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A green protocol for one-pot three-component synthesis of α-amino phosphonates catalyzed by succinic acid

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Abstract A simple, efficient, and general method has been developed for the one-pot, three-component synthesis of α-amino phosphonates from a condensation reaction of trialkyl phosphite, aldehydes, and amines in the presence of a catalytic amount of succinic acid (8.5 mol %) (for the first time) under solvent-free conditions. The advantages of this protocol are excellent yields, short reaction time, mild reaction conditions, higher availability, low costs, more environmentally friendly, lack of need for column chromatography and simple work-up procedure.

Keywords α-amino phosphonate · Succinic acid · Trialkyl phosphite · Amine · Aldehyde

Introduction

Organophosphorus compounds have found a wide range of applications in the industrial, agricultural, and medicinal chemistry fields, owing to their biological and physical properties as well as their utility as synthetic intermediates [1, 2]. In recent years, α-amino phosphonates have received attention as structural analogs of the corresponding α-amino acids. The activities of α-amino phosphonates, such as HIV protease [3], enzyme inhibitors [4], antibiotics [5], peptide mimics [6], herbicide, insecticides and fungicides [7, 8], antithrombotic agents [9], antimicrobial agents [10], and inhibitors of UDP-galactopyranose mutase [11], are reported in the literature. As a result, a number of methods have been developed for the synthesis of α-amino phosphonates [12]. Of these, the nucleophilic addition of phosphites to imines, catalyzed by a base or an acid, is the most convenient [13–16]. However, these methods

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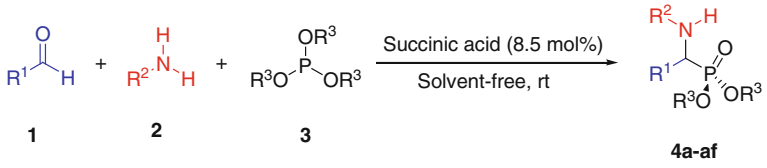
are not devoid of limitation as many imines are hygroscopic and are not sufficiently stable for isolation. In the other words, these reactions cannot be processed in a one-pot reaction involving a carbonyl compound, an amine and a trialkyl phosphite, because the existing amines and water during imine formation can decompose or deactivate the Lewis acid. This drawback has been overcome by some recent methods using metal triflates [$M(OTf)_n$, M = Li, Mg, Al, Cu, Ce] [17], $InCl_3$ [18], $TaCl_5$–SiO_2 [19], CF_3CO_2H [20], $In(OTf)_3$ [21], magnesium perchlorate [22], $PhNMe_3Cl$ [23], $H_3PW_{12}O_{40}$ [24], Amberlyst-15 [26], Amberlite-IR 120 [26], sulfamic acid [27], TiO_2 [28], oxalic acid [29], boric acid [30], Yttria-zirconia [31], $SbCl_3/Al_2O_3$ [32], $CoCl_2.6H_2O$ [33], microwave-assisted [34], and ionic liquid [35]. However, most of these methods have drawbacks, such as toxic catalysts, environmental pollution caused by using an organic solvent, expensive catalyst, difficulty of preparation, unavailable reagents, prolonged reaction times, unsatisfactory yields, and harsh reaction conditions. Therefore, it is necessary to further develop an efficient one-pot multi-component synthesis of α-amino phosphonates which is devoid of these problems.

It is well known that homogeneous catalysts have gained increasing attraction in recent years due to their operational simplicity, low cost, ease of preparation and handling, stability, lack of toxicity, and economic and environmental advantages. One of these homogeneous catalysts is succinic acid (c_4-dicarboxylic acid). Succinic acid, c_4-dicarboxylic acid, a common metabolite in plants, animals and microorganisms, has been used widely in the agricultural, food and pharmaceutical industries [36]. This acid holds good industrial applications and is used in industries such as resins, polymer, paints, cosmetics and inks, etc. [38–40]. To date, the economically renewable resources used in succinic acid production reported are cheese whey [40–43], cane molasses [44, 45], Jerusalem artichoke [46], wheat flour [47], wood hydrolysate [48, 49, 50], and corn straw hydrolysate [51]. As part of our continuing interest in developing methods for the synthesis of α-amino phosphonates [52–56], here we report a green, simple and efficient protocol for the synthesis of α-amino phosphonates through one-pot three-component reactions of aldehydes, amines, and trialkyl phosphite using catalytic amounts of succinic acid (8.5 mol %) under solvent-free conditions at room temperature (Scheme 1).

