Heteroatom Chemistry
Volume 22, Number 3/4, 2011

Facile Synthesis of α-Amino Phosphonates in Water by Kabachnik–Fields Reaction Using Magnesium Dodecyl Sulfate

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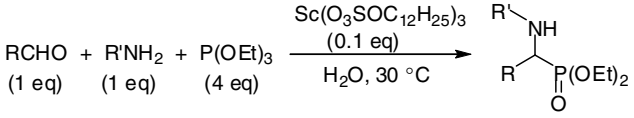
Received 29 September 2010; revised 29 October 2010

ABSTRACT: *The three-component reaction of aldehydes, amines and triethyl or diphenyl phosphite was efficiently catalyzed by magnesium dodecyl sulfate at room temperature in water to give various α-amino phosphonates in high yields. The catalyst is easily available from inexpensive sodium dodecyl sulfate and magnesium bromide.* © 2011 Wiley Periodicals, Inc. Heteroatom Chem 22:358–362, 2011; View this article online at wileyonlinelibrary.com. DOI 10.1002/hc.20689

INTRODUCTION

The synthesis of α-amino phosphonates has attracted much attention due to their significant biological activities such as antibacterial and anti-HIV, structural analogy to α-amino acids, and transition state mimics of peptide hydrolysis [1,2]. Nucleophilic addition reaction of phosphites to imines is one of the most preferred methods [3]. A one-pot reaction of aldehydes (or ketones), amines, and dialkyl, or diaryl phosphites via the Kabachnik–Fields reaction [4] is one of the most attractive alternates. Recently, various catalysts have been developed for this reaction [5]. However, still there remains a need to develop a more efficient method, particularly from the viewpoint of today's environmental concerns. Especially, use of aqueous media as a reaction solvent has attracted much attention. Kobayashi et al. reported that the three-component reaction of aldehydes, amines, and triethyl phosphite was effectively catalyzed by scandium tris(dedecyl sulfate) to give various α-amino phosphonates in high yields in water [6]. However, scandium(III) salts are expensive and 4 equiv of $P(OEt)_3$ was needed in order to get high yields. Therefore, we decided to study the effect of other metal dodecyl sulfates and the analogues for the one-pot Kabachnik–Fields reaction to find a more efficient and less expensive catalyst. Here, we would like to report our results.



First, the reaction of benzaldehyde, aniline, and $P(OEt)_3$ **3a** was tested as a model reaction in the presence of various catalysts in water (Table 1). The catalysts **1** were prepared from $MgBr_2$, $CaCl_2$, and $AlCl_3$ with sodium dodecyl sulfate by following Kobayashi's procedure [7]. The catalyst **2a** was prepared from $Mg(OH)_2$ with dodecylbenzensulfonic acid, and **2b** was prepared from $CaCl_2$ with sodium dodecylbenzensulfonate. When 0.1 equiv of **1a** was used as a catalyst, the reaction proceeded smoothly to give α-amino phosphonate **4a** in 99% yield in 1 h (entry 1). Reduction of the quantity of **3a** to 2.0 and 1.2 equiv reduced the yield only slightly

Dedicated to Professor Kin-ya Akiba on the occasion of his 75th birthday.

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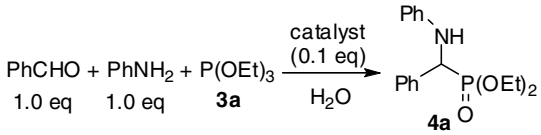
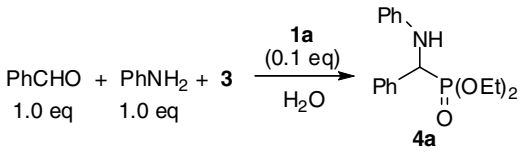
TABLE 1 Three-Component Synthesis of α-Amino Phospnonate **4a** in Water



	3a (Equiv)	*Catalyst*	*Time (h)*	*Yield (%)*
1	4.0	**1a**	1	99
2	2.0	**1a**	1	92
3	1.2	**1a**	1	89
4	4.0	**1b**		93
5	1.2	**1b**	1	82
6	1.2	**1c**	1.5	78
7	1.2	**1d**	1	70
8	1.2	$MgSO_4$	2.5	47
9	1.2	$MgSO_4$–**1d**	2	84
10	1.2	$MgSO_4$-THF[a]	5	80
11	1.2	**2a**	1	84
12	1.2	**2b**	1	84

The reaction was performed in 0.17 mol/L water.
[a]5 equiv.

