



SYNTHESIS AND CHARACTERISATION OF μ -OXY-BIS

[TRIARYLANTIMONY (V)] DICARBOXYLATES AND HALO-CARBOXYLATES

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ABSTRACT

Several hitherto to unknown μ -oxybis[triarylantimony(V)]dicarboxylates & μ -oxy-bis[triarylantimony]halo-carboxylates of the general formula $R_3Sb(L)-O-Sb(L)R_3$ & $R_3Sb(Cl)-O-Sb(L)R_3$ respectively have been synthesised by the metathetical reactions of μ -oxybis(triarylantimony)dichloride reactions and silver salts of corresponding carboxylic acids in 1:2 & 1:1 ratio [where L= 2-pyrazine carboxylic acid, *p*-methoxy medallic acid, salicylic acid, benzillic acid,). The newly synthesised antimony derivatives have been characterised on the basis of melting point, elemental analysis, IR 1H & ^{13}C NMR spectra. The molecular weight & conductivity data indicate the monomeric & non electrolytic behaviour in solution, these have been found to exhibit moderate to significant antimicrobial activity.

Keywords: μ -oxybis(triphenylantimony)dicarboxylates, μ -oxybis(triphenylantimony)chloro-carboxylates, IR, (1H & ^{13}C)NMR spectra antimicrobial activity.

INTRODUCTION

As compared to exhaustive studies carried out on organometallic derivatives of antimony(III) and antimony(V) of the general formula R_nSbX_{3-n} and R_nSbX_{5-n} respectively, very few studies have been devoted to the synthesis and characterization of compounds of the type $(R_3Sb-O-SbR_3)X_2$. Such compounds are important not only from synthetic point of view but they also offer structural novelty as well. Moreover, μ -oxy compounds of antimony having Sb-O-Sb skeleton are parallel to sodium stibogluconate compound which are still practised for the treatment of Leishmaniasis disease. Leishmaniasis is caused by different species belonging to the genus leishmania, a protozoa transmitted by the bite of 2-3 mm long insect vector, the phlebotomine sandfly^[1]. Leishmaniasis has an overwhelming impact on health especially in tropical and subtropical countries. It may be noted that metal containing compounds may offer some advantage over purely organic compounds in drug therapy for example, coordination of an organic molecule to a metal may lead to a slow release mechanism for delivery of the organic molecule as well as may alter the normal metabolic pathway. In other words the metal complex may function as a pro-drug. One obvious drawback in metal therapy is toxicity associated with the metal. Nevertheless, the importance of metal based drug in therapy is undisputed. It is well known that significant changes in bonding and biological activity are observed on changing the nature and content of organic groups bonded to antimony nature of anion attached, oxidation state of metal ion and substituent on organic group bond to antimony. It is worth mentioning that the presence of OH and fluoro group enhances solubility in water as well in lipid and thus enhances the biological activity. Similar properties, though to a lesser extent, are shown by chlorosubstituted ligands.

Lucknow workers have earlier reported the formation of some representative binuclear oxo-bridged antimony (V) derivatives of the type $(Ph_3SbX)_2O$ (X= amide, succinimide, 2-methylimidazole acetoxime, trichloroacetic acid etc.^[2]. Raj et al. reported the synthesis and characterisation of pentacoordinated tris(pentafluorophenyl)antimony (V) derivatives and corresponding $[(C_6F_5)_3SbX]_2O$ ^[3]. On the basis of IR spectra, the mode of bonding of the pseudohalide groups to antimony was established. In addition to this, the synthesis and characterisation of Group 15 oxo-bridged organometal selenocyanates have also been reported by the same authors^[4]. Molecular and crystal structure of an oxo-bridged chlorophenyl antimony(V) benzene solvate^[5] and of an oxo-bis $[BrPh_3Sb(V)]$ compound has been studied^[6].

