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One-pot three-component synthesis of the spiroacenaphthylene derivatives

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ABSTRACT

A facile and efficient one-pot three-component synthesis of the spiroacenaphthylene derivatives was achieved, via the reaction of acenaphthequinone, malononitrile/ethylcyanoacetate and various reagents including α-methylencarbonyl compounds/enols.

1. Introduction

In multicomponent reactions (MCRs), three or more reactants come together in a single reaction vessel to form new products that contain structural units of all the components. This type of reaction becomes increasingly important in organic and medicinal chemistry because it allows to obtain highly sophisticated polyfunctional molecules through simple one-pot procedures. Multicomponent reactions have been successfully employed to generate highly diverse combinatorial libraries for high-throughput screening of biological and pharmacological activities. The use of three or more building blocks in a one-pot, high-yield multicomponent reaction leads to a wide structural and functional diversity combined with excellent combinatorial efficiency. Over the past decade, industrial and academic research has made powerful MCR strategies into one of the most efficient and cost-effective tools for combinatorial synthesis.[1–3] The development of novel MCRs is an intellectually challenging task since one has to consider not only the reactivity match of the starting materials but also the reactivity of the intermediate molecules generated in situ, their compatibility and their compartmentalization.[4]

Heterocycles containing the pyran ring are important targets in synthetic and medicinal chemistry because this fragment is a key moiety in numerous biologically active compounds and natural products.[5,6] Spiro compounds represent an important class of naturally occurring substances characterized by highly pronounced biological properties. The spiro functionality has been known for a long time to be present in phytochemicals either in alkaloids, lactones or terpenoids.[7] Biological activities of spiro compounds containing pyrans have also been proved. They also show good activity as hypertensive agents.[8] They have been the subject of great interest as potential novel analgesic agents.[9]

The synthesis of heterocycles by multicomponent reactions often involves classic carbonyl condensation chemistry[10,11] and basic catalysts play important role in MCRs. Triethylamine is an efficient and inexpensive base and its efficiency as a basic catalyst has been explored frequently.[12,13] The broad spectrum of applications of acenaphthenequinone **1** and its derivatives as biologically active compounds, dyes, etc.[14] has prompted us to explore its reaction with malononitrile/ethylcyanoacetate **2** and α-methylencarbonyl compounds/enols **3** in MCR (Scheme 1).

2. Results and discussion

In continuation of our investigations of the synthesis of novel heterocyclic compounds and designing novel procedures in multicomponent reactions,[15–18] we have developed an efficient protocol for the solution phase synthesis of new spiroacenaphthylene derivatives, through the three-component condensation of acenaphthequinone, malononitrile/ethylcyanoacetate and various reagents including α-methylencarbonyl compounds/enols (Scheme 1).

During our work we found out that, Dabiri et al.[19] reported the synthesis of **4e** and **4f** through the three-component condensation of acenaphthequinone, malononitrile/ethylcyanoacetate and dimedon in the presence of ammonium salt. Herein, we wish to reveal our results, using a wide variety of compounds **3** and a straightforward procedure in the absence of complex and expensive catalyst.

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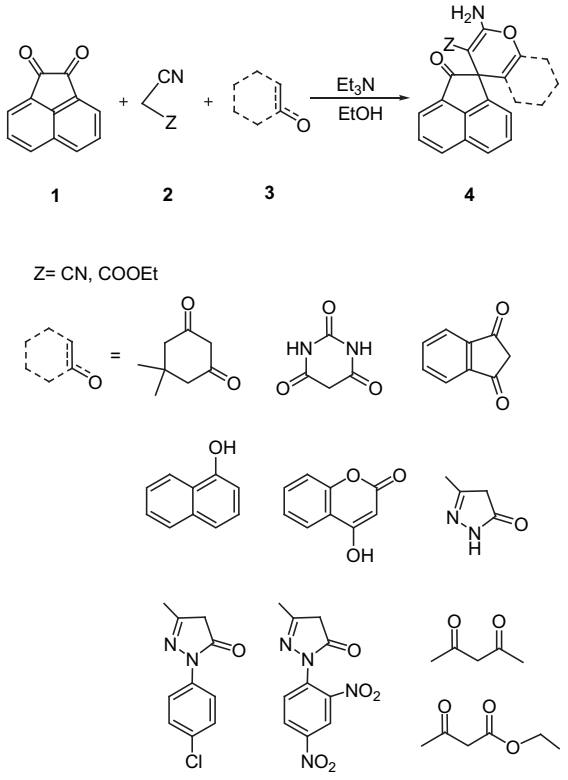
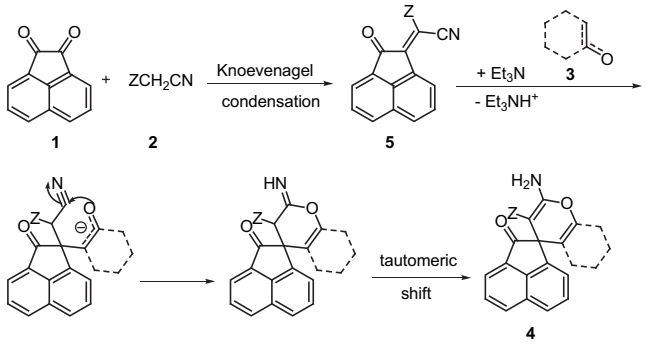


