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Volume 54
Number 1
4 January 2018
Pages 1-112

ChemComm
Chemical Communications
rsc.li/chemcomm

ISSN 1359-7345

ROYAL SOCIETY OF CHEMISTRY

COMMUNICATION
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Conformational control of nonplanar free base porphyrins: towards bifunctional catalysts of tunable basicity

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DOI: 10.1039/D0CC06652D

Selective Nickel-catalyzed Fluoroalkylations of Olefins

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Received 00th January 20xx,
Accepted 00th January 20xx

DOI: 10.1039/x0xx00000x

Mild and selective nickel-catalyzed trifluoromethylation and perfluoroalkylation reactions of alkenes were developed to provide fluorinated olefins, including natural products, pharmaceuticals, and variety of synthetic building blocks in good to excellent yields (38 examples). Control experiments, kinetic measurements and *in situ* EPR studies reveal the importance of radical species and the formation of 1,2-adducts as intermediates.

Organofluorine compounds have significantly different chemical and physical properties compared to their parent hydrocarbons because of the high electronegativity (3.98, Pauling scale) and at the same time very small size of fluorine atoms (50 pm).[1] Hence, their synthesis and applications continue to attract the attention of many researchers in industry and academia.[2] Apart from the fundamental scientific interest in fluorinated chemicals, they are also used in diverse applications ranging from material (e.g. refrigerants, oil and water repellents) to life sciences (pharmaceuticals, agrochemicals, etc.).[3] Especially, in this latter area several methodologies enabling the synthesis of new bioactive compounds were disclosed in recent years.[4]

Nevertheless, the preparation of advanced fluorinated building blocks generally requires special techniques associated with the handling of sensitive and expensive reagents.[5] To overcome these limitations, we started a program investigating catalytic coupling processes of commercially available perfluoroalkyl halides including trifluoromethyl bromide and iodide with (hetero)arenes.[6] Most recently, we also reported a facile and practical method for the stereoselective synthesis of fluorinated olefins from ketones,[7] and the first selective carbonylations of *gem*-difluoroalkenes leading to difluoromethylated esters.[8]

Inspired by these works and the known versatility of (per)fluoroalkyl-substituted olefins, we became interested in their preparations (Scheme 1).[9] An obvious approach to access this kind of molecules makes use of metal-mediated cross coupling reactions between vinyl halides and the (*in situ)* formed fluoroalkyl metal species.[10] However, the need of stoichiometric amounts of transition metals and the availability of the substrates limits such procedures. Following the same strategy, (per)fluoroalkyl-substituted olefins have been synthesized by reaction of perfluoroalkyl reagents with vinyl boronic acids,[11] vinyl zirconium complexes,[12] or alkynes.[13] In all these cases only very few examples could be realized, and the reaction conditions are sensitive. Alternatively, the direct functionalization of alkenes with (per)fluoroalkyl halides R_FX (X = I, Br) represents an advantageous and straightforward methodology. Notably, both kinds of substrates (alkenes and R_FX) are commercially available at reasonable prices. Hence, it is surprising that this transformation was scarcely investigated. In fact, only photochemical[14] and noble metal catalyzed[15] protocols are known. Complementary to these examples, herein we report a general nickel-catalyzed protocol for the perfluoroalkylation of alkenes to offer selectively fluorinated olefins.

For our initial studies, the perfluorodecylation of 3,3-dimethylbutene was selected as a model system to avoid unwanted side reactions (e.g. olefin isomerization). As alternative catalysts we envisioned nickel complexes, which can be often used as a substitute for palladium in cross coupling reactions.[16] More specifically, the commercially available air- and moisture-stable dppfNi(*o*-tol)Cl complex was applied as pre-catalyst,[17] because of its activity in the perfluoroalkylation of (hetero)arenes.[18] To trap the potentially formed acid (HX), most test reactions were performed in the presence of base (2 equiv). Indeed, without base no conversion occurred at 50-80°C even after 16 h. Similar results were obtained in the presence of alkali carbonates and acetates (Na_2CO_3, Cs_2CO_3, and NaOAc)

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Electronic Supplementary Information (ESI) available: [details of any supplementary information available should be included here]. See DOI: 10.1039/x0xx00000x

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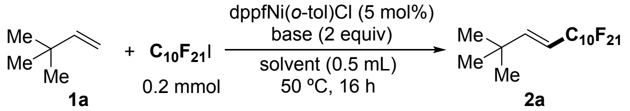
DOI: 10.1039/D0CC06652G

(Table 1, entries 1-3). Likewise, using a weak organic base (TMEDA) showed no desired product formation (Table 1, entry 4). However, applying stronger bases *t*-BuONa and *t*-BuOK a Heck-type vinylation occurred and *E*-1-perfluorodecyl-3,3-dimethyl-but-1-ene **2a** is obtained in 86-87% yield (Table 1, entries 5-6). It is noteworthy that the test reaction also took place at room temperature giving 48% of the desired product, while reactions at higher temperature did not improve the product yield further on (Table 1, entries 7-8). Blank experiments without nickel catalyst gave only negligible conversion.

Table 1. Ni-catalyzed perfluorodecylation of 3,3-dimethylbutene.

