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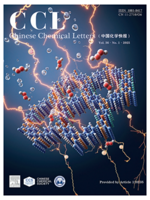

I_2-DMSO mediated tetra-functionalization of enaminones for the construction of novel furo[2′,3′:4,5]pyrimido[1,2-*b*]indazole skeletons *via in situ* capture of ketenimine cations

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ABSTRACT

The first-ever synthesis of the unknown furo[2′,3′:4,5]pyrimido[1,2-*b*]indazole skeleton was demonstrated based on the undiscovered tetra-functionalization of enaminones, with simple substrates and reaction conditions. The key to realizing this process lies in the multiple trapping of the *in situ* generated ketenimine cation by the 3-aminoindazole, which results in the formation of four new chemical bonds and two new rings in one pot. Moreover, the products of this new reaction were found to exhibit aggregation-induced emission (AIE) without modification.

Polycyclic nitrogen heterocyclic skeletons are widely found in naturally occurring compounds and also have applications as luminescent materials and semiconductors [1–3]. The discovery of new polycyclic nitrogen heterocyclic skeletons offers more possibilities for the screening of drug molecules and luminescent materials, as well as an important means of exploring organic chemistry. However, the development of simple and practical new methods for the direct assembly of novel large π-conjugated molecules of this type using readily available substrates has long been a challenge [4–6].

The development of novel reaction modes enables the discovery of new reaction mechanism and unknown skeletons [7,8]. Enaminones are emerging synthetic building blocks capable of providing multiple nucleophilic or electrophilic reaction sites and thus have the potential to allow new reactions modes. In recent years, numerous elegant research works have been reported by Wan and Yu *et al.*, and the multiple reactivity of enaminone has been deeply investigated [9–17]. Specifically, the currently reported mono-functionalisation reactions of enaminones were mainly focused on the regionally selective modification of the α- or β-positions of C=C with heteroatoms to construct C–X bonds (Scheme 1a) [18–23]. As shown in this scheme, there are numerous reaction modes available for the bi-functionalization of enaminones. (a) Using “CO” and β-positions as triatomic synthons for the construction of five- or six-membered rings [24–31]. (b) On the basis of the cleavage of a C–N bond at the β-position, the β,β-disubstituted phenylacetones or 2-substituted chromones can also be obtained [32–36]. (c) The different activities of α- and β-positions can be used to achieve the installation of bifunctional groups [37,38], the construction of heterocycles by [2 + n] cyclization reactions [39–46], and abundant use of *o*-hydroxyphenyl enaminones to construct 3-substituted chromone skeletons (Scheme 1b) [47–53]. In contrast, tri-functionalization reactions of enaminones are rarely and only a few examples have been reported. Among these, works involving the “CO”, α- and β-positions have allowed the synthesis of trisubstituted pyrazoles and pyrrolo[3,4-*c*]quinolineones. Recently, our group achieved the construction of chromeno[2,3-*b*]pyrrolones skeleton using *o*-hydroxyphenyl enaminones *via* multiple cyclization reactions at the α-, β- and β-positions

