A novel $Cu(OTf)_2$ mediated three component high yield synthesis of *α*-aminophosphonates

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Abstract
Copper(II) triflate catalyzes efficiently the three-component condensation reaction of an aldehyde, amine and $P(OMe)_3$ in acetonitrile at room temperature to afford the corresponding *α*-aminophosphonates in high yields.

Keywords: Aldehydes, catalysts, *α*-aminophosphonates, copper triflate

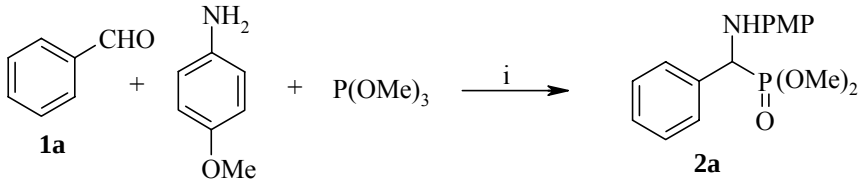
Introduction

In the recent years, *α*-aminophosphonates have received enormous attention because they are considered to be structural analogues of the corresponding α-amino acids and transition state mimics of peptide hydrolysis.[1] Due to their structural analogy to α-aminoacids, they function as inhibitors of enzymes involved in the metabolism of proteins and aminoacids. For example, the phosphonic analogue of phenylalanine is an inhibitor of phenylalanyl-5-RNA-synthetase[2] and phosphonodipeptide alafosfalin is an antimicrobial agent.[3]

Substantial progress has been made towards the development of efficient methods for the preparation of these compounds.[4] Although many methods are available in the literature for hydrophosphonylation of imines, they suffer from certain drawbacks such as use of expensive and hazardous phosphorous sources, stoichiometric amounts of catalysts, high temperatures, multistep synthesis, lower product selectivities and yields. Recently, nucleophilic addition of phosphate onto imines, catalyzed by a base or an acid, has emerged as an important alternative for the synthesis of such α-aminophosphonates derivatives.[5] Generally, Lewis acids such as $SnCl_2$, $SnCl_4$, $BF_3{\cdot}OEt_2$, $ZnCl_2$, $MgBr_2$, and $InCl_3$ have been used as catalysts.[6] However, these reactions could not be carried out efficiently in a single step operation with the carbonyl, amine and phosphite functionalities because amines and water that are formed during imine formation can decompose or deactivate these Lewis acids.[7] So there is a need to develop one-pot synthesis of *α*-aminophosphonates catalyzed by a water-tolerant Lewis acid.

Results and Discussion

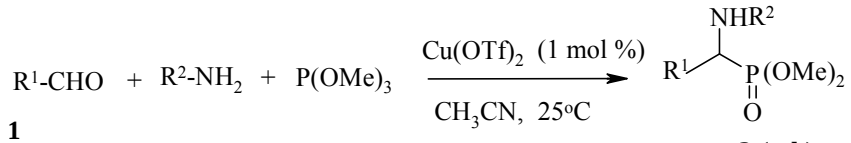
In continuation of our interest in Cu(II) mediated organic reactions,[8] we explored the effectiveness of $Cu(OTf)_2$ as a catalyst for the generation of *α*-amino phosphonates. Thus, the reaction of trimethyl phosphite with imines generated *in-situ* from benzaldehyde and *p*-anisidine in CH_3CN at room temperature in the presence of 1 mol % of $Cu(OTf)_2$ without using internal drying agent, afforded the corresponding α-amino phosphonate (**2a**) in 97% yield (**Scheme 1, Table 1**). The reaction was complete in 5 h at room temperature and the product was isolated by usual work-up, with high purity. To the best of our knowledge, this is the first demonstration of $Cu(OTf)_2$ catalyzed synthesis of *α*-amino phosphonates.



