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Facile synthesis of oxazolidinones catalyzed by n-Bu_4NBr_3/n-Bu_4NBr directly from olefins, chloramine-T and carbon dioxide

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ABSTRACT

A binary catalyst system composed of n-Bu_4NBr_3/n-Bu_4NBr was developed for facile synthesis of 5-substituted 2-oxazolidinones with perfect regioselectivity in a single operation directly from olefins, chloramine-T and CO_2. The choice of efficient binary catalysts for two steps, i.e. aziridination and cycloaddition, and the optimization of reaction condition are keys to the one-pot synthesis of 5-substituted 2-oxazolidinones. A possible mechanism for the present one-pot synthesis of oxazolidinones was also proposed.

1. Introduction

Carbon dioxide is the most abundant greenhouse gas and can be also regarded as a typical renewable natural resource. The chemical fixation of CO_2 received much attention from the viewpoint of resources utilization and green chemistry [1]. In this context, one of the major successes is the utilization of CO_2 as the starting materials to prepare the five-membered cyclic compounds. 2-Oxazolidinones are an important class of five-membered cycles [2] showing a plethora of applications as intermediates [3] and chiral auxiliaries [4] in organic synthesis.

Currently, there are four main synthetic strategies for 2-oxazolidinones starting from CO_2 as C1 resources: (i) direct addition of CO_2 to 1, 2-aminoalcohols; [5–7] (ii) reaction of propargylamines with CO_2; [8–10] (iii) reaction of amine/aminoalcohol with cyclic carbonate from CO_2 [11,12] and (iv) insertion of CO_2 into the aziridines. Among them, cycloaddition of CO_2 with aziridines is one of the most promising methods for synthesis of oxazolidinones. Several catalyst systems have been explored for this reaction [13–17]. However, such a cycloaddition generally involves the initial synthesis of aziridine from olefin and a nitrogen source, [18–31] which requires a tedious workup for separation and involves metal catalysts in most cases. In other words, this two-step manipulation is one of the main drawbacks of this process. So it is more desirable to synthesize an oxazolidinone directly from olefin, a nitrogen source and CO_2.

We have developed an effective catalytic process for the synthesis of 5-substituted 2-oxazolidinone from aziridine and CO_2 [14,15]. In this work, we would like to report a binary catalyst system comprising n-Bu_4NBr_3/n-Bu_4NBr for highly regioselective synthesis of 5-substituted 2-oxazolidinones in a single operation via the three-component reaction of olefin, chloramine-T (sodium p-toluenesulfonchloramide, TsNClNa, a practical nitrogen source) with CO_2.

2. Experimental

2.1. General procedure for the synthesis of tetrabutylammonium tribromide according to the published procedure

To a solution of tetrabutylammonium bromide (3.2 g, 10 mmol) and sodium bromate (0.5 g, 3.3 mmol) in water (20 mL) was added dropwise hydrobromic acid (40%, 4 mL) under stirring at r.t. After the mixture was stirred for 15 min, the immediate orange precipitate was filtered and recrystallized from petroleum ether-dichloromethane (4:1) to give tetrabutylammonium tribromide as orange crystals; yield 4.65 g (96.5%); m.p. 72–73 °C (lit. 70–72 °C)[32,33].

2.2. General procedure for the synthesis of oxazolidinones from olefins, chloramine-T and CO_2

A 50 mL autoclave reactor was charged with olefin (3 mmol), chloramine-T (4 mmol, the commercially available chloramine-T trihydrate was dried to constant weight at ca. 80 °C for 12 h in a drying pistol [34,35]), n-Bu_4NBr (0.3 mmol), n-Bu_4NBr_3 (0.3 mmol), hydroquinone (0.15 mmol), biphenyl (100 mg, as internal standard) and CH_3CN (10 mL). CO_2 was introduced into the autoclave and then the

