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Synthetic, Spectroscopic and Biological Studies on Some μ-Oxy-*bis*[triphenylantimony(V)]carboxylates and Cyclic Organoantimonates

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A series of *hitherto* unreported μ-oxy-*bis*[triphenylantimony(V)]dicarboxylates and μ-oxy-*bis*[triphenylantimony(V)] chlorocarboxylates of general formula $Ph_3Sb(L)$-O-$Sb(L)Ph_3$ and $Ph_3Sb(Cl)$-O-$Sb(L)Ph_3$, respectively have been synthesized by the metathetical reaction of μ-oxy*bis*-[triphenylantimony(V)]dichloride and silver salts of corresponding carboxylic acids in 1:2 and 1:1 molar ratio [where L = thiosalicyclic acid, *p*-nitrobenzoic acid, *p*-aminobenzoic acid, *p*-fluorobenzoic acid, *o*-chlorobenzoic acid]. The newly isolated antimony carboxylates have been identified on the basis of melting points, elemental analysis, FT-IR, ^{1}H NMR,^{13}C NMR, ^{19}F NMR. The molecular weight and conductivity data indicate the monomeric and non-electrolytic behaviour in solution. Compounds have been evaluated for their antifungal and antibacterial activity.

Keywords: μ-Oxy-*bis*[triphenylantimony(V)]dicarboxylates and mixed carboxylates, Thiosalicyclic acid, Antimicrobial activity.

INTRODUCTION

Compared to a large number of organoantimony(V) derivatives reported in the preceding past, oxo-bridged $[R_3SbX]_2O$ (R = alkyl or aryl X = halogen, pseudohalogens, chlorates, nitrates, *etc.*) compounds have been studied to a limit extent [1-4]. Although the synthesis of such compounds was reported earlier [5-7]. Goel *et al.* [8] were the first to do a systematic study in this direction and synthesized a number of compounds of the type $(Ph_3SbX)_2O$ (X = Cl, Br, NCS, N_3, NCO, NO_3 and ClO_4). The work was extended further to cover oxyanion derivatives as well of the type $(R_3SbX)_2O$ (X = SO_4, SeO_4 and C_2O_4) [8-10]. The structural features of this noble class of compounds were determined by a crystallographic study as well as by vibrational spectral study [11]. The infrared spectral data were interpreted in favour of non-ionic pentacoordinated structure possibly involving bridging anionic groups and planer triphenylantimony moieties, but when X is replaced by highly electronegative group *viz.* perchlorate and nitrate, the antimony is no longer pentavalent but ions are present instead [12].

Pandey & Srivastava [13] 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.*. Raj *et al.* [14] reported the synthesis and characterization of pentacoordinated *tris*-(pentafluorophenyl)antimony(V) derivatives and corresponding $[(C_6F_5)_3SbX]_2O$. On the basis of IR spectra, the mode of bonding of pseudohalide group(s) to antimony was established. In addition to this, the synthesis and characterization of group 15, oxo-bridged organometalselenocyanates have also been reported by the same authors [15]. Molecular and crystal structure of an oxo-bridged chlorophenylantimony(V) benzene solvate [16] and of an oxo-*bis*$[BrPh_3Sb(V)]$ compound has been studied [17].

Oxygen bridged hexa(organo)di-antimony compounds with the help of NNR spectra has been investigated by Gibbons *et al.* [18]. Crystal structure is reported for *o*- and *p*-tolyl isomers of $[(tolyl)_3SbBr]_2O$. Later on, Gibbons & Sowerby [19] reported reactions of $[Ph_3SbBr]_2O$ and $[Me_3SbCl]_2O$ with a number of carboxylates. This lead to halogen substitution and formation of either $[SbR_3(O_2CR')]_2O$ or $SbR_3(O_2CR')_2$. It was observed from the X-ray structure of two products, $[Ph_3Sb(O_2CCF_3)]_2O$ and $[Me_3Sb(O_2CCH_3)]_2O$ where carboxylates are unidentate.

