Synthesis of 2-aminobutene-1,4-diones by gold-catalysed three-component coupling reactions

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A gold-catalysed three-component coupling reaction of phenylglyoxal derivatives, alkynes and secondary amines was developed to provide a novel one-pot synthesis of 2-aminobutene-1,4-diones in moderate to good yields.

Keywords: 2-aminobutene-1,4-dione, gold-catalysis, phenylglyoxal derivative, secondary amine, alkynes, three-component coupling reaction

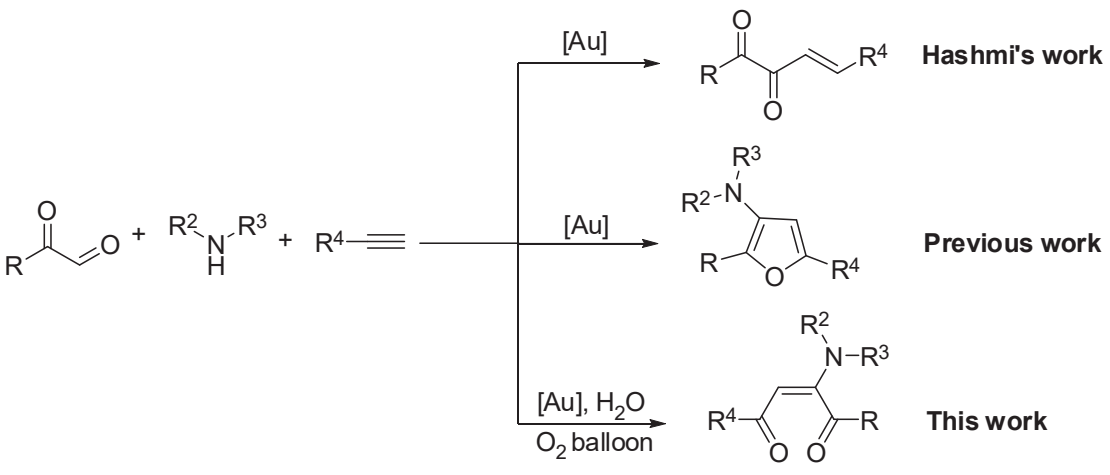
Compounds which have the 2-aminobutene-1,4-dione skeleton can be found in many drugs, bio-active compounds and industrial products.[1–4] Additionally, these 2-aminobutene-1,4-diones can also serve as useful tools for constructing heterocyclic compounds in organic synthesis containing, for example, pyrrole, hydrazine, furan and cyclopentenone moieties.[5–8] As a consequence, various methods for their preparation have been reported previously, for example, conjugate addition of diaroylacetylenes or sulfur ylides to aryl azides,[9,10] using α,β-unsaturated γ-dicarbonyl or acetophenones with amines,[11,12] reaction of pyridinium bromides and primary aromatic amines with phenylglyoxal or ethyl glyoxalate[13] and other ways.[14,15] Recently, Wu and co-workers developed an efficient Cu-catalysed tandem reaction to synthesise 2-aminobutene-1,4-diones in good yields, but a long reaction time is needed.[16] Here, we develop a simple and convenient method by a multi-component reaction process to access 2-aminobutene-1,4-diones rapidly.

Gold catalysis offers new ways for the efficient construction of complex molecules and has attracted much attention because of the catalyst's soft and carbophilic Lewis acidity in recent years.[17–20] Furthermore, Au-catalysed reactions generally proceed under very mild conditions with small amounts of catalyst. In 2013 we reported a new gold-catalysed three-component coupling (TCC) reaction of phenylglyoxal derivatives, secondary amines and alkynes under an N_2 atmosphere.[21] This method afforded the efficient synthesis of three-substituted furans in good yields and provided a powerful approach for the preparation of furan derivatives. Meanwhile, Hashmi and co-workers demonstrated a reaction to form 1,2-dicarbonyl-3-enes with the same substrates under AuCl catalysis.[22] Secondary amine mediated alkyne-to-allene isomerisation and hydrolysis of the enamine substructure during the work-up delivered the formal hydroacylation products. During our previous work, we found traces of 2-aminobutene-1,4-diones formed under aerobic conditions with gold catalysts, so we decided to study this product. Now we wish to introduce a gold-catalysed one-pot, three-component synthesis of 2-aminobutene-1,4-dione derivatives utilising the same substrates (phenylglyoxal derivatives, secondary amine and alkynes) in the presence of H_2O (Scheme 1).

