Catalytic Addition of Nitroalkanes to Unactivated Alkenes via Directed Carbopalladation

Amit Kumar Simlandy, Warabhorn Rodphon, Hui-Qi Ni, John A. Gurak, Jr., Keary M. Engle*

[a] Dr. Amit Kumar Simlandy, Warabhorn Rodphon, Hui-Qi Ni, John A. Gurak, Jr., and Prof. K. M. Engle
Department of Chemistry
The Scripps Research Institute
10550 N. Torrey Pines Road, La Jolla, CA 92037, USA
E-mail: keary@scripps.edu

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Abstract: We report a redox-neutral catalytic coupling of nitroalkanes and unactivated alkenes that proceeds by a directed carbopalladation mechanism. The reaction is uniquely enabled by the combination of PdI_2 as the precatalyst and HFIP solvent. Structurally complex nitroalkane products, including nitro-containing carbo- and heterocycles, are prepared under operationally convenient conditions without the need for toxic or corrosive reagents. Deuterium labeling experiments and isolation of a catalytically relevant intermediate shed light on the reaction mechanism. By taking advantage of different catalytic activation modes, we demonstrate orthogonal methods for site-selective functionalization of a polyfunctional nitroalkyl ketone.

The nitro group is a versatile synthetic handle that can easily be transformed into other useful functional groups.[1] Nitroalkanes are a readily available and shelf-stable family of nitrated synthetic building blocks that commonly serve as pronucleophiles; upon deprotonation at the α-C–H bond ($MeNO_2$ pK_a (DMSO) = 17.2), the resulting nitronate anion is able to engage various carbon-based electrophiles to enable C–C bond formation. Compatible electrophiles include, carbonyl compounds (Henry reaction),[2] enones and other conjugated alkenes (Michael addition),[3] and organometallic species generated from oxidative addition of a low-valent metal catalyst to allyl electrophiles[4] or aryl/alkyl halides (Figure 1A).[5] Expansion of this toolkit to unactivated alkenes through a catalytic π-Lewis acid activation mechanism would broaden the scope of nitro-containing product structures that could be accessed, yet such a transformation remains unknown to date.

In recent years, substrate-directed Heck- and Wacker-type alkene addition reactions have gained prominence as enabling tools in synthesis.[6, 8, 10] We have previously employed 1,3-dicarbonyl compounds and other C–H pronucleophiles in a pK_a range of 10–18 in directed carbo-Wacker-type additions to alkenes under Pd(II) catalysis.[6b] Based on this precedent we questioned whether it would be possible to enlist nitroalkanes in this type of process (Figure 1B).[7] Successful realization of such a catalytic coupling would be practically significant as it would facilitate expedient synthesis of highly substituted nitroalkane products without the need for harsh reagents (e.g., nitric acid). Conceptually, it would demonstrate the feasibility of using nitrated nucleophiles in the modular approach to alkene difunctionalization that our lab and others have developed during the past five years.[8]

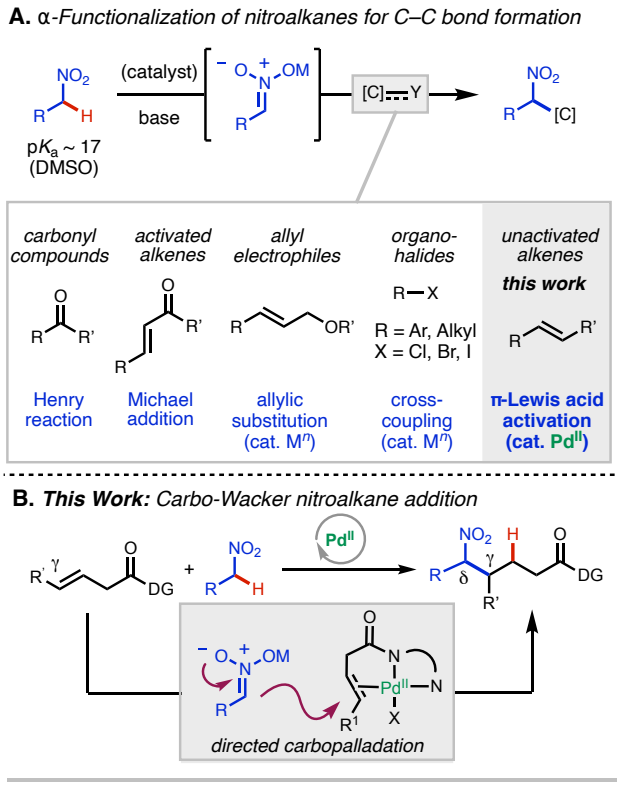
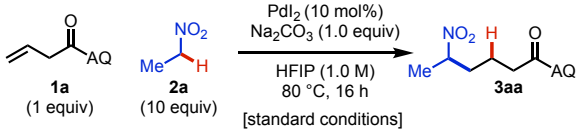


