A Novel Method for the Synthesis of Spiro[indoline-Pyrazolo[4′,3′:5,6]pyrido[2,3-*d*]pyrimidine]triones by Alum as a Reusable Catalyst

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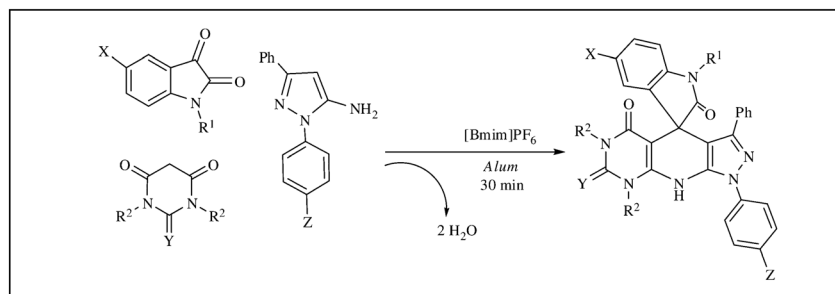[d]Immunology Research Center, Iran University of Medical Sciences, Tehran, Iran
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Synthesis of spiro[indoline-pyrazolo[4′,3′:5,6]pyrido[2,3-*d*]pyrimidine]trione derivatives by a cyclocondensation reaction of indolin-2-ones, barbituric acids, and 1,3-diphenyl-1*H*-pyrazol-5-amines with the ionic liquid as an effective green reaction media and in the presence of Alum as a reusable catalyst was reported. Excellent yields of products, green media, use of a reusable catalyst, and short reaction time are the main advantages of this new method.

INTRODUCTION

With the emphasis on the search for atom-efficient transformations of easily available starting materials into complex organic molecules, reactions that provide maximum diversity are especially desirable. Here, expeditious domino and multicomponent reactions (MCRs) have emerged as powerful strategies. These methodologies have great utility, particularly when they lead to the formation of privileged medicinal heterocyclic compounds. MCRs are economically and environmentally very advantageous, because multistep syntheses produce considerable amounts of waste mainly due to complex isolation procedures often involving expensive, toxic, and hazardous solvents after each step [1–3].

In recent years, the synthesis of combinatorial small-molecule heterocyclic libraries has emerged as a valuable tool in the search for novel lead structures. Thus, the success of combinatorial chemistry in drug discovery is considerably dependent on further advances in heterocyclic MCR methodology [4].

Polyfunctionalized heterocyclic compounds play important roles in the drug discovery process, and analysis of drugs in late development or on the market shows that 68% of them are heterocycles. Therefore, it is not surprising that research in the field of synthesis of polyfunctionalized heterocyclic compounds has received special attention [1].

Spirocyclic systems containing one carbon atom common to two rings are structurally interesting [5]. The asymmetric characteristic of the molecule due to the chiral spiro carbon is one of the important criteria of the biological activities. The presence of the sterically constrained spiro structure in various natural products also adds to the interest in the investigations of spiro compounds [6]. Spiro compounds represent an important class of naturally occurring substances characteristic by their highly pronounced biological properties [7,8].

The heterocyclic spirooxindole ring system is a widely distributed structural framework present in a number of pharmaceuticals and natural products [9], including such cytostatic alkaloids as spirotryprostatins A, B, and strychnophylline (Fig. 1) [10–12]. The unique structural array and the highly pronounced pharmacological activity displayed by the class of spirooxindole compounds have made them attractive synthetic targets [13].
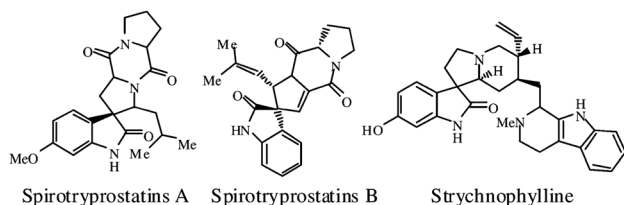
Figure 1. Representatives of important indenone-fused heterocycles.