Scheme 1. Three-component synthesis of spiroacenaphthylene derivatives.

The synthesis of **4** includes a two-step reaction: Knoevenagel condensation to obtain unsaturated nitriles **5** from acenaphthenequinone **1** and malononitrile/ethylcyanoacetate **2** and interaction of **5** with the active α-methylencarbonyl compounds/enols **3** catalyzed by triethylamine (Et_3N).

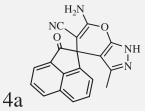
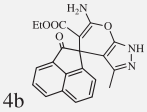
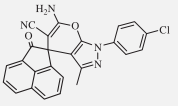
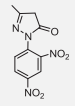
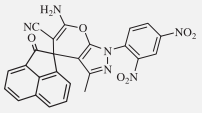
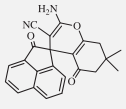
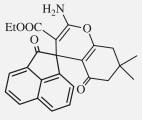
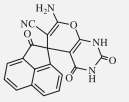
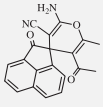
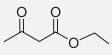
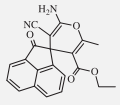
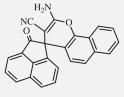
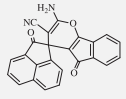
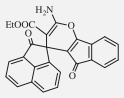
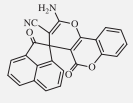
A plausible mechanism for the reaction is shown in Scheme 2. Compound **1** undergoes Knoevenagel condensation with malononitrile/ethylcyanoacetate. Unsaturated nitriles **5** are formed very easily in ethanol even in the absence of Et_3N. Reactant **3** undergoes Michael addition to reagent **5**, followed by cycloaddition onto the nitrile. Eventually, after tautomeric proton shift compound **4** is formed.



Scheme 2. Plausible mechanism for the formation of **4**.

As shown in Table 1, to establish the generality of the method, a wide variety of suitable substrates were employed. Various reactants **3** including 1,3-diketones, pyrazolones and enoles were used and the reactions afforded the corresponding products. In addition, the reaction with ethylcyanoacetate or malononitrile also proceeded smoothly; however, the reaction times needed, when ethylcyanoacetate employed as one of the substrates, were longer

Table 1
Synthesis of spiroacenaphthylene derivatives **4**

Entry	Z	Reactant **3**	Product	Time (h)	Yield[a] (%)
1	CN		 4a	4	85
2	COOEt		 4b	4.5	80
3	CN		 4c	6	75
4	CN		 4d	6	65
5	CN		 4e	4	80
6	COOEt		 4f	4.5	80
7	CN		 4g	8	60
8	CN		 4h	4	65
9	CN		 4i	4	65
10	CN		 4j	5	70
11	CN		 4k	5	65
12	COOEt		 4l	7	40
13	CN		 4m	4	60

[a] Isolated yield.

than those of malononitrile, which is probably due to the lower reactivity of cyanoacetates (Table 1).

3. Conclusion

In summary, we have developed a new facile protocol for the synthesis of new spiroacenaphthylene derivatives from the reaction of acenaphthenequinone, malononitrile/ethylcyanoacetate and α-methylencarbonyl compounds/enols.

4. Experimental section

4.1. General

Melting points were measured, using a capillary tube method with a Bamstead Electrothermal 9200 apparatus. ^{1}H and ^{13}C NMR spectra were recorded on a Bruker AQS-AVANCE spectrometer at 500 and 125 MHz, using TMS as an internal standard ($CDCl_3$ solution). FTIR spectra were recorded using, KBr disks on FT-IR Bruker Tensor 27 instrument. Mass spectra were documented on an Agilent Technology (HP) mass spectrometer operating at an ionization potential of 70 eV. The elemental analysis was performed with an Elemetar Analysensystem GmbH VarioEL CHNS mode.

