RESEARCH PAPER

A Benzotriazole Enabled Lewis Acid Strategy for the Controlled Monofunctionalization of Donor–Acceptor Cyclopropanes

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ABSTRACT

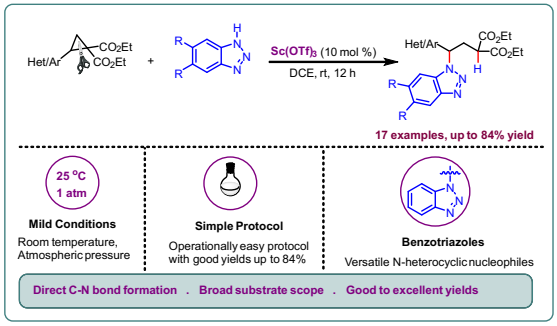
Donor–Acceptor (D–A) cyclopropanes serve as useful synthetic precursors that make them suitable substrates for Lewis acid-mediated ring-opening transformations. Herein, we report a $Sc(OTf)_3$-catalyzed ring-opening monofunctionalization of D–A cyclopropanes with benzotriazoles as N-nucleophiles. The reaction proceeds under mild conditions in DCE at room temperature, providing γ-benzotriazolyl malonate derivatives through C–N bond formation. The protocol exhibits good functional-group tolerance across a range of substituted D–A cyclopropanes and benzotriazole derivatives, affording 17 examples in moderate to good yields up to 84%. The operationally simple protocol provides a convenient approach to the synthesis of benzotriazole-functionalized malonate derivatives from readily accessible activated cyclopropanes.

Keywords: Donor–Acceptor cyclopropanes; benzotriazoles; Lewis acid catalysis; ring-opening reaction; C–N bond formation.

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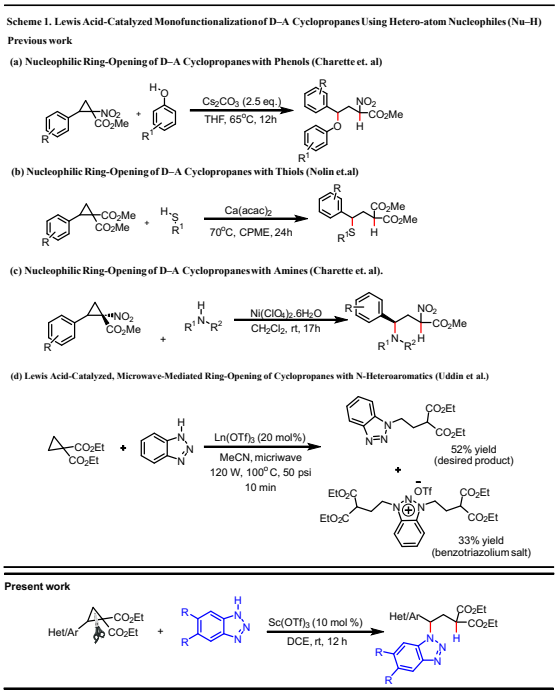
Introduction

Donor–Acceptor (D–A) cyclopropanes possess a highly polarized, strained three-membered ring with adjacent donor and acceptor substituents, that have found broad applications in organic synthesis.[1,2,3] Due to the high ring strain (≈27.5 kcal/mol), D–A cyclopropanes readily undergo ring opening reactions upon Lewis acid activation under mild conditions.[4] This process generates a 1,3-dipolar intermediate[5], which has been extensively explored in various [3 + n] annulation reactions, including formal [3 + 2], [3 + 3], and [3 + 4] cycloadditions.[1,6,7] Beyond annulation chemistry, Lewis acid activation of D-A cyclopropanes also facilitates nucleophile-induced ring-opening reactions, providing access to a library of acyclic functionalized products. Heteroatom nucleophiles of type Nu–H such as amines,[8] azides,[9] alcohols,[10] and thiols,[11] as well as carbon nucleophiles,[12,13] have been employed in these transformations. Nucleophilic attack on the activated cyclopropane generates a 1,3-zwitterionic intermediate that can undergo protonation to furnish monofunctionalized products or react with an external electrophile to afford 1,3-bifunctionalized products. These three component reactions can be studied by the attack of a nucleophile to the strained D–A cyclopropane followed by the interception with an electrophile.[14,15,16] This reactivity has enabled a variety of multicomponent functionalization strategies of D–A cyclopropanes. The reactivity of D–A cyclopropane has also been very well explored by the insertion of the three-membered ring into a heteroatom-heteroatom bonds under Lewis acid catalysis.[17,18]

