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ORIGINAL PAPER

TfOH/C-catalyzed one-pot three-component synthesis of α-amino phosphonates under solvent-free conditions

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Abstract The TfOH/C was readily prepared via simple absorption of triflic acid onto activated charcoal. This solid acid was used as an efficient catalyst for the synthesis of α-aminophosphonates through the Kabachnik–Fields reaction of carbonyl compound, amine and diethyl phosphite under solvent-free conditions.

Keywords α-Aminophosphonates · Kabachnik–Fields reaction · Three-component synthesis · Solvent free · Triflic acid supported on carbon

Introduction

Solid-supported acids are unique catalysts that have become popular over the last two decades. The activity and selectivity of a catalyst dispersed on the surface of a support are improved, as its effective surface area is increased significantly and hence is more effective than the individual acidic catalysts [1, 2]. Low toxicity, moisture resistance, air tolerance and low prices are other common features that make the use of solid-supported reagents attractive alternatives to conventional Lewis acids or metal triflates.

Triflic acid (TfOH) absorbed on solid inorganic or organic support has received considerable attention as an inexpensive, recyclable, easily handled, low toxic, non-corrosive and free-flowing powder with superior thermal and mechanical stability under catalytic conditions [3–10]. To the best of our knowledge, there has been no report on the use of unsupported and supported TfOH as catalyst to promote the synthesis of α-aminophosphonates via the Kabachnik–Fields reaction.

From organic chemistry to biology, molecules containing phosphonate moiety have attracted the interest of organic chemists for their structural modifications and synthetic developments [11–13]. α-Aminophosphonates are important in medicinal chemistry, since they are considered as structural analogs of the corresponding α-amino acids and their utilities as enzyme inhibitors, antibiotics, pharmacological agents and peptidomimetics which are well documented [14–16]. Among the various synthetic approaches for their preparation, the Kabachnik–Fields reaction is established as the most useful method. This one-pot procedure has been carried out using $InCl_3$ [17], $In(OTf)_3/MgSO_4$ [18], GaI_3 [19], $BiCl_3$ [20], I_2 [21], $SbCl_3/Al_2O_3$ [22], metal perchlorates [23–27], metal triflates [28], $CeCl_3$ [29], $Na_2CaP_2O_7$ [30], Sulfamic acid [31], TFA [32], (bromodimethyl)sulfonium bromide [33], TsCl [34], Amberlyst-15 [35], $Cd(ClO_4)_2.xH_2O$ [36], ZnO nanoparticle [37], $NaHSO_4–SiO_2$ [38] and acidic ionic liquids [39]. A few catalyst-free as well as solvent-free protocols have been developed which necessitates thermal [40–42], ultrasonic [43] or MW [44, 45] activation. However, some of the reported procedures are associated with disadvantages like costly and moisture-sensitive catalysts, use of trialkylphosphite as phosphorus nucleophile with a nasty smell, use of additional reagents and long reaction times. Furthermore, in all of the reported methods, a non-environmental-friendly organic solvent was uses as reaction media and/or for workup. Keeping in view the increasing importance of α-aminophosphonates in pharmaceutical/

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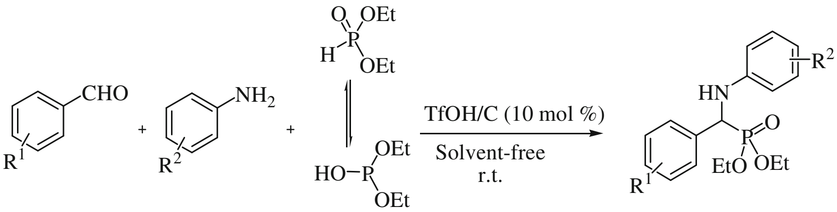
Scheme 1 TfOH/C catalyzed synthesis of α-aminophosphonates



industrial chemistry, there is a need to develop their synthesis. Therefore, in continuation of our researches on developing environmentally benign methodologies [29, 46–48], herein we describe a convenient and simple procedure via a one-pot reaction of aldehydes, amines and diethylphosphonate for the preparation of α-aminophosphonates using trifluoromethanesulfonic acid supported on carbon (TfOH/C) under solvent-free conditions as a reusable and efficient solid catalyst (Scheme 1).