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mixture was stirred at desired temperature (for instance 100 °C) for 15 min to allow equilibration. Finally, the pressure was adjusted to the reaction pressure (e.g. 8 MPa) and the mixture was stirred continuously. When the reaction finished, the reactor was cooled in ice-water and CO_2 was ejected slowly. An aliquot of sample was taken from the resultant mixture for GC analysis. The residue was purified by column chromatography on silica gel (200–300 mesh, eluting with 5:1 to 20:1 petroleum ether/ethyl acetate) to afford the desired product. The products were further identified by ^1H NMR and ^{13}C NMR spectroscopy, ESI-MS and IR, which are consistent with those reported in the literature and in good agreement with the assigned structures. The polymeric side-product was purified by reprecipitation from THF/H_2O to remove low-molecular-weight products, and finally, the product was dried in vacuo. Molecular weights and molecular weight distributions of the oligomers were measured using Agilent Technologies 1200 Series GPC-SEC Analysis System (concentration, 1 mg/mL; eluent, THF; flow rate, 1 mL/min; temperature, 23 °C). Calibration was made with monodispersed polystyrenes as standards (see Electronic supplementary information).

3. Results and discussion

3.1. One-pot binary catalytic process for the synthesis of 5-phenyl 2-oxazolidinone

$PhMe_3NBr_3$ was found to be active towards the aziridination of olefins and *n*-Bu_4NBr was effective for the cycloaddition reaction of aziridine and CO_2, in well agreement with the published results. [13,21] We reasoned the binary catalysts comprising *n*-Bu_4NBr_3/*n*-Bu_4NBr could be effective for synthesis of 5-substituted 2-oxazolidinones in a single operation directly from olefins, chloramine-T and CO_2 as depicted in Scheme 1. Styrene was selected as a model substrate for further investigation.

Table 1 summarizes experimental results on catalyst effect on one-pot process for the synthesis of 2-oxazolidinones. Neither product **2**, **3** nor **4** was detected at all without any catalyst (entry 1, Table 1). And *n*-Bu_4NBr alone was also inactive for this process (entry 2). On the other hand, *n*-Bu_4NBr_3 itself showed poor active to afford the desired product **3** in 27% yield (entry 3) and increasing its amount enhanced formation of aziridine **2** (entry 4), so Br_3^- is necessary for the generation of 2-oxazolidinones **3**. Interestingly, addition of *n*-Bu_4NBr could improve the activity (entry 3 vs.7). Notably, the anion of catalyst **II** has great influence on reactivity. The yield of **3** was found to be *n*-Bu_4NBr > *n*-Bu_4NCl > *n*-Bu_4NF (entries 5–7), being presumably correlated with nucleophilicity and leaving ability of the anions. However, both *n*-Bu_4NI and I_2 gave no desired product (entry 11 and 12), which could be ascribed to possible oxidation of *n*-Bu_4NI or I_2 by Br_3^-. Indeed, *n*-Bu_4NI_3 in place of *n*-Bu_4NBr_3 produced product **3** even low yield (entry 14 vs. 10), also supporting this hypothesis. Subsequently, a series of tetraalkylammonium tribromide and tetraalkylammonium bromide with various alkyl chain lengths were also investigated (entries 15–17). The oxazolidinone yield increased in the order of *n*-Bu_4N^+ > *n*-Pr_4N^+ > Me_4N^+ (entries 7, 15, 16), *n*-$CetMe_3N^+$ > Me_4N^+ (entries 15, 17). The LiBr which has been reported [13] to be active for the coupling of aziridine and CO_2, also gave relatively good yield (entry 13). All the results demonstrated a homogeneous binary catalyst system of *n*-Bu_4NBr_3/*n*-Bu_4NBr was the most effective for the present one-pot process to regioselectively afford 5-phenyl 2-oxazolidinone **3** in 49.6% yield without the formation 4-phenyl isomer, alongside with 11.4% yield of aziridine **2** and 9.6% hydrolyzed product **4**. In addition, the influence of the amount of catalyst was also evaluated. The yield of **3** was 31.1% when 5 mol% catalyst was used; and increased to 49.6% with 10 mol% of catalyst loading (entries 7 and 10). Moreover, more polymerized products were detected without hydroquinone (entry 8). And the use of the trihydrate of chloramine-T gave more hydrolytic product **4** (entry 9).

