Rapid and efficient synthesis of spiro-oxindoles using Fe^{3+}-montmorillonite under ultrasonic irradiation

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A simple and efficient one-pot three-component approach was devised for the synthesis of spiro-oxindoles through the reaction of isatin derivatives, malononitrile and 1,3-dicarbonyl compounds in the presence of modified montmorillonite (Fe^{3+}-mont.) under ultrasonic irradiation. The reaction furnished the desired products in excellent yields (85–95%) and short reaction times (2–7 min).

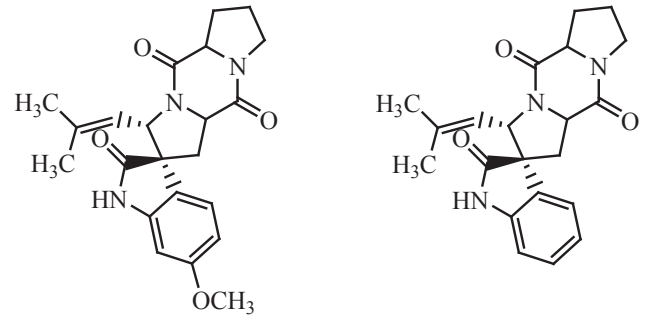
Keywords: spiro-oxindole, ultrasounic irradiations, Fe^{3+}-montmorillonite, three-component reaction

The indole nucleus is found in a variety of medicinal agents and natural products[1] including antibacterial and antifungal agents.[2] It has been reported that in the formation of C-3 spiroindoline derivatives enhances the biological activity.[3,4] The spiro-oxindole system is found in a number of alkaloids and pharmacological agents. For example, spirotryprostatins A and B (Fig. 1), alkaloids isolated from the fermentation broth of *Aspergillus fumigatus*, have been found to have anti-mitotic properties, and are of interest as anti-cancer drugs,[5,6] whilst pteropodine and isopteropodine have been shown to modulate the function of muscarinic serotonin receptors.[7]

Many compounds that containing the spiro-oxindole core exhibit significant biological activity, which has a role in drug development.[8-14]

Several strategies have been reported for the synthesis of spiro-oxindoles.[15-22] Although most of these processes have particular advantages, some use expensive starting materials, harsh reaction conditions, long reaction times, and give low yields. In some cases, their catalyst is harmful to the environment and cannot be reused. Therefore, the development of an efficient and versatile method is still required. Ultrasonic conditions have been increasingly used to provide a green, clean and environmentally benign source of energy for the preparation of organic compounds.[23, 24] A large number of organic reactions can be carried out in higher yield and shorter reaction time using a sono-chemical method.[25, 26]

Consequently, a convenient one-pot three-component approach was developed for the synthesis of spiro-oxindoles using modified montmorillonite (Fe^{3+}-mont.) under ultrasonic irradiation (Scheme 1).



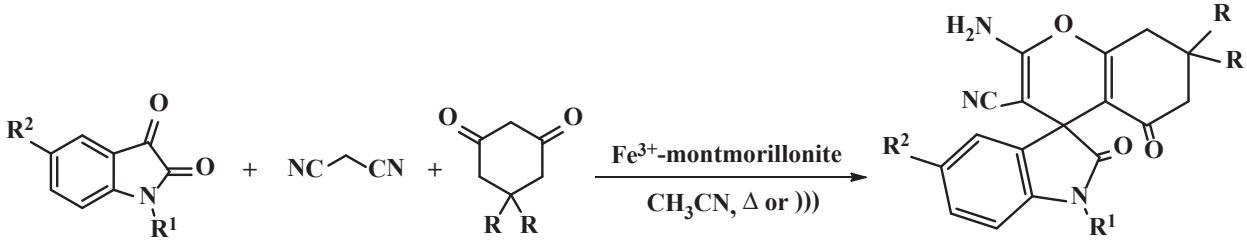
Spirotryprostatin A **Spirotryprostatin B**

Fig. 1 Representatives of spiro-oxindole containing compounds.

