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Stereoselective Synthesis of Bicyclic Nitrocyclopropanes by a Radical–Anion Domino Reaction

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Cyclopropanation is regarded as an important transformation in organic synthesis.[1] Synthesis of 3-azabicyclo-[3.1.0]hexane structures has been of interest because compounds with these structures, for example, indolizomycin,[2] trovafloxacin,[3] duocarmycin, and CC-1065[4] show significant biological activity. Preparation of these structures involves low-valent titanium-mediated cyclization (the Kulinkovich reaction), which has recently been explored extensively by de Meijere and others.[5,6] Another method using cyclopropyllithium reagents has also been reported.[7]

Aliphatic nitro compounds are recognized as useful synthetic building blocks because the nitro group has a strong electron-withdrawing property.[8] The α-anions of the nitro compounds are easily oxidized to the corresponding carbon radicals by treatment with ceric ammonium nitrate, silver(I) salts, $K_3Fe(CN)_6$, and $Mn(OAc)_3$.[9] These radicals are frequently used to form new carbon–carbon bonds[10] through radical cyclization.[11] However, to the best of our knowledge, there are currently no successful studies reported to show a direct cyclopropanation process using primary nitro compounds. In this paper, we report a novel intramolecular cyclopropanation from β-nitroamides or β-nitroethers through a higher-order domino reaction to give bicyclic nitrocyclopropanes in a stereoselective manner.

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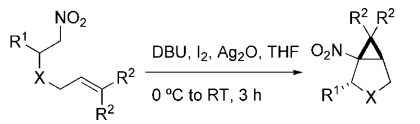
Preparation of the cyclization precursors **1**, **2** and **3** was carried out by the conjugate addition of formamides, alkoxides, and thiolate to nitroalkenes.[12] Treatment of **1a** with Ag_2O and iodine in the presence of DBU resulted in a smooth consumption of **1a**. After the usual workup, bicyclic adduct **4a** was isolated in 72% yield as a mixture of two diastereomers. GC analysis revealed that the diastereomeric ratio was 88:12 (Scheme 1, Table 1, entry 1).

Cyclopropanation from β-nitroamide **1** occurred smoothly to yield **4** in moderate to good yields. For example, treatment of **1b** with DBU, Ag_2O and I_2 resulted in the formation of **4b** in 69% yield (entry 2). The product ratio was found to be 83:17. When R^1 was a primary alkyl group, the diastereoselectivity was slightly lowered (entries 3 and 4).

Table 1. Preparation of bicyclic nitrocyclopropanes **4** and **5** by domino cyclopropanation.

Entry	X	R^1	R^2	Product	Yield [%][a]	dr[b]
1	NCHO	*i*Pr	H	**4a**	72	88:12
2	NCHO	*c*C_6H_{11}	H	**4b**	69	83:17
3	NCHO	Pr	H	**4c**	74	65:35
4	NCHO	C_5H_{11}	H	**4d**	57	66:34
5	NCHO	Ph	H	**4e**	55	90:10
6	NCHO	*i*Pr	Me	**4f**	45	99:1
7	O	*i*Pr	H	**5a**	54	93:7
8	O	*c*C_6H_{11}	H	**5b**	64	92:8
9	O	Pr	H	**5c**	72	96:4
10	O	Ph	H	**5d**	58	97:3
11	O	1-naphthyl	H	**5e**	56	99:1
12	S	Ph	H	**6a**	0	–

[a] Isolated yield. [b] Determined by GC analyses.



1: X = NCHO
2: X = O
3: X = S

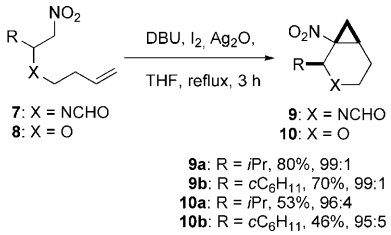
4: X = NCHO
5: X = O
6: X = S

Scheme 1. Dominocyclopropanation of **1**, **2** and **3**.