Results and discussion

Initially, phenylglyoxal monohydrate (**1a**), morpholine (**2a**) and phenylacetylene (**3a**) were chosen as the model substrates to determine the catalytic activity of different metal catalysts under an O_2 atmosphere (Table 1). Among the various metal catalysts, $AuBr_3$ was found to be the most effective catalyst for this TCC transformation affording the product in 34% yield (Table 1, entry 5). This reaction did not work in the absence of any catalyst (Table 1, entry 1). CuI showed no catalytic activity to the reaction (Table 1, entry 2). We were happy to find that a yield of 82% of **4a** was formed using 5 mol% $AuBr_3$ in MeOH/H_2O (ratio of 10:1) at 50 °C after 5 h (Table 1, entry 7), while using H_2O only led to a lower yield (Table 1, entry 8). Various other solvents such as DMF, THF and toluene were also used for the reaction but none of them could produce a higher yield, even at higher temperature (Table 1, entries 9–12).

With the optimised reaction conditions in hand, the scope of this TCC reaction was explored. As shown in Table 2, the results revealed that all the desired products could be obtained in moderate to good yields. Alkyne substrates bearing electron-withdrawing groups or electron-donating groups were converted efficiently into 2-aminobutene-1,4-dione products



Scheme 1

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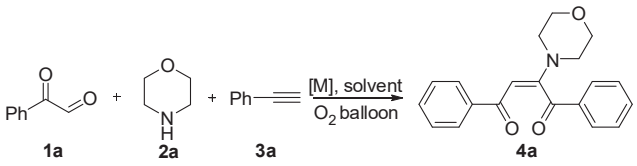
Table 1 Optimisation studies for the preparation of **4a**[a]

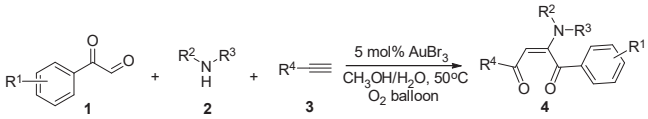


1a **2a** **3a** **4a**

Entry	Catalyst	T/°C	Solvent	Yield[b]/%
1	None	25	MeOH	0
2	CuI	25	MeOH	0
3	AuCl	25	MeOH	16
4	$AuCl_3$	25	MeOH	11
5	$AuBr_3$	25	MeOH	34
6	$AuBr_3$	50	MeOH	42
7	$AuBr_3$	50	MeOH/H_2O (10:1)	82
8	$AuBr_3$	50	H_2O	19
9	$AuBr_3$	50	DMF	16
10	$AuBr_3$	50	THF	31
11	$AuBr_3$	50	Toluene	34
12	$AuBr_3$	110	Toluene	25

[a]Standard reaction conditions: **1a** (1.0 mmol), **2a** (1.5 mmol), **3a** (2.0 mmol), catalyst (5 mol%), solvent (2.0 mL) were heated in a tube under O_2, 6 h.
[b]Isolated yield.

Table 2 Gold-catalysed three-component coupling reaction to form *E*-2-aminobutene-1,4-diones[a]



1 **2** **3** **4**

Product	R^1	Amine	R^4	Yield[b]/%
4a	**1a**; H	**2a**; morpholine	**3a**; C_6H_5	82
4b	**1a**	**2a**	**3b**; 4-FC_6H_4	85
4c	**1a**	**2a**	**3c**; 4-$CH_3C_6H_4$	67
4d	**1a**	**2a**	**3d**; 4-$C_5H_{11}C_6H_4$	71
4e	**1b**; 2-Cl	**2a**	**3a**	54
4f	**1c**; 4-Cl	**2a**	**3a**	58
4g	**1d**; 4-br	**2a**	**3a**	72
4h	**1a**	**2b**; piperidine	**3a**	76
4i	**1a**	**2b**	**3c**	73
4j	**1c**	**2b**	**3a**	51
4k	**1a**	**2c**; diethylamine	**3a**	62

[a]Reaction conditions: 1 (0.20 mmol), 2a (0.25 mmol), $AuBr_3$ (0.02 mmol), MeOH/H_2O (2.0 mL), at 50 °C under O_2, 6h.
[b]Isolated yield.

