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ORIGINAL PAPER

A simple and environmentally benign synthesis of novel spiro[indolin e-3,5′-pyrano[2,3-*d*]pyrimidine] derivatives in water

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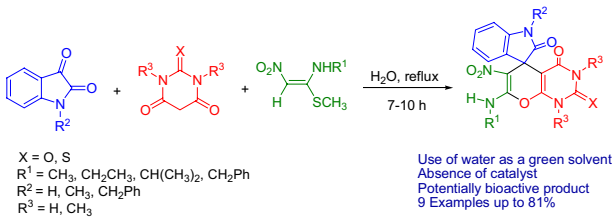
Abstract

A green, convenient, and efficient one-pot synthesis of a new class of spiro[indolinepyranopyrimidine] derivatives was achieved in good yields by the multi-component reaction of *N*-alkyl-1-(methylthio)-2-nitroethenamine derived from the addition of various amines to nitroketene dithioacetal with isatin and barbituric acid derivatives in water at reflux conditions. Notably, the present method offers desirable advantages including good yields, use of water as green solvent, absence of catalyst, simple workup procedure, and easy purification process with no chromatographic technique.

Graphical abstract



Keywords Spiro[indolinepyranopyrimidine] · Amines · Nitroketene dithioacetal · Isatin · Barbituric acid

Introduction

Indole derivatives have been a topic of substantial research interest and continue to be one of the most active areas of heterocyclic chemistry. They exhibit a wide range of biological activities [1–4] such as antibacterial, antimicrobial, antiviral, antifungal, antihypertensive, anti-inflammatory [5], antitumor [6], anticancer, anti-HIV, antioxidant [7], antimalarial, anticonvulsant [8], and anti-alzheimer [9] properties. In addition, it was reported that sharing of the indole 3-carbon atom in the formation of spiroindole derivatives (Fig. 1) significantly improves biological properties [10]. Spiroindoles have generated considerable synthetic interest due to their occurrence in diverse natural products and notable biological activities [11–21].

Pyranopyrimidine derivatives are very important and valuable compounds, due to their potential importance in the medicine and biological fields [22]. They have diverse pharmacological properties such as antimalarial, antibacterial [23], antifungal, antiviral, antitumor [23, 24], antibronchitic [25, 26], anti-AIDS [27], antipyretic [28], anti-inflammatory [29], and antihypertensive [30] evaluation activities. Considering the above reports, the development of new and simple synthetic methods for the efficient preparation of the spiroindoles containing pyranopyrimidine fragment could potentially lead to a series of structurally and biologically interesting heterocycles.

During the past decades, specific strategies have been reported for the synthesis of spiroindole-annulated

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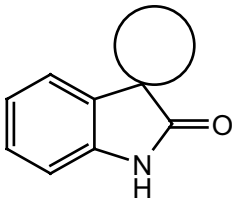


Fig. 1 Spiroindole derivatives linked at position three of the indole

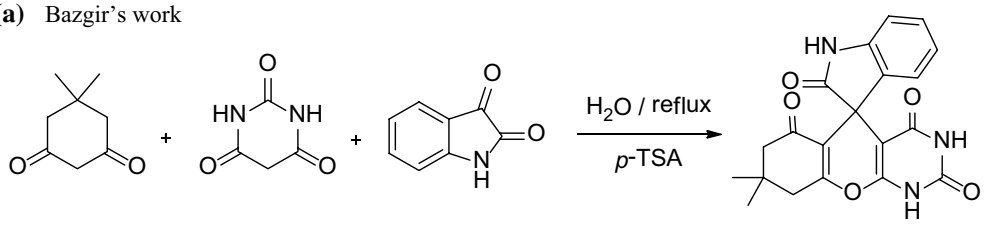
heterocycles. In 2010, Bazgir et al. reported an efficient, one-pot synthesis of spiro[chromenopyrimidineindoline] from cyclohexane-1,3-diones, isatins, and barbituric acids in refluxing water in the presence of *p*-TSA for 10 h (Scheme 1, entry a) [10, 13]. In 2015, Esmaeili et al. developed a rapid and convenient protocol for the synthesis of novel spirooxindole derivatives in excellent yields by the three-component reaction of malononitrile, isatin, and 2,3-dihydro-5*H*-[1,3]thiazolo[3,2-*a*]pyrimidine-5,7(6*H*)-dione in the presence of diisopropylethylamine (Scheme 1, entry b) [14]. In 2018, Deka et al. described a simple and cost-effective, micelle-catalyzed one-pot strategy for the synthesis of spiro[indolinepyranopyrazoles] by reacting isatins, malononitrile, and 3-methyl-1*H*-pyrazol-5(4*H*)-ones in water at room temperature (Scheme 1, entry c) [11]. Herein we report an environmentally benign synthesis of a new class of spiro[indolinepyranopyrimidine] derivatives via a catalyst free, one-pot, multi-component condensation reaction of various amines, nitroketene dithioacetal, isatin derivatives, and barbituric acids in refluxing water (Scheme 1, entry d).

