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A green, one-pot, three-component and microwave assisted synthesis of α-sulfamidophosphonates

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Abstract

An easy and handy synthesis of α-sulfamidophosphonates directly by Three-Component reactions is reported. The reaction involves the use of aldehyde, sulfonamides and trimethylphosphite. A wide range of substrates is compatible in this reaction, producing excellent yields in short time. The reaction is performed under solvent- and catalyst-free conditions using microwave irradiation, greener conditions and lower generation of waste or pollution are the main advantages of this method.

1. Introduction

α-aminophosphonate derivatives belong to an important class of organophosphorus compounds that attracted much attention because these phosphonates scaffolds as core unit for a wide spectrum of natural product and pharmacological compounds such as *N*-phosphono-amino acids (NPAA) inhibitors of some enzymes [1-3]. phosphonoleucine inhibitors of leucineaminopeptidase [4].

Moreover, these derivatives exhibit various potential biological and pharmaceutical activities including exhibit enzyme inhibitory [5-9], antiviral [10], antibiotic [11], antibacterial [12], antifungal [13], herbicidal activities [14] and several applications in the agricultural industry [13,15], as well as effective ligands and extractants [16,17].

These important compounds have been synthesized by various routes [18]. Among the literature methods, the Kabachnik–Fields reaction is one of the most convenient approaches to α-aminophosphonates. The reaction usually needs various catalyst such as; SmI_2 [19], $LiClO_4$ [20], metal triflates ($M(OTf)_n$, M = Li, Mg, Al, Cu, Ce) [21], $TaCl_5$-SiO_2 [22], montmorillonite clay [23], Al_2O_3 [24], CF_3CO_2H [25], scandium(tris dodecyl sulfate) [26] $BF_3.Et_2O$ [27] and tetra-tertbutyl-substituted phthalocyanines Pht-1-Pht-3 [28] and Lewis acid such as Al_2O_3, BF_3, $ZnCl_2$, $MgBr_2$, and $SnCl_4$etc [21,19] have also been reported as the catalysts. However, many of these catalysts are expensive and have to be used in stoichiometric amount. The catalyst-free synthesis of α-aminophosphonates is rather limited [29].

Recently, we have synthesized new sulfamidophosphonates [30] by three components, one-pot reaction under solvent and catalyst free conditions using ultrasonic irradiation.

The phosphonates containing sulfonamide moiety have been described and have interesting biological properties. They act as potent inhibitors of protein tyrosine phosphatase 1B and HIV protease inhibitors [31-32].

Microwave-promoted solvent-free heterogeneous reactions have received much attention due to their high efficiency, cost effective, and environmentally friend characteristics.

In view of this, development of a green chemical synthesis for them is desirable. Microwave-assisted solvent-free reactions are the best option. They are environmentally benign and provide greater selectivity in the product formation, enhanced reaction rates, and simple manipulation. In continuation of our studies on eco-friendly

synthetic methodologies herein, we report the syntheses of α-sulfamidophosphonates by three components, one-pot Kabachnik–Fields reaction under solvent-and catalyst-free conditions using microwave irradiation (MW) (Scheme 1).

2. Experimental

2.1. General information

All chemicals were purchased from common commercial sources and were used as received without any further purification. All reactions were monitored by TLC on silica Merck 60 F_{254} percolated aluminum plates and were developed by spraying with ninhydrin solution. All reactions were carried out in the LG microwave Lightwave Oven MJ3281BCS, using 60 W of microwave power at 40 °C. Proton nuclear magnetic resonance (^{1}H NMR) spectra were recorded on a Brückerspectrometer at 250, 300 or 400 MHz. Chemical shifts are reported in δ units (ppm) with TMS as reference (δ0.00). All coupling constants (*J*) are reported in hertz. Multiplicity is indicated by one of the following: s (singlet), d (doublet), t (triplet), q (quartet), m (multiplet). Carbon nuclear magnetic resonance (^{13}C NMR) spectra were recorded on a Brücker spectrometer at 60, 75 or 100 MHz. Chemical shifts are reported in δ units (ppm) relative to $CDCl_3$ (δ 77.0). Infrared spectra were recorded on a SCHIMADZU FT-IR 8000 spectrometer. Melting points were recorded on a Büchi B-545 apparatus in open capillary tubes.

