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ORIGINAL PAPER

Formic acid catalyzed one-pot synthesis of α-aminophosphonates: an efficient, inexpensive and environmental friendly organocatalyst

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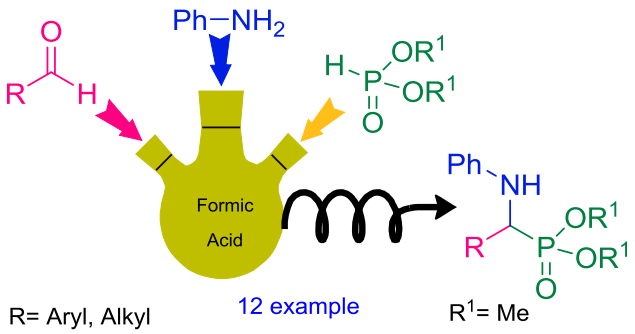
Abstract
Aqueous formic acid is used for the synthesis of α-aminophosphonates through Kabachnik–Fields reaction applying aromatic amine, phosphite, and carbonyl compounds. Using formic acid as an efficient and low-cost organocatalyst provides environmental friendly, high yields, low reaction time and mild reaction condition. The isolated products were analyzed by IR, NMR, and mass techniques.

Graphical abstract



Keywords α-Aminophosphonates · Formic acid · Organocatalyst · Kabachnik–Fields reaction

Introduction

α-Aminophosphonates have always been in the center of attention among the organophosphorus compounds. α-Aminophosphonates are structural analogues of the corresponding α-amino acids in which a carboxylic motif is replaced by phosphonic acid or related groups. Due to interesting biological activities such as antifungal (Maier and Diel 1991), antibacterial (Atherton et al. 1986; Eshtiaghi 2013), anticancer activity (Huang and Chen 2000; Kafarski and Lejczak 2001; Lavielle et al. 1991; Prasad and Rao 2013), enzyme inhibitors (Giannousis and Bartlett 1987), catalytic antibodies (Hirschmann et al. 1994), antibiotics and pharmacologic agents (Atherton et al. 1986; Baylis et al. 1984), aminophosphonates are considered as an important class of compounds. Additionally, it is found that they can act as antagonists of amino acids through inhibition of the metabolism of the involved enzymes. Thus, affects the physiological activities of the cell. These effects may be used as peptide mimics, plant growth regulatory or neuromodulatory (Kafarski and Lejczak 1991). Anti-HIV effect of some α-aminophosphonates and phosphoramidates derivatives

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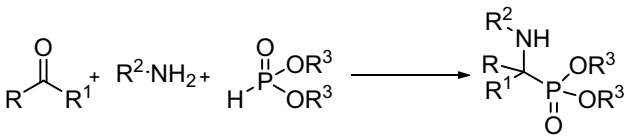
have been reported (Bhattacharya et al. 2012; Bonini et al. 2005; Peyman et al. 1994). Furthermore, aminophosphonates were used in the synthesis of new radioprotecting agents (Sal'keeva et al. 2002).

Therefore, a variety of synthetic approaches have been developed for synthesis of α-amino phosphonates during the last decades. Two main pathways have been developed for the synthesis of α-aminophosphonates: Pudovik reaction and Kabachnik–Fields reaction (Scheme 1). The Kabachnik–Fields reaction is a Mannich-type three-component coupling of a carbonyl, an amine, and a hydrophosphoryl compound in the presence of either a base or an acid that leads to α-aminophosphonates (Wang 2010). This reaction was discovered by Kabachnik and Fields (Fields 1952; Kabachnik and Medved 1952, 1953).