Result and discussions

As part of our research into the synthesis of heterocycles with medicinal applications,[27-30] we carried out a simple one-pot preparation of spiro-oxindoles **(4a–j)** by the reaction of equimolar amounts of isatin derivatives (**1**), malononitrile (**2**) and 1,3-dicarbonyl compounds (**3**) using Fe^{3+}-mont. (0.05 g $mmol^{-1}$ substrate) as efficient catalyst under ultrasonic irradiation at 50 °C (Scheme 1).

To optimise the reaction conditions, the preparation of (2-amino-5´-iodo-7,7-dimethyl-2´,5-dioxo-5,6,7,8-tetrahydro-spiro[chromen-4,3´- indoline]-3- carbonitrile) **4a** was examined in several solvents such as 1,4-dioxane, CH_2Cl_2, CH_3CN and EtOH (Table 1). The result showed that CH_3CN was the most effective solvent giving the product in high yield (92%). We also



1 **2** **3** **4a-j**

R^1 = H, Bn
R^2 = H, Cl, NO_2, OMe, I
R = H, CH_3

Scheme 1 Synthesis of derivatives of spiro-oxindole (**4a–j**).

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established the amount of the catalyst required for the preparation of **4a** in CH_3CN. The best result was obtained using 0.05 g Fe^{3+}-mont./mmole substrate (Table 2). The results in Table 2 showed that, the reaction in the presence of montmorillonite-K10 (entry 7) gave the product in 50% yield but without Fe^{3+}–montmorillonite (entry 1), only traces (10 %) of the product **(4a)** was produced.

The reaction under optimised conditions (Table 2, entry 6) furnished the desired spiro-oxindoles (**4a–j**) in excellent yields (85–95%) and short reaction times (2–7 min.) (Scheme 1 and Table 3). From the results shown in Table 3, it is evident that both electron-deficient and electron-rich isatin derivatives afford fairly high yields of the desired spiro-condensates in reactions with malononitrile and a variety of 1,3-dicarbonyl compounds in a few minutes.

The structures of all the products were established on the basis of their analytical and spectroscopic data (Table 3).

A plausible mechanism for the formation of **4a–j** is outlined in Scheme 2. The formation of these products may occur by an initial Knoevenagel condensation of isatin **(1)** and malononitrile **(2)**. Isatin **(1)** activated by Fe^{3+}-montmorillonite could be a good acceptor for a Knoevenagel condensation with malononitrile to afford isatylidene malononitrile **(5)**. This is subsequently attacked by **(6↔3)** to furnish the central intermediate **(7↔8)** thus producing products **(4a–j)**.

Table 1 Effect of various solvents (5 mL $mmol^{-1}$ substrate) in the synthesis of **4a** using Fe^{3+}-montmorillonite under ultrasonic irradiation

Solvent	Time/min	Yield/%[a]
Dioxane	5	60
CH_2Cl_2	5	65
CH_3CN	5	92
EtOH	5	80

[a]Isolated yield.

The catalyst, prepared according to the literature method,[31,32] was also recycled and reused in the preparation of **4a** under the optimised conditions. After each run the catalyst was filtered, washed, dried and activated at 120 °C. The result showed that after four successive runs, the activity of the catalyst was retained without significant loss.

Conclusions

In conclusion, we have developed a simple, efficient, and versatile one-pot three-component protocol for the synthesis of functionalised spiro-oxindole derivatives **4a–j** by the reaction of isatin derivatives, malononitrile, and 1,3-dicarbonyl compounds using Fe^{3+}-mont. under ultrasonic irradiations. This method involves mild reaction conditions, easy work-up, and cleaner reaction profiles using a recyclable solid acid catalyst.

Experimental

Melting points were measured on an Electrothermal 9100 apparatus. IR spectra were determined on a Shimadzo IR-470 spectrometer. ^1H NMR and ^{13}C NMR spectra were recorded on 400 MHz Bruker DRX-400 and 500MHz Bruker DRX-500 in DMSO-d_6 as solvent and TMS as an internal standard. Chemical shifts in the ^1H and ^{13}C NMR were expressed in ppm downfield from tetramethylsilane. Sonication was performed in Elmasonic S 40H ultrasonic cleaning unit. Elemental analyses were carried out on a Carlo-Erba EA1110CNNO-S analyser and agreed with the calculated values. All the chemicals were purchased from Merck and used without further purification. All solvents used were dried and distilled according to standard procedures.