4.2. General procedure for the synthesis of compounds 4

Acenaphthenequinone **1** (0.182 g, 1 mmol), malononitrile/ethylcyanoacetate **2** (1 mmol), compound **3** (1 mmol) were dissolved under heating in ethanol (5 mL) and Et_3N (0.3 mL) was then added. The reaction mixture was then heated at reflux for the indicated time (Table 1) and cooled to 4 °C overnight. The solid, which separated was filtered off, washed three times with ethanol and dried at 60–70 °C. Spiroacenaphthylene derivatives were analytically pure without recrystallization.

*4.2.1. 3′-Amino-7′-methyl-2-oxo-2H,5H′-spiro[acenaphthylene-1,1′-pyrano[2,3-c]pyrazolo-2′-carbonitrile (**4a**).* Pale yellow powder (0.28 g, 85%). Mp 298–299 °C. Found: C, 69.90; H, 3.36; N, 17.32. $C_{19}H_{12}N_4O_2$ requires C, 69.51; H, 3.68; N, 17.06%; ν_{max} (KBr) 3422, 3321, 3146, 2187, 1710, 1640, 1580, 1517 cm^{-1}; δ_H (500 MHz, DMSO-d_6) 8.48–7.54 (6H, m, ArH), 7.38 (2H, s, NH_2), 4.43 (1H, s, N*H*), 1.15 (3H, s, *Me*); δ_C (125 MHz, DMSO-d_6) 204.7, 163.4, 156.1, 141.9, 141.8, 135.6, 133.4, 131.4, 130.8, 130.3, 129.8, 125.8, 123.4, 122.1, 119.7, 97.2, 56.8, 52.5, 9.9; MS, *m*/*z* (%): 328 (M^+, 5), 299 (25), 230 (30), 43 (65).

*4.2.2. Ethyl-3′-amino-7′-methyl-2-oxo-2H,5′H-spiro[acenaphthylene-1,1′-pyrano[2,3-c]pyrazole]-2′-carboxylate (**4b**).* Light yellow powder (0.30 g, 80%). Mp 247–248 °C. Found: C, 67.57; H, 4.44; N, 11.37. $C_{21}H_{17}N_3O_4$ reqires C, 67.19; H, 4.56; N, 11.19%; ν_{max} (KBr) 3387, 3264, 3057, 2978, 2920, 1714, 1688, 1599, 1549 cm^{-1}; δ_H (500 MHz, DMSO-d_6) 12.19 (1H, s, N*H*), 8.37–7.31 (6H, m, ArH), 8.35 (2H, s, NH_2), 3.38 (2H, q, *J* 7.0 Hz, −OCH_2Me), 1.02 (3H, s, *Me*), 0.04 (3H, t, *J* 7.0 Hz, −OCH$_2$*Me*); δ_C (125 MHz, DMSO-d_6) 205.7, 168.9, 163.9, 155.1, 146.3, 141.2, 135.5, 133.5, 132.3, 130.4, 129.9, 129.2, 124.3, 121.9, 120.0, 99.2, 75.8, 59.1, 52.5, 12.9, 10.2; MS, *m*/*z* (%): 375 (M^+, 5), 336 (100), 305 (30), 83 (25).

*4.2.3. 3′-Amino-5′-(4-chlorophenyl)-7′-methyl-2-oxo-2H,5H′-spiro[acenaphthylene-1,1′-pyrano[2,3-c]pyrazole]-2′-carbonite (**4c**).* Light yellow powder (0.33 g, 75%). Mp >300 °C. Found: C, 68.02; H, 3.29; N, 12.81. $C_{25}H_{15}N_4O_2Cl$ reqires C, 68.42; H, 3.45; N, 12.77%; ν_{max} (KBr) 3423, 3206, 2216, 1720, 1621, 1576 cm^{-1}; δ_H (500 MHz, DMSO-d_6) 8.52–7.54 (10H, m, ArH), 7.38 (2H, s, NH_2), 1.15 (3H, s, *Me*); δ_C (125 MHz, DMSO-d_6) 203.4, 163.5, 156.5, 151.4, 150.8, 150.6, 146.8, 146.2, 144.7, 143.6, 141.4, 141.3, 132.8, 129.7, 129.3, 124.8, 124.7, 124.2, 124.0, 120.7, 116.4, 105.3, 56.0, 52.0, 11.0; MS, *m*/*z* (%): 438 (M^+, 15), 402 (30), 326 (100), 69 (35).