*2.2. Procedure for synthesis of α-sulfamidophosphonate (**2a**)*

In a 10 mL round bottom flask taken a mixture of aldehyde (1 mmol) and sulfonamide (1 mmol) at room temperature and then triethylphosphite (1 mmol) was added. Then reaction mixture was exposed to microwave irradiation for the appropriate time. After completion of the reaction, as indicated by TLC, silica gel; dichloromethane: methanol (9:1), a (4:1) mixture of diethyl ether and n-hexane was added and the mixture was cooled to 6 °C overnight. The product was finally filtered and dried

2.3. Spectral data

*2.3.1. Dimethyl (N-cyclohexylsulfamoylamino)(phenyl)methylphosphonate (Table 1, Entry **2a**):*

White crystal.mp = 137-139 °C. Yield 94%.R_f(DCM-MeOH: 95/5) = 0.39. Ms (*m/z*): 399.05 [M+23]. ν_{max} (KBr)/cm^{-1} 3468, 3065, 1631, 1368, 1259, 1152, 1100. δ_P (120 MHz, $CDCl_3$) 19.98. δ_H (300 MHz, $CDCl_3$) 0.9 (m, 4H, $2CH_2$), 1.1 (m, 2H, CH_2), 1.39 (m, 2H, CH_2), 1.55 (m, 1H, CH_2), 1.84 (m, 1H, CH_2), 2.45 (m, 1H, CH), 3.47 (d, *J* 9 Hz, 3H, CH_3), 3.67 (d, *J* 7.2 Hz, 3H, CH_3), 4.61 (dd, *J* 6.8 Hz, 1H, CH), 6.72 (d, *J* 6.5 Hz, 1H, NH), 7.27-7.51 (m, 5H, H-Ar).δ_C(75 MHz, $CDCl_3$) 21.5, 24.6, 25.1, 33.9, 53.4, 53.5, 56.1, 126.2, 129.5, 129.8, 136.5. Anal. Calc. for $C_{15}H_{25}N_2O_5PS$: C 47.86, H 6.69, N 7.44. Found: C 48.03, H 6.65, N 7.40 %. M=376.

2.*3.2. Diethyl phenyl(N-propylsulfamoylamino)methylphosphonate (Table 1, Entry **2b**):*

White powder.mp = 136-138 °C. Yield 88%.R_f (DCM-MeOH: 95/5) = 0.41. Ms (*m/z*): 366.12 [M+1].ν_{max} (KBr)/cm^{-1} 3265, 2978, 1593, 1372, 1264, 1154, 1077.δ_P (120 MHz, $CDCl_3$) 19.88.δ_H (300 MHz, $CDCl_3$) 0.72 (t, *J* 7.3 Hz, 3H, CH_3), 1.09 (t, *J* 7.1 Hz, 3H, CH_3), 1.14-1.29 (m, 2H, CH_2), 1.37 (t, *J* 7.1 Hz, 3H,CH_3), 2.54 (m, 1H, CH_2-N), 2.82 (m, 1H, CH_2-N), 3.67-3.76 (m, 1H, CH_2), 3.89-3.97 (m, 1H, CH_2), 4.07 (t, *J* 6 Hz, 1H, NH), 4.18-4.27 (m, 2H, CH_2), 4.75 (dd, *J* 8.8 Hz, 1H, CH), 6.03 (t, *J* 6.6 Hz, 1H, NH), 7.28-7.52 (m, 5H, H-Ar).δ_C (75 MHz, $CDCl_3$) 11.1, 16.3, 16.4, 21.7, 24.5, 55.4, 66.2, 66.3, 124.6, 127.1, 127.3, 129, 129.2, 137.1. Anal. Calc. for $C_{14}H_{25}N_2O_5PS$: C 46.14, H 6.92, N 7.69. Found: C 46.10, H 7.08, N 7.55 %. M=365.