Raj *et al.* [20] reported the synthesis and reactions of sterically hindered α-naphthyl and cyclohexyl derivatives having Sb-O-Sb linkage, hydrolysis of R_3SbCl_2 (R = α-$C_{10}H_7$, cyclo-C_6H_{11}) afforded $(R_3SbCl)_2O$ type compounds. 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

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 derivatives have been assigned a trigonal bipyramidal geometry around the antimony atom with a Sb-O-Sb linkage on the basis of spectral analysis. 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 130-180º.

In the present investigation, some novel oxo-bridged carboxylates having Sb-O-Sb linkage have been synthesized and characterized. Most of the synthesized compounds have been exclusively tested for their antifungal and antibacterial activity. New μ-oxy-*bis*[triphenylantimony(V)] derivatives of general formula $(R_3SbL)_2O$ and $R_3Sb(Cl)$-O-$Sb(L)R_3$ obtained by the interaction of $(R_3SbCl)_2O$ with the appropriate metal salt of the ligand. The newly synthesized compounds have been assigned a trigonal bipyramidal geometry around antimony atom with a Sb-O-Sb linkage on the basis of spectral data.

EXPERIMENTAL

All the chemicals used were of analytical grade. The complex, μ-oxy*bis*[(triphenylantimony(V)] chloride was synthesized by the reported method [21]. The molar conductance values of 10^{-3} M solutions were determined at 25 ºC with a Phillips conductive assembly PR-9500. Molecular weights were determined cryoscopically in benzene using a Beckmann thermometer (accuracy ± 0.01 ºC).

The stringent precautions were taken to avoid moisture. Solvents were dried and distilled before use.

Reaction of $(Ph_3SbCl)_2O$ with silver salt of thiosalicylic acid: A solution of $(Ph_3SbCl)_2O$ (0.396 g, 0.5 mmol) and silver salt of thiosalicylic acid (0.131 g, 0.5 mmol) in a molar ratio of 1:1 in THF (20 mL) was stirred at room temperature for 24 h. On filtration of heterogeneous solution containing precipitate of AgCl, 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 ºC affording a white crystalline solid which was recrystallized from a mixture of THF and *n*-hexane (1:3). The compound was characterized as μ-oxy-*bis*(triphenyl antimony)chlorothiosalicylate (**1a**).

In the same manner, 1:2 molar ratio reaction of $(Ph_3SbCl)_2O$ (0.396, 0.5 mmol g) with silver salt of thiosalicylic acid (0.262 g, 0.5 mmol) in THF (15 mL) afforded a white crystalline μ-oxy-*bis*[(triphenyl antimony thiosalicylate)] (**1b**).

Reaction of $(Ph_3SbCl)_2O$ with silver salt of *p*-nitro benzoic acid: A solution of $(Ph_3SbCl)_2O$ (0.396 g, 0.5 mmol) and silver salt of *p*-nitrobenzoic acid (0.272 g, 0.5 mmol) in a molar ratio of 1:2 in THF (20 mL) was stirred at room temperature for 24 h. On filtration of heterogeneous solution containing precipitate of silver chloride, a clear solution was obtained and concentrated *in vacuo* (2-3 mL). After the addition of *n*-hexane (3 mL), solution was allowed to stand overnight at 0 ºC affording a white crystalline solid, which was recrystallized from a mixture of THF and *n*-hexane (1:3). The compound was characterized as μ-oxy*bis*(triphenyl antimony *p*-nitro benzoate) (**1c**).

Reaction of $(Ph_3SbCl)_2O$ with silver salt of *p*-amino-benzoic acid: A solution of $(Ph_3SbCl)_2O$ (0.396 g, 0.5 mmol) and silver salt of *p*-aminobenzoic acid (0.122 g, 0.5 mmol) in a molar ratio of 1:1 in THF (20 mL) was stirred at room temperature for 24 h. On filtration of heterogeneous solution containing precipitate of silver chloride, a clear solution was obtained and concentrated *in vacuo* (2-3 mL). After the addition of *n*-hexane (3 mL) the solution was allowed to stand overnight at 0 ºC affording a white crystalline solid and recrystallized from a mixture of THF and *n*-hexane (1:3). The compound was characterized as μ-oxy*bis*(triphenyl antimony)chloro-*p*-amino benzoate (**1d**).

