

Ionic liquids as efficient catalysts for synthesis of phosphorylated dialkyl succinates containing phthalazin-1-one moieties

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Received 20 January 2014; received in revised form 1 February 2014; accepted 6 March 2014

ABSTRACT

Ionic liquids such as 1,3-dialkylimidazolium salts make excellent catalysts and solvents for synthesis of dialkyl 2-(dialkoxyphosphoryl)-3-(1-oxo-1-*H*-phthalazin-2-yl)succinates from 2*H*-phthalazin-1-one, dialkyl acetylenedicarboxylates, and trialkyl phosphites. Under similar conditions, triphenyl phosphite led to dialkyl 2-(diphenoxyphosphoryl)-3-(1-oxo-1-*H*-phthalazin-2-yl)succinates. The ionic liquid was readily recycled and can be reused four times without significant loss of activity or mass.

Keywords: Phosphorylation, Organocatalysis, 2*H*-Phthalazin-1-one, Acetylenic ester, Ionic liquid.

1. Introduction

Ionic liquids (ILs) are ionic compounds that have a melting point below 100 °C. Most of the commonly used ILs are liquids at room temperature. Ionic liquids have a high polarity (usually between acetonitrile and methanol) and low vapor pressure. These features combined with the fact that most ionic liquids are immiscible with less polar organic solvents led to their use as media or co-solvent in catalysis. The importance of this area is highlighted by the increasing number of reviews and books dedicated to the topic [1-4]. Room temperature ionic liquids (RTILs), especially those based on the 1,3-di-alkylimidazolium salts, have shown great promise as an attractive alternative to conventional solvents [3]. RTILs possess the unique advantages of high thermal stability, negligible vapor pressure, immiscibility with a number of organic solvents and recyclability [1,3].

Organophosphorus compounds, *i.e.* those bearing a carbon atom directly bound to a phosphorus atom, are synthetic targets of interest, not least because of their value for a variety of industrial, biological, and chemical synthetic uses [4,5]. The successful attack by nucleophilic trivalent phosphorus on a carbon center is facilitated when the latter is part of, or conjugated with, a carbonyl group, or when it is part of an unsaturated bond otherwise activated [6-8].

There are many studies on the reaction between trivalent phosphorus nucleophiles and α,β -unsaturated carbonyl compounds in the presence of a proton source such as alcohol or phenol [8-10].

Herein, we report the results of our studies involving the reaction of zwitterions derived from trialkyl phosphites **1** and dialkyl acetylenedicarboxylates **2** with 2*H*-phthalazin-1-one (**3**), in 1-butyl-3-methylimidazolium trifluoromethanesulfonate ([bmim]CF₃SO₃), which constitute a facile one-pot diastereoselective synthesis of dialkyl 2-(dialkoxyphosphoryl)-3-(1-oxo-1-*H*-phthalazin-2-yl)succinates **4** in good yields. Under similar conditions, triphenyl phosphite led to dialkyl 2-(diphenoxyphosphoryl)-3-(1-oxo-1-*H*-phthalazin-2-yl)succinates.

2. Experimental

2.1. General

Compounds **1-3** and the ILs were obtained from Fluka and were used without further purification. M.p.: Electrothermal-9100 apparatus. IR Spectra: Shimadzu IR-460 spectrometer. ¹H- and ¹³C-NMR spectra: Bruker DRX-300 AVANCE instrument; in CDCl₃ at 300 and 75 MHz, respectively; δ in ppm, *J* in Hz. EI-MS (70 eV): Finnigan-MAT-8430 mass spectrometer, in *m/z*. Elemental analyses (C, H, N) were performed with a Heraeus CHN-O-Rapid analyzer. The results agreed favorably with the calculated values.

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2.2. General procedure for the preparation of compound 4a-4f

To a stirred solution of 2*H*-phthalazin-1-one (3, 0.146 g, 1 mmol) and ester 2 (1 mmol) in 2 mL of [bmim]CF₃SO₃ was added phosphite 1 (1 mmol) at room temperature over 5 min. After completion of the reaction (30 min), as indicated by TLC (EtOAc/*n*-hexane, 2:1), the products were extracted with Et₂O (2 × 5 mL). The solvent was evaporated under reduced pressure to leave the crude product, which was purified by column chromatography on silica gel and eluted with a mixture of *n*-hexane:EtOAc (3:1) to afford pure product. The IL was recovered by addition of water (5 mL), separated and dried under vacuum. These reactions were performed without any protective atmosphere of inert gas.

