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Furyl(aryl)methanes and their Derivatives. Part 21:[1] Cinnoline Derivatives from 2-Aminophenylbisfurylmethanes

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Abstract—(*Z*)-4-[4-(5-Methyl-2-furyl)-3-cinnolinyl]-3-buten-2-ones and (*Z*)-1-[4-(5-ethyl-2-furyl)-3-cinnolinyl]-1-penten-3-ones have been obtained from 2-aminoarylbisfurylmethanes under treatment with isoamyl nitrite/trimethylchlorosilane in dry acetonitrile. Intramolecular cyclisation and furan oxidative ring opening follow the formation of an intermediate diazonium salt. X-Ray analysis proved the *cis*-configuration of the alkene side chain. © 2000 Elsevier Science Ltd. All rights reserved.

Introduction

The use of furan derivatives as versatile synthons in organic synthesis is well documented.[2] As was shown by our group, ortho-functionalised aryldifurylmethanes are convenient precursors in the syntheses of various condensed heterocyclic systems: benzofurans,[3] oxazulenes,[4] azaazulenes,[5] indoles[5,6] and benzothiazines.[6] Carbazole derivatives were obtained in the deoxygenation reaction of 2-nitrophenyldifurylmethanes with triethylphosphite.[7]

Jones' and McKinley's earlier attempt[7] to substitute an amino-group for the azido-group in 2-aminophenyldifurylmethanes via diazotisation in aqueous H_2SO_4 failed due to tar formation in the reaction mixture. We have found that performance of the above-mentioned reaction under anhydrous conditions leads to cinnoline derivatives.[6] In the present paper, we wish to present our data about this new cinnoline synthesis from furyl(aryl)methanes.

Results and Discussions

2-Nitrophenyldifurylmethanes **1a–g** were obtained in good yields by the condensation of the appropriate 2-nitrobenzaldehydes with 2-methyl- and 2-ethylfurans in dioxane in the presence of $HClO_4$. Reduction of the compounds with $NaBH_4$ in the presence of 5% Pd/C in methanol gave 2-aminophenyldifurylmethanes **2a–g** (Scheme 1).

Diazotisation was carried out in dry acetonitrile at 0°C with isoamyl nitrite and trimethylchlorosilane. There was no noticeable tar formation under these conditions, and after the usual workup seven cinnoline derivatives **3a–g** were isolated in high yields (Scheme 2). We suppose that cinnoline formation is a result of intramolecular electrophilic attack of the diazonium group at one of the furan rings, with its subsequent cleavage.

Structures **3a–g** were confirmed by elemental analysis, along with the presence of C=O stretching IR-spectra at 1680 cm^{-1} and vinyl proton signals in ^1H NMR-spectra (J=12.2 Hz). The value of the coupling constant indicates that the alkene side chain has a *cis*-configuration. For further support of the structure X-ray analysis of the compound **3g** monocrystal was made, and its ORTEP drawings are shown below (Fig. 1).

Cis-configuration of the side chain is in accordance with the elecrocyclic mechanism of the furan oxidative ring cleavage, as shown in Scheme 2.

Experimental

General

^1H NMR spectra were recorded with Bruker AC-200 (200 MHz), Bruker AM-300 (300 MHz) and Tesla BS-467A (60 MHz) spectrometers. IR-spectra were recorded on Specord-M80 spectrophotometer. Commercial 2-methylfuran and 2-ethylfuran was allowed to stand over powdered NaOH overnight, with subsequent filtration and distillation. Substituted 2-nitrobenzaldehydes were obtained via nitration of the appropriate aldehydes according to the described methods.[8,9]

Keywords: diazo compounds; furans; ring transformations; cyclisation; aromatisation; bicyclic heterocyclic compounds; nitrogen heterocycles.

