

Reaction between 1,*n*-Diamines, Diketene, and Dibenzoylacetylene in the Presence of Triphenylphosphine: One-Pot, Pseudo-Five-Component Synthesis of Bisfuramides

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Abstract: Using diketene as a basic reagent, a one-pot, pseudo-five-component reaction for the synthesis of highly substituted bisfuramides is described. In this method, ring opening of diketene occurs in mild condition and without a catalyst. Thus, the reaction between diketene, 1,*n*-diamine, and dibenzoylacetylene, in the presence of triphenylphosphine, produces bisfuramides.

Keywords: diketene, dibenzoylacetylene, 1,*n*-diamine, triphenylphosphine, bisfuramide

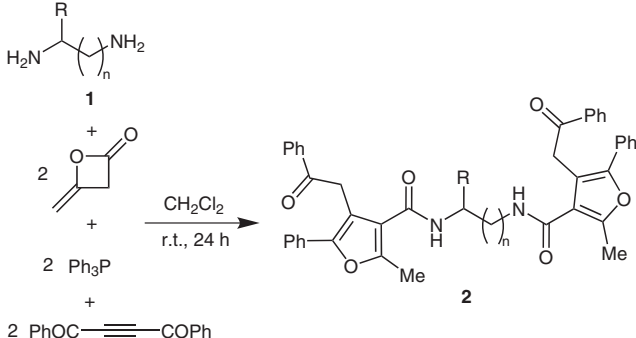
Furan derivatives, obtained from both synthetic and natural sources, have been attracting much interest due to the wide range of pharmaceutical applications.¹ They are frequently found in natural products, such as the kallolides^{2a} and combranolides^{2b} and in important pharmaceuticals as well as in flavoring and fragrance compounds. They are often used as building blocks in organic synthesis.³ On the other hand, a general way to improve synthetic efficiency and also to address other criteria is the development of multicomponent reactions.⁴ Multicomponent reactions (MCRs), by virtue of their convergence, productivity, facile execution, and generally high yields of products, have attracted much attention from the point of combinatorial chemistry.^{4–8}

We recently reported a new class of diketene-based multicomponent reactions mediated by zwitterionic intermediates,⁹ and ring opening of the diketene cycle.¹⁰ In previous studies we were interested in developing diketene-based one-pot, multicomponent reactions. These researches led to the discovery of the reaction between amines, dibenzoylacetylenes, and diketene. In our recent investigations, treatment of amines with diketene and dibenzoylacetylene in the presence of triphenylphosphine at 25 °C led to the formation of the furamide derivatives.^{10a} Although the mechanism of the reaction between triphenylphosphine and dibenzoylacetylene in the presence of *N*-alkyl-3-oxobutanamide, derived from the addition of an amine to diketene, has not yet been established in an experimental manner, a possible explanation was proposed.^{10a}

In the present study we have incorporated 1,*n*-diamines under similar conditions. To this end, as a preliminary

step in diketene-based multicomponent reaction, we could synthesize 2-methyl-*N*³-[*n*-({[2-methyl-4-(2-oxo-2-phenylethyl)-5-phenyl-3-furyl]carbonyl}amino)alkyl]-4-(2-oxo-2-phenylethyl)-5-phenyl-3-furamides **2** in good yields. Thus, the reaction of diketene with 1,*n*-diamine **1**, dibenzoylacetylene (DBA) in the presence of triphenylphosphine proceeded spontaneously at 25 °C in anhydrous CH₂Cl₂ and furnished furamides **2** within 24 hours in 82–90% yields (Table 1).

Table 1 Bisfuramides **2** Prepared



Entry	Compound	R	n	Yield (%) of 2
1	2a	H	1	90
2	2b	Me	1	88
3	2c	H	3	84
4	2d	H	5	85
5	2e	H	7	82

The structures of compounds **2a–e** were deduced from their elemental analyses, IR, and high-field ¹H and ¹³C NMR spectra. The mass spectrum of **2a** displayed the molecular ion (M⁺) peak at *m/z* 664, which is consistent with the proposed structure. The IR spectrum of **2a** exhibited absorption bands due to the carbonyl groups of the ketone and the amide at 1681 and 1638 cm^{–1} respectively, and the NH group at 3260 cm^{–1}.

