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$Yb(OTf)_3$-Catalyzed Construction of Indole Derivatives through Formal [3+3] Cycloaddition of 1,1-Vinylidenecyclopropanediesters with Nitrones

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The 1,3-dipolar cycloaddition reaction is a highly efficient and powerful method for the assembly of a variety of biologically important heterocycles in a convergent fashion from simple precursors.[1] Of particular interest to us is the Lewis acid catalyzed cycloaddition reaction of highly strained small rings with nitrones **2** for the construction of N-containing heterocycles. Thus far, the groups of Brandi and de Meijere have systematically studied the 1,3-dipolar cycloaddition of methylenecyclopropanes (MCPs) with nitrones,[2] affording the exocyclic type of [3+2] addition products in good yields. Later on, Young and Kerr[3] reported a [3+3] cycloaddition of donor–acceptor (D-A) type of cyclopropane diesters with nitrones, catalyzed by $Yb(OTf)_3$ under mild conditions. Recently, the group of Wang[4] has reported the first example of $Yb(OTf)_3$-promoted distal [3+3] cycloaddition of MCP diesters with nitrone 1,3-dipoles to give the N,O-heterocyclic products in good yields. On the basis of above examples, it is quite clear that the release of highly strained energy associated with the ring opening of a cyclopropane moiety in an organic molecule might lead to multiple transformations, the selectivity of which depends on the nature and pattern of the substituents on the cyclopropane ring as well as the adjacent positions.[5] Inspired by these results, we anticipated that the geminal installation of two electron-withdrawing groups (EWGs) at the cyclopropane ring connected with an allene moiety, for example, vinylidenecyclopropanes, might further activate the carbon–carbon bond cleavage and lead to high regioselectivity of the ring-opening reaction, providing novel cycloadducts.[6] Thus far, the chemistry of vinylidenecyclopropanes (VDCPs) has been extensively explored in our laboratory and in other groups during the last several years. Many novel intramolecular rearrangements or cycloaddition reactions with carbon–carbon or carbon–heteroatom multiple bonds, such as imines, aldehydes, nitriles, and α,β-unsaturated ketones, took place smoothly either upon heating and photoirradiation[7] or catalyzed/mediated by Lewis and Brønsted acids, giving the corresponding [2+2], [3+2], or [3+3] cycloadducts in moderate to good yields under mild conditions.[8–10] To our delight, we found that in the $Yb(OTf)_3$-catalyzed reactions of vinylidenecyclopropanes containing two ester groups (VDCP diesters) **1**[11] with nitrones **2**, the proximal [3+3] cycloaddition products **3** were exclusively obtained with a high regioselectivity. To the best of our knowledge, this is the first example of Lewis acid catalyzed proximal [3+3] cycloaddition of VDCPs with 1,3-dipoles.

Initial experiments were carried out by using VDCP diester **1a** and nitrone **2a** as the substrates in the presence of a series of Lewis acids to determine the optimal reaction conditions (please see the Supporting Information for the details). The typical results are summarized in Table 1. We found that the reaction could be finished within 10 min at room temperature and the corresponding product **3aa** was obtained in the yield of 32% using **1a** (1.0 equiv) and nitrone **2a** (3.0 equiv) catalyzed by $Yb(OTf)_3$ (20 mol%) in THF (Table 1, entry 1). Solvent screening showed that the highest yield was achieved in toluene (Table 1, entries 1–4) and decreasing the amount of nitrone **2a** resulted in a little higher yield of 53% (Table 1, entry 5). Several commonly used Lewis acids were also tested and, of these, $Yb(OTf)_3$ afforded better results than $Zn(OTf)_2$, $Sc(OTf)_3$, $BF_3{\cdot}Et_2O$, or $Sn(OTf)_2$ etc. (Table 1, entries 6–9, see the Supporting Information for the details). Decreasing the amount of $Yb(OTf)_3$ to 10 mol%, the similar result was obtained without the distinct diminishment of the yield (Table 1, entry 10). The highest yield of 72% was achieved when the reaction was conducted in toluene at room temperature using a excess amount of VDCP **1a** (1.5 equiv) and nitrone **2a**

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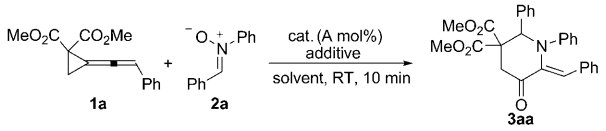
Supporting information for this article is available on the WWW under http://dx.doi.org/10.1002/chem.200902510. It contains ^{13}C and ^{1}H NMR spectroscopic and analytic data for **1**, **3** and **4** and X-ray crystal data of **3ae** and **4aa**.

