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Microwave Assisted Synthesis, Characterization and Antibacterial Activity of Some Arsenic (III) Derivatives of O-Alkyl or O-Aryl Trithiophosphates

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Abstract

Arsenic (III) O-alkyl or O-aryl trithiophosphate of the type ClAs[S_2(S)P(OR)] and ROP(S)[SAsS_2P(S)OR)]$_2$ (R=Me, Et, Pr^i, Bu^i, Ph, CH_2Ph) have been synthesized by solvent free microwave assisted procedure from the reaction of arsenic trichloride with potassium salts of O-alkyl or O-aryl trithiophosphate in 1:1 and 2:3 motar ratio respectively. These derivatives have been characterized by elemental analysis, molecular weight determinations and spectroscopic (IR, ^{1}H and ^{31}P NMR) studies. On the basis of them distorted tetrahedral geometry has been proposed for these derivatives. The newly synthesized derivatives show good activity against gram positive and gram negative bacteria and a comparative study of antibacterial effect has also been made with standard drugs.

Keywords: Arsenic trichloride, potassium salts of O-alkyl or O-aryl trithiophosphate, antibacterial activity.

Introduction

In continuation to our earlier investigation on metal, organometal and organic derivatives of phosphate and dithiophosphate (open chain and cyclic) ester[1-6], it was considered of interest to extend the investigation to trithiophosphate ligand[7-9]. Potassium salts of trithiophosphate exist in two isomeric form [(RO)P(S)S_2] (thiono) and [(RS)P(O)S_2] (thiolo)[10].

In the recent years considerable interest have been evinced for synthesizing and screening the antibacterial activity of various metal derivatives of thiophosphates ligand[11]. Although a few O-alkyltrithiophosphate derivatives of tin[12-13] have been studied in our laboratories, the arsenic derivatives of this ligand have not been synthesized as yet.

Arsenic trichloride is poisonous and toxic in nature but it has certain industrial and commercial uses. Arsenic halides are used in the manufacturing of ceramics and in the synthesis of organic arsenochlorine compounds. These include in the blister agent lewisite and the monochloro and Dichlorophenyl arsines, which can be used as either riot control agent or blister agent[14-15].

Microwave chemistry is the science of applying microwave irradiation to chemical reactions[16-18]. Microwave heating have certain benefits over conventional methods like reaction rate acceleration. It required milder reaction conditions. It gives higher chemical yield. It requires lower energy.

In view of this it was considered worthwhile to synthesize O-alkyl or O-aryl trithiophosphate derivatives of arsenic 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.

Material and Methods

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. All the solvents used during present investigation were of reagent grade. Carbon and hydrogen were estimated by Coleman C, H and N analyzer. Arsenic and sulfur were estimated by iodometric method[19] and Messenger's method[19], respectively chlorine is estimated by method reported in literature[19]. 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 spectrophotometer using TMS (for ^{1}H) and H_3PO_4 (for ^{31}P) as an external reference.

Synthesis of ClAs[S_2(S)P(OCH$_3$)]: Arsenictrichloride 1.5386g[8.4808 mmol] and dipotassium salt of O-methyltrithiophosphate 2.0015g [8.4809 mmol] in (1:1) molar ratio were taken in R.B.F. The mixture was put into microwave for 2 minutes. Product obtained as yellow to brown coloured powdery solids. It has been washed three-four times with aqueous ethanol and recrystalize it by method of recrystallization (table –1).
Analysis calcd. for ClAs[S_2(S)P(O CH$_3$)]
C = 4.47; H = 1.12; S = 35.76; As = 27.91; Cl = 13.22
Found C = 4.38, H = 1.11, S = 34.68, As = 26.45, Cl = 12.78
Rest derivatives were synthesized by similar method.

Synthesis of $CH_3OP(S)[SAsS_2P(S)(OCH_3)]_2$: Arsenictrichloride 1.02539g [5.6520 mmol] and dipotassium salt of O-methyltrithiophosphate 2.008g [8.4779 mmol] in (2:3) molar ratio were taken in R.B.F. The mixture was put into microwave for 2 minutes. Product obtained as yellow to brown coloured powdery solids. It has been washed three-four times with aqueous ethanol and recrystalize it. (Table – 1)
Analysis calcd. for $ROP(S)[SAsS_2P(S)(OR)]_2$
C = 5.77; H = 1.44; S = 46.16; As = 24.01
Found C = 5.23, H = 1.23, S = 45.18, As = 23.86.
Rest derivatives were synthesized by similar method.