Results and discussion

Succinic acid is a readily available, low cost reagent, which can be conveniently handled, and removed from the reaction mixture. Thus, its remarkable catalytic



Scheme 1 Synthesis of α-amino phosphonates **4a–af**

activities together with its operational simplicity make it the most suitable catalyst for the synthesis of α-amino phosphonates. First, in order to optimize the reaction conditions, the reaction of benzaldehyde (1 mmol), aniline (1 mmol), and trimethyl phosphite (1 mmol) was carried out using different quantities of succinic acid under different conditions at room temperature. As can be seen in Table 1, maximum yield was obtained with 8.5 mol % of the catalyst under solvent-free conditions. Poor yield (41 %) was obtained when the reaction was carried out in the absence of succinic acid at room temperature under solvent-free conditions for 24 h. We have also studied the effect of various solvents on the model reaction. The various solvents screened were EtOAc, EtOH, MeOH, THF, $CHCl_3$, Et_2O, acetone, CH_2Cl_2, and MeCN. Due to the growing concern for the influence of the organic solvent on the environment as well as on the human body, organic reactions without the use of conventional organic solvents have attracted the attention of synthetic organic chemists. It is observed that the solvent-free conditions gave an excellent yield of product and the shortest reaction time than in the presence of solvents. Development of solvent-free organic reactions is thus gaining prominence.

Thus, several reactions of different aldehydes, amines, and trialkyl phosphite were examined in the presence of succinic acid as a catalyst under solvent-free conditions at room temperature (Table 2). In all cases, the one-pot, three-component reaction proceeded smoothly to afford the corresponding α-amino phosphonates in good to excellent yields. As shown in Table 2, the reaction of anilines with a variety of aromatic aldehydes containing electron-deficient and/or electron-releasing groups and trimethyl/triethylphosphite proceeded to afford α-amino phosphonates in good

Table 1 Optimization reaction conditions for the reaction of benzaldehyde (1 mmol), aniline (1 mmol), and trimethyl phosphite (1 mmol) at room temperature in the presence of succinic acid

Entry	Catalyst (mol %)	Solvent	Time (min)	Yield[a] (%)
1	4	–	30	93
2	6	–	15	97
3	8.5	–	5	98
4	17	–	5	98
5	25	–	5	98
6	35	–	5	98
7	8.5	EtOAc	20	79
8	8.5	EtOH	30	65
9	8.5	MeOH	20	76
10	8.5	THF	25	70
11	8.5	$CHCl_3$	28	67
12	8.5	Et_2O	35	69
13	8.5	Acetone	25	70
14	8.5	CH_2Cl_2	15	82
15	8.5	MeCN	30	69
16	–	–	24 h	41

[a] Yield refers to the pure isolated products

to excellent yields. On the basis of experimental results, the rates of all reactions in the presence of triethyl phosphite were reduced in comparison with trimethyl phosphite under constant conditions.

We have also prepared four new analogs of these compounds in excellent yields (Table 2, entries 30–33). These new compounds were characterized by elemental analyses, IR, ^{1}H, ^{13}C, ^{31}P NMR, and mass spectroscopies.

To show the merit of the present work in comparison with reported results in the literature, we compared the results of succinic acid with the other catalysts for the synthesis of compounds **4a**. As shown in Table 3, succinic acid can act as more effective catalyst with respect to reaction times and yields of products.

A plausible mechanism is shown in scheme 2. It is believed to involve the formation of activated imine **A** by the addition of aldehyde and amine. Then phosphite is added to the C=N bond of imine **A** to give phosphonium intermediate **B**. This phosphonium intermediate undergoes reaction with water to give the α-aminophosphonates **4** and ethanol [30, 57–60].

Experimental

Melting points and IR spectra were measured on an Electrothermal 9100 apparatus and a JASCO FT/IR-460 plus spectrometer, respectively. The ^{1}H, ^{13}C, and ^{31}P NMR spectra were obtained on Bruker DRX-400 Avance instruments with $CDCl_3$ as a solvent. Elemental analyses were performed using a Heraeus CHN-O-Rapid analyzer. Mass spectra were recorded on an Agilent Technology (HP) spectrometer operating at an ionization potential of 70 eV. All reagents and solvents obtained from Fluka and Merck were used without further purification.

