Chinese Chemical Letters 34 (2023) 107823

Contents lists available at ScienceDirect

Chinese Chemical Letters

journal homepage: www.elsevier.com/locate/cclet

[4 + 3] Cycloaddition of ketenimines with furocarbenoids: Divergent and efficient synthesis of fused cycloheptatriene and tropone scaffolds

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ARTICLE INFO

Article history:
Received 12 June 2022
Revised 8 September 2022
Accepted 13 September 2022
Available online 15 September 2022

Keywords:
Domino reactions
Isocyanides
Ene-yne-ketones
Cycloheptafuran scaffolds
[4+3] cycloaddition

ABSTRACT

An unprecedent [4 + 3] cycloaddition of furoketenimines with furocarbenoids has been disclosed for the divergent and efficient synthesis of cycloheptafuran and cycloheptapyrrole scaffolds. Zinc chloride acted as promoters for both the formation of these two transient intermediates from isocyanides and ene–yne-ketones, and the subsequent construction of seven-membered ring. Three rings and five bonds were constructed successively in this three-component one-pot domino reaction.

Cycloheptafuran and cycloheptapyrrole scaffolds are widely found in many natural products with significant biological activities (Fig. 1). For example, rubrolone B has cardioprotective activity [1,2], frondosin B is served as an interleukin-8 receptor antagonist [3], myrrhterpenoid H possesses neuroprotective effects, and exotines A and B show inhibition of nitric oxide production [4,5]. While a number of synthetic methods for the construction of these fused frameworks have been documented [6–13], the development of highly efficient synthetic approaches is still desirable.

Ketenimines, a type of unique heterocumulenes, have been explored as versatile synthons for the synthesis of carbocycles and heterocycles *via* cycloaddition reactions (Scheme 1) [14,15]. In most cases, ketenimines served as a 2π component to undergo [2 + 2] [16–19], [3 + 2] [20–23] and [4 + 2] [24,25] cycloaddition reactions (Scheme 1a). Occasionally, vinyl, phenyl or furyl substituted ketenimines can act as 4π component in [4 + 2] [26–29] and [4 + 1] [30] cycloadditions, respectively (Scheme 1b). Additionally, difluoroketenimines acting as 1,3-dipoles was disclosed by Wang and coworkers [31,32]. Despite these well-developed reaction activity the higher-order cycloaddition of ketenimines remain unexplored.

Isocyanide has been proven to be a useful precursor for the synthesis of ketenimines [14,15,33,34] *via* coupling reactions with carbenes [14,29,31,32,35] and allyl- or propargyl carbonates [36,37], as well as isocyanide-based multicomponent reactions [14,38,39]. Recently, the research groups of Li [30] and us [28] independently developed a convenient method to generate furyl-ketenimines *in situ* following with [4 + 1] and [4 + 2] cycloadditions by treatment of ene–yne-ketones with isocyanides. With this background and in continuation to our interest in isocyanide-based annulations [40–45], we report herein a [4 + 3] cycloaddition between the *in situ* generated furyl-ketenimines with zinc furyl-carbenoids, both of which are formed by a zinc promoted reaction of isocyanides with ene–yne-ketones (Scheme 1c). This transformation not only provides a novel protocol for the synthesis of fused cycloheptatriene and tropone scaffolds with highly efficiency, but also represents the first example of higher-order [4 + 3] cycloaddition of ketenimines with perfect atom economy.

We began our investigation by treating aryl isocyanide **1a** with ene–yne-ketone **2a** (for more details, see Supporting information). To our delight, under $ZnCl_2$ promoted conditions cycloheptafuran product **3aa** was formed in 46% yield, while the cycloheptapyrrole **4aa** was obtained in 24% yield (Table 1, entry 1). The structures of **3aa** and **4aa** were unambiguously assigned by X-ray crystallography (Fig. 2, CCDC Nos. 1974901 and 2018184). Fortunately, rising the reaction temperature to 90 °C resulted in improved 74% yield for **3aa** along with only 8% of **4aa** obtained (entry 2), while higher temperature showed adverse effect on **3aa** formation (entry 3). Lowering the $ZnCl_2$ loading from 1.0 equiv. to 0.8 equiv. dramati-