Scheme 1

Further, the chemistry of spiro-indoles in which an indole ring is joined to sulfur- and nitrogen-containing heterocycles at the C-3 position through a spiro carbon atom is of great interest due to their physiological and biological activities [14,15].

In addition, the synthesis of pyridopyrimidine and their derivatives is of high interest in organic chemistry due to their potential biological and pharmacological activities such as antiviral, anti-inflammatory, insecticidal, antifolate, tyrosine kinase inhibitor, antimicrobial, calcium channel antagonists, antileishmanial, diuretic, and potassium-sparing [16–24].

In continuation of our previous works for the synthesis of heterocyclic compounds [25–30], recently, we reported novel one-pot, three-component synthesis of spiro[indoline-pyrazolo[4′,3′:5,6]pyrido[2,3-d]pyrimidine]trione in refluxing condition at 24 h [31]. Herein, we report a simple and efficient method for the synthesis of these spirooxindoles with fused pyrazole, pyridine, and pyrimidine rings, through the three-component one-pot reaction in ionic liquid media, using Alum as a catalyst at very short reaction times.

RESULTS AND DISCUSSION

In this research, to reach the efficiency of three-component reaction of isatin **1a**, barbirutic acid **2a**, and 1,3-diphenyl-1*H*-pyrazol-5-amine **3a**, various conditions were considered.

At first, we examined this simple model reaction in the presence of diverse types of lewis and bronsted acids such as Alum, SSA, SnCl$_4$, *p*-TSA, and CAN as catalysts. We also tested the reaction in different ionic liquids media such as [Bmim]Br, [Bmim]Cl, [Bmim]CF$_3$COO, [Bmim]BF$_4$, and [Bmim]PF$_6$. We have found that use of Alum has a unique capability and best promoter to enhance the reaction rate in [Bmim]PF$_6$ medium (Scheme 1). The results are summarized in Table 1.

As follow, we evaluated the amount of catalyst required for this transformation. It was found that using 10 mol % Alum in ionic liquid is sufficient to push the reaction forward. We also checked the reusability of the catalyst by recovering the Alum and using it for new runs and found that the catalyst could be reused several times without any
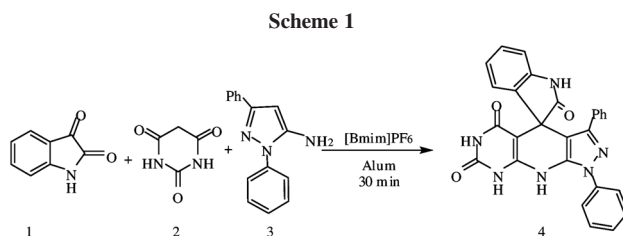
decrease in the product yield. Apparently, recycling of catalyst is possible for three successive times without significant loss of activity (Table 2, entry **4c**).

After optimizing the conditions, to delineate this approach, particularly in regard to library construction, this methodology was evaluated by using different barbituric acids, 1,3-diphenyl-1*H*-pyrazol-5-amine, and isatins. Five substituted isatins **1(a–e)**, three commercially available barbituric acids **2(a–c)** and two 1,3-diphenyl-1*H*-pyrazol-5-amine **3(a,b)**, were chosen for the library validation (Scheme 2).

Corresponding spiro[indoline-pyrazolo[4′,3′:5,6]pyrido[2,3-d]pyrimidine]triones **4(a–t)** were synthesized by the one-pot, three-component condensation reaction in excellent yields at 100°C in the presence of catalytic amount of Alum for 30 min. The reaction can be represented as in Table 2.

EXPERIMENTAL

Melting points were measured on an Elecrtothermal 9100 apparatus. ^{1}H-NMR spectra were recorded on a BRUKER DRX-300 AVANCE spectrometer at 300.13 MHz. The starting materials and TLC papers were obtained from Merck and Aldrich.

