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Base-Controlled Divergent Synthesis of 5-Cyanobenzoxepines and Benzofuro[2,3-*b*]pyridines from 2-Bromophenylacetonitriles and Ynones

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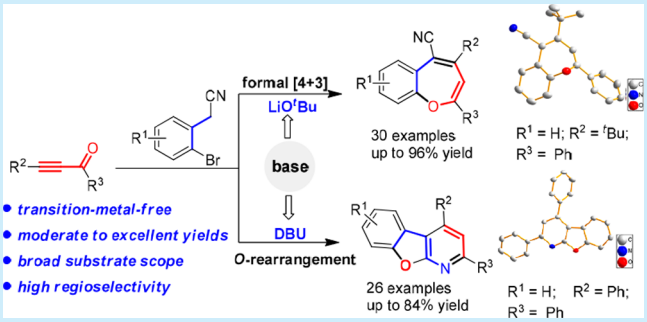
Supporting Information

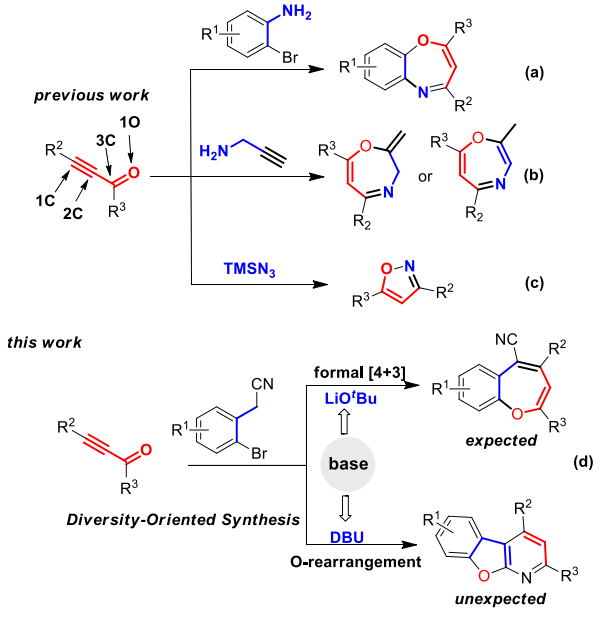
ABSTRACT: An effective base-controlled divergent annulation reaction of 2-bromophenylacetonitriles and ynones has been developed. Various functionalized 5-cyanobenzoxepines and benzofuro[2,3-*b*]pyridines were obtained with a broad substrate scope and high regioselectivity in moderate to excellent yield. Of importance, an unexpected O-rearrangement reaction to access benzofuro[2,3-*b*]pyridines was observed using 1,8-diazabicyclo[5.4.0]undec-7-ene (DBU) as the base, and the possible mechanism was supported by ^{18}O-labeled experiments. In addition, the gram-scale synthesis and further transformation of the product were studied.



Benzoxepines and benzofurans, as important seven- and five-membered oxygen-containing heterocyclic compounds, are vital structural units due to their broad application in biomedicinal natural molecules and pharmaceuticals.[1,2] Consequently, various methods for preparing benzoxepines or benzofurans from easily available materials by intermolecular or intramolecular cyclization have been developed, respectively.[3,4] Although the above methodologies are impressive, the development of an efficient and transition-metal-free approach for the diversity-oriented synthesis of both benzoxepines and benzofurans is still a challenging but attractive task.

On the contrary, ynones, as electron-deficient and polarized alkynes, have been widely investigated because of their convenient preparation by the Sonogashira coupling of terminal alkynes with acyl chlorides[5] and easy functionalized transformation to valuable compounds.[6] It is noteworthy that few cyclization reactions including the C−C triple bond and carbonyl groups (1C, 2C, 3C, 1O) as the four-atom synthon have been explored.[7] For example, the synthesis of 1,4-oxazepines from ynones and 2-bromoaniline or propargylamine was described (Scheme 1a,b).[7a−d] A direct approach to isoxazoles from ynones and trimethylsilyl azide under metal-/catalyst-free conditions was also reported (Scheme 1c).[7e] Inspired by the above excellent studies and our continuing interest in the catalytic transitions of alkyne,[8] we initially envisioned that ynones as a four-atom synthon may react with 2-bromophenylacetonitriles via a formal [4 + 3] cyclization reaction to construct benzoxepines in the presence of a suitable base. To our surprise, the expected benzoxepines and unexpected benzofuro[2,3-*b*]pyridines were both obtained using different bases. Herein we report for the first time a novel and efficient base-controlled divergent synthesis of 5-