Table 1. Optimization of Lewis acid catalyzed reaction conditions of VDCP-diester **1a** with nitrone **2a**.[a]



	1a:**2a**	Cat. ([mol%])	Additive	Solvent	Yield **3aa** [%][b]
1	1.0:3.0	$Yb(OTf)_3$ (20)	–	THF	32
2	1.0:3.0	$Yb(OTf)_3$ (20)	–	CH_2Cl_2	29
3	1.0:3.0	$Yb(OTf)_3$ (20)	–	toluene	50
4	1.0:3.0	$Yb(OTf)_3$ (20)	–	MeOH	decomposed
5	1.0:1.0	$Yb(OTf)_3$ (20)	–	toluene	53
6	1.0:1.0	$Zn(OTf)_2$ (20)	–	toluene	30
7	1.0:1.0	$Sc(OTf)_3$ (20)	–	toluene	25
8	1.0:1.0	$BF_3{\cdot}Et_2O$ (20)	–	toluene	NR[c]
9	1.0:1.0	$Sn(OTf)_2$ (20)	–	toluene	NR[c]
10	1.0:1.0	$Yb(OTf)_3$ (10)	–	toluene	51
11	1.5:1.0	$Yb(OTf)_3$ (10)	4 Å MS	toluene	72

[a] All the reactions were carried out with VDCP diester **1a** and nitrone **2a** in solvent catalyzed by Lewis acid at RT. [b] Isolated yield. [c] NR = no reaction.

(1.0 equiv) catalyzed by $Yb(OTf)_3$ (10 mol%) with the addition of 4 Å molecular sieves (50 mg for 0.2 mmol of nitrone) (Table 1, entry 11).

With these optimal reaction conditions identified, the scope of the reaction was investigated further with respect to various VDCP diesters **1** and nitrones **2**. The results are summarized in Table 2. As for VDCP diesters **1a** and **1c** with an electron-deficient substituent (Cl) on the aryl ring, the reactions proceeded smoothly to give **3** in moderate to good yields under optimized conditions, irrespective of the electronic nature of the aryl rings on the C,N-diarylnitrones (Table 2, entries 1–4, 7, and 8). However, VDCPs **1b** with a moderately electron-rich methyl group on the aryl ring and **1d** with an aliphatic *n*Pr group connected to the carbon atom of the allene moiety led to low yields under the same reaction conditions. To improve yields, we optimized the reaction conditions again with respect to substrates **1b** and **1d**. After variation of reaction temperature and time, the moderate yields of the cycloadducts were obtained (Table 2, entries 5, 6, and 9).

The structure of the compound **3ae** was established by single-crystal X-ray diffraction (Figure 1; CIF data are presented in the Supporting Information).[12]

