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ORIGINAL PAPER

Synthesis of spirooxindole pyrimidines catalyzed by silica-bonded *N*-propyltriethylenetetramine as a recyclable solid base catalyst in aqueous medium

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Abstract Silica-bonded *N*-propyltriethylenetetramine was used as a heterogeneous solid base catalyst for the synthesis of spirooxindole pyrimidines by a one-pot condensation reaction of isatin, activated methylene reagent, and barbituric acid or thiobarbituric acid in aqueous medium in good to high yields. Catalyst could be recycled for several times without any additional treatment.

Keywords Heterogeneous catalyst · Solid base · Silica-bonded *N*-propyltriethylenetetramine · Barbituric acid · Spirooxindole pyrimidines · Water

Introduction

Spirooxindoles are commonly occurring heterocyclic ring systems and are important structural motifs found in many natural products and pharmaceuticals [1–5] (Fig. 1). For example, spirotryprostatins A and B (**1** and **2**) were isolated from the fermentation broth of *Aspergillus fumigatus* and have been shown to completely inhibit the G2/M progression of mammalian tsFT210 cells at concentrations in excess of 12.5 mg cm^{-3} [6, 7]. Pteropodine (**3**) has been shown to modulate the function of muscarinic serotonin receptors [8]. (−)-Horsfiline (**4a**) was isolated in 1991 [9] is one of the relatively unsubstituted spirooxindole cores. The related compound coerulescine (**4b**), which possesses an even simpler structure, was isolated in 1998; its synthesis is often reported together with that of horsfiline [10]. Further, strychnofoline (**5**) has been found to inhibit mitosis in a number of cell lines including mouse melanoma B16, Ehrlich, and Hepatom HW165 [11]. Alstonisine (**6**), a natural alkaloid, was first isolated from *Alstonia muelleriana* [12–14] and has been identified for its biomimetic transformations [15]. Chitosenine (**7**) is another structurally interesting natural product that exhibits short-lived inhibitory activity of ganglionic transmission "in vivo" in rats and rabbits [16–18].

In recent years, numerous efficient transformations have been developed for the construction of spirooxindole structures [19–42]. In 1998, Edmonson and Danishefsky reported the first total synthesis of spirotryprostatin A; a stereoselective bromohydrin formation was used for the oxidative rearrangement to form the spirooxindole [19, 20]. Also, the synthesis of the precursor to the oxidative rearrangement was made in a simple three-step sequence in 1997 by Ganesan and co-workers [21]. Horne and co-workers [23, 24] synthesized a number of spirooxindole natural products using a novel *N*-acyliminium cyclization of 2-halotryptamine derivatives. Fuji et al. [27, 28] developed an asymmetric nitroolefination protocol using a chiral auxiliary that was applied to the synthesis of chiral oxindoles. Overman and co-workers [29–32] used an aza-Cope-Mannich rearrangement to put together the pyrrolidine ring in order, the synthesis of asymmetric synthesis of strychnine. Carreira and co-workers [36–38] have developed a reliable methodology to access the pyrrolidinyl-spirooxindole structure starting from imines and spirocyclopropyl oxindoles. Williams accessed both enantiomers of the target molecule and prepared analogs of the natural products in the synthesis of spirotryprostatin B [39–41]. Gong and co-workers [42] described an enantioselective

