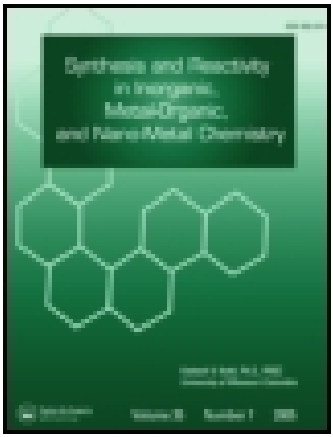

Synthesis and Reactivity in Inorganic and Metal-Organic Chemistry

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Synthesis, Spectroscopic Characterization and Biological Studies of Organotin Derivatives of 2- (2,6-dichlorophenyl)aminophenylacetic

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SYNTHESIS, SPECTROSCOPIC CHARACTERIZATION AND BIOLOGICAL STUDIES OF ORGANOTIN DERIVATIVES OF 2-(2,6-DICHLOROPHENYL)AMINOPHENYLACETIC ACID

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ABSTRACT

Infrared, multinuclear NMR, mass and Mössbauer spectral techniques have been used to characterize new organotin compounds of 2-(2,6-dichlorophenyl)aminophenyl-acetic acid anion with the general formulae R_3SnL, R_2SnL_2 and $R_2Sn(Cl)L$ where R = CH_3, C_4H_9, C_6H_5, $C_6H_5CH_2$ and L = 2-(2,6-dichlorophenyl)-aminophenylacetic acid anion. These techniques were used to compare the geometry of these compounds as solids and in solution. Antibacterial and antifungal activities were determined in order to study their biological significance.

INTRODUCTION

In recent years, a large number of organotin carboxylates have been synthesized and characterized by different spectroscopic techniques[1-7]. These techniques, such as IR, multinuclear NMR, mass and Mössbauer spectroscopy are not only used to predict the geometry around the tin atom but also to compare the chemistry of organotin carboxylates as solids and in solution[8-11]. Our previous reports reveal the synthesis and

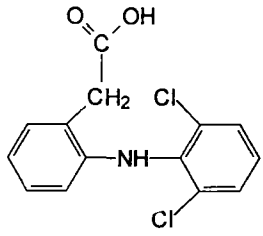


Figure 1. Structure of the Ligand HL

characterization of many organotin carboxylates by spectroscopic techniques[12-16] as well as X-ray crystal structures[16-22]. In the present report we discuss the synthesis and the characterization of organotin derivatives of 2-(2,6-dichlorophenyl)aminophenylacetic acid. The structure of the ligand is shown in Fig. 1. The antibacterial and antifungal activities of these derivatives are also tested.

RESULTS AND DISCUSSION

The tri- and diorganotin compounds of 2-(2,6-dichlorophenyl)aminophenylacetic acid were prepared by the reaction of the sodium salt of the acid with the respective organotin chlorides [equations (1) and (2)].

$$R_3SnCl + NaL \longrightarrow R_3SnL + NaCl \qquad (1)$$

[R = Me (I), Bu (II), Ph (III), Benz (IV)]

$$R_2SnCl_2 + 2NaL \longrightarrow R_2SnL_2 + 2NaCl \qquad (2)$$

[R = Bu (V), Ph (VI), Benz (VII)]

The redistribution reaction of diorganotin dicarboxylate with diorganotin dichloride yielded chloro derivatives [equation (3)].

$$R_2SnL_2 + R_2SnCl_2 \longrightarrow 2R_2Sn(Cl)L \qquad (3)$$

[R = Bu (VIII), Ph (IX), Benz (X)]

The physical data of the compounds are given in Table I.

