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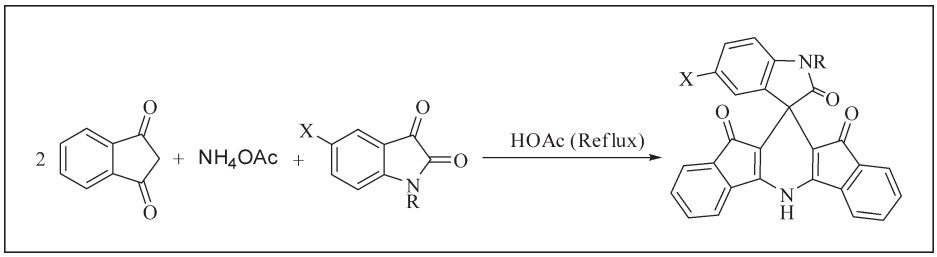

A one-pot and pseudo four-component synthesis of spiro[diindeno[1,2-*b*:2′,1′-*e*]pyridine-11,3′-indoline]-trione derivatives by cyclo-condensation reaction of isatins, 1,3-indandione, and ammonium acetate in refluxing acetic acid is reported.

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INTRODUCTION

Multicomponent reactions (MCRs) have been frequently used by synthetic chemists as a facile means to generate molecular diversity from bifunctional substrates that react sequentially in an intramolecular fashion [1,2]. Devising such types of MCRs that achieve the formation of multiple bonds in a single operation is one of the major challenges in modern organic synthesis [3,4]. As such processes avoid time consuming and costly purification processes, as well as protection–deprotection steps, they are inherently more environmentally benign and atom economic [5]. They provide a powerful tool toward the one-pot synthesis of diverse and complex compounds as well as small and drug-like heterocycles [6].

Indenone-fused heterocycles represent important biological and medicinal scaffolds. Thus, the indenopyridine skeleton is present in the 4-azafluorenone group of alkaloids, represented by its simplest member onychnine (Fig. 1) [7]. Indenopyrazoles (**A**) and indenopyridazines (**B**) have been investigated as cyclin-dependent kinase [8] and selective monoamine oxidase B (MAO-B) [9] inhibitors, respectively.

Further, indenopyridines (**C**) exhibit cytotoxic [10], phosphodiesterase inhibitory [11], adenosine A2a receptor antagonistic [12], anti-inflammatory/antiallergic [13], coronary dilating [14], and calcium modulating activities [15]. These compounds have also been investigated for the treatment of hyperlipoproteinemia and arteriosclerosis [16] as well as neurodegenerative diseases [17].

Indole and indoline fragments are important moieties of a large number of a variety of natural products and medicinal agents [18], and some of indolines, spiro-annulated with heterocycles in the 3-position, have shown high biological activity [19–21]. The spirooxindole system is the core structure of many pharmacological agents and natural alkaloids [22–24]. Therefore, a number of methods have been reported for the preparation of spirooxindole fused heterocycles [25].

As part of our continuing efforts on the synthesis of biologically active heterocyclic compounds [26], we recently described an efficient synthesis of spiropyrimidoquinoline-pyrrolopyrimidines and spiroindoline-pyridodipyrimidines *via* a condensation reaction between amino-uraciles and isatines [27]. We have also developed an efficient synthesis of spiro[dibenzo[*b*,*i*]xanthene-13,3′-indoline]-pentaones via a reaction of isatins and 2-hydroxy-naphthoquinone in water [28].

Considering the important biological properties of spirooxindole fused heterocycles, we report herein a one-pot, pseudo four-component synthesis of spiro[diindeno[1,2-*b*:2′,1′-*e*]pyridine-11,3′-indoline]-triones 4 through a one-pot condensation reaction of 1,3-indandione 1, ammonium acetate 2 and isatins 3 in refluxing acetic acid (Scheme 1).

RESULTS AND DISCUSSION

In a pilot experiment, a mixture of 1,3-indandione **1**, ammonium acetate **2**, and isatin **3a** at refluxing acetic acid were stirred to afford the 5*H*-spiro[diindeno[1,2-*b*:2′,1′-*e*]pyridine-11,3′-indoline]-2′,10,12-trione **4a** in 87% for 4 h.

