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A simple and convenient synthesis of substituted furans and pyrroles by $CuCl_2$-catalyzed heterocyclodehydration of 3-yne-1,2-diols and *N*-Boc- or *N*-tosyl-1-amino-3-yn-2-ols

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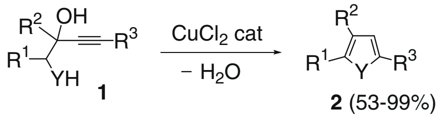
ABSTRACT

A simple and economical synthesis of substituted furans and pyrroles, by ligand-free $CuCl_2$-catalyzed heterocyclodehydration of readily available 3-yne-1,2-diols and *N*-Boc- or *N*-tosyl-1-amino-3-yn-2-ols, respectively, is presented. Reactions are carried out in MeOH at 80–100 °C for 1–24 h and afford the corresponding heterocyclic derivatives in 53–99% isolated yields.

Copper catalysis has recently acquired an increasing importance, in view of the higher availability, lower toxicity, and lower environmental impact of copper-based catalysts when compared with other commonly employed transition metal catalysts.[1] We have recently reported several examples of synthesis of heterocyclic derivatives by heteroannulation reactions by using inexpensive $CuCl_2$ as catalyst under ligand-free conditions.[2]

We have now found that $CuCl_2$ is also an excellent catalyst for realizing the 5-*endo-dig* heterocyclodehydration of readily available 3-yne-1,2-diols[3] and *N*-Boc- or *N*-tosyl-1-amino-3-yn-2-ols,[4] to produce substituted furans and pyrroles, respectively, in good to high yields (Eq. (1)).



(1)

(R^1 = H, alkyl; R^2 = H, alkynyl, aryl; R^3 = alkyl, aryl; Y = O, NR, R = Boc or Ts)

It is important to point out that the heterocyclodehydration of 3-yne-1,2-diols to give the corresponding furans was previously reported under Au,[5a,b] Ru,[5c] Ag,[5d,e] Mo,[5f,g] or Pd[5h,i] catalysis. In particular, mild and efficient reaction conditions have been recently developed under Au–Ag co-catalysis.[5a,b] To the best of our knowledge, however, no examples of copper-catalyzed formation of furans from 3-yne-1,2-diols have been reported so far in the literature. Also, the heterocyclodehydration of N-substituted 1-amino-3-yn-2-ols to give the corresponding pyrroles was previously reported to occur under palladium[5i] and gold catalysis.[5a,b] However, no general method for the conversion of N-substituted 1-amino-3-yn-2-ols into pyrroles in the presence of catalytic amounts of copper has so far appeared in the literature.[6–8]

We began our investigations with 3-yne-1,2-diols. When 2-methyl-4-phenylbut-3-yne-1,2-diol **1a** (R^1 = H, R^2 = Me, R^3 = Ph, Y = O) was let to react at 80 °C in MeOH for 1 h in the presence of 2 mol % of $CuCl_2$, we observed the formation of 4-methyl-2-phenylfuran **2a** in 37% GLC yield at 47% substrate conversion (Table 1, entry 1). Substrate conversion achieved 100% after 5 h, with a GLC yield of **2a** of 60% (55% isolated, Table 1, entry 2). The same reaction, carried out at 100 °C for 2 h, led to furan **2a** in 75% isolated yield (Table 1, entry 3). The reaction did not take place in aprotic solvents, such as 1,2-dimethoxyethane (DME), dioxane, or acetonitrile (Table 1, entries 4–6), or using CuI as the catalyst (Table 1, entry 7), while CuCl led to less satisfactory results (Table 1, entry 8).