Infrared Spectra

The infrared spectra of the sodium salt and the tin compounds have been recorded as KBr discs in the range 4000-250 cm^{-1}. The bands of interest are ν(COO), ν(Sn-C), ν(Sn-O) and ν(Sn-Cl), which are given in Table II. The COO stretching vibrations are very important to predict the bonding mode of the ligand. As shown in Table II, the Δν values [$\Delta\nu = \nu_{asym}(COO) - \nu_{sym}(COO)$] are comparable to the sodium salt which clearly indicate the bidentate nature of the ligand[8,10]. In case of the chloro compounds, the Δν values also show the bidentate nature of the ligand[15]. The bands in the range of 600-500 cm^{-1} and 500–400 cm^{-1} indicate the Sn-C and Sn-O bonds, respectively[8,10,15]. In the chloro derivatives the Sn-Cl band is observed in the 335-330 cm^{-1} region.

NMR Spectra

The ^1H and ^{13}C NMR data are presented in Tables III and IV, respectively. The expected resonances are assigned by their multiplicity and intensity pattern, integration, and coupling constants. The integration of the spectra is in good agreement with the expected values in the complex molecules. The coupling constant values $^1J[^{119}Sn\text{-}^{13}C]$ and $^2J[^{119}Sn\text{-}^1H]$ for the compound (I) show tetrahedral geometry around the tin atom[9], which is further supported by the C-Sn-C bond angle (Table V). Due to complex multiple patterns, the $^2J[^{119}Sn\text{-}^1H]$ coupling constants are not observed for the other compounds. Various literature methods[23,24] have been applied to calculate the C-Sn-C bond angles in solution based on $^1J[^{119}Sn\text{-}^{13}C]$ coupling constants (Table V). As far as the geometry of the diorganotin dicarboxylates in non-coordinating solvents is concern, it is not defined with certainty due to the fluxional behaviour of the carboxylate oxygens in their coordination with the tin atom. However, earlier reports[9,25] suggest a geometry in-between penta- and hexa-coordination. For the chlorodiorganotin carboxylates, the presence of oxygen and chlorine atoms on tin increase their Lewis acidity which

Table I. Physical Data of the Investigated Organotin Carboxylates[a]

Compounds (Formula weight)	M.p. ^0C	% Yield	%C Cacl./(found)	%H Cacl./(found)	%N Cacl./(found)
(I) Me_3SnL $C_{17}H_{19}Cl_2NO_2Sn$ (459)	125	86	44.44 (44.29)	4.14 (4.20)	3.04 (3.03)
(II) Bu_3SnL $C_{26}H_{37}Cl_2NO_2Sn$ (585)	35	80	53.33 (52.96)	6.32 (6.29)	2.39 (2.41)
(III) Ph_3SnL $C_{32}H_{25}Cl_2NO_2Sn$ (645)	95	82	59.53 (59.48)	3.88 (3.84)	2.17 (2.16)
(IV) $Benz_3SnL$ $C_{35}H_{31}Cl_2NO_2Sn$ (687)	110	69	61.14 (61.10)	4.51 (4.48)	2.03 (1.92)
(V) Bu_2SnL_2 $C_{36}H_{38}Cl_4N_2O_4Sn$ (823)	70	75	52.49 (52.62)	4.62 (4.72)	3.40 (3.61)
(VI) Ph_2SnL_2 $C_{40}H_{30}Cl_4N_2O_4Sn$ (863)	100	78	55.62 (55.47)	3.48 (3.48)	3.24 (3.19)
(VII) $Benz_2SnL_2$ $C_{42}H_{34}Cl_4N_2O_4Sn$ (891)	80	72	56.56 (56.39)	3.82 (3.79)	3.14 (3.20)
(VIII) $Bu_2Sn(Cl)L$ $C_{22}H_{28}Cl_3NO_2Sn$ (563.5)	75	97	46.85 (46.76)	4.97 (5.01)	2.48 (2.52)
(IX) $Ph_2Sn(Cl)L$ $C_{26}H_{20}Cl_3NO_2Sn$ (603.5)	110	95	51.70 (51.60)	3.31 (3.29)	2.31 (2.32)
(X) $Benz_2Sn(Cl)L$ $C_{28}H_{24}Cl_3NO_2Sn$ (631.5)	70	97	53.21 (52.98)	3.80 (3.80)	2.20 (2.21)

[a]In all other tables the formulation and numbering of the compounds are the same as given in this table.

