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A Critical Overview of the Kabachnik–Fields Reactions Utilizing Trialkyl Phosphites in Water as the Reaction Medium: A Study of the Benzaldehyde-Benzylamine Triethyl Phosphite/Diethyl Phosphite Models

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ABSTRACT: *α-Aminophosphonates may be synthesized by the three-component condensation of oxo-compounds, amines, and dialkyl phosphites or trialkyl phosphites. In the latter case, mostly water is the reaction medium and a catalyst is also needed. This approach has been studied critically by us, exploring the background of this version of the Kabachnik–Fields condensation. The possibilities for the Kabachnik–Fields condensation of benzaldehyde, benzylamine, and triethyl phosphite or diethyl phosphite including the accomplishment in water were studied in detail.* © 2014 Wiley Periodicals, Inc. Heteroatom Chem. 25:282–289, 2014; View this article online at wileyonlinelibrary.com. DOI 10.1002/hc.21192

INTRODUCTION

The synthesis of α-aminophosphonates is of importance due to their significant biological activity [1–4]. The classical method for the preparation of α-aminophosphonates is the Kabachnik–Fields condensation of an aldehyde, amine, and dialkyl phosphite [5–8]. A lot of variations were elaborated during the past decades using different model compounds and applying different conditions. A great number of catalyst were described that made possible carrying out the reactions under mild conditions [9–13], including microwave (MW)-assisted and solvent-free accomplishments [12,13]. However, it was found by the senior author of this article that, in most cases, there is no need to use any catalyst, as the Kabachnik–Fields reaction takes place efficiently under catalyst- and solvent-free conditions on MW irradiation [14–16]. It is a modification of the classical Kabachnik–Fields reaction, when the Shiff base (imine) is performed by the condensation of the aldehyde and the primary amine and the intermediate so formed is reacted with the dialkyl phosphite [17]. There were attempts to accomplish the

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condensations under discussion in water medium [18,19]. This, however, offers no specific advantage, as there is no need for solvent at all, and what is more, if water is used, a catalyst is also needed [19].

There is another variation for the Kabachnik–Fields reaction, when a trialkyl phosphite is used instead of the dialkyl phosphite. In these cases, mostly water was the reaction medium, and it was necessary to use a catalyst. A few examples are summarized in Table 1.

If water is used as the solvent, the solubility of the organic components is problematic. For this reason, surfactants such as scandium tris(dodecyl sulfate) and magnesium bis(dodecyl sulfate) were used to promote the three-component condensation. Using a variety of aldehydes and amines, along with triethyl phosphite as the P-reagent at room temperature (or at 30°C), the α-aminophosphonates were obtained in variable, mostly in good yields (29–84%) after reaction times of 2–6 h (Table 1, entries 1 and 2) [19,20].

In another method, acyclic and cyclic quaternary onium salts (ionic liquids) were used as co-solvents and at the same time as acid catalysts. The condensation of aromatic aldehydes, aromatic amines, and triethyl phosphite at room temperature gave the corresponding α–aminophosphonates in yields of 89–96% in relatively short reaction times (10–60 min) (Table 1, entry 3) [18,21].

Mandhane and co-workers reported an efficient methodology for the synthesis of α–aminophosphonates using thiamine hydrochloride (VB_1) as the catalyst. The condensations were carried out not only under conventional stirring, but under ultrasound. It was found that the reaction time was reduced to few minutes under ultrasound (Table 1, entry 4) [22].

The reaction of aromatic aldehydes, amines, and triethyl phosphite was studied in the presence of tetramethyl-tetra-3,4-pyridinoporphyrazinato copper (II) methyl sulphate in water at 80°C by Sobhani and his research group. The corresponding α-aminophosphonates were obtained in high yields (90–98%) after reaction times of 0.5–3 h (Table 1, entry 5) [23].

In the last case, the reaction of substituted salicylaldehydes, aniline derivatives, and triphenyl phosphite was investigated at ambient temperature in water in the presence of p–toluenesulfonic acid (PTSA) as the catalyst. The products were obtained in yields of 82–94% after reaction times of 3–5 h (Table 1, entry 6) [24].