Results and Discussion

Reactions of arsenictrichloride 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 $ClAs[S_2(S)P(OR)]$ and $ROP(S)[SAsS_2P(S)(OR)]_2$, respectively.

$$ROP(S)(SK)_2 + AsCl_3 \longrightarrow ROP(S)S_2AsCl + 2KCl$$

$$ROP(S)(SK)_2 + AsCl_3 \longrightarrow ROP(S)\left[SAsS_2P(S)(OR)\right]_2$$

(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 brown to yellow powdery solid. Potassium chloride was removed in filtrated. These compounds were washed 3-4 times and recrystallized. The products were isolated as yellow to brown colored powdery solids. These complexes were insoluble in common organic solvents but soluble in coordinated solvents like DMSO, DMF, etc.

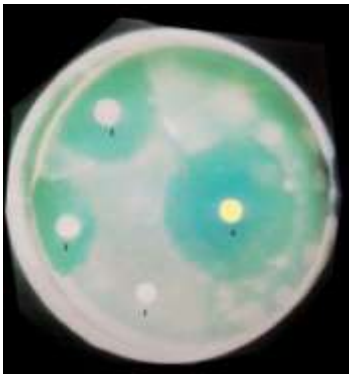
Conventional method was also used for the synthesis of these derivatives. It was observed that product yield was more in microwave assisted method than from conventional method.

IR Spectra: IR spectra were recorded in the region 4000-200 cm^{-1} and following characteristic changes were observed: i. The absorption band at 710.2-664.3 $cm^{-1}$and 550.4-518.1cm^{-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. ii. The ν(P)-O-C and νP-O-(C) linkage were present in the region 1020.3-969.2cm^{-1} and 880.4-828.4 cm^{-1}, respectively. iii. The appearance of a new medium and weak intensity absorption band in the region 390.6-374.3cm^{-1} indicates the formation of arsenic sulfur bond[20-21]. iv. A medium and weak intensity absorption band in the 665.2-660.4 cm^{-1} was assigned for bending vibration of arsenic chlorine bond. Which was absent in 2 : 3 ratios product.

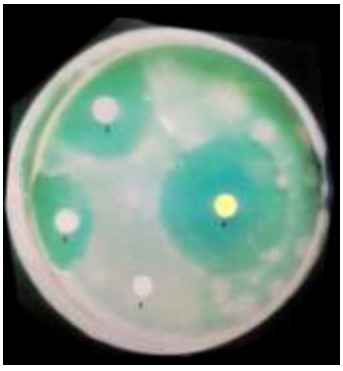
NMR Spectra: ^{1}H NMR Spectra: The PMR spectra were recorded in 300.13 MHz region. These derivatives show characteristic resonance signals due to alkoxy and phenyl protons (Table-3). The characteristic resonance signals due to OCH_3, OCH_2, OCH, OC_6H_5, $OCH_2C_6H_5$ protons are present in the expected region[22-24].

^{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 103.47-95.79ppm show the deshielding of the phosphorus atom to the extent of about 12-15ppm from the parent trithiophosphate ligand (Table-3). 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-4). 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. The observations show that compounds 12, 13, 16, 18 are more effective against gram-negative bacteria and compounds 10, 11, 17, 19 are more effective against gram positive bacteria.



