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Synthesis, Spectroscopic (FT-IR, ^{1}H, ^{13}C, Mass Spectrometry), and Biological Investigation of Five-Coordinated Germanium-Substituted Tricyclohexyl Antimony Dipropionates: Crystal Structure of Tricyclohexylantimony Dibromide

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A series of five-coordinated germanium-substituted tricyclohexylantimony dipropionates have been synthesized and characterized by different instrumental techniques, such as elemental analyses, FT-IR, multinuclear NMR (^{1}H, ^{13}C) and mass spectrometry. These compounds have also been screened against different microbes and they showed good activity against different bacteria that was comparable to the reference drugs. The crystal structure of the precursor $(C_6H_{11})_3SbBr_2$ is reported here, which showed that the antimony atom in an asymmetric unit exists in trigonal bipyramidal geometry, having space group C2/c with the monoclinic crystal system.

Key Words: Organoantimony, organogermanium, spectroscopy, biological studies.

Introduction

A substantial literature exists on the synthesis, structure, and biological activities of R_nSbX_{5-n}(R = Alkyl, aryl; X = Carboxylate; n = 3,4) because of their wide range of biological and catalytic applications.[1–6] In recent years, some germanium-containing organic compounds have received considerable attention because of their potential clinical applications.[7] Although not markedly toxic, organogermanium compounds are of interest for their erythropoitic, bactericidal, and fungicidal properties.[8,9] However, studies of derivatives of germanium-substituted carboxylic acids with main group metalloids are relatively few. In order to explore the scope of biological activity, nature of bonding, and structures of compounds, we have synthesized a new series of tricyclohexyl antimony germanium-substituted dipropionates that contain 2 active centers,

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the tricyclohexyl antimony(V) moiety and the germanium-substituted carboxylate group. In addition, we present the crystal structure of the precursor $(C_6H_{11})_3SbBr_2$.

Experimental

Materials

Substituted propenoic acids and $SbCl_3$ were purchased from Aldrich (Germany), germanium dioxide (99.9% purity) was purchased from the People's Republic of China, and each was used as received. All chemicals were of analytical grade and used without further purification. The organic solvents were dried before use over sodium benzophenone by the standard method.[10]

Instrumentation

Elemental analyses were carried out at Midwest Micro-Lab, Indianapolis, USA. Melting points were determined with an MP-D Mitamura Riken Kogyo (Japan) and are uncorrected. FT-IR spectra were recorded on a Bio-Rad Excalibur FT-IR Model FTS 3000 MX using a KBr disc. The ^{1}H and ^{13}C NMR spectra were recorded on a Varian Mercury 300 spectrometer, with $CDCl_3$ as a solvent and TMS as a reference, operating at 300 and 75.5 MHz, respectively. The crystallographic data were collected at 173 K on a Nonius Kappa CCD diffractometer.

Synthesis of $(C_6H_{11})_3SbBr_2$

$(C_6H_{11})_3SbBr_2$was synthesized according to the literature by the method of oxidative addition reaction of $(C_6H_{11})_3Sb$.[11] $(C_6H_{11})_3Sb$ was prepared by dissolving 0.05 mol of freshly distilled $SbCl_3$ in dry diethyl ether. Cyclohexyl magnesium bromide (0.15 mol) was added dropwise at 273 K over 1 h, with regular stirring. The temperature was allowed to rise slowly and the mixture was subsequently refluxed for 1 h. The reaction mixture was cooled and hydrolyzed with cold distilled water. The organic layer was separated and dried over anhydrous $MgSO_4$; the solvent was evaporated under reduced pressure. The solid $(C_6H_{11})_3Sb$ was recrystallized in hot petroleum ether. The resulting $(C_6H_{11})_3Sb$ was converted to $(C_6H_{11})_3Sb\ Br_2$by direct bromination, and the solid product was recrystallized from a toluene-petroleum ether mixture (3:1).