Selected spectral data

Dimethyl 2-(dimethoxyphosphoryl)-3-(1-oxo-1*H*-phthalazin-2-yl)succinate (4a):

Colorless powder; m.p.: 144-145 °C; Yield: 0.37 g (92%). IR (KBr): $\bar{\nu}$ = 1743, 1663 (C=O), 1270 (P=O) cm⁻¹. ¹HNMR (300 MHz, CDCl₃): δ = 3.63 (3H, d, ³J_{HP} = 10.8 Hz, MeO), 3.69 (3H, d, ³J_{HP} = 10.8 Hz, MeO), 3.71 (3H, s, MeO), 3.87 (3H, s, MeO), 4.34 (1H, dd, ³J_{HH} = 11.2 Hz, ²J_{HP} = 20.4 Hz, P-CH), 6.23 (1H, br s, P-C-CH), 7.72 (1H, d, ³J_{HH} = 7.2 Hz, CH), 7.78 (1H, t, ³J_{HH} = 7.6 Hz, CH), 7.82 (1H, t, ³J_{HH} = 7.6 Hz, CH), 8.2 (1H, s, CH), 8.44 (1H, d, ³J_{HH} = 7.6 Hz, CH) ppm. ¹³CNMR (75 MHz, CDCl₃): δ = 44.3 (d, ¹J_{CP} = 129.8 Hz, P-C), 53.1 (MeO), 53.2 (MeO), 53.5 (MeO), 53.5 (MeO), 60.2 (P-C-C), 126.4 (CH), 127.0 (CH), 127.7 (C), 129.6 (C), 131.9 (CH), 133.6 (CH), 138.1 (CH), 138.6 (CH), 159.4 (C=O), 164.1 (C=O), 167.8 (d, ²J_{CP} = 3.3 Hz, C=O), 169.1 (d, ³J_{CP} = 19.0 Hz, C=O). MS (EI, 70 eV): *m/z* (%) = 398 (M⁺, 8), 366 (49), 339 (23), 307 (100), 289 (55), 257 (62), 109 (13) ppm. Anal. Calcd for C₁₆H₁₉N₂O₈P (398.09): C, 48.25; H, 4.81; N, 7.03. Found: C, 47.91; H, 4.87; N, 7.09%.

Diethyl 2-(dimethoxyphosphoryl)-3-(1-oxo-1*H*-phthalazin-2-yl)succinate (4b):

Colorless oil; Yield: 0.37 g (86%). IR (KBr): $\bar{\nu}$ = 1664, 1740 (C=O), 1255 (P=O) cm⁻¹. ¹HNMR (300 MHz, CDCl₃): δ = 1.17 (3H, t, ³J_{HH} = 7.0 Hz, Me), 1.35 (3H, t, ³J_{HH} = 7.0 Hz, Me), 3.64 (3H, d, ³J_{HP} = 11.0 Hz, MeO), 3.69 (3H, d, ³J_{HP} = 11.0 Hz, MeO), 4.19 (2H, q, ³J_{HH} = 7.0 Hz, OCH₂), 4.31 (2H, q, ³J_{HH} = 7.0 Hz, OCH₂), 4.32 (1H, dd, ³J_{HH} = 11.5 Hz, ²J_{HP} = 20.5 Hz, P-CH), 6.20 (1H, br s, P-C-CH), 7.71 (1H, d, ³J_{HH} = 7.5 Hz, CH), 7.78 (1H, t, ³J_{HH} = 7.5 Hz, CH), 7.82 (1H, t, ³J_{HH} = 7.5 Hz, CH), 8.19 (1H, s, CH), 8.44 (1H, d, ³J_{HH} = 8.0, CH) ppm. ¹³CNMR (75 MHz, CDCl₃): δ = 13.8 (Me), 14.0 (Me), 44.5 (d, ¹J_{CP} = 129.3 Hz, P-C), 53.4 (d, ²J_{CP} = 5.0 Hz, MeO), 53.5 (d, ²J_{CP} = 5.0 Hz, MeO), 60.2 (P-C-C), 61.8 (OCH₂), 62.2 (OCH₂), 126.4 (CH),

