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ORIGINAL PAPER

A regioselective and diastereoselective synthesis of new spiro-isoxazolidines via 1,3-dipolar cycloaddition of stable isatin ketonitrone and various dipolarophiles

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Abstract The 1,3-dipolar cycloaddition of stable isatin ketonitrone with various dipolarophiles has been conducted under classical, ionic liquid, and solvent-free conditions to give novel spiro[oxindole-isoxazolidine] derivatives with similar diastereoselectivity. In the presence of the ionic liquid 1-butyl-3-methylimidazolium bromide highly diastereoselective and regioselective cyclocondensation products were obtained in good to excellent yields in the absence of any catalyst. The reaction workup was simple and the ionic liquid was easily recovered from the reaction and reused.

Keywords Isatin ketonitrone · 1,3-Dipolar cycloaddition · Ionic liquids · Spiro[oxindole-isoxazolidine] derivatives

Introduction

The reactivity of nitrones has been studied widely, because they are very useful intermediates in organic synthesis, predominantly in 1,3-dipolar cycloaddition chemistry [1–5]. These intermediates are particularly useful in synthetic approaches toward natural products and biologically active compounds [6, 7]. Many endeavors have been devoted to the aldonitrones, mainly because of their ease of preparation via the condensation of an *N*-alkylhydroxylamine or *N*-arylhydroxylamine with an aldehyde. In contrast, the preparation of ketonitrones may not always be accomplished by simple condensation reactions, and occasional reports of synthetic routes toward these compounds are typically of narrow scope and are rare for linear ketones [8–16].

Isatin ketonitrones belong to the group of rarely found stable nitrones for which few synthetic procedures have been reported so far [17, 18]. Thus, the application of such 1,3-dipolar compounds in organic synthesis remains a challenge for chemists because of their low reactivity.

Isoxazolidines are structural key moieties in many bioactive substances and herbicides [19]. They are also easily ring opened to the aminoalcohols which can serve as attractive building blocks for the construction of natural products and other important bioactive substances [20]. The most important biological and pharmacological properties of isoxazolidines are antitumor [21, 22], antibacterial [23, 24], antimalarial [25], and anti-HIV activity [26]. Therefore, these considerations prompted us to attempt, as a part of our ongoing research program [27–32], to establish an efficient and robust novel synthetic procedure for a rare class of new spiro[oxindole-isoxazolidine] derivatives generated from stable isatin ketonitrone and various dipolarophiles, regioselectively and diastereoselectively by 1,3-dipolar cycloaddition under classical and solvent-free conditions. Because of the principles of green chemistry and in order to use eco-friendly conditions [33], we have developed the synthesis of these heterocyclic building blocks in neutral 1-butyl-3-methylimidazolium bromide ionic liquid as a green solvent.

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Results and discussion

Condensation of isatin **1** or isatin imine **2** with phenylhydroxylamine led to the formation of stable isatin ketonitrone **3** in high yield. The product was obtained nearly quantitatively when isatin imine **2** was used. An intense contour between H_4 and hydrogen atoms of the phenyl ring (N-Ph) in the ROESY spectrum showed the configuration of the stable isatin ketonitrone **3** was *Z* and not *E*. This was also confirmed from the NOE experiment (Scheme 1).

The 1,3-dipolar cycloaddition reaction of stable isatin ketonitrone (*Z*)-**3** with various cyclic and acyclic dipolarophiles (**4** and **5**; Scheme 2) was then studied. This resulted in the formation of a series of new spiro[oxindole-isoxazolidine] derivatives **6** and **7** in a highly regioselective and diastereoselective manner. As can be seen in Table 1, the reaction was primarily carried out under two different conditions. When the reaction was conducted in acetonitrile under reflux, yields of the products were moderate to good after 1 h. The process led to similar yields but with much longer reaction times under solvent-free conditions.