For example, the same treatment of **1c** resulted in the formation of **4c** in 74% yield, but the product contained two diastereomers in 65:35 ratio (entry 3). The stereoselectivity was improved to 71:29 when the reaction was carried out at 0°C, although the yield of **4c** decreased to 45%. Thus, the stereoselectivity was higher with a secondary alkyl group as R^1 than a primary alkyl group as R^1. Placing a phenyl group at the R^1 position offered good stereoselectivity (entry 5). The prenyl derivative stereoselectively formed **4f**, which was isolated as a single diastereomer in 45% yield (entry 6).

The same treatment for β-nitroether **2** also formed corresponding oxabicyclo[3.1.0]hexane (**5**) in good yields (entries 7–11). Cyclopropanation again occurred in a highly stereoselective manner. For example, β-nitroether **2a** was converted into oxabicyclopropane **5a** in 54% yield with 93:7 stereoselectivity (entry 7). This selectivity was higher than that of the corresponding formation of aza-derivative **4a**. The present high selectivity was also observed in the preparation of **5c** which has a primary alkyl group at the R^1 position (entry 9). A major isomer of **5c** was obtained with 96:4 selectivity, slightly higher compared with the corresponding aza-derivative **4c** (65:35). The highest selectivity was achieved in the formation of **5e**, where mostly a single isomer was formed (entry 11). Treatment of β-nitrosulfide **3a** under the same conditions did not produce the desired **6a** and the corresponding nitroalkene was observed in the reaction mixture (entry 12).

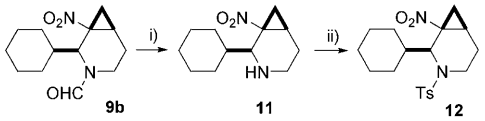
Application of the present reaction to homoallyl amide or ether derivatives also successfully yielded the corresponding bicyclic [4.1.0]heptanes **9** and **10** (Scheme 2). For example,



Scheme 2. Dominocyclopropanation of **7** and **8**.

treatment of a homoallylic amide with Ag_2O/I_2/DBU resulted in the smooth formation of **9a** in 80% yield. Compound **9a** consisted mostly of a single isomer, indicating that the stereoselectivity of the reaction was very high. Thus, the present method is applicable to homoallylic amides and ethers that yielded a one-carbon extension analogue of the bicyclopropanes.

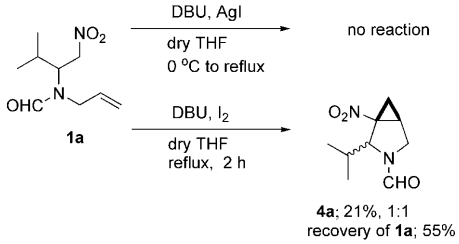
The stereochemistry was determined by X-ray analyses. For example, CH_2Cl_2 solution of rotameric mixture of **4a** gave a crystal of single rotamer, which was used for the X-ray crystallographic analysis.[13] The X-ray data revealed the configuration of **4a**, in which the R^1 group and the cyclopropane ring were located in a *trans* relationship. Compound **5b** also gave nice crystals, X-ray analysis of which unambiguously indicated *trans* configuration.[14] Thus, the stereochemical course of the present cyclopropanation of the ether derivatives was the same as that of the amide derivatives. Compound **9b** was liquid and did not give good crystals for X-ray analysis. We thus attempted to remove the formyl group under acidic conditions (Scheme 3), which was



Scheme 3. Conversion to *N*-tosyl derivative **12**. i) HCl, MeOH, reflux, 12 h, 92%; ii) TsCl, Et_3N, DMAP, CH_2Cl_2, 76%.