Results and discussion

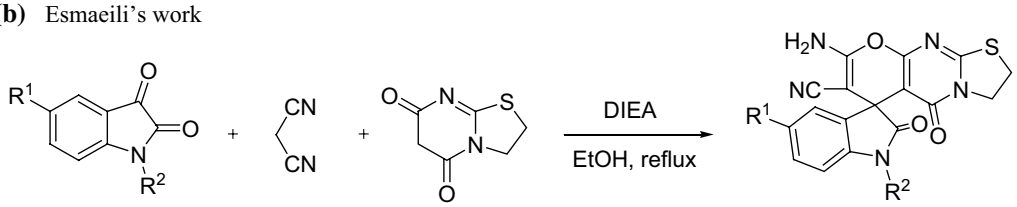
In this paper, we would like to report an easy and efficient procedure for synthesizing novel spiro[indolinepyranopyrimidine] derivatives. The products were obtained from the addition of various amines **1** to nitroketene dithioacetal **2** with isatin **3** and barbituric acid derivatives **4** in water as a green solvent at reflux conditions (Scheme 2).

Scheme 1

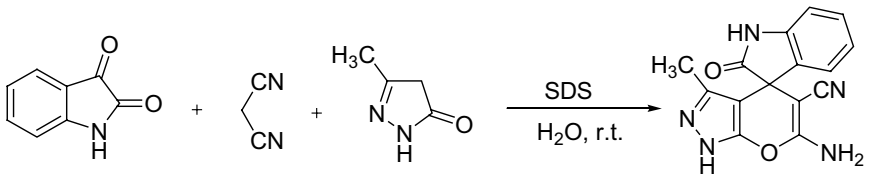
(a) Bazgir's work



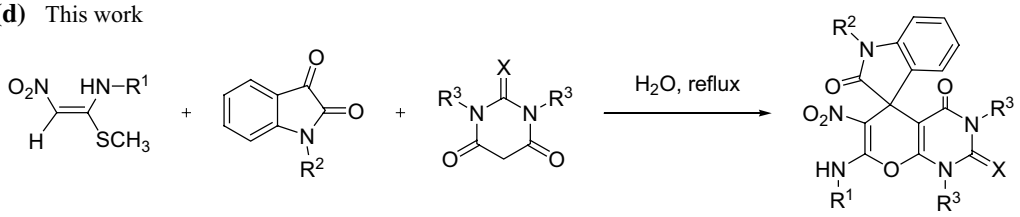
(b) Esmaeili's work



(c) Deka's work



(d) This work



Scheme 2

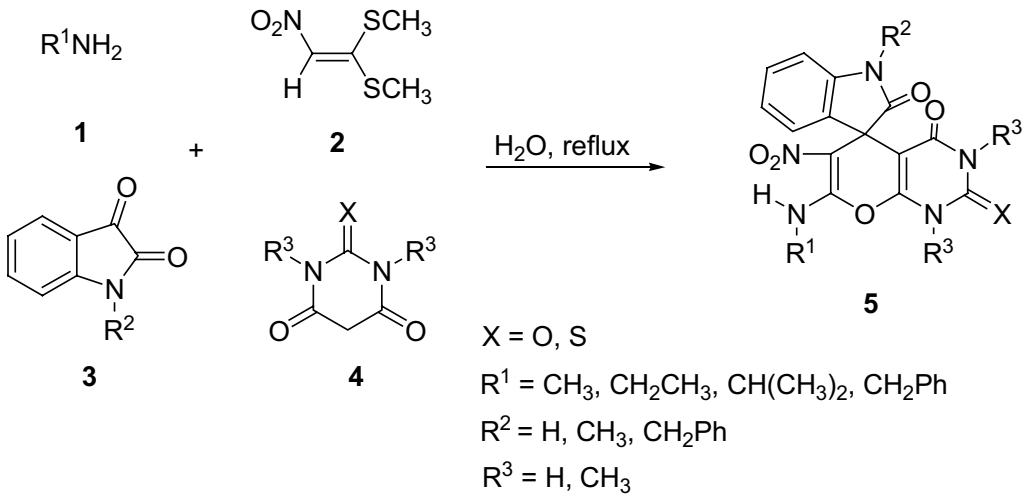


Table 1 Optimization of reaction conditions for **5c**

Entry	Conditions	Catalyst	Time/h	Yield/%
1	EtOH, r.t.	–	24	65
2	EtOH, reflux	–	7	75
3	EtOH, reflux	Piperidine	3	59
4	EtOH, reflux	Et_3N	3	54
5	EtOH/H_2O, reflux	–	7	70
6	**H_2O, reflux**	**–**	**7**	**72**
7	H_2O, 60 °C	–	12	60
8	H_2O, r.t.	–	24	20
9	CH_3CN, reflux	–	24	Trace
10	$CHCl_3$, reflux	–	24	n.r.
11	DMF, reflux	–	24	Trace

Bold numbers represent the best values

r.t. room temperature, *n.r.* no reaction

Several solvents in the presence and absence of catalyst were examined to develop standard reaction conditions and the results are summarized in Table 1. Experimental results showed that the reaction proceeded very cleanly with good yield when the EtOH and water were used as solvent at reflux conditions without any catalyst (Table 1, entries 2 and 6). The yield of product was low, when the water was used as solvent at 60 °C and room temperature (Table 1, entries 7 and 8). Also the yield of product was low, when the reaction was performed in the presence of piperidine or Et_3N as catalyst in EtOH (Table 1, entries 3 and 4). The reaction did not proceed well, when the CH_3CN and DMF were used as solvent (Table 1, entries 9 and 11). Also the reaction did not work in $CHCl_3$, so a lot of spots were observed on TLC (Table 1, entry 10).