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https://doi.org/10.1016/j.cclet.2022.107823
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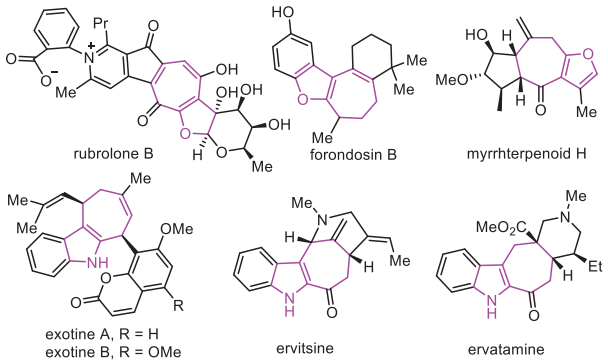
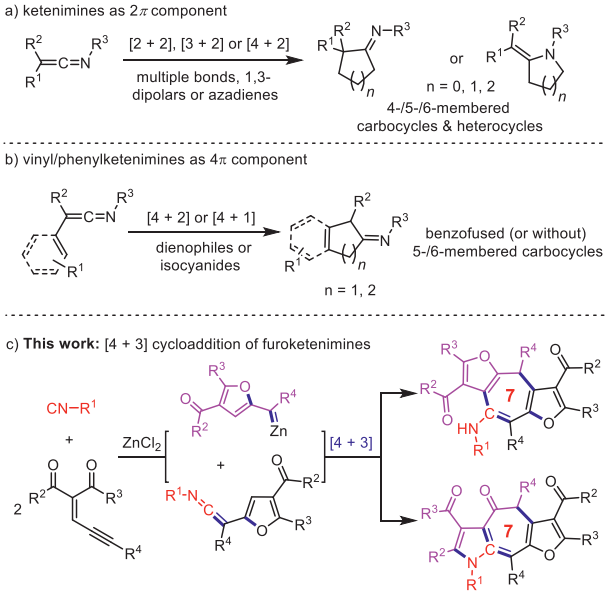


Fig. 1. Cycloheptafuran and cycloheptapyrrole scaffold alkaloids.



Scheme 1. Cycloadditions of ketenimines.

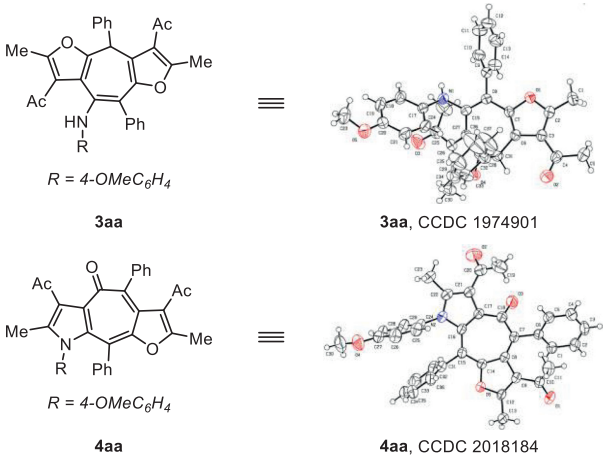
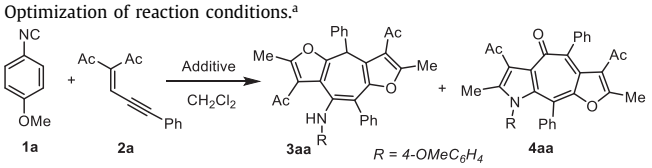


Fig. 2. X-ray crystallography of **3aa** and **4aa**.

cally affected the reaction efficiency (entry 4). Other zinc salts such as $ZnBr_2$, ZnI_2, $Zn(OTf)_2$ and $Zn(OAc)_2$ provided inferior results regarding the yield of **3aa** (entries 5–8). This reaction only took place in halogenated solvents (entries 9 and 10); minimal amount of **3aa** was formed in other reaction media (entries 11 and 12).

