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Copper-Catalyzed Cross-Coupling of Acylzirconocenes and Diaryliodonium Salts: An Efficient Synthesis of Alkyl-aryl-ketones from Alkenes

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An efficient and unprecedented cross-coupling between acylzirconocenes, readily available starting materials conveniently prepared by hydrozirconation of alkenes and a subsequent carbonylation with carbon monoxide, and diaryliodonium tetrafluoroborates is reported. This procedure enables the synthesis of a broad variety of alkyl-aryl-ketones upon simple catalysis with copper cyanide without the need of additional ligands and only requires a low pressure of carbon monoxide generated *in situ*, in a two-chamber reactor, from *N*-formylsaccharin.

Introduction

Since the discovery of the use of zirconocene hydrochloride, Cp_2ZrHCl, for the functionalization of olefins by Hart and Schwartz in 1974,[1] a variety of reactions using organozirconocene complexes have been developed, notably enabling the design of innovative and efficient processes for the formation of carbon-carbon bonds.[2] One of the most interesting, but still understudied, transformation of organozirconocenes is certainly their carbonylation to the corresponding acyl zirconium complexes **2**.[3] Such intermediates can be readily obtained by a one-pot hydrozirconation with the Schwartz reagent/carbonylation under a low pressure of carbon monoxide from alkenes **1** or alkynes, and they were shown to be remarkably versatile intermediates acting as acyl anion equivalents which can be smoothly converted to the corresponding aldehydes, acyl halides or esters.[4] They are in addition excellent reagents for carbon-carbon bond forming reactions since they readily add, for example, to a range of electrophiles **3** such as ketones, imines[5] and enones,[6] to generate the corresponding α-hydroxy ketones, α-amino-ketones and 1,4-diketones, respectively (Figure 1, top).

More recently, and in addition to these reactions, the reactivity of acylzirconocene complexes **2** as acyl anions and their easy transmetallation to various transition metals[7] has been elegantly exploited for the development of novel cross-coupling reactions enabling the direct introduction of carbonyl group from nucleophilic precursors (Figure 1, middle), an interesting alternative strategy compared to more traditional approaches based on electrophilic acyl species. In this perspective, palladium catalysis has clearly contributed to expand the reactivity of acyl zirconium complexes towards a much broader range of electrophiles and, hence, their synthetic usefulness.[8] Although efficient, the main limitation of the catalytic systems utilized lies in their cost and there is clearly a strong need for alternative systems based on more available non-noble metal, which could moreover further expand the chemistry of acylzirconium complexes.[9] In this perspective, copper complexes are especially appealing. However, since the first report of transmetallation of an organozirconocene to copper by Schwartz in 1977,[10] only a limited number of processes have been reported with acylzircononcenes, despite the strong potential of their transmetallation to copper for the generation of acylcopper species. Examples reported so far on the use of

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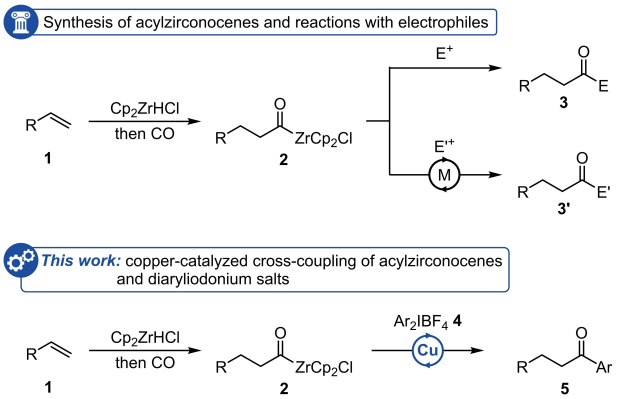

Figure 1. Cross-coupling of acylzirconocenes and electrophiles: state of-the-art and current work.

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copper catalysis with acylzirconocene complexes include their cross-coupling reactions with activated electrophiles such as allylic and propargylic halides[11] or alkynyliodonium tosylates,[12] and their addition to α,β-enones, allenyl ketones,[13] and *N*-acyl-isoquinolinium chlorides.[14]

One of the main limitations for the further development of such unique reagents most probably comes from difficulties associated to the safe handling of carbon monoxide. This issue was however efficiently addressed by the Skrydstrup group who designed a set of efficient and safe procedures for the *in situ* generation of carbon monoxide in a two-chamber system.[15] This, combined with our joint interests in copper catalysis[16,17] and carbonylation chemistry,[18] prompted us to investigate this procedure for the safe generation of acylzirconocenes **2** from alkenes **1** and their use in copper-catalyzed cross-coupling with diaryliodonium salts[19,20] **4**, which would, in addition to further expend the usefulness of acylzirconocenes in synthesis, provide an efficient entry to alkyl-aryl-ketones **5** from readily available alkenes as starting materials (Figure 1, bottom).

