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Phosphine-catalyzed (3+2)/(2+3) sequential annulation involving a triple nucleophilic addition reaction of  $\gamma$ -vinyl allenoates†‡

Jiaxu Feng and You Huang 🗅 \*

A phosphine-catalyzed (3+2)/(2+3) sequential annulation involving a triple nucleophilic addition reaction of  $\gamma$ -vinyl allenoates was successfully developed. The reaction provided efficient and more practical access to functionalized hydropyrroloimidazolones with good to excellent yields under mild reaction conditions. Notably,  $\gamma$ -vinyl allenoate served as a triple-electrophilic intermediate in this protocol.

Hydropyrroloimidazolones constitute a valuable class of nitrogen heterocycles and are widely prevalent in biologically active products and pharmaceuticals (Fig. 1).<sup>1</sup> While effective methods for their syntheses have been reported with substituted N-heterocycles,<sup>2</sup> these syntheses remain a formidable challenge.

Phosphine-catalyzed nucleophilic addition<sup>3</sup> is one of the most popular tools for the assembly of C–C and C–N bonds in organic synthesis.<sup>4</sup> In this field, activated allenoates have been recognized as one of the most effective starting materials<sup>5</sup> and the seminal work has been vastly expanded upon, leading to great potential for the synthesis of natural products.<sup>6</sup> The formidable breakthrough in phosphine catalysis was reported by Trost, who discovered that the  $\gamma$ -umpolung addition of nucleophiles to activated allenoates and alkynoates leads to the formation of C–C bonds<sup>7</sup> (Scheme 1, (a)). Subsequently, many efforts have been devoted to the  $\gamma$ -umpolung addition of various pro-nucleophiles.<sup>8</sup> Later on, Huang and Lu reported phosphine-catalyzed  $\beta$ -Michael addition<sup>9</sup> under mild reaction conditions (Scheme 1, (b)).

Phosphine-catalyzed nucleophilic addition of allenoates using di-nucleophiles as substrates has emerged as a valuable approach for forming functionalized carbocycles and heterocycles from acyclic reactants (Scheme 1, (c)).<sup>10</sup> In 2002, Lu reported using a tandem reaction involving phosphinecatalyzed umpolung addition and intramolecular conjugate addition to form carbocycles and heterocycles.<sup>11</sup> Later, Lu<sup>12</sup> and Kwon<sup>13</sup> delivered, respectively, the efficient syntheses of carbocycles and heterocycles using allenoates and various dinucleophiles in a tandem reaction. Moreover, Tong<sup>14</sup> and Shi<sup>15</sup> similarly described a phosphine-catalyzed tandem annulation with  $\delta$ -acetoxy allenoates to 1,3-bisnucleophiles, which could be converted to various heterocycles. Furthermore, the reactions of 1,1- and 1,2-bisnucleophiles with allenoates to form five- and six-membered carbocyclic and heterocyclic products have also been reproted.<sup>16</sup> To the best of our knowledge, there has been no report about reactions between allenoates and nucleophiles involving triple nucleophilic additions, probably because of several challenges that could not be ignored, such as (1) competing reactions at multiple reaction sites in allenoates, (2) difficulty in finding suitable nucleophiles, and (3) difficulty in controlling the regioselectivity. In order to overcome these issues and accomplish this desired reaction, it is critical to carefully design the allenoates and screen multifunctional nucleophiles. On the basis of our group's previous work in the phosphine catalysis of sequential annulation,<sup>17</sup> we designed and synthesized a new kind of  $\gamma$ -vinyl allenoate. Our group provided here the first examples of phosphine-catalyzed (3+2)/(2+3) sequential annulation mediated by a triple nucleophilic addition reaction of  $\gamma$ -vinyl allenoates with urea derivatives (Scheme 1, (d)). This work featured a fine control



OCH<sub>3</sub>

<sup>Br</sup>Me

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 $<sup>{\</sup>it State Key \ Laboratory \ and \ Institute \ of \ Elemento-Organic \ Chemistry, \ College \ of}$ 

Chemistry, Nankai University, Tianjin 300071, China. E-mail: hyou@nankai.edu.cn † Dedicated to 100th anniversary of Nankai University and dedicated to the 100th anniversary of the birth of the academician Ruyu Chen.

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Previous work:



Scheme 1 Nucleophilic addition of the allenoate in the phosphine catalyst.

of the regioselectivity and a wide substrate scope, which provided a new and practical route to the construction of hydropyrroloimidazolone derivatives.