Figure 1. Background and envisioned transformation

To reduce this idea to practice, we initially focused on the model reaction shown in Table 1, in which a 3-butenoic acid substrate containing an 8-aminoquinoline amide (AQ) directing auxiliary (**1a**) is coupled with excess nitroethane (**2a**) (10 equiv). Pilot experiments revealed that published conditions for 1,3-dicarbonyl nucleophiles were unsuccessful in this case (entry 2).[6b] After extensive optimization, we identified optimal conditions consisting of PdI_2 as catalyst, Na_2CO_3 as base, and HFIP as solvent at 80 °C (entry 1). The presence of iodide as the counteranion was found to be critical, as other commonly used Pd(II) sources like $PdCl_2$ and $Pd(OAc)_2$ were ineffective (entries 3–5). Other inorganic bases like K_2CO_3 were found to be inferior (entry 6). While the reaction provided synthetically useful yields with as low as 2.0 equiv $EtNO_2$, the yield steadily decreased with lower loadings of $EtNO_2$ (entries 7–8). Fluorinated alcohol solvents were found to be uniquely effective in promoting this coupling (entries 9–12), while aprotic

solvents with different dielectric constants (entry 9) and non-fluorinated polar protic solvents (e.g., ^{t}BuOH and ^{t}AmOH) (entry 10) failed to facilitate the reaction. Compared to HFIP, TFE and $(CF_3)_3COH$ were competent though led to lower yield (entries 11–12).

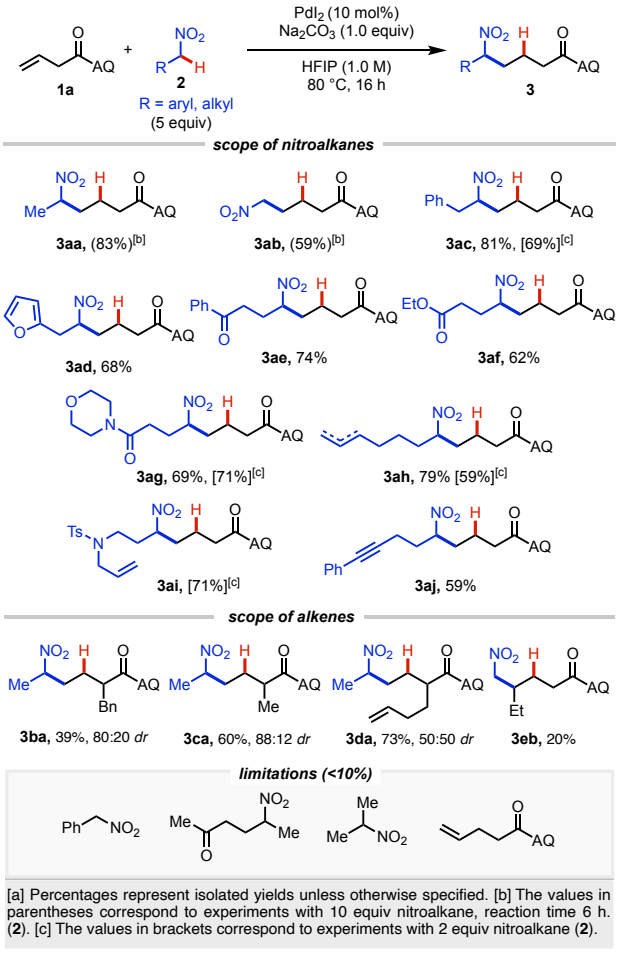
Table 1. Reaction optimization



entry	deviation from standard conditions	yield (%)[a]
1	None	83[b]
2	AcOH, $Pd(OAc)_2$, MeCN, 120 °C	<10[ref. 6b]
3	$PdCl_2$ instead of PdI_2	11
4	$Pd(OAc)_2$ instead of PdI_2	13
5	$Pd(OAc)_2$, TBAI	60
6	K_2CO_3 instead of Na_2CO_3	36
7	5.0 equiv. of $EtNO_2$	57
8	2.0 equiv. of $EtNO_2$	37
9	Toluene or MeCN instead of HFIP	<10
10	^{t}BuOH or ^{t}AmOH instead of HFIP	<10
11	$(CF_3)_3COH$ instead of HFIP	50
12	TFE instead of HFIP	63

[a] ^{1}H NMR yield of the crude reaction mixture was determined using mesitylene as an internal standard. [b] Isolated yield, reaction time 6 h.

