$\begin{array}{l} CeCl_{3}\cdot 7H_{2}O\text{-}Catalyzed \ One-Pot\\ Kabachnik-Fields \ Reaction: \ A \ Green \ Protocol\\ for \ Three-Component \ Synthesis \ of\\ \alpha\text{-}Aminophosphonates \end{array}$

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ABSTRACT: $CeCl_3 \cdot 7H_2O$ has been utilized as an efficient Lewis acid catalyst for the three-component coupling of aldehydes, aromatic amines, and diethylphosphite to produce α -aminophosphonates under solvent-free conditions. The advantages of this protocol are high yield, mild reaction conditions, less environmental pollution, and simple work-up procedure. © 2010 Wiley Periodicals, Inc. Heteroatom Chem 21:397–403, 2010; View this article online at wileyonlinelibrary.com. DOI 10.1002/hc.20635

INTRODUCTION

Phosphonate-containing molecules are biologically potent. Their diverse applications include as inhibitors of synthase [1], HIV protease [2], rennin [3], PTPases [4–6], antibiotics [5], enzymes [6], UDP-galactopyranose mutase [6a], and herbicides [7] and as surrogates of α -amino acids [8]. α -Aminophosphonates have attracted attention as substrates in the synthesis of phosphonopeptides (peptido mimetics) [9]. However, there are also a few reports available in which antitumor activity of α -aminophosphonates has been investigated [10].

Generally, α -aminophosphonates are prepared by the addition of phosphorous nucleophiles to imines in the presence of acids [11]. Recently, three-component synthesis starting from aldehydes, amines, and diethylphosphite or triethylphosphite has been reported by using Lewis and Brønsted acid catalysts such as LiClO₄ [12], InCl₃ [13a], FeCl₃ [13b], ZrCl₄ [13c], ZrOCl₂·8H₂O [13d], lanthanide triflates/magnesium sulfate [14], SbCl₃/Al₂O₃ [15a], TaCl₅-SiO₂ [15b], BiNO₃·5H₂O [15c], amberlyst-15 [16a], montmorillonite clay-MW [16b], silica sulfuric acid [16c], sulfamic acid [16d], Al₂O₃-MW [17a], TiO₂ [17b], CF₃CO₂H [18], Sc(DS)₃ [19a], β-cyclodextrine [19b], SDS [19c], BF₃·Et₂O [20], $M(OTf)_n$ [21], $M(ClO_4)_n$ [22], in an ionic liquid [23] and under thermal conditions [24]. By considering the importance of α -aminophosphonates as biologically active compounds, there is a great demand for the development of more convenient and practically efficient methods for the synthesis of α aminophosphonates.

Cerium(III) chloride heptahydrate [25] (CeCl₃·7H₂O) has emerged as a potential catalyst in affecting various organic transformations due to its high catalytic ability, water tolerance, being a "friendly" reagent, economic viability, and easy availability. To our knowledge, no Kabachnik– Fields reaction catalyzed by CeCl₃·7H₂O has been reported. In this paper, as a part of our ongoing green organic chemistry program [26], we wish to report an efficient and versatile procedure for

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SCHEME 1

affecting the one-pot, three-component reaction of an aldehyde, an amine, and diethylphosphite for the preparation of α -aminophosphonates in the presence of catalytic amounts of the CeCl₃·7H₂O under solvent-free conditions (Scheme 1).

RESULTS AND DISCUSSION

At the outset, the three-component reaction of benzaldehyde, aniline, and diethylphosphite was investigated in various conditions at room temperature (Table 1). To optimize the reaction conditions, the model reaction was examined under solvent-free conditions and in solvents such as CH₃CN, acetone, CH₂Cl₂, and EtOH in the presence of cerium salts. The best reaction condition resulted when the molar ratio of benzaldehyde (2 mmol)/aniline (2 mmol)/diethylphosphate (2.2 mmol)/CeCl₃·7H₂O (5 mol%) was used at room temperature under solvent-free conditions. In these conditions, the reaction proceeded well and was completed after 5 h to produce the desired diethyl phenyl(phenylamino)methylphosphonate in 95% isolated yield. To show the merit of the catalyst, a similar reaction in the absence of CeCl₃·7H₂O catalyst was studied (Table 1, entry 1). The reaction did not proceed even after 24 h.