Reaction of $(Ph_3SbCl)_2O$ with silver salt of *p*-fluoro-benzoic acid: A solution of $(Ph_3SbCl)_2O$ (0.396 g, 0.5 mmol) and silver salt of *p*-aminobenzoic acid (0.123 g, 0.5 mmol) in in a molar ratio of 1.1 in THF (20 mL) was stirred at room temperature for 24 h. On filtration of heterogeneous solution containing precipitate of silver chloride, a clear solution was obtained and concentrated *in vacuo* (2-3 mL). After the addition of *n*-hexane (3 mL) the solution was allowed to stand overnight at 0 ºC affording a white crystalline solid and recrystallized from a mixture of THF and *n*-hexane (1:3). The compound was characterized as μ-oxy*bis*[(triphenyl antimony)chloro-*p*-fluorobenzoate (**1e**).

In the same manner, 1:2 molar ratio reaction of $(Ph_3SbCl)_2O$ (0.396 g, 0.5 mmol) with silver salt of *p*-fluorobenzoic acid (0.246 g, 0.5 mmol) in THF (15 mL) afforded a white crystalline μ-oxy*bis*(triphenyl antimony-*p*-fluorobenzoate) (**1f**).

Reaction of $(Ph_3SbCl)_2O$ with silver salt of *o*-chloro benzoic acid: A solution of $(Ph_3SbCl)_2O$ (0.396 g, 0.5 mmol) and silver salt of *o*-chlorobenzoic acid (0.263 g, 0.5 mmol) in a molar ratio of 1:2 in THF (20 mL) was stirred at room temperature for 24 h. On filtration of heterogeneous solution containing precipitate of silver chloride, a clear solution was obtained and concentrated *in vacuo* (2-3 mL). After the addition of *n*-hexane (3 mL), the solution was allowed to stand overnight at 0 ºC affording a white crystalline solid and recrystallized from a mixture of THF and *n*-hexane (1:3). The compound was characterized as μ-oxy*bis*(triphenyl antimony *o*-chlorobenzoate) (**1g**).

In the same manner 1:1 molar ratio reaction of $(Ph_3SbCl)_2O$ (0.396 g, 5 mmol) with silver salt of *o*-chlorobenzoic acid (0.131 g, 0.5 mmol) in THF (15 mL) afforded of white crystalline compounds characterized as μ-oxy-*bis*(triphenyl antimony)-chloro, *o*-chlorobenzoate) (**1h**).

Biological activity: Antifungal activity of the compounds was determined against two pathogenic fungal strains *viz. Candida albicans* and *Aspergillus niger*. 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 1 mL or DMSO/100 mL 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 5 mm diameter from actively growing plates of the test fungi were inoculated in the center of petridish with the help of inoculating needle. The plates were incubated for 10 days at 30 ºC. Visual observation for fungal growth was taken daily.

Antibacterial activity of the compounds was carried out using disc diffusion method [22,23] against the bacterial strains. In this method, filter paper discs (Whatman No. 1) of 5 mm diameter were impregnated with the test compounds. The compounds were dissolved in an appropriate organic solvent DMSO of analytical grade and the concentration of the compounds was made to 10 μg/mL. The dried discs containing the compounds were placed on nutrient agar plates spread with the test organism and incubated at 25 ºC for 72 h. A control was maintained where the solvent in which the compounds are prepared was used.

RESULTS AND DISCUSSION

The interaction of μ-oxy-*bis*(triphenyl antimony chloride) with the silver salt of carboxylic acid in 1:1 and 1:2 molar ratio afford mono and disubstituted μ-oxy-*bis*(triphenyl antimony) derivatives.

$$(R_3SbCl)_2O + AgL \longrightarrow R_3Sb(Cl)\text{-}O\text{-}Sb(L)R_3 + AgCl \quad (1)$$

$$(R_3SbCl)_2O + 2AgL \longrightarrow R_3Sb(L)\text{-}O\text{-}Sb(L)R_3 + 2AgCl \quad (2)$$

where R = C_6H_5; L = thiosalicylic acid, *p*-nitrobenzoic acid, *p*-aminobenzoic acid, *p*-fluorobenzoic acid, *o*-chlorobenzoic acid.

