Conference paper

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Direct esterification of phosphinic and phosphonic acids enhanced by ionic liquid additives

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Abstract: The beneficial combination of microwave (MW) and ionic liquid (IL) additives was exploited in the direct esterification of a series of acyclic phosphinic and phosphonic acids giving rise to phenyl-*H*-phosphinates/methyl-phenylphosphinates/diphenylphosphinates and phenylphosphonic mono- and diesters, respectively. The latter is the first example for the direct esterification of a phosphonic acid.

Keywords: esterification; ICPC-22; ionic liquid; microwave irradiation; phosphinic derivatives; phosphonic derivatives.

Introduction

The interest in the preparation of phosphinates and phosphonates is continuously expanding due to their many applications and the understanding of their role in biological systems. Phosphinates and phosphonates are important reagents in organic transformations. *H*-phosphinates can undergo a series of synthetically useful reactions, such as oxidation and oxidative coupling [1–3], P–C coupling [4], addition to carbonyl groups and imines [5, 6], as well as to C=C unsaturations [7–9], etc. Phosphinates are also versatile building blocks for the preparation of heterocycles of biological interest [10], while phosphonates are important intermediates in the preparation of agricultural chemicals [11] and pharmaceuticals [12, 13]. P-esters can also be used as catalyst ligands [14], metal extractants for liquid-liquid extraction [15] or as halogen-free flame retardants [16, 17].

As the corresponding phosphinic and phosphonic acids do not undergo direct esterification, the desired phosphinates and phosphonates are usually synthesized via acylation applying the corresponding P-chlorides [18–23]. However, in this case, the hydrochloric acid formed in the course of the substitution means an environmental burden, and has to be removed by a base. It is also possible to activate the *P*-acids. Such activating agent is the dicyclohexylcarbodiimide (DCC) [24], or the trimeric propylphosphonic anhydride (T3P®) [25, 26]. Another general method for the synthesis of phosphinates and phosphonates involves the Michaelis–Arbuzov reaction of phosphonous diesters and trialkyl phosphites, respectively, with an alkyl halide [21, 27].

The most desirable esterification method would be the direct esterification of phosphinic and phosphonic acids. Previously it was found by us that the direct esterification of phosphinic acids takes place under

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microwave (MW) irradiation at a high temperature under pressure [28–30]. Preliminary experiments with a phosphonic acid referred to even a lower reactivity [31]. The optimum reaction temperatures of 200–235 °C and the long reaction times of up to 6 h mean disadvantages. Another difficulty is that steric hindrance makes the esterifications rather reluctant. Later on, a more efficient esterification method of a cyclic phosphinic acid, 1-hydroxy-3-methyl-3-phospholene 1-oxide was developed utilizing ionic liquids (ILs) as additives [32]. Among the imidazolium salts tested, [bmim][BF_4] and [bmim][PF_6] were found to be the most efficient. It is a recent observation that several organic chemical transformations may benefit from the IL-catalysis, especially, when ILs are applied together with MW irradiation [33, 34].

In this paper, we describe the MW-assisted direct esterification of a series of P-acids of type "PhP(O)(OH)Y", where Y is H, Me, Ph or OH (Scheme 1) in the presence or absence of [bmim][BF_4] or [bmim][PF_6] as additives.

Experimental

The MW-assisted reactions were carried out in a CEM Discover MW reactor equipped with a stirrer and a pressure controller applying 100–300 W irradiation.

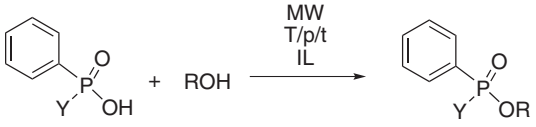
The ^{31}P NMR spectra were taken on a Bruker DRX-300 spectrometer operating at 202.4 MHz. The couplings are given in Hz. The exact mass measurements were performed using a Q-TOF Premier mass spectrometer in positive electrospray mode.

