractometer for **2**, in $\theta-2\theta$ scan mode with graphite monochromated MoKα radiation (λ = 0.7107 Å).

In both compounds a correction for absorption was applied.[15]

The structures were solved by direct methods using SIR92[16] for **1** and the three-dimensional Patterson analysis for **2**. In the final refinements all non-hydrogen atoms were anisotropically refined.

Hydrogens were calculated with standard geometries for **1** and not considered for **2**. In **2** an unexpected toluene molecule was found. It was refined as a rigid body, statistically distributed in two positions around a symmetry center with s.o.f 0.5.

All calculations were performed on ENCORE 91 computer of the Centro di Studio per la Strutturistica Diffrattometrica del CNR (Parma) using SHELX93,[17] PARST,[18] ORTEP,[19] and PLUTO[20] programs. Atomic scattering factors were taken from ref 21.

Results and Discussion

The thermogravimetric analysis shows that the compounds **1** and **2** are stable up to the melting point (160 and 175 °C, respectively), decomposition immediately following.

The IR spectra of [2.2.2.2]paracyclophane and of its compounds with $SbBr_3$ and $BiBr_3$ in the 3000−2500 cm^{-1} spectral range (−CH_2 region) are quite similar.

In the 850−500 cm^{-1} spectral range both in the ligand and in the compounds strong bands at 820, 811, 584, and 567 cm^{-1} typical of paracyclophane are present.[22]

In the far-IR spectra (500−60 cm^{-1}) of the ligand, the band at 458 cm^{-1} is assignable to the ring-out-of plane frequencies; the same band strongly decreases in intensity upon the formation of compounds with $SbBr_3$ and $BiBr_3$. The far-IR spectra of compound **1** shows medium bands at 241 and 100 cm^{-1} and a strong band at 224 cm^{-1} characteristic of the Sb-halogen stretching vibrations.

The strong bands at 199, 181, 166, and 93 cm^{-1} of compound **2** are in good agreement with terminal and bridging Bi(III)−halogen stretching vibrations.[23]

As shown in Figure 1 the structure of the complex **1** consists of dimeric Sb_2Br_6 edge-sharing units in which Br1 forms a bridge connecting two adjacent metal atoms: Sb···Sb(−*x*, −*y*, 1 − *z*) = 4.707(2) Å. The antimony atoms are closely bonded to three bromine atoms (Sb−Br1 = 2.538(2) Å, Sb−Br2 =

