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Enviro-economic Synthesis of Some Antimony (III) Derivatives of O-Alkyl or O-Aryl Trithiophosphates and Studies of Their Antibacterial Activity

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Abstract: An environmentally benign, efficient and facile route is used for the preparation of antimony (III) derivatives of O-alkyl or O-aryl trithiophosphate of the type $ClSb[S_2(S)P(OR)]$ and $RoP(S)[SSbS_2P(S)(OR)]_2$ (R=Me, Et, Pr^i, Bu^i, Ph, CH_2Ph) .They were synthesized by solvent free conditions and microwave exposure procedure resulting from the reaction of antimony trichloride with potassium salts of O-alkyl or O-aryl trithiophosphate in 1:1 and 2:3 molar ratio, respectively. In comparison to conventional synthesis involving tedious work up, excessive use of solvent and extra labour for separation and purification of compounds, the present method indicates operational simplicity, shorter reaction time and higher yields which can prove this procedure as a useful alternative, for the synthesis of antimony(III) derivatives. They are brick red powdery solids, monomer in nature and soluble in common organic solvents. These compounds have been characterized by elemental analysis, molecular weight determinations and spectroscopic (IR, ^1H and ^{31}P NMR) studies.
The potent antibacterial effect of the synthesized compounds has also been investigated.
Keywords: Antimony trichloride, Potassium salts of O-alkyl or O-aryl trithiophosphate, Antibacterial activity, Microwave exposure, Solvent free conditions.

Introduction

Microwave chemistry is the science of applying microwave irradiation to chemical reactions[1-4]. It frequently leads to dramatically reduction in reaction time, higher yields, cleaner reaction profiles and eco-friendliness. In conventional synthesis expensive and toxic solvents are used which are hazardous and cause severe health problem.
On the other hand solvent free conditions offer green chemical route for synthesis of organic compounds[5-8]. In the recent years considerable interest has been evinced in the metal, organometal and organic derivatives of phosphate and dithiophosphate (open chain and cyclic) ester[9-12] and trithiophosphate ligands[13-16].

O-alkyl trithiophosphate ester have been used as defoliants[17], insecticides[18], nematodicides and inhibitor of steel corrosion[19]. Trithiophosphates derivatives of elemental antimony have received very little attention[20] to the best of our knowledge. The survey of literature reveals that some metal derivatives of thiophosphates ligand[21-22] have been evinced for synthesizing and screening antibacterial activity.
Antimony trichloride has been used in the past to dissolve and remove horn stubs from calves without having cut them off. It is also used as a catalyst for polymerization, haydrocraking and chlorination reactions. Its solution is used as an analytical reagent for chloral aromatics and vitamin A[23].
Although a few O-alkyl trithiophospate derivatives of tin[24-25] have been studied as

antibacterial agents in our laboratory yet. The antimony derivatives of this ligand have not been studied for their antibacterial effect as yet.
In view of this it was considered worthwhile to synthesize O-alkyl or O-aryl trithiophosphate derivatives of antimony by microwave assisted method and to study the chemical bonding modes, their antibacterial action and to make comparison of their antibacterial activities with standard drugs.

Experimental

Stringent precautions were taken to exclude moisture throughout all the experimental manipulations. Dipotassium salts of O-alkyl or O-aryltrithiophosphates have been synthesized by the methods reported in the literature[26]. All the solvents used during present investigation were of reagent grade. Carbon and hydrogen were estimated by Coleman C, H and N analyzer. Antimony and sulphur were estimated by iodometric method[27] and Messenger's method[27], respectively. Chlorine is estimated by method reported in literature[27]. Molecular weights were determined by Knauer vapour pressure osmometer in chloroform. FT IR spectra were recorded on Shimadzu 8201 PC spectrophotometer in the range of 4000-200cm^{-1} using CsI cell. ^{1}H NMR spectra were recorded in $CDCl_3$ and ^{31}P NMR spectra were recorded in benzene on Brucker –DRX-300.13MHz spectrophotometer using TMS (for ^{1}H) and H_3PO_4 (for ^{31}P) as an external reference.