General procedure for the MW-assisted direct esterification of phosphinic acids (1, 3 and 5) (Tables 3–5)

A mixture of 0.75 mmol (0.11 g of phenyl-*H*-phosphinic acid (**1**), 0.12 g of methyl-phenylphosphinic acid (**3**) or 0.16 g of diphenylphosphinic acid (**5**)) and 11.0 mmol of the alcohol (0.64 mL of ethanol, 1.0 mL of *n*-butanol, 1.0 mL of *i*-butanol, 1.2 mL of *n*-pentanol, 1.4 mL of *i*-pentanol or 1.8 mL of *n*-octanol), and 10 % of [bmim][PF_6] (15.5 μL) or [bmim][BF_4] (20.5 μL) was measured in a standard vessel that was irradiated in the MW reactor at the temperatures and for the reaction times given in Tables 3–5. Then, the excess of the alcohol was removed under reduced pressure, and the residue so obtained purified by flash column chromatography using silica gel and ethyl acetate as the eluent to afford phosphinates (**2**, **4** and **6**) as oils. Comparative characterization is listed in Table 1.

General procedure for the MW-assisted direct esterification of phenylphosphonic acid (7) (Table 6)

A mixture of 0.75 mmol (0.12 g) of phenylphosphonic acid (**7**), and 11.0 mmol of the alcohol (1.0 mL of *n*-butanol, or 1.8 mL of *n*-octanol), and 10 % of [bmim][PF_6] (15.5 μL) or [bmim][BF_4] (20.5 μL) was subjected to MW irradiation at the temperatures and for the reaction times given in Table 6. The work-up was performed as described in the previous procedure to provide phosphonate mono- (**8**) and diesters (**9**). The characterization is summarized in Table 2.



Y = H (**1**), Me (**3**), Ph (**5**), OH (**7**) Y = H (**2**), Me (**4**), Ph (**6**), OH (**8**), OR (**9**)

R = Et (**a**), ^{n}Bu (**b**), ^{i}Bu (**c**), ^{n}Pent (**d**), ^{i}Pent (**e**), ^{n}Oct (**f**)

Scheme 1: MW-assisted esterification of acyclic phosphinic and phosphonic acids.

Table 1: ^{31}P NMR and HRMS data for phosphonates **2**, **4**, and **6**.

Product	δ_P	δ_P[lit]	$[M+H]^+_{found}$	$[M+H]^+_{requires}$	Formula
2a	24.7	25.7 [35]	171.0569	171.0575	$C_8H_{11}O_2P$
2b	24.9	25.3 [35]	199.0881	199.0888	$C_{10}H_{15}O_2P$
2c	24.9	25.0 [36]	199.0881	199.0888	$C_{10}H_{15}O_2P$
2d	25.6	25.7 [31]	213.1037	213.1044	$C_{11}H_{18}O_2P$
2f	25.1	25.0 [31]	255.1517	255.1514	$C_{14}H_{24}O_2P$
4b	42.1	42.8 [37]	213.1030	213.1039	$C_{11}H_{18}O_2P$
4f	42.0	42.2 [37]	269.1670	269.1671	$C_{15}H_{26}O_2P$
6b	31.2	31.2 [36]	275.1193	275.1201	$C_{16}H_{19}O_2P$
6d	31.2	30.0 [38]	289.1358	289.1357	$C_{17}H_{22}O_2P$
6e	30.1	31.3 [39]	289.1359	289.1357	$C_{17}H_{22}O_2P$
6f	31.2	32.2 [40]	331.1825	331.1827	$C_{20}H_{28}O_2P$

Table 2: ^{31}P NMR and HRMS data for phosphinates **8** and **9**.

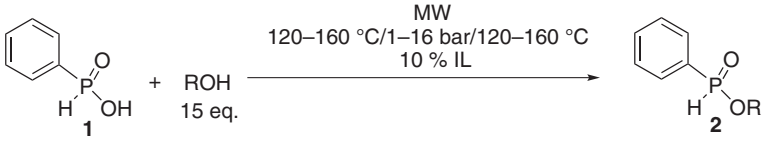
Product	δ_P	δ_P[lit]	$[M+H]^+_{found}$	$[M+H]^+_{requires}$	Formula
8b	21.8	21.8 [31]	215.0840	215.0837	$C_{10}H_{16}O_3P$
8f	22.6	22.8 [31]	271.1467	271.1463	$C_{14}H_{24}O_3P$
9b	19.5	18.8 [31]	271.1465	271.1463	$C_{14}H_{24}O_3P$
9f	18.0	18.9 [31]	383.2716	383.2715	$C_{22}H_{40}O_3P$

Results and discussion

MW-assisted direct esterification of phenyl-*H*-phosphinic acid (1)