Table II. Infrared Data for the Investigated Compounds

Comp. No.	ν(COO)		Δν	ν(Sn-C)	ν(Sn-O)	ν(Sn-Cl)
	Asym..	Sym.				
(I)	1546 s	1380 s	166	551 m	446 w	-
(II)	1560 s	1390 s	170	550 s	450 s	-
(III)	1565 s	1395 s	170	540 m	450 m	-
(IV)	1570 s	1380 s	190	535 m	447 m	-
(V)	1575 s	1405 s	170	529 m	424 w	-
(VI)	1579 s	1400 s	179	541 m	447 m	-
(VII)	1572 s	1379 s	193	530 s	440 m	-
(VIII)	1553 s	1389 s	164	530 m	450 m	335 s
(IX)	1572 s	1398 s	174	525 m	445 w	333 s
(X)	1572 s	1378 s	194	550 m	445 m	330 s
NaL	1580 s	1402 s	178	-	-	-

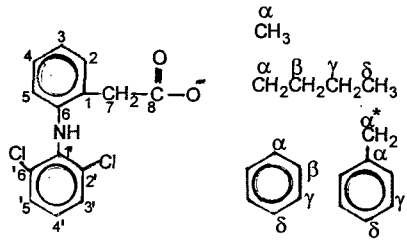
facilitates intramolecular coordination[26] of the carboxylate group resulting in a trigonal bipyramidal geometry.

^{119}Sn NMR is also a powerful technique for the determination of the coordination number of tin. We report here ^{119}Sn NMR data for two compounds, Me_3SnL and Ph_2SnL_2 (Table VI). The chemical shift observed for Me_3SnL is in the same range as reported for other $Me_3SnOOCR$ derivatives where R = H, CH_3, the δ ^{119}Sn values are 150 and 129 ppm, respectively, suggesting a coordination number ≥ 4[27]. For Ph_2SnL_2 we observed δ ^{119}Sn at -103.7 ppm whereas Wrackmeyer[27] in his review has reported -101.2 ppm for diphenyltin dithiocarboxylate having the coordination number ≥ 4.

Mössbauer Spectra

Mössbauer spectra were recorded for the investigated compounds in order to predict the geometry around the tin atom and to compare with their structures in solution

Table III. ^{1}H NMR Data (in ppm) for the Investigated Compounds[a]



