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One-pot synthesis of iodine-substituted 1,4-oxazepines

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ABSTRACT

A facile one-pot method for the synthesis of iodine-substituted 1,4-oxazepines is reported. When reacted with $ZnCl_2$ and I_2 in DCM at 40 °C, *N*-propargylic β-enaminones, prepared by the conjugate addition of propargylamine to α,β-alkynic ketones, underwent 7-exo-dig cyclization by zinc chloride and concomitant reaction with molecular iodine to afford 2-(iodomethylene)-2,3-dihydro-1,4-oxazepines in good to high yields. This cyclization was found to occur with broad scope of substrates and high tolerance of functional groups. The resulting iodine-containing 1,4-oxazepines can be further elaborated to more complex structures by subsequent cross-coupling reactions, which may provide a platform for biological studies.

1,4-Oxazepines represent a privileged class of heterocyclic compounds and appear in many bioactive molecules and pharmaceutical compounds.[1] Indeed, 1,4-oxazepines have been comprehensively studied in the last decades and still gain importance for their exciting biological and medicinal activities.[2] Many 1,4-oxazepine derivatives have exhibited remarkable medicinal properties,[3] including antidepressant,[4] antiulcer,[5] antipsychotic,[6] anxiolytic,[7] and antitumor[8] activities. Moreover, they have been used for the treatment of a range of diseases, such as bronchial asthma,[9] breast cancer,[10] epilepsy,[11] and psychotic disorders.[6] Notably, antidepressants *Amoxapine*[12] and *Sintamil* (nitroxazepine),[13] and antipsychotic and antischizophrenic *Loxapine*[14] are the well-known examples of 1,4-oxazepine-containing drugs. Although partially and fully saturated benzo-, dibenzo- and pyrido-fused, and/or oxo derivatives of 1,4-oxazepines are very common, half and fully unsaturated monocyclic 1,4-oxazepines are far less known. Especially, the fully unsaturated monocyclic derivatives are very scarce. Notably, very few approaches have been described for the synthesis of such compounds.[15] Recently, *N*-propargylic β-enaminones have attracted great attention as precious substrates in organic synthesis since their cyclizations have been recognized as an attractive way to synthesize a broad range of important heterocycles, including pyrroles, 1-pyrrolines, pyridines and dihydropyridines.[16] Although the cyclizations of *N*-propargylic β-enaminones have mainly produced five- and six-membered heterocycles, the studies regarding the synthesis of seven-membered ring systems, such as 1,4-oxazepines, have begun to show up.[17] Moreover, through the intermediacy of in situ-generated 1,4-oxazepines, a range of pyridine derivatives was synthesized.[18]

Our growing interest in novel heterocyclic compounds as potential pharmaceuticals and scaffolds has encouraged us to explore new reactivity routes of *N*-propargylic β-enaminones. In this regard, we have shown that when reacted with molecular iodine in the presence of sodium bicarbonate, *N*-propargylic β-enaminones afforded iodo-substituted pyridines in good to high yields via electrophilic cyclization with high functional group tolerance (Scheme 1a, left).[19] Iodopyridines were further elaborated to more functional structures by Suzuki–Miyaura and Sonogashira coupling reactions.[20] Recently, we have reported zinc chloride-mediated synthesis of 1,4-oxazepines from *N*-propargylic β-enaminones, as well (Scheme 1a, right).[21] This 7-exo-dig cyclization proceeded well and yielded 2-methylene-2,3-dihydro-1,4-oxazepine derivatives in good to high yields with large functional group tolerance and high efficiency. Importantly, the incorporation of an iodine atom into the structures of 1,4-oxazepines may provide opportunities for constructing more complex frameworks. In fact, iodine-containing compounds are very important building blocks for the synthesis of diverse natural products and pharmaceuticals since they can be easily modified to more sophisticated molecules by metal-catalyzed cross-coupling reactions. To the best of our knowledge, there is only one report in this regard. Boruah and coworkers showed that iodocyclization of *N*-propargylic β-(hydroxylmethyl)enamides led to in situ formation of 2-(iodomethylene)-2,3-dihydro-1,4-oxazepines, which, upon treatment