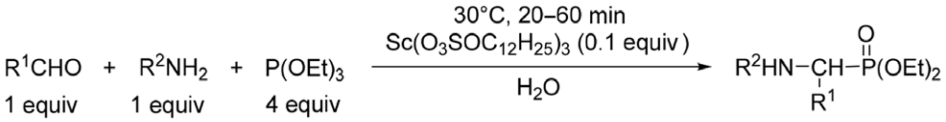
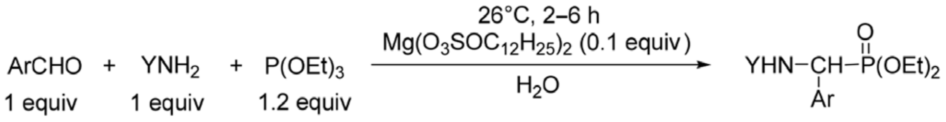
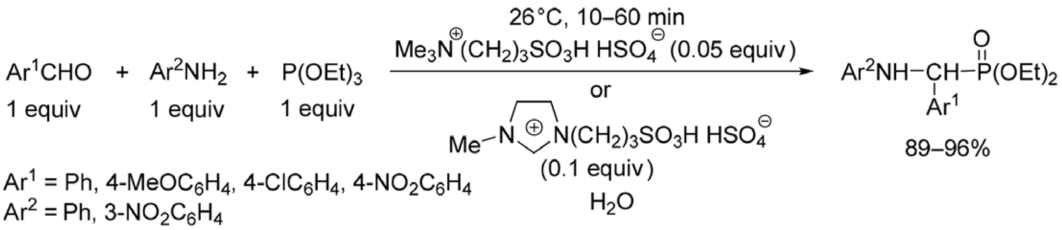
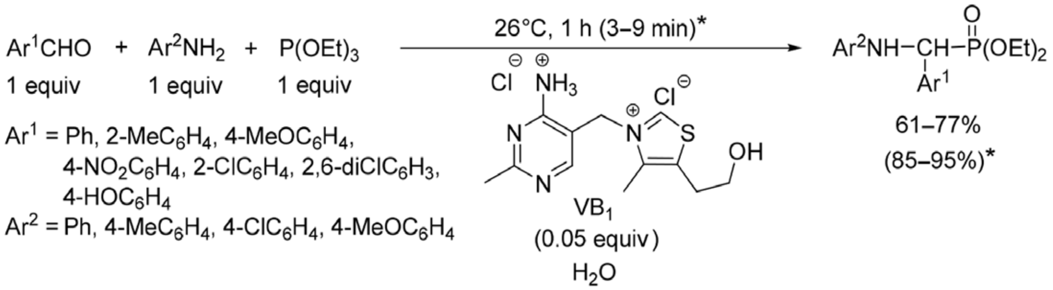
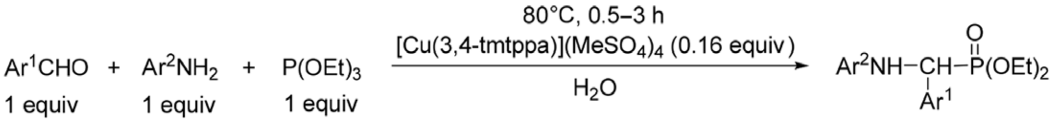
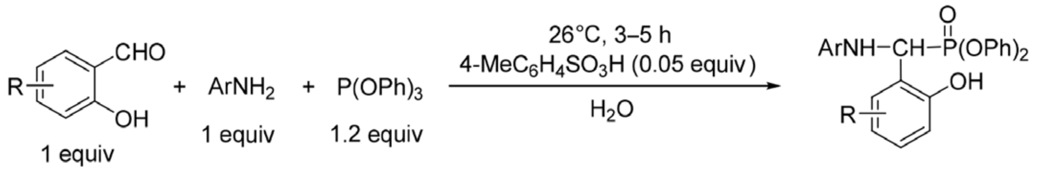
It can be seen that the special Kabachnik–Fields reactions applying trialkyl phosphites (instead of dialkyl phosphites) in water as the reaction medium may be accomplished efficiently. But what may be the reason that a few chemists chose water as the solvent for the reactions under discussion? On the one hand, these days there are efforts to carry out reactions in green solvents, such as water. This is, however, not a right argument here; as it was shown above, the Kabachnik–Fields condensations may be best accomplished without any solvent if a dialkyl phosphite is the P-component [14]. Moreover, water is not a real solvent here as from among the organic components applied, only diethyl phosphite and benzylamine are soluble in water. The other reason supporting the use of water is that it may have some role in the course of the reaction. The plausible mechanism assumed also by other authors is shown in Scheme 1 [25]. In the original version [25], the acid is boric acid and the anion is $(HO)_2BO^-$ (or in general, a deprotonated acid); however, the proton may also derive from water. The first step is the condensation of the aldehyde and primary amine to provide the imine by dehydration. Then, a molecule of trialkyl phosphite is added on the protonated C=N bond of the imine. Then, the adduct is stabilized by an Arbuzov fission to result in the α-aminophosphonate. One can see that water has two roles in the course of the reaction. The first role is to protonate the nitrogen atom of the imine. The second role is to serve as a counter ion in the phosphonium salt intermediate before the Arbuzov stabilization.

