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Unprecedented acceleration of the domino reaction between methyl 4-hydroxyalk-3-ynoates and amines in ionic liquids

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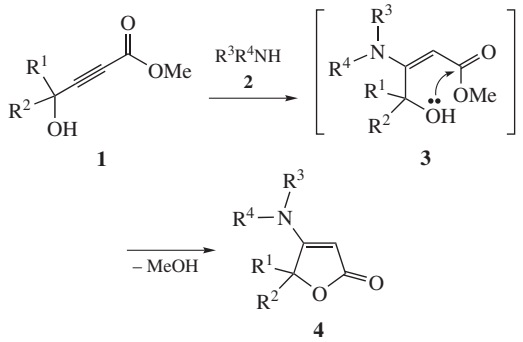
Reaction between methyl 4-hydroxyalk-3-ynoates and amines leading to 4-aminofuran-2(5*H*)-ones is significantly accelerated in ionic liquids, which can be recycled at least five times.

The butenolide [furan-2(5*H*)-one] fragment is an integral part of a variety of biologically active compounds and natural products such as vitamin C, steroids, pheromones, acetogenins, digitoxin and related cardenolides.[1,2] Commonly, it is formed by multi-step reaction sequences, which require the use of absolute solvents and/or expensive reagents.[3–6] Examples of more efficient one-pot synthetic procedures are scarce. Among them there is a reaction of methyl 4-hydroxyalk-3-ynoates **1** with secondary amines **2** that includes aza-Michael addition of amines **2** to activated triple bond of **1** followed by the lactonisation of thus *in situ* generated adducts **3** (Scheme 1).[7] This reaction is a convenient protocol for the synthesis of 4-aminobut-2-en-4-olides **4**, which are versatile intermediates for the preparation of various furan-2(5*H*)-ones. However, it runs slowly (1–10 days) in organic solvents (diethyl ether, alcohols, acetone, dioxane, *etc*.).

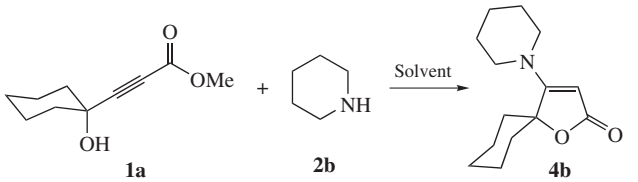


Scheme 1

Taking into account that reactants **1** and **2** as well as intermediates **3** are polar molecules, we anticipated that the reaction would run faster in ionic liquids (ILs) that have attracted attention over the last decade as neoteric solvents and catalysts in the organic synthesis.[8–12] In particular, they promote reactions of nucleophiles with compounds bearing electron-deficient double bonds C=C[13–20] or C=O,[21–24] among them a cascade reaction that includes a lactonisation stage.[24] However, to the best of our knowledge, ILs have not so far been applied to Michael additions involving electron-deficient alkynes.

First, we studied a model reaction between alkyne **1a** and piperidine **2b** in various ILs and compared the obtained results with those attained earlier in Et_2O and under neat conditions. Commercial 1-butyl-3-methylimidazolium tetrafluoroborate [bmim][BF_4] and hexafluorophosphate [bmim][PF_6], 1-hexyl-3-methylimidazolium tris(pentafluoroethyl) trifluorophosphate [hmim][$PF_3(C_2F_5)_3$],

Table 1 Reaction between compounds **1a** and **2b**.[a]



Entry	Solvent	*T*/°C	*t*/h	Yield of **4b** (%)
1	[bmim][BF_4]	20	1	85
2	[bmim][PF_6]	20	1	80
3	[hmim][$PF_3(C_2F_5)_3$]	20	1	82
4	[bmpl][TfO]	20	1	73
5	[bmpl][NTf_2]	20	1	85
6[b]	Et_2O	20	140	67
7[c]	Et_2O	20	140	64
8	neat	20	22	58
9[b]	neat	100	4	62

[a]The reactions were carried out with **1a** (1 mmol), **2b** (1.5 mmol) and the indicated solvent (3 mmol for entries 1–5 or 6 ml for entry 6). [b]Data according to ref. 7(*a*). [c]The reaction was carried out in the presence of HCl (cat.).

