International Journal of Science and Research (IJSR)
ISSN (Online): 2319-7064
Index Copernicus Value (2013): 6.14 | Impact Factor (2013): 4.438

Synthesis, Characterization and Biological Activity of Some Haloorganophosphonates (III) Compounds

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Abstract: ***Three of the haloorganophosphonates (III) compounds of the chemical formula $(CH_3)_4N[RPCl_2X]$, where R represents phenyl or propyl groups, and X represents Cl or Br were prepared. These compounds were characterized by I.R , atomic absorption, elemental analysis (CHN) techniques, and conductivity measurments. The biological activity of the prepared compounds was studied, they showed antimicrobial activity against three pathogenic bacteria viz ; Staphylococcus aureus (gram positive), Escherichia coli, and Pesduomonsaeruginosa (gram negative). The effectiveness of these compounds was measured according to inhibition zone and colonies count methods.***

Keywords: Preparation , Haloorganophosphonates, Antibacterial activity

1. Introduction

At the outset, a wide range of pesticides, insecticides and antibiotics were emanated from phosphorus compounds [1,2]. A large number of small organic and inorganic compounds containing phosphorous are extremely important in biological and chemical profiles, including nucleosides, organophosphates (OP) and phosphate ion. Among them, phosphate ion is one of the limiting nutrients and its analysis is of great importance in biological and environmental chemistry[3].

Molecules with five-coordinate phosphorus are essential to life[4] and the recognition of the role played by the five-coordinate state of the element in biochemistry has spurred interest in this field. On this basis, a consistent interpretation has been made of a number of significant problems of biochemistry[5].

Located at the crossroad of various bioinformation exchange pathways phosphorus-containing compounds play a key role in living organisms as carriers of genetic information and important signalling, regulatory, energy transfer, and structural compounds[6] . Due to this pivotal role, biologically important phosphorus compounds have become therapeutic targets in various modern medicinal techniques, such as antisense[7] and antigene[8] approaches to modulation gene expression or a gene silencing technique using short interfering RNA (siRNA)[9]

2. Materials and Methods

2.1 Materials, and Methods

The reagents used are of analytical grade and were used without further purifications. Haloorganophosphonates were prepared according to well established methods in literature [10].

2.2 Preparation of haloorganophosphonates (III) compounds

These compounds were prepared following the reported method by Jalil[10] according to the following equations :-

A- $(CH_3)_4NBr + C_3H_7PCl_2 \rightarrow (CH_3)_4N[C_3H_7PCl_2Br]$
B- $(CH3)4NBr+C_6H_5PCl_2 \rightarrow (CH_3)_4N[C_6H_5PCl_2Br]$
C- $(CH_3)_4NCl + C_6H_5PCl_2 \rightarrow (CH_3)_4N[C_6H_5PCl_3]$

2.3 General Procedure

(0.0028mole) of $RPCl_2$ in 5ml absolule ethanol was added gradually to (0.0028mole) of $(CH_3)_4NX$ in 5ml absolute ethanol with continuous stirring at room temperature for one hour. A white precipitate was formed, washed with small amounts of ethyl alcohol and then with a little amount of diethyl ether.The products were dried in the oven at a temperature90°C.

2.4 Results and Discussion

The chemical structures of the new compounds were confirmed by atomic absorpation, elemental analysis, IR [11-13], conductivity measurements, and melting points .The molar conductivities of the complexes in 10^{-3} M ethanol were found to be (2.3-55.6) µs . cm^{-1} indicating their electrolytic nature[14] .

Table 1: Some physical characteristics of the prepared compounds

Comp.	*Mol. wt.*	*colour*	*Yield%*	*M.P.°C*	*$\lambda\mu S/cm^{-1}$*
A	*299.012*	*White Powder*	*59*	*336 d*	*55.6*
B	*333.026*	*White Powder*	*68*	*140*	*20.9*
C	*288.57*	*White Powder*	*54*	*285 d*	*2.3*

d : decomposition

Table 2: Element analysis date of the prepared compounds

Comp.	*Element analysis found (calc.)*					
	C%	*H%*	*N%*	*Cl%*	*Br %*	*P%*
A	*27.50 (28.12)*	*5.80 (6.40)*	*4.10 (4.68)*	*23.20 (23.74)*	*26.00 (26.72)*	*9.85 (10.35)*
B	*35.40 (36.06)*	*4.50 (5.14)*	*3.70 (4.20)*	*20.80 (21.31)*	*23.20 (23.99)*	*8.70 (9.29)*
C	*41.00 (41.61)*	*5.20 (5.93)*	*4.40 (4.85)*	*36.20 (36.90)*	--------- ---------	*9.90 (10.73)*

