## Solvent-Controlled Oxidative Cyclization for Divergent Synthesis of Highly Functionalized Oxetanes and Cyclopropanes

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## ABSTRACT



An efficient solvent-controlled oxidative cyclization of Michael adducts of malonates with chalcones with the combination of iodosobenzene and tetrabutylammonium iodide is reported. Highly functionalized oxetanes and cyclopropanes were divergently synthesized in moderate to excellent yields with high diastereoselectivity.

The efficient composition of highly strained compounds such as three- or four-membered cycles is of significant synthetic interest.<sup>1-4</sup> Their rigid scaffolds not only appear as important

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10.1021/ol9012102 CCC: \$40.75 © 2009 American Chemical Society Published on Web 06/17/2009 substructures in a large number of medicinally and biologically active natural and unnatural substances but also can undergo a variety of transformations to afford other useful synthetic building blocks.<sup>5</sup> The Michael addition of malonates to  $\alpha,\beta$ -unsaturated ketones is one of the important C-C bond forming reactions that offer access to synthetically useful functionalized organic molecules.<sup>6</sup> In the last decades,

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many methods have been developed for the stereoselective reaction.<sup>7</sup> As a result, this transformation is practical for use in organic synthesis. In this communication, we present a solvent-controlled stereoselective oxidative cyclization of Michael adducts of malonates with chalcones for the divergent synthesis of highly functionalized oxetanes and cyclopropanes.

Polyvalent iodine derivatives have been used extensively in organic synthesis as a result of their benign environmental character, ready availability and versatility.<sup>8</sup> Some of our recent efforts<sup>9</sup> have been addressed in the development of synthetic application of iodobenzene diacetate, PhI(OAc)<sub>2</sub>, to construct aziridines<sup>9b</sup> and nitrocyclopropanes.<sup>9d</sup> With the aim of extending this approach, we investigated the oxidative cyclization of Michael adduct of ethyl malonate with chalcone.



No cyclopropanation was observed with PhI(OAc)2 as the oxidant under the typical conditions. Interestingly, with

Table	1.	Evaluation	of	Reaction	Conditions <sup>a</sup>
ant	1.	Lvaluation	01	Reaction	Conditions

EtO	$\begin{array}{c} \text{OC}  \text{COOEt}  \text{EtOOC}  \text{COOEt} \\ \text{Ph}  \text{PhIO, Bu_4NI}  \text{Ph}  Ph$		Ph
		3a	2a
entry	conditions	(%)0	(%)0
1	Toluene, 30 °C, 28 h	25	32
2	CH <sub>2</sub> Cl <sub>2</sub> , 30 °C, 36 h	28	27
3	DMF, 30 °C, 30 h	47	30
4	<i>t</i> -BuOH, 30 °C, 10 h	28	26
5	EtOH, 30 °C, 10 h	59	7
6	MeOH, 30 °C, 3 h	90	0
7	H <sub>2</sub> O, 30 °C, 128 h	4	60
8	H <sub>2</sub> O, 50 °C, 30 h	17	40
$9^c$	$C_{12}H_{25}C_6H_4SO_3Na, H_2O, 30 \ ^\circ C, 128 \ h$	7	0
10	Ultrasonic, H <sub>2</sub> O, 30 °C, 128 h	6	0
$11^d$	$\rm SiO_2, Na_2CO_3$ (0.5 equiv), $\rm H_2O, 30\ ^\circ C, 96\ h$	$^{2}$	63
$12^e$	MeOH, 30 °C, 3 h	92	0
$13^{f}$	$SiO_2,Na_2CO_3$ (0.5 equiv), H_2O, 30 °C, 96 h	2	68

<sup>*a*</sup> Reaction conditions: substrate **1a** (0.5 mmol), PhIO (1 mmol), and Bu<sub>4</sub>NI (1 mmol), solvent (2 mL), unless noted. <sup>*b*</sup> Isolated yield based on substrate **1a**. <sup>*c*</sup> C<sub>12</sub>H<sub>25</sub>C<sub>6</sub>H<sub>4</sub>SO<sub>3</sub>Na (100 mg) was added. <sup>*d*</sup> Na<sub>2</sub>CO<sub>3</sub> (0.5 equiv, 0.25 mmol) and silica gel (100 mg) were added. <sup>*e*</sup> Bu<sub>4</sub>NI (1.5 equiv, 0.75 mmol) was used. <sup>*f*</sup> PhIO (3 equiv, 1.5 mmol) and 1.2 equivalents of Bu<sub>4</sub>NI (0.6 mmol), 0.5 equivalents of Na<sub>2</sub>CO<sub>3</sub> (0.25 mmol), and silica gel (100 mg) were used.

iodosobenzene (PhIO) instead of PhI(OAc)2, the desired cyclopropane 3a was obtained in 20% yield, and an unexpected product, which was identified as the oxetane 2a, was isolated in 38% yield (Scheme 1).



