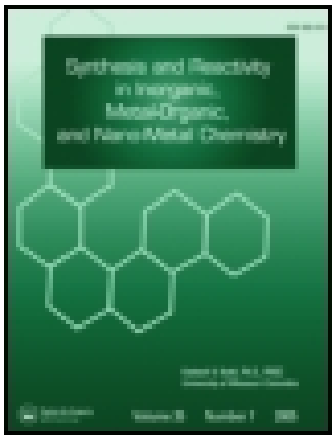

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Synthesis, Spectral (FT-IR, ^{1}H, ^{13}C, ^{119}Sn) and Biological Studies of Bimetallic Trimethyltin(IV) Germapropionates: X-Ray Structure of $(CH_3)_3Sn(C_{22}H_{21}O_2Ge)$

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Synthesis, Spectral (FT-IR, ^{1}H, ^{13}C, ^{119}Sn) and Biological Studies of Bimetallic Trimethyltin(IV) Germapropionates: X-Ray Structure of $(CH_3)_3Sn(C_{22}H_{21}O_2Ge)$

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New compounds of bimetallic trimethyltin(IV) germapropionates have been synthesized and characterized by elemental analyses, FT-IR, and multinuclear nmr (^{1}H,^{13}C,^{119}Sn) spectroscopies. These compounds show four-coordinated structure in solution and penta-coordinated structure in solid state. The crystal structure of representative compound, $(CH_3)_3Sn(C_{22}H_{21}O_2Ge)$, shows one dimensional polymeric penta-coordinated structure in its solid state. The synthesized compounds were subjected for a bioassay screening and show promising antibacterial and antifungal activities.

Keywords organotin, carboxylates, germanium, X-ray studies, biological studies

INTRODUCTION

In the past two decades, organotin chemistry has received much attention because of its extensive applications in different fields of life especially industrial, agricultural and biocidal applications.[1–5] The use of inorganic tin, considered the third most pollutant element in the ecosystem, has increased general concern that it may eventually enter the human food chain, accumulate in the environment and finally in the biological system.[6] The biological importance of organotins has been supported by studies concentrating on structure-activity correlations[7,8] and it has been observed that several diorganotin and triorganotin species show potential as anti-neoplastic agents.[9] Triorganotin compounds demonstrate an interesting range of structural variations leading to an indefinite structure correlation with biocidal activity.[10] Organogermanium is another kind of compound that has a wide range of biocidal applications.[11]

It has been reported that triorganotin carboxylates containing the biologically active germyl group in the carboxylate ligand, exhibit good acaricidal activity. Biological testing of germanium-tin compounds has demonstrated that these compounds can be used against bacterial, viral and antifungal ailments found in both humans and in animals. Some of these compounds were found to be more active for certain bacteria than the reference drug.[12] To link biological properties of organotin and organogermanium compounds, we have synthesized some new triorganotin carboxylates containing triorganogermyl group as a part of the carboxylate ligand and report their antibacterial and antifungal activities. In parallel, we also studied the nature of bonding and structure of these compounds in solution as well as in solid state. The general synthetic procedure, structural characterization using variety of spectroscopic techniques (^{1}H,^{13}C,^{119}Sn) and crystal structure of representative compound (**1**) are reported here.

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EXPERIMENTAL

Chemicals

Trimethyltin chloride, substituted cinnamic acids were purchased from Aldrich Chemicals (USA) and Germanium dioxide (99.9% purity) was purchased from the People's Republic of China and was used without further purification. All organic solvents were purchased from E-Merk, Germany and were dried over sodium benzophenone before use according to the standard method.[12]

Instrumentation

Elemental analyses were carried out at Midwest MicroLab Indianapolis, USA. Melting points were determined with Mitamura Riken Kogyo (Japan). IR was recorded on Bio-Rad Excalibure FT-IR Model FTS 3000 MX using KBr disc. The ^{1}H and ^{13}C NMR spectra were recorded on Varian Mercury 300 spectrometer using deuterated solvents and TMS as a reference operating at 300 and 75.5 MHz, respectively. ^{119}Sn NMR spectra were obtained on Bruker 250 ARX spectrometer (Germany) with TMS as a reference. The crystallographic data were collected at 173K on Nonius Kappa CCD diffractometer and the crystals suitable for X-ray diffraction were isolated from solution of chloroform and acetone mixture (3:1) over a prolonged period at room temperature.

