

## Accepted Article

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## FULL PAPER

# Chemical Upcycling of Waste Poly(bisphenol A carbonate) to 1,4,2-Dioxazol-5-ones and One-pot C–H Amidation

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**Abstract:** Chemical upcycling of poly(bisphenol A carbonate) (PC) was achieved in this study with hydroxamic acid nucleophiles, giving rise to synthetically valuable 1,4,2-dioxazol-5-ones and bisphenol A. Using 1,5,7-triazabicyclo[4.4.0]-dec-5-ene (TBD), non-green carbodiimidazole or phosgene carbonylation agents used in conventional dioxazolone synthesis were successfully replaced with PC, and environmentally harmful bisphenol A was simultaneously recovered. Assorted hydroxamic acids exhibited good to excellent efficiencies and green chemical features, promising broad synthetic application scope. In addition, a green aryl amide synthesis process was developed, involving one-pot depolymerization from polycarbonate to dioxazolone followed by rhodium-catalyzed C–H amidation, including gram-scale examples with used compact discs.

## Introduction

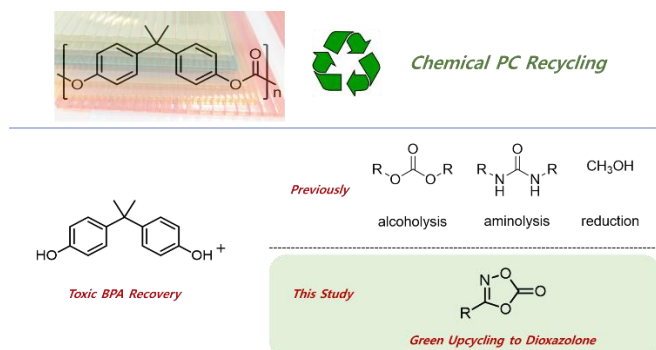
Plastics are essential materials in the modern world. They are lightweight, strong, durable, easy to process, and inexpensive, and their benefits have been reaped over the last 100 years. However, once plastics serve their designated purpose, they pose a serious problem as they do not degrade.<sup>[1]</sup> A recent report estimated that 4.9 billion tons of plastic waste ends up in landfill. Only 0.8 billion tons are recycled, mostly mechanically. However, even recycled plastics eventually end up in landfill or used for energy recovery after a single recycle.<sup>[2]</sup> As an approach to improve the processing of plastic waste, chemical recycling, which is the recovery of chemical feedstocks through pyrolysis or depolymerization of used plastics, has gained attention.<sup>[3–5]</sup> Waste plastics are rich reservoirs in carbon, hydrogen, oxygen, and nitrogen. Thus, chemical recycling could reduce the amount of waste and diminish fossil fuel dependency.

Poly(bisphenol A carbonate) (polycarbonate or PC) is a leading engineering plastic with excellent heat resistance, mechanical strength, and transparency.<sup>[6,7]</sup> Thus, it is widely used in electronics, automobiles, construction, optical parts, and

protective goods. Over 5 million tons of PC are produced, and a similar amount of PC is wasted annually. It is classified under code 7 (other plastics) according to the ASTM plastic recycling system, and most used PCs end up in landfills.<sup>[8]</sup> As a result, a large amount of bisphenol A (BPA) slowly enters the ecosystem. Considering the health and environmental impact of BPA, efficient recycling of PC is critical.<sup>[9,10]</sup> Therefore, chemical recycling of PC is of interest.<sup>[11]</sup> The carbonate functionality can undergo various chemical transformations, mostly toward nucleophilic additions. While pyrolysis yields a mixture of lightweight organic compounds, chemical transformations that are more selective have shown promise.<sup>[12,13]</sup> In particular, the carbonyl moiety could replace the highly toxic phosgene and carbon monoxide in carbonylation reactions.<sup>[14–16]</sup> Besides hydrolysis, which yields BPA and carbon dioxide,<sup>[17,18]</sup> alcoholysis,<sup>[19–23]</sup> aminolysis,<sup>[24–26]</sup> and hydrogenation<sup>[27–29]</sup> yield value-added organic carbonates, carbamates, and urea under mild conditions.