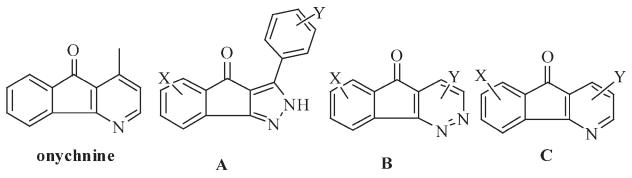


Figure 1. Representatives of important indenone-fused heterocycles

Encouraged by this success, we extended this reaction of 1,3-indandione **1** and ammonium acetate **2** with a range of other isatins **2b-l** under similar conditions, furnishing the respective 5*H*-spiro[diindeno[1,2-*b*:2′,1′-*e*]pyridine-11,3′-indoline]-2′,10,12-triones **4b-l** in good yields. The optimized results are summarized in Table 1. We have shown that the use of a wide diversity of substituents in isatins **3** in this reaction makes possible the synthesis of libraries under similar circumstances.

^{1}H and ^{13}C NMR spectra of the crude products clearly indicated the formation of spirooxindol fused diindenopyridines **4**. The nature of these compounds as 2:1:1 adducts was apparent from their mass spectra, which displayed, in each case, the molecular ion peak at appropriate m/z value. Compounds **4a–l** are stable solids whose structures were established by IR, ^{1}H and ^{13}C NMR spectroscopy and elemental analysis.

For the investigation of the reaction mechanism, it is notable that when the 1,3-indandione **1**, ammonium acetate **2**, and isatin **3a** were reacted for 2 h, the intermediate **6** were isolated and characterized by spectroscopic methods. When intermediate **6** was reacted with NH_4OAc **2** under the same reaction conditions, the product **4a** was obtained in 83% yield (Scheme 2).

Therefore, the formation of products **4** can be rationalized *via* initial addition of 1,3-indanedione **1** to the isatins **3** to yield intermediate **5**, which reacted further with another molecule of **1**. Finally, reaction of ammonium acetate **2** with the intermediate **6**, followed by cyclization afforded the corresponding product **4** (Scheme 3).

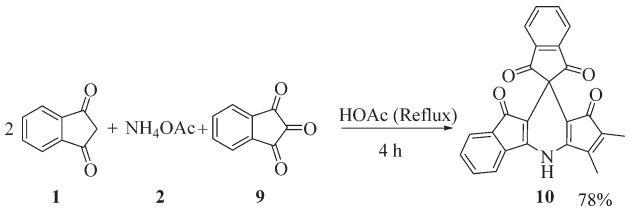
As expected, when the isatins **3** was replaced by acenaphthylene-1,2-dione **7**, 2*H*,5′*H*-spiro[acenaphthylene-1,11′-diindeno[1,2-*b*:2′,1′-*e*]pyridine]-2,10′,12′-trione **8** was obtained in 82% yield under the same reaction conditions (Scheme 4).

To further explore the potential of this protocol for spirofused heterocycle synthesis, we investigated reaction of 1,3-indandione **1** and ammonium acetate **2** with ninhydrine **9** and obtained 5*H*-spiro[diindeno[1,2-*b*:2′,1′-*e*]pyridine-11,2′-indene]-1′,3′,10,12-tetraone **10** in 78% yield (Scheme 3).

Table 1

Synthesis of spiro[diindenopyridine-indoline]-triones **4**.

Product **4**	R	X	Yield (%)
a	H	H	87
b	Me	H	85
c	Et	H	82
d	$PhCH_2$	H	80
e	H	Br	91
f	H	Me	88
g	H	F	79
h	H	NO_2	92
i	Me	Br	76
j	Et	Br	78
k	Me	NO_2	79
l	Et	NO_2	77

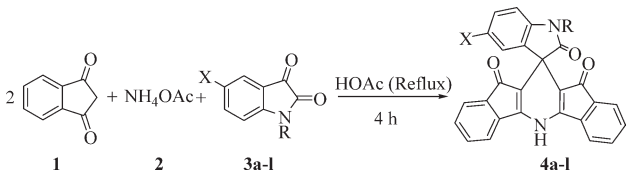


In conclusion, we have demonstrated an efficient and simple method for the preparation of some spirooxindole fused heterocycles using readily available starting materials. Prominent among the advantages of this new method are operational simplicity, good yields, and easy work-up procedures employed. Moreover, it is worth noting that two C—C and one two C—N bonds were formed with concomitant creation of a spirooxindoles in this one-pot, pseudo four-component process.