1-butyl-3-methylpyrrolidinium triflate [bmpl][TfO] and bis(triflyl)-amide [bmpl][NTf_2] were used as ILs. In all cases 4-aminofuran-2(5*H*)-one **4b** was formed much faster, at lower temperature and in improved yield than in Et_2O or under solvent-free conditions (Table 1). This acceleration cannot be caused by acidic impurities in ILs because they (if any) should be instantly neutralized by the amine **2b** taken in excess. Furthermore, the addition of catalytic amount of HCl did not influence the same reaction in Et_2O (Table 1, entry 7).

Then, the least expensive IL [bmim][BF_4], in which butenolide **4b** was obtained in high yield (85%), was applied to reactions between various alkynes **1** and amines **2** (Table 2).[†] Alkynes containing cyclohexanol (entries 1–11, 16–19), cyclopentanol (entries 12, 13), propan-2-ol (entry 14) and butan-2-ol (entry 15)

[†] *Typical procedure*. A mixture of alkyne **1** (1.0 mmol), amine **2** (1.5 mmol) and IL (3 mmol) or organic solvent (6 ml) was stirred under the conditions specified in Tables 1 and 2 and successively extracted with light petroleum (40–70 °C) (5 ml), the Et_2O–light petroleum mixture (1:1) (5 ml) and Et_2O (5 ml). The combined extracts were passed through a silica gel pad, solvents were evaporated *in vacuo* (15 Torr) and the residue was crystallized or purified by column chromatography on silica gel (Acros, 40–60 μm). In the case of compounds **4j**,**r**,**s**, reaction mixtures were diluted with water (3 ml) and precipitated products were filtered off and recrystallized.

Table 2 Synthesis of 4-aminofuran-2(5*H*)-one derivatives **4** from compounds **1** and **2** in [bmim][BF_4].

Entry	Product	R^1	R^2	R^3	R^4	*T*/°C (lit.)	*t*/h (lit.)	Yield (%) (lit.)
1	**4a**	$-(CH_2)_5-$		$-(CH_2)_4-$		20 (20[a])	1 (48[a])	80 (73[a])
2	**4b**	$-(CH_2)_5-$		$-(CH_2)_5-$		20 (20[a], 100[b])	1 (140[a], 4[b])	85 (67[a], 62[b])
3	**4c**	$-(CH_2)_5-$		$-(CH_2)_6-$		30 (20[a])	1 (48[a])	88 (53[a])
4	**4d**	$-(CH_2)_5-$		$-(CH_2)_2O(CH_2)_2-$		20 (20[a])	1 (140[a])	85 (72[a])
5	**4e**	$-(CH_2)_5-$		$-(CH_2)_2N(Me)(CH_2)_2-$		40 (20[a], 65[c])	4 (48[a], 7[c])	90 (59[a], 47[c])
6	**4f**	$-(CH_2)_5-$		$-CH(Me)(CH_2)_4-$		20 (20[a])	2 (250[a])	87 (82[a])
7	**4g**	$-(CH_2)_5-$		$-(CH_2)_2N[C(O)Ph](CH_2)_2-$		20 (65[c])	8 (6[c])	86 (62[c])
8	**4h**	$-(CH_2)_5-$		$-(CH_2)_2N(CO_2Et)(CH_2)_2-$		20 (20[a])	6 (140[a])	80 (71[a])
9	**4i**	$-(CH_2)_5-$		**2** = imidazole		60 (65[c])	2 (12[c])	18 + 52[d] (35 + 8[c,d])
10	**4j**	$-(CH_2)_5-$		**2** = (*S*)-anabasine		20 (65[c], 100[e])	6 (20[c], 12[e])	99 (62[c], 74[e])
11	**4k**[f]	$-(CH_2)_5-$		**2** = (*S*)-cytisine		20 (20[g])	8 (150[g])	70 (64[g])
12	**4l**[f]	$-(CH_2)_4-$		$-(CH_2)_5-$		20 (20[h])	1 (48[h])	95 (65[h])
13	**4m**[f]	$-(CH_2)_4-$		$-(CH_2)_2N(Me)(CH_2)_2-$		20 (20[h])	1 (45[h])	96 (60[h])
14	**4n**[f]	Me	Me	$-(CH_2)_4-$		20 (20[h])	1 (48[h])	88 (66[h])
15	**4o**[f]	Me	Et	$-(CH_2)_4-$		20 (20[h])	1 (48[h])	90 (68[h])
16	**4p**[f]	$-(CH_2)_5-$		$Me_2CH(CH_2)_2$	$Me_2CH(CH_2)_2$	20 (20[g])	5 (25[g])	88 (60[g])
17	**4q**	$-(CH_2)_5-$		Bn	Bn	20 (20[a])	4 (250[a])	82 (78[a])
18	**4r**	$-(CH_2)_5-$		H	Bn	20 (20[c], 100[e])	3 (24[c], 8[e])	80 (70[c], 67[e])
19	**4s**	$-(CH_2)_5-$		H	CH(Me)Ph	20 (20[a])	8 (12[a])	70 (58[a])

