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$Zn(OAc)_2 \cdot 2H_2O$-Catalyzed Synthesis of α-Aminophosphonates under Neat Reaction

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ABSTRACT: *A series of novel α-aminophosphonates have been prepared by one-pot three-component condensation in the presence of a zinc acetate ($Zn(OAc)_2 \cdot 2H_2O$) catalyst at 50°C under solvent-free conditions with excellent yields. The major advantages of the present method are high yields, a short reaction time, and solvent-free reaction conditions. Their antimicrobial activity was evaluated, and some of them showed significant activity.* © 2011 Wiley Periodicals, Inc. Heteroatom Chem 23:160–165, 2012; View this article online at wileyonlinelibrary.com. DOI 10.1002/hc.20765

INTRODUCTION

α-Aminophosphonates have attracted much attention, owing to their biological activities. Their utilities as enzyme inhibitors, antibiotics, peptide mimics, herbicides, pharmacological agents, and many other applications are well documented [1–5]. A number of synthetic methods for the preparation of α-aminophosphonates have been carried out under various conditions. However, the one-pot synthesis of α-aminophosphonates remains a favorable method because of its versatile route and high yielding reactions. Recently, the three-component synthesis starting from aldehydes, amines, and diethylphosphite or triethylphosphite has been reported using Lewis and Brønsted acid catalysts such as $LiClO_4$ [6], $InCl_3$ [7], lanthanide triflates/magnesium sulfate [8], $TaCl_5$–SiO_2 [9], Amberlyst-15 [10], Al_2O_3-MW [11], sulfonic acid [12], scandium *tris*(-dodecyl sulfate) [13], $M(OTf)_n$ [14], and $M(ClO_4)_n$ [15]. More recently, $CoCl_2 \cdot 6H_2O$ [16], ytterbium perfluorooctanate [$Yb(PFO)_3$] [17], nano Fe_3O_4 [18], $Mg(ClO_4)_2$[19], and $BF_3 \cdot SiO_2$[20] were reported to be effective catalysts for the formation of α-aminophosphonates using a three-component system composed of aldehydes/ketones, amines, and triethylphosphite under neat conditions. However, the above-mentioned catalysts have several disadvantages, such as a long reaction time, low yield of the products, requiring a stoichiometric amount of catalysts, costly, moisture-sensitive, corrosive, toxic or volatile catalysts, and generating a large amount of waste products. $Zn(OAc)_2 \cdot 2H_2O$ is a bench-top catalyst that is easy to handle, cheap, readily available, ecofriendly, and versatile. Additionally, it enables better accessibility of the reactants to the active sites and is efficient for the promotion of many acid-catalyzed organic reactions [21,22].

Hence, there is a need to develop a convenient, environmentally benign, and practicably feasible method for the synthesis of α-aminophosphonates. In this article, we report for the first time an efficient and environmentally benign protocol for the synthesis of α-aminophosphonates (**4a–l**) by the condensation of various aldehydes, amines, and phosphites. The method is a simple, one-pot, practical protocol for the synthesis of α-aminophosphonates in the

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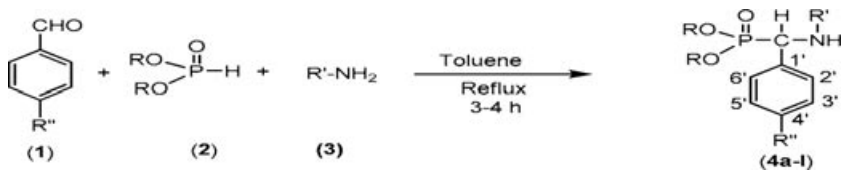


SCHEME 1 Synthesis of α-aminophosphonates **4a–1.**

presence of catalyst $Zn(OAc)_2 \cdot 2H_2O$ at 50°C under solvent-free conditions.

RESULTS AND DISCUSSION

Initially, we attempted a three-component coupling of *p*-hydroxybenzaldehyde (**1**), 2,4-dichloroaniline (**2**), and dimethyl phosphite (**3**) in the presence of a catalytic amount (12 mol%) of $Zn(OAc)_2 \cdot 2H_2O$ under solvent-free conditions at 50°C, and the desired product, **4a**, was obtained with a 94% yield (Scheme 1). We have studied the catalyst concentration on the model reaction. Various concentrations of the catalyst 2, 4, 6, 8, 10, 12, and 14 mol% were used. The results revealed that when the reaction was carried out in the presence of 2, 4, 6, and 8 mol% of the catalyst, it gave a lower yield of the product, even after a prolonged reaction time. At the same time, when the concentration of the catalyst was 12 or 14 mol%, an excellent yield of products was obtained in a short span of time. Even after increasing the catalyst concentration, the yield of the products was found to be constant. Therefore, the use of 12 mol% of $Zn(OAc)_2 \cdot 2H_2O$ is sufficient to push the reaction forward. The results obtained are summarized in Table 1.

