

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

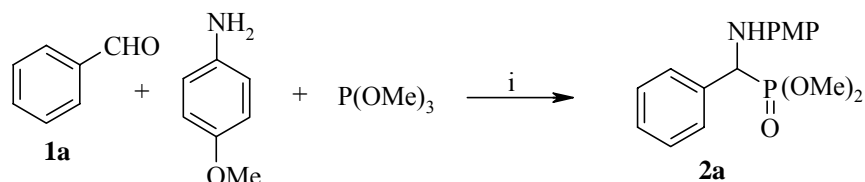
Introduction

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-synthetase² 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 SnCl₂, SnCl₄, BF₃·OEt₂, ZnCl₂, MgBr₂, and InCl₃ 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 CH₃CN 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.



Scheme 1. i cat. Cu(OTf)₂ (1 mol%), CH₃CN, 25 °C., 5 h

Table 1. Cu(OTf)₂-catalyzed one pot addition of P(OMe)₃ onto imines: synthesis of α -aminophosphonates^a

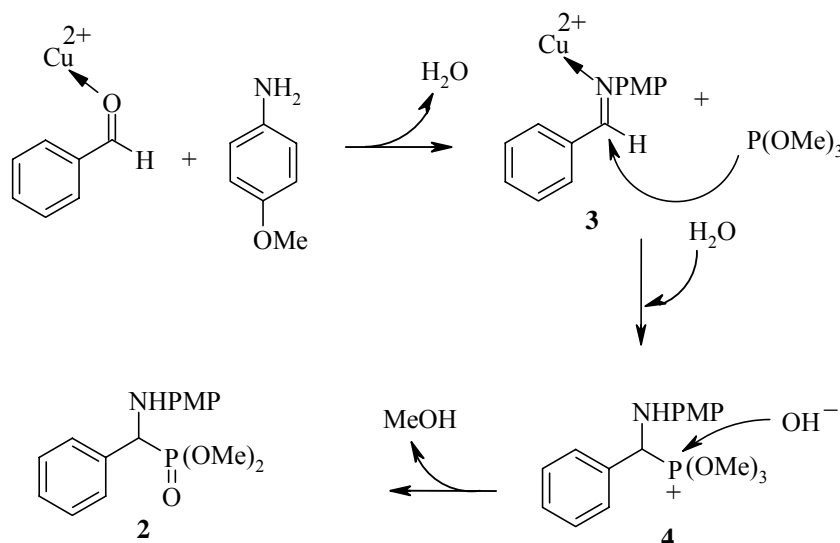
Entry	R ¹	R ²	Yield (%) ^b
a	C ₆ H ₅	4-MeO-C ₆ H ₄	97
b	C ₆ H ₅	C ₆ H ₅	84
c	C ₆ H ₅	C ₆ H ₅ CH ₂	88
d	C ₆ H ₅	C ₆ H ₅ (CH ₃)CH	56
e	4-Cl-C ₆ H ₄	4-MeO-C ₆ H ₄	92
f	4-MeO-C ₆ H ₄	4-MeO-C ₆ H ₄	95
g	4-F ₃ C-C ₆ H ₄	4-MeO-C ₆ H ₄	80
h	4-NC-C ₆ H ₄	4-MeO-C ₆ H ₄	82
i	4-O ₂ N-C ₆ H ₄	4-MeO-C ₆ H ₄	75
j	4-HO-C ₆ H ₄	4-MeO-C ₆ H 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 %), CH₃CN, 25 °C, 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 $\text{Cu}(\text{OTf})_2$. 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. In case 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 $\text{P}(\text{OMe})_3$ addition onto imines

In conclusion, we have successfully demonstrated the use of $\text{Cu}(\text{OTf})_2$ 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 $\text{Cu}(\text{OTf})_2$ 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°C was used. Melting points are uncorrected. Infrared spectra were recorded on a Shimadzu FTIR-8400 spectrometer. ¹H-NMR, ¹³C-NMR and ³¹P-

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; ¹H-NMR (200 MHz, CDCl₃): 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); ¹³C-NMR (50 MHz, CDCl₃ + DMSO-d₆): 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; ³¹P-NMR (80 MHz, CDCl₃ + DMSO-d₆): δ 25.19; Analysis: C₁₆H₂₀NO₄P 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; ¹H-NMR (200 MHz, CDCl₃): 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); ¹³C-NMR (50 MHz, CDCl₃): 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; ³¹P-NMR (80 MHz, CDCl₃): δ 25.51; Analysis: C₁₅H₁₈NO₃P 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 CHCl₃); IR (Neat, cm⁻¹): 1029, 1103, 1233, 1600, 1805, 1886, 1951, 2857, 3295; ¹H-NMR (200 MHz, CDCl₃): δ 3.40-3.45 (d, *J* = 10.7 Hz, 3H), 3.73-3.89 (m, 6H), 6.85-7.30 (m, 10H); ¹³C-NMR (50 MHz, CDCl₃ + DMSO-d₆): 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; ³¹P-NMR (80 MHz, CDCl₃ + DMSO-d₆): δ 23.72; Analysis: C₁₆H₂₀NO₃P Calculated C, 62.94; H, 6.60; N, 4.59; found C, 63.00; H, 6.66; N, 4.60%.

