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Diastereoselective Synthesis of 3,4-Benzomorphan Derivatives via Tandem [5 + 1]/Hemiaminalization of (2-Aminoaryl)divinyl Ketones

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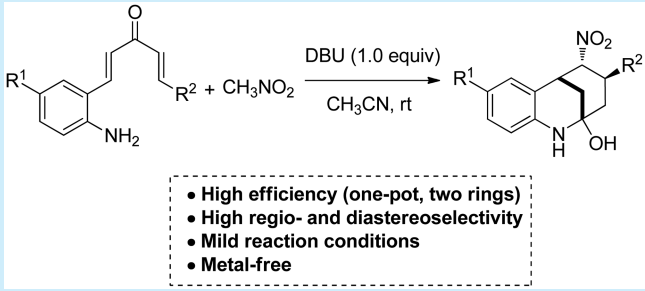
Supporting Information

ABSTRACT: A novel tandem formal [5 + 1]/hemiaminalization reaction based on the readily available (2-aminoaryl)-divinyl ketones and various nucleophiles has been developed. The reaction represents a highly efficient and convenient methodology for the synthesis of 3,4-benzomorphan derivatives with high diastereoselectivity, and three new bonds and two rings are successively formed in one step under mild, metal-free conditions.



The morphan ring system is present in many natural products, such as morphine, and some synthetic compounds with analgesic activity.[1] 3,4-Benzomorphan, a related morphan skeleton with potential biological activity, can also be found in natural products, such as strychnochromine[2] (an unusual alkaloid from *Strychnos gossweileri*) and aspernomine[3] (a cytotoxic anti-insectan metabolite from the sclerotia of *Aspergillus nomius*). Although numerous methods for the synthesis of the morphan structure have been developed,[4,5] only few reports exist in the literature on the construction of 3,4-benzomorphan scaffolds.[2a,6−9] In 2001, Bonjoch's group reported a Pd-mediated intramolecular coupling of aryl iodide with the α-carbon of a carbonyl group to give 3,4-benzomorphan derivatives (Scheme 1, eq 1).[6a] Two years later, a novel rearrangement of 2-(2-bromophenyl)-3-(3-butenyl)-3*H*-indol-3-ol to generate the 3,4-benzomorphan derivative was documented under acidic conditions in McWhorter's work (Scheme 1, eq 2).[7] Very recently, Streuff et al. reported an elegant double-reductive umpolung strategy of quinolones with acrylonitrile to generate the 3,4-benzomorphan derivative in the presence of a titanium(III) catalyst (Scheme 1, eq 3).[8] However, the above methods suffer from drawbacks such as limited substrate scope, lack of readily available precursors, or harsh reaction conditions. Therefore, the development of a new and general methodology for the efficient construction of the 3,4-benzomorphan derivatives from simple starting materials under mild conditions is highly desirable.

Creation of complicated structural molecules from simple substrates,[10] while combining economic aspects,[11,12] constitutes a great challenge in modern organic chemistry. In this context, the one-pot tandem strategy is highly attractive since multiple bonds and stereocenters can be formed in such a single operation without the need to isolate intermediates.[13,14] Recently, we[15−20] and others[21,22] developed a series of new tandem reactions on the basis of divinyl ketones for the efficient construction of structural heterocycles and carbocycles, such as pyrrolizidines,[16] C_2-tethered pyrrole/oxazole pairs,[17] 8-azabicyclo[5.2.1]dec-8-enes,[18] 7-azatetrahydroindoles,[19] and indolizidines.[20] Herein, we report a novel and general tandem [5 + 1]/hemiaminalization reaction of (2-aminoaryl)divinyl ketones with nitroalkanes and activate methylene compounds for the direct and efficient construction of 3,4-benzomorphans in one step under mild conditions (Scheme 1, eq 4). This new general approach allows the formation of two C−C bonds and one C−N bond in a regio- and diastereoselective manner in a single reaction.

