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Synthesis and spectral characterization of some new dibutyl / diphenyl α -aminophosphonates by one-pot three - component Kabachnik-fields reaction

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ABSTRACT

A simple procedure for the synthesis of α – aminophosphonates in high yields by Kabachnik – Fields reaction in one pot three component operation involving 4-aminophenol and halo/methoxy substituted benzaldehydes and dibutyl/diphenyl phosphites without using a catalyst has been employed. The structures of novel α - aminophosphonates were established by elemental analysis, IR, ^{1}H, ^{13}C. ^{31}P NMR and Mass spectral data.

KEYWORDS

4 - Aminophenol;
Aldehydes;
dibutyl/diphenyl phosphites;
α - aminophosphonates;
Spectral characterization.

INTRODUCTION

α – Aminophosphonates are important class of compounds, since they are considered to be structural analogs of the corresponding α – amino acids and find applications as enzyme inhibitors, antibiotics, pharmacological agents and catalytic antibodies.[1] Due to their structural analogy with α –amino acids, these compounds are widely used as neuroactive compounds and plant growth regulators.[2,3] α – Aminophosphonates are chief substrates in the synthesis of phosphonopeptides.[4] In addition, they have been used as antibacterials[5], anti HIV agents[6], attractive peptide mimics.[7] Some of these derivatives are potential herbicides[8] and many other applications of α – aminophosphonates are well documented.

The synthesis of α – aminophosphonates exhibiting high bio-activity and large number of modified conditions in their syntheses have drawn considerable attention over the past few years. The three - component coupling of a carbonyl compound, an amine and hydrophosphoryl compound yielding α – aminophosphonates is called Kabachnik - Fields reaction.[9]

However, this method usually applied to aldehydes, has been much less studied with ketones.[10,11] The low activity of ketones in comparison to aldehydes, necessitates either harsh conditions like heating in an autoclave or the use of various catalysts.[12,13] Meanwhile, the application of microwave irradiation has often been demonstrated to enable facile realization of reactions, which otherwise require harsh conditions and are too slow for practical purposes.[14,15]

The mechanistic pathway of the Kabachnik-Fields

reaction depends on the nature of the substrates. The amine and hydro phosphoryl compound form a complex in which either one of the partners may react with carbonyl compound. Weakly basic amines such as anilines favour the formation of an imine, whereas alkyl amines such as cyclohexyl amines do not form imines. The key step in the Kabachnik-Fields synthesis of aminophosphonates is the nucleophilic addition of amine to a carbonyl compound followed by the addition of dialkyl or diaryl phosphite to the resulting imine. The formation of amino phosphonate is frequently accompanied by the formation of a hydroxyphosphonate or a product of its rearrangement[16] and this is due to the presence of one electrophile (carbonyl compound) and two nucleophiles (amine and phosphite) in the reaction mixture which may compete for the electrophilic centre.[17]

Naryana Reddy *et al.*[18] synthesized novel α – aminophosphonates from one-pot simulataneous reaction of indole-3-carboxaldehyde, a dialkyl - or diphenyl phosphite and different heterocyclic-, cyclic- or other primary amines in the presence of tetramethylguanidine (TMG) as catalyst in toluene at reflux temperature by Kabachnik - Fields reaction. Novel α – aminophosphonates were reported by Srinivasulu *et al.*[19] through one-pot three-component reaction of substituted salicylaldehydes, amines and dialkyl / diaryl phosphites in dry toluene, at reflux temperature.

Recent literature reports indicate that tetramethylguanidine (TMG)[20], quinine[21], magnesium perchlorate or molecular iodine[22] were successfully used as catalysts in the synthesis of α– aminophosphonates.

Ali Karimian[23] prepared α-aminophosphonates in a solution of lithium perchlorate / diethyl ether (5.0 M) in high yields.

A simple, efficient and general method has been developed by Babak Kaboudin[24] for the synthesis of α-aminophosphonates from α-hydroxyphosphonates under solvent-free conditions using microwave irradiation. α-Aminophosphonates were obtained in high yields by the reaction of diethyl α-hydroxyalkylphosphonate with amines in the presence of acidic alumina.