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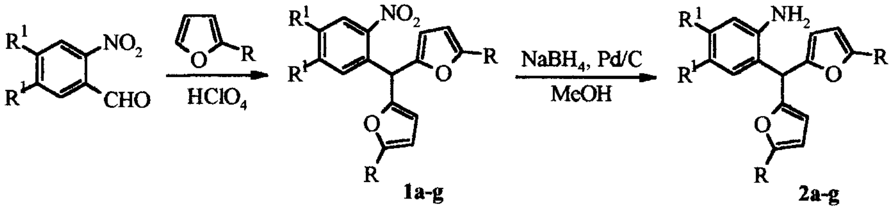
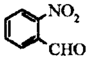
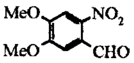
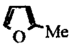
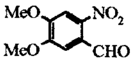
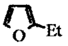
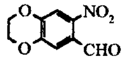
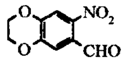
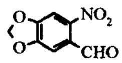
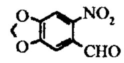
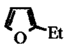
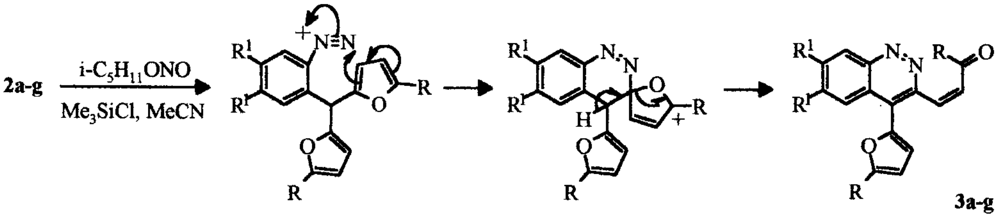


1a-g **2a-g**

Aldehyde	Furan	Isolated Yield	
		1 (%)	2 (%)
		**1a** (74)	**2a** (70)
		**1b** (69)	**2b** (73)
		**1c** (78)	**2c** (78)
		**1d** (58)	**2d** (67)
		**1e** (81)	**2e** (80)
		**1f** (64)	**2f** (67)
		**1g** (86)	**2g** (83)

Scheme 1.



3a-g

Compound	R	$R^1 = R^1$	Isolated Yield, %
3a	CH_3	H	79
3b	CH_3	OCH_3	86
3c	C_2H_5	OCH_3	89
3d	CH_3	OCH_2CH_2O	76
3e	C_2H_5	OCH_2CH_2O	87
3f	CH_3	OCH_2O	79
3g	C_2H_5	OCH_2O	83

Scheme 2.

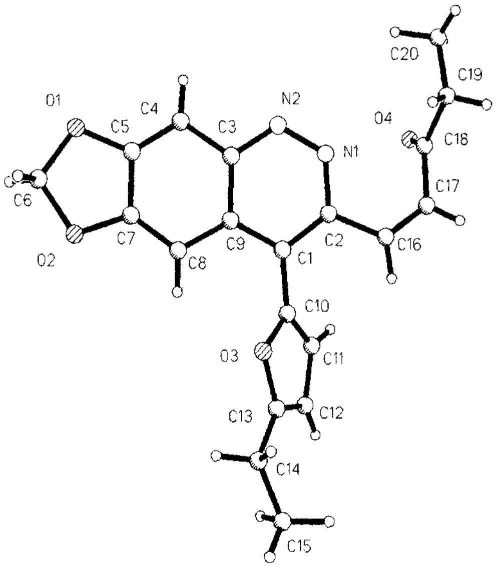


Figure 1. ORTEP Drawing of **3g**.

X-Ray single crystal analysis of 3g

Data were collected on a SYNTEX P-1 diffractometer using the $\theta/2\theta$ technique, Cu-Ka radiation ($\lambda=1.5418$), scan speed 3.0–12.0 deg min^{-1}, scan range 3.5–120° and beta-filter. Data were corrected for Lorentz and polarisation effects. The structure was resolved by a direct method using SHELXS-86,[10] and the model was refined by a full-matrix least-square technique.