*3.3.3. Diethyl phenyl(N-phenylsulfamoylamino)methylphosphonate (Table 1, Entry **2c**):*

White crystal.m.p = 152-154 °C. Yield 95%.R_f (DCM-MeOH: 95/5) = 0.32. Ms (*m/z*): 399.30 [M+1]. ν_{max} (KBr)/cm^{-1} 3210, 1675, 1387, 1262, 1151, 1023.δ_P (160 MHz, $CDCl_3$) 19.61. δ_H (400 MHz, $CDCl_3$) 1.03 (t, *J* 7.2 Hz, 3H, CH_3), 1.29 (t, *J* 6.8 Hz, 3H,CH_3), 3.59-3.67 (m, 1H, CH_2), 3.82-3.89 (m, 1H, CH_2), 4.06-4.17 (m, 2H, CH_2), 4.81 (dd, *J* 8.8 Hz,1H, CH), 5.92 (t, *J* 6.8 Hz, 1H, NH), 6.47 (s, 1H, NH), 6.79-6.81 (m, 2H, H-Ar), 7.01-7.25 (m, 8H, H-Ar).δ_C(100 MHz, $CDCl_3$) 16.3, 16.5, 54.8, 63.9, 64.1, 119.7, 124.4, 128.3, 128.4, 128.7, 128.9, 129.3, 134.2, 136.8. Anal. Calc. for $C_{17}H_{23}N_2O_5PS$: C 51.25, H 5.82, N 7.03. Found: C 51.15, H 5.80, N 7.20 %. M= 398.

*3.3.4. Diethyl phenyl(N-(1-phenylethyl)sulfamoylamino)methylphosphonate (Table 1, Entry **2d**):*

White powder.mp = 138-140 °C. Yield 90%.R_f (DCM-MeOH: 95/5) = 0.36. Ms (*m/z*): 427.21 [M+1].ν_{max} (KBr)/cm^{-1} 3330, 3040, 1622, 1355, 1230, 1155, 1103.δ_P (160 MHz, $CDCl_3$) 20.16.δ_H (400 MHz, $CDCl_3$) 1.08 (t, *J* 7.2 Hz, 3H, CH_3), 1.12 (d, *J* 6.6 Hz, 3H, CH_3), 1.32 (t, *J* 7 Hz, 3H, CH_3), 3.69 (m, 1H, CH), 3.92 (m, 1H, CH),

4.10-4.19 (m, 2H, CH_2), 4.27-4.39 (m, 2H, CH_2), 4.73 (dd, *J* 11.2 Hz, 1H, CH), 5.41 (t, *J* 7.7 Hz, 1H, NH), 7.15-7.27 (m, 5H, H-Ar), 7.28-7.45 (m, 5H, H-Ar).δ_C (100 MHz, $CDCl_3$) 17, 21.3, 48.2, 56, 62.2, 115.7, 122.8, 124, 126.3, 128.5, 129.4, 142.5, 145.1. Anal. Calc. for $C_{19}H_{27}N_2O_5PS$: C 53.51, H 6.38, N 6.57. Found: C 53.02, H 6.21, N 6.35 %. M=426.