Synthesis

Synthesis of Triarylgermyl Substituted Propionic Acids

The 3-(triarylgermyl) substituted propionic acids were synthesized according to the reported procedure.[12] Phenyl/*p*-tolyl magnesium bromide was prepared by reaction between magnesium (0.176 mole) and bromobenzene/*p*-bromotoluene (0.16 mol) in THF (30 cm^3) under nitrogen. The resulting Phenyl/*p*-tolyl magnesium bromide was added drop wise at 0°C to 3-(trichlorogermyl) substituted propionic acid (0.04 mole) in THF. After the addition, the mixture was refluxed for 4 hours. Reaction mixture was cooled and 10% aqueous hydrochloric acid was added. The water phase was extracted three times with chloroform. The chloroform phase was dried over anhydrous magnesium sulfate and filtered. The solvent was removed under vacuum to give white solid. The solid was then recrystallized from chloroform and acetone (3:1) to obtain a colorless crystalline solid.

Synthesis of Title Compounds

Title compounds were synthesized according to the literature method.[13] Equimolar of triarylgermyl substituted propionic acid and trimethyltin(IV) chloride was refluxed for 8–10 hours in toluene (50 cm^3) in the presence of triethylamine. Reaction mixture was cooled down, filtered and the solvent was removed under reduced pressure. Recrystallization of resulting solid was taking placed from a mixture of chloroform and acetone (3:1) yielded fine crystals.

Biological Studies

The synthesized compounds (1–10) were evaluated for their antibacterial activity by agar well diffusion method[13] against six different types of bacteria. The bacteria cultures used were *Escherichia coli, Bacillus subtilis, Shigella flexenari* and *Salmonella typhi*. Imipenum was used as standard antibiotic. The 24-hours-old culture containing approximately 10^4–10^6 colony forming unit

TABLE 1
Physical data of triorganotin derivatives of general formula: $((R^1)_3\ Ge\ CH\ (R^2)\ CH_2COO)\ Sn\ (CH_3)_3$

Comp.	R^1	R^2	Molecular formula	M. Wt.	M.P.°C	Yield%	Elemental analysis found (Calcd.) C%	H%
1	C_6H_5	CH_3	$C_{25}H_{30}O_2GeSn$	554	178–180	59	54.05 (54.15)	5.40 (5.41)
2	C_6H_5	C_6H_5	$C_{30}H_{32}O_2GeSn$	616	134–136	63	58.50 (58.44)	5.16 (5.19)
3	C_6H_5	*o*-$CH_3OC_6H_4$	$C_{31}H_{34}O_3GeSn$	646	165–168	64	57.61 (57.58)	5.21 (5.26)
4	C_6H_5	*m*-$CH_3OC_6H_4$	$C_{31}H_{34}O_3GeSn$	646	140–143	62	57.52 (57.58)	5.23 (5.26)
5	C_6H_5	*p*-$CH_3C_6H_4$	$C_{31}H_{34}O_2GeSn$	630	165	76	58.97 (59.04)	5.34 (5.39)
6	C_6H_5	*p*-ClC_6H_4	$C_{30}H_{31}O_2GeSnCl$	651	182–184	85	55.30 (55.29)	4.77 (4.76)
7	*p*-$CH_3C_6H_4$	*o*-$CH_3OC_6H_4$	$C_{34}H_{40}O_3GeSn$	688	133–135	81	59.29 (59.30)	5.79 (5.81)
8	*p*-$CH_3C_6H_4$	*m*-$CH_3OC_6H_4$	$C_{34}H_{40}O_3GeSn$	688	142–143	84	59.31 (59.30)	5.82 (5.81)
9	*p*-$CH_3C_6H_4$	*p*-$CH_3OC_6H_4$	$C_{34}H_{40}O_3GeSn$	688	161–162	79	59.28 (59.30)	5.79 (5.81)
10	*p*-$CH_3C_6H_4$	C_6H_5	$C_{33}H_{38}O_2GeSn$	658	178–180	83	60.10 (60.18)	5.76 (5.77)
11	*p*-$CH_3C_6H_4$	*p*-$CH_3C_6H_4$	$C_{34}H_{40}O_2GeSn$	672	181–182	76	60.74 (60.71)	5.93 (5.95)