Over the past two decades, the D–A cyclopropanes have been widely employed in nucleophile-induced monofunctionalization

reactions, through a nucleophile triggered ring opening reaction under Lewis acid conditions. Charette et al. have reported the nucleophilic ring-opening monofunctionalization of activated cyclopropanes with alcohols in the presence of Cs_2CO_3 base **(Scheme 1a).**[10(a)] Nolin and co-workers have demonstrated the $Ca(acac)_2$ -catalyzed synthesis of γ-sulfanyl malonates through the addition of thiols to the D–A cyclopropanes **(Scheme 1b).**[11(a)] In the same year, Charette and co-workers have explored the insertion of D–A cyclopropane into amine nucleophiles to generate 1,3-functionalized molecules. They have demonstrated that 1-nitrocyclopropanecarboxylates could undergo Lewis acid-catalyzed ring opening with a range of amine nucleophiles, while retaining the enantiomeric purity of the resulting products (**Scheme 1c**).[8(b)]

Uddin et al. previously reported the microwave-assisted $Ln(OTf)_3$-catalyzed ring-opening addition of N-heteroaromatics to activated cyclopropane derivatives.[8(c)] Although benzotriazole was demonstrated as a competent nucleophile as a single derivative, the reported transformation required 20 mol% $Ln(OTf)_3$, microwave irradiation at 100 °C and 50 psi in MeCN, and afforded the benzotriazole-derived product in 52% yield together with 33% of a benzotriazolium salt (**Scheme 1d**). The requirement for specialized microwave equipment, elevated temperature and pressure, together with the formation of a significant amount of quaternized benzotriazolium salt byproduct, limits the operational simplicity and efficiency of this approach. These relatively demanding reaction conditions and the formation of a substantial amount of quaternized byproduct highlight the scope for developing a simpler and milder approach to the direct incorporation of benzotriazole into donor-acceptor cyclopropanes. More recently, Biju and co-workers reported a benzotriazole-triggered three-component 1,3-aminofunctionalization of D–A cyclopropanes under Lewis acid catalysis.[16] In contrast to these approaches, we sought to develop a direct two-component ring-opening monofunctionalization of D–A cyclopropanes with benzotriazoles under mild and operationally simple conditions, without the involvement of an additional electrophilic reaction partner.

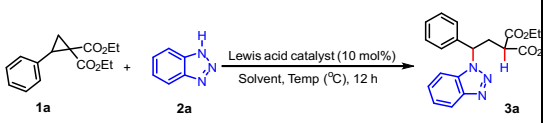


Herein, we report a $Sc(OTf)_3$-catalyzed ring-opening monofunctionalization of D–A cyclopropanes with benzotriazoles in DCE at room temperature, affording γ-benzotriazolyl malonate derivatives in moderate to good yields. The protocol operates without microwave irradiation or elevated pressure and accommodates a range of substituted D–A cyclopropanes and benzotriazole derivatives. Interestingly, the benzotriazole compounds are known to be a potent pharmacophore in medicinal chemistry, which are effective as anticancer, antifungal, antibacterial, antitubercular and antiviral agents.[19]

Results and discussion

The reaction conditions for the insertion[20] of the D–A cyclopropane **1a** into N–H bond of benzotriazole **2a** using Lewis acid catalyst were systematically optimized by varying the effects of the substrate stoichiometry, Lewis acid identity, catalyst loading, solvent, and temperature. The reaction between diethyl 2-phenylcyclopropane-1,1-dicarboxylate **1a** (0.25 mmol) and 1*H*-benzo[*d*][1,2,3]triazole **2a** (0.375 mmol) were selected as the model substrates for the ring-opening monofunctionalization of D–A cyclopropane, and the formation of product **3a** was monitored under various reaction conditions (**Table 1**).

Table 1. Optimization of the $Sc(OTf)_3$-catalyzed monofunctionalization of D–A cyclopropane **1a** with benzotriazole **2a**.[a]



Entry	1a (equiv.)	2a (equiv.)	Lewis acid	Solvent	Temp	Yield

			catalyst	**(3mL)**	**(°C)**	**(%)[b]**
1	1.0	1.0	$Sc(OTf)_3$	DCE	25	69
2	1.5	1.0	$Sc(OTf)_3$	DCE	25	75
3	**1.0**	**1.5**	**$Sc(OTf)_3$**	**DCE**	**25**	**83**
4	1.0	1.5	$Yb(OTf)_3$	DCE	25	72
5	1.0	1.5	$Sn(OTf)_2$	DCE	25	<20
6[c]	1.0	1.5	$Sc(OTf)_3$	DCE	25	65
7	1.0	1.5	$Sc(OTf)_3$	THF	25	69
8	1.0	1.5	$Sc(OTf)_3$	DCM	25	62
9	1.0	1.5	$Sc(OTf)_3$	$CHCl_3$	25	60
10	1.0	1.5	$Sc(OTf)_3$	DCE	0	34
11	1.0	1.5	$Sc(OTf)_3$	DCE	50	53
12	1.0	1.5	$Sc(OTf)_3$	DCE	85	37
13	1.0	1.5	None	DCE	25	<5