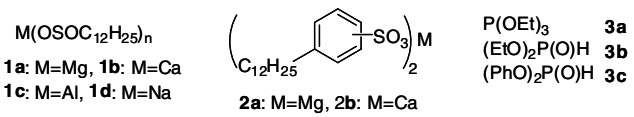
TABLE 2 Three-Component Synthesis of **4** Catalyzed by **1a**



Entry	***3*,equivs**	*Solvent (mol/L)*	*Time (h)*	*Yield (%)*
1	**3a**, 1.2	H_2O, 0.17	1	89
2	**3a**, 1.2	H_2O, 1	1	91
3	**3a**, 1.2	H_2O, 1.7	1	87
4[a]	**3a**, 1.2	H_2O, 0.17	1	81
5	**3a**, 1.2	THF, 0.17	6	72
6	**3a**, 1.2	CH_2Cl_2, 0.17	6	73
7	**3b**, 2.0	H_2O, 0.17	1	82[b]
8	**3b**, 2.0	H_2O, 0.17	6	92[c]
9	**3b**, 1.2	neat[d]	24	55
10	**3c**, 2.0	H_2O, 0.17	2	97[e]

[a]**1d** and $MgCl_2$ was used instead of **1a**.
[b]After work-up, the concentrated crude mixture was left overnight.
[c]After work-up, the concentrated crude mixture was left for 28 h.
[d]Without **1a**.
[e]The corresponding diphenyl phosphonate.

(entries 2 and 3). The yield of the reaction using 1.2 equiv of **3a** was 89% and higher than that of the literature yield using 4 equiv of **3a** and expensive $Sc(O_3SOC_{12}H_{25})_3$ (88% yield) [6]. Thus, the quantity of **3a** was set to 1.2 equiv. The use of the calcium catalyst **1b** and the alminum catalyst **1c** gave slightly reduced 82% and 78% yields (entries 5 and 6). Although Kobayashi reported that sodium dodecyl sulfate **1d** (0.3 equiv) gave α-amino phosphonate in a very low yield, the reaction using **1d** gave **4a** in 70% yield (entry 7). Recently, the use of **1d** as a catalyst in water at 50°C was reported to give α-amino phosphonate [8]. In the presence of 0.1 equiv of $MgSO_4$, the reaction proceeded to give **4a** in 47% yield after 2.5 h (entry 8). From the comparison of the structure with **1a**, $MgSO_4$ lacks long alkyl chains, which are needed to dissolve the organic substrates in water. Adding **1d** (0.1 equiv) or THF (5 equiv) to



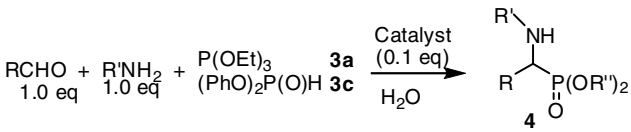
the reaction mixture, the yield of **4a** increased from 47% to 84% and 80%, respectively (entries 9 and 10). Thus, $MgSO_4$ also works as an effective catalyst if a catalytic amount of dissolving reagents is present. Recently, the use of $Mg(ClO_4)_2$ as a catalyst for the Kabachnik–Fields reaction was also reported [9]. Both magnesium and calcium dedecylbensensulfonate, **2a** and **2b**, can also catalyze the reaction to give **4a** in slightly reduced 84% yield after 1 h (entries 11 and 12). Thus, the magnesium salt **1a** is the most effective catalyst. Since our method requires neither expensive reagents nor heating, the superiority of the $Mg(O_3SOC_{12}H_{25})_2$ **1a** to both the corresponding Sc and Na salts is clear.