EXPERIMENTAL

Melting points were determined in open capillary tubes on a Mel-Temp apparatus and are uncorrected. IR spectra were recorded in KBr pellets on a Perkin-Elmer FT-IR 1000 spectrophotometer. The ^{1}H, ^{13}C and ^{31}P NMR spectra were taken on Bruker ACF NMR spectrophotometer operating at 400 MHz, 100MHz and 161.89 MHz respectively in $CDCl_3$ or DMSO – d_6 and were referenced to TMS (^{1}H and ^{13}C) and 85% H_3PO_4 (^{31}P). Mass spectral data were collected on LC-MS – 2010 A, Shimadzu spectrometer.

Synthesis of dibutyl [(4-hydroxyanilino) (4-methoxyphenyl) methyl] phosphonate (8)

A mixture of 4-methoxy benzaldehyde (0.68 g, 0.005 mole), 4-aminophenol (0.55 g, 0.005 mole) and dibutyl phosphite (0.97 g, 0.005 mole), in dry toluene (30 mL) was stirred at reflux temperature for 5 hours to obtain the corresponding product. After completion of the reaction, as indicated by TLC (silica gel) using hexane and ethyl acetate (3:1) as a mobile phase, the solvent was removed in a rota-evaporator and the crude product obtained was purified by column chromatography on silica gel (60-120 mesh) using hexane and ethyl acetate (3:1) as an eluent to afford the analytically pure dibutyl [(4-hydroxyanilino) (4-methoxyphenyl) methyl] phosphonate **(8)**. Yield: 71%, m.p: 182-184^{0}C. The remaining compounds were synthesized by adopting the above procedure.

RESULTS AND DISCUSSION

A simple procedure, for the synthesis of α – aminophosphonates in high yields by Kabachnik-Fields reaction in a one-pot three-component operation involving aminophenol and halo/methoxy substituted benzaldehydes and dibutyl/diphenyl phosphites without using a catalyst, has been employed.

Syntheses of new α – aminophosphonates **(4-11)** were accomplished by the one-pot reaction of equimolar quantities of different substituted aromatic aldehydes **(1)** 4-aminophenol **(2)** and dibutyl / diphenyl phosphites **(3)** in the same vessel in toluene, at reflux temperature for 4 - 5 hours with 67-72% yields. TLC was employed to monitor the progress of the reaction and to determine the purity of the products (hexane- ethyl acetate, 3:1). All the title compounds were readily soluble in polar solvents and melted in the range of 182-206^{o}C.

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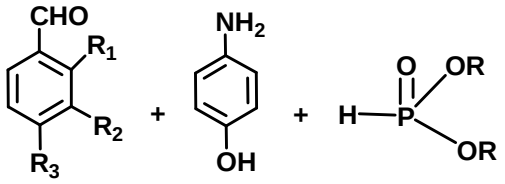
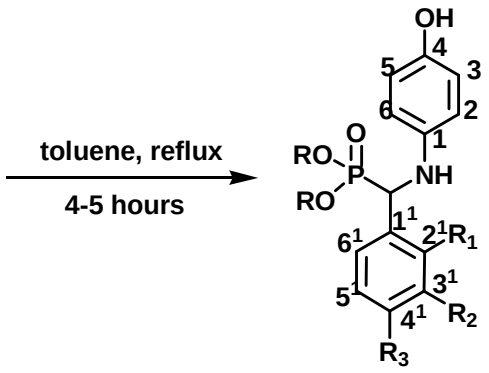
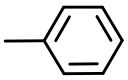
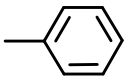
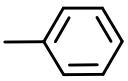
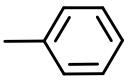


(1) (2) (3)



(4-11)

Compound	R_1	R_2	R_3	R
4	H	Cl	H	$-CH_2-CH_2-CH_2-CH_3$
5	H	Cl	H	
6	H	Br	H	$-CH_2-CH_2-CH_2-CH_3$
7	H	Br	H	
8	H	H	OCH_3	$-CH_2-CH_2-CH_2-CH_3$
9	H	H	OCH_3	
10	Cl	H	Cl	$-CH_2-CH_2-CH_2-CH_3$
11	Cl	H	Cl	

Scheme 1

The synthetic and analytical data of compounds **(4-11)** are given in the TABLE 1. The infrared spectral data are given in the TABLE 1. The compounds **(4-11)** exhibited P=O, P-$C_{(aliphatic)}$ and N-H stretching frequencies in the regions 1219-1249, 721-753 and 3255-3374 cm^{-1} respectively.[25] The characteristic absorption for Ar–OH stretching frequency[25] of these compounds is observed in the region 3430-3508 cm^{-1}.