General procedure for 2-nitroarylbisfurylmethanes

To a solution of 2-nitrobenzaldehyde (50 mmol) and 2-methylfuran (15 mL, 167 mmol) in dioxane (70 mL) perchloric acid (0.5 mL) was added. The mixture was left overnight, then poured into water (500 mL), and extracted with warm benzene (3×50 mL); the combined organic layers were dried with Na_2SO_4 and evaporated to dryness. The oily residue was dissolved in hot hexane and the solution was filtered through a pad of Al_2O_3 and left overnight. The crystalline product was filtered off and air dried.

Bis(5-methyl-2-furyl)-(2-nitrophenyl)methane (1a). Yield 11.0 g, 74%; mp 82–83°C (hexane/CH_2Cl_2); [Found: C, 68.71; H, 5.12; N, 4.69. $C_{17}H_{15}NO_4$ requires C, 68.68; H, 5.09; N, 4.71%]; δ_H (200 MHz, $CDCl_3$) 2.23 (6H, s, CH_3), 5.88 (2H, d, J=3.2 Hz, 4-H_{Fur}), 5.96 (2H, d, J=3.2 Hz, 3-H_{Fur}), 6.16 (1H, s, CH), 7.30 (1H, d, J=7.8 Hz, 6-H_{Ar}), 7.53 (1H, dd, J=7.8, 8.0 Hz, 4-H_{Ar}), 7.65 (1H, dd, J=7.8, 8.0 Hz, 5-H_{Ar}), 7.95 (1H, d, J=7.8 Hz, 3-H_{Ar}).

Bis(5-methyl-2-furyl)-(4,5-dimethoxy-2-nitrophenyl)-methane (1b). Yield 12.3, 69%; mp 94–95°C (methanol); [Found: C, 63.89; H, 5.40; N, 3.91. $C_{19}H_{19}NO_6$ requires C, 63.86; H, 5.36; N, 3.92%]; δ_H (300 MHz, DMSO-d$_6$) 2.22 (6H, s, CH_3), 3.73 (3H, s, OCH_3), 3.88 (3H, s, OCH_3), 5.90 (2H, d, J=3.2 Hz, 4-H_{Fur}), 5.96 (2H, d, J=3.2 Hz, 3-H_{Fur}), 6.16 (1H, s, CH), 6.71 (1H, s, 6-H_{Ar}), 7.60 (1H, s, 3-H_{Ar}).

Bis(5-ethyl-2-furyl)-(4,5-dimethoxy-2-nitrophenyl)-methane (1c). Yield 15.0 g, 78%; oil; [Found: C, 64.35; H, 6.13; N, 3.51. $C_{21}H_{23}NO_6$ requires C, 65.44; H, 6.01; N, 3.63%]; δ_H (300 MHz, DMSO-d$_6$) 1.18 (6H, t, J=7.5 Hz, CH$_2$CH_3), 2.56 (4H, q, J=7.5 Hz, CH_2CH$_3$), 3.72 (3H, s, OCH_3), 3.87 (3H, s, OCH_3), 5.90 (2H, d, J=3.2 Hz, 4-H_{Fur}), 5.95 (2H, d, J=3.2 Hz, 3-H_{Fur}), 6.18 (1H, s, CH), 6.69 (1H, s, 6-H_{Ar}), 7.59 (1H, s, 3-H_{Ar}).

Bis(5-methyl-2-furyl)-(4,5-ethylenedioxy-2-nitrophenyl)-methane (1d). Yield 10.3 g, 58%; mp 138–139°C (hexane); [Found: C, 64.15; H, 4.95; N, 3.84. $C_{19}H_{17}NO_6$ requires C, 64.22; H, 4.82; N, 3.94%]; δ_H (300 MHz, $CDCl_3$) 2.25 (6H, s, CH_3), 4.26–4.32 (4H, m, OCH_2CH_2O), 5.88 (2H, d, J=3.2 Hz, 4-H_{Fur}), 5.91 (2H, d, J=3.2 Hz, 3-H_{Fur}), 6.29 (1H, s, CH), 6.80 (1H, s, 6-H_{Ar}), 7.64 (1H, s, 3-H_{Ar}).

