

## One-pot three-component synthesis of $\alpha$ -amino phosphonates using 2,3-dibromosuccinic acid as catalyst under solvent-free conditions

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**Abstract:** For the first time, 2,3-dibromosuccinic acid was applied as catalyst for the one-pot, three-component synthesis of  $\alpha$ -aminophosphonates from aldehydes, amines and trialkylphosphite under solvent-free condition at room temperature. This procedure has some advantages such as excellent yields, mild reaction conditions, short reaction time, lack of need for column chromatography, simple work-up, cheap and biodegradable catalyst.

**Keywords:**  $\alpha$ -Aminophosphonate, Amines, Aldehydes, 2,3-Dibromosuccinic acid, Solvent-free.

### Introduction

Due to the growing concern for the influence of organic solvents on the environment as well as on the human body, organic reactions without use of conventional organic solvents have attracted the attention of synthetic organic chemists [1]. Although a number of modern solvents such as fluorous media [2],  $\text{ScCO}_2$  [3], ionic liquids [4], and water [5-6] have been extensively studied recently, not using a solvent at all is definitely the best option. Development of solvent-free organic reactions is thus gaining prominence [7-9].

The nitrogen atom is present in many natural products, biologically relevant molecules, pharmaceuticals, and dyes [10-12]. Among compounds containing nitrogen atom,  $\alpha$ -aminophosphonates are important specially.  $\alpha$ -Aminophosphonates as a kind of natural amino acid analogs occupy an important place, reveal diverse, interesting biological and biochemical properties [13-15]. Their potential as plant growth regulators [16], herbicidals [17], pharmacogenic agents [18], antibiotics [19], enzyme inhibitors [20], HIV protease [21], and antithrombotic agents [22] is well documented. As a result, various methodologies have been developed for the synthesis of  $\alpha$ -amino

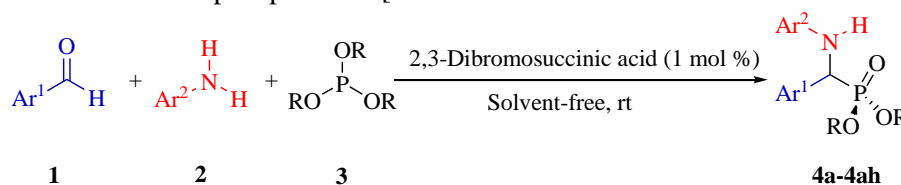
phosphonates. Many of these methods are based on nucleophilic addition of phosphate to imines catalyzed by protic [23] Lewis acids like  $\text{BF}_3 \cdot \text{OEt}_2$  [24],  $\text{SnCl}_4$  [25],  $\text{ZnCl}_2$  and  $\text{MgBr}_2$  [26], or by base [27]. However, these reactions can not proceed in one-pot procedure from reaction between an aldehyde, an amine and a phosphite, because the amines and water that exist during imine formation can decompose or deactivate the Lewis acids [28]. This drawback has been overcome by some recent methods using metaltriflates [ $\text{M}(\text{OTf})_n$ ,  $\text{M} = \text{Li}, \text{Mg}, \text{Al}, \text{Cu}, \text{Ce}$ ] [29],  $\text{NaH}_2\text{PO}_4$  [30],  $\text{TaCl}_5 \cdot \text{SiO}_2$  [31],  $\text{InCl}_3$  [32],  $\text{ZrOCl}_2 \cdot 8\text{H}_2\text{O}$  [33-34],  $\text{H}_3\text{PW}_{12}\text{O}_{40}$  [35],  $\text{SmI}_2$  [36], oxalic acid [37], CSA [38],  $\text{Mg}(\text{ClO}_4)_2$  [39], CAN [40], Boric acid [41],  $\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$  [42],  $\text{PhNMe}_3\text{Cl}$  [43], Yttria-zirconia [44],  $\text{TiO}_2$  [45],  $\text{SbCl}_3/\text{Al}_2\text{O}_3$  [46], Amberlite-IR 120 [47], Amberlyst-15 [48],  $\text{In}(\text{OTf})_3$  [49], microwave assisted [50], and  $\text{CF}_3\text{CO}_2\text{H}$  [51]. 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  $\alpha$ -amino phosphonates which is devoid of these problems.

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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, and economic advantages. One of these homogeneous catalysts is 2,3-dibromosuccinic acid.