As shown in Table 2, various primary amines, isatin derivatives, and barbituric acids were tolerated. The reaction proceeds cleanly under the same reaction conditions to afford a series of spiro[indolinepyranopyrimidine] derivatives **5a**–**5i** in 64–81% yields.

Table 2 Products **5a**–**5i** (cf. Scheme 2)

Entry	R^1	R^2	R^3	X	Product[a]	Time/h	Yield/%
1	CH_3	CH_2Ph	H	O	**5a**	9	68
2	CH_3	CH_3	H	O	**5b**	7	64
3	CH_3	H	H	O	**5c**	7	72
4	$CH(CH_3)_2$	H	H	O	**5d**	8	75
5	CH_2CH_3	H	H	O	**5e**	8	70
6	CH_3	H	CH_3	O	**5f**	9	81
7	CH_2Ph	H	H	O	**5g**	9	68
8	CH_2Ph	H	CH_3	O	**5h**	10	67
9	CH_2Ph	H	H	S	**5i**	10	70

[a]Various amines (1 mmol), nitroketene dithioacetal (1 mmol), isatin (1 mmol), and barbituric acid (1 mmol) were used. The reactions were run in refluxing water, without any catalyst

The structures of compounds **5a–5i** were elucidated from their mass, IR, and ^{1}H and ^{13}C NMR spectra. The IR spectrum of **5a** showed absorption bonds due to the NH groups at 3267 and 3194 cm^{-1}, and C=O groups at 1721 and 1615 cm^{-1}. Stretching frequencies related to the Ar and NO_2 groups appeared at 1528, 1463, and 1384 cm^{-1}, respectively. The ^{1}H NMR spectrum of **5a** exhibited a doublet recognized as arising from the CH_3 group ($\delta = 3.14$ ppm, $^{3}J_{HH} = 5.1$ Hz), one AB quartet due to CH_2 group (4.85 ppm), one multiplet for $NHCH_3$ group (10.55 ppm) and two singlets for NH groups (11.19 and 12.58 ppm), together with characteristic signals for the aromatic moiety (6.55–7.51 ppm). ^{1}H-decoupled ^{13}C NMR spectrum showed 20 distinct signals in agreement with the proposed structure. Resonances due to CH_3, CH_2, spiro carbon and three C=O groups appeared at $\delta = 29.3$, 44.6, 48.2, 156.9, 161.3, and 175.7 ppm, respectively.