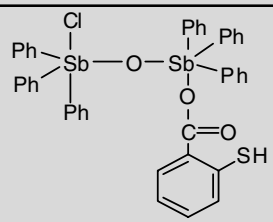
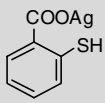
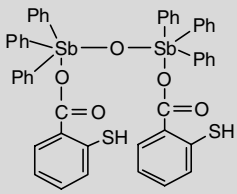
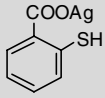
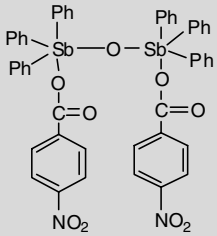
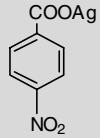
The physico-chemical data of the the newly synthesized μ-oxy derivatives of triphenyl-antimony(V) are given in Table-1.

Infrared spectra: The infrared spectra of the synthesized μ-oxy derivatives of triphenylantimony(V) were recorded in the range 4000-400 cm^{-1}. Band due to antimony-oxygen-antimony (Sb-O-Sb) bond is at similar position in 735-731 cm^{-1}as strong to very strong band. The Sb-C stretching correspond to mass sensitive Y mode was observed at 463-451 cm^{-1}.

The position of asymmetric and symmetric OCO stretching modes and separation (Δν) between them provides a method of assessing carboxylates coordination modes. Singhal *et al*. [4,14,15] reported that this correlation is limited to recognition of complexes with unidentate carboxylates where $\Delta\nu_{unidentate} > \Delta\nu_{ionic}$ and complexes with chelating or bridging carboxylates where $\Delta\nu_{briging\ or\ chelating}$ is often < $\Delta\nu_{ionic}$ ($\Delta\nu_{ionic}$ = 233-164 cm^{-1}). In case of newly synthesized μ-oxy*bis*-(triphenyl antimony)carboxylates, asymmetric and symmetric vibrations of diagnostic values were identified in comparison with other reported organoantimony carboxylates [1-4,20,24].

Asymmetric OCO stretching modes were assigned in the range 1652-1601 cm^{-1} as medium to very strong band. While symmetric OCO stretching vibrations appear in the range 1344-1255 cm^{-1}. The extent of separation between these two bands comes in the range 364-257 cm^{-1} between the two modes of carboxylates derivatives and suggests the presence of monodentate ester type carboxylates groups imparting a pentacoordinate environment around the antimony atom (Table-4). The non-conducting carboxylate ions [20] in the IR spectra, further rule out the possibility of an ionic structure.

TABLE-1
PHYSICO-CHEMICAL DATA OF SYNTHESIZED μ-OXY-*bis* [(TRIPHENYL ANTIMONY)] DERIVATIVES

Compd. No.	Complex	$(Ph_3SbCl)_2O$ (g)	Ligand (g)	Molar ratio/Solvent (mL)	m.p. (ºC)	Yield (%)	Colour	Recrystallization solvent
1a		0.396	 (0.131)	1:1/THF (15 mL)	98	50	White	Chloroform/ Acetonitrile
1b		0.396	 (0.262)	1:2/THF (15 mL)	90	55	White	Chloroform/ Acetonitrile
1c		0.396	 (0.272)	1:2/THF (15 mL)	170	56	White	Chloroform/ Acetonitrile