All the products **4(a–t)** are known compounds and were characterized by comparison of their ^{1}H-NMR spectrum with authentic samples synthesized by reported procedures [31].

Table 1

Model reaction, conditions, and yields.[a]

Conditions	Catalyst	Time (min)	Temp. (°C)	Yield (%)
[Bmim]Br	CAN	30	100	<50
[Bmim]Br	*p*-TSA	30	100	60
[Bmim]Br	SnCl$_4$	30	100	57
[Bmim]Br	SSA	30	100	62
[Bmim]Br	Alum	30	100	70
[Bmim]CF$_3$CO$_2$	Alum	30	100	82
[Bmim]Cl	Alum	30	100	63
[Bmim]BF$_4$	Alum	30	100	76
[Bmim]PF$_6$	Alum	30	100	95

[a]Isatin (1 mmol), barbituric acid (1 mmol), 1,3-diphenyl-1*H*-pyrazol-5-amine (1 mmol), catalyst (10 mol %), and ionic liquid (0.2 g).

Table 2

Model reaction and yield.

Product[a]	R^1	R^2	X	Y	Z	Yield[b] (%)
4a	H	H	H	O	H	95
4b	H	H	Br	O	H	96
4c	H	H	NO_2	O	H	98 (98,96,96)[c]
4d	Me	H	H	O	H	92
4e	H	H	Me	O	H	88
4f	Et	H	H	O	H	86
4g	Me	H	NO_2	O	H	87
4h	Me	H	Br	O	H	84
4i	Et	H	Br	O	H	83
4j	H	Me	Br	O	H	89
4k	H	Me	NO_2	O	H	91
4l	Me	Me	H	O	H	85
4m	H	H	H	O	NO_2	89
4n	H	H	Br	O	NO_2	88
4o	H	H	NO_2	O	NO_2	90
4p	H	H	Me	O	NO_2	84
4q	H	H	H	S	H	85
4r	H	H	Br	S	H	89
4s	H	H	NO_2	S	H	91
4t	Me	H	H	S	H	83

[a]All the products are known compounds [31].
[b]Isolated yields.
[c]Isolated yield after recycling of catalyst.

Typical procedure for the preparation of spiro[indoline-pyrazolo[4′,3′:5,6]pyrido[2,3-d]pyrimidine]triones (4a). A mixture of barbituric acid (1 mmol), 1,3-diphenyl-1H-pyrazol-5-amine (1 mmol), isatin (1 mmol), [Bmim]PF$_6$ (0.2 g), and Alum (0.02 g) was stirred for 30 min at 100°C (the progress of the reaction was monitored by TLC). After completion, the reaction mixture dissolved in water and filtered then the precipitate washed with water (10 mL) and recrystallized by EtOH to afford the pure product **4** as Cream powder (95%). m.p > 300°C.

^{1}H-NMR (300 MHz, DMSO-d_6): δ$_H$ (ppm) 6.50–7.69 (m, 14H, H-Ar), 9.36 (s, 1H, NH), 9.93 (s, 1H, NH), 10.18 (s, 1H, NH), 10.67 (s, 1H, NH).

5-Bromo-1′,3′-diphenyl-spiro[indoline-3,4′-pyrazolo[4′,3′:5,6]pyrido[2,3-d]pyrimidine]-2,5′, 7′(6′H,8′H,9′H)-trione (4b). White powder (96%); m.p > 300°C. ^{1}H-NMR (300 MHz, DMSO-d_6): δ$_H$ (ppm) 6.43–7.70 (m, 13H, H-Ar), 9.43 (s, 1H, NH), 10.12 (s, 1H, NH), 10.19 (s, 1H, NH), 10.77 (s, 1H, NH).

