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Ultrasound assisted green synthesis of α-hydroxyphosphonates under solvent-free conditions

Abdeslem Bouzina[1] · **Nour-eddine Aouf**[1] · **Malika Berredjem**[1]

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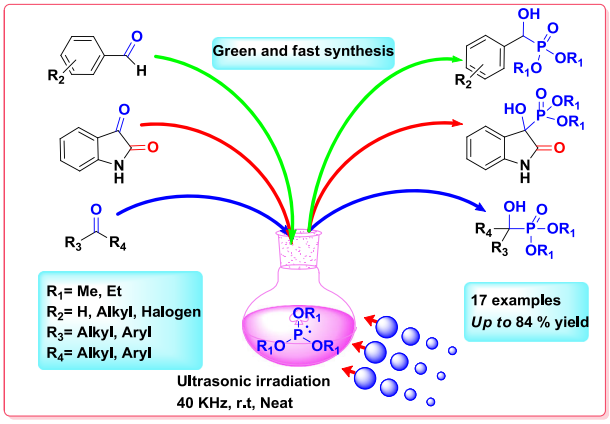
Abstract A simple, efficient and environmentally benign method for the synthesis of α-hydroxyphosphonates by reaction of an aldehyde or a ketone, and trialkylphosphite is effectively accomplished under ultrasound irradiation and solvent-free and catalyst-free conditions. This rapid method produces α-hydroxyphosphonates in high yields and short reaction times.

Graphical Abstract



Electronic supplementary material The online version of this article (doi:10.1007/s11164-015-2420-8) contains supplementary material, which is available to authorized users.

✉ Malika Berredjem
mberredjem@yahoo.fr; malika.berredjem@univ-annaba.org

[1] Laboratory of Applied Organic Chemistry, Synthesis of Biomolecules and Molecular Modelling Group, Department of Chemistry, Sciences Faculty, Badji-Mokhtar - Annaba University, Box 12, 23000 Annaba, Algeria

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Keywords α-Hydroxyphosphonates · Green chemistry · Ultrasound irradiation · Solvent-free

Introduction

The synthesis of α-hydroxyphosphonates and their derivatives has attracted much attention due to their potential biological activities with broad applications as synthetic intermediates [1–8]. They exhibit a variety of interesting and useful properties that make them attractive as herbicides [9], antibiotics [10], pesticides [11], and antiviral [12] and anticancer agents [13].

Recently, various methodologies have been developed for the synthesis of α-hydroxyphosphonates from aldehydes [14–17]. Most of them have reported the use of expensive catalysts, strong acidic or basic conditions, higher temperatures, longer reaction times and have required stoichiometric amount of reagents. These include various bases such as sodium alkoxide [18], triethylamine [19], ethyl magnesium bromide [20], potassium or cesium fluoride [21], LDA [22], MgO [23] etc., or acid catalyst such as BF_3 Et_2O and $AlCl_3$ or HCl [24], alumina [25], TFA or TfOH [26], Ti(OiPr) [27], and other catalyst complexes such as chiral tethered bis (8-quinolinolato) (TBOx) aluminum(III) [28], ambertlyst [29], lanthanide amides [$(Me_3Si)_2N$] 3Ln(μ-Cl)Li$(THF)_3$ [6] and 1,4-dimethylpiperazine under ultrasonic irradiation [30].

One of the current major difficulties is to develop synthetic methods that are less polluting, i.e., to design green chemical transformations and clean technology. In this context, the use of ultrasonic irradiation 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 requirements [31–36]. Applications have been found in synthetic chemistry, in materials science, in life sciences, as well as in medicinal chemistry [37, 38]. Improvement of chemical reactions by ultrasound is often ascribed to the high temperature formed when cavitations formed by the ultrasound collapse, sometimes leading to products different than those obtained without sonication. Although the energy delivered through the cleansing bath is not that high, there is still acoustic cavitation in the reaction flask. In heterogeneous systems, the acoustic cavitation is known to vastly improve diffusion rates and accelerate the reactions through micro jets formed from asymmetrical collapses of the bubbles, and similar effects can be achieved with high speed mechanical stirring.