Raj et al. has reported synthesis and reactions of sterically hindered α -naphthyl and cyclohexyl derivatives having Sb-O-Sb linkage, hydrolysis of R_3SbCl_2 (R= α -C₁₀H₇, cyclo-C₆H₁₁) afforded $(R_3SbCl)_2O$ type compounds^[7]. In addition to this, in a latter publication they reported several new μ -oxy-bis[tris(α -naphthyl) antimony(V)] derivatives of general formula $(R_3SbL)_3O$ obtained by the interaction of $(R_3SbCl)_2O$ with the appropriate metal salt of the ligand^[8]. It was observed that $(R_3SbN_3)_2O$ could also be obtained by the interaction of R_3SbCl_2 with NaN_3 (1:2 molar ratio) in ether/water solvent mixture. The newly synthesized compounds have been assigned a trigonal bipyramidal geometry around the antimony atom with a Sb-O-Sb linkage on the basis of spectral data. Roughly, it may be concluded that all the oxo-bridged compounds contain linear Sb-O-Sb system but the residual electron density about the bridging oxygen and along the halo and pseudohalo moiety suggest disorder and the actual bridge angle varies between 130-

180°.

Motivated by the aforementioned findings the author directed systematic efforts towards the synthesis of new oxo-bridged organoantimony(V) derivatives of carboxylic acids, the analogs of sodium stibogluconate.

In continuation to our work on the synthetic reactivity and biological aspect of organoantimony derivatives, we now report metathetical reactions of μ -Oxy-bis [tri(*p*-chlorophenyl) antimony] halide with silver salt (MY) (M=Na, Ag, Y=RCOO⁻) to give μ -Oxy-bis[tri(*p*-chlorophenyl) antimony(V)] derivatives.

The newly synthesized compounds have been characterised on the basis of IR, ¹H, and ¹³C (NMR) spectra.

EXPERIMENTAL

1:2 Molar Ratio of Reaction of [(*p*-ClC₆H₄)₃SbCl]₂O with Silver Salt of Benzilic Acid:

A solution of [(*p*-ClC₆H₄)₃SbCl]₂O (0.99gm 1mmol) and silver salt of benzilic acid (0.672gm 2mmol) in THF (20mL) was stirred at room temp. for 24h. On filtration of heterogenous solution contain precipitate of silver chloride a clear solution was obtain which was concentrated on *vacuum* (2-3ml). After the addition of (n-Hexane) 3ml the solution was allowed to stand overnight at 0°C affording a white crystalline solid. The compound was crystallised from n-Hexane to afford μ -oxybis[tri(*p*-chlorophenyl)antimony(V)]benzylate.

M.P.:- 90°C

Yield:- 67%

1:2 Molar Ratio Reaction of [(*p*-ClC₆H₄)₃SbCl]₂O with Silver Salt of 2,4dichlorophenoxy Acetic Acid:

A solution of [(*p*-ClC₆H₄)₃SbCl]₂O (0.99gm 1mmol) and silver salt of 2,4 dichlorophenoxy acetic acid (0.656gm 2mmol) in THF was stirred at room temp. for 24hrs. On filtration of heterogenous solution containing precipitate of silver chloride a clear solution was obtained which was concentrated on vacuum (2-3ml). After the addition of hexane yielded white crystalline solid. The compound was crystallised from chloroform to afford μ -Oxy-bis[tri(*p*-chlorophenyl)antimony(V)] 2,4dichlorophenoxy acetate.

M.P.:- 120°C

Yield :- 45%

1:2 Molar Ratio Reaction of [(*p*-ClC₆H₄)₃SbCl]₂O with Silver Salt of 2-Pyrazine Carboxylic Acid:

In an oxygen and moisture free atmosphere, a solution of [(*p*-ClC₆H₄)₃SbCl]₂O (0.99g, 1mmol) with silver salt of 2-Pyrazine carboxylic acid (0.464g, 2mmol) in dry acetone (20mL) 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* (3ml) followed by addition of petroleum ether (60-80°C) yielded white crystalline solid. The compound was

crystallised from chloroform/n-hexane mixture (1:2) to afford μ -oxy-bis[tri(*p*-chlorophenyl)antimony(V)(2-pyrazine carboxylate)]