[a]Solvent – Et_2O, data according to ref. 7(a). [b]Neat conditions, data according to ref. 7(*a*). [c]Solvent – MeOH, data according to ref.7(*a*). [d]Yield of compound **3i**. [e]Solvent – BuOH, data according to ref. 7(*b*). [f]New compounds. [g]Solvent – Et_2O/MeOH (2:1, v/v). [h]Solvent – Et_2O.

fragments acted as Michael acceptors. Cyclic amines (entries 1–15), in particular alkaloids (entries 10, 11), as well as acyclic secondary (entries 16, 17) or primary (entries 18, 19) amines were used as nucleophiles.

As a rule, yields of corresponding butenolides **4a**–**s** were close or higher than those reported in literature. Furthermore, the reaction time was much shorter in the IL medium than that in traditional organic solvents (TLC monitoring). The sole exception was the reaction between alkyne **1a** and imidazole (entry 9), in which furan derivative **4i** was formed in only 18% yield and the major product was enamine **3i**.‡ We succeeded in synthesizing several new compounds in the IL medium, in particular (*S*)-cytisine-derived butenolide **4k** (entry 11) and compounds **4l**–**p** (entries 12–16).§ For comparison, we synthesized these compounds in organic solvents as well and became convinced that the IL medium gave much better results in terms of both the product yield and the reaction rate (Table 2). Positive IL impact may be attributed to ion–dipole interactions in the IL medium, which facilitate the charge separation in compounds **1** and/or **3** and promote reactions (Figure 1).

‡ *Synthesis of compounds* **3i** *and* **4i**. A mixture of alkyne **1a** (0.18 g, 1.0 mmol), imidazole (0.12 g, 1.5 mmol) and [bmim][BF_4] (0.69 g, 3.0 mmol) was stirred at 60 °C for 2 h, cooled to ambient temperature and successively extracted with light petroleum (5 ml), light petroleum–diethyl ether (1:10) (5 ml) and light petroleum–benzene (1:1) (5 ml). The combined extracts were evaporated to a half volume, the precipitated solid was filtered off and crystallized from the light petroleum–Et_2O–EtOAc–$CHCl_3$ (5:5:2:1) mixture to afford methyl 3-(1-hydroxycyclohexyl)-3-(1*H*-imidazol-1-yl)-acrylate **3i**, yellowish solid, 0.13 g (52%), mp 110–111 °C. IR (KBr, ν/cm^{-1}): 1660, 1732, 3112. ^{1}H NMR (300 MHz, DMSO-d_6) δ: 0.95–1.75 (m, 10H, CH_2, cyclohexyl), 3.50 (s, 3H, OMe), 5.25 (s, 1H, OH), 6.35 (s, 1H, =CH), 6.90, 7.20, 7.45 (3s, 1H each, imidazole). Found (%): C, 62.61; H, 7.13; N, 10.98. Calc. for $C_{13}H_{18}N_2O_3$ (%): C, 62.38; H, 7.25; N,11.19.

Evaporation of mother liquor afforded 4-(1*H*-imidazol-1-yl)-1-oxaspiro-[4.5]dec-3-en-2-one **4i**, white solid, 40 mg (18%), mp 180–181 °C. IR (KBr, ν/cm^{-1}): 1632, 1748, 3148. ^{1}H NMR (300 MHz, DMSO-d_6) δ: 1.43–1.85 (m, 10H, CH_2, cyclohexyl), 6.52 (s, 1H, =CH), 7.21, 7.89, 8.50 (3s, 1H each, imidazole). Found (%): C, 66.27; H, 6.68; N, 12.57. Calc. for $C_{12}H_{14}N_2O_2$ (%): C, 66.04; H, 6.47; N, 12.84.