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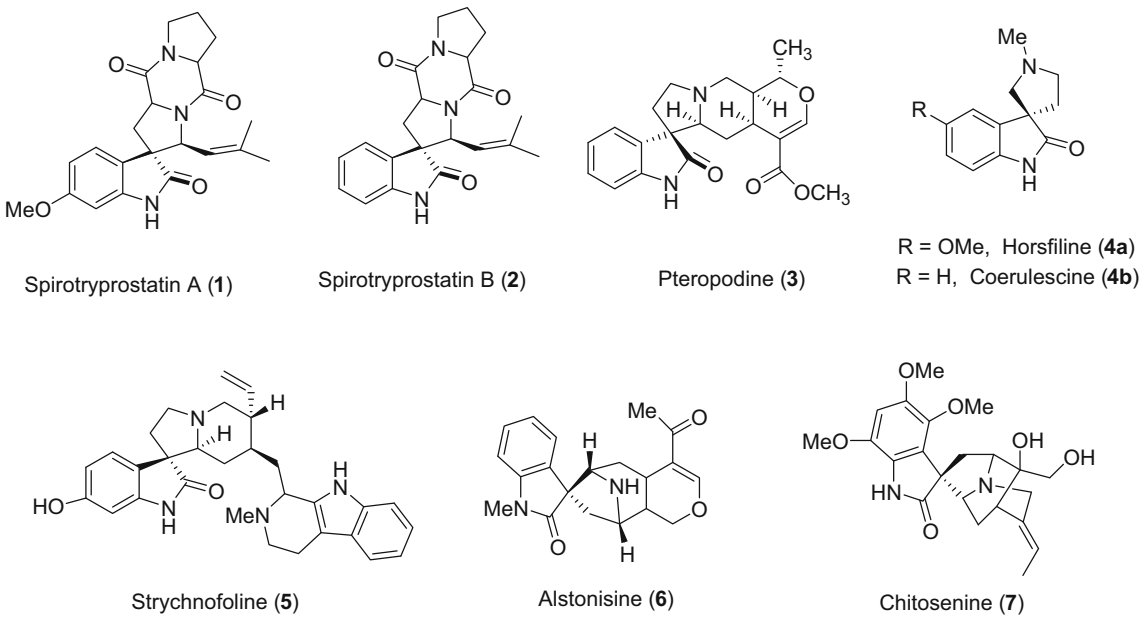


Fig. 1 Natural products based on spirooxindole core

organocatalytic approach for the rapid synthesis of spiro[pyrrolidin-3,3′-oxindole] derivatives in the presence of a chiral phosphoric acid.

Among them, the one-pot condensation reaction of isatin and activated methylene reagent could readily afford various spirooxindole pyramidine structures and has attracted wide attention, and this type of reaction has been achieved by a number of different catalysts and conditions, such as $InCl_3$ [43], electrochemical methods [44], triethylbenzylammonium chloride (TEBA) [45], Et_3N [46], tetrabutylammonium fluoride (TBAF) [47], NH_4Cl [48], lipase [4], *p*-TSA [49], ZnS nanoparticles [50], NaCl under sonication [51], butylmethylimidazolium tetrafluoroborate ([BMIm]BF_4) [52], amino-functionalized SBA-15 [53], $CaCl_2$ under ultrasonic irradiation [54], piperidine under ultrasonic irradiation [55], nanocrystalline MgO [56], gluconic acid [57], $MnFe_2O_4$ nanoparticles [58], 1,8-diazabicyclo[5.4.0]undec-7-ene (DBU) [59], ethylenediammonium diformate (EDDF) [60], and sulfated choline based heteropolyanion [61] which some of them suffer from technical intricacy and generation of mixtures of pyrans and unsaturated nitriles.

The potential use of microporous and mesoporous base catalysts in fine chemical production is enormous [62, 63]. These heterogeneous catalysts are known to suppress side reactions, which include self-condensation and oligomerization, resulting in better selectivity and product yield. Also, heterogeneous catalysts are advantageous over conventional homogeneous catalysts as they can be easily recovered from the reaction mixture by simple filtration and can be reused after activation, thereby making the process economically viable. Recently, we prepared silica-bonded *N*-propyltriethylenetetramine (SBNPTT) as solid base catalyst (Fig. 2) and its catalytic activity was investigated for the synthesis of 4,4′-(arylmethylene)bis(1*H*-pyrazol-5-ols) [63] and chromenes [64].

Along the line of our studies in application of heterogeneous catalysts in chemical transformations [63–73] herein, we wish to describe the application of SBNPTT as a solid base catalyst for the synthesis of spirooxindole derivatives.