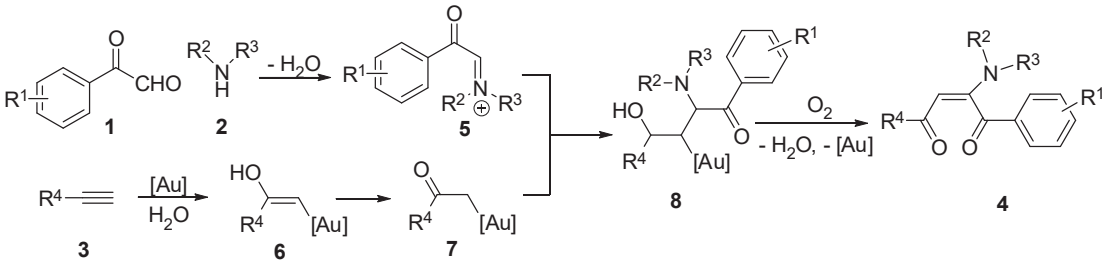


Fig. 1 Proposed reaction mechanism and possible explanation of the observed stereochemistry.

with good yields (products **4b**–**d** and **4i**). A long chain alkyl-substituted aryl alkyne such as 1-ethynyl-4-pentylbenzene could also be smoothly transformed into the desired product in 71% yield (**4d**). Reaction of different phenylglyoxal derivatives was then examined. Phenylglyoxal derivatives with electron-withdrawing groups at the *para*- or *ortho*-position of the aryl ring all delivered the desired products in moderate yields (products **4e**–**g** and **4j**). Finally, with respect to amines, those such as morpholine or piperidine reacted smoothly under these mild reaction conditions. It is noteworthy that an acyclic amine, diethylamine, was also suitable for the reaction to give **4k** in 62% yield. However, aromatic amines such as diphenylamine could not be used for this reaction.

The structures of the prepared diones **4a**–**k** were fully characterised by ^{1}H NMR, ^{13}C NMR and HRMS. Results were in agreement with the proposed structures. The characteristic NMR ($CDCl_3$) features for 2-aminobut-2-ene-1,4-dione H-19/H-20 signals (6.08–6.15 ppm) and C-19/C-20 signals (91.73–93.70 ppm), demonstrated that the molecules adopted an *E*-configuration.

A plausible mechanism is proposed in Figure 1. Condensation of the phenylglyoxal with the amine initially affords the corresponding imine **5**. The alkyne was activated by the gold catalyst and underwent nucleophilic attack by H_2O to generate intermediate **6**.[23–25] Subsequent nucleophilic attack of **7** on **5** afforded the 1,4-enedione **8**, which could form the product **4** by gold-catalysed aerobic oxidative dehydrogenation.[26–28]

Experimental

All experiments were conducted under an O_2 atmosphere. All solvents were commercially available. For column chromatography, 200–300 mesh silica gel was employed. ^{1}H NMR and ^{13}C NMR were recorded on Bruker Advance 300, 400 or 500 MHz spectrometers in $CDCl_3$ solution and the chemical shifts were reported in parts per million (δ) relative to internal standard TMS (0 ppm). HRMS was performed on a TOF Agilent 6540 instrument. Melting points were determined with a WRS-1B apparatus and were uncorrected. Unless otherwise noted, materials obtained from commercial suppliers were used without further purification. Compounds **4a**, **4h** and **4k** are reported in the literature.[16] Others are new compounds.

General procedure

A solution of phenylglyoxal derivative (1.0 mmol), amine (1.5 mmol), $AuBr_3$ (8.7 mg, 0.02 mmol) and alkyne (2.0 mmol) in solvent (MeOH/H_2O = 10/1, 2.0 mL) was heated to 50 °C under O_2 for 6 h. After completion of the reaction (observed on TLC), the solvent was evaporated under reduced pressure to obtain the crude mixture. The residues were purified by silica gel column chromatography [ethyl acetate/ petroleum ether (b.p. 60–90 °C) = 1/10–1/4] to afford the pure product.

(E)-2-Morpholino-1,4-diphenylbut-2-ene-1,4-dione (**4a**): Light yellow solid; m.p. 169–171 °C (lit.[16] 171–172 °C); ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.02 (d, *J* = 7.5 Hz, 2H), 7.84 (d, *J* = 7.4 Hz, 2H), 7.57–7.55 (m, 1H), 7.49–7.44 (m, 3H), 7.36 (t, *J* = 7.8 Hz, 2H), 6.15 (s, 1H), 3.74 (br s, 4H), 3.42 (br s, 2H), 3.32 (br s, 2H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 193.9, 187.2, 160.9, 138.8, 135.7, 133.6, 131.7, 129.0, 128.2, 128.1, 127.7, 93.5, 66.2, 47.6; MS (EI) *m*/*z*: 322.14.