5-Nitro-1′,3′-diphenyl-spiro[indoline-3,4′-pyrazolo[4′,3′:5,6]pyrido[2,3-d]pyrimidine]-2,5′, 7′(6′H,8′H,9′H)-trione (4c). White powder (98%); m.p > 300°C. ^{1}H-NMR (300 MHz, DMSO-d_6): δ$_H$ (ppm) 6.63–8.06 (m, 13H, H-Ar), 9.54 (s, 1H, NH), 10.28 (s, 1H, NH), 10.73 (s, 1H, NH), 10.82 (s, 1H, NH).

1-Methyl-1′,3′-diphenyl-spiro[indoline-3,4′-pyrazolo[4′,3′:5,6]pyrido[2,3-d]pyrimidine]- 2,5′, 7′(6′H,8′H,9′H)-trione (4d). White powder (92%); m.p > 300°C. ^{1}H-NMR (300 MHz, DMSO-d_6): δ$_H$ (ppm) 2.67 (s, 3H, CH3), 6.60–7.70 (m, 14H, H-Ar), 9.45 (s, 1H, NH), 10.24 (s, 1H, NH), 10.71 (s, 1H, NH).

5-Methyl-1′,3′-diphenyl-spiro[indoline-3,4′-pyrazolo[4′,3′:5,6]pyrido[2,3-d]pyrimidine]-2,5′, 7′(6′H,8′H,9′H)-trione (4e). White powder (88%); m.p > 300°C. ^{1}H-NMR (300 MHz, DMSO-d_6): δ$_H$ (ppm) 2.21 (s, 3H, CH$_3$), 6.42–7.70 (m, 13H, H-Ar), 9.38 (s, 1H, NH), 9.86 (s, 1H, NH), 10.20 (s, 1H, NH), 10.70 (s, 1H, NH).

1-Ethyl-1′,3′-diphenyl-spiro[indoline-3,4′-pyrazolo[4′,3′:5,6]pyrido[2,3-d]pyrimidine]-2,5′, 7′(6′H,8′H,9′H)-trione (4f). White powder (86%); m.p > 300°C. ^{1}H-NMR (300 MHz, DMSO-d_6): δ$_H$ (ppm) 0.77 (t, J = 6.93 Hz, 3H, CH$_3$), 3.10–3.35 (m, 2H, CH$_2$), 6.56–7.70 (m, 14H, H-Ar), 9.44 (s, 1H, NH), 10.23 (s, 1H, NH), 10.70 (s, 1H, NH).

1-Methyl-5-nitro-1′,3′-diphenyl-spiro[indoline-3,4′-pyrazolo[4′,3′:5,6]pyrido[2,3-d]pyrimidine]-2,5′,7′(6′H,8′H,9′H)-trione (4g). White powder (87%); m.p > 300°C. ^{1}H-NMR (300 MHz, DMSO-d_6): δ$_H$ (ppm) 2.78 (s, 3H, CH$_3$), 6.66–8.12 (m, 13H, H-Ar), 9.58 (s, 1H, NH), 10.29 (s, 1H, NH), 10.82 (s, 1H, NH).

1-Methyl-5-bromo-1′,3′-diphenyl-spiro[indoline-3,4′-pyrazolo[4′,3′:5,6]pyrido[2,3-d]pyrimidine]-2,5′,7′(6′H,8′H,9′H)-trione (4h). White powder (84%); m.p > 300°C. ^{1}H-NMR (300 MHz, DMSO-d_6): δ$_H$ (ppm) 2.67 (s, 3H, CH$_3$), 6.59–7.71 (m, 13H, H-Ar), 9.48 (s, 1H, NH), 10.22 (s, 1H, NH), 10.78 (s, 1H, NH).

1-Ethyl-5-bromo-1′,3′-diphenyl-spiro[indoline-3,4′-pyrazolo[4′,3′:5,6]pyrido[2,3-d]pyrimidine]-2,5′,7′(6′H,8′H,9′H)-trione (4i). White powder (83%); m.p > 300°C. ^{1}H-NMR (300 MHz, DMSO-d_6): δ$_H$ (ppm) 0.78 (t, J = 6.90 Hz, 3H, CH$_3$), 3.08–3.39 (m, 2H, CH$_2$), 6.63–7.71 (m, 13H, H-Ar), 9.47 (s, 1H, NH), 10.21 (s, 1H, NH), 10.77 (s, 1H, NH).