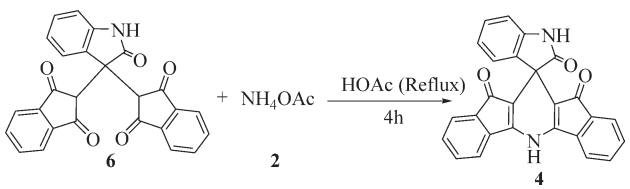
EXPERIMENTAL

Melting points were measured on an Elecrtothermal 9100 apparatus. Mass spectra were recorded on a FINNIGAN-MAT 8430 mass spectrometer operating at an ionization potential of 70 eV. ^{1}H and ^{13}C NMR spectra were recorded on a BRUKER DRX-300 AVANCE spectrometer at 300.13 and 75.47 MHz, respectively. IR spectra were recorded using a Shimadzu IR-470 apparatus. Elemental analyses were performed using a Heracus CHN-O-Rapid analyzer.

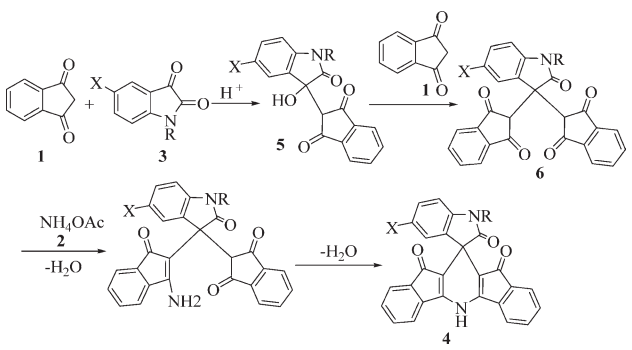
Scheme 1



Scheme 2



Scheme 3



Because of very low solubility of the products, we cannot report the ^{13}C NMR data for these products.

Typical procedure for the preparation of 5*H*-Spiro[diindeno[1,2-*b*:2′,1′-*e*]pyridine-11,3′-indoline]-2′,10,12-trione (4a). A mixture of 1,3-indandione **1a** (0.30 g, 2 mmol), ammonium acetate **2** (0.46 g, 3 mmol), and isatin **3a** (0.15 g, 1 mmol) in refluxing (5 mL) was stirred for 4 h (the progress of the reaction was monitored by TLC). After completion, the reaction mixture was filtered and the precipitate washed with water (10 mL) and recrystallized by EtOH to afford the pure product **4a** as red powder (87%); m.p >300°C (dec). IR (KBr) (ν_{max}/cm^{-1}): 3169, 2996, 1694, 1672, 1631. MS (EI, 70 eV) *m/z*: 402 (M^+). ^1H NMR (300 MHz, DMSO-d_6): δ_H 6.83–7.74 (m, 12H, ArH), 10.66 (s, 1H, NH), 11.62 (s, 1H, NH). Anal. Calcd for $C_{26}H_{14}N_2O_3$: C, 77.60; H, 3.51; N, 6.96%. Found: C, 77.51; H, 3.45; N, 6.88%.

1′-Methyl-5*H*-spiro[diindeno[1,2-*b*:2′,1′-*e*]pyridine-11,3′-indoline]-2′,10,12-trione (4b). Dark red powder (85%); m.p > 300°C. IR (KBr) (ν_{max}/cm^{-1}): 2926, 1701, 1678, 1617. MS (EI, 70 eV) *m/z*: 416 (M^+). ^1H NMR (300 MHz, DMSO-d_6): δ_H 3.27 (s, 3H, NCH_3), 7.07–8.53 (m, 12H, ArH), 11.13 (s, 1H, NH). Anal. Calcd for $C_{27}H_{16}N_2O_3$: C, 77.87; H, 3.87; N, 6.73%. Found: C, 77.95; H, 3.80; N, 6.66%.

1′-Ethyl-5*H*-spiro[diindeno[1,2-*b*:2′,1′-*e*]pyridine-11,3′-indoline]-2′,10,12-trione (4c). Red powder (82%); m.p > 300°C. IR (KBr) (ν_{max}/cm^{-1}): 3219, 2921, 1707, 1652 . MS (EI, 70 eV) *m/z*: 430 (M^+). ^1H NMR (300 MHz, DMSO-d_6): δ_H 1.23–1.27 (m, 3H, CH_3), 3.78–3.80 (m, 2H, NCH_2), 6.90–7.80 (m, 12H, ArH), 11.63(1H, s, NH). Anal. Calcd for $C_{28}H_{18}N_2O_3$: C, 78.13; H, 4.21; N, 6.51%. Found: C, 78.01; H, 4.13; N, 6.62%.