*3.3.5. Diethyl (N-(3-fluorophenyl)sulfamoylamino)(phenyl)methylphosphonate (Table 1, Entry **2e**):*
White crystal.mp = 109-111 °C. Yield 92%.R_f (DCM-MeOH: 95/5) = 0.45. Ms (*m/z*): 417.30 [M+1].ν_{max} (KBr)/cm^{-1} 3315, 1688, 1319, 1225, 1140, 1032. δ_F (375 MHz, $CDCl_3$) -111.62.δ_P (160 MHz, $CDCl_3$) 22.21.δ_H (400 MHz, $CDCl_3$) 1.1 (t, *J* 7.1 Hz, 3H, CH_3), 1.29 (t, *J* 7 Hz, 3H, CH_3), 3.59-3.69 (m, 1H, CH_2), 3.87-3.96 (m, 1H, CH_2), 4.04-4.18 (m, 2H, CH_2), 4.71 (dd, *J* 7.7 Hz, 1H, CH), 4.97 (t, *J* 8.6 Hz, 1H, NH), 6.24-7.03 (m, 4H, H-Ar), 7.25-7.46 (m, 5H, H-Ar).δ_C (100 MHz, $CDCl_3$) 16.1, 16.4, 56.2, 64, 64.1, 106.5, 106.7, 110.6, 110.8, 114.7, 128.2, 128.7, 130.2, 130.3, 134, 138.5, 161.8. Anal. Calc. for $C_{17}H_{22}FN_2O_5PS$: C 49.03, H 5.33, N 6.73. Found: C 48.50, H 5.62, N 6.53 %. M=416.

*3.3.6.Diethyl (N-(4-chlorophenyl)sulfamoylamino)(phenyl)methylphosphonate (Table 1, Entry **2f**):*
White crystal.mp = 116-118 °C. Yield 91%.R_f (DCM-MeOH: 95/5) = 0.46. Ms (*m/z*): 433.43 [M+1].ν_{max} (KBr)/cm^{-1} 3210, 1675, 1387, 1262, 1151, 1023.δ_P (100 MHz, $CDCl_3$) 19.66. δ_H (300 MHz, $CDCl_3$) 1.10 (t, *J* 6.9 Hz, 3H, CH_3), 1.28 (t, *J* 7.1 Hz, 3H,CH_3), 3.61-3.69 (m, 1H, CH_2), 3.88-3.96 (m, 1H, CH_2), 4.07-4.14 (m, 2H, CH_2), 4.78 (dd, *J* 7.7 Hz, 1H, CH), 4.82 (t, *J* 8.2 Hz, 1H, NH), 6.50 (d, *J* 8.85 Hz, 2H,H-Ar), 7.03 (d, *J* 7.1 Hz, 2H,H-Ar), 7.29-7.35 (m, 5H, H-Ar).δ_C (75 MHz, $CDCl_3$) 16.3, 16.4, 55.5, 63.5, 63.7, 109.3, 111.2, 115.2, 124.1, 126.8, 128.3, 128.9, 131.4, 136.6, 148.4, 149.3, 163.2. Anal. Calc. for $C_{17}H_{22}ClN_2O_5PS$: C 47.17, H 5.12, N 6.47. Found: C 46.95, H 5.58, N 6.46 %. M=432.

*3.3.7. Diethyl (N-(4-methoxyphenyl)sulfamoylamino)(phenyl)methylphosphonate (Table 1, Entry **2g**):*
Oil.Yield 87%.R_f (DCM-MeOH: 95/5) = 0.39. Ms (*m/z*): 429.12 [M+1].ν_{max} (KBr)/cm^{-1} 3323, 1610, 1315, 1236, 1167, 1050.δ_P (160 MHz, $CDCl_3$) 21.37. δ_H (400 MHz, $CDCl_3$) 1.21 (t, *J* 7 Hz, 3H, CH_3), 1.26 (t, *J* 7 Hz, 3H, CH_3), 3.75 (s, 3H, CH_3), 3.94-4.01 (m, 2H, CH_2), 4.00-4.15 (m, 2H, CH_2), 5.03 (d, *J* 10.8 Hz, 1H, CH), 6.62-6.81 (2d, *J* 8.9 Hz, 2H, H-Ar), 7.24-7.49 (m, 7H, H-Ar). δ_C (100 MHz, $CDCl_3$) 16.3, 16.4, 55.4, 63.1, 63.3, 70, 71.6, 114.1, 123.2, 127, 127.1, 128, 128.1, 128.2, 128.3, 128.5, 129.6, 136.5, 136.6. Anal. Calc. for $C_{18}H_{25}N_2O_6PS$: C 50.46, H 5.88, N 6.54. Found: C 50.99, H 6.15, N 6.80 %. M=428.