Next, we turned our attention to optimizing the formation of **4aa**. Initial effort to convert **3aa** directly to **4aa** under previously

Table 1
Optimization of reaction conditions.[a]

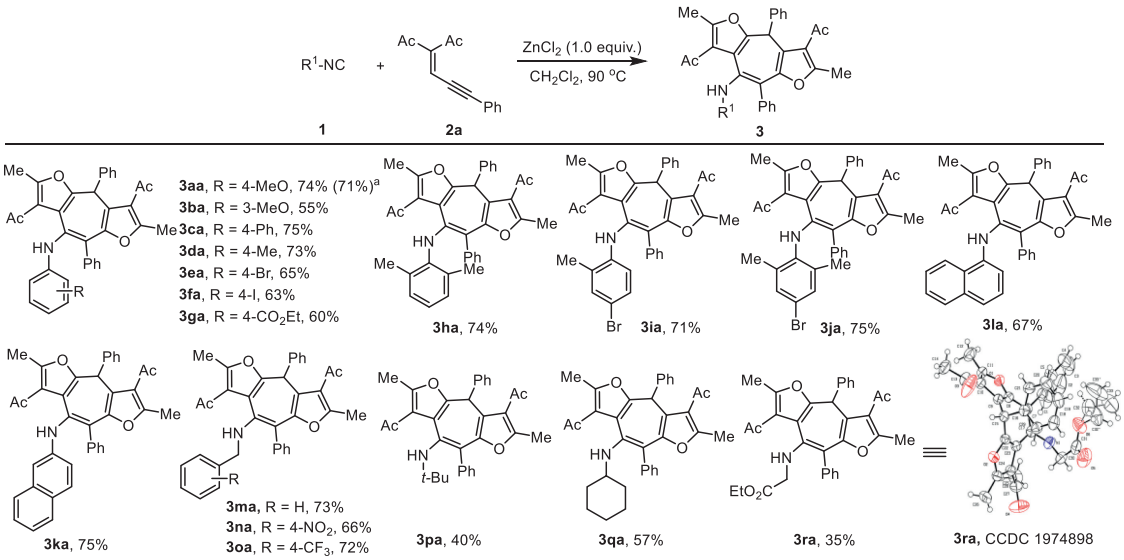


Entry	Additive (equiv.)	Solvent	Temp. (°C)	Time (h)	Yield (%)[b]	
					3aa	**4aa**
1	$ZnCl_2$ (1.0)	CH_2Cl_2	60	24	46	24
2	$ZnCl_2$ (1.0)	CH_2Cl_2	90	1.5	74	8
3	$ZnCl_2$ (1.0)	CH_2Cl_2	100	0.5	62	7
4	$ZnCl_2$ (0.8)	CH_2Cl_2	90	3	55	15
5	$ZnBr_2$ (1.0)	CH_2Cl_2	90	1.5	62	trace
6	ZnI_2 (1.0)	CH_2Cl_2	90	3	47	trace
7	$Zn(OTf)_2$ (1.0)	CH_2Cl_2	90	13	53	14
8	$Zn(OAc)_2$ (1.0)	CH_2Cl_2	90	17	trace	
9	$ZnCl_2$ (1.0)	$CHCl_3$	90	1.5	64	11
10	$ZnCl_2$ (1.0)	DCE	90	2	54	13
11	$ZnCl_2$ (1.0)	EtOH	90	20	–	–
12	$ZnCl_2$ (1.0)	Toluene	90	5	–	–
13	$ZnCl_2$ (1.0)	CH_2Cl_2	90	72	–	30
14[c]	$ZnCl_2$ (1.0), *m*-CPBA (1.0)	CH_2Cl_2	90	18	–	26
15[d]	$ZnCl_2$ (1.0), *m*-CPBA (1.0)	CH_2Cl_2	25	15	–	74
16[d,e]	$ZnCl_2$ (1.0), *m*-CPBA (1.0)	CH_2Cl_2	25	18	–	82