The proton NMR spectral data of the compounds **(4-11)** are furnished in the TABLE 2. The aromatic protons showed a complex multiplet in the region δ 6.10-7.74. The P-C*-H proton signal appeared as a multiplet in the region δ 4.04 - 4.56 ppm due to its coupling with phosphorous and the neighbouring N-H proton. The N-H proton signal appeared as a singlet in the region δ 5.12 – 5.68 ppm. The OH proton gave a signal in the region δ 8.90 – 9.86 ppm, which was confirmed by D_2O exchange experiments. The methylene proton signals of P-O-*CH_2-CH_2-CH_2-CH_3* in **(4)**, **(6)**, **(8)**, **(10)** showed a multiplet in the region δ 3.94-4.56 and those of P-O-*CH_2-CH_2-CH_2-CH_3*, P-O-*CH_2-CH_2-CH_2-CH_3* appeared as multiplets in the regions δ 1.42 – 1.72 and δ 1.20 – 1.48 respectively. P-O-*CH_2-CH_2-CH_2-CH_3* appeared as a triplet in the region δ 0.76 - 0.90 (t, 3H, J = 12 Hz – 16 Hz). The proton signal of Ar-O-CH_3 in **(8)** and **(9)** appeared as a singlet at δ 3.65 and 3.74 respectively. The ^{13}C NMR spectral data of **(4-11)** are presented in the TABLE 3. The chemical shifts for the aromatic carbons of the title compounds are observed in the expected regions and N-C-P is observed in the region δ 51.5-57.3. The carbon chemical shifts for Ar-O-CH_3 in **(8)** and **(9)** are observed at δ 55.8 and 44.8 respectively. P-O-$CH_2CH_2CH_2CH_3$ in **(4)**, **(6)**, **(8)**, **(10)** are resonated in the region δ 66.8-

TABLE 1 : Physical, analytical, IR and ^{31}P NMR spectral data for the compounds (4-11)

Compound	m.p (^0C)	Yield (%)	Molecular Formula	Elemental analysis Found (Calcd)%			IR Frequencies in cm^{-1}				^{31}P NMR[a,b] ppm
				C	H	N	P=O	-NH	Ar - OH	P-C(Ali.)	
4	192-194	72	$C_{21}H_{29}PNO_4Cl$	59.32 (59.22)	6.79 (6.82)	3.34 (3.29)	1220	3362	3502	743	18.96
5	198-200	69	$C_{25}H_{21}PNO_4Cl$	64.56 (64.45)	4.46 (4.51)	3.07 (3.01)	1219	3281	3430	751	21.49
6	182-184	67	$C_{21}H_{29}PNO_4Br$	53.59 (53.63)	6.23 (6.17)	2.90 (2.98)	1221	3374	3512	738	21.16
7	188-190	69	$C_{25}H_{21}PNO_4Br$	58.81 (58.82)	4.09 (4.12)	2.71 (2.75)	1220	3318	3456	732	20.22
8	182-184	71	$C_{22}H_{32}PNO_5$	62.69 (62.71)	7.64 (7.60)	3.34 (3.33)	1249	3348	3506	721	21.49
9	188-190	69	$C_{26}H_{24}PNO_5$	67.72 (67.68)	5.28 (5.21)	2.96 (3.04)	1248	3272	3470	736	20.30
10	194-196	68	$C_{21}H_{28}PNO_4Cl_2$	54.83 (54.78)	6.22 (6.09)	3.03 (3.04)	1242	3255	3492	753	21.00
11	204-206	67	$C_{25}H_{20}PNO_4Cl_2$	60.07 (60.00)	3.97 (4.00)	2.79 (2.80)	1224	3289	3508	748	19.78

[a]Recorded in $CDCl_3$; [b]Chemical shifts were expressed in ppm from 85% ortho-phosphoric acid as external standard.