TABLE 2
Characteristics IR absorption frequencies in cm^{-1} of triorganotin(IV) derivatives of general formula: $((R^1)_3$ Ge CH (R^2) $CH_2COO)$ Sn $(CH_3)_3$

Comp.	$\nu(COO)_{asym}$	$\nu(COO)_{sym}$	$\Delta\nu$	ν(Ge-C)	ν(Sn-C)	ν(Sn-O)
1	1680	1440	240	650	545	462
2	1678	1443	235	656	549	465
3	1685	1452	233	665	562	460
4	1682	1458	224	658	549	465
5	1679	1457	222	655	551	468
6	1690	1453	237	648	552	462
7	1688	1459	229	658	562	469
8	1683	1457	226	664	552	468
9	1697	1466	231	667	554	466
10	1690	1452	238	674	561	465
11	1682	1454	228	664	552	462

(CFU) was spread on the surface of Muller Hinton Agar (MHA) plates. Wells were created in medium with the help of a sterile metallic borer. Test samples of different concentrations were added in their respective wells. Experimental plates were incubated at 37°C for 24 hours and zones of inhibition (%) were measured and compared with standard antibiotic imipenum with zone inhibition of 20 and 22 mm, respectively.

The agar tube dilution protocol[13] was applied to study the activity of the compounds against various fungal strains such as *Trichophyton longifusus, Candida albicans, Aspergillus flavus, Microsporum canis, Fusarium solani* and *Candida glaberata.* The standard antifungal drugs used were Amphotericin B and Micronazole for comparison test. The tubes containing sterile sabour and dextrose agar were incubated with the test compound at different concentrations and solidified at room temperature. Test fungal cultures were inoculated on the slant, and growth inhibition (%) was observed after an incubation period of 7 days.

The shrimp larvae were applied as a tool to monitor the cytotoxicity of samples using *Etoposide* as standard cytotoxic drug.[13] Brine Shrimp eggs (50 mg) were placed in a hatching tray half-filled with brine solution and incubated for 2 days at 27°C. Test samples (20 mg) was dissolved in DMSO and diluted to 1000 μg/ mL, 100 μg/ mL, 10 μg/ mL in 500 μL, 50 μL and 5 μL vials using Pasteur pipettes. Then 30 larvae were placed in each vial and 5 mL volume was made with seawater. The contents were incubated at 25–27°C for 24 hours under illumination. The number of survivors were counted and compared with standard cytotoxic drug.

X-Ray Crystallography

Intensity data for a colorless crystal ($0.20 \times 0.16 \times 0.10$ mm^3) were measured at 295(2)K on Nonius Kappa CCD diffractometer with (graphite monochromater, $\lambda = 0.71073$Å). The data were corrected for Lorentz and polarization effects for absorption, using the multi-scan method. The details of crystal data and structure refinement have been included here 5. The structure was solved by direct method and refined by full-matrix least squares procedure using SHELXL-97.[13] The hydrogen atoms were included at geometrically idealized positions.