In the present study, initially, the reaction of (2-aminoaryl)-divinyl ketone **1a**[23] with nitromethane **2a** was examined carefully to optimize the reaction conditions. As shown in Table 1, the 3,4-benzomorphan derivative **3a** could be obtained in 80% isolated yield from the reaction of (2-aminoaryl)divinyl ketone **1a** (0.30 mmol) with nitromethane **2a** (1.2 equiv, 0.36 mmol) in CH_3CN (5 mL) in the presence of DBU (0.5 equiv, 0.15 mmol) at 25 °C for 9 h, and byproduct quinoline **4a** was produced in 5% yield (Table 1, entry 1). The yield of **3a** was increased to 87% within 8 h by increasing the amount of DBU to 1.0 equiv (0.30 mmol) (Table 1, entry 2). Decreasing the amount of DBU (0.3 or 0.2 equiv) decreased the yield of **3a**. Different bases were also screened, and it was found that NaOH gave a low yield of **3a** with prolonged reaction time (Table 1, entry 5). TMG afforded the desired product **3a** in reduced yield, along with byproduct **4a** in 21% yield (Table 1, entry 6), and when Et_3N was employed, the starting material **1a** was recovered in 99% yield (Table 1, entry 7). The related amidine base DBN showed reaction results similar to those of DBU but with slightly lower yield (Table 1, entry 8). Other solvents, such as THF, DMF, and dichloromethane, gave lower yields of **3a** (Table 1, entries 9−11).

Nitromethane was sometimes found to be unreactive in the double Michael addition with divinyl ketones;[21a,b] however, it is

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Scheme 1. Synthesis of 3,4-Benzomorphan Derivatives

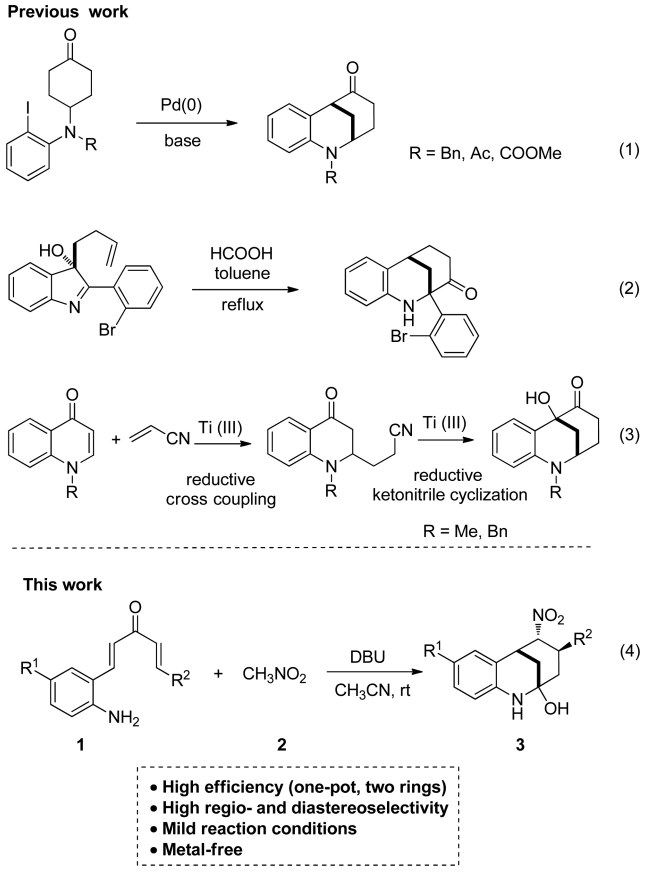
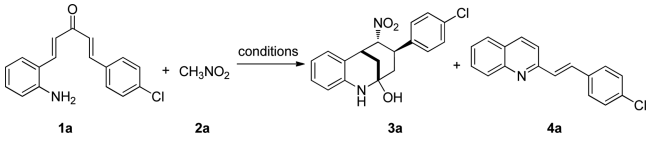


Table 1. Optimization of the Reaction Conditions[a]

