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Tetrahydroquinolines via Stereospecific [3 + 3]-Annulation of Donor−Acceptor Cyclopropanes with Nitrosoarenes

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Supporting Information

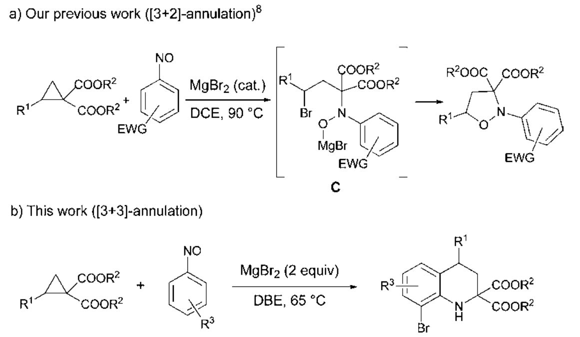
ABSTRACT: A stereospecific [3 + 3]-annulation of donor−acceptor cyclopropanes with nitrosoarenes under the influence of $MgBr_2$ as a stoichiometric Lewis acid and reagent offers a novel approach to various structurally diverse C-8-brominated tetrahydroquinolines. In these cascades C−C, C−N, and C−Br bonds are formed. The reactions are easy to conduct and proceed under mild conditions, and the products can readily be further functionalized, rendering the method highly valuable.

Nitrogen-containing heterocycles occur as key structural elements in various natural products and biologically relevant compounds such as alkaloids, nucleic acids, and antibiotics. Tetrahydroquinoline (THQ) derivatives, in particular, have attracted great attention as they are the key subunits found in various complex molecules that show interesting biological activity.[1] In addition, the tetrahydroquinoline structural motif has found applications in materials science.[2] Consequently, the development of novel synthetic methodology for the construction of this particular core structure starting from simple components is highly desirable. Among the various known synthetic routes to heterocycles, the use of donor−acceptor (DA) cyclopropanes as three-carbon synthons has emerged as a valuable approach to access heterocycles. Importantly, DA cyclopropanes are readily obtained and show versatile reactivity.[3] For example, DA cyclopropanes engage in [3 + 3]-annulations with 1,3-dipoles. These annulations occur via a stepwise reaction where the DA cyclopropane is ring-opened in situ in the presence of a Lewis acid into a reactive 1,3-zwitterion that can then undergo cycloadditions.[4,5] Although such reactions have been studied for more than a decade, the scope with respect to the 1,3-dipoles used in the [3 + 3]-annulations is limited. Notably, preparation of tetrahydroquinoline derivatives via this approach using a formal C−C−N-type 1,3-zwitterionic synthon has not been well explored to date.[6]

Within the framework of our program devoted to the development of stereoselective cycloaddition reactions of nitrosoarenes,[7] we recently disclosed a $MgBr_2$-catalyzed stereospecific formal [3 + 2]-cycloaddition with DA cyclopropanes to afford isoxazolidines with high yields and complete regioselectivity (Scheme 1a).[8] Exploration of the substrate scope of this reaction revealed that electron-deficient nitrosoarenes are the most suitable substrates, and we postulated that cyclization occurs via magnesiated hydroxylamine **C**.

Based on these findings, we became interested in investigating the reaction of nitrosoarenes bearing an electron-donating group at the arene with DA cyclopropanes.

Scheme 1. Reactivity of Nitrosoarenes toward D−A Cyclopropanes in the Presence of $MgBr_2$



We assumed that the weak N−O bond[9] of intermediate **C** might be cleaved, assisted by a Lewis acid and the electron-donating substituent, to generate a reactive iminoquinone-type species which in turn should undergo interesting follow-up chemistry. Along these lines, we herein disclose [3 + 3]-annulation of DA cyclopropanes with nitrosoarenes bearing mostly electron-donating substituents to give C-8-brominated tetrahydroquinolines (Scheme 1b).

We commenced our studies by examining the reaction between diethyl 2-phenylcyclopropane-1,1-dicarboxylate (**1a**) and easily prepared *p*-methoxynitrosobenzene (**2a**) as the model substrates. Lewis acid, solvent, and reaction temperature were systematically varied (Table 1). With 1.5 equiv of $MgBr_2$ in dichloroethane (DCE) at 90 °C, the brominated tetrahydroquinoline **3a** was formed in 19% yield (Table 1, entry 1). Notably, the corresponding isoxazolidine previously obtained in the presence of a catalytic amount of $MgBr_2$ in combination with electron-poor nitrosoarenes was not identified. The yield of **3a** could be increased to 43% by