M.P. :- 140°C

Yield :- 56%

1:2 Molar Ratio Reaction of [(*p*-ClC₆H₄)₃SbCl]₂O with Silver Salt of Salicylic Acid:

A solution of [(*p*-ClC₆H₄)₃SbCl]₂O (0.99 gm 1mmol) and silver salt of salicylic acid (0.488g 2.0mmol) in THF (20mL) was stirred at room temperature for 24h. On filtration of heterogenous solution containing precipitate of silver chloride a clear solution was obtained which was concentrated in *vacuo* (2-3mL) the solution was allowed to stand overnight at 0°C affording a white crystalline solid which was recrystallised from n-Hexane. The compound was characterised as μ -oxy-bis[tri(*p*-chlorophenyl)antimony(V) salicylate].

M.P.:- 120°C

Yield :- 67%

In the same manner 1:1 molar ratio reaction of [(*p*-ClC₆H₄)₃SbCl]₂O (0.99g 1mmol) with silver salt of salicylic acid (0.244g 1mmol) in THF (15mL) afforded off white crystalline compound characterised as tri(*p*-chlorophenyl)antimony(V) salicylate μ -oxy-bis-tri(*p*-chlorophenyl)antimony(V) chloride.

M.P.:- 180°C

Yield :- 76%

1:2 Molar Ratio of Reaction of (Ph₃SbCl)₂O with Silver Salt of Benzilic Acid:

A solution of (Ph₃SbCl)₂O (0.388gm, 0.5mmol) and silver salt of benzilic acid (0.335gm, 1mmol) in THF (20mL) was stirred at room temp. for 24h on filtration of heterogenous solution contain in precipitate of silver chloride a clear solution was obtain which was concentrated on *vacuo* (2-3ml). After the addition of (n-Hexane) 3ml the solution was allowed to stand overnight at 0°C affording a white crystalline solid. The compound was crystallised from n Hexane to afford μ -oxy-bis(triphenyl antimony(V) (benzylate)].

M.P.:- 125°C

Yield :- 68%

1:1 Molar Ratio Reaction of (Ph₃SbCl)₂O with Silver Salt of DL Aspartic Acid:

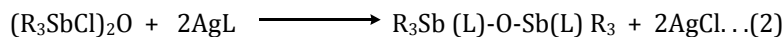
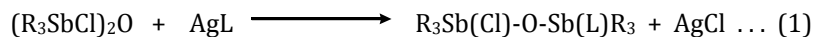
A heterogenous solution of (Ph₃SbCl)₂O (0.77g, 1mmol) and silver salt of DL aspartic acid (0.120 1mmol) on filtration of heterogenous solution contain in precipitate of silver chloride a clear solution was obtain. Which was concentrated on *vacuo* (2-3ml). After the addition of n-Hexane the solution was allowed to stand overnight 0°C affording a white crystalline solid. The compound was characterised as μ -oxy-bis(triphenylantimony) aspartate.

M.P. :- 168°C

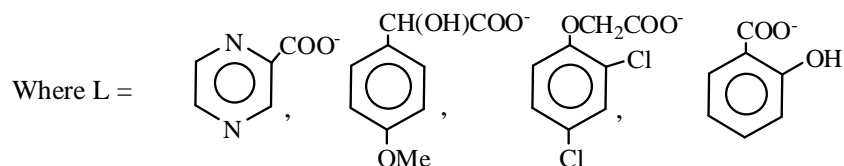
Yield :- 59%

RESULTS AND DISCUSSION

The interaction of μ -oxy-bis(triphenylantimony chloride), and μ -oxy-bis [tri(*p*-chlorophenyl) antimony] chloride with the silver salt of corresponding carboxylic acid in 1:1 and 1:2 molar ratio afford mono- and di-substituted μ -oxy-bis [tri(*p*-chlorophenyl)antimony] and μ -oxy-bis(triphenylantimony) derivatives respectively.