Bis(5-ethyl-2-furyl)-(4,5-ethylenedioxy-2-nitrophenyl)-methane (1e). Yield 15.5 g, 81%; mp 85–86°C (hexane); [Found: C, 65.92; H, 5.67; N, 3.54. $C_{21}H_{21}NO_6$ requires C, 65.79; H, 5.52; N, 3.65%]; δ_H (300 MHz, $CDCl_3$) 1.20 (6H, t, J=7.5 Hz, CH$_2$CH_3), 2.61 (4H, q, J=7.5 Hz, CH_2CH$_3$), 4.27–4.33 (4H, m, OCH_2CH_2O), 5.89 (2H, d, J=3.2 Hz, 4-H_{Fur}), 5.92 (2H, d, J=3.2 Hz, 3-H_{Fur}), 6.30 (1H, s, CH), 6.79 (1H, s, 6-H_{Ar}), 7.65 (1H, s, 3-H_{Ar}).

Bis(5-methyl-2-furyl)-(4,5-methylenedioxy-2-nitrophenyl)-methane (1f). Yield 10.9 g, 64%; mp 108–109°C (hexane); [Found: C, 63.27; H, 4.53; N, 3.98. $C_{18}H_{15}NO_6$ requires C, 63.34; H, 4.43; N, 4.10%]; δ_H (60 MHz, $CDCl_3$) 2.17 (6H, s, CH_3), 5.80 (2H, d, J=3.2 Hz, 4-H_{Fur}), 5.90 (2H, d, J=3.2 Hz, 3-H_{Fur}), 6.03 (2H, s, OCH_2O), 6.23 (1H, s, CH), 6.73 (1H, s, 6-H_{Ar}), 7.45 (1H, s, 3-H_{Ar}).

Bis(5-ethyl-2-furyl)-(4,5-methylenedioxy-2-nitrophenyl)-methane (1g). Yield 15.9 g, 86%; mp 80–81°C (hexane); [Found: C, 64.96; H, 5.21; N, 3.83. $C_{20}H_{19}NO_6$ requires C, 65.03; H, 5.18; N, 3.79%]; δ_H (60 MHz, $CDCl_3$) 1.13 (6H, t, J=7.5 Hz, CH$_2$CH_3), 2.53 (4H, q, J=7.5 Hz, CH_2CH$_3$), 5.83 (2H, d, J=3.2 Hz, 4-H_{Fur}), 5.92 (2H, d, J=3.2 Hz, 3-H_{Fur}), 6.01 (2H, s, OCH_2O), 6.25 (1H, s, CH), 6.73 (1H, s, 6-H_{Ar}), 7.45 (1H, s, 3-H_{Ar}).

General procedure for 2-aminoarylbisfurylmethanes

To a solution of 2-nitroarylbisfurylmethane **1** (100 mmol) in hot methanol (70 mL), 5% palladium on charcoal (0.5 g) was added, and then $NaBH_4$ (1 g, 26 mmol) in small portions. The mixture was stirred for 2 hours until the starting compound disappeared (TLC), then poured into water (500 mL). After extracting with warm benzene (3×50 mL), the combined organic layers were dried with Na_2SO_4 and evaporated to dryness. The oily residue was dissolved in hot hexane, and the solution was filtered through a pad of Al_2O_3 and left overnight in the cold to crystallise. The crystalline product **2b** was filtered off and air-dried; the oily products were used in the next step without further purification.