127.0 (CH), 127.8 (C), 129.6 (C), 131.9 (CH), 133.5 (CH), 138.4 (CH), 159.4 (C=O), 167.2 (d, ²J_{PC} = 5.3 Hz, C=O), 168.4 (d, ³J_{PC} = 18.5 Hz, C=O) ppm. MS (EI, 70 eV): *m/z* (%) = 426 (M⁺, 6), 394 (45), 353 (25), 322 (100), 317 (35), 285 (70), 109 (11). Anal. Calcd for C₁₈H₂₃N₂O₈P (426.12): C, 50.71; H, 5.44; N, 6.57. Found: C, 51.32; H, 5.40; N, 6.64%.

Dimethyl 2-(diethoxyphosphoryl)-3-(1-oxo-1*H*-phthalazin-2-yl)succinate (4c):

Colorless oil; Yield: 0.35 g (82%). IR (KBr): $\bar{\nu}$ = 1663, 1744 (C=O), 1250 (P=O) cm⁻¹. ¹HNMR (300 MHz, CDCl₃): δ = 1.09 (3H, t, ³J_{HH} = 7.0 Hz, Me), 1.24 (3H, t, ³J_{HH} = 7.0 Hz, Me), 3.71 (3H, s, MeO), 3.86 (3H, s, MeO), 3.97-4.01 (2H, m, ABX₃, OCH₂), 4.03-4.07 (2H, m, ABX₃, OCH₂), 4.32 (1H, dd, ³J_{HH} = 11.5 Hz, ²J_{HP} = 20.5 Hz, P-CH), 6.25 (1H, dd, ³J_{HH} = 11.5 Hz, ³J_{HP} = 7.0 Hz, P-C-CH), 7.70 (1H, d, ³J_{HH} = 7.4 Hz, CH), 7.77 (1H, t, ³J_{HH} = 7.4 Hz, CH), 7.83 (1H, t, ³J_{HH} = 7.4, CH), 8.18 (1H, s, CH), 8.43 (1H, d, ³J_{HH} = 8. Hz, CH) ppm. ¹³CNMR (75 MHz, CDCl₃): δ = 16.0 (d, ³J_{CP} = 5.8, Me), 16.1 (d, ³J_{CP} = 5.8, Me), 45.0 (d, ¹J_{CP} = 129.0 Hz, P-C), 52.7 (MeO), 52.9 (MeO), 60.4 (br s, P-C-C), 63.0 (d, ²J_{CP} = 6.0 Hz, OCH₂), 63.1 (d, ²J_{CP} = 6.0, OCH₂), 126.3 (CH), 127.2 (CH), 129.6 (C), 131.8 (C), 133.5 (CH), 138.0 (CH), 138.3 (CH), 159.3 (C=O), 167.9 (d, ²J_{CP} = 6.5 Hz, C=O), 169.0 (d, ³J_{PC} = 18.3 Hz, C=O) ppm. MS (EI, 70 eV): *m/z* (%) = 426 (M⁺, 15), 380 (55), 367 (20), 322 (100), 289 (60), 285 (58), 137 (15). Anal. Calcd for C₁₈H₂₃N₂O₈P (426.12): C, 50.71; H, 5.44; N, 6.57. Found: C, 50.36; H, 5.48; N, 6.65%.

Diethyl 2-(diethoxyphosphoryl)-3-(1-oxo-1*H*-phthalazin-2-yl)succinate (4d):

