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A Highly Regio- and Diastereoselective Four-Component Reaction to Construct Polycyclic Bispiroindolines from 2-Isocyanoethylindoles and Isocyanates

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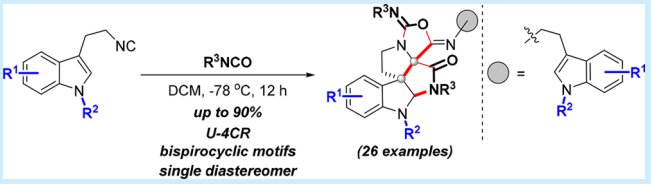
Supporting Information

ABSTRACT: A one-pot multicomponent domino reaction between 2-isocyanoethylindoles and isocyanates for the diastereoselective construction of polycyclic bispiroindolines was developed. Fused polycyclic bispiroindolines containing two contiguous spiral atoms were afforded in moderate to good yields with excellent regio- and diastereoselectivities through a four-component Ugi-type reaction (U-4CR) under mild conditions.



Polycyclic spiroindolines are privileged complex molecular skeletons that often exist in both natural products and pharmaceutical molecules with pronounced biological activities.[1,2] For example, strychnine,[1a,2b,c] communesin A-H,[2a,d,e,g,i,l−n,n] and (+)-perophoramidine[2h,j,m,o] are characteristic members of the alkaloid family that continue to capture the imagination of organic chemists (Figure 1). Strychnine is a

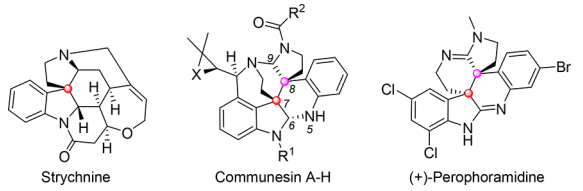


Figure 1. Structures of polycyclic spiroindoline alkaloids.

notorious poison, causing postsynaptic inhibition in the spinal cord where it antagonizes the transmitter glycine.[3] Communesins demonstrate significant cytotoxicity and insecticidal activity.[4] (+)-Perophoramidine possesses modest cytotoxicity against the HCT 116 human colon carcinoma cell line (IC_{50} = 60 μM). Notable features of communesin or (+)-perophoramidine are the two contiguous spiral atoms at C7/8 and the presence of the bisaminal or bisamidine moieties.

Multicomponent reactions involving cascade processes provide powerful strategies for the total synthesis of natural products and synthetically useful building blocks.[5,6] Among them, isocyanide-based multicomponent reactions are particularly important.[5b,c] Recently, 2-isocyanoethylindoles, bearing a nucleophilic isocyanide at the C3-position of the indoles, have been used as precursors for the rapid construction of spiroindolines.[7] Because the chemoselective synthesis of spiroindolines and pyrroles have been achieved by using a 2-isocyanoethylindole-based domino reaction,[7b,c] a number of chemo- and enantioselective synthetic methods have been established for the dearomatization of 2-isocyanoethylindoles to afford spiroindolines. In this aspect, Feng and co-workers have made significant contributions to the catalytic asymmetric transformation of 2-isocyanoethylindoles into polycyclic spiroindolines in the presence of *N,N′*-dioxide ligands, thus affording a series of enantiomerically enriched polycyclic spiroindolines in good yields and high ee values.[7d,e] However, the chemoselectivity on the construction of spiroindolines from 2-isocyanoethylindoles still faces some challenges. For example, a Bischler−Napieralski reaction may take place as a side reaction, and 2-isocyanoethylindoles can only perform as normal isocyanides to react with the other reagents,[8] making the polycyclic spiroindolines ultimately difficult to achieve. Moreover, the substrate scopes in the cascade cyclization of 2-isocyanoethylindoles to construct polycyclic spiroindolines are still limited. As shown in Scheme 1, two-component domino cyclization of 2-isocyanoethylindoles and electron-deficient olefins to give spiroindolines has been disclosed by Ji and Feng's groups (Scheme 1a);[7c−e] Ji's group and Ruijter's group also independently reported a three-component cyclization of 2-isocyanoethylindoles with aldehydes and malononitriles as well as 2-isocyanoethylindoles with primary amines and aldehydes (Scheme 1b).[7a,b] However, in all of these cases, only one spiral atom was incorporated in the present products. Herein, we report a novel one-pot, catalyst-free, four-component Ugi-type reaction (U-4CR) to construct polycyclic bispiroindolines bearing two spiral atoms and having bispirocyclic motifs from simple benzsulfonyl isocyanates and readily available 2-isocyanoethylindoles under mild conditions (Scheme 1c, this work).

