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# Synthesis of Carbodiimides by $I_2$/CHP-Mediated Cross-Coupling Reaction of Isocyanides with Amines under Metal-Free Conditions

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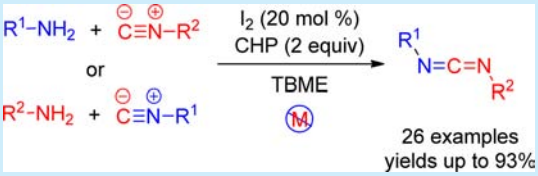
Supporting Information

**ABSTRACT:** An $I_2$/CHP-mediated cross-coupling reaction of isocyanides with readily accessible amines via C−N formation is described for carbodiimide synthesis in moderate to excellent yields. This represents a metal-free strategy for a coupling reaction of isocyanides with amines, and it provides an efficient approach for symmetric and unsymmetric functionalized carbodiimide derivative synthesis under mild conditions.




Carbodiimides such as *N,N′*-dicyclohexylcarbodiimide (DCC) and *N,N′*-diisopropylcarbodiimide (DIC) are some of the most important chemical reagents in organic chemistry,[1] and they have several useful applications including the synthesis of nucleotides, dehydration agents for peptide synthesis,[2] and intermediates or precursors for heterocycles.[3−5] In view of their high synthetic value, various methods have been developed for their preparation such as thermolysis−decarboxylation of isocyanates, dehydration of ureas, dehydrosulfurization of thioureas, and rearrangements of amidoximes.[6] However, these methods suffer from longer reaction time, toxic reagents, as well as poor atom economy. Palladium-catalyzed reactions of isocyanide insertion with amines provide an alternative strategy for the construction of *N,N′*-dialkylcarbodiimides with high atom economy.[7] It is interesting to note that Au particles could also catalyze similar reactions in micromolar scales.[8] However, these methods have the drawbacks of limited substrates, low reaction scales, and expensive catalysts, which limit their further applications. Therefore, the development of an efficient protocol to construct carbodiimides under metal-free and mild conditions is more desirable.

More recently, we have reported a Co-catalyzed isocyanide insertion reaction with 2-arylanilines under $O_2$ atmosphere for the creation of 6-aminophenanthridine derivatives.[9] Inspired by this previous work and in continuation of our work on isocyanide-based reactions, we herein report an $I_2$/CHP-mediated cross-coupling reaction of isocyanides with readily accessible amines via C−N formation to construct various carbodiimides in moderate to excellent yields (Scheme 1).

Our investigations began with the reaction of methyl 3-(2-aminophenyl)acrylate **1a** and *tert*-butyl isocyanide **2a** under different cobalt-catalyzed conditions. However, the reaction was messy, and no expected product was detected. To our surprise, carbodiimide **3aa** was formed in 12% GC yield instead of indole derivative product by the reaction **1a** and **2a** in the presence of 5 mol % of $I_2$ and 1 equiv of TBHP in 1,4-dioxane (Table 1, entry 4). Only trace amounts of **3a** were detected in the absence of catalyst or oxidant (Table 1, entries 1 and 8). Increasing the amount of $I_2$ to 20 mol % gave **3aa** in 42% GC yield (Table 1, entry 6). Other iodo-source catalysts such as TBAI, and NIS led to **3aa** in poor yields (Table 1, entries 2 and 3). Further screening of different oxidants, such as TBPB, DTBP, $O_2$, $K_2S_2O_8$, and CHP, showed that CHP was the best oxidant (Table 1, entries 9−13). Increasing the amount of CHP to 2 equiv furnished **3aa** in 69% GC yield (Table 1, entry 13). It should be noted that further reducing or increasing the amount of CHP resulted in a decrease in the yield of **3aa**. When the reaction was carried out in toluene, DMF, DCE, DME, and THF, respectively, **3aa** was observed in less than 46% GC yield in all cases (Table 1, entries 17−22). To our delight, the reaction in MTBE led to **3aa** in 77% GC yield (Table 1, entry 23). Further examination of the reaction temperature (Table 1, entries 24 and 25) showed **3aa** could be obtained in 81% GC yield (76% isolated yield) under reflux conditions (Table 1, entry 25). Therefore, the optimum reaction conditions feature 20 mol % of $I_2$ and 2 equiv of CHP as the oxidant in MTBE at 55 °C for 3 h (Table 1, entry 25, 81% for **3aa**).