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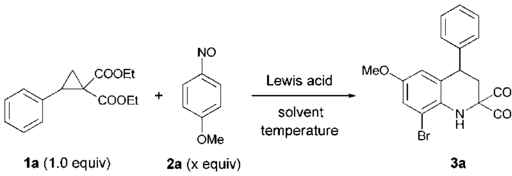
Table 1. Reaction Optimization between 1a and 2a



entry	2a (equiv)	Lewis acid	LA (equiv)	T (°C)	solvent	yield[a] (%)
1	1.5	$MgBr_2$	1.5	90	DCE	19
2	1.5	$MgBr_2$	2.0	90	DCE	43
3	1.5	$MgBr_2$	3.0	90	DCE	48
4	1.5	$MgBr_2$	2.0	90	CH_2Cl_2	25
5	1.5	$MgBr_2$	2.0	90	CH_2Br_2	42
6	1.5	$MgBr_2$	2.0	90	DBE	52
7	2.0	$MgBr_2$	3.0	65	DBE	54
8	2.5	$MgBr_2$	3.5	65	DBE	46
9[b]	2.0	$MgBr_2$	2.0	65	DBE	59
10[b,c]	**2.0**	**$MgBr_2$**	**2.0**	**65**	**DBE**	**65**
11[b,d]	2.0	$MgBr_2$	2.0	65	DBE	44
12[b,c]	2.0	$MgBr_2$	2.0	20	DBE	48

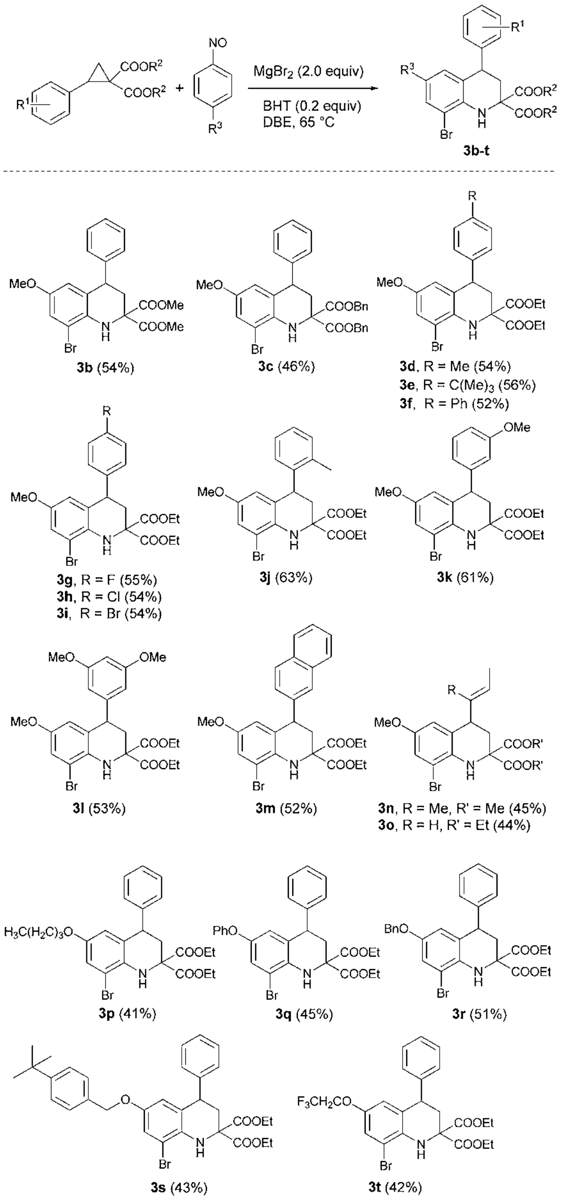
[a] Isolated yield. [b] **2a** was added over 30 min by a syringe pump. [c] 0.2 equiv of BHT was used. [d] 1.0 equiv of BHT was used. DCE = 1,2-dichloroethane. DBE = 1,2-dibromoethane.

using 2.0 equiv of $MgBr_2$ (Table 1, entry 2). However, a further increase in the stoichiometry of $MgBr_2$ led to little improvement of the yield (Table 1, entry 3). We continued the studies by screening different Lewis acids. The use of MgI_2, $MgCl_2$, $NiCl_2$, $Cu(OTf)_2$, $CuBr_2$, $ZnBr_2$, $FeBr_3$, and $InBr_3$ did not deliver the [3 + 3]-annulation product **3a** (not shown in Table 1).