We report herein a highly efficient reaction for the synthesis of α-hydroxyphosphonates **1-13a**, **1-2b** and **1-3c** under ultrasound irradiation and catalyst-free and solvent-free conditions in high yields.

Experimental

General

All chemicals and solvents were purchased from common commercial sources and were used as received without any further purification. All reactions were monitored

by thin-layer chromatography (TLC) on silica Merck 60 F_{254} percolated aluminium plates and were developed by spraying with ninhydrin solution. Column chromatography was performed with Merck silica gel (230–400 mesh). Proton nuclear magnetic resonance (^{1}H NMR) spectra were recorded on a Brücker spectrometer at 250 or 400 MHz. Chemical shifts are reported in δ units (ppm) with TMS as reference (δ 0.00). All coupling constants (J) are reported in Hertz. Multiplicity is indicated by one or more 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 at 60 or 100 MHz. Phosphorus nuclear magnetic resonance (^{31}P NMR) spectra were recorded on a Brücker at 60 or 100 MHz. Chemical shifts are reported in δ units (ppm) relative to $CDCl_3$ (δ 77.0). Infrared spectra were recorded on a Perkin Elmer 600 spectrometer. Mass spectra were recorded on a Shimadzu QP 1100 Ex mass spectrometer operating at an ionization potential of 70 eV. Elemental analysis was recorded on a EURO E.A 3700. Melting points were recorded on a Büchi B-545 apparatus in open capillary tubes. Ultrasound assisted reactions were carried out using a FUNGILAB ultrasonic bath with a frequency of 40 kHz and a nominal power of 250 W. The reactions were carried out in an open glass tube (diameter: 25 mm; thickness: 1 mm; volume: 20 mL) at room temperature.

Typical experimental procedure for the synthesis of α-hydroxyphosphonates

In a glass tube (diameter: 25 mm; thickness: 1 mm; volume: 20 mL), a mixture of aldehyde (1 mmol) and trialkylphosphite (1.2 mmol) was subjected to sonication for appropriate time (Tables 1, 2, 3). After completion of the reaction, as indicated by TLC, silica gel; dichloromethane:methanol (90:10) and a (6:4) mixture of diethyl ether and *n*-hexane was added to the reaction mixture and pure product was crystallized by cooling the solution to 6 °C overnight.

*Diethyl (hydroxy(phenyl)methyl)phosphonate (**1a**, $C_{11}H_{17}O_4P$)*

Cristal; 94 % yield; ^{31}P NMR (100 MHz, $CDCl_3$): $\delta = 21.44$ ppm; ^{1}H NMR (400 MHz, $CDCl_3$): $\delta = 1.20$ (t, $J = 7.20$ Hz, 3H, CH_3-CH_2O), 1.25 (t, $J = 6.93$ Hz, 3H, CH_3-CH_2O), 3.90–3.96 (m, 2H, CH_2-O), 3.97–4.07 (m, 2H, CH_2-O), 5.02 (d, $J = 11.2$ Hz, 1H, CH*), 7.25–7.37 (m, 3H, H-Ar), 7.46–7.49 (m, 2H, H-Ar). pm; ^{13}C NMR (100 MHz, $CDCl_3$): $\delta = 16.53$, 16.58, 63.26, 63.55, 70.22, 71.80, 127.27, 128.26, 128.44, 130.30, 136.80, 136.82 ppm; IR (KBr): $v = 3382.45$, 1514.49, 1251.33, 1033.44 cm^{-1}; MS: (m/z) = 245.1 (M + 1); Anal. Calc. for $C_{11}H_{17}O_4P$: C, 54.10; H, 7.02; Found: C, 54.16; H, 7.03.