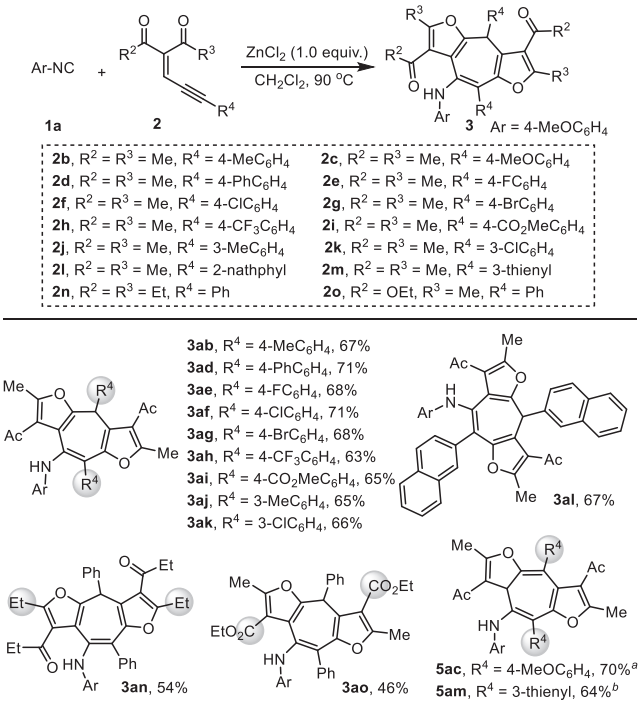
[a] Reaction conditions: Additive (1.0 equiv.) was added to a solution of isocyanide **1a** (0.3 mmol) and ene–yne-ketone **2a** (0.6 mmol) in CH_2Cl_2 (2 mL) under air atmosphere.
[b] Isolated yields.
[c] 1.0 equiv. of *m*-CPBA was added after the reaction mixture was stirred for 1.5 h.
[d] 1.0 equiv. of *m*-CPBA was added after the reaction mixture was stirred for 10 min.
[e] 2.5 equiv. of **2a** was used.

optimized conditions in prolonged reaction time only provided 30% yield (entry 13). When oxidant *m*-CPBA (or HOAc, see Supporting information for details) was added the conversion and decomposition of **3aa** was promoted (entry 14). A more detailed investigation into the reaction procedure revealed when 1.0 equiv. of *m*-CPBA was added after the reaction mixture was stirred for 10 min at 25 °C, the desired product **4aa** was obtained in 74% yield (entry 15). Finally, 82% yield of **4aa** was obtained when 2.5 equiv. of **2a** was used at 25 °C (entry 16). Herein, the optimal reaction conditions for **3aa** and **4aa** were determined as shown in entry 2 and entry 16, respectively.

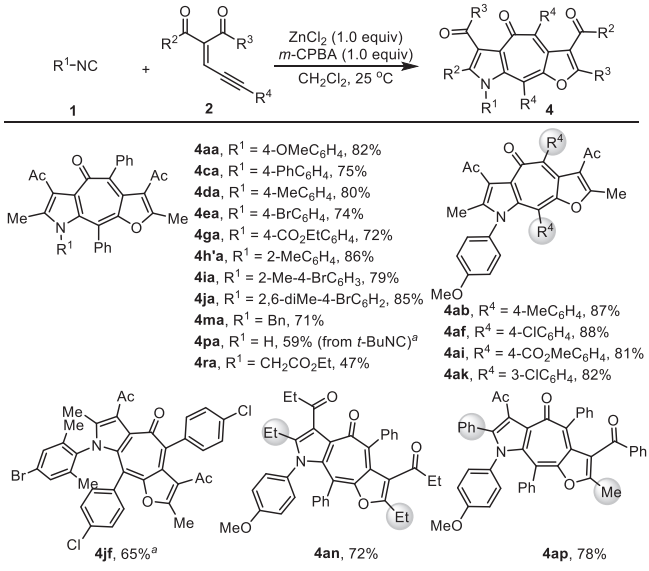
With the optimized conditions in hand (Table 1, entry 2), we began to investigate the generality of this strategy for synthesis of cycloheptafuran **3** (Scheme 2). In general, isocyanide substrates **1** with both electron-donating and electron-withdrawing substituents on the phenyl ring are compatible with this reaction, providing the desired products **3aa**–**3ga** with good yields. Gratifyingly, phenyl isocyanides with sterically hindered 2-methyl or 2,6-dimethyl substitution provided satisfactory yields (**3ha**–**3ja**). Naphthyl isocyanides could also be participated in this reaction to afford **3ka** and **3la** with good yields. In the case of alkyl isocyanides, benzyl isocyanides were converted into corresponding cycloheptafurans in good yields (**3ma**–**3oa**). The *tert*–butyl, cyclohexyl and active methylene isocyanide substrates could also be compatible in this method to afford **3pa**–**3ra** with slightly lower yields. Notably, the structure of product **3ra** was further confirmed by X-ray crystallography (CCDC: 1974898). It is worth mentioning that bromo, iodo and ester groups were tolerated in this reaction, thus providing a useful handle for further modification. Particularly, a gram-scale synthesis of **3aa** (0.989 g, 71% yield) was carried out, demonstrating the practicability of this protocol.