Synthesis of spiro-oxindole derivatives **(4a–j);** *general procedure*

A mixture of isatin derivatves (1 mmole), malononitrile (1 mmole), 1,3-dicarbonyl compounds (1 mmole), and Fe^{3+}-mont. (0.05 g) in CH_3CN (5 mL) was heated using an Elmasonic S 40 H ultrasonic cleaning unit at 50 °C under silent condition. The progress of the reaction was monitored by

Table 2 Optimisation of amount of the catalyst in the synthesis of **4a** under ultrasonic irradiation

Entry	Catalyst	Catalyst/g	Conventional method		Sonochemical method[b]	
			Time/min	Yield/%[a]	Time/min	Yield/%[a]
1	–	–	180	5	5	10
2	Fe^{3+}-mont.	0.01	180	7	5	25
3	Fe^{3+}-mont.	0.02	180	20	5	37
4	Fe^{3+}-mont.	0.03	180	37	5	45
5	Fe^{3+}-mont.	0.04	180	40	5	55
6	Fe^{3+}-mont.	0.05	180	45	5 (5)[c]	92 (68)[c]
7	Montmorillonite-k10	0.05	180	35	5	50
8	Fe^{3+}-mont.	0.08	180	42	5	90

[a]Isolated yield.

[b]Conditions under ultrasonic irradiations: 40 KHz, 50 °C.

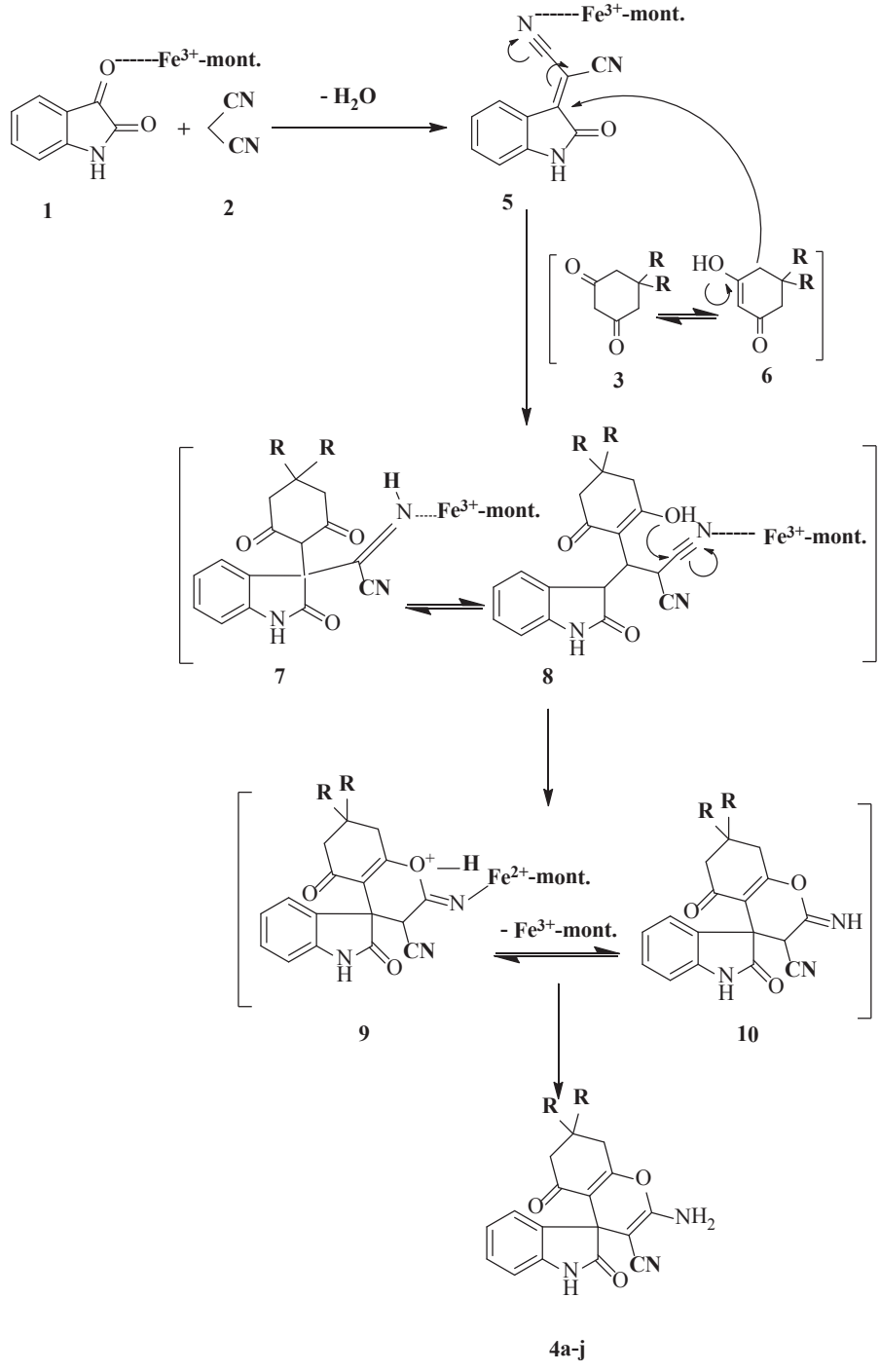
[c]Reaction at room temperature.