General reaction conditions: [a]All the reactions were carried out on a 0.25 mmol scale using diethyl 2-phenylcyclopropane-1,1-dicarboxylate **1a** (0.25 mmol, 1.0 equiv), 1*H*-benzo[*d*][1,2,3]triazole **2a** (0.375 mmol, 1.5 equiv), Lewis acid catalyst (10 mol%), solvent (3.0 mL), 25 °C, 12 h, under N_2. [b]Isolated yields are reported; [c]$Sc(OTf)_3$ (5 mol%).

Initially, the reaction was performed using equimolar amounts of diethyl 2-phenylcyclopropane-1,1-dicarboxylate **1a** and 1*H*-benzo[*d*][1,2,3]triazole **2a** in the presence of 10 mol% $Sc(OTf)_3$ in DCE at 25 °C for 12 h, furnishing **3a** in 69% yield (entry 1). When the loading of cyclopropane **1a** was increased to 1.5 equiv., the yield increased to 75% (entry 2). In contrast, employing benzotriazole **2a** in 1.5 equiv. gave **3a** in 83% yield (entry 3). Accordingly, the use of 1.5 equiv. of **2a** was selected for subsequent optimization studies.

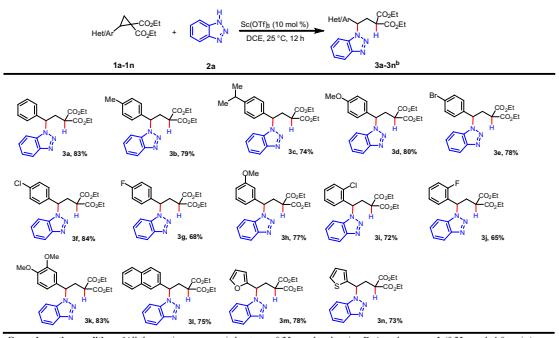
With the substrate stoichiometry optimized, the influence of the Lewis acid was next examined. Under otherwise identical conditions, $Yb(OTf)_3$ and $Sn(OTf)_2$ afforded the desired product in 72% and <20% yields, respectively, both lower than that obtained with $Sc(OTf)_3$ (entries 4 and 5). A subsequent reduction of the $Sc(OTf)_3$ loading from 10 to 5 mol% led to a diminished yield of 65% (entry 6), indicating that 10 mol% catalyst loading was preferable. The solvent effect was subsequently investigated under the optimized substrate stoichiometry and catalyst loading. Replacing DCE with THF, DCM, or $CHCl_3$ resulted in lower yields of 69%, 62%, and 60%, respectively (entries 7-9). In comparison, DCE afforded the highest yield, and was therefore retained as the solvent for subsequent studies. The influence of temperature was subsequently investigated, with diminished yields observed at both 0 and 50 °C (entries 10 and 11), while increasing the temperature to 85 °C further decreased the yield to 37% (entry 12). Finally, to assess the necessity of the Lewis acid catalyst, the reaction was performed in the absence of $Sc(OTf)_3$ under otherwise identical conditions. Only a negligible amount of product **3a** (<5%) was detected (entry 13), demonstrating the essential role of Lewis acid activation in promoting the ring-opening process.

Having established the optimal reaction conditions, we subsequently examined the scope and generality of the reaction using a range of D–A cyclopropane and benzotriazole substrates (**Schemes 2 and 3**). We have tested benzotriazole **2a** with a range of aromatic and heteroaromatic D–A cyclopropanes containing both electron-donating and electron-withdrawing substituents at the donor site. (**Scheme 2**).