Results and Discussion

Reaction Setup and Optimization

We first began our investigations using Skrydstrup's COware two-chamber reactor[15] depicted in Figure 2. To first assess the hydrozirconation/carbonylation steps, 4-phenyl-1-butene **1a** was selected as a model substrate and was placed in the CO-consuming chamber A together with Schwartz reagent Cp_2ZrHCl while the CO-generating chamber B was charged with *N*-formylsaccharin **6**, a readily available crystalline compound which was selected as the CO-surrogate.[21] A brief evaluation of the influence of both the solvent and the temperature[22] revealed that the hydrozirconation was best performed in 1,2-dichloroethane at 60 °C for 2 hours followed by a carbonylation, initiated by the addition of triethylamine and 1,2-dichloroethane to the CO-generating chamber B, at room temperature for an additional 2 hours. Under these conditions, linear aldehyde **7** could be obtained as a single regioisomer and in 76% NMR yield upon acidic hydrolysis, while other conditions resulted in the formation of minor amounts of branched aldehyde **7'**, thus demonstrating the feasibility of the hydrozirconation/carbonylation sequence.

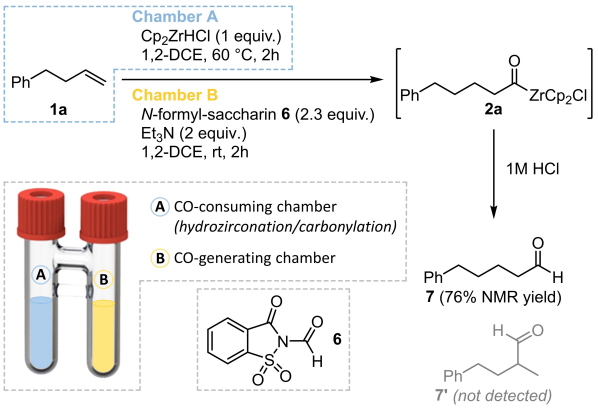

Figure 2. Reaction setup with the COware two-chamber system and optimized conditions for the hydrozirconation/carbonylation steps.

With these optimized setup and conditions for the hydrozirconation/carbonylation steps, we next focused our efforts on the evaluation of the copper-catalyzed cross-coupling with diaryliodonium salts and its optimization. A set of representative sources of copper(I) commonly used in copper catalysis was thus evaluated using *in situ*-generated acylzirconocene **2a** and diphenyliodonium tetrafluoroborate **4a** as model substrates. Results from this screening are shown in Figure 3 and demonstrate the feasibility of our working hypothesis as well as the superiority of copper(I) cyanide as a catalyst. Using 20 mol% of CuCN indeed promoted the cross-coupling to ketone **5a** in 84% NMR and isolated yields. Importantly, **5a** was obtained as a single regioisomer resulting from a fully regioselective hydrozirconation since no branched isomer could be detected in the crude reaction mixture. As an important note, while the use of an excess of acylzirconocene **2a** gave the best result, the cross-coupling is still operative, although less efficient, using a 1:1 ratio of **2a** compared to **4a** since **5a** was obtained in 70% NMR yield in this case, an important point when starting from less available alkenes.

Scope and limitations

With the optimized reaction conditions in hand, we then explored the scope of the process, first with various acylzircono-