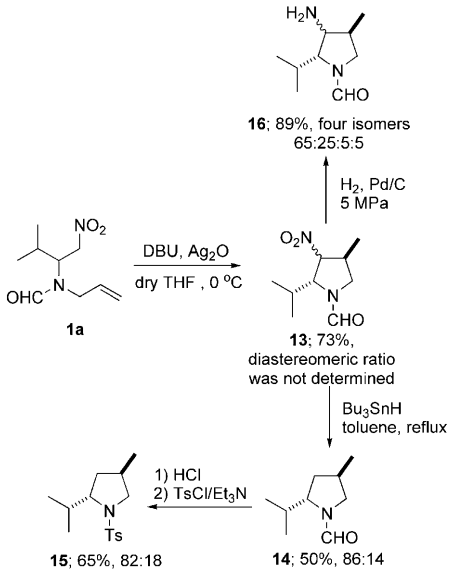
successfully achieved; *N*-H bicyclopiperidine **11** was obtained in 76% yield as a single isomer.[15] No epimerization occurred during the deprotection. Compound **11** was then converted to the corresponding tosylate **12** by treatment with TsCl. Recrystallization of tosylamide **12** afforded good crystals for the X-ray analysis.[16] It should be mentioned that the structure obtained for **12** showed a *cis*-configuration between the cyclohexyl group and the cyclopropane ring, which was opposite to the configuration of **4a**. Fortunately, **10b** gave good crystals for the X-ray analysis that clearly showed a *cis*-configuration.[17] Again the stereochemical courses of homoallylic amides and ethers derivatives are the same. Thus, the present domino cyclopropanation occurred in a *trans*-selective manner in the formation of aza- and oxabicyclo[3.1.0]hexane and a *cis*-selective manner in the formation of aza- and oxabicyclo[4.1.0]heptane. The stereochemistry of the reaction depended on the length of the tether. We attempted to prepared seven-membered ring by use of the one-carbon extended precursor of **7**, but no bicyclo[5.1.0]octane derivatives were observed in the reaction mixture.

To clarify the reaction mechanism, we carried out the reaction using **1a** under various conditions (Scheme 4). First, we attempted the reaction with AgI instead of Ag_2O and I_2, but no reaction occurred. Then the reaction in the absence of Ag_2O was examined. Compound **1a** was consumed slowly and remained after 2 h under refluxing conditions. The reaction became sluggish and the yield of **4a** dropped to 21%. It should be mentioned that after isolation **4a** contained the two isomers in an almost 1:1 ratio along with recovery of the starting material. Thus, the stereoselectivity was totally lost, when the reaction was performed without Ag_2O.



Scheme 4. Reactions of **1a** under various conditions.

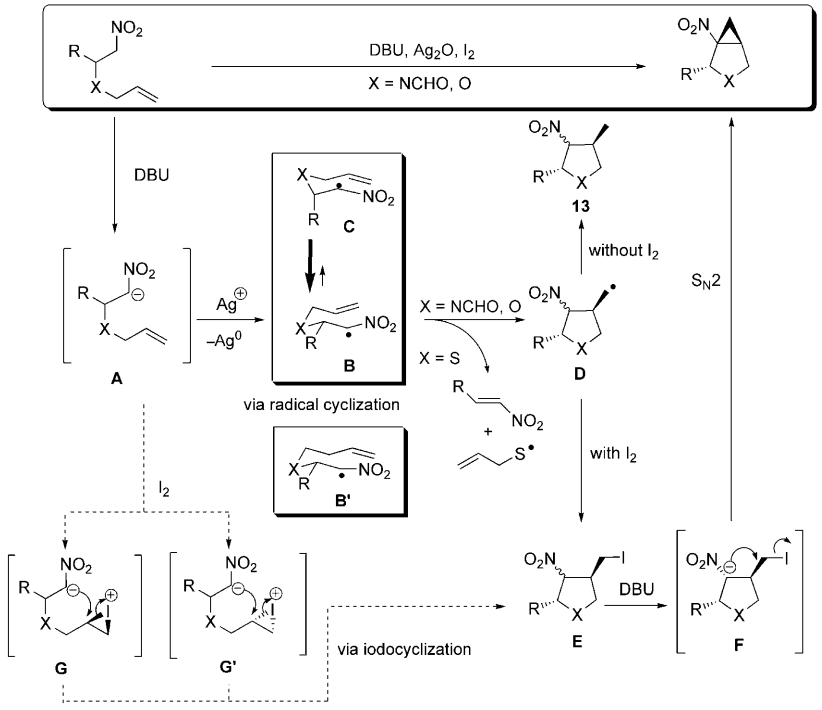
The reaction without iodine also afforded an interesting result (Scheme 5). Treatment of **1a** with Ag_2O in the absence of I_2 did not give **4a**; instead, substituted pyrrolidine