*Diethyl (hydroxy(4-chlorophenyl)methyl)phosphonate (**2a**, $C_{11}H_{16}ClO_4P$)*

Cristal; 91 % yield; ^{31}P NMR (100 MHz, $CDCl_3$): $\delta = 21.21$ ppm; ^{1}H NMR (250 MHz, $CDCl_3$): $\delta = 1.16$ (t, $J = 7.16$ Hz, 3H,CH_3-CH_2O), 1.23 (t, $J = 7.04$ Hz, 3H, CH_3-CH_2O), 3.88 (m, 2H, CH_2-O), 3.96 (m, 2H, CH_2-O), 5.00

Table 1 Synthesis of α-hydroxyphosphonates from aldehydes

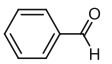
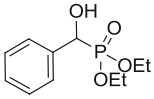
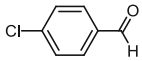
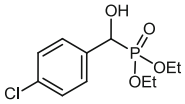
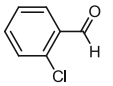
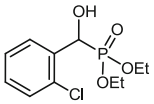
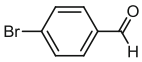
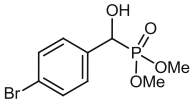
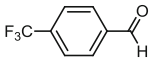
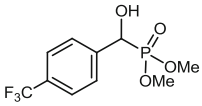
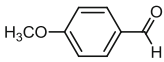
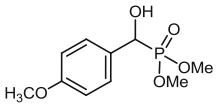
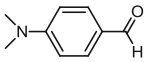
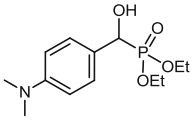
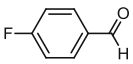
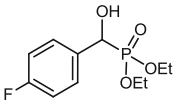
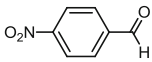
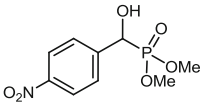
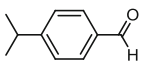
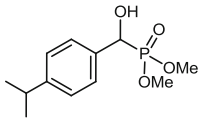
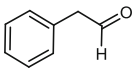
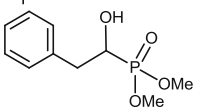
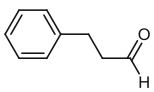
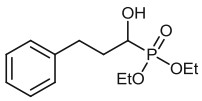
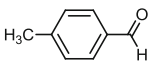
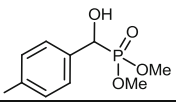
Entry	Aldehyde	Product	Time (min)	Yield %
1a			10	94
2a			12	91
3a			15	90
4a			17	88
5a			17	93
6a			12	89
7a			14	88
8a			13	92
9a			10	94
10a			10	92
11a			15	90
12a			14	91
13a			15	87

Table 2 Synthesis of α-hydroxyphosphonates from isatin

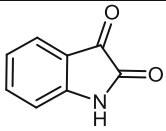
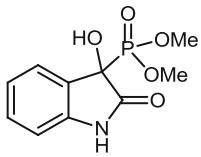
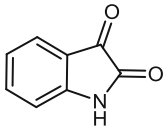
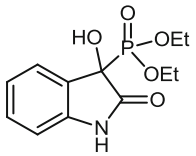
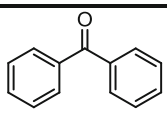
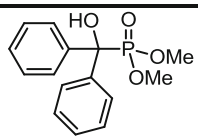
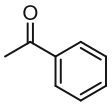
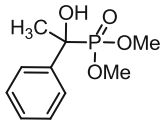
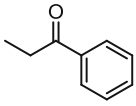
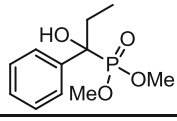
Entry	isatin	Product	Time (min)	Yield %
1b			35	85
2b			37	86