The ¹H NMR spectrum of **2a**, based on the symmetrical structure, exhibited three sharp lines readily recognized as arising from methyl group (δ = 2.56), the CH₂NH (δ = 3.43) and the CH₂COPh (δ = 4.51) protons. A broad signal of the NH group arises at δ = 7.16. The phenyl moi-

ety gave rise to characteristic signals in the aromatic region of the spectrum. The ^1H decoupled ^{13}C NMR spectrum of **2a** showed 17 distinct resonances in agreement with the structure. The ^1H and ^{13}C NMR spectra of compounds **2b–e** are similar to those of **2a**, except for the alkyl chain, which exhibit characteristic signals with appropriate chemical shifts.

In conclusion, the present one-pot multicomponent method carries the advantages that the ring opening of diketene is performed under neutral conditions and that the total reaction afforded good yields. The simplicity of the present procedure makes it an interesting alternative to complex multi-step approaches.

Diamines and diketene were obtained from Merck (Germany) and Fluka (Switzerland) and were used without further purification. Dibenzoylacetylene was prepared according to the literature procedure.¹¹ Melting points were measured on an Electrothermal 9100 apparatus. Elemental analyses for C, H, and N were performed using a Heraeus CHN-O-Rapid analyzer. Mass spectra were recorded on a Finnigan MATT 8430 mass spectrometer operating at an ionization potential of 70 eV. ^1H and ^{13}C NMR spectra were measured (CDCl_3) with a Bruker DRX-500 Avance spectrometer at 500.1 and 125.8 MHz, respectively. IR spectra were recorded on a Shimadzu IR-460 spectrometer. Chromatography columns were prepared from Merck silica gel 230–240 mesh.

2-Methyl-*N*³-[2-([2-methyl-4-(2-oxo-2-phenylethyl)-5-phenyl-3-furyl]carbonyl)amino]ethyl]-4-(2-oxo-2-phenylethyl)-5-phenyl-3-furamide (2a): Typical Procedure

A solution of 1,2-diamine (0.60 g, 1 mmol) and diketene (0.16 g, 2 mmol) in anhyd CH_2Cl_2 (5 mL) was magnetically stirred for 5 h and then Ph_3P (0.52 g 2 mmol), and a solution of dibenzoylacetylene (0.46 g, 2 mmol) in anhyd CH_2Cl_2 (3 mL) was added dropwise at 25 °C over 15 min. The mixture was then allowed to stir for 24 h. The solvent was removed under reduced pressure, and the residue was purified by silica gel column chromatography using a mixture of hexane–EtOAc (5:1) as eluent. The bisfuramides were then recrystallized from EtOH or EtOAc; yield: 0.59 g (90%); white powder; mp 170–172 °C.

IR (KBr): 3260 (NH), 1681 (C=O), 1638 (CONH), 1539 and 1486 cm^{-1} (Ph).

^1H NMR (500.13 MHz, CDCl_3): δ = 2.56 (6 H, s, 2 CH_3), 3.43 (4 H, d, $^3J_{\text{H,H}}$ = 4.5 Hz, 2 CH_2NH), 4.51 (4 H, s, 2 CH_2COPh), 7.16 (2 H, br, 2 NH), 7.30 (2 H, t, $^3J_{\text{H,H}}$ = 7.4 Hz, 2 CH_{para} of 2 Ph), 7.34 (4 H, t, $^3J_{\text{H,H}}$ = 7.2 Hz, 4 CH_{meta} of 2 Ph), 7.41 (4 H, d, $^3J_{\text{H,H}}$ = 8.2 Hz, 4 CH_{ortho} of 2 Ph), 7.44 (4 H, t, $^3J_{\text{H,H}}$ = 7.8 Hz, 4 CH_{meta} of 2 Ph), 7.56 (2 H, t, $^3J_{\text{H,H}}$ = 7.5 Hz, 2 CH_{para} of 2 Ph), 7.97 (4 H, d, $^3J_{\text{H,H}}$ = 8.2 Hz, 4 CH_{ortho} of 2 Ph).

^{13}C NMR (125.7 MHz, CDCl_3): δ = 13.46 (2 CH_3), 34.83 (2 CH_2COPh), 39.28 (2 CH_2NH), 113.57 (2 C-3 of furan), 120.0 (2 C-4 of furan), 126.66 (4 CH of 2 Ph), 127.86 (2 CH of 2 Ph), 128.29 (4 CH of 2 Ph), 128.64 (4 CH of 2 Ph), 128.72 (4 CH of 2 Ph), 130.42 (2 C_{ipso} -furyl), 133.49 (2 CH of 2 Ph), 136.66 (2 C_{ipso} -COCH₂ of 2 Ph), 149.0 (2 C-5 of furan), 152.08 (2 C-2 of furan), 165.72 (2 CONH), 199.14 (2 COPh).