Literature survey reveals that the corresponding three-component reaction is carried out in the presence of Lewis and Brønsted acids such as: LPDE (Heydari et al. 1998), $SnCl_4$ (Laschat and Kunz 1992), $Mg(ClO_4)_2$ (Wu et al. 2006), BF_3–OEt_2 (Ha and Nam 1992), $FeCl_3$ (Rezaei et al. 2009), $AlCl_3$ (Manjula et al. 2003), TiO_2 (Hosseini-Sarvari 2008), TiO_2–SiO_2 (Chinthaparthi et al. 2013), SiO_2/$ZnCl_2$ (Subba et al. 2013), ZnI_2 (Karimi-Jaberi et al. 2012), $BiCl_3$ (Zhan and Li 2005), $Bi(NO_3)_3$ (Bhattacharya and Kaur 2007), $Bi(OTf)_3$ (Banik et al. 2010), $H_3PW_{12}O_{40}$ (Heydari et al. 2007), $Cu(OTf)_2$ (Paraskar and Sudalai 2006), $SbCl_3/Al_2O_3$ (Ambica et al. 2008), $Ln(OTf)_3$ (Qian and Huang 1998), SmI_2 (Xu et al. 2003), $InCl_3$ (Ranu et al. 1999), $Yb(PFO)_3$ (Tang et al. 2011), $TaCl_5$–SiO_2 (Chandrasekhar et al. 2001), Zr^{4+} (Yadav et al. 2001a), $ZrOCl_2{\cdot}8H_2O$ (Bhanushali et al. 2009), $ZrCl_4$ (Yadav et al. 2001a), TMSCl (Pokalwar et al. 2010), CF_3COOH (Akiyama et al. 2003), scandium tris(dodecylsulfate) (Manabe and Kobayashi 2000), scandium(III)-*N*,*N*′-dioxide (Zhou et al. 2009), zinc di(L-prolinate) (Domingues et al. 2016), $HfCl_4$ (Li et al. 2016) and hypophosphorous acid (Kaboudin and Jafari 2008). Rostamnia and Amini (2014) used combined ultrasonic (US) and [bmim]$AlCl_4$ ionic liquid for Kabachnik–Fields reaction. Catalyst-free ultrasonic-promoted synthesis of some tertiary α-amino phosphonates was reported by Kalla et al. (2017).

Quantitative synthesis of α-hydroxyphosphonates and α-aminophosphonates from aldehydes and imines in solvent-free condition in the presence of chlorotrimethylsilane (Pokalwar et al. 2010), solid $LiClO_4$ (Azizi et al. 2004), $Al(H_2PO_4)_3$ (Maghsoodlou et al. 2010) and $Na_2CaP_2O_7$ (Zahouily et al. 2005) at room temperature and short reaction time was introduced as a green process. Veeranjaneyulu reported the synthesis of α-amino phosphonates by reaction of α-amido sulfones with dialkyl trimethylsilyl phosphites in the catalytic presence of $FeCl_3$ (Veeranjaneyulu and Das 2017).



Scheme 1 Kabachnik–Fields reaction

The above-mentioned approaches mainly suffer from significant limitations in terms of long reaction time and low yields. In addition to high-cost, moisture sensitivity, highly toxic or toxic, using the stoichiometric amount of catalyst is the constant feature of many transformations in this regard. In recent years many efforts have been conducted to improve the reaction condition. There are some reports using one-pot procedures, microwave or solvent-free conditions (Bálint et al. 2015; Chandra Sekhar Reddy et al. 2014; Gyorgy and Anna 2008; Keglevich and Balint 2012; Thaslim Basha et al. 2016; Tibhe et al. 2012).

In recent years many efforts have been conducted to improve the reaction condition. Among these cases, oxalic and citric acid (Hellal et al. 2016; Vahdat et al. 2008), tetramethyl guanidine (TMG) (Reddy et al. 2010), dehydroascorbic acid (DHAA) (Saberi et al. 2013), graphene oxide (Dhopte et al. 2015), Triton X-100 (Reddy et al. 2016), 2,3-dibromo succinic acid (Hazeri and Aboonajmi 2014), bromodimethylsulfonium bromide (Kudrimoti and Bommena 2005), glycerol (Azizi et al. 2014), $PhNMe_3Cl$ (Heydari and Arefi 2007), [BMIM]Cl (Bai et al. 2011), Trifluoroethanol (Heydari et al. 2009) and pentafluorophenylammonium triflate (PFPAT) (Malamiri and Khaksar 2014) are used as catalyst for this reaction. Additionally, some recyclable catalysis such as $HClO_4$–SiO_2 (Maghsoodlou et al. 2011), amberlite IRC-748 (Shashikumar 2013), natrolite zeolite (Bahari and Sajadi 2012), montmorillonite clay (Yadav et al. 2001b), Fe_3O_4@ZrO_2/SO_4 (Ghafuri et al. 2016b), DHAA-Fe_3O_4 (Saberi et al. 2013), Fe_3O_4@SiO_2-imid-PMA^n (Esmaeilpour et al. 2016), have been documented for synthesis of α-aminophosphonates.