RESULTS AND DISCUSSION

Triorganotin (IV) derivatives containing germanium have been synthesized by the reaction of germanium-substituted carboxylic acid with trimethyltin chloride in (1:1) molar ratio in dry toluene (50 cm^3) in the presence of triethylamine. However, a prolonged refluxion (8–10 hours) is required for good yield.[13] The general reaction

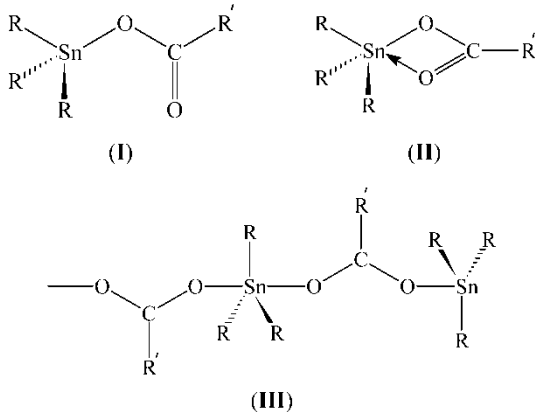

FIG. 1. Different structure of triorganotin(IV) compounds.

TABLE 3
^{1}H NMR data[a–e] for triorganotin derivatives of general formula: $((R^1)_3Ge\ CH\ (R^2)\ CH_2COO)\ Sn\ (CH_3)_3$

Comp.	CH	CH_2	R^1	R^2	Sn-CH_3	C-Sn-C angle (°)
1	3.15–3.61 (m,1H)	2.41–2.58 (m,2H)	7.3–7.61 (m,15H)	1.35 (d,6.98)	0.58 ^{2}J[58]	111.0
2	3.42–3.51 (m,1H)	2.54–2.61 (m,2H)	6.65–7.21 (m,15H)	6.65–7.21 (m,5H)	0.15 ^{2}J[59]	112
3	3.71–3.8 (m,1H)	2.58–2.61 (m,2H)	6.25–7.1 (m,15H)	6.25–7.1 (m,4H) 2.95 (s,3H)	0.10 ^{2}J[58.5]	111
4	3.41–3.62 (m,1H)	2.65–2.81 (m,2H)	6.95–7.21 (m,15H)	6.45–6.65 (m,4H) 3.51 (s,3H)	0.15 ^{2}J[58]	111
5	3.79–3.81 (m,1H)	2.53–2.64 (m,2H)	6.91–7.65 (m,15H)	6.91–7.65 (m,4H) 2.41 (s,3H)	0.59 ^{2}J[58.7]	111
6	3.61–3.63 (m,1H)	2.51–2.60 (m,2H)	6.27–7.14 (m,15H)	6.56–6.65 (m,4H)	0.81 ^{2}J[58.6]	111
7	3.31–3.41 (m,1H)	2.61–2.67 (m,2H)	6.80–7.51 (m,12H) 2.10 (s,9H)	6.38–6.46 (m,4H) 3.24 (s,3H)	0.16 ^{2}J[58.2]	111
8	3.38–3.40 (m,1H)	2.65–2.75 (m,1H)	6.82–7.14 (m,12H) 2.15 (s,9H)	6.3–6.51 (m,4H) 3.21 (s,3H)	0.15 ^{2}J[59]	112
9	3.41–3.45 (m,1H)	2.61–2.67 (m,2H)	6.86–7.11 (m,12H) 2.05 (s,9H)	6.31–6.67 (m,4H) 3.51 (s,3H)	0.15 ^{2}J[59.5]	112
10	3.52–3.77 (m,1H)]	2.46–2.71 (m,2H)	6.95–7.41 (m,12H) 2.45 (s,9H)	6.95–7.41 (m,5H)	0.15 ^{2}J[58.1]	111
11	3.45–3.57 (m,1H)	2.61–2.28 (m,2H)	6.61–7.15 (m,12H) 2.15 (s,9H)	6.61–7.15 (m,4H) 2.01 (s,3H)	0.14 ^{2}J[59.8]	112