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



Effect on gram negative bacteria
$ClAs[S_2(S)P(OCH_2C_6H_5)]_2$

1. Solvent 2. Ligand 3. $AsCl_3$ 4.Compound

Conclusion

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

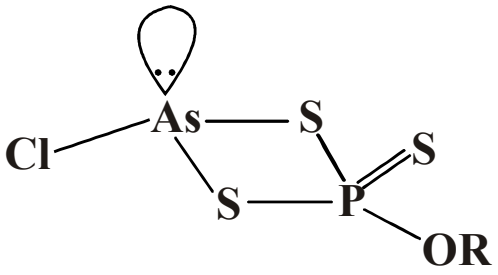


Figure-1

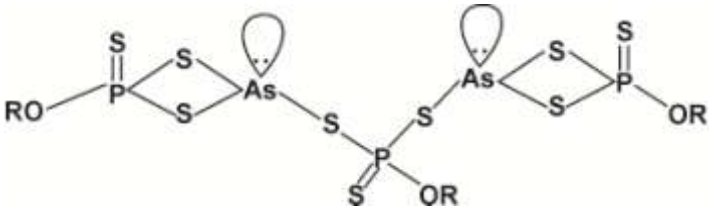


Figure-2

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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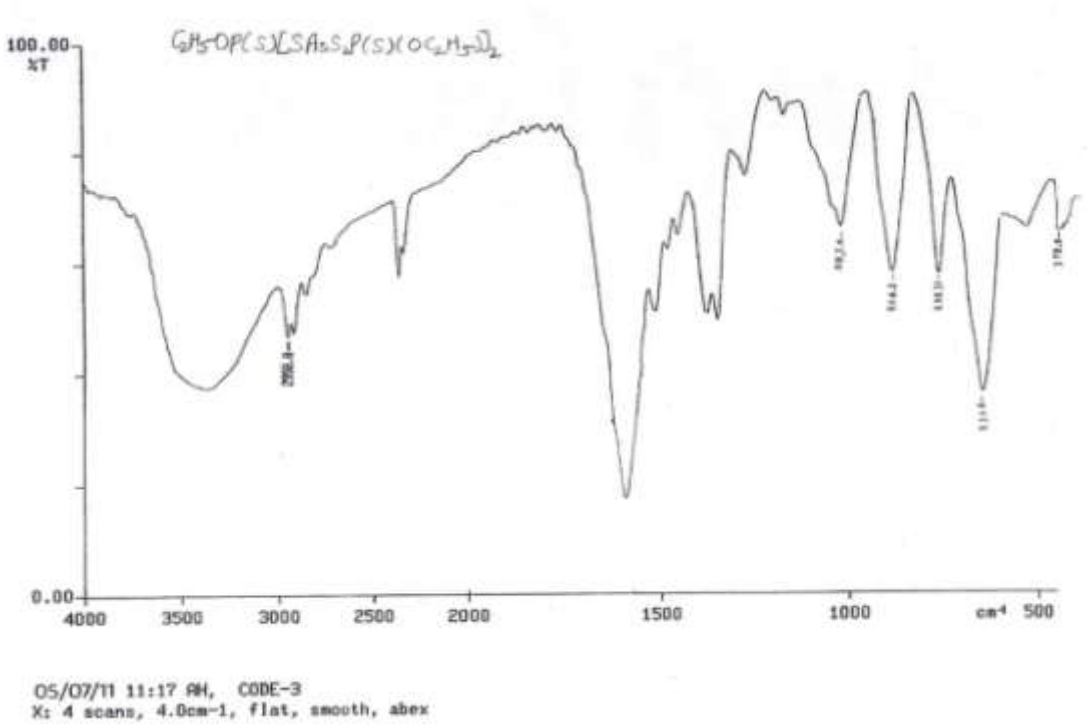

Figure-3

Table -1
Synthetic and Analytical Data of $ClAs[S_2(S)P(OR)]$ and $ROP(S)[SAsS_2P(S)OR)]_2$