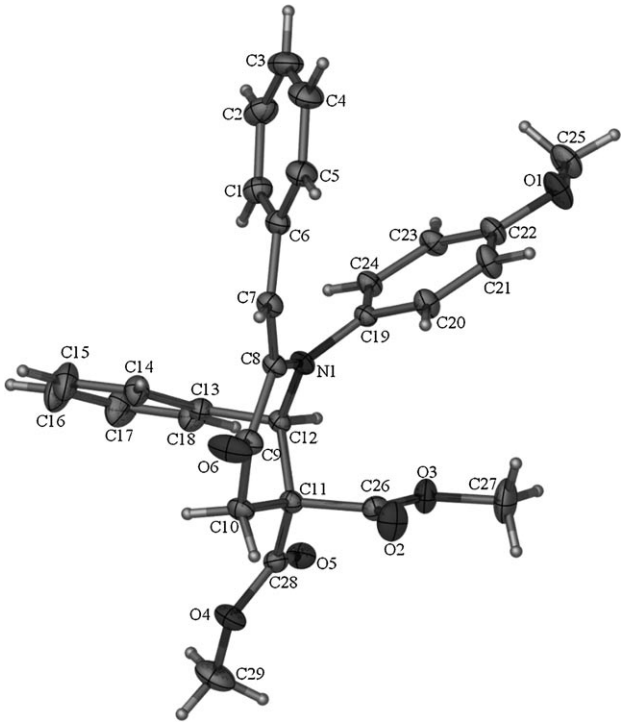


Figure 1. X-ray crystal structure of **3ae**.

Table 2. $Yb(OTf)_3$-catalyzed reactions of VDCP diesters **1** with nitrones **2**.[a]

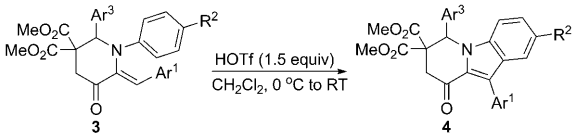
		Ar^1		Ar^2	Ar^3	Product, Yield [%][b]
1	**1a**	Ph	**2b**	Ph	*p*-ClC_6H_4	**3ab**, 66
2	**1a**	Ph	**2c**	Ph	*p*-$MeOC_6H_4$	**3ac**, 62
3	**1a**	Ph	**2d**	*p*-BrC_6H_4	Ph	**3ad**, 68
4	**1a**	Ph	**2e**	*p*-$MeOC_6H_4$	Ph	**3ae**, 60
5[c]	**1b**	*p*-MeC_6H_4	**2a**	Ph	Ph	**3ba**, 61
6[d]	**1b**	*p*-MeC_6H_4	**2e**	*p*-$MeOC_6H_4$	Ph	**3be**, 60
7	**1c**	*p*-ClC_6H_4	**2a**	Ph	Ph	**3ca**, 66
8	**1c**	*p*-ClC_6H_4	**2d**	*p*-BrC_6H_4	Ph	**3cd**, 65
9[e]	**1d**	*n*Pr	**2a**	Ph	Ph	**3da**, 42

[a] All reactions were conducted by adding VDCP diesters **1** (0.3 mmol, 1.5 equiv) in toluene (6.0 mL) to the solution of nitrones **2** (0.2 mmol, 1.0 equiv), $Yb(OTf)_3$ (10 mol%) and 4 Å MS (50 mg) in toluene (2.0 mL) at RT unless otherwise specified. [b] Isolated yield. [c] The reaction was conducted at 0 °C. [d] The reaction was conducted at −20 °C. [e] The reaction mixtures were heated at 80 °C for 20 min.

Interestingly, all the products **3** from the formal [3+3] cycloaddition of VDCP diesters and nitrones could undergo ring closure to afford the indole derivatives **4** in good yields under mild reaction conditions in the presence of trifluoromethanesulfonic acid CF_3SO_3H (HOTf) (1.5 equiv) (Table 3), thus opening up a new route to synthesize indole derivatives. The structure of the compound **4aa** was characterized by spectroscopic analysis and confirmed by single-crystal X-ray diffraction (Figure 2; CIF data are given in the Supporting Information).[12]

Although the mechanism of this reaction is still under investigation, a plausible mechanism involving a formal [3+3] cycloaddition pathway is proposed in Scheme 1 to rationalize the reaction. The strained bonds in the cyclopropane ring have long been considered to have a significant π character[13] and in 1,1-cyclopropane diesters, this bond can be polarized and further weakened by coordination of a Lewis acid to one or both of the ester moieties.[3] Thus, initially VDCP diester **1a** is activated by $Yb(OTf)_3$, forming a com-

Table 3. Translation of compounds **3** mediated by HOTf to indole derivatives **4**.[a]