Scheme 1. Previous Work and This Work for the Cyclization Reaction of Ynones Used as the Four-Atom (1C, 2C, 3C, 1O) Synthon



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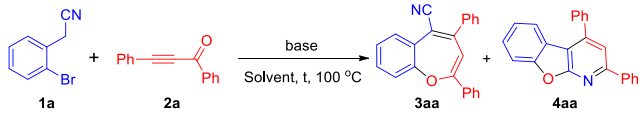
cyanobenzoxepines and benzofuro[2,3-*b*]pyridines from ynones and 2-bromophenylacetonitriles with high regioselectivity and a broad substrate scope under transition-metal-free conditions (Scheme 1d).

Initially, we started to screen reaction conditions by the model reaction of 2-bromophenylacetonitrile **1a** and ynone **2a** (Table 1). To our delight, product **3aa** was isolated in 58%

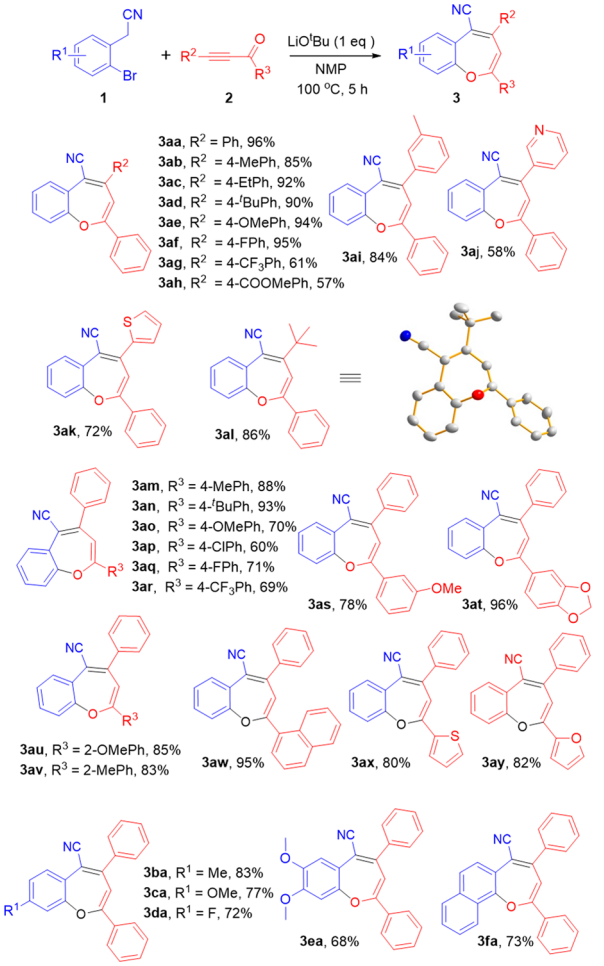
Table 1. Optimization of the Reaction Conditions[a]



