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# Phenyliodine(III) Diacetate Mediated Oxidative Cyclization of 1-Alkenoyl-1-carbamoyl Cycloalkanes: Access to Spiro-Fused Dihydrofuran-3(2*H*)-ones

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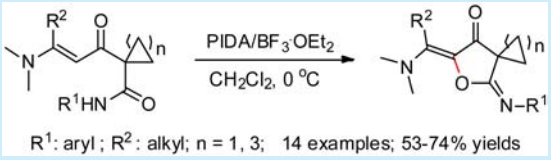
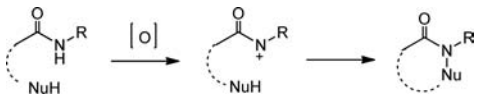
Supporting Information

**ABSTRACT:** Facile and efficient synthesis of spiro-fused dihydrofuran-3(2*H*)-ones was developed via phenyliodine(III) diacetate (PIDA) mediated oxidative cyclization of 1-alkenoyl-1-carbamoyl cycloalkanes under very mild conditions.




Hypervalent iodine(III) reagents have drawn considerable research attention in recent decades in view of their moderate oxidizing ability, required mild reaction conditions, low toxicity, and ready availability in comparison to that of heavy metal reagents.[1,2] Extensive work on the metal-free oxidation of a wide range of functionalities, such as alcohols, phenols, amines, amides, sulfides, and carbonyl compounds, has been reported.[3−7] Particularly, it has been demonstrated that the substituted amides can be easily oxidized by hypervalent iodine reagents to the corresponding *N*-acylnitrenium ions, which are powerful intermediates that may subsequently undergo inter- or intramolecular cyclization to form aza-heterocycles (Scheme 1).[6b,8,9]

**Scheme 1. Amidation Process Mediated by Hypervalent Iodine Reagents**



During the course of our studies on the synthesis of aza-heterocycles from *β*-oxo amide derivatives,[10−12] we investigated the reaction behaviors of *α*-oxo-ketene-*S*,*S*-acetals, cyclo-alkyloximes, enaminones, and *β*-oxo thioamides toward phenyliodine(III) bis(trifluoroacetate) (PIFA), respectively, and achieved efficient synthesis of substituted isothiazol-3(2*H*)-ones, spiro-fused cycloalkano-(C4)-pyrazolin-5-one *N*-oxides, pyrrolin-4-ones, and benzo[*d*]thiazoles. Most recently, we developed a one-pot synthesis of substituted 2,5-dihydrofurans from *β*-oxo amides and cinnamaldehydes in the presence of phenyliodine(III) diacetate (PIDA).[14] The above results reveal that there exists a chemoselective N−C or O−C bond formation during the oxidation of amides in the presence of varied hypervalent iodine reagents. In light of this, we envisaged that under appropriate conditions the oxidative process of 1-alkenoyl-1-carbamoyl cycloalkanes derived from *β*-oxo amides might lead to heterocyclization. After a series of investigations, we developed an efficient synthesis of spiro-fused dihydrofuran-3(2*H*)-ones via a chemoselective C−O bond formation. Herein, we wish to report our experimental results.

The substrates, 1-alkenoyl-1-carbamoyl cycloalkanes **1**, were prepared from *β*-oxo amides according to our previously reported procedure.[12b] Thus, we selected 1-[3-(dimethyl-amino)acryloyl]-*N*-phenyl cyclopentane carboxamide **1a** as the model compound and examined its behavior in the presence of a hypervalent iodine reagent. Upon treatment of **1a** with PIDA (1.5 equiv) and acetic acid (4.5 equiv) in dichloromethane (DCM) at room temperature for 5.0 h, the reaction could proceed sluggishly as indicated by TLC and furnished two products after workup and purification by silica column chromatography (Table 1, entry 1). The two products were characterized as 3-[(dimethylamino)methylene]-1-(phenylimino)-2-oxaspiro[4.4]nonan-4-one **2a** and 1-(dimethylamino)-3-oxo-3-[1-(phenylcarbamoyl)cyclopentyl]-prop-1-en-2-yl acetate **3a** based on its spectral and analytical data, respectively. In fact, the similar results in acetoxylation of acyclic *β*-enaminones in the presence of PIDA were reported recently by Chen and co-workers.[15] Thus, we next briefly examined the effect of different additives (acetic acid, TFA, $BF_3$·$OEt_2$), sources of hypervalent iodine (PIDA, PIFA), solvents (DCM, toluene, THF, acetonitrile), the loaded amount of hypervalent iodine or additive, and temperature on the success of the oxidative cyclization reaction to **2a**. As shown in Table 1, the experiments revealed that $BF_3$·$OEt_2$ was the most effective additive (entries 2−4), which had been extensively reported to increase the reactivity of hypervalent iodine reagents,[16] and the catalytic amount of addictive $BF_3$·$OEt_2$ turned out to be

