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Carbonylative Negishi-type Coupling of 2-iodoglycals with Alkyl and Aryl Halides

Henrique A. Esteves,[a]* Mariana P. Darbem,[a] Daniel C. Pimenta,[b] Hélio A. Stefani.[a]*

Abstract. *C*-glycosides are valuable organic compounds in the field of medicinal chemistry due to their ubiquity inside living systems and pronounced biological activity. Herein, we describe an approach to alkyl-ketones bearing glycal units via the Pd-catalyzed carbonylative coupling of 2-iodoglycals and alkyl and aryl halides. Examples bearing a variety of functional groups are presented as well as a mechanistic proposal for this transformation.

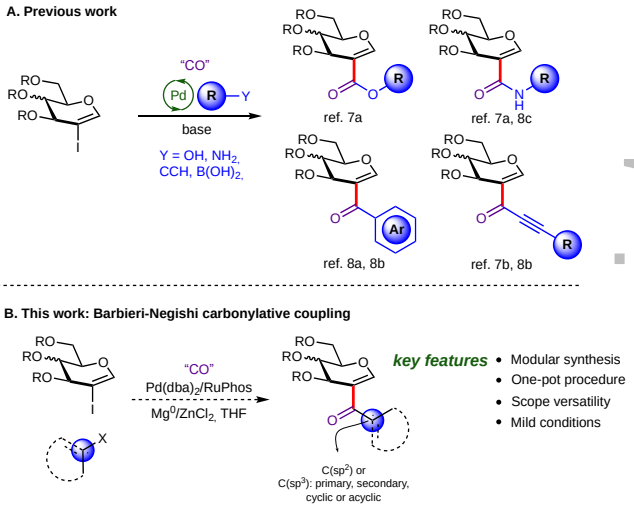
Introduction

In the field of carbohydrate chemistry, a *C*-glycoside is a class of molecules in which a carbohydrate unit is connected to an aglycone by a C–C bond, a modification that directly impacts its chemical stability in a biological environment.[1] These compounds are found in a variety of biologically active natural products, as well as in important therapeutic agents, making them desirable targets.[2]

Given their importance, many strategies have been developed for *C*-glycosylation and, among them, some of the most attractive involve the functionalization of unsaturated sugars derivatives, known as glycals.[3] Typical approaches rely on classical cross-coupling procedures, such as Heck's,[4] Suzuki-Miyaura's[5] and Stille's.[6]

Our group[7] and others,[8] on the other hand, have been focusing on the use of carbonylative cross-coupling reactions of 2-iodoglycals for glycal functionalization. In this approach, the carbohydrate unit is connected to different nucleophiles through a carbonyl link originated from a carbon monoxide molecule. This approach permits not only a rapid connection of two fragments, but also gives rise to a rich variety of functional groups (Scheme 1a).[9]

While carbonylative reactions to obtain amides,[10] esters,[11] aryl-ketones[12] and alkynones[13] are widespread, those aiming alkyl-ketones[14] are much more limited, due to the challenges associated with making C(sp^2)–C(sp^3) bonds.



Scheme 1. Pd-catalyzed carbonylative coupling reactions of 2-iodoglycals.

One common strategy involves the intermediacy of an alkyl-metal species, such as an organozinc, either pre-formed of generated in situ (Barbier-Negishi conditions). Transmetalation with a Pd-aryl complex followed by reductive elimination then forges a new C–C bond.[15] For this process to be viable, however, some limitations have to be addressed: a) the dehalogenation of the aryl halide substrate via the formation of an undesired organometallic species followed by protonolysis and b) the formation of isomeric products caused by chain migration of the alkylpalladium intermediate. A fine tune in the electronic and steric properties of the ligand employed, however, can reduce these side reactions and render the process viable.[16]

With these considerations in mind, we wondered whether a carbonylative Negishi-type coupling of 2-iodoglycals with alkyl halides would be feasible (Scheme 1b). Unlike previous reports (Scheme 1a), this process would deliver valuable alkyl-ketones bearing acidic α-protons that can give rise to a rich variety of enolate-based functionalizations, such as electrophile trapping,[17] *de novo* synthesis of heterocycles,[18] α-arylation,[19] hydrogen-borrowing alkylation,[20] α-fluorination,[21] etc.