Table 3 Synthesis of spiro-oxindoles (**4a–j**) using Fe^{3+}-montmorillonite under ultrasonic irradiation at 50 °C

Entry	R	R_1	R_2	Time/min	Yield/%[a]	M.p. found/°C	M.p. reported/°C[lit]
4a	CH_3	H	I	5	92	265–268	–
4b	H	H	I	5	94	264–266	–
4c	CH_3	H	OCH_3	2	96	270–272	–
4d	H	H	OCH_3	2	95	272–274	–
4e	CH_3	H	H	6	90[b]	269–270	268–270[18]
4f	CH_3	H	NO_2	7	85[b]	303–305	302–304[19]
4g	H	H	NO_2	6	82	306–307	–
4h	CH_3	Bn	H	4	90[b]	268–270	267–269[20]
4i	CH_3	H	Cl	3	94[b]	289–290	289–291[21]
4j	H	H	Cl	3	95[b]	297–298	294–296[22]

[a]Isolated yield.

[b]Identified by comparison of their melting points and spectral data with those reported.



Scheme 2 A possible mechanism for the formation of **4a–j**.

TLC (EtOAc/hexane 3:7). After completion of the reaction, the catalyst was removed by filtration. Water was added to the residue. The solid which was obtained was filtered off and dried to provide pure products of the spiro-oxindole derivatives **(4a–j)** (Table 3).

Synthesis of Fe^{3+}-montmorillonite[32]

A total of 1% suspension of montmorillonite (K10) in a 1.5 mol dm^{-3} solution of $FeCl_3.6H_2O$ was stirred overnight. On settling, the supernatant solution was discarded and exchange process repeated three times. The ion-exchange material was filtered and washed free of chloride ion (checked by 0.1 M $AgNO_3$) with deionised water and dried in air.

2-Amino-5′-iodo-7,7-dimethyl-2′,5-dioxo-5,6,7,8-tetrahydrospiro[chromen-4,3′-indoline]-3-carbonitrile **(4a)**: White powder; IR (KBr) (ν_{max}/cm^{-1}): 3380, 3300, 3170, 3020, 2950, 2200, 1720, 1680, 1650, 1595, 1464, 1345,1220,1160, 1050, 803. ^{1}H NMR (500 MHz, DMSO-d_6): δ 10.49 (1H, s, NH), 7.44 (1H, d, *J* = 7.0 Hz, ArH), 7.27 (3H, br. s, ArH, NH_2), 6.61 (1H, d, *J* = 7.0 Hz, ArH), 2.56 (1H, d, *J* = 17.2 Hz, –CH_2–C=O), 2.46 (1H, *d*, *J* = 17.22 Hz, –CH_2–C=O), 2.14 (1H, *d*, *J* = 16.1 Hz, –CH_2–), 2.09 (1H, *d*, *J* = 16.1 Hz, –CH_2–), 0.98 (6H, s, CH_3) ppm. ^{13}C NMR (125 MHz, DMSO-d_6): δ 196.0, 178.3, 165.4, 159.7, 142.8, 138.9, 138.0, 137.6, 135.7, 132.2, 118.1, 112.6, 111.1, 85.3, 57.7, 50.8, 32.9, 28.4, 28.0. Anal. calcd for $C_{19}H_{16}IN_3O_3$ (461.25): C, 49.47; H, 3.50; N, 9.11; found: C, 49.33; H, 3.41, N, 9.00%.