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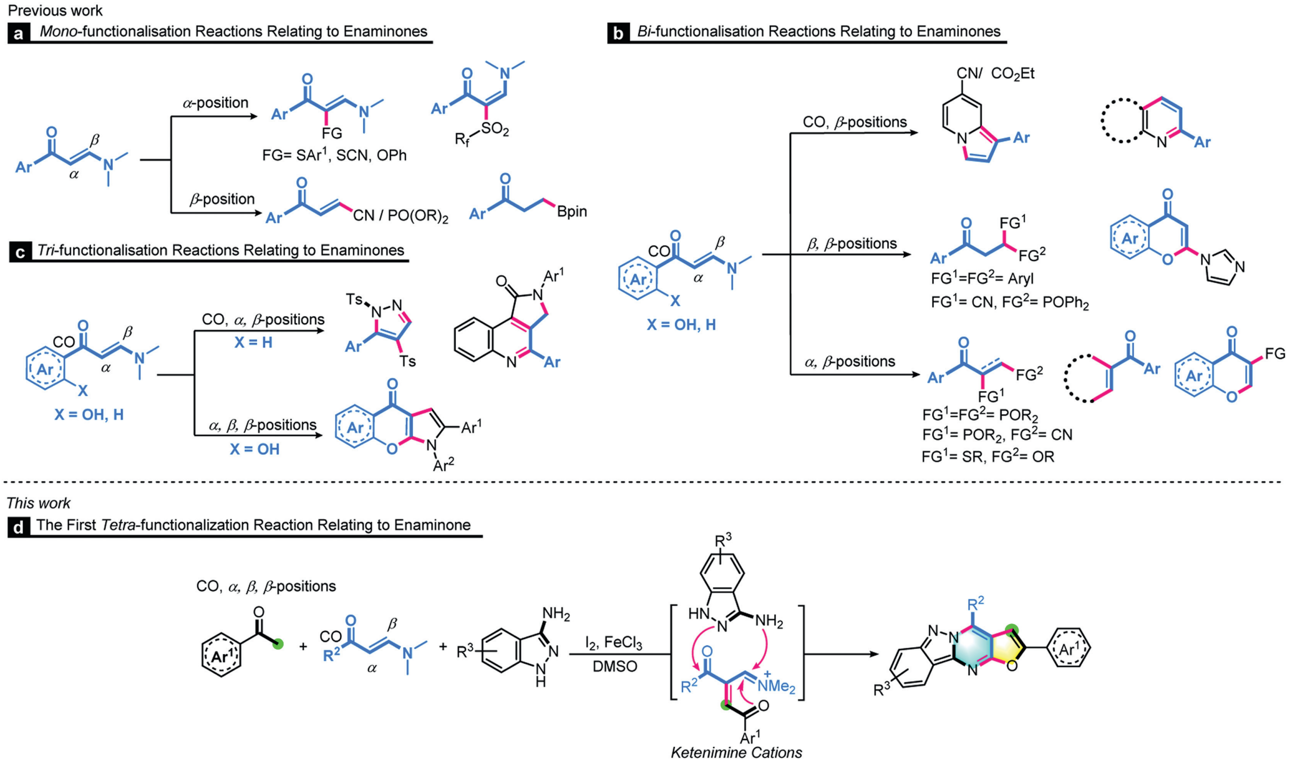