The D–A cyclopropane with aromatic ring at the donor position, bearing either electron-donating and electron-withdrawing substituents smoothly reacted with **2a** to afford the corresponding γ-benzotriazolyl malonate derivatives **3a-3n** in moderate to good yields (65-84%). The reaction tolerated both electron-donating and electron-withdrawing substituents, although variations in yield were observed depending on the nature and position of the substituent. *Para*-substituted substrates bearing electron-donating groups, such as methyl, isopropyl, and methoxy, provided **3b-3d** in good yields. *Para*-halogen substitution was also well tolerated furnishing **3e-3g** in moderate to good yields. These results indicate that both electron-rich and electron-deficient para-substituted aromatic substrates are compatible with the transformation. The influence of substituent position was further examined with *meta*- and *ortho*-substituted substrates. The *meta*-methoxy-substituted cyclopropane gave **3h** in 77% yield, whereas the *ortho*-chloro- and *ortho*-fluoro-substituted substrates afforded **3i** and **3j** in 72% and 65% yields, respectively. The disubstituted 3,4-dimethoxy-substituted substrate was also compatible with the reaction conditions, delivering the corresponding γ-benzotriazolyl malonate product **3k** in 83% yield. The scope was further extended beyond simple substituted phenyl derivatives. A naphthyl-substituted D–A cyclopropane underwent the reaction smoothly to afford **3l** in 75% yield, demonstrating the compatibility of a larger polyaromatic substituent. Heteroaryl-substituted cyclopropanes were also

tolerated, the 2-furyl and 2-thienyl derivatives furnished the corresponding products **3m** and **3n** in 78% and 73% yields, respectively. Overall, the results demonstrate that the developed protocol accommodates diverse aromatic, polyaromatic, and heteroaromatic D–A cyclopropanes, with moderate to good yields across the examined substrate range.

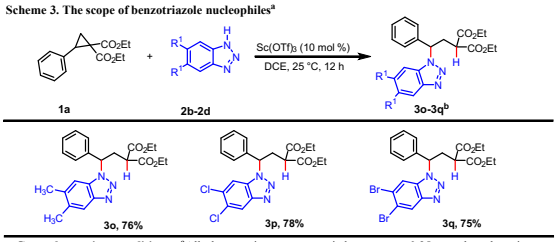
Scheme 2. The substrate scope of D-A Cyclopropanes[a]



General reaction conditions: [a]All the reactions were carried out on a 0.25 mmol scale using D–A cyclopropane **1** (0.25 mmol, 1.0 equiv.), benzotriazole **2** (0.375 mmol, 1.5 equiv.), Lewis acid catalyst $Sc(OTf)_3$ (10 mol%), in 3.0 mL DCE at 25 °C for 12 h, [b]Isolated yields are reported.

Next, we explored the scope of the benzotriazole nucleophiles **2b-2d** under the optimized reaction conditions. The different benzotriazole substrates were compatible with the developed protocol, furnishing the corresponding products **3o–3q** in 75-78% yields (**Scheme 3**).

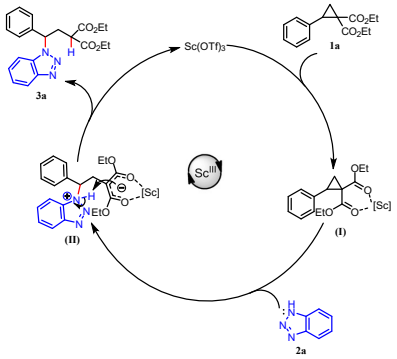
Scheme 3. The scope of benzotriazole nucleophiles[a]



General reaction conditions: [a]All the reactions were carried out on a 0.25 mmol scale using D–A cyclopropane **1** (0.25 mmol, 1.0 equiv.), benzotriazole **2** (0.375 mmol, 1.5 equiv.), Lewis acid catalyst $Sc(OTf)_3$ (10 mol%), in 3.0 mL DCE at 25 °C for 12 h, [b]Isolated yields are reported.

A plausible mechanism involves initial coordination of $Sc(OTf)_3$ to the ester carbonyls **(I)**, followed by nucleophilic ring opening of the activated D–A cyclopropane **1a** by benzotriazole **2a** to generate a zwitterionic intermediate **(II)**. Subsequent proton transfer affords the γ-benzotriazolyl malonate product with regeneration of the Lewis acid catalyst (**Scheme 4**).

Scheme 4. Proposed mechanism for the monofunctionalization of D–A cyclopropane with benzo



Conclusion

In conclusion, we have developed a $Sc(OTf)_3$-catalyzed ring-opening monofunctionalization of D–A cyclopropanes with benzotriazoles under mild conditions. The protocol provides access to a range of γ-benzotriazolyl malonate derivatives in moderate to good yields and accommodates variation in both the D–A cyclopropane and benzotriazole components. The reaction proceeds in DCE at room temperature with the use of a readily accessible Lewis acid catalyst without microwave irradiation or elevated pressure, providing an operationally simple approach to these benzotriazole-functionalized products. The methodology also supports further research and can be utilized to explore biological evaluation of the γ-benzotriazolyl malonate derivatives.

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Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this article.

Supplementary data

Experimental procedures, synthesis and characterization data for compounds, and copies of ^{1}H and ^{13}C NMR spectra for products.

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