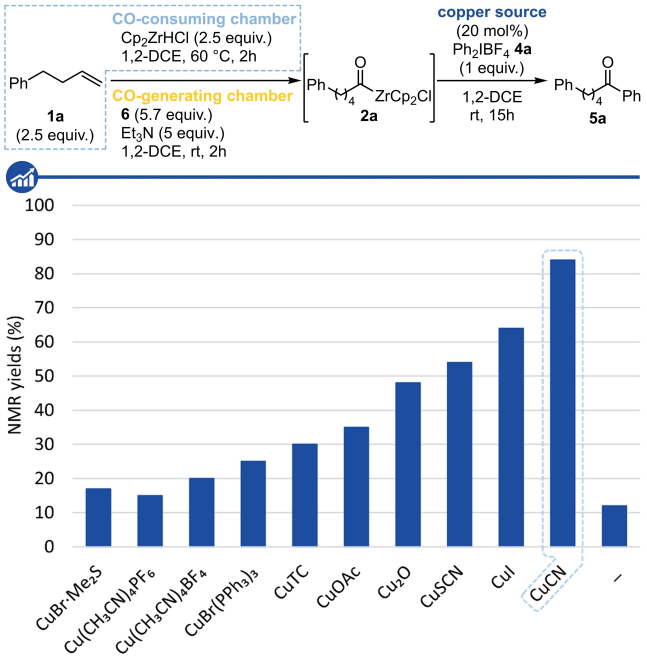

Figure 3. Optimization of the copper-catalyzed cross-coupling of *in situ*-generated acylzirconocenes with diaryliodonium salts. *^{1}H NMR yields obtained using 1,3,5-trimethoxybenzene as an internal standard; TC = thiophene-2-carboxylate.*

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cenes **2** *in situ*-generated from the corresponding alkenes **1** and using diphenyliodonium tetrafluoroborate **4a** as the model arylating agent. As shown with results collected in Figure 4, the scope of the hydrozirconation/carbonylation/arylation sequence was found to be rather broad, the corresponding aryl-alkyl-ketones **5** being obtained in good to excellent yields and as single linear regioisomers, except in some peculiar cases. Simple acylzirconocenes derived from terminal alkenes such as 4-phenyl-1-butene, allylbenzene, 4-allyl-1,2-dimethoxybenzene, vinylcyclohexane or 1-dodecene were readily coupled with diphenyliodonium tetrafluoroborate to give the corresponding unsymmetrical ketones **5a**–**5e** with yields in the 65–84% range. The presence of an aryl ether, a thioether, a chloride or a silyl group had a limited effect on the efficiency of the process, the corresponding ketones **5f**, **5g**, **5h** and **5i** being isolated in 67, 53, 67 and 77% yield, respectively.

Gratifyingly, the reaction could be extended to the use of geminal di-substituted alkenes such as 2-methylhept-1-ene, although the corresponding ketone **5j** was obtained with a lower yield of 64%, mostly due to a more difficult hydrozirconation. Readily available cyclopentene and cyclohexene were also found to be efficient starting materials yielding to aryl-cycloalkyl-ketones with different ring sizes **5k** and **5l** in 72% and 75% yields, respectively. Unfortunately, the sequence failed when starting from less reactive and more hindered alkenes such as stilbene and trisubstituted alkenes, most certainly due to a too slow and unfavorable hydrozirconation,[2a] the corresponding ketones **5m**–**p** being undetectable in crude reaction mixtures.

When starting from aryl-substituted alkenes, the sequence was found to strongly depend on the nature of the aromatic ring. Indeed, when using styrene and 4-vinyl-toluene, the desired aryl-homobenzyl-ketones **5q** and **5r** were obtained in good yields (75 and 82% yields, respectively), although they were formed in this case together with minor amounts of their inseparable branched isomers **5q'** and **5r'** with linear/branched ratio of 85:15 and 75:25, respectively. For such conjugated alkenes, hydrozirconation usually favors the formation of linear alkylzirconocenes in similar ratio.[23] Switching to 1-vinylnaphthalene and 9-vinylanthracene however restored a full regioselectivity, which might result from steric effects, the corresponding ketones **5s** and **5t** being obtained as sole regioisomers, with reduced yields, however (69% and 52%, respectively).

Having studied in detail the scope of our process with respect to the nature of the acylzirconocene, we next focused our efforts on the influence of the diaryliodonium salt. As show from results summarized in Figure 5, the scope with respect to diaryliodonium tetrafluoborates was found to be fairly broad and the presence of a substituent at their *ortho*, *meta* or *para* positions had virtually no impact on the outcome of the cross-coupling, as exemplified with the synthesis of **5u**–**x** in comparable yields. Halogen atoms, which represent useful handles for post-functionalization, were well-tolerated and left untouched, aryl-alkyl-ketones **5z**, **5aa**, **5ab** and **5ac** being obtained in fair to good yields. Other electron-withdrawing groups such as a trifluoromethyl group (**5ad**) or a methyl ester (**5ae**) also worked well, as well as an electron-donating methyl ether (**5af**), although with a slightly reduced efficiency.