 TABLE 1
 Optimization of Reaction Conditions^a

Entry	Solvent	Catalyst (mol%)	Time (h)	Yield (%)
1	CH₃CN	_	24	<10
2	CH ₃ CN	Ce(NO ₃) ₃	24	70
3	CH ₃ CN	(NH ₄)Ce(NO ₃) ₆	14	94
4	CH ₃ CN	CeCl ₃ ·7H ₂ O (5)	12	93
5	CH ₃ CN	CeCl ₃ ·7H ₂ O +	12	95
	-	Nal (5)		
6	CH₃CN	CeCl ₃ ·7H ₂ O +	24	80
	-	Nal/Al ₂ O ₃ (5)		
7	Acetone	CeCl ₃ ·7H ₂ O (5)	10	94
8	CH ₂ Cl ₂	CeCl ₃ ·7H ₂ O (5)	24	70
9	Ethanol	CeCl ₃ ·7H ₂ O (5)	24	70
10	Solvent-free	CeCl ₃ ·7H ₂ O (5)	5	95
11	Solvent-free	CeCl ₃ ·7H ₂ O (2.5)	8	92

^aReaction conditions: solvent (0.5 mL), room temperature, benzaldehyde (2 mmol), aniline (2 mmol), and diethylphosphate (2.2 mmol). Then, to show the general application of the method [25], several aldehydes, amines, and diethylphosphate with similar molar ratios as mentioned earlier were reacted smoothly in appropriate reaction times (2.5–24 h) to yield the corresponding α -aminophosphonates in high to excellent isolated yields (87–95%). The examples studied covered electron-rich and electron- deficient aromatic aldehydes with aniline derivatives containing strong electron-donating and electronwithdrawing groups and also show that cyclohexanone as a ketone reacts smoothly to give the corresponding α -aminophosphonate. The results of this study are summarized in Table 2.

A reasonable pathway for the reaction of an aldehyde with an amine and diethylphosphite conducted in the presence of CeCl₃·7H₂O is presented in Fig. 1. The reaction proceeds via formation of imine that undergoes addition with diethylphosphite to afford α -aminophosphonates. CeCl₃·7H₂O shows dual activity: One action is to activate the in situ generated imine, and its second function is to bring diethylphosphite molecule close to the imine by the template effect.

Finally, the efficacy of $CeCl_3 \cdot 7H_2O$ was compared with that of other catalysts reported earlier



FIGURE 1 A plausible mechanism for the CeCl3·7H₂O catalyzed three-component reaction for the preparation of α aminophosphonates.

Entry	Aldehyde	Amine	Product ^b	Time (h)	Yield (%)	mp Found (Reported)	Reference
1	Н	NH2	HIN PO(OE1)2	5	95	87 (86)	[17b]
2	MeO H OMe	NH2	HN H3CO OCH3 H3CO H3CO H3CO H3CO H1 HN PO(OEt)2 H3CO H3 HN PO(OEt)2 H3 HN HN PO(OEt)2 H3 HN HN HN H3 HN HN HN HN HA HA HA HA HA HA HA HA HA HA HA HA HA	2.5	94	77	-
3	OMe O H OMe	NH2	OMe HN PO(OEt) ₂	5	92	120	-
4	MeO H	NH2	HN PO(OEt) ₂	4.5	91	108	[12]
5	CI	NH2	HN PO(OEt) ₂	9	90	57 (57)	[17b]
6	O CI	NH2	Cl HN PO(OEt) ₂	14	88	89 (88)	[17b]
7	ОН	NH2	OH HN PO(OEt) ₂	8	94	91	[15b]
8	F	NH2	HN PO(OEt) ₂	20 (9) ^c	94 (95) ^c	79	[23]

TABLE 2 One-Pot Synthesis of α -Aminophosphonates Catalyzed by CeCl₃·7H₂O at Room Temperature under Solvent-Free Conditions^{*a*}

(Continued)

TABLE 2 Continued

Entry	Aldehyde	Amine	Product ^b	Time (h)	Yield (%)	mp Found (Reported)	Reference
10	O ₂ N H	NH2	HN PO(OEt) ₂	13	90	120 (120)	[17b]
11	NC H	NH2	HN PO(OEt) ₂	10.5	89	105	[15c]
12	0 H	CI NH2	HN PO(OEt) ₂	9	93	119	[23]
13	0 H	Cl NH ₂	HN PO(OEt) ₂	5	95	129	[17b]
14	O H	Br NH ₂	HN PO(OEt) ₂	8	92	122	-
15	O H	MeO NH2	HN PO(OEt) ₂	5	92	115	[15b]
17	Н	OH NH ₂	HO HN PO(OEt) ₂	10	90	120	[17b]
18	Н	HO NH2	HN PO(OEt) ₂	8	93	127	[17b]

(Continued)



TABLE 2 Continued

^aThe ratio of aldehyde (2 mmol), amine (2 mmol), and diethylphosphate (2.2 mmol). ^bProducts were confirmed by physical and spectroscopic methods such as mp, IR, and NMR.