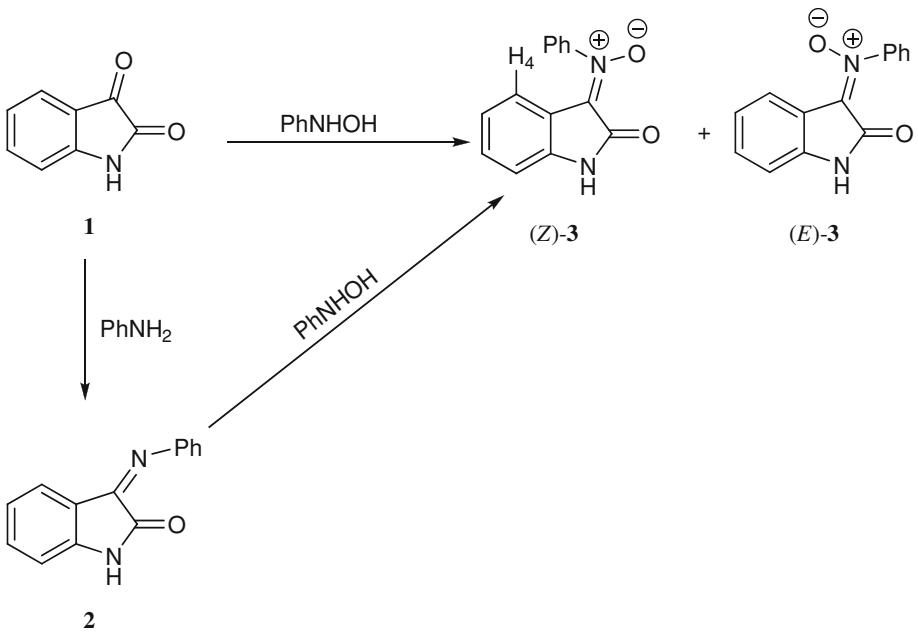
Because of the importance of green chemistry and the disadvantages of classical and solvent-free methods, the application of environmentally friendly media for this procedure was taken into account leading to accomplishing the reaction of isatin ketonitrone **3** with different dipolarophiles in [bmim]X (X = Br, PF_6, BF_4) ionic liquids as part of green alternative solvents known as designer solvents [33]. As can be seen in Table 2, the products were obtained in higher yields with high regioselectivity and diastereoselectivity and more rapidly than with the above mentioned classical and solvent-free conditions. The best results were obtained when [bmim]Br was used at 70 °C for 15 min; under these mild conditions the products were obtained using simple work-up and no impurities were observed by TLC. Recovery and reusability of [bmim]Br were the other advantages of using ILs as solvents. Reactivity in IL did decrease substantially even after four runs of reuse in the model reaction (**6d**; Table 2).

The exact stereochemistry of products **6** and **7** was determined using ^{1}H and ^{13}C and several 2D NMR spectroscopic techniques. For example, the ^{1}H NMR spectrum of **6a** contains two doublet signals at $\delta = 4.29$ ($J = 7.7$ Hz) and 5.61 ($J = 7.7$ Hz) ppm which are related to H_a, H_b. An intense contour between H_a and H_b in the ROESY spectrum shows that these two hydrogens are *cis* to each other. Also, the relative stereochemistry of **6a** was defined by the observation of an NOE from H_a to H_b and H_4. Therefore, the correct stereochemistry could be as shown in Scheme 2.

The regiochemistry proposed for product **7a** in Scheme 2 was established on the basis of its ^{1}H NMR spectrum containing a doublet of doublets at $\delta = 5.61$–5.68 ppm, attributed to the H_a proton and two other doublet of doublets signals at 3.04–3.08 and 3.32–3.38 ppm, corresponding to two diastereotopic protons named H_b. This regiochemistry was also confirmed from the 2D HMBC NMR spectrum. The H_b signals make HMBC cross peaks with carbons C2 and C3a. Also the NOE between H_a, H_b, and H_4 was observed proving the observed stereochemistry.

