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ORIGINAL PAPER

One-pot of three-component synthesis of novel amino-spiroindene derivatives

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Abstract A facile and efficient one-pot three-component synthesis of the amino-spiroindene derivatives was achieved, via the reaction of ninhydrin, malononitrile or ethylcyanoacetate and various reagents including 1, 2- and 1,3-dicarbonyl compounds/enols.

Keywords Ninhydrin · Amino-spiroindene derivatives · Multi-component reactions

Introduction

In multi-component 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.

The development of MCRs designed to produce elaborate biologically active compounds has become an important area of research in organic, combinatorial, and medicinal chemistry [1–4]. The MCR strategy offers significant advantages over conventional linear-type synthesis due to its flexible, convergent, and atom efficient nature [5, 6].

Electronic supplementary material The online version of this article (doi:10.1007/s13738-013-0332-3) contains supplementary material, which is available to authorized users.

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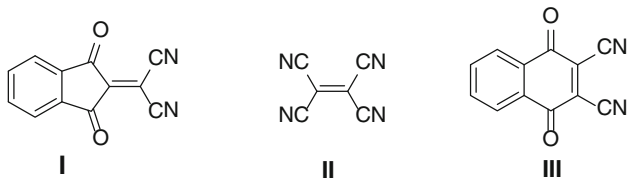
In recent years, the synthesis of combinatorial small-molecule heterocyclic libraries has emerged as a valuable tool in the search for novel lead structures [7, 8]. Thus, the success of combinatorial chemistry in the drug discovery process is considerably dependent on further advances in heterocyclic MCR methodology and, according to the current synthetic requirements; environmentally benign multi-component procedures are particularly welcome.

(1,3-Dioxo-2,3-dihydro-1H-inden-2-ylidene) propanedinitrile (**I**, also referred to as 2-(dicyanomethylene)-1,3-indanedione, Scheme 1) may be considered to be analogous to ethenetetracarbonitrile (**2**) in its reactions [9]. This compound can be considered as an intermediate for the synthesis of pyran rings.

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 [10, 11]. 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 [12]. Biological activities of spiro compounds containing pyrans have also been proved. They also show good activity as hypertensive agents [13]. They have been the subject of great interest as potential novel analgesic agents [14].

Spiro-cyclic systems containing one sp^3 carbon atom common to two rings are structurally interesting [15]. One of the important criteria of the biological activities of asymmetric structure spiro-molecule, is chiral spiro-carbon atom [16, 17]. The presence of the sterically constrained spiro structure in various natural products also adds to the interest in the investigations of spiro compounds [18].



Scheme 1 Carbonitrile derivatives: **a** 2-(1,3-dioxo-1H-inden-2(3H)-ylidene) malononitrile **I**; **b** ethene-1,1,2,2-tetracarbonitrile **II**; **c** 1,4-dioxo-1,4-dihydronaphthalene-2,3-dicarbonitrile **III**

The synthesis of heterocycles by MCRs often involves classic carbonyl condensation chemistry [19, 20] and basic catalysts play an important role in MCRs. Triethylamine is an efficient and inexpensive base and its efficiency as a basic catalyst has been explored frequently [21, 22].

As we were synthesizing new amino-spiroindene derivatives using 2-(dicyanomethylene)-1, 3-indanedione derivatives and cyclic ketone, 1, 2 and 1, 3-bicarbonyl, cumarin and 1-naphthol, we were interested in the synthesis of spiro and spiroindene rings and their properties. The reaction was carried out in ethanol or dioxin under reflux conditions. The results of ^{1}H- NMR, ^{13}C- NMR, FT-IR and elemental analysis confirmed the formation of these products.

Experimental

General

Chemicals and solvents such as EtOAc, EtOH, DMF, and MeOH obtained from Merck Chemical. Co. were used without further purification. Melting points were determined on a Melt-Tem II melting point apparatus and were uncorrected. IR spectra were obtained on a Matson-1000 FT-IR spectrometer. Peaks are reported in wave numbers (cm^{-1}). All of the NMR spectra were recorded on a Bruker model DRX-500 avance (^{1}H: 500 MHz) (^{13}C: 125 MHz) and Bruker model DRX-400 AVANCE (^{1}H: 400 MHz) (^{13}C: 100 MHz) NMR spectrometer. Chemical shifts are reported in parts per million (ppm) from tetramethylsilane (TMS) as an internal standard in DMSO-d_6 as a solvent. Elemental analyses (C, H, and N) were performed with a Heracus CHN-O-Rapid analyzer. Purity of the compounds is checked by thin layer chromatography (TLC) on Merck silica gel 60 F_{254} pre-coated sheets in *n*-Hexane/ethyl acetate mixture and spots were developed using either iodine or ultraviolet light as visualizing agent.

