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One-pot three-component synthesis of functionalized spirolactones by means of reaction between aromatic ketones, dimethyl acetylenedicarboxylate, and *N*-heterocycles

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ABSTRACT

A simple and convenient one-pot multi-component reaction has been described for the synthesis of functionalized spirolactones. This strategy demonstrated three-component reaction between aromatic ketones (11*H*-indeno[1,2-*b*]quinoxalin-11-one) and dimethyl acetylenedicarboxylate (DMAD) in the presence of *N*-heterocycles, such as pyridine, quinoline, and isoquinoline in CH_2Cl_2 at ambient temperature without use of any catalyst or activator.

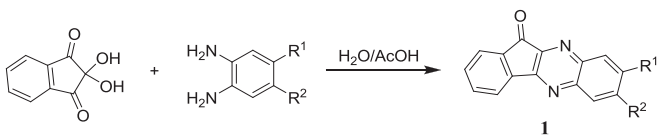
1. Introduction

The development of simple synthesis routes for widely used organic compounds using readily available reagents is one of the most important tasks in organic chemistry.[1] Heterocyclic systems with oxygen, nitrogen, sulfur, and other heteroatoms in five and six-membered rings are of interest due to their pharmaceutical and biological activities, such as anti-inflammatory, cardiotonic, inotropic, antihypertensive, antimicrobial, and antibacterial properties.[2–9] In this respect, spiro compound and specially spirolactone derivatives have been reported as anti-convulsants[10] and antitumorals.[11,12] As a results, a number methods have been describing novel synthesis for spiro compounds.[13–23]

In continuation of our work on multi-component reactions,[24–29] we now describe a one-pot, three-component synthesis of new functionalized spirolactones **4** from reaction between aromatic ketones **1**, dimethyl acetylenedicarboxylate (DMAD) **2** and *N*-heterocycles **3**, such as pyridine, quinoline, and isoquinoline in high to excellent yields.

2. Results and discussion

First, the aromatic ketones (11*H*-indeno[1,2-*b*]quinoxalin-11-one) **1** were synthesized according to previous work,[30] by means of reaction between ninhydrin (2,2-dihydroxyindane-1,3-dione) and 1,2-diaminobenzene derivatives (Scheme 1).



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Scheme 1. Synthesis of 11*H*-indeno[1,2-*b*]quinoxalin-11-one derivatives.[30]

The reaction of aromatic ketones **1** and DMAD **2** in the presence of *N*-heterocycles **3** proceeds smoothly in CH_2Cl_2 at ambient temperature to produce spirolactones **4** in high to excellent yield (Scheme 2). The results are summarized in Table 1. It is noteworthy that the reaction in the presence of pyridine and/or isoquinoline (as a *N*-heterocycle) generates only one diastereoisomer, while with quinoline produce two diastereoisomers. Our attempt to separate these diastereoisomers was unsuccessful (Scheme 3).

The structures of the products **4a**–**f** were deduced by IR, ^{1}H NMR, and ^{13}C NMR spectroscopy. The mass spectra of these

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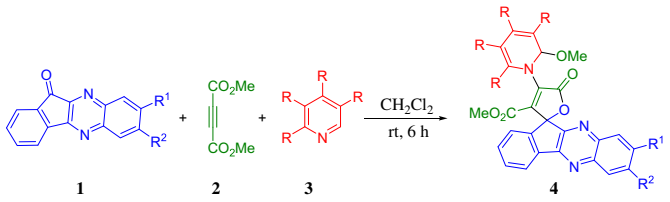
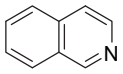
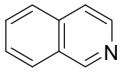
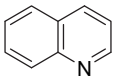
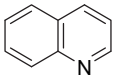
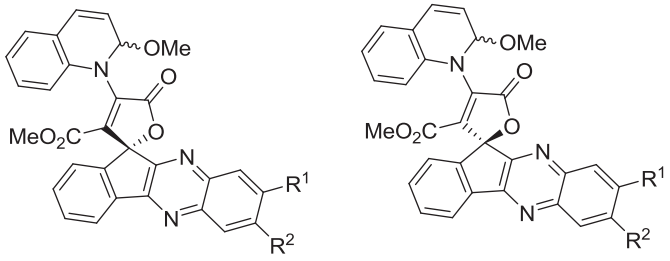


Scheme 2. Synthesis of spirolactone **4**.