As part of our continuing interest in developing methods for the synthesis of  $\alpha$ -aminophosphonates [52-

60], herein, we report a green, simple and efficient protocol for the synthesis of  $\alpha$ -aminophosphonates through one-pot three-component reactions of aldehydes, amines, and trialkylphosphite using catalytic amounts of 2,3-dibromosuccinic acid (1 mol%) under solvent-free conditions at room temperature (Scheme 1).



**Scheme 1:** Synthesis of  $\alpha$ -aminophosphonates **4a-4ah**.

## Results and discussion

First, in order to optimize the reaction conditions, the reaction of 4-chlorobenzaldehyde (1 mmol), aniline (1 mmol), and trimethylphosphite (1 mmol) was carried out using different quantities of 2,3-dibromosuccinic acid under different conditions at room temperature. As can be seen in (Table 1, entry 6), the best result in the presence of 2,3-dibromosuccinic acid (0.0027 g, 1 mol %) of the catalyst under solvent-free conditions. Poor yield (52%) was obtained when the reaction was carried out in the absence of 2,3-dibromosuccinic acid at room temperature under solvent-free conditions for 24 h (Table 1, entry 7). Structurally diverse aldehydes, amines and phosphites were used in optimized conditions to afford the corresponding  $\alpha$ -aminophosphonates in high to excellent yields.

**Table 1:** Optimization of the reaction conditions for the synthesis of  $\alpha$ -aminophosphonates **4**<sup>a</sup>.

Entry	Catalyst (mol%)	Time (min)	Isolated Yield (%)
1	20	1	94
2	15	1	95
3	10	1	97
4	5	1	97
5	2	1	97
6	1	1	97
7	No	24 h	52

<sup>a</sup> Reaction conditions: 4-chlorobenzaldehyde, aniline, and trimethylphosphite under solvent-free conditions at room temperature.

Thus, several reactions of different aldehydes, amines, and trialkylphosphite were examined in the presence of 2,3-dibromosuccinic 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  $\alpha$ -aminophosphonates 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  $\alpha$ -aminophosphonates in shorter reaction times and in good to excellent yields. On the basis of experimental results, the rates of all reactions in the presence of triethylphosphite were reduced in comparison with trimethylphosphite under constant conditions.

We have also prepared one new analogs of these compounds in excellent yields (Table 2, entry 34). These new compounds were characterized by elemental analyses, IR, <sup>1</sup>H, <sup>13</sup>C, <sup>31</sup>P NMR, and mass spectroscopies.

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  $\alpha$ -aminophosphonates **4** and ethanol [38, 41].

To show the capability of the present work in comparison with reported conditions in the literature, we compared the results of 2,3-dibromosuccinic acid with the other catalysts for the synthesis of compounds **4c**. As shown in (Table 3), 2,3-dibromosuccinic can act as effective catalyst with respect to reaction times and yields of products.

## Conclusion

In summary, we have developed a simple and efficient method for the one-pot, three-component

synthesis of  $\alpha$ -aminophosphonates in the presence of catalytic amount of 2,3-dibromosuccinic acid (1 mol%) 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.

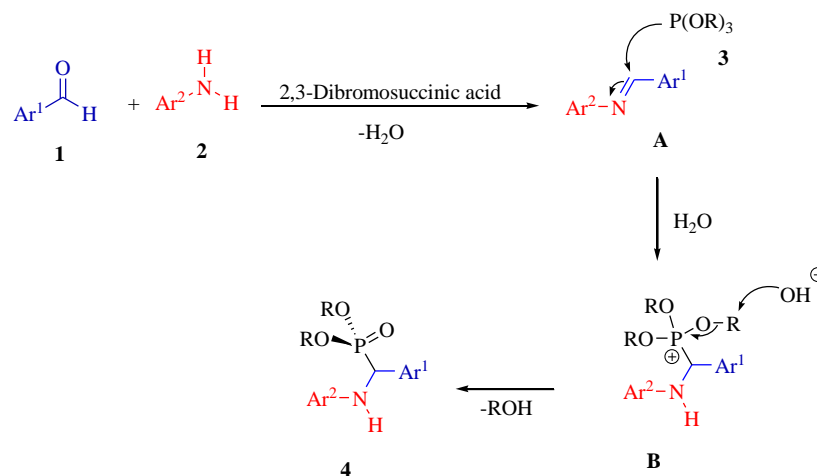
**Table 2:** Preparation of  $\alpha$ -aminophosphonates 4a-4ah.