Formic acid or methanoic acid, HCOOH, is a colorless, corrosive liquid with a pungent odor. It is completely soluble in water and many polar solvents. It is sufficiently stronger than carboxylic acids with longer chains. Formic acid has aldehydic nature and reducing properties and it is used in many of organic transformations. In comparison with mineral acids, formic acid evaporates without leaving any residue (Hietala et al. 2000).

In recent years, much attention has been made to formic acid. Formic acid (HCOOH, FA) is the simplest carboxylic acid. Formic acid has low toxicity (hence its use as a food additive), with an LD_{50} of 1.8 g/kg (tested orally in mice) (Hietala et al. 2000). Its liquid form and low toxicity render its transportation, refuelling, and handling straightforward. It is used as a hydrogen storage (Czaun et al. 2011; Eppinger

and Huang 2017; Joó 2008; Müller et al. 2017; Sordakis et al. 2017). Formic acid is decomposed to H_2 and CO_2 in the presence of $RuCl_3$ (Czaun et al. 2014). The catalytic effect of formic acid in the rearrangement of Thevinols to Thevinals is documented (Grundt et al. 2003). Very recently, aqueous formic acid is used as efficient, inexpensive and environmentally friendly organocatalyst for the synthesis of β-amino carbonyl derivatives (Ghafuri et al. 2015). Additionally, three-component Strecker synthesis of α-aminonitriles and imines with excellent yields is available (Ghafuri and Roshani 2014).

Based on these reports, we decided to explore the possibility of three-component Kabachnik–Fields reaction for the preparation of α-aminophosphonates from amines, phosphites, and carbonyl compounds in aqueous formic acid–ethanol (Scheme 2). Herein, we report a green and effective method for the synthesis of α-aminophosphonates in the presence of aqueous formic acid and ethanol, which is an inexpensive and highly efficient catalyst. This multicomponent reaction was done at room temperature with easy work-up.

Experimental

All purchased solvents and chemicals were of analytical grade and used without further purification. FT-IR spectra were obtained over the region 400–4000 cm^{-1} with a NICOLET IR100 FT-IR spectrometer with spectroscopic grade KBr. Mass spectra were measured on an agilent spectrometer (equipped with a direct inlet probe). The ^1H NMR spectra were obtained with a BRUKER 250.1 MHz instrument using $CDCl_3$ as the applied solvent and TMS as the internal standard.

Results and discussion

General procedure for synthesis of α-aminophosphonates

Aqueous formic acid–ethanol solution was prepared from 1 ml formic acid (37%) and 4 ml ethanol (final concentration of formic acid is about 7%). Aldehyde (1.0 mmol) and aniline (1.1 mmol) was added to aqueous formic acid–ethanol solution at room temperature and after 5 min dialkyl phosphite (1.1 mmol) was added to the mixture. After completion the reaction, solvents were evaporated under reduced pressure. Water was added to the reaction mixture and the resulting solution was neutralized by sodium bicarbonate. The reaction product was extracted with dichloromethane. The crude mixture was purified by chromatography (hexane:ethylacetate; 3:1) to afford pure products.

First, the three-component reaction of benzaldehyde, aniline, and dimethyl phosphite in aqueous formic acid–ethanol at room temperature was selected as a model reaction.

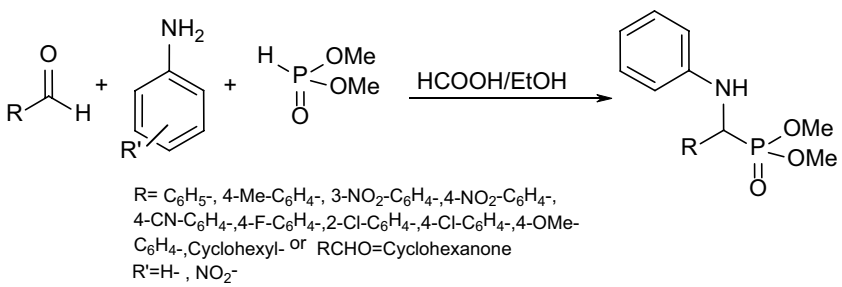
Next, the optimization of time and temperature was investigated. The reaction was carried out at three different temperatures, 20, 40 and 60 °C. According to these experiments, formation of imine was completed in the first 10 min at each temperature (with a little difference). After that, dimethylphosphite was added. The disappearance of aldehyde and formation of α-aminophosphonate was followed with TLC. Among the evaluated conditions, performing the reaction at room temperature during 30 min was selected as an optimized condition.