Synthesis of germanium-substituted tricyclohexylantimony dipropionates

These compounds compounds were synthesized under mild conditions according to the literature.[12] To 3-triphenylgermyl (substituted) propionic acid (1 mol) and triethylamine (0.8 cm^3) in toluene (50 cm^3) was added $(C_6H_{11})_3SbBr_2$ (0.5 mol) as shown in Eq. 1. The reaction mixture was stirred at room temperature for 8 h and then filtered. The filtrate was evaporated under reduced pressure. The obtained solid was crystallized from a CH_2Cl_2- Pet-ether mixture (1:3).

$$2Ph_3GeCHRCH_2CO_2H + (C_6H_{11})_3SbBr_2 \xrightarrow[\substack{\text{toluene}\\ \text{r.t. 8h}}]{Et_3N} (Ph_3GeCHRCH_2CO_2)_2Sb(C_6H_{11})_3 + Et_3N.HCl \quad (1)$$

$$\begin{aligned} R^1 \quad = \quad & p-ClC_6H_4(1), CH_3(2), C_6H_5(3), n-C_3H_7(4), o-CH_3OC_6H_4(5), \\ & m-CH_3OC_6H_4(6), p-CH_3OC_6H_4(7), p-CH_3C_6H_4(8) \end{aligned}$$

Biological Studies

The biological activity of germanium-substituted tricyclohexylantimony dipropionates was determined against various bacteria, including *Escherichia coli, Bacillus subtilis, Shigella flexenari*, and *Salmonella typhi*, by the agar well diffusion method.[13,14] Imipenum was used as the standard antibiotic. The 24-h-old culture containing approximately 104-106 colony forming units (CFU) was spread on the surface of Muller-Hinton Agar (MHA) plates. Wells were created in the medium with the help of a sterile metallic borer. Test samples of different concentrations were added to their respective wells. Experimental plates were incubated at 310 K for 24 h, and zones of inhibition (%) were measured and compared with the standard antibiotic imipenum, with zone inhibition of 20 and 22 mm, respectively.

The cytotoxicity data were collected by the brine shrimp method using Etoposide as the standard cytotoxic drug.[15] Brine shrimp eggs (50 mg) were placed in a hatching tray half filled with brine solution and incubated for 2 days at 300 K. Test samples (20 mg) were dissolved in DMSO and diluted to 1000, 100, and 10 μg/mL in 500, 50, and 5 μL vials using a Pasteur pipette. In each vial, 30 larvae were placed and seawater added to make a volume of 5 mL. The contents were incubated at 298-300 K for 24 h under illumination. The numbers of survivors were counted and compared with those from the standard cytotoxic drug.

X-ray Crystallography

The crystals of the precursor $(C_6H_{11})_3SbBr_2$ suitable for X-ray diffraction were grown by dissolving 0.5 g of the sample in a mixture of toluene and pet-ether (3:1). Slow evaporation at room temperature yielded fine crystals. Diffraction measurements were carried out at 173 K on a Nonius Kappa CCD diffractometer for a colorless block of suitable size, 0.30 x 0.20 x 0.10 mm^3. Cell constants from the refinement of 2892 reflections in the range of $4.82 < \theta < 30.05^\circ$ corresponded to the monoclinic cell.

The data were corrected for Lorentz and polarization effects. Absorption correction using the multi-scan method[16]was applied with SHELXL-97[17].

Results and Discussion

The results of elemental analyses and other physical properties of the synthesized compounds are reported in Table 1. The compounds are quite stable and are soluble in common organic solvents, such as CH_2Cl_2, $CHCl_3$, C_6H_6, $CH_3C_6H_4$, and DMSO.

Infrared Spectroscopy

Infrared spectroscopy provides a method of assigning carboxylate coordination modes from the position of and separation ($\Delta\upsilon$) between antisymmetric and symmetric CO_2 stretching modes. The infrared spectrum of the synthesized compounds has been recorded in the range 4000-400 cm^{-1}. The stretching vibrations due to Sb-C(459-474 cm^{-1}), Sb-O(538-574 cm^{-1}), and Ge-C(642-669 cm^{-1}) can be assigned on the basis of earlier publications[18−20] and are listed in Table 2. The IR stretching vibration of the CO_2 group in organoantimony carboxylates are very important for determining their structures. When there are interactions between the carbonyl oxygen atoms of the carboxylate groups and the antimony atom, the asymmetric absorption vibration frequencies [$\upsilon_{asy}(CO_2)$] of the carboxylate groups decrease and the symmetric absorption vibration

frequencies $\upsilon_{sy}(CO_2)$ increase. In the IR spectra of the title compounds, the carboxylate bands are observed in the characteristic regions for $\upsilon_{asy}(CO_2)$ between 1636 and 1665 cm^{-1}, and for $\upsilon_{sym}(CO_2)$ between 1308 and 1335 cm^{-1}.