1a **2a**

Scheme 1. i cat. $Cu(OTf)_2$ (1 mol%), CH_3CN, 25 ^oC. , 5 h

Table 1. $Cu(OTf)_2$-catalyzed one pot addition of $P(OMe)_3$ onto imines: synthesis of α-aminophosphonates [a]



1 **2 (a-k)**

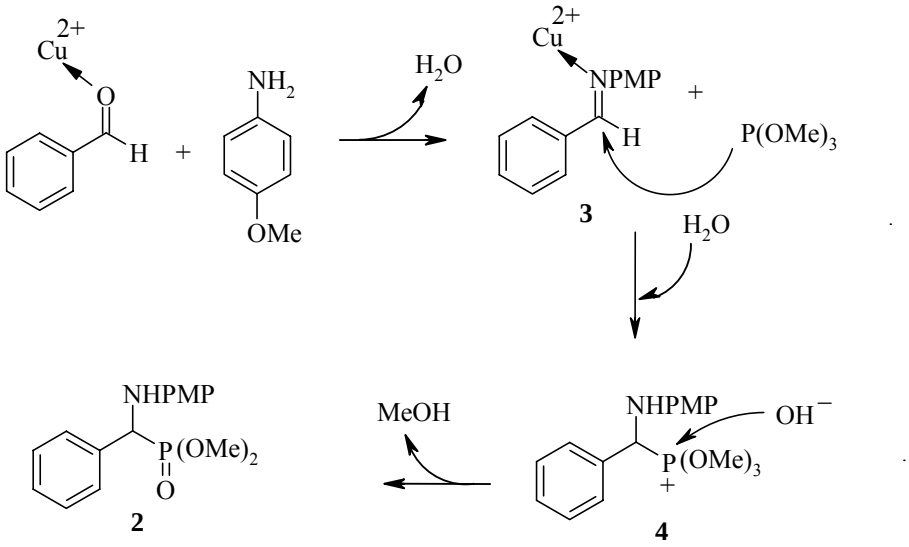
Entry	R^1	R^2	Yield (%)[b]
a	C_6H_5	4-MeO-C_6H_4	97
b	C_6H_5	C_6H_5	84
c	C_6H_5	$C_6H_5CH_2$	88
d	C_6H_5	$C_6H_5(CH_3)CH$	56
e	4-Cl-C_6H_4	4-MeO-C_6H_4	92
f	4-MeO-C_6H_4	4-MeO-C_6H_4	95
g	4-F_3C-C_6H_4	4-MeO-C_6H_4	80
h	4-NC-C_6H_4	4-MeO-C_6H_4	82
i	4-O_2N-C_6H_4	4-MeO-C_6H_4	75
j	4-HO-C_6H_4	4-MeO-C_6H_4	79
k	$(CH_3)CH$	$(CH_3)CH$	57

a: Conditions. Aldehyde (10 mmol), amine source (10 mmol), $P(OMe)_3$ (11 mmol), $Cu(OTf)_2$ (1 mol %), CH_3CN, 25 ^oC, 5h.

b: Isolated yield after chromatographic purification

In order to gauge the scope, several aromatic aldehydes (**1**) were examined under the optimized conditions using 1 mol % of $Cu(OTf)_2$. The results are shown in Table 1. In all cases studied, the reaction proceeded smoothly to give the corresponding *α*-amino phosphonates (**2**) in high yields. Most importantly, aromatic aldehydes carrying either electron-donating or electron-withdrawing substituents including hydroxy groups reacted efficiently giving excellent yields. Incase of aliphatic aldehyde (**2k**) the yield was found to be moderate.

The mechanism of this reaction is believed to involve, at first, the formation of activated imine **3** so that addition of phosphite is facilitated to afford phosphonium intermediate **4,** which then undergoes reaction with water generated during formation of imine to give *α*-amino phosphonates **2** and MeOH as shown in **Scheme 2**.[9]



Scheme 2. Cu^{2+}- activation in three-component $P(OMe)_3$ addition onto imines

In conclusion, we have successfully demonstrated the use of $Cu(OTf)_2$ as an efficient Lewis acid catalyst, for the first time, to the three-component high yield synthesis of *α*-aminophosphonates (**2a-k**) at room temperature. The $Cu(OTf)_2$ is stable and does not show any decrease in its catalytic activity due to *in-situ* generated water during the course of reaction. Finally, the present procedure appears attractive for its operational simplicity and generally high yields of products.