A plausible mechanistic pathway for the formation of **5** is outlined in Scheme 3. Initially, the Knoevenagel condensation between isatin **3** and barbituric acid **4** derivatives affords **7** which undergoes Michael addition with *N*-alkyl-1-(methylthio)-2-nitroethenamine 6 (derived from the addition of various amines **1** to nitroketene dithioacetal 2) to give **8**. Thus the intermediate **8** undergoes imine-enamine tautomerisation to form **9** followed by O-cyclization to form **5** via the elimination of MeSH (Scheme 3).

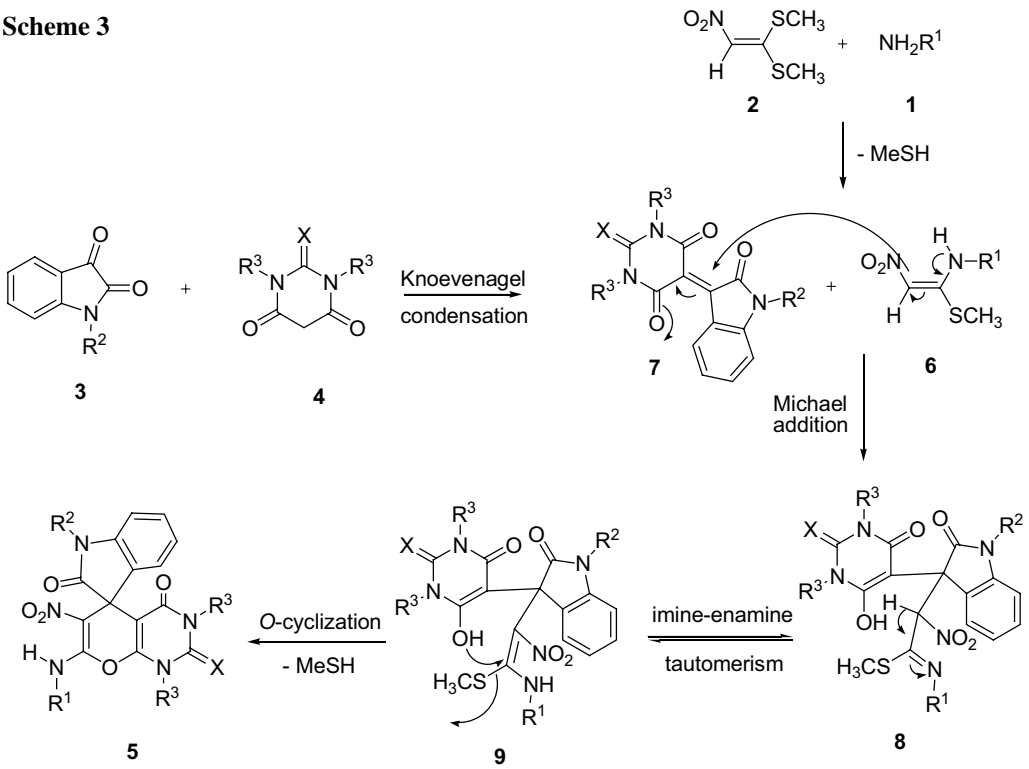
Conclusion

In conclusion, we have developed a simple, green and novel one-pot, multi-component synthesis of spiro[indolinepyranopyrimidine] derivatives, through sequential Knoevenagel condensation, Michael addition, and O-cyclization sequences in refluxing water, without any catalyst. This procedure offers several advantages, such as use of water as a green solvent, good yields of products, easy accessibility of reactants, easy workup procedure, and high atom economy.

Experimental

The various amines, nitroketene dithioacetal, isatin, barbituric acid, and other chemicals and solvents were obtained from Merck and Aldrich and were used without

Scheme 3



X = O, S

R^1 = CH_3, CH_2CH_3, $CH(CH_3)_2$, CH_2Ph

R^2 = H, CH_3, CH_2Ph

R^3 = H, CH_3

further purification. NMR spectra were recorded with a Bruker DRX-300 Avance instrument (300 MHz for ^{1}H and 75.4 MHz for ^{13}C) with DMSO-d_6 and $CDCl_3$ as solvent. Chemical shifts are given in ppm (δ), and coupling constant (J) are reported in hertz (Hz). Melting points were measured with an electrothermal 9100 apparatus. Mass spectra were recorded with an Agilent 5975C VL MSD with Triple-Axis Detector operating at an ionization potential of 70 eV. IR spectra were measured with Bruker Tensor 27 spectrometer. Elemental analyses for C, H, and N were performed using a PerkinElmer 2004 series [II] CHN elemental analyzer.