Entry	Ar <sup>1</sup>	Ar <sup>2</sup>	R	Product	Time (min)	Yield <sup>a</sup> (%)	M.p. / °c	Ref. <sup>b</sup>
1	Ph	Ph	Me	4a	5	90	91-93	39
2	Ph	Ph	Et	4b	18	88	87-88	45
3	4-Cl-C <sub>6</sub> H <sub>4</sub>	Ph	Me	4c	1	97	137-140	37
4	4-Cl-C <sub>6</sub> H <sub>4</sub>	Ph	Et	4d	6	95	58-59	45
5	4-OMe-C <sub>6</sub> H <sub>4</sub>	Ph	Me	4e	1	97	122	41
6	4-OMe-C <sub>6</sub> H <sub>4</sub>	Ph	Et	4f	17	97	108	46
7	2,4-di-OMe-C <sub>6</sub> H <sub>3</sub>	4-OMe-C <sub>6</sub> H <sub>4</sub>	Me	4g	5	91	131-133	54
8	4-NO <sub>2</sub> -C <sub>6</sub> H <sub>4</sub>	Ph	Me	4h	7	93	124-125	43
9	4-NO <sub>2</sub> -C <sub>6</sub> H <sub>4</sub>	Ph	Et	4i	14	90	126-127	47
10	2,4-di-Cl-C <sub>6</sub> H <sub>3</sub>	Ph	Me	4j	3	85	109-111	59
11	2,4-di-OMe-C <sub>6</sub> H <sub>3</sub>	Ph	Me	4k	1	95	146-148	57
12	2,4-di-OMe-C <sub>6</sub> H <sub>3</sub>	Ph	Et	4l	15	84	98-99	59
13	2-Cl-C <sub>6</sub> H <sub>4</sub>	Ph	Me	4m	1	95	132-134	37
14	2-Cl-C <sub>6</sub> H <sub>4</sub>	Ph	Et	4n	8	94	88-89	45
15	2,5-di-OMe-C <sub>6</sub> H <sub>3</sub>	Ph	Me	4o	1	95	121-122	58
16	4-Me-C <sub>6</sub> H <sub>4</sub>	Ph	Me	4p	1	98	113-115	41
17	4-Me-C <sub>6</sub> H <sub>4</sub>	Ph	Et	4q	10	92	63-65	59
18	3-NO <sub>2</sub> -C <sub>6</sub> H <sub>4</sub>	3-NO <sub>2</sub> -C <sub>6</sub> H <sub>4</sub>	Et	4r	30	86	158-159	49
19	Ph	4-Cl-C <sub>6</sub> H <sub>4</sub>	Me	4s	3	93	88-89	50
20	4-NO <sub>2</sub> -C <sub>6</sub> H <sub>4</sub>	4-OMe-C <sub>6</sub> H <sub>4</sub>	Me	4t	15	90	117-119	50
21	4-OMe-C <sub>6</sub> H <sub>4</sub>	4-OMe-C <sub>6</sub> H <sub>4</sub>	Me	4u	8	85	184-185	51
22	4-F-C <sub>6</sub> H <sub>4</sub>	Ph	Et	4v	22	93	79-80	47
23	2,5-di-OMe-C <sub>6</sub> H <sub>3</sub>	4-Cl-C <sub>6</sub> H <sub>4</sub>	Me	4w	1	90	111-113	54
24	2-Me-C <sub>6</sub> H <sub>4</sub>	Ph	Me	4x	1	94	132-134	57
25	3-Cl-C <sub>6</sub> H <sub>4</sub>	Ph	Me	4ay	2	95	98-100	48
26	4-OH-3-OMe-C <sub>6</sub> H <sub>3</sub>	Ph	Me	4z	33	83	110-112	56
27	4-OH-3-OMe-C <sub>6</sub> H <sub>3</sub>	4-Me-C <sub>6</sub> H <sub>4</sub>	Me	4aa	40	81	104-107	56
28	2,4-di-OMe-C <sub>6</sub> H <sub>3</sub>	2-CN-C <sub>6</sub> H <sub>4</sub>	Me	4ab	38	75	88-90	56
29	3-NO <sub>2</sub> -C <sub>6</sub> H <sub>4</sub>	Ph	Et	4ac	10	98	95-97	47
30	4-OMe-C <sub>6</sub> H <sub>4</sub>	3-NO <sub>2</sub> -C <sub>6</sub> H <sub>4</sub>	Et	4ad	26	86	152-153	49
31	4-OMe-C <sub>6</sub> H <sub>4</sub>	4-F-C <sub>6</sub> H <sub>4</sub>	Et	4ae	20	94	52-53	45
32	2,6-di-Cl-C <sub>6</sub> H <sub>3</sub>	4-Br-C <sub>6</sub> H <sub>4</sub>	Me	4af	2	90	94-96	60
33	2,3-di-OMe-C <sub>6</sub> H <sub>3</sub>	4-Br-C <sub>6</sub> H <sub>4</sub>	Me	4ag	1	97	135-136	60
34	4-F-C <sub>6</sub> H <sub>4</sub>	4-Cl-C <sub>6</sub> H <sub>4</sub>	Me	4ah	30	90	102-103	C