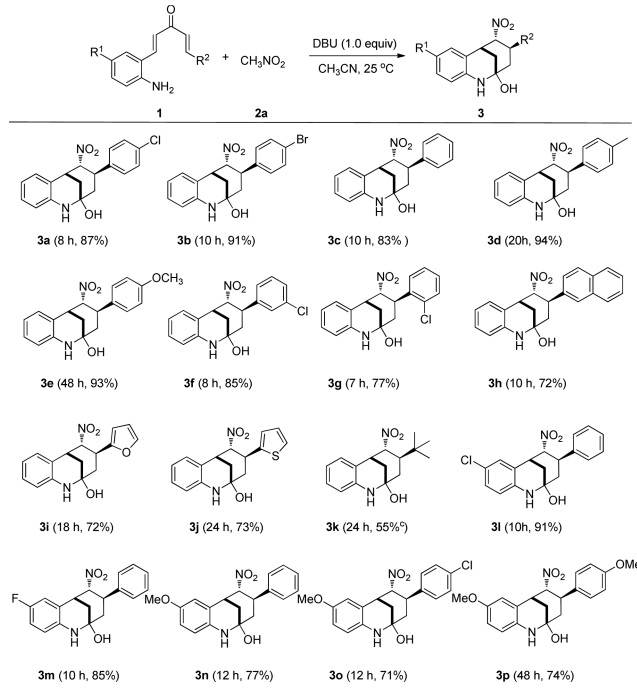


entry	base (equiv)	solvent	temp (°C)	time (h)	**3a** (%)[b]	**4a** (%)[b]
1	DBU (0.5)	CH_3CN	25	9.0	80	5
2	***DBU (1.0)***	***CH_3CN***	***25***	***8.0***	***87***	***trace***
3	DBU (0.3)	CH_3CN	25	9.0	72	10
4[c]	DBU (0.2)	CH_3CN	25	72.0	60	10
5	NaOH (1.0)	CH_3CN	25	36.0	45	5
6	TMG (1.0)	CH_3CN	25	5.5	64	21
7[d]	Et_3N(1.0)	CH_3CN	25	72.0	0	0
8	DBN (1.0)	CH_3CN	25	8.0	78	trace
9[e]	DBU (1.0)	THF	25	72.0	17	20
10	DBU (1.0)	DMF	25	6.0	63	5
11	DBU (1.0)	CH_2Cl_2	25	48.0	20	37

[a]Reactions were carried out with **1a** (0.3 mmol), **2a** (0.36 mmol), and DBU (1 equiv) in solvent (5 mL) at 25 °C. [b]Isolated yields. [c]**1a** was recovered in 23% yield. [d]**1a** was recovered in 99% yield. [e]**1a** was recovered in 52% yield.

an active binucleophile in our tandem reactions. To probe the scope of this transformation, reactions of nitromethane with a range of (2-aminoaryl)divinyl ketones were carried out under the optimal conditions (Table 1, entry 2), and the results are summarized in Scheme 2. It was found that the tandem reaction showed broad tolerance for various R^1 and R^2 groups of

Scheme 2. Synthesis of 3,4-Benzomorphan Derivatives 3[a,b]



[a]Reactions were carried out with **1** (0.3 mmol), **2a** (0.36 mmol), and DBU (1 equiv) in CH_3CN (5 mL) at 25 °C. [b]Isolated yields. [c]Byproduct quinoline **4k** was obtained in 40% yield.

substrates **1**. (2-Aminoaryl)divinyl ketones **1** having electron-deficient (**1a,b** and **1f,g**) and electron-rich (**1d,e**) aryl, phenyl (**1c**), *β*-naphthyl (**1h**), heteroaromatic (**1i,j**), and *tert*-butyl (**1k**) R^2 groups can afford the corresponding 3,4-benzomorphan derivatives **3a−i** in good to high yields with high diastereoselectivity. In addition, (2-aminoaryl)divinyl ketones **1** with both electron-donating and electron-withdrawing R^1 groups gave the polysubstituted 3,4-benzomorphans (**3l−p**) in high yields. It is worth mentioning that the tandem reaction of (2-aminoaryl)-divinyl ketones **1** with nitromethane **2a** proceeded in a highly regio- and diastereoselective manner to set four stereocenters in the 3,4-benzomorphan frameworks on the basis of ^1H and ^{13}C NMR spectroscopy data of products **3** (no diastereoisomers of **3a−p** were detected). The configurations of **3a−p** were assigned according to the X-ray diffraction analysis of **3a**.[24]