§ *4-[(*S*)-2-(3-Pyridyl)piperidin-1-yl]-1-oxaspiro[4.5]dec-3-en-2-one* **4j**: white solid, 0.31 g (99%), mp 220.5–222 °C (lit.,[7(b)] 221–222 °C), $[\alpha]_D^{20}$ –169 (*c* 0.47, $CHCl_3$), Chiralcel AD-H, eluent hexane–Pr^{i}OH (1:1), 0.8 ml min^{-1}, 254 nm, retention time 8.2 min (single); eluent hexane–Pr^{i}OH (7:3), 0.8 ml min^{-1}, 254 nm, retention time 9.9 min (single).

*(1*R*,5*S*)-3-(2-Oxo-1-oxaspiro[4.5]dec-3-en-4-yl)-1,2,3,4,5,6-hexahydro-8*H*-1,5-methanopyrido[1,2-a][1,5]diazocin-8-one* **4k**: white solid, 0.24 g (70%), mp 285–287 °C (decomp.), $[\alpha]_D^{20}$ –413 (*c* 0.45, $CHCl_3$). IR (KBr, ν/cm^{-1}): 1740 (C=O), 1630 (C=C). ^{1}H NMR (300 MHz, $CDCl_3$) δ: 0.98–1.98 (m, 10H, CH_2, cyclohexyl), 1.98–2.24 (m, 2H, H^8-cytisine), 2.6 (s, 1H, H^7-cytisine), 3.15 (s, 1H, H^9-cytisine), 3.30 (d, 2H, H^{10}-cytisine, *J* 12.5 Hz), 3.65–3.95 (m, 3H, NCH_2), 4.20 (d, 1H, NCH_2, *J* 12.5 Hz), 4.60 (s, 1H, =CH), 6.07 (d, 1H, H^5-cytisine, *J* 10.4 Hz), 6.46 (d, 1H, H^3-cytisine, *J* 12.5 Hz), 7.31 (t, 1H, H^4-cytisine, *J* 10.4 Hz). Found (%): C, 70.81; H, 7.17; N, 8.09. Calc. for $C_{20}H_{24}N_2O_3$ (%): C, 70.56; H, 7.11; N, 8.23. Chiralcel AD-H, eluent hexane–Pr^{i}OH (1:1), 0.8 ml min^{-1}, 220 nm, retention time 8.5 min (single); eluent hexane–Pr^{i}OH (7:3), 0.8 ml min^{-1}, 220 nm, retention time 16.1 min (single).

4-Piperidino-1-oxaspiro[4.4]non-3-en-2-one **4l**: white solid, 0.25 g (95%), mp 110–111 °C. IR (KBr, ν/cm^{-1}): 1722 (C=O), 1596 (C=C). ^{1}H NMR (300 MHz, $CDCl_3$) δ: 1.56–1.90 (m, 4H, CH_2), 1.90–2.18 (m, 6H, CH_2, piperidine), 3.19–3.37 (m, 4H, NCH_2, piperidine), 4.61 (s, 1H, =CH). Found (%): C, 70.31; H, 8.79; N, 6.15. Calc. for $C_{13}H_{19}NO_2$ (%): C, 70.55; H, 8.65; N, 6.33.

4-(4-Methylpiperazin-1-yl)-1-oxaspiro[4.4]non-3-en-2-one **4m**: white solid, 0.23 g (96%), mp 109–110 °C. IR (KBr, ν/cm^{-1}): 1722 (C=O), 1588 (C=O). ^{1}H NMR (300 MHz, $CDCl_3$) δ: 1.69–2.15 (m, 8H, CH_2, cyclopentyl), 2.42 (s, 3H, NMe), 2.48, 3.33 (2t, 4H each, NCH_2, *J* 4.6 Hz), 4.63 (s, 1H, =CH). Found (%): C, 66.25; H, 8.61; N, 11.64. Calc. for $C_{13}H_{20}N_2O_2$ (%): C, 66.07; H, 8.53; N, 11.86.

*5,5-Dimethyl-4-pyrrolidinofuran-2(5*H*)-one* **4n**: yellowish solid, 0.16 g (88%), mp 105–106 °C. IR (KBr, ν/cm^{-1}): 1722 (C=O), 1588 (C=C). ^{1}H NMR (300 MHz, $CDCl_3$) δ: 1.6 (s, 6H, Me), 1.92–2.09 (m, 4H, CH_2, pyrrolidine), 3.38 (br. s, 4H, NCH_2), 4.38 (s, 1H, =CH). Found (%): C, 65.92; H, 8.39; N, 7.58. Calc. for $C_{10}H_{15}NO_2$ (%): C, 66.27; H, 8.34; N, 7.73.