Results and discussion

At first, a one-pot three-component reaction of benzaldehyde (2.0 mmol), aniline (2.0 mmol) and diethylphosphite (2.5 mmol) was studied as a model reaction in the presence of supported TfOH catalyst at room temperature. As the results show, 10 mol% of TfOH/C was the most effective catalyst for this reaction, which led to the production of the desired diethyl phenyl (phenyl amino)methyl phosphonate in 97 % isolated yield after 3 h. In the presence of solvents, the product was achieved in low yield overnight (Table 1, entries 5–7). To show the merit of the supported catalyst, a similar reaction was studied in the presence of unsupported TfOH and charcoal and in the absence of any catalyst (Table 1, entries 8–10). These results showed the strong synergistic effect of charcoal on the catalytic activity of TfOH in this reaction (Table 1).

Under the optimized conditions, the scope of this method was examined for various aromatic aldehydes and amines. By this method, the corresponding α-amino phosphonates were achieved in high to excellent yields (85–98 %).

Table 1 Optimization of the reaction conditions

Entry	Solvent	Catalyst (mol%)	Time (h)	Conversion (%)
1	–	PEG-SO$_3$H (5)	6	40
2	–	TfOH/SiO$_2$ (5)	6	80
3	–	TfOH/C (5)	6	98
4	–	TfOH/C (10)	3	100
5	CH$_3$CN	TfOH/C (10)	24	70
6	CH$_2$Cl$_2$	TfOH/C (10)	24	30
7	Ethanol	TfOH/C (10)	24	60
8	–	TfOH (10)	6	80
9	–	Activated charcoal	24	Trace
10	–	–	24	N.R. [31, 54]

When aldehydes with electron-donating groups and aniline with electron-withdrawing groups were used, the reaction was performed with shorter reaction times and higher yields (Table 2, entries 5, 6, 11, 12, 13, 14, 16, 18 and 19). In addition, new diethyl(3-methylphenyl)(phenylamino) methyl phosphonate (3c), diethyl (3,4-dihydroxyphenyl)(phenylamino)methyl phosphonate (3h) and diethyl(4-bromophenylamino) (3,4 dimethoxyphenyl) methyl phosphonate (3m) were produced under the same conditions to show the selectivity and the scope of this green procedure. In all cases, after the reaction completion, the reaction mixture was dissolved in ethanol. The catalyst was filtered off and the pure product was isolated by adding ice-water to stirring residue and simple filtration. Thus the workup procedure is very simple and the use of choromatography and harmful organic solvents for isolation and purification of products is not necessary. Cyclohexanone as a ketone was also coupled with aniline and diethylphosphite to produce the corresponding α-aminophosphonates in 70 % isolated yield after 10 h.

Furthermore, the possibility of this method for preparation of bis(α-aminophosphonates) was investigated. Thus, the reaction of 4,4′-methylenedianiline (1.0 mmol) with benzaldehyde (2.0 mmol) and diethyl phosphite (2.5 mmol) was completed in 5 h to produce the desired bis(α-aminophosphonate)(3u) in 90 % yield (Scheme 2).

To show the efficiency of this protocol for Kabachnik–Fields reaction, the catalytic activity of TfOH/C with some of the other recent catalysts was compared. As demonstrated in Table 3, TfOH/C is an effective catalyst for performing this reaction at ambient temperature and under solvent-free conditions.