Experimental Section

General Procedures. Solvents were purified and dried by standard procedures before use; petroleum ether of boiling range 60-80^{o}C was used. Melting points are uncorrected. Infrared spectra were recorded on a Shimadzu FTIR-8400 spectrometer. ^{1}H-NMR, ^{13}C-NMR and ^{31}P-

NMR were recorded on Bruker AC-200 NMR spectrometer. Elemental analysis was carried on a Carlo Erba CHNS-O analyzer.

General experimental procedure for hydrophosphonylation of imines

A mixture of aldehydes (10 mmol), p-anisidine (10 mmol), $P(OMe)_3$ (11 mmol) and $Cu(OTf)_2$ (0.1 mmol) in acetonitrile (10 ml) was stirred at 25°C for 5 h. The reaction was monitored by TLC. After completion of the reaction, it was extracted with ethyl acetate (20 ml) and washed with water and brine. The organic layer was dried over anhydrous sodium sulfate and concentrated under reduced pressure to give the crude products, which were purified by column chromatography on silica gel using petroleum ether as eluent to afford the pure product.

Dimethyl (4-methoxyphenylamino)(phenyl)methylphosphonate (2a). Yield: 97%; brown colored solid; mp: 85–88°C (crystallized from EtOAc); IR (Neat, cm^{-1}): 1032, 1230, 1452, 1609, 1879, 2855, 2932, 3294; ^{1}H-NMR (200 MHz, $CDCl_3$): δ 3.28-3.34 (d, *J* = 10.6 Hz, 3H), 3.70 (s, 3H), 3.75-3.80 (d, *J* = 10.6 Hz, 3H), 4.22 (brs, 1H), 4.68-4.80 (d, *J* = 24.2 Hz, 1H), 6.54-6.59 (d, *J* = 9.0 Hz, 2H), 6.70-6.75 (d, *J* = 9.0 Hz, 2H), 7.19-7.24 (m, 5H); ^{13}C-NMR (50 MHz, $CDCl_3$ + DMSO-d_6): δ 51.53, 54.49, 62.51, 112.77, 113.59, 114.19, 115.42, 116.40, 121.42, 127.28, 130.04, 131.67, 152.79; ^{31}P-NMR (80 MHz, $CDCl_3$ + DMSO-d_6): δ 25.19; Analysis: $C_{16}H_{20}NO_4P$ Calculated C, 59.81; H, 6.27; N, 4.36; found C, 59.63; H, 6.21; N, 4.29%.

Dimethyl phenyl(phenylamino)methylphosphonate (2b). Yield: 84%; green colored gum; ^{1}H-NMR (200 MHz, $CDCl_3$): δ 2.14 (s, 1H), 3.45-3.50 (d, *J* = 10.7 Hz, 3H), 3.76-3.82 (d, *J* = 10.7 Hz, 3H), 4.79-4.92 (d, *J* = 24.4 Hz, 1H), 6.63-6.67 (m, 3H), 7.09-7.17 (m, 2H), 7.28-7.41 (m, 3H), 7.50-7.53 (m, 2H); ^{13}C-NMR (50 MHz, $CDCl_3$): δ 53.59, 53.73, 53.88, 56.89, 113.72, 118.35, 127.58, 127.69, 128.53, 128.98, 135.33, 145.70, 145.99; ^{31}P-NMR (80 MHz, $CDCl_3$): δ 25.51; Analysis: $C_{15}H_{18}NO_3P$ Calculated C, 61.85; H, 6.23; N, 4.81; found C, 61.70; H, 6.12; N, 4.60%.