To evaluate the effect of the solvent, various solvents such as tetrahydrofuran, dioxane, toluene, acetonitrile, dichloromethane, and ethanol were used for the model reaction. The desired product was obtained with 55, 65, 69, 70, 74, and 75% yields, respectively, after a prolonged reaction time of 4 h, whereas the neat condition afforded a product with an excellent (94%) yield within a very short time of 15 min. This shows that the use of solvent retards the rate of reaction, which decreases the yield of product. The results obtained are summarized in Table 2.

After optimizing the conditions, the generality of this method was examined by the reaction of several substituted benzaldehydes, anilines, and diethyl phosphite using $Zn(OAc)_2 \cdot 2H_2O$ as the catalyst under neat reaction conditions at 50°C. The results are presented in Table 3. Here, we carried out a similar reaction with various aromatic aldehydes containing electron-donating or electron-withdrawing functional groups at different positions, but it did not show any remarkable difference in the yields of product and reaction time. This result provided an incentive to extend this process to various substrates. This methodology offers a significant improvement with regard to the scope of this transformation, simplicity in operation, and green aspects by avoiding expensive or corrosive catalysts. The synthetic, analytical, and spectral data of α-aminophosphonates (**4a–l**) are given in Tables 4–7.

TABLE 1 Influence of the Catalyst on the Synthesis of α-Aminophosphonates **4a–l**[a]

Entry	*Catalyst (mol%)*	*Yield (%)*[b]
1	2	55
2	4	66
3	6	67
4	8	70
5	10	80
6	12	94
7	14	94

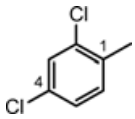
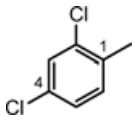
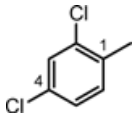
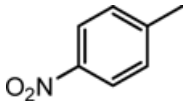
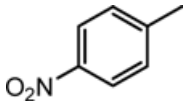
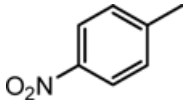
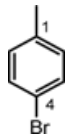
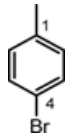
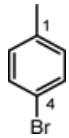
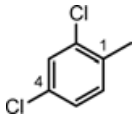
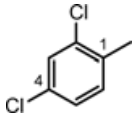
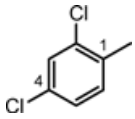
[a]Reaction of *p*-hydroxybenzaldehyde, *p*-bromoaniline, and diethyl phosphite in the presence of $Zn(OAc)_2 \cdot 2H_2O$ under solvent-free conditions at 50°C for 15 min.
[b]Isolated yield.

TABLE 2 Influence of the Solvent on the Synthesis of α-Aminophosphonates[a]

Entry	*Solvents*	*Yield (%)*[b]	*Time*
1	Tetrahydrofuran	55	4 h
2	Dioxane	65	4 h
3	Toluene	69	4 h
4	Acetonitrile	70	4 h
5	Dichloromethane	74	4 h
6	Ethanol	75	4 h
7	Solvent-free	94	15 min

[a]Reaction of *p*-hydroxybenzaldehyde, *p*-bromoaniline, and diethyl phosphite catalyzed by $Zn(OAc)_2 \cdot 2H_2O$ under solvent-free conditions at 50°C for 15 min.
[b]Isolated yield.

TABLE 3 $Zn(OAc)_2 \cdot 2H_2O$ Catalyzed Synthesis of α-Aminophosphonates **4a–l**

Compound	*R*	*R′*	*R″*	*Time (min)*	*Yield (%)*
4a	CH_3		OH	20	94
4b	C_2H_5		OH	18	90
4c	C_6H_5		OH	22	85
4d	CH_3		OH	21	92
4e	C_2H_5		OH	16	88
4f	C_6H_5		OH	19	89
4g	CH_3		OH	17	93
4h	C_2H_5		OH	15	94
4i	C_6H_5		OH	18	93
4j	CH_3		OMe	18	92
4k	C_2H_5		OMe	15	92
4l	C_6H_5		OMe	20	90

EXPERIMENTAL

Melting points were determined in open capillary tubes on a Mel-temp apparatus and were uncorrected. IR spectra (ν_{max} in cm^{-1}) were recorded using KBr disks on a Nicolet-380 FT-IR spectrophotometer. ^1H, ^{13}C, and ^{31}P NMR spectra were recorded on an AMX 400 MHz spectrometer operating at 400 MHz for ^1H, 100 MHz for ^{13}C, and 161.9 MHz for ^{31}P using deuterated chloroform as the solvent.