Scheme 2. Scope of isocyanides **1**. Reaction conditions: $ZnCl_2$ (1.0 equiv.) was added to a solution of isocyanide **1** (0.3 mmol) and ene–yne-ketone **2** (0.6 mmol) in CH_2Cl_2 (2 mL) at 90 °C in an air atmosphere for 1.5 h. Isolated yields. [a] Gram scale synthesis, 0.989 g **3aa** was obtained.



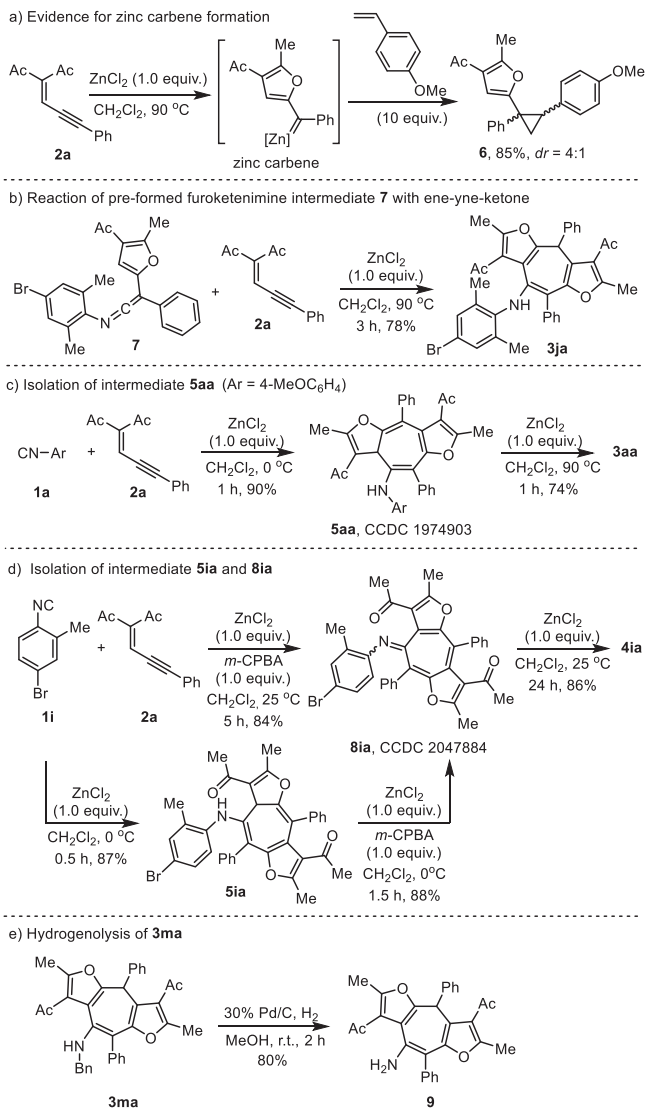
Scheme 3. Scope of ene–yne-ketones **2**. Reaction conditions: $ZnCl_2$ (1.0 equiv.), **1a** (0.3 mmol) and ene–yne-ketone 2 (0.6 mmol) in CH_2Cl_2 (2 mL) at 90 °C in an air atmosphere for 1.5 h. Isolated yields. [a] At 0 °C for 10 min. [b] At 0 °C for 15 min.