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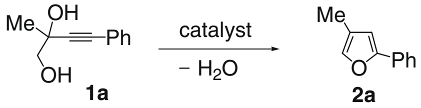
Table 1

Heterocyclodehydration reactions of 2-methyl-4-phenylbut-3-yne-1,2-diol **1a** under different conditions[a]



Entry	Catalyst	Solvent	*T* (°C)	Time (h)	Conversion of **1a**[b] (%)	Yield of **2a**[c] (%)
1	$CuCl_2$	MeOH	80	1	47	37
2	$CuCl_2$	MeOH	80	5	100	60 (55)
3	$CuCl_2$	MeOH	100	2	100	80 (75)
4	$CuCl_2$	DME	80	1	3	0
5	$CuCl_2$	Dioxane	80	1	1	0
6	$CuCl_2$	MeCN	80	1	3	0
7	CuI	MeOH	80	1	0	0
8	CuCl	MeOH	80	1	30	19

[a] All reactions were carried out using 0.2 mmol of **1a** per mL of solvent (1 mmol scale based on **1a**) in the presence of 2% of catalyst.

[b] Based on starting **1a**, by GLC.

[c] GLC yields (isolated yields), based on starting **1a**.

Having established the possibility to realize the heterocyclodehydration of **1a** in MeOH with $CuCl_2$ as catalyst, we then tested the reactivity of differently substituted 3-yne-1,2-diols, in order to assess the generality of the method. The reactivity of 2,4-diphenylbut-3-yne-1,2-diol **1b** was similar to that of **1a**, with the corresponding furan **2b** being formed in 53% isolated yield (Table 2, entry 1). On the other hand, 2-phenyloct-3-yne-1,2-diol **1c**, bearing an alkyl rather than a phenyl group at C-4, turned out to be more reactive, and the reaction could be carried out at 80 °C for 2 h, with an isolated yield of furan **2c** of 80% (Table 2, entry 2).

The reaction also worked nicely with substrates bearing an additional alkynyl group at C-2, as in the case of 3-hex-1-ynylnon-4-yne-2,3-diol **1d**, which was converted into the corresponding 5-butyl-3-hex-1-ynyl-2-methylfuran **2d** with an isolated yield as high as 91% working at 80 °C for 2 h (Table 2, entry 3).[9,10]

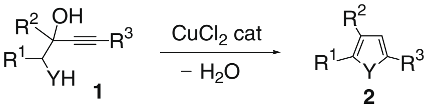
The reaction was then extended to *N*-Boc-1-amino-3-yn-2-ols, for the synthesis of substituted pyrroles. Under the same conditions already optimized for 3-yne-1,2-diols **1a–d** (2 mol % of $CuCl_2$, in MeOH as the solvent at 80–100 °C), *N*-Boc-2-amino-1-phenylnon-4-yn-3-ol **1e** ($Y = NBoc$, $R^1 = Bn$, $R^2 = H$, $R^3 = Bu$) turned out to be less reactive, as shown by the results reported in Table 2, entry 4 (to be compared with those reported in Table 2, entry 1). In any case, the formation of *N*-Boc-2-benzyl-5-butylpyrrole **2e** was indeed observed, thus confirming the possibility to obtain pyrroles by $CuCl_2$-catalyzed heterocyclodehydration of *N*-Boc-1-amino-3-yn-2-ols. In order to compensate for the lower reactivity of **1e** with respect to **1a–d**, we carried out the reaction with a lower substrate-to-catalyst ratio: with 5 mol % of $CuCl_2$ at 100 °C, substrate conversion reached 100% after 15 h, with an isolated yield of **2e** of 70% (Table 2, entry 5). *N*-Boc-2-aminononon-4-yn-3-ol **1f** ($Y = NBoc$, $R^1 = Me$, $R^2 = H$, $R^3 = Bu$) behaved similarly, as shown in Table 2, entries 6 and 7 (to be compared with entries 4 and 5, respectively). On the other hand, a substrate bearing an additional alkynyl group at C-2, such as *N*-Boc-7-(1-aminoethyl)trideca-5,8-diyn-7-ol **1g** ($Y = NBoc$, $R^1 = Me$, $R^2 = C{\equiv}CBu$, $R^3 = Bu$), was significantly more reactive, leading to the corresponding pyrrole **2g** in practically quantitative yield after only 1 h reaction time at 80 °C (Table 2, entry 8). *N*-Ts-1-amino-3-yn-2-ols could also be successfully used, as shown by the result obtained in the case of *N*-Ts-7-(1-aminoethyl)trideca-5,8-diyn-7-ol **1h** ($Y = Ts$, $R^1 = Me$, $R^2 = C{\equiv}CBu$, $R^3 = Bu$) (Table 2, entry 9).[9,10]

