

Synthesis and spectroscopic characterization of some sterically hindered μ -oxo bis [tricyclohexyl antimony (V)] carboxylates and -halocarboxylates

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Abstract— A series of sterically hindered μ -oxo bis [tricyclohexylantimony (V)] carboxylates [(cyclo-C₆H₁₁)₃Sb-O-Sb(cyclo-C₆H₁₁)₃](OCOR)₂ where -OCOR = p-trifluoromethyl mandelate, p-methoxy mandelate, salicylate, 2pyrazine carboxylate, mandelate has been synthesised and characterised by solid state infrared, ¹H, ¹⁹F and ¹³C NMR spectroscopy, molecular weight and conductance measurements. The newly synthesized derivatives are monomeric in benzene and non ionic in acetonitrile with pentacoordination dispensation around antimony.

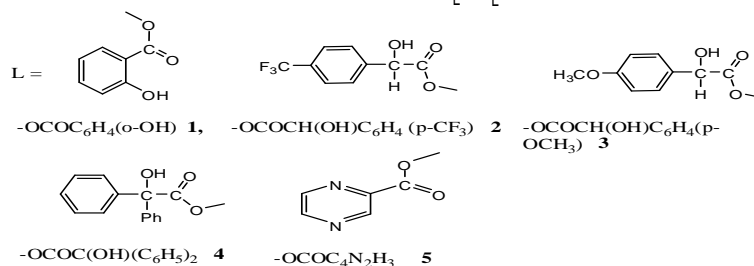
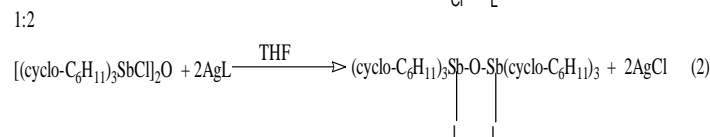
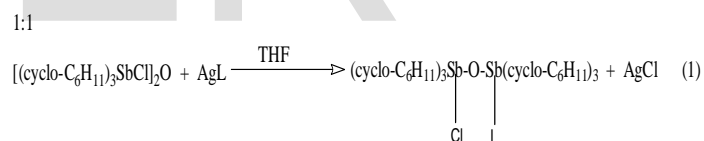
Keywords— μ -oxo bis [tricyclohexylantimony (V)] carboxylates, halocarboxylates, IR, ¹H, ¹⁹F NMR, ¹³CNMR spectra, monomeric, non ionic.

1 INTRODUCTION

Despite a considerable amount of work done on the synthesis and reactivity of organoantimony (V) carboxylates, R_n-Sb(OCOR)_{5-n} (n= 1-4), R = alkyl, aryl, those having Sb-O-Sb framework have been studied to a limited extent [1-8]. The latter class of compounds is important in the light of fact that sodium stibogluconate (pentostam) a well known drug for the treatment of leishmaniasis have the similar framework coupled with gluconate constitution. Our recent in vitro antileishmaniasis and antimicrobial studies indicate that μ -oxobis [(triarylantimony (V))] dicarboxylates exhibit high antifungal, antibacterial, antitumoral and antileishmanial activity [9-15]. Since the biological activity is largely affected by the nature of anion and organic group bound to the central metal atom apart from its nature of metal, geometrical dispensation around metal and oxidation state, we considered it worthwhile to synthesize a series of μ -oxo bridged organoantimony carboxylates and -halocarboxylates having biologically potential organic moieties. It may be noted that cyclohexyltin derivatives exhibit potential antimicrobial and antitumoral activity. In addition to this the introduction of cyclohexyl group is known to render the hydrolytic stability to the system [16-23]. The present investigation concerned with the synthesis of μ -oxobis [(tricyclohexylantimony (V))] dicarboxylates and their characterization by IR, NMR spectroscopy. Solution phase studies complementary to above have been carried out to as-

2 Results and Discussion

The interaction of μ -oxy bis (tricyclohexyl antimony chloride) [(cyclo-C₆H₁₁)₃SbCl]₂O, with the silver salt of corresponding carboxylic acid in 1:1 and 1:2 molar ratio afforded mono and di-substituted μ -oxy bis (tricyclohexyl antimony) derivatives, respectively (Eq. 1-2).