67.5. The carbon chemical shifts for P-O-$CH_2\underline{C}H_2CH_2CH_3$, P-O-$CH_2CH_2\underline{C}H_2CH_3$ and P-O-$CH_2CH_2CH_2\underline{C}H_3$ are observed in the regions δ 31.9-32.4, 18.5-18.6 and 13.2-13.5 respectively.

The ^{31}P NMR data of α-aminophosphonates **(4-11)** are presented in the TABLE 1. The ^{31}P NMR signals[27] of 4-11 appeared in the range of δ 18.96 - 21.49 ppm. The LC Mass Spectra of the compounds **(7-10)** were recorded and interpreted. The data are given in the TABLE 4.

TABLE 2 : ^{1}H NMR spectral data [a,b] of compounds (4-11)

Comp.	Ar-H	P-C-H	N- H	Ar-O-CH_3	P-O$CH_2$$CH_2CH_2CH_3$	P-O$CH_2CH_2$$CH_2CH_3$	P-O$CH_2CH_2CH_2$$CH_3$	P-O$CH_2CH_2CH_2CH_2$-OH
4	6.10-7.53 (m,8H)	4.05-4.17 (m,1H)	5.34 (s,1H)	-	4.02-4.25 (m, 2H)	1.52-1.64 (m,2H)	1.16-1.27 (m,2H)	0.82-0.90 (t,3H, *J*=16Hz)	9.14 (s, 1H)
5	6.38-7.38 (m,18H)	4.04-4.19 (m,1H)	5.21 (s,1H)	-	-	-	-	-	9.10 (s, 1H)
6	6.42-7.58 (m,8H)	4.10-4.26 (m,1H)	5.52 (s,1H)	-	4.42-4.56 (m, 2H)	1.42-1.56 (m,2H)	1.21-1.29 (m,2H)	0.84-0.90 (t,3H, *J*=12Hz)	8.90 (s, 1H)
7	6.27-7.70 (m,18H)	4.32-4.46 (m,1H)	5.40 (s,1H)	-	-	-	-	-	9.55 (s, 1H)
8	6.37-7.74 (m,8H)	4.22-4.56 (m,1H)	5.18 (s,1H)	3.65 (s, 3H)	3.94-4.16 (m, 2H)	1.60-1.72 (m,2H)	1.21-1.48 (m,2H)	0.76-0.84 (t,3H, *J*=16Hz)	9.15 (s, 1H)
9	6.42-7.36 (m,18H)	4.40-4.55 (m,1H)	5.68 (s,1H)	3.74 (s, 3H)	-	-	-	-	9.62 (s, 1H)
10	6.40-7.38 (m,7H)	4.05-4.17 (m,1H)	5.62 (s,1H)	-	4.12-4.32 (m, 2H)	1.51-1.66 (m,2H)	1.20-1.33 (m,2H)	0.76-0.82 (t,3H, *J*=12Hz)	9.13 (s, 1H)
11	6.48-7.62 (m.17H)	4.10-4.23 (m,1H)	5.12 (s.1H)	-	-	-	-	-	9.86 (s,1H)

- No such type of proton present, [a]Recorded in $CDCl_3$ [b]Chemical shifts in δ ppm

TABLE 3 : ^{13}C NMR spectral data [a,b] of compounds (4-11)