Bis(5-methyl-2-furyl)-(4,5-dimethoxy-2-aminophenyl)-methane (2b). Yield 23.9 g, 73%; oil, oxalate salt—mp 119–120°C; [Found: C, 69.75; H, 6.51; N, 4.26. $C_{19}H_{21}NO_4$ requires C, 69.71; H, 6.47; N, 4.28%]; ν_{max}(Nujol) 3450, 3380 cm^{-1}; δ_H (300 MHz, DMSO-d$_6$) 2.22 (6H, s, C*H*$_3$), 3.53 (3H, s, OC*H*$_3$), 3.69 (3H, s, OC*H*$_3$), 4.46 (2H, s, N*H*$_2$), 5.39 (1H, s, C*H*), 5.89 (2H, d, *J*=3.2 Hz, 4-*H*$_{\mathrm{Fur}}$), 5.97 (2H, d, *J*=3.2 Hz, 3-*H*$_{\mathrm{Fur}}$), 6.38 (1H, s, 3-*H*$_{\mathrm{Ar}}$), 6.49 (1H, s, 6-*H*$_{\mathrm{Ar}}$).

General procedure for cinnolines

To a stirred solution of 2-aminoarylbisfurylmethane **2** (10 mmol) in acetonitrile (15 mL), trimethylchlorosilane (2 mL, 16 mmol) and isoamyl nitrite (1.5 mL, 11 mmol) were added successively. The mixture was stirred for an additional 15 min, then poured into water (200 mL) and made alkaline with solid $NaHCO_3$. The aqueous solution was extracted with ethyl acetate (3×50 mL), the organic layer was separated and dried over Na_2SO_4. Then picric acid (4 g, 18 mmol) in ethyl acetate (10 mL) was added to the solution and the mixture was left overnight. The precipitated salt was filtered and washed with ether. Treatment with 3% aqueous sodium hydrogen carbonate gave an easily crystallisable oil. The crude cinnoline was filtered off, washed with methanol and air dried. The product was recrystallised from ethyl acetate.

(Z)-4-[4-(5-Methyl-2-furyl)-3-cinnolinyl]-3-buten-2-one (3a). Yield 2.20 g, 79%; mp 94–95°C (benzene); [Found: C, 73.24; H, 5.19; N, 10.13. $C_{17}H_{14}N_2O_2$ requires C, 73.37; H, 5.07; N, 10.06%]; $\nu_{\max}$(Nujol) 1680 cm^{-1}; δ_H (300 MHz, $CDCl_3$) 2.31 (3H, s, COC*H*$_3$), 2.49 (3H, s, C*H*$_3$), 6.30 (1H, d, *J*=3.2 Hz, 4-*H*$_{\mathrm{Fur}}$), 6.47 (1H, d, *J*=12.2 Hz, CH═C*H*–CO), 6.80 (1H, d, *J*=3.2 Hz, 3-*H*$_{\mathrm{Fur}}$), 7.13 (1H, d, *J*=12.2 Hz, C*H*═CHCO), 7.75 (1H, dd, *J*=7.8, 8.0 Hz, 7-*H*$_{\mathrm{Cin}}$), 7.83 (1H, dd, *J*=7.8, 8.0 Hz, 6-*H*$_{\mathrm{Cin}}$), 8.33 (1H, d, *J*=7.8 Hz, 8-*H*$_{\mathrm{Cin}}$), 8.53 (1H, d, *J*=7.8 Hz, 5-*H*$_{\mathrm{Cin}}$).

(Z)-4-[6,7-Dimethoxy-4-(5-methyl-2-furyl)-3-cinnolinyl]-3-buten-2-one (3b). Yield 2.91 g, 86%; mp 169–170°C (ethyl acetate); [Found: C, 67.47; H, 5.40; N, 8.30. $C_{19}H_{18}N_2O_4$ C, 67.45; H, 5.36; N, 8.28%]; $\nu_{\max}$(Nujol) 1680 cm^{-1}; δ_H (200 MHz, $CDCl_3$) 2.26 (3H, s, COC*H*$_3$), 2.47 (3H, s, C*H*$_3$), 4.01 (3H, s, OC*H*$_3$), 4.10 (3H, s, OC*H*$_3$), 6.28 (1H, d, *J*=3.2 Hz, 4-*H*$_{\mathrm{Fur}}$), 6.40 (1H, d, *J*=12.2 Hz, CH═C*H*CO), 6.75 (1H, d, *J*=3.2 Hz, 3-*H*$_{\mathrm{Fur}}$), 7.10 (1H, d, *J*=12.2 Hz, C*H*═CHCO), 7.55 (1H, s, 8-*H*$_{\mathrm{Cin}}$), 7.75 (1H, s, 5-*H*$_{\mathrm{Cin}}$).