Where R = C₆H₅, *p*-ClC₆H₄



The newly synthesised μ -oxy-derivatives of triphenylantimony(V) are white crystalline solids with sharp melting points and are moderately 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 summarised in Table (2). All the carboxylates isolated were identified characterised on the basis of elemental analysis IR and NMR (¹H, ¹³C) spectroscopy the spectral data obtained are given in Tables (3).

Comp. (No.)	complex	$[(p\text{-ClC}_6\text{H}_4)_3\text{SbCl}]_2\text{O}$	Ligand (g)	Molar ratio/Solvent (mL)	M.P. (°C)	Yield (g) (%)	Colour	Recrystallisation Solvent
1		0.99		1:2 THF (20mL)	90	67	white	n-Hexane
2		0.99		1:2 THF (30mL)	140	56	white	Chloroform/ n-Hexane
3		0.99		1:2 THF (20mL)	120	45	white	n-Hexane
4		0.99		1:2 THF (20mL)	120	67	white	n-Hexane
5		0.99		1:1 THF (20mL)	180	76	white	n-Hexane
6		0.77		1:1 THF (20mL)	168	59	white	n-Hexane
7		0.388		1:2 THF (20mL)	125	68	white	THF/ n-Hexane
8		0.388		1:2 THF (20mL)	134	61	white	n-Hexane

Table 1: Preparation and properties of μ -Oxy-bis[triarylantimony(V)] Derivatives

Comp. (No.)	Empirical Formula	Molecular Weight	Found (Calcd.) % - C	Found (Calcd.) % - H	Found (Calcd.) % - N
1	C ₆₄ H ₄₆ Cl ₆ O ₇ Sb ₂	1383.28	55.78 (55.57)	3.98 (3.35)	-
2	C ₄₆ H ₃₀ Cl ₆ N ₄ O ₅ Sb ₂	1174.99	47.56 (47.02)	2.12 (2.57)	4.90 (4.77)
3	C ₅₂ H ₃₄ Cl ₁₀ O ₇ Sb ₂	1368.87	44.98 (45.63)	2.09 (2.50)	-
4	C ₅₀ H ₃₄ Cl ₆ O ₇ Sb ₂	1203.04	49.45 (49.92)	3.04 (2.85)	-
5	C ₄₃ H ₂₉ Cl ₇ O ₄ Sb ₂	1101.38	46.90 (45.89)	2.90 (2.65)	-
6	C ₄₁ H ₃₈ NO ₅ Sb ₂	868.26	56.09 (56.72)	4.67 (4.41)	1.89 (1.61)
7	C ₆₄ H ₅₂ O ₇ Sb ₂	1176.61	65.09 (65.33)	4.98 (4.45)	-
8	C ₅₄ H ₄₈ O ₉ Sb ₂	1084.47	59.03 (59.81)	4.56 (4.46)	-

Table 2: Elemental Analysis of μ -Oxy-bis[triarylantimony(V)] Derivatives

Comp. (No.)	V(OCO) V _{asym}	V(OCO) V _{sym}	ΔV	V(OH)	V(Sb-C) (y-mode)	V (Sb-O-Sb)
(1)	1649(m)	1404(m)	320	3467(m)	489(w)	763(vs)
(2)	1675(w)	1382(m)	322	-	488(w)	767(s)
(3)	1684(s)	1384(w)	380	-	488.8(w)	767(s)
(4)	1620(m)	1326(s)	325	3280(m)	477(m)	769(vs)
(5)	1620(m)	1320(s)	324	3289(m)	451(s)	778(vs)
(6)	1630(s)	1321(m)	367	-	459(m)	735(vs)
(7)	1686(s)	1303(m)	345	3425(w)	456(s)	731(s)
(8)	1698(s)	1314	376	3450(w)	454(s)	730(s)

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

Table 3: Characteristic IR Absorption of μ -Oxy-bis[triarylantimony(V)] Derivatives in cm⁻¹

Infrared spectra:

The infrared spectra of these [μ -oxy-bis(triaryl) antimony(V)] derivatives were recorded in the range 4000-400cm⁻¹. The characteristic absorption frequencies are listed in Table (3).