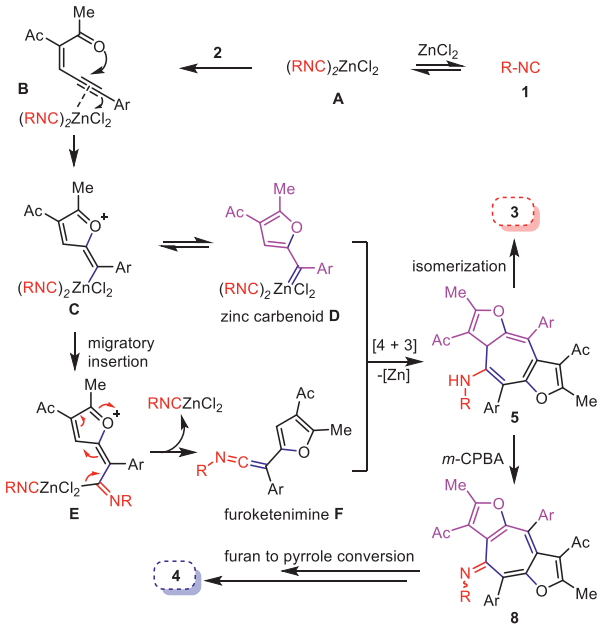
Scheme 4. Synthesis of cycloheptapyrroles **4**. Reaction conditions: unless otherwise noted, $ZnCl_2$ (1.0 equiv.) was added to a solution of isocyanide **1** (0.3 mmol) and ene–yne-ketone **2** (0.75 mmol) in CH_2Cl_2 (2 mL), and the reaction mixture was kept at 25 °C in an air atmosphere for 10 min, then *m*-CPBA (1.0 equiv.) was added. The reaction mixture was stirred for another 18 h. Isolated yields. [a] $ZnCl_2$ (1.5 equiv.) was used.

Next, the scope of the ene–yne-ketones **2** was tested (Scheme 3). For acetylacetone derived substrates **2a–2m** ($R^2 = R^3 = Me$), when aryl groups were adopted at R^4 position, including electron-rich (**2b** and **2j**), electron-neutral (**2a** and **2d**), and electron-deficient phenyl groups (**2e–2i** and **2k**), as well as naphthyl group (**2l**), those yield furo-fused cycloheptatriene products **3ab**, **3ad**–**3al** in good result. However, substrates **2c** with a 4-methoxylphenyl R^4 group and **2m** with a 3-thienyl R^4 group led to a complex mixture, respectively. Notably, when the reaction temperature was decreased to 0 °C for a few minutes, compounds **5ac** and **5am**, the isomers of **3**, were obtained in good yields. In addition, ene–yne-ketone substrates **2n** and **2o** derived from heptane-3,5–dione and ethyl acetoacetate underwent this transformation smoothly, giving the corresponding products **3an** and **3ao** in moderate yields. Disappointingly, ene–yne-ketone with alkyl group at R^4 position failed to yield desired product **3**. It is probably because the alkyl substituted ene–yne-ketone led to a much unstable alkyl substituted zinc furo-carbenoids.

To explore the applicability of this methodology for synthesis of cycloheptapyrrole **4**, the scope of both isocyanides **1** and ene–yne-ketones **2** was tested and summarized in Scheme 4. In terms of phenyl isocyanides, various substituent groups on the aromatic ring were tolerated to afford the furo-fused cycloheptapyrrole products **4aa–4ja**, **4jf** with good yields. The cycloheptapyrrole products **4ma** (71%) and **4ra** (47%) could also be obtained



Scheme 5. Mechanistic investigation.



Scheme 6. Proposed mechanism.

by using active methylene isocyanide **1m** and **1r**, respectively. However, when treated with *tert*–butyl isocyanide **1p**, the corresponding product **4pa** was obtained with eliminating the *tert*–butyl group. In general, sterically hindered isocyanide required longer reaction time and excess of $ZnCl_2$ were necessary, suggesting that the formation of the pyrrole ring in product **4** may be the rate-determining step (**4ja**, **4pa** and **4jf**). The ene–yne-ketones **2** containing both electron-donating and electron-withdrawing phenyl group on R^4 position were all suitable for this reaction, providing **4ab–4ak** in high yields. Ene–yne-ketone substrates **2n** and **2p** derived from other 1,3-diketones underwent this reaction smoothly, and desired products (**4an** and **4ap**) were obtained in good yields. However, ene–yne-ketone **2o** derived from β-ketoester was not viable in this reaction.