General procedure for the synthesis of product 5

A mixture of various amines (1 mmol), 0.165 gnitroketene dithioacetal (1 mmol) and 10 cm^3 H_2O in a 50 cm^3 flask was refluxed for 6 h. After completion of the reaction (monitored by TLC, ethyl acetate/*n*-hexane, 6:4), isatin derivatives (1 mmol) and barbituric acids (1 mmol) were added to the reaction mixture, and it was stirred under reflux for 7–10 h. Then, the reaction mixture was cooled to room temperature and filtered to give the crude product. The solid was washed with water to give pure product **5** in good yield.

1-Benzyl-7′-(methylamino)-6′-nitrospiro[indoline-3,5′-pyrano[2,3-*d*]pyrimidine]-2,2′,4′(1′*H*,3′*H*)-trione (5a, $C_{22}H_{17}N_5O_6$) White solid; m.p.: 308–310 °C (dec.); yield: 0.304 g (68%); IR (KBr): $\bar{\nu}$ = 3267 and 3194 (NH), 1721 (C=O), 1615 (C=O), 1528 (Ar), 1463 and 1384 (NO_2) cm^{-1}; MS (EI, 70 eV): *m/z* (%) = 447 (M^+, 11), 390 (9), 347 (5), 299 (30), 247 (10), 169 (8), 91 (100), 51 (2); ^{1}H NMR (300 MHz, DMSO-d_6): δ = 3.14 (d, $^3J_{HH}$ = 5.1 Hz, 3H), 4.85 (AB q, 2H), 6.55 (d, $^3J_{HH}$ = 7.5 Hz, 1H), 6.86 (t, 1H), 7.07 (t, 1H), 7.21–7.30 (m, 4H), 7.51 (d, $^3J_{HH}$ = 7.2 Hz, 2H), 10.55 (m, 2H), 11.19 (s, 1H), 12.58 (br s, 1H) ppm; ^{13}C NMR (75.4 MHz, DMSO-d_6): δ = 29.3 ($NHCH_3$), 44.6 (CH_2), 48.2 (C-spiro), 89.3, 107.5, 108.4, 122.2, 123.3, 127.4, 127.6, 128.6, 128.7, 130.3, 137.1, 145.6, 149.3, 151.9, 156.9 (C=O), 161.3 (C=O), 175.7 (C=O) ppm.

1-Methyl-7′-(methylamino)-6′-nitrospiro[indoline-3,5′-pyrano[2,3-*d*]pyrimidine]-2,2′,4′(1′*H*,3′*H*)-trione (5b, $C_{16}H_{13}N_5O_6$) White solid; m.p.: 320–322 °C (dec.); yield: 0.237 g (64%); IR (KBr): $\bar{\nu}$ = 3246 (NH), 1711 (C=O), 1656 (C=O), 1533 (Ar), 1467 and 1388 (NO_2) cm^{-1}; MS (EI, 70 eV): *m/z* (%) = 371 (M^+, 60), 310 (44), 284 (25), 240 (11), 211 (100), 171 (83), 143 (18), 114 (24), 91 (30), 57 (17); ^{1}H NMR (300 MHz, DMSO-d_6): δ = 3.11 (s, 6H), 6.87–7.19 (m, 4H), 10.48 (m, 1H), 11.11 (s, 1H), 12.55 (br s, 1H) ppm; ^{13}C NMR (75.4 MHz, DMSO-d_6): δ = 26.9 (CH_3), 29.2 (CH_3), 48.0 (C-spiro), 89.3, 107.4, 107.7, 122.0, 123.1, 128.7, 130.3, 146.2, 149.3, 151.8, 156.9 (C=O), 161.0 (C=O), 175.4 (C=O) ppm.