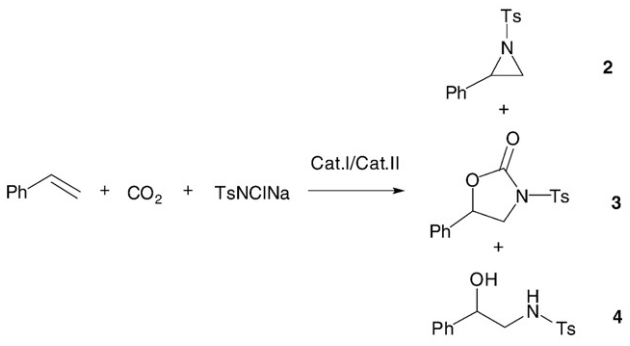
Table 1
Catalyst effect on one-pot process for the synthesis of 5-phenyl 2-oxazolidinone[a].

Entry	Cat. I	Cat. II	Yield /% [b]		
			2	3	4
1	–	–	0	0	0
2	–	*n*-Bu_4NBr	0	0	0
3	*n*-Bu_4NBr_3	–	2.8	27.0	4.1
4 [c]	*n*-Bu_4NBr_3	–	11.3	31.0	11.1
5	*n*-Bu_4NBr_3	*n*-Bu_4NF	9.0	10.8	3.8
6	*n*-Bu_4NBr_3	*n*-Bu_4NCl	9.9	31.5	3.2
7	*n*-Bu_4NBr_3	*n*-Bu_4NBr	11.4	49.6	9.6
8 [d]	*n*-Bu_4NBr_3	*n*-Bu_4NBr	3.3	14.9	2.4
9 [e]	*n*-Bu_4NBr_3	*n*-Bu_4NBr	4.9	27.9	22.1
10 [f]	*n*-Bu_4NBr_3	*n*-Bu_4NBr	12.2	31.1	2.1
11	*n*-Bu_4NBr_3	*n*-Bu_4NI	0.86	0	0.28
12	*n*-Bu_4NBr_3	I_2	0.77	0	0.82
13	*n*-Bu_4NBr_3	LiBr	0.56	35.1	3.7
14	*n*-Bu_4NI_3	Bu_4NI	0.56	4.9	4.7
15	Me_4NBr_3	Me_4NBr	0.69	12.6	1.8
16	*n*-Pr_4NBr_3	*n*-Pr_4NBr	6.1	20.3	3.7
17 [g]	*n*-$CetMe_3NBr_3$	*n*-$CetMe_3NBr$	1.7	25.6	6.5

[a] All the reaction were performed with styrene (3 mmol), chloramine-T (4 mmol, 1.33 equiv.), Cat. I (0.3 mmol, 10 mol%), Cat. II (0.3 mmol, 10 mol%), hydroquinone (0.15 mmol, 5 mol%, as polymerization inhibitor) and CH_3CN (10 mL) at 8 MPa CO_2, 100 °C for 24 h unless otherwise noted.
[b] Determined by GC using biphenyl as internal standard.
[c] Cat. I (0.9 mmol, 30 mol%).
[d] Without hydroquinone 0.3526 g of oligomer ($Mn=6.6869\times10^2$ g/mol, $Mw=6.7158\times10^2$ g/mol, $Mz=6.7452\times10^2$ g/mol) was obtained.
[e] Chloramine-T trihydrate (4 mmol).
[f] Cat. I (0.15 mmol, 5 mol%), Cat. II (0.15 mmol, 5 mol%).
[g] *n*-$CetMe_3NBr$: cetyl trimethylammonium bromide.