Table 3 Synthesis of α-hydroxyphosphonates from ketones

Entry	Ketone	Product	Time (min)	Yield %
1c			27	85
2c			35	84
3c			25	85

(d, $J = 10.13$ Hz, 1H, CH*), 7.31–7.45 (m, 4H, H-Ar), ppm; ^{13}C NMR (100 MHz, $CDCl_3$): $\delta = 16.15$, 16.51, 61.21, 62.35, 69.04, 70.75, 120.15, 125.17, 126.45, 132.45, 136.50 ppm; IR (KBr): $v = 3249.69$, 1491.85, 1234.20, 1029.13 cm^{-1}; MS: $(m/z) = 279$ (M + 1); Anal. Calc. for $C_{11}H_{16}ClO_4P$: C, 47.41; H, 5.79; Found: C, 47.37; H, 5.78.

*Diethyl (hydroxy(2-chlorophenyl)methyl)phosphonate (**3a**, $C_{11}H_{16}ClO_4P$)*

Color powder; 90 % yield; ^{31}P NMR (100 MHz, $CDCl_3$): $\delta = 21.45$ ppm; ^{1}H NMR (250 MHz, $CDCl_3$): $\delta = 1.21$ (t, $J = 7.21$ Hz, 3H,CH_3-CH_2O), 1.29 (t, $J = 7.06$ Hz, 3H, CH_3-CH_2O), 3.91 (m, 2H, CH_2-O), 4.02 (m, 2H, CH_2-O), 5.08 (d, $J = 10.81$ Hz, 1H, CH*), 7.31–7.42 (m, 4H, H-Ar), ppm; ^{13}C NMR (75 MHz, $CDCl_3$): $\delta = 16.30$, 16.75, 59.87, 60.20, 68.87, 69.25, 122.65, 124.39, 125.63, 133.15, 134.00 ppm; IR (KBr): $v = 3319.75$, 1614.61, 1254.76, 1056.29 cm^{-1};

MS: (m/z) = 279 (M + 1); Anal. Calc. for $C_{11}H_{16}ClO_4P$: C, 47.41; H, 5.79; Found: C, 47.42; H, 5.83.

*Dimethyl (hydroxy(4-bromophenyl)methyl)phosphonate (**4a**, $C_9H_{12}BrO_4P$)*

White powder; 88 % yield; ^{31}P NMR (100 MHz, $CDCl_3$): δ = 21.45 ppm; ^{1}H NMR (250 MHz, $CDCl_3$): δ = 3.91 (d, J = 9.61 Hz, 3H, CH_3-O), 3.95 (d, J = 10.12 Hz, 3H, CH_3-O), 5.08 (d, J = 10.81 Hz, 1H, CH*), 7.15–7.40 (m, 4H, H-Ar), ppm; ^{13}C NMR (75 MHz, $CDCl_3$): δ = 53.17, 53.74, 72.61, 76.65, 127.08, 127.11, 127.65, 129.36, 137.97 ppm; IR (KBr): v = 3362.24, 1415.85, 1225.35, 1066.21 cm^{-1}; MS: (m/z) = 293.9 (M + 1); Anal. Calc. for $C_9H_{12}BrO_4P$: C, 36.63; H, 4.10; Found: C, 36.70; H, 4.13.

*Dimethyl (hydroxy(4-methoxyphenyl)methyl)phosphonate (**6a**, $C_{10}H_{15}O_5P$)*

White powder; 89 % yield; ^{31}P NMR (100 MHz, $CDCl_3$): δ = 21.61 ppm; ^{1}H NMR (250 MHz, $CDCl_3$): δ = 3.61 (d, J = 9.11 Hz, 3H,CH_3-O), 3.79 (d, J = 10.81 Hz, 3H, CH_3-O), 3.82 (s, 3H, CH_3-O), 5.02 (d, J = 14.81 Hz, 1H, CH*), 7.01–7.35 (m, 2H, H-Ar), 7.37–7.48 (m, 2H, H-Ar) ppm; ^{13}C NMR (75 MHz, $CDCl_3$): δ = 53.82, 53.94, 55.40, 69.98, 71.54, 114.02, 120.40, 128.55, 128.64, ppm; IR (KBr): v = 3319.75, 1479.54, 1235.79, 1066.29 cm^{-1}; MS: (m/z) = 247 (M + 1); Anal. Calc. for $C_{10}H_{15}O_5P$: C, 48.78; H, 6.14; Found: C, 48.81; H, 6.09.