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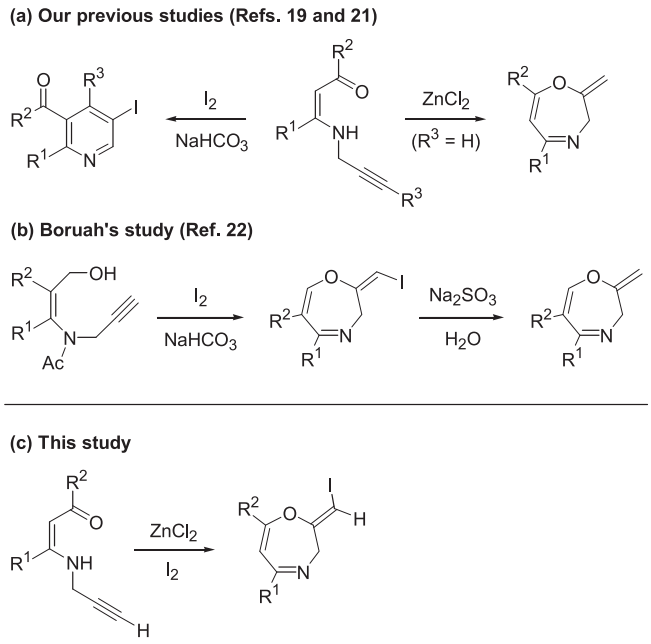


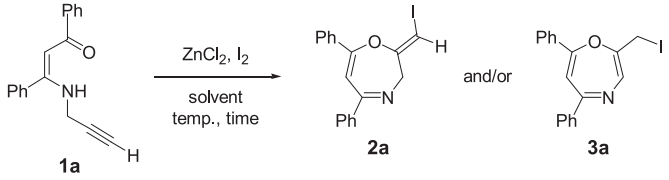
Scheme 1. Approaches for the synthesis of pyridines and 1,4-oxazepines.

with aqueous sodium sulfite, afforded 2-methylene-2,3-dihydro-1,4-oxazepines (Scheme 1b).[22] In this study, iodine-substituted 1,4-oxazepines were not isolated and converted directly into 1,4-oxazepines. We anticipated that if the zinc chloride-mediated cyclization of *N*-propargylic β-enaminones is carried out in the presence of molecular iodine, it would produce iodine-substituted 1,4-oxazepines, which might be useful for elaboration to new molecular entities. Considering the valuable potential of iodine-containing products, we have decided to evaluate the feasibility of this reaction. We have found that when treated with $ZnCl_2$ in the presence of I_2, *N*-propargylic β-enaminones afforded 2-(iodomethylene)-2,3-dihydro-1,4-oxazepines in one-pot manner (Scheme 1c). Herein, we report the initial results of this study.

Initially, we prepared the required *N*-propargylic β-enaminones **1** by the conjugate addition of propargylamine to α,β-alkynic ketones according to our recent study.[21] For this purpose, 10 kinds of *N*-propargylic β-enaminone derivatives **1** were synthesized (For identity of R groups and yields, see ESI).