Scheme 5. Reactions of **1a** in the absence of I_2.

13 was isolated in 73% yield as a mixture of diastereomers. Due to overlapping signals, the GC analysis of **13** showed only two peaks. To confirm the diastereomeric ratio, the nitro group was reduced to the amine under hydrogenation conditions. The corresponding amine **16** was prepared in 89% yield. The present diastereomeric mixture of **16** gave well-resolved GC peaks, which allowed determination of the diastereomeric ratio of **16** as 65:25:5:5. To reduce the number of diastereomers, we then removed the nitro group by treatment with Bu_3SnH. The removal took place smoothly and **14** was isolated in 50% yield. As expected, it contained only two diastereomers in the ratio 86:14. Removal of the formyl group under acidic conditions followed by *N*-tosylation produced **15** in 65% yield. Compound **15** contained two diastereomers in the ratio 82:18, which was slightly different from the ratio found for **14**. The two isomers were separated by careful HPLC treatment and the major isomer of **15** (major-**15**) was recrystallized. X-ray crystallographic analysis for major-**15** unambiguously indicated 2,4-*trans* configuration.[18] It should be noted that the *trans* relationship in major-**15** was the same as in **4a**.

Based on these results, we assumed the following reaction mechanism (Scheme 6): The starting compound **1** or **2** is deprotonated by DBU to give a nitronate anion **A**, which immediately undergoes single-electron oxidation by Ag_2O to generate the α-nitro radical **B**.[9f] If β-nitrosulfide **3** is used in the reaction, the generated radical **B** readily loses a sulfenyl radical through β-elimination to afford nitroalkene so that no cyclization takes place. The allylic or homoallylic double bond is located in a good position for radical cyclization by the α-nitro radical generated from **1** and **2** that forms cyclized radical **D**. During the cyclization, the two conformations, **B** and **C**, are proposed. Conformer **B** is preferred to conformer **C** because of the steric bias of the R group. Radical **D** is likely to be trapped by iodine to give iodomethylpyrrolidine **E**. If the reaction is carried out without iodine, the intermediate radical **D** abstracts hydrogen from the solvent to give monocyclic pyrrolidine **13**. This is strongly supported by the observation that both compounds **13** and **4a** have the same 2,4-*trans* configuration and the formation of these compounds occurs with almost the same level of diastereoselectivity. Iodine in intermediate **E**, being a good leaving group, is displaced through intramolecular S_N2 attack by the α-nitroanion to form bicyclo[3.1.0]hexanes **4** and **5** in a *trans*-selective manner. Thus, the diastereoselectivity of the present domino cyclopropanation is determined in the radical cyclization step. The opposite *cis*-selectivity during the formation of bicyclo[4.1.0]heptanes **9** and **10** from the homoalylic derivatives should also be explained by

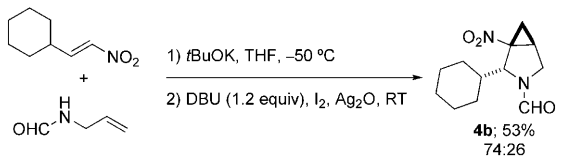


Scheme 6. Plausible reaction mechanism.

the assumption that the radical cyclization takes place through an analogous conformation **B′**.