The tandem process mentioned above represents a concise and highly efficient methodology for the construction of 3,4-benzomorphan derivatives from simple precursors under very mild conditions. To further test the generality of this new reaction, the reactions of (2-aminoaryl)divinyl ketone **1a** with various readily available nucleophiles **2** were investigated. As shown in Scheme 3, under the aforementioned optimal conditions (Table 1, entry 2), the domino reactions of (2-aminoaryl)divinyl ketone **1a** with nitroethane **2b** and nitropropane **2c** proceeded smoothly, affording the corresponding 3,4-benzomorphan derivatives in high yields with diastereoisomers **5b/b′** and **5c/c′** in a ratio of approximately 1:1. Besides nitroalkane, nucleophilic active methylene compounds, such as ethyl cyanoacetate **2d** or ethyl nitroacetate **2e**, also gave high yield of 3,4-benzomorphan derivatives with diastereoismers **5d/5d′** and **5e/5e′**. Malononitrile **2f** and malonates **2g** and **2h** afforded the 3,4-benzomorphan derivatives **5f−h** in good yields, and no other diastereoismers could be detected. The isomers of **5b/b′−5e/e′** were readily isolated with column chromatog-

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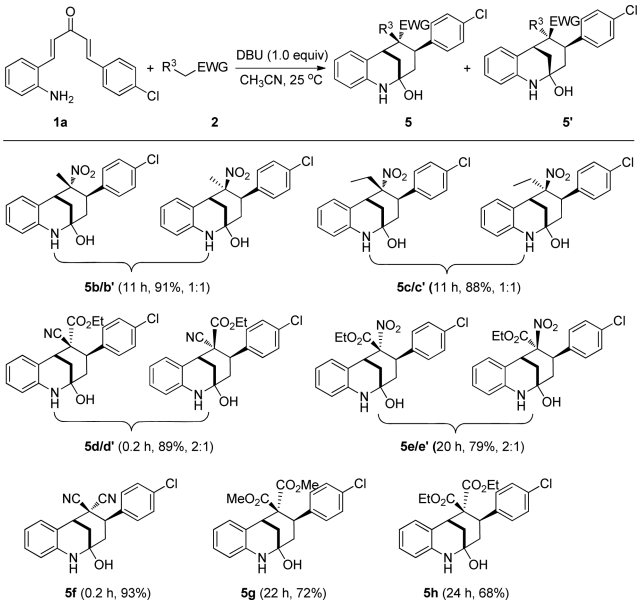
Scheme 3. Synthesis of 3,4-Benzomorphan Derivatives 5[a,b]

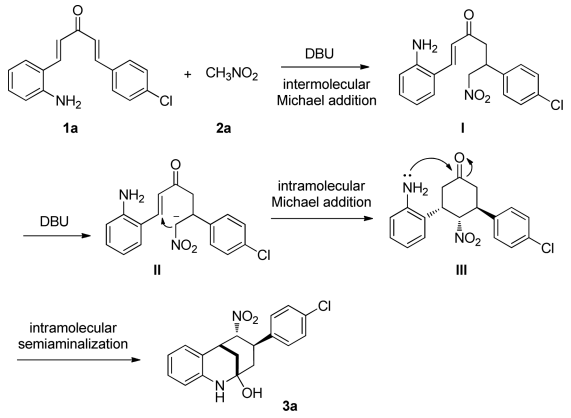


[a]Reactions were carried out with **1a** (0.3 mmol), **2** (0.36 mmol), and DBU (1 equiv) in CH_3CN (5 mL) at 25 °C. [b]Isolated yields.