4.2.4. 3′-Amino-5′-(2,4-dinitrophenyl)-7′-methyl-2-oxo-2H,5H′-spiro[acenaphthylene-1,1′-pyrano[2,3-c]pyrazole]-2′-carbonite. Light yellow powder (0.32 g, 65%). Mp >300 °Cz. Found: C, 60.37; H, 2.81; N, 17.11. $C_{25}H_{14}N_6O_6$ requires C, 60.73; H, 2.85; N, 17.00%; ν_{max} (KBr) 3440, 3315, 2222, 1726, 1662, 1616, 1593, 1497, 1423, 1339 cm^{-1}; δ_H (500 MHz, DMSO-d_6) 8.64–7.68 (10H, m, ArH), 7.26 (2H, s, NH_2), 1.21 (3H, s, *Me*); δ_C (125 MHz, DMSO-d_6) 204.7, 164.0, 157.0, 151.7, 150.8, 150.4, 146.9, 146.7, 144.9, 143.8, 141.9, 141.8, 132.5, 129.4, 129.0, 125.0, 124.3, 124.2, 124.1, 120.5, 116.7, 105.9, 56.7, 52.2, 9.7; MS, *m*/*z* (%): 494 (M^+, 10), 327 (100), 258 (30), 69 (35).

*4.2.5. 2′-Amino-7′,7′-dimethyl-2,5′-dioxo-5′,6′,7′,8′-tetrahydro-2H-spiro[acenaphthylene-1,4′-chromene]-3′-carbonitrile (**4e**).* Light yellow powder (0.30 g, 80%). Mp 268–270 °C; [Found: C, 74. 63; H, 4.79; N, 7.47. $C_{23}H_{18}N_2O_3$ requires C, 74.58; H, 4.90; N, 7.56%; ν_{max} (KBr) 3368, 3296, 2957, 2193, 1718, 1664, 1600 cm^{-1}; δ_H (500 MHz, DMSO-d_6) 8.35–7.46 (6H, m, ArH), 7.39 (2H, s, NH_2), 2.71 (2H, s, CH_2), 2.17 (1H, d, *J* 16.1 Hz, CH_aH$_b$), 2.12 (1H, d, *J* 16.0 Hz, CH$_a$$H_b$), 1.12 (3H, s, *Me*), 1.10 (3H, s, *Me*); δ_C (125 MHz, DMSO-d_6) 204.4, 196.2, 165.4, 159.7, 144.1, 141.4, 133.1, 132.3, 130.7, 129.8, 129.3, 125.4, 122.3, 120.7, 118.4, 112.9, 64.4, 58.9, 51.9, 50.6, 32.9, 28.3, 28.1; MS, *m*/*z* (%): 370 (M^+, 75), 286 (100), 259 (44), 83 (25).

*4.2.6. Ethyl-2′-amino-7′,7′-dimethyl-2,5′-dioxo-5′,6′,7′,8′-tetrahydro-2H-spiro[acenaphthylene-1,4′-chromene]-3′-carboxylate (**4f**).* White powder (0.33 g, 80%). Mp 259–262 °C. Found: C, 71.63; H, 5.80; N, 3.40. $C_{25}H_{23}NO_5$ requires C, 71.93; H, 5.55; N, 3.36%; ν_{max} (KBr) 3379, 3269, 2958, 1719, 1688, 1520 cm^{-1}; δ_H (500 MHz, DMSO-d_6) 8.18–7.31 (6H, m, ArH), 8.01 (2H, s, NH_2), 3.36 (2H, q, *J* 3.3, 7.3 Hz, −CH_2Me), 2.71 (1H, d, *J* 17.6, CH_aH$_b$), 2.61 (1H, d, *J* 17.6 Hz, CH$_a$$H_b$), 2.14 (1H, d, *J* 16.0 Hz, C$H_{a'}$H$_{b'}$), 1.99 (1H, d, *J* 16.0 Hz, CH$_{a'}$$H_{b'}$), 1.09 (3H, s, *Me*), 1.02 (3H, s, *Me*), 0.04 (3H, t, *J* 7.1 Hz, −CH$_2$*Me*); δ_C (125 MHz, DMSO-d_6) 206.0, 196.1, 168.3, 163.6, 160.3, 146.2, 141.5, 136.9, 130.3, 130.1, 129.1, 128.6, 124.6, 120.0, 119.8, 115.7, 78.1, 68.5, 59.3, 51.6, 50.9, 32.5, 28.6, 27.6, 13.1; MS, *m*/*z* (%): 417 (M^+, 20), 344 (100), 271 (28), 83 (25).