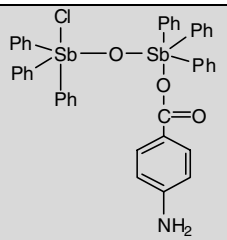
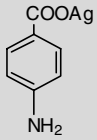
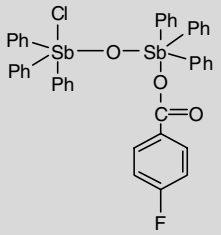
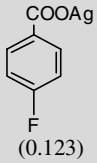
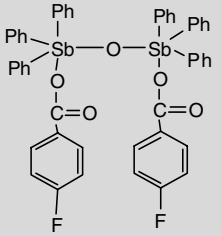
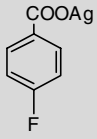
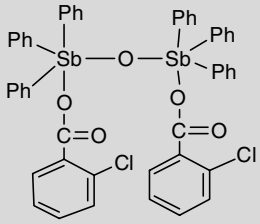
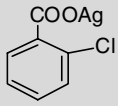
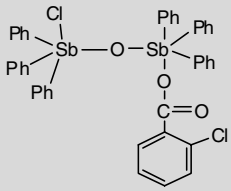
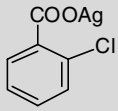
1d		0.396	 (0.122)	1:1/THF (20 mL)	198	64	White	*n*-Hexane
1e		0.396	 (0.123)	1:1/THF (15 mL)	178	56	White	*n*-Hexane
1f		0.396	 (0.246)	1:2/THF (20 mL)	162	62	White	*n*-Hexane
1g		0.396	 (0.263)	1:2/THF (15 mL)	148	60	White	*n*-Hexane
1h		0.396	 (0.131)	1:1/THF (20 mL)	126	66	White	*n*-Hexane

TABLE-2
CHARACTERISTICS IR ABSORPTION BANDS (cm^{-1}) OF μ-OXY-*BIS*[TRIPHENYLANTIMONY(V)] DERIVATIVES

Compd. No.	ν(OCO)			ν(Sb-C)	ν(Sb-O-Sb)
	ν_{asy}	ν_{sym}	Δν		
1a	1636(s)	1305(vs)	331	456(s)	731(s)
1b	1635(s)	1297(m)	338	456(s)	731(s)
1c	1646(s)	1309(w)	327	459(m)	734(s)
1d	1602(s)	1291(s)	311	459(s)	734(vs)
1e	1601(s)	1344	257	463(s)	735(vs)
1f	1620(s)	1288(m)	332	456(s)	734(s)
1g	1640(s)	1333(s)	307	452(s)	732
1h	1624(s)	1344(vs)	280	456(s)	733(vs)

^{1}H NMR spectra: The proton NMR spectra of representative derivatives of μ-oxy derivatives of triphenylantimony(V) were recorded on Bruker DRX-300(300 MHZ FT NMR). The peak for protons of dimethyl sulfoxide appeared at 2.50 ppm. All the compounds show multiplet in the range of δ 6.5-8.5 ppm, which can be attributed to the presence of phenyl group protons (Table-2).

^{13}C NMR spectra: The ^{13}C NMR of the synthesized compounds was obtained on 300 MHz FT NMR instrument (Bruker DRX-300) at ~ 75 MHz using DMSO as solvent. The signals for DMSO appeared at ~ 40.0 ppm as septet (Table-3). 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 tend indicated the participation of carboxylic group in coordination to antimony. The carboxylic acids give the distinct signals for magnetically non-equivalent carbons.

^{19}F NMR spectra: Presence of fluorine atom in compound **1e** was confirmed by the observed fluorine signals (δ -107.24 to -107.61, as a multiplet) in fluorine NMR spectrum.

Thus on the basis of IR and NMR spectra, elemental analysis and ionic nature of the compounds the newly synthesized μ-oxy-*bis*[(triphenylantimony(V)]carboxylates may be assigned a trigonal bipyramidal structure (Fig. 1). Carboxylate group being more electronegative would occupy apical positions.

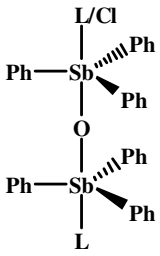
Biological activity: A perusal of literature reveals that in comparison to a large number of organoantimony(V) derivatives reported earlier [22], oxo-bridged $(R_3SbX)_2O$ (R = *p*-

TABLE-3
^{1}H NMR SPECTRAL DATA OF SYNTHESIZED μ-OXY-*BIS*[TRIPHENYLANTIMONY(V)] DERIVATIVES IN δ (ppm)