1′-Benzyl-5*H*-spiro[diindeno[1,2-*b*:2′,1′-*e*]pyridine-11,3′-indoline]-2′,10,12-trione (4d). Dark red powder (80%); m.p = 270°C IR (KBr) (ν_{max}/cm^{-1}): 3048, 1706, 1666, 1607. MS (EI, 70 eV) *m/z*: 492 (M^+). ^1H NMR (300 MHz, DMSO-d_6): δ_H 5.00 (bs, 2H, NCH_2), 6.72–7.81 (m, 17H, ArH), 11.68 (s, 1H, NH). Anal. Calcd for $C_{33}H_{20}N_2O_3$: C, 80.47; H, 4.09; N, 5.69%. Found: C, 80.38; H, 4.01; N, 5.58%.

5′-Bromo-5*H*-spiro[diindeno[1,2-*b*:2′,1′-*e*]pyridine-11,3′-indoline]-2′,10,12-trione (4e). Red powder (91%); m.p > 300°C. IR (KBr) (ν_{max}/cm^{-1}): 3222, 2922, 1698, 1640, 1603. MS (EI, 70 eV) *m/z*: 482 (M^++2), 480 (M^+). Anal. ^1H NMR (300 MHz, DMSO-d_6): δ_H 6.84–7.94 (m, 11H, ArH), 10.78 (s, 1H, NH), 11.66 (s, 1H, NH). Calcd for $C_{26}H_{13}BrN_2O_3$: C, 64.88; H, 2.72; N, 5.82%. Found: C, 64.81; H, 2.78; N, 5.89%.

5′-Methyl-5*H*-spiro[diindeno[1,2-*b*:2′,1′-*e*]pyridine-11,3′-indoline]-2′,10,12-trione (4f). Red powder (88%); m.p > 300°C. IR (KBr) (ν_{max}/cm^{-1}): 3169, 2996, 1682, 1645. MS (EI, 70 eV) *m/z*: 416 (M^+). ^1H NMR (300 MHz, DMSO-d_6): δ_H 2.13 (s, 3H, CH_3), 6.72–7.76 (m, 11H, ArH), 10.53 (s, 1H, NH), 11.60 (s, 1H, NH). Anal. Calcd for $C_{27}H_{16}N_2O_3$: C, 77.87; H, 3.87; N, 6.73%. Found: C, 77.74; H, 3.77; N, 6.64%.

5′-Fluoro-5*H*-spiro[diindeno[1,2-*b*:2′,1′-*e*]pyridine-11,3′-indoline]-2′,10,12-trione (4g). Red powder (79%); m.p > 300°C. IR (KBr) (ν_{max}/cm^{-1}): 3048, 2901, 1681, 1640. MS (EI, 70 eV) *m/z*: 420 (M^+). ^1H NMR (300 MHz, DMSO-d_6): δ_H 6.81–7.80 (m, 11H, ArH), 10.63 (s, 1H, NH), 11.66 (s, 1H, NH). Anal. Calcd for $C_{26}H_{13}FN_2O_3$: C, 74.28; H, 3.12; N, 6.66%. Found: C, 74.37; H, 3.06; N, 6.60%.

5′-Nitro-5*H*-spiro[diindeno[1,2-*b*:2′,1′-*e*]pyridine-11,3′-indoline]-2′,10,12-trione (4h). Red powder (92%); m.p > 300°C. IR (KBr) (ν_{max}/cm^{-1}): 3211, 3048, 1706, 1631, 1600. MS (EI, 70 eV) *m/z*: 447 (M^+). ^1H NMR (300 MHz, DMSO-d_6): δ_H 7.07–8.15 (m, 11H, ArH), 11.36 (s, 1H, NH), 11.93 (s, 1H, NH). Anal. Calcd for $C_{26}H_{13}N_3O_5$: C, 69.80; H, 2.93; N, 9.39%. Found: C, 69.72; H, 2.86. N, 9.31%.

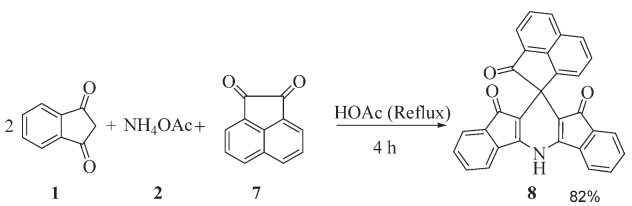
5′-Bromo-1′-methyl-5*H*-spiro[diindeno[1,2-*b*:2′,1′-*e*]pyridine-11,3′-indoline]-2′,10,12 -trione (4l). Dark red powder (76%); m.p > 300°C. IR (KBr) (ν_{max}/cm^{-1}): 2917, 1680, 1640, 1608. MS (EI, 70 eV) *m/z*: 496 (M^++2), 494 (M^+). ^1H NMR (300 MHz, DMSO-d_6): δ_H 3.21 (s, 3H, NCH3), 7.04-7.81 (m, 11H, ArH), 11.72 (s, 1H, NH). Anal. Calcd for $C_{27}H_{15}BrN_2O_3$: C, 65.47; H, 3.05; N, 5.66%. Found: C, 65.35; H, 3.14; N, 5.75%.