TABLE 4 Synthetic, Analytical, Infrared, and ^{31}P NMR Spectral Data of α-Aminophosphonates **4a–l** [23,24]

			Elemental Analysis (%)				*IR (cm^{-1})*		
Compound	*Molecular Formula*	*MP (°C)*	*C*	*H*	*N*	*NH*	*P═O*	*P─$C_{aliphatics}$*	*^{31}P NMR/ δ (ppm)*
4a	$C_{15}H_{16}Cl_2NO_4P$	142–144	47.81 (47.89)	4.23 (4.29)	3.69 (3.72)	3386	1215	759	25.10
4b	$C_{17}H_{20}Cl_2NO_4P$	138–140	50.45 (50.51)	4.96 (4.99)	3.42 3.47)	3394	1222	749	24.83
4c	$C_{25}H_{20}Cl_2NO_4P$	150–152	59.95 (60.02)	3.99 (4.03)	2.76 2.80)	3341	1223	763	24.55
4d	$C_{19}H_{20}NO_4P$	135–137	63.82 (63.86)	5.60 (5.64)	3.89 3.92)	3397	1224	756	25.44
4e	$C_{21}H_{24}NO_4P$	175–178	65.41 (65.45)	6.23 (6.28)	3.59 3.63)	3339	1209	756	25.82
4f	$C_{29}H_{24}NO_4P$	162–164	72.30 (72.34)	5.00 (5.03)	2.89 2.91)	3386	1208	760	24.95
4g	$C_{15}H_{17}NO_4PBr$	180–182	46.61 (46.65)	4.40 (4.44)	3.60 3.63)	3220	1224	745	24.63
4h	$C_{17}H_{21}NO_4PBr$	167–169	49.25 (49.29)	4.68 (4.70)	3.32 3.38)	3346	1238	786	24.85
4i	$C_{25}H_{21}NO_4PBr$	179–181	56.75 (56.83)	4.34 (4.38)	2.60 2.65)	3357	1206	757	24.52
4j	$C_{16}H_{18}Cl_2NO_4P$	143–145	49.20 (49.250	4.61 (4.65)	3.53 3.59)	3389	1236	757	26.94
4k	$C_{18}H_{22}Cl_2NO_4P$	146–148	51.64 (51.69)	5.26 (5.30)	3.31 3.35)	3364	1233	753	26.75
4l	$C_{26}H_{22}Cl_2NO_4P$	184–186	60.68 (60.71)	4.29 (4.31)	2.70 2.72)	3378	1228	759	26.88

TABLE 5 ^1H NMR Spectral Data(δ/ppm) of α-Aminophosphonates **4a–l** [25]

Compound	*Ar─H*	*P─C─H*	*N─H*	*P─OCH_2CH_3/P─OCH_3*	*P─OCH_2CH_3*	*OH/OCH_3*
4a	6.71–7.46 (m, 7H)	4.86	5.76 (br, s)	3.42 (s, 6H)	–	10.2
4b	6.69–7.43 (m, 7H)	5.08	5.89 (br, s)	3.62–3.87 (m, 4H)	1.02 (t)	9.8
4c	6.76–7.54 (m, 17H)	4.94	5.72 (br, s)	–	–	9.01
4d	6.68–7.34 (m, 11H)	5.02	5.56 (br, s)	3.44 (s, 6H)	–	10.10
4e	6.63–7.22 (m, 11H)	5.13	5.66 (br, s)	3.74–3.88 (m, 4H)	1.16 (t)	10.20
4f	6.72–7.41 (m, 21H)	4.96	5.42 (br, s)	–	–	9.99
4g	6.64–7.38 (m, 8H)	4.83	5.88 (br, s)	3.71 (s, 6H)	–	10.12
4h	6.58–7.36 (m, 8H)	4.87	5.33 (br, s)	3.66–3.82 (m, 4H)	1.22 (t)	9.23
4i	6.54–7.48 (m, 18H)	4.99	5.28 (br, s)	–	–	10.02
4j	6.74–7.42 (m, 7H)	5.06	5.49 (br, s)	3.45 (s, 6H)	–	4.7
4k	6.79–7.44 (m, 7H)	4.92	5.27 (br, s)	3.71–3.86 (m, 4H)	1.18 (t)	4.9
4l	6.52–7.26 (m, 17H)	4.72	5.62 (br, s)	–	–	4.3