Results and discussion

In our initial study, evaluation of a series of silica immobilized bases (SBNPTT, SBPP, SBPM, and 3-SPA) was carried out for the synthesis of spirooxindole pyrimidines in aqueous medium. After some preliminary experiments, it was found that a mixture of isatin, malononitrile, and barbituric acid in refluxing water in the presence of a catalytic amount of these heterogeneous solid bases could afford 7′-amino-2,2′,4′-trioxo-1′,2′,3′,4′-tetrahydrospiro[indoline-3,5′-pyrano[2,3-d]pyrimidine]-6′-carbonitrile one (**4a**) in excellent yield (Table 1).

We examined this reaction in the absence of catalyst and it was found that when the reaction was carried out without any catalyst resulted in poor yield (Table 1, entry 1). The best result was obtained when SBNPTT was used for which the yield was up to 95 % (Table 1, entry 3). In addition, the result of this condensation in the presence of other silica immobilized bases (3-SPA, SBPP, and SBPM) gave

Fig. 2 Silica immobilized bases

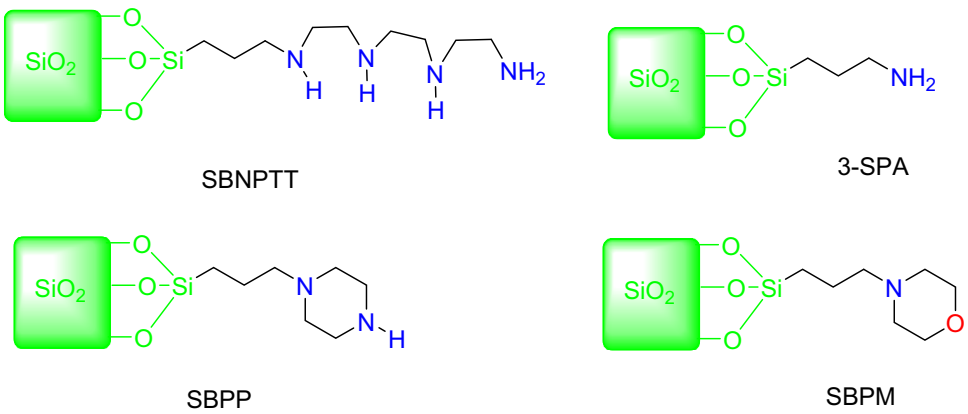
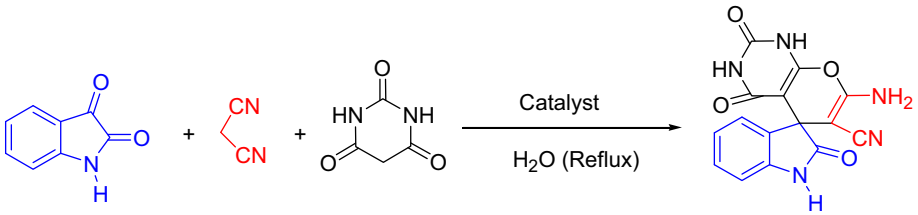


Table 1 Condensation reaction of isatin, malononitrile with barbituric acid in the presence of different amounts of catalysts



Entry	Catalyst	Catalyst loading/g	Time/min	Yield/%[a]
1	No catalyst	–	600	Trace
2	SBNPTT	0.01	60	82
3	SBNPTT	0.03	30	95
4	SBNPTT	0.05	30	95
5	3-SPA	0.03	40	91
6	SBPP	0.03	50	90
7	SBPM	0.03	60	89
8	SBNPTT	0.03	120	80[b]

Reaction conditions; isatin (1 mmol), malononitrile (1 mmol), barbituric acid (1 mmol), 3 cm^3 water, reflux conditions