In this regard, 1,4,2-dioxazol-5-one has recently attracted research interest.<sup>[30]</sup> Though first reported in 1950, its synthetic usage was seldom investigated until 2015.<sup>[31,32]</sup> It was recently shown to be a safe and highly reactive acyl nitrene source for various reactions, including C–H amidations.<sup>[33–36]</sup> However, the synthesis of dioxazolones has received little attention. The reaction between hydroxamic acid and highly reactive carbonyl sources, such as phosgene, triphosgene, or 1,1'-carbodiimidazole (CDI), has been carried out for a long time.<sup>[12,36]</sup> However, considering the instability and toxicity of these reagents, as well as the inconvenience of processing them, a better synthetic process involving easy handling and safe carbonylation reagents is necessary.<sup>[37]</sup> Usage of waste chemicals as starting materials promotes greenness and economy. Based on our experience with the chemical recycling of PC,<sup>[11,21]</sup> we envisioned that PC could replace phosgene or CDI in dioxazolone synthesis (Scheme 1). The details are discussed as follows.

## FULL PAPER



**Scheme 1.** Chemical recycling of PC: BPA recovery and green upcycling to obtain value-added organic compounds.

**Table 1.** Optimization results: phenyl dioxazolone from DPC.

Entry	Catalyst	Solvent	Yield of <b>2a</b> <sup>a</sup>
1	None	2-Me-THF	8%
2	TBD (2 mol%)	2-Me-THF	99%
3	DBU (2 mol%)	2-Me-THF	96%
4	Triethylamine (2 mol%)	2-Me-THF	68%
5	DMAP (2 mol%)	2-Me-THF	51%
6	Pyridine (2 mol%)	2-Me-THF	17%
7	Imidazole (2 mol%)	2-Me-THF	17%
8	NaOH (2 mol%)	2-Me-THF	91%
9	TBD (1 mol%)	2-Me-THF	98%
10	TBD (0.5 mol%)	2-Me-THF	97%
11	TBD (0.1 mol%)	2-Me-THF	86% (50 °C)
12	TBD (2 mol%)	Acetone	98%
13	TBD (2 mol%)	Dimethyl Carbonate	96%
14	TBD (2 mol%)	Ethyl Acetate	92%
15	TBD (2 mol %)	<i>t</i> -BuOH	54%

<sup>a</sup> Yields were determined via <sup>1</sup>H NMR using hexamethylbenzene as an internal standard.

## Results and Discussion

To optimize the dioxazolone formation conditions, we used a model compound, diphenyl carbonate (DPC) (Table 1). The aromatic carbonate, structurally similar to polycarbonate, has exhibited similar chemical behavior in many studies.<sup>[38,16]</sup> The formation of 1,4,2-dioxazol-5-one from highly reactive CDI and phenyl hydroxamic acid (**1a**) does not require a catalyst,<sup>[36]</sup> but the direct reaction between DPC and **1a** afforded only 8% conversion (entry 1). To compensate for such low reactivity, a series of catalysts were applied. In our previous study, we achieved facile alcoholysis of DPC or PC to organic carbonate with the organic superbases, 1,5,7-triazabicyclo[4.4.0]-dec-5-ene (TBD), and 1,8-diazabicyclo[5.4.0]undec-7-ene (DBU).<sup>[16,21]</sup> Likewise, excellent catalytic efficiency was maintained with nucleophile **1a**. TBD (2 mol%) was used to successfully produce the desired phenyl dioxazolone (**2a**) quantitatively in 2-methyl tetrahydrofuran (2-Me-