Table 1. Physical data of triorganoantimony(V) derivatives of the general formula: $[(C_6H_5)_3GeCHRCH_2COO]_2$ $Sb[C_6H_{11}]_3$.

Comp. No.	R^1	Molecular Formula	M.P.(K)	Yield %	Elemental Analysis Found (Calcd.)	
					C %	H %
1	*p*-ClC_6H_4	$C_{72}H_{77}O_4Ge_2SbCl_2$	495-496	76	65.16 (65.15)	4.45 (4.44)
2	CH_3	$C_{62}H_{75}O_4Ge_2Sb$	458-460	83	64,60 (64.63)	6.53 (6.51)
3	C_6H_5	$C_{72}H_{79}O_4Ge_2Sb$	503-504	85	67.78 (67.76)	6.20 (6.19)
4	n-C_3H_7	$C_{66}H_{83}O_4Ge_2Sb$	483-485	84	65.59 (65.61)	6.84 (6.87)
5	*o*-$CH_3OC_6H_4$	$C_{74}H_{83}O_6Ge_2Sb$	491-493	79	66.53 (66.51)	6.20 (6.21)
6	*m*-$CH_3OC_6H_4$	$C_{74}H_{83}O_6Ge_2Sb$	497-498	81	66.49 (66.51)	6.20 (6.21)
7	*p*-$CH_3OC_6H_4$	$C_{74}H_{83}O_6Ge_2Sb$	500-501	77	66.50 (66.51)	6.23 (6.21)
8	*p*-$CH_3C_6H_4$	$C_{74}H_{83}O_4Ge_2Sb$	488-489	76	68.17 (68.15)	6.35 (6.36)

The $\Delta\nu(CO_2)$ values (310-333 cm^{-1}) are higher than $\Delta\nu$(ionic) clearly point to the weaker interaction or no interaction between the carbonyl oxygen atom of the carboxylate groups and antimony atom. The high difference of $\Delta\nu$ may be attributed to the steric effect of cyclohexyl groups that decrease the capacity of antimony atom to accept lone electron pair from the carbonyl oxygen atom. Reduction of antimony Lewis acidity leads to basically unidentate carboxylate[21]. Further the absence of a strong band in the 3500-3300 cm^{-1} regions due to ν (OH), of all organoantimony(V) compounds indicating the deportation and coordination of the carboxylate group with antimony.

Table 2. Characteristic IR absorption frequencies in cm^{-1} of triorganoantimony(V) derivatives of the general formula: $[(C_6H_5)_3GeCHRCH_2COO]_2Sb[C_6H_{11}]_3$.

Comp.	$\nu(COO)_{asym}$	$\nu(COO)_{sym}$	$\Delta\nu$	ν(Ge-C)	ν(Sb-C)	ν(Sb-O)
1	1647	1315	332	642	463	560
2	1643	1314	329	653	467	563
3	1636	1318	318	669	467	571
4	1646	1311	335	657	466	558
5	1642	1314	328	675	469	542
6	1639	1314	325	664	468	554
7	1641	1308	333	655	467	538
8	1648	1317	331	658	467	574

^{1}H NMR Spectroscopy

The ^{1}H NMR data of the synthesized compounds are presented in Table 3. All the protons in the compounds have been identified by intensity and multiplicity patterns, and the total number of protons calculated from the integration curve is in close agreement with the expected molecular formulae. The ^{1}H NMR data of the investigated compounds showed the absence of an OH signal at 11.00 ppm for COOH of the germanium-substituted acids, which indicates the bonding of the antimony with the germanium moiety through the deprotonated carboxylic oxygen. The proton signals of the cyclohexyl group attached to the antimony show 2 sets of multiplets in the region 1.15-1.94 ppm.[9]

Another important observation in these compounds is the extent of diastereotopy that is due to the presence of the GeCH chiral center and the CH_2 prochiral center. The two-diastereotopic protons of CH_2 have become non-equivalent because of the chiral center, and would have geminal coupling as well as vicinal coupling with the GeCH proton and appears as 2 multiplets in the regions 3.15-3.74 ppm and 2.15-2.95 ppm[22].