<sup>a</sup> Yields refer to the pure isolated products. <sup>b</sup> All known products have been reported previously in the literature and were characterized by comparison of IR and NMR spectra with authentic samples. <sup>c</sup> The new compound synthesized in this work.

**Table 3:** Comparison of 2,3-dibromosuccinic acid with previously reported catalyst for the synthesis of  $\alpha$ -aminophosphonate 4c.

Entry	Catalyst	Conditions	Time	Yield (%)	Ref.
1	Boric acid	Solvent-free/ rt	15 min	96	41
2	CoCl <sub>2</sub> .6H <sub>2</sub> O	Solvent-free/ rt	10 min	95	42
3	Oxalic acid	Solvent-free/ 50 °C	2h	91	37
4	Al(H <sub>2</sub> PO <sub>4</sub> ) <sub>3</sub>	Solvent-free/ 100 °C	12 min	95	58
5	PPA-SiO <sub>2</sub>	Solvent-free/ 80 °C	20 min	95	57
6	H <sub>3</sub> PW <sub>12</sub> O <sub>40</sub>	CH <sub>2</sub> Cl <sub>2</sub> / rt	<15 min	92	35
7	PhNMe <sub>3</sub> Cl	CH <sub>2</sub> Cl <sub>2</sub> / 40 °C	3 h	74	43
8	2,3-Dibromosuccinic acid	Solvent-free/ rt	1 min	97	This work

**Scheme 2:** Plausible mechanism for the synthesis of  $\alpha$ -aminophosphonates 4a-4ah.

## Experimental

### Apparatus and analysis:

Melting points and IR spectra were measured on an Electrothermal 9100 apparatus and a JASCO FT/IR-460 plus spectrometer, respectively. The <sup>1</sup>H, <sup>13</sup>C, and <sup>31</sup>P NMR spectra were obtained on Bruker DRX-400 Avance instruments with CDCl<sub>3</sub> and Acetone-*d*<sub>6</sub> 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 $\alpha$ -Aminophosphonates (4a-4ah):

The mixture of aldehyde (1 mmol), amine (1 mmol), and 2,3-dibromosuccinic acid (1 mol%) were stirred for a few minutes. Then trimethyl / triethylphosphite (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 soluble in water and was removed from the reaction mixture. The crude product was washed with the mixture of *n*-hexane/Et<sub>2</sub>O (5/2) to give pure products.

### Spectral data for the synthesized compounds (4a, 4c, 4aa and 4ah):

Compound **4a**: white solid, mp. 91-93 °C; FT-IR (KBr)  $\nu$ : 3294 (NH); 1236 (P=O); <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>):  $\delta_{\text{H}}$  3.51 (3H, d, <sup>3</sup>J<sub>PH</sub> = 10.4 Hz, P-OCH<sub>3</sub>), 3.80 (3H, d, <sup>3</sup>J<sub>PH</sub> = 10.4 Hz, P-OCH<sub>3</sub>), 4.83 (1H, br, NH), 4.84 (1H, d, <sup>2</sup>J<sub>PH</sub> = 24.8 Hz, CHP), 6.64 (2H, d, *J* = 7.6 Hz, ArH), 6.74 (1H, t, *J* = 7.4 Hz, ArH), 7.14 (2H, t, *J* = 8.0 Hz, ArH), 7.31 (1H, t, *J* = 7.8 Hz, ArH), 7.38 (2H, t, *J* = 7.6 Hz, ArH), 7.52 (2H, d, *J* = 8.0 Hz, ArH).