To determine the best experimental conditions, the reaction of benzaldehyde and aniline was studied using the catalyst **1a** (Table 2). First, the effect of the concentration of the reaction was studied. The yields obtained from the reactions with **3a** performed in 0.17, 1.0, and 1.7 mol/L aqueous solution were 89%, 91%, and 87% yields, respectively (entries 1–3). Thus, the concentration did not have a big influence on the product's yields. When the catalyst **1a** was prepared in situ, that is, 0.1 equiv of **1d** and 0.1 equiv of $MgCl_2$ were used instead of **1a, 4a** was obtained in 81% yield after 1 h (entry 4). Thus, the catalyst prepared in situ was slightly less effective than **1a**. The use of organic solvents (THF and CH_2Cl_2) required longer reaction time, and moderate 72% and 73% yields were obtained after 6 h, respectively (entries 5 and 6). When diethyl phosphite **3b** was used instead of **3a**, the product **4a** was not detected by TLC during the reaction. However, **4a** was isolated by column chromatography in 82% yield (entry 7). Careful monitoring of the reaction by TLC showed that the reaction did not proceed in water even after 6 h. The main product right after the work-up is the corresponding imine. Since the reaction mixture was left as a concentrated state for a while, we

speculated that the reaction between the imine and **3b** occurred in the solvent-free condition. In fact, the yield of **4a** was increased to 92% by leaving the reaction mixture as a concentrated state for 28 h after the work-up (entry 8). Furthermore, **4a** was obtained in 55% yield when benzaldehyde, aniline, and diethyl phosphite **3b** was stirred for 24 h without catalyst and without solvent (entry 9). The improvement of the yield was obtained when diphenyl phosphite **3c** was used instead of **3b**. The reaction of the imine with **3c** occurred smoothly in water and the α-amino phosphonate was obtained in 97% yield after 2 h.

To establish the generality, various aldehydes and amines were subjected to the one-pot three-component reaction using the catalyst **1a** in water. The results are summarized in Table 3. Excellent results were obtained from the reaction of benzaldehyde and triethyl phosphite **3a** with aromatic amines (entries 1–3). The reaction with *o*-anisidine gave α-amino phosphonate **4b** in 78% yield along with the corresponding imine (5%). When 1.0 equiv of **3a** was added to the reaction mixture, and 0.5 equiv of **3a** was added again 1 h later, the yield was improved to 84%. Since the reactivity of the intermediate imine having *o*-anisidine group (electron-donating *o*-methoxy-substituted phenyl group) is supposed to be lower than that of the *N*-phenylimine, decomposition of **3a** in water may compete with the reaction. Therefore, excess amount of **3a** improved the yield. The reaction with 4-chloroaniline proceeded smoothly to give the product **4c** in 81% yield. In this case, there is no intermediate imine left but 5% of benzaldehyde was detected after the work-up. Since the nucleophilicity of 4-chloroaniline is lower than that of aniline, the imine formation is slower than that from aniline. The reactions of benzaldehyde and aliphatic amines were next studied. The reaction with *i*-propylamine gave only trace amount of α-amino phosphonate and the main product was the intermediate imine from benzaldehyde and

TABLE 3 Synthesis of α-Aminophosphonate from Various Aldehydes, Amines, and Phosphites