S. No	Reactant …… g …… (mmol)		Product …… g …… %	MP °C	Analysis % Found (Calcd)					Molecular Weight found (Calcd)
	$AsCl_3$	$ROP(S)(SK)_2$ R… or Ar …			C	H	S	As	Cl	
1.	1.5386 [8.4808]	CH_3 2.0015 [8.4809]	$CH_3OP(S)S_2AsCl$ 2.15 95	129	4.38 (4.47)	1.11 (1.12)	34.68 (35.76)	26.45 (27.91)	12.78 (13.22)	256.79 (268.42)
2.	1.4519 [8.0029]	C_2H_5 2.0008 [8.0032]	$CH_3CH_2OP(S)S_2AsCl$ 2.10 93	138	7.98 (8.49)	1.38 (1.78)	32.78 (33.99)	24.69 (26.59)	11.98 (12.56)	nil
3.	1.3732 [7.5691]	$^iC_3H_7$ 1.9984 [7.5696]	$^iC_3H_7OP(S)S_2AsCl$ 2.05 91	Nil	11.96 (12.21)	2.02 (2.36)	30.86 (32.38)	23.96 (25.27)	10.70 (11.97)	284.69 (296.42)
4.	1.3126 [7.2351]	$^iC_4H_9$ 2.0114 [7.2352]	$^iC_4H_9OP(S)S_2AsCl$ 2.18 98	Nil	14.76 (15.58)	2.85 (2.90)	29.16 (30.92)	23.04 (24.13)	10.62 (11.43)	297.97 (310.42)
5.	1.2203 [6.7263]	C_6H_5 2.0045 [6.7265]	$C_6H_5OP(S)S_2AsCl$ 2.01 90	128	20.98 (21.93)	1.39 (1.51)	27.86 (29.05)	20.98 (22.67)	9.98 (10.74)	318.18 (330.42)
6.	1.1667 [6.4309]	$C_6H_5CH_2$ 2.0065 [6.4311]	$C_6H_5CH_2OP(S)S_2AsCl$ 1.96 89	136	23.91 (24.54)	1.99 (2.03)	26.08 (27.87)	20.68 (21.75)	9.76 (10.30)	nil
7.	1.02539 [5.6520]	CH_3 2.0008 [8.4779]	$CH_3OP(S)[SAsS_2P(S)(OCH_3)]_2$ 1.69 96	nil	5.48 (5.77)	1.38 (1.44)	46.03 (46.16)	23.90 (24.01)	nil	621.76 623.84
8.	0.97120 [5.3533]	C_2H_5 2.0075 [8.0300]	$C_2H_5OP(S)[SAsS_2P(S)(OC_2H_5)]_2$ 1.68 93	189	11.15 (11.28)	2.02 (2.35)	44.98 (45.15)	21.01 (23.49)	nil	nil
9.	0.9193 [5.0672]	$^iC_3H_7$ 2.0068 [7.6015]	$^iC_3H_7OP(S)[SAsS_2P(S)(O^iC_3H_7)]_2$ 1.52 92	nil	15.07 (16.57)	2.96 (3.22)	43.02 (44.18)	21.17 (22.98)	nil	650.76 (651.84)
10.	0.8722 [4.8076]	$^iC_4H_9$ 2.0048 [7.2115]	$^iC_4H_9OP(S)[SAsS_2P(S)(O^iC_4H_9)]_2$ 1.50 94	196	20.28 (21.63)	3.78 (4.05)	42.18 (43.25)	21.17 (22.50)	nil	779.96 (809.0)
11.	0.8149 [4.4917]	C_6H_5 2.0079 [6.7379]	$C_6H_5OP(S)[SAsS_2P(S)(OC_6H_5)]_2$ 1.61 89	nil	25.03 (26.67)	1.07 (1.85)	34.86 (35.56)	17.98 (18.50)	nil	nil
12.	0.7787 [4.2922]	$C_6H_5CH_2$ 2.0089 [6.4387]	$C_6H_5CH_2OP(S)[SAsS_2P(S)(OCH_2C_6H_5)]_2$ 1.55 88	206	29.81 (30.58)	2.01 (2.54)	33.27 (34.95)	17.81 (18.19)	nil	850.78 (851.84)

Table-2
IR Spectral Data of $ClAs[S_2(S)P(OR)]$ and $ROP(S)[SAsS_2P(S)OR)]_2$

S.No	Compound	ν(P)-O-C	νP-O-(C)	νP=S	νP-S	νAs-S	νAs-Cl
1.	$CH_3OP(S)S_2AsCl$	1010.2s	880.4s	700.2s	545.5s	380.8w	660.0w
2.	$C_2H_5OP(S)S_2AsCl$	990.6s	865.2s	690.9s	540.7s	382.2m	662.8m
3.	$^{i}C_3H_7OP(S)S_2AsCl$	980.5s	850.6s	680.7s	530.3s	387.8m	664.7w
4.	$^{i}C_4H_9OP(S)S_2AsCl$	970.4s	830.8s	665.4s	520.2s	390.6w	665.2w
5	$C_6H_5OP(S)S_2AsCl$	1020.3s	860.7s	710.2vs	550.4s	375.4m	660.5w
6.	$C_6H_5CH_2OP(S)S_2AsCl$	1015.8s	870.5s	705.3s	545.2s	382.2w	663.1w
7.	$CH_3OP(S)[SAsS_2P(S)(OCH_3)]_2$	1005.1s	876.7s	698.3s	544.3s	379.6w	nil
8.	$C_2H_5OP(S)[SAsS_2P(S)(OC_2H_5)]_2$	987.4s	864.2s	688.3s	539.6s	378.8w	nil
9.	$^{i}C_3H_7OP(S)[SAsS_2P(S)(O^{i}C_3H_7)]_2$	978.2s	849.5s	679.3s	528.4s	383.1m	nil
10.	$C_4H_9OP(S)[SAsS_2P(S)(O^{5}C_4H_9)]_2$	969.2s	828.4s	664.3s	518.1s	387.6m	nil
11.	$C_6H_5OP(S)[SAsS_2P(S)(OC_6H_5)]_2$	1018.4s	859.8s	708.1s	549.2s	374.3m	nil
12.	$C_6H_5CH_2OP(S)[SAsS_2P(S)(OC_6H_2C_6H_5)]_2$	1014.7s	869.3s	704.3s	543.2s	381.8w	nil