2-Amino-5′-iodo-2′,5-dioxo-5,6,7,8-tetrahydrospiro[chromen-4,3′-indoline]-3-carbonitrile **(4b)**: White powder; IR IR (KBr) (ν_{max}/cm^{-1}): 3390, 3300, 3180, 3050, 2200, 1705, 1652, 1630,1605, 1590, 1468, 1220, 1165, 1075, 809. ^{1}H NMR (500 MHz, DMSO-d_6): δ 10.51 (1H, s, NH), 7.47 (1H, d, *J* = 7.8 Hz, ArH), 7.35 (1H, s, ArH), 7.28 (2H, s, NH_2), 6.64 (1H, d, *J* = 7.8 Hz, ArH), 2.64 (2H, br. s, CH_2–C=O), 2.24 (2H, br. s, –CH_2–), 1.93 (2H, m, –CH_2–CH_2– CH_2–) ppm. ^{13}C NMR (125 MHz, DMSO-d_6): δ 195.7, 178.0, 167.0, 159.2, 142.3, 137.7, 137.2, 132.0, 117.8, 112.2, 111.8,

85.0, 36.8, 27.2, 20.2 ppm. Anal. calcd for $C_{17}H_{12}IN_3O_3$ (433.2): C, 47.13; H, 2.79; N, 9.70; found: C, 47.18; H, 2.61, N, 9.60%.

2-Amino-5′-methoxy-7,7-dimethyl-2′,5-dioxo-5,6,7,8-tetrahydrospiro[chromen-4,3′-indoline]-3-carbonitrile (**4c**): White powder; IR (KBr) (ν_{max}/cm^{-1}): 3390, 3300, 3250, 3200,3050, 3020, 2960, 2200, 1670, 1650, 1599 1480, 1460, 1358, 1220, 1160, 1045, 800, 742. ^{1}H NMR (500 MHz, DMSO-d_6): δ 10.16 (1H, s, NH), 7.17 (2H, s, NH_2), 6.66 (2H, s, ArH), 6.56 (1H, s, ArH), 3.62 (3H, s, OCH_3), 2.46 (2H, s, –CH_2–C=O), 2.12 (1H, *d*, *J* = 16.0 Hz, –CH_2–), 2.07 (1H, *d*, *J* = 16.0 Hz, –CH_2–), 0.99, 0.98 (6H, s, 2CH_3) ppm. ^{13}C NMR (125 MHz, DMSO-d_6), δ 195.7, 178.8, 165.0, 159.6, 155.8, 136.6, 136.3, 118.2, 113.4, 111.6, 111.0, 110.4, 56.2, 50.9, 48.1, 32.8, 28.4, 28.0 ppm. Anal. calcd for $C_{20}H_{19}N_3O_4$ (365.38): C, 65.74; H, 5.24; N, 11.50; found: C, 65.88; H, 5.11, N, 11.37%.