General procedure

A mixture of ninhydrin (2 mmol, 0.36 g), malononitrile (2 mmol, 0.13 g), or ethyl cyanoacetate (2 mmol, 0.23 g), in EtOH (10 ml) was stirred at 60 °C for 15 min. Then, the desired ketone (2 mmol) EtOH (4 ml) and piperidine (0.3 ml) were added to the mixture; upon completion, monitored by TLC (*n*-hexane/ethyl acetate), the reaction mixture was allowed to cool to room temperature. The solid was filtered off, washed with ether or ethanol several times, and was purified with EtOH to give the desired products.

2′-amino-1,3,5′-trioxo-1,3-dihydro-5′H-spiro[indene-2,4′indeno[1,2-b]pyran]-3′-carbonitrile (5a)

Brown powder, M.P = 335–339 °C. Yield = 70 %. IR KBr (υ_{max}, cm^{-1}): 3,395 (NH_2, broad), 3,045 (C–H aromatic), 2,194 (CN), 1,705, 1,680 (C=O). ^{1}H-NMR (DMSO-d_6, 500 MHz): δ (ppm) = 7.31 (1H, H-aromatic(1,3-indandione), d, J = 10.0 Hz), 7.37 (1H, H-aromatic(1,3-indandione), d, J = 10.0 Hz), 7.45 (1H, H-aromatic(1,3-indandione), t, J = 10 Hz), 7.57 (1H, H-aromatic(1,3-indandione), t, J = 10.0 Hz),7.93–8.11 (2H, NH_2), 8.13 (4H, H-aromatic) ppm. ^{13}C-NMR (DMSO-d_6, 125.77 MHz): δ (ppm) = 52.18 (1C, C–CN), 53.23 (1C, C-spiro), 105.57 (C–C=O), 116.81 (1C, CN), 119.17, 122.59, 122.92, 123.07, 123.77, 129.92, 131.73, 133.94, 134.60, 136.26, 136.52, 137.64, 140.63 (C-aromatic), 161.39 (1C, C–NH_2), 169.05 (1C, C–CO), 189.32 (1C, C=O(1,3-indandione)), 198.41 (2C, C=O).

2-amino-1′,3′,5-trioxo-1′,3′,5,6,7,8-hexahydrospiro [chromene-4,2′-indene]-3-carbonitrile (5b)

Yellow powder, M.P = 295–298 °C. Yield = 69 % IR KBr (υ_{max}, cm^{-1}): 3,401 (NH_2, broad), 3,085 (C–$H_{aromatic}$), 2,922 (C–$H_{aliphatic}$), 2,203 (CN), 1,711, 1,675 (C=O) cm^{-1}. ^{1}H-NMR (DMSO-d_6, 500 MHz): δ (ppm) = 1.94 (2H, CH_2, t, J = 5.0 Hz), 2.27 (2H, CH_2, t, J = 5.0 Hz), 2.69 (2H, CH_2, t, J = 4.0 Hz), 7.64 (2H, NH_2, s), 8.01 (4H, aromatic) ppm. ^{13}C-NMR (DMSO-d_6, 125.77 MHz): δ (ppm) = 19.81 (1C, CH_2), 26.37 (1C, CH_2), 35.27 (1C, CH_2), 51.89 (1C, C–CN), 53.18 (1C, C-spiro), 111.04 (1C, C=C spiro ring), 116.89 (1C, C–CN), 123.13 (2C, C-aromatic), 136.59 (2C, C-aromatic), 140.55 (2C, C-aromatic), 159.79 (1C, C–O), 168.31 (1C, C–NH_2),196.11 (2C, C=O), 199.76 (1C, C=O, 1,3-diketon).