Our first model reaction was the esterification of phenyl-*H*-phosphinic acid (**1**) (Scheme 2, Table 3). It was previously found by us that phosphinic acid **1** can be esterified directly with an excess of alcohol, when irradiated by MWs. Moreover, as compared to 5- or 6-membered cyclic phosphinic acids, such as 1-hydroxy-3- or 2-phospholene oxides, hydroxyphospholane oxides, or a hydroxy-hexahydrophosphinine oxide, the reactivity was much higher. Despite the increased reactivity, that may be possible due to the tautomerization of the starting acid, completion of the reaction of phosphinic acid **1** and alcohols required 60 min at 160 °C (Table 3/Entries 1, 5 and 9) or 30 min at an elevated temperature of ≥180 °C (Table 3/Entries 4, 12 and 15).

In order to promote the reaction, we added 10 % of [bmim][PF_6] to the reaction mixture prior to irradiation. In all cases, complete conversions could be obtained at 140 °C after an irradiation time of 30 min (Table 3/Entries 3, 6, 10, 13 and 16). [Bmim][BF_4] was also tried out, and it was found to be as beneficial as the [PF_6] salt (Table 3/Entries 7 vs 6). Although we could not attain complete conversions with ^{n}BuOH and ^{i}BuOH at 120 °C (Table 3/Entries 8 and 11), the esterification took place quantitatively with *n*-pentanol at this temperature after 45 min (Table 3/Entry 14).



Scheme 2: Direct esterification of phenyl-*H*-phosphinic acid (**1**).

Table 3: MW-assisted esterification of phenyl-*H*-phosphinic acid (**1**) with different alcohols in the absence or presence of an IL.

R	T (°C)	T (min)	IL	Conversion[a] (%)	Yield (%)	Entry
Et	160	60	–	100 (**2a**)	80 [37]	1
	160	25	10 % [bmim][PF_6]	100 (**2a**)		2
	140	30	10 % [bmim][PF_6]	100 (**2a**)	94	3
^{n}Bu	180	30	–	100 (**2b**)		4
	160	60	–	100 (**2b**)	85 [37]	5
	140	30	10 % [bmim][PF_6]	100 (**2b**)	94	6
	140	30	10 % [bmim][BF_4]	100 (**2b**)	95	7
	120	30	10 % [bmim][PF_6]	88 (**2b**)		8
^{i}Bu	160	60	–	100 (**2c**)	75 [37]	9
	140	30	10 % [bmim][PF_6]	100 (**2c**)	93	10
	120	60	10 % [bmim][PF_6]	90 (**2c**)		11
^{n}Pent	190	30	–	100 (**2d**)	89	12
	140	30	10 % [bmim][PF_6]	100 (**2d**)	92	13
	120	45	10 % [bmim][PF_6]	100 (**2d**)		14
^{n}Oct	180	30	–	100 (**2f**)	84	15
	140	30	10 % [bmim][PF_6]	100 (**2f**)	88	16

[a]On the basis of ^{31}P NMR.

MW-assisted direct esterification of methyl-phenyphosphinic acid (3)

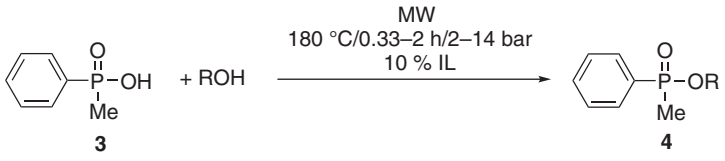
As was mentioned, the direct esterification of phosphinic acids is possible under MW conditions, however, certain limitations apply. Such is the volatility of alcohols due to the pressure limit of 20 bar of the MW reactor applied. At a temperature of 220 °C, the direct esterification of methyl-phenylphosphinic acid **3** and ^{n}BuOH resulted in a conversion of only 15 % after a reaction time of 3 h (Table 4/Entry 1). The pressure limit prevented further elevation of the temperature. It is worth mentioning that the replacement of the H atom by a methyl group on the P atom significantly decreased the reactivity of the phosphinic acid. This may be the result of the lack of tautomerization, and also the electron donation and steric hindrance exerted by the methyl group.