H No.	(I)	(II)	(III)	(IV)	(V)	(VI)	(VII)	(VIII)	(IX)	(X)
2	7.12 (d, 9)	7.11 (d, 8)	7.36 (d, 8)	7.42 (d, 8)	7.15 (d, 9)	7.13 (d, 10)	7.45 (d, 10)	7.21 (d, 8)	7.31 (d, 8)	7.42 (d, 8)
3	7.09 (dd, 8, 8)	7.08 (dd, 8, 8)	7.25 (dd, 8, 8)	7.35 (dd, 8 8)	7.07 (dd, 8, 8)	7.10 (dd, 8, 8)	7.32 (dd, 8, 8)	7.12 (dd, 7, 7)	7.24 (dd, 7, 7)	7.39 (dd, 7, 7)
3',5'	7.34 (d, 9)	7.32 (d, 9)	7.22 (d, 9)	7.28 (d, 9)	7.35 (d, 9)	7.28 (d, 9)	7.31 (d, 9)	7.39 (d, 8)	7.16 (d, 8)	7.30 (d, 8)
4	6.93 (dd, 9, 9)	6.95 (dd, 8, 8)	6.97 (dd, 7, 7)	6.98 (dd, 7, 7)	6.84 (dd, 7, 7)	6.94 (dd, 7, 7)	6.96 (dd, 7, 7)	6.97 (dd, 8, 8)	6.95 (dd, 8, 8)	6.79 (dd, 8, 8)
4'	7.24 (dd, 8, 8)	7.24 (dd, 8, 8)	7.13 (dd, 8, 8)	7.18 (dd, 8, 8)	7.18 (dd, 8, 8)	7.28 (dd, 8, 8)	7.20 (dd, 8, 8)	7.29 (dd, 8, 8)	7.01 (dd, 8, 8)	7.22 (dd, 8, 8)
5	6.94 (d, 9)	6.92 (d, 9)	6.50 (d, 9)	6.60 (d, 9)	6.82 (d, 9)	6.91 (d, 9)	6.67 (d, 9)	6.91 (d, 9)	6.95 (d, 9)	6.51 (d, 9)
7	3.80 (s)	3.81 (s)	3.81 (s)	3.81 (s)	3.83 (s)	3.99 (s)	3.91 (s)	3.82 (s)	3.72 (s)	3.89 (s)
α	0.56(s) ^{2}J[58.3]	1.5 – 1.6 (m)	-	-	1.7-1.8 (m)	-	-	1.71 (m)	-	-
β	-	1.5 – 1.6 (m)	7.73 (m)	7.69 (m)	1.7-1.8 (m)	7.73-7.71 (m)	7.65-7.70 (m)	1.36-1.39 (m)	7.67 (m)	7.65 (m)
γ	-	1.3-1.4 (m)	7.69 (m)	7.71 (m)	1.3-1.4 (m)	7.73-7.71 (m)	7.65-7.70 (m)	1.36-1.39 (m)	7.4 (m)	7.41 (m)
δ	-	0.61 (t)	7.50 (m)	7.60 (m)	0.78 (t)	7.4 (m)	7.41 (m)	0.67 (t)	7.66 (m)	7.8 (m)
NH	9.1 (s)	9.3 (s)	9.2 (s)	9.5 (s)	9.4 (s)	9.5 (s)	9.2 (s)	9.3 (s)	9.4 (s)	9.5 (s)
α*	-	-	-	2.36 (s)	-	-	2.34 (s)	-	-	2.34 (s)

[a] ^{2}J[^{119}Sn-^{1}H] Hz, ^{3}J(H-H) Hz are listed in parenthesis, s = singlet, d = doublet, dd = doublet of doublets, m = multiplet

Table IV. ^{13}C NMR Data (in ppm) for the Investigated Compounds[a]

C No.	(I)	(II)	(III)	(IV)	(V)	(VI)	(VII)	(VIII)	(IX)
1	138.0	138.3	138.7	138.3	137.9	138.0	139.2	138.8	138.0
1′	129.4	129.5	129.8	129.7	129.5	129.4	129.8	129.9	129.4
2	122.7	123.6	123.7	123.0	123.9	123.6	124.7	123.4	123.5
2′,6′	130.8	130.8	130.7	130.6	130.8	130.9	131.7	132.0	131.6
3	121.8	121.7	121.6	122.2	122.0	121.9	121.9	121.5.	121.4
5	124.2	124.4	124.0	123.9	124.4	124.3	124.3	123.8	123.9
3′,5′	128.9	128.8	128.7	128.6	128.8	129.0	128.2	128.9	128.9
4	118.0	118.0	118.2	119.7	118.2	118.2	117.9	118.2	118.0
4′	125.8	126.0	125.1	125.3	125.3	125.2	125.9	126.1	125.1
6	127.5	127.4	127.0	127.8	127.9	127.7	127.9	127.8	127.5
7	42.8	42.9	40.0	42.1	42.7	42.8	42.8	51.6	49.0
8	177.4	177.9	177.5	179.1	178.2	178.7	178.5	180.0	179.1
α	-2.8 ^{1}J[398]	16.4 ^{1}J[362]	136.9 ^{1}J[649]	130.2	23.4 ^{1}J[569]	137.9 ^{1}J[644]	131.1	25.6 ^{1}J[499]	140.2 ^{1}J[652]
β	-	27.8 ^{2}J[22]	138.9 ^{2}J[50]	116.8	26.4 ^{2}J[34]	136.9 ^{2}J[48]	129.9	26.4 ^{2}J[35]	139 ^{2}J[50]
γ	-	27.0	132.0	112.8	26.1 ^{3}J[93]	130.2 ^{3}J[65]	118.0	26.1 ^{3}J[93]	131.1 ^{3}J[67]
δ	-	13.6	129.2 ^{4}J[14]	110.0	13.4	128.9 ^{4}J[34]	113.2	13.9	128.0
α*	-	-	-	26.9	-	-	25.9	-	-