5-Bromo-6′,8′-dimethyl-1′,3′-diphenyl-spiro[indoline-3,4′-pyrazolo[4′,3′:5,6]pyrido[2,3-d]pyrimidine]2,5′,7′(6′H,8′H,9′H)-trione (4j). White powder (89%); m.p > 300°C. ^{1}H-NMR (300 MHz, DMSO-d_6): δ$_H$ (ppm) 3.03 (s, 3H, CH$_3$), 3.54 (s, 3H, CH$_3$), 6.41–7.80 (m, 13H, H-Ar), 9.73 (s, 1H, NH), 10.14 (s, 1H, NH).
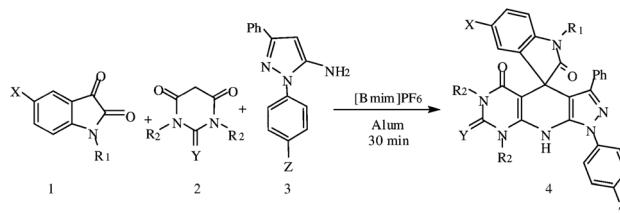
5-Nitro-6′,8′-dimethyl-1′,3′-diphenyl-spiro[indoline-3,4′-pyrazolo[4′,3′:5,6]pyrido[2,3-d]pyrimidine] 2,5′,7′(6′H,8′H,9′H)-trione (4k). White powder (91%); m.p >300°C. ^{1}H-NMR (300 MHz, DMSO-d_6): δ$_H$ (ppm) 3.01 (s, 3H, CH$_3$), 3.56 (s, 3H, CH$_3$), 6.61–8.06 (m, 13H, H-Ar), 9.86 (s, 1H, NH), 10.75 (s, 1H, NH).

1,6′,8′-Trimethyl-1′,3′-diphenyl-spiro[indoline-3,4′-pyrazolo[4′,3′:5,6]pyrido[2,3-d]pyrimidine] 2,5′,7′(6′H,8′H,9′H)-trione (4l). White powder (85%); m.p >300 °C. ^{1}H-NMR (300 MHz, DMSO-d_6): δ$_H$ (ppm) 2.68 (s, 3H, CH$_3$), 3.01 (s, 3H, CH$_3$), 3.57 (s, 3H, CH$_3$), 6.60–7.81 (m, 14H, H-Ar), 9.77 (s, 1H, NH).

1′-Phenyl-3′-(4-nitrophenyl)-spiro[indoline-3,4′-pyrazolo[4′,3′:5,6]pyrido[2,3-d]pyrimidine]2,5′,7′(6′H,8′H,9′H)-trione (4m). White powder (89%); m.p > 300°C. ^{1}H-NMR (300 MHz, DMSO-d_6): δ$_H$ (ppm) 6.49–7.96 (m, 9H, H-Ar), 7.97 (d, J = 9.0 Hz, 2H, H-Ar), 8.45 (d, J = 9.0 Hz, 2H, H-Ar), 9.64 (s, 1H, NH), 9.99 (s, 1H, NH), 10.52 (s, 1H, NH), 10.77 (s, 1H, NH).

5-Bromo-1′-phenyl-3′-(4-nitrophenyl)-spiro[indoline-3,4′-pyrazolo[4′,3′:5,6]pyrido[2,3-d]pyrimidine]2,5′,7′(6′H,8′H,9′H)-trione (4n). White powder (88%); m.p > 300°C. ^{1}H-NMR (300 MHz, DMSO-d_6): δ$_H$ (ppm) 6.63–7.99 (m, 8H, H-Ar), 7.98 (d, J = 8.8 Hz, 2H, H-Ar), 8.46 (d, J = 8.7 Hz, 2H, H-Ar), 9.68 (s, 1H, NH), 10.14 (s, 1H, NH), 10.48 (s, 1H, NH), 10.83 (s, 1H, NH).