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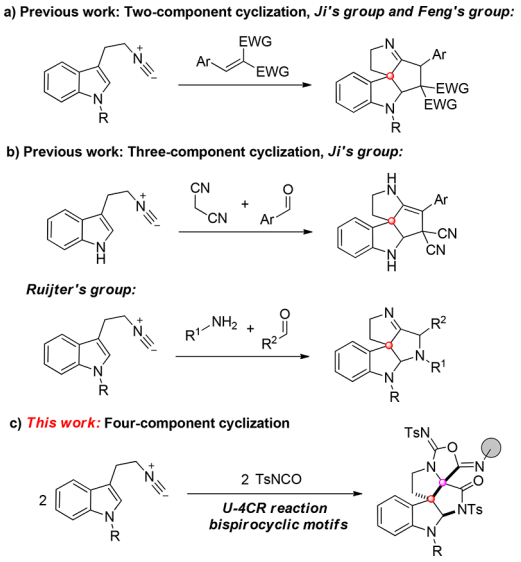
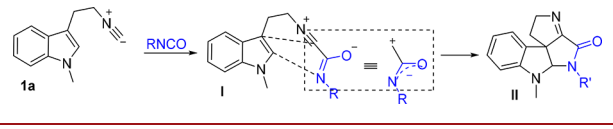
Scheme 1. Strategies of Constructing Polycyclic Spiroindolines from 2-Isocyanoethylindoles



During our ongoing investigation on the multicomponent reactions of 2-isocyanoethylindoles under catalyst-free conditions, we assumed that 2-isocyanoethylindole **1a** would react with isocyanate, producing 1,3-dipolar intermediate **I** and then undergo an intramolecular [3 + 2] dearomative cyclization to give polycyclic spiroindoline **II** (Scheme 2). Unlike previous

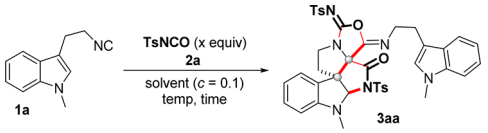
Scheme 2. Our Working Hypothesis of the Dearomative Cyclization of 2-Isocyanoethylindoles with Isocyanates



works, because of the effect of an adjacent carbonyl group, spiroindoline **II** as a much more reactive imide that could undergo an Ugi-type reaction with another **1a** and isocyanate **2a** to give a polycyclic bispirocyclic framework under the same conditions. Indeed, we found that, in the reaction of **1a** with **2a**, the polycyclic bispirocyclic product **3aa** derived from a four-component reaction was obtained in 61% yield in dichloromethane (DCM) from −65 °C to room temperature within 12 h (Table 1, entry 1). Then, the optimization of the reaction conditions was performed, and the results are shown in Table 1. Examination of the employed amount of **2a** revealed that the use of 2.0 equiv of **2a** afforded **3aa** in 76% yield (entries 1−5). Subsequently, other solvents such as tetrahydrofuran, methyl *tert*-butyl ether, ethyl acetate, and toluene were also tested, but none of them could give **3aa** in better yields (entries 6−9). Furthermore, changing the reaction time and carrying out the reaction at −78 °C did not significantly improve the yield of **3aa** (entries 10−14). The influence of the reaction mixture's concentration on the reaction outcome has also been examined, and we pleasingly found that using **1a** in DCM (c = 0.2) gave **3aa** in 80% isolated yield (entries 15−17).