At the outset of our studies, we chose 1-(1-methyl-2-oxoindolin-3-yl)-3-(*p*-tolyl)urea (1a) and  $\gamma$ -vinyl allenoates (2a) as model starting materials to explore the optimal conditions for this (3+2)/(2+3) nucleophilic addition reaction. As a result of our initial screening of conditions, the use of PPh<sub>3</sub> in toluene at 110 °C led to the corresponding heterocyclic product diastereomer **3aa** in 23% yield (Table 1, entry 1).

Screening of phosphine catalysts revealed that PBu<sub>3</sub> and  $(p-FC_6H_4)_3P$  were ineffective in this reaction (entries 2 and 3). Gratifyingly, when  $(p-MeOC_6H_4)_3P$  was used, two diastereomers were obtained in 94% total yield. Pleasingly, we were able to completely separate the diastereomers **3aa** and **4aa** using silica-gel

| Table 1         Optimization of the reaction conditions <sup>a</sup> |                    |            |                        |    |
|--|--------------------|------------|------------------------|----|
|  | in catalyst (0.3   |            |                        |    |
|  | Lu                 |            | Yield <sup>b</sup> (%) |    |
| Entry  | Cat. (30 mol%)     | Temp. (°C) | 3                      | 4  |
| 1  | PPh <sub>3</sub>   | 110        | 23                     | _  |
| 2  | PBu <sub>3</sub>   | 110        | _                      | _  |
| 3  | $(p-FC_6H_4)_3P$   | 110        | _                      | _  |
| 4  | $(p-MeOC_6H_4)_3P$ | 110        | 45                     | 49 |
| 5  | $(p-MeOC_6H_4)_3P$ | 50         | 37                     | 32 |
| 6  | $(p-MeOC_6H_4)_3P$ | 25         | 32                     | 24 |
| 7 <sup>c</sup>   | $(p-MeOC_6H_4)_3P$ | 110        | 46                     | 50 |
| $8^d$  | $(p-MeOC_6H_4)_3P$ | 110        | 50                     | 42 |
| $9^{c,e}$  | $(p-MeOC_6H_4)_3P$ | 110        | 36                     | 32 |

<sup>*a*</sup> Reaction conditions: 0.20 mmol **1a** (1.0 equiv.), 0.60 mmol **2a** (3.0 equiv.), cat. (30 mol%) in toluene (2.0 mL) at 110 °C. <sup>*b*</sup> Yield of isolates product. <sup>*c*</sup> Cat. (20 mol%) was added. <sup>*d*</sup> Cat. (40 mol%) was added. <sup>*e*</sup> 0.40 mmol **2a** (2.0 equiv.) was used.



Scheme 2 The scope of this reaction. The reaction was performed under optimum conditions. Isolated yield is shown.

column chromatography and achieved yields of 45% and 49%, respectively (entry 4). Subsequent testing of the effect of temperature confirmed that 110 °C was the best temperature (entries 5 and 6) and in fact showed that the reaction outcome was highly affected by the reaction temperature. Moreover, further evaluation here of reaction conditions showed a higher yield when lowering the catalyst loading to 20% (entry 7), but increasing the (*p*-MeOC<sub>6</sub>H<sub>4</sub>)<sub>3</sub>P loading led to a similar result (entry 8). Lowering the amount of **2a** did not result in a satisfactory outcome (entry 9).

With the optimal reaction conditions in hand, we turned our attention to validating the potential and generality of this (3+2)/(3+2)(2+3) annulation reaction. We first investigated a variety of multifunctional nucleophiles (Scheme 2). A broad range of substituted ureas, including those bearing electron-donating groups (Me or MeO) and electron-withdrawing groups (F, Cl or Br) at the *para*- or *meta*-position of the aryl ring moiety  $(R^1)$ , were compatible with the reaction conditions and gave the corresponding diastereoisomer products in good to excellent yields (3aa-3ia, 4aa-4ia), respectively, showcasing the excellent functional group tolerance of the reaction. The relative configurations of the diastereoisomers 3aa and 4aa were determined by carrying out X-ray crystal analysis and others were assigned by analogy. In addition, urea 1j with a 3-CF<sub>3</sub>, 4-Cl substitution on the aryl group (3-CF<sub>3</sub>, 4-Cl bis-substituted urea 1j) was found to be suitable and afforded desired products with moderate yields. Delightedly, 5-fluorine and the 5-MeO group in the oxindole core also proved to yield good substrates, affording the products (3ka-3la, 4ka-4la) with good yields. Then we explored the effects of the protection of the nitrogen atom in the oxindole core by protecting groups such as CH<sub>2</sub>CH(OEt)<sub>2</sub> (1m-1r), n-Pr (1s-1t) and Bn (1u). It was found that the methyl protecting group was most effective. A gram-scale reaction of urea 1m and allenoates 2a was tested, and resulted in



an 83% overall yield. Meanwhile, other protecting groups were also well tolerated, which highlighted the broad substrate scope of this reaction. Furthermore, various  $\gamma$ -vinyl allenoates were tested, and produced the target compounds with good yields (**3ab**, **3ac**, **3bb**, **3bc**, **4ab**, **4ac**, **4bb**, **4bc**).