The presence of 10 % of [bmim][PF_6] had a positive effect, and a conversion of 100 % could be attained after 2 h at a lower temperature of 180 °C. Phosphinate **4b** was isolated in a yield of 73 % (Table 4/Entry 2). When *n*-octanol was used as the reaction partner, the IL-catalyzed reaction carried out at 180 °C led to complete conversion after 20 min (Table 4, entry 4), while the catalyst-free variation was

Table 4: Direct esterification of methyl-phenylphosphinic acid (**4**) with alcohols.

R	T (°C)	t (h)	IL	Conversion[a] (%)	Yield (%)	Entry
^{n}Bu	220	3	–	15 (**4b**)	–	1
	180	2	10 % [bmim][PF_6]	100 (**4b**)	73 [36]	2
^{n}Oct	200	2	–	32 (**4f**)	–	3
	180	0.33	10 % [bmim][PF_6]	100 (**4f**)	92 [36]	4

[a]On the basis of ^{31}P NMR.



3 4

Scheme 3: Direct esterification of methyl-phenylphosphinic acid (3).

rather ineffective: after an irradiation of 2 h at 200 °C the conversion was only 32 % in respect of *n*-octyl phosphinate **4f** (Table 4/Entry 3) (Scheme 3).

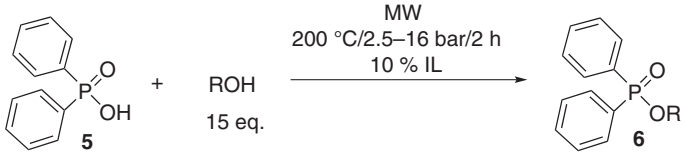
MW-assisted direct esterification of diphenyphosphinic acid (5)

The MW assisted direct esterification was then extended to the sterically more hindered diphenylphosphinic acid (**5**) (Scheme 4). A low reactivity was expected in this case. After a 3 h irradiation time at 220 °C, the butyl phosphinate (**6b**) was formed in a conversion of 28 % (Table 5/Entry 1). Applying *n*-pentanol or *i*-pentanol, the higher boiling points allowed the increase of the reaction temperature. However, even at 235 °C, the conversions remained low (36 % and 34 %, respectively) (Table 5/Entries 3 and 5). The reaction with *n*-octanol was somewhat more effective. After an irradiation time of 6 h at 235 °C, the conversion was 61 % (Table 5/Entry 7).

The IL catalysis was then utilized in the above esterifications. The effect of [bmim][PF_6] was significant, and in these cases higher conversions and yields were obtained at lower temperatures, and in shorter reaction times. Carrying out the reactions at 200 °C for 2 h, the corresponding esters (**6**), in all but one cases, were formed in complete conversions, and could be prepared in good yields (92–95 %) (Table 5/Entries 2, 4 and 8). The reaction with the branched *i*-pentanol was somewhat more reluctant. Applying the same conditions, a conversion of 75 %, and a yield of 65 % could be obtained (Table 5/Entry 6).

MW-assisted direct esterification of phenylphosphonic acid (7)

Encouraged by the results obtained by the IL-catalyzed MW-assisted direct esterification of phosphinic acids, we aimed at extending our method to the esterification of a phosphonic acid as well (Scheme 5). First, we attempted the direct esterification of phenylphosphonic acid (**7**) with ^{n}BuOH in the absence of any additives. The reaction was rather reluctant. Irradiating the solution of phosphonic acid **7** and a 15-fold excess of ^{n}BuOH in a closed vessel at 180 °C, a conversion of 70 % could be obtained after 6 h, and the monoester (**8b**) was formed almost exclusively (Table 6/Entry 1). In a comparative thermal experiment carried out in a bomb tube applying the same reaction conditions, the conversion was only 17 %. Higher, but still incomplete conversions

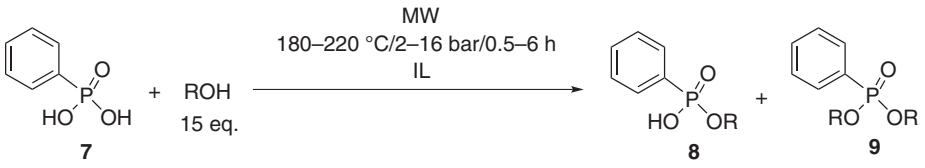


Scheme 4: Direct esterification of diphenylphosphinic acid (**5**).

Table 5: Esterification of sterically hindered phosphinic acids in the presence or absence of [bmim][PF_6] as a catalyst.