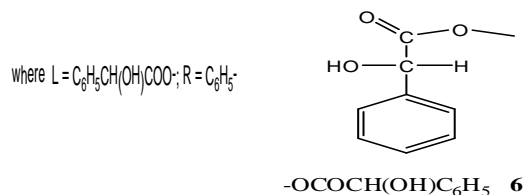
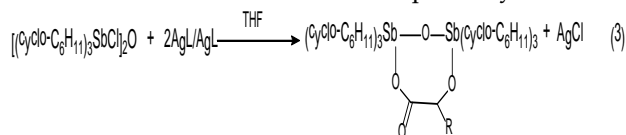


As is well known α -hydroxy carboxylic acid may undergo cyclisation using hydroxyl and carboxylic hydrogen's [23], β -hydroxy, carboxylic acids normally prefer monodentate and linear salts [24]. The only carboxylic acid undergoing cyclisation was mandelic acid to form cyclic μ -oxy-bis (tricyclohexyl antimony) derivatives (**7**, **8**), irrespective of molar ratio i.e. the same product was formed in 1:1 and 1:2 molar ratio reactions. The formation of ring compound was established on the basis of amount of residue after filtration, melting points, elemental

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certain the chemical behavior in solution.

analysis, super imposable IR spectra and other spectral analysis. On the other hand, the other α -hydroxy acid viz. benzoic acid, p. (trifluoromethyl) mandelic acid, p-methoxy mandelic acid do not form the cyclometallates and mono or disubstituted oxo-bridged derivatives of antimony are formed for 1:2 and 1:2 molar ratio reactions, respectively.



The newly synthesized μ -oxy, derivatives of tricyclohexyl antimony (v) are white or off white crystalline solid with sharp melting points and are soluble in organic solvents. The compounds are listed with their physical properties in Table-1. The elemental analysis was found satisfactory and within permissible limits. The data obtained are summarized in Table (3-5). The molar conductance values of the compound in acetonitrile (10^{-3} solution) were found in the appropriate value to show their non-conducting behavior in solution. The molecular weight and thus Vant Hoff factor 'i' determined cryoscopically in nitro benzene confirmed their monomeric nature.

2.1 INFRARED SPECTRA

The infrared absorption for all the μ -oxy bis [tricyclohexylantimony (v)] derivatives were recorded in the range $4000\text{--}400\text{ cm}^{-1}$ using KBr pellets on FT IR spectrophotometer (Shimadzu 8201/PC). The Characteristic absorption frequencies are listed in Table (3). Absorption associated with various internal modes of vibrations of μ -oxy bis [tricyclohexyl antimony (v)] derivatives have been identified and indicate the nature of bonding. Bond due to antimony – oxygen – antimony (Sb – O – Sb) bond is at almost similar position in the range $740\text{--}725\text{ cm}^{-1}$ as strong to very strong band and come in the range of earlier published data [20, 25-26, 27]. The position of asymmetric and symmetric modes and separation ($\Delta\nu$) between them provides a method of assessing carboxylate coordination modes. The non-conducting the monomeric nature and the absence of band at 1556 , 1413 and 650 cm^{-1} due to carboxylate ion [28-31] in the IR spectra further rule out the possibility of an ionic structure. The $\nu(\text{OH})$ for the compounds (**1-6**, **9-10**) was observed in the range $3465\text{--}3240\text{ cm}^{-1}$ as a weak or medium hand. In case of mandelic acid derivatives (**7**, **8**) where the cyclic product was obtained due to participation of hydrogen of carboxylate group and hydroxyl group, $\nu(\text{OH})$ was not recorded.