^{13}C NMR Spectroscopy

^{13}C NMR spectral data of the triorganoantimony carboxylates containing germanium are given in Table 4. The number of signals found corresponds with the presence of a magnetically non-equivalent carbon atom, which was assigned by comparison with the experimental chemical shift with those calculated from the incremental method.[23] The involvement of the germanium-substituted carboxylic group in bonding to Sb is confirmed by the downfield shift of carboxylic carbon upon coordination, as compared with the germanium-substituted carboxylic acid. The group with a strong electron withdrawing effect, for example, the methoxy group attached to the phenyl ring (R^1), resonate at low field, while the ^{13}C signal of the cyclohexyl group attached to the antimony in compounds resonates in the region 23.85-31.65 ppm.

Mass Spectrometry

The main fragments of the synthesized compounds with their relative abundance are listed in Table 5. Mass spectral data are in agreement with the expected and proposed molecular formulae of all synthesized compounds. During fragmentation, decarboxylation/dealkylation from the metal atoms are the main

breakdown patterns for the synthesized compounds. The peak of highest intensity is found at 305 and 370 m/z in the spectral fragmentation of phenyl-substituted germanium, $[(C_6H_5)_3GeCHRCH_2COO]_2Sb[C_6H_{11}]_3$. $(C_6H_5)_3Ge^+$ and $(C_6H_{11})_3Sb^+$, respectively.[8]

Table 3. ^{1}H NMR data[(a–d)] of triorganoantimony(V) derivatives of the general formula: $[(C_6H_5)_3GeCHRCH_2COO]_2Sb[C_6H_{11}]_3$.

Comp.	CH	CH_2	R	$C_6H_{11}Sb$	C_6H_5Ge
1	3.33 (m, 2H)	2.45-2.59 (m, 4H)	7.38-7.42 (m,8H)	1.20-1.73 (m, 33H)	7.13-7.35 (m,30H)
2	3.15 (m, 2H)	2.46-2.65 (m, 4H)	0.85 [d,6H, ^{3}J(6.9)]	1.24-1.75 (m, 33H)	7.5-7.65 (m, 30H)
3	3.48-3.61 (m, 2H)	2.45-2.91 (m, 4H)	6.95-7.31 (m, 10H)	1.21-1.85 (m, 33H)	6.95-7.31 (m, 30H)
4	2.91-3.24 (m, 2H)	2.20-2.65 (m, 4H)	1.15 [t, 6H, ^{3}J(6.8)] 1.60-1.95 (m, 8H)	1.42-1.95 (m, 33H)	7.11-7.46 (m, 30H)
5	3.15-3.25 (m, 2H)	2.5-2.89 (m, 4H)	6.48-6.92 (m, 8H) 3.27 (s, 6H)	1.15-1.94 (m, 33H)	7.05-7.29 (m, 30H)
6	3.21-3.45 (m, 2H)	2.15-2.31 (m, 4H)	6.61-6.84 (m, 8H) 3.36 (s, 6H)	1.15-1.67 (m, 33H)	6.95-7.18 (m, 30H)
7	3.55-3.74 (m, 2H)	2.51-2.95 (m, 4H)	6.50-6.81 (m, 8H) 3.65 (s, 6H)	1.11-1.85 (m, 33H)	7.15-7.34 (m, 30H)
8	3.39-3.42 (m, 2H)	2.81-2.94 (m, 4H)	6.81-6.97 (m, 8H) 2.25 (s, 6H)	1.17-1.85 (m, 33H)	7.14-7.25 (m, 30H)