With the optimized reaction conditions in hand (Table 1, entry 16), we next surveyed the substrate scope of this reaction, and the results are summarized in Scheme 3. Various substituted *N*-methyl-2-isocyanoethylindoles **1**, regardless of whether they have electron-poor or -rich substituents on the aromatic rings,

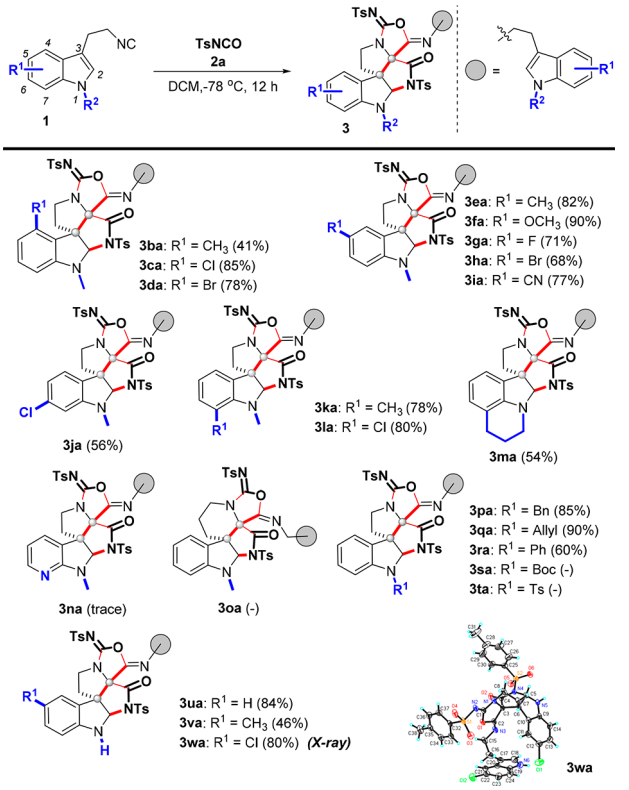
Table 1. Optimization of the Reaction Conditions[a]



entry	x equiv	solvent	temp (°C)	time (h)	yield (%)
1	1.0	DCM	−65∼rt	12	61
2	1.2	DCM	−65∼rt	12	64
3	1.5	DCM	−65∼rt	12	67
4	2.0	DCM	−65∼rt	12	76
5	2.5	DCM	−65∼rt	12	76
6	2.0	THF	−65∼rt	12	n.p.
7	2.0	MTBE	−65∼rt	12	38
8	2.0	EA	−65∼rt	12	23
9	2.0	toluene	−65∼rt	12	58
10	2.0	DCM	−78	1	71
11	2.0	DCM	−78	2	72
12	2.0	DCM	−78	3	71
13	2.0	DCM	−78	5	74
14	2.0	DCM	−78	12	76
15	2.0	DCM (c = 0.05)	−78	12	70
16	***2.0***	***DCM (c = 0.2)***	***−78***	***12***	***82 (80)***[b]
17	2.0	DCM (c = 0.4)	−78	12	76

[a]Reactions were carried out using **1a** (0.4 mmol) and **2a** (x equiv). Yields were determined by ^{1}H NMR spectroscopic data of the crude reaction mixture using benzyl methyl ether as an internal standard. [b]Isolated yield in parentheses.

Scheme 3. Substrate Scope for 2-Isocyanoethylindoles with TsNCO 2a[a]



[a]Reaction conditions: **1** (0.4 mmol), **2a** (0.8 mmol), DCM (2.0 mL), −78 °C, 12 h, under Ar; yields of isolated products are given.