Herein we describe our efforts towards the synthesis of this important class of C2-branched sugars via the carbonylative coupling of 2-iodoglycals with alkyl and aryl halides.

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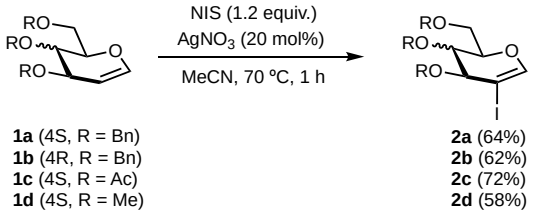
Results and Discussion

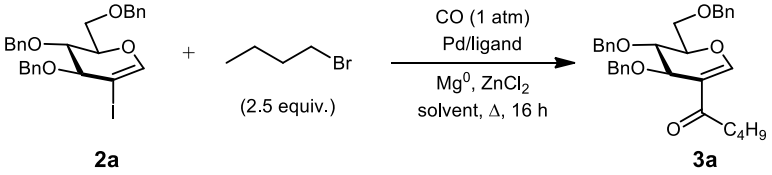
We began our studies by synthesizing four different 2-iodoglycals (**2a-d**) via Ag-catalyzed electrophilic iodination of glycals **1a-d** (Scheme 2).[22]



Scheme 2. Synthesis of 2-iodoglycals.

Tri-*O*-benzyl-2-iodoglucal (**2a**) was then selected for the optimization of the reaction conditions for the Negishi-type carbonylative coupling with 1-bromobutane (Table 1). Initial conditions based on a previous report by Wu and co-workers[14a] gave **3a** in encouraging 56% (Entry 1). Changing the catalyst to a NHC-based Pd complex, PEPPSI,[23] led to a poor conversion of **2a** and the formation of significant amounts of **1a**, resulting from de dehalogenation of the starting material (Entry 2). Bidentate phosphine ligand dppf, on the other hand, considerably increased the reaction yield (Entry 3) and bulky monophosphine RuPhos was identified as the best choice for this transformation with no significant formation of **1a**, result in agreement with previous findings by Buchwald (Entry 4).[16c]

Table 1. Optimization of the reaction conditions



Entry	Pd (mol%)/ligand (mol%)	Additives	Temperature	Solvent	Yield[a]
Effect of catalyst/ligand					
1	$Pd(dba)_2$ (6)/PPh_3 (6)	Mg^0 (2.5 equiv.), $ZnCl_2$ (2.5 equiv.)	55 °C	THF	56%
2	PdPEPPSI (6)	Mg^0 (2.5 equiv.), $ZnCl_2$ (2.5 equiv.)	55 °C	THF	30%
3	$Pd(dppf)Cl_2$ (6)	Mg^0 (2.5 equiv.), $ZnCl_2$ (2.5 equiv.)	55 °C	THF	62%
4	$Pd(dba)_2$ (6)/RuPhos (6)	Mg^0 (2.5 equiv.), $ZnCl_2$ (2.5 equiv.)	55 °C	THF	72%
5	Pd(dba)2	Mg^0 (2.5 equiv.), $ZnCl_2$ (2.5 equiv.)	55 °C	THF	0%
Effect of solvent					
6	$Pd(dba)_2$ (6)/RuPhos (6)	Mg^0 (2.5 equiv.), $ZnCl_2$ (2.5 equiv.)	55 °C	Toluene	0%
7	$Pd(dba)_2$ (6)/RuPhos (6)	Mg^0 (2.5 equiv.), $ZnCl_2$ (2.5 equiv.)	55 °C	1,4-Dioxane	7%
8	$Pd(dba)_2$ (6)/RuPhos (6)	Mg^0 (2.5 equiv.), $ZnCl_2$ (2.5 equiv.)	55 °C	MeCN	5%
Effect of temperature					
9	$Pd(dba)_2$ (6)/RuPhos (6)	Mg^0 (2.5 equiv.), $ZnCl_2$ (2.5 equiv.)	40 °C	THF	50%
10	$Pd(dba)_2$ (6)/RuPhos (6)	Mg^0 (2.5 equiv.), $ZnCl_2$ (2.5 equiv.)	50 °C	THF	74%
11	$Pd(dba)_2$ (6)/RuPhos (6)	Mg^0 (2.5 equiv.), $ZnCl_2$ (2.5 equiv.)	65 °C	THF	16%
Effect of the additive concentration					
12[b]	$Pd(dba)_2$ (6)/RuPhos (6)	Mg^0 (5.0 equiv.), $ZnCl_2$ (6.0 equiv.)	50 °C	THF	86%
Effect of the catalyst loading					
13[b]	$Pd(dba)_2$ (5)/RuPhos (5)	Mg^0 (5.0 equiv.), $ZnCl_2$ (6.0 equiv.)	50 °C	THF	85%
14[b]	$Pd(dba)_2$ (3)/RuPhos (3)	Mg^0 (5.0 equiv.), $ZnCl_2$ (6.0 equiv.)	50 °C	THF	56%