THF) under mild conditions (30 °C, 2 h) (entry 2). DBU (2 mol%) afforded a good yield (96%, entry 3). Subsequently, the selected organic bases were further analyzed. Triethylamine exhibited intermediate conversion to **2a** (68%, entry 4). *N*-heterocyclic bases, 4-dimethylaminopyridine (DMAP), pyridine, and imidazole produced **2a** in 51%, 17%, and 17% yields, respectively (entries 5–7). These results suggest that the basicity is closely related to the reactivity. The order of basicity, pyridine (pK<sub>a</sub> (MeCN) = 12.53) < imidazole (pK<sub>a</sub> (MeCN) = 15.07) < DMAP (pK<sub>a</sub> (MeCN) = 17.96) < triethylamine (pK<sub>a</sub> (MeCN) = 18.83) < DBU (pK<sub>a</sub> (MeCN) = 24.31) < TBD (pK<sub>a</sub> (MeCN) = 26.02), is consistent with the yields of dioxazolone **2a** under the given conditions.<sup>[39]</sup> An inorganic base NaOH also promoted the reaction efficiently under standard conditions (91%, entry 8).

In previous studies of DPC and polycarbonate alcoholysis to organic carbonates, the strong nucleophilic nature of TBD and DBU played a principal role in activating the aromatic carbonate group.<sup>[16,20,21]</sup> Thus, weak nucleophiles such as DMAP, pyridine, imidazole, and triethylamine did not promote the reaction. In this study, catalyst-free conditions produced conversion, and a more basic catalyst exhibited better efficiency. These observations led us to hypothesize that the reaction commences with the deprotonation of hydroxamic acid, followed by the nucleophilic addition of the conjugated anion. The plausible mechanism is described in scheme 2. In the presence of a base, hydroxamic acid **1a** forms hydroxamates **A**. The nucleophilic attack of **A** to DPC generates oxy-carbonyl-hydroxamate **B** via tetrahedral intermediate and phenoxide departure. The second deprotonation by either catalytic base or phenoxide gives anion **C**, which is known to undergo Lossen rearrangement. However, intramolecular nucleophilic addition of its resonance structure **D** is favored over Lossen rearrangement and furnishes dioxazolone **2a**.<sup>[40,41]</sup>

We further evaluated the most active TBD catalyst systems. The synthetic efficiency remained high at lower catalyst loadings of 1.0 mol% and 0.5 mol% (entries 9 and 10). In case of a 0.1 mol% loading, elevated temperature (50 °C) was required for high conversion (86%, turn-over-number = 860, entry 11). Environmentally and process-friendly solvents were tested to enhance the green chemistry metrics. Aprotic polar solvents such as acetone, dimethyl carbonate, and ethyl acetates promoted excellent DPC to dioxazolone conversion, with 98% (entry 12), 96% (entry 13), and 92% (entry 14) yields, respectively; polar protic *t*-BuOH decreased the efficiency (54%, entry 15).

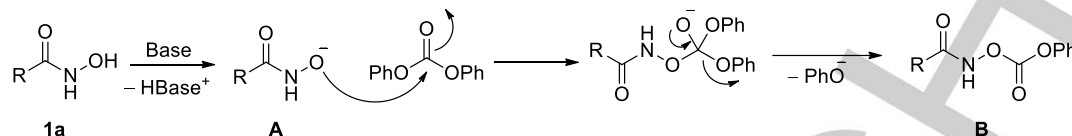
The optimized conditions were successfully applied for the depolymerization of PC (Table 2). The complete depolymerization to BPA and dioxazolone required a longer reaction time than the DPC did. Under the TBD (2 mol%) condition, 4 h was required for near-complete depolymerization, as after 2 h some BPA units containing the carbonates BPAMC and BPADC remained (entries 1 and 2). The use of either TBD (1 mol%) (entry 3) or DBU as a catalyst (2 mol%) (entry 4) could not achieve the same reactivity as in the case of 2 mol% TBD. We monitored the catalytic depolymerization using <sup>1</sup>H NMR (Figure 1A) and size-exclusion chromatography (SEC) (Figure 1B). In 5 min, most of the polymeric BPA was converted to small molecular structures such as BPAMC and BPA. Only 22% of the polymeric carbonate unit, BPADC, remained and no BPADC signal was observed after 30 min. These distributions were highly correlated with changes in molecular weights. The high molecular weight portion of PC (blue line) disappeared in 5 min (red line). The oligomeric peaks