Dimethyl (1-phenylethylamino)(phenyl)methylphosphonate (2d). Yield: 56%; gum; ¹H-NMR (200 MHz, CDCl₃): 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); ¹³C-NMR (50 MHz, CDCl₃ + DMSO-d₆): 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; ³¹P-NMR (80 MHz, CDCl₃ + DMSO-d₆): δ 23.98; Analysis: C₁₇H₂₂NO₃P 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; $^1\text{H-NMR}$ (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); $^{13}\text{C-NMR}$ (50 MHz, $\text{CDCl}_3 + \text{DMSO-d}_6$): 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; $^{31}\text{P-NMR}$ (80 MHz, $\text{CDCl}_3 + \text{DMSO-d}_6$): δ 24.73; Analysis: $\text{C}_{16}\text{H}_{19}\text{ClNO}_4\text{P}$ 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; $^1\text{H-NMR}$ (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); $^{13}\text{C-NMR}$ (50 MHz, $\text{CDCl}_3 + \text{DMSO-d}_6$): 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; $^{31}\text{P-NMR}$ (80 MHz, $\text{CDCl}_3 + \text{DMSO-d}_6$): δ 24.89; Analysis: $\text{C}_{17}\text{H}_{22}\text{NO}_5\text{P}$ 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; $^1\text{H-NMR}$ (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); $^{13}\text{C-NMR}$ (50 MHz, $\text{CDCl}_3 + \text{DMSO-d}_6$): 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; $^{31}\text{P-NMR}$ (80 MHz, $\text{CDCl}_3 + \text{DMSO-d}_6$): δ 29.06; Analysis: $\text{C}_{17}\text{H}_{19}\text{F}_3\text{NO}_4\text{P}$ 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°C (crystallized from EtOH); IR (Neat, cm^{-1}): 1030, 1100, 1380, 1600, 1805, 1951, 2931, 3294; $^1\text{H-NMR}$ (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); $^{13}\text{C-NMR}$ (50 MHz, $\text{CDCl}_3 + \text{DMSO-d}_6$): 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; $^{31}\text{P-NMR}$ (80 MHz, $\text{CDCl}_3 + \text{DMSO-d}_6$): δ 23.88; Analysis: $\text{C}_{17}\text{H}_{19}\text{N}_2\text{O}_4\text{P}$ 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°C (crystallized from MeOH); IR (Neat, cm^{-1}): 1031, 1234, 1447, 1598, 1950, 2933, 3298; $^1\text{H-NMR}$ (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); $^{13}\text{C-NMR}$ (50 MHz, $\text{CDCl}_3 + \text{DMSO-d}_6$): 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; $^{31}\text{P-NMR}$ (80 MHz, $\text{CDCl}_3 + \text{DMSO-d}_6$): δ 23.88.

d₆): δ 27.41; Analysis: C₁₆H₁₉N₂O₆P 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; ¹H-NMR (200 MHz, CDCl₃): 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); ¹³C-NMR (50 MHz, CDCl₃ + DMSO-d₆): 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; ³¹P-NMR (80 MHz, CDCl₃ + DMSO-d₆): δ 24.36; Analysis: C₁₆H₂₀NO₅P 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; ¹H-NMR (200 MHz, CDCl₃): 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); ¹³C-NMR (50 MHz, CDCl₃ + DMSO-d₆): δ 18.06, 20.61, 22.74, 23.48, 29.28, 47.54, 52.35, 52.87, 57.42; ³¹P-NMR (80 MHz, CDCl₃ + DMSO-d₆): δ 29.72; Analysis: C₉H₂₂NO₃P Calculated C, 48.42; H, 9.93; N, 6.27; found C, 48.40; H, 9.88; N, 6.21%.

Acknowledgements

ASP thanks CSIR, New Delhi, for the award of research fellowships. Financial Grants from DST, New Delhi (Sanction No SR/S1/OC-22/2002) is gratefully acknowledged. The authors thank Dr. B. D. Kulkarni, Head, CE-PD Division, for his constant encouragement and support.

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