In the light of the above mechanism, it is clear that the presence of water may be advantageous for the Kabachnik–Fields condensations using trialkyl phosphites. However, there is no need to add water to the reaction mixture, as one molecule of water is formed in the reaction of aldehyde and primary amine that, basically, may promote further reaction as a protonating agent and as the source of the hydroxy counter ion in the phosphonium salt. This may have been recognized by Karimi-Jaberi and co-worker, when they reacted aromatic aldehydes, aromatic amines, and trimethyl phosphite at room temperature without any solvent. Ten mole percent of boric acid was used as the catalyst that may also act a protonating agent and may also provide an anion. The α-aminophosphonates were obtained after 15–60 min reaction times in yields of 87–98% (Scheme 2) [25].

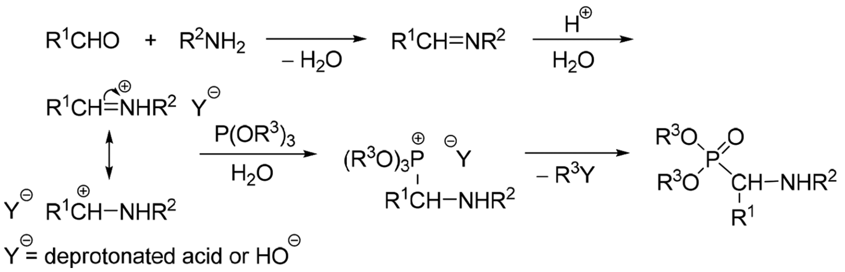
In other solvent-free variations, an ionic liquid with sulfonic acid function [26] or nano-TiO_2 [27] was used as catalysts or $TiCl_4$ was applied in dichloromethane [28].

In overall, the addition of a certain quantity (a few equivalents) of water to the reaction

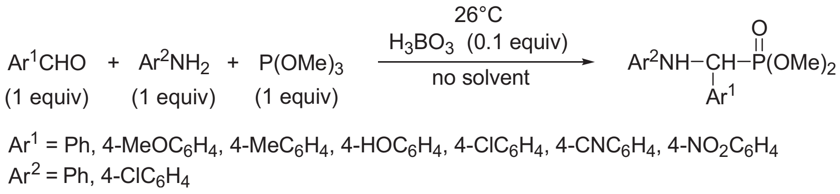
TABLE 1 Kabachnik–Fields Reactions Applying Trialkyl Phosphite as the P-Reagent in Water as the Medium