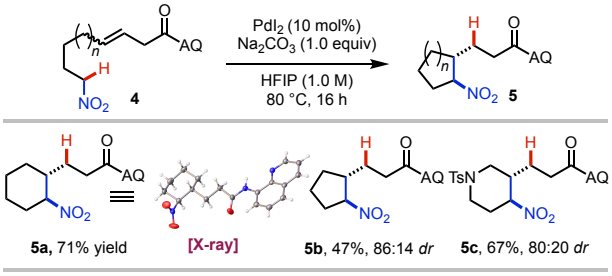
Having identified optimal reaction conditions, the scope of the method was next evaluated (Scheme 1). A diverse collection of nitroalkane coupling partners participated in the reaction. It is important to mention that for valuable nitroalkanes, use of either 5.0 equiv or 2.0 equiv was sufficient to generate the nitro-containing products in good to excellent yields. Beyond the bulk chemicals nitroethane (**3aa**) and nitromethane (**3ab**), more structurally complex nitroalkane coupling partners were effective (**3ac–3aj**), including those containing potentially reactive or inhibitory functional groups, such as electron-rich heterocycles (**3ad**), ketones (**3ae**), esters (**3af**), amides (**3ag**), alkenes (**3ah**), protected amines (**3ai**), and alkynes (**3aj**). Regarding the alkene scope, substituents were well tolerated at the position α- with respect to the amide directing group (**3ba**–**3da**), albeit with variable *d.r.* An internal alkene was competent, though the yield was low (**3eb**). A number of limitations were also identified, as α,α-disubstituted nitroalkanes and substrates with longer spacers between the alkene and directing group were unreactive.



[a] Percentages represent isolated yields unless otherwise specified. [b] The values in parentheses correspond to experiments with 10 equiv nitroalkane, reaction time 6 h. (**2**). [c] The values in brackets correspond to experiments with 2 equiv nitroalkane (**2**).

Scheme 1. Substrate and coupling partner scope.[a]

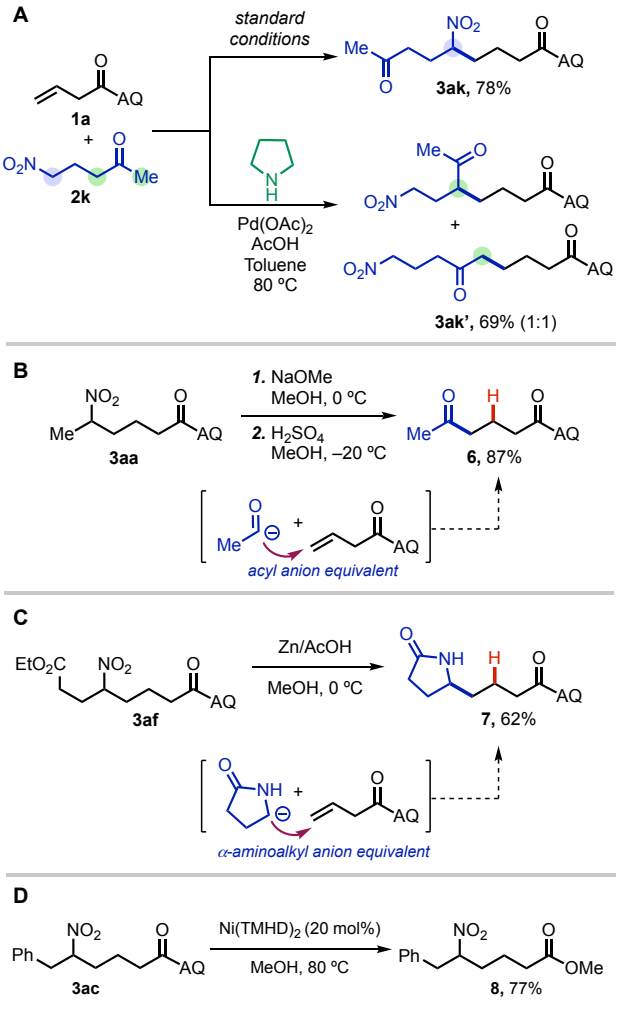
By using cross-metathesis[9] it was possible to quickly access substrates in which the nitroalkane is intramolecularly tethered to the alkene. Under the optimal conditions from Table 1, redox-neutral carbocyclization[6l, 10] took place to furnish nitro-substituted carbo- and heterocycles in moderate yield and with good to excellent diastereoselectivity (Scheme 2).[11] An X-ray crystal structure of **5a** confirmed the *trans* relationship between the nitro group and the alkyl chain containing AQ in the major product.