Colorless oil; Yield: 0.34 g (75%). IR (KBr): $\bar{\nu}$ = 1664, 1744 (C=O), 1250 (P=O) cm⁻¹. ¹HNMR (300 MHz, CDCl₃): δ = 1.11 (3H, t, ³J_{HH} = 7.0 Hz, Me), 1.17 (3H, t, ³J_{HH} = 7.0 Hz, Me), 1.24 (3H, t, ³J_{HH} = 7.0 Hz, Me), 1.35 (3H, t, ³J_{HH} = 7.0 Hz, Me), 3.98-4.02 (2H, m, ABX₃, OCH₂), 4.02-4.10 (2H, m, ABX₃, OCH₂), 4.18 (2H, q, ³J_{HH} = 7.0 Hz, OCH₂), 4.29 (2H, q, ³J_{HH} = 7.0 Hz, OCH₂), 4.30 (1H, dd, ³J_{HH} = 11.5 Hz, ²J_{HP} = 20.5 Hz, P-CH), 6.20 (1H, br s, P-C-CH), 7.69 (1H, d, ³J_{HH} = 7.5 Hz, CH), 7.77 (1H, t, ³J_{HH} = 7.5 Hz, CH), 7.82 (1H, t, ³J_{HH} = 7.5 Hz, CH), 8.18 (1H, s, CH), 8.43 (1H, d, ³J_{HH} = 7.7 Hz, CH) ppm. ¹³CNMR (75 MHz, CDCl₃): δ = 13.8 (Me), 14.0 (Me), 16.0 (d, ³J_{CP} = 5.3, Me), 16.2 (d, ³J_{CP} = 5.3, Me), 45.2 (d, ¹J_{CP} = 128.8 Hz, P-C), 60.5 (P-C-C), 61.8 (OCH₂), 62.2 (OCH₂), 62.9 (d, ²J_{CP} = 6.0 Hz, OCH₂), 64.0 (d, ²J_{CP} = 6.0, OCH₂), 126.3 (CH), 127.0 (CH), 127.8 (C), 129.6 (C), 131.8 (CH), 133.4 (CH), 138.2 (CH), 159.3 (C=O), 167.4 (d, ²J_{CP} = 6.0 Hz, C=O), 168.5 (d, ³J_{CP} = 18.5 Hz, C=O) ppm. MS (EI, 70 eV): *m/z* (%) = 454 (M⁺, 17), 408 (45), 381 (28), 337 (100), 317 (51), 313 (68), 137 (15). Anal. Calcd for C₂₀H₂₇N₂O₈P (454.15): C, 52.86; H, 5.99; N, 6.16. Found: C, 53.26; H, 6.07; N, 6.22%.

Dimethyl 2-(diphenoxyphosphoryl)-3-(1-oxo-1H-phthalazin-2-yl)succinate (4e):

Colorless oil; Yield: 0.44 g (85%). IR (KBr): $\bar{\nu}$ = 1665, 1743 (C=O), 1249 (P=O) cm^{-1} . $^1\text{H NMR}$ (300 MHz, CDCl_3): δ = 3.74 (3H, s, MeO), 3.90 (3H, s, MeO), 4.67 (1H, dd, $^3J_{\text{HH}} = 11.5$ Hz, $^2J_{\text{HP}} = 21.3$ Hz, P-CH), 6.50 (1H, dd, $^3J_{\text{HH}} = 11.5$ Hz, $^3J_{\text{HP}} = 6.5$ Hz, P-C-CH), 7.02-7.13 (m, 5H, OPh), 7.2-7.25 (m, 5H, OPh), 7.65 (1H, d, $^3J_{\text{HH}} = 7.0$ Hz, CH), 7.70 (1H, t, $^3J_{\text{HH}} = 7.0$ Hz, CH), 7.81 (1H, t, $^3J_{\text{HH}} = 7.0$ Hz, CH), 8.11 (1H, s, CH), 8.42 (1H, d, $^3J_{\text{HH}} = 7.7$ Hz, CH) ppm. $^{13}\text{C NMR}$ (75 MHz, CDCl_3): δ = 45.9 (d, $^1J_{\text{CP}} = 129.5$ Hz, P-C), 52.6 (MeO), 52.9 (MeO), 60.2 (br s, P-C-C), 120.2 (CH), 120.2 (CH), 125.2 (CH), 125.2 (CH), 125.9 (CH), 126.1 (CH), 127.2 (CH), 127.8 (C), 129.2 (C), 131.8 (CH), 133.4 (CH), 138.5 (CH), 141.2 (C), 142.2 (C), 159.4 (C=O), 167.9 (d, $^2J_{\text{CP}} = 6.4$ Hz, C=O), 169.0 (d, $^3J_{\text{CP}} = 18.2$, C=O) ppm. MS (EI, 70 eV): m/z (%) = 524 (M^+ , 14), 430 (48), 465 (30), 383 (40), 372 (100), 289 (53), 235 (20), 71 (17). Anal. Calcd for $\text{C}_{26}\text{H}_{23}\text{N}_2\text{O}_8\text{P}$ (522.12): C, 59.77; H, 4.44; N, 5.36. Found: C, 60.14; H, 4.49; N, 5.42%.