5′-Bromo-1′-ethyl-5*H*-spiro[diindeno[1,2-*b*:2′,1′-*e*]pyridine-11,3′-indoline]-2′,10,12-trione (4j). Red powder (78%); m.p > 300°C. IR (KBr) (ν_{max}/cm^{-1}): 2931, 1714, 1667. MS (EI, 70 eV) *m/z*: 510 (M^++2), 508 (M^+). ^1H NMR (300 MHz, DMSO-d_6): δ_H 1.21 (bs, 3H, CH_3), 3.78 (bs, 2H, NCH_2), 7.06–7.79 (m, 11H, ArH), 11.96 (s, 1H, NH). Anal. Calcd for $C_{28}H_{17}BrN_2O_3$: C, 66.03; H, 3.36; N, 5.50%. Found: C, 66.14; H, 3.30; N, 5.59%.

1′-Methyl-5′-nitro-5*H*-spiro[diindeno[1,2-*b*:2′,1′-*e*]pyridine-11,3′-indoline]-2′,10,12-trione (4k). Red powder (79%); m.p > 300°C. IR (KBr) (ν_{max}/cm^{-1}): 2996, 1693, 1608. MS (EI, 70 eV) *m/z*: 461 (M^+). ^1H NMR (300 MHz, DMSO-d_6): δ_H 3.33 (s, 3H, NCH3), 7.21–8.29 (m, 11H, ArH), 11.85(s, 1H, NH). Anal. Calcd for $C_{27}H_{15}N_3O_5$: C, 70.28; H, 3.28; N, 9.11%. Found: C, 70.19; H, 3.34; N, 9.04%.

1′-Ethyl-5′-nitro-5*H*-spiro[diindeno[1,2-*b*:2′,1′-*e*]pyridine-11,3′-indoline]-2′,10,12-trione (4l). Red powder (77%); m.p > 300°C. IR (KBr) (ν_{max}/cm^{-1}): 2964, 1692, 1645. MS (EI, 70 eV) *m/z*: 475 (M^+). ^1H NMR (300 MHz, DMSO-d_6): δ_H 1.26 (t, $^3J_{HH}$ = 6.9 Hz, 3H, CH_3), 3.90 (q, $^3J_{HH}$ = 6.6 Hz, 2H, NCH_2), 7.27–8.27 (m, 11H, ArH), 11.83 (s, 1H, NH). Anal. Calcd for $C_{28}H_{17}N_3O_5$: C, 70.73; H, 3.60; N, 8.84%. Found: C, 70.67; H, 3.55; N, 8.91%.

Scheme 4



2*H*,5′*H*-Spiro[acenaphthylene-1,11′-diindeno[1,2-*b*:2′,1′-*e*]pyridine]-2,10′,12′-trione (8). Dark red powder (82%); m.p > 270°C. IR (KBr) (ν_{max}/cm^{-1}): 3059, 1692, 1640. MS (EI, 70 eV) *m*/*z*: 437 (M^+). ^{1}H NMR (300 MHz, DMSO-d_6): δ_H 7.14–8.29 (m, 15H, ArH, NH). ^{13}C NMR (75 MHz, DMSO-d_6): δ_C (ppm) 51.5, 11.8, 120.5, 121.2, 121.3, 123.9, 125.0, 128.9, 129.3, 129.7, 130.2, 131.3, 132.9, 133.7, 1373.3, 137.9, 141.3, 143.9, 158.0, 190.5, 205.4. Anal. Calcd for $C_{30}H_{15}NO_3$: C, 82.37; H, 3.46; N, 3.20%. Found: C, 82.48; H, 3.38; N, 3.29%.

5*H*-Spiro[diindeno[1,2-*b*:2′,1′-*e*]pyridine-11,2′-indene]-1′,3′,10,12-tetraone (10). Dark red powder (78%); m.p > 260°C dec. IR (KBr) (ν_{max}/cm^{-1}): 2922, 1703, 1651. MS (EI, 70 eV) *m*/*z*: 415 (M^+). ^{1}H NMR (300 MHz, DMSO-d_6): δ_H 7.29–8.09 (m, 12H, ArH), 11.94 (s, 1H, NH). Anal. Calcd for $C_{27}H_{13}NO_4$: C, 78.07; H, 3.15; N, 3.37%. Found: C, 77.97; H, 3.09; N, 3.29%.

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