entry	base	**1a**/**2a**/base	solvent	*t* (h)	yield (%) **3aa**/**4aa**[b]
1	K_2CO_3	1/1/1.5	DMF	5	58/trace
2	Cs_2CO_3	1/1/1.5	DMF	5	68/trace
3	KO^{t}Bu	1/1/1.5	DMF	5	70/10
4	LiO^{t}Bu	1/1/1.5	DMF	5	89/trace
5	KOH	1/1/1.5	DMF	5	50/15
6	DBU	1/1/1.5	DMF	5	trace/47
7	DABCO	1/1/1.5	DMF	5	trace/–
8	LiO^{t}Bu	1/1/0.5	DMF	5	44/–
9	LiO^{t}Bu	1/1/1	DMF	5	94/–
10	LiO^{t}Bu	1/1/2	DMF	5	91/–
11	**LiO^{t}Bu**	**1/1/1**	**NMP**	**5**	**96**/–
12	LiO^{t}Bu	1/1/1	DMSO	5	82/trace
13	LiO^{t}Bu	1/1/1	CH_3CN	5	trace/–
14	DBU	1/1/1.5	DMSO	5	trace/56
15	DBU	1/1/1.5	NMP	5	–/45
16	DBU	1/1/1.5	CH_3CN	5	10/41
17	DBU	1/1.5/1.5	DMSO	12	trace/68
18	DBU	1/2/1.5	DMSO	12	21/65
19	**DBU**	**1/1.5/2**	**DMSO**	**12**	**trace/76**
20[c]	DBU	1/1.5/2	DMSO	12	trace/65
21[d]	DBU	1/1.5/2	DMSO	12	trace/71

[a]Reaction conditions: **1a** (0.30 mmol, 1.0 equiv), **2a**, base, and 1.5 mL of solvent. [b]Isolated yields. [c]110 °C. [d]90 °C.

Scheme 2. Scope of the Cyclization Reactions Using LiO^{t}Bu as Base[a,b]



[a]Reaction conditions: 0.3 mmol **1**, 0.3 mmol **2**, 0.3 mmol LiO^{t}Bu, 1.5 mL of NMP, 100 °C, 5 h. [b]Isolated yield.

yield using K_2CO_3 as an inorganic base (Table 1, entry 1). On the basis of the above experimental result, various inorganic and organic bases were then investigated. The higher yield of product **3aa** was obtained in the presence of LiO^{t}Bu (Table 1, entry 4). When the base was changed to DBU, an unexpected major product **4aa** was given in 47% yield (Table 1, entry 6). Subsequently, the effects of different reaction parameters, such as solvent, reaction time, reaction temperature, material ratio of reactants, and usage of base, were further explored. The experimental results show that the base LiO^{t}Bu is suitable for product **3aa** in 96% yield (Table 1, entry 11), and the base DBU exhibits superior activity to prepare product **4aa** in 76% yield (Table 1, entry 19).

With the optimal reaction conditions in hand, we set out to explore the formal [4 + 3] cyclization reaction to construct benzoxepines in the presence of LiO^{t}Bu. A variety of ynones **2** with 2-bromophenylacetonitriles **1** were studied, and the results are listed in Scheme 2. The R^2 and R^3 substituents anchoring the ynones were first studied. Substrates **2b**–**i** and **2m**–**v** with common substituents containing alkyl, ether, halo, CF_3, or an ester group at the ortho, meta, or para positions of the aryl groups could smoothly deliver the corresponding products **3ab**–**ai** and **3am**–**av** in moderate to excellent yield. Further investigation showed that heteroaromatic substrates **2j**,**k** and **2x**,**y** and naphthyl substrate **2w** were also tolerated under the standard conditions. It is noteworthy that product **3al** was obtained in 86% yield, the structure of which was unambiguously characterized by X-ray crystallography. Then, we turned to investigate the scope of various 2-bromophenylacetonitriles with ynone **2a**. The electronic effect on the four-position of 2-bromophenylacetonitriles has little influence on the reactivity, and the 4-Me (**1b**), 4-OMe (**1c**), and 4-F (**1d**) substitutive substrates were also competent in this cyclization reaction. Additionally, substrate **1e** with an electron-donating substituent (R^1 = 4,5-OMe) or naphthyl substrate **1f** was smoothly transformed into the corresponding product in satisfactory yield.