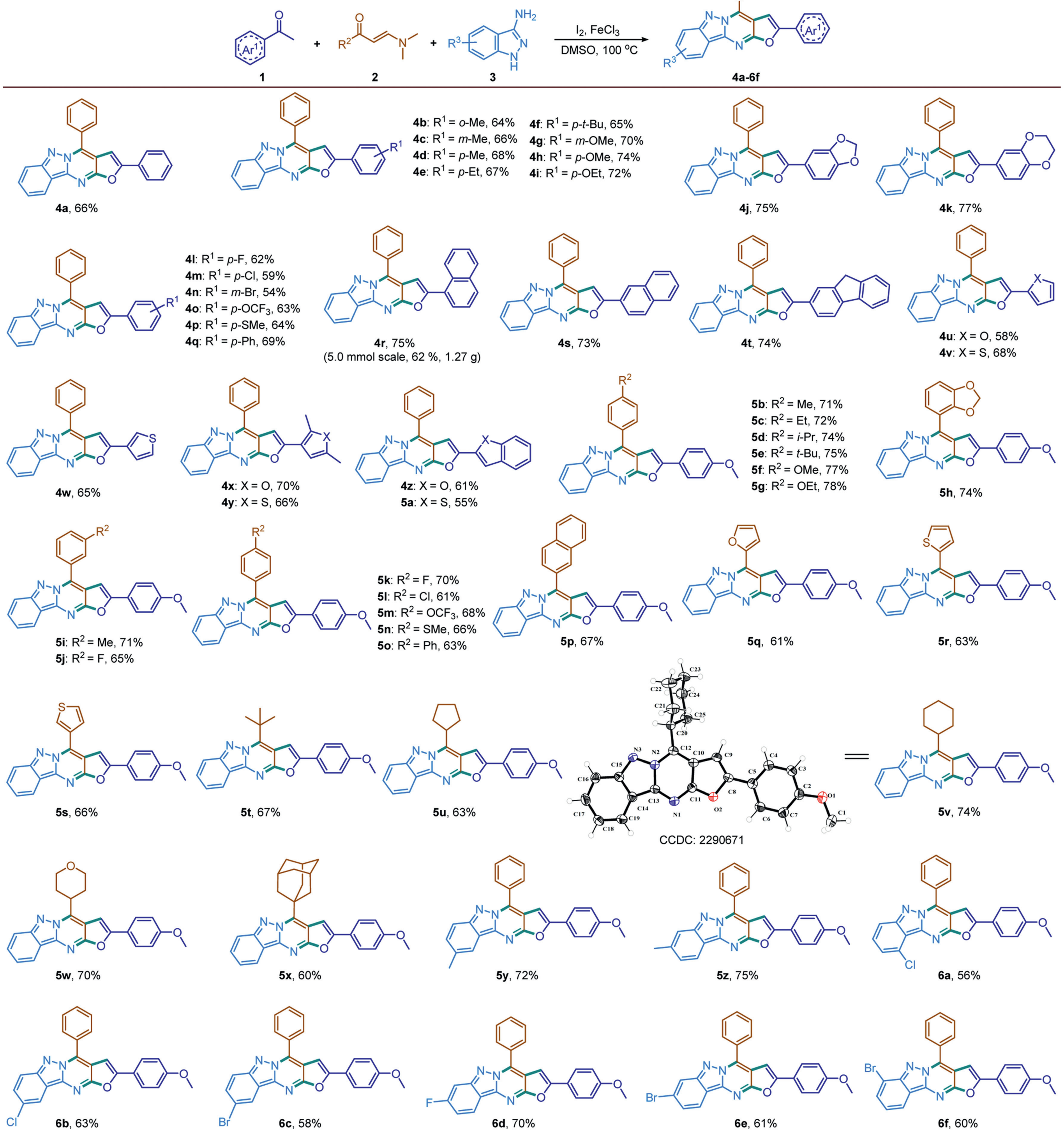
Scheme 1. Related research work of enaminones.

(Scheme 1c) [54–57]. However, the higher order functionalization reaction mode of enaminones has been undiscovered even to date. Here, we exhaustively used the four reaction sites, "CO", α-, β-, and β-positions, to successfully achieve the tetra-functionalization reaction of enaminones, resulting the discovery of the unknown furo[2′,3′:4,5]pyrimido[1,2-*b*]indazole skeleton. Furthermore, this efficiently assembled synthetic method provided a new strategy for the design of luminescent materials for the obtained polycyclic heterocyclic skeleton showed typical AIE feature (Scheme 1d).

To evaluate the viability of this proposal, the reaction conditions were optimized using acetophenone (**1a**), 3-(dimethylamino)-1-phenylprop-2-en-1-one (**2a**) and 1*H*-indol-3-amine (**3a**) as model substrates (Table 1). First, the reaction was mediated only by I_2 and the isolated yield of the target product was only 38% (entry 1). When Brönsted acids or Lewis acids were used as additives, the optimal yield of product is achieved with $FeCl_3$ (entries 2–11). Then the equivalents of I_2 and $FeCl_3$ were optimized and found that 1.5 equiv. of I_2 and 1.0 equiv. of $FeCl_3$ were the optimum (entries 12–17). Finally, by comparing the reaction at different temperatures, 110 °C was determined to be the best (entries 18–21).