To investigate the effect of formic acid as a catalyst, the reaction of benzaldehyde, aniline, and dimethyl phosphite was repeated in the absence of formic acid. After 12 h at 60–70 °C, no product was isolated.

Several α-aminophosphonates were prepared based on the optimized reaction conditions (Table 1). Both aromatic and aliphatic aldehydes provided excellent yields of the desired products (60–90%) in short reaction times. Additionally, cyclohexanone gave corresponding phosphonate in good yield (70%). 4-Methoxybenzaldehyde afforded the desired adduct in a longer time and higher temperature.

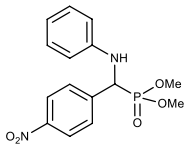
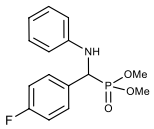
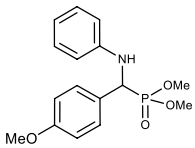
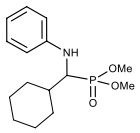
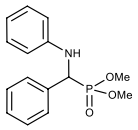
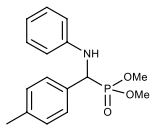
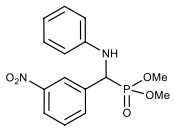
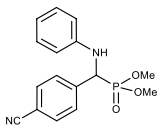
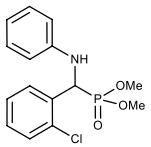
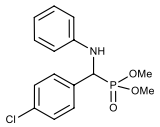
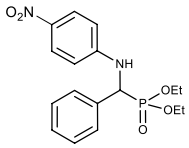
Aliphatic amines give very poor results in this test. Additionally, the better results were obtained for aromatic amines rather than aliphatic amines. The reaction of benzaldehyde, *n*-butylamine, aniline and dimethyl phosphite in aqueous formic acid–ethanol solution gave the aromatic derivative α-aminophosphonate (Scheme 3).

It seems that the hydrogen-bonding activation or protonation of aldehydes and imines with formic acid is in place for catalysis of the Kabachnik–Fields reaction. The proposed



Scheme 2 Kabachnik–Fields reaction in aqueous formic acid–ethanol

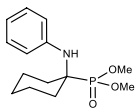
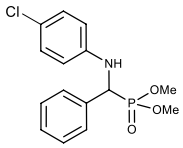
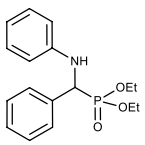
Table 1 Products of the Kabachnik–Fields reaction in aqueous formic acid–ethanol

Entry	Product	Temp (°C)	Time (h)	Yield (isolated) (%)	Melting point (Lit. mp) °C	References
1		r.t	2	93	123–125 (124–128)	Ghafuri et al. (2016b); Heydari and Arefi (2007); Lukanov and Venkov (1992); Mu et al. (2006); Zhan and Li (2005)
2		r.t	3	80	103–105 (105–106)	Lukanov and Venkov (1992)
3		60	3	75	121–124 (123–124)	Bhagat and Chakraborti (2007); Ghafuri et al. (2016b); Karimi-Jaberi et al. (2012); Kudrimoti and Bommena (2005); Mu et al. (2006); Saidi and Azizi (2002)
4		r.t	3	91	73–75 (74–75)	Bhagat et al. (2014)
5		r.t	3	90	88–90 (90–92)	Azizi and Saidi (2003); Bhagat and Chakraborti (2007); Ghafuri et al. (2016b); Mu et al. (2006); Mulla et al. (2014); Saidi and Azizi (2002); Zhan and Li (2005)
6		r.t	3	90	116–118 (116–118)	Karimi-Jaberi et al. (2012); Kudrimoti and Bommena (2005); Lukanov and Venkov (1992); Mulla et al. (2014)
7		r.t	2	85	121–123 (122–124)	Ghafuri et al. (2016b); Saidi and Azizi (2002)
8		r.t	2	90	Oily	Bhagat and Chakraborti (2008)
9		r.t	2	75	127–129 (128–129)	Ghafuri et al. (2016b)
10		r.t	2	85	137–140 (139–140)	Bhagat and Chakraborti (2007); Lukanov and Venkov (1992); Mulla et al. (2014); Saidi and Azizi (2002); Vahdat et al. (2008)
11		60 °C	3	90	124–126 (123–125)	Peng et al. (2015)