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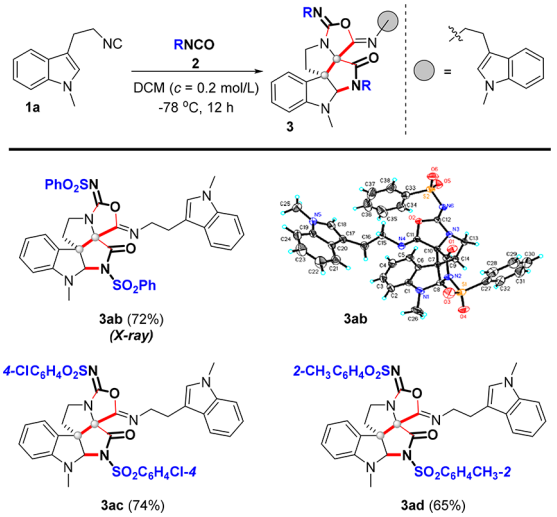
were all compatible, affording the corresponding products **3ba**–**3la** in moderate to good yields ranging from 41 to 90%. It is worth mentioning that using **1b**, **1c**, and **1d** as substrates, in which the substituent was introduced at the 4-position of indole, afforded desired products **3ba**, **3ca**, and **3da** in 41–85% yields, suggesting that the steric effect may have some impact on the reaction proceeding because the sterically more bulky substrate **1b** having a methyl group at the 4-position gave corresponding product **3ba** in lower yield. For substrate **1m**, in which N1 and C7 were connected with an aliphatic chain, the reaction proceeded smoothly, giving desired product **3ma** in 54% yield. The use of the 7-aza-indole derivative as substrate afforded a trace of **3na**. Extending the aliphatic carbon chain of isonitrile from two to three carbon atoms did not give the corresponding product **3oa**. For *N*-benzyl, allyl, and phenyl group-substituted indoles, the desired products **3pa**, **3qa**, and **3ra** were obtained in 60–90% yields, respectively. However, for *N*-Boc- and *N*-Ts-substituted substrates, desired products **3sa** and **3ta** were not formed under identical conditions, perhaps due to the electronic effect. Delightfully, *N*-H indole derivatives were also tolerated, furnishing the products **3ua**, **3va**, and **3wa** in 46–84% yields. The structure of **3wa** has been unambiguously confirmed by X-ray diffraction, and its CIF data are presented in the Supporting Information (SI). The ORTEP drawing is shown in Scheme 3.

Next, we investigated the scope with respect to a series of isocyanate derivatives **2** (Scheme 4). All these reactions

Scheme 4. Substrate Scope for *N*-Methyl-2-Isocyanoethylindole 1a with Isocyanate Derivatives[a]

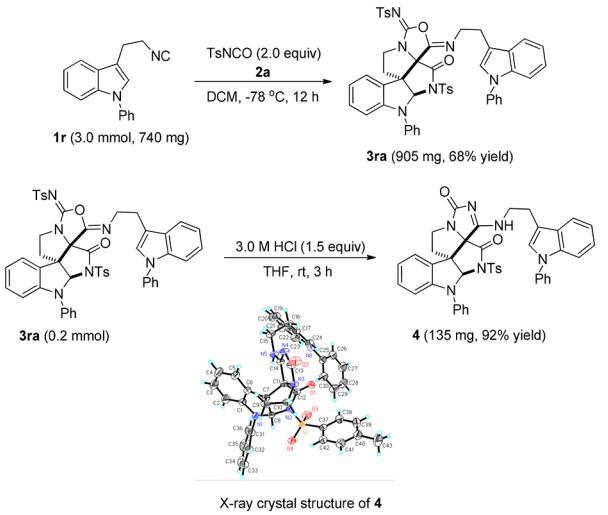


[a]Reaction condition: **1a** (0.4 mmol), **2** (0.8 mmol), DCM (2.0 mL), −78 °C, 12 h, under Ar; yields of isolated products are given.

proceeded smoothly, giving the corresponding products **3ab**–**3ad** in 65–74% yields. The structure of **3ab** was also unambiguously determined by X-ray diffraction, and its CIF data are presented in the SI. The ORTEP drawing is shown in Scheme 4. It is worth noting that benzoyl-, phenyl-, benzyl-, and *tert*-butylisocyanates did not react with **1a** under the standard reaction conditions.