[a] Isolated yield

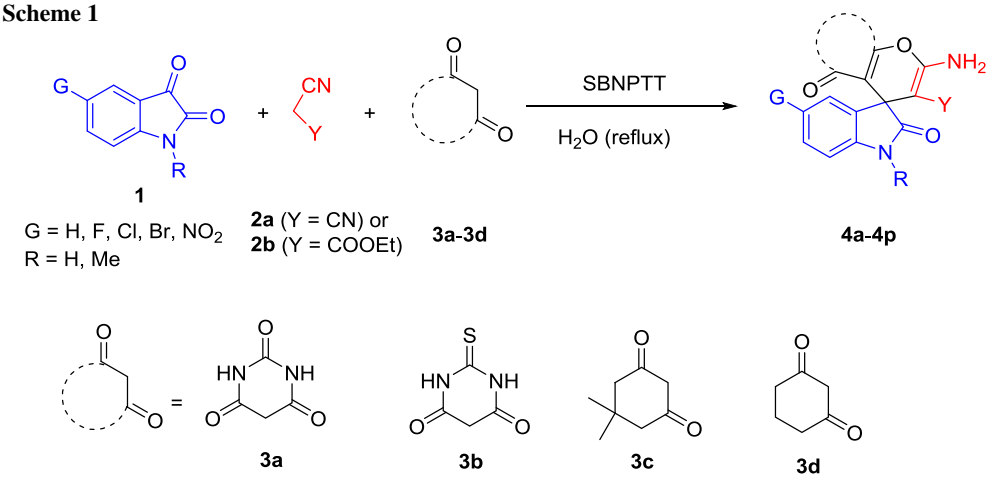
[b] The reaction was carried out at 120 °C under solvent-free conditions

corresponding product in slightly longer reaction time and lower yield (Table 1, entries 5–7). The condensation reaction under solvent-free conditions at 120 °C gave corresponding product in 80 % yield after 120 min (Table 1, entry 8). So, the optimal amount of SBNPTT was 0.03 g (2.77 mol% [63]) per 1 mmol of isatin in refluxing water.

Therefore, we employed the optimized conditions (0.03 g $mmol^{-1}$ of SBNPTT in refluxing water) for the condensation reaction of isatin, malononitrile with 1,3-dicarbonyl compounds [barbituric acid (**3a**), thiobarbituric acid (**3b**), dimedine (**3c**), and 1,3-cyclohexadione (**3d**)] into the corresponding spirooxindole derivatives (Scheme 1).

As shown in Table 2, it was found that this method works with a wide variety of substrates. A series of different position substituted isatins including either electron-withdrawing or halogen groups reacted with malononitrile and barbituric acid under optimized conditions and corresponding products were obtained in high yields. Also, thiobarbituric acid was treated with isatins and malononitrile gave corresponding products in good to high yields (Table 2, entries 6–8). In addition, dimedone and 1,3-cyclohexanedione were used in this three-component condensation reaction (Table 2, entries 9–16). Dimedone reacted with isatines and malononitrile or ethyl cyanoacetate under optimized conditions and corresponding products were obtained in high yields (Table 2, entries 9–14). The reaction with ethyl cyanoacetate or malononitrile also proceeded smoothly; however, the reaction time

Scheme 1



of ethyl cyanoacetate with isatins and 1,3-dicarbonyl compounds was longer than those of malononitrile, which is probably due to the lower reactivities of the cyanoacetates.

Proposed mechanism for the synthesis of spiro derivative **4** was described in Scheme 2 [45, 64, 72]. The process represents a typical cascade reaction in which the isatin **1** first condenses with malononitrile (**2a**) to afford isatylidene malononitrile derivative **5** in the presence of SBNPTT in water. This step was regarded as a fast Knoevenagel condensation. Then, **5** is attacked via Michael addition of 1,3-dicarbonyl compound **3** to give the intermediate **6** followed by the cycloaddition of hydroxyl group to the cyano moiety to form the desired product **4** (Scheme 2).

The possibility of recycling the catalyst SBNPTT was examined using the reaction of isatin, malononitrile, and barbituric acid under the optimized conditions. Upon completion, the reaction mixture was washed with warm ethanol (3×5 cm^3). The recovered catalyst was washed with diethyl ether, dried, and reused for subsequent runs. The recycled catalyst was reused four times without any additional treatment. No observation of any appreciable loss in the catalytic activity of SBNPTT was observed (Fig. 3).