Entry	*RCHO*	*R'NH$_2$*	*P(OEt)$_3$ (Eq)*	*Catalyst*	*Time (h)*	*Product*	*Yield (%)*	*Ratio*[a]
1	PhCHO	*o*-MeOC$_6$H$_4$NH$_2$	**3a**, 1.2	**1a**	5	**4b**	78	
2	PhCHO	*o*-MeOC$_6$H$_4$NH$_2$	**3a**, 1.0 + 0.5	**1a**	2	**4b**	84	
3	PhCHO	p-ClC$_6$H$_4$NH$_2$	**3a**, 1.2	**1a**	2	**4c**	81	
4	PhCHO	*i*-PrNH$_2$	**3a**, 1.2	**1a**	3	**-**	-[b]	
5	PhCHO	*i*-PrNH$_2$	**3c**, 2.0	**1a**	2	**4d**	(58)[c]	
6	PhCHO	*i*-PrNH$_2$	**3c**, 2.0	**1a, Et$_3$N**	2	**4d**	(74)[c]	
7	PhCHO	PhCH$_2$NH$_2$	**3a**, 1.2	**1a**	6	**4e**	29[b]	
8	PhCHO	PhCH$_2$NH$_2$	**3c**, 1.5	**1a**	4	**4f**	77	
9	PhCHO	(*S*)-PhMeCHNH$_2$	**3a**, 1.2	**1a**	6	**4g**	77	76:24
10	PhCHO	(*S*)-PhMeCHNH$_2$	**3c**, 2.0	**1a**	5	**4h**	53	76:24
11	PhCHO	(*S*)-PhMeCHNH$_2$	**3c**, 2.0	**1a, Et$_3$N**	5	**4h**	77	76:25
12	PhCHO	(*S*)-PhMeCHNH$_2$	**3c**, 1.5	**1a, sparteine**	2	**4h**	87	76:24
13	PhCHO	(*R*)-PhMeCHNH$_2$	**3c**, 1.5	**1a, Pr$_3$N**	3	**4h**	80	76:24
14	*p*-ClC$_6$H$_4$CHO	PhNH$_2$	**3a**, 1.2	**1a**	2	**4i**	77	
15	*p*-ClC$_6$H$_4$CHO[d]	PhNH$_2$	**3a**, 1.2	**1a**	2	**4i**	81	
16	*p*-MeOC$_6$H$_4$CHO	PhNH$_2$	**3a**, 1.2	**1a**	2	**4j**	70	
17	*p*-MeOC$_6$H$_4$CHO	PhNH$_2$	**3a**, 1.0 + 0.5	**1a**	2	**4j**	78	
18	*n*-Octanal	(*S*)-PhMeCHNH$_2$	**3c**, 1.5	**1a, Et$_3$N**	4	**4k**	74	76:25
19	*n*-Octanal	PhCH$_2$NH$_2$	**3c**, 1.5	**1a, Et$_3$N**	3	**4l**	52	
20	*c*-HexCHO	(*S*)-PhMeCHNH$_2$	**3c**, 1.5	**1a, Et$_3$N**	3	**4m**	58	76:24

[a]Diastereomer ratio.
[b]The corresponding imine was obtained mainly.
[c]After the aqueous work-up, the concentrated crude mixture was analyzed in the presence of methyl benzoate as a reference.
[d]2.5 mol/L THF solution was used instead of solid *p*-ClC$_6$H$_4$CHO.

i-propylamine. It seems that the nucleophilicity of **3a** is not enough for the imine having the electron-donating alkyl substituent. When **3c** was used instead of **3a**, α-amino phosphonate **4d** was isolated in 25% yield after column chromatography. Since the NMR spectrum of the crude mixture showed the yield of **4d** was much higher than that, the purified **4d** was submitted to column chromatography again. As a result, 80% of **4d** was recovered and 20% of **4d** decomposed. Owing to the instability of **4d**, the yield was measured by the comparison with the peak of methyl benzoate (0.2 mmol) added after the aqueous work-up. This showed that the yield of **4d** was 58% (entry 5). Furthermore, the yield was improved to 74% when the reaction was performed in the presence of 0.1 equiv of triethylamine. The reaction with benzaldehyde, benzylamine, and $P(OEt)_3$ **3a** gave only 29% yield of the α-amino phosphonate **4e** along with 70% of the corresponding imine (entry 7). The product **4f** was obtained in 77% yield by using diphenyl phosphite **3c** and 0.1 equiv of triethylamine. Interestingly, more lipophilic phenethylamine reacts faster with benzaldehyde and **3a** in the presence of the catalyst **1a** to give **4g** in 77% yield after 6 h. The reaction with **3c** was also improved using 0.1 equiv of amines, triethylamine, sparteine, and tripropylamine (entries 10–13). The yield of **4h** was higher by using more lipophilic sparteine (87% yield). The diastereoselectivity was 76:24 and did not much influenced by the reaction conditions. The reaction of *p*-chlorobenzaldehyde with aniline and **3a** gave **4i** in 77% yield (entry 14). Since *p*-chlorobenzaldehyde is solid at room temperature and was not soluble well in the reaction media, the yield of **4i** was improved to 81% by using 2.5 mol/L THF solution of *p*-chlorobenzaldehyde instead of solid *p*-chlorobenzaldehyde (entry 15). For the reaction of *p*-methoxybenzaldehyde with aniline and **3a, 4j** was obtained in 78% yield when 1.0 and 0.5 equiv of **3a** were added separately (entry 17). For the aliphatic aldehydes, the reaction with amines and **3a** hardly occurred. However, the use of **3c** in the presence of 0.1 equiv of **1a** and 0.1 equiv of triethylamine promoted the reaction efficiently (entries 18–20).