Dimethyl (benzylamino)(phenyl)methylphosphonate (2c). Yield: 88%; green colored solid; mp: 77–78°C (crystallized from $CHCl_3$); IR (Neat, cm^{-1}): 1029, 1103, 1233, 1600, 1805, 1886, 1951, 2857, 3295; ^{1}H-NMR (200 MHz, $CDCl_3$): δ 3.40-3.45 (d, *J* = 10.7 Hz, 3H), 3.73-3.89 (m, 6H), 6.85-7.30 (m, 10H); ^{13}C-NMR (50 MHz, $CDCl_3$ + DMSO-d_6): δ 52.48, 52.57, 53.44, 54.12, 126.45, 126.72, 127.23, 127.33, 128.13, 128.58, 138.90, 129.28, 129.52, 143.60; ^{31}P-NMR (80 MHz, $CDCl_3$ + DMSO-d_6): δ 23.72; Analysis: $C_{16}H_{20}NO_3P$ Calculated C, 62.94; H, 6.60; N, 4.59; found C, 63.00; H, 6.6.62; N, 4.60%.

Dimethyl (1-phenylethylamino)(phenyl)methylphosphonoate (2d). Yield: 56%; gum; ^{1}H-NMR (200 MHz, $CDCl_3$): δ 1.30-1.33 (d, *J* = 6.5 Hz, 3H), 2.05 (brs, 1H), 3.41-3.52 (q, *J* = 10.3 Hz, 3H), 3.76-3.82 (m, 5H), 6.82-6.89 (q, *J* = 4.7 Hz, 2H), 7.17-7.33 (m, 8H); ^{13}C-NMR (50 MHz, $CDCl_3$ + DMSO-d_6): δ 22.13, 24.59, 53.57, 54.91, 58.18, 113.87, 126.59, 126.87, 128.24, 129.28, 129.41, 129.64, 143.60, 144.75, 159.16; ^{31}P-NMR (80 MHz, $CDCl_3$ + DMSO-d_6): δ 23.98; Analysis: $C_{17}H_{22}NO_3P$ Calculated C, 63.94; H, 6.94; N, 4.39; found C, 63.87; H, 6.82; N, 4.40%.

Dimethyl (4-methoxyphenylamino)(4-chlorophenyl)methylphosphonate (2e). Yield: 92%; green colored solid; mp: 105–108°C (crystallized from EtOAc); IR (Neat, cm^{-1}): 1030, 1105,

1234, 1451, 1603, 1800, 1950, 2960, 3295; ^{1}H-NMR (200 MHz, $CDCl_3$): δ 3.52-3.57 (d, *J* = 10.7 Hz, 3H), 3.68 (s, 3H), 3.74-3.80 (d, *J* = 10.7 Hz, 3H), 4.61-4.74 (d, *J* = 24.2 Hz, 1H), 6.47-6.52 (d, *J* = 9.1 Hz, 2H), 6.65-6.70 (d, *J* = 9.1 Hz, 2H), 7.26-7.42 (m, 4H); ^{13}C-NMR (50 MHz, $CDCl_3$ + DMSO-d_6): δ 51.66, 51.83, 52.15, 53.74, 54.90, 111.92, 113.52, 126.80, 128.13, 128.24, 131.38, 131.46, 133.66, 138.71, 139.03, 150.62; ^{31}P-NMR (80 MHz, $CDCl_3$ + DMSO-d_6): δ 24.73; Analysis: $C_{16}H_{19}ClNO_4P$ Calculated C, 54.02; H, 5.38; N, 3.94; found C, 54.00; H, 5.52; N, 3.89%.