Synthesis of ClSb [S_2(S)P(OCH_3)]

Antimony trichloride 1.9361g [8.4819mmol] and dipotassium salt of O-methyltrithiophosphate 2.0018g [8.4822 mmol] in (1:1) molar ratio were taken in R.B.F. The mixture was put into microwave for 2 minutes. Then reaction mixture was dissolve by minimum amount of distilled water, after filteration brick red coloured powdery solid product was dissolve in distilled water. The compound has been washed three-four times with n-hexane and recrystalize it from benzene/petroleum ether mixture (1:4) by method of recrystallization. (**Table – 4).**
Analysis calcd. For ClSb [S_2(S)P(O CH_3)]
C = 3.81; H = 0.95; S = 30.45; Sb = 38.62; Cl = 11.26
Found C = 3.76, H = 0.87, S = 30.01, Sb = 37.96, Cl = 10.98
Rest derivatives were synthesized by similar method.

Synthesis of $CH_3OP(S)[SSbS_2P(S)(OCH_3)]_2$

Antimony trichloride 1.2907g [5.6545mmol] and dipotassium salt of O-methyltrithiophosphate 2.0018g [8.4822 mmol] in (2:3) molar ratio were taken in R.B.F. The mixture was put into microwave for 2 minutes. Then reaction mixture was dissolve by minimum amount of distilled water, after filtration brick red coloured powdery solid product was dissolve in distilled water. The compound has been washed three-four times with n-hexane and recrystalize it from benzene/petroleum ether mixture (1:4) by method of recrystallization (**Table – 4).**
Analysis calcd. For ROP(S)[$SSbS_2P(S)(OR)]_2$
C = 5.02; H = 1.25; S = 40.14; Sb = 33.94
Found C = 4.07, H = 1.01, S = 39.12, Sb = 32.08
Rest derivatives were synthesized by similar method.

Results and Discussion

Reactions of antimony trichloride with dipotassium salt of O-alkyl or O-aryltrithiophosphates in 1:1 and 2:3 molar ratio by using solvent free microwave assisted procedure resulted in the high yield ClSb[S_2(S)P(OR)] and ROP(S)[$SSbS_2$P(S)(OR)]$_2$, respectively.

$$ROP(S)(SK)_2 + SbCl_3 \longrightarrow ROP(S)S_2SbCl + 2KCl$$

$$3ROP(S)(SK)_2 + 2SbCl_3 \longrightarrow ROP(S)[SSbS_2P(S)(OR)]_2 + 6KCl$$

(Where R=Me, Et, Pr^i, Bu^i, Ph, CH_2Ph)

These reactions were completed within 2 minutes in microwave. Then the reaction mixture was dissolved in minimum amount of distilled water after filtration dried derivatives were separated as brick red powdery solid. Potassium chloride was removed in filtrate. These compounds were washed 3-4 times with n-hexane from benzene/petroleum ether mixture(1:4) and recrystallized. The products were isolated as brick red coloured powdery solids. These complexes were soluble in organic solvents like DMSO, DMF, etc.
Conventional method was also used for the formation of these derivatives. In this method antimony trichloride was taken with dipotassium salts of O-alkyl or O-aryl trithiophosphate in 1:1 and 2:3 molar ratios in 30mL methanol, respectively. Reaction mixture was refluxed for 5-6hours. Potassium chloride thus formed as precipitate got filtered off and solvent was

removed under vacuum. They were washed and recrystallized.
It was observed that product yield was more in microwave assisted method than from conventional method.