To elucidate the mechanism, several control experiments were carried out as shown in Scheme 5. Cyclopropane derivative **6** was isolated in 85% yield when treating ene–yne-ketone **2a** with 4-methoxystyrene under the zinc-promoted reaction conditions, suggesting the formation of a zinc furyl-carbenoid intermediate during the reaction process (Scheme 5a) [46–51]. Presumably, zinc carbenoid can react with isocyanide to form ketenimine [30]. Thus, we synthesized furyl-ketenimine **7** under the known Pd-catalysis conditions [30] and employed it to react with ene–yne-ketone **2a** under our zinc-promoted conditions. Gratefully, the desired cycloheptafuran **3ja** was obtained in good yield (Scheme 5b). This evidence supports the furyl-ketenimine intermediate is involved in the mechanism. Interestingly, when treating **1a** and **2a** in the presence of $ZnCl_2$ at 0 °C, a cyclized product **5aa** was obtained in excellent yield. This thermodynamically labile intermediate **5aa** was readily isomerized to **3aa** upon heating (Scheme 5c). These results suggested that the [4 + 3] cycloaddition likely gave rise to compound **5** firstly, which served as a key intermediate to provide cycloheptafuran **3** by isomerization.

In addition, we found a 4–bromo-2-methylphenyl isocyanide derived **5ia** intermediate, formed under similar zinc-promoted reaction conditions, can be oxidized rapidly by *m*-CPBA at 0 °C to provide imine **8ia**, which could be isolated and further converted to cycloheptapyrrole **4ia** after reacting at 25 °C for 24 h. Moreover, imine **8ia** could be isolated in excellent yield when reacting **1i** and **2a** under zinc-promoted oxidative conditions for a shorter time (Scheme 5d). Notably, both structures of the key intermediate **5aa** (CCDC: 1974903) and **8ia** (CCDC: 2047884) have been confirmed by X-ray crystallography. These results suggested that the [4 + 3] cycloaddition likely gave rise to compound **5** first, which served as a common intermediate to either provide cycloheptafuran **3** by isomerization or form cycloheptapyrrole **4** by an *m*-CPBA oxidation-rearrangement sequence (see Supporting information for more details). $ZnCl_2$ played multiple roles in this reaction. First, it initiated the intramolecular cyclization to form the zinc carbenoid; second, it promoted the [4 + 3] cycloaddition and subsequent isomerization to form furan product 3; third, it accelerated *m*-CPBA oxidation to yield imine 8 and triggered rearrangement to form pyrrole product 4. Thus, the synergistic and benign effect of a single zinc accelerator is essential for this methodology. Lastly, product **3ma** can undergo hydrogenolysis to provide cycloheptatriene amine **9**, which can serve as a useful intermediate to access diverse substitutions on the nitrogen (Scheme 5e).

Based on these preliminary studies and literatures [31,32,46,52], we proposed a tentative mechanism (Scheme 6). First, Coordination of isonitrile **1** with zinc formed complex **A** [46], which upon activation of alkyne moiety of **2** afforded furyl-carbenoid intermediate **C** or **D** [47–52]. Isocyanide migratory insertion from inter-

mediate **C** or **D** occurred to form furyl-ketenimine **F** [30]. Then, ketenimine intermediate F participated in a formal [4 + 3] cycloaddition with another zinc furyl-carbenoid **D** to yield the furo-fused cycloheptatriene **5** [53]. This process was rather fast and could be completed at 0 °C. At last, isomerization of **5** provided the thermally stable furo-fused cycloheptatriene product **3**. While addition of *m*-CPBA in the presence of $ZnCl_2$ caused the formation of imine **8**, which eventually led to the cycloheptapyrrole product **4** *via* a $ZnCl_2$ promoted rearrangement (for more details, see Supporting information).

In summary, we have developed an efficient and divergent approach to access cycloheptafuran and cycloheptapyrrole scaffolds, respectively. A novel [4 + 3] cycloaddition process between the *in situ* generated furyl-ketenimine and zinc furyl-carbenoid is supported by mechanistic investigation. This reaction utilizes readily available starting materials and inexpensive metal additive. Three rings and five bonds are successively constructed, which exemplified the concise and green aspect of this multicomponent domino reaction. Further investigation of this [4 + 3] cycloaddition in the synthesis of structurally relevant natural products and pharmaceuticals is currently under investigation in our group.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Acknowledgments

The authors gratefully acknowledge financial support from National Natural Science Foundation of China (Nos. 22171168, 22001152 and 22101159), Taishan Scholar Program of Shandong Province and the Natural Science Foundation of Shandong Province of China (No. ZR2020QB019).

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