A scale-up synthesis using 3.0 mmol of **1r** and further hydrolysis of **3ra** were performed as shown in Scheme 5. The use of 3.0 mmol of **1r** afforded **3ra** in 68% yield (905 mg) under the standard conditions. In addition, the hydrolysis of **3ra** with 1.5 equiv of 3.0 M aqueous HCl solution produced the

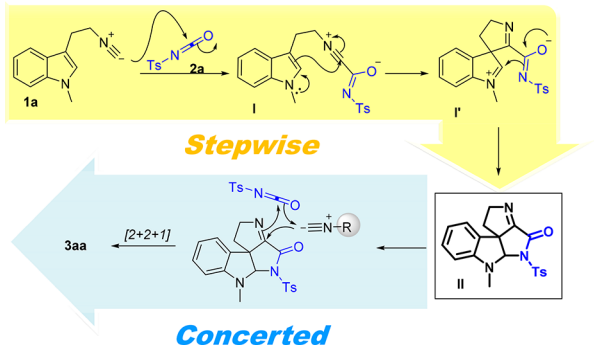
Scheme 5. Scale-up Synthesis and Hydrolysis Experiment



corresponding product **4** in 92% yield. The structure of **4** was unambiguously determined by X-ray diffraction and its CIF data are presented in the SI.

On the basis of previous reports and the obtained results described above, a plausible mechanism for this four-component cascade cyclization reaction of 2-isocyanoethylindoles and isocyanates is depicted in Scheme 6. First, *p*-toluenesulfonyl

Scheme 6. Proposed Reaction Mechanism



isocyanate **2a** accepts the nucleophilic attack of *N*-methyl-2-isocyanoethylindole **1a** to produce 1,3-dipolar intermediate **I**, which subsequently undergoes an intramolecular [3 + 2] dearomative cyclization to give polycyclic spiroindoline **II** via intermediate **I′**. Then, spiroindoline **II** reacts with another **1a** and **2a** again through a synergistic [2 + 2 + 1] ring-closure process to give **3aa**. The regioselective insertion of C–N and C–O bonds of TsNCO in the formation of **3aa** may be due to the existence of a stepwise process and a concerted pathway during the reaction proceeding. The concerted process took place faster than the stepwise process once spiroindoline **II** was produced, which may be a reasonable explanation for why polycyclic spiroindoline **II** can not be separated even by controlling the reaction parameters or switching the reaction sequences.

In summary, we have developed a novel catalyst-free one-pot U-4CR between 2-isocyanoethylindoles and isocyanates for the construction of polycyclic bispiroindolines in moderate to good yields under mild conditions. A variety of polycyclic bispiroindolines containing two contiguous spiral atoms could be prepared with a good substrate scope and excellent regio- and

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diastereoselectivity. Further studies for extending the application of this method and biological evaluation of these complex polycyclic bispiroindoline compounds are in progress.

■ ASSOCIATED CONTENT

Supporting Information

The Supporting Information is available free of charge on the ACS Publications website at DOI: 10.1021/acs.orglett.8b03019.

Experimental procedure and characterization data for all compounds (PDF)

Accession Codes

CCDC 1525040, 1583908, and 1874462 contain the supplementary crystallographic data for this paper. These data can be obtained free of charge via www.ccdc.cam.ac.uk/data_request/cif, or by emailing data_request@ccdc.cam.ac.uk, or by contacting The Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB2 1EZ, UK; fax: +44 1223 336033.

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Notes

The authors declare no competing financial interest.

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