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

Table-3
^{1}H NMR and ^{31}P NMR Spectral Data of $ClAs[S_2(S)P(OR)]$ and $ROP(S)[SAsS_2P(S)OR)]_2$

S. No.	Compound	^{1}H Chemical Shift (δ ….ppm)	^{31}P Chemical Shift (δ ….ppm)
1.	$CH_3OP(S)S_2AsCl$	3.61, s, 3H (OCH_3)	103.47
2.	$C_2H_5OP(S)S_2AsCl$	1.65, t, 3H (CH_3) 3.05, q, 2H (OCH_2)	99.08
3.	$^{i}C_3H_7OP(S)S_2AsCl$	1.54, d, 6H (CH_3) 3.02-3.14, m, 1H (OCH)	97.90
4.	$^{i}C_4H_9OP(S)S_2AsCl$	1.14, d, 6H (CH_3) 2.36-2.46, m, 1H (CH) 3.46, d, 2H (OCH_2)	95.80
5.	$C_6H_5OP(S)S_2AsCl$	6.71-6.92, m, 5H (OC_6H_5)	102.17
6.	$C_6H_5CH_2OP(S)S_2AsCl$	6.47-6.73, m, 5H (C_6H_5) 3.47, s, 2H (OCH_2)	101.06
7.	$CH_3OP(S)[SAsS_2P(S)(OCH_3)]_2$	363 s, 3H (OCH_3) 368 s, 6H (OCH_3)	104.43
8.	$C_2H_5OP(S)[SAsS_2P(S)(OC_2H_5)]_2$	1.68 3H (CH_3) 3.06, q, 2H(OCH_2) 1.70 6H(CH_3) 3.08, q, 2H(OCH_2)	99.86
9.	$^{i}C_3H_7OP(S)[SAsS_2P(S)(O^{i}C_3H_7)]_2$	1.5b, d, 6H(CH_3) 3.02-3.14,m,1H(OCH) 1.58d, 12H(CH_3) 3.04-3.18, m,2H(OCH)	97.60
10.	$^{i}C_4H_9OP(S)[SAsS_2P(S)(O^{i}C_4H_9)]_2$	1.16, d, 6H(CH_3) 2.38-2.47,m 1H(CH) 3.47, d, 2H(OCH_2) 1.18, d, 12H(CH_3) 2.39-2.49m2H(CH) 3.49, d, 4H(OCH_2)	95.79
11.	$C_6H_5OP(S)[SAsS_2P(S)(OC_6H_5)]_2$	6.72-6.95m 10H(OC_6H_5) 6.74-6.95m 10H(OC_6H_5)	102.28
12.	$C_6H_5CH_2OP(S)[SAsS_2P(S)(OCH_2C_6H_5)]_2$	6.49-6.76m, 5H(C_6H_5) 3.48-5, 2H(OCH_2) 6.51-6.78m, 10H(C_6H_5) 3.49s 4H(OCH_2)	101.86

Table-4
Antibacterial Activity of $ClAs[S_2(S)P(OR)]$ and $ROP(S)[SAsS_2P(S)OR)]$