**Scheme 1. Oxidative Reactions of Amine with Isocyanide**

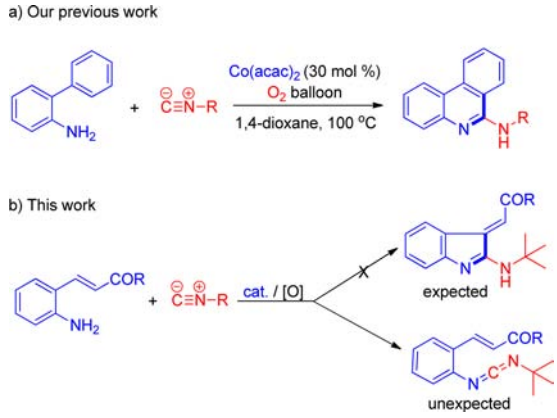



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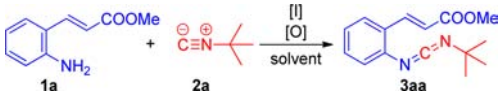
**Table 1. Optimization of Reaction of Methyl 3-(2-Aminophenyl)acrylate 1a and *tert*-Butyl Isocyanide 2a[a]**

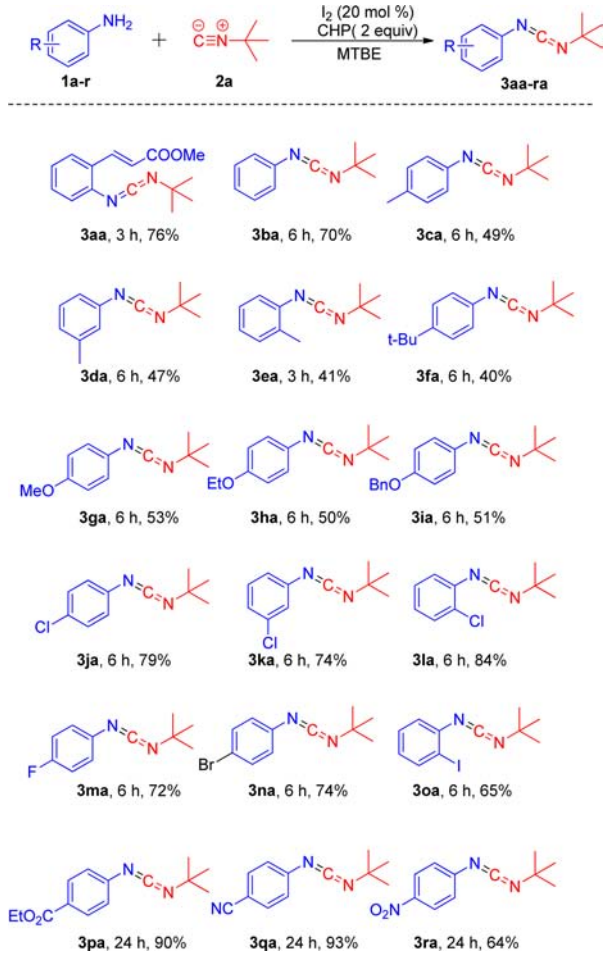



| entry | catalyst (mol %) | oxidant (equiv) | solvent | yield[b] (%) |
|---|---|---|---|---|
| 1 | | TBHP[e] (1) | 1,4-dioxane | trace |
| 2 | TBAI[c] (20) | TBHP (1) | 1,4-dioxane | trace |
| 3 | NIS[d](20) | TBHP (1) | 1,4-dioxane | 11 |
| 4 | $I_2$ (5) | TBHP (1) | 1,4-dioxane | 12 |
| 5 | $I_2$ (10) | TBHP (1) | 1,4-dioxane | 26 |
| 6 | $I_2$ (20) | TBHP (1) | 1,4-dioxane | 42 |
| 7 | $I_2$ (30) | TBHP (1) | 1,4-dioxane | 41 |
| 8 | $I_2$ (20) | Ar | 1,4-dioxane | trace |
| 9 | $I_2$ (20) | TBPB[f] (1) | 1,4-dioxane | 46 |
| 10 | $I_2$ (20) | DTBP[g] (1) | 1,4-dioxane | trace |
| 11 | $I_2$ (20) | $O_2$ | 1,4-dioxane | trace |
| 12 | $I_2$ (20) | $K_2S_2O_8$ (1) | 1,4-dioxane | trace |
| 13 | $I_2$ (20) | CHP[h] (1) | 1,4-dioxane | 53 |
| 14 | $I_2$ (20) | CHP (0.5) | 1,4-dioxane | 18 |
| 15 | $I_2$ (20) | CHP (2) | 1,4-dioxane | 69 |
| 16 | $I_2$ (20) | CHP (3) | 1,4-dioxane | 22 |
| 17 | $I_2$ (20) | CHP (2) | toluene | trace |
| 18 | $I_2$ (20) | CHP (2) | DMF[i] | trace |
| 19 | $I_2$ (20) | CHP (2) | MeCN | 20 |
| 20 | $I_2$ (20) | CHP (2) | DCE[j] | 22 |
| 21 | $I_2$ (20) | CHP (2) | DME[k] | 46 |
| 22 | $I_2$ (20) | CHP (2) | THF[l] | 26 |
| 23 | $I_2$ (20) | CHP (2) | MTBE[m] | 77 |
| 24[n] | $I_2$ (20) | CHP (2) | MTBE | 22 |
| 25[o] | $I_2$ (20) | CHP (2) | MTBE | 81 |