## FULL PAPER

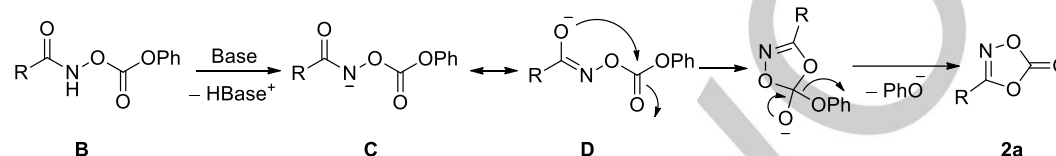
between 5 and 6.5 mL elution volume were completely gone after 30 min. All polycarbonates were depolymerized in 30 min to BPA and BPAMC. These observations suggest that depolymerization

occurs through random chain scission rather than chain-end degradation, which could result in gradual molecular weight reduction and low BPAMC accumulation.

## Step 1) Hydroxamic acid addition to carbonate

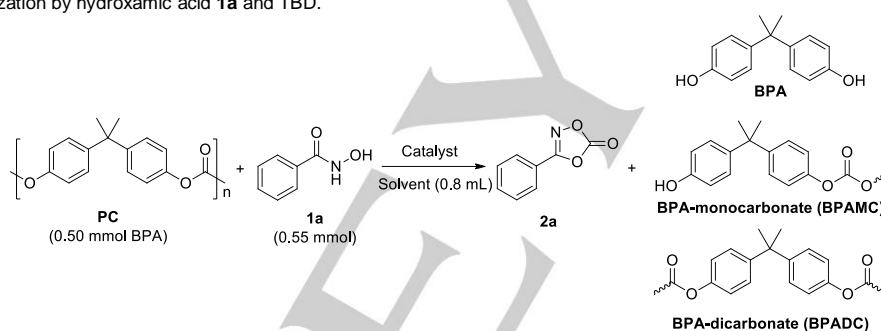


## Step 2) Cyclization



**Scheme 2.** Plausible reaction mechanism of **2a** formation from **1a** and DPC

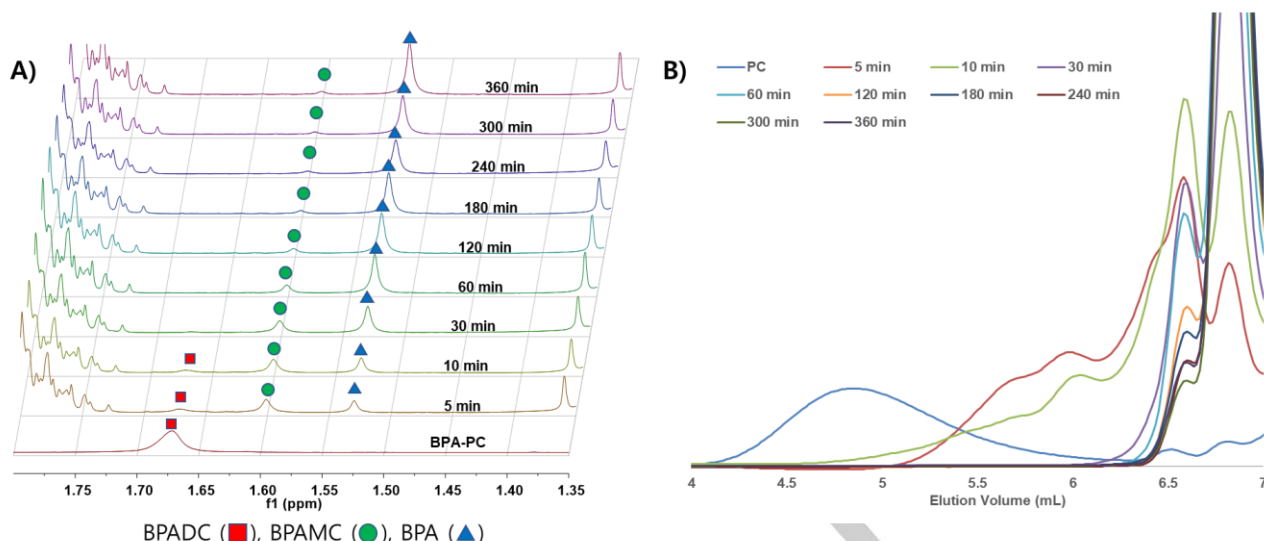
**Table 2.** PC depolymerization by hydroxamic acid **1a** and TBD.