If Ag_2O is absent in the reaction mixture, bicyclic propane can be formed through an alternative route. Thus, iodine attacks the allylic double bond to form the *epi*-iodonium ion **G**. In this route, the rate of the reaction can become very slow. No stereoselectivity should be expected because there are no substituents close to the double bond to provide a proper steric bias. As a result, two diastereomeric *epi*-iodonium ions **G** and **G′** should be generated in a 1:1 ratio. The iodomethylpyrrolidine **E** intermediate should be generated by an intramolecular nucleophilic attack by the nitronate anion to give intermediate **E**, but this should be formed as a 1:1 diastereomeric mixture through the process. This assumption is in good agreement with the observation that the reaction without Ag_2O occurred very slowly, and a 1:1 diastereomeric mixture of **4a** was obtained in 21% yield along with recovery of the starting material **1a**. No reaction takes place in the absence of Ag_2O and I_2. For example, AgI does not oxidize the nitronate anion **A** so no reaction occurred. Hence, we conclude that the present reaction is a novel higher-order domino process in which oxidation of the nitronate, radical cyclization and intramolecular S_N2 reaction takes place in a one-pot process in a highly stereoselective manner. The combination use of Ag_2O and iodine is a key factor for the smooth formation of bicyclopropanes **4, 5, 9** and **10**.

Finally, we examined a much higher-order domino reaction starting from nitroalkene and formamide. Treatment of a mixture of formamide and nitroalkene in the presence of a base smoothly formed the nitronate anion of **1b**, which underwent the present domino cyclization process by addition of Ag_2O/I_2 to give **4b** in 53% yield (Scheme 7). Addition of DBU improved the yield of **4b**. Thus, a carbon–nitrogen bond and two carbon–carbon bonds were formed in a one-pot process through five domino reactions: a conjugate addition, oxidation, radical cyclization, trapping by iodine, and an intramolecular S_N2 reaction. The reaction showed almost the same level of stereoselectivity as the reaction using **1b**.



Scheme 7. One-pot conversion to **4b**.

In conclusion, we found a new reaction to prepare bicyclic cyclopropanes in one step from the conjugate adducts of formamides or alcohols with nitroalkenes. The present method involves a higher-order domino process in which three reactions occur sequentially in a one-pot process. With this reaction, primary nitroalkanes are regarded as an equivalent to α-nitrocarbene. This method will be useful for the preparation of heterocyclic bicyclo[3.1.0]hexanes and bicyclo[4.1.0]heptanes which are of interest for their unique bioactivity. Further investigation of this reaction is now underway in our laboratory.

Experimental Section

See Supporting Information for additional details.