Compd. No.	Aromatic ring			Ligands			
	Phenyl rings attached to Sb	SH	NH_2	PhH1	PhH2	PhH3	PhH4
1a	7.33-8.19(m)	3.31	–	6.72-6.74(m)	6.75-7.08(m)	7.11-7.14(m)	7.17-7.24(m)
1b	7.52-7.64(m)	3.06	–	6.48-6.58(m)	7.10-7.14(m)	7.14-7.16(m)	7.18-7.34(m)
1c	7.58-7.64(m)	–	–	7.18-7.11(m)			
1d	7.64-7.70(m)	–	3.43	7-14-6.74(m)			
1e	8.28-8.13(m)	–	–	7.97-6.99(m)			
1f	7.87-7.92(m)	–	–	7.88-6.46(m)			
1g	7.76-7.98(m)			7.59-6.88(m)			
1h	7.75-7.96(m)			7.56-6.96(m)			

TABLE-4
^{13}C NMR SPECTRAL DATA OF SYNTHESIZED μ-OXY-*BIS*[TRIPHENYLANTIMONY(V)] DERIVATIVES IN δ (ppm)

Compd. No.	Aromatic ring		Ligands						
	C1/C2	C3/C4	C=O	PhC1	PhC2	PhC3	PhC4	PhC5	PhC6
1a&1b	138.8/136.5	128.7/128.7	170.4	129.6	135.5	133.9	129.6	125.2	133.8
1c	138.8/136.5	128.7/128.7	171.0	136.3	131.2	123.8	153.1	–	–
1d	138.8/136.5	128.7/128.7	171.0	120.2	131.1	114.1	153.6	–	–
1e & 1f	138.8/136.5	128.7/128.7	171.0	125.8	131.9	115.4	168.1	–	–
1g/1h	138.7/136.4	129.9/128.8	171.2	126.5	131.4	116.9	163.2	126.3	134.4



where, L = thiosalicylic acid, *p*-nitrobenzoic acid, *p*-amino benzoic acid, *p*-fluoro benzoic acid, *o*-chloro benzoic acid

Fig. 1. Tentative geometry of newly synthesized μ-oxy-*bis*[(triphenylantimony(V)]carboxylates

$C_6H_4(Cl)$, C_6H_5, X = halogen, thiocyanogen and halopseudohalide, *etc.*) compounds have been studied to a limited extent [5,6,25]. Antibacterial activity of μ-oxo-briged antimony derivatives was determined against two pathogenic bacterial strains *viz.*, *Staphylococcus aureus* and *Escherichia coli.* Antibacterial 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. Antibacterial activity of the synthesized compounds **1f**, **1g**, against *S. aureus* and compounds **1b**, **1c**, **1d**, **1f**, **1g**, against *E. coli* was found to be best amongst the tested compounds. Compounds **1e**, **1h**, showed moderate good activity against the *S. aureus* and *E. coli* (Table-5).

Compounds containing chloro and fluorogroup bonded to antimony also enhance the antimicrobial activity of organoantimony compounds. The biological activity of Sb-O-Sb containing organometallic compounds is yet to be assayed in detail. Keeping in view these facts it was considered work to synthesize different μ-oxy-derivatives of antimony with varying ligand/anions to ascertain their biological activity.

TABLE-5
ANTIBACTERIAL ACTIVITY OF μ-OXY-*BIS*[(TRIPHENYL ANTIMONY(V)]DICARBOXYLATES AND HALOCARBOXYLATES

Sample (100 ppm in DMSO)	Inhibition zone (mm)					
	S. aureus	Inhibition (%)		*E. coli*	Inhibition (%)	
		Streptomycin	Tetracycline		Streptomycin	Tetracycline
1a	19	59	79	-	-	-
1b	15	47	62	28	84	164
1c	14	44	58	27	81	158
1d	17	53	71	27	81	158
1e	16	50	66	20	60	117
1f	24	75	100	25	75	147
1g	22	68	91	23	69	135
1h	15	46	62	16	48	94
Control DMSO	0	–	–	0	–	–
*Streptomycin 100 ppm	32	–	–	33	–	–
#Tetracycline 30 ppm	24	–	–	17	–	–

Conc. = 100 μg/mL of tested compound, 10 μL was used for evaluation. For control 10 μL each of streptomycin (100 μg/mL) and tetracycline (30 μg/mL) was used. DMSO was used as control (10 μL).

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CONFLICT OF INTEREST

The authors declare that there is no conflict of interests regarding the publication of this article.

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