(Z)-1-[4-(5-Ethyl-2-furyl)-6,7-dimethoxy-3-cinnolinyl]-1-penten-3-one (3c). Yield 3.26 g, 89%; mp 97–96°C (benzene); [Found: C, 68.92; H, 6.13; N, 7.75. $C_{21}H_{22}N_2O_4$ requires C, 68.84; H, 6.05; N, 7.65%]; $\nu_{\max}$(Nujol) 1680 cm^{-1}; δ_H (200 MHz, $CDCl_3$) 1.13 (3H, t, *J*=7.2 Hz, COCH$_2$C*H*$_3$), 1.36 (3H, t, *J*=7.2 Hz, CH$_2$C*H*$_3$), 2.58 (2H, q, *J*=7.2 Hz, COC*H*$_2$CH$_3$), 2.81 (2H, q, *J*=7.2 Hz, C*H*$_2$CH$_3$), 4.00 (3H, s, OC*H*$_3$), 4.09 (3H, s, OC*H*$_3$), 6.28 (1H, d, *J*=3.2 Hz, 4-*H*$_{\mathrm{Fur}}$), 6.41 (1H, d, *J*=12.2 Hz, CH═C*H*CO), 6.76 (1H, d, *J*=3.2 Hz, 3-*H*$_{\mathrm{Fur}}$), 7.07 (1H, d, *J*=12.2 Hz, C*H*═CHCO), 7.52 (1H, s, 8-*H*$_{\mathrm{Cin}}$), 7.74 (1H, s, 5-*H*$_{\mathrm{Cin}}$).

(Z)-4-[4-(5-Methyl-2-furyl)-7,8-dihydro[1,4]dioxino[2,3-*g*]cinnolin-3-yl]-3-buten-2-one (3d). Yield 2.56 g, 76%; mp 135–136°C (ethyl acetate); [Found: C, 67.94; H, 4.91; N, 8.25. $C_{19}H_{16}N_2O_4$ requires C, 67.85; H, 4.79; N, 8.33%]; $\nu_{\max}$(Nujol) 1680 cm^{-1}; δ_H (300 MHz, $CDCl_3$) 2.27 (3H, s, COC*H*$_3$), 2.48 (3H, s, C*H*$_3$), 4.43 (4H, s, OC*H*$_2$C*H*$_2$O), 6.27 (1H, d, *J*=3.2 Hz, 4-*H*$_{\mathrm{Fur}}$), 6.39 (1H, d, *J*=12.4 Hz, CH═C*H*CO), 6.72 (1H, d, *J*=3.2 Hz, 3-*H*$_{\mathrm{Fur}}$), 7.09 (1H, d, *J*=12.4 Hz, C*H*═CHCO), 7.69 (1H, s, 8-*H*$_{\mathrm{Cin}}$), 7.92 (1H, s, 5-*H*$_{\mathrm{Cin}}$).