[a]In $CDCl_3$ at 295 K.
[b]Chemical shifts in ppm. $^{n}J(^{1}H–^{1}H)$ in Hz.
[c]Multiplicity is given as; s = singlet, d = doublet, t = triplet, m = multiplet
[d]R = *p*-ClC_6H_4 (1), CH_3 (2), C_6H_5 (3), n-C_3H_7 (4), *o*-$CH_3OC_6H_4$ (5), *m*-$CH_3OC_6H_4$ (6), *p*-$CH_3OC_6H_4$ (7), *p*-$CH_3C_6H_4$(8)

$$\begin{array}{cccccccc} (C_6H_5)_3Ge^+ & \longrightarrow & (C_6H_5)_2Ge^+ & \longrightarrow & (C_6H_5) & \longrightarrow & Ge^+GeH^+ \\ 305(m/z) & & 228(m/z) & & 151(m/z) & & 75(m/z) \\ (C_6H_{11})_3Sb^+ & \longrightarrow & (C_6H_{11})_2Sb^+ & \longrightarrow & (C_6H_{11})Sb^+ & \longrightarrow & Sb^+ \\ 370(m/z) & & 287(m/z) & & 204(m/z) & & 121(m/z) \end{array}$$

Table 4. ^{13}C NMR data$^{(a-c)}$ of triorganoantimony(V) derivatives of the general formula.

Comp	CH	CH_2	R	$C_6H_{11}Sb$	C_6H_5Ge	CO
1	32.85	37.5	137.41,135.23 129.51,130.40	136.32,135.50 129.85,134.01	141.5, 138.1, 131.61,134.45	178.4
2	33.48	38.5	13.83	29.11, 27.76, 25.95, 23.85	134.00,133.91 28.97, 131.11	177.5
3	30.75	38.4	135.54,135.36, 127.90, 128.22	29.81, 28.15, 25.99, 25.87	135.57,135.47 128.01,129.12	176.9
4	33.12	39.2	23.85, 22.45, 14.62	30.51, 29.67, 27.84, 25.80	136.95,135.87 129.24,132.52	178.4
5	29.81	37.4	136.16, 135.33, 155.90, 110.00, s = 54.45	29.81, 28.19, 26.26, 25.92	137.81,135.37 127.82,130.58	177.1
6	32.51	38.7	138.11, 135.25, 157.81, 113.52, s = 56.31	30.65, 29.74, 27.83, 26.95	138.42,136.73 129.24,132.46	178.8
7	32.30	38.6	135.47, 133.59, 157.21,113.38, s = 55.09	30.73, 29.81, 28.12, 26.00	135.58,135.33 128.15,129.24	178.5
8	31.79	38.5	135.95, 133.42, 129.35, 134.27, s = 22.41	31.65, 29.41, 27.52, 26.47	136.51,135.42 129.77,131.28	177.8

[a]In $CDCl_3$ at 297 K.
[b]Chemical shifts in ppm
s = substituent on 'R' phenyl ring
[c]R = *p*-ClC_6H_4 (1), CH_3 (2), C_6H_5 (3), n-C_3H_7 (4), *o*-$CH_3OC_6H_4$ (5), *m*-$CH_3OC_6H_4$ (6), *p*-$CH_3OC_6H_4$ (7), *p*-$CH_3C_6H_4$(8)

Table 5. Mass fragments observed for triorganoantimony derivatives.