Entry	Catalyst	Conditions	2a (%)	BPA unit distribution		
				BPA (%)	BPAMC (%)	BPADC (%)
1	TBD (2 mol%)	2-Me-THF, 2h, 30°C	89	80	17	2
2	TBD (2 mol%)	2-Me-THF, 4h, 30°C	95	98	2	0
3	TBD (1 mol%)	2-Me-THF, 4h, 30°C	93	94	5	0
4	DBU (2 mol%)	2-Me-THF, 4h, 30°C	91	83	17	0

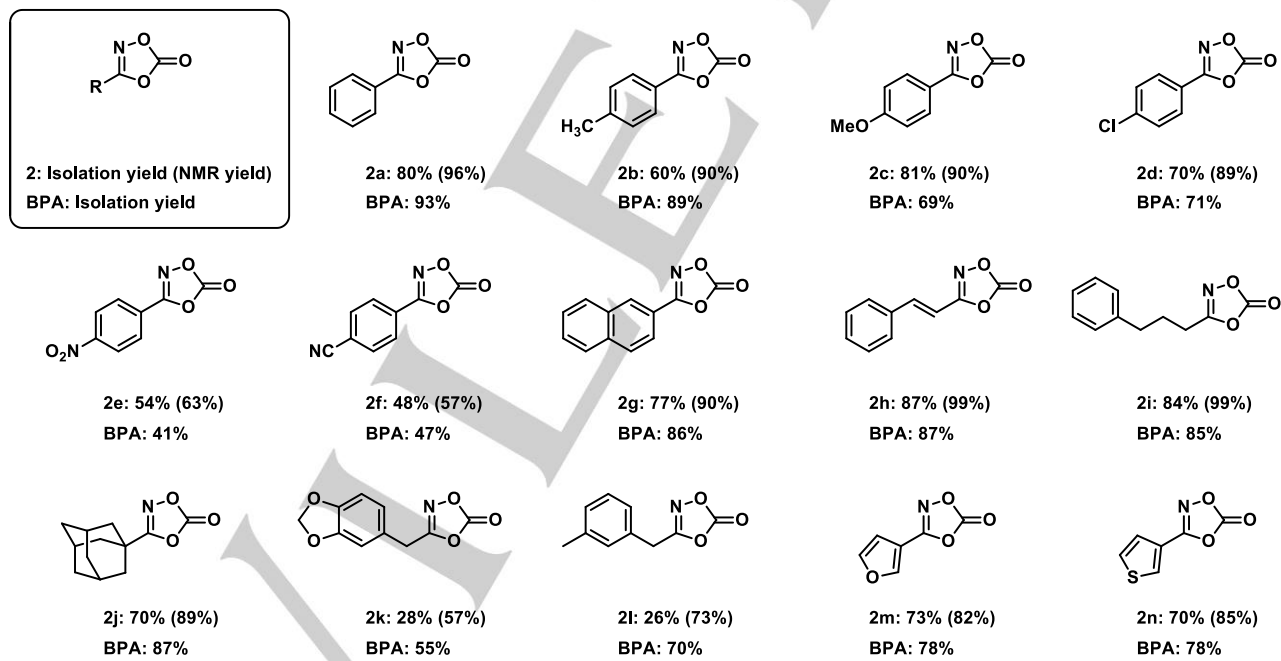
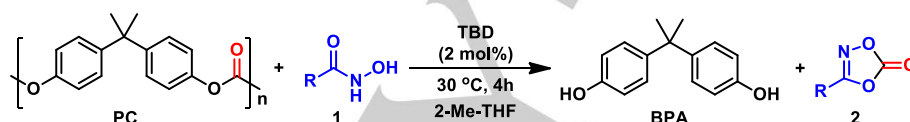
<sup>a</sup> Yields were determined by <sup>1</sup>H NMR using hexamethylbenzene as an internal standard.