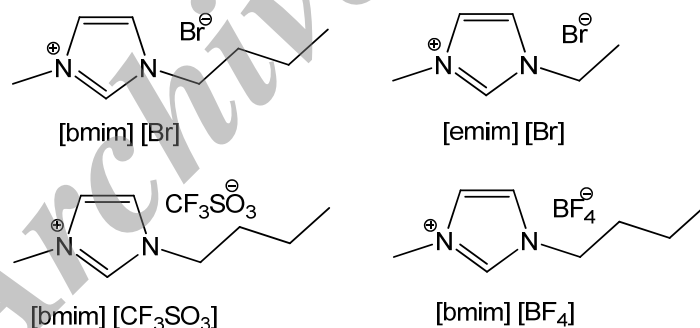
Diethyl 2-(diphenoxyphosphoryl)-3-(1-oxo-1H-phthalazin-2-yl)succinate (4f):

Colorless oil; Yield: 0.46 g (83%). IR (KBr): $\bar{\nu}$ = 1665, 1744 (C=O), 1249 (P=O) cm^{-1} . $^1\text{H NMR}$ (300 MHz, CDCl_3): δ = 1.19 (3H, t, $^3J_{\text{HH}} = 7.2$ Hz, Me), 1.34 (3H, t, $^3J_{\text{HH}} = 7.2$, Me), 4.23 (2H, q, $^3J_{\text{HH}} = 7.2$ Hz,

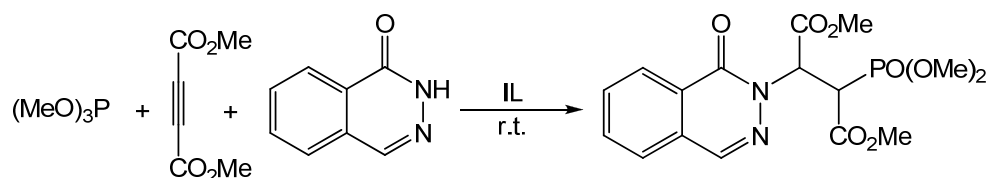
OCH₂), 4.35 (2H, q, $^3J_{\text{HH}} = 7.2$ Hz, OCH₂), 4.64 (1H, dd, $^3J_{\text{HH}} = 11.1$ Hz, $^3J_{\text{HP}} = 21.2$ Hz, P-CH), 6.47 (1H, dd, $^3J_{\text{HH}} = 11.1$ Hz, $^3J_{\text{HP}} = 6.5$ Hz, P-C-CH), 7.04-7.11 (m, 5H, OPh), 7.20-7.28 (m, 5H, OPh), 7.64 (1H, d, $^3J_{\text{HH}} = 8.0$ Hz, CH), 7.74 (1H, t, $^3J_{\text{HH}} = 8.0$ Hz, CH), 7.80 (1H, t, $^3J_{\text{HH}} = 8.0$ Hz, CH), 8.11 (1H, s, CH), 8.42 (1H, d, $^3J_{\text{HH}} = 8.0$ Hz, CH) ppm. $^{13}\text{C NMR}$ (75 MHz, CDCl_3): δ = 14.0 (Me), 14.1 (Me), 46.0 (d, $^1J_{\text{CP}} = 134.0$ Hz, P-C), 62.2 (OCH₂), 62.4 (OCH₂), 60.5 (br s, P-C-C), 120.4 (CH), 120.5 (CH), 125.2 (CH), 125.2 (CH), 125.7 (CH), 125.8 (CH), 126.2 (CH), 127.1 (CH), 127.8 (C), 129.1 (C), 131.8 (CH), 133.5 (CH), 138.5 (CH), 141.2 (C), 141.3 (C), 159.5 (C=O), 167.3 (d, $^2J_{\text{CP}} = 5.5$ Hz, C=O), 168.5 (d, $^3J_{\text{CP}} = 18.1$ Hz, C=O) ppm. MS (EI, 70 eV): m/z (%) = 552 (M^+ , 17), 479 (28), 458 (50), 410 (63), 386 (100), 317 (40), 235 (27), 71 (15). Anal. Calcd for $\text{C}_{28}\text{H}_{27}\text{N}_2\text{O}_8\text{P}$ (550.15): C, 61.09; H, 4.94; N, 5.09. Found: C, 61.46; H, 5.02; N, 5.14%.