Fragment Ion	**(1)** m/z(%)	**(2)** m/z(%)	**(3)** m/z(%)	**(4)** m/z(%)	**(5)** m/z(%)	**(6)** m/z(%)	**(7)** m/z(%)	**(8)** m/z(%)
$((C_6H_5)_3GeCHRCH_2CO_2)_2Sb(C_6H_{11})_3$	1343 (n.o)	1152 (n.o)	1276 (n.o)	1208 (n.o)	1336 (n.o)	1336 (n.o)	1336 (n.o)	1304 (n.o)
$((C_6H_5)_3GeCHRCH_2CO_2)Sb(C_6H_{11})_3$	856 (35.6)	761 (11.5)	824 (36.3)	789 (25.6)	853 (45.7)	853 (32.2)	853 (11.3)	837 (15.9)
$((C_6H_5)_3GeCHRCH_2CO_2)Sb(C_6H_{11})_2$	773 (28.1)	595 (45.7)	741 (29.6)	706 (18.5)	770 (38.3)	770 (21.4)	770 (18.6)	754 (10.5)
$((C_6H_5)_3GeCHRCH_2CO_2)$	487 (20.4)	391 (35.4)	452 (40.4)	419 (55.7)	483 (22.8)	483 (31.2)	483 (33.1)	467 (12.1)
$(C_6H_{11})_3Sb^+$	370 (31.2)	370 (21.5)	370 (100)	370 (52.5)	370 (22.6)	370 (100)	370 (65.2)	370 (25.8)
$(C_6H_{11})_2Sb^+$	286 (15.2)	286 (18.4)	286 (23.5)	286 (15.8)	286 (18.4)	286 (22.2)	286 (24.7)	286 (19.3)
$(C_6H_{11})Sb^+$	204 (27.2)	204 (35.5)	204 (36.5)	204 (42.1)	-	-	204 (35.8)	204 (12.5)
Sb^+	121 (10.9)	-	-	-	121 (8.5)	-	121 (11.4)	-
$(C_6H_5)_3Ge^+$	305 (100)	305 (100)	305 (85.2)	305 (100)	305 (100)	305 (65.0)	305 (100)	305 (100)
$(C_6H_5)_2Ge^+$	228 (35.4)	228 (38.2)	228 (45.6)	228 (33.8)	228 (25.3)	228 (35.4)	228 (23.9)	228 (38.6)
$[(C_6H_5)_2Ge\text{-}H]^+$	227 (20.1)	-	227 (42.6)	-	-	-	227 (48.3)	227 (41.5)
$(C_6H_5)Ge^+$	151 (33.0)	151 (43.5)	151 (54.1)	151 (29.4)	151 (43.6)	151 (37.8)	151 (41.6)	151 (29.2)
GeH^+	75 (4.1)	75 (7.3)	75 (2.5)	75 (4.6)	75 (7.2)	75 (5.6)	75 (4.4)	75 (5.3)

Crystal structure of $(C_6H_{11})_3SbBr_2$

The ORTEP diagram of the precursor $(C_6H_{11})_3SbBr_2$is shown in Figure 1 with its atomic labeling scheme, while the details of the crystal data and structure refinement are listed in Table 6. The selected bond lengths and bond angles are listed in Table 7. The molecular structure of tricyclohexyl antimony(V) dibromide is a distorted trigonal bipyramidal with bromine atoms occupying the axial sites. The SbC_3 unit is planar, but the C1-Sb1-C1^i [125.14 (10)°] and C1-Sb1-C7 [125.33 (10)°] angles are somewhat enlarged at the expense of the C1-Sb1-C7 [109.35 (10)°]. The C-Sb-Br angles lie close to 90° for the ordered rings, C1-Sb1-Br1 [89.92 (5)°] and C1^i−Sb1-Br1 [87.68 (5)°], while those involving the disordered ring based on C7 show a significant deviation from a regular trigonal bipyramidal arrangement.

The disorderedness of the ring is based on C7 and C10 because both are out of plane with -44.37 and -21.68 ϕ, respectively. Furthermore, the asymmetric unit contains one half of a molecule with a central antimony atom located on a 2-fold rotation axis. While this axis generates a second C_6H_{11} ring (based on C1^i and the second bromine Br1^i), the fact that it does not contain the C7 and C10 of the remaining cyclohexyl ring means that the latter is disordered over 2 sites, each of a fragment exhibiting half occupancy. To accommodate the chair conformation of the ring, C8 and C9 are each further disordered over 2 sites of equal occupancies, C8/C8^i and C9/C9^i. For clarity, one complete ring containing C7, C8, C9, C10^i, C9^i, and C8^i is shown in the Figure. The second component of this disordered ring contains C7^i, C8A, C9A, C10, C8A^i, and C9A, though it is not shown in the Figure.