Different solvents were tested next, keeping $MgBr_2$ as the Lewis acid, and it was found that halogenated solvents such as dichloromethane and dibromomethane gave **3a** in 25% and 42% yield (Table 1, entries 4 and 5). In toluene, CCl_4, THF, DMSO, or acetone, reaction to yield **3a** failed (not shown in Table 1). A further enhancement in the product yield was achieved upon changing the solvent to 1,2-dibromoethane (52%) (Table 1, entry 6). Increasing the amount of $MgBr_2$ did not improve the result (Table 1, entries 7 and 8). With the observation that a significant quantity of **1a** remained unreacted after full consumption of the nitrosoarene, the amount of **2a** was increased. Addition of 2.0 equiv of **2a** over 30 min via a syringe pump at 65 °C provided complete conversion of **1a**, affording the targeted [3 + 3]-annulation product **3a** in 59% isolated yield (Table 1, entry 9). The reaction was further optimized by studying various additives (the full screening table is listed in the Supporting Information). Pleasingly, we found that yield was increased to 65% by using 0.2 equiv of 3,5-di-*tert*-butyl-4-hydroxytoluene (BHT) as an additive (Table 1, entry 10). The exact role of BHT on the cascade is currently not understood. Increasing the amount of BHT (Table 1, entry 11) and lowering the reaction temperature (Table 1, entry 12) resulted in decreased yields.

With the optimized reaction conditions in hand (Table 1, entry 10), we next investigated the scope and limitations of the method by using various racemic DA cyclopropanes in combination with *p*-methoxynitrosobenzene (**2a**) as the reaction partner. The results are presented in Scheme 2.

Scheme 2. Scope of the [3 + 3]-Annulation Reaction



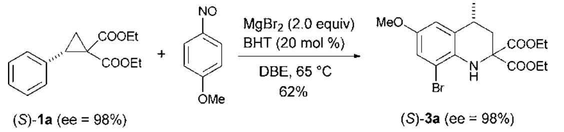
The activating ester functionality in the DA cyclopropane was varied, and the bismethyl ester **1b** gave **3b** in 54% yield. A slightly lower yield was achieved with the bisbenzyl ester (see **3c**). To investigate electronic effects exerted by the donor substituent in the DA cyclopropane component, the phenyl group in the parent **1a** was replaced by substituted aryl groups. Neither electron-donating nor electron-withdrawing substituents at the *para*-position of the phenyl ring affected the efficiency of this reaction, and the corresponding products **3d–i** were obtained in 52–56% yield. Moreover, steric effects seem to be less important as *o*-tolylcyclopropane **1j** gave **3j** in 63% yield. As expected on the basis of these results, a substituent at the *meta*-position of the phenyl group and also disubstitution at the phenyl ring do not influence reaction efficiency to a large extent, and tetrahydroquinolines **3k** and **3l** were obtained in 61% and 53% yield, respectively. The naphthyl congener **1m** worked well to provide **3m** in 52% yield. The structure of **3m** was unambiguously confirmed by X-ray analysis (see the Supporting Information).

We also tested nonaromatic donor substituents at the DA cyclopropane moiety and found that alkenylcyclopropanes can be used, although slightly lower yields were achieved and products **3n** and **3o** were isolated in 45% and 44% yield. To examine the viability of this methodology for larger scale synthesis, the [3 + 3]-annulation reaction of **1a** with **2a** was repeated on a 1.0 mmol scale to give **3a** in 58% yield.

Next, the scope of the reaction was examined with respect to the nitrosoarene component. We found oxygen-containing electron-donating groups at the *para*-position of the nitrosobenzene to be tolerated. Alkoxy- and aryloxy-substituted nitrosoarenes reacted with **1a** to give the corresponding tetrahydroquinolines **3p−s** in moderate to good yields. Notably, even with the electron-poor *p*-(2,2,2-trifluoroethoxy)-nitrosobenzene, the annulation occurred smoothly and **3t** was isolated in 42% yield. However, in the reaction with **1a** under optimized conditions nitrosoarenes bearing nitrogen containing electron-donating groups at the *para* position did not afford the corresponding [3 + 3]-annulation products.

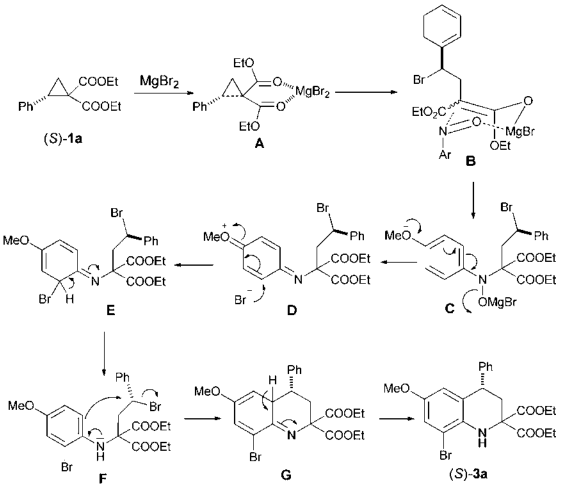
To gain insight into the stereochemical course of the reaction, we investigated the stereospecificity of the [3 + 3]-annulation using highly enantioenriched phenyl cyclopropane (*S*)-**1a** (98% ee).[10] Pleasingly, *p*-methoxynitrosobenzene reacted with (*S*)-**1a** with complete stereospecificity to provide (*S*)-**3a** in 62% yield (Scheme 3) (98% ee as analyzed by HPLC, see the Supporting Information). The absolute configuration was unambiguously assigned by X-ray analysis showing that the [3 + 3]-annulation occurred with net retention at the stereogenic center of the starting DA cyclopropane (see the Supporting Information).