*4.2.7. 3′-Amino-2,6′,8′-trioxo-5′,6′,7′,8′-tetrahydro-2H-spiro[acenaphthylene-1,1′-pyrano[2,3-d]pyrimidine]-2-carbonitrile (**4g**).* Light yellow powder (0.21 g, 60%). Mp >300 °C. Found: C, 63.31; H, 2.89; N, 15.56. $C_{19}H_{10}N_4O_4$ requires C, 63.69; H, 2.81; N, 15.64%; ν_{max} (KBr) 3407, 3134, 2983, 2682, 2195, 1687, 1579 cm^{-1}; δ_H (500 MHz, DMSO-d_6) 9.63 (1H, s, N*H*), 8.39–7.33 (6H, m, ArH), 8.18 (2H, s, NH_2), 4.71 (1H, s, N*H*); δ_C (125 MHz, DMSO-d_6) 203.6, 167.9, 166.9, 165.4, 159.7, 145.0, 141.9, 133.9, 132.9, 130.9, 129.9, 129.8, 125.8, 122.8, 118.7, 112.3, 79.0, 67.9, 58.4; MS, *m*/*z* (%): 358 (M^+, 10), 272 (100), 57 (35).

*4.2.8. 5′-Acetyl-2′-amino-6′-methyl-2-oxo-2H-spiro[acenaphthylene-1,4′-pyran]-3′-carbonitrile (**4h**).* Light yellow powder (0.21 g, 65%). Mp >300 °C. Found: C, 72.32; H, 4.31; N, 8.51. $C_{20}H_{14}N_2O_3$ requires C, 72.72; H, 4.27; N, 8.48%; ν_{max} (KBr) 3425, 2983, 2200, 1712, 1566 cm^{-1}; δ_H (500 MHz, DMSO-d_6) 8.48–7.54 (6H, m, ArH), 7.38 (2H, s, NH_2), 2.09 (3H, s, *Me*), 1.61 (3H, s, -CO*Me*); δ_C (125 MHz, DMSO-d_6) 204.4, 199.5, 163.2, 156.1, 141.4, 135.4, 133.6, 131.6, 130.1, 130.0, 129.0, 126.0, 124.0, 123.0, 119.9, 99.3, 60.8, 56.9, 29.8, 19.9; MS, *m*/*z* (%): 330 (M^+, 5), 287 (100), 271 (10), 43 (30).

*4.2.9. Ethyl-2′-amino-3′-cyano-6′-methyl-2-oxo-2H-spiro[acenaphthylene-1,4′-pyran]-5′-carboxylate (**4i**).* Light yellow powder (0.23 g, 65%). Mp >300 °C. Found: C, 70.01; H, 4.51; N, 7.81. $C_{21}H_{16}N_2O_4$ requires C, 69.99; H, 4.48; N, 7.77%; ν_{max} (KBr) 3440, 2983, 2223, 1719, 1623, 1573 cm^{-1}; δ_H (500 MHz, DMSO-d_6) 8.07–7.20 (6H, m, ArH), 8.04 (2H, s, NH_2), 3.38 (2H, q, *J* 7.0 Hz,

−CH_2Me), 2.38 (3H, s, *Me*), 1.17 (3H, t, *J* 7.0 Hz, −CH_2*Me*); δ_C (125 MHz, DMSO-d_6) 203.9, 168.2, 165.9, 156.0, 141.5, 135.5, 133.2, 132.4, 130.3, 130.1, 129.4, 129.3, 124.1, 121.7, 120.3, 99.9, 75.5, 59.9, 52.5, 19.0, 10.2; MS, *m*/*z* (%): 360 (M^+, 5), 287 (100), 272 (35), 83 (30).