## FULL PAPER



**Figure 1.** A)  $^1\text{H}$  NMR spectral changes in dimethyl protons in a BPA unit and B) molecular weight changes by SEC as a function of depolymerization time.

**Scheme 3.** Scope of dioxazolone synthesis beginning with PC.<sup>a</sup>



<sup>a</sup> Reaction conditions: PC (0.11 mmol, 1.0 equiv), **1** (0.12 mmol, 1.1 equiv), TBD (2 mol %), and 2-methyltetrahydrofuran (0.3 mL).

Using the optimized conditions, we explored the scope of PC depolymerization to dioxazolones and BPA with representative hydroxamic acids (Scheme 2). The reaction with phenyl hydroxamic acid (**1a**) afforded the corresponding phenyl dioxazolone (**2a**) in 96% yield via  $^1\text{H}$  NMR and 80% isolation yield. BPA recovery was recorded at 93% simultaneously. The positioning of electron-donating groups, such as methyl and

methoxy, afforded good conversion to the corresponding dioxazolones **2b** and **2c**. Both compounds achieved 90% yield as per  $^1\text{H}$  NMR but had different isolation efficiencies 60% and 81%, respectively. The BPA isolation rate were 89% and 69%, respectively. The effect of electron-withdrawing groups was prominent. Weakly withdrawing chlorine substitution exhibited minimal influence, as demonstrated by the 70% isolation yield of