raphy, and the relative configurations of **5b**/**b′** and **5c**/**c′** were confirmed by NOESY spectroscopy (see Supporting Information). The structure of product **5e** was confirmed by X-ray single-crystal analysis.[24]

With the previous[15−22,25] and present results, a plausible mechanism for the formation of 3,4-benzomorphan derivatives **3** and **5** may involve Scheme 4 (with the transformation of **1a** with

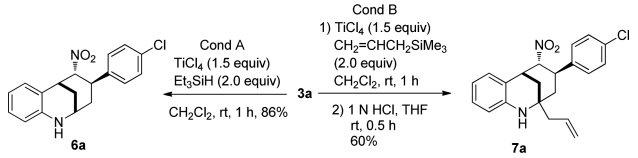
Scheme 4. Proposed Mechanism for Formation of 3,4-Benzomorphan Derivative 3a



2a as an example). The overall process may involve the following steps: (1) a DBU-promoted intermolecular Michael addition of (2-aminoaryl)vinyl ketone **1a** and nitromethane **2a** to provide intermediate **I**; (2) deprotonation of **I** by DBU to form anion intermediate **II**; (3) consecutive intramolecular Michael addition of anion **II** by selectively attacking the less hindered face to generate anti-diastereomer cyclohexanone intermediate **III** in a diastereoselective manner;[15] (4) intramolecular semiaminalization of **III** to furnish 3,4-benzomorphan derivative **3a**. The reason for the diastereoselectivity of this tandem process is not very certain at this stage. Alternatively, thermodynamic control (equilibrium isomerization of one diastereomer into the stable one through the nitronate form of the nitro compounds under basic conditions) is also possible.

To highlight the synthetic potential of this domino process, two possible transformations of the hydroxylation 3,4-benzomorphan derivative **3a** were conducted to form the benzomorphan frameworks, which are found in strychnochromine[2] and aspernomine[3] (Scheme 5). First, the hydroxyl group

Scheme 5. Transformations of 3,4-Benzomorphan Derivatives 3a



of **3a** was readily removed to give the 3,4-benzomorphan derivative **6a** in high yield when **3a** was treated with Et_3SiH (2.0 equiv) and $TiCl_4$ (1.5 equiv). Then, in the presence of allyltrimethylsilane (2.0 equiv) and $TiCl_4$ (1.5 equiv), **3a** was transformed to an allylic 3,4-benzomorphan **7a**, which has a new tetrasubstituted tertiary carbon center.

In conclusion, we have developed a novel domino strategy for the direct and practical synthesis of 3,4-benzomorphan derivatives through (2-aminoaryl)divinyl ketones. The reaction involves a sequential double Michael addition/intramolecular hemiaminalization and allows the diastereoselective construction of 3,4-benzomorphan scaffolds in a single step from easily available substrates in good to high yields under mild metal-free conditions. This strategy shows the highly efficient use of the reactive sites of (2-aminoaryl)divinyl ketones and further expands the synthetic potential of (2-aminoaryl)divinyl ketones in organic synthesis.

■ ASSOCIATED CONTENT

Supporting Information

The Supporting Information is available free of charge on the ACS Publications website at DOI: 10.1021/acs.orglett.5b03506.

Experimental procedures, characterization data for all compounds, and X-ray data of **3a** and **5e** (PDF)

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Notes

The authors declare no competing financial interest.

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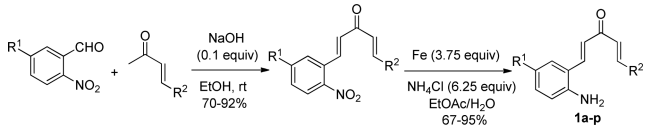
(23) Substrates **1a**−**p** were readily prepared in two steps in high overall yields. See Supporting Information.



(24) CCDC 1402478 (**3a**) and CCDC 1425724 (**5e**) contain the supplementary crystallographic data for this paper. These data can be obtained free of charge from the Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif.

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