TABLE 6 ^{13}C NMR Spectral Data of α-Aminophosphonates **4a, 4b, 4g,** and **4h** [25b]

Compound	*Chemical Shifts (δ, in ppm)*
4a	136.2 (C-1), 115.5 (C2 and C6), 128.3 (C3 and C5), 156.2 (C-4), 141.2 (C-1′), 127.8 (C-2′) (10), 129.0 (C-3′), 125.0 (C-4′), 130.0 (C-5′), 128.0 (C-6′), 53.6 (d,$^1J_{p\text{-}c} = 152$ Hz, C-7), 51.8 (d,$^2J_{p\text{-}c} = 7.2$ Hz, O$\underline{C}H_3$), 54.3 (d,$^2J_{p\text{-}c} = 7.4$ Hz, O$\underline{C}H_3$)
4b	137.0 (C-1), 115.3 (C2 and C6), 128.9 (C3 and C5), 156.9 (C-4), 141.1 (C-1′), 127.7 (C-2′), 128.2 (C-3′), 125.1 (C-4′), 129.0 (C-5′), 128.4 (C-6′), 58.9 (d,$^1J_{p\text{-}c} = 148.2$ Hz, C-7), 64.2 (d,$^2J_{p\text{-}c} = 7.1$ Hz, O$\underline{C}H_2CH_3$), 62.8. (d,$^2J_{p\text{-}c} = 6.9$ Hz, O$\underline{C}H_2CH_3$), 16.3 (d,$J = 11.8$ Hz, OCH_2 $\underline{C}H_3$), 16.2 (d,$J = 12.6$ Hz, OCH_2 $\underline{C}H_3$)
4g	135.6 (C-1), 115.8 (C2 and C6), 132.1 (C3 and C5), 157.5 (C-4), 135.4 (C-1′), 126.9 (C-2′ and C-6′), 129.1 (C-3′ and C-5′), 129.4 (C-4′), 54.2 (d,$^1J_{p\text{-}c} = 152.4$ Hz, C-7), 52.7 (d,$^2J_{p\text{-}c} = 7.4$ Hz, O$\underline{C}H_3$), 50.9 (d, $^2J_{p\text{-}c} = 7.1$ Hz, O$\underline{C}H_3$)
4h	136.9 (C-1), 115.6 (C2 and C6), 132.6 (C3 and C5), 157.9 (C-4), 132.9 (C-1′), 126.3 (C-2′ and C-6′), 128.7 (C-3′ and C-5′), 129.7 (C-4′), 50.9 (d,$^1J_{p\text{-}c} = 147.2$ Hz, C-7), 63.3 (d,$^2J_{p\text{-}c} = 6.7$ Hz, O$\underline{C}H_2CH_3$), 60.9. (d,$^2J_{p\text{-}c} = 6.9$ Hz, O$\underline{C}H_2CH_3$), 17.6 (d,$J = 11.4$ Hz, OCH_2 $\underline{C}H_3$), 17.4 (d,J = 11.3 Hz, OCH_2 $\underline{C}H_3$)

TABLE 7 Mass Spectral Data of α-Aminophosphonates **4a, 4b**, and **4k**

Compound	*m/z (% Relative Abundance)s*
4a	375 [69 ($M^{+\bullet}$)], 267 (82), 265 (100), 231 (29), 154 (20), 136 (15), 120 (10), 93 (8)
4b	406 [40 (M^{+2})], 405 [55 (M^{+1})], 404 [75 ($M^{+\bullet}$)], 368 (20), 391 (25), 268 (97), 267 (15), 266 (100), 243 (50), 172 (14), 154 (37), 136 (23), 107 (20)
4k	390 [63 ($M^{+\bullet}$)], 284 (12), 280 (17), 229 (100), 215 (20), 196 (24), 139 (15), 74 (9)