2.2 ^1H NMR SPECTRA

The ^1H NMR spectra of some derivatives of μ -oxy bis [tricyclohexyl antimony (v)] were recorded on Bruker DRX-300 (300

MHz FT NMR) in solvent (DMSO + CDCl_3) with chemical shift being reported as $\delta(\text{ppm})$ taking tetramethylsilane as reference. The peak for protons of DMSO appeared at δ 83.3 ppm. The ^1H NMR data of the title compounds are listed in Table-4. The chemical shifts of the protons of cyclohexyl ring appear in the range $\delta(1.15\text{--}3.80)$ ppm in the form of multiplet. The appearance of signal of protons of cyclohexyl ring as multiplet is due to low to high long range coupling. When four sigma bonds between interacting protons adopt a 'W-arrangement' as in the case of 1, 3 diequatorial protons of a rigid cyclohexyl system such type of coupling are observed (fig. 1).

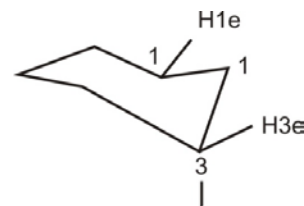


Figure 1

In other word this is due to diaxial, axial, equatorial and diequatorial coupling of protons of rigid cyclohexyl system. All the axial and equatorial protons of cyclohexyl ring give signals at different δ -values. The signals for proton (H1) bonded to ipso carbon of cyclohexyl ring appear in the range $\delta(3.60\text{--}3.80)$ ppm. The axial proton at position – 2 (H2a) and equatorial proton at position – 3 experienced the same electronic environment and were found magnetically equivalent giving out the signal at same position in the range $\delta(1.75\text{--}1.85)$ ppm. In this way, the signals for cyclohexyl protons were found in good agreement with those reported earlier [31, 32]. The α -hydroxy acids containing α -hydrogen (**2-6**, **9**, **10**) shows the proton signal for α -hydrogen in the range $\delta(4.95\text{--}5.15)$ ppm. In the case of compounds, the methoxy proton was observed at $\delta(3.78, 3.81)$ ppm, respectively, as singlet. The phenyl ring protons appear in the range $\delta(6.85\text{--}7.95)$ ppm as multiplet, the details are summarized in the Table (4). The three, magnetically non equivalent protons (H2, H3, H4) of 2-pyrazine carboxylate were observed at $\delta(9.0315)$, $8.75(\text{d})$ and $8.85(\text{d})$ ppm, respectively, the peaks of ligands are comparable with free carboxylic acids and with those of similar organometallic compounds reported earlier [23, 24, 28-40].

2.3 ^{13}C NMR SPECTRA

^{13}C NMR spectra of representative compounds were recorded on 300 MHz FT NMR (Bruker DRX – 300) spectrometer operating at 75 MHz using CDCl_3 as solvent and reference ($\delta 77.0$) ppm with chemical shift being reported as $\delta(\text{ppm})$. The peaks are compared, identified and are listed in Table-5. The chemical shift of four magnetically non-equivalent of cyclohexyl ring were observed at different δ -values in the region $\delta(23.03\text{--}56.72)$ ppm. The ipso-carbon (C1) was more shielded in all cases and the signal appears in the range $\delta(55.09\text{--}56.72)$ ppm. The chemical shift behaviour of carbon centers of phenyl ring is also dependent on the pKa values of the carboxylic acids. The signals for magnetically non-equivalent carbon centers of ligand acid appeared at different δ -value; and the signal for carboxyl ($>\text{C}=\text{O}$) carbon was observed in the range $\delta(165.1\text{--}$

174.75) ppm with a shiftment towards lower field in comparison to that of free acid which indicates the participation of carboxylate group in the formation of Sb-O-C(O) bond. In case of compounds (3, 4, 6 – 8) the chemical shift values for α -C appears in the range δ (73.02 – 81.60) ppm. These values are at lower field in comparison to free acids showing the deshielding effect at α -carbon center due to the coordination of carboxylate group to antimony. The 2-pyrazine carboxylate derivatives give the distinct signals for magnetically non-equivalent carbons. The ^{19}F NMR spectra, in case of compound (5, 6) which contains trifluoromethyl group was also recorded and a signal appeared at δ 62.9 ppm for fluorine atom. Thus, on the basis of molecular weight, conductivity, IR and NMR (^1H , ^{13}C and ^{19}F) spectral data, the μ -oxy bis [tricyclohexyl antimony (v)] derivatives prepared in the present investigation may be assigned a trigonal bipyramidal structure with a Sb-O-Sb linkage and on each antimony atom more electronegative group are situated at apical position (fig. 1-2). The similar structure has also been established for other μ -oxo, derivatives of antimony [9, 13, 15, 19, and 20].