Table 1
Synthesis of spirolactone **4** in the presence of various *N*-Heterocycle compounds

Entry	R^1	R^2	*N*-heterocycle	Product	Yield[a] (%)
1	H	H		**4a**	91
2	Me	Me		**4b**	89
3	H	H		**4c**	90
4	Me	Me		**4d**	87
5	H	H		**4e**	84
6	Me	Me		**4f**	81

[a] Isolated yields.



Scheme 3. Two diastereoisomers of spirolactones **4e** and **4f**.

compounds displayed molecular ion peaks at the appropriate *m*/*z* values and initial fragmentation involves the loss of the side chains. For example, the ^{1}H NMR spectrum of **4a** exhibited two singlets at δ 3.16 and 4.00 ppm for methoxy groups. The hemiaminal proton of **4a** was observed at δ 6.46 ppm as doublet with coupling constant ^3J=7.5 Hz. The other dihydropyridine protons and the aromatic protons resonance appeared as mixture of singlets, doublets, triplets or multiplets around δ 5.36–8.16 ppm. The ^{13}C NMR spectrum of compound **4a** showed 26 distinct resonances consistent with the spirolactone structure. The characteristic signal for the spiro carbon was observed at δ 79.4 ppm.

Although the mechanism of this reaction has not been established experimentally, the proposed mechanism for the formation of these spirolactones **4** is illustrated in Scheme 4. It is reasonable to assume that the spirolactone **4** results from the initial formation of a 1,3-dipolar intermediate **5** between *N*-heterocycle **3** and DMAD **2**, which reacts with the carbonyl group of aromatic ketone **1** for generating intermediate **6**. Cyclization of the intermediate **6** leads to the intermediate **7**, next the reaction between intermediate **7** and methoxy anion generate the spirolactone **4**.

3. Conclusion

In summary, we demonstrated an effective one-pot three-component approach for the synthesis of spirolactone derivatives via the reaction between the aromatic ketones (11*H*-indeno[1,2-*b*] quinoxalin-11-one), DMAD and *N*-heterocycles, such as pyridine, quinoline, and isoquinoline in CH_2Cl_2 at ambient temperature in high to excellent yields. The present procedure has the advantage that, not only is the reaction performed under neutral conditions, but also the substances can be mixed without any activation or modification.