3.2. The effects of the reaction parameters

The reaction conditions were also carefully examined for the reaction of styrene with chloramine-T and CO_2 using the *n*-Bu_4NBr_3/*n*-Bu_4NBr binary catalyst system, and the results are summarized in Table 2. The yield of 2-oxazolidinone **3** increased with reaction time within 24 h (entries 1–5, Table 2). On the other hand, the yield of **3** increased rapidly with reaction temperature up to 100 °C, whereas, further increase in the temperature caused a decrease in the yield (entries 4, 6–9), possibly due to the decomposition of chloramine-T. [32,33] The influence of CO_2 pressure on the reaction was also investigated, the most proper pressure was around 8 MPa. The yield of the target product **3** increased with CO_2 pressure up to 8 MPa (entry 4, 10–13, Table 2). This enhancement would be ascribed to less mass transfer limitation under near critical CO_2 conditions. Whereas, further increase in the CO_2 pressure caused a decrease in the yield. This presumably is owing to CO_2 diluent effect, which could decrease the polarity of the reaction medium. So the appropriate reaction conditions are at 100 °C under 8 MPa CO_2 pressure and 24 h.



Scheme 1. One-pot synthesis of 5-phenyl-2-oxazolidinone via the three-component coupling of styrene, chloramine-T and CO_2.

Table 2
Screening reaction conditions[a].

Entry	Temp./°C	Pressure /MPa	Time/ h	Yield /%[b]		
				2	3	4
1	100	8	6	13.9	14.2	5.2
2	100	8	12	11.2	25.5	8.5
3	100	8	18	11.1	49.4	9.4
4[c]	100	8	24	11.4	49.6	9.6
5	100	8	48	8.07	48.0	9.2
6	25	8	24	8.13	0	1.5
7	50	8	24	12.7	6.7	1.6
8	80	8	24	8.25	15.3	6.0
9	120	8	24	0.39	36.7	11.5
10	100	0.1	24	18.4	0	4.1
11	100	2	24	5.66	19.7	6.9
12	100	6	24	3.01	22.5	9.7
13	100	13	24	8.77	43.3	9.4

[a] Reactions were conducted with styrene (3 mmol), chloramine-T (4 mmol, 1.33 equiv.), *n*-Bu_4NBr (0.3 mmol, 10 mol%), *n*-Bu_4NBr_3 (0.3 mmol, 10 mol%), hydroquinone (0.15 mmol, 5 mol%, as polymerization inhibitor), and CH_3CN (10 mL).
[b] Determined by GC using biphenyl as internal standard.
[c] The polymeric side-product was purified by reprecipitation from THF/H_2O. 0.0919 g of oligomers (Mn = 5.5245×10^2 g/mol, Mw = 5.8025×10^2 g/mol, Mz = 6.1788×10^2 g/mol) was isolated.

3.3. Solvent effect

The effect of solvent on the reaction was evaluated as shown in Table 3. As a result, catalytic activity is strongly dependent on the solvent (entries 1–9, Table 3). Among the solvents, acetonitrile showed the best results under the otherwise identical conditions (entry 1), possibly due to the polarity and the solvent power for dissolving chloramine-T. Particularly, CCl_4 is a bifunctional initiator to initiate atom transfer radical polymerization of styrene. Indeed, it gave polymers as major product. [36]

3.4. Synthesis of various carbamates

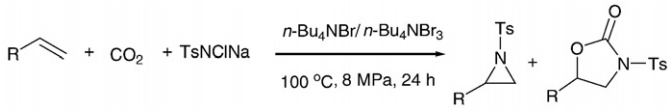
To demonstrate the utility and generality of this approach to formation of 5-substituted 2-oxazolidinones, we examined the reactions of various olefins with chloramine-T and CO_2 by performing the reaction under the identical conditions (Scheme 2).