Dimethyl (4-methoxyphenylamino)(4-methoxyphenyl)methylphosphonate (2f). Yield: 95%; gum; IR (Neat, cm^{-1}): 1014, 1093, 1369, 1611, 1802, 1950, 2930, 3294; ^{1}H-NMR (200 MHz, $CDCl_3$): δ 3.53-3.58 (d, *J* = 10.7 Hz, 3H), 3.67 (s, 6H), 3.74-3.80 (d, *J* = 10.7 Hz, 3H), 4.17 (brs, 1H), 4.61-4.73 (d, *J* = 24.3 Hz, 1H), 6.43-6.48 (d, *J* = 8.9 Hz, 2H), 6.63-6.68 (d, *J* = 8.9 Hz, 2H), 6.71-6.89 (m, 4H); ^{13}C-NMR (50 MHz, $CDCl_3$ + DMSO-d_6): δ 51.12, 52.15, 52.44, 53.89, 55.72, 55.89, 112.27, 113.81, 117.27, 127.46, 127.63, 138.62, 141.36, 151.06, 158.73; ^{31}P-NMR (80 MHz, $CDCl_3$ + DMSO-d_6): δ 24.89; Analysis: $C_{17}H_{22}NO_5P$ Calculated C, 58.12; H, 6.31; N, 3.99; found C, 58.10; H, 6.28; N, 4.00%.

Dimethyl (4-methoxyphenylamino)(4-(trifluoromethyl)phenyl)methylphosphonate (2g). Yield: 80%; gum; IR (Neat, cm^{-1}): 1011, 1087, 1107, 1378, 1457, 1886, 2860, 2930, 3289; ^{1}H-NMR (200 MHz, $CDCl_3$): δ 3.54-3.59 (d, *J* = 10.7 Hz, 3H), 3.68 (s, 3H), 3.75-3.81 (d, *J* = 10.7 Hz, 3H), 4.71-4.83 (d, *J* = 24.6 Hz, 1H), 6.47-6.51 (d, *J* = 8.9 Hz, 2H), 6.66-6.70 (d, *J* = 8.9 Hz, 2H), 7.59 (s, 4H); ^{13}C-NMR (50 MHz, $CDCl_3$ + DMSO-d_6): δ 51.84, 51.98, 52.44, 52.57, 53.89, 55.54, 113.14, 113.66, 123.74, 127.22, 127.32, 138.75, 139.04, 139.87, 150.92; ^{31}P-NMR (80 MHz, $CDCl_3$ + DMSO-d_6): δ 29.06; Analysis: $C_{17}H_{19}F_3NO_4P$ Calculated C, 52.45; H, 4.92; N, 3.36; found C, 52.52; H, 4.98; N, 3.59%.

Dimethyl (4-methoxyphenylamino)(4-cyanophenyl)methylphosphonate (2h). Yield: 82%; brown colored solid; mp: 88^{o}C (crystallized from EtOH); IR (Neat, cm^{-1}): 1030, 1100, 1380, 1600, 1805, 1951, 2931, 3294; ^{1}H-NMR (200 MHz, $CDCl_3$): δ 3.57-3.62 (d, *J* = 10.7 Hz, 3H), 3.68 (s, 3H), 3.76-3.82 (d, *J* = 10.7 Hz, 3H), 4.23 (brs, 1H), 4.70-4.83 (d, *J* = 24.9 Hz, 1H), 6.45-6.50 (d, *J* = 8.9 Hz, 2H), 6.66-6.70 (d, *J* = 8.9 Hz, 2H), 7.55-7.66 (m, 4H); ^{13}C-NMR (50 MHz, $CDCl_3$ + DMSO-d_6): δ 52.06, 52.20, 52.80, 54.02, 55.80, 109.87, 113.24, 113.79, 117.25, 127.65, 127.74, 130.72, 138.58, 138.90, 141.21, 151.10; ^{31}P-NMR (80 MHz, $CDCl_3$ + DMSO-d_6): δ 23.88; Analysis: $C_{17}H_{19}N_2O_4P$ Calculated C, 58.96; H, 5.53; N, 8.09; found C, 58.89; H, 5.55; N, 7.96%.