Reaction scale: 0.1 mmol (**2a**). [a] Isolated yield. [b] 5.0 equiv. of 1-bromobutane.

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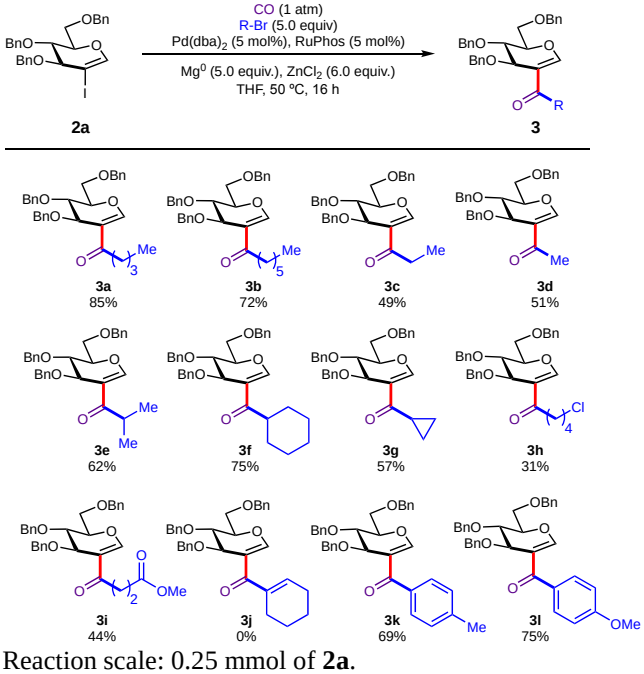
No conversion was observed in the absence of an external ligand (Entry 5). Next, a solvent screening revealed this to be a key factor in this transformation: replacing THF with toluene, 1,4-dioxane or MeCN dramatically decreased the efficiency of the carbonylative coupling and only trace amounts of **3a** were observed (Entries 6-8). The reaction temperature was then surveyed and the optimum temperature was established as 50 °C (Entry 10). Increasing the equivalents of Mg^0 and $ZnCl_2$ added had a positive impact in the reaction outcome: full conversion of **2a** was observed, while **1a** was not detected, leading to an isolated yield of 86% (Entry 12). Finally, we investigated different catalyst loadings and these experiments showed that a reduction to 5 mol% was tolerated with no significant decrease in the reaction yield (Entry 13). It is worth mentioning that some aspects proved fundamental for reproducibility and high yields of the coupling reaction: a vigorous stirring to allow an adequate gas transfer between the reaction medium and the tube headspace and a proper degasification of the reaction solvent prior to use (see Supporting Information for details).

With the optimal conditions in hand, we set out to demonstrate the reaction scope for the alkyl halide partner using **2a** as the 2-iodoglycal (Table 2).