*5-Methyl-5-ethyl-4-pyrrolidinofuran-2(5*H*)-one* **4o**: white solid, 0.17 g (90%), mp 65–66 °C. IR (KBr, ν/cm^{-1}): 1722 (C=O), 1588 (C=C). ^{1}H NMR (300 MHz, $CDCl_3$) δ: 0.8 (t, 3H, CH_2*Me*, *J* 7.4 Hz), 1.51 (s, 3H, CMe), 1.83 (sept., 2H, C*H*$_2$Me, *J* 7.4 Hz), 1.85–2.05 (m, 4H, CH_2 cycl.), 3.36 (br. s, 4H, NCH_2), 4.37 (s, 1H, =CH). Found (%): C, 67.89; H, 8.64; N, 6.95. Calc. for $C_{11}H_{17}NO_2$ (%): C, 67.66; H, 8.78; N, 7.17.

4-[Bis(4-methylbutyl)amino]-1-oxaspiro[4.5]dec-3-en-2-one **4p**: white solid, 0.24 g (88%), mp 92–93 °C. IR (KBr, ν/cm^{-1}): 1720 (C=O), 1590 (C=C). ^{1}H NMR (300 MHz, $CDCl_3$) δ: 0.95 (d, 12H, Me, *J* 6.4 Hz), 1.05–2.00 (m, 14H, CH_2), 2.1 (sept., 2H, CH, *J* 6.5 Hz), 3.1 (d, 4H, NCH_2, *J* 7.6 Hz), 4.47 (s, 1H, =CH). Found (%): C, 73.88; H, 10.68; N, 4.89. Calc. for $C_{19}H_{33}NO_2$ (%): C, 74.22; H, 10.81; N, 4.56.

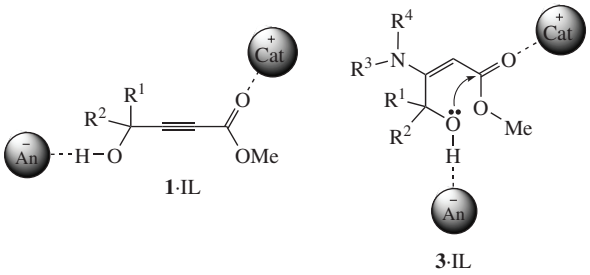


Figure 1 A plausible charge separation in compounds **1** and **3** in the IL medium.

According to HPLC (Chiralcel AD-H, eluent *n*-hexane/Pr^{i}OH 1:1–7:3) the obtained chiral compounds **4j** and **4k** were individual enantiomers. Their specific optical rotations remained high {$[\alpha]_D^{20}$ –169 (*c* 0.47, $CHCl_3$) for **4j** and –413 (*c* 0.45, $CHCl_3$) for **4k**} irrespective of the reaction period (6–24 h), the fact testifying against the racemisation of chiral centers in the IL medium.

The recyclability of IL [bmim][BF_4] was demonstrated in the syntheses of butenolides **4b**,**l**,**m**. After the extraction of the products, portions of the reactants **1** and **2** were added to the remaining IL and the reactions were re-performed. Reaction rates and product yields remained the same over at least five reaction cycles (Table 3).

Table 3 Recyclability of the IL in the studied reactions.[a]

Product	Yield (%) (cycle)
4b	85 (1), 87 (2), 90 (3), 90 (4), 90 (5)
4l	95 (1), 96 (2), 94 (3), 95 (4), 95 (5)
4m	96 (1), 96 (2), 97 (3), 96 (4), 95 (5)

[a]The reactions were carried out with **1** (1 mmol), **2** (1.5 mmol) and [bmim][BF_4] (3 mmol) at 20 °C for 1 h.

In conclusion, we have discovered that ionic liquids are solvents of choice for Michael additions of N-nucleophiles to activated alkynes. Their application allowed the time of the domino reaction between methyl 4-hydroxyalk-3-ynoates and amines affording 4-aminofuran-2(5*H*)-ones to become 10–30-fold shorter than that in traditional organic solvents. Moreover, ionic liquids can be recovered and reused at least five times without any decrease in reaction rates and product yields.

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