Entry	*Model Reaction*	*Ref.*
1	 53–95% R^1 = Ph, 4-ClC_6H_4, 3-ClC_6H_4, PhC_2H_4, PhC_2H_2 R^2 = Ph, Ph_2CH, 2-$MeOC_6H_4$, Bn	[20]
2	 29–84% Ar = Ph, 4-ClC_6H_4, 4-$MeOC_6H_4$ Y = ^{i}Pr, Bn, PhMeCH, Ph, 2-$MeOC_6H_4$, 4-ClC_6H_4	[19]
3	 89–96% Ar^1 = Ph, 4-$MeOC_6H_4$, 4-ClC_6H_4, 4-$NO_2C_6H_4$ Ar^2 = Ph, 3-$NO_2C_6H_4$	[18,21]
4	 61–77% (85–95%)* Ar^1 = Ph, 2-MeC_6H_4, 4-$MeOC_6H_4$, 4-$NO_2C_6H_4$, 2-ClC_6H_4, 2,6-diClC_6H_3, 4-HOC_6H_4 Ar^2 = Ph, 4-MeC_6H_4, 4-ClC_6H_4, 4-$MeOC_6H_4$	[22]
5	 tmtppa = tetramethyl-tetra-3,4-pyridinoporphyrazinato 90–98% Ar^1 = Ph, 4-$MeOC_6H_4$, 4-ClC_6H_4, 2,6-diClC_6H_3 Ar^2 = Ph, 4-MeC_6H_4, 4-ClC_6H_4	[23]
6	 82–94% R = H, 5-Cl, 5-Br, 5-NO_2 Ar = Ph, 2-$MeOC_6H_4$, 4-MeC_6H_4, 2-MeC_6H_4, 4-ClC_6H_4, 4-BrC_6H_4, 2,5-diClC_6H_3, 3-$NO_2C_6H_4$,	[24]

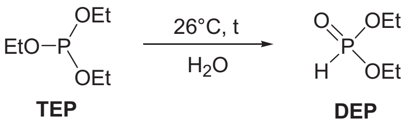
*Under ultrasound.



SCHEME 1



SCHEME 2



SCHEME 3

mixture seems to be reasonable to promote the Kabachnik–Fields condensation. However, there is no need to use water in an unlimited quantity, as it cannot serve as a real solvent. We planned to study the Kabachnik–Fields condensations in water medium ensuring a 2–4 mmol/mL concentration of the components (0.11 mL (1.0 mmol) of benzylamine, 0.10 mL (1.0 mmol) of benzaldehyde, and 1.2 mmol of the P-reagent (0.20 mL of triethyl phosphite or 0.15 mL of diethyl phosphite), or 2.0 mmol of the P-reagent (0.34 mL of triethyl phosphite or 0.26 mL of diethyl phosphite) in 0.5 mL of water).

TABLE 2 Hydrolysis of Triethyl Phosphite on Stirring in Water at 26°C[a]

			Composition (%)[b]	
Entry	*Additive*	*Time*	*TEP*	*DEP*
1/1	–	5 min	87	13
1/2	–	15 min	32	68
1/3	–	30 min	7	93
1/4	–	1 h	0	100
2	10% of PTSA	<10 min	0	100

[a] Stirring 0.20 mL TEP in 0.5 mL of water.
[b] On the basis of GC.

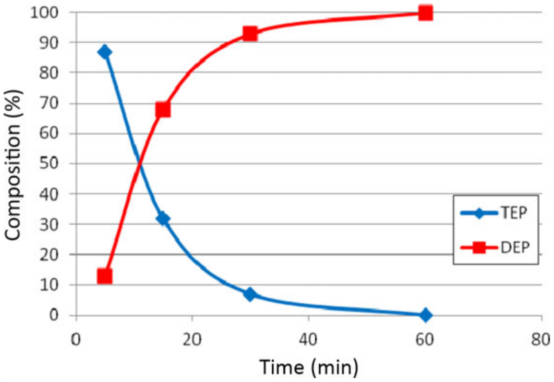


FIGURE 1 Hydrolysis of TEP to DEP in the absence of catalyst at 26°C.