Next, we investigated electrophilic cyclizations of *N*-propargylic β-enaminones **1** with zinc chloride in the presence of molecular iodine. In order to test the reaction and optimize the conditions, we first studied the cyclization of *N*-propargylic β-enaminone **1a** as depicted in Table 1. In the light of our previous study,[21] reactions were performed in refluxing DCM with varying amounts of $ZnCl_2$ and I_2 (Table 1, Entries 1–7). Interestingly, all reactions afforded 2-(iodomethylene)-2,3-dihydro-1,4-oxazepine **2a**, but in varying yields (23–81%), where the highest yield was obtained by using 2.5 equiv of $ZnCl_2$ and 1.0 equiv of I_2 (Table 1, Entry 7). Notably, higher equivalents of $ZnCl_2$ increased the yield of 1,4-oxazepine **2a**. For instance, when the reaction using 1.0 equiv of I_2 was performed in turn with 1.0, 1.5, 2.0 and 2.5 equivalents of $ZnCl_2$, it produced 1,4-oxazepine **2a** in 52, 62, 76 and 81% yields, respectively (Table 1, Entries 1, 3, 4 and 7). On the other hand, higher equivalents of I_2 decreased the yield of **2a**. For example, when the reaction using 1.0 equiv of $ZnCl_2$ was conducted in turn with 1.0 and 2.0 equivalents of I_2, it yielded 1,4-oxazepine **2a** in 52 and 23% yields, respectively (Table 1, Entries 1 and 2). Similarly, when the reaction using 2.0 equiv of $ZnCl_2$ was carried out in turn with 1.0, 1.5 and 2.0 equivalents of I_2, **2a** was obtained in 76, 56 and 46% yields, respectively (Table 1, Entries 4–6). Clearly, a higher equivalent of I_2 interferes with the reaction and lowers the yield of **2a** considerably. Then, the reaction giving the highest yield was tested at relatively higher temperatures. When the reaction was performed in refluxing THF, ACN, DCE and 1,4-dioxane, 1,4-oxazepine **2a** resulted in 40, 12, 19 and 56% yields, respectively (Table 1, Entries 8–11), indicating that higher temperatures significantly decreased the yield of **2a**. Finally, the same reaction was conducted with $ZnBr_2$ and ZnI_2, instead of $ZnCl_2$, in refluxing DCM, which afforded **2a** in 52 and 39% yields, respectively (Table 1, Entries 12 and 13). Noticeably, $ZnBr_2$ and ZnI_2 were not effective as $ZnCl_2$ was. In summary, the highest yield (81%) of 1,4-oxazepine **2a** was obtained with 2.5 equiv of $ZnCl_2$ and 1.0 equiv of I_2 in DCM

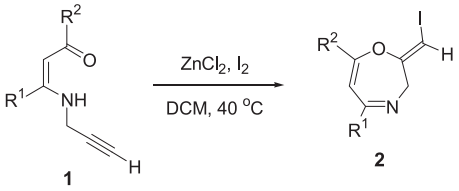
Table 1
Optimization of the reaction conditions for the formation of iodine-substituted 1,4-oxazepines.[a]

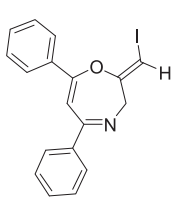


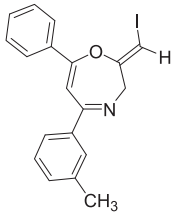
Entry	ZnX_2 (equiv)	I_2 (equiv)	Solvent	Temp. (°C)	Time (h)	Product(s) (% Yield)[b]
1	$ZnCl_2$ (1.0)	1.0	DCM	40	5.0	**2a** (52)
2	$ZnCl_2$ (1.0)	2.0	DCM	40	6.0	**2a** (23)
3	$ZnCl_2$ (1.5)	1.0	DCM	40	7.0	**2a** (62)
4	$ZnCl_2$ (2.0)	1.0	DCM	40	5.0	**2a** (76)
5	$ZnCl_2$ (2.0)	1.5	DCM	40	6.0	**2a** (56)
6	$ZnCl_2$ (2.0)	2.0	DCM	40	5.0	**2a** (46)
7	$ZnCl_2$ (2.5)	1.0	DCM	40	6.0	**2a** (81)
8	$ZnCl_2$ (2.5)	1.0	THF	65	5.0	**2a** (40)
9	$ZnCl_2$ (2.5)	1.0	ACN	81	5.5	**2a** (12)
10	$ZnCl_2$ (2.5)	1.0	DCE	84	3.5	**2a** (19)
11	$ZnCl_2$ (2.5)	1.0	1,4-Dioxane	100	3.5	**2a** (56)
12	$ZnBr_2$ (2.5)	1.0	DCM	40	5.0	**2a** (52)
13	ZnI_2 (2.5)	1.0	DCM	40	5.0	**2a** (39)

[a] Reactions were carried out on a scale of 0.38 mmol of *N*-propargylic β-enaminone **1a** in 10 mL of solvent under argon with the indicated conditions. For work-up and purification, see ESI.
[b] Isolated yield.