*4.2.10. 2′-Amino-2-oxo-2H-spiro[acenaphthylene-1,4′-benzo[h]chromene]-3′-carbonitrile (**4j**).* Light yellow powder (0.26 g, 70%). Mp >300 °C. Found: C, 80.31; H, 3.58; N, 7.32. $C_{25}H_{14}N_2O_2$ requires C, 80.20; H, 3.77; N, 7.48%; ν_{max} (KBr) 3426, 3062, 2200, 1714, 1622, 1576 cm^{-1}; δ_H (500 MHz, DMSO-d_6) 8.18−7.25 (12H, m, ArH), 8.03 (2H, s, NH_2); δ_C (125 MHz, DMSO-d_6) 203.8, 163.5, 156.1, 142.9, 142.0, 141.8, 139.9, 138.6, 135.6, 133.5, 132.7, 131.5, 130.8, 130.3, 129.9, 127.8, 126.8, 125.8, 123.7, 123.4, 122.2, 119.7, 98.2, 56.8, 52.6; MS, *m*/*z* (%): 374 (M^+, 5), 353 (10), 236 (15), 144 (20), 86 (100).

*4.2.11. 3′-Amino-2,9′-dioxo-2H,9′H-spiro[acenaphthylene-1,1′-indeno[1,2-b]pyran]-2′-carbonitrile (**4k**).* Light yellow powder (0.24 g, 65%). Mp >300 °C. Found: C, 76.28; H, 3.13; N, 7.43. $C_{24}H_{12}N_2O_3$ requires C, 76.59; H, 3.21; N, 7.44%; ν_{max} (KBr) 3419, 2980, 2204, 1706, 1568 cm^{-1}; δ_H (500 MHz, DMSO-d_6) 8.23−7.35 (10H, m, ArH), 8.21 (2H, s, NH_2); δ_C (125 MHz, DMSO-d_6) 202.9, 188.8, 168.4, 163.4, 146.2, 145.6, 144.4, 141.4, 141.3, 138.3, 135.5, 133.7, 132.7, 132.6, 132.0, 131.1, 130.6, 130.1, 129.4, 125.4, 123.6, 122.6, 66.5, 65.1; MS, *m*/*z* (%): 376 (M^+, 5), 324 (30), 176 (40), 57 (80).

*4.2.12. Ethyl-3′-amino-2,9′-dioxo-2H,9′H-spiro[acenaphthylene-1,1′-indeno[1,2-b]pyran]-2′-carboxylate (**4l**).* Light yellow powder (0.17 g, 40%). Mp >300 °C. Found: C, 73.08; H, 4.13; N, 3.33. $C_{26}H_{17}NO_5$ requires C, 73.75; H, 4.05; N, 3.31%; ν_{max} (KBr) 3423, 3053, 2979, 2187, 1709, 1672 cm^{-1}; δ_H (500 MHz, DMSO-d_6) 8.27−7.20 (10H, m, ArH), 8.19 (2H, s, NH_2), 3.36 (2H, q, *J* 7.1 Hz, −OCH_2Me), 1.04 (3H, t, *J* 7.1 Hz, −OCH_2*Me*); δ_C (125 MHz, DMSO-d_6) 202.6, 188.6, 170.4, 168.4, 163.4, 144.2, 144.1, 142.0, 141.4, 138.4, 135.4, 133.0, 132.6, 132.3, 131.3, 131.2, 130.1, 130.0, 129.0, 125.0, 123.9, 122.9, 95.4, 70.1, 66.5, 14.0; MS, *m*/*z* (%): 423 (M^+, 10), 350 (15), 298 (10), 97 (40).

*4.2.13. 2′-Amino-2,5′-dioxo-2H,5′H-spiro[acenaphthylene-1,4′-pyrano[3,2-c]chromene]-3′-carbonitrile (**4m**).* Light yellow powder (0.23 g, 60%). Mp >300 °C. Found: C, 73.10; H, 3.21; N, 7.11. $C_{24}H_{12}N_2O_4$ requires C, 73.47; H, 3.08; N, 7.14%; ν_{max} (KBr) 3424, 2978, 2987, 2199, 1723, 1607, 1577 cm^{-1}; δ_H (500 MHz, DMSO-d_6) 8.33−7.60 (10H, m, ArH), 7.33 (2H, s, NH_2); δ_C (125 MHz, DMSO-d_6) 203.6, 167.91, 166.9, 165.4, 159.7, 145.0, 141.9, 136.5, 133.9, 132.9, 130.9, 130.0, 129.9, 125.8, 122.8, 120.8, 119.2, 118.7, 115.6, 112.3, 98.0, 79.0, 67.9, 58.4; MS, *m*/*z* (%): 392 (M^+, 5), 340 (5), 151 (25).

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Supplementary data

Supplementary data associated with this article can be found in online version at doi:10.1016/j.tet.2010.05.067.

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