[a] $^{n}J[^{119}Sn\text{-}^{13}C]$ in Hz are listed in brackets, numbering scheme is given in Table III

Table V. C-Sn-C Angles (°) Based on NMR Data[a]

Compound No.	$^{1}J[^{119}Sn-^{13}C]$	θ(°)
(I)	398.0	111.66
(II)	361.9	112.6
(III)	649.0	116.46
(V)	568.9	131.98
(VI)	644	116.0
(VIII)	499	128.26
(IX)	652	116.65

[a]Formulae used for the calculation of bond angles are from refs. 23 and 24.

(NMR) and in the solid (IR and Mössbauer). Mössbauer parameters are given in Table VI. The QS values are very important for the prediction of the geometry around the tin atom. The triorganotin compounds, except triphenyltin derivative, show a *trans*-R_3SnO_2 arrangement with bridging carboxylate [(1) in Fig. 2][28], which is further supported by the crystal structure of the analogous trimethyltin derivative of [(2,3-dimethylphenyl)amino]benzoate[20]. Triphenyltin derivative show QS values at 2.51 mms^{-1} which suggest a monomeric trigonal bipyramidal geometry [(2) in Fig. 2].

As reported earlier[9a], if the ρ value (QS/IS) is greater than 2.1, the geometry around the tin atom is octahedral, thus the diorganotin compounds in the present study show monomeric *trans*-hexa-coordinated geometry [(3) in Fig. 2], except Ph_2SnL_2 for which *cis*-hexa-coordination is proposed on the basis of the low QS value[9b,c].

The chloro derivatives show higher values of IS and QS as compared to triorganotin compounds which is accord with a *cis*-$R_2(X)SnO_2$ geometry [(4) in Fig. 2][15].

Mass Spectra

The 80 eV monoisotopic mass spectral fragmentation patterns of the investigated compounds are cited in Tables VII and VIII. The molecular ion peak in most compounds

Table VI. ^{119m}Sn Mössbauer and ^{119}Sn NMR Data for the Investigated Compounds[a]

Comp. No.	IS	QS	Γ_1	Γ_2	ρ = QS/IS	^{119}Sn δppm
(I)	1.24	3.46	0.87	0.87	2.79	141.8
(II)	1.45	3.68	0.90	0.90	2.54	-
(III)	1.16	2.51	0.94	0.98	2.16	-
(IV)	0.70	1.69	1.04	1.04	2.41	-
(V)	1.37	3.28	0.89	0.94	2.39	-
(VI)	0.87	2.78	0.90	0.95	3.19	-103.7
(VIII)	1.54	3.8	0.89	1.00	2.47	-
(IX)	1.23	2.81	0.98	1.02	2.28	-
(X)	1.54	3.48	0.99	1.04	2.26	-

[a]IS = isomer shift (mm s^{-1}), QS = quadruple splitting (mm s^{-1}), Γ = line width at half hight

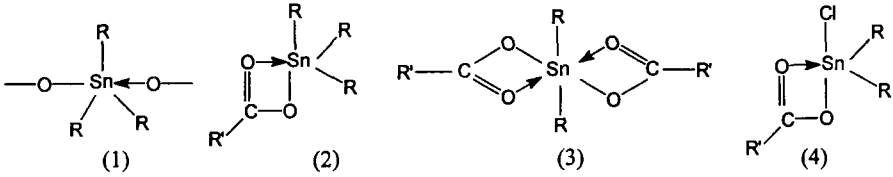


Figure 2. Suggested Structures of the Complexes.