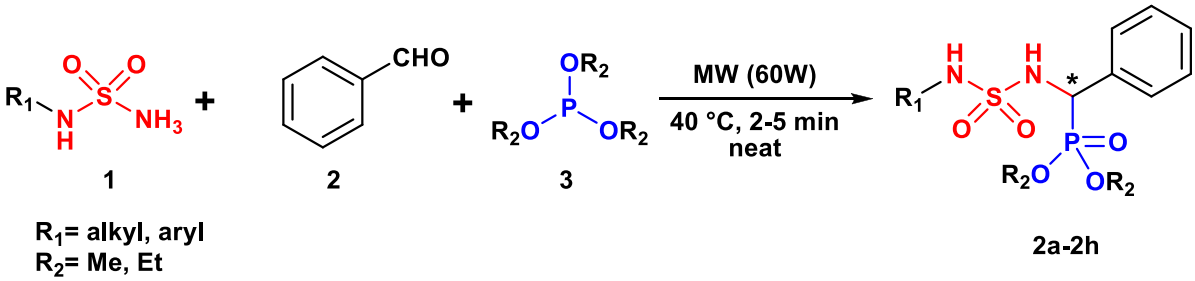
*3.3.8. Diethyl (N-(2-methoxyphenyl)sulfamoylamino)(phenyl)methylphosphonate (Table 1, Entry **2h**):*
White crystal.mp = 145-147 °C. Yield 85%.R_f (DCM-MeOH: 95/5) = 0.38. Ms (*m/z*): 429.15 [M+1].ν_{max} (KBr)/cm^{-1} 3300, 1655, 1311, 1228, 1151, 1080.δ_P (160 MHz, $CDCl_3$) 19.53. δ_H (400 MHz, $CDCl_3$) 1.01 (t, *J* 7.1 Hz, 3H, CH_3), 1.29 (t, *J* 7.1 Hz, 3H, CH_3), 3.81 (s, 3H, CH_3), 3.54-3.83 (m, 1H, CH2), 3.84-3.87 (m, 1H, CH_2), 4.04-4.16 (m, 2H, CH_2), 4.74 (dd, *J* 8.8 Hz, 1H, CH), 6.00 (t, *J* 7.1 Hz, 1H, NH), 6.55-6.57 (d, *J* 8 Hz, 1H, H-Ar), 6.74 (s, 1H, NH), 6.83-6.87 (t, *J* 7.6 Hz, 1H, H-Ar), 6.91-6.95 (t, *J* 7.5 Hz, 1H, H-Ar), 7.08-7.14 (m, 5H, H-Ar), 7.39-7.43 (d, *J* 6.9 Hz, 1H, H-Ar).δ_C (100 MHz, $CDCl_3$) 16.3, 16.4, 55.9, 58.2, 68.2, 69, 109.9, 117.4, 120.8, 123.6, 123.9, 127.7, 127.7, 128.1, 128.3, 129.1, 138.4, 139.6. Anal. Calc. for $C_{18}H_{25}N_2O_6PS$: C 50.46, H 5.88, N 6.54. Found: C 50.95, H 5.71, N 6.68 %. M=428.