MS: m/z (%) = 664 (M^+ , 2), 646 (3), 302 (100), 284 (36), 260 (27), 202 (37), 105 (68), 77 (78), 43 (50).

Anal. Calcd for $\text{C}_{42}\text{H}_{36}\text{N}_2\text{O}_6$ (664.75): C, 75.89; H, 5.46; N, 4.21. Found: C, 76.00; H, 5.60; N, 4.10.

2-Methyl-*N*³-[1-methyl-2-([2-methyl-4-(2-oxo-2-phenylethyl)-5-phenyl-3-furyl]carbonyl)amino]ethyl]-4-(2-oxo-2-phenylethyl)-5-phenyl-3-furamide (2b)

Yield: 0.59 g (88%); white powder; mp 165–167 °C.

IR (KBr): 3320 (NH), 1690 (C=O), 1673 (CONH), 1590 and 1518 cm^{-1} (Ph).

^1H NMR (500.13 MHz, CDCl_3): δ = 1.15 (3 H, d, $^3J_{\text{H,H}}$ = 6.6 Hz, CH_3CHNH), 2.49 (3 H, s, CH_3), 2.51 (3 H, s, CH_3), 3.33–3.39 (1 H, m, CH_2NH), 3.49–3.52 (1 H, m, CH_2NH), 4.16–4.21 (1 H, m, CH_3CHNH), 4.39–4.51 (4 H, m, 2 CH_2COPh), 6.90 (1 H, d, $^3J_{\text{H,H}}$ = 7.5 Hz, NH), 7.20 (1 H, br, NH), 7.22–7.65 and 7.95–8.11 (20 H of 4 Ph).

^{13}C NMR (125.7 MHz, CDCl_3): δ = 13.46 and 13.55 (2 CH_3), 14.18 (CH_3CHNH), 29.68 and 34.96 (2 CH_2COPh), 45.01 (CH_2NH), 46.0 (CH_3CHNH), 113.12 and 113.34 (2 C-3 of furan), 119.98 and 120.0 (2 C-4 of furan), 126.56 (2 CH of Ph), 126.59 (2 CH of Ph), 127.83 (CH of Ph), 127.87 (CH of Ph), 128.42 (2 CH of Ph), 128.45 (2 CH of Ph), 128.62 (4 CH of 2 Ph), 128.71 (4 CH of 2 Ph), 130.12 and 130.36 (2 C_{ipso} -furyl), 133.43 (CH of Ph), 133.54 (CH of Ph), 136.54 (2 C_{ipso} -COCH₂ of 2 Ph), 149.46 (2 C-5 of furan), 153 and 153.20 (2 C-2 of furan), 164.88 and 165.42 (2 CONH), 199.35 (2 COPh).

MS: m/z (%) = 678 (M^+ , 2), 277 (100), 199 (21), 183 (22), 152 (16), 105 (21), 77 (57), 51 (47).

Anal. Calcd for $\text{C}_{43}\text{H}_{38}\text{N}_2\text{O}_6$ (678.78): C, 76.09; H, 5.64; N, 4.13. Found: C, 76.50; H, 5.70; N, 4.10.

2-Methyl-*N*³-[4-([2-methyl-4-(2-oxo-2-phenylethyl)-5-phenyl-3-furyl]carbonyl)amino]butyl]-4-(2-oxo-2-phenylethyl)-5-phenyl-3-furamide (2c)

Yield: 0.58 g (84%); yellow oil.

IR (KBr): 3320 (NH), 1663 (C=O), 1593 (CONH), 1537 and 1481 cm^{-1} (Ph).

^1H NMR (500.13 MHz, CDCl_3): δ = 1.16–1.24 (4 H, m, 2 CH_2), 2.00 (3 H, s, CH_3), 2.30 (3 H, s, CH_3), 2.95–2.99 (2 H, m, CH_2NH), 3.04–3.08 (2 H, m, CH_2NH), 3.13 (2 H, s, CH_3COPh), 4.18 (2 H, s, CH_2COPh), 6.33 (1 H, br, NH), 6.73 (1 H, br, NH), 7.04 (2 H, t, $^3J_{\text{H,H}}$ = 7.1 Hz, 2 CH_{meta} of Ph), 7.07 (2 H, t, $^3J_{\text{H,H}}$ = 7.4 Hz, 2 CH_{meta} of Ph), 7.12 (4 H, d, $^3J_{\text{H,H}}$ = 7.2 Hz, 4 CH_{ortho} of 2 Ph), 7.21 (2 H, t, $^3J_{\text{H,H}}$ = 7.7 Hz, 2 CH_{meta} of Ph), 7.25 (2 H, t, $^3J_{\text{H,H}}$ = 7.5 Hz, 2 CH_{meta} of Ph), 7.33 (2 H, t, $^3J_{\text{H,H}}$ = 7.5 Hz, 2 CH_{para} of Ph), 7.38 (2 H, t, $^3J_{\text{H,H}}$ = 7.1 Hz, 2 CH_{para} of 2 Ph), 7.68 (2 H, d, $^3J_{\text{H,H}}$ = 7.8 Hz, 2 CH_{ortho} of Ph), 7.80 (2 H, d, $^3J_{\text{H,H}}$ = 7.7 Hz, 2 CH_{ortho} of Ph).