Subsequently, the cyclization reactions of 2-bromophenylacetonitriles **1** and ynones **2** to prepare benzofuro[2,3-*b*]pyridines were investigated, and the results are shown in Scheme 3. To our delight, substrates **2b**–**i** and **2m**–**t** at meta or para positions of the aryl groups (R^2, R^3) with electron-donating groups or electron-withdrawing groups were well tolerated, and the corresponding products **4ab**–**ai** and **4am**–**at** were obtained in moderate to good yield. Unfortunately, substrates **2u**,**v** at the ortho position of the aryl groups (R^3) failed to give products **4au**,**av**, and benzoxepine products **3au**,**av** were given in 51 and 85% yield, respectively. Similar

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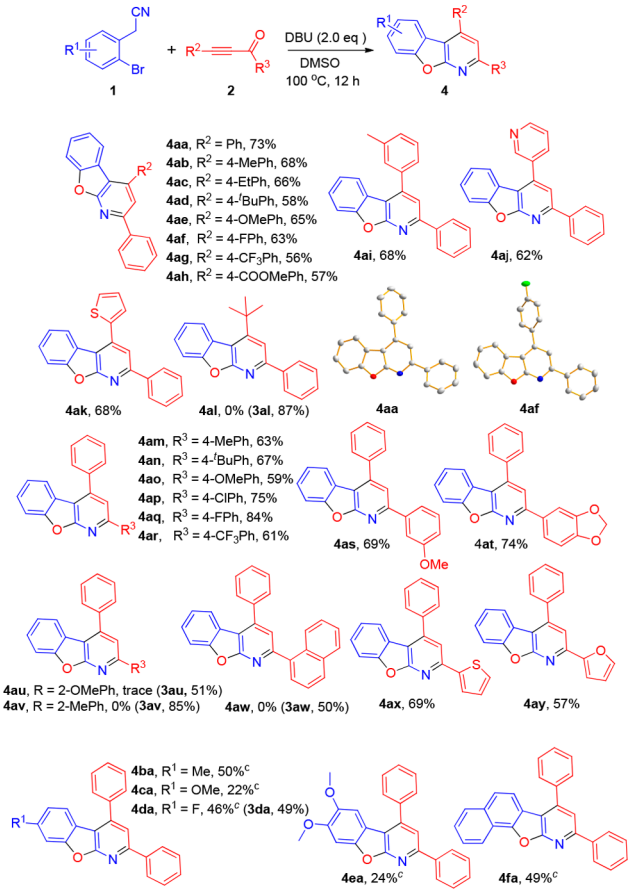
Scheme 3. Scope of the Reactions Using DBU as Base[a,b]

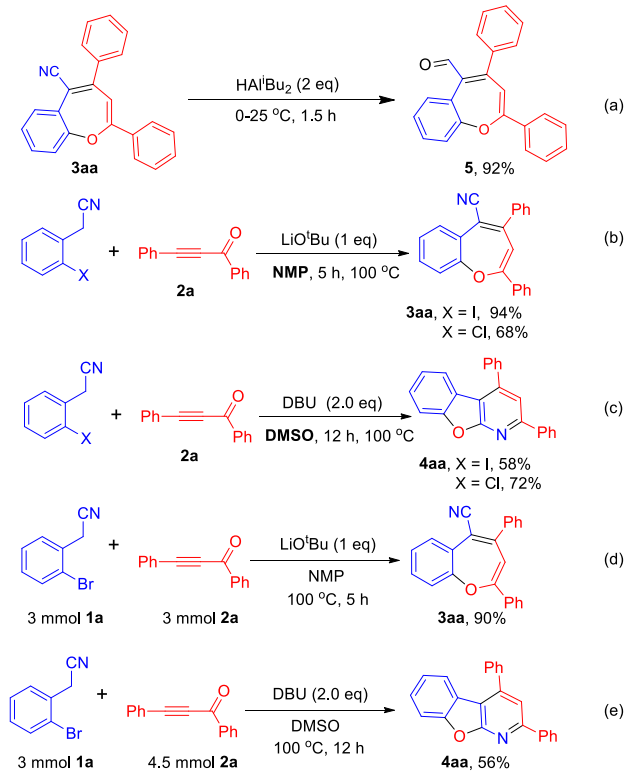


[a]Reaction conditions: 0.3 mmol **1**, 0.45 mmol **2**, 0.6 mmol DBU, 1.5 mL of DMSO, 100 °C, 12 h. [b]Isolated yield. [c]3 equiv DBU.