IR Spectra

IR spectra were recorded in the region 4000-200 cm^{-1}(**Table-1)** and following characteristic changes were observed: -

1. The absorption band at 608.7-576.4 $cm^{-1}$and 535.7-507.1cm^{-1} assigned to νP=S and νP-S linkage, respectively. Shifting of bands towards lower frequency (30-40cm^{-1}) from parent trithiophosphate indicate strong chelation of thiophosphoryl group to metal atom and also indicates the bidentate nature of this group.
2. The ν(P)-O-C and νP-O-(C) linkage were present in the region 1045.3-1002.3cm^{-1} and 835.4-803.1 cm^{-1}, respectively.
3. The appearance of a new medium and weak intensity absorption band in the region 450.4-426.7cm^{-1} indicates the formation of antimony sulfur bond[28].
4. A medium and weak intensity absorption band in the 704.8-689.7 cm^{-1} was assigned for bending vibration of antimony chlorine bond, which was absent in 2 : 3 ratio product.

NMR Spectra

^{1}H NMR Spectra

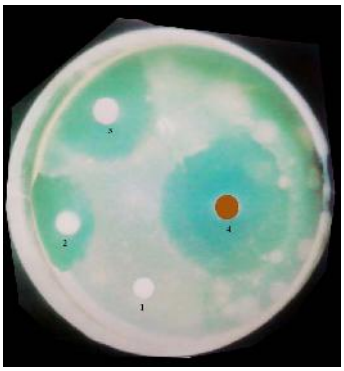
The PMR spectra were recorded in 300.13 MHz region. These derivatives show characteristic resonance signals due to alkoxy and phenyl protons. (**Table-2)** The characteristic resonance signals due to OCH_3, OCH_2, OCH, OC_6H_5, $OCH_2C_6H_5$ protons are present in the expected region[29-30].

^{31}P NMR Spectra

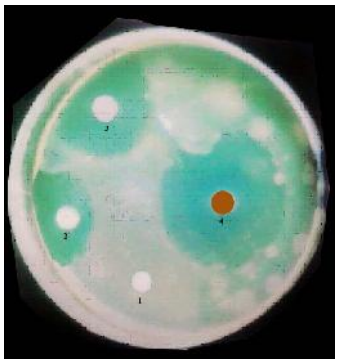
^{31}P NMR spectra were recorded in 121.49 MHz region. Proton decoupled ^{31}P NMR spectra observed in the region 99.26-93.28ppm show the deshielding of the phosphorus atom to the extent of about 12-15ppm from the parent trithiophosphate ligand.(**Table-2**) This is indicative of a bidentate mode of bonding of the ligand moiety in these complexes.

Antibacterial Activity

All the newly synthesized compounds were screened for their antibacterial activity against gram-negative and gram-positive bacteria (**Table-3**). The activity was carried out by using the paper disc method. The zone of inhibition was measured in mm. DMF was used as a solvent. The compounds were tested at 100µg/mL concentration.



Effect on gram positive bacteria
ClSb [S_2(S)P($O^iC_3H_7$)]



Effect on gram negative bacteria
ClSb[S_2(S)P($OCH_2C_6H_5$)]

1. Solvent 2. Ligand 3. $SbCl_3$ 4.Compound

The observations show that compounds 8, 11, 13, 17 are more effective against gram-negative bacteria and compounds 10, 12, 16, 19 are more effective against gram positive bacteria.

Table 1 : Synthetic and Analytical Data of $ClSb[S_2(S)P(OR)]$ and $ROP(S)[SSbS_2P(S)OR)]_2$