R	T (°C)	t (h)	IL	Conversion[a] (%)	Yield (%)	Entry
^{n}Bu	220	3	–	28 (**6b**)	20	1
	200	2	10 % [bmim][PF_6]	100 (**6b**)	94	2
^{n}Pent	235	6	–	36 (**6d**)	30	3
	200	2	10 % [bmim][PF_6]	100 (**6d**)	95	4
^{i}Pent	235	6	–	34 (**6e**)	22	5
	200	2	10 % [bmim][PF_6]	75 (**6e**)	65	6
^{n}Oct	235	6	–	61 (**6f**)	42	7
	200	2	10 % [bmim][PF_6]	100 (**6f**)	92	8

[a]On the basis of ^{31}P NMR.



Scheme 5: Direct esterification of diphenylphosphinic acid (**5**).

Table 6: Direct esterification of phenylphosphonic acid in the presence or absence of an ionic liquid.

R	IL	T (°C)	t (h)	Conv.[a] (%)	Composition[a]		Yield of the monoester (%)	Entry
					Monoester (%) (product)	Diester (%) (product)		
Bu	–	180	6	70[b]	98 (**8b**)	2 (**9b**)		1
	–	200	4[c]	83	92 (**8b**)	8 (**9b**)	75 (**8b**)	2
	–	220	4	88	89 (**8b**)	11 (**9b**)		3
	10 % [bmim][PF_6]	180	1	100	89 (**8b**)	11 (**9b**)		4
	10 % [bmim][BF_4]	180	0.75	100[d]	95 (**8b**)	5 (**9b**)	82 (**8b**)	5
	10 % [bmim][BF_4]	200	0.5	100	90 (**8b**)	10 (**9b**)		6
	10 % [bmim][BF_4]	200	4	100	70 (**8b**)	30 (**9b**)		7
^{n}Oct	–	220	4	96	75 (**8f**)	25 (**9f**)		8
	10 % [bmim][BF_4]	180	0.75	100	96 (**8f**)	4 (**9f**)	90 (**8f**)	9
	10 % [bmim][BF_4]	220	4	100	34 (**8f**)	66 (**9f**)		10

[a]On the basis of ^{31}P NMR.
[b]In a comparative thermal experiment carried out in a bomb tube, the conversion was 17 %, no diester was formed.
[c]No change in the conversion on further irradiation.
[d]In a comparative thermal experiment carried out in a bomb tube, the conversion was 22 % (composition: 93 % **8b** and 7 % **9b**).

could be reached at somewhat higher temperatures under MW conditions, however, at 200/220 °C, 8–11 % of the corresponding diester (**9b**) was also present in the crude mixture (Table 6/Entries 2 and 3). The addition of 10 % of IL allowed complete conversions at 180 °C after 1 h using [bmim][PF_6], or after 45 min applying [bmim][BF_4] (Table 6/Entries 4 and 5). In the latter case, the selectivity was better (95 %). It is worth mentioning, that when the same reaction was carried out in a bomb tube (Table 6/Entry 5[d]), a conversion of 22 % was observed, meaning that the IL additive somewhat promoted the reaction (compare Table 6/Entry 5[d] and Table 6/Entry 1[b]). A beneficial synergism between MWs and the IL could be observed. Increasing the reaction temperature to 200 °C, although the reaction was complete already after 30 min, somewhat more diester (**9b**) was formed. A prolonged heating resulted in an increase in the proportion of the diester (**9b**) (Table 6/Entries 6 vs. 7). Applying *n*-octanol, the same tendencies could be observed (Table 6/Entries 8 and 9). A further increase in the temperature and reaction time resulted in the formation of diester **9f** as the main component (Table 6/Entry 10). Thus, we can conclude that by changing the conditions, the reaction may be fine-tuned, and hence either the mono- (**8**), or the diester (**9**) may be obtained in a high selectivity.

Conclusions

In summary, our novel MW-assisted IL-supported direct esterification method could be used well in the esterification of acyclic phosphinic and phosphonic acids. Phenyl-*H*-phosphinates, methyl-phenylphosphinates, diphenylphosphinates and phenylphosphonic acid monoesters were obtained in good to excellent yields at 140–200 °C after 0.5–2 h reaction times in the presence of a catalytic amount of an IL. The first direct esterifications of a phosphonic acid were elaborated.

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