2-amino-7,7-dimethyl-1′,3′,5-trioxo-1′,3′,5,6,7,8-hexahydrospiro [chromene-4,2′-indene]-3-carbonitrile (5c)

Brown powder, M.P = 297 °C. Yield = 63 %. IR KBr (υ_{max}, cm^{-1}): 3,398 (NH_2, broad), 3,058 (C–$H_{aromatic}$), 2,912 (C–$H_{aliphatic}$), 2,199 (CN), 1,713, 1,668 (C=O), cm^{-1}. ^{1}H-NMR (DMSO-d_6, 500 MHz): δ (ppm) = 1.05 (6H, CH_3), 2.21 (2H, CH_2, s), 2.63 (2H, CH_2, s), 7.66 (2H, NH_2, s), 8.03 (4H, H-aromatic) ppm. ^{13}C-NMR (DMSO-d_6,

125.77 MHz): δ (ppm) = 28.06 (2C, CH_3), 33.31 (1C), 40,90 (1C, CH_2), 49.82 (1C, CH_2), 52.68 (1C, C–CN), 54.01 (1C, C-spiro), 110.89 (1C, C=C, spiro ring), 117.70 (1C, CN), 124.03, 137.48, 141.45 (6C, C-aromatic), 160.76 (1C, C–O), 167.34 (1C, C-NH_2), 196.91 (2C, C=O), 200.59 (1C, C=O).

2-amino-1′,3′,8-trioxo-1′,3′,5,6,7,8-hexahydrospiro [chromene-4,2′-indene]-3-carbonitrile (5d)

Brown powder, M.P = 240 °C. Yield = 79 %. IR KBr (υ_{max}, cm^{-1}): 3,375 (NH_2, broad), 3,029 (C–$H_{aromatic}$), 2,203 (CN), 2,913 (C–$H_{aliphatic}$), 1,726, 1,715 (C=O) cm^{-1}. ^{1}H-NMR (DMSO-d_6, 500 MHz): δ (ppm) = 1.96 (2H, CH_2, t, J = 5.0 Hz), 2.28 (2H, CH_2, t, J = 5.0 Hz), 2.71 (2H, CH_2, t, J = 5.0 Hz), 7.63 (2H, NH_2, S), 8.00 (2H, H-aromatic), 8.03 (2H, H-aromatic) ppm. ^{13}C-NMR (-DMSO-d_6, 125.77 MHz): δ (ppm) = 19.79 (1C, CH_2), 26.35 (1C, CH_2), 35.25 (2H, CH_2), 51.87 (1C, C–CN), 53.17 (1C, C-spiro), 111.01 (1C, CN), 116.84 (1C, C=C), 123.10, 136.57, 140.52 (C-aromatic), 159.76 (1C, C–O), 168.28 (1C, C–NH_2), 196.08 (1C, C = O 1,2-cyclohexanone), 199.72 (2C, C=O).

7′-amino-1,2′,3,4′-tetraoxo-1,1′,2′,3,3′,4′-hexahydrospiro[indene-2,5′-pyrano[2,3-d]pyrimidine]-6′-carbonitrile (5e)

Yield = 88 %.IR (KBr, υ_{max}, cm^{-1}): 3,316(NH_2, broad), 3,183 (NH), 3,096, 2,206 (CN), 1,724, 1,699 (C=O).^{1}H-NMR (DMSO-d_6, 500 MHz) δ:7.68 (s, 2H, NH_2), 8.15–8.22 (m, 4H, ArH), 11.27 (s, 1H, NH), 12.35 (s, 1H, NH); MS m/z(%): 336 (M+, 38.64).

7′-amino-1′,3′-dimethyl-1,2′,3,4′-tetraoxo-1,1′,2′,3,3′,4′-hexahydrospiro[indene-2,5′-pyrano[2,3-d]pyrimidine]-6′-carbonitrile (5f)