The results are listed in Table 4. The substrates with different alkyl chain lengths were investigated (entries 1–4, Table 4), which gave relative different reactivity, presumably owing to the sterically hindered influence in terms of reaction mechanistic consideration (see Scheme 3).

Table 3
Solvent effect[a].

Entry	solvent	Yield /%[b]		
		2	3	4
1	CH_3CN	11.4	49.6	9.6
2	–	3.86	0.96	0
3	THF	2.06	26.1	4.7
4	DMF	5.06	15.2	2.2
5	NMP	0.60	0	0
6	CH_2Cl_2	4.12	19.5	7.2
7	$CHCl_3$	8.36	3.34	1.9
8	Toluene	2.64	28.3	2.8
9	CCl_4	2.55	2.21	0.59

[a] Reaction conditions: styrene (3 mmol), chloramine-T (4 mmol, 1.33 equiv.), *n*-Bu_4NBr (0.3 mmol, 10 mol%), *n*-Bu_4NBr_3 (0.3 mmol, 10 mol%), hydroquinone (0.15 mmol, 5 mol%, as polymerization inhibitor), and solvent (10 mL), 8 MPa CO_2, 100 °C for 24 h.
[b] Determined by GC using biphenyl as internal standard.



Scheme 2. Substrate scope.

However, cyclohexene showed no 2-oxazolidinone other than 39.8% isolated yield of the aziridine (entry 1 vs 5), probably because of inactivity of its corresponding aziridine originating from the steric effect of the internal olefin. Notably, the electron-donating group at benzene ring is likely to be more favorable than electron-deficient counterpart (entries 6–10) to improve the reaction, presumably owing to the electronic influence (see Scheme 3). In particular, the electron-withdrawing group gave just 4% yield of the aziridine without formation of the 2-oxazolidinone, whereas electron-donating group showed higher reactivity in comparison with electron-withdrawing group affording the 2-oxazolidinone with full conversion of the aziridine and a moderate yield (entry 9 vs. 10). Accordingly, electronic effect could have greater impact on generation of 2-oxazolidinone than aziridine. Interestingly, phenyl substituted aliphatic olefins and a diene showed better activities among the tested substrates (entries 11–13). It is also worth noting that only one carbon–carbon double bond was converted to the oxazolidinone (entry 13). Its structure could be proved by NMR spectra (see Supporting Information).