^{13}C NMR (125.7 MHz, CDCl_3): δ = 13.34 (2 CH_3), 26.12 and 29.44 (2 CH_3), 30.64 and 34.23 (2 CH_2CO), 39.13 and 39.54 (2 CH_2NH), 112.34 (2 C-3 of furan), 119.55 (2 C-4 of furan), 125.34 (4 CH of 2 Ph), 126.63 (2 CH of 2 Ph), 127.54 (4 CH of 2 Ph), 129.48 (4 CH of 2 Ph), 129.73 (4 CH of 2 Ph), 129.34 (2 C_{ipso} -furyl), 132.66 (2 CH of 2 Ph), 135.94 (2 C_{ipso} -COCH₂ of 2 Ph), 148.72 (2 C-5 of furan), 153.24 (2 C-2 of furan), 164.78 and 165.36 (2 CONH), 198.23 and 203.25 (2 COPh).

MS: m/z (%) = 692 (M^+ , 2), 302 (24), 301 (100), 271 (6), 207 (17), 193 (8), 77 (4), 43 (15).

Anal. Calcd for $\text{C}_{44}\text{H}_{40}\text{N}_2\text{O}_6$ (692.81): C, 76.28; H, 5.82; N, 4.04. Found: C, 76.50; H, 5.90; N, 3.95.

2-Methyl-*N*³-[6-([2-methyl-4-(2-oxo-2-phenylethyl)-5-phenyl-3-furyl]carbonyl)amino]hexyl]-4-(2-oxo-2-phenylethyl)-5-phenyl-3-furamide (2d)

Yield: 0.61 g (85%); yellow oil.

IR (KBr): 3310 (NH), 1693 (C=O), 1645 (CONH), 1537 and 1481 cm^{-1} (Ph).

^1H NMR (500.13 MHz, CDCl_3): δ = 1.16–1.29 and 1.41–1.53 (8 H, m, 4 CH_2), 2.24 (3 H, s, CH_3), 2.55 (3 H, s, CH_3), 3.17–3.24 (2 H, m, CH_2NH), 3.29–3.33 (2 H, m, CH_2NH), 3.38 (2 H, s, CH_2COPh), 4.43 (2 H, s, CH_2COPh), 6.58 (1 H, br, NH), 6.98 (1 H, br, NH), 7.28 (2 H, t, $^3J_{\text{H,H}}$ = 7.1 Hz, 2 CH_{meta} of Ph), 7.32 (2 H, t, $^3J_{\text{H,H}}$ = 7.6 Hz, 2 CH_{meta} of Ph), 7.37 (4 H, d, $^3J_{\text{H,H}}$ = 7.5 Hz, 4 CH_{ortho} of 2 Ph), 7.46 (2 H, t, $^3J_{\text{H,H}}$ = 7.2 Hz, 2 CH_{meta} of Ph), 7.51 (2 H, t, $^3J_{\text{H,H}}$ = 7.5 Hz, 2 CH_{meta} of Ph), 7.58 (2 H, t, $^3J_{\text{H,H}}$ = 8.6 Hz, 2 CH_{para} of Ph), 7.63 (2 H, t, $^3J_{\text{H,H}}$ = 7.3 Hz, 2 CH_{para} of 2 Ph), 7.93 (2 H, d, $^3J_{\text{H,H}}$ = 7.8 Hz, 2 CH_{ortho} of Ph), 8.05 (2 H, d, $^3J_{\text{H,H}}$ = 7.8 Hz, 2 CH_{ortho} of Ph).