4. Experimental

4.1. General

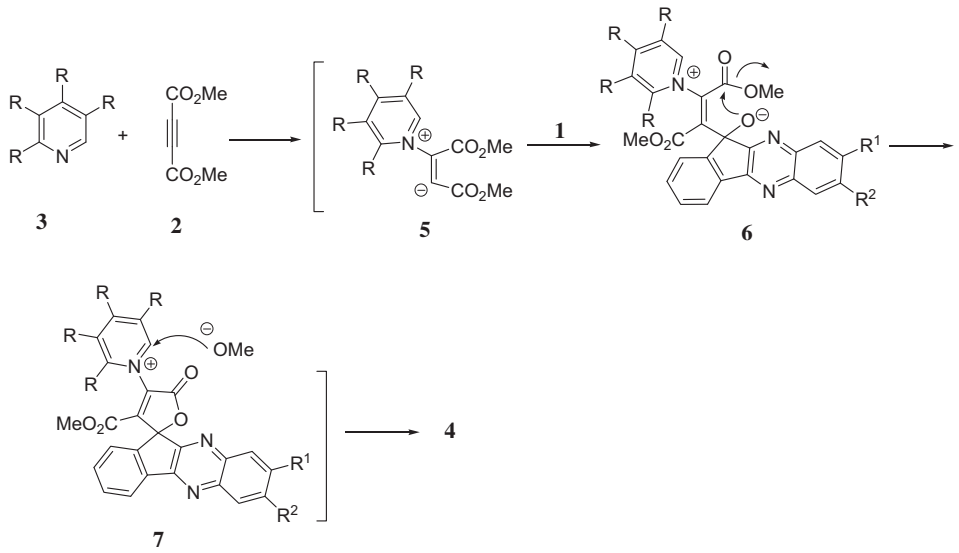
Melting points and IR spectra were taken on an Electrothermal 9100 apparatus and a JASCO FT-IR-460 plus spectrometer, respectively. Elemental analyses for C, H, and N were performed using a Heraeus CHN-O-Rapid analyzer at Iranian Central Research of Petroleum Company. The ^{1}H and ^{13}C NMR spectra were recorded on a Bruker (Rheinstetten, Germany) DRX-250 and 300 Avanve instrument with $CDCl_3$ as solvent and using TMS as internal reference at 250, 300, 62.9, and 75 MHz, respectively. The mass spectra were recorded on a shimadzu GC–MS-QP5050 mass spectrometer, operating at an ionization potential of 70 eV. All reagents and solvent were obtained from Merck (Darmastadt, Germany), Acros (Geel, Belgium), and Fluka (Buchs, Switzerland), and use without further purifications.

4.2. General procedure for synthesis of spirolactone 4

To a stirred solution of aromatic ketone **1** (1 mmol) and *N*-heterocycle **3** (1 mmol) in CH_2Cl_2 (10 mL) was added dropwise a mixture of DMAD **2** (1 mmol) in CH_2Cl_2 (2 mL) at ambient temperature over 15 min. The mixture was allowed to stir for 6 h. After completion of the reaction (monitored by TLC), the solvent was removed under reduced pressure and the residue was washed with cold ethanol to afford pure product **4**.

*4.2.1. Methyl 4-(2-methoxypyridin-1(2H)-yl)-5-oxo-5H-spiro[furan-2,11′-indeno[1,2-b]quinoxaline]-3-carboxylate (**4a**).* Yellow powder, yield 0.413 g (91%). [Found: C, 69.08; H, 4.26; N, 9.33. $C_{26}H_{19}N_3O_5$ requires C, 68.87; H, 4.22; N, 9.27%]; R_f (50% EtOAc/hexane) 0.54; mp: 167–169 °C. IR (KBr) (λ_{max}, cm^{-1}): 2945, 3057 (CH), 1700 and 1735 (C=O). ^{1}H NMR (250 MHz, $CDCl_3$): 3.16 (3H, s, OCH_3), 4.00 (3H, s, OCH_3), 5.36 (1H, m, $CH_{dihydropyridine}$), 5.53 (1H, m, $CH_{dihydropyridine}$), 6.24 (1H, m, $CH_{dihydropyridine}$), 6.46 (1H, d, J=7.5, $CH_{dihydropyridine}$), 7.01 (H, s, CH), 7.46 (5H, m, 5CH), 8.07 (1H, d, J=7.5, $CH_{dihydropyridine}$), 8.16 (2H, m, 2CH). ^{13}C NMR (62.9 MHz, $CDCl_3$): 51.4, 53.3 ($2OCH_3$), 79.4 (C_{spiro}), 80.0, 101.6 (2CH), 107.4 (C), 116.5, 123.3, 123.6, 124.8, 125.0, 128.8, 129.2, 129.8, 130.3 (9CH), 132.0, 138.4 (2C), 142.2 (CH), 142.7 (C), 145.7 (CH), 146.5, 147.2, 154.0, 161.7 (4C), 163.6, 163.8 (2C=O). MS (*m*/*z*, %): 453 (M^+, 29), 421 (5), 394 (16), 362 (100).