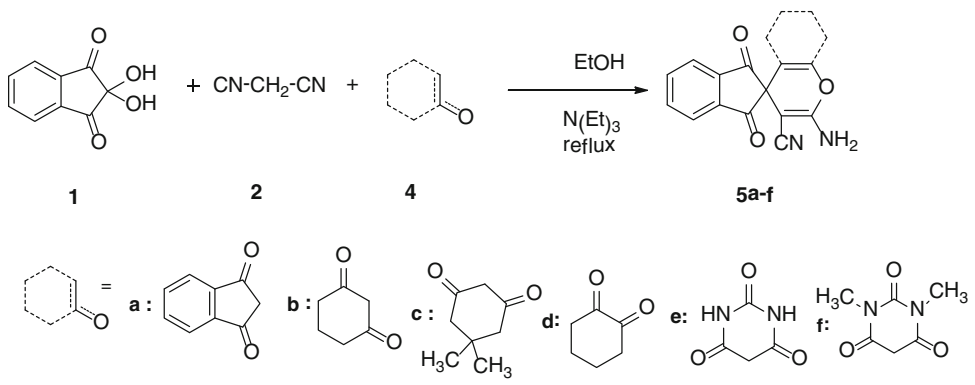
Yield = 89 %. IR (KBr, υ_{max}, cm^{-1}): 3,384 (NH_2, broad), 3,321, 3,253, 3,210, 2,193 (CN), 1,731, 1,680 (C=O). ^{1}H-NMR (DMSO-d_6, 500 MHz) δ: 3.10 (s, 3H, CH_3), 3.39 (s, 3H, CH_3), 7.89 (s, 2H, NH_2), 8.08–8.13 (m, 4H, ArH), MS m/z (%): 364 (M+, 17.23).

2′-amino1,3,5′-trioxo-1,3-dihydro-5′H-spiro[indene-2,4′-pyrano[3,2-c]chromene]-3′-carbonitrile (5g)

Brown powder, M.P = 215 °C (Decompose). Yield = 75 % IR KBr (υ_{max}, cm^{-1}): 3,398 (NH_2, broad), 3,039 (C–$H_{aromatic}$), 2,201(CN), 1,700, 1,682(C=O), cm^{-1}. ^{1}H-NMR (DMSO-d_6, 500 MHz): δ (ppm) = 7.51 (1H-aromatic, d, J = 8.4 Hz), 7.54 (1H, H- aromatic, t, J = 7.65 Hz), 7.78 (1H, H-aromatic, t, J = 7.4 Hz), 7.90 (1H, H-aromatic, d, J = 7.75 Hz), 8.09–8.13 (m, NH_2, H-aromatic) ppm. ^{13}C-NMR (DMSO-d_6, 125.77 MHz): δ (ppm) = 52.89 (1C, C–CN), 65.39 (1C, C-spiro), 99.76 (1C, C- C-spiro), 111.9 (1C, CN), 115.57, 116.50, 117.02, 122.23, 122.99, 123.83, 120.57, 125.33, 125.43, 134.28, 137.32, 140.53, 152.22, 156.74, 159.61, 159.74 (C-aromatic), 160.00 (C=O, C-cumarin), 198.78 (2C=O, 1,3-indandione ring).

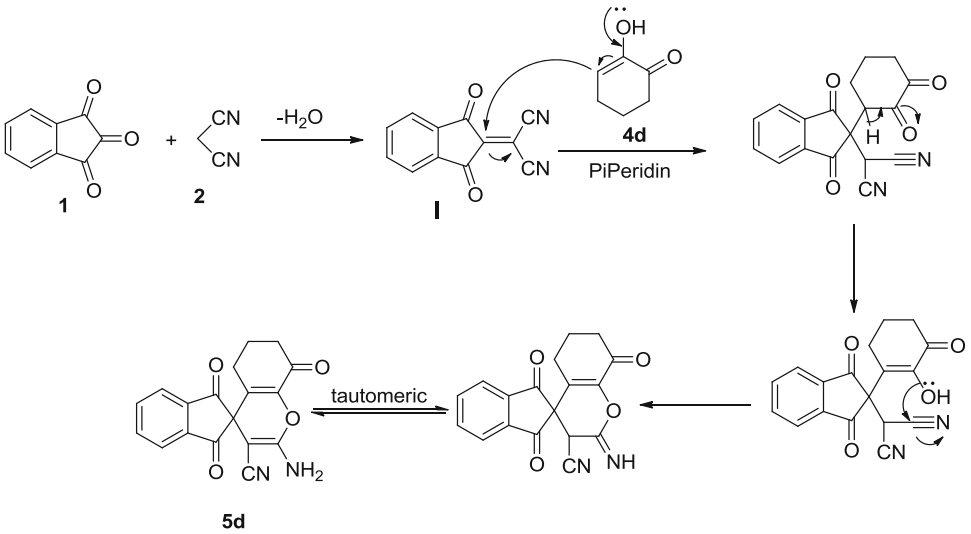
Ethyl-2′-amino-1,3,5′-trioxo-1,3-dihydro-5′H-spiro[indene-2,4′-pyrano[3,2-c]chromene]-3′-carboxylate (5g')