The plausible mechanism for the formation of heterocyclic derivatives **2** starting from substrates **1** is shown in Scheme 1. It involves the intramolecular 5-*endo-dig* nucleophilic attack of the –YH group to the triple bond coordinated to $CuCl_2$, followed by protonolysis and dehydration or vice versa.

In conclusion, we have developed a convenient, practical, and economical synthesis of substituted furans and pyrroles, by hetero-

Table 2

$CuCl_2$-catalyzed synthesis of substituted furans and pyrroles **2** by 5-*endo-dig* heterocyclodehydration of 3-yne-1,2-diols and *N*-Boc- or *N*-tosyl-1-amino-3-yn-2-ols **1**[a]

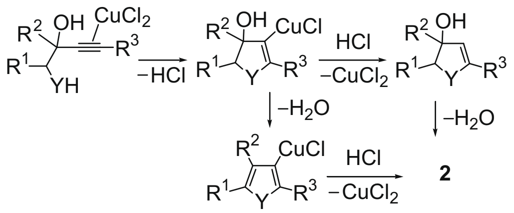


Entry	**1**	Y	R^1	R^2	R^3	Mol % of $CuCl_2$	*T* (°C)	Time (h)	Conversion of **1a**[b] (%)	**2**	Yield of **2**[c] (%)
1	**1b**	O	H	Ph	Ph	2	100	3	100	**2b**	53
2	**1c**	O	H	Ph	Bu	2	80	2	100	**2c**	80
3	**1d**	O	Me	$C{\equiv}CBu$	Bu	2	80	2	100	**2d**	91
4	**1e**	NBoc	Bn	H	Bu	2	100	24	94	**2e**	42
5	**1e**	NBoc	Bn	H	Bu	5	100	15	100	**2e**	70
6	**1f**	NBoc	Me	H	Bu	2	100	24	95	**2f**	56
7	**1f**	NBoc	Me	H	Bu	5	100	15	100	**2f**	56
8	**1g**	NBoc	Me	$C{\equiv}CBu$	Bu	2	80	1	100	**2g**	99
9	**1h**	NTs	Me	$C{\equiv}CBu$	Bu	2	80	8	100	**2h**	83

[a] All reactions were carried out in MeOH in the presence of $CuCl_2$, using 0.2 mmol of **1** per mL of solvent (1 mmol scale based on **1**).

[b] Based on starting **1**, by GLC.

[c] Isolated yield, based on starting **1**.



Scheme 1. Plausible reaction mechanism for the formation of substituted furans and pyrroles **2** by $CuCl_2$-catalyzed heterocyclization of 3-yne-1,2-diols and *N*-Boc- or *N*-tosyl-1-amino-3-yn-2-ols **1**.

cyclodehydration of readily available 3-yne-1,2-diols and N-substituted 1-amino-3-yn-2-ols, catalyzed by $CuCl_2$ under ligand-free conditions. The possibility to obtain furan and pyrrole derivatives starting from readily available substrates and employing a simple and inexpensive catalyst appears particularly attractive, also in view of the importance of these classes of heterocyclic compounds.[11,12]