[a]Reaction conditions: methyl 3-(2-aminophenyl)acrylate **1a** (0.5 mmol) *tert*-butyl isonitrile **2a** (1.2 equiv, 0.6 mmol), catalyst, and oxidant in 3 mL of solvent at 110 °C for 3 h. [b]Yields were determined by GC analysis with biphenyl as the internal standard. [c]TBAI = tertabutylammonium iodide. [d]NIS = *N*-iodosuccinimide. [e]70% TBHP solution in water, TBHP = *tert*-butyl hydroperoxide. [f]TBPB = *tert*-butyl peroxybenzoate. [g]DTBP = 2-(*tert*-butylperoxy)-2-methylpropane. [h]CHP = cumene hydroperoxide. [i]DMF = *N*,*N*-dimethylformamide. [j]DCE = 1,2-dichloroethane. [k]DME = ethylene glycol dimethyl ether. [l]THF = terahydrofuran. [m]MTBE = methyl *tert*-butyl ether. [n]The system was carried out at room temperature. [o]The system was carried out at 55 °C.

To explore the scope and limitations of this approach, various anilines **1a**−**r** were applied to the reaction with *tert*-butyl isocyanide **2a** under the optimized metal-free conditions, and the results are summarized in Scheme 2. Most of the reactions proceeded smoothly to afford the desired carbodiimide products in moderate to excellent yields. The reaction of aniline **1b** with **2a** afforded the desired product **3ba** in 70% yield. Anilines **1c**−**e** with an *o*-, *m*-, or *p*-methyl group could be converted to the corresponding carbodiimides **3ca**−**ea** in moderate yields (41−49%). Typically, functional groups including electron-donating *tert*-butyl, OMe, OEt, OBn (**1f**−**i**) and electron-withdrawing groups such as CN, COOEt, $NO_2$, **1q**, **1p**, **1r** were tolerated under identical conditions. However, anilines bearing electron-withdrawing groups showed less reactivity, and the reaction time should be extended to 24 h. When halogen-substituted anilines **1j**−**o** were subjected to the reactions, the desired carbodiimides **3ja**−**oa** were observed in good yields (65%−84%). It is worth noting that more bulky substrates **1e**, **1l**, and **1o** could also undergo the transformation to furnish the desired products **3ea**, **3la**, and **3oa** in 41%, 84%, and 65% yields, respectively. Unfortunately, the reaction of pyridin-2-amine **1s** could only give the corresponding product **3sa** in trace yield.