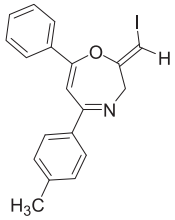
Table 2
Synthesis of 2-(iodomethylene)-2,3-dihydro-1,4-oxazepines **2**.[a,b]



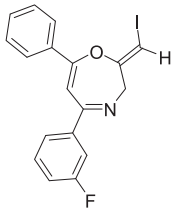

2a (81%)



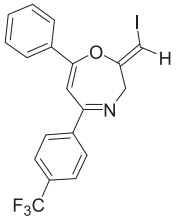
2b (78%)



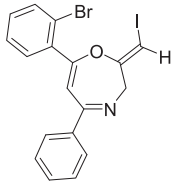
2c (42%)



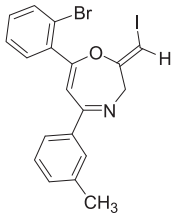
2d (85%)



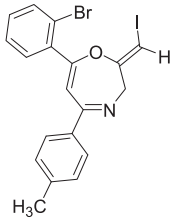
2e (72%)



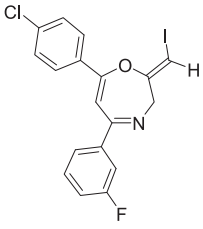
2f (66%)



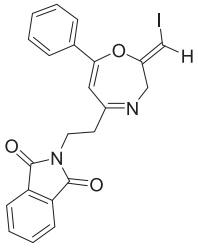
2g (59%)



2h (55%)



2i (68%)



2j (28%)

[a] Reaction conditions: *N*-propargylic β-enaminone **1** (0.38 mmol), $ZnCl_2$ (0.95 mmol), I_2 (0.38 mmol), DCM (10 mL) at 40 °C under argon. For the full procedure including work-up and purification, see ESI.
[b] Isolated yield.

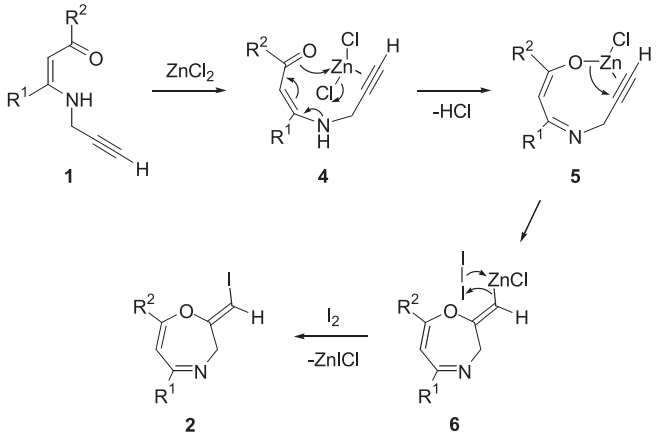
at 40 °C (i.e. with the conditions in Entry 7 of Table 1). The generality of the reaction and the scope of the substrates were studied under these optimized conditions, as depicted in Table 2.

It should be mentioned that 2-(iodomethylene)-2,3-dihydro-1,4-oxazepine **2a** was isolated as single isomer from the reactions in Table 1. The geometry of the double bond in this compound was assigned as *Z* on the basis of NOESY experiment. In the NOESY spectrum of **2a**, an NOE interaction was noticed between exo double bond hydrogen (=C*H*I) and methylene hydrogens (CH_2) on the ring, confirming the *Z* configuration of exo double bond (see ESI for NOESY spectrum and the observed NOE interaction). Furthermore, 2-(iodomethylene)-2,3-dihydro-1,4-oxazepine **2a** was obtained as the only 1,4-oxazepine product from these reactions (Table 1). 2-(Iodomethyl)-1,4-oxazepine **3a**, a fully unsaturated isomer of **2a**, was never observed in these reactions. In the other words, **2a** did not convert into 1,4-oxazepine **3a** under reaction conditions.

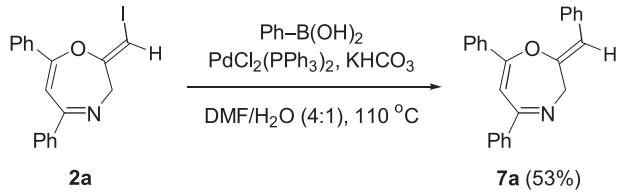
As illustrated in Table 2, a diverse range of *N*-propargylic β-enaminone derivatives **1** was used in these electrophilic cyclizations. In most cases, cyclizations proceeded efficiently and afforded the corresponding 1,4-oxazepines **2** in good to high yields (55–85%). In two cases, 1,4-oxazepines **2** were obtained in low to moderate yields (28–42%). Incorporation of fluorine-containing groups into an organic molecule may lead to improvements in pharmacological properties.[23] So three derivatives of fluorine-bearing 1,4-oxazepines, **2d**, **2e** and **2i**, were synthesized in 68–85% yields (Table 2). In summary, cyclization was found to be general for a variety of *N*-propargylic β-enaminones **1** and tolerated the presence of aryl groups with electron-withdrawing and electron-donating substituents. Besides, during derivatization studies, we did not encounter formation of any of fully unsaturated 1,4-oxazepine derivatives **3**.