results were observed for the substrates bearing alkyl (**2l**) and naphthyl (**2w**) groups, which were all converted into benzoxepine products. Failure to access the desired products may be affected by the steric and electronic effect using the above ynones (**2l**, **2u**−**2w**) as substrates. In addition, the reaction of heteroaromatic substrates **2j**,**k** and **2x**,**y** with 2-bromophenylacetonitrile **1a** proceeded smoothly, leading to the desired products (**4aj**,**ak**, **4ax**,**ay**). The effect on the catalytic activity of 2-bromophenylacetonitriles **1** with ynone **2a** was further studied. Compared with the ynones **2**, the substituted 2-bromophenylacetonitriles **1b**−**f** showed low catalytic activity and underwent a cyclization reaction to deliver the target products **4ba**−**fa** in the presence of 3 equiv DBU. When 2-bromophenylacetonitriles with 4-Me (**1b**) or 4-F (**1d**) group was used as the substrate, the corresponding product **4ba** or **4da** was obtained in moderate yield. In particular, product **3da** was obtained in 49% yield, accompanied by product **4da**. However, 2-bromophenylacetonitriles with strong electron-donating group 4-OMe (**1c**) or 3,4-OMe (**1e**) gave product **4ca** or **4ea** in low yield. The naphthyl substrate **1f** was also suitable for this reaction in satisfactory yield. The structures of **4aa** and **4af** were determined unambiguously by single-crystal X-ray crystallography (see Figure S1). Additionally, the photophysical properties of **4aa** were analyzed using ultraviolet−visible and photoluminescence (PL) spectrometry (see Figure S2).

Products **3** with a cyano group have an opportunity to prepare new functionalized compounds, and product **5** was obtained by the reduction of **3aa** with excellent yield in the presence of $HAl(^iBu)_2$ (Scheme 4a). Moreover, when 2-

Scheme 4. Further Chemical Transformations of Product 3aa, Application Using Other 2-Halophenylacetonitriles as Substrates, and Gram-Scale Reaction for the Synthesis of Products 3aa and 4aa



iodophenylacetonitrile or 2-chlorophenylacetonitrile was employed to react with **2a** under standard conditions, the identical product **3aa** or **4aa** was obtained in moderate to excellent yield (Scheme 4b,c). To further demonstrate the practicality of our current method, gram-scale reactions were carried out. When 3.0 mmol of substrate **1a** was used under standard conditions, the desired products **3aa** and **4aa** could be obtained in 90 and 56% yield, respectively (Scheme 4d,e).

On the basis of the present results and previous literature,[3l,7a−d] a plausible mechanism for the base-controlled divergent preparation of 5-cyanobenzoxepines and benzofuro-[2,3-*b*]pyridines from 2-bromophenylacetonitriles **1** and ynones **2** is proposed in Scheme 5. First, 2-bromophenylacetonitriles **1** could add to the triple bond of ynones **2** by nucleophilic attack in the presence of a base, affording intermediate **A**. For path I using LiO^tBu as the base, deprotonation and allyl and keto−enol tautomerism of **A** successively occur to yield the new zwitterions **B**, the intramolecular nucleophilic aromatic substitution (S_NAr) of which would give product **3** (path I). Both intermediates **A** and **B** could be detected by MS and ^1H NMR (see Figures S3 and S4). For path II using DBU as base, the ethenimines **C** were obtained by the hydrogen transfer of **A**, the intramolecular cyclization of which between ethenimines with the carbonyl carbon occurs to release the bridged intermediates **D**. Subsequently, the base snatches the hydrogen of **D**; then, the