Absorption associated with various internal modes of vibrations of μ -oxy-bis[triarylantimony(V)] derivatives have been identified and indicate the nature of bonding. Band due to antimony-oxygen-antimony (Sb-O-Sb) bond is at almost similar position, in the range 730-778 cm⁻¹ as strong to very strong band and come in the range of earlier published data^[9]. The Sb-C stretching frequency corresponding to mass sensitive Y-mode recorded in the range 451-

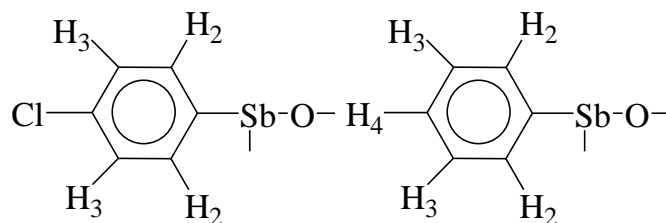
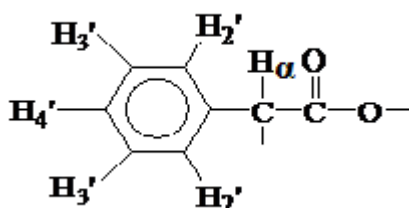
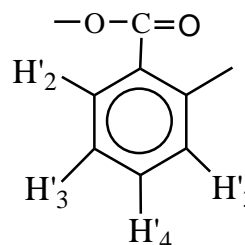
489 cm^{-1} and is in good agreement to those reported earlier.^[9]

The position of asymmetric and symmetric OCO stretching modes, and separation ($\Delta\nu$) between them provides a method of assessing carboxylate coordination modes. Deacon and co-workers ^[10] point out that this correlation is limited to recognition of complexes with unidentate carboxylates where $\Delta\nu_{\text{unidentate}} > \nu_{\text{ionic}}$ and complexes with chelating or bridging carboxylates where $\Delta\nu_{\text{bridging or chelating}}$ is often $< \Delta\nu_{\text{ionic}}$ ($\Delta\nu_{\text{ionic}}$ values are in the range 233-164 cm^{-1}). In case of newly synthesised the μ -oxy-bis tri(*p*-chlorophenyl)antimony(V) carboxylates, asymmetric and symmetric stretching vibration of diagnostic value were identified in comparison with other reported organoantimony carboxylates^[11]. Asymmetric $\nu(\text{OCO})$ stretching modes were assigned in the range 1698-1620 cm^{-1} as medium to very strong band. While symmetric $\nu(\text{OCO})$ stretching vibration appear in the range 1384-1303 cm^{-1} . The extent of separation between these two bands come in the range 327-286 cm^{-1} between the two modes of carboxylate derivatives and suggest the presence of monodentate ester type carboxylate groups imparting a penta-coordinate environment around the antimony atom. The non-conducting the monomeric nature and the absence of bands at 1556, 1413 and 650 cm^{-1} due to carboxylate ion^[12] in the IR spectra further rule out the possibility of an ionic structure. The $\nu(\text{OH})$ for compounds (1,4,5,7,8) was observed in the range 3467-3280 cm^{-1} as a weak or medium band.

¹H NMR spectra:

The proton magnetic resonance spectra of representative derivatives of μ -oxy-bis[tri(*p*-chlorophenyl)antimony(ν)] were recorded on Bruker DRX-300(300 MHz FT NMR) in solvent (DMSO + CDCl_3), with chemical shifts being reported as δ (ppm) taking tetramethylsilane as reference. The peak for protons of dimethyl sulfoxide appeared at 82.3ppm. The ¹H NMR data of the title compounds are listed in Table (4).