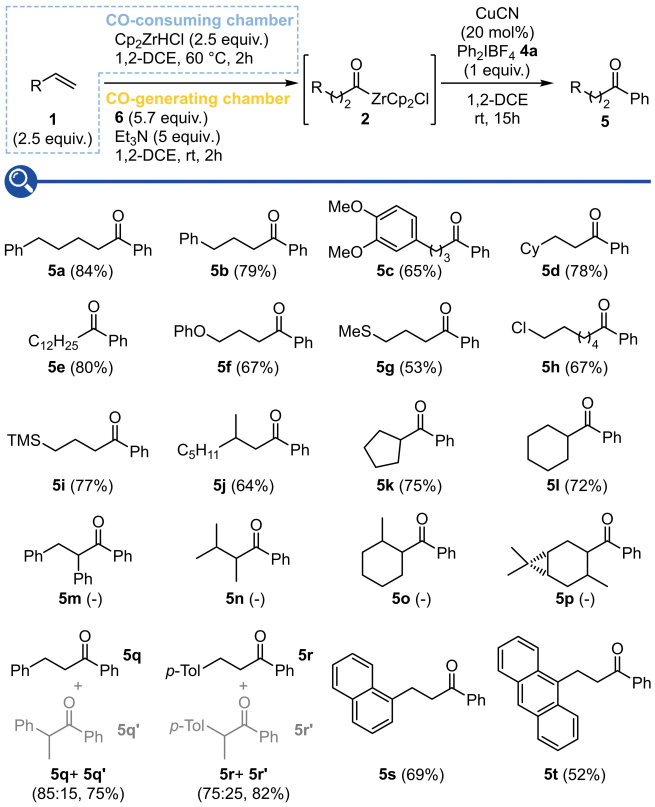

Figure 4. Scope of the copper-catalyzed cross-coupling of acylzirconocenes with diaryliodonium salts: acylzirconocenes.

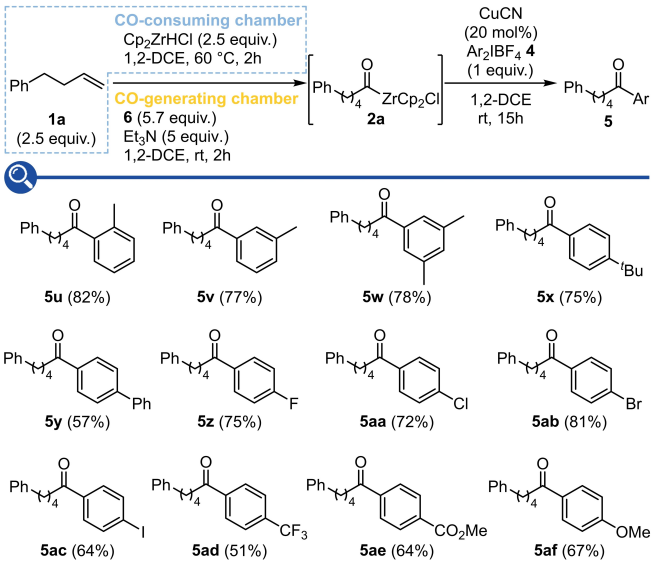

Figure 5. Scope of the copper-catalyzed cross-coupling of acylzirconocenes with diaryliodonium salts: diaryliodonium salts.

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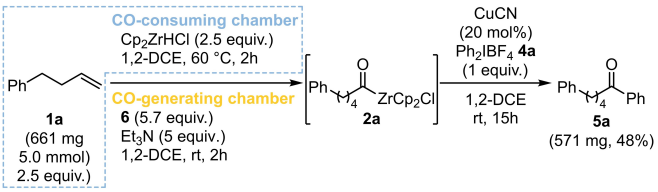
Scale-up

Moreover, a scale-up of the reductive carbonylative arylation was also explored using 4-phenyl-1-butene **1a** and diphenyliodonium tetrafluoroborate **4a**, as depicted in Scheme 1. Using a two-chamber reactor of 100 mL and performing the reaction on a 5.0 mmol scale, ketone **5a** was isolated in 48% yield, a yield that was not further optimized but still demonstrate the possibility of a scale-up. In this case, the lower yield observed on a bigger scale is most probably due to a less efficient diffusion of carbon monoxide in a greater amount of solvent.

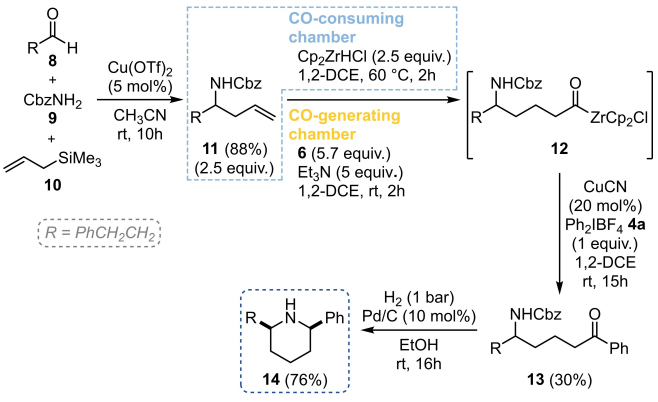
Application to the synthesis of a *cis*-2,6-disubstituted piperidine

Lastly, in a further effort to demonstrate the synthetic potential of our process and inspired by the seminal work of the Szymoniak group on the synthesis of *cis*-2,6-disubstituted piperidines via a hydrozirconation/acylation/intramolecular reductive amination sequence,[24] we sought to target this biologically relevant structure commonly found in a variety of natural products and drugs.