3. Results and Discussion

Initially, we employed four ILs, shown in Scheme 1, as reaction media and catalysts for phosphorylation reaction. Thus, trimethyl phosphite (**1a**), dimethyl acetylenedicarboxylate (DMAD, **2a**), and 2H-phthalazine-1-one (**3**) were chosen as model compounds for optimization of the reaction conditions. The effects of the type of IL used in the reaction, are investigated. As shown in Scheme 2, [bmim]CF₃SO₃



Scheme 1. The structures of different ILs used in this study.



IL	Yield/ %
[bmim][Br]	56
[emim][Br]	50
[bmim][CF ₃ SO ₃]	92
[bmim][BF ₄]	62

Scheme 2. The reaction of **1a**, DMAD, and **3** in different ILs as solvent and catalyst.

exhibited better results. When the reactions were carried out in a 1:1 mixture of [bmim]CF₃SO₃ and H₂O or CH₂Cl₂, the yields were much lower.

The reaction of trialkyl (aryl) phosphites **1** and acetylenic esters **2**, and 2*H*-phthalazin-1-one (**3**) in [bmim]CF₃SO₃ proceeded smoothly at room temperature and afforded **4a-f** in good yields (Scheme 3). The reactions were complete within 30 min.

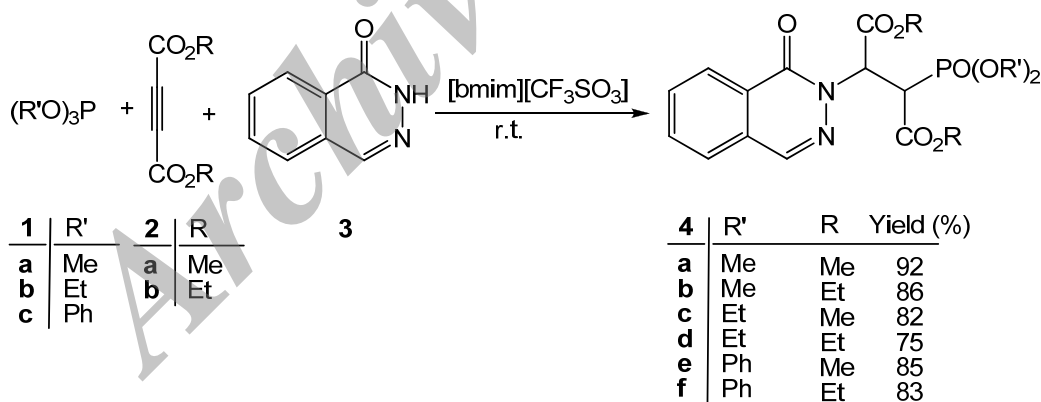
The essential structures of compound **4** were apparent from their mass spectra, which display, in each case, the molecular ion peak at appropriate *m/z* values. The ¹H and ¹³C NMR spectroscopic data, as well as IR spectra, are in agreement with the proposed structures. For example, the ¹H NMR spectrum of **4a** showed two doublets for the two diastereotopic methoxy ($\delta = 3.63$ ppm, ³*J*_{HP} = 10.8 Hz and $\delta = 3.69$ ppm, ³*J*_{HP} = 10.8 Hz) groups. The two singlets at $\delta = 3.71$ and 3.87 ppm belong to the methoxycarbonyl protons. Observation of ³*J*_{HH} = 11.2 Hz for the vicinal methane proton in **4a** indicates the anti arrangement. Since compound **4a** possesses two stereogenic centers, two diastereoisomers with anti-HCCH arrangements are possible (see Experimental).