2-Amino-5′-methoxy-2′,5-dioxo-5,6,7,8-tetrahydrospiro[chromen-4,3′-indoline]-3-carbonirile (**4d**): White powder; IR (KBr) (ν_{max}/cm^{-1}): 3400, 3300, 3220, 3180, 3002, 2200, 1700, 1680, 1650, 1600, 1500, 1460, 1210, 1190, 1080, 1010, 856, 818, 780. ^{1}H NMR (400 MHz, DMSO-d_6), δ 10.24 (1H, s, NH), 7.23 (2H, s, NH_2), 6.72–6.66 (3H, m, ArH), 3.68 (3H, s, OCH_3), 3.39 (2H, s, –CH_2–C=O), 2.24 (2H, s, –CH_2–), 1.94 (2H, s, –CH_2–$\underline{CH_2}$– CH_2–) ppm. ^{13}C NMR (100 MHz, DMSO-d_6): δ 195.5, 178.5, 166.5, 159.1, 155.4, 136.3, 135.8, 117.9, 113.0, 112.3, 110.8, 109.9, 55.8, 36.9, 27.2, 20.2 ppm. Anal. calcd for $C_{18}H_{15}N_3O_4$ (337.33): C, 64.09; H, 4.48; N, 12.46; found: C, 64.28; H, 4.35, N, 12.58%.

*2-Amino-7,7-dimethyl-2′,5-dioxo-5,6,7,8-tetrahydrospiro [chromen-4,3′-indoline]-3-carbonitril*e (**4e**): White powder; IR (KBr) (ν_{max}/cm^{-1}): 3380, 3300, 3140, 3020, 2950, 1708, 1680, 1650, 1600, 1345 (C-H bend, CH3), 1220, 1160, 1050, 740. ^{1}H NMR (500 MHz , DMSO-d_6): δ 10.38 (1H, s, NH), 7.20 (2H, s, NH_2), 7.14 (1H, t, *J* = 7.35 Hz, ArH), 6.98 (1H, d, *J* = 7.0 Hz, ArH), 6.89 (1H, t, *J* = 7.21 Hz, ArH), 6.79 (1H, d, *J* = 7.54 Hz, ArH), 2.55 (2H, dd, *J* = 8.0 Hz, –CH_2–C=O), 2.13 (2H, dd, *J* = 15.98 Hz, –CH_2–), 1.03 (3H, s, CH_3), 1.0 (3H, s, CH_3) ppm. ^{13}C NMR (125 MHz, DMSO-d_6) : δ 195.7, 178.9, 165.0, 159.6, 142.9, 135.3, 129.0, 123.9, 122.5, 118.2, 111.7, 110.1, 50.9, 47.7, 32.8, 28.5, 27.9 ppm. Anal. calcd for $C_{19}H_{17}N_3O_3$ (335.36): C, 68.05; H, 5.11; N, 12.53; found: C, 68.16; H, 5.27, N, 12.64%.

2-Amino-7,7-dimethyl-5′-nitro-2′,5-dioxo-5,6,7,8-tetrahydrospiro[chromen-4,3′-indoline]-3-carbonitrile (**4f**): Yellow powder; IR (KBr) (ν_{max}/cm^{-1}): 3460, 3350, 3100, 2200, 1722, 1680, 1650, 1595, 1480, 1460, 1510, 1345, 1360, 1210, 1180, 1050, 822, 740, 700; ^{1}H NMR (500 MHz, DMSO-d_6): δ 11.14 (1H, s, NH), 8.11 (1H, d, *J* = 7.1 Hz, ArH), 7.92 (1H, s, ArH),7.41 (2H, s, NH_2), 6.98 (1H, d, *J* = 7.4 Hz, ArH), 2.62 (1H, *d*, *J* = 17.4 Hz, –CH_2–C=O) 2.48 (1H, *d*, *J* = 17.4 Hz, –CH_2–C=O), 2.16(1H, *d*, *J* = 15.11 Hz, –CH_2–), 2.10 (1H, *d*, *J* = 15.11 Hz, –CH_2–), 0.99 (6H, s, 2CH_3) ppm. $C_{19}H_{16}N_4O_5$ (380.36): C, 60.0; H, 4.24; N, 14.73; found: C, 60.16; H, 4.12, N, 14.61%.