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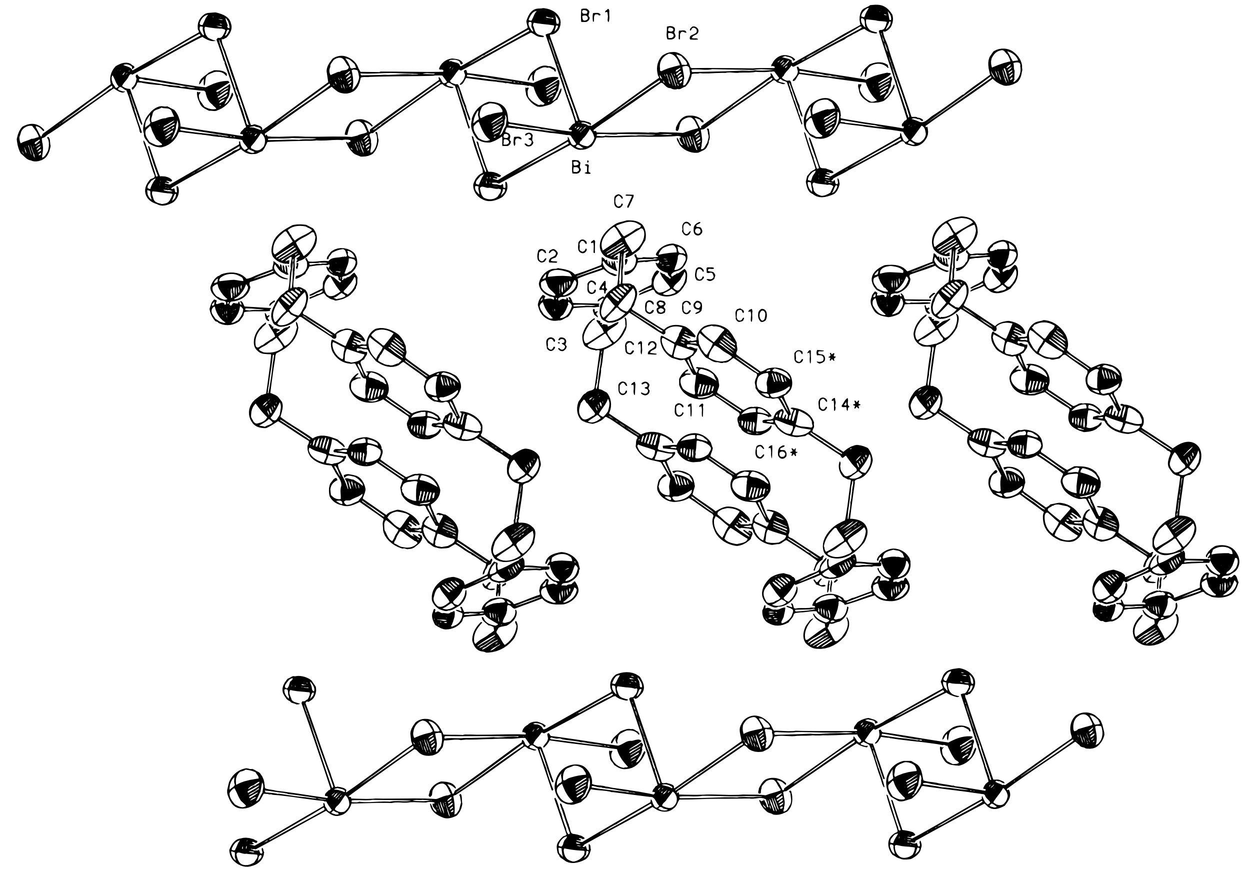

Figure 2. Perspective view of the structure of compound **2** with thermal ellipsoids at 50% probability level. Toluene molecules are omitted for clarity.

2.528(2) Å, and Sb−Br3 = 2.522(2) Å) and loosely to Br1(−*x*, −*y*, 1 − *z*) at 3.733(2) Å (Table 2). The equatorial plane of the distorted square pyramid of coordination is occupied by Br2, Br3, Br1 (−*x*, −*y*, 1 − *z*) atoms and the midpoint (CM) of the C25−C30 bond (Sb⋯CM = 3.28(1) Å) of a benzene ring of the [2.2.2.2]paracyclophane molecule (Sb⋯C contacts range from 3.34(1) to 3.72(1) Å). The Br1 atom lies in the axial direction of the pyramid while a weaker interaction with the C6 atom (Sb⋯C6 = 3.43(2) Å) of another ring of the organic molecule completes the coordination sphere of the metal leading to six-coordination at each Sb atom in the binuclear complex. Br1 and C6 fill the axial positions of a distorted octahedron.