General procedure for the synthesis of α-amino phosphonates **4a–af**

The mixture of aldehyde (1 mmol), amine (1 mmol), and succinic acid (8.5 mol %, 0.010 g) were stirred for a few minutes. Then trimethyl/triethyl phosphite (1 mmol) was added, and the reaction mixture was stirred at room temperature for the appropriate time as indicated in Table 2. The progress of reactions was monitored by TLC (ethyl acetate/*n*-hexane, 1/4). After completion of the reaction, the reaction mixture was washed with water (3 × 10 mL). The catalyst is solvable in water and was removed from the reaction mixture. The crude product was washed with the mixture of *n*-hexane/Et_2O (5/2) to give pure products.

Compound **4ad**: white solid, mp. 111–113 °C; IR (KBr) υ: 3,360 (NH), 3,253 (br, OH), 1,269 (P=O); ^{1}H NMR ($CDCl_3$, 400 MHz) δ: 3.52, 3.78 (2d, 6H, J_{HP} = 10.8 Hz, $P(OMe)_2$), 3.88 (s, 3H, OMe), 4.74 (d, 1H, J_{HP} = 24.0 Hz, P–C–H), 4.71, 5.82 (2br, 2H, NH, OH), 6.64 (d, 2H, J_{HH} = 8.0, H_{Ar}), 6.74 (t, 1H, J_{HH} = 8.0 Hz, H_{Ar}), 6.90 (d, 1H, J_{HH} = 8.0 Hz, H_{Ar}), 6.98 (d, 1H, J_{HH} = 8.0 Hz, H_{Ar}), 7.02 (s, 1H, H_{Ar}), 7.14 (t, 2H, J_{HH} = 8.0 Hz, H_{Ar}); ^{13}C NMR ($CDCl_3$, 100 MHz) δ: 53.8, 53.9 (2d, J_{CP} = 7.0 Hz, $P(OMe)_2$), 55.4 (d, J_{CP} = 153.0 Hz, P–CH), 55.9 (s, OMe), 110.2 (d, J_{CP} = 5.0 Hz, C_{Ar}), 113.9 (s, C_{Ar}), 114.7 (d, J_{CP} = 3.0 Hz, C_{Ar}), 118.5 (s, C_{Ar}), 120.9 (d, J_{CP} = 7.0 Hz, C_{Ar}), 127.1 (d, J_{CP} = 3.0 Hz, C_{Ar}), 129.2

Table 2 Preparation of α-amino phosphonates **4a–af**

Entry	R^1	R^2	R^3	Product	Time (min)	Yield[a](%)	Ref.[b]
1	Ph	Ph	Me	**4a**	5	98	[22]
2	Ph	Ph	Et	**4b**	20	97	[28]
3	4-NO_2–C_6H_4	Ph	Me	**4c**	17	90	[23]
4	4-NO_2–C_6H_4	Ph	Et	**4d**	38	85	[26]
5	2,4-di-Cl–C_6H_3	Ph	Me	**4e**	12	90	[51]
6	2,4-di-OMe-C_6H_3	Ph	Me	**4f**	4	94	[53]
7	2,4-di-OMe–C_6H_3	Ph	Et	**4g**	30	90	[51]
8	4-Cl–C_6H_4	Ph	Me	**4h**	4	88	[29]
9	4-Cl–C_6H_4	Ph	Et	**4i**	35	88	[28]
10	4-OMe–C_6H_4	Ph	Me	**4j**	40	98	[30]
11	4-OMe–C_6H_4	Ph	Et	**4k**	65	95	[32]
12	2,4-di-OMe–C_6H_3	4-OMe-C_6H_4	Me	**4l**	10	90	[54]
13	2-Cl–C_6H_4	Ph	Me	**4m**	8	96	[29]
14	2-Cl–C_6H_4	Ph	Et	**4n**	14	95	[28]
15	2,5-di-OMe–C_6H_3	Ph	Me	**4o**	20	90	[52]
16	4-CN–C_6H_4	Ph	Me	**4p**	90	85	[29]
17	Ph	4-OMe-C_6H_4	Me	**4q**	6	88	[29]
18	4-F–C_6H_4	Ph	Et	**4r**	30	91	[26]
19	2,5-di-OMe–C_6H_3	4-Cl-C_6H_4	Me	**4 s**	2	95	[54]
20	2-Me–C_6H_4	Ph	Me	**4t**	2	95	[53]
21	3-OMe–C_6H_4	Ph	Me	**4u**	25	92	[32]
22	3-Cl–C_6H_4	Ph	Me	**4v**	6	95	[26]
23	4-NMe_2–C_6H_4	Ph	Me	**4w**	2	95	[26]
24	3-NO_2–C_6H_4	3-NO_2–C_6H_4	Et	**4x**	50	89	[21]
25	4-NO_2–C_6H_4	4-NO_2–C_6H_4	Me	**4y**	20	90	[22]
26	Ph	4-Cl–C_6H_4	Me	**4z**	7	95	[34]
27	4-NO_2–C_6H_4	4-OMe–C_6H_4	Me	**4aa**	14	93	[34]
28	4-OMe–C_6H_4	4-OMe–C_6H_4	Me	**4ab**	35	92	[20]
29	Ph	4-Me–C_6H_4	Me	**4ac**	16	85	[34]
30	4-OH-3-OMe–C_6H_3	Ph	Me	**4ad**	40	80	c
31	4-OH-3-OMe–C_6H_3	4-Me–C_6H_4	Me	**4ae**	35	77	c
32	2,4-di-OMe–C_6H_3	2-CN–C_6H_4	Me	**4af**	45	70	c