S. No.	Reactant …… g …… (mmol)		Molar Ratio	Product …… g …… %	M. P. °C	Analysis % Found (Calcd)					Molecular Weight found (Calcd)
	$SbCl_3$	$ROP(S)(SK)_2$ R… or Ar …				C	H	S	Sb	Cl	
1.	1.9361 [8.4819]	CH_3 2.0018 [8.4822]	1:1	$CH_3OP(S)S_2SbCl$ 2.56 96	129	3.76 (3.81)	0.87 (0.95)	30.01 (30.45)	37.96 (38.62)	10.98 (11.26)	312.02 (315.26)
2.	1.8340 [8.0346]	C_2H_5 2.0087 [8.0348]	1:1	$CH_3CH_2OP(S)S_2SbCl$ 2.61 98	138	6.87 (7.28)	1.41 (1.52)	28.76 (29.15)	35.04 (36.97)	10.01 (10.78)	---
3.	1.7410 [7.6273]	$^iC_3H_7$ 2.0136 [7.6272]	1:1	$^iC_3H_7OP(S)S_2SbCl$ 2.54 97	---	9.86 (10.48)	1.86 (2.03)	26.01 (27.96)	34.87 (35.47)	9.84 (10.34)	338.17 (343.26)
4.	1.6658 [7.2978]	$^iC_4H_9$ 2.0289 [7.2982]	1:1	$^iC_4H_9OP(S)S_2SbCl$ 2.45 94	---	12.08 (13.43)	2.02 (2.52)	25.89 (26.87)	33.02 (34.08)	8.49 (9.94)	354.76 (357.26)
5.	1.5507 [6.7935]	C_6H_5 2.0245 [6.7936]	1:1	$C_6H_5OP(S)S_2SbCl$ 2.45 95	128	18.82 (19.08)	1.02 (1.33)	24.11 (25.45)	31.16 (32.27)	8.86 (9.41)	375.46 (377.26)
6.	1.5307 [6.7059]	$C_6H_5CH_2$ 2.0789 [6.7061]	1:1	$C_6H_5CH_2OP(S)S_2SbCl$ 2.46 94	136	20.88 (21.47)	1.07 (1.79)	23.78 (24.54)	30.86 (31.12)	8.09 (9.07)	---
7.	1.2907 [5.6545]	CH_3 2.0018 [8.4822]	2 : 3	$CH_3OP(S)[SSbS_2P(S)(OCH_3)]_2$ 1.89 94	135	4.07 (5.02)	1.01 (1.25)	39.12 (40.14)	32.08 (33.94)	---	714.50 717.52
8.	1.2203 [5.3460]	C_2H_5 2.0048 [8.0192]	2 : 3	$C_2H_5OP(S)[SSbS_2P(S)(OC_2H_5)]_2$ 1.85 90		8.76 (9.47)	1.06 (1.97)	36.89 (37.92)	31.05 (32.06)	---	------
9.	1.1623	$^iC_3H_7$	2 : 3	$^iC_3H_7OP(S)[SSbS_2P(S)($	149	12.68	1.83	35.08	29.07	---	794.20

	[5.0920]	2.0164 [7.6378]		$O^{i}C_3H_7)]_2$ 1.85 90		(13.47)	(2.62)	(35.93)	(30.38)		(801.52)
10.	1.1164 [4.8909]	$^{i}C_4H_9$ 2.0396 [7.3367]	2 : 3	$^{i}C_4H_9OP(S)[SSbS_2P(S)(O^{i}C_4H_9)]_2$ 1.81 87		16.08 (17.07)	2.76 (3.20)	33.19 (34.14)	27.86 (28.81)	---	841.20 (843.52)
11.	1.0288 [4.5071]	C_6H_5 2.0148 [6.7610]	2 : 3	$C_6H_5OP(S)[SSbS_2P(S)(OC_6H_5)]_2$ 1.83 90		22.05 (23.90)	1.63 (1.66)	30.89 (31.87)	30.89 (31.87)	---	----
12.	0.9885 [4.3307]	$C_6H_5CH_2$ 2.0138 [6.4961]	2 : 3	$C_6H_5CH_2OP(S)[SSbS_2P(S)(OCH_2C_6H_5)]_2$ 1.79 87	160	25.69 (26.65)	1.96 (2.22)	30.04 (30.45)	30.04 (30.45)	---	941.25 (945.52)