Preparation of *N*-formyl-2-isopropyl-1-nitro-3-azabicyclo[3.1.0]hexane (4a): Ag_2O (0.5562 g, 2.40 mmol) and I_2 (0.6091 g, 2.40 mmol) were added to a solution of **1a** (0.2403 g, 1.20 mmol) and DBU (0.21 mL, 1.44 mmol) in anhydrous THF (5 mL) at 0 °C, and the mixture was allowed to stir for 3 h. The reaction mixture was filtered and the filtrate was concentrated in vacuo. The obtained crude product was purified by flash chromatography (silica gel, hexane/ethyl acetate 2:1 then 1:1) to give **4a** as oil (0.1700 g, 72%). GC analysis revealed that the diastereomeric ratio of **4a** was 88:12. Pure *trans*-**4a** contained two rotational isomers at room temperature (ratio ca. 2:1), and the ratio was about 2:1. ^{1}H NMR (270 MHz, $CDCl_3$, TMS): δ = 0.86 (d, ^3J = 6.9 Hz, 3H, major rotamer), 0.87 (d, ^3J = 6.9 Hz, 3H, minor rotamer), 1.04 (d, ^3J = 6.9 Hz, 3H, major rotamer), 1.05 (d, ^3J = 6.9 Hz, 3H, minor rotamer), 1.19 (t, ^3J = 5.9 Hz, 1H, minor rotamer), 1.24 (t, ^3J = 5.9 Hz, 1H, major rotamer), 1.82 (m, 1H, minor rotamer), 1.86 (dd, ^3J = 5.9, 8.9 Hz, 1H, major rotamer), 2.58–2.74 (m, 1H), 2.74–2.90 (m, 1H), 3.45 (dd, ^3J = 14.0, 12.4 Hz, 1H, major rotamer), 3.52 (d, ^3J = 5.9 Hz, 1H, minor rotamer), 3.82 (dd, ^3J = 3.0, 9.9 Hz, 1H, minor rotamer), 3.97 (d, ^3J = 11.9 Hz, 1H, major rotamer), 4.24 (d, ^3J = 2.0 Hz, 1H, major rotamer), 4.84 (d, ^3J = 3.0 Hz, 1H, minor rotamer), 8.13 ppm (s, 1H); ^{13}C NMR (67.5 MHz, $CDCl_3$) δ = 16.8, 17.2, 20.1, 20.7, 20.8, 21.6, 24.5, 24.8, 29.0, 29.6, 45.2, 48.8, 59.8, 63.7, 162.5, 163.2 ppm; IR ($CHCl_3$): $\tilde{\nu}$ = 1373, 1530, 1672 cm^{-1}; elemental analysis calcd (%) for $C_9H_{14}N_2O_3$; C 54.54, H 7.12, N 14.13; found: C 54.49, H 7.03, N 14.10.

Preparation of *N*-formyl-2-isopropyl-4-methyl-3-nitropyrrolidine (13): Ag_2O (0.463 g, 2.0 mmol) was added to a solution of **1a** (0.200 g, 1.0 mmol) and DBU (0.18 mL, 1.2 mmol) in anhydrous THF (15 mL) at 0 °C, and the mixture was allowed to stir for 2 h. The reaction mixture was filtered and the filtrate was concentrated in vacuo. The obtained crude product was purified by flash chromatography (silica gel, hexane/ethyl acetate 1:1) to give **13** as pale yellow oil (0.1456 g, 73%). GC analysis showed two peaks: ratio 67:33. ^{1}H NMR (270 MHz, $CDCl_3$): δ = 0.71–1.20 (m, 9H), 1.65–2.20 (m, 1H), 2.53–2.83 (m, 1H), 2.90–3.45 (m, 1H), 3.50–4.38 (m, 2H), 4.38–4.56 (m, 0.5H), 4.80 (t, ^3J = 5.7 Hz, 1H), 8.07 (s, 0.2H), 8.17 (s, 0.6H), 8.24 ppm (s, 0.2H); ^{13}C NMR (67.5 MHz, $CDCl_3$): δ = 12.1, 12.3, 14.9, 15.8, 16.8, 17.4, 17.5, 18.2, 18.4, 18.8, 18.9, 28.6,29.5, 30.3, 32.0, 32.2, 35.2, 35.6, 40.1, 40.6, 48.7, 48.9, 51.2, 52.5, 64.8, 65.7, 67.4, 67.9, 89.5, 90.1, 92.7, 92.9, 160.7, 161.1, 162.0, 162.1 ppm; IR ($CHCl_3$): $\tilde{\nu}$ = 1375, 1551, 1674, 2880, 2940, 2967 cm^{-1}; HRMS (EI$^+$): *m*/*z*: calcd for $C_9H_{16}N_2O_3$: 200.1161; found: 200.1164 $[M]^+$.

Keywords: cyclopropanes · domino reactions · oxidation · radical cyclization · stereoselective reaction

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[13] CCDC 713684 (**4a**) contains 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.
[14] CCDC 739560 (**5b**) contains 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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[16] CCDC 713685 (**12**) contains 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.
[17] CCDC 739561 (**10b**) contains 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.
[18] CCDC 713686 (major-**15**) contains 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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