Compound **4c**: white solid, mp. 137-140 °C; FT-IR (KBr)  $\nu$ : 3310 (NH); 1232 (P=O); <sup>1</sup>H NMR (400 MHz,

Acetone- $d_6$ ):  $\delta$ : 3.57 (3H, d,  $^3J_{\text{PH}} = 10.8$  Hz, P-OCH<sub>3</sub>), 3.76 (3H, d,  $^3J_{\text{PH}} = 10.4$  Hz, P-OCH<sub>3</sub>), 5.10 (1H, d,  $^2J_{\text{PH}} = 25.2$  Hz, CHP), 5.76 (1H, br, NH), 6.63 (1H, t,  $J = 7.2$  Hz, ArH), 6.81 (2H, d,  $J = 8.0$  Hz, ArH), 7.08 (2H, t,  $J = 7.8$  Hz, ArH), 7.39 (2H, d,  $J = 8.0$  Hz, ArH), 7.62 (2H, d,  $J = 8.4$  Hz, ArH).

Compound **4aa**: white solid, mp. 104-107 °C; FT-IR (KBr)  $\nu$ : 3365 (NH), 3260 (br, OH), 1265 (P=O);  $^1\text{H NMR}$  (CDCl<sub>3</sub>, 400 MHz)  $\delta$ : 2.22 (s, 3H, ArMe), 3.53, 3.79 (2d, 6H,  $J_{\text{HP}} = 10.4$  Hz, P(OMe)<sub>2</sub>), 3.88 (s, 3H, OMe), 4.71 (d, 1H,  $J_{\text{HP}} = 22.4$  Hz, P-C-H), 4.50, 5.75 (2br, 2H, NH, OH), 6.55 (d, 2H,  $J_{\text{HH}} = 8.0$ , H<sub>Ar</sub>), 6.90 (d, 1H,  $J_{\text{HH}} = 8.0$  Hz, H<sub>Ar</sub>), 6.95 (d, 2H,  $J_{\text{HH}} = 8.0$  Hz, H<sub>Ar</sub>), 6.96 (s, 1H, H<sub>Ar</sub>), 7.02 (s, 1H, H<sub>Ar</sub>).

Compound **4ah**: white solid, mp. 102-103 °C; FT-IR (KBr)  $\nu$ : 3301 (NH); 1234 (P=O);  $^1\text{H NMR}$  (CDCl<sub>3</sub>, 400 MHz)  $\delta$ : 3.54, 3.87 (2d, 6H,  $J_{\text{HP}} = 10.4$  Hz, P(OMe)<sub>2</sub>), 4.64 (br, H, NH), 5.18 (d, 1H,  $J_{\text{HP}} = 24.8$  Hz, P-C-H), 6.57 (d, 2H,  $J_{\text{HH}} = 8.0$ , H<sub>Ar</sub>), 7.08-7.32 (m, 5H, H<sub>Ar</sub>), 7.52 (t, 1H,  $J_{\text{HH}} = 8.0$  Hz, H<sub>Ar</sub>);  $^{13}\text{C NMR}$  (CDCl<sub>3</sub>, 100MHz)  $\delta$ : 47.8 (d,  $J_{\text{CP}} = 156.0$  Hz, P-CH), 53.9, 54.0 (2d,  $J_{\text{CP}} = 7.0$  Hz, 2P(OMe)<sub>2</sub>), 114.8 (s, C<sub>Ar</sub>), 115.4 (dd,  $J_{\text{CF}} = 20.0$  Hz, C<sub>Ar</sub>), 122.8 (d,  $J_{\text{CF}} = 13.0$  Hz, C<sub>Ar</sub>), 129.1 (s, C<sub>Ar</sub>), 129.9 (d,  $J_{\text{CP}} = 8.0$  Hz, C<sub>Ar</sub>), 140.3 (d,  $J_{\text{CP}} = 15.0$  Hz, C<sub>Ar</sub>), 160.6 (d,  $J_{\text{CF}} = 244.0$  Hz, C<sub>Ar</sub>);  $^{31}\text{P NMR}$  (CDCl<sub>3</sub>, 162 MHz)  $\delta$ : 24.08; MS (EI, 70 eV)  $m/z$  (%): 345 (M<sup>+</sup>+2, 7), 343 (M<sup>+</sup>, 20), 234 (100), 110 (6); Anal. calcd for C<sub>15</sub>H<sub>16</sub>ClFNO<sub>3</sub>P: C, 52.42; H, 4.69; N, 4.08; found C, 52.57; H, 4.86; N, 4.16.

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