Table 2: IR Spectral Data of $ClSb[S_2(S)P(OR)]$ and $ROP(S)[SSbS_2P(S)OR)]_2$

S.No	Compound	¬(P)-O-C	¬P-O-(C)	¬P=S	¬P-S	¬Sb-S	¬Sb-Cl
1.	$CH_3OP(S)S_2SbCl$	1005.4s	810.2s	580.2vs	510.1vs	450.4m	690.8w
2.	$C_2H_5OP(S)S_2SbCl$	1015.2s	820.4s	595.3ss	515.4vs	445.2w	695.4w
3.	$^{i}C_3H_7OP(S)S_2SbCl$	1030.8vs	825.6m	605.6vs	525.8s	435.4w	698.7m
4.	$^{i}C_4H_9OP(S)S_2SbCl$	1045.3s	835.4s	608.7s	535.7s	430.8m	704.8m
5.	$C_6H_5OP(S)S_2SbCl$	1035.2s	830.2s	600.2m	530.8s	440.8m	692.7w
6.	$C_6H_5CH_2OP(S)S_2SbCl$	1030.5s	815.4s	590.4m	520.7m	445.7m	689.7w
7.	$CH_3OP(S)[SSbS_2P(S)(OCH_3)]_2$	1002.3s	803.1s	576.4vs	507.1vs	446.2m	—
8.	$C_2H_5OP(S)[SSbS_2P(S)(OC_2H_5)]_2$	1013.6s	812.3s	592.7s	511.6vs	441.1w	—
9.	$^{i}C_3H_7OP(S)[SSbS_2P(S)(O^{i}C_3H_7)]_2$	1026.4vs	819.2m	598.4vs	521.7s	432.2w	—
10.	$C_4H_9OP(S)[SSbS_2P(S)(O^{5}C_4H_9)]_2$	1042.2s	832.2s	604.6s	531.2s	426.7m	—
11.	$C_6H_5OP(S)[SSbS_2P(S)(OC_6H_5)]_2$	1032.1s	824.3s	598.7m	526.4s	437.7m	—
12.	$C_6H_5CH_2OP(S)[SSbS_2P(S)(OCH_2C_6H_5)]_2$	1026.4s	812.2s	586.2m	516.6m	442.7m	—

Vs = very strong, s = strong, m = medium, w = weak

Table 3 : ^{1}H NMR and ^{31}P NMR Spectral Data of $ClSb[S_2(S)P(OR)]$ and $ROP(S)[SSbS_2P(S)OR)]_2$

S. No.	Compound	^{1}H Chemical Shift (δ ….ppm)	^{31}P Chemical Shift (δ ….ppm)
1.	$CH_3OP(S)S_2SbCl$	3.48, s, 3H (OCH_3)	98.69
2.	$C_2H_5OP(S)S_2SbCl$	1.85, t, 3H (CH_3) 3.01, q, 2H (OCH_2)	96.79
3.	$^{i}C_3H_7OP(S)S_2SbCl$	1.36, d, 6H (CH_3) 2.98-3.06, m, 1H (OCH)	94.38
4.	$^{i}C_4H_9OP(S)S_2SbCl$	1.46, d, 6H (CH_3) 2.25-2.16, m, 1H (CH) 3.38, d, 2H (OCH_2)	93.28
5.	$C_6H_5OP(S)S_2SbCl$	6.65-6.89, m, 5H (OC_6H_5)	98.89
6.	$C_6H_5CH_2OP(S)S_2SbCl$	6.64-6.79, m, 5H (C_6H_5) 3.26, s, 2H (OCH_2)	96.45
7.	$CH_3OP(S)[SSbS_2P(S)(OCH_3)]_2$	3.52 s, 3H (OCH_3) 3.59 s, 6H (OCH_3)	99.78
8.	$C_2H_5OP(S)[SSbS_2P(S)(OC_2H_5)]_2$	1.87 3H (CH_3) 3.04, q, 2H(OCH_2) 1.89,t, 6H(CH_3) 3.09, q, 2H(OCH_2)	97.67
9.	$^{i}C_3H_7OP(S)[SSbS_2P(S)(O^{i}C_3H_7)]_2$	1.38, d, 6H(CH_3) 3.01-3.08,m,1H(OCH) 1.41, d, 12H(CH_3) 3.08-3.06, m,2H(OCH)	96.28
10.	$^{i}C_4H_9OP(S)[SSbS_2P(S)(O^{i}C_4H_9)]_2$	1.48, d, 6H(CH_3) 2.28-2.32,m 1H(CH) 3.40, d, 2H(OCH_2) 1.51, d, 12H(CH_3) 2.29-2.38, m, 2H(CH) 3.44, d, 4H(OCH_2)	94.86
11.	$C_6H_5OP(S)[SSbS_2P(S)(OC_6H_5)]_2$	6.68-6.90m 10H(OC_6H_5) 6.66-6.79m 10H(OC_6H_5)	99.26
12.	$C_6H_5CH_2OP(S)[SSbS_2P(S)(OCH_2C_6H_5)]_2$	6.65-6.80m, 5H(C_6H_5) 3.28,s, 2H(OCH_2) 6.67-6.84, m, 10H(C_6H_5) 3.34s 4H(OCH_2)	97.89