Subsequent trials investigated the ranges of arylmethyl ketones, enaminones and 3-aminoindazoles (Scheme 2). Firstly, acetophenone with alkyl and alkoxy substitutions at different sites on the benzene ring could be readily converted to the target products (**4a**-**4k**, 64%–77%). In addition, with halogen, methylthio and phenyl substituted acetophenones, the target products were obtained smoothly (**4l**-**4q**, 54%–69%). For 1-acetylnaphthalene, 2-acetylnaphthalene and 2-acetylfluorene with condensed ring structures the target molecules were obtained in relatively high yields (**4r**-**4t**, 73%–76%). Fortunately, heterocycles with furan, thiophene and their derived structures were also converted to the products in moderate yields (**4u**-**5a**, 55%–70%). Furthermore, the compatibility of aryl and alkyl enaminones was also assessed. The substitution of alkyl and alkoxy groups on the benzene ring of the enaminones allowed the formation of the target products (**5b**-**5i**, 71%–78%). Phenyl enaminones having halogen, trifluoromethoxy, methylthio and phenyl substituents were also compatible with the reaction (**5j**-**5o**, 61%–70%). Aromatic enaminones with non-benzene rings such as naphthalene, furan and thiophene could also be converted to the target structures successfully (**5p**-**5s**, 61%–67%). Fortunately, alkyl type enaminones incorporating *tert*-butyl, cyclopentyl, cyclohexyl, 4-tetrahydropyranyl and 1-adamantyl groups also yield the products satisfactorily (**5t**-**5x**, 60%–74%). The structure of product **5v** was confirmed by X-ray single crystal diffraction (CCDC: 2290671). Finally, the range of substrates for substituted 3-aminoindazoles were examined and found that both 5-Me and 6-Me substituted 3-aminoindazoles gave the target products in appreciable yields (**5y** and **5z**, 72% and 75%). Moreover, the polycyclic nitrogen heterocyclic were also successfully achieved when using 4-, 5-, 6-, 7-site halogen-substituted 3-aminoindazole as substrates (**6a**-**6f**, 56%–70%). It is noteworthy that the product **4r** obtained from 1-acetylnaphthalene (**1r**) was scaled up on a 5.0 mmol scale and the isolated yield was still maintained at 62%.

The photophysical properties of the unknown molecules were also investigated (Fig. 1). These trials primarily explored the AIE properties and solvated fluorescence characteristics of products **4r** and **4a** at 360 nm (see Supporting information for details). Molecules **4r** was examined in equal concentrations of $MeCN/H_2O$ mixed solvents with different water fractions, when the water fraction increased from 0 to 60% its fluorescence emission intensity gradually weakened. However, the fluorescence intensity increased rapidly when the f_w went from 70% to 98% (Fig. 1a). Further, the trend of maximum emission intensity with water fraction was showed in Fig. 1b, which demonstrates the weakest fluorescence intensity occurred at 60% water fraction. The tendency of first reducing and then increasing of the emission intensity may be caused by the superposition of the intrinsic AIE effect and the solvent effect of fluorescence [58–60]. These results demonstrated their typical AIE properties and have potencial in organic functional luminophores [61–63].



Scheme 2. Scope of the substrates. Reaction conditions: **1** (1.2 mmol), **2** (1.0 mmol), **3** (1.0 mmol), I_2 (1.5 mmol), $FeCl_3$ (1.0 mmol), DMSO (4 mL), 110 °C, and 8 h. Isolated yield.

The reaction mechanism was examined by performing a series of controll experiments (Scheme 3). In initial trials, acetophenone (**1a**) was almost quantitatively converted to phenylglyoxal (**1ab**) or phenylglyoxal monohydrate (**1ac**) mediated by I_2 and DMSO (Scheme 3a) [64–66]. These experiments further confirmed that α-iodoacetophenone (**1ad**) and phenylglyoxal monohydrate (**1ac**) were intermediates in the reaction (Schemes 3b and c). In the case that intermediate **1ac** was used as substrate, $FeCl_3$ was found to play a more important role than I_2, whereas the target product could not be obtained if both were absent (Scheme 3c). The generation of substituted furan **7a** under the standard conditions, indirectly proved the existence of ketenimine cation **B-H**, which may experience self-cyclization and subsequent deprotonation process (Scheme 3d). Finally, the use of deuterated acetophenone-d_3 (**1a-D**) as substrate gave the deuterated product **4a-D** in 68% isolated yield and the retention of deuterium atoms up to 90% in the product.