In conclusion, we have prepared some new spirooxindole derivatives by three-component condensation reaction of isatin, reactive methylene reagents, with 1,3-dicarbonyl compounds in the presence of SBNPTT as a solid base catalyst in refluxing water.

Experimental

Chemicals were purchased from Fluka, Merck, and Aldrich chemical companies. Known compounds were characterized by comparison of their IR, ^{1}H NMR, and ^{13}C NMR spectroscopic data and their melting points with reported values [4, 5, 44–55].

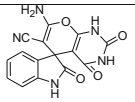
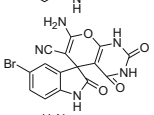
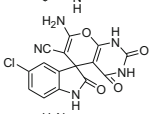
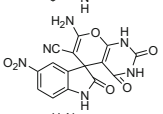
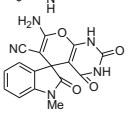
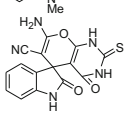
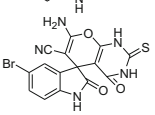
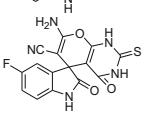
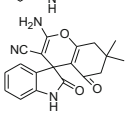
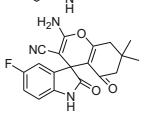
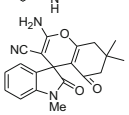
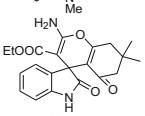
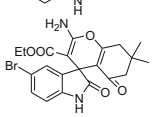
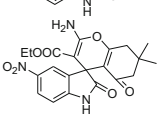
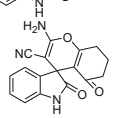
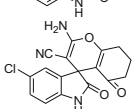
General procedure for the synthesis of spirooxindoles

A mixture of isatin (1 mmol), reactive methylene compound (1 mmol), and 1,3-dicarbonyl compound (1 mmol) in the presence of 0.03 g SBNPTT (2.77 mol%) in 3 cm^3 water was refluxed with stirring in an oil bath. The progress of the reaction was monitored by TLC. After completion of the reaction, the mixture was filtered and remaining was washed with warm ethanol (3×5 cm^3) to separate SBNPTT catalyst. After cooling the ethanol phase the precipitates were filtered. The crude products were purified by recrystallization from ethanol (95 %). The recovered catalyst was washed with diethyl ether, dried, and reused for subsequent runs.

7′-Amino-5-bromo-2,2′,4′-trioxo-1′,2′,3′,4′-tetrahydrospiro[indoline-3,5′-pyrano[2,3-d]pyrimidine]-6′-carbonitrile (**4b**, $C_{15}H_8BrN_5O_4$)
White solid; m.p.: 258–260 °C; IR (KBr): $\bar{\nu}$ = 3,450, 3,291, 3,160, 2,197, 1,693, 1,535 cm^{-1}; ^{1}H NMR (400 MHz, DMSO-d_6): δ = 6.70–6.87 (m, 1H), 7.23–7.80 (m, 4H), 10.57 (s, 1H, NH), 11.11 (s, 1H, NH), 12.28 (brs, 1H, NH) ppm; ^{13}C NMR (100 MHz, DMSO-d_6): δ = 48.5, 58.7, 87.9, 112.8, 115.1, 118.5, 128.4, 132.7, 137.6, 143.0, 150.9, 155.2, 160.0, 163.1, 178.7 ppm.

7′-Amino-5-chloro-2,2′,4′-trioxo-1′,2′,3′,4′-tetrahydrospiro[indoline-3,5′-pyrano[2,3-d]pyrimidine]-6′-carbonitrile (**4c**, $C_{15}H_8ClN_5O_4$)
White solid; m.p.: 237–240 °C; IR (KBr): $\bar{\nu}$ = 3,452, 3,292, 3,160, 2,196, 1,694, 1,535, 1,477, 1,335 cm^{-1}; ^{1}H NMR (400 MHz, DMSO-d_6): δ = 6.80 (d, 1H,

Table 2 Preparation of spirooxindole derivatives catalyzed by SBNPTT in water at reflux conditions (Scheme 1)