3 EXPERIMENTAL

Tricyclohexylantimony dichloride and μ -oxo bis [tricyclohexylantimony (V)] chloride were prepared by the reported method [21]. The carboxylic acids (all from Aldrich) were used in the form of their silver salts. IR spectra were recorded in solid state, using KBr pellets, on FT-IR spectrophotometer (Schimadzu 8201 PC, Shimadzu Corporation Kyoto, Japan) over the spectral range 4000-400 cm^{-1} . ^1H , ^{19}F , and ^{13}C NMR spectra were recorded on a Bruker spectrometer (Bruker Corporation, US), using TMS, CF_3COOH and CDCl_3 as reference, respectively. The stoichiometry of compounds was established by elemental analysis on a semimicro scale using an elemental analyzer (Elementar Vario EL III, Carlo Erba 1108, Milan, Italy). All the reactions of $[(\text{Cyclo}-\text{C}_6\text{H}_{11})_3\text{SbCl}]_2\text{O}$ with silver salt of corresponding – carboxylic acids were performed under dark condition in presence of inert atmosphere. The corresponding silver salts of carboxylic acids were prepared from their sodium salt by reaction with silver nitrate. Tetrahydrofuran (THF), was distilled purified and dried under an inert atmosphere from sodium – benzophenone. The typical experiments for some representative μ -oxy-bis [tricyclohexyl antimony (v)] carboxylates are given below and the further details for the other reactions are summarized in Table (1). The analytical data are listed in Tables (2-5).

3.1 1:2 Molar Ratio Reaction of $[(\text{Cyclo}-\text{C}_6\text{H}_{11})_3\text{SbCl}]_2\text{O}$ with silver salt of Salicylic Acid (2)

A solution of $[(\text{Cyclo}-\text{C}_6\text{H}_{11})_3\text{SbCl}]_2\text{O}$ (0.829g, 1.0 mmol) and silver salt of salicylic acid (0.490g, 2.0 mmol) in THF (25 mL) was stirred at room temperature for 24h. On filtration of heterogeneous solution containing precipitate of silver chloride, a clear solution was obtained which was concentrated in vacuo (2-3 mL). After the addition of n-hexane (3 mL), the solution was allowed to stand overnight at 0°C affording a white crystalline solid which was recrystallised from a mixture of THF and n-Hexane (1:3). The Compound was characterized as μ -oxy bis [tricyclohexylantimony (v)] (salicylate) (1). In the same manner, 1:1 molar ratio reaction of $[(\text{Cyclo}-\text{C}_6\text{H}_{11})_3\text{SbCl}]_2\text{O}$ (0.829g, 1.0 mmol) with silver salt of salicylic

acid (0.245 g, 1.0 mmol) in THF (20 mL) afforded off white crystalline compound characterized as tricyclohexyl antimony (V) salicylate μ -oxo tricyclohexyl- antimony (v) chloride (1).