°10 mol% CeCl₃·7H₂O was used.

TABLE 3 Comparison of the Reaction of Benzaldehyde, Aniline, Diethylphosphate Catalyzed by CeCl₃·7H₂O with the Other Catalysts Recently Used for This Reaction

Entry	Catalyst (mol%)	Solvent	Temperature (° C)	Time (h)	Yield (%)	References
1	CF3CO2H (25)	_	r.t.	24	96	[18]
2	ŽrCl₄ (10)	CH ₃ CN	r.t.	4.5	92	[13c]
3	InCl ₃ (10)	ТŇF	r.t.	11	93	[13a]
4	TiO ₂ (20)	_	50	2.5	98	[17a]
5	[bmim]BF ₆ (1 mL)	_	r.t.	8	84	[23]
6	Bi(NO ₃) ₃ ,5H ₂ O (10)	_	r.t.	10	93	[15c]
7	SbCl _{3/} Al ₂ Ō ₃ (5)	CH ₃ CN	r.t.	3	91	[15a]
8	$TaCl_5 - SiO_2$ (10)	CH ₂ Cl ₂	r.t.	20	90	[15b]
9			80	0.5	92	[24a]
10	_	_	r.t.	24	N.R.	[23], [16d]
11	CeCl ₃ ·7H ₂ O (5)	_	r.t.	5	95	This work

r.t., room temperature; N.R., no reaction.

in the synthesis of α -aminophosphonates. The data summarized in Table 3 clearly demonstrate the superiority of CeCl₃·7H₂O in terms of time, cost, as well as operational simplicity of the reaction.

CONCLUSION

In conclusion, the results reported here show that $CeCl_3 \cdot 7H_2O$ efficiently catalyzes the one-pot synthesis of biologically active α -aminophosphonates in excellent yields via a three-component coupling reaction of aldehydes, amines, and diethylphosphite at room temperature. This protocol has advantages

of mild reaction conditions, no environmental pollution, avoiding use of any harmful organic solvent in reaction media and/or in work-up procedure, use of an inexpensive, water-tolerant, and environment-friendly catalyst, and simple work-up procedure.

EXPERIMENTAL

General Remarks

Merck and Fluka chemicals were purchased from Kimia Exir Chemical Co., Tehran, Iran. All the products are known and were characterized by comparison of their physical data with those reported in the literature. IR spectra were run on a Shimadzu model 8300 FT-IR spectrophotometer. NMR spectra were recorded on a Bruker Avance DPX-250 or 500 MHz. The purity of the products and the progress of the reactions were accomplished by thinlayer chromatography (TLC) on silica-gel polygram SILG/UV₂₅₄ plates.

General Procedure for Preparation of α -Aminophosphonates

To a mixture of aldehyde (2 mmol), amine (2 mmol), and diethyl phosphite (2.2 mmol), CeCl₃·7H₂O (0.02 g, 5 mol%) was added and stirred at room temperature for the appropriate reaction time (Table 2). After completion of the reaction (TLC), the mixture was dissolved in hot EtOH (2 mL) and the product was precipitated by adding ice water. The pure α -aminophosphonates were collected with a simple filtration and consequently washed with water (Table 2).