Dimethyl (4-methoxyphenylamino)(4-nitrophenyl)methylphosphonate (2i). Yield: 75%; yellowish brown colored solid; mp: 114–117^{o}C (crystallized from MeOH); IR (Neat, cm^{-1}): 1031, 1234, 1447, 1598, 1950, 2933, 3298; ^{1}H-NMR (200 MHz, $CDCl_3$): δ 3.59-3.64 (d, *J* = 10.7 Hz, 3H), 3.67 (s, 3H), 3.80-3.86 (d, *J* = 10.7 Hz, 3H), 4.29 (brs, 1H), 4.72-4.84 (d, *J* = 24.2 Hz, 1H), 6.47-6.52 (d, *J* = 8.9 Hz, 2H), 6.71-6.75 (d, *J* = 8.9 Hz, 2H), 7.51-8.12 (m, 4H); ^{13}C-NMR (50 MHz, $CDCl_3$ + DMSO-d_6): δ 52.56, 52.70, 52.81, 54.28, 56.13, 113.22, 113.81, 120.25, 127.72, 127.81, 130.79, 139.61, 139.96, 148.22, 151.14; ^{31}P-NMR (80 MHz, $CDCl_3$ + DMSO-

d_6): δ 27.41; Analysis: $C_{16}H_{19}N_2O_6P$ Calculated C, 52.46; H, 5.23; N, 7.65; found C, 52.44; H, 5.20; N, 7.59%.

Dimethyl (4-methoxyphenylamino)(4-hydroxyphenyl)methylphosphonate (2j). Yield: 79%; green colored solid; mp: 107-108°C (crystallized from MeOH); IR (Neat, cm^{-1}): 1012, 1078, 1231, 1385, 1602, 1653, 1951, 2937, 3296; ^{1}H-NMR (200 MHz, $CDCl_3$): δ 3.44-3.49 (d, *J* = 10.6 Hz, 3H), 3.67 (s, 3H), 3.72-3.77 (d, *J* = 10.6 Hz, 3H), 3.92 (brs, 1H), 4.56-4.67 (d, *J* = 23.5 Hz, 1H), 6.52-6.56 (d, *J* = 8.9 Hz, 2H), 6.64-6.68 (d, *J* = 8.9 Hz, 2H), 6.77-6.81 (d, *J* = 8.2 Hz, 2H), 7.21-7.25 (d, *J* = 8.2 Hz, 2H); ^{13}C-NMR (50 MHz, $CDCl_3$ + DMSO-d_6): δ 51.10, 52.13, 52.41, 53.87, 55.70, 55.85, 112.32, 113.74, 117.23, 127.55, 127.62, 138.59, 141.71, 151.00, 157.68; ^{31}P-NMR (80 MHz, $CDCl_3$ + DMSO-d_6): δ 24.36; Analysis: $C_{16}H_{20}NO_5P$ Calculated C, 56.97; H, 5.98; N, 4.15; found C, 56.82; H, 5.87; N, 4.12%.

Dimethyl 1-(isopropylamino)-2-methylpropylphosphonate (2k). Yield: 57%; colorless oil; IR (Neat, cm^{-1}): 1061, 1251, 1469, 3325; ^{1}H-NMR (200 MHz, $CDCl_3$): δ 0.99-1.08 (m, 12H), 2.10 (m, 1H), 2.85 (dd, *J* = 16.7, 3.8 Hz, 1H), 3.07 (m, 1H), 3.77 (d, *J* = 9.9 Hz, 3H), 3.81 (d, *J* = 10.2 Hz, 3H); ^{13}C-NMR (50 MHz, $CDCl_3$ + DMSO-d_6): δ 18.06, 20.61, 22.74, 23.48, 29.28, 47.54, 52.35, 52.87, 57.42; ^{31}P-NMR (80 MHz, $CDCl_3$ + DMSO-d_6): δ 29.72; Analysis: $C_9H_{22}NO_3P$ Calculated C, 48.42; H, 9.93; N, 6.27; found C, 48.40; H, 9.88; N, 6.21%.

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