RESULTS AND DISCUSSION

First of all, we wished to investigate the hydrolysis of triethyl phosphite (TEP) to diethyl phosphite (DEP) at 26°C in water (Scheme 3), as this side reaction may obviously influence the Kabachnik–Fields condensation. Our results are summarized in Table 2 and Fig. 1. It was found that the hydrolysis is relatively fast at room temperature. After 30 min, 93% of triethyl phosphite was hydrolyzed to diethyl phosphite (Table 2, entry 1/3). The hydrolysis was completed after 1 h (Table 2, entry 1/4). In the presence of 10% of PTSA, the hydrolysis was much faster, it was almost completed within 10 min (Table 2, entry 2).

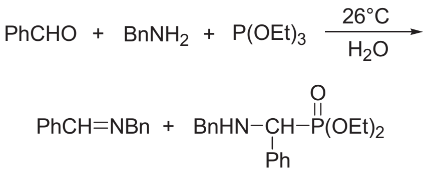
The ^{31}P NMR shifts of the P-species involved in our study are listed in Table 3. A small amount (≤3%) of triethyl phosphate was formed in all reactions as

TABLE 3 ^{31}P NMR Shifts for the Simple P-Species Involved

Compound	*δ_P (ppm)*	*δ_P^{lit} (ppm)*	*Reference*
$P(OEt)_3$	138.6	138.9	[29]
$(EtO)_2P(O)H$	7.31	8.0	[30]

$$\text{PhCHO} + \text{BnNH}_2 \xrightarrow[\text{H}_2\text{O}]{26°\text{C}} \text{PhCH=NBn} + \text{H}_2\text{O}$$

SCHEME 4



$$\text{PhCHO} + \text{BnNH}_2 + \text{P(OEt)}_3 \xrightarrow[\text{H}_2\text{O}]{26°\text{C}} \text{PhCH=NBn} + \text{BnHN-}\underset{\text{Ph}}{\text{CH}}\text{-}\overset{\text{O}}{\overset{\|}{\text{P}}}\text{(OEt)}_2$$

AP

SCHEME 5

a consequence of oxidation of the triethyl phosphite (δ_P (CDCl$_3$) –1.3, δ_P [29] –0.8).

It can be seen that the hydrolysis of the trialkyl phosphites may be a competent side reaction, when the Kabachnik–Fields reaction is carried out in water. In any case, it is advisable to use the trialkyl phosphite in an excess to provide an enough quantity of the reactant.

In the next part of our work, we studied the formation of imine from the aldehyde in water. Our model was the reaction of benzaldehyde with benzylamine (Scheme 4).

LC-MS analysis revealed that using equimolar quantities of the reactants (2–2 mmol) in water (1 mL), the condensation was completed after 6 min. One can conclude that the formation of an imine is quite fast under the conditions applied.

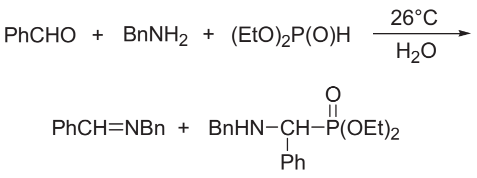
Then, we tried to carry out the Kabachnik–Fields condensation of benzaldehyde, benzylamine, and 1.2 or 2 equiv of triethyl phosphite in water at 26°C (Scheme 5, Table 4, Fig. 2). Without any catalyst and using only 1.2 equiv of the phosphite, the reaction was slow and incomplete even after a prolonged reaction time (Table 4, entry 1/4). Using 2 equiv of triethyl phosphite, the situation was better and the reaction was almost complete (Table 4, entry 2/4). The presence of 10% of PTSA was helpful in the case, when only 1.2 equiv of phosphite was used (Table 4, entries 3/4 and 4/3 vs. entries 1/4 and 2/4).