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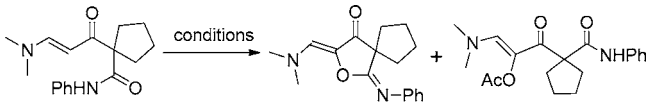
**Table 1. Selected Assays Performed on 1-Alkenoyl-1-carbamoyl Cycloalkane 1a**[a]




1a 2a 3a

| entry | oxidant (equiv) | additive (equiv) | solvent | temp (°C) | time (h) | yield (%)[b] |
|---|---|---|---|---|---|---|
| 1 | PIDA (1.5) | $CH_3COOH$ (3.0) | $CH_2Cl_2$ | rt | 5 | 23(26)[c] |
| 2 | PIDA (1.5) | $CH_3COOH$ (1.0) | $CH_2Cl_2$ | rt | 5 | mixture |
| 3 | PIDA (1.5) | TFA (1.0) | $CH_2Cl_2$ | rt | 3.0 | 60 |
| 4 | PIDA (1.5) | $BF_3{\cdot}OEt_2$ (1.0) | $CH_2Cl_2$ | rt | 1.0 | 65 |
| 5 | PIDA (1.5) | $BF_3{\cdot}OEt_2$ (0.3) | $CH_2Cl_2$ | rt | 1.0 | 64 |
| 6 | PIDA (1.5) | $BF_3{\cdot}OEt_2$ (0.1) | $CH_2Cl_2$ | rt | 12 | 55 |
| 7 | PIDA (1.5) | $BF_3{\cdot}OEt_2$ (0.3) | $CH_2Cl_2$ | reflux | 0.5 | 59 |
| 8 | **PIDA (1.5)** | **$BF_3{\cdot}OEt_2$ (0.3)** | **$CH_2Cl_2$** | **0** | **0.5** | **72** |
| 9 | PIFA (1.5) | $BF_3{\cdot}OEt_2$ (0.3) | $CH_2Cl_2$ | 0 | 1.0 | 56 |
| 10 | PIFA (1.5) | TFA (0.3) | $CH_2Cl_2$ | 0 | 1.0 | 68 |
| 11 | PIDA (1.0) | $BF_3{\cdot}OEt_2$ (0.3) | $CH_2Cl_2$ | 0 | 12 | 61 |
| 12 | PIDA (2.0) | $BF_3{\cdot}OEt_2$ (0.3) | $CH_2Cl_2$ | 0 | 0.5 | 63 |
| 13 | PIDA (1.5) | $BF_3{\cdot}OEt_2$ (0.3) | $CH_3CN$ | 0 | 2 | 22(49) |
| 14 | PIDA (1.5) | $BF_3{\cdot}OEt_2$ (0.3) | THF | 0 | 2 | 35(41) |
| 15 | PIDA (1.5) | $BF_3{\cdot}OEt_2$ (0.3) | toluene | 0 | 2 | 44(38) |

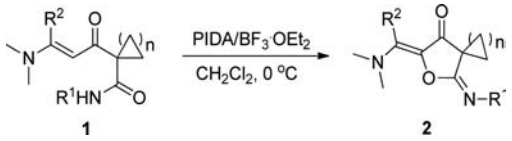
[a]Reagents and conditions: **1a** (1.0 mmol), solvent (10.0 mL). [b]Isolated yield of **2a** (data in parentheses for the recovery of **1a**). [c]28% yield of **3a**.

sufficient for the oxidative cyclization reaction (entries 5 and 6). It was observed that a higher temperature would result in a slightly low yield of **2a** (entry 7), whereas a higher yield of **2a** was obtained by decreasing the temperature to 0 °C (entry 8). The reaction could proceed in the presence of hypervalent iodine reagent PIFA (entries 9 and 10). The loaded amount of PIDA also had a significant influence on the reaction (entries 11 and 12). It should be mentioned that the nature of the solvent played a crucial role during the cyclization process (entries 13–15). The employment of polar aprotic solvents such as THF and acetonitrile afforded the desired product **2a** in low yields. Therefore, the optimal reaction conditions were obtained when 1-alkenoyl-1-carbamoyl cycloalkane **1a** was treated with PIDA (1.5 equiv) in DCM at 0 °C in the presence of $BF_3{\cdot}OEt_2$ (0.3 equiv), whereby the yield of **2a** reached 72% (entry 8).