Scheme 2

5-Nitro-1′-phenyl-3′-(4-nitrophenyl)-spiro[indoline-3,4′-pyrazolo[4′,3′:5,6]pyrido[2,3-d]pyrimidine]2,5′,7′(6′H,8′H,9′H)-trione (4o). White powder (90%); m.p > 300°C. ¹H-NMR (300 MHz, DMSO-d_6): δ_H (ppm) 6.60–8.46 (m, 12H, H-Ar), 9.77 (s, 1H, NH), 10.55 (s, 1H, NH), 10.74 (s, 1H, NH), 10.87 (s, 1H, NH).

1-Methyl-1′-phenyl-3′-(4-nitrophenyl)-spiro[indoline-3,4′-pyrazolo[4′,3′:5,6]pyrido[2,3-d]pyrimidine]2,5′,7′(6′H,8′H,9′H)-trione (4p). White powder (84%); m.p > 300°C. ¹H-NMR (300 MHz, DMSO-d_6): δ_H (ppm) 2.66 (s, 3H, CH₃), 6.61–7.30 (m, 9H, H-Ar), 7.97 (d, J = 9.0 Hz, 2H, H-Ar), 8.46 (d, J = 9.0 Hz, 2H, H-Ar), 9.70 (s, 1H, NH), 10.55 (s, 1H, NH), 10.78 (s, 1H, NH).

1′,3′-Diphenyl-7′-thioxo-spiro[indoline-3,4′-pyrazolo[4′,3′:5,6]pyrido[2,3-d]pyrimidine]-2,5′(6′H,8′H,9′H)-dione (4q). White powder (85%); m.p > 300°C. ¹H-NMR (300 MHz, DMSO-d_6): δ_H (ppm) 6.52–7.71 (m, 14H, H-Ar), 9.41 (s, 1H, NH), 10.04 (s, 1H, NH), 11.75 (s, 1H, NH), 12.17 (s, 1H, NH).

5-Bromo-1′,3′-diphenyl-7′-thioxo-spiro[indoline-3,4′-pyrazolo[4′,3′:5,6]pyrido[2,3-d]pyrimidine]-2,5′(6′H,8′H,9′H)-dione (4r). White powder (89%); m.p >300°C. ¹H-NMR (300 MHz, DMSO-d_6): δ_H (ppm) 6.45–7.68 (m, 13H, H-Ar), 9.44 (s, 1H, NH), 10.21 (s, 1H, NH), 11.73 (s, 1H, NH), 12.24 (s, 1H, NH).

5-Nitro-1′,3′-diphenyl-7′-thioxo-spiro[indoline-3,4′-pyrazolo[4′,3′:5,6]pyrido[2,3-d]pyrimidine]-2,5′(6′H,8′H,9′H)-dione (4s). White powder (91%); m.p > 300°C. ¹H-NMR (300 MHz, DMSO-d_6): δ_H (ppm) 6.65–8.01 (m, 13H, H-Ar), 9.54 (s, 1H, NH), 10.82 (s, 1H, NH), 11.79 (s, 1H, NH), 12.28 (s, 1H, NH).

1-Methyl-1′,3′-diphenyl-7′-thioxo-spiro[indoline-3,4′-pyrazolo[4′,3′:5,6]pyrido[2,3-d]pyrimidine]-2,5′(6′H,8′H,9′H)-dione (4t). White powder (83%); m.p >300°C. ¹H-NMR (300 MHz, DMSO-d_6): δ_H (ppm) 2.69 (s, 3H, CH₃), 6.62–7.71 (m, 14H, H-Ar), 9.46 (s, 1H, NH), 11.77 (s, 1H, NH), 12.19 (s, 1H, NH).

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