IR KBr (υ_{max}, cm^{-1}): 3,398 (NH_2, broad), 3,039 (C–$H_{aromatic}$), 2,201(CN), 1,700, 1,682(C=O), cm^{-1}. H-NMR (DMSO-d_6, 500 MHz): δ (ppm) 1.10 (m, 3H, CH_3), 4.20 (m, 2H, CH_2), 7.51 (1H-aromatic, d, J = 8.4 Hz), 7.54 (1H, H-aromatic, t, J = 7.65 Hz), 7.78 (1H, H-aromatic, t, J = 7.4 Hz), 7.90 (1H, H-aromatic, d, J = 7.75 Hz), 8.09–8.13 (m, NH_2, H-aromatic) ppm ^{13}C-NMR (DMSO-d_6, 125.77 MHz): δ (ppm) = 14.00 (CH_3), 65.00 (CH_2–O), 52.89 (1C, C–CN), 65.39 (1C, C-spiro), 99.76 (1C, C- C-spiro), 111.9 (1C, CN), 115.57, 116.50, 117.02, 122.23, 122.99, 123.83, 120.57, 125.33, 125.43, 134.28, 137.32, 140.53, 152.22, 156.74, 159.61, 159.74 (C-aromatic), 160.00 (C=O, C=O esteric), 198.78 (2C=O, 1, 3-indandione ring).

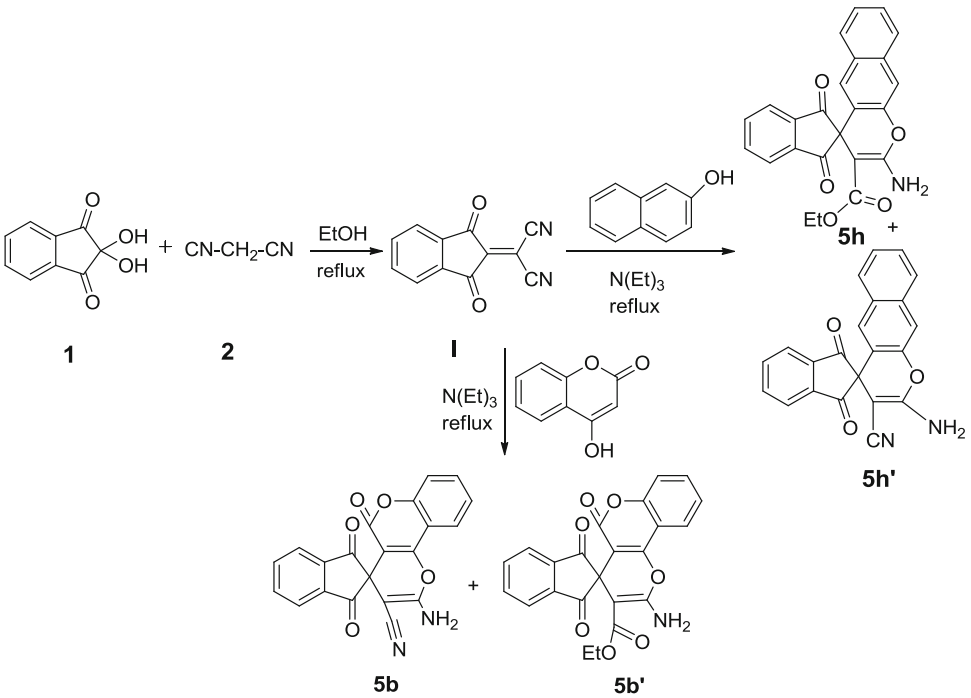


Scheme 2 Three-component synthesis of amino-spiroindene derivatives

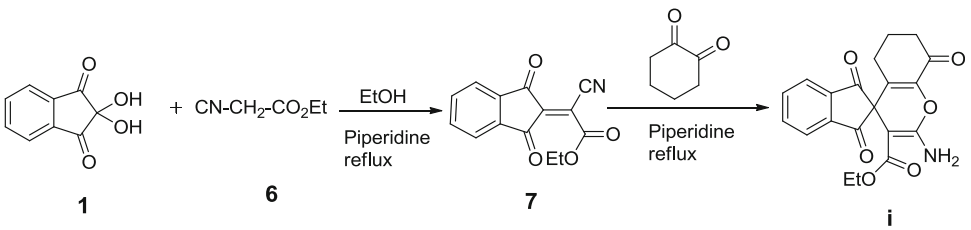
Scheme 3 A plausible mechanism for synthesis of 2-amino-1′,3′,8-trioxo-1′,3′,5,6,7,8-hexahydrospiro[chromene-4,2′-indene]-3-carbonitrile