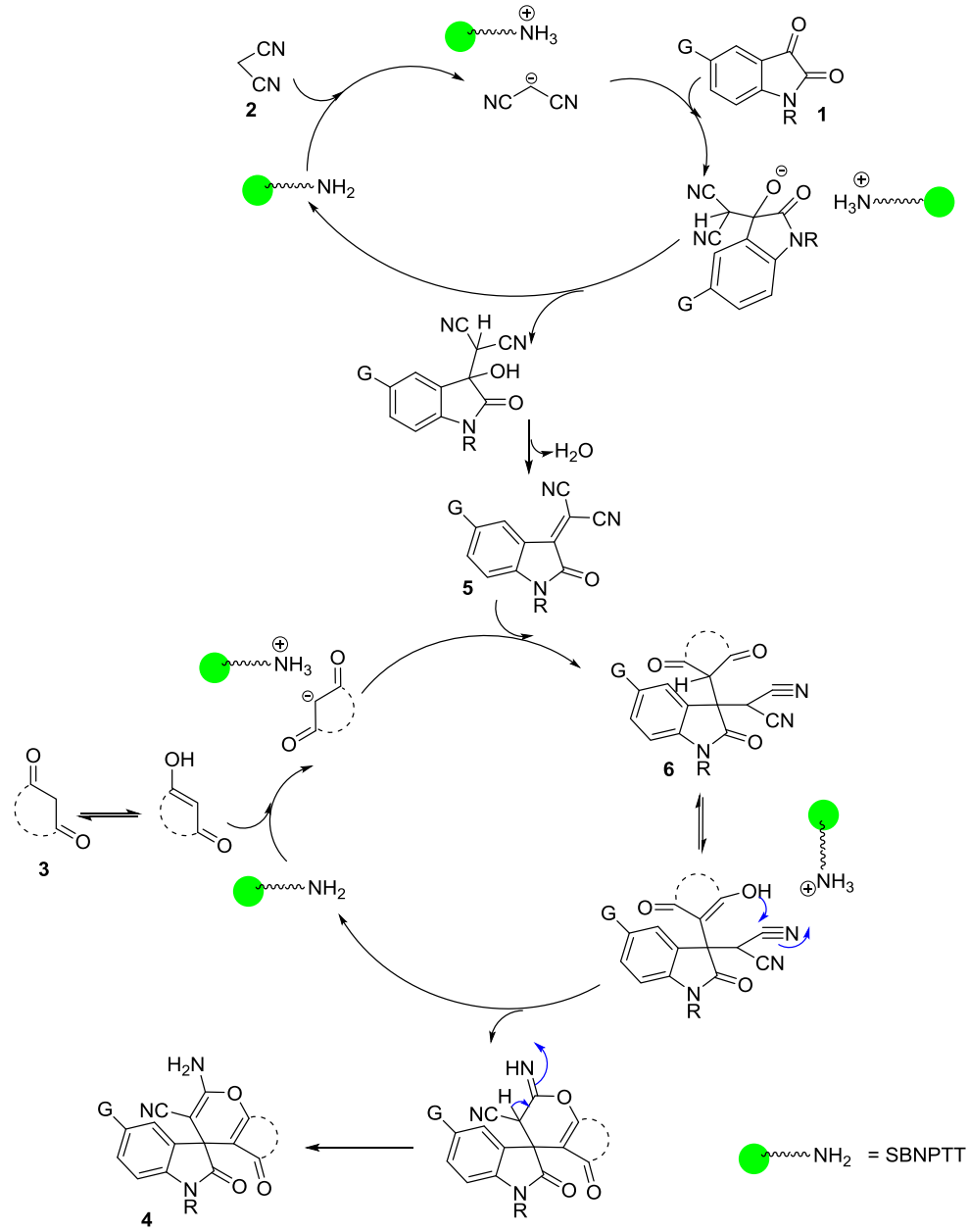
Entry	Dicarbonyl	G	Y	Product	Time /min	Yield /%[a]	M.p. /°C	Lit. m.p. /°C
1	**3a**	H	CN	 **4a**	30	95	276-278	275-276 [27]
2	**3a**	Br	CN	 **4b**	50	92	258-260	-
3	**3a**	Cl	CN	 **4c**	40	95	237-240	-
4	**3a**	NO_2	CN	 **4d**	25	92	286-288	-
5	**3a**	H	CN	 **4e**	50	97	240-242	-
6	**3b**	H	CN	 **4f**	60	95	235-237	238-242 [27]
7	**3b**	Br	CN	 **4g**	60	95	249-251	-
8	**3b**	F	CN	 **4h**	55	67	252-255	-
9	**3c**	H	CN	 **4i**	20	97	268-270	284-285 [4]
10	**3c**	F	CN	 **4j**	25	95	285-287	270-273 [30]
11	**3c**	H	CN	 **4k**	30	95	247-249	254-256 [27]
12	**3c**	H	CO_2Et	 **4l**	210	96	268-270	269-271 [4]
13	**3c**	Br	CO_2Et	 **4m**	210	95	247-249	294-296 [32]
14	**3c**	NO_2	CO_2Et	 **4n**	110	93	274-276	276-278 [33]
15	**3d**	H	CN	 **4o**	20	97	250-252	251-252 [30]
16	**3d**	Cl	CN	 **4p**	25	95	265-267	288-290 [4]