The presence of ³¹P nucleus in compounds **4**, helps in the assignment of the signals by long range couplings with ¹H and ¹³C nuclei. The three-bond carbon-

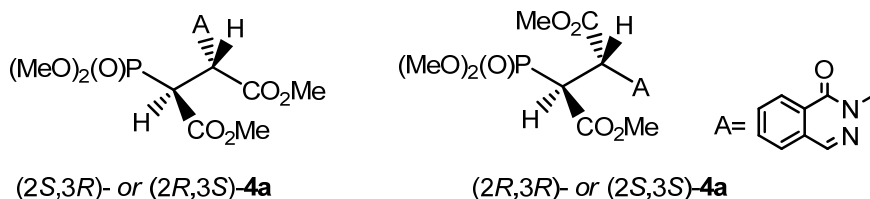
phosphorus couplings, ³*J*_{CP}, depends on configuration, as expected, transoid couplings being larger than cisoid ones. The Karplus relation can be derived from the data for organophosphorus compound with tetra and pentavalent phosphorus [11]. Observation of ³*J*_{CP} = 19.0 Hz for the carbonyl carbon atom of the CO₂Me group is in agreement with the (2*S*,3*R*)-**4a** or (2*R*,3*S*)-**4a** diastereoisomer (Scheme 4).

Phosphonate ester **4** apparently results from the initial addition of phosphite **1** to the acetylenic ester **2** and subsequent protonation of the 1:1 adduct by the NH-acid **3**. Then, the positively charged ion **6** might be attacked by the conjugate base of the NH-acid to form phosphorane **8**, which is hydrolyzed to the phosphonate ester **4** (see Scheme 5). Hydrolysis of alkyl triphenoxyphosphonium salts in water has been reported to yield diphenyl alkylphosphonates [12]. There are also examples in the literature of the use of methyltriphenoxyphosphonium iodide as a dehydrating agent [12].

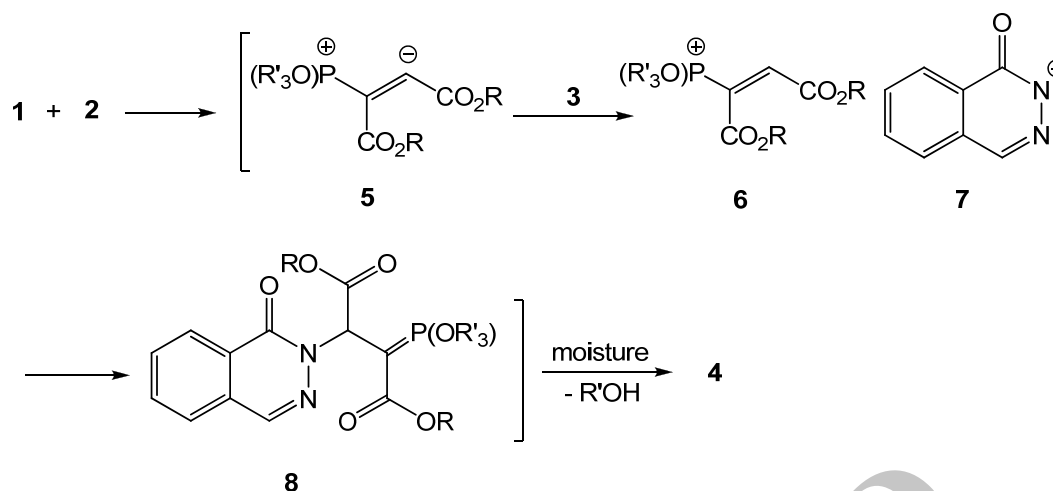
The role of the IL may be postulated in terms of some Lewis/Bronsted acidity of the imidazolium cation leading to its interaction with the carbonyl oxygen of the acetylenic ester resulting in its increased polarization and electrophilicity [13]. Thus, promoting the addition and cyclization steps to give product **4** (Scheme 5).



Scheme 3. Preparation of compound **4a-f** from phosphites, acetylenic esters, and **3** in [bmim]CF₃SO₃.



Scheme 4. The diastereoisomers of product **4a**.



Scheme 5. Plausible mechanism for the formation of products 4a-4f.

The ionic liquid was readily recycled and can be reused four times without significant loss of activity or mass.

4. Conclusion

In conclusion, ionic liquids are proved to be useful catalysts and solvents for diastereoselective synthesis of phosphorylated 2*H*-phthalazin-1-ones from 2*H*-phthalazin-1-one, dialkyl acetylenedicarboxylates, and trialkyl(aryl) phosphites. The effects of the type of four ILs were investigated. The ionic liquids are successfully regenerated and reused. The one-pot nature of the present procedure makes it an alternative to multistep approaches.

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