3.2 1:2 Molar Ratio Reaction of $[(\text{Cyclo}-\text{C}_6\text{H}_{11})_3\text{SbCl}]_2\text{O}$ with silver salt of (RS) – Mandelic Acid (8)

In an inert atmosphere a solution of $[(\text{Cyclo}-\text{C}_6\text{H}_{11})_3\text{SbCl}]_2\text{O}$ (0.414g, 0.5 mmol) and silver salt of (RS) mandelic acid (0.259g, 1.0 mmol) in THF (20 mL) was stirred at room temperature for 24 h. The insoluble portion of heterogeneous solution containing precipitate of silver chloride was filtered. The filtrate on concentration in vacuo followed by addition of petroleum ether (40-60 $^\circ\text{C}$) afforded white crystalline solid. The compound was crystallized from a mixture of benzene and petroleum ether (40-60 $^\circ\text{C}$) in the ratio (1:2) and was characterized as cyclometallated (RS) - μ -oxy bis [tricyclohexyl antimony (V)] mandelate (8). Similarly, 1:1 molar ratio reaction $[(\text{Cyclo}-\text{C}_6\text{H}_{11})_3\text{SbCl}]_2\text{O}$ (0.829g, 1.0 mmol, with silver salt of (RS) mandelic acid (0.259 g, 1.0 mmol) in THF (20 mL) afforded the same cyclometallates (RS) - μ -oxy bis [tricyclohexyl antimony (V)] mandelate (7).

3.3 1:2 Molar Ratio Reaction of $[(\text{Cyclo}-\text{C}_6\text{H}_{11})_3\text{SbCl}]_2\text{O}$ with silver salt of (RS) p – (Trifluoromethyl) Mandelic Acid (5)

A heterogeneous solution of $[(\text{Cyclo}-\text{C}_6\text{H}_{11})_3\text{SbCl}]_2\text{O}$ (0.415g, 0.5 mmol) and silver salt of (RS) – p – (trifluoromethyl) mandelic acid (0.327 g, 1.0 mmol) in THF (15 mL) was stirred together at room temperature for 24h. The white precipitate of AgCl thus formed was filtered off. The filtrate on concentration under vacuum (2-3 mL) afforded white solid after addition of petroleum ether (60-80 $^\circ\text{C}$) (2mL). The solid compound was crystallized from a mixture of THF and n-Hexane (1:3) and was characterized as μ -oxy bis [tricyclohexyl antimony (V) p-(trifluoromethyl) mandelate] (5).

In the similar fashion 1:1 molar ratio reaction of $[(\text{Cyclo}-\text{C}_6\text{H}_{11})_3\text{SbCl}]_2\text{O}$ (0.829g, 1.0 mmol) and silver salt of p-(trifluoromethyl) mandelic acid (0.327g, 1.0 mmol) in THF (20 mL) afforded the compound (RS) tricyclohexyl antimony (V) p-(trifluoromethyl) mandelate μ -oxo-tricyclohexylantimony (V) chloride (6).

3.4 1:2 Molar Ratio Reaction of $[(\text{Cyclo}-\text{C}_6\text{H}_{11})_3\text{SbCl}]_2\text{O}$ with silver Salt of 2-Pyrazine carboxylic acid (11)

In an oxygen and moisture free atmosphere, a solution of $[(\text{Cyclo}-\text{C}_6\text{H}_{11})_3\text{SbCl}]_2\text{O}$ (0.829g, 1.0 mmol) with silver salt of 2-Pyrazine carboxylic acid (0.462 of, 1.0 mmol) in dry acetone (20 mL) was stirred at room temperature for 12h followed by refluxion for 2h. The silver chloride thus formed was filtered off and filtrate on concentration in vacuo (2mL) was stirred at

room temperature for 12h followed by addition of petroleum ether (60-80°C) (3mL), yielded white crystalline/4-hexane mixture (1:3) to afford μ -oxybis [tricyclohexylantimony (V) (2-pyrazine carboxylate)] (11).