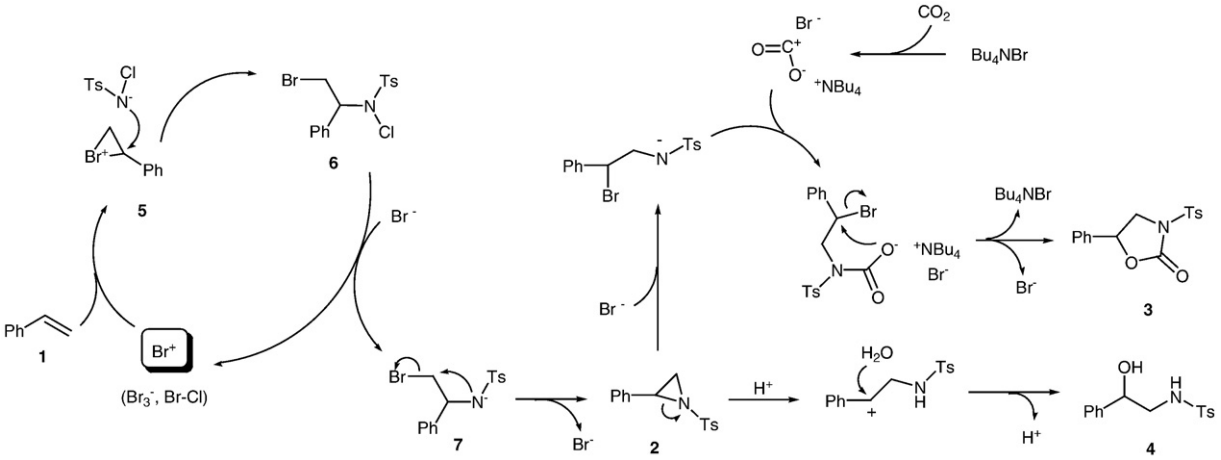
3.5. Possible mechanism

On the basis of the literature [13,21] and experiment results, a possible mechanism for the present *n*-Bu_4NBr_3/*n*-Bu_4NBr catalyzed one-pot process of forming 5-substituted 2-oxazolidinone via the three-component reaction of olefin, chloramine-T and CO_2 is shown in Scheme 3. For the first step, the olefin (**1**) reacts with a Br^+ source (Br_3^-, Br–Cl) to give the bromonium ion (**5**), which then undergoes benzylic opening by $TsNCl^-$ forming the β-bromo-N-chloro-N-toluenesulfonamide (**6**). Attack of Br^- on the N–Cl group in putative intermediate (**6**) generates the anion (**7**). Expulsion of Br^- from the anion (**7**) obtained the aziridine (**2**). [21] For the second step, the mechanism involves three steps: the nucleophilic species Br^-initiates the ring-opening of the aziridine (**2**), followed by reacting with the activated CO_2 and subsequent cyclization via an intramolecular nucleophilic attack leading to the target product 5-substituted 2-oxazolidinone (**3**) and regeneration of the catalyst. On the other hand,

Table 4
Substrate scope of this approach[a].

Entry	Olefin	Isolated yield of aziridines /%	Isolated yield of oxazolidinones /%
1	1-hexene	6.0	63.7
2	1-heptene	8.8	55.9
3	1-octene	12.6	38.2
4[b]	1-dodecene	18.2	14.6
5	cyclohexene	39.8	0
6	Styrene	11.4	47.6
7	2-methylstyrene	4.6	36.9
8	3-methylstyrene	0	47.1
9	4-methylstyrene	0	42.7
10	4-chlorostyrene	4.0	0
11	3-phenyl-1-propene	6.4	52.1
12	4-phenyl-1-butene	5.8	53.0
13	1,5-hexadiene	6.8	56.4

[a] All the experiments were conducted at 8 MPa CO_2, 100 °C for 24 h in a 50 mL stainless steel autoclave containing olefin (3 mmol), chloramine-T (4 mmol, 1.33 equiv.), *n*-Bu_4NBr (0.3 mmol, 10 mol%), *n*-Bu_4NBr_3 (0.3 mmol, 10 mol%), hydroquinone (0.15 mmol, 5 mol%, as polymerization inhibitor), and CH_3CN (10 mL).
[b] 48 h.



Scheme 3. A putative mechanism.

the aziridine (**2**) hydrolyzes in the presence of water to furnish 2-(N-tosylamino)-1-phenyl-1-ethanol (**4**) as a by-product. Furthermore, aziridine species **2** is assumed to be its polymerization and/or copolymerization with CO_2 to afford oligomers under the reaction conditions, as previously reported in the literature. [16,17]

4. Conclusions

In conclusion, we developed a simple and effective process catalyzed by *n*-Bu_4NBr_3/*n*-Bu_4NBr in a single operation for the synthesis of 5-substituted 2-oxazolidinones with perfect regioselectivity directly from olefins, chloramine-T, and CO_2. The choice of efficient binary catalysts for two steps, i.e. aziridination and cycloaddition, and the optimization of reaction condition are keys to the one-pot synthesis of 5-substituted 2-oxazolidinones. The present protocol could show an additional example for efficiently utilizing CO_2 as a carbon source in the field of green chemistry and catalysis.

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Appendix A. Supplementary data

Supplementary data associated with this article can be found, in the online version, at doi:10.1016/j.catcom.2010.04.003.

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