Having established the optimal conditions for the oxidative cyclization process, we aimed to determine its scope with respect to the amide motif of 1-alkenoyl-1-carbamoyl cycloalkanes **1**. Thus, a series of reactions of substrates **1b–n** with PIDA and $BF_3{\cdot}OEt_2$ were carried out in DCM at 0 °C, and some of the results are summarized in Table 2. It was observed that all the reactions of 1-alkenoyl-1-carbamoyl cyclopentanes **1b–i** bearing electron-withdrawing/-donating aryl amide groups $R^1$ could proceed smoothly to afford the corresponding spiro-fused dihydrofuran-3(2*H*)-ones **2b–i** in good yields (entries 2–9). In the case of **1j** bearing a methyl group $R^2$ also rendered successfully the corresponding spiro-fused dihydrofuran-3(2*H*)-one **2j** in moderate yield (entry 10). The versatility of this spiro-fused dihydrofuran-3(2*H*)-one synthesis was further evaluated by performing 1-alkenoyl-1-carbamoyl cyclopropanes **1k–n** under identical conditions to afford the corresponding spiro-fused dihydrofuran-3(2*H*)-ones **2k–n** (entries 11–14). It should be mentioned that the structure of **2b** was further confirmed by X-ray single crystal analysis and its spectral and analytical data (Figure 1).

To further expand the scope of the oxidative cyclization protocol, we investigated the reaction of **1o** bearing a benzyl amide group $R^1$ in the same fashion. It was observed that the reaction proceeded smoothly, and a product was obtained after

**Table 2. Synthesis of Spiro-Fused Dihydrofuran-3(2*H*)-ones 2 from 1-Alkenoyl-1-carbamoyl Cycloalkanes 1**[a]




1 2

| entry | 1 | $R^1$ | $R^2$ | *n* | 2 | yield (%)[b] |
|---|---|---|---|---|---|---|
| 1 | **1a** | Ph | H | 3 | **2a** | 72 |
| 2 | **1b** | 4-$MeC_6H_4$ | H | 3 | **2b** | 68 |
| 3 | **1c** | 4-$ClC_6H_4$ | H | 3 | **2c** | 74 |
| 4 | **1d** | 4-$MeOC_6H_4$ | H | 3 | **2d** | 67 |
| 5 | **1e** | 2,4-$Me_2C_6H_3$ | H | 3 | **2e** | 69 |
| 6 | **1f** | 2-$MeC_6H_4$ | H | 3 | **2f** | 71 |
| 7 | **1g** | 2-$ClC_6H_4$ | H | 3 | **2g** | 66 |
| 8 | **1h** | 2-$MeOC_6H_4$ | H | 3 | **2h** | 62 |
| 9 | **1i** | 3-$ClC_6H_4$ | H | 3 | **2i** | 64 |
| 10 | **1j** | 4-$ClC_6H_4$ | Me | 3 | **2j** | 53 |
| 11 | **1k** | 4-$MeC_6H_4$ | H | 1 | **2k** | 65 |
| 12 | **1l** | 4-$ClC_6H_4$ | H | 1 | **2l** | 61 |
| 13 | **1m** | 2,4-$Me_2C_6H_3$ | H | 1 | **2m** | 73 |
| 14 | **1n** | 2-$MeOC_6H_4$ | H | 1 | **2n** | 60 |

[a]Reagents and conditions: **1** (1.0 mmol), $BF_3{\cdot}OEt_2$ (0.3 mmol), PIDA (1.5 mmol), DCM (10.0 mL), 0 °C, 25–45 min. [b]Isolated yield.

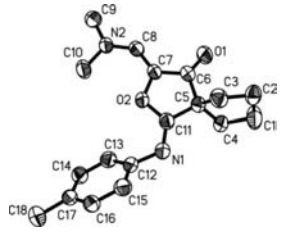



**Figure 1.** ORTEP drawing of **2b**.



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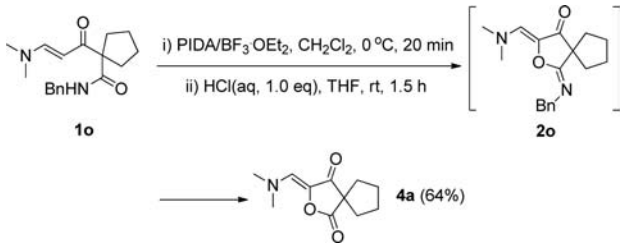
workup and purification of the resulting mixture, which was characterized as 3-[(dimethylamino)methylene]-2-oxaspiro-[4.4]nonane-1,4-dione **4a**. As indicated by TLC results, **2o** was unstable, especially during the workup process. Obviously, the formation of product **4a** is attributed to the hydrolysis of the intervening spiro-fused dihydrofuran-3(2*H*)-one **2o**. Thus, we envisaged a one-pot two-step procedure as shown in Scheme 2, by which **4a** was obtained in 64% yield. When (*E*)-1-

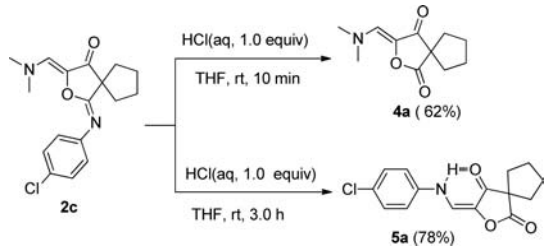
**Scheme 2. One-Pot Synthesis of Dihydrofuran-3(2*H*)-one 4a from 1o**




[3-(dimethylamino)acryloyl]-*N*-methylcyclopentane carboxamide **1p** was subjected to the conditions used for **2a** in Table 1, entry 8, the corresponding **4a** rather than **2p** was isolated, which is similar to the reaction of **1o**.