4 FIGURES

Figure 1

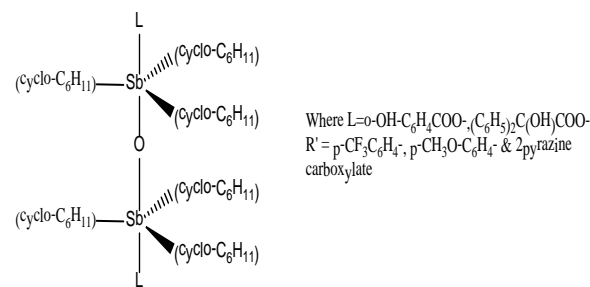


Fig 1 Showing structure of μ -Oxo derivatives

Figure 2

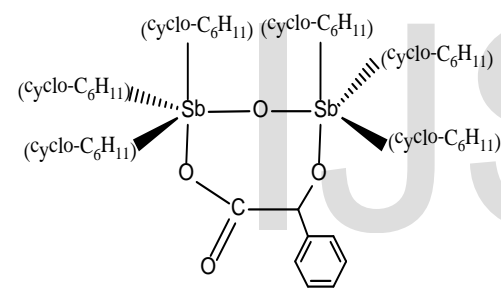


Fig 2 Showing structure of μ -Oxo Cyclometallate derivatives

5 TABLES

TABLE – 1 Preparation and Properties of μ -Oxy bis [tricyclohexyl antimony (v)] derivatives

Comp. No.	Complex	[Cyclo-C ₆ H ₁₁) ₃ SbCl ₂ O]	Ligand No. (g)	Molar Ratio/ Solvent	M.P. (°C)	Colour	Recrystallization Solvent
(1)	[(cyclo-C ₆ H ₁₁) ₃ Sb ₂ O(Cl)(OCOC ₆ H ₄ OH-o)]	0.829g	1, (0.245)	1:1 /THF	142	White	n-Hexane Benzene
(2)	[(cyclo-C ₆ H ₁₁) ₃ Sb ₂ O(OCOC ₆ H ₄ OH-o) ₂]	0.829g	1, (0.490)	1:2 /THF	165	off white	THF/n-Hexane
(3)	[(cyclo-C ₆ H ₁₁) ₃ Sb ₂ O(OCOC(OH)(C ₆ H ₅) ₂) ₂]	0.414g	4, (0.335)	1:2 /THF	115	White	THF Pet-ether (40-60°)
(4)	[(cyclo-C ₆ H ₁₁) ₃ Sb ₂ O(Cl)(OCOC(OH)(C ₆ H ₅) ₂) ₂]	0.829g	4, (0.335)	1:1/ THF	98	White	THF, n-Hexane
(5)	[(cyclo-C ₆ H ₁₁) ₃ Sb ₂ O(OCOC(OH)(C ₆ H ₄ CF ₃ -p) ₂]	0.415g	2, (0.327)	1:2/ THF	122	white	THF, n-Hexane
(6)	[(cyclo-C ₆ H ₁₁) ₃ Sb ₂ O(Cl)(OCOC(OH)(C ₆ H ₄ CF ₃ -p) ₂]	0.829g	2, (0.327)	1:1/ THF	119	White	THF, n-Hexane
(7)	[(cyclo-C ₆ H ₁₁) ₃ Sb ₂ O(OCOC(OH)(C ₆ H ₅) ₂)]**	0.829g	6, (0.259)	1:1 /THF	98	White	Benzene/ Pet. ether (40-60°C)
(8)	[(cyclo-C ₆ H ₁₁) ₃ Sb ₂ O(OCOC(OH)(C ₆ H ₅) ₂)]**	0.414g	6, (0.259)	1:2/ THF	98	White	Pet. Ether(40-60°C)
(9)	[(cyclo-C ₆ H ₁₁) ₃ Sb ₂ O(OCOC(OH)(C ₆ H ₄ OCH ₃ -p) ₂]	0.414g	4, (0.290)	1:2 /THF	135	off white	Acetone, n-Hexane
(10)	[(cyclo-C ₆ H ₁₁) ₃ Sb ₂ O(Cl)(OCOC(OH)(C ₆ H ₄ OCH ₃ -p) ₂]	0.414g	4, (0.145)	1:1/ THF	85	off white	Acetone/ n-hexane
(11)	[(cyclo-C ₆ H ₁₁) ₃ Sb ₂ O(OCOC(N ₃ H ₅) ₂)]*	0.829g	5, (0.462)	1:2/Acetone	127	white	Chloroform, n-Hexane