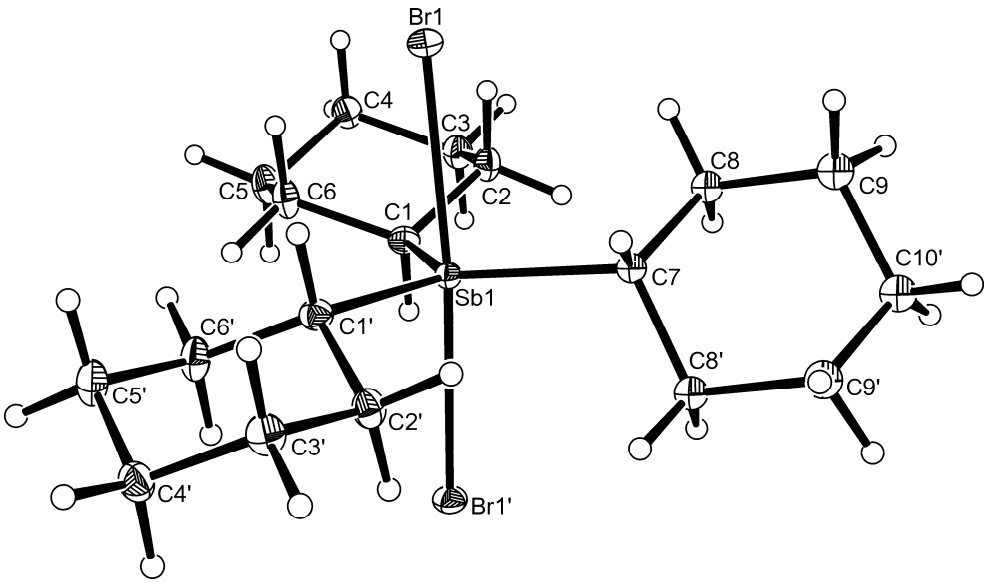

Figure. ORTEP diagram of precursor $(C_6H_{11})_3SbBr_2$.

Biological Studies

The selected number of synthesized organoantimony(V) dipropionates containing germanium was tested for their microbial toxicity against various sets of bacterial populations. The compounds were also evaluated for their toxicity using the brine shrimp method.

Table 6. Crystal data and structure refinement for $(C_6H_{11})_3SbBr_2$.

Empirical formula	$C_{18}H_{33}Br_2Sb$
Formula weight	531.01
Crystal system	Monoclinic
Space group	C2/c
a(Å)	13.0460(2)
b(Å)	19.7930(3)
c(Å)	8.6820(1)
$\beta(^o)$	117.211(1)
Volume	1993.75(5)Å^3
Z	4
Absorption coefficient	5.386 mm^{-1}
$\theta_{max}(^o)$	32.02
Reflections collected	22951
Independent reflections	34632 [R(int) = 0.0486]
Reflected observed (>2σ)	3176
Max. and min. transmission	0.37 and 0.27
Goodness-of-fit on F^2	1.100
Final R indices [I>2sigma(I)]	R1 = 0.0298, wR2 = 0.0755
R indices (all data)	R1 = 0.0335, wR2 = 0.0779
δ_{max}(e.Å^{-3})	1.434

Table 7. Selected bond lengths [Å] and angles [^{o}] for $(C_6H_{11})_3SbBr_2$.

Bond Lengths			
Sb1-C1^i	2.172 (18)	Sb1-C1	2.172(18)
Sb1)-C7	2.180(3)	Sb1-C7^i	2.180(3)
Sb1-Br1^i	2.670(2)	Sb1-Br1	2.6701(2)
C1-C6	1.526(3)	C1-C2	1.529(3)
Bond Angles			
C1^i-Sb1-C1	125.14(10)	C1^i-Sb1-C7	125.33(10)
C(1)-Sb(1)-C(7)	109.35(10)	C1^i-Sb1-C7^i	109.35(10)
C1-Sb1-C7^i	125.33(10)	C7-Sb1-C7^i	18.28(16)
C1^i-Sb1-Br1^i	89.92(5)	C1-Sb1-Br1^i	87.68(5)
C7-Sb1-Br1^i	88.01(9)	C7^i-Sb1-Br1^i	97.15(10)
C1^i-Sb1-Br1	87.68(5)	C1-Sb1-Br1	89.92(5)
C7-Sb1-Br1	97.15(10)	C7^{i}Sb1-Br1	88.01(10)
Br1^i -Sb1-Br1	174.796(10)	C6-C1-C2	111.98(16)
C6-C1-Sb1	112.47(12)	C2-C1-Sb1	111.28(12)
C1-C2-C3	109.81(16)	C4-C3-C2	111.05(17)