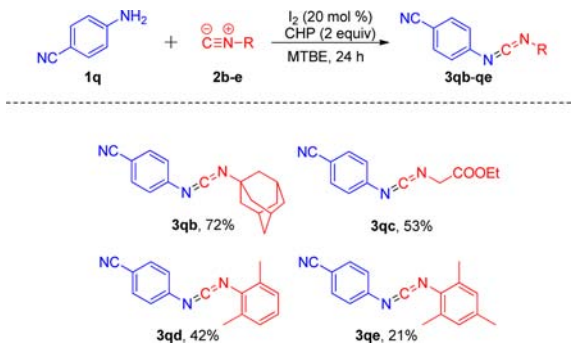
**Scheme 2. Scope of Anilines 1a−s[a,b]**




[a]Reaction condition: anilines **1a**−**r** (0.5 mmol), *tert*-butyl isonitrile **2a** (1.2 equiv, 0.6 mmol), $I_2$ (20 mol %), and CHP (2 equiv) in 3 mL of MTBE at 55 °C. [b]Isolated yields.

Next, we investigated the scope of isocyanides (Scheme 3). The reaction of 4-aminobenzonitrile **1q** with adamantanyl isocyanide **2b** performed well under the optimal conditions and led to the desired product **3qb** in 72% yield. When the functional isocyanide **2c** was used, we could also obtain the carbodiimide **2qc** in 53% yield. When more bulky aryl

**Scheme 3. Scope of Isocyanides 2b−d with 4-Aminobenzonitrile 1q**



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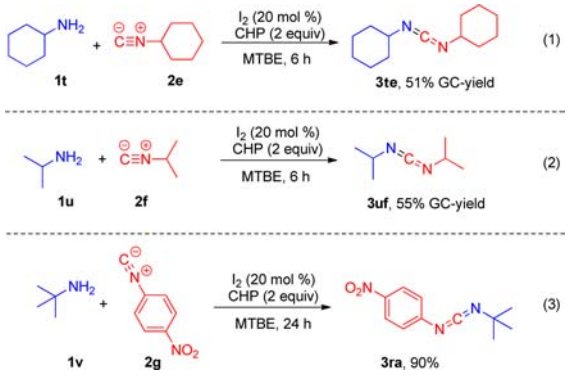
isocyanides **2d,e** were subjected to the reactions, the reactions could also furnish carbodiimide derivatives **3qc,qd** in 21% and 42% yields, respectively.

The reaction is extendable to construct some useful carbodiimides such as DCC and DIC by the reaction of aliphatic amines with aliphatic isocyanides under the optimal conditions. The reaction of cyclohexanamine **1t** with cyclohexyl isocyanide **2e** proceeded well to furnish symmetric carodiimide DCC **3te** in 51% GC yield (Scheme 4, eq 1). Similarly, DIC **3uf** could also be