Encouraged by the above-mentioned results, we further examined the reactivity of spiro-fused dihydrofuran-3(2*H*)-ones **2** under acidic conditions. Thus, **2c** was selected as a model compound and treated with aqueous HCl (1.0 equiv) in THF. The reaction was completed within 10 min at room temperature as indicated by TLC results and furnished **4a** in 62% yield, whereas the reaction when performed at room temperature for 3.0 h could afford another product in 78% yield, which was characterized as 3-{[(4-chlorophenyl)amino]-methylene}-2-oxa spiro[4.4]nonane-1,4-dione **5a** (Scheme 3).

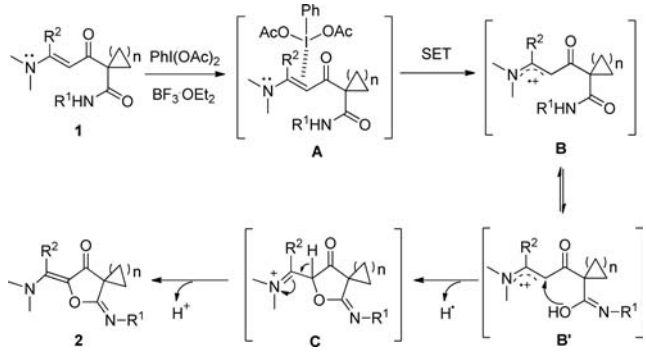
**Scheme 3. Reaction of Dihydrofuran-3(2*H*)-one 2c in Aqueous HCl**




Both products **4a** and **5a** contain the main framework of γ-lactones. Actually, γ-lactone and its analogues are an important class of oxa-heterocycles, which display a wide range of natural products along with biological activities[17,18] and are extensively used as building blocks in organic synthesis.[19]

As mentioned above, *N*-acyl nitrenium ions can be generated by the treatment of amides with hypervalent iodine reagents and, therefore, widely applied in the synthesis of a number of aza-heterocycles.[6b,8,9] In contrast, however, the preferential formation of spiro-fused iminolactones **2** over their counterpart spiro-fused lactams was achieved in the present work. This implies that the transformation might not arise from the intermediate *N*-acyl nitrenium ions. On the basis of the above-mentioned experimental results together with some reported literature, a mechanism for the synthesis of spiro-fused dihydrofuran-3(2*H*)-ones **2** is proposed as depicted in Scheme 4. In the presence of $BF_3{\cdot}OEt_2$, a CT-complex **A** is initially

**Scheme 4. Plausible Mechanism for the Reaction of 1 with PIDA/$BF_3{\cdot}OEt_2$**




formed from the enamine moiety of **1** with PIDA, which is oxidized via a SET process to afford a cation radical **B**.[16,20] The resonance interactions between the nitrogen atom and the carbonyl system in an amide promote accumulation of electronic density on the oxygen atom, which therefore becomes a more nucleophilic site.[21] Then, an intramolecular cyclization of the cation radical **B′**, i.e. resonance isomer of **B**, takes place by a one-electron oxidation to produce a cation intermediate **C**, followed by deprotonation to give rise to the final product spiro-fused dihydrofuran-3(2*H*)-one **2**.[14,21,22]

In summary, a facile and efficient synthesis of spiro-fused dihydrofuran-3(2*H*)-ones **2** is described via PIDA-mediated oxidative cyclization of 1-alkenoyl-1-carbamoyl cycloalkanes **1** with an intramolecular C−O bond formation. The hydrolysis of dihydrofuran-3(2*H*)-ones **2** to spiro-fused γ-lactones **4** and **5** is also achieved. The mild reaction conditions, simple execution, good yields, and synthetic potential of the products make this novel protocol very attractive. Further work on the mechanism of the reactions and the utilization and extension of the scope of the methodology is currently under investigation in our laboratory.

## ■ ASSOCIATED CONTENT

### Supporting Information

The Supporting Information is available free of charge on the ACS Publications website at DOI: 10.1021/acs.orglett.5b02485.

Experimental details, spectral and analytical data, copies of $^1$H NMR and $^{13}$C NMR spectra for new compounds **2**−**5** (PDF)

CIF data for **2b** (CIF)

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### Notes

The authors declare no competing financial interest.



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