Comp. (No.)	Phenyl ring H ₂	Phenyl ring H ₃	Phenyl ring H ₄	Ligand H ₂ '	Ligand H ₃ '	Ligand H ₄ '	OCH ₃	H α
1	7.21-7.34(m)	7.28-7.40(m)	-	7.24-7.47(m)	7.16-7.19(m)	-	-	-
2	7.19-7.23(m)	7.23-7.37(m)	-	9.15(s)	8.78(d)	8.89(d)	-	-
3	7.25-7.30(m)	7.25-7.39(m)	-	7.40-7.70(m)	6.50-6.65(m)	7.10-7.15(m)	-	-
4	7.51-7.60(m)	7.24-7.42(m)	-	7.24-7.46(m)	7.14-7.18(m)	-	-	-
5	7.67-7.70(m)	7.44-7.78(m)	-	7.47-7.84(m)	-	3.87(s)	5.35(s)	-

Table 4: ¹HNMR Spectral Data for μ -Oxy-bis[triarylantimony(V)] Derivatives in δ in (ppm)**(Phenyl ring)****(α -Hydroxy acids)****(Salicylate)**

All the compounds show multiplet in the range δ (6.50-9.15) ppm which can be attributed to the presence of phenyl group protons. Compounds (8) showed a singlet δ (3.87)ppm which is due to the methyl proton of OCH_3 .

¹³C NMR spectra:

The ¹³C NMR spectra of representative compounds were obtained on 300MHz FT NMR instrument (Bruker DRX-300) at \sim 75MHz using a mixture of CDCl_3 and DMSO as solvent and reference. The signals for CDCl_3 and DMSO appear at \sim 77.0 ppm and \sim 40.0ppm as triplet and septet, respectively. The chemical shift values for different carbon centres in δ (ppm) are listed in Table (5).

The carbon centre of carboxylic group was found deshielded and shifted to lower field in each compound when compared with that of free acid. This trend indicated the participation of carboxylate group in coordination to antimony^[13]. The α -carbon in case of compound (1,7) appeared in the range δ (80-81.56) ppm and was also deshielded because of the coordination of carboxylate group to antimony. The 2-pyrazine carboxylate and DL-aspartate (2,6) give the distinct signals for magnetically non-equivalent carbons. The two carbonyl groups of DL-aspartate derivative (6) appeared at different δ values.

Thus on the basis of IR and NMR spectra, molecular weight and non-ionic nature of the compounds the newly synthesized μ -oxy-bis(triarylantimony) carboxylate may be assigned a trigonal bipyramidal structure. Carboxylate group being more electronegative would occupy apical positions (Fig. 1)

Compound (No.)	C1	C2	C3	C4	C α	>C=O	C1'	C2'	C3'	C4'
1	134.2 3	134.2 9	128.7 8	137.0 7	80.0 0	173.5 9	142.0 5	126.72	126.24	126.3 1
2	134.1 5	134.2 3	128.4 5	137.6 6	-	165.5 4	144.5 3	143.73	144.26	146.1 3
3	135.1 5	133.8 1	128.5 0	130.5 2	-	172.0 9	141.2 5	130.28 161.15(OH)	116.90 &117.5 0	134.2 0
4	135.2 6	135.7 6	129.3 0	131.6 9	-	172.3 2 174.2 4	50.26	36.22	-	-
5	135.2 4	135.3 0	129.8 0	132.1 0	81.5 6	174.6 1	143.0 2	127.72	127.28	127.3 2

Table 5: ^{13}C NMR Spectral Data for μ -Oxy-bis[triarylantimony(V)] Derivatives in δ in (ppm)

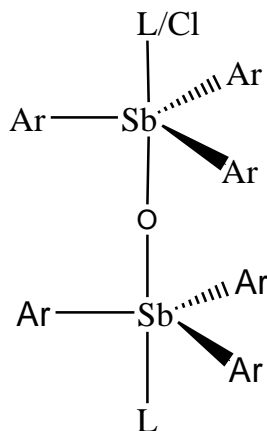
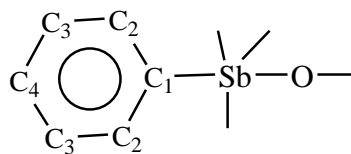