[a] In $CDCl_3$ at 295 K.
[b] Chemical shift in ppm $^{2}J[^{119}Sn - {}^{1}H]$ in Hz.
[c] Multiplicity is given as s = singlet, t = triplet, m = multiplet.
[d] $R^1 = C_6H_5$ for compounds (1-6), p-$CH_3C_6H_4$ for (7–11).
[e] $R^2 = CH_3$ (1), C_6H_5 (2,10), o-$CH_3OC_6H_4$(3,7), m-$CH_3OC_6H_4$ (4,8), p-$CH_3C_6H_4$ (5,11), p-ClC_6H_4 (6), p-$CH_3OC_6H_4$ (9).

scheme is shown as the following.

$$(R^1)_3GeCH(R^2)CH_2CO_2H + (CH_3)_3SnCl \xrightarrow[\text{Toluene}]{Et_3N} (R^1)_3 GeCH(R^2)CH_2CO_2Sn(CH_3)_3 + Et_3N.HCl$$

$$R^1 = C_6H_5(1-6), p-CH_3C_6H_4(7-11)$$

$$R^2 = CH_3(1), C_6H_5(2,10), o-CH_3OC_6H_4(3,7), m-CH_3OC_6H_4(4,8), p-CH_3C_6H_4(5,11), p-ClC_6H_4(6), p-CH_3OC_6H_4(9)$$

All the compounds are solid, which are stable in light and dry air. They are soluble in organic solvents such as $CHCl_3$, CH_2Cl_2, C_6H_6 and DMSO. The analytical and physical data of the synthesized compounds are given in Table 1.

INFRARED SPECTROSCOPY

The infrared spectra of synthesized compounds have been recorded in the range of 4000–400 cm^{-1} and the main spectral data is listed in Table 2. Tentative assignments of C═O, Sn-C, Sn-O and Ge-C bonds have been made on the basis of earlier publications.[14,15] It is well established that triorganotin carboxylates adopt a three structural motifs in its solid state[16] as shown in Figure 1. The mode of coordination of the carboxylate group has been related to the magnitude of the separation ($\Delta\nu$) of the $\nu(COO)_{asy}$ and $\nu(COO)_{sym}$ vibrations.[15,17] The $\Delta\nu$, which indicates the coordination number around the tin, in all complexes increased by over 200 cm^{-1} and this was characteristic of a structural change wherein the carboxylate group passes from the zwitterionic COO^{-1} form in free ligand[18,19] to ester type O-C═O form after organotin coordination resulting in a five-coordinate polymeric structure[20] as confirmed from the crystal structure of compound (1).

^{1}H NMR Spectroscopy

The ^{1}H NMR data of the compounds is given in Table 3. The expected resonances are ascribed by their multiplicity, intensity pattern, integration, coupling constant and tin satellites. All the protons in the compound have been identified and total numbers of protons calculated from the integration curve are in agreement with the expected molecular composition. The ^{1}H NMR data show a well-defined singlet in the region 1.0 to −0.81 ppm for methyl protons directly

TABLE 4
^{13}C NMR data[a–e] for triorganotin derivatives of general formula ((R^1)$_3$ Ge CH (R^2) CH_2 COO) Sn (CH_3)$_3$

Comp. no.	1	2	3	4	5	6	7	8	9	10	11
R^1-Ge											
1	138.04	138.0	138.00	138.5	137.81	137.42	140.51	141.12	138.50	141.60	138.89
2	137.53	134.42	134.51	134.1	136.12	136.84	138.24	138.59	137.01	139.51	135.32
3	130.13	128.56	129.10	128.41	128.79	128.91	128.61	122.50	126.31	126.70	128.81
4	132.61	130.50	131.46	129.50	131.80	132.30	131.34	129.54	129.52	129.94	129.95
s	—	—	—	—	—	—	21.81	21.93	20.85	21.71	21.94
R^2											
1		136.15	138.5	136.42	136.41	138.1	137.42	136.43	136.51	141.9	136.41
2		133.51	133.67	136.00	134.32	137.2	135.61	132.64	132.42	134.6	132.51
3	18.01	128.47	159.51	157.13	130.20	132.5	159.51	158.10	159.10	127.04	128.43
4		130.04	113.41	113.89	131.32	—	112.65	113.52	114.52	129.61	129.31
s		—	57.12	55.15	21.89	—	54.84	55.01	55.43	129.61	21.01
CH	21.52	30.01	34.51	32.15	31.98	29.71	29.91	27.53	31.48	34.01	32.14
CH_2	40.45	39.45	39.46	37.91	40.16	40.43	36.9	37.98	43.14	38.51	37.95
COO	182	178	178.5	178	178.56	178.62	178.43	178.5	178.5	177.69	178.0
Sn-CH_3	−2.20	−2.14	−2.52	−2.10	−2.45	−1.86	−2.45	−2.23	−2.01	−2.21	−2.00
^{119}Sn	131.45	133.64	131.00	133.35	132.63	132.61	130.99	132.92	131.90	125.81	132.91