*Diethyl (hydroxy(4-fluorophenyl)methyl)phosphonate (**8a**, $C_{11}H_{16}FO_4P$)*

White powder; 92 % yield; ^{31}P NMR (100 MHz, $CDCl_3$): δ = 20.90 ppm; ^{1}H NMR (250 MHz, $CDCl_3$): δ = 1.17 (t, J = 7.07 Hz, 3H,CH_3-CH_2O), 1.23 (t, J = 6.96 Hz, 3H, CH_3-CH_2O), 3.97 (m, 2H, CH_2-O), 4.01 (m, 2H, CH_2-O), 5.05 (d, J = 10.78 Hz, 1H, CH*), 7.28–7.53 (m, 4H, H-Ar) ppm; ^{13}C NMR (75 MHz, $CDCl_3$): δ = 16.23, 16.71, 64.30, 65.70, 69.85, 70.61, 119.87, 121.20, 122.60, 126.55, 131.20, 134.51, 157.23 ppm; IR (KBr): v = 3273.39, 1416.56, 1239.88, 1066.06 cm^{-1}; MS: (m/z) = 263 (M + 1); Anal. Calc. for $C_{11}H_{16}FO_4P$: C, 50.39; H, 6.15; Found: C, 50.42; H, 6.13.

*Diethyl (1-hydroxy-3-phenylpropyl)phosphonate (**12a**, $C_{13}H_{21}O_4P$)*

White powder; 91 % yield; ^{31}P NMR (100 MHz, $CDCl_3$): δ = 20.20 ppm; ^{1}H NMR (250 MHz, $CDCl_3$): δ = 1.18 (t, J = 7.24 Hz, 3H,CH_3-CH_2O), 1.25 (t, J = 6.99 Hz, 3H, CH_3-CH_2O), 1.75 (m, 2H, CH_2-CH), 2.68 (m, 2H, CH_2-Ar), 3.94 (m, 2H, CH_2-O), 4.10 (m, 2H, CH_2-O), 5.12 (d, J = 10.85 Hz, 1H, CH*), 7.31–7.39 (m, 3H, H-Ar), 7.43–7.46 (m, 2H, H-Ar) ppm; ^{13}C NMR (100 MHz, $CDCl_3$): δ = 16.53, 16.58, 25.30, 34.7, 63.26, 63.55, 70.22, 71.80, 127.27, 128.26, 128.44, 130.30, 136.80, 136.82 ppm; IR (KBr): v = 3325.97, 1514.29, 1250.96, 1034.85 cm^{-1}; MS: (m/z) = 273.2 (M + 1); Anal. Calc. for $C_{13}H_{21}O_4P$: C, 57.35; H, 7.77; Found: C, 57.40; H, 7.81.

*Dimethyl (hydroxy(4-methylphenyl)methyl)phosphonate (**13a**, $C_{10}H_{15}O_5P$)*