7′-(Methylamino)-6′-nitrospiro[indoline-3,5′-pyrano[2,3-*d*]pyrimidine]-2,2′,4′(1′*H*,3′*H*)-trione (5c, $C_{15}H_{11}N_5O_6$) White solid; m.p.: 354–356 °C (dec.); yield: 0.257 g (72%); IR (KBr): $\bar{\nu}$ = 3382 and 3235 (NH), 1725 (C=O), 1693 (C=O), 1531 (Ar), 1472 and 1325 (NO_2) cm^{-1}; MS (EI, 70 eV): *m/z* (%) = 357 (M^+, 39), 313 (9), 283 (63), 240 (100), 197 (94), 168 (85), 140 (55), 103 (35), 57 (69); ^{1}H NMR (300 MHz, DMSO-d_6): δ = 3.09 (s, 3H), 6.67–7.12 (m, 4H), 10.45 (s, 2H), 11.10 (s, 1H), 12.45 (br s, 1H) ppm; ^{13}C NMR (75.4 MHz, DMSO-d_6): δ = 29.2 ($NHCH_3$), 48.4 (C-spiro), 89.4, 107.8, 108.9, 121.3, 123.3, 128,5, 131.0, 144.8, 149.4, 151.8, 156.9 (C=O), 161.1 (C=O), 176.7 (C=O) ppm.

7′-(Isopropylamino)-6′-nitrospiro[indoline-3,5′-pyrano[2,3-*d*]pyrimidine]-2,2′,4′(1′*H*,3′*H*)-trione (5d, $C_{17}H_{15}N_5O_6$) White solid; m.p.: 255–260 °C (dec.); yield: 0.288 g (75%); ^{1}H NMR (300 MHz, DMSO-d_6): δ = 1.13 (d, 3H), 1.30 (d, 3H), 4.15–4.30 (m, 1H), 6.60–7.05 (m, 4H), 7.60 (br s, 1H), 9.30 (s, 1H), 10.15 (s, 1H), 10.65 (d, 1H) ppm; ^{13}C NMR (75.4 MHz, DMSO-d_6): δ = 20.8 (CH_3), 22.8 (CH_3), 44.6 (CH), 49.5 (C-spiro), 85.0, 108.4, 108.6, 120.7, 122.4, 127.5, 132.8, 144.9, 157.8, 158.4, 161.3 (C=O), 163.8 (C=O), 178.1 (C=O) ppm.

7′-(Ethylamino)-6′-nitrospiro[indoline-3,5′-pyrano[2,3-*d*]pyrimidine]-2,2′,4′(1′*H*,3′*H*)-trione (5e, $C_{16}H_{13}N_5O_6$) White solid; m.p.: 306–312 °C (dec.); yield: 0.259 g (70%); ^{1}H NMR (300 MHz, DMSO-d_6): δ = 1.24 (t, $^3J_{HH}$ = 6.9 Hz, 3H), 3.51–3.59 (m, 2H), 6.68–7.12 (m, 4H), 10.47 (s, 1H), 10.59 (t, $^3J_{HH}$ = 5.7 Hz, 1H), 11.12 (s, 1H) ppm; ^{13}C NMR (75.4 MHz, DMSO-d_6): δ = 15.6 (CH_3), 37.5 (CH_2), 48.4 (C-spiro), 89.4, 107.6, 108.9, 121.3, 123.3, 128.5, 131.0, 144.8, 149.3, 151.7, 156.4 (C=O), 161.1 (C=O), 176.7 (C=O) ppm.

1′,3′-Dimethyl-7′-(methylamino)-6′-nitrospiro[indoline-3,5′-pyrano[2,3-*d*]pyrimidine]-2,2′,4′(1′*H*,3′*H*)-trione (5f, $C_{17}H_{15}N_5O_6$) White solid; m.p.: 287–289 °C (dec.); yield: 0.312 g (81%); IR (KBr): $\bar{\nu}$ = 3431 and 3192 (NH), 1726 (C=O), 1687 (C=O), 1455 and 1355 (NO_2) cm^{-1}; MS (EI, 70 eV): *m/z* (%) = 385 (M^+, 53), 339 (55), 324 (100), 280 (37), 228 (24), 197 (41), 157 (31), 114 (22), 58 (35); ^{1}H NMR (300 MHz, DMSO-d_6): δ = 3.00 (s, 3H), 3.16 (d, $^3J_{HH}$ = 4.8 Hz, 3H), 3.45 (s, 3H), 6.69–7.11 (m, 4H), 10.48 (s, 1H) ppm; ^{13}C NMR (75.4 MHz, DMSO-d_6): δ = 28.2 ($NHCH_3$), 29.4 (NCH_3), 30.0 (NCH_3), 48.9 (C-spiro), 90.0, 107.8, 108.9, 121.2, 123.4, 128.6, 130.9, 144.9, 149.7, 150.7, 156.6 (C=O), 159.0 (C=O), 176.6 (C=O) ppm.