It can be said that within a time range of 10 min, triethyl phosphite is the major P-reagent, and then diethyl phosphite, which is formed from triethyl phosphite by hydrolysis, becomes the predominant P-nuchleophile that may react with the protonated imine. After 40 min, there is practically no triethyl phosphite in the mixture. In case of a higher excess of the phosphite, there is a bigger chance for the reaction of imine with the triethyl phosphite. As it was shown, the PTSA catalyst somewhat accelerated the process.

TABLE 4 Kabachnik–Fields Reaction of Benzaldehyde, Benzylamine, and Triethyl Phosphite at 26°C in Water

				Composition (%)^a		
Entry	*Additive*	*TEP (equiv)*	*Time*	*Benzaldehyde*	*Imine*	*AP*
1/1	–	1.2	3 h	3	86	11
1/2			6 h	3	81	35
1/3			10 h	3	33	64
1/4			1 Day	4	19	77^b
2/1	–	2	1 h	3	90	7
2/2			3 h	5	31	64
2/3			6 h	3	17	80
2/4			10 h	4	4	92^b
3/1	10% of PTSA	1.2	1 h	6	48	46
3/2			3 h	4	24	72
3/3			10 h	6	18	76
3/4			1 Day	4	10	86^b
4/1	10% of PTSA	2	1 h	4	48	48
4/2			3 h	4	13	83
4/3			10 h	2	3	95^b

^{a}On the basis of GC.
^{b}There was no change for further stirring.



$$\text{PhCHO} + \text{BnNH}_2 + \text{(EtO)}_2\text{P(O)H} \xrightarrow[\text{H}_2\text{O}]{26°\text{C}} \text{PhCH=NBn} + \text{BnHN-}\underset{\text{Ph}}{\text{CH}}\text{-}\overset{\text{O}}{\overset{\|}{\text{P}}}\text{(OEt)}_2$$

AP

SCHEME 6

On the basis of our experience, the use of triethyl phosphite in aqueous medium in Kabachnik–Fields reactions can be questioned due to the hydrolysis of the reagent to diethyl phosphite. In these condensations, diethyl phosphite is an in situ formed reagent concurring with triethyl phosphite, and replacing it at a certain point.

For this, the condensation was investigated with diethyl phosphite as well. The reaction conditions were similar as applied above (Scheme 6, Table 5). It was found that the condensation with diethyl phosphite at 26°C in water took place slower than the reaction with triethyl phosphite. The increase in the amount of diethyl phosphite was somewhat helpful, as the α-aminophosphonate was formed in a bigger proportion (Table 5, entries 1/2 and 2/2). In the presence of PTSA, the bigger amount of DEP had a

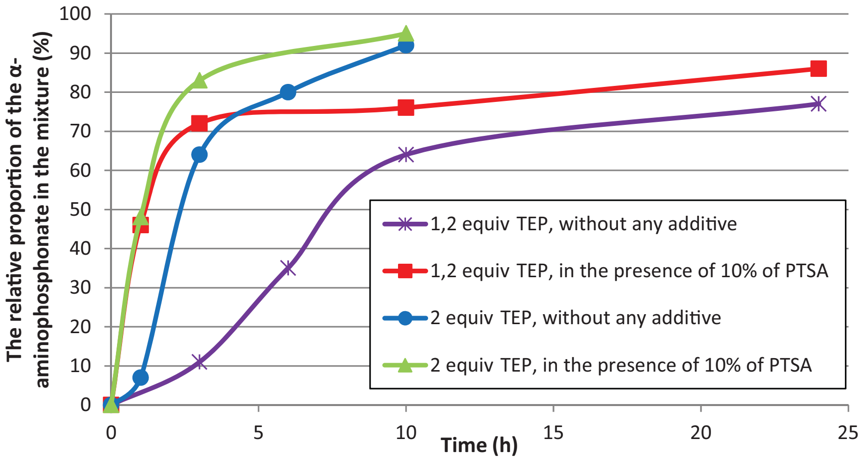


FIGURE 2 Time dependence of the relative proportion of aminophosphonate (AP) in the reaction of benzaldehyde, benzylamine, and triethyl phosphite at 26°C in water.