Scheme 2. Scope of intramolecular cyclization.

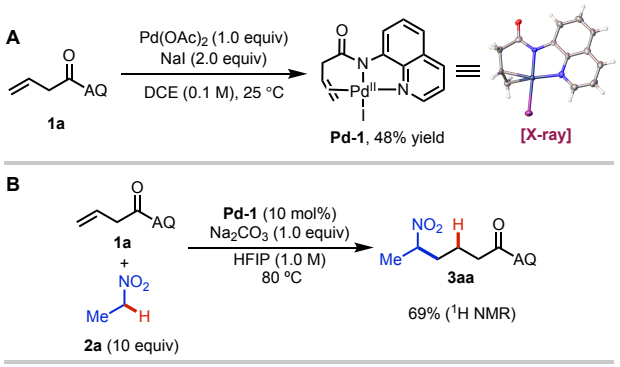
We investigated keto-nitroalkane **2k** as a model bis-pronucleophile with different potential reaction sites that we thought could be addressed in a chemodivergent manner

(Scheme 3A). Indeed, when **2k** was subjected to the optimized reaction conditions, **3ak** was obtained in 78% yield, with sites adjacent to the ketone unperturbed. Alternatively, a dual Pd(II)/organocatalytic activation strategy[6i, 6j, 8j] was applied to unlock reactivity α to the ketone through enamine catalysis, and the product **3ak'** was obtained in 69% yield as a 1:1 regioisomeric mixture with both α-positions proving to be reactive. Efforts to improve the regioisomeric ratio through optimization of the reaction conditions were unsuccessful (see ESI). To demonstrate how the nitroalkane pronucleophiles can serve as synthetic equivalents for other useful synthons, we performed a Nef reaction[12] with **3aa** and were pleased to see the formation of **6**, the product of formal hydroacylation of **1a**, in 87% yield (Scheme 3B). Alternatively, the nitro group could also be easily reduced under Zn/AcOH conditions, and in the presence of a pendant ester, in situ cyclization took place to furnish compound **7**, representing a formal α-aminoalkyl anion addition (Scheme 3C). The AQ protecting group in **3ac** was easily removed by $Ni(TMHD)_2$ in presence of methanol to obtain ester **8** (Scheme 3D).[13]



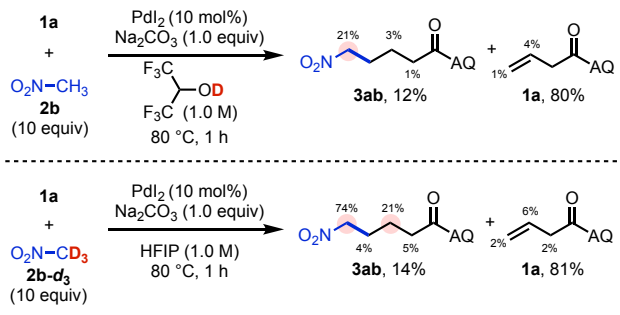
Scheme 3. [A] Chemodivergent addition of nitroalkane. [B] Nef reaction. [C] Reduction. [D] Removal of AQ protecting group. TMHD = 2,2,6,6-tetramethyl-3,5-heptanedionate.

To shed light on the mechanistic details of this transformation, particularly the importance of HFIP as solvent and PdI_2 as catalyst (Scheme 4), several experiments were performed. By treating **1a** with stoichiometric $Pd(OAc)_2$ and NaI in DCE, we were able to prepare substrate-bound Pd(II) complex **Pd-1** and determine its X-ray crystal structure.[6b] This complex was found to be catalytically competent in the reaction, consistent with the notion that it may be an intermediate on the catalytic cycle.