[a] Yields refer to the pure isolated products

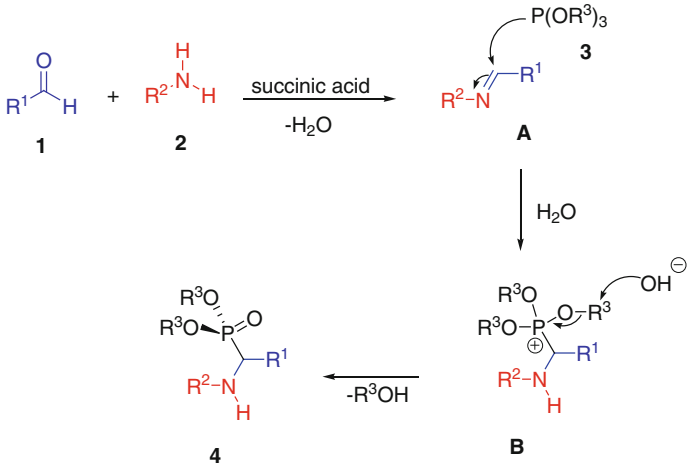
[b] All known products have been reported previously in the literature and were characterized by comparison of IR and NMR spectra with authentic samples

[c] The new compounds synthesized in this work

(s, C_{Ar}), 145.7 (d, $J_{CP} = 3.0$ Hz, C_{Ar}), 146.2 (d, $J_{CP} = 14.0$ Hz, C_{Ar}), 147.1 (d, $J_{CP} = 2.0$ Hz, C_{Ar}); ^{31}P NMR ($CDCl_3$, 162 MHz) δ: 25.27; MS (EI, 70 eV) *m/z* (%): 337 (M^+, 6), 267 (54), 228 (100), 93 (8); Anal. calcd for $C_{16}H_{20}NO_5P$: C, 56.97; H, 5.98; N, 4.15; found C, 57.09; H, 5.80; N, 4.30.

Table 3 Comparison of succinic acid with previously reported catalyst for the synthesis of α-amino phosphonate **4a**

Entry	Catalyst	Conditions	Time	Yield (%)	Ref.
1	Oxalic acid	Solvent-free/50 °C	2 h	98	[29]
2	Boric acid	Solvent-free/rt	15 min	98	[30]
3	$H_3PW_{12}O_{40}$	CH_2Cl_2/rt	<15 min	94	[24]
4	PPA–SiO_2	Solvent-free/80 °C	35 min	97	[53]
5	$Al(H_2PO_4)_3$	Solvent-free/100 °C	15 min	95	[52]
6	$NaHSO_4$–SiO_2	Solvent-free/80 °C	45 min	95	[55]
7	Yttria–zirconia	MeCN/60 °C	2 h	99	[31]
8	$CoCl_2.6H_2O$	Solvent-free/rt	15 min	96	[33]
9	Amberlyst-15	MeCN/rt	25 min	92	[26]
10	Succinc acid	Solvent-free/rt	5 min	98	This work