*4.2.2. Methyl 4-(2-methoxypyridin-1(2H)-yl)-7′,8′-dimethyl-5-oxo-5H-spiro[furan-2,11′-indeno[1,2-b]quinoxaline]-3-carboxylate (**4b**).* Yellow powder, yield 0.430 g (89%). [Found: C, 69.99; H, 4.80; N, 8.78. $C_{28}H_{23}N_3O_5$ requires C, 69.84; H, 4.81; N, 8.73%]; R_f (50% EtOAc/hexane) 0.61; mp: 169–172 °C. IR (KBr) (λ_{max}, cm^{-1}): 2951 (CH), 1709 and 1739 (C=O). ^{1}H NMR (250 MHz, $CDCl_3$): 2.48 (6H, s,



Scheme 4. The proposed mechanism for synthesis of spirolactone **4**.

2CH_3), 3.13 (3H, s, OCH_3), 4.00 (3H, s, OCH_3), 5.35 (1H, m, $CH_{dihydropyridine}$), 5.51 (1H, m, $CH_{dihydropyridine}$), 6.22 (1H, m, $CH_{dihydropyridine}$), 6.46 (1H, d, J=7.25, $CH_{dihydropyridine}$), 7.00 (1H, s, CH), 7.49 (3H, m, 3CH), 7.82 (1H, s, CH), 7.88 (1H, s, CH), 8.11 (1H, d, J=6.5, $CH_{dihydropyridine}$). ^{13}C NMR (62.9 MHz, $CDCl_3$): 20.1, 20.2 (2CH_3), 51.4, 53.3 (2OCH_3), 79.5 (C_{spiro}), 79.9, 101.4 (2CH), 107.5 (C), 116.5 (CH), 121.3 (C), 122.0 (CH), 123.0 (C), 124.9, 125.0, 128.5 (3CH), 129.1 (C), 130.1 (CH), 131.5 (C), 139.1, 140.1 (2CH), 141.1, 145.3 (2C), 147.0 (CH), 150.9, 153.0, 160.8 (3C), 163.6, 163.8 (2C=O). MS (*m*/*z*, %): 481 (M^+, 11), 449 (3), 422 (4), 389 (39), 312 (13), 128 (47), 77 (100).

*4.2.3. Methyl 4-[1-methoxyisoquinolin-2(1H)-yl]-5-oxo-5H-spiro [furan-2,11′-indeno[1,2-b]quinoxaline]-3-carboxylate (**4c**).* Yellow powder, yield 0.454 g (90%). [Found: C, 71.67; H, 4.18; N, 8.31. $C_{30}H_{21}N_3O_5$ requires C, 71.56; H, 4.20; N, 8.35%]; R_f (50% EtOAc/hexane) 0.58; mp: 190–193 °C. IR (KBr) (λ_{max}, cm^{-1}): 2949 (CH), 1708 and 1734 (C=O). ^{1}H NMR (250 MHz, $CDCl_3$): 3.19 (3H, s, OCH_3), 4.03 (3H, s, OCH_3), 5.87 (1H, d, J=7.5, $CH_{dihydroisoquinoline}$), 6.51 (1H, d, J=7.5, $CH_{dihydroisoquinoline}$), 7.02 (1H, s, CH), 7.11 (3H, m, 3CH), 7.56 (6H, m, 6CH), 8.17 (3H, m, 3CH). ^{13}C NMR (62.9 MHz, $CDCl_3$): 51.5, 53.4 (2OCH_3), 79.9 (C_{spiro}), 80.8, 105.1 (2CH), 122.6 (C), 123.4, 123.9, 125.2 (3CH), 126.3 (CH), 127.0, 127.8 (2CH), 128.9 (C), 129.0, 129.4 (2CH), 129.7 (C), 129.8, 130.0, 130.4 (3CH), 132.2, 137.9 (2C), 141.3 (CH), 142.2, 145.5 (2C), 147.4 (CH), 153.9, 162.2 (2C), 163.7, 163.9 (2C=O). MS (*m*/*z*, %): 503 (M^+, 2), 444 (10), 412 (13), 128 (14), 42 (100).