According to the stereochemistry of the cycloadducts, it can be suggested that the pathway of this reaction would be

Scheme 1



Scheme 2

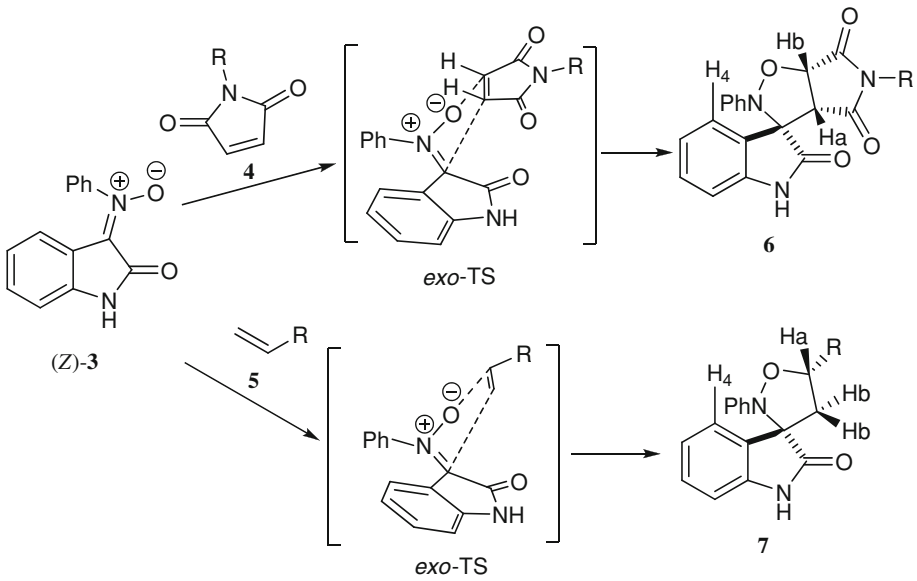


Table 1 Results of the reaction between isatin ketonitrone and various dipolarophiles

Dipolarophile	R	Yield (%) of products **6** and **7**[a]	
		Classic/CH_3CN, 45 min	Solvent-free/ 70 °C, 3 h
4a	C_6H_5	55	55
4b	4-Cl–C_6H_4	53	54
4c	4-Br–C_6H_4	58	54
4d	H	54	56
4e	C_2H_5	56	57
5a	CN	40	50
5b	Ph	37	45

[a] Isolated yield

Table 2 Results of the reaction between isatin ketonitrone and a variety of dipolarophiles under green reaction conditions

Product	Yield (%)[a] [bmim]PF_6	Yield (%)[a] [bmim]BF_4	Yield (%)[a] [bmim]Br/70 °C, 15 min
6a	63	60	72
6b	73	65	78
6c	71	73	81
6d	80	75	85 (84, 84, 80)[b]
6e	79	82	85
7a	52	50	68
7b	48	42	68

[a] Isolated yield

[b] Ionic liquid used four times

through the *exo* transition state. It is highly possible that the *endo* transition state is disfavored because of steric interaction of the phenyl group and benzene moiety on the nitrone with *R* substituents of the dipolarophile.

We herein report, for the first time, the synthesis of a rare class of new spiro[oxindole-isoxazolidine] derivatives **6** and **7**, with special biological and pharmacological properties, by highly regioselective and diastereoselective 1,3-dipolar cycloaddition reaction between stable isatin ketonitrone (*Z*)-**3** and various dipolarophiles (**4** and **5**) under classical, solvent free, and ionic liquid conditions. In general, the ionic liquid was found to be an excellent medium for the 1,3-dipolar cycloaddition reaction, and it promotes the reaction between unreactive isatin ketonitrone and various dipolarophiles. Thus, application of this green solvent was advantageous because it produced cleaner products in greater yields, and the reaction proceeded faster in comparison with the other conditions. All conditions produced identical diastereomers, as evidenced by TLC and NMR analysis.

Experimental

Melting points were determined by use of a electrothermal 9100 apparatus. FT-IR spectra were recorded on a Bomen FT-IR-MB-Series instrument. ^1H and ^{13}C NMR spectra were recorded, using TMS as an internal standard, on a Bruker DRX-300 Avance instrument, at 300.13 and

75.47 MHz, respectively. Mass spectra were recorded on a Finnigan MAT 8430 spectrometer operating at an ionization potential of 70 eV. Elemental analyses for C, H, N were performed using a Heraeus CHN-O-Rapid analyzer, and the results agreed favorably with calculated values. In order to purify the synthesized compounds column chromatography was performed on silica gel 60 (Merck).