		Ar^1	R^2	Ar^3	Product, Yield [%][b]
1	**3aa**	Ph	H	Ph	**4aa**, 83
2	**3ab**	Ph	H	*p*-ClC_6H_4	**4ab**, 82
3	**3ac**	Ph	H	*p*-$MeOC_6H_4$	**4ac**, 77
4	**3ad**	Ph	Br	Ph	**4ad**, 80
5	**3ae**	Ph	MeO	Ph	**4ae**, 85
6	**3ba**	*p*-MeC_6H_4	H	Ph	**4ba**, 81
7	**3be**	*p*-MeC_6H_4	MeO	Ph	**4be**, 82
8	**3ca**	*p*-ClC_6H_4	H	Ph	**4ca**, 80
9	**3cd**	*p*-ClC_6H_4	Br	Ph	**4cd**, 79

[a] All reactions were conducted with the substrates **3** (0.2 mmol) in CH_2Cl_2 (5.0 mL) cooled in ice water for a while before adding HOTf (1.5 equiv) and then warmed to RT to continue the reaction. [b] Isolated yield.

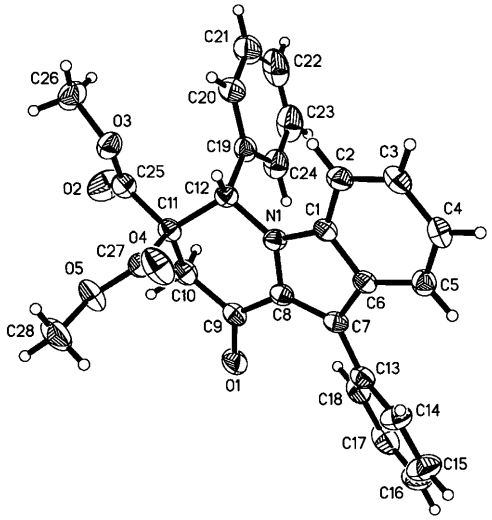
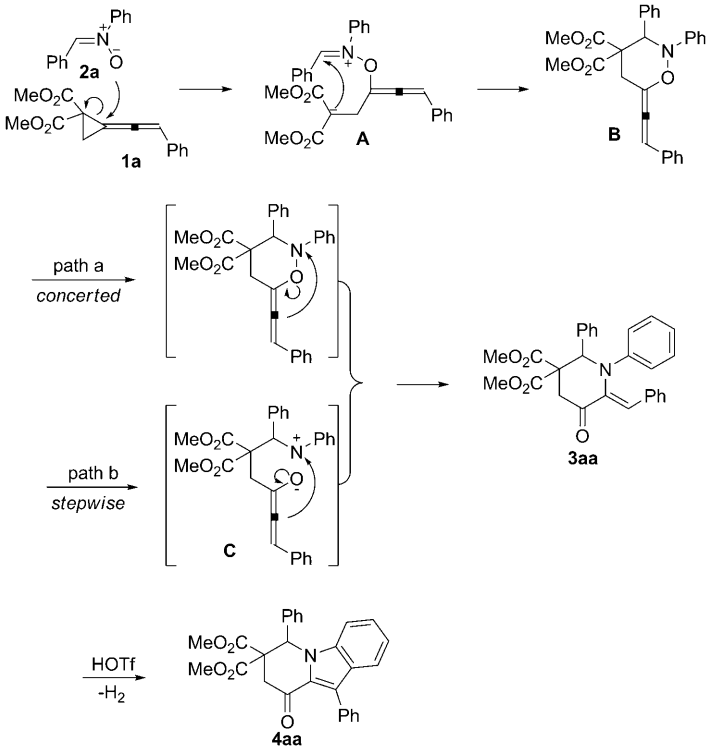


Figure 2. X-ray crystal structure of **4aa**.

plex between one or two of the carbonyl groups.[3,14] Subsequently, a stepwise annulation mechanism is proposed involving an initial nucleophilic attack of the negatively charged oxygen atom of nitrone dipole **2a** onto the cyclopropane, leading to the opening of the cyclopropyl ring and the formation of zwitterionic intermediate **A**, which undergoes a cyclization to afford the formal [3+3] cycloaddition intermediate **B**. Presumably, the nitrogen–oxygen bond of intermediate **B** can be easily cleaved and subsequently rearranges to afford the final product **3aa** either in a concerted way (path a)[15] or in a stepwise way through a zwitterionic intermediate **C** (path b). The product **3aa** mediated by HOTf may undergo an intramolecular Friedel–Crafts arylation (a typical Scholl reaction),[16] affording the indole derivative **4aa**.