3. Results and discussion

The use of microwave to accelerate reactions has proven to be a particularly important tool for meeting the Green Chemistry goals of minimization of waste and reduction of energy requirement [33, 34]. Applications of microwave are playing an increasing role in organic synthesis, especially in cases where classical methods require drastic conditions or prolonged reaction times. The sulfonamides (1) presented here were prepared easily by a simple and efficient protocol described by our group [35, 36]. The one-pot tree-component reaction of benzaldehyde (1 mmol), sulfonamide (1 mmol) and triethylphosphite (1 mmol) was examined under microwave irradiation to give the target compound (2a-2h) (Scheme 1).

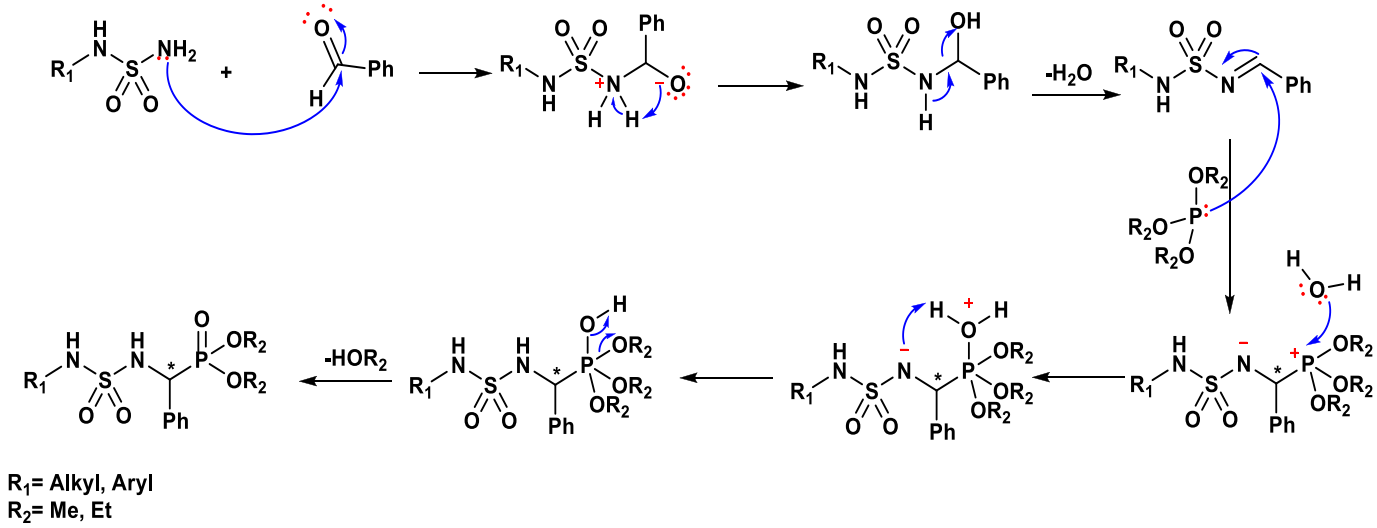
Initially, the reaction was attempted in different solvents such as ethanol, dichloromethane and water. After the reaction mixture was stirred at room temperature for 3 h, no desired product was detected (Table 1, entries 1, 2 and 3). Then, the reactions were explored under the solvent-free conditions. The reaction of benzaldehyde, sulfonamide, and triethylphosphite at 50 °C for 3 h gave desired product 2a in 10% yield. A higher yield 50% was obtained after heating the reaction mixture at 80°C for 3 h (Table 1, entry 6).

Finally, reaction mixture was irradiated in a microwave at 60 W and 30-40 °C for only 2-5 min, the reaction was completed and the yield of 2a was increased to 93% (Table 1, entry 8).



Scheme 1: Microwave assisted synthesis of α-sulfamidophosphonates.

A possible mechanism for the synthesis of α-sulfamidophosphonates is suggested in Scheme 2. Microwave irradiation not only quickly heats the system to high temperature facilitating the reaction, but also activates the carbonyl group, making them more susceptible for nucleophilic attack by the amine, which forms sulfonylimine as intermediate. During this reaction, the chemical bonds break, and water eliminated for sulfonylimine formation. Then the triethylphosphite attacks the imine function to form the desired product.