Table 3:FTIR bands (cm^{-1}) of the prepared compounds

Comp.	*γ(P-Cl)*	*γ(P-Br)*	*γ(P-C)*	*Other bonds*
A	*501*	*447,470*	*948*	*C-H al. 2958,2908 C-N al. 1400 CH_3- 1485*

				C-C al. 1296
B	*493*	*424*	*1481*	*C-H ar. 3062, 3016* *C-H a 1303* *C-N al. 1400* *Ph-M-S 744,694*
C	*497*		*1473*	*C-H ar. 3047* *C-N al. 1388* *Ph-M-S 744, 694*

ar.= aromatic
al.= aliphatic
Ph-M-S= (C_6H_5-Mono-Substition)

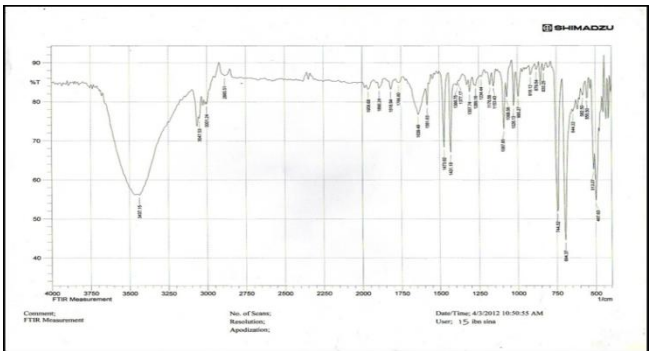


Figure 1: FTIR spectrum of $(CH_3)_4N[PhPCl_3]$

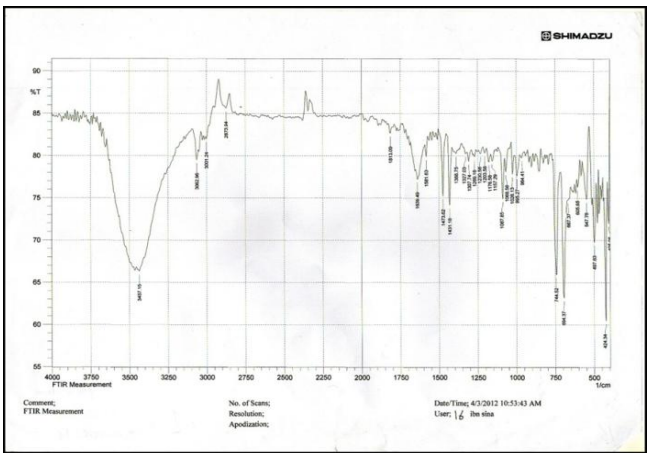
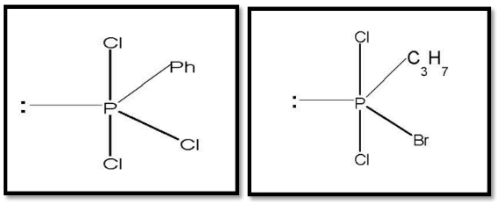


Figure 2: FTIR spectrum of $(C_3H_7)_4N[PhPCl_2Br]$

2.5 The suggested structure

With the help of what is given in the results, a trigonalbipyramide geometry around P (III) ion can be suggested,as illustrated below:-



2.6 Antibacterial Activity

Antibacterial activity was determined by inhibition zone method and colonies count method[16] . The compounds were diluted in ethanol for bioassay. Solvent control was included, although no antibacterial activity has been noted for the solvent employed.The compounds were assayed for antibacterial activity against three bacteria.The screening antibacterial results as illustrated below indicated that the biological activity is mostly dependent on the used concentration and the type of substituent groups (X and R) in the complexes as in Tables(4-9).The concentration [5 mg/ml] showed the highest inhibitory effect against Pesduomonsaeruginosa , the same concentration has the higher activity against Staphylococcus areus bacteria , while has a moderate activity against Escherichia coli bacteria.The biological activity of these compounds follows the order:
$(CH_3)_4N[PhPCl_3]$ > $(CH_3)_4N[PhPCl_2Br]$ > $(CH_3)_4N[C_3H_7PCl_2Br]$

The effect of compounds on the three types of bacteria is due to many reasons, including the ability of solutions of these compounds to destroy the fat layer of the wall of the bacteria causing cell fluids out. It may also be due to the cells membrane which stop the activity of cytoplasme that prevents passage of compounds necessary to metabolism[17].