TABLE 5 Kabachnik–Fields Reaction of Benzaldehyde, Benzylamine, and Diethyl Phosphite at 26°C in Water

Entry	*Additive*	*DEP (equiv)*	*Time*	*Composition (%)*[a] *Benzaldehyde*	*Imine*	*AP*
1/1	–	1.2	3 h	9	65	26
1/2			1 Day	13	33	54[b]
2/1	–	2	3 h	11	68	21
2/2			1 Day	16	12	72[b]
3/1	10% of PTSA	1.2	3 h	9	63	28
3/2			1 Day	13	24	63[b]
4/1	10% of PTSA	2	3 h	10	62	28
4/2			1 Day	15	9	76[b]

[a]On the basis of GC.
[b]There was no change for further stirring.

less effect (Table 5, entries 3/2 and 4/2). While with triethyl phosphite, the best result was 95% after 10 h and with diethyl phosphite the maximum conversion was 76% after 1 day (Table 4, entry 4/3 and Table 5, entry 4/2). The reason for the longer reaction time is that the aqueous medium slows down the bimolecular reaction of diethyl phosphite and the imine, as water dilutes the reaction components, and the imine is only slightly soluble in water.

We wished to study the effect of the temperature on the reaction, thus a few experiments were carried out with 1.2 equiv of diethyl phosphite without catalyst in water at 40, 80, and 100°C (Table 6). It was shown that at 26°C after 1 day, 54% of aminophosphonate was obtained (Table 5, entry 1/2). At 40, 80, and 100°C, the ratio of the product was 51, 59, and 73%, respectively. No complete reaction could be achieved without a catalyst. In the presence of 10% of PTSA, the reaction was completed after 4 h at 100°C (Table 6, entry 4).

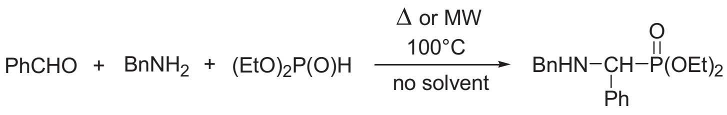
TABLE 6 Temperature Dependence of Kabachnik–Fields Reaction of Benzaldehyde, Benzylamine, and Diethyl Phosphite in Water

Entry	*Additive*	*T (°C)*	*Time*	*Composition (%)*[a] *Benzaldehyde*	*Imine*	*AP*
1	–	40	1 Day	11	30	51
2	–	80	6 h	23	26	59[b]
3	–	100	6 h	11	16	73[b]
4	10% of PTSA	100	4 h	8	12	80[b]

[a]On the basis of GC.
[b]There was no change for further stirring.

It can be seen that the Kabachnik–Fields reactions with diethyl phosphite in water were not complete even at higher temperature without catalyst. The aqueous medium inhibited the reaction of diethyl phosphite. Finally, the reaction of benzaldehyde, benzylamine, and 1.2 equiv of diethyl phosphite was carried out under solvent-free conditions, first on conventional heating and then under MW conditions (Scheme 7, Table 7). It was found that the condensation was complete under both variations; however, under MW conditions, the reaction was much faster (Table 7, entries 1 and 2).

In conclusion, in the condensation with diethyl phosphite, there is no need for any solvent (water) and catalyst as compared to the reaction with triethyl phosphite. In addition, the triethyl phosphite is malodorous and the use of excess of this reagent means extra cost. Moreover, using water as the solvent in the reaction with triethyl phosphite, it is hydrolyzed fast resulting in the formation of diethyl



$PhCHO + BnNH_2 + (EtO)_2P(O)H \xrightarrow[\text{no solvent}]{\Delta \text{ or MW, } 100°C} BnHN{-}CH(Ph){-}P(O)(OEt)_2$

AP

SCHEME 7

TABLE 7 Kabachnik–Fields Reaction of Benzaldehyde, Benzylamine, and Diethyl Phosphite under Solventless Conditions

Entry	*Mode of Heating*	*Time*	*Composition of AP (%)*[a]
1	Δ	>1 h	~100
2	MW	20 min	100

[a]On the basis of GC.

phosphite and, hence resulting in a complicated reaction mixture, in which both P-reagents may react in a competitive way.