Scheme 2 Plausible mechanism for the synthesis of α-amino phosphonates **4a–af**

Compound **4ae**: white solid, mp. 104–107 °C; IR (KBr) υ: 3,365 (NH), 3,260 (br, OH), 1,265 (P=O); ^{1}H NMR ($CDCl_3$, 400 MHz) δ: 2.22 (s, 3H, ArMe), 3.53, 3.79 (2d, 6H, J_{HP} = 10.4 Hz, $P(OMe)_2$), 3.88 (s, 3H, OMe), 4.71 (d, 1H, J_{HP} = 22.4 Hz, P–C–H), 4.50, 5.75 (2br, 2H, NH, OH), 6.55 (d, 2H, J_{HH} = 8.0, H_{Ar}), 6.90 (d, 1H, J_{HH} = 8.0 Hz, H_{Ar}), 6.95 (d, 2H, J_{HH} = 8.0 Hz, H_{Ar}), 6.96 (s, 1H, H_{Ar}), 7.02 (s, 1H, H_{Ar}); ^{13}C NMR ($CDCl_3$, 100 MHz) δ: 20.4 (s, ArMe), 53.8 (d, J_{CP} = 6.0 Hz, $2P(OMe)_2$), 55.7 (d, J_{CP} = 142.0 Hz, P–CH), 55.9 (s, OMe), 110.2 (d, J_{CP} = 5.0 Hz, C_{Ar}), 114.0 (s, C_{Ar}), 114.6 (d, J_{CP} = 2.0 Hz, C_{Ar}), 120.9 (d, J_{CP} = 6.0 Hz, C_{Ar}), 127.2 (d, J_{CP} = 3.0 Hz, C_{Ar}), 127.8 (s, C_{Ar}), 129.7 (s, C_{Ar}), 143.8 (d, J_{CP} = 15.0 Hz, C_{Ar}), 145.6 (d, J_{CP} = 3.0 Hz, C_{Ar}), 147.0 (d, J_{CP} = 2.0 Hz, C_{Ar}); ^{31}P NMR ($CDCl_3$, 162 MHz) δ: 25.41; MS (EI, 70 eV) *m/z* (%): 351 (M^+, 8), 242 (100), 234 (17); Anal. calcd for $C_{17}H_{22}NO_5P$: C, 58.12; H, 6.31; N, 3.99; found C, 58.25; H, 6.45; N, 4.06.

Compound **4af**: white solid, mp. 88–90 °C; IR (KBr) υ: 3,383 (NH), 2,211 (CN), 1,246 (P=O); ^{1}H NMR ($CDCl_3$, 400 MHz) δ: 3.61, 3.81 (2d, 6H, $J_{HP} = 10.4$ Hz, $P(OMe)_2$), 3.79, 3.94 (2 s, 6H, 2OMe), 5.35 (dd, 1H, $J_{HP} = 23.6$ Hz, $J_{HH} = 8.8$ Hz, P–C-H), 5.60 (t, 1H, $J_{HH} = 8.0$ Hz, NH), 6.50 (d, 1H, $J_{HH} = 6.4$, H_{Ar}), 6.51 (s, 1H, H_{Ar}), 6.64 (d, 1H, $J_{HH} = 8.4$ Hz, H_{Ar}), 6.70 (t, 1H, $J_{HH} = 8.0$ Hz, H_{Ar}), 7.29–7.38 (m, 3H, H_{Ar}); ^{13}C NMR ($CDCl_3$, 100 MHz) δ: 48.5 (d, $J_{CP} = 157.0$ Hz, P–CH), 53.8 (d, $J_{CP} = 7.0$ Hz, $P(OMe)_2$), 55.3, 55.8 (2 s, 2OMe), 97.2 (s, C_{Ar}), 98.7 (d, $J_{CP} = 1.0$ Hz, C_{Ar}), 105.0 (d, $J_{CP} = 3.0$ Hz, C_{Ar}), 111.8 (s, CN), 115.1 (d, $J_{CP} = 1.0$ Hz, C_{Ar}), 117.4 (s, C_{Ar}), 117.7 (s, C_{Ar}), 129.1 (d, $J_{CP} = 5.0$ Hz, C_{Ar}), 132.7 (s, C_{Ar}), 134.2 (s, C_{Ar}), 149.6 (d, $J_{CP} = 13.0$ Hz, C_{Ar}), 158.0 (d, $J_{CP} = 6.0$ Hz, C_{Ar}), 160.9 (d, $J_{CP} = 3.0$ Hz, C_{Ar}); ^{31}P NMR ($CDCl_3$, 162 MHz) δ: 24.08; MS (EI, 70 eV) *m/z* (%): 376 (M^+, 2); Anal. calcd for $C_{18}H_{21}N_2O_5P$: C, 57.45; H, 5.62; N, 7.44; found C, 57.53; H, 5.75; N, 7.33.

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