In conclusion, we have succeeded in developing TfOH/C as an effective heterogeneous TfOH catalyst for one-pot three-component reaction of aldehyde, amine and diethylphosphite. This novel catalyst provides a clean and convenient alternative for the Kabachnik–Fields reaction in view of the following advantages. The reaction proceeds smoothly in the presence of moisture under solvent-free condition and produces α-aminophosphonate in high to excellent yield at room temperature. The catalyst is stable and a non-polluting solid that offers easy handling and ready workup. In addition, no harmful organic solvent was used as reaction media or in the workup procedure.

Table 2 TfOH/C-catalyzed synthesis of α-amino phosphonates under solvent-free conditions at room temperature

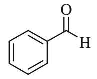
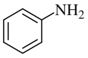
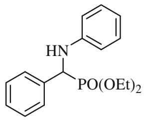
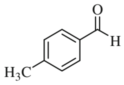
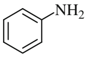
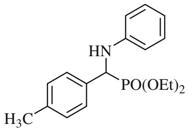
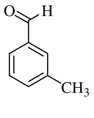
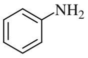
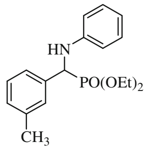
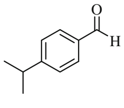
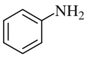
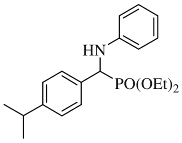
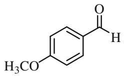
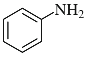
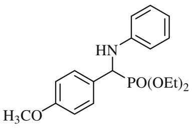
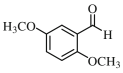
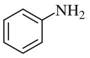
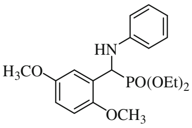
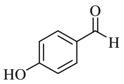
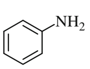
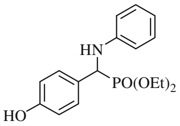
Entry	Aldehyde	Amine	Product	Time (h)	Yield (%)	M. P.	Ref.
1	 1a	 2a	 3a	3	97	88-89 (87-89)	[49]
2	 1b	 2a	 3b	5	95	99-100 (64-65)	[50]
3	 1c	 2a	 3c	5	90	80-82	
4	 1d	 2a	 3d	3	90	121	[51]
5	 1e	 2a	 3e	2.25	94	104-105 (102-103)	[52]
6	 1f	 2a	 3f	1.5	90	126-127	[29]
7	 1g	 2a	 3g	4	96	88-89	[53]

Table 2 continued

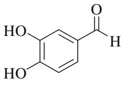
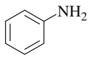
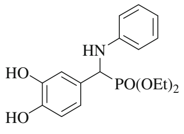
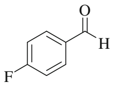
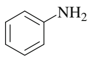
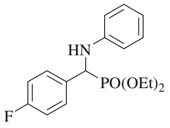
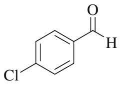
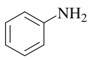
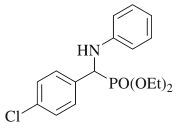
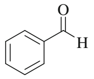
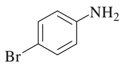
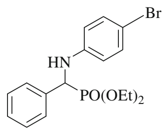
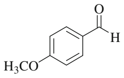
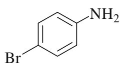
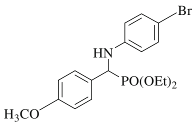
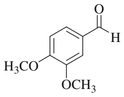
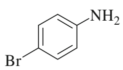
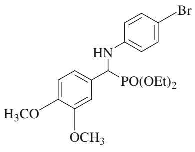
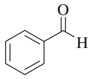
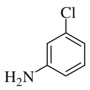
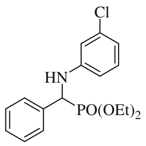
Entry	Aldehyde	Amine	Product	Time (h)	Yield (%)	M. P.	Ref.
8	 1h	 2a	 3h	6	85	137-138	
9	 1i	 2a	 3i	9	85	84-85 (78-79)	[54]
10	 1j	 2a	 3j	4.5	87	68-69 (58-59)	[53]
11	 1a	 2b	 3k	2	93	123-124 (121-123)	[52]
12	 1e	 2b	 3l	2	92	109-110 (107-108)	[41]
13	 1k	 2b	 3m	1	95	92-94	
14	 1a	 2c	 3n	1.25	98	130-131 (129)	[29]