Scheme 4 Three-component reaction of ninhydrin/malonitrile and 2-naphthol or 4-hydroxy cumarin



Scheme 5 Synthesis of ethyl 2-amino-1′,3′,8-trioxo-1′,3′,5,6,7,8-hexahydrospiro[chromene-4,2′-indene]-3-carboxylate



2-amino-1′,3′-dioxo-1′,3′-dihydrospiro [benzo[h]chromene-4,2′-indene]-3-carbonitrile (5h')

Brown powder, M.P = 319 °C Decompose. Yield = 88 %. IR (KBr, υ_{max}, cm^{-1}): 3,399 (NH_2, broad), 3,068 ($C–H_{aromatic}$), 2,926 ($C–H_{aliphatic}$), 1,709 (C=O), 1,620 (C=C) cm^{-1}. ^1H -NMR (DMSO-d_6, 500 MHz) δ: 7.90 (s, 2H, NH_2), 8.14–8.18 (m, 10H, ArH); MS *m/z* (%): 352 (M+, 19.23), 180 (100.00).

Ethyl-3-amino-1′,3′-dioxo-1′,3′-dihydrospiro[benzo[f]chromene-1,2′-indene]-2-carboxylate (5h)

IR (KBr, υ_{max}, cm^{-1}): 3,410 (NH_2, broad), 1,718 (C=O), 1,648, cm^{-1}. H-NMR (DMSO-d_6, 500 MHz) δ: 1.09 (m, 3H, CH_3), 4.18 (m, 2H, CH_2), 7.57 (s, 2H, NH_2), 7.86–9.08 (m, 10H, ArH); MS *m/z* (%): 399 (M+, 23.05), 327 (100.00).

2-amino-3-(2-methoxyacetyl)-6,7-dihydrospiro[chromene-4,2′-indene]-1′,3′,8(5H)-trione (i)

Yield = 69 %. IR (KBr, υ_{max}, cm^{-1}): 3,395 (NH_2, broad), 3,064 (C–$H_{aromatic}$), 2,209 (CN), 2917 (C–$H_{aliphatic}$), 1,726, 1,715, 1,687 (C=O) cm^{-1}. ^{1}H-NMR (DMSO-d_6, 500 MHz): δ (ppm) = δ: 1.12 (m, 3H, CH_3), 4.21 (m, 2H, CH_2) 1.96 (2H, CH_2, t, J = 5.0 Hz), 2.28 (2H, CH_2, t, J = 5.0 Hz), 2.71 (2H, CH, t, J = 5.0 Hz), 7.63 (2H, NH_2, s), 8.00 (2H, H-aromatic), 8.03 (2H, H-aromatic) ppm. ^{13}C-NMR (DMSO-d_6, 125.77 MHz): δ (ppm) = 14.53 9 (1C, CH_3), 19.46 (1C, CH_2), 27.23 (1C, CH_2), 35.11 (2C, CH_2), 52.07 (1C, C–CN), 53.63 (1C, C-spiro), 62.17 (1C, OCH_2), 112.05 (1C, CN), 117.55 (1C, C=C), 123.79, 138.61, 140.82 (C-aromatic), 160.96 (1C, C–O), 169.98 (1C, C–NH_2), 197.04 (1C, C=O 1,2-cyclohexanone),178, 199.72 (3C, C=O).

Results and discussion

We have developed an efficient protocol for the solution phase synthesis of new amino- spiroindene derivatives through the three-component condensation of ninhydrin, malononitrile/ethylcyanoacetate and various reagents including α-methylencarbonyl compounds/enols (Scheme 2). In a closely relevant study Khurana et al. [23] reported the synthesis of a series of Spiropyrans derivatives through the three-component condensation of ninhydrin, malononitrile and different 1,3-dicarbonyl compounds in the presence of polyethylene glycol (PEG)-stabilized Ni nanoparticles. But the present study revealed the results, using a wide variety

Table 1 Structures, yield and conditions synthesis of amino-spiroindene derivatives **5a–f**, **5g**, **5g'**, **5h**, **5h'** and **i**