Selected Spectral Data for New Compounds

[(3, 4 - Dimethoxy-phenyl) - phenylamino - methyl] phosphonic acid diethyl ester (Table 2, entry 2): white solid, mp 77–78°C; ¹H NMR (500 MHz, CDCl₃): $\delta = 7.12$ (dd, $J_1 = 8.4$, $J_2 = 7.5$, 2H), 7.05–7.02 (m, 2H), 6.84 (d, J = 7.9, 1H), 6.71 (t, J = 7.5, 1H), 6.63 (d, J = 8.1, 2H), 4.85 (b, 1H, NH), 4.72 (d, J = 23.8, 1H), 4.16–4.10 (m, 2H), 3.99–3.95 (m, 1H), 3.88 (s, 3H), 3.86 (s, 3H), 3.75-3.70 (m, 1H), 1.30 (t, J = 7.1, 3H), 1.16 (t, J = 7.1, 3H); ¹³C NMR (125 MHz, CDCl₃): $\delta = 149.5$, 149.1, 146.9, 129.6, 128.6, 120.6, 118.8, 114.3, 111.5, 111.3, 63.6 (d, J = 6.9), 63.6 (d, J = 6.9), 56.3, 56.2, 56.2 (d, J =151.0), 16.8 (d, J = 5.6, CH₃), 16.7 (d, J = 5.6, CH₃); IR = 3284, 2995, 1602, 1515, 1498, 1230, 1020cm⁻¹; C₁₉H₂₆NO₅P (379): calcd C 60.15, H 6.91; found C 60.03, H 6.90. [(2,5-Dimethoxy-phenyl)phenylamino-methyl]-phosphonic acid diethyl ester (Table 2, entry 3): white solid, mp $120-121^{\circ}$ C; ¹H NMR (500 MHz, CDCl₃): $\delta = 7.16-7.12$ (m, 3H), 6.86 (d, J = 8.9, 1H), 6.8 (dt, $J_1 = 8.9$, $J_2 = 2.4$, 1H), 6.73–6.70 (m, 3H), 5.47 (d, J = 25.1, 1H), 5.1 (b, 1H, NH), 4.28-4.24 (m, 2H), 3.99-3.97 (m, 1H), 3.93 (s, 3H), 3.76 (s, 3H), 3.74-3.68 (m, 1H), 1.34 (t, J = 7.1, 3H), 1.10 (t, J = 7.1, 3H); ¹³C NMR (125 MHz, CDCl₃): $\delta = 154.4$, 151.9, 146.7, 146.6, 129.5, 125.6, 118.7, 114.8, 114.2, 114.1, 112.1, 64.2, 56.8, 56.2, 48.5 (d, J = 155.1), 16.8 (d, J = 6.0), 16.5 (d, J = 6.0); IR = 3304, 2984, 2832, 1604, 1500, 1232, 1026 cm⁻¹; C₁₉H₂₆NO₅P (379): calcd C 60.15, H 6.91; found C 60.08, H 6.88. Diethyl (4-bromophenylamino)(phenyl)methylphosphonate

(Table 2, entry 14): white solid, mp 122°C; ¹H NMR $(500 \text{ MHz}, \text{CDCl}_3)$: $\delta = 7.47 \text{ (d, } J = 7.2, 2\text{H}), 7.37 \text{ (d, } J = 7.2, 3\text{H}), 7.37 \text{ (d, } J = 7.2, 3\text{H}), 7.37 \text{ (d, } J = 7.2, 3\text{H}$ J = 7.2, 2H, 7.31 (t, J = 7.2, 1H), 7.20 (d, J = 8.5, 1H) 2H), 6.50 (d, J = 8.5, 2H), 4.92 (b, 1H, NH), 4.74 (d, J = 23.2, 1H), 4.31-4.28 (m, 2H), 3.99-3.97 (m, 2H)1H), 3.69-3.70 (m, 1H), 1.32 (t, J = 7.0, 3H), 1.14(t, J = 7.0, 3H) ppm; ¹³C NMR (125 MHz, CDCl₃): $\delta = 145.7, 135.8, 132.3, 129.1, 128.5, 128.2, 115.9,$ 110.6, 63.9, 63.7, 56.6 (d, *J* = 150.5), 16.8, 16.6 ppm; IR (KBr): $\tilde{\nu} = 3388$, 2983,1615, 1516, 1235 cm⁻¹; C₁₇H₂₁BrNO₃P (398): calcd C 51.27, H 5.32; found C 51.24, H 5.30. [(4-Cyano-phenylamino)-phenylmethyl]-phosphonic acid diethyl ester (Table 2, entry 20): white solid, mp 130–131°C; ¹H NMR (500 MHz, CDCl₃): δ = 7.49 (d, J = 7.5, 2H), 7.39–7.31(m, 5H), 6.64 (d, J = 8.6, 2H), 5.78 (b, 1H, NH), 4.80 (d, J = 24.1, 1H), 4.19–4.12 (m, 2H), 3.97–3.92 (m, 1H), 3.69-3.64 (m, 1H), 1.33 (t, J = 7.0, 3H), 1.13(t, J = 7.0, 3H) ppm; ¹³C NMR (125 MHz, CDCl₃): $\delta = 150.3, 125.2, 134, 129.2, 128.8, 128.2, 120.5,$ 113.9, 100.5, 64.1 (d, J = 6.9), 63.7 (d, J = 6.9), 56.4 (d, J = 150.5), 16.8 (d, J = 5.6), 16.5 (d, J = 5.6) ppm; IR (KBr): $\tilde{\nu} = 3306$, 2986, 2211, 1604, 1527, 1233, 1026 cm⁻¹; C₁₈H₂₁N₂O₃P (344): calcd C 62.78, H 6.15; found C 62.68, H 6.12.

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