Due to Dunitz-Schomaker strain (1,3-carbon-carbon interaction),<sup>10</sup> which is significant in four-membered systems,<sup>11</sup> the efficient synthesis of oxetanes remains a challenge for organic chemists.<sup>4</sup> Motivated by the synthetic potential of the possible method, the reaction was further optimized by examining various reaction conditions, and some of the results are shown in Table 1. Compared with the reactions in polar or nonpolar aprotic solvents, the reactions in alcohols completed in the shorter time. A satisfactory selectivity and isolated yield of cyclopropane 3a (90% yield) was achieved when methanol was utilized (Table 1, entry 6). When the reaction was carried out in an air-open system with water as the solvent, the reaction gave rise to oxetane 2a as the major product in 60% yield, albeit in a longer time (Table 1, entry 7). Higher temperature accelerated the reaction but resulted in an inferior result (Table 1, entry 8). Addition of a surfactant or ultrasonic treatment resulted in complicated reactions, and no oxetane 2a was obtained (Table 1, entries 9 and 10). Moreover, when  $SiO_2$  and  $Na_2CO_3$  (0.5 equiv) were used as the additives, the reaction was accelerated and

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gave rise to a better selectivity and a higher isolated yield of oxetane **2a** (Table 1, entry 11). In the further investigation, we found that the best ratio of substrate, PhIO, and Bu<sub>4</sub>NI for the reaction in MeOH was 1:2:1.5, with which the yield of **3a** increased to 92%, while the best ratio for the reaction in  $H_2O$  was 1:3:1.2, with which the yield of **2a** increased to 68% (Table 1, entries 12 and 13).

To understand the reaction pathway, several control experiments were conducted (Scheme 2). While no reaction occurred in the absence of  $Bu_4NI$ , cyclopropane **3a** was obtained in 48% yield with the combination of PhIO with a base (*t*-BuOK). Although the generation of iodine was observed during the reactions, no cyclopropane or oxetane was formed with  $I_2$  instead of  $Bu_4NI$ . The above results indicated that the reaction might not be mediated by I<sup>+</sup>. A small amount of 2-hydroxy substituted Michael adduct **4** was detected from the reaction in water. After the purification and identification, compound **4** was treated with PhIO and  $Bu_4NI$  in water. The reaction was completed in 24 h and gave rise to oxetane **2a** in 82% yield.

A plausible reaction pathway, which involves the ligand exchange reaction and reductive elimination reaction of organic iodine(III) compounds,<sup>8,12</sup> is outlined in Scheme 3. In the presence of Bu<sub>4</sub>NI, polymeric iodosobenzene is depolymerized and generates an oxidizing monomeric species  $\mathbf{A}$ ,<sup>13</sup> which can be transformed into an intermediate  $\mathbf{B}$  in MeOH. Michael adduct **1a** reacts with the highly reactive species  $\mathbf{A}$  or  $\mathbf{B}$  via a ligand exchange reaction to form an intermediate  $\mathbf{C}$ , which undergoes an intramolecular reductive elimination to afford cyclopropane **3a**. When the reaction is carried out in water, the intermediate  $\mathbf{C}$  reacts with water to

Table 2. Solvent-Controlled Oxidative Cyclization of Michael Adducts of Malonates with Chalcones

PhIO (3 equiv), Bu <sub>4</sub> NI (1.2 equiv) SiOo NacCOo (0.5 equiv)	
H <sub>2</sub> O, 30 °C	
condition A	R' // K 2 O +
condition B	R <sup>3</sup> OOC COOR <sup>3</sup>
PhIO (2 equiv), Bu <sub>4</sub> NI (1.5 equiv) MeOH, 30 °C	R <sup>1</sup> 3 0
	PhIO (3 equiv), Bu <sub>4</sub> NI (1.2 equiv) SiO <sub>2</sub> , Na <sub>2</sub> CO <sub>3</sub> (0.5 equiv) H <sub>2</sub> O, 30 °C condition A <u>condition B</u> PhIO (2 equiv), Bu <sub>4</sub> NI (1.5 equiv) MeOH, 30 °C