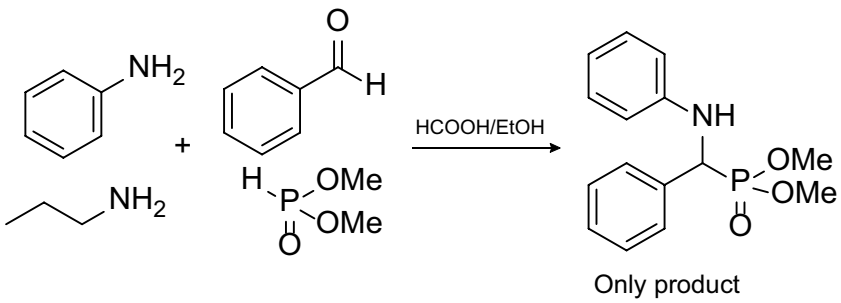
mechanism for synthesis of α-aminophosphonates is shown in Scheme 4.

According to this mechanism, formic acid catalyzed the in situ formation of the imine intermediate, through generation of hydrogen bonds between the hydroxyl groups and the oxygen atom of the carbonyl group. In the presence of formic acid, the imine carbon is attacked by dimethyl phosphite to give the desired product. According to our findings, the results show that the electronic of substitutes (on amine and aldehyde) have a pivotal role in the reaction. This is as follows, electron donating substitutes that increase the electron density on carbonyl and imine group decreases nucleophilic addition. Spectral analysis were consistent with the known derivatives (Azizi et al. 2004; Azizi and Saidi 2003; Bhagat and Chakraborti 2007; Bhattacharya and Kaur 2007; Dhopte et al. 2015; Ghafuri et al. 2016b; Hazeri and Aboonajmi

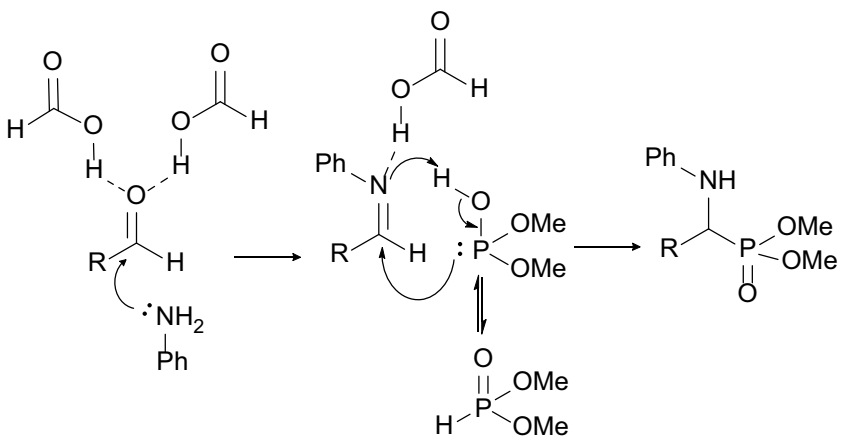
Table 1 (continued)

Entry	Product	Temp (°C)	Time (h)	Yield (isolated) (%)	Melting point (Lit. mp) °C	References
12		r.t	3	70	103–105 (99–102)	Bhagat and Chakraborti (2007); Ghafuri et al. (2016a); Saidi and Azizi (2002)
13		60 °C	3	90	87–89 (88–90)	Mu et al. (2006)
14		r.t	3	85	87–88 (86)	Hosseini-Sarvari (2008)

Scheme 3 Selectivity of the Kabachnik–Fields reaction (α-aminophosphonates)



Scheme 4 A plausible mechanism for synthesis of α-aminophosphonates in aqueous formic acid–ethanol



2014; Hosseini-Sarvari 2008; Maghsoodlou et al. 2010; Saberi et al. 2013; Vahdat et al. 2008). Because most of the products have been characterized, analytical identification of the synthesized adducts is limited to the infrared, MS and melting point for some products. Spectral data for selected derivatives:

Dimethyl [anilino(4-nitrophenyl)methyl] phosphonate (1): Melting point: 123–125 °C, IR (KBr cm^{-1}): 3305 (N–H), 2924 (C–H), 1601 (C=C), 1518 (C=C), 1284 (P=O), 1039 (P–O–C); ^{1}H NMR (250 MHz, $CDCl_3$): $\delta = 3.62$ (3H, d, $^{3}J_{PH} = 10.7$ Hz, OCH_3), 3.80 (3H, d, $^{3}J_{PH} = 10.7$ Hz, OCH_3), 4.9 (1H, d, $^{2}J_{PH} = 25.5$ Hz, CH), 5.30 (1H, br, NH), 6.55 (d, 2H), 6.69 (t, 1H), 7.1 (t, 2H), 7.66 (d, 2H), 8.2 (d, 2H); ^{13}C NMR (62.9 MHz, $CDCl_3$): $\delta = 53.9$ (d, $^{2}J_{CP} = 6.9$ Hz, OCH_3), 54.3 (d, $^{2}J_{CP} = 6.9$ Hz, OCH_3), 55.5 (d, $^{1}J_{CP} = 149.1$ Hz, CH), 113.8, 119.2, 123.9 (d, $^{4}J_{CP} = 3.5$ Hz), 128.6 (d, $^{3}J_{CP} = 11$ Hz), 129.4, 143.6 (d, $^{3}J_{CP} = 3.2$ Hz), 145.3 (d, $^{2}J_{CP} = 14.2$ Hz), 147.7; ^{31}P NMR (101.25 MHz, $CDCl_3$): $\delta = 23.9$; $C_{15}H_{17}N_2O_5P$ (M^+) 336.09, found 336.4.

Dimethyl [anilino(4-fluorophenyl)methyl] phosphonate (2): Melting point: 103–105 °C, IR (KBr cm^{-1}): 3306 (N–H), 2954-3045 (C–H), 1602 (C=C), 1504 (C=C), 1239 (P=O), 1039 (P–O–C); 1H NMR (250 MHz, $CDCl_3$): $\delta = 3.50$ (3H, d, $^{3}J_{PH} = 10.5$ Hz, OCH_3), 3.75 (3H, d, $^{3}J_{PH} = 10.7$ Hz, OCH_3), 4.81 (1H, d, $^{2}J_{PH} = 24.3$ Hz, CH), 5.03 (1H, br, NH), 6.59 (d, 2H), 6.69 (t, 1H), 7.01 (t, 2H), 7.09 (t, 2H), 7.46 (m, 2H); ^{13}C NMR (62.9 MHz, $CDCl_3$): $\delta = 53.4$ (d, $^{2}J_{CP} = 6.9$ Hz, OCH_3), 53.5 (d, $^{2}J_{CP} = 6.8$ Hz, OCH_3), 54.46 (d, $^{1}J_{CP} = 153.2$ Hz, CH), 113.5, 115.3 (dd, $^{2}J_{CF} = 21.7$ Hz, $^{4}J_{CP} = 2.8$ Hz), 118.2, 128.8, 129.1 (dd, $^{3}J_{FC}$, $^{3}J_{PC} = 5.3$, 8.3 Hz), 131.1, 145.6 (d, $^{3}J_{PC} = 14.9$ Hz), 162.1 (dd, $^{3}J_{FC} = 247.6$ Hz, $^{5}J_{PC} = 4.3$ Hz); ^{31}P NMR (101.25 MHz, $CDCl_3$): $\delta = 24.9$; ^{19}F NMR (235.3 MHz, $CDCl_3$): $\delta = -113.9$; $C_{15}H_{17}FNO_3P$ (M^+) 309.28, found 309.4.

Dimethyl [anilino(4-methoxyphenyl)methyl] phosphonate (3): Melting point: 121–124 °C, IR (KBr cm^{-1}): ν 3290 (N–H), 2839–3038 (C–H), 1602 (C=C), 1503 (C=C), 1242 (P=O), 1024 (P–O–C); ^{1}H NMR (500 MHz, $CDCl_3$): $\delta = 3.47$ (d, 3H, $^{3}J_{PH} = 10.4$ Hz, P-OCH_3), 3.67 (d, 3H, $^{3}J_{PH} = 10.5$ Hz, P-OCH_3), 3.70 (s, 3H, OCH_3), 5.03 (dd, 1H, $^{2}J_{PH} = 25.6$ Hz, $^{3}J_{HH} = 107$ Hz, CH), 6.30 (br s, 1H, NH), 6.53 (t, 1H), 6.79 (d, 2H), 6.88 (d, 2H), 7.01 (t, 2H), 7.45 (d, 2H); ^{31}P NMR (101.25 MHz, CDCl3): $\delta = 25.51$.