[a]In $CDCl_3$ at 295 K.
[b]s = Substituents on phenyl ring.
[c]Chemical Shifts in ppm.
[d]R^1 = C_6H_5 (1–6), *p*-$CH_3C_6H_4$ (7–11).
[e]R^2 = CH_3 (1), C_6H_5 (2,10), *o*-$CH_3OC_6H_4$(3,7), *m*-$CH_3OC_6H_4$ (4,8), *p*-$CH_3C_6H_4$ (5,11), *p*-ClC_6H_4 (6), *p*- $CH_3OC_6H_4$ (9).

attached to the tin atom in all synthesized compounds. The well-resolved coupling constant, $^2J[^{119}Sn - {}^1H]$, falls in the range of 58–59.8 Hz and is indicative of tetrahedral geometry of the compounds in a non-coordinated solvent,[21,22] which is further supported by bond angle (111–112°) calculated by using Lockhart's equation.[23] Another characteristic feature of these compounds is the presence of GeCH chiral center and CH_2 is prochiral center, and three hydrogen atoms of the unit $GeCHCH_2$ comprising of three hydrogen atoms that appear as two sets of multiplets in the region of 3.15–3.82 ppm and 2.41–2.81 ppm, respectively.[24]

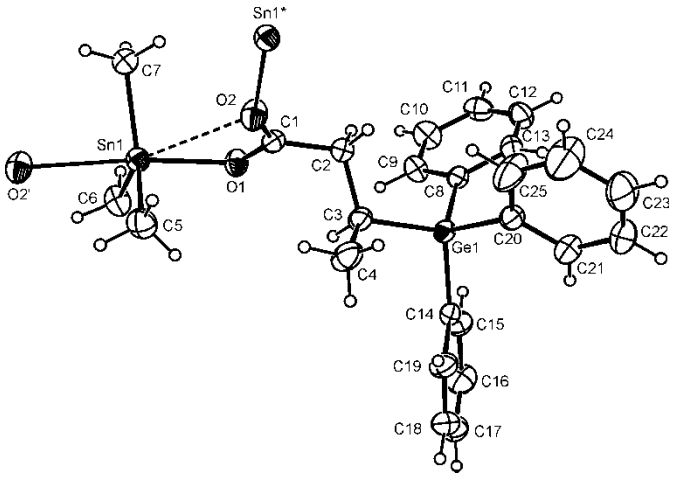

FIG. 2. X-ray structure of: [Trimethyltin(IV)-3-(Triphenylgermyl)-3-methyl) propionate].