Table 2 continued

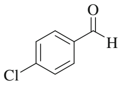
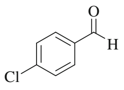
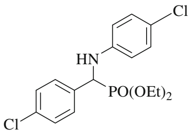
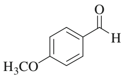
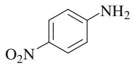
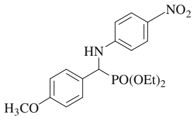
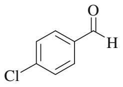
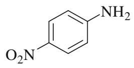
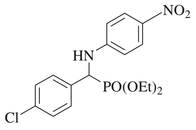
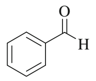
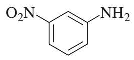
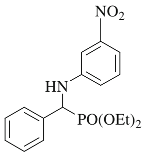
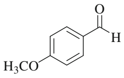
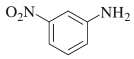
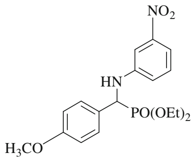
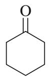
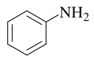
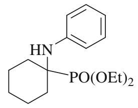
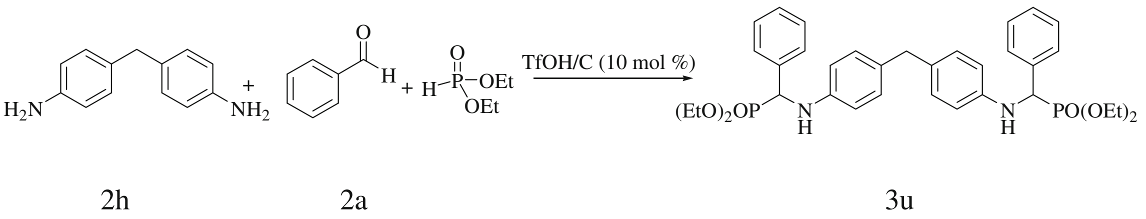
Entry	Aldehyde	Amine	Product	Time (h)	Yield (%)	M. P.	Ref.
15	 1j	 2d	 3o	3	90	123-124 (117-118)	[41]
16	 1i	 2e	 3p	2.5	93	114-116 (115)	[55]
17	 1e	 2f	 3q	10	85	138-140 (130-132)	[54]
18	 1a	 2g	 3r	1.5	89	127-128 (129-130)	[53]
19	 1e	 2g	 3s	1	86	151-152 (150-152)	[54]
20	 1l	 2a	 3t	10	70	108 (104)	[53]

Table 3 Comparison of the effect of catalysts in the reaction of benzaldehyde, aniline and diethylphosphite

Entry	Catalyst (mol%)	Solvent	Temperature (°C)	Time (h)	Yield (%)	References
1	CF_3CO_2H (25)	–	r.t.	24	96	[31]
2	β-CD (10)	H_2O	100	24	61	[56]
3	$InCl_3$ (10)	THF	r.t.	11	92	[17]
4	TiO_2 (20)	–	50	3.5	90	[53]
5	$Bi(NO_3)_3{\cdot}5H_2O$ (10)	–	r.t.	10	93	[57]
6	$TaCl_5$–SiO_2 (10)	CH_2Cl_2	r.t.	20	90	[58]
7	$FeCl_3$	THF	60	0.75	92	[59]
8	Mesoporous alumino silicate nano cage	CH_3CN	80	4	86	[60]
9	[Cu(3,4-tmtppa)]$(MeSO_4)_4$	H_2O	80	1	90	[61]
10	$SbCl_3/Al_2O_3$ (5)	CH_3CN	r.t.	3	91	[22]
11	$Al(ClO_4)_3$ (5)	–	60	12	100	[54]
12	$NbCl_5$ (5)	–	60	0.5	95	[55]
13	TfOH/C (10)	–	r.t.	3	97	This method