In conclusion, the three-component reaction of aldehydes, amines, and triethyl or diphenyl phosphite in water was effectively catalyzed by magnesium dodecyl sulfate (0.1 equiv) to give various α-amino phosphonates in high yields. Probably, magnesium dodecyl sulfate forms stable dispersion systems with organic substrates in water and the magnesium cation combines aldehydes and amines to form the corresponding imines, which reacts with phosphites. The reactions for either aliphatic aldehydes or aliphatic amines more effectively proceeded with diphenyl phosphite in the presence of magnesium dodecyl sulfate (0.1 equiv) and triethylamine (0.1 equiv). The catalyst is easily available from inexpensive sodium dodecyl sulfate and magnesium bromide, and the reaction can be performed in water at room temperature. These features make the present methodology an environment friendly chemical process.

EXPERIMENTAL

Instrumentation and Chemicals

Melting points were uncorrected. The ^{1}H NMR spectra were recorded on a JEOL α-400 (400 MHz) in $CDCl_3$ using tetramethylsilane as an internal standard ($\delta = 0$ ppm). ^{1}H NMR data are reported as follows: chemical shift, integration, multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, br = broad, m = multiplet), coupling constants (Hz). The mass spectra (MS) were taken on a JEOL JNM 700 mass spectrometer. The column chromatography was carried out using silica gel 60N of Kanto Chemical Co., Ltd. Commercially available reagents were used without purification. The α-amino phosphonates **4a**, [8] **4b**, [10a] **4c**, [8] **4e**, [8] **4h**, [10b] **4i**, [8] and **4j** [5a] are known compounds and their ^{1}H NMR spectra are identical to the reported values.

*Magnesium dodecyl sulfate (***1a***)*: To a solution of sodium dodecyl sulfate (3.036 g, 10 mmol) in H_2O (15 mL) was added $MgBr_2 \cdot 6H_2O$ (1.411 g, 5 mmol) at room temperature. Viscous white precipitates appeared immediately, and the mixture was stirred for 2 h. The white lustrous solid was filtrated and dried (1 mmHg/15 h at room temperature).

Typical experimental procedure (Table 1, entry 3): To a suspension of **1a** (0.028 g, 0.05 mmol) in H_2O (3.0 mL), aniline (0.046 mL, 0.5 mmol), benzaldehyde (0.052 mL, 0.50 mmol), and $P(OEt)_3$ (0.105 mL, 0.6 mmol) were added successively at room temperature. The mixture was stirred for 1 h. 1 mol/L NaOH (10 mL × 2) was added, and the aqueous layer was extracted with ethyl acetate (10 mL). The combined organic extracts were washed with brine, dried ($MgSO_4$), and concentrated under reduced pressure. The residue was purified by flash chromatography (silica gel/hexane-AcOEt (2:3)) to give pure **4a** (0.1427 g, y. 89%) [8].

Diphenyl phenyl-(i-propylamino)methylphosphonate **4d**: Colorless oil. ^{1}H NMR δ 1.01 (3H, d, J = 6.2 Hz), 1.05 (3H, d, J = 6.2 Hz), 1.96 (1H, brs), 2.79 (1H, septet, J = 6.2 Hz), 4.48 (1H, d, J = 22.0 Hz), 6.85–6.90 (2H, m), 7.05–7.40 (11H, m), 7.46–7.51 (2H, m); MS m/e 382 ($M + 1^+$).

Diphenyl (benzylamino)(phenyl)methylphosphonate **4f**: Colorless solid recrystalized from CH_2Cl_2-hexanes, mp 110–111°C. ^{1}H NMR δ 2.42 (1H, brs), 3.64 (1H, d, J = 13.3 Hz), 3.90 (1H, d, J = 13.3 Hz), 4.37 (1H, d, J = 20.3 Hz), 6.86–7.53 (20H, m); MS m/e 430 ($M + 1^+$).