The units cell of **2** contains one [2.2.2.2]paracyclophane centrosymmetrical molecule, two $BiBr_3$ units and a disordered toluene molecule as solvate (Figure 2). The $BiBr_3$ units are outside the paracyclophane ring and the environment of the metal could be described as a very distorted octahedron. The bismuth atom is surrounded by three bromines Br1, Br2, and Br3 with shorter distances (2.668(2), 2.659(2), 2.598(3) Å, respectively), two asymmetrically bridging bromines from different adjacent polyhedra (Bi⋯Bi(1 − *x*, 1 − *y*, −*z*)= 4.607(2); Bi⋯Bi(1 − *x*, 1 − *y*, 1 − *z*) = 4.512(2) Å) with longer distances (3.221(2), 3.419(2) Å) and by a phenyl ring (Bi⋯C, 3.27(2)−3.56(2) Å) (Table 3). The equatorial plane of the polyhedron including Br2, Br1(1 − *x*, 1 − *y*, −*z*), Br2(1 − *x*, 1 − *y*, 1 − *z*), and Br3 is parallel to the plane of the interacting phenyl ring (angle of 1.1(4)°). Br1 and the electrons donated by the π-system fill the axial positions of the distorted octahedron. The lines Bi−Br1 and Bi−CM (CM is the midpoint of the C1−C6 bond) form angles of 8.5 and 15.2°, respectively with the normal equatorial coordination plane. The metal atom is out of this plane by 0.246(1) Å toward the benzene ring.

Table 3. Selected Bond Distances (Å) and Angles (deg) with ESDs in Parentheses for **2**[a]

Bi−Br1	2.668(2)	Bi−C2	3.40(1)
Bi−Br1^{i}	3.221(2)	Bi−C3	3.49(1)
Bi−Br2	2.659(2)	Bi−C4	3.56(2)
Bi−Br2^{ii}	3.419(3)	Bi−C5	3.42(2)
Bi−Br3	2.598(3)	Bi−C6	3.27(2)
Bi−C1	3.31(2)		
Br2^{ii}−Bi−Br3	171.8(1)	Br1^{i}−Bi−Br2	161.6(1)
Br2−Bi−Br3	96.6(1)	Br1−Bi−Br3	90.3(1)
Br2−Bi−Br2^{ii}	85.0(1)	Br1−Bi−Br2^{ii}	81.6(1)
Br1^{i}−Bi−Br3	97.1(1)	Br1−Bi−Br2	90.3(1)
Br1^{i}−Bi−Br2^{ii}	79.8(1)	Br1−Bi−Br1^{i}	77.4(1)
C1−C7−C8−C9	75(2)	C4−C12−C13−C14	−81(2)

[a] i: 1 − *x*, 1 − *y*, −*z*. ii: 1 − *x*, 1 − *y*, 1 − *z*.

The lengthening of the bismuth−halogen bond (Bi−Br1) opposite to the π-system is explained by considering both the arene−metal π-donation and the bridging effect, the latter also present in the Bi−Br2 distance. The Bi−Br3 bond (2.559 Å), not bridging, is the shortest. In **1** instead the three shorter Sb−Br distances are similar, with the bridging slightly longer.

The average of the Bi−ring distances (Table 3) is shorter than that of the Sb−ring (Table 2) in **1**, indicating stronger bonding as previously found in $C_{24}H_{24}(SbCl_3)_2 \cdot {}^{1}/_{2}C_6H_6$ and in $C_{24}H_{24}(BiCl_3)_3 \cdot C_6H_6$.[8]

In **1** two benzene rings of the asymmetric paracyclophane molecule are coordinated to a single metal atom in a centrosym-

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Table 4. Comparison of Distances (Å) of Sb and Bi from the Interacting Rings Carbon Atoms in Related Structures

a		b		c		d		e	
Sb···C(1)	3.364(10)	Sb1···C11	3.27	Sb···C(1)	3.61(3)	Bi···C(7)	3.37(4)	Bi···C(1)	3.64(2)
Sb···C(2)	3.394(8)	Sb2···C1−C2 bond	2.94	Sb···C(2)	3.75(3)	Bi···C(8)	3.23(4)	Bi···C(2)	3.53(2)
Sb···C(3)	3.549(8)			Sb···C(3)	3.77(3)	Bi···C(9)	3.35(5)	Bi···C(3)	3.34(2)
Sb···C(4)	3.736(8)			Sb···C(4)	3.66(3)	Bi···C(10)	3.44(6)	Bi···C(4)	3.30(2)
Sb···C(5)	3.719(8)			Sb···C(5)	3.44(3)	Bi···C(11)	3.66(5)	Bi···C(5)	3.17(2)
Sb···C(6)	3.505(11)			Sb···C(6)	3.48(3)	Bi···C(16)	3.56(5)	Bi···C(6)	3.36(6)