Scheme 3. Testing Stereospecificity with Enantioenriched (*S*)-1a and 2a



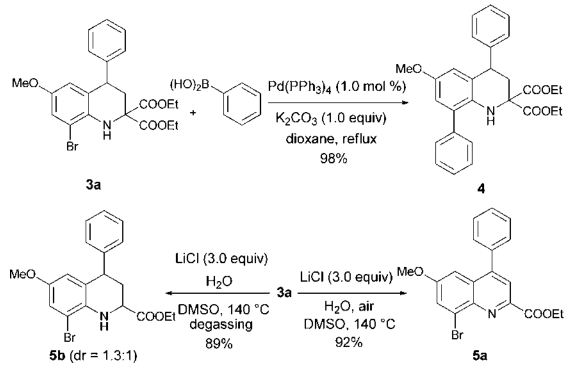
On the basis of the above observation, our previous work, and literature reports, a plausible mechanism for the [3 + 3]-annulation reaction is suggested in Scheme 4. $MgBr_2$ first interacts with the donor−acceptor cyclopropane (*S*)-**1a** providing the activated $MgBr_2$-complexed cyclopropane **A**. The bromide anion then stereoselectively opens the cyclopropane ring at the benzylic position to generate the corresponding enolate. This enolate further reacts with the *p*-methoxynitrosobenzene in which the oxygen atom of the nitroso compound is bound to the oxophilic Mg atom via a six-membered chair transition state **B** to give magnesiated hydroxylamine **C**.[8] The electron-donating effect of the *p*-methoxy group now plays an important role in cleaving the N−O bond-forming reactive iminoquinone-type species **D**, which is directly intercepted by the bromide anion to form **E**. Tautomerization leads to intermediate **F**, which reacts stereoselectively in an intramolecular Friedel−Crafts-type alkylation to **G**. Final tautomerization eventually affords product (*S*)-**3a** with net retention at the stereogenic center with respect to the starting cyclopropane (*S*)-**1a**. Importantly, when *p*-methoxynitrosobenzene was allowed to react with diethyl 2-(4-(trifluoromethyl)phenyl)cyclopropane-1,1-dicarboxylate, the corresponding intermediate **F** could be isolated (see the Supporting Information), which supports our mechanistic picture.

Scheme 4. Plausible Mechanism



To demonstrate the synthetic value of the method, we investigated follow-up chemistry using **3a** as a substrate. Tetrahydroquinoline **3a** engaged in a Suzuki−Miyaura coupling with phenylboronic acid to provide **4** in 98% yield (Scheme 5).

Scheme 5. Follow-up Chemistry



When **3a** was subjected to Krapcho decarboxylation conditions, we noted that in the presence of air decarboxylation is accompanied by oxidation to give quinoline **5a** in 92% overall yield. However, in the absence of oxygen, the tetrahydroquinoline **5b** was obtained in 89% yield as a 1.3:1 diastereoisomeric mixture.

In conclusion, we have reported a [3 + 3]-annulation involving DA cyclopropanes and nitrosoarenes in the presence of a stoichiometric amount of $MgBr_2$ to provide valuable C-8 brominated tetrahydroquinolines. Further examination of the process with an enantiometrically enriched cyclopropane showed that the product tetrahydroquinolines are formed with complete stereospecificity. Notably, since the synthesis of highly enantioenriched DA cyclopropanes is well-established, our novel process offers access to highly enantioenriched tetrahydroquinolines in a one pot cascade. The Lewis acid has a dual role in this sequence: First, it acts as a "classical" Lewis acid for activation of the DA cyclopropane; second, it acts as a reagent (bromide anion donor) in the arene bromination step. In these cascades, a C−C bond along with one C−N bond and one C−Br bond are formed. Reactions are easy to conduct and occur under mild conditions in a short time (2−4 h). The synthetic value of the products has been demonstrated by high-yielding follow-up reactions.

■ ASSOCIATED CONTENT

Ⓢ Supporting Information

The Supporting Information is available free of charge on the ACS Publications website at DOI: 10.1021/acs.orglett.6b01302.

> Experimental details, characterization data for the products, and supplementary crystallographic data (CCDC 1475167 (**3m**), 1475168 [(*S*)-**3a**)] (PDF)
>
> X-ray data for **3m** and (*S*)-**3a** (CIF)

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Notes

The authors declare no competing financial interest.

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