is not observed except in compounds (I) and (VI)[11]. In triorganotin derivatives, the primary fragmentation is due to loss of the R group, which follows the elimination of CO_2[8,11]. The rest of the fragmentation follows the same pattern as reported earlier[10]. In the dibutyltin compounds the first decomposition is due to the loss of the R group and the second due to R and CO_2 while diphenyl and dibenzyl derivatives eliminate the R group and one ligand in the second step. Chloro derivatives follow elimination of chloride first and then behave similar to triorganotin derivatives[15].

Table VII. Fragmentation Pattern and Relative Abundance of Triorganotin and Chlorodiorganotin Derivatives

Fragment ion	(I)	(II)	(III)	(IV)	(VII)	(IX)	(X)
$C_6H_3Cl_2NHC_6H_4CH_2COOSnR_3^+$	32	-	-	-	-	-	-
$C_6H_3Cl_2NHC_6H_4CH_2COOSn(Cl)R_2^+$	-	-	-	-	-	2	-
$C_6H_3Cl_2NHC_6H_4CH_2COOSnR_2^+$	24	35	35	15	100	100	100
$C_6H_3Cl_2NHC_6H_4CH_2SnR_2^+$	52	100	49	40	-	-	-
$C_6H_3Cl_2NHC_6H_4CH_2COOSnR^+$	-	-	-	-	52	59	49
$C_6H_3Cl_2NHC_6H_4CH_2Sn^+$	4	22	71	100	41	70	50
$C_6H_3Cl_2NHC_6H_4CH_2^+$	-	-	-	-	68	69	25
R_3Sn^+	67	9	21	50	-	-	-
R_2Sn^+	7	15	25	31	14	22	-
RSn^+	8	6	15	11	-	-	-
$C_6H_3Cl_2NH^+$	-	43	100	82	85	28	12
SnH/Sn^+	2.8	28	30	14	44	40	38
$C_6H_4CH_2^+$	-	15	10	8	18	15	10

Table VIII. Fragmentation Pattern and Relative Abundance of Diorganotin Derivatives

Fragment ion	(V)	(VI)	(VII)
$(C_6H_3Cl_2NHC_6H_4CH_2COOSn)_2SnR_2^+$	-	3	-
$(C_6H_3Cl_2NHC_6H_4CH_2COO)_2SnR^+$	30	5	62
$C_6H_3Cl_2NHC_6H_4CH_2COOSn^+$	55	100	45
$C_6H_3Cl_2NHC_6H_4CH_2Sn^+$	-	-	100
$C_6H_3Cl_2NHC_6H_4CH_2^+$	100	78	29
$C_6H_3CH_2Sn^+$	25	48	21
SnH/Sn^+	3	25	15
$C_6H_4CH_2^+$	6	10	7

Biological Activity

The biological activity studies of some representative compounds were carried out by the "agar well diffusion method" [29] and the results are given in Tables IX-XI.

The triorganotin compounds are more active against gram positive and gram negative bacteria than the diorganotin and chloro derivatives[9a]. It is very interesting that NaL itself is almost inactive but its organotin derivatives are either highly active or moderately active against these bacteria. Among the diorganotin and chlorodiorganotin derivatives, the diorganotin compounds are more active. Thus the activity decreases in the following order:

$$R_3SnOCOR' > R_2Sn(OCOR')_2 > R_2Sn(Cl)OCOR'$$

The antifungal activity of NaL and its organotin derivatives is given in Table XI which shows that the triorganotin derivatives are more active than the diorganotin and chloro derivatives. Amongst the triorganotin derivatives, the trimethyltin compound shows the highest activity.