In this perspective, we could apply our procedure to a three-step synthesis of *cis*-2,6-disubstituted piperidine **14**, in a complementary approach to the one of Szymoniak based on the use of an acylzirconocene. The starting homoallylic amine **11** could be readily obtained in good yields by a copper-catalyzed allylation of an imine generated *in situ* from 3-phenylpropionaldehyde **8** and benzyl carbamate **9** with allyltrimethylsilane **10** (Scheme 2). We then used our optimized protocol on protected homoallylamine **11** and isolated the desired ketone **13** in a modest 30% yield that could be attributed to undesired reactions of the CBz protecting group with the strongly reducing zirconium hydride. The final deprotection/cyclization/reduction sequence was then achieved in a single step by simple hydrogenation over Pd/C and furnished the desired *cis*-2,6-disubstituted piperidine **14** in a fair 76% yield and as a single diastereoisomer.



Scheme 1. Scale-up of the copper-catalyzed cross-coupling of acylzirconocenes with diaryliodonium salts.



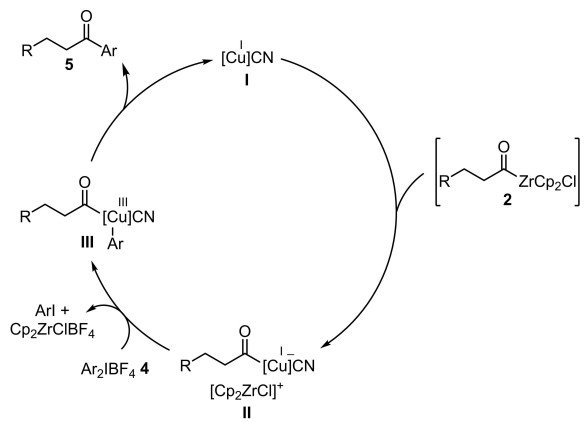
Scheme 2. Application of the copper-catalyzed cross-coupling of acylzirconocenes with diaryliodonium salts to the synthesis of a *cis*-2,6-disubstituted piperidine.

Proposed Catalytic Cycle

Based on mechanisms typically proposed for copper-catalyzed cross-coupling reactions,[25] notably involving diaryliodonium salts, a proposed catalytic cycle that could account for our copper-catalyzed cross-coupling of acylzirconocenes with diaryliodonium salts is shown in Scheme 3. It would start by a transmetallation of the acylzirconocene to copper, generating an acylcopper(I) intermediate **II**. Oxidative addition of the diaryliodonium tetrafluoroborate would yield a transient arylacylcopper(III) **III** species whose reductive elimination would finally close the catalytic cycle and afford the desired aryl-alkyl-ketone **5**.

Conclusions

In conclusion, we have developed an efficient and unprecedented cross-coupling between acylzirconocenes, readily available starting materials conveniently prepared by hydrozirconation of alkenes and a subsequent carbonylation with carbon monoxide, and diaryliodonium tetrafluoroborates. This procedure enables the synthesis of a broad variety of alkyl-aryl-ketones upon simple catalysis with copper cyanide without the need of additional ligands and only requires a low pressure of carbon monoxide generated *in situ*, in a two-chamber reactor, from *N*-formylsaccharin. In addition to providing a straightforward entry to alkyl-aryl-ketones, it also brings useful insights into the unique reactivity of acylzirconocenes under copper catalysis.



Scheme 3. Proposed, simplified catalytic cycle for the copper-catalyzed cross-coupling of acylzirconocenes with diaryliodonium salts.

Experimental Section

Supporting Information: Experimental details and copies of the ^{1}H and ^{13}C NMR spectra of all new compounds.

Supporting Information

Additional references cited within the Supporting Information.[26–29]

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Conflict of Interests

The authors declare no conflict of interest.

Data Availability Statement

The data that support the findings of this study are available in the supplementary material of this article.

Keywords: acylzirconocenes · carbonylation · copper catalysis · diaryliodonium salts · hydrozirconation

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