f		g		h		i		j	
Bi···C(1)	3.62(3)	Bi···C	3.15(3)−3.47(2)	Bi···C(4)	3.68(5)	Sb···C(25)	3.34(1)	Bi···C(1)	3.31(2)
Bi···C(2)	3.37(5)	Bi−mean plane	3.028(1) Å	Bi···C(5)	3.62(3)	Sb···C(26)	3.47(1)	Bi···C(2)	3.40(1)
Bi···C(3)	3.25(4)			Bi···C(7)	3.25(7)	Sb···C(27)	3.67(1)	Bi···C(3)	3.49(1)
Bi···C(4)	3.39(5)			Bi···C(8)	3.22(6)	Sb···C(28)	3.72(1)	Bi···C(4)	3.56(2)
Bi···C(5)	3.56(5)			Bi···C(9)	3.10(5)	Sb···C(29)	3.56(2)	Bi···C(5)	3.42(2)
				Bi···C(10)	3.36(5)	Sb···C(30)	3.36(1)	Bi···C(6)	3.27(2)

[a] SbCl$_2$·naphthalene.[26] [b] 2SbCl$_3$·phenanthrene.[27] [c] 2SbBr$_3$·pyrene.[24] [d] Bi$_4$Br$_{12}$·2fluoranthene.[28] [e] Bi$_2$Br$_6$·[2.2]paracyclophane.[3a] [f] 2BiCl$_3$·pyrene.[29] [g] Bi$_2$Br$_6$·[2.2]paracyclophane:pyrene.[3b] [h] [Bi$_4$Br$_{12}$]·3perylene.[30] [i] Present paper **1**. [j] Present paper **2**.

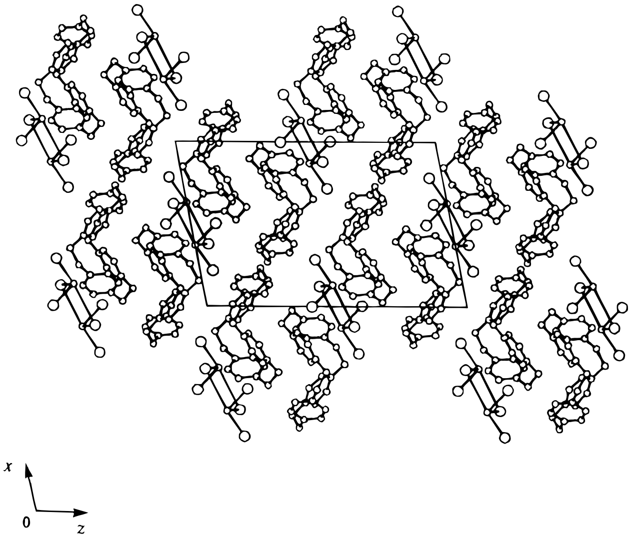

Figure 3. Packing arrangement of compound **1** on plane (010).

metric dimeric structure. In fact two SbBr$_3$ molecules, related by a center of symmetry, are situated between two organic molecules.

The dimer packing is determined only by van der Waals interactions (Figure 3).

In **2** the organic molecule is centrosymmetric and two rings are bonded to two bismuth atoms related by symmetry.[3,24] The Bi−Br bridges allow the formation of one-dimensional polymeric chains of edge-sharing polyhedra running along the *c* axis at $^1/_2b$. These chains are connected by the paracyclophane molecules forming layers packed with the solvated toluene rings (Figure 4).