Scheme 2 The reaction of 4,4′-methylenedianiline with benzaldehyde and diethyl phosphite

Experimental

Preparation of carbon-supported triflic acid (TfOH/C)

The activated carbon (4.0 g) was activated by refluxing in HNO_3 (100 mL, 5.0 M) for 6 h. Activated carbon was filtered off and washed with deionized water until pH 6–7. The residue was dried at 120 °C for 12 h [62]. Then, triflic acid (3.0 mmol) was added dropwise to activated carbon (3.0 g) and the mixture was stirred magnetically for 6 h at room temperature to yield TfOH/C (1.0 mmol/g).

General procedure for the preparation of α-aminophosphonates

TfOH/C (0.20 mmol, 0.20 g) was added to a stirring mixture of benzaldehyde (2.0 mmol), aniline (2.0 mmol) and diethylphosphite (2.5 mmol) at room temperature for an appropriate time (Table 2). The progress of the reaction was monitored by TLC using hexane and ethyl acetate as eluent. After completion, the reaction mixture was dissolved in hot EtOH. The catalyst was filtered off and the ethanol volume was decreased. The pure products were solidified by adding ice water and, after a simple filtration, consequently washed with water to achieve good to excellent yields.

Selected spectral data for new compounds

Diethyl(3-methylphenyl)(phenylamino)methyl phosphonate (3c): white solid; m.p.: 80–82 °C; FT-IR: $\nu_{max} = 3{,}291$ (NH stretching), 2,984 (CH stretching), 1,603 (C=C stretching), 1,521 (NH bending), 1,499 (C=C stretching), 1,232 (P=O stretching), 1,021 (C–O stretching), 963 (C–O stretching), 744, 691 cm^{-1}; ^1H-NMR(400 MHz, $CDCl_3$): $\delta = 1.12$(t, $J = 6.6$ Hz, 3H, $OCH_2\mathbf{CH_3}$), 1.3(t, $J = 6.6$ Hz, 3H, $OCH_2\mathbf{CH_3}$), 2.34(s, 3H, Ar-CH_3), 3.63–3.69 (m, 1H, $O\mathbf{CH_2}CH_3$), 3.91–3.97 (m, 1H, $O\mathbf{CH_2}CH_3$), 4.09–4.15 (m, 2H, $O\mathbf{CH_2}CH_3$), 4.73 (d, $J = 25.2$ Hz, 1H, HC-P), 4.8 (br, 1H, NH), 6.61 (d, $J = 7.2$ Hz, 2H, Ar–H), 6.7 (t, $J = 7.2$ Hz, 1H, Ar–H), 7.07–7.14 (m, 3H, Ar–H), 7.22–7.29 (m, 3H, Ar–H) ppm; ^{13}C-NMR (100 MHz, CDCl3): $\delta = 16.21$(d, $^3J_{PC} = 5.5$ Hz), 16.46 (d, $^3J_{PC} = 5.5$ Hz), 21.5, 56 (d, $J_{CP} = 149$ Hz), 63.2 (d, $^2J_{PC} = 7.5$ Hz), 63.3 (d, $^2J_{PC} = 7.5$ Hz), 113.5, 113.8, 118.3,125, 128.5, 128.8, 129.2, 135.8, 138.2, 146.5(d, $^3J_{PC} = 14$ Hz) ppm.