(E)-4-(4-Fluorophenyl)-2-morpholino-1-phenylbut-2-ene-1,4-dione (**4b**): Light yellow solid; m.p. 178–180 °C, ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.02 (d, *J* = 7.1 Hz, 2H), 7.85 (dd, *J* = 5.5, 8.8 Hz, 2H), 7.57–7.55 (m, 1H), 7.51–7.47 (m, 2H), 7.04 (t, *J* = 8.6 Hz, 2H), 6.09 (s, 1H), 3.74 (br s, 4H), 3.42 (br s, 2H), 3.32 (br s, 2H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 193.8, 185.7, 163.3*, 161.2, 135.6, 133.7, 130.1 (d, J_{F-C} = 8.9 Hz), 129.1, 128.1, 115.2 (d, J_{F-C} = 21.6 Hz), 92.9, 66.2, 47.6; HRMS (ESI) calcd for $C_{20}H_{19}FNO_3$: 340.1349; found: 340.1348.

*It is thought that this is part of the doublet for the C bonded to F, the other part of which is overlapped.

*(E)-2-Morpholino-1-phenyl-4-*p*-tolylbut-2-ene-1,4-dione* (**4c**): Light yellow solid; m.p. 176–178 °C. ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.03 (d, *J* = 7.8 Hz, 2H), 7.74 (d, *J* = 7.9 Hz, 2H), 7.55 (d, *J* = 7.2 Hz, 1H), 7.47 (t, *J* = 7.6 Hz, 2H), 7.16 (d, *J* = 7.9 Hz, 2H), 6.15 (s, 1H), 3.73 (br s, 4H), 3.40 (br s, 2H), 3.30 (br s, 2H), 2.36 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 186.9, 160.8, 142.3, 133.5, 129.0, 128.9, 128.1, 127.8, 93.5, 66.2, 47.6, 21.6; HRMS (ESI) calcd for $C_{21}H_{22}NO_3$: $[M+H]^+$: 336.1599; found: 336.1601.

(E)-2-Morpholino-4-(4-pentylphenyl)-1-phenylbut-2-ene-1,4-dione (**4d**): Light yellow solid; m.p. 185–187 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.03 (d, *J* = 7.5 Hz, 2H), 7.76 (d, *J* = 8.0 Hz, 2H), 7.56 (t, *J* = 7.3 Hz 1H), 7.48–7.45 (m, 2H), 7.17 (d, *J* = 8.0 Hz, 2H), 6.15 (s, 1H), 3.74 (br s, 4H), 3.41 (br s, 2H), 3.31 (br s, 2H), 2.60 (t, *J* = 7.6 Hz, 2H), 1.59 (m, 2H), 1.30 (s, 4H), 0.87 (t, *J* = 6.5 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 194.0, 186.9, 160.7, 147.2, 136.4, 135.8, 133.5, 128.9, 128.3, 128.1, 127.8, 93.7, 66.2, 47.6, 35.8, 31.4, 30.8, 22.5, 13.9; HRMS (ESI) calcd for $C_{25}H_{30}NO_3$: $[M+H]^+$: 392.2225; found: 392.2230.

(E)-1-(3-Chlorophenyl)-2-morpholino-4-phenylbut-2-ene-1,4-dione (**4e**): Light yellow solid; m.p. 177–179 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.00 (t, *J* = 1.7 Hz, 1H), 7.90–7.88 (m, 1H), 7.84–7.83 (m, 2H), 7.55–7.52 (m, 1H), 7.47–7.35 (m, 4H), 6.16 (s, 1H), 3.76 (br s, 4H), 3.43 (br s, 2H), 3.32 (br s, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 187.4, 160.4, 138.5, 137.4, 135.3, 133.5, 131.9, 130.4, 128.3, 127.8, 126.1, 93.8, 66.1, 47.7; HRMS (ESI) calcd for $C_{20}H_{19}ClNO_3$: $[M+H]^+$: 356.1053; found: 356.1059.

(E)-1-(4-Chlorophenyl)-2-morpholino-4-phenylbut-2-ene-1,4-dione (**4f**): Light yellow solid; m.p. 176–178 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.89 (d, *J* = 8.5 Hz, 2H), 7.83 (d, *J* = 7.4 Hz, 2H), 7.62 (d, *J* = 8.5 Hz, 2H), 7.47–7.44 (m, 1H), 7.37 (t, *J* = 7.4 Hz, 2H), 6.15 (s, 1H), 3.75 (br s, 4H), 3.41 (br s, 2H), 3.30 (br s, 2H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 192.9, 187.3, 160.4, 138.5, 134.5, 132.4, 131.9, 129.5, 128.8, 128.3, 127.8, 93.7, 66.2, 47.6; HRMS (ESI) calcd for $C_{20}H_{19}ClNO_3$: $[M+H]^+$: 356.1053; found: 356.1055.