1,3-Dihydro-3-(oxidophenylimino)-2H-indol-2-one (**3**)
Phenylhydroxylamine (18.30 mmol) was dissolved in 50 cm^3 chloroform, then isatin imine **2** (18 mmol) was added to the well-stirred reaction mixture. After a few minutes at 50 °C the reaction mixture was left to warm. When reaction was complete (indicated by TLC), the orange crystals of isatin nitrone were isolated by filtration and washed with cold ethanol. Yield: 4.22 g (98.5%); m.p.: 218–220 °C; Ref. [17]: m.p.: 216 °C.

*General procedure for synthesis of **6** and **7** with classical heating*

A stirred mixture of isatin ketonitrone (**3**, 1 mmol) and different dipolarophiles (**4** or **5**, 1.5 mmol) was heated under reflux for 1 h in 5 cm^3 acetonitrile. After completion of the reaction, as indicated by TLC, the mixture was cooled, the solvent was removed under high vacuum, and the crude product was purified by column chromatography using hexane–ethyl acetate (9:1) and was recrystallized from ethanol to give colorless crystals of **6** or light yellow crystals of **7**.

*General procedure for synthesis of **6** and **7** in ionic liquid as medium*

A mixture of isatin nitrone (*Z*)-**3** (1 mmol), different dipolarophiles (**4** or **5**) (1.5 mmol), and 1-butyl-3-methylimidazolium bromide (4.5 mmol) was stirred for 15 min at 70 °C. After completion of the reaction (as monitored by TLC), the mixture was washed with water, cooled to room temperature, and the crude product was purified by column chromatography using hexane–ethyl acetate (9:1) and was recrystallized from ethanol to give **6** as colorless crystals or **7** as light yellow crystals.

Water was evaporated and the recycled IL was washed with diethyl ether and reused for the second run. The isolated yield was measured for the second run and compared with that for the first run (Table 2, entry 4). No decrease in reaction yield was observed. Repeating the experiment showed that even after the fourth run the activity of the IL did not decrease significantly.

2′,5′-Diphenylspiro[3H-indol-3,3′(3′aH)-[2H]pyrrolo-[3,4-d]isoxazole]-2,4′,6′(1H,5′H,6′aH)-trione
(**6a**, $C_{24}H_{17}N_3O_4$)
White crystals; m.p.: 187–189 °C; IR (KBr): $\bar{\nu}$ = 1,731 (CO), 3,178 (NH) cm^{-1}; ^{1}H NMR (300 MHz, acetone-d_6): δ = 4.29 (d, 1H, J = 7.7 Hz), 5.61 (d, 1H, J = 7.7 Hz), 6.83 (d, 1H, J = 7.7 Hz), 6.93–6.96 (m, 2H), 7.01–7.08 (m, 2H), 7.11–7.16 (m, 2H), 7.27–7.30 (m, 1H), 7.33–7.38 (m, 1H), 7.43–7.50 (m, 3H), 7.53–7.58 (m, 2H), 9.55 (s, 1H) ppm; ^{13}C NMR (75 MHz, acetone-d_6): δ = 56.4, 74.7, 76.7 (spiro carbon), 110.3, 119.7, 122.1, 122.8, 125.5, 126.7, 126.9, 128.2, 128.7, 129.1, 130.7, 132.3, 142.9, 144.7 (Ar), 171.3 (CO), 173.2 (CO), 173.3 (CO) ppm; MS: *m*/*z* (%) = 411 (M^+, 23), 238 (M-$C_{10}H_7NO_2$, 6), 173 (M-$C_{14}H_{10}N_2O_2$, 100).