In **1** the A and C rings are slightly distorted (maximum deviation 0.03(2) Å), while B and above all D (ring coordinated to Sb) are more planar (maximum deviation 0.016(16) Å). The dihedral angles between two adjacent rings are A,B = 42.9(5)°, B,C = 51.9(4)°, C,D = 47.1(4)°, and A,D = 56.2(5)°. This arrangement is similar to that found in the structure of [2.2.2.2]paracyclophane.[25]

In **2** the four aromatic rings are little distorted (maximum deviation 0.03(2) Å for C4). The cyclophane molecule is folded in such a way that the phenyl planes form an angle of 48.6(6)° with each other, and, for symmetry, the C1,C6 rings are on parallel planes, while the C9,C16 rings are coplanar (dihedral angle is 180(1)°).

Figure 1, Figure 2, and the torsion angle values (Tables 2 and 3) show that in **1**, between the four possible gauche conformations, the G$^+$G$^+$G$^+$G$^+$conformation is observed in the solid state, while in **2** a G$^+$G$^-$G$^-$G$^+$G$^-$ type is observed. Both conformation are probably energetically equivalent and may be governed by steric interactions within the crystal.[25]

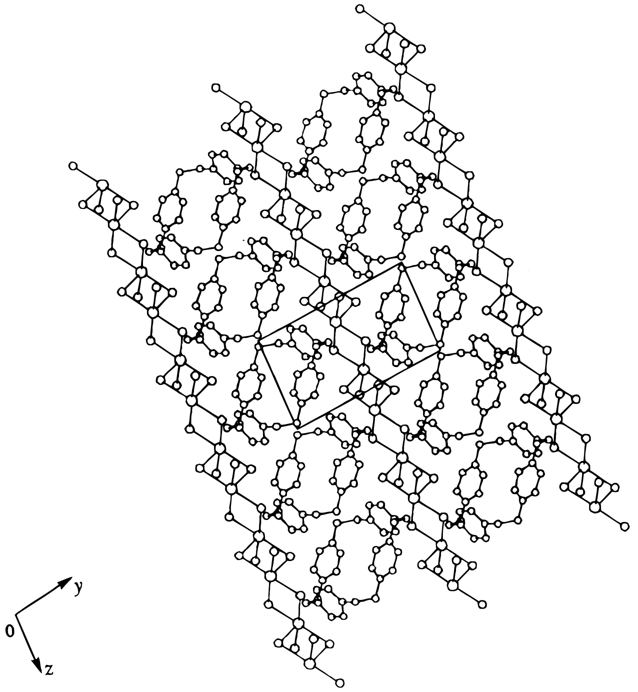

Figure 4. Packing arrangement of compound **2** projected on plane (100). Toluene molecules are omitted for clarity.

The bismuth−C distances in [2.2.2.2]paracyclophane complex are on average longer (Table 3) than those in [2.2]paracyclophane compounds[3] probably due to better π-basicity of the latter. This can be correlated with the more rigid structure of the [2.2]-paracyclophane.

In Table 4 the Sb and Bi distances from interacting ring carbon atoms in analogous structures are compared.

The compounds are more stable to air and moisture compared to similar complexes with usual arenes.

The CT compounds are characterized by weak interactions between the organic and inorganic components. Also in this

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case these weak interactions are present for both compounds but the weaker complexing properties of [2.2.2.2]paracyclophane with Sb^{III}bromide with respect to Bi^{III}bromide are shown by the longer interactions with the metal (3.34(1); 3.72(1) Å) (Table 2) in accordance with a better π-acceptor ability of Bi with respect to Sb.

In both compounds a comparison of the individual Bi···C and Sb···C distances shows that the π-bonding is closer to an η^2 mode than to an η^6 one, as previously found in $(perylene)_3[Bi_4Br_{12}]$.[30]

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Supporting Information Available: Tables of crystallographic data, fractional atomic coordinates, hydrogen atom coordinates, anisotropic thermal parameters, and remaining bond angles and bond distances (8 pages). Ordering information is given on any current masthead page.

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