(Z)-1-[4-(5-Ethyl-2-furyl)-7,8-dihydro[1,4]dioxino[2,3-*g*]cinnolin-3-yl]-1-penten-3-one (3e). Yield 3.17 g, 87%; mp 113–114°C (ethyl acetate); [Found: C, 69.33; H, 5.52; N, 7.60. $C_{21}H_{20}N_2O_4$ requires C, 69.22; H, 5.53; N, 7.69%]; $\nu_{\max}$(Nujol) 1680 cm^{-1}; δ_H (200 MHz, $CDCl_3$) 1.13 (3H, t, *J*=7.2 Hz, COCH$_2$C*H*$_3$), 1.35 (3H, t, *J*=7.2 Hz, CH$_2$C*H*$_3$), 2.59 (2H, q, *J*=7.2 Hz, COC*H*$_2$CH$_3$), 2.81 (2H, q, *J*=7.2 Hz, C*H*$_2$CH$_3$), 4.41 (4H, s, OC*H*$_2$C*H*$_2$O), 6.26 (1H, d, *J*=3.2 Hz, 4-*H*$_{\mathrm{Fur}}$), 6.41 (1H, d, *J*=12.4 Hz, CH═C*H*CO), 6.73 (1H, d, *J*=3.2 Hz, 3-*H*$_{\mathrm{Fur}}$), 7.06 (1H, d, *J*=12.4 Hz, C*H*═CHCO), 7.66 (1H, s, 8-*H*$_{\mathrm{Cin}}$), 7.90 (1H, s, 5-*H*$_{\mathrm{Cin}}$).

(Z)-4-[4-(5-Methyl-2-furyl)[1,3]dioxolo[4,5-*g*]cinnolin-3-yl]-3-buten-2-one (3f). Yield 2.55, 79%; mp 118–119°C (benzene); [Found: C, 67.14; H, 4.35; N, 8.73. $C_{18}H_{14}N_2O_4$ requires C, 67.08; H, 4.38; N, 8.69%]; $\nu_{\max}$ (Nujol) 1680 cm^{-1}; δ_H (200 MHz, $CDCl_3$) 2.28 (3H, s, COC*H*$_3$), 2.47 (3H, s, C*H*$_3$), 4.43 (4H, s, OC*H*$_2$O), 6.25 (1H, d, *J*=3.2 Hz, 4-*H*$_{\mathrm{Fur}}$), 6.38 (1H, d, *J*=12.4 Hz, CH═C*H*CO), 6.70 (1H, d, *J*=3.2 Hz, 3-*H*$_{\mathrm{Fur}}$), 6.99 (1H, d, *J*=12.4 Hz, C*H*═CHCO), 7.43 (1H, s, 8-*H*$_{\mathrm{Cin}}$), 7.61 (1H, s, 5-*H*$_{\mathrm{Cin}}$).

(Z)-1-[4-(5-Ethyl-2-furyl)[1,3]dioxolo[4,5-*g*]cinnolin-3-yl]-1-penten-3-one (3g). Yield 2.91, 83%; mp 112–113°C (benzene); [Found: C, 68.49; H, 5.20; N, 7.97. $C_{20}H_{18}N_2O_4$ requires C, 68.56; H, 5.18; N, 8.00%]; $\nu_{\max}$(Nujol) 1680 cm^{-1}; δ_H (200 MHz, $CDCl_3$) 1.12 (3H, t, *J*=7.2 Hz, COCH$_2$C*H*$_3$), 1.32 (3H, t, *J*=7.2 Hz, CH$_2$C*H*$_3$), 2.57 (2H, q, *J*=7.2 Hz, COC*H*$_2$CH$_3$), 2.78 (2H, q, *J*=7.2 Hz, C*H*$_2$CH$_3$), 4.41 (4H, s, OC*H*$_2$O), 6.23 (1H, d, *J*=3.2 Hz, 4-*H*$_{\mathrm{Fur}}$), 6.39 (1H, d, *J*=12.4 Hz, CH═C*H*CO), 6.69 (1H, d, *J*=3.2 Hz, 3-*H*$_{\mathrm{Fur}}$), 6.97 (1H, d, *J*=12.4 Hz, C*H*═CHCO), 7.41 (1H, s, 8-*H*$_{\mathrm{Cin}}$), 7.60 (1H, s, 5-*H*$_{\mathrm{Cin}}$).

Supporting samples of the compounds are available independently from Molecular Diversity Preservation International, http://www.mdpi.org.

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