Reaction conditions: isatin (1 mmol), activated methylene reagents (1 mmol), 1,3-dicarbonyl compounds (1 mmol), 0.03 g catalyst SBNPTT in 3 cm^3 water at reflux conditions

[a] Isolated yield

$J = 8.1$ Hz), 7.21 (dd, 1H, $J_1 = 4.3$ Hz, $J_2 = 2.2$ Hz), 7.34 (d, 1H, $J = 2.2$ Hz), 7.45 (s, 2H, NH_2), 10.61 (s, 1H, NH), 11.16 (s, 1H, NH), 12.33 (brs, 1H, NH) ppm; ^{13}C NMR (100 MHz, DMSO-d_6): $\delta = 46.9$, 57.0, 86.2, 110.6, 116.8, 124.1, 125.8, 128.2, 135.6, 141.0, 153.5, 158.3, 161.5, 177.5 ppm.

Scheme 2



7′-Amino-5-nitro-2,2′,4′-trioxo-1′,2′,3′,4′-tetrahydrospiro[indoline-3,5′-pyrano[2,3-d]pyrimidine]-6′-carbonitrile (**4d**, $C_{15}H_8N_6O_6$)

White solid; m.p.: 286–288 °C; IR (KBr): $\bar{\nu}$ = 3,442, 3,296, 3,173, 2,199, 1,748, 1,687, 1,632, 1,521, 1,477, 1,345, 1,253, 1,230 cm^{-1}; ^1H NMR (400 MHz, DMSO-d_6): δ = 6.80–7.30 (m, 1H), 7.53 (s, 2H, NH_2), 8.07–8.40 (m, 2H), 11.15–11.30 (m, 2H, 2 × NH), 12.38 (brs, 1H, NH) ppm; ^{13}C NMR (100 MHz, DMSO-d_6): δ = 48.5, 57.7, 87.4, 111.0, 118.4, 121.6, 127.5, 136.2, 144.2, 150.2, 150.9, 155.5, 160.3, 163.3, 180.0 ppm.

7′-Amino-1′-methyl-2,2′,4′-trioxo-1′,2′,3′,4′-tetrahydrospiro[indoline-3,5′-pyrano[2,3-d]pyrimidine]-6′-carbonitrile (**4e**, $C_{16}H_{11}N_5O_4$)