5′-(4-Chlorophenyl)-2′-phenylspiro[3H-indol-3,3′(3′aH)-[2H]pyrrolo[3,4-d]isoxazole]-2,4′,6′(1H,5′H,6′aH)-trione
(**6b**, $C_{24}H_{16}ClN_3O_4$)
White crystals; m.p.: 218–219 °C; IR (KBr): $\bar{\nu}$ = 1,721 (CO), 3,168 (NH) cm^{-1}; ^{1}H NMR (300 MHz, acetone-d_6): δ = 4.28 (d, 1H, J = 7.7 Hz), 5.60 (d, 1H, J = 7.7 Hz), 6.82 (d, 1H, J = 7.7 Hz), 6.92 (d, 2H, J = 7.9 Hz), 7.01–7.15 (m, 4H), 7.28–7.34 (m, 2H), 7.48 (d, 2H, J = 8.5 Hz), 7.59 (d, 2H, J = 7.8 Hz), 9.54 (s, 1H) ppm; ^{13}C NMR (75 MHz, acetone-d_6): δ = 56.3, 74.7, 76.7 (spiro carbon), 110.3, 119.8, 122.1, 122.8, 125.5, 126.8, 128.2, 128.4, 129.2, 130.8, 130.9, 133.8, 142.9, 144.7 (Ar), 171.1 (CO), 173.1 (CO), 173.2 (CO) ppm; MS: *m*/*z* (%) = 445 (M^+, 32), 238 (M-$C_{10}H_6NO_2Cl$, 19), 207 (M-$C_{14}H_{10}N_2O_2$, 88), 91 (M-$C_{18}H_{11}N_2O_4Cl$, 100).

5′-(4-Bromophenyl)-2′-phenylspiro[3H-indol-3,3′(3′aH)-[2H]pyrrolo[3,4-d]isoxazole]-2,4′,6′(1H,5′H,6′aH)-trione
(**6c**, $C_{24}H_{16}BrN_3O_4$)
White crystals; m.p.: 206–208 °C; IR (KBr): $\bar{\nu}$ = 1,722 (CO), 3,162 (NH) cm^{-1}; ^{1}H NMR (300 MHz, acetone-d_6): δ = 4.28 (d, 1H, J = 7.6 Hz), 5.61 (d, 1H, J = 7.6 Hz), 6.83 (d, 1H, J = 7.6 Hz), 6.92 (d, 2H, J = 7.6 Hz), 7.04–7.15 (m, 4H), 7.28–7.34 (m, 2H), 7.42 (d, 2H, J = 8.2 Hz), 7.74 (d, 2H, J = 8.2 Hz), 9.55 (s, 1H) ppm; ^{13}C NMR (75 MHz, acetone-d_6): δ = 56.3, 74.7, 76.7 (spiro carbon), 110.3, 119.8, 121.9, 122.1, 122.8, 125.5, 126.8, 128.1, 128.6, 130.8, 131.4, 132.2, 142.9, 144.7 (Ar), 171.1 (CO), 173.0 (CO), 173.2 (CO) ppm; MS: *m*/*z* (%) = 251 (M-$C_{14}H_{10}N_2O_2$, 56), 238 (M-$C_{10}H_6NO_2Br$, 19), 91 (M-$C_{18}H_{11}N_2O_4Br$, 100).

2′-Phenylspiro[3H-indol-3,3′(3′aH)-[2H]pyrrolo[3,4-d]-isoxazole]-2,4′,6′(1H,5′H,6′aH)-trione (**6d**, $C_{18}H_{13}N_3O_4$)
White crystals; m.p.: 243–244 °C; IR (KBr): $\bar{\nu}$ = 1,703 (CO), 1,741 (CO), 1,793 (CO), 3,217 (NH), 3,396

(NH) cm^{-1}; ^1H NMR (300 MHz, acetone-d_6): $\delta = 4.03$ (d, 1H, $J = 7.4$ Hz), 5.53 (d, 1H, $J = 7.4$ Hz), 6.79–6.87 (m, 1H), 6.91–6.96 (m, 1H), 6.98–7.04 (m, 3H), 7.06–7.14 (m, 2H), 7.16–7.22 (m, 1H), 7.47 (d, 1H, $J = 7.4$ Hz), 9.74 (s, 1H), 10.62 (s, 1H) ppm; ^{13}C NMR (75 MHz, acetone-d_6): $\delta = 60.4$, 75.3, 77.5 (spiro carbon), 110.1, 118.5, 119.9, 122.4, 124.4, 125.2, 127.6, 128.0, 128.2, 129.9, 141.3, 145.2 (Ar), 171.8 (CO), 172.4 (CO), 174.5 (CO) ppm; MS: m/z (%) = 335 (M^+, 39), 238 (M-$C_4H_3NO_2$, 31), 97 (M-$C_{14}H_{10}N_2O_2$, 26), 91 (M-$C_{12}H_8N_2O_4$, 100).