Notably, the synthesized 2-(iodomethylene)-2,3-dihydro-1,4-oxazepine derivatives **2** display some characteristic peaks in their ^{1}H and ^{13}C NMR spectra, which relates to the regiochemistry of 1,4-oxazepine ring formation. In the ^{1}H NMR spectra, exo double bond hydrogen (=C*H*I) resonates in the range of 5.82–6.02 ppm as a singlet. The peak of methylene hydrogens (CH_2) comes between 4.50 and 4.94 ppm as a singlet while the double bond hydrogen (=C*H*–) on the ring appears at 5.94–6.40 ppm as a singlet, consistent with the data reported for similar compounds.[21] In the ^{13}C NMR spectra, exo double bond carbon (=*C*HI) is relatively upfield, due to the effect of iodine atom, and observed around 59.4–60.5 ppm. The peak of methylene carbon (*C*H_2) comes between 54.7 and 55.2 ppm while the double bond carbon with one hydrogen (=*C*H–) on the ring appears at 99.4–105.2 ppm, which are in agreement with those reported for similar compounds.[21] In short, the combined NMR data briefly support the indicated regiochemistry of 1,4-oxazepine ring formation.

The mechanism proposed for the formation of iodine-substituted 1,4-oxazepines **2** is shown in Scheme 2. Initially, interaction of zinc chloride with alkyne moiety of **1** generates intermediate **4**, which increases the electrophilicity of alkyne moiety. Subsequently, coordination of carbonyl oxygen to zinc through vinylo-



Scheme 2. Proposed mechanism for the synthesis of 2-(iodomethylene)-2,3-dihydro-1,4-oxazepines.



Scheme 3. Suzuki-Miyaura reaction of iodine-substituted 1,4-oxazepine **2a** with phenylboronic acid.

gous amido-imido tautomerization produces intermediate **5**, which brings the alkyne and carbonyl groups in close proximity. Then intramolecular 7-exo-dig electrophilic cyclization occurs to yield vinyl zinc intermediate **6**. Finally, the reaction of **6** with molecular iodine affords 2-(iodomethylene)-2,3-dihydro-1,4-oxazepines **2** (Scheme 2).

In order to show further applicability of iodine-substituted 1,4-oxazepines **2**, we examined one example of Suzuki-Miyaura reaction between 1,4-oxazepine **2a** and phenylboronic acid (Scheme 3), which was performed under the conditions we employed in our previous study.[20a] This reaction produced the expected 2-benzylidene-2,3-dihydro-1,4-oxazepine **7a** in 53% yield, indicating that iodine-substituted 1,4-oxazepines are stable enough for the further cross-coupling reactions. It should be mentioned that optimization of the reaction conditions could improve the yields of cross-coupling products.

In summary, we established an unprecedented one-pot method for the construction of iodine-substituted 1,4-oxazepine rings from readily available starting materials. When treated with zinc chloride and molecular iodine in refluxing DCM, *N*-propargylic β-enaminones produced 2-(iodomethylene)-2,3-dihydro-1,4-oxazepines in good to high yields through 7-exo-dig electrophilic cyclization and concomitant reaction with molecular iodine. High reaction efficiency and wide reaction scope make this method a fast and straightforward route to diversely substituted iodine-containing 1,4-oxazepines, an important group of biologically and pharmaceutically relevant molecules. More importantly, the presence of the iodine atom in these compounds may create possibilities for building more complex substances through a subsequent transformation of the iodine functionality.

Acknowledgments

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A. Supplementary data

Supplementary data associated with this article can be found, in the online version, at https://doi.org/10.1016/j.tetlet.2018.01.048.

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