^{13}C NMR Spectroscopy

The ^{13}C NMR spectral data of the synthesized compounds is given in Table 4. The number of the signals found corresponds with the presence of magnetically non-equivalent carbon atoms, which were assigned by comparison with literature values.[25,26] The carbon atom directly attached to tin resolved in the expected region −1.86 to −2.54 ppm. The aromatic carbon present in germanium-substituted ligand acids were assigned by comparing the experimental chemical shifts with those calculated form the incremental method.[27]

^{119}Sn NMR data for these compounds, listed in Table 4, show single resonance in the region 125.87–133.64 ppm and this further supports the four coordination of tin in solution.[28]

X-Ray Crystallography of $(CH_3)_3Sn$ ($C_{22}H_{21}O_2Ge$)

The crystal structure of $(CH_3)_3Sn(C_{22}H_{21}O_2Ge)$ is depicted in Figure 2. The relevant structure refinement data, selected

bond length and bond angles are given in Table 5 and Table 6. In this compound, germanium adopts a distorted tetrahedral geometry C-Ge-C, with its angles lying in the range of 107.34(13) and 113.98(13)°. The Ge(1)-C(3) distance, 1.976(3), is significantly longer than the Ge(1)-C(8), Ge(1)-C(14) and Ge(1)-C(20) within 3 σ limits with mean value of 1.952(3)Å. The geometry around tin atom is trigonal bipyramid with the equatorial plane being defined by three methyl carbon atoms with oxygen atoms occupying the axial positions. The three Sn-C distances are equal within the experimental error [2.110(3), 2.115(3), 2.117(3) Å]. The Sn-O bond lengths are significantly different [Sn1-O1 2.155(2) and Sn1-O2 2.533(2) Å]. The shorter C1-O1 bond, 1.287 Å, and the longer Sn1-O1 bond, 2.533(2) Å, share the same oxygen atom. This could be because one of the carboxyl oxygen atom is coordinated to tin with a longer Sn-O distance because the C-O bond keeps some of its double bond character. The central methyl tin group bridges two neighboring germanium substituted carboxylic acid ligand via ester type O-C═O form after organotin coordination to form a one-dimensional polymeric structure.[29]

BIOLOGICAL ACTIVITY

The antibacterial activity of synthesized compounds containing germanium were screened against different types of bacteria, *E. coli, B. substilis, S. flexinari, S. auereus, P. aeruginosa* and *S. typhi*. The results have been compared with the reference drug imipenum and are listed in Table 7. It is well documented that the trimethyl complexes are more biocidally active than the other class of alkyl species. All compounds show good activity; compounds (6) was found to be more active than the reference drug. The greater activity of this compound is probably due to the presence of chlorine in the ligand acid, which itself antibacterial.[30]

The antifungal activity of the compounds was tested against various fungi, *T. longifusus, C. albicans, A. flavus, M. canis, F. solani* and *C. glaberata.* The results are summarized in Table 8.

The results showed that most of the compounds show significant activity against all tested fungi except *C. albicans*. The increased fungicidal activity of trimethyltin derivatives is due to the triarylgermyl substituted ligand acid which could act as carrier in this series.[31]

Brine Shrimp (*Artemia Salina*), a tiny crustacean, was used for the determination of toxicity of compounds. The results are given in Table 9. The LD_{50} values were found in between 5.01–501.00 μg/mL which suggests, comparatively, a high degree of toxicity for these compounds except compounds I, II and III which show lower toxicity with lethal dose greater than 35 μg/mL.

TABLE 5
Crystal data and structure refinement of compound (**1**)

Empirical formula	$C_{25}H_{30}GeO_2Sn$
Formula weight	553.77
Crystal system	Monoclinic
Space group	$P2_1/c$
a(Å)	13.901(3)
b(Å)	9.440(2)
c(Å)	19.017(5)
β(°)	97.632(14)
Volume	2473.4(10)Å^3
Z	4
Absorption coefficient	2.242 mm^{-1}
θ_{max}(°)	27.5
Reflections collected	10526
Independent reflections	5632 [R(int) = 0.029]
Max. and min. transmission	0.807 and 0.663
Goodness-of-fit on F^2	1.02
Final R indices [I > 2sigma(I)]	R1 = 0.032, wR2 = 0.068
R indices (all data)	R1 = 0.053, wR2 = 0.077
δ_{max}(e.Å^{-3})	0.55

TABLE 6
Selected bond lengths [Å] and bond angels [°] of compound (**1**)