In order to further underscore the synthetic utility of this method, we also explored the reactivities of alkyne compounds. Intriguingly, when methyl hex-5-en-2-ynoate was employed in this annulation, the desired diastereoisomer products **3aa**, **4aa**, **3ma** and **4ma** were also obtained under the same conditions with satisfactory yields (Scheme 3).

To gain further insight into the mechanism of the reaction, control experiments were performed (Scheme 4). Treatment of 1-butyl-3-(1-methyl-2-oxoindolin-3-yl)urea 1v with  $\gamma$ -vinyl allenoates under the optimal conditions did not yield the corresponding products 3va and 4va, but the key intermediate product spirooxindole 5va was isolated (Scheme 4, (1)). The structure of 5va was confirmed from NMR and HRMS spectra. Additionally, the reaction of 1-benzyl-3-(1-methyl-2-oxoindolin-3-yl)urea, 1w, with 2a was tested, and the intermediate spirooxindole 5wa was also obtained with 20% yield (Scheme 4, (2)). The structure and relative configuration of intermediate 5wa was confirmed using X-ray crystallography. Next, two different pro-nucleophiles, 1x and 1y, were synthesized and we also examined their reactivities under optimal reaction conditions. However, when the pro-nucleophile 1x was employed as a substrate, no desired product was afforded under optimal conditions (Scheme S1, (1), see ESI<sup>‡</sup> for details), which indicated that the urea nitrogen atom played a crucial role in the annulation. Moreover, substrate 1y was also inefficient (Scheme S1, (2), ESI<sup>‡</sup>). These outcomes indicated that the oxindole backbone was indispensable and its structurally unique property may have triggered the formation of the important intermediate D.

On the basis of the detailed mechanistic understanding and our previous works,<sup>18</sup> a plausible reaction mechanism was



Scheme 4 Preliminary mechanism study



Scheme 5 Proposed reaction mechanism.

derived (shown in Scheme 5). This mechanism differs from the phosphine-catalyzed reaction mechanisms of allenoates reported before in which the key intermediates of allenoates were the (1,n)-zwitterion or the 1,4-bis-electrophile intermediates.

According to the currently proposed mechanism, first nucleophilic addition to vinyl allenoates 2 by PPh<sub>3</sub> afforded zwitterionic intermediate A, which then acted as a base to deprotonate the pro-nucleophile 1 leading to the construction of the intermediates B and C. Subsequently, according to the mechanism, nucleophilic attack of the vinyl group of intermediate B by intermediate C produced intermediate D-I, which formed an equilibrium with intermediate D-II, and then D-II deprotonated to generate the corresponding intermediate E. Next, according to the proposal, intramolecular Michael addition (the second nucleophilic addition) occurred to give the intermediate F, which then underwent a proton shift and the third nucleophilic addition (see path a in Scheme 5), finally producing the desired (3+2)/(2+3) annulation product. Through an alternative path (path b in Scheme 5), byproduct 5 was obtained through an H-shift and subsequent 1,2-elimination of PPh<sub>3</sub>.

In summary, we have developed a novel and efficient phosphinecatalyzed (3+2)/(2+3) sequential annulation mediated by triple nucleophilic addition. By using substituted urea compounds as readily accessible precursors of triple-functional nucleophiles, a wide range of hydropyrroloimidazolones were obtained in good to excellent yields under mild conditions, showing the great tolerance of the reaction to a wide variety of functional groups. This stepeconomical and regionally selective method involved formation of a C-C bond and two C-N bonds and the production of functionalized N-hetero-bicyclic derivatives bearing three stereogenic centers. In this sequence,  $\gamma$ -vinyl allenoates were shown to serve as versatile triple electrophiles and to be able to participate in three nucleophilic additions with pro-nucleophiles. Interestingly, the (3+2)/(2+3) sequential annulation was also compatible with the use of an envne, and in this case afforded hydropyrroloimidazolone with a satisfactory outcome. This approach provides a potential method

for the preparation of natural products and pharmaceutical molecules. Investigations of further applications of this method are ongoing in our laboratory.

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## Conflicts of interest

There are no conflicts to declare.

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