Diethyl(3,4 dihydroxyphenyl)(phenylamino) methyl phosphonate(3 h): white solid; m.p.: 137–138 °C; FT-IR:

ν_{max} (KBr) = 3,443(OH stretching), 3,367 (NH stretching), 2,985 (CH stretching), 1,606 (C=C stretching), 1,517 (NH bending), 1,497 (C=C stretching), 1,209 (P=O stretching), 1,051 (C–O stretching), 977 (C–O stretching) cm^{-1}; ^1H-NMR(400 MHz, DMSO): $\delta = 1.05$(s, 3H, $OCH_2\mathbf{CH_3}$), 1.18 (s, 3H, $OCH_2\mathbf{CH_3}$), 3.67 (m, 1H, $O\mathbf{CH_2}CH_3$), 3.84–3.86 (m, 1H, $O\mathbf{CH_2}CH_3$), 4.02 (m, 2H, $O\mathbf{CH_2}CH_3$), 4.75 (dd, $J_1 = 23.6$ Hz, $J_2 = 8.8$ Hz, 1H, HN-C**H**-P), 6.09 (s, 1H, NH), 6.51 (s, 1H, Ar–H), 6.64 (d, $J = 6.4$ Hz, 1H, Ar–H), 6.75 (s, 3H, Ar–H), 6.88 (s, 1H, Ar–H), 6.99 (s, 2H, Ar–H), 8.82 (1H, OH), 8.86 (1H, OH) ppm; ^{13}C-NMR (100 MHz, DMSO): $\delta = 16.6$ (d, $^3J_{PC} =$ 5 Hz), 16.8 (d, $^3J_{PC} = 5$ Hz), 54.1 (d, $J_{CP} = 153$ Hz), 62.7 (d, $^2J_{PC} = 6$ Hz), 62.8 (d, $^2J_{PC} = 6$ Hz), 114, 115.6, 116.3, 117.2, 119.9, 127.8, 129.1, 145.3 (d, $^3J_{PC} = 14$ Hz), 147.7, 147.9 ppm.

Diethyl(4-bromophenylamino)(3,4 dimethoxyphenyl) methyl phosphonate (3m): white solid; m.p.: 92–94 °C; FT-IR: $\nu_{max} = 3,293$ (NH stretching), 2,986 (CH stretching), 1,592 (C=C stretching), 1,516 (NH bending) 1,489 (C=C stretching), 1,229 (P=O stretching), 1,143, 1,020 (C–O stretching), 973 (C–O stretching) cm^{-1}; ^1H-NMR (400 MHz, $CDCl_3$): $\delta = 1.13$ (t, $J = 7$ Hz, 3H $OCH_2\mathbf{CH_3}$,), 1.29 (t, $J = 7$ Hz, 3H, $OCH_2\mathbf{CH_3}$), 3.66–3.7 (m, 1H, $O\mathbf{CH_2}CH_3$), 3.85 (s, 3H, OCH_3), 3.86 (s, 3H, OCH_3), 3.86–3.97 (m, 1H, $O\mathbf{CH_2}CH_3$), 4.07–4.13 (m, 2H, $O\mathbf{CH_2}CH_3$), 4.62 (dd, $J_1 = 25.6$ Hz, $J_2 = 7.2$ Hz, 1H, NH-C**H**-P), 4.81–4.85(dd, $J_1 = J_2 = 7.2$ Hz, 1H, NH), 6.48 (d, $J = 8.4$ Hz, 2H, Ar–H), 6.82 (d, $J = 8.4$ Hz, 1H, Ar–H), 6.96 (br s, 2H, Ar–H), 7.18 (d, $J = 8.4$ Hz, 2H, Ar–H) ppm; ^{13}C-NMR (100 MHz, $CDCl_3$): $\delta = 16.39$ (d, $^3J_{PC} =$ 6 Hz), 16.4 (d, $^3J_{PC} = 6$ Hz), 55.7 (d, $J_{CP} =$ 152 Hz), 55.8, 55.9, 63.2 (d, $^2J_{PC} = 7$ Hz), 63.3 (d, $^2J_{PC} = 7$ Hz), 110, 110,7 111, 115.5, 120.2, 127.6, 131.8, 145.5 (d, $^3J_{PC} = 15$ Hz), 148.7, 149.1 ppm.

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