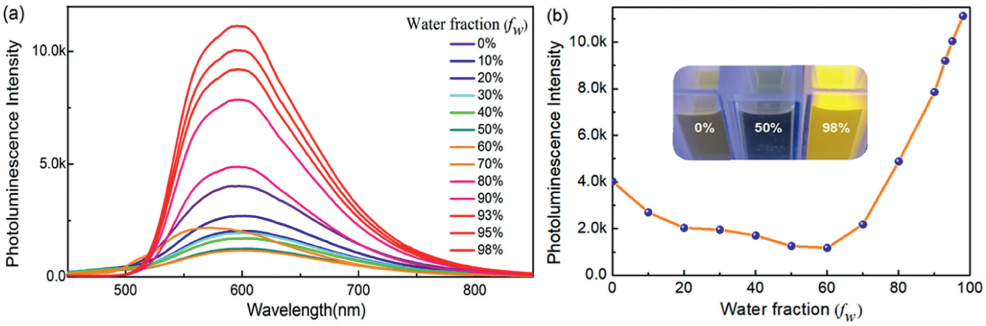
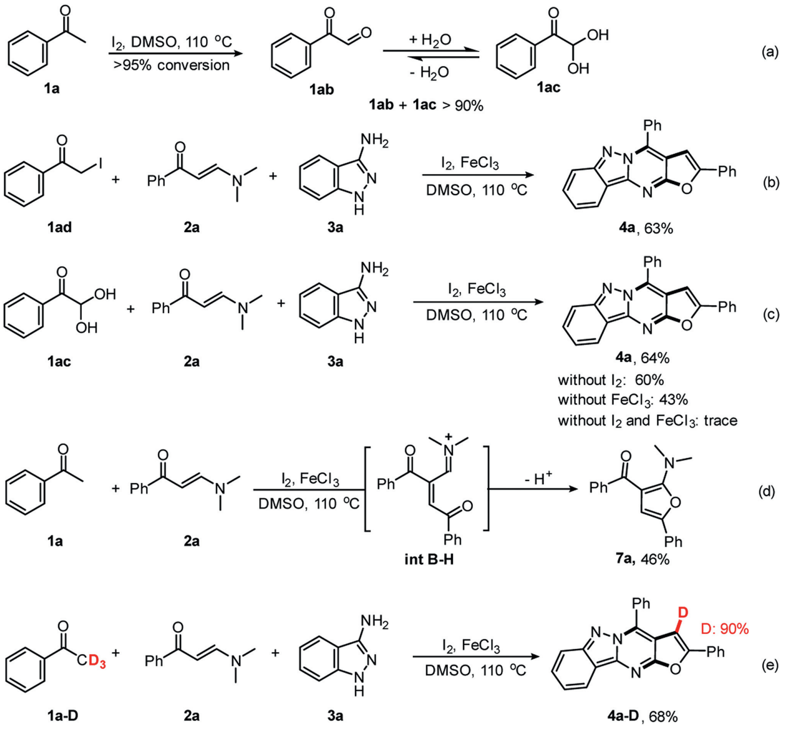


Fig. 1. (a) Photoluminescence (PL) spectra of **4r** MeCN/H_2O mixtures with different water fractions (f_w). (b) Maximum fluorescence emission intensity of **4r** as a function of f_w. Inset: Luminescence photographs of **4r** in MeCN/H_2O mixtures (f_w = 0%, 50% and 98%) taken under 365 nm excitation.