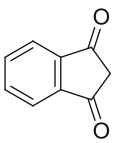
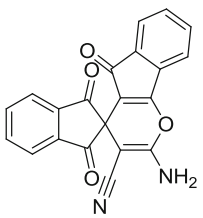
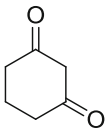
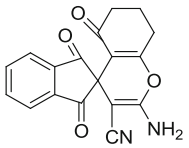
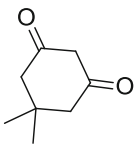
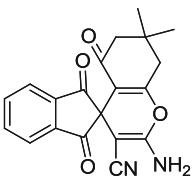
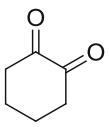
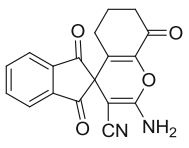
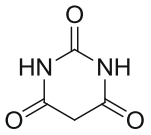
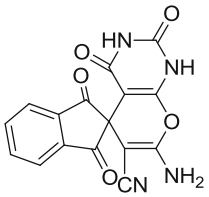
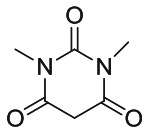
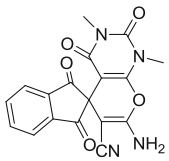
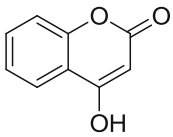
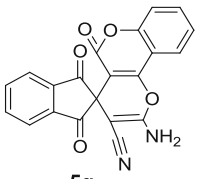
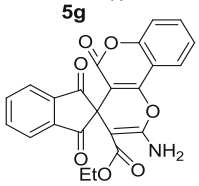
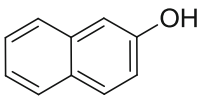
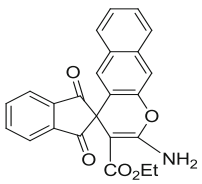
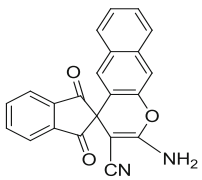
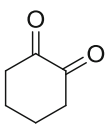
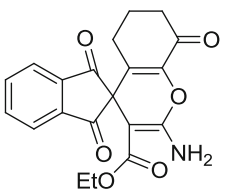
Entry	Dicarbonyl compounds	Product	Time (h)	Isolated yield (%)
1		 **5a**	8	70
2		 **5b**	4	69
3		 **5c**	6	63
4		 **5d**	5	79

Table 1 continued

Entry	Dicarbonyl compounds	Product	Time (h)	Isolated yield (%)
5		 **5e**	8	88
6		 **5f**	8	89
7		 **5g**  **5g'**	7	75
8		 **5h** +  **5h'**	6	88
9		 **i**	10	69

of compounds **4**, 4-hydroxy cumarin and 2-naphthol by a straightforward procedure in the absence of complex and expensive catalyst.

The synthesis of **5a-f** includes a two-step reaction: Knoevenagel condensation to obtain unsaturated nitriles **I** from ninhydrin **1** and malononitrile **2** and interaction of **I** with the active α-methylencarbonyl compounds/enols **4** catalyzed by triethylamine or piperazine (Et_3N or $C_5H_{11}N$). A plausible mechanism for the reaction is shown in Scheme 3. Compound **1** undergoes Knoevenagel condensation with malononitrile/ethyl cyanoacetate. Unsaturated nitriles **I** are formed very easily in ethanol even in the absence of Et_3N. Reactant **I** undergoes Michael addition to reagent **4**, followed by cycloaddition onto the nitrile. Eventually, after tautomeric proton shift compound **5** is formed.

In this reaction, as shown in Scheme 4 when ninhydrin/malononitrile reacted with 2-naphthol, or 4-hydroxy cumarin, a mixture of compounds **5h** and **5h'** or **5g** and **5g**' was formed.

As shown in Scheme 2 and Table 1, to establish the generality of the method, wide variety of suitable substrates was employed. Various reactants **4** including 1,3-diketones and 1,2-diketone were used and the reactions afforded the corresponding products. Table 1 shows the reaction yields, products structures, and conditions for synthesis of amino-spiroindene derivatives. In addition, the reaction with ethyl cyanoacetate also proceeded smoothly and included two-steps. In the first step, ninhydrin reacted with ethyl cyanoacetate to produce intermediate **7** and then this intermediate was reacted with cyclohexane-1,2-dione (Scheme 5). However, the reaction times were longer, when ethyl cyanoacetate was employed as one of the substrates.

Conclusion

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

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