Table 4: Inhibition zone of $(CH_3)_4N(C_3H_7PCl_2Br)$ in mm

Conc. mg/ml	*Staphylococcus aureus*	*Escherchia coli*	*Pseudomonas aeruginosa*
5	*15*	*14*	*28*
4	*12*	*13*	*27*
3	*11*	*11*	*25*
2	*10*	*10*	*24*
1	*9*	*8*	*23*

Table 5: Colonies count and percentage of killing of $(CH_3)_4N(C_3H_7PCl_2Br)$

Conc. mg/ml	*Colonies count of Staph. aureus*	*Percent of killing%*	*Colonies count of E.coli*	*Percent of killing%*	*Colonies count of pseud.*	*Percenof killing%*
control	140		110		200	
5	83	40.7	70	36.4	115	42.5
4	85	39.3	73	33.6	119	40.5
3	89	36.4	75	31.8	123	38.5
2	91	35.0	77	30.0	126	37.0
1	93	33.5	78	29.1	130	35.0

Table 6: Inhibition zone of $(CH_3)_4N(C_6H_5PCl_2Br)$ in mm

Conc. mg/ml	*Staphylococcus aureus*	*Escherchia coli*	*Pseudomonas aeruginosa*
5	*25*	*18*	*32*
4	*24*	*17*	*31*
3	*20*	*15*	*30*
2	*15*	*12*	*29*
1	*12*	*10*	*28*

Table 7: Colonies count and percentage of killing of $(CH_3)_4N(C_6H_5PCl_2Br)$

Conc. Mg /ml	*Colonies count of Staph. aureus*	*Percent of killing %*	*Colonies count of E.coli*	*Percent of killing %*	*Colonies count of pseud.*	*Percent of killing %*
control	*140*		*110*		*200*	
5	*62*	*55.7*	*55*	*50.0*	*88*	*57.5*
4	*67*	*52.1*	*58*	*47.3*	*92*	*54.0*
3	*71*	*49.3*	*60*	*45.5*	*95*	*52.5*
2	*75*	*46.4*	*65*	*40.9*	*100*	*50.0*
1	*80*	*42.9*	*67*	*39.1*	*110*	*45.0*

Table 8: Inhibition zone of $(CH_3)_4N(C_6H_5PCl_3)$ in mm

Conc. mg/ml	*Staphylococcus aureus*	*Escherchia coli*	*Pseudomonas aeruginosa*
5	*30*	*20*	*36*
4	*28*	*19*	*35*
3	*27*	*18*	*34*
2	*25*	*15*	*33*
1	*22*	*12*	*32*

Table 9: Colonies count and percentage of killing of $(CH_3)_4N(C_6H_5PCl_3)$

Conc. mg/ml	*Colonies count of Staph. aureus*	*Percent. of killing %*	*Colonies count of E.coli*	*Percent. of killing %*	*Colonies count of psedo*	*Percent. of killing %*
control	*140*		*110*		*200*	
5	*52*	*62.8*	*45*	*59.1*	*70*	*65.0*
4	*55*	*60.7*	*48*	*56.3*	*73*	*63.5*
3	*59*	*57.9*	*50*	*54.5*	*77*	*61.5*
2	*63*	*55.0*	*54*	*50.9*	*84*	*58.0*
1	*66*	*52.8*	*57*	*48.2*	*87*	*56.5*

3. Conclusions

The biological activity depends on the concentration used for inhibition; i.e. it increases with increasing the concentration. Types of alkyl & aryl groups, halogens bonded to phosphorus affect the biological activity of the compounds.All compounds showed their effectiveness against the three types of bacteria according to the order; Pseud. aeruginosa > Staph. aureus > E.coli

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