The overall conclusion is that for the preparation of the desired α-aminophosphonates, the application of dialkyl phosphites is preferable in the Kabachnik–Fields reaction, as this reaction can be accomplished without any catalyst, in a solvent-free manner. However, together with suitable catalysts, trialkyl phosphites may also be used with water as the solvent. A better alternative is the solvent-free use of trialkyl phosphites in the presence of suitable catalysts.

EXPERIMENTAL

General

The MW-assisted reaction was carried out in a CEM Discover (300 W) MW reactor equipped with a pressure controller using 50 W irradiation.

GC was carried out on an HP5890 series 2 GC-FID chromatograph using a 15 m × 0.18 mm Restek, Rtx-5 column with a film layer of 0.20 μm. The temperature of the column was initially held at 40°C for 1 min, followed by a 25°C/min program up to 300°C and a final isothermal period at 300°C for 10 min. The temperature of the injector was 290°C and of the FID detector was 300°C. The carrier gas was N_2.

GC-MS was carried out on an Agilent 6890 N-GC-5973 N-MSD chromatograph using a 30 m × 0.25 mm Restek, Rtx-5SILMS column with a film layer of 0.25 μm. The initial temperature of column was 45°C for 1 min, followed by a 10°C/min program up to 310°C and a final isothermal period at 310°C for 17 min. The temperature of the injector was 250°C. The carrier gas was He and the operation mode was splitless.

^{31}P NMR spectra were obtained in $CDCl_3$ solution on a Bruker AV-300 spectrometer operating at 121.5 MHz. Chemical shifts are downfield relative to 85% H_3PO_4. In the experiments, distilled water was used with a pH of 7.0.

General Procedure for the Hydrolysis of Triethyl Phosphite

The mixture of 0.20 mL (1.2 mmol) of triethyl phosphite, 0.50 mL (28.0 mmol) of distilled water, and in certain cases 0.019 g (0.1 mmol) of PTSA was stirred at 26°C for the appropriate time. The reaction mixture was then washed with 10 mL of dichloromethane in a separating funnel and 10 mL of distilled water was also added, then the water phase was extracted two times with 10 mL of dichloromethane and the combined organic phase dried (Na_2SO_4). Evaporation of the volatile components provided the crude product that was analyzed by GC and/or GC-MS (see Table 2).

General Procedure for the Kabachnik–Fields Reaction in Aqueous Medium

The mixture of 1.0 mmol (0.11 mL) of benzyl amine, 1.0 mmol (0.10 mL) of benzaldehyde, 1.2 mmol of phosphite (0.20 mL of triethyl phosphite or 0.15 mL of diethyl phosphite) or 2.0 mmol of phosphite (0.34 mL of triethyl phosphite or 0.26 mL of diethyl phosphite), 0.50 mL (28 mmol) of distilled water, and in certain cases 0.019 g (0.10 mmol) of PTSA was stirred at appropriate temperature for the appropriate time (see Tables 4 and 5). The reaction mixture was worked up similarly as above. The crude products were analyzed by GC and/or GC-MS.

The reactions at higher temperature (Table 6) were carried out similarly.

General Procedure for the Kabachnik–Fields Reaction under Solvent-Free Condition

The mixture of 1.0 mmol (0.11 mL) of benzyl amine, 1.0 mmol (0.10 mL) of benzaldehyde, and 1.2 mmol (0.15 mL) of diethyl phosphite was heated at 100°C in a vial in an oil bath or in a CEM Discover MW reactor for the appropriate time. Then, the water formed was removed in vacuum. The crude product obtained as a yellow oil was analyzed by GC and/or GC-MS (for the details, see Table 7).

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