Scheme 1. A plausible reaction mechanism.

In conclusion, we have established a novel method for the construction of indole derivatives through $Yb(OTf)_3$-catalyzed formal [3+3] cycloaddition of vinylidenecyclopropanes bearing two EWGs at the cyclopropane ring with nitrones. The reaction is applicable to a variety of VDCPs and nitrones with a moderate yield under mild reaction conditions. Further efforts are in progress regarding the scope and mechanistic details.

Experimental Section

General remarks: ^1H and ^{13}C NMR spectra were recorded at 400 and 100 MHz, respectively. Mass and HRMS spectra were recorded by EI method. Organic solvents used were dried by standard methods when necessary. Satisfactory CHN microanalyses were obtained with an analyzer. Commercially obtained reagents were used without further purification. All these reactions were monitored by TLC with silica gel coated plates. Flash column chromatography was carried out using silica gel at increased pressure. VDCP diesters **1** were prepared according to the literature.[11]

General procedure for $Yb(OTf)_3$-catalyzed formal [3+3] cycloaddition of 1,1-vinylidenecyclopropanediesters with nitrones: Under an argon atmosphere, nitrone **2** (0.2 mmol, 1.0 equiv), $Yb(OTf)_3$ (10 mol%) and 4 Å molecular sieves (50 mg) were placed in a Schlenk tube, and then toluene (2.0 mL) was added. The mixture was stirred at room temperature (20 °C) for 5 min and then a solution of VDCP diester **1** (0.3 mmol, 1.5 equiv) in toluene (6.0 mL) was added into the reaction mixture. The reaction completed within 10 min. Then the solvent was quickly removed under reduced pressure and the residue was purified by a short path silica gel flash column chromatography immediately.

General procedure for HOTf-mediated translations of compounds 3 to indole derivatives: Compound **3** (0.2 mmol) was dissolved in dichlorome-

thane (5.0 mL) in a Schlenk tube and cooled to 0 °C, and then at this temperature HOTf (0.3 mmol, 1.5 equiv) was added in. The mixture was stirred at room temperature (20 °C) for about additional 50 h. After the reaction was completed the solvent was removed under reduced pressure and the residue was purified by silica gel flash column chromatography.

Vinylidenecyclopropanediester 1a: A light yellow oil. ^{1}H NMR ($CDCl_3$, 300 MHz, TMS): $\delta = 2.63$ (d, $J = 4.5$ Hz, 2H; CH_2), 3.80 (s, 3H; CH_3), 3.81 (s, 3H; CH_3), 6.59 (t, $J = 4.5$ Hz, 1H; CH), 6.02 (s, 1H; CH), 7.21–7.26 (m, 2H; Ar), 7.29–7.32 ppm (m, 3H; Ar); ^{13}C NMR ($CDCl_3$, 75 MHz, TMS): $\delta = 19.9$, 35.2, 52.57, 52.61, 86.8, 102.2, 126.9, 127.4, 128.4, 133.5, 166.9, 167.3, 188.8 ppm; IR (CH_2Cl_2): $\tilde{\nu} = 2925$, 2927, 2854, 2370, 2342, 23013, 1738, 1436, 1276, 1234, 1108, 693 cm^{-1}; MS (%): *m/z* (%): 258 (2) $[M]^+$, 242 (9), 155 (13), 113 (28), 99 (31), 85 (64), 71 (81), 57 (100); HRMS (EI): *m/z* calcd for $C_{15}H_{14}O_4$: 258.0892; found: 258.0891.

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Keywords: cycloaddition · homogeneous catalysis · indoles · nitrones · ytterbium

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[12] CCDC-731462 (**3ae**) and 719445 (**4aa**) 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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