Diethyl (α-phenylethylamino)(phenyl)methylphosphonate **4g**: Colorless oil. ^{1}H NMR major isomer: δ 1.10 (3H, t, J = 7.1 Hz), 1.25–1.38 (6H, m), 2.25 (1H, brs), 3.55–4.20 (6H, m), 7.21–7.33 (10H, m); minor isomer: δ 1.05 (3H, t, J = 7.1 Hz), 1.25–1.38 (6H, m), 2.25 (1H, brs), 3.55–4.20 (6H, m), 7.21–7.33 (10H, m); MS m/e 348 ($M + 1^+$), 210 ($M + 1^+$-HP(O)$(OEt)_2$).

Diphenyl (α-phenylethylamino)(phenyl)methylphosphonate **4h**: Colorless oil. ^{1}H NMR major isomer: δ 1.32 (3H, d, J = 6.6 Hz), 2.36 (1H, bs), 3.97 (1H, q, J = 6.6 Hz), 4.44 (1H, d, J = 20.0 Hz), 6.85–7.43 (20H, m); minor isomer (*S*, *S*): [10b] δ 1.34 (3H, d, J = 6.6 Hz m), 2.36 (1H, bs), 3.66 (1H, dq, J = 1.5, 6.8 Hz), 4.11 (1H, d, J = 22.7 Hz), 6.85–7.43 (20H, m); MS m/e 444 ($M + 1^+$).

Diphenyl (α-phenylethylamino)octylphosphonate **4k**: Colorless oil. ^{1}H NMR major isomer: δ 0.87 (3H, t, J = 7.1 Hz), 1.33 (3H, d, J = 6.4 Hz), 1.01–1.38 (8H, m), 1.48–2.06 (4H, m), 3.03 (1H, dt, J = 3.7, 9.1 Hz), 4.32 (1H, dq, J = 3.4, 6.4 Hz), 7.08–7.35 (15H, m); minor isomer: δ 0.88 (3H, t, J = 7.1 Hz), 1.37 (3H, d, J = 6.4 Hz), 1.01–1.38 (8H, m), 1.48–2.06 (4H, m), 3.21 (1H, dt, J = 17.1, 5.9 Hz), 4.16 (1H, q, J= 6.4 Hz), 7.08–7.35 (15H, m); MS m/e 466 ($M + 1^+$).

Diphenyl (benzylamino)octylphosphonate **4l**: Colorless oil. ^{1}H NMR δ 0.88 (3H, t, J = 6.8 Hz), 1.17–1.35 (8H, m), 1.36–1.50 (1H, m), 1.56–1.68 (1H, m), 1.70–1.78 (1H, m), 1.94–2.07 (1H, m), 3.27 (1H, dd, J = 4.2, 9.2, 10.6 Hz), 3.99 (1H, dd, J = 2.2, 13.2 Hz), 4.08(1H, dd, J = 1.0, 13.2 Hz), 7.12–7.35 (15H, m); MS m/e 452 ($M + 1^+$), 218 ($M + 1^+$-HP(O)$(OPh)_2$).

Diphenyl (α-phenylethylamino)cyclohexylmethylphosphonate **4m**: Colorless oil. ^{1}H NMR major isomer: δ 1.10–2.10 (11H, m), 1.34 (3H, d, J = 6.6 Hz), 2.99 (1H, dd, J = 2.9, 12.2 Hz), 3.91 (1H, brs), 4.30 (1H, dq, J = 4.2, 6.6 Hz), 7.03–7.385 (15H, m); minor isomer: 1.10, 2.10 (11H, m), 1.35 (3H, d, J = 6.4 Hz), 3.17 (1H, dd, J = 3.2, 18.3 Hz), 3.91 (1H, brs), 4.22 (1H, dq, J = 1, 6.4 Hz), 7.03–7.385 (15H, m); MS m/e 216 ($M + 1^+$-HP(O)$(OPh)_2$).

ACKNOWLEDGMENTS

This work was partially supported by the Novartis Foundation (Japan) for the Promotion of Science.

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