*4.2.4. Methyl 4-(1-methoxyisoquinolin-2(1H)-yl)-7′,8′-dimethyl-5-oxo-5H-spiro[furan-2,11′-indeno [1,2-b]quinoxaline]-3-carboxylate (**4d**).* Yellow powder, yield 0.463 g (87%). [Found: C, 72.55; H, 4.77; N, 7.93. $C_{30}H_{21}N_3O_5$ requires C, 72.30; H, 4.74; N, 7.91%]; R_f (50% EtOAc/hexane) 0.63; mp: 205–207 °C. IR (KBr) (λ_{max}, cm^{-1}): 2950, 3037 (CH), 1703 and 1737 (C=O). ^{1}H NMR (250 MHz, $CDCl_3$): 2.50 (3H, s, CH_3), 2.52 (3H, s, CH_3), 3.16 (3H, s, OCH_3), 4.03 (3H, s, OCH_3), 4.86 (1H, d, J=7.5, $CH_{dihydroisoquinoline}$), 6.50 (1H, d, J=7.5, $CH_{dihydroisoquinoline}$), 7.01 (1H, s, CH), 7.14 (3H, m, 3CH), 7.7 (4H, m, 4H), 7.87 (1H, s, CH), 8.00 (1H, s, CH), 8.27 (1H, m, CH). ^{13}C NMR (62.9 MHz, $CDCl_3$): 20.2, 20.3 (2CH_3), 51.4, 53.4 (2OCH_3), 80.0 (C_{spiro}), 80.8, 105.1, 123.4, 123.9, 125.2 (5CH), 126.3 (C) 127.0, 127.7, 129.2, 129.4 (4CH), 129.7, 130.5, 132.4 (3C), 139.9, 140.3, 145.5 (3CH), 147.5, 155.0, 161.6 (3C), 163.7, 163.9 (2C=O). MS (*m*/*z*, %): 530 (M^+–H, 11), 499 (3), 472 (5), 128 (100), 76 (30).

*4.2.5. Methyl 4-(2-methoxyquinolin-1(2H)-yl)-5-oxo-5H-spiro[furan-2,11′-indeno[1,2-b]quinoxaline]-3-carboxylate (**4e**).* Yellow powder, yield 0.424 g (84%). [Found: C, 71.78; H, 4.25; N, 8.33. $C_{30}H_{21}N_3O_5$ requires C, 71.56; H, 4.20; N, 8.35%]; mp: 216–220 °C. IR (KBr) (λ_{max}, cm^{-1}): 3064 and 2951 (CH), 1714 and 1740 (C=O). *NMR data for the major isomer* (55%). ^{1}H NMR (300 MHz, $CDCl_3$): 3.26 (3H, s, OCH_3), 3.92 (3H, s, OCH_3), 5.94 (1H, m, $CH_{dihydroquinoline}$), 6.50 (1H, d, J=4.3, $CH_{dihydroquinoline}$), 6.87 (1H, d, J=9.7, $CH_{dihydroquinoline}$), 7.08 (2H, m, 2CH), 7.26–8.24 (10H, m, 10CH). ^{13}C NMR (75 MHz, $CDCl_3$): 52.0, 53.2 (2OCH3), 79.9 (CH), 81.0 (C_{spiro}), 114.3, 118.5 (2CH), 121.7 (C), 122.5, 122.5, 123.7, 124.7, 128.7, 129.6, 129.7, 129.8, 130.2, 131.6, 132.5 (11CH), 135.8 (C), 136.8 (CH), 141.4, 142.1, 142.1, 142.6, 143.6, 146.4, 153.8, 161.2 (8C), 163.7, 164.4 (2C=O). *NMR data for the minor isomer* (45%). ^{1}H NMR (300 MHz, $CDCl_3$): 3.24 (3H, s, OCH_3), 3.89 (3H, s, OCH_3), 5.94 (1H, m, $CH_{dihydroquinoline}$), 5.99 (1H, d, J=4.3, $CH_{dihydroquinoline}$), 6.83 (1H, d, J=9.7, $CH_{dihydroquinoline}$), 7.08 (2H, m, 2CH), 7.26–8.24 (10H, m, 10CH). ^{13}C NMR (75 MHz, $CDCl_3$): 52.0, 53.2 (2OCH_3), 79.9 (CH), 81.0 (C_{spiro}), 115.3, 117.9 (2CH), 121.2 (C), 122.1, 122.7, 124.3, 128.6, 128.9, 129.1, 129.2, 129.9, 130.4, 130.6, 132.1, 132.4 (12CH), 138.1, 140.8, 141.4, 142.1, 143.2, 144.6, 147.1, 156.45, 161.2 (9C), 164.1, 164.7 (2C=O). MS: (*m*/*z*, %): 503 (M^+, 9), 443 (7), 412 (4), 314 (9), 128 (100).