s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet

Table 4:- Antibacterial Activity of $ClSb[S_2(S)P(OR)]$ and $ROP(S)[SSbS_2P(S)OR)]_2$

S.No.	Compounds	Gram Positive Bacteria Zone of inhibition in mm.	Gram Negative Bacteria Zone of inhibition in mm.
1.	Solvent	0	0
2.	$CH_3OP(S)(SK)_2$	8	6
3.	$C_2H_5OP(S)(SK)_2$	7	4
4.	$^{i}PrOP(S)(SK)_2$	9	3
5.	$^{i}BuOP(S)(SK)_2$	5	8
6.	$PhOP(S)(SK)_2$	7	9
7.	$PhCH_2OP(S)(SK)_2$	10	12
8.	$CH_3OP(S)S_2SbCl$	18	27
9.	$C_2H_5OP(S)S_2SbCl$	26	19
10.	$^{i}C_3H_7OP(S)S_2SbCl$	36	22
11.	$^{i}C_4H_9OP(S)S_2SbCl$	24	26
12.	$C_6H_5OP(S)S_2SbCl$	32	20
13.	$C_6H_5CH_2OP(S)S_2SbCl$	24	32
14.	$CH_3OP(S)[SSbS_2P(S)(OCH_3)]_2$	28	19
15.	$C_2H_5OP(S)[SSbS_2P(S)(OC_2H_5)]_2$	22	26
16.	$^{i}C_3H_7OP(S)[SSbS_2P(S)(O^{i}C_3H_7)]_2$	29	18
17.	$^{i}C_4H_9OP(S)[SSbS_2P(S)(O^{i}C_4H_9)]_2$	19	28
18.	$C_6H_5OP(S)[SSbS_2P(S)(OC_6H_5)]_2$	27	20
19.	$C_6H_5CH_2OP(S)[SSbS_2P(S)(OCH_2C_6H_5)]_2$	30	24
20.	Imipenem	12	30
21.	Linezolid	18	10

Conclusion

On the basis of physico-chemical and spectroscopic data the structure of these complexes may be as follow:-

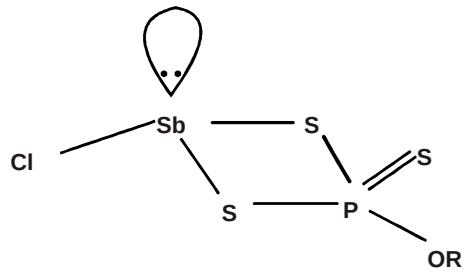


Figure - 1

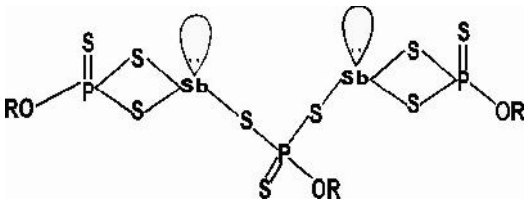


Figure - 2

Due to non-availability of suitable crystals the authentic structure of the complexes synthesized by us could not be determined by X-ray crystallography, however on the basis of spectroscopic studies a distorted tetrahedral geometry for these complexes has been suggested.

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