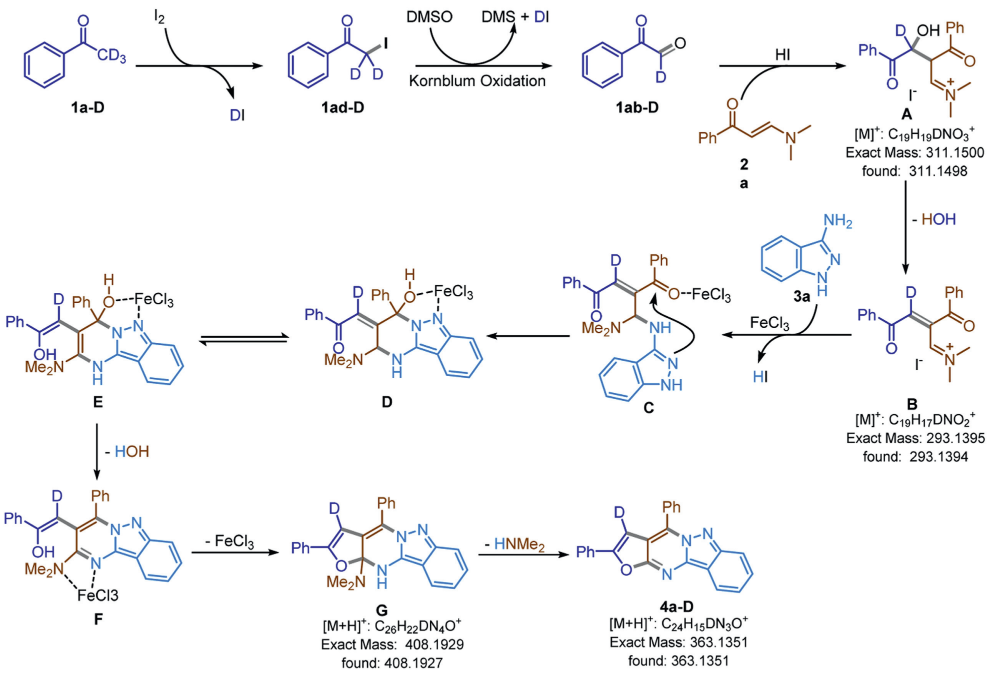


Scheme 3. Mechanistic studies.

This result demonstrated that the mechanism involved the cleavage of two C–D bonds, meaning that the only retained C–D bond in phenylglyoxal monohydrate (**1ac**) remains unbroken during the reaction (Scheme 3e).

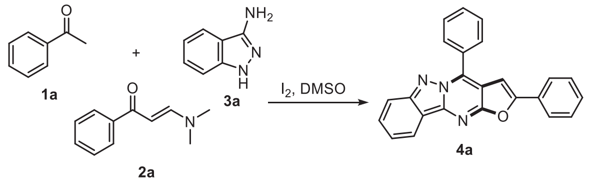
On the basis of these control experiments and related literature, we propose a reaction mechanism around the capture of ketenimine cation (Scheme 4) [67–75]. In this process, the deuterated acetophenone-d_3 (**1a-D**) is initially converted to phenylglyoxal-d_1 (**1ab-D**) *via* intermediate α-iodoacetophenone-d_2 (**1ad-D**), mediated by I_2 and DMSO. Subsequently, intermediate **1ab-D** is attacked by the electron-rich α-site of enaminone (**2a**) to afford the unstable cationic intermediate **A**. The intermediate **A** convert to the key ketenimine cation **B** with the release of water. The imine structure of intermediate **B** is then attacked by the amino group of 3-aminoindazole (**3a**) to form **C**, following intramolecular nucleophilic addition to yield intermediate **D** with the coordination of FeCl$_3$. The tautomerism of **D** occurs to generate the enol form **E**, which subsequently undergoes dehydration to give intermediate **F**. Then, the two nitrogen atoms of the intermediate **F** may coordinate with FeCl$_3$, increasing the electrophilicity of the carbon atom between the two nitrogen atoms, thus allowing the hydroxyl group to undergo nucleophilic cyclization to form intermediate **G**. Finally, intermediate **G** along with the lost HNMe$_2$ gas under heating to form the target product **4a-D**. Delightedly, intermediates **A**, **B**, **G** and product **4a-D** were detected *in situ* during analysis of the reaction solution by high-resolution mass spectrometry, providing evidence for the proposed mechanism.