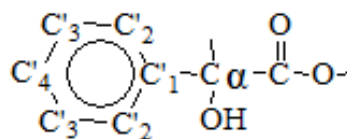
Figure 1

where, Ar = *p*-ClC₆H₄, C₆H₅

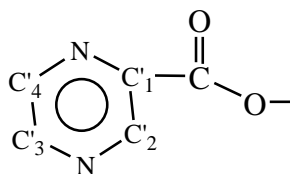
L = OHC₆H₄COO, Ph₂(OH)COO, R'CH(OH)COO
(R' = *p*-OCH₃C₆H₄) and 2-Pyrazine carboxylate;



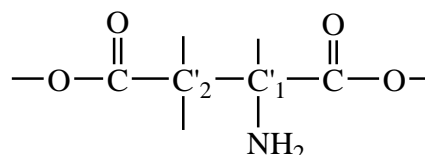
(Phenyl Ring)



(α Hydroxy Acids)



(2-Pyrazine Carboxylate)



(Aspartate)

METHOD

Antifungal activity:

The antifungal activity was determined by supplementation of the test compounds in media. The test compounds were added to the medium just before pouring, keeping constant 1ml ethanol / 100ml medium or the test compounds were added in the wells cut into the agar plates. A control was maintained with equal amount of solvent which has no effect on fungal growth. After the medium was solidified agar plugs of 5mm diameter from actively growing plates of the test fungi were inoculated in the centre of petridish with the help of inoculating needle. The plates were incubated for 10 days at 30°C. Visual observation for fungal growth was taken daily.

Antibacterial activity:

Antibacterial activity of the compounds was carried out using disc diffusion method against the bacterial strains. In this method filter paper (Whatman No. 1) discs of 5 mm diameter were impregnated with the test compounds. The compounds were dissolved in an appropriate organic solvent (ethanol) of analytical grade and the concentration of the compounds was made to 10 μ g/ml. The dried discs containing the compounds were placed on nutrient agar plates spread with the test organism and incubated at 25°C for 72 hrs. Zone of inhibition was measured and categorized as follows, the compounds showing less than 5 mm inhibition zone were considered to be inactive (+), 5-10 mm are slightly active (++) , 10-15mm moderately active (+++) and >15mm highly active (++++). A control was maintained where the solvent in which the compounds are prepared is used.

Antifungal Activity of μ -oxo-briged Antimony Derivative:

Antifungal activity of μ -oxo-briged antimony derivative was determined against two pathogenic fungal strains viz. *Candida albicans* and *Aspergillus niger*.

Antifungal activity of the compounds was carried out on agar plates using the test compounds placed in wells cut into the agar or supplemented into the growth medium in the required concentration. No fungal growth could be observed in 250 and 500 μ g/ml concentrations of test compounds even after 7 days of incubation. Hence lower concentrations (10 μ g/ml) of these compounds were used for their evaluation. Zone of inhibition was measured and observations were taken for up to 10 days (Table 7). The compounds showing less than 5 mm inhibition zone were considered to be ineffective (+), moderate(++), moderately good (+++), and good (++++). Antifungal activity of compounds No. (5 & 6) were found to be best amongst the tested compounds against *Candida albicans*. Compounds No. (2, 3 & 4) showed moderate good activity against the *Aspergillus niger*.

Antibacterial activity of μ -oxo-briged Antimony Derivative:

All the μ -oxo-briged Antimony compounds were assayed for antibacterial activity against two human pathogenic strains viz. *S. Aureus* and *K. Pneumoniae*. The result obtained are tabulated in table (8).

The highest activity against both strains was shown by compound no. (3) among all the μ -oxo-briged antimony derivatives. In general most of the μ -oxobis[triarylantimony (V)]derivatives were found to exhibit moderate to high activity.

CONCLUSION

μ -oxybis(triarylantimony)dicarboxylates and halocarboxyaltes having Sb-O-Sb linkage are stable and non-hygroscopic complexes. they are effective antibacterial and antifungal compounds and expected to display antileishmaniasis activity.

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