5′-Ethyl-2′-phenylspiro[3H-indol-3,3′(3′aH)-[2H]-pyrrolo[3,4-d]isoxazole]-2,4′,6′(1H,5′H,6′aH)-trione (**6e**, $C_{20}H_{17}N_3O_4$)

White crystals; m.p.: 157–159 °C; IR (KBr): $\overline{v} = 1{,}708$ (CO), 1,792 (CO), 3,196 (NH) cm^{-1}; ^1H NMR (300 MHz, acetone-d_6): $\delta = 1.22$ (t, 3H, $J = 7.2$ Hz), 3.63 (q, 2H, $J = 7.2$ Hz), 4.09 (d, 1H, $J = 7.5$ Hz), 5.42 (d, 1H, $J = 7.5$ Hz), 6.80 (d, 1H, $J = 7.8$ Hz), 6.85 (d, 2H, $J = 7.7$ Hz), 6.98–7.12 (m, 4H), 7.18 (d, 1H, $J = 7.4$ Hz), 7.29 (t, 1H, $J = 7.7$ Hz), 9.48 (s, 1H) ppm; ^{13}C NMR (75 MHz, acetone-d_6): $\delta = 12.3$, 33.7, 56.0, 74.3, 76.5 (spiro carbon), 110.1, 119.7, 121.8, 122.8, 125.3, 126.8, 128.1, 130.6, 142.9, 144.7 (Ar), 172.0 (CO), 173.3 (CO), 173.7 (CO) ppm; MS: m/z (%) = 363 (M^+, 100), 263 (M-CO-N-C_2H_5-CO, 30), 238 (M-$C_6H_7NO_2$, 30), 91 (M-$C_{14}H_{12}N_2O_4$, 80).

1,2-Dihydro-2-oxo-2′-phenylspiro[3H-indole-3,3′-isoxazolidin]-5′-carbonitrile (**7a**, $C_{17}H_{13}N_3O_2$)

Light yellow crystals; m.p.: 187–189 °C; IR (KBr): $\overline{v} = 1{,}710$ (CO), 2,350 (C–N) cm^{-1}; ^1H NMR (300 MHz, acetone-d_6): $\delta = 3.05$ (dd, 1H, $J = 2.8$, 13.2 Hz), 3.35 (dd, 1H, $J = 8.6$, 13.2 Hz), 5.63 (dd, 1H, $J = 2.8$, 8.6 Hz), 6.74–7.69 (m, 9H) ppm; ^{13}C NMR (75 MHz, acetone-d_6): $\delta = 47.2$ (CH_2), 68.7 (spiro carbon), 72.6 (CH), 110.5 (CN), 117.7, 120.2, 122.9, 124.8, 125.8, 128.4, 129.4, 130.5, 142.2, 145.9 (Ar), 174.8 (CO) ppm; MS: m/z (%) = 291 (M^+, 100), 184 (M-$C_{11}H_8N_2O$, 41).

2′,5′-Diphenylspiro[3H-indole-3,3′-isoxazolidin]-2(1H)-one (**7b**, $C_{22}H_{18}N_2O_2$)

Light yellow crystals; m.p.: 187–189 °C; IR (KBr): $\overline{v} = 1{,}710$ (CO) cm^{-1}; ^1H NMR (300 MHz, acetone-d_6): $\delta = 2.69$ (dd, 1H, $J = 8.7$, 12.5 Hz), 3.22 (dd, 1H, $J = 6.5$, 12.5 Hz), 5.63 (dd, 1H, $J = 6.5$, 8.7 Hz), 6.65–7.64 (m, 14H), 10.68 (s, 1H) ppm; ^{13}C NMR (75 MHz, acetone-d_6): $\delta = 50.1$ (CH_2), 69.5 (spiro carbon), 78.5 (CH), 111.2, 113.7, 116.9, 120.3, 121.6, 121.8, 124.2, 125.2, 126.5, 128.2, 129.6, 132.4, 142.3, 146.3 (Ar), 172.1 (CO) ppm; MS: m/z (%) = 342 (M^+, 86.8), 235 (M-$C_{16}H_{13}NO$, 100).

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