Compound	Chemical shifts in δ/ppm
4	141.7 (C-1), 115.4 (C-2/C-6), 116.3 (C-3/C-5), 149.7 (C-4), 136.0 (C-1'), 127.4(C-2'), 132.9(C-3'), 128.4(C-4'), 129.0(C-5'), 125.7(C-6'), 53.3(N-C-P), 66.8(P$\underline{C}H_2CH_2CH_2CH_3$), 32.3(POCH$_2\underline{C}H_2CH_2CH_3$), 18.5 (P-OCH$_2CH_2\underline{C}H_2CH_3$), 13.2 (P-OCH$_2CH_2CH_2\underline{C}H_3$)
5	139.4 (C-1), 115.9 (C-2/C-6), 116.3 (C-3/C-5), 149.8 (C-4), 138.9 (C-1'), 126.8 (C-2'), 134.6 (C-3'), 128.7(C-4') 129.7 (C-5'), 125.2 (C-6') 53.5 (N-C-P), 150.4(C-1''), 115.7 (C-2''/(C-6''), 129.7 (C-3''/(C-5''), 120.6 (C-4'').
6	139.9 (C-1), 115.5 (C-2/C-6), 116.7 (C-3/C-5), 148.9 (C-4), 138.8 (C-1'), 127.3(C-2'), 130.9(C-3'), 130.0 (C-4'), 122.7 (C-5'), 131.9 (C-6'), 56.1(N-C-P),67.3(P-O-$\underline{C}H_2CH_2CH_2CH_3$), 32.2 (P-O-$CH_2\underline{C}H_2CH_2CH_3$), 18.6 (P-O-$CH_2CH_2\underline{C}H_2CH_3$), 13.5 (P-O-$CH_2CH_2CH_2\underline{C}H_3$)
7	140.9 (C-1), 115.6 (C-2/C-6), 116.2 (C-3/C-5), 148.0 (C-4), 137.6 (C-1'), 130.0 (C-2'), 124.5 (C-3'), 130.6(C-4'/ C-5'), 125.0(C-6') 53.2(N-C-P), 150.3(C-1''), 112.2(C-2''/(C-6''), 130.4(C-3'/C-5''), 120.5 (C-4'').
8	139.7 (C-1), 115.7 (C-2/C-6), 116.2 (C-3/C-5), 149.4 (C-4), 130.4 (C-1'), 129.0(C-2'/ C-6'), 114.1 (C-3'/ C-5'), 159.3(C-4'), 57.3(N-C-P),55.8 (Ar-O-$\underline{C}H_3$), 67.1(P-O-$\underline{C}H_2CH_2CH_2CH_3$), 32.4(POCH$_2\underline{C}H_2CH_2CH_3$), 18.5 (P-OCH$_2CH_2\underline{C}H_2CH_3$), 13.5 (P-OCH$_2CH_2CH_2\underline{C}H_3$).
9	139.0 (C-1),114.9 (C-2/C-6), 116.0(C-3/C-5), 148.2(C-4), 128.7(C-1'), 128.2 (C-2'/ C-6'), 134.6 (C-3'/ C-5'),158.7(C-4'), 56.1 (N-C-P), 44.8(Ar-O-CH_3), 149.4(C-1''), 115.2 (C-2''/(C-6''), 128.7 (C-3''/(C-5''), 119.6 (C-4'').
10	139.2 (C-1), 115.1 (C-2/C-6), 116.3 (C-3/C-5), 149.0 (C-4), 139.0 (C-1'), 129.8 (C-2'/ C-6'), 127.0(C-3'), 133.2 (C-4'), 129.9(C-5'), 51.5 (N-C-P), 67.5(P-O$\underline{C}H_2CH_2CH_2CH_3$), 31.9(POCH$_2\underline{C}H_2CH_2CH_3$), 18.6 (P-OCH$_2CH_2\underline{C}H_2CH_3$), 13.5 (P-OCH$_2CH_2CH_2\underline{C}H_3$).
11	140.2 (C-1), 114.5 (C-2/C-6), 118.0 (C-3/C-5), 147.9 (C-4), 136.5 (C-1'), 132.5 (C-2'/ C-6'), 132.5 (C-3'/ C-5'), 133.2(C-4'), 53.1 (N-C-P), 148.1(C-1''), 114.5 (C-2''/C-6''), 130.8 (C-3''/C-5''), 118.9 (C-4'').

[a]Recorded in $CDCl_3$, [b]Chemical shifts in δ/ppm

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TABLE 4 : Mass spectral data of compounds (7-10).

Compound	m/z (% relative abundance)
7	514[$(M+4)^+$,10], 512[$(M+2)^+$,35], 495(25), 494(100), 481 (3), 402(5), 369(10).
8	420 [$(M-1)^+$, 34], 419 [$(M-2)^+$,100], 391 (5), 343 (15), 287(2).
9	461 ($M^{+\bullet}$, 10), 450 (100), 406 (50), 433 (5).
10	465[$(M+2)^+$,64], 463 ($M^{+\bullet}$, 100), 459 (2), 405 (5), 391 (2), 370 (4).

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