**Scheme 4. Select Carbodiimides Synthesis by the Reaction of Isocyanides with Amines**



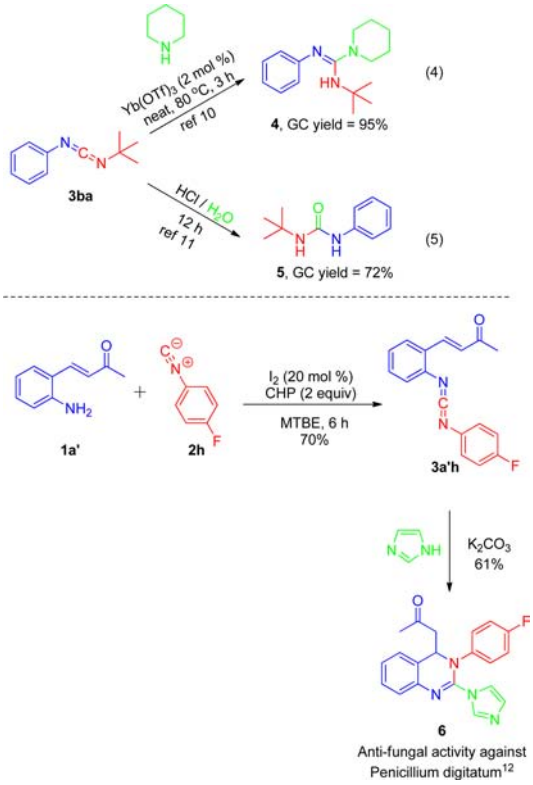

obtained in 55% GC yield by the reaction of propan-2-amine **1u** with isopropyl isocyanide **2f** (Scheme 4, eq 2). Carbodiimide **3ra** could also be generated in 90% yield by the reaction of **1v** with **2g** (Scheme 4, eq 3). This result indicated that the unsymmetric carbodiimide could be prepared by two different sets of the corresponding amine and isocyanide.

To further demonstrate the utility of our above reactions, we applied the carbodiimides in other transformations (Scheme 5). For example, the reaction of *N*-((*tert*-butylimino)methylene)-aniline **3ba** with piperidine in the presence of a catalytic amount of $Yb(OTf)_3$ easily led to the trisubstituted guanidine **4** in 95% GC yield (Scheme 5, eq 4).[10] When **3ba** was treated with water, urea **5** could be formed in 72% GC yield (Scheme 5, eq 5).[11] Our protocol has also been successfully applied in the total synthesis of 1-(3-(4-fluorophenyl)-3,4-dihydro-2-(1*H*-imidazol-1-yl)-quinazolin-4-yl)propan-2-one **6**, which shows good to significant fungicidal activity against *Penicillium digitatum* and strong binding interaction with the cytochrome P450-dependent sterol 14$\alpha$-demethylase (CYP51).[12] $I_2$/CHP mediated cross-coupling reaction of isocyanide **2h** insertion reaction with amine **1a′** gave carbodiimide **3a′n** in 70% yield. Following the reaction of **3a′h** with imidazole in the presence of $K_2CO_3$ furnished **6** in 61% yield (Scheme 6). Compound **6** could be easily prepared over two steps based on our method instead of 5 steps as reported in the literature.

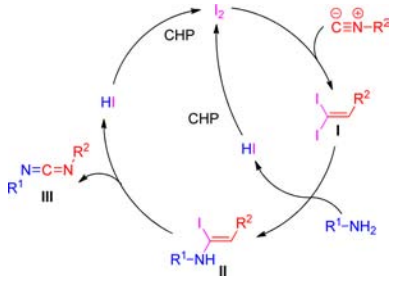
On the basis of the above results, a plausible reaction mechanism is proposed in Scheme 6. The reaction of isocyanide with iodine via 1,1-addition affords intermediate **I**.[13] Compound **I** reacts with amine to give intermediate **II** by dehydrohalogenation. Following the subsequent second time dehydrohalogenation, carbodiimide **III** is formed. Hydrogen iodide is oxidized by CHP to furnish iodine and complete the catalytic cycle.

In conclusion, we have developed a novel $I_2$/CHP-mediated cross-coupling reaction of isocyanides with readily accessible amines via C−N bond formation, affording carbodiimides in moderate to excellent yields under metal-free conditions. This

**Scheme 5. Transformations of Carbodiimides and Total Synthesis of Antifungal Activity Molecule 6**




**Scheme 6. Proposed Mechanism**




work not only represents a metal-free strategy for cross-coupling of isocyanides with amines but also provides an efficient approach to symmetric and unsymmetric functionalized carbodiimides under mild conditions. Further studies to understand this isocyanide insertion mechanism and their other synthetic applications are ongoing in our laboratory.

## ■ ASSOCIATED CONTENT

### Supporting Information

Detailed experimental procedures and characterization data. This material is available free of charge via the Internet at http://pubs.acs.org.

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### Notes

The authors declare no competing financial interest.



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