Symmetry transformations used to generate equivalent atoms: (i)1-xy-z+3/2

Antibacterial Activity

The selected number of synthesized compounds containing germanium and antimony were evaluated for their antibacterial activity by the agar well diffusion method[13] against various bacteria, including *Escherichia coli, Bacillus subtilis, Shigella flexenari*, and *Salmonella typhi*. The zone of inhibition was measured in millimeters. The results obtained were compared with the reference drug (Imipenum) and are listed in Table 8. The preliminary tests indicated that these compounds exhibited, to a certain extent, antibacterial

activity. Compound (1) shows significant activity against all the tested bacteria, as compared to standard drug. The greater activity of this compound was probably due to the presence of chlorine in the ligand acid, which itself is antibacterial.[24] The germanium-substituted acids, themselves, were found to be active against different bacteria and their organoantimony carboxylate showed more antibacterial activity.

Table 8. Antibacterial activity data[(a−c)] of organoantimony(V) derivatives containing germanium (in vitro).

Name of Bacteria	Zone of Inhibition of Sample (mm)								Zone of Inhibition of Std. Drug (mm)
	1	**2**	**3**	**4**	**5**	**6**	**7**	**8**	
Escherichia coli	32	25	22	19	26	28	25	25	33
Bacillus subtilis	29	26	22	28	27	22	28	23	30
Shigella flexenari	10	5	-	-	13	11	24	-	35
Staphylococcus aureus	42	35	32	41	28	33	35	39	43
Pseudomonas aeruginosa	-	-	-	-	-	-	-	-	25
Salmonella typhi	39	34	31	37	35	38	36	37	40

[a]Concentration of sample = 5 mg/mL of DMSO
[b]Concentration of standard drug (Imipenum) = 10 μg/mL
[c](-) = No activity

Cytotoxicity

The brine shrimp lethality bioassays of the compounds are presented in Table 9. Bioactive compounds are often toxic to shrimp larvae. So the cytotoxicity of synthesized compounds was determined by the brine shrimp method[18], using Etoposide as the standard cytotoxic drug. These results show positive lethality with LD_{50}values of 1.15-26.91 μg/mL. The highest toxicity was shown by compound (5), whose LD_{50} was 26.91 μg/mL, while the lowest toxicity was shown by compound (4).

Table 9. Cytotoxicity data[(a−d)] against brine shrimp.

Comp. No.	LD_{50} (μg/mL)
1	8.92
2	7.64
3	4.66
4	1.15
5	26.91
6	1.24

[a]Organism = Brine Shrimp (in vitro)
[b]Std. drug = Etoposide
[c]Conc. of std. drug = λ_{50}(μg/mL) = 7.46
[d]LD_{50} = Lethal dose at which 50% organisms die

Conclusions

Various physicochemical studies confirmed the formation and purity of the synthesized compounds. The geometry of tricyclohexylantimony dibromide is trigonal bipyramidal in solid state, which was confirmed by

X-ray crystallography, and this organoantimony retained the geometry after complexation with germanium-substituted carboxylic acids. Infrared spectroscopy proved the monodentate nature of the germanium-containing carboxylic unit in solid state and suggests the geometry of compounds as trigonal bipyramidal around antimony(V). Mass spectral data are in agreement with expected and proposed molecular formulae of all the synthesized compounds. Biological screening results revealed that some compounds show good activity against different bacteria.

Supplementary data

A complete list of crystallographic data and parameters, including atomic coordinates, has been deposited at the Cambridge Crystallographic Data Center as CCDC Number: 232851. Copies of the data can be obtained upon request to CCDC, 12 Union Road, Cambridge CB2 1EZ, UK. E-mail: deposited@ccdc.cam.ac.uk or http//www.ccdc.cam.ac.uk.

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