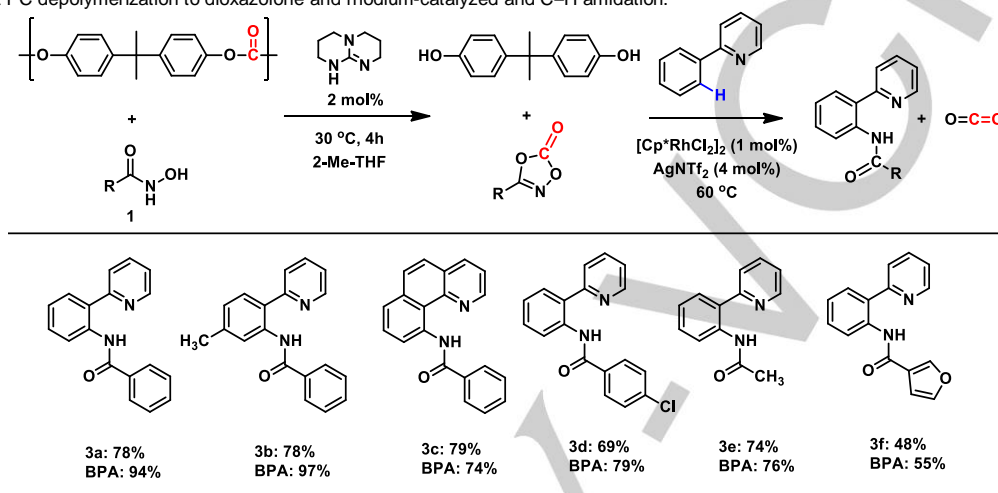


## FULL PAPER

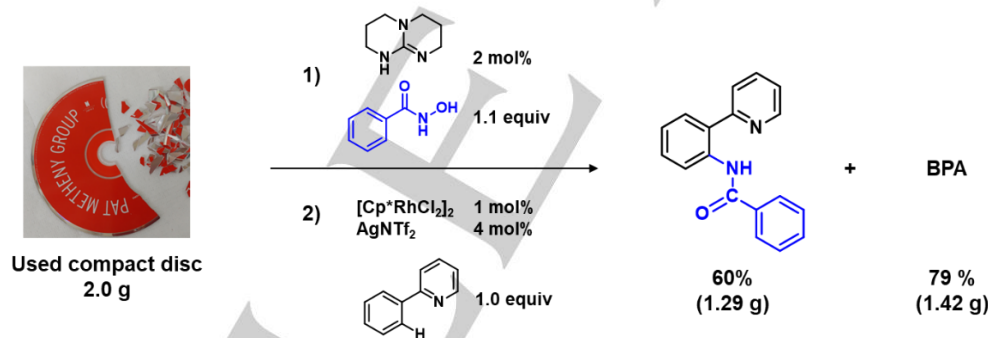
**2d.** Stronger substituents such as 4-nitro (**2e**) and 4-cyano (**2f**) groups resulted in reduced reactivity, 63% and 57%  $^1\text{H}$  NMR yields, and 54% and 48% isolation yields, respectively. Electron-withdrawing substituents reduced the nucleophilicity of the conjugated anion of hydroxamic acids, explaining their low efficiencies. The long conjugated moieties, namely the 2-naphthyl (**2g**) and cinnamic (**2h**) units, gave high yields. The aliphatic substituents produced mixed effects. Steric influence was

insignificant. Both primary alkyl and bulky adamantyl hydroxamic acids exhibited good conversions to the corresponding dioxazolones 99% (**2i**) and 89% (**2j**) respectively with good isolation yields. In contrast, the benzylic-type groups exhibited low conversions and isolation yields (**2k** and **2l**). Hydroxamic acids with heterocycles, herein furan and thiophene, converted BPA-PC into dioxazolones (**2m** and **2n**) smoothly.

**Scheme 4.** One-pot PC depolymerization to dioxazolone and rhodium-catalyzed and C–H amidation.



<sup>a</sup> Reaction conditions: step 1) PC (0.22 mmol, 1.0 equiv), **1** (0.24 mmol, 1.1 equiv), TBD (2 mol %), and 2-methyltetrahydrofuran (0.4 mL); step 2)  $[\text{Cp}^*\text{RhCl}_2]_2$  (1 mol %),  $\text{AgNTf}_2$  (4 mol %), and 2-phenyl pyridine (0.20 mmol, 1.0 equiv).



**Scheme 5.** Gram-scale upcycling of used PC to aryl amides.

The primary use of dioxazolone is direct C–H amidation.<sup>[42]</sup> With an appropriate catalyst, amides can be introduced in various transformations. To maximize the synthetic value of the chemical upcycling of PC, we evaluated tandem PC-depolymerization to dioxazolone and C–H amidations (Scheme 3). The original C–H amidation system developed by Chang was applied to a mixture of BPA and dioxazolone.<sup>[35,36]</sup> BPA and TBD did not significantly affect the  $\text{Cp}^*\text{Rh(III)}$  catalyst reactivity. Temperature adjustment to 60 °C was required while the original reaction was performed at room temperature. Combinations of 2-phenyl pyridine and hydroxamic acid derivatives exhibited promise in a one-pot recycling and amidation sequence. Yields in the range of 69–79% amide (**3a** – **3e**) were recorded with high BPA isolation yields. Only furanoic acid derived dioxazolone afforded low yields of amide (**3f**) and BPA. The upcycling of waste polycarbonates was tested using compact discs (Scheme 4). The one-pot

transformation was applied on a 2-gram scale, and produced **3a** in 60% and BPA in 79% isolation yields, respectively.