White powder; 87 % yield; ^{31}P NMR (100 MHz, $CDCl_3$): $\delta = 21.20$ ppm; ^{1}H NMR (250 MHz, $CDCl_3$): $\delta = 2.35$ (s, 3H, CH_3-CAr), 3.63 (d, $J = 9.05$ Hz, 3H,CH_3-O), 3.74 (d, $J = 10.64$ Hz, 3H, CH_3-O), 5.01 (d, $J = 14.78$ Hz, 1H, CH*), 7.10–7.48 (m, 4H, H-Ar) ppm; ^{13}C NMR (75 MHz, $CDCl_3$): $\delta = 25.13$, 54.06, 54.59, 72.15, 73.50, 116.52, 121.64, 127.80, 129.90, 132.56 ppm; IR (KBr): $v = 3254.00$, 1474.13, 1255.83, 1031.07 cm^{-1}; MS: (m/z) = 231.1 (M + 1); Anal. Calc. for $C_{10}H_{15}O_5P$: C, 52.18; H, 6.57; Found: C, 52.24; H, 6.62.

*Dimethyl (3-hydroxy-2-oxoindolin-3-yl)phosphonate (**1b**, $C_{10}H_{12}NO_5P$)*

Color powder; 85 % yield; ^{31}P NMR (100 MHz, $CDCl_3$): $\delta = 19.98$ ppm; ^{1}H NMR (250 MHz, $CDCl_3$): $\delta = 3.73$ (d, $J = 7.32$ Hz, 3H,CH_3-O), 3.81 (d, $J = 9.45$ Hz, 3H, CH_3-O), 7.13–7.23 (m, 2H, H-Ar), 7.26–7.35 (m, 2H, H-Ar) ppm; ^{13}C NMR (75 MHz, $CDCl_3$): $\delta = 54.22$, 55.36, 108.10, 118.70, 119.23, 120.40, 128.55, 129.64, 134.15, 158.25 ppm; IR (KBr): $v = 3321.20$, 3180.63, 1690.34, 1237.75 cm^{-1}; MS: (m/z) = 258 (M + 1); Anal. Calc. for $C_{10}H_{12}NO_5P$: C, 46.70; H, 4.70; N, 5.45; Found: C, 46.78; H, 4.75; N, 5.50.

*Dimethyl (1-hydroxy-1-phenylethyl)phosphonate (**2c**, $C_{10}H_{15}O_4P$)*

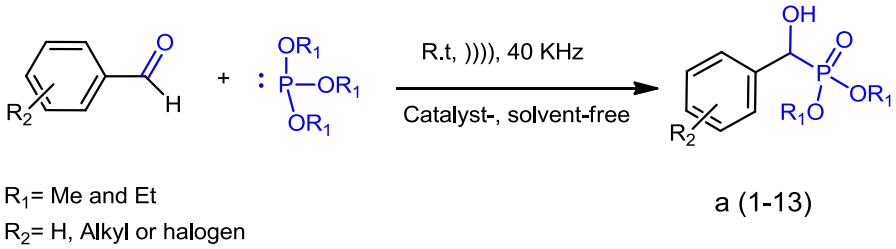
Oil; 84 % yield; ^{31}P NMR (100 MHz, $CDCl_3$): $\delta = 22.15$ ppm; ^{1}H NMR (250 MHz, $CDCl_3$): $\delta = 1.64$ (s, 3H, CH_3-C), 3.63 (d, $J = 8.40$ Hz, 3H,CH_3-O), 3.96 (d, $J = 9.14$ Hz, 3H, CH_3-O), 7.15–7.40 (m, 5H, H-Ar) ppm; ^{13}C NMR (75 MHz, $CDCl_3$): $\delta = 15.20$, 54.40, 55.25, 101.02, 119.10, 122.58, 128.57, 129.71, ppm; IR (KBr): $v = 3315.05$, 1510.21, 1251.08, 1025.20 cm^{-1}; MS: (m/z) = 231 (M + 1); Anal. Calc. for $C_{10}H_{15}O_4P$: C, 58.18; H, 6.57; Found: C, 58.14; H, 6.59.

Results and discussion

As part of our research program focused towards the development of highly opportune methods for the synthesis of diverse phosphonate derivatives [39–44], we have developed a simple and very efficient method for the preparation of α-hydroxyphosphonates under green chemical conditions.