Scheme 4. [A] Synthesis of substrate-bound palladium complex **Pd-1**. [B] Assessment of the catalytic competence of **Pd-1.**

We then focused on determining the main source of the H-atom in the product with a series of deuterium labeling experiments. Reactions were halted at low conversion to avoid complications from secondary H/D exchange processes (see SI). Whereas use of deuterated solvent, $(CF_3)_2CHOD$, led primarily to D-incorporation at the δ position (directly adjacent to the nitro group), when the reaction was performed with CD_3NO_2 (**2b-*d*$_3$**) as nucleophile, 21% D-incorporation at the β-position was observed. In both cases H/D exchange at the alkenyl positions of the substrate[6f] was comparatively slow. This data indicates that the acidic protons/deuterons of the solvent and nucleophile do not freely exchange under the reaction conditions and also that the nitroalkane serves the dual role as nucleophile and proton/deuteron source in this catalytic alkene hydrofunctionalization. Given that HFIP is not the primary proton source in the reaction, a potential explanation for its role in promoting the reaction is stabilizing the charged sodium nitronate anion.[14] Meanwhile, iodide may play a role as a Lewis basic site for sodium coordination to template nucleophilic attack.[15]



Scheme 5. Deuterium labelling study

Based on previous reports[6, 8] and the mechanistic experiments above, a plausible catalytic cycle is drawn in Figure 2. First, **Pd-1** complex is formed via the ligand exchange between PdI_2 and substrate (**1a**). Next, nucleopalladation delivers an alkyl palladacycle.[6b] This conformationally constrained is resistant to β-H elimination and is instead intercepted by an acidic proton originating from the nitroalkane to furnish the product (**3ab**). During this cycle acidic protons will be exchanged with deuterium in the presence of base.

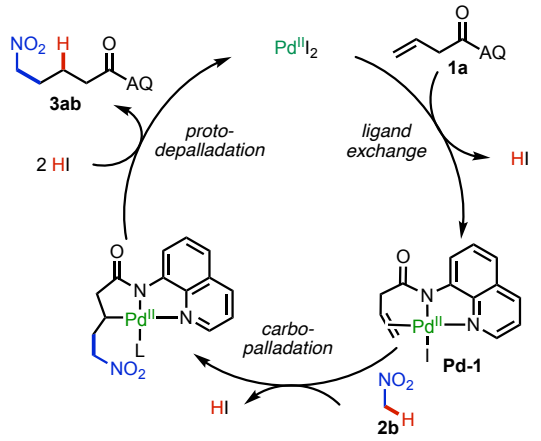


Figure 2. Plausible catalytic cycle with a representative alkene and nitroalkane.

In conclusion, we have developed a redox-neutral addition of nitroalkanes to unactivated alkenes via a directed nucleopalladation strategy, granting convenient access to various nitro-containing products in regioselective fashion. Whereas previously published protocols are incompatible with nitroalkane pronucleophiles, the discovery of specially tailored conditions comprised of PdI_2 as catalyst in HFIP as solvent allows for selective engagement of sites adjacent to nitro groups. In this way, chemodivergent addition of keto-nitroalkane was demonstrated under different activation modes.

Acknowledgements

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Keywords: palladium • directing group • nitroalkane • alkene functionalization

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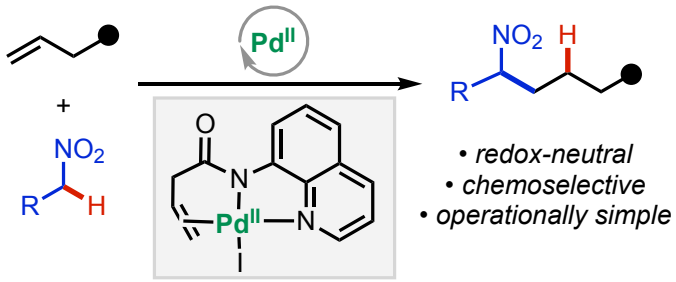

We demonstate a redox-neutral palladium-catalyzed coupling of nitroalkanes and unactivated alkenes for the synthesis of diverse range of nitro-containing products under operationally simple conditions. Synthetic transformations of the nitro group provided access to molecules that are otherwise difficult to synthesize via olefin functionalization.

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