*4.2.6. Methyl 4-(2-methoxyquinolin-1(2H)-yl)-7′,8′-dimethyl-5-oxo-5H-spiro[furan-2,11′-indeno[1,2-b]quinoxaline]-3-carboxylate (**4f**).* Yellow powder, yield 0.453 g (85%). [Found: C, 72.59; H, 4.80; N, 7.96. $C_{30}H_{21}N_3O_5$ requires C, 72.30; H, 4.74; N, 7.91%]; mp: 217–220 °C. IR (KBr) (λ_{max}, cm^{-1}): 2920, 2920 (CH), 1721 and 1741 (C=O). *NMR data for the major isomer* (60%). ^{1}H NMR (300 MHz, $CDCl_3$): 2.49 (6H, s, 2CH_3), 3.23 (3H, s, OCH_3), 3.90 (3H, s, OCH_3), 5.93 (1H, m, $CH_{dihydroquinoline}$), 6.49 (1H, d, J=4.4, $CH_{dihydroquinoline}$), 6.85 (1H, d, J=9.5, $CH_{dihydroquinoline}$), 7.03–8.11 (10H, m, 10CH). ^{13}C NMR (75 MHz, $CDCl_3$): 20.2, 20.5 (2CH_3), 51.9, 53.2 (2OCH_3), 79.8 (CH), 81.0 (C_{spiro}), 114.3, 118.5, 122.1 (3CH), 122.6 (C) 124.5, 129.0, 129.2, 129.7, 130.7, 132.0 (6CH), 136.0, 136.1, 136.4 (3C), 136.5 (CH), 139.5 (C), 140.9 (CH) 141.3, 14.5, 141.7, 141.9 (4C), 143.5 (CH), 146.3, 148.3, 156.1 (3C), 163.7, 164.4 (2C=O). *NMR data for the minor isomer* (40%). ^{1}H NMR (300 MHz, $CDCl_3$): 2.51 (6H, s, 2CH_3), 3.20 (3H, s, OCH_3), 3.87 (3H, s, OCH_3), 5.93 (1H, m, $CH_{dihydroquinoline}$), 5.99 (1H, d, J=4.4, $CH_{dihydroquinoline}$), 6.82 (H, d, J=9.5, $CH_{dihydroquinoline}$), 7.03–8.11 (10H, m, 10CH). ^{13}C NMR (75 MHz, $CDCl_3$): 20.2, 20.3 (2CH_3), 51.9, 53.2 (2OCH_3), 79.8 (CH),

81.0 (C_{spiro}), 115.2, 117.9, 121.7, 121.2, 122.3 (5CH), 122.6 (C), 123.6, 124.2, 128.3, 128.6, 129.6, 129.6, 130.4 (7CH), 136.0, 136.1, 136.4, 139.5, 141.3, 141.5, 141.7, 141.9, 146.3, 148.3, 156.13 (11C), 163.7, 164.4 (2C=O). MS (*m*/*z*, %): 531 (M^+, 7), 439 (12), 343 (10), 284 (14), 128 (100).

Acknowledgements

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