In conclusion, this work utilized arylmethyl ketones and enaminones to generate ketenimine cations *in situ*, which subsequently captured by 3-aminoindazole to achieve the bicyclization process. This transformation realized the first-ever tetra-functionalization reaction of enaminones, allowing the construction of novel polycyclic nitrogen heterocyclic furo[2′,3′:4,5]pyrimido[1,2-*b*]indazole skeletons. The photophysical properties of the new skeletons were further investigated and shown favorable AIE properties, which may have potential applications in the organic functional luminescent materials. Research to develop synthetic novel molecular skeletons and assessments of the photophysical properties are currently underway in our laboratory.



Scheme 4. Proposed mechanism.

Table 1
Optimization of reaction conditions.[a]



Entry	I_2 (equiv.)	Additives (equiv.)	Temp (°C)	Yield (%)[b]
1	1.5	-	100	38
2	1.5	50% HI (1.0)	100	32
3	1.5	CF_3CO_2H (1.0)	100	45
4	1.5	CF_3SO_2H (1.0)	100	15
5	1.5	TsOH (1.0)	100	39
6	1.5	$CuCl_2$ (1.0)	100	34
7	1.5	$CuSO_4$ (1.0)	100	10
8	1.5	$FeCl_2$ (1.0)	100	49
9	1.5	$FeCl_3$ (1.0)	100	60
10	1.5	$Fe(NO_3)_3$ (1.0)	100	28
11	1.5	$Fe_2(SO_4)_3$ (1.0)	100	35
12	1.5	$FeCl_3$ (0.5)	100	50
13	1.5	$FeCl_3$ (1.5)	100	55
14	1.5	$FeCl_3$ (2.0)	100	57
15	0.5	$FeCl_3$ (1.0)	100	48
16	1.0	$FeCl_3$ (1.0)	100	53
17	2.0	$FeCl_3$ (1.0)	100	58
18	1.5	$FeCl_3$ (1.0)	80	48
19	1.5	$FeCl_3$ (1.0)	90	56
20	1.5	$FeCl_3$ (1.0)	110	66
21	1.5	$FeCl_3$ (1.0)	120	63

[a] Reaction conditions: **1a** (0.60 mmol), **2a** (0.50 mmol) and **3a** (0.50 mmol) at different temperatures for 8 h under air.

[b] Isolated yields.

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.

CRediT authorship contribution statement

You Zhou: Conceptualization, Data curation, Formal analysis, Investigation, Methodology, Project administration, Resources, Software, Supervision, Validation, Visualization, Writing – original draft, Writing – review & editing. **Li-Sheng Wang:** Data curation, Formal analysis, Supervision, Writing – review & editing. **Shuang-Gui Lei:** Data curation, Formal analysis, Investigation, Supervision, Writing – review & editing. **Bo-Cheng Tang:** Investigation, Methodology, Writing – review & editing. **Zhi-Cheng Yu:** Formal analysis, Supervision, Writing – review & editing. **Xing Li:** Data curation. **Yan-Dong Wu:** Conceptualization, Funding acquisition, Investigation, Methodology, Project administration, Supervision, Validation, Writing – review & editing. **Kai-Lu Zheng:** Investigation, Methodology, Project administration, Resources, Software, Supervision, Validation, Visualization, Writing – review & editing. **An-Xin Wu:** Conceptualization, Funding acquisition, Investigation, Methodology, Project administration, Resources, Software, Supervision, Validation, Visualization, Writing – review & editing.

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Supplementary materials

Supplementary material associated with this article can be found, in the online version, at doi:10.1016/j.cclet.2024.109799.

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