Table 2. Scope for alkyl halides

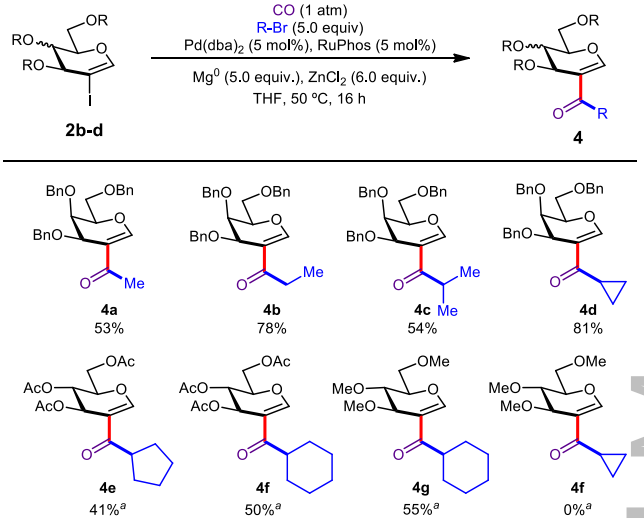


Reaction scale: 0.25 mmol of **2a**.

Linear primary alkyl groups were successfully installed, giving products **3a-d** in moderate to good yields. Secondary alkyl bromides also proved to be well tolerated, with both acyclic (**3e**) and cyclic (**3f-g**) structures forming the desired products in moderate to good yields. In the case of cyclopropyl-ketone **3g**, the alkyl moiety can also be used as a useful handle for further functionalization.[24] Despite the low yield observed for ketone **3h**, this example demonstrates a valuable selectivity for alkyl bromides over chlorides, regardless the excess Mg^0 required for this reaction.[16] Methyl 3-bromopropionate was also compatible with this reaction and dicarbonyl compound **3i** was isolated in moderate yield. Allyl bromides, on the other hand, were not amenable to this transformation, possibly due to competitive η^3-allyl-Pd formation. Gratifyingly, aryl halides were well tolerated and aryl-ketones **3l-m** were isolated in good yields.

Next, we sought to investigate the scope for the 2-iodoglycal coupling partner (Table 3).

Table 3. Scope for 2-iodoglycals



Reaction scale: 0.25 mmol of **2b-d**. [a]Catalyst loading: 10 mol%

Tri-*O*-benzyl-D-galactal **2b** was first tested and cleanly reacted under the standard reaction conditions, delivering ketones bearing primary (**4a-b**), secondary (**4c**) and cyclic alkyl moieties (**4d**) in moderate to good yields. Tri-*O*-acetyl-D-glucal **2c** and tri-*O*-methoxy-D-Glucal **2d**, on the other hand, required a higher catalyst loading to achieve full conversion of the 2-iodoglycal starting materials. Despite the higher catalyst concentration, only moderate yields were obtained (**4e-g**). In the case of some bromoalkanes (*e.g.* bromocyclopropane), the reactions yielded intractable mixtures containing large amounts of the corresponding dehalogenated glycal and a complex mixture of by-products.

In order to increase the attractiveness of the methodology, ketones **3d** and **3l** were deprotected under Lewis-acidic conditions, affording deprotected glycals **5d** and **5l** in 64% yield.

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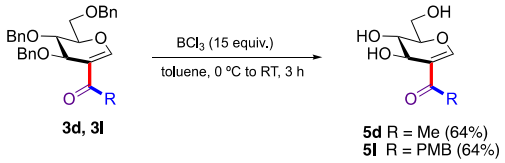

Scheme 3. Deprotection of ketones **3d** and **3l**.

The proposed mechanism for the Carbonylative coupling of **2a** with 1-bromobutane is depicted in Figure 1. Oxidative addition of Pd to 2-iodoglycal **2a** gives complex **ii**. CO complexation, followed by transmetallation with the alkylzinc species formed in situ from Mg^0 and the corresponding halide (**iv**), leads to complex **v**. Carbonylation generates acylpalladium complex **vi** that undergoes reductive elimination to deliver the alkyl-ketone **3a** and regenerate the active Pd^0 species. In some cases, as observed during the screening of the reaction conditions (Table 1) and the reaction scope (Table 2 and 3), significant amounts of dehalogenated glycals were observed. We speculate that this by-product is formed via *β*-hydride elimination of complex **v** followed by reductive elimination of the complex formed or by the formation of an organomagnesium species from **2a** followed by its protonolysis.