TABLE 8 Antibacterial Activity of α-Aminophosphonates **4a–l**

	Zone of Inhibition (%)					
	E. coli			S. aureus		
Compound	*100*	*50*	*25*	*100*	*50*	*25*
4a	14	8	4	11	8	6
4b	13	8	4	10	8	6
4c	15	12	8	12	9	5
4d	15	12	7	15	10	8
4e	10	6	5	10	8	5
4f	10	5	3	14	11	8
4g	12	8	6	11	8	5
4h	8	5	5	–	–	–
4i	8	7	6	9	7	5
4j	10	6	4	9	7	6
4k	12	9	6	10	8	6
4l	11	9	6	9	8	6
Penicillin	12	8	–	10	7	–

TABLE 9 Antifungal Activity of α-Aminophosphonates **4a–l**

	Zone of Inhibition (%)					
	A. niger			H. oryzae		
Compound	*100*	*50*	*25*	*100*	*50*	*25*
4a	10	7	5	11	6	5
4b	11	8	4	11	9	5
4c	13	9	6	13	10	7
4d	12	10	8	15	9	4
4e	9	5	3	13	11	9
4f	10	6	4	9	8	–
4g	14	10	9	13	12	8
4h	13	9	8	10	9	7
4i	13	10	8	11	9	5
4j	9	7	6	12	7	8
4k	12	10	8	14	10	7
4l	10	8	7	12	10	7
Griseofulvin	10	7	–	12	9	–

The ^{1}H and ^{13}C chemical shifts were referenced to tetramethylsilane and ^{31}P chemical shifts to 85% H_3PO_4(*ortho*-phosphoric acid). Mass spectra were recorded on a Jeol SX 102 DA/600 mass spectrometer, using argon/xenon (6 KV, 10 mA) as the FAB gas, and also on a Shimadzu QP-2000 GC-MS instrument.

General Procedure for the Preparation of α-Aminophosphonates **4a–l**

A mixture of *p*-hydroxybenzaldehyde (1 mmol), 2,4-dichloroaniline (1 mmol), dimethylphosphite (1 mmol), and $Zn(OAc)_2{\cdot}2H_2O$ (12%) was stirred vigorously at 50°C for the appropriate time, as indicated in Table 3. The progress of the reaction was monitored by thin layer chromatography. After completion of the reaction, the mixture was washed with rectified spirit and purified by column chromatography using a short silica-gel column with hexane and ethyl acetate (8:2) as the eluent. This procedure was applied successfully for the preparation of other compounds **4a–l** (Table 3).

ANTIMICROBIAL ACTIVITY

The Whatman no. 1 filter paper disk method [26] was employed for the in vitro study of antibacterial and antifungal activity against *Escherichia coli, Staphylococcus aureus, Aspergillus niger,* and *Helminthosporium oryzae*. The inhibitory effects of compounds **4a–l** against these microorganisms are presented in Tables 8 and 9.

Antibacterial Activity

The antibacterial activity of all the title compounds **4a–l** was assayed [26] against the growth of *S. aureus* (gram positive) and *E. coli* (gram negative) at concentrations of 100, 50, and 25 ppm (Table 8). The majority of the compounds exhibited high activity against both of the bacteria, and two compounds, **4c** and **4d**, were more effective than the standard compound.

Penicillin was tested as a standard reference compound to compare the activity of these compounds.

Antifungal Activity

Compounds **4a–l** (Table 9) were screened for their antifungal activity against *A. niger* and *H. oryzae* species, along with the standard fungicide, Griseofulvin. The Disk diffusion method [27] was followed for the screening of the compounds at three different concentrations of 100, 50, and 25 ppm.

It is gratifying to observe that all the compounds **4a–l** exhibited higher antifungal activity when compared with the reference compound. All the compounds exhibited very high activity against fungi, and the compounds **4c** and **4g** were more effective than the standard Griseofulvin.

CONCLUSION

In summary, $Zn(OAc)_2{\cdot}2H_2O$ was found to be an efficient catalyst in the one-pot reaction of various aldehydes, amines, and phosphites to afford α-aminophosphonates in excellent yields under

solvent-free conditions. The procedure has many advantages, such as a short reaction time and a small amount of catalyst used. The major advantages of $Zn(OAc)_2 \cdot 2H_2O$ are that it is a reusable, eco-friendly, inexpensive, and efficient catalyst. Short reaction times, high yields, and easy workup are the advantages of this protocol, making the procedure a green and efficient method for the synthesis of α-aminophosphonates.

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