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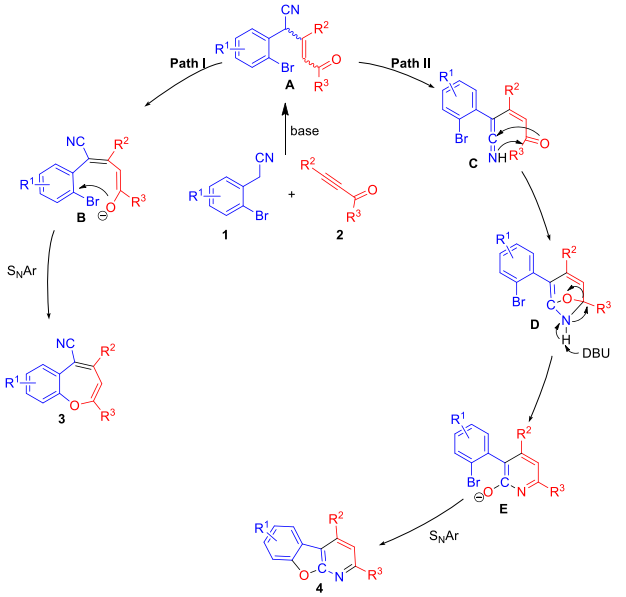
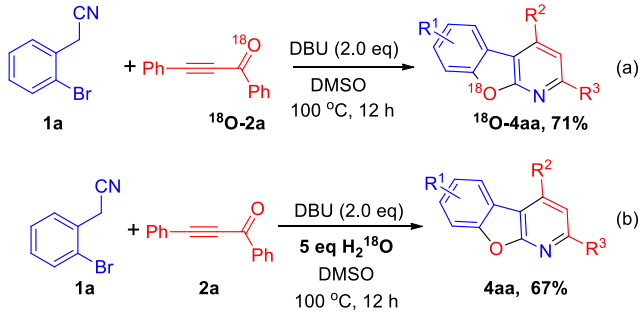
Scheme 5. Plausible Mechanism for the Synthesis of Products 3 and 4



O-rearrangement intermediates **E** were obtained by the C−O bond cleavage promoted by electron transfer. The target products **4** were formed by the intramolecular nucleophilic aromatic substitution (S_NAr). According to previous reports,[3l,7a] path I to access the expected products **3** was easily understood by the formal [4 + 3] cyclization reaction. To further understand the mechanisms of this novel cyclization for the preparation of products **4** and to clarify path II undergoing the O-rearrangement reaction, the ^{18}O-labeled substrate **2a** was synthesized (see Figure S5), and ^{18}O-labeled experiments were carried out (Scheme 6). When **1a** reacted with ^{18}O-labeled **2a** under the standard conditions, ^{18}O-labeled **4aa** was detected (see Figure S6). However, when **1a** reacted with **2a** in the presence of $H_2{}^{18}O$, ^{18}O-labeled **4aa** failed to be detected. The above results imply that the oxygen atom of products **4** comes from the substrates **2** by the O-rearrangement. Meanwhile, this also excludes the possible interference of trace water in this cyclization reaction.

Scheme 6. Controlled Experiments Using ^{18}O-2a or $H_2{}^{18}O$



In conclusion, we have developed the first example of the base-controlled divergent annulation of 2-bromophenylacetonitriles and ynones to prepare 5-cyanobenzoxepines and benzofuro[2,3-*b*]pyridines with moderate to excellent yields. The usage of readily available materials, a broad substrate scope, and high regioselectivity makes this current methodology very practical. A plausible mechanism to access the unexpected products **4** by O-rearrangement was proposed and supported by the ^{18}O-labeled experiments. Further research of the catalytic transition involving ynones as four-atom synthons is ongoing in our laboratory.

■ ASSOCIATED CONTENT

Supporting Information

The Supporting Information is available free of charge on the ACS Publications website at DOI: 10.1021/acs.orglett.9b01700.

Experimental procedures, characterization of products, copies of ^1H and ^{13}C NMR, X-ray crystal structures of **3al**, **4aa**, and **4af**, and photophysical properties of **4aa** and mechanistic studies (PDF)

Accession Codes

CCDC 1913757−1913759 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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