2-Amino-5′-nitro2′,5-dioxo-5,6,7,8-tetrahydrospiro[chromen-4,3′-indoline]-3-carbonitrile (**4g**)**:** Yellow powder; IR IR (KBr) (ν_{max}/cm^{-1}): 3460, 3350, 3198, 3060, 2200, 1740, 1719, 1678, 1650, 1620 (N–H bend), 1585, 1482, 1443, 1502, 1330, 1200, 1180, 1065, 822, 740, 720; ^{1}H NMR (400 MHz, DMSO-d_6): δ 11.20 (1H, s, NH), 8.16 (1H, d, *J* = 8.0 Hz, ArH), 8.0 (1H, s, ArH),7.46 (2H, s, NH_2),7.03 (1H, d, *J* = 8.4 Hz, ArH), 2.70 (2H, s, br., –CH_2–C=O), 2.26 (2H, s, br., –CH_2–), 1.96 (2H, br. s, –CH_2–$\underline{CH_2}$–CH_2–) ppm. $C_{17}H_{12}N_4O_5$ (352.08): C, 57.96; H, 3.43; N, 15.90; found: C, 57.65; H, 4.10, N, 15.60%.

2-Amino-1′-benzyl-7,7-dimethyl-2′,5-dioxo-5,6,7,8-tetrahydrospiro[chromen-4,3′-indoline]-3- carbonitrile (**4h**): White powder; IR (KBr) (ν_{max}/cm^{-1}): 3380, 3305, 3180, 3020, 2920, 2200, 1708, 1680, 1662, 1598, 1482, 1350, 1220, 1200, 1055, 800, 770, 700; ^{1}H NMR (500 MHz, DMSO-d_6): δ 7.46, 7.45 (2H, s, NH_2), 7.29–7.22 (5H, m, ArH), 7.09 (1H, t, *J* = 7.44 Hz, ArH), 7.05 (1H, d, *J* = 7.06 Hz, ArH), 6.92 (1H, t, *J* = 7.18 Hz, ArH), 6.65 (1H, d, *J* = 7.57 Hz, ArH), 4.88 (2H, AB quart., *J* = 16.14 Hz, –$\underline{H_2C}$– Ar), 2.60 (1H, *d*, *J* = 17.65 Hz, –CH_2–C=O), 2.54 (1H, *d*, *J* = 17.65 Hz, –CH_2–C=O), 2.18 (1H, d, *J* = 15.97 Hz, –CH_2–), 2.10 (1H, *J* = 15.97 Hz, –CH_2–), 1.02 (3H, s, CH_3), 0.98 (3H, s, CH_3) ppm.

2-Amino-5′-chloro-7,7-dimethyl-2′,5-dioxo-5,6,7,8-tetrahydrospiro[chromen-4,3′-indoline]-3-carbonitrile (**4i**): Cream powder, IR (KBr) (ν_{max}/cm^{-1}): 3380, 3300,3155,3060, 2950, 2200, 1718, 1680, 1647, 1600, 1475, 1345, 1220, 1160, 1055, 805; ^{1}H NMR (500 MHz, DMSO-d_6): δ 10.49 (1H, s, NH), 7.27 (2H, s, NH_2), 7.15 (1H, d, *J* = 7.53 Hz, ArH), 7.06 (1H, s, ArH), 6.76 (1H, *d*, *J* = 7.78 Hz, ArH), 2.55 (1H, d, *J* = 17.20 Hz, –CH_2–C=O) , 2.48 (1H, d, *J* = 17.20 Hz, –CH_2–C=O), 2.11 (2H, s, –CH_2–), 0.96 (6H, s, 2CH_3) ppm.

2-Amino-5′-chloro-2′,5-dioxo-5,6,7,8-tetrahydrospiro[chromen-4,3′-indoline]-3-carbonitrile (**4j**): Cream powder; IR (KBr) (ν_{max}/cm^{-1}): 3350, 3300, 3150, 2950, 2199, 1718, 1670, 1647, 1618, 1595, 1475, 1210, 1190, 1070, 830, 805, 722; ^{1}H NMR (DMSO-d_6, 400 MHz): δ 10.56 (1H, s, NH), 7.32 (2H, s, NH_2), 7.19 (1H, d, *J* = 7.2 Hz, ArH), 7.16 (1H, s, ArH), 6.81 (1H, d, *J* = 7.6 Hz, ArH), 2.66 (2H, s, br., –CH_2–C=O), 2.26 (2H, s, br., –CH_2–), 1.95 (2H, s, br., –CH_2–$\underline{CH_2}$– CH_2–) ppm.

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