## Conclusion

In this study, PC was chemically recycled to dioxazolones and BPA in the presence of a TBD catalyst and hydroxamic acid. The highly reactive organocatalytic system was applied to a broad range of hydroxamic acids and produced a range of synthetically valuable dioxazolones. In addition to the recovery of environmentally harmful BPA, PC served as a green carbonyl source in place of CDI or phosgene. The resulting dioxazolones were subjected to one-pot C–H amidations. The  $\text{Cp}^*\text{Rh}$  catalyst maintained good efficiency under one-pot conditions. Real PC upcycling from used compact discs demonstrated the practicality

## FULL PAPER

of the dioxazolone formation and one-pot C–H amidation. Further PC upcycling to other synthetically valuable heterocycles is currently in progress.

## Experimental Section

**General experimental procedure for dioxazolone synthesis beginning with BPA-PC.** PC (0.028 g, 0.11 mmol of carbonate functionality), *N*-hydroxybenzamide (0.016 g, 0.12 mmol), TBD (0.30 mg, 0.0022 mmol, 2 mol%), 2-methyl THF (0.3 ml), and dibromomethane (7.7  $\mu$ l, 0.22 mmol, internal standard) were added to a 4 mL glass vial. The vial was placed in a preheated reaction block at 30 °C. After stirring for 4 h, a few drops of acetic acid were added to quench the reaction. An aliquot of the reaction mixture was used for <sup>1</sup>H NMR analysis. The crude mixture was directly purified by preparative thin layer chromatography with RP18F plates (hexane:acetone = 60:1) to afford BPA (23 mg, 93%) and 3-phenyl 1,4,2-dioxazol-5-one (29 mg, 80%).

**General experimental procedure for one-pot C–H amidation.** PC (0.056 g, 0.22 mmol of carbonate functionality), *N*-hydroxybenzamide (0.033 g, 0.24 mmol), TBD (0.60 mg, 0.0044 mmol, 2 mol%), and 2-methyl THF (0.4 ml) were added to a 4 mL glass vial. The vial was placed in a preheated reaction block at 30 °C. After stirring for 4 h, [Cp\*RhCl<sub>2</sub>]<sub>2</sub> (1.2 mg, 1.0 mol%), AgNTf<sub>2</sub> (3.1 mg, 4.0 mol%), and 2-phenylpyridine (31 mg, 0.20 mmol) were added to the mixture. The reaction mixture was stirred in a heating block at 60 °C for 15 h. The reaction was cooled to room temperature, filtered through a pad of celite, and then washed with CHCl<sub>3</sub>. The solvent was then removed under reduced pressure. The resulting mixture was purified by column chromatography (hexane:EtOAc from 99:1 to 70:30) to afford BPA (47 mg, 94%) and *N*-(2-pyridin-2-yl)phenylbenzamide (48 mg, 78%).

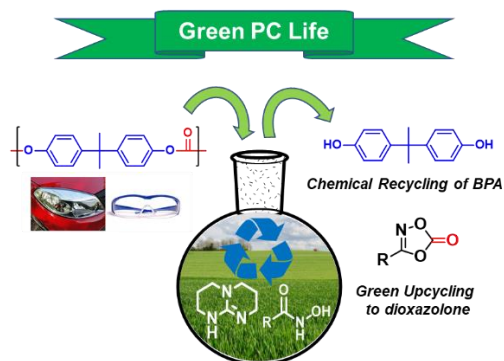
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**Keywords:** Chemical recycling • Upcycling • Polycarbonate • Dioxazolone • C-H amidation

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## Entry for the Table of Contents



Chemical upcycling of poly(bisphenol A carbonate) to synthetically valuable 1,4,2-dioxazol-5-one is realized. In addition to toxic bisphenol A recovery, waste PC replaces phosgene or its derivatives in a carbonylation reaction, boosting the metrics of green chemistry.

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