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.	$CH_3OP(S)S_2AsCl$	19	15
9.	$C_2H_5OP(S)S_2AsCl$	25	18
10.	$^{i}C_3H_7OP(S)S_2AsCl$	35	21
11.	$^{i}C_4H_9OP(S)S_2AsCl$	28	19
12.	$C_6H_5OP(S)S_2AsCl$	22	35
13.	$C_6H_5CH_2OP(S)S_2AsCl$	20	33
14.	$CH_3OP(S)[SAsS_2P(S)(OCH_3)]_2$	21	24
15.	$C_2H_5OP(S)[SAsS_2P(S)(OC_2H_5)]_2$	18	26
16.	$^{i}C_3H_7OP(S)[SAsS_2P(S)(O^{i}C_3H_7)]_2$	19	32
17.	$^{i}C_4H_9OP(S)[SAsS_2P(S)(O^{i}C_4H_9)]_2$	27	14
18.	$C_6H_5OP(S)[SAsS_2P(S)(OC_6H_5)]_2$	17	31
19.	$C_6H_5CH_2OP(S)[SAsS_2P(S)(OCH_2C_6H_5)]_2$	23	19
20.	Imipenem	12	30
21.	Linezolid	18	10

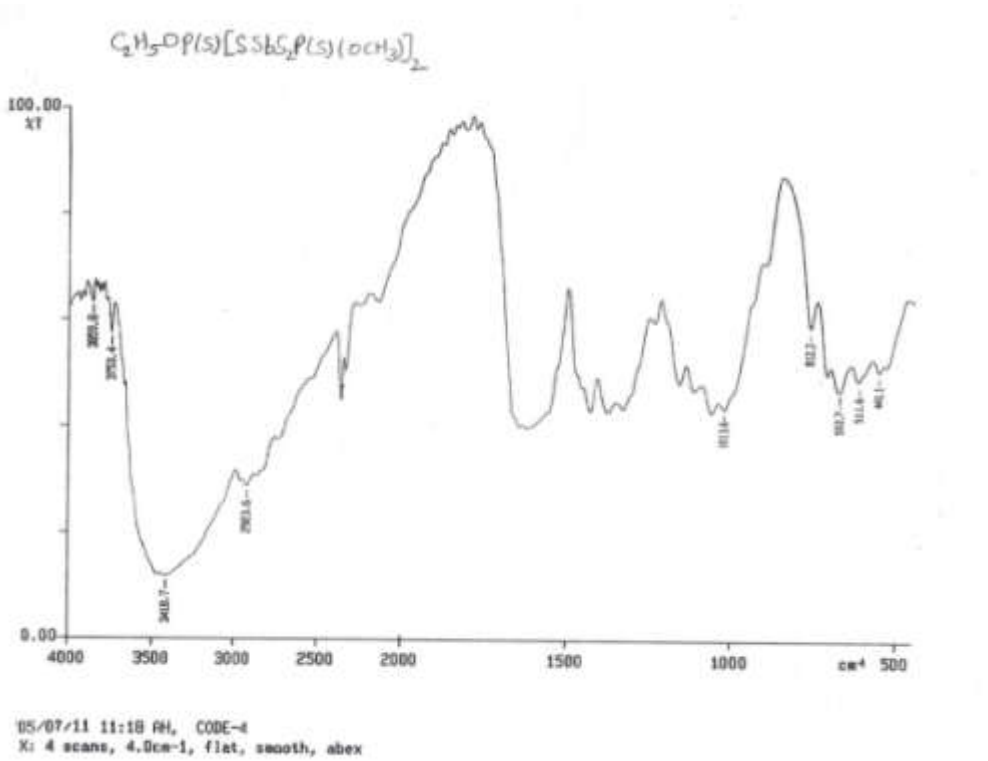

Figure-4

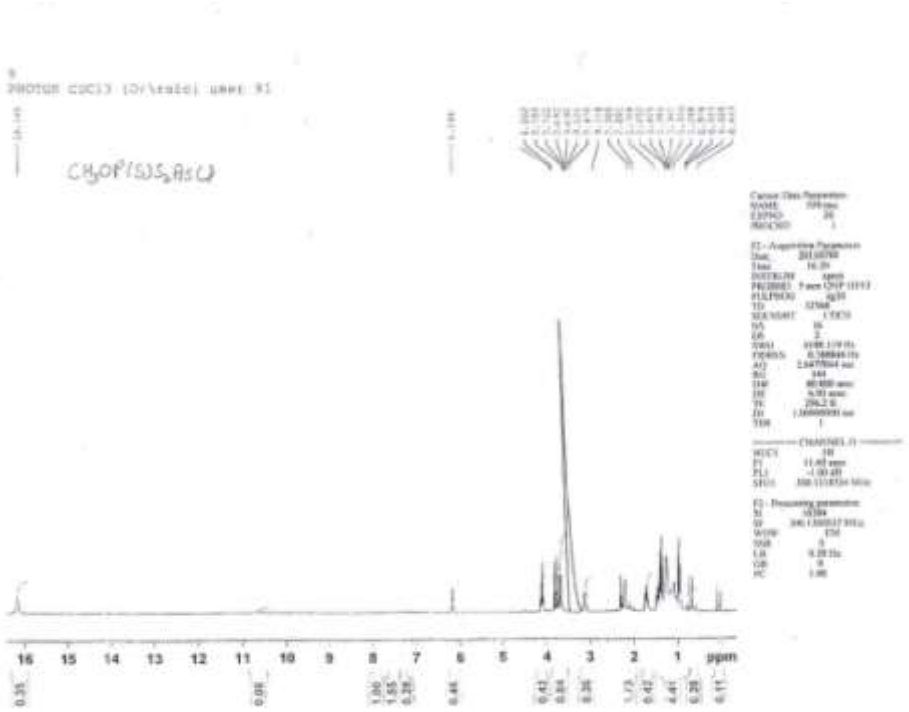

Figure-5

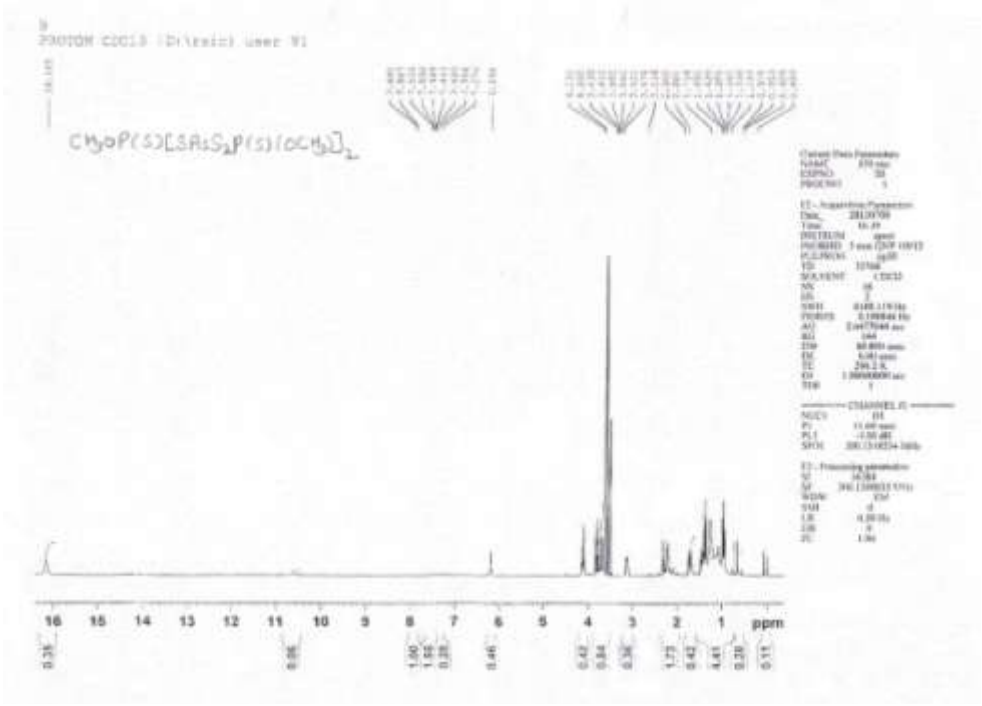

Figure-6

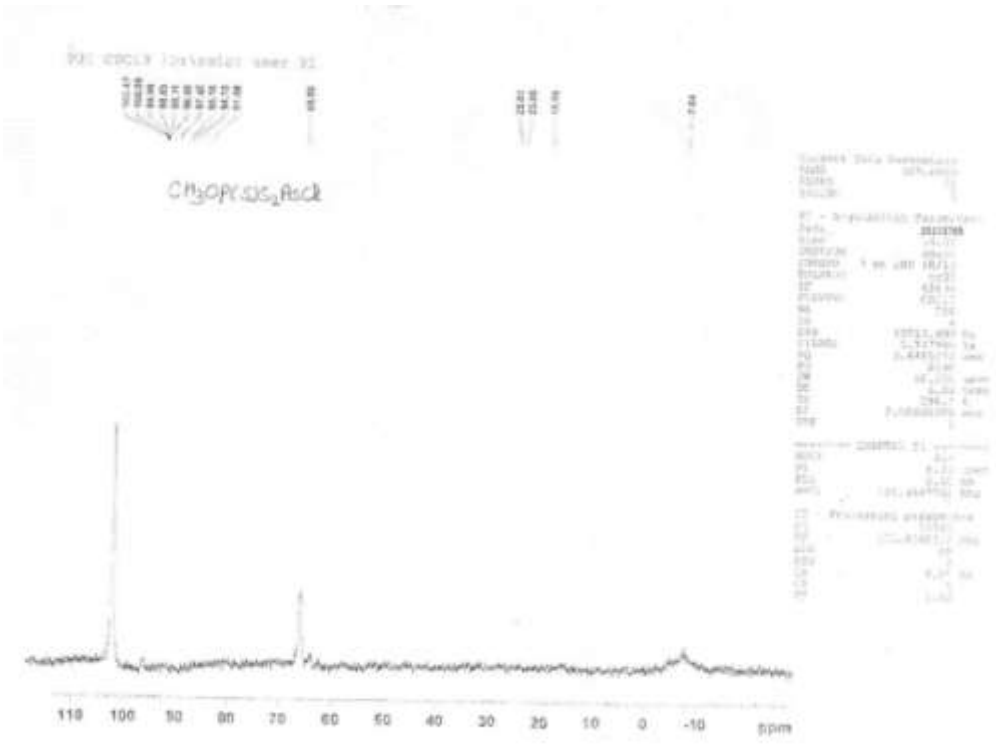

Figure-7

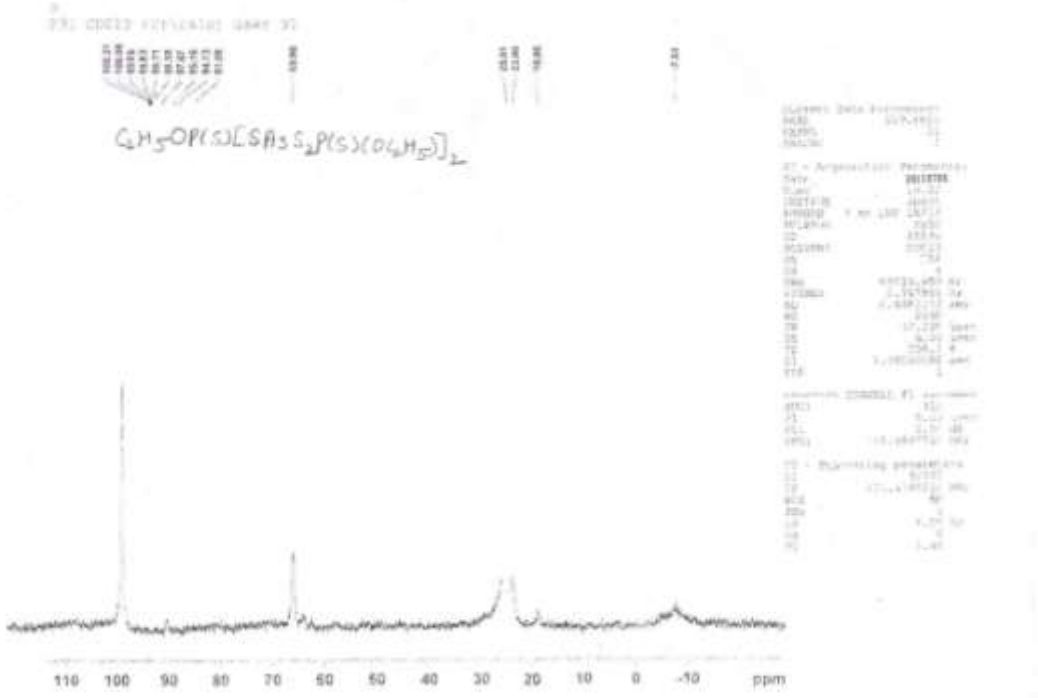

Figure-8