White solid; m.p.: 240–242 °C; IR (KBr): $\bar{\nu}$ = 3,467, 3,383, 3,164, 2,206, 1,705, 1,610, 1,526, 1,470, 1,324, 1,244, 1,113 cm^{-1}; ^1H NMR (400 MHz, DMSO-d_6): δ = 3.19 (s, 3H), 7.04–7.07 (m, 2H), 7.26 (d, 1H, J = 6.3 Hz), 7.33 (dt, 1H, J_1 = 6.1 Hz, J_2 = 1.0 Hz), 7.48 (s, 2H, NH_2), 11.17 (s, 1H, NH), 12.39 (brs, 1H, NH) ppm; ^{13}C NMR (125 MHz, DMSO-d_6): δ = 26.9, 46.8, 57.8, 87.2, 108.7, 117.3, 123.0, 124.0, 129.1, 133.2, 144.1, 149.7, 153.8, 158.8, 161.8, 176.6 ppm.

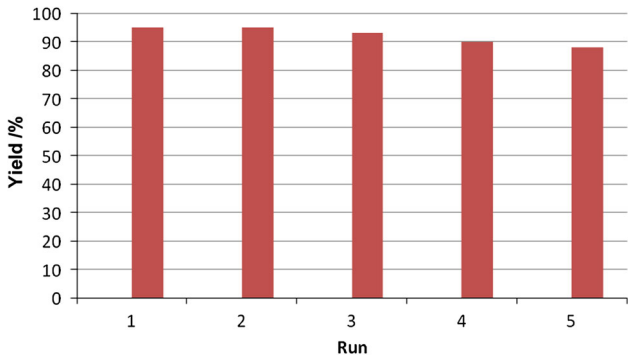


Fig. 3 Recyclability of SBNPTT as catalyst in the condensation reaction of isatin (1 mmol), malononitrile (1 mmol), and barbituric acid (1 mmol) in the presence of 0.03 g of SBNPTT in refluxing water. Reaction time: 30 min

7′-Amino-5-bromo-2,4′-dioxo-2′-thioxo-1′,2′,3′,4′-tetrahydrospiro[indoline-3,5′-pyrano[2,3-d]pyrimidine]-6′-carbonitrile (**4g**, $C_{15}H_8BrN_5O_3S$)
White solid; m.p.: 249–251 °C; IR (KBr): $\bar{\nu} = 3{,}426$, 3,311, 3,160, 2,200, 1,693, 1,655, 1,615, 1,570, 1,460 cm^{-1}; ^1H NMR (400 MHz, DMSO-d_6): $\delta = 6.77$ (s, 1H), 7.31–7.75 (m, 4H), 10.69 (s, 1H, NH), 12.54 (s, 1H, NH), 13.81 (brs, 1H, NH) ppm; ^{13}C NMR (100 MHz, DMSO-d_6): $\delta = 46.8$, 56.0, 90.9, 111.1, 113.6, 116.8, 127.0, 131.2, 135.4, 141.5, 152.9, 158.1, 159.2, 173.9, 177.0 ppm.

7′-Amino-5-fluoro-2,4′-dioxo-2′-thioxo-1′,2′,3′,4′-tetrahydrospiro[indoline-3,5′-pyrano[2,3-d]pyrimidine]-6′-carbonitrile (**4h**, $C_{15}H_8FN_5O_3S$)
White solid; m.p.: 252–255 °C; IR (KBr): $\bar{\nu} = 3{,}425$, 3,313, 3,161, 2,201, 1,694, 1,656, 1,615, 1,570, 1,469 cm^{-1}; ^1H NMR (400 MHz, DMSO-d_6): $\delta = 6.76$–6.79 (m, 1H), 6.97–7.02 (m, 1H), 7.23 (dd, 1H, $J_1 = 8.2$ Hz, $J_2 = 2.6$ Hz), 7.48 (s, 2H, NH_2), 10.56 (s, 1H, NH), 12.50 (s, 1H, NH), 13.84 (brs, 1H, NH) ppm; ^{13}C NMR (125 MHz, DMSO-d_6): $\delta = 47.6$, 57.4, 91.6, 110.4, 110.4, 112.4, 112.6, 115.2, 115.4, 117.2, 135.1, 135.2, 138.8, 153.4, 159.1 ($J_{C-F} = 128.2$ Hz), 174.5, 177.8 ppm.

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