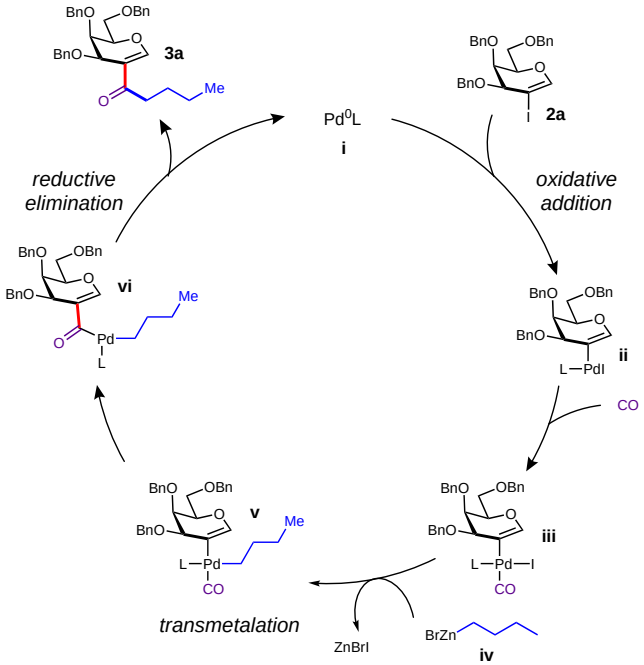

Figure 1. Proposed mechanism for the carbonylative coupling of 2-iodoglycal **2a** with 1-bromobutane.

Conclusion

In summary, we have presented a catalytic atom-economical methodology for the synthesis of valuable *C*-branched sugars bearing alkyl-ketones via Pd-catalyzed carbonylative Negishi-type coupling. 16 examples were synthesized from 4 different 2-iodoglycals in moderate to good yields. Several substituents were tolerated, demonstrating the versatility of this methodology.

Experimental Section

General procedure B: Pd-catalyzed carbonylative Neigishi-type coupling of 2-iodoglycals and alkyl halides
To a flame-dried 25-mL reaction tube capped with a rubber septum were added Mg^0 (30 mg, 1.3 mmol, 5.0 equiv.) and $ZnCl_2$ (205 mg, 1.5 mmol, 6.0 equiv.). The solid mixture was thoroughly dried under vacuum while heated by a heat gun and then backfilled with dry N_2. $Pd(dba)_2$ (7.0 mg, 12 μmol, 5 mol%) and Ruphos (6 mg, 12 μmol, 5 mol%) were then added to the reaction tube under a flow of dry N_2, the tube was then evacuated and backfilled with CO. Dry and degassed THF was added to the system (2.5 mL), followed by the corresponding 2-iodoglycal (0.25 mmol, 1.0 equiv.). The mixture was then stirred at 50 °C for 5 min and then the corresponding alkyl halide (1.25 mmol, 5.0 equiv.) was added via syringe. A CO-filled balloon was connected to the reaction tube and the system was stirred at 50 °C for 16 h. The resulting mixture was filtered through a plug of silica using EtOAc as eluent, concentrated under reduced pressure and purified by flash column chromatography.
Notes for this procedure:
1) THF was degassed via freeze-thaw technique in 3 cycles. Vacuum broken with CO in the last freeze-thaw cycle;
2) The reaction yield is influenced by the quality of the glycal and the alkyl halide used. Freshly purified glycals produce superior results;
3) Vigorous stirring is also important, therefore the size of the reaction vessel as well as the magnetic bar rotation should to be taken into account.

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Keywords

2-iodoglycals, cross-coupling, palladium, carbon monoxide, carbonylative coupling.

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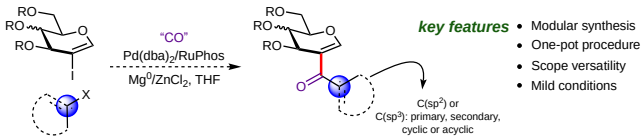
Graphical abstract



A versatile C(sp^2)-C(sp^3) carbonylative Negishi-type reaction allowing the access to glyco-ketones bearing alkyl and aryl groups is described in this report. The tolerance for different functional groups as well as protecting groups denote the usefulness of the methodology.

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