Dimethyl [anilino(cyclohexyl)methyl]phosphonate (4): Melting point: 73–75 °C, IR (KBr cm^{-1}): ν 3317 (N–H), 2926, 2855 (C–H), 1601 (C=C), 1507 (C=C), 1262 (P=O), 1053 (P–O–C); ^{1}H NMR (250 MHz, $CDCl_3$): $\delta = 0.883$–2.176 (m, 11H, $-C_6H_{11}$), 3.65 (d, 3H, $^{3}J_{PH} = 10.5$ Hz, OCH_3), 3.70 (d, 3H, $^{3}J_{PH} = 10.5$ Hz, OCH_3), 3.86 (br, 1H), 5.30 (s, 1H), 6.65 (d, 2H), 6.71 (t, 1H), 7.17 (t, 2H); ^{13}C NMR (62.9 MHz, $CDCl_3$): $\delta = 16.1$ (CH_2), 18.4 (CH_2), 20.9 (CH_2), 29.8 (CH), 42.4 (d, $^{2}J_{CP} = 7.5$ Hz, OCH_3), 44.7(d, $^{2}J_{CP} = 7.5$ Hz, OCH_3), 45.8 (d, $^{1}J_{CP} = 154.1$ Hz, CH), 103.1, 107.9, 119.4, 145.7; ^{31}P NMR (101.25 MHz, CDCl3): $\delta = 119.4$; $C_{15}H_{24}NO_3P$ (M^+) 297.34, found 297.4.

Table 2 shows the comparison of the ^{1}H NMR results of synthesized products with previous reports, which shows that the results are in agreement.

Conclusions

Aqueous formic acid has been demonstrated to be a green, effective, inexpensive, insensitive to moisture and more accessible catalyst for synthesis of α-aminophosphonates through Kabachnik–Fields reaction in high yield and mild condition. To conclude, easy work-up, low-cost of catalyst and high efficiency make our method to be efficient and practical for synthesis of α-aminophosphonates. Further works need to be done to establish whether formic acid can be used in other acid catalyzed, nucleophilic addition and multi-component reactions in catalyst-free conditions.

Table 2 Comparison of the ^{1}H NMR results of synthesized products with previous reports

Entry	Obtained	Reported	References
(1)	3.62 (d, 3H), 3.80 (d, 3H), 4.9 (d, 1H), 5.30 (br, 1H), 6.55 (d, 2H), 6.69 (t, 1H), 7.1 (t, 2H), 7.66 (d, 2H), 8.2 (d, 2H);	3.60 (d, 3H), 3.78 (d, 3H), 4.80 (d, H), 6.53–8.21 (m, 9H)	Bhagat et al. (2014)
(2)	3.50 (d, 3H), 3.75 (d, 3H), 4.81 (d, 1H), 5.03 (br, 1H), 6.59 (d, 2H), 6.69 (t, 1H), 7.01 (t, 2H), 7.09 (t, 2H), 7.46 (m, 2H);	3.45–3.86(2s, 3H), 3.65–3.70 (2s, 3H), 4.58–4.95 (2S, 1H), 4.80(s, 1H), 6.55 (d, 2H), 6.60–7.60 (m, 5H), 7.05 (d, 2H)	Lukanov and Venkov (1992)
(3)	3.47 (d, 3H), 3.67 (d, 3H), 3.70 (s, 3H), 5.03 (dd, 1H), 6.30 (br, 1H), 6.53 (t, 1H), 6.79 (d, 2H), 6.88 (d, 2H), 7.01 (t, 2H), 7.45 (d, 2H)	3.47–3.50 (3H, d), 3.73–3.77 (6H, 2s), 4.70–4.78 (1H, d). 6.58–7.39 (9H, m)	Bhagat et al. (2014)
(4)	0.88–2.17 (m, 11H), 3.65 (d, 3H), 3.70 (d, 3H), 3.86 (br, 1H), 5.30 (s, 1H), 6.65 (d, 2H), 6.71 (t, 1H), 7.17 (t, 2H)	1.08–1.98 (11H, m), 3.6(3H, d), 3.7 (3H, d), 3.84–3.88 (1H, m) 6.6–7.2 (5H, m)	Bhagat et al. (2014)

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