				condition $A^a$			condition $B^b$				
entry	$\mathbb{R}^1$	$\mathbb{R}^2$	$\mathbb{R}^3$	2	yield $(\%)^c$	3	yield $(\%)^c$	2	yield $(\%)^c$	3	yield (%) <sup>c</sup>
1	Ph	Ph	Et	2a	68	3a	2	2a	0	3a	92
2	$4-MeO-C_6H_4$	Ph		<b>2b</b>	83	3b	3	<b>2b</b>	0	3b	95
3	$4-Me-C_6H_4$	Ph		<b>2c</b>	70	3c	3	<b>2c</b>	0	3c	96
4	$4-Cl-C_6H_4$	Ph		<b>2d</b>	60	3d	12	<b>2d</b>	0	3d	96
5	$2-Cl-C_6H_4$	Ph		<b>2e</b>	61	<b>3e</b>	28	<b>2e</b>	0	<b>3e</b>	92
6	$4-NO_2-C_6H_4$	Ph		<b>2f</b>	0	3f	0	<b>2f</b>	0	3f	92
7	$3-NO_2-C_6H_4$	Ph		$2\mathbf{g}$	41	3g	21	$2\mathbf{g}$	0	3g	95
8	Ph	$4\text{-MeO-C}_6\text{H}_4$		<b>2h</b>	73	3h	trace	2h	0	3h	98
9	Ph	$4\text{-Me-C}_6\text{H}_4$		<b>2i</b>	70	3i	trace	<b>2i</b>	0	<b>3i</b>	98
10	Ph	$4-Cl-C_6H_4$		2j	63	3j	21	2j	0	3j	94
11	Ph	$4-NO_2-C_6H_4$		$2\mathbf{k}$	0	3k	68	$2\mathbf{k}$	0	3k	75
12	$4-MeO-C_6H_4$	$4\text{-Me-C}_6\text{H}_4$		2L	66	3L	trace	2L	0	3L	95
13	$4-MeO-C_6H_4$	$4-Cl-C_6H_4$		<b>2m</b>	71	3m	11	<b>2m</b>	0	3m	96
14	Ph	Ph	Me	<b>2n</b>	60	3n	trace	<b>2n</b>	0	3n	94
15	Ph	Ph	<sup>t</sup> Bu	<b>2o</b>	0	30	0	<b>2o</b>	0	30	89
16	t-Bu	Ph	$\mathbf{Et}$	2p	0	3р	0	2p	0	3p	0
17	Ph	$\mathrm{CH}_3$	$\mathbf{Et}$	2q	0	3q	0	$2\mathbf{q}$	0	3q	0

<sup>*a*</sup> Reaction conditions A: substrate **1** (0.5 mmol), PhIO (1.5 mmol), Bu<sub>4</sub>NI (0.6 mmol), silica gel (100 mg) and Na<sub>2</sub>CO<sub>3</sub> (0.25 mmol), H<sub>2</sub>O (2 mL). <sup>*b*</sup> Reaction conditions B: substrate **1** (0.5 mmol), PhIO (1 mmol), Bu<sub>4</sub>NI (0.75 mmol), MeOH (2 mL). <sup>*c*</sup> Isolated yield based on substrate **1**. yield 2-hydroxy compound **4**. After the second ligand exchange reaction with **A** to generate an intermediate **D** and its subsequent reductive elimination, oxetane **2a** is formed.

The generality of the present PhIO/Bu<sub>4</sub>NI induced divergent oxidative cyclization of Michael adducts of malonates with chalcones was then investigated under the established conditions (Table 2). When the reactions were carried out in water, a moderate electronic substrate effect was observed. Compounds with an electron-donating substituent were better substrates than those with an electron-withdrawing substituent, and their reactions afforded the corresponding oxetanes in better selectivities and yields. This electronic substrate effect accorded with the proposed mechanism. Oxetanes and cyclopropanes are derived from the same intermediate C. Due to the higher reactivity of the proton on the carbon alpha to the electron-poor aryl ketone, the intramolecular reductive elimination reaction of the intermediate C is preferred to yield cyclopropane. For the electron-rich aryl ketone substrates, the lower reactivity of the corresponding proton leads the intermediate C to be readier to undergo the intermolecular reaction with water to yield 2-hydroxy compound and to afford oxtane. No oxetane was obtained from the reactions of *p*-nitro substrates or ditert-butyl malonate derivative. When the reactions were carried out in MeOH, all substrates showed a good reactivity. The corresponding cyclopropanes were generated in good to excellent yields, while no oxetane

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Figure 1. X-ray diffraction structure of 2d.

In conclusion, we report here an efficient solventcontrolled oxidative cyclization of Michael adducts of malonates with chalcones with the combination of iodosobenzene and tetrabutylammonium iodide. Highly functionalized oxetanes and cyclopropanes are divergently synthesized in moderate to excellent yields with high diastereoselectivity. Current dedication has been made to extend its scope, asymmetric transformations, and possible synthetic applications.

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**Supporting Information Available:** Experimental details and spectral data for the major products. This material is available free of charge via the Internet at http://pubs.acs.org.

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