^{13}C NMR (125.7 MHz, CDCl_3): δ = 13.38 (2 CH_3), 26.25, 26.3, 29.06 and 29.37 (4 CH_2), 30.93 and 35.13 (2 CH_2CO), 39.16 and 39.24 (2 CH_2NH), 112.78 (2 C-3 of furan), 120.55 (2 C-4 of furan), 126.49 (4 CH of 2 Ph), 127.89 (2 CH of 2 Ph), 128.47 (4 CH of 2 Ph), 128.63 (4 CH of 2 Ph), 128.82 (4 CH of 2 Ph), 130.29 (2 C_{ipso} -furyl), 133.76 (2 CH of 2 Ph), 136.34 (2 C_{ipso} - COCH_2 of 2 Ph), 149.62 (2 C-5 of furan), 152.70 (2 C-2 of furan), 164.80 and 165.42 (2 CONH), 199.32 and 204.65 (2 COPh).

MS: m/z (%) = 720 (M^+ , 2), 302 (14), 277 (66), 199 (13), 183 (15), 152 (11), 122 (8), 105 (100), 77 (81), 57 (33), 44 (47).

Anal. Calcd for $\text{C}_{46}\text{H}_{44}\text{N}_2\text{O}_6$ (720.86): C, 76.65; H, 6.15; N, 3.89. Found: C, 77.00; H, 6.30; N, 3.70.

2-Methyl-N³-[8-([2-methyl-4-(2-oxo-2-phenylethyl)-5-phenyl-3-furyl]carbonyl)amino]octyl]-4-(2-oxo-2-phenylethyl)-5-phenyl-3-furamide (2e)

Yield: 0.61 g (82%); yellow oil.

IR (KBr): 3315 (NH), 1693 (C=O), 1657 (CONH), 1539 and 1439 cm^{-1} (Ph).

^1H NMR (500.13 MHz, CDCl_3): δ = 1.15–1.32 and 1.40–1.56 (12 H, m, 6 CH_2), 2.25 (3 H, s, CH_3), 2.55 (3 H, s, CH_3), 3.21–3.25 (2 H, m, CH_2NH), 3.28–3.32 (2 H, m, CH_2NH), 3.39 (2 H, s, CH_2COPh), 4.43 (2 H, s, CH_2COPh), 6.54 (1 H, br, NH), 6.96 (1 H, br, NH), 7.28 (2 H, t, $^3J_{\text{H,H}}$ = 7.1 Hz, 2 CH_{meta} of Ph), 7.32 (2 H, t, $^3J_{\text{H,H}}$ = 7.7 Hz, 2 CH_{meta} of Ph), 7.37 (4 H, d, $^3J_{\text{H,H}}$ = 7.1 Hz, 4 CH_{ortho} of 2 Ph), 7.38 (2 H, t, $^3J_{\text{H,H}}$ = 8.7 Hz, 2 CH_{meta} of Ph), 7.46 (2 H, t, $^3J_{\text{H,H}}$ = 7.8 Hz, 2 CH_{meta} of Ph), 7.51 (2 H, t, $^3J_{\text{H,H}}$ = 7.7 Hz, 2 CH_{para} of 2 Ph), 7.63 (2 H, t, $^3J_{\text{H,H}}$ = 7.5 Hz, 2 CH_{para} of 2 Ph), 7.93 (2 H, d, $^3J_{\text{H,H}}$ = 7.4 Hz, 2 CH_{ortho} of Ph), 8.05 (2 H, d, $^3J_{\text{H,H}}$ = 7.3 Hz, 2 CH_{ortho} of Ph).

^{13}C NMR (125.7 MHz, CDCl_3): δ = 13.39 (2 CH_3), 26.68, 26.77, 28.95, 29.00, 29.28 and 29.44 (6 CH_2), 31.00 and 35.19 (2 CH_2CO), 39.47 and 39.52 (2 CH_2NH), 112.78 (2 C-3 of furan), 120.65 (2 C-4 of furan), 126.51 (4 CH of 2 Ph), 127.90 (2 CH of 2 Ph), 128.50 (4 CH of 2 Ph), 128.65 (4 CH of 2 Ph), 128.83 (4 CH of 2 Ph), 130.34 (2 C_{ipso} -furyl), 133.77 (2 CH of 2 Ph), 136.38 (2 C_{ipso} - COCH_2 of 2 Ph), 149.64 (2 C-5 of furan), 152.73 (2 C-2 of furan), 164.75 and 165.34 (2 CONH), 199.36 and 204.78 (2 COPh).

MS: m/z (%) = 748 (M^+ , 2), 702 (48), 701 (45), 700 (100), 438 (17), 317 (97), 96 (23).

Anal. Calcd for $\text{C}_{48}\text{H}_{48}\text{N}_2\text{O}_6$ (748.91): C, 76.98; H, 6.46; N, 3.74. Found: C, 77.00; H, 6.50; N, 3.70.

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