Scheme 2: Mechanistic proposal for synthesis of α-sulfamidophosphonate.

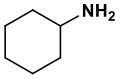
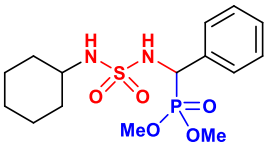
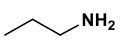
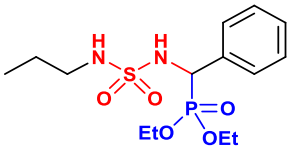
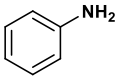
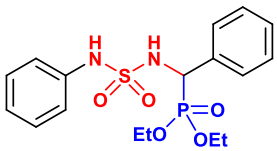
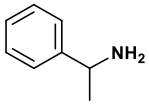
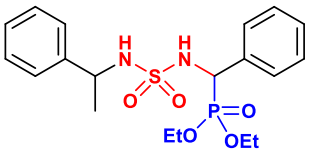
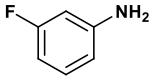
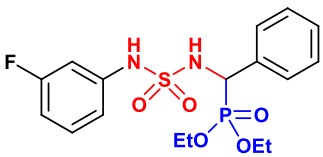
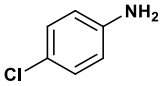
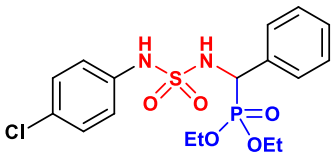
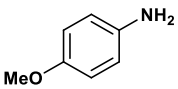
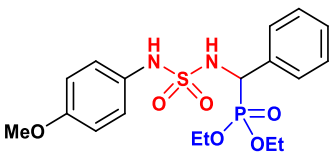
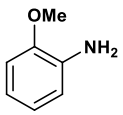
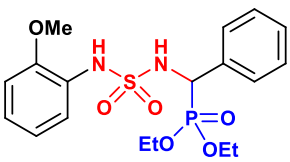
Table 1: Catalyst-free synthesis of α-sulfamidophosphonates.

Entry	*Solvent*	*Temp (°C)*	*Time(min)*	*Yield(%)*
1	EtOH	rt	180	nr[c]
2	CH_2Cl_2	rt	180	nr
3	H_2O	rt	180	nr
4	Solvent-free	rt	180	nr
5	Solvent-free	50	180	10
6	Solvent-free	80	180	50
7	Solvent-free	rt, us[a]	180	95
8	Solvent-free	40, mw[b]	5	93

[a]us =ultrasound. [b]mw =microwave. [c]nr =no réaction.

Under these conditions, we found that microwave irradiation causes a strong acceleration of this process (reaction time was shorten) to give the α-sulfamidophosphonates in 82-93% yield.

Table 2: One-pot synthesis of α-sulfamidophosphonate under microwave irradiation[a].

Entry	*Amine*	*Compound*	*Ultrasound*		*Microwave*		*M.p. (°C)*
			Time (min)	*Yield (%)*	*Time (min)*	*Yield (%)*	
2a			90	94	4	86	137-139
2b			120	88	5	90	136-138
2c			120	95	4	93	152-154
2d			150	90	5	89	138-140
2e			90	92	2	87	109-111
2f			120	91	2	82	116-118
2g			180	87	3	85	-
2h			180	85	3	80	145-147

[a]Conditions: aldehyde (1 mmol), sulfonamide (1 mmol), triethylphosphite (1 mmol), 60 W, 40 °C.

Conclusions

In conclusion, this paper reports a new microwave assisted efficient and green approach for the synthesis of new α-sulfamidophosphonates without the use of any catalyst. This new protocol offers advantages like short reaction times, no use of solvent, no side reactions, facile isolation of products, excellent yields. The reaction process is highly efficient, economic, and also environmentally friendly.

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