EXPERIMENTAL

Instrumentation

Infrared spectra were recorded as KBr discs on a Hitachi model 270-1117 spectrophotometer. NMR spectra were obtained on Bruker SF 300 or SF 400 spectrometers using $CDCl_3$ as internal reference and a Jeol FX90Q instrument with Me_4Sn as external reference for ^{119}Sn NMR. Mass spectral data were measured on a JMS-DX 300 mass spectrometer. ^{119m}Sn Mössbauer spectra were recorded with a constant acceleration microprocessor-controlled spectrometer (Cryoscopic Ltd., Oxford, UK). A barium stannate source was used at room temperature and samples were packed in perspex discs and cooled to 80K. Isomer shift data are relative to SnO_2.

Synthesis

The general method for the synthesis of tri- and diorganotin derivatives is given below: A quantity of 0.01 mole (2.96 g) of the sodium salt of 2-(2,6-

Table IX. Antibacterial Activity (Gram Positive)[a]

Bacterium	NaL	(I)	(II)	(III)	(IV)	(V)	(VI)	(VIII)
Staphylococcus aureus	-	+++	+++	++	+++	+	++	++
Staphylococcus epidermiedis	-	++	+++	++	++	+	+	-
Strepotococous pyogenes	-	++	++	0	++	+	-	+
Bacillus anthracis	-	++	0	+++	++	+	+	+
Corynebacterium species	-	0	++	++	+++	0	+	-
Clostridium species	0	-	++	++	+	+	+	+
Peptococcus species	-	++	+++	+++	+++	+++	++	++
Streptococcus pneumonial	-	++	+	++	++	-	0	++
Streptofaecates	-	+	+++	+++	+++	++	0	+
Listeris monocytogenes	-	++	+++	-	0	+	+	+
Micrococci	-	++	++	+++	+++	0	0	++

[a] 0 = not tested, - = no activity, + = low activity, ++ = moderate activity +++ = high activity

Table X. Antibacterial Activity (Gram Negative)[a]

Bacterium	NaL	(I)	(II)	(III)	(IV)	(V)	(VI)	(VIII)
Escherichia coli	-	++	+++	+++	++	++	+	+
Proteus mirablis	-	++	+++	++	+	0	++	-
Proteus vulgeris	-	-	++	++	+++	-	+	++
Sallmonella typhi	+	++	+	-	+	+	-	++
C. diptherial	0	+	+++	++	++	++	++	+
P. aeruginosa	-	+	+	+	+	++	++	+
Aeromans sobrial	+	++	++	++	+	+	+	0
Shigella boydie	-	++	++	+	++	-	++	++
Vibrio cholera	-	+	+	++	+	++	++	+
Brucella species	-	+	++	++	++	+	+	+

[a] See footnotes of Table IX

Table XI. Antifungal Activity[a]

Fungus	NaL	(I)	(II)	(III)	(IV)	(V)	(VI)	(VIII)
Candida albican	-	+++	+++	++	-	+	+	+++
Pencillium notatum	0	+++	+++	+++	+	+	++	++
Dutarium notatum	0	++	++	+	-	++	+	++
Gurvularia lunata	+++	+++	+++	0	0	0	++	++
Alterneria solani	0	++	++	-	++	+++	++	++
Fusarium solani	0	+++	-	+++	+++	++	++	++
E. flocosum	+++	++	0	++	++	+	+	-
Candida tropicalis	+	++	++	+	+	++	++	+++
Aspergillus nigar	++	+++	+	+++	++	++	+++	0
Ascomycetes	0	++	+	+++	++	+++	++	+++
Microsponum canis	+++	++	++	-	+	+	++	0

[a] See footnotes of Table IX

dichlorophenyl)aminophenylacetic acid was refluxed with 0.01 mole of triorganotin chloride/0.005 mole of diorganotin dichloride in 50 mL dry chloroform for 6-7 hours under an inert atmosphere. After cooling the reaction mixture to room temperature, sodium chloride was filtered and the solvent was removed by a rotary evaporator. The solid mass left was crystallized from dichloromethane/hexane (1:1). The chloro derivatives were prepared by the method reported earlier[15].

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