Bond lengths	Bond angles
Sn(1)-C(5)	2.110(3)
Sn(1)-C(6)	2.115(3)
Sn(1)-C(7)	2.117(3)
Sn(1)-O(1)	2.155(2)
Sn(1)-O(2)#1	2.533(2)
Ge(1)-C(20)	1.948(3)
Ge(1)-C(8)	1.951(3)
Ge(1)-C(14)	1.957(3)
Ge(1)-C(3)	1.976(3)
O(1)-C(1)	1.287(4)
O(2)-C(1)	1.226(3)
C(5)-Sn(1)-C(6)	114.77(15)
C(5)-Sn(1)-C(7)	127.50(15)
C(6)-Sn(1)-C(7)	115.02(14)
C(5)-Sn(1)-O(1)	94.25(12)
C(6)-Sn(1)-O(1)	92.82(11)
C(7)-Sn(1)-O(1)	98.91(11)
C(20)-Ge(1)-C(8)	107.34(13)
C(20)-Ge(1)-C(14)	108.73(12)
C(8)-Ge(1)-C(14)	108.41(12)
C(20)-Ge(1)-C(3)	113.98(13)
C(8)-Ge(1)-C(3)	110.27(12)
C(14)-Ge(1)-C(3)	107.99(12)

Symmetry transformations used to generate equivalent atoms: #1-x,y + 1/2, -z + 1/2, #2-x,y-1/2, -z + 1/2.

TABLE 7
Antibacterial activity data[a–c] of organotin derivatives of (1–10) 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	9	10	
Escherichia coli	29	30	25	28	14	28	10	29	26	15	30
Bacillus subtilis	10	10	10	10	10	30	—	—	10	10	31
Shigella flexenari	—	—	8	10	12	30	—	10	—	8	33
Staphylococcus	42	40	8	35	14	40	—	—	—	8	43
Pseudomonas aeruginosa	10	10	7	8	8	23	6	—	9	5	25
Salmonella typhi	40	39	36	40	14	39	15	32	39	12	41

[a]Concentration of sample = 3 mg/mL.
[b]Concentration of standard drug (Imipenum) = 10 μg/disc.
[c](−) = No activity.

TABLE 8
Antifungal activity data[a–c] of organotin(IV) derivatives containing germanium (in vitro)

Name of fungus	Zone of inhibition of sample										Std. drug MIC μg/mL	Zone of inhibition of std. drug
	1	2	3	4	5	6	7	8	9	10		
Trichophyton longifusus	69	60	65	61	40	68	65	70	35	30	Miconazole	70
Candida albicans	99	86	98	60	40	109	109	105	65	30	″	110
Aspergillus flavus	—	—	—	—	—	—	—	—	—	—	Amphotericine in B	20
Microsporum canis	88	84	44	51	25	92	88	92	35	20	Miconazole	98
Fusarium solani	30	30	40	35	20	69	20	30	25	20	″	73
Candida glaberata	106	93	85	95	95	105	98	102	20	72	″	110

[a]Concentration of sample = 400 μg/mL of DMSO.
[b]Incubation temperature (period) = 27 ± 1°C (7 days).
[c](−) = No activity.

TABLE 9
Cytotoxicity data[a–c] of organotin(IV) derivatives containing germanium

Comp. no.	1	2	3	4	5	6	7	5	9	10	Std. drug
LD50 (μg/mL)	61.00	59.98	501.00	60.00	35.86	05.01	05.91	16.90	16.90	6.00	7.46

[a]Organism = Artemia salina (brine shrimp) in vitro
[b]Standard drug = Etoposide.
[c]LD_{50} = Lethal dose at which 50% organisms die.

Supplementary Data

Crystallographic data for the compound (I) have been deposited with Cambridge Crystallographic Data Center as CCDC Number: 252060. Copies of the data can be obtained on request to CCDC,12 Union Road, Cambridge CB21 EZ, UK. E-mail: deposited@ccdc.cam.ac.uk or <http//www.ccdc.cam.ac.uk>.

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