We report here the application of ultrasound irradiation in the synthesis of α-hydroxyphosphonates under catalyst-free and solvent-free conditions. This reaction involves the condensation of aromatic aldehydes or ketones and trialkylphosphite.

The process was promoted by directly immersing of reaction vessels into the ultrasonic cleaning bath, which provides a fairly even distribution of energy into the reaction medium. The reaction was completed within 10–37 min, with a substantial increase in the yield of product. The collapse of cavitation bubbles results in the formation of very reactive chemical species having short lifetimes, facilitating the rapid synthesis of α-functionalized phosphonates derivatives. This is an efficient and



Scheme 1 Ultrasound assisted synthesis of α-hydroxyphosphonates from aldehydes

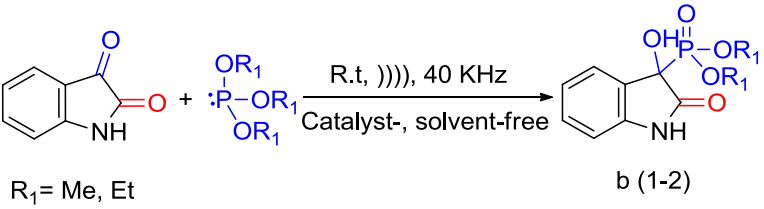
environmentally benign methodology for the synthesis of α-hydroxyphosphonates at ambient temperature (Scheme 1).

The results are summarized in (Table 1, Entry **1a**–**13a**). This is an efficient and eco-sustainable methodology for the synthesis of α-hydroxyphosphonates at ambient temperatures. The reaction is easily scalable and products are obtained in excellent yields.

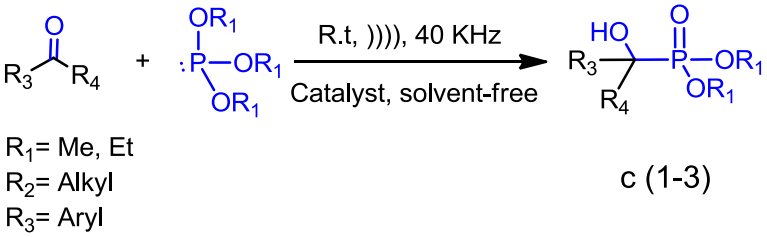
For the resulting compounds, IR spectra showed a band at 1220–1260 cm^{-1} (P=O) of phosphonate moiety, and a band at 3250–3350 cm^{-1} (OH) of hydroxyl group. In 1H-NMR spectra, the structure of α-hydroxyphosphonates was confirmed by a signal at 5.0–5.2 ppm, corresponding to *CH.

To explore the scope and limitations of this procedure, we extended our study of isatin derivative. The isolated yields of products (Table 2, Entry **1b**–**2b**) were in the range of **85**–**86** % after 35–37 min of reaction (Scheme 2).

Encouraged by the preliminary results and to increase the scope of this reaction, we examined this method to ketones (Scheme 3). The reaction gave good yields



Scheme 2 Ultrasound assisted synthesis of α-hydroxyphosphonates from isatin



Scheme 3 Ultrasound assisted synthesis of α-hydroxyphosphonates from ketones

(**84–85** %) after (25–35 min). The results are summarized in (Table 3, Entry **1c**–**3c**).

The structures of the synthesized compounds are confirmed by elemental analysis as well as by IR and ^{1}H, ^{13}C, ^{31}P NMR spectral data and MS.

Conclusions

In conclusion, a green, efficient, and environmentally benign methodology towards the synthesis of α-hydroxyphosponate has been reported. α-hydroxyphosphonates were synthesized by the addition of trialkylphosphite to a variety of arylaldehydes or ketone under neat conditions by simple grinding process; this has proven to be an excellent method. The effect of ultrasound has mostly been shown by the increasing yields of reactions, and in some cases, the ratio of formed products.

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