7′-(Benzylamino)-6′-nitrospiro[indoline-3,5′-pyrano[2,3-*d*]pyrimidine]-2,2′,4′(1′*H*,3′*H*)-trione (5g, $C_{21}H_{15}N_5O_6$) Orange solid; m.p.: 240–242 °C (dec.); yield: 0.294 g (68%); IR (KBr): $\bar{\nu}$ = 3422 and 3217 (NH), 1702 (C=O), 1687

(C=O), 1641 (C=O), 1605 (Ar), 1515 and 1326 (NO_2), 1461 and 1388 (NO_2) cm^{-1}; MS (EI, 70 eV): *m/z* (%) = 433 (M^+, 2), 417 (6), 283 (9), 240 (20), 197 (19), 168 (25), 133 (45), 91 (100), 51 (19); ^{1}H NMR (300 MHz, DMSO-d_6): δ = 4.63 (ABX, J_{AB} = 66 Hz, J_{AX} = J_{BX} = 5.7 Hz, δ_A = 4.52 and δ_B = 4.74 ppm, 2H), 7.18–7.72 (m, 9H), 8.20 (t, 1H), 9.37 (t, $^3J_{HH}$ = 5.7 Hz, 1H) ppm; ^{13}C NMR (75.4 MHz, DMSO-d_6): δ = 43.2 (CH_2), 44.4 (C-spiro), 118.5, 124.3, 126.6, 127.1, 127.6, 127.9, 128.1, 128.6, 128.8, 129.8, 133.6, 138.8, 140.1, 141.7, 147.9, 149.0, 163.5 (C=O) ppm.

7′-(Benzylamino)-1′,3′-dimethyl-6′-nitrospiro[indoline-3,5′-pyrano[2,3-*d*]pyrimidine]-2,2′,4′(1′*H*,3′*H*)-trione (5h, $C_{19}H_{19}N_5O_6$) White solid; m.p.: 306–312 °C (dec.); yield: 0.309 (67%); ^{1}H NMR (300 MHz, DMSO-d_6): δ = 2.96 (s, 3H), 3.23 (s, 3H), 4.82 (A_2X, d, J_{AX} = 6.0 Hz, δ_A = 4.82, 2H), 6.70–7.43 (m, 9H), 10.53 (s, 1H), 10.98 (t, $^3J_{HH}$ = 6.0 Hz, 1H) ppm; ^{13}C NMR (75.4 MHz, DMSO-d_6): δ = 28.2 (NCH_3), 29.9 (NCH_3), 45.9 (CH_2), 48.9 (C-spiro), 90.0, 108.2, 109.0, 121.3, 123.4, 127.2, 128.0, 128.7129.2, 130.8, 137.6, 144.9, 149.6, 150.6, 156.2 (C=O), 159.0 (C=O), 176.6 (C=O) ppm.

7′-(Benzylamino)-6′-nitro-2′-thioxo-2′,3′-dihydrospiro[indoline-3,5′-pyrano[2,3-*d*]pyrimidine]-2,4′(1′*H*)-dione (5i, $C_{21}H_{15}N_5O_5S_5$) Orange solid; m.p.: 240–242 °C (dec.); yield: 0.314 g (70%); ^{1}H NMR (300 MHz, DMSO-d_6): δ = 4.63 (ABX, J_{AB} = 65 Hz, J_{AX} = J_{BX} = 5.4 Hz, δ_A = 4.52 and δ_B = 4.74, 2H), 7.18–7.67 (m, 11H), 8.17 (br s, 1H), 9.35 (br s, 1H) ppm; ^{13}C NMR (75.4 MHz, DMSO-d_6): δ = 43.2 (CH_2), 44.5 (C-spiro), 118.5, 124.3, 126.6, 127.1, 127.6, 127.9, 128.1, 128.6, 128.8, 129.8, 133.6, 138.8, 140.1, 141.7, 147.9, 149.0, 163.5 (C=S) ppm.

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