(E)-1-(4-Bromophenyl)-2-morpholino-4-phenylbut-2-ene-1,4-dione (**4g**): Light yellow solid; m.p. 177–179 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.89 (d, *J* = 8.3 Hz, 2H), 7.82 (d, *J* = 7.6 Hz, 2H), 7.61 (d, *J* = 8.3 Hz, 2H), 7.46–7.44 (m, 1H), 7.37 (t, *J* = 7.5 Hz, 2H), 6.15 (s, 1H), 3.75 (br s, 4H), 3.40 (br s, 2H), 3.30 (br s, 2H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 192.9, 187.3, 160.5, 138.5, 134.6, 132.4, 131.8, 129.5, 128.8, 127.7, 93.7, 66.2, 47.6; HRMS (ESI) calcd for $C_{20}H_{19}BrNO_3$: $[M+H]^+$: 400.0548; found: 400.0542.

(E)-1,4-Diphenyl-2-(piperidin-1-yl)but-2-ene-1,4-dione (**4h**): Light yellow solid; m.p. 166–169 °C (lit.[16] 169–171 °C); ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.03 (dd, *J* = 8.5 Hz, 1.5 Hz, 2H), 7.84 (dd, *J* = 8.4 Hz, 1.5 Hz, 2H), 7.57–7.52 (m, 1H), 7.48–7.46 (m, 2H), 7.43–7.39 (m, 1H), 7.37–7.32 (m, 2H), 6.11 (s, 1H), 3.37 (br s, 4H), 1.66 (br s, 6H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 194.3, 186.8, 161.0, 139.3, 135.9, 133.3, 131.2, 128.9, 128.1, 127.7, 92.0, 49.1, 25.7, 23.9; MS (EI) *m/z*: 320.16.

*(E)-1-Phenyl-2-(piperdin-1-yl)-4-*p*-tolylbut-2-ene-1,4-dione* (**4i**): Light yellow solid; m.p. 173–175 °C; ^{1}H NMR (300 MHz, $CDCl_3$): δ 8.03 (d, *J* = 7.3 Hz, 2H), 7.74 (d, *J* = 8.0 Hz, 2H), 7.55–7.52 (m, 1H), 7.45 (t, *J* = 7.6 Hz, 2H), 7.15 (d, *J* = 7.9 Hz, 2H), 6.09 (s, 1H), 3.40–2.25 (br m, 4H), 2.35 (s, 3H), 1.7–1.55 (br s, 6H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 194.3, 186.6, 160.8, 141.7, 136.6, 136.0, 133.2, 131.6, 129.0, 128.8, 128.3, 127.8, 126.8, 92.1, 25.6, 23.9, 21.5; HRMS (ESI) calcd for $C_{22}H_{24}NO_2$: $[M+H]^+$: 334.1807; found: 334.1810.

(E)-1-(4-Chlorophenyl)-4-phenyl-2-(piperidin-1-yl)but-2-ene-1,4-dione (**4j**): Light yellow solid; m.p. 177–179 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.98 (d, *J* = 8.2 Hz, 2H), 7.84 (d, *J* = 7.6 Hz, 2H), 7.45–7.42 (m, 2H), 7.38–7.34 (m, 3H), 6.10 (s, 1H), 3.4–3.2 (br m, 4H), 1.7–1.55 (br m, 6H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 193.0, 187.0, 160.5, 139.6, 139.1, 134.4, 131.4, 129.4, 129.3, 128.2, 127.7, 92.2, 49.4, 23.9; HRMS (ESI) calcd for $C_{21}H_{21}ClNO_2$: $[M+H]^+$: 354.1261; found: 354.1263.

(E)-2-(Diethylamino)-1,4-diphenylbut-2-ene-1,4-dione (**4k**): Light yellow solid; m.p. 156–159 °C (lit[16] 159–162 °C); ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.01 (d, *J* = 7.2 Hz, 2H), 7.83 (d, *J* = 7.0 Hz, 2H), 7.54–7.52 (m, 1H), 7.47–7.42 (m, 3H), 7.38–7.36 (m, 2H), 6.00 (s, 1H), 3.47 (br s, 4H), 1.41 (br s, 3H), 1.10 (br s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 193.6, 186.5, 160.6, 139.4, 135.9, 133.2, 131.1, 128.9, 128.1, 128.0, 127.6, 91.6, 46.2, 44.3, 14.3; MS (EI) *m/z*: 308.16.

Electronic Supplementary Information

The ESI (NMR spectra of **4a**–**k**) is available through: stl.publisher.ingentaconnect.com/content/stl/jcr/supp-data

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