Acknowledgments

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4. N-Substituted 1-amino-3-yn-2-ols **1e**–**h** were easily prepared by alkynylation, with an excess of $R^3C{\equiv}CMgBr$, of the appropriate *N*-Boc-α-amino aldehyde, *N*-Boc-α-amino ester, or *N*-tosyl-α-amino ester (*N*-Boc-2-amino-3-phenylpropionaldehyde in the case of **1e**; *N*-Boc-2-aminopropionaldehyde in the case of **1f**; methyl *N*-Boc-2-aminopropionate in the case of **1g**; methyl *N*-tosyl-2-aminopropionate in the case of **1h**).
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9. *Typical procedure for the* $CuCl_2$*-catalyzed heterocyclodehydration of 3-yne-1,2-diols* **1a**–**d** *and N-substituted 1-amino-3-yn-2-ols* **1e**–**g** *to the corresponding furans* **2a**–**d** *and pyrroles* **2e**–**g**: In a typical experiment, to a solution of **1** (1.0 mmol) in anhydrous MeOH (5.0 mL) was added $CuCl_2$ (2.7 mg, 2.0×10^{-2} mmol, or 6.8 mg, 5×10^{-2} mmol, see Tables 1 and 2) under nitrogen in a Schlenk flask. The resulting mixture was stirred under nitrogen at 80 °C or 100 °C for the required time (see Tables 1 and 2). The solvent was evaporated, and the crude products were purified by column chromatography on silica gel (eluent: 99:1 hexane–acetone for **2a**, **2b**, and **2c**; hexane–AcOEt from 9:1 to 8:2 for **2e**, **2f**, **2g**, and **2h**) or neutral alumina (for **2d**; eluent: 99:1 hexane–acetone) to give the pure products **2**, which were fully characterized by spectroscopic techniques and elemental analysis.[10] The yields obtained in each experiment are given in Tables 1 and 2.
10. *Characterization data for selected products*: For **2d**: Pale yellow oil. IR (film): ν = 2932 (m), 2862 (m), 2230 (w), 1580 (m), 1465 (m), 1232 (m), 1124 (w), 951 (w), 799 (w), 734 (w) cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$): δ = 5.87 (s, 1H, H-4), 2.51 (t, *J* = 7.7, 2H, $=CCH_2$), 2.37 (t, *J* = 6.9, 2H, $\equiv CCH_2$), 2.28 (s, 3H, Me at C-2), 1.63–1.24 (m, 8H, $2CH_2CH_2CH_3$), 0.93 (t, *J* = 7.4, 3H, CH_2CH_3), 0.91 (t, *J* = 7.4, 3H, CH_2CH_3); ^{13}C NMR (75 MHz, $CDCl_3$): δ = 154.0, 153.5, 107.6, 103.7, 92.0, 72.9, 31.1, 30.1, 27.5, 22.2, 22.0, 19.2, 13.8, 13.7, 12.5; GC–MS (EI, 70 eV): *m*/*z* = 218 (M^+, 28), 176 (14), 175 (100), 145 (4), 133 (11), 117 (4), 105 (5), 91 (8), 77 (6); Anal. Calcd for $C_{15}H_{22}O$ (218.33): C, 82.52; H, 10.16. Found: C, 82.45; H, 10.19. For **2g**: Pale yellow oil. IR (film): ν = 2968 (m), 2878 (m), 2229 (w), 1752 (s), 1548 (w), 1459 (w), 1338 (s), 1171 (m), 1108 (m), 856 (w) cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$): δ = 5.87 (t, *J* = 0.9, 1H, H-4), 2.77–2.69 (m, 2H, $=CCH_2$), 2.43 (s, 3H, Me at C-2), 2.39 (t, *J* = 7.0, 2H, $\equiv CCH_2$), 1.62–1.30 (m, 8H, $2CH_2CH_2CH_3$), 1.58 (s, 9H, *t*-Bu), 0.93 (t, *J* = 7.1, 3H, CH_2CH_3), 0.92 (t, *J* = 7.1, 3H, CH_2CH_3); ^{13}C NMR (75 MHz, $CDCl_3$): δ = 150.1, 135.3, 134.4, 111.8, 106.4, 91.6, 83.7, 74.9, 31.3, 29.0, 28.1, 22.5, 22.0, 19.3, 14.8, 14.0, 13.6; MS (ESI+): *m*/*z* = 340 [$(M+Na)^+$]; Anal. Calcd for $C_{20}H_{31}NO_2$ (317.47): C, 75.67; H, 9.84; N, 4.41. Found: C, 75.75; H, 9.81; N, 4.43.
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