*OCOC(N₃H₅)₂ = 2-pyrazine carboxylate
** = cyclohexanones

TABLE – 2 Elemental Analysis of μ -oxy bis [tricyclohexyl antimony (v)] Derivatives

Comp. No.	Empirical Formula	Molecular weight	Found (Calcd.)%		
			C	H	N
(1)	C ₄₃ H ₇₁ ClO ₄ Sb ₂	930.99	55.50 (55.47)	7.59 (7.69)	-
(2)	C ₅₀ H ₇₆ O ₅ Sb ₂	1032.65	63.35 (63.38)	7.39 (7.42)	-
(3)	C ₆₄ H ₈₈ O ₅ Sb ₂	1212.90	63.35 (63.38)	7.38 (7.31)	-
(4)	C ₅₀ H ₇₇ ClO ₄ Sb ₂	1021.12	58.51 (58.81)	7.52 (7.60)	-
(5)	C ₅₄ H ₇₈ F ₆ O ₄ Sb ₂	1196.70	54.31 (54.20)	6.53 (6.57)	-
(6)	C ₄₈ H ₇₇ ClF ₃ O ₄ Sb ₂	1013.02	53.25 (53.35)	7.25 (7.16)	-
(7)	C ₄₄ H ₇₇ O ₄ Sb ₂	908.56	58.25 (58.17)	7.87 (7.99)	-
(8)	C ₄₄ H ₇₇ O ₄ Sb ₂	1120.76	57.88 (57.87)	7.60 (7.55)	-
(9)	C ₄₈ H ₇₈ ClO ₅ Sb ₂	975.05	55.35 (55.43)	7.70 (7.75)	-
(11)	C ₄₆ H ₇₂ N ₄ O ₅ Sb ₂	1004.61	54.98 (55.00)	7.18 (7.22)	5.57 (5.58)

TABLE – 3 Characteristic TR Absorption of μ -oxy-bis [tricyclohexyl antimony (V)] Derivatives in cm^{-1}

Comp. No.	$\nu(\text{OCO})$			$\nu(\text{OH})$	$\nu(\text{Sb-C})$	$\nu(\text{Sb-O-Sb})$
	ν_{asym}	ν_{sym}	$\Delta\nu$			
1	1635 (s)	1390 (vs)	245	3287 (m)	459 (m)	742 (s)
2	1637 (m)	1309 (s)	328	3280 (m)	459 (m)	745 (s)
3	1686 (s)	1303 (m)	383	3425 (w)	456 (s)	731 (s)
4	1682 (s)	1310 (w)	372	3431 (w)	455 (m)	735 (s)
5	1686 (s)	1321 (s)	365	3461 (w)	451 (m)	728 (vs)
6	1690 (s)	1325 (vs)	365	3463 (w)	456 (s)	736 (vs)
7	1684 (s)	(O)	-	-	458 (s)	731 (vs)
8	1684 (s)	1304	380	-	458 (s)	731 (vs)
9	1687 (s)	1315 (vs)	372	3449 (w)	454 (s)	735 (s)
10	1687 (s)	1301 (m)	386	3449 (s)	459 (s)	738 (vs)
11	1660 (vs)	1327 (vs)	333	-	456 (s)	738 (s)

Where, w = weak, m = medium, s = strong, vs = very strong, O = overlapped

TABLE – 4 ^1H NMR Spectral Data for μ -Oxybis [tricyclohexyl antimony (v)] Derivatives in $\delta(\text{ppm})$

Comp. No.	Cyclohexyl						Ligand				
	H1	H2e	H2a/H3a	H3e	H4e	H4a	H2'	H3'	H4'	OCH3	H α
(2)	3.75-3.81 (m)	2.80-2.88 (m)	1.71-1.75 (m)	1.52-1.55 (m)	1.57-1.61 (m)	1.16-1.24 (m)	7.48-7.77	6.51-6.60	7.02-7.08	-	-
(3)	3.80-3.85 (m)	2.91-2.87 (m)	1.73-1.78 (m)	1.52-1.56 (m)	1.18-1.20 (m)	1.19-1.25 (m)	7.25-7.48	7.14-7.17 (m)	-	-	-
(4)	3.77-3.80 (m)	2.82-2.89 (m)	1.71-1.77 (m)	1.54-1.58 (m)	1.60-1.65 (m)	1.16-1.21 (m)	7.23-7.74	7.10-7.12 (m)	-	-	-
(5)	3.79-3.83 (m)	2.82-2.90 (m)	1.72-1.76 (m)	1.53-1.58 (m)	1.61-1.64 (m)	1.16-1.19 (m)	7.40-7.78 (m)	-	-	-	5.37 (s)
(7) & (8)	3.64-3.69 (m)	2.70-2.81 (m)	1.70-1.76 (m)	1.55-1.62 (m)	1.64-1.68 (m)	1.13-1.20 (m)	7.34-7.59	7.13-7.18 (m)	-	-	5.28 (s)
(9)	3.81-3.85 (m)	2.89-2.95 (m)	1.73-1.79 (m)	1.51-1.57 (m)	1.60-1.63 (m)	1.14-1.19 (m)	7.45-7.82 (m)	-	-	3.80 (s)	5.34 (s)

Where m = multiplet, d = doublet, s = singlet

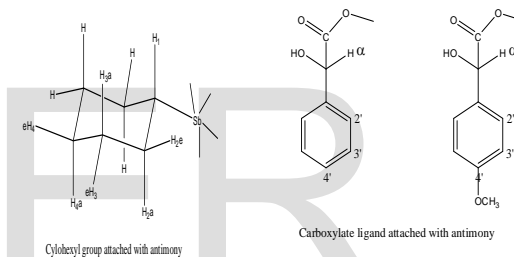
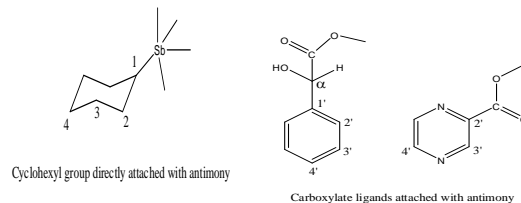


TABLE – 5 ^{13}C NMR Spectral Data for μ -oxybis [tricyclohexylantimony (V)] derivatives in $\delta(\text{ppm})$

Comp.	Cyclohexyl						Ligand				
No.	C1	C2	C3	C4	C α	> C=O	C1'	C2'	C3'	C4'	
(2)	55.43	30.01	26.14	26.00	-	173.09	141.52	130.54, 161.48 (OH)	116.98 & 118.56	134.83	
(3)	55.09	29.61	26.34	26.02	81.58	174.61	143.02	127.73	127.28	127.32	
(7) & (8)	55.31	29.63	28.23	25.90	73.68	174.13	139.71	128.26	126.58	127.46	
(6)	55.24	29.45	28.20	25.87	73.02	173.14	139.85	126.44	126.49	128.06	
(11)	56.58	28.05	25.03	25.55	-	165.01	145.61	143.98	144.53	197.66	

^{13}C NMR Spectra for compound (5) gave the signal at 862.9 ppm for -CF $_3$ group



CONCLUSION

Thus, from the IR and NMR (¹H, ¹⁹F and ¹³C) spectral studies aided by molecular weight and conductance measurements, it is evident that carboxylic acids behave as monodentate except in case of mandelic acid which forms cyclometallates. Thus in these newly synthesised carboxylates, the antimony is in pentacoordinated state imparting trigonal-bipyramidal (TBP) structure around the antimony atom, in which electronegative groups occupy apical positions and three cyclohexyl group are situated at the equatorial positions (Fig. 1). The preferred geometry of five coordinated group 15 element is TBP, which is fluxional, stereochemically non rigid, or pseudorotating arrangement rapidly interconverting to square pyramidal structure. This has been established in case of antimony [23]. The compounds are monomeric and non electrolyte in solution having pentacoordinate dispensation around antimony atom.

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