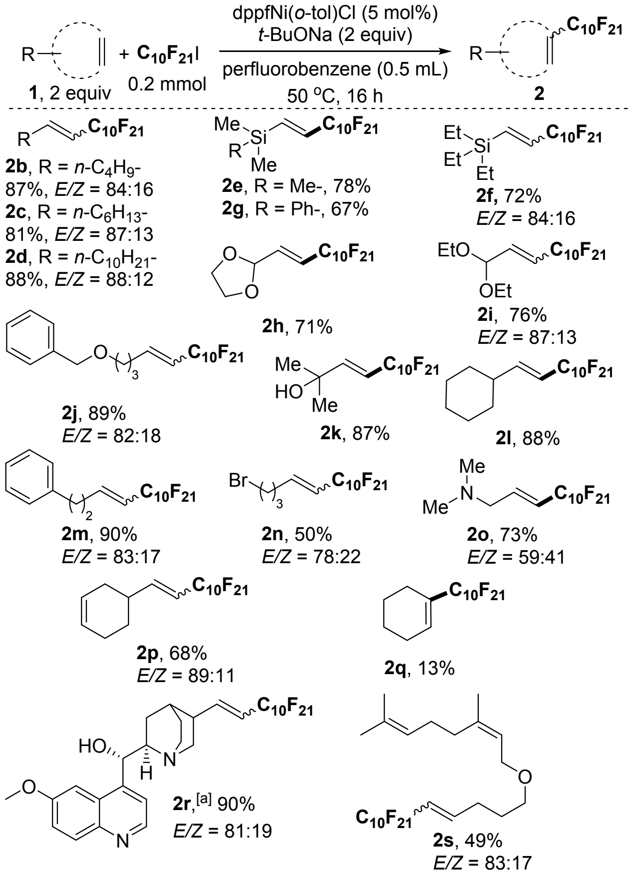


Entry	Base	Solvent	T / °C	Yield / %
1	Na_2CO_3	--	50	-
2	Cs_2CO_3	--	50	-
3	NaOAc	--	50	-
4	TMEDA	--	50	-
5	*t*-BuONa	--	50	87
6	*t*-BuOK	--	50	86
7	*t*-BuONa	--	25	48
8	*t*-BuONa	--	120	85
9	*t*-BuONa	benzene	50	19
10	*t*-BuONa	DMF	50	17
11	*t*-BuONa	perfluorobenzene	50	75
12	*t*-BuONa	CH_3CN	50	15
13	*t*-BuONa	THF	50	21

Reaction conditions: **1a** (2 equiv in case of using additional solvent, 0.5 mL for all other entries), Ar, yields were determined by ^{19}F NMR.

In all previously described experiments, 3,3-dimethylbutene was used both as substrate and solvent. Although it is beneficial for inexpensive olefins to run this transformation in neat media, for structurally more advanced olefins the use of an external solvent will be obviously preferred. Thus, experiments in the presence of different solvents were investigated. As shown in Table 1 (entries 9-13), this transformation is quite sensitive towards solvents. While standard aromatic (benzene) and dipolar aprotic (DMF) solvents showed low product yields (17-19%), in the presence of perfluorobenzene a comparable yield of **2a** (75%) with the neat condition could be obtained. Using THF and CH_3CN as solvent, as the major reaction pathway reductive dehalogenation occurred and a significant amount of $C_{10}F_{21}H$ is detected by ^{19}F NMR (33% and 76%, respectively).

With suitable reaction conditions in hand, the perfluoroalkylation of various alkenes was tested. Apart from 3,3-dimethylbutene, linear aliphatic alkenes with different chain length are converted to the corresponding products **2b** – **2d** in high yields (>81%). Here, no olefin isomerization is occurring and the *E*-products prevail. Fluoroalkylated vinylsilanes **2e** – **2g** are obtained (67–78% yields) by smooth transformation of vinylsilanes, which can be easily transformed into other useful products.[19] Next, various alkenes with 1,3-dioxa-2-cyclopentyl (**2h**), ethoxy (**2i**), benzyloxy (**2j**), hydroxy (**2k**), cyclohexyl (**2l**), and phenyl (**2m**) groups were all successfully perfluoroalkylated to give the corresponding products in good to excellent yields. Interestingly, perfluorodecyl iodide is selectively activated in the presence of an alkenyl bromide (**2n**). Furthermore, amino groups (**2o**) are well tolerated in this procedure, too. Using substrate **1p**, containing internal and terminal double bonds, only the latter reacted affording **2p** in 68% yield. Nevertheless, without terminal double bond present also cyclohexene can be perfluoroalkylated, albeit in lower yield. From a synthetic point of view, the reactions of quinidine to give **2r** in 90% and naturally occurring nerol to provide **2s** are notable.



Scheme 1. Nickel-catalyzed perfluorodecylation of alkenes. Yields were determined by ^{19}F NMR. [a] 4 equiv *t*-BuONa.

To demonstrate the generality of our protocol and this specific catalyst system, other perfluoroalkyl reagents including trifluoromethyl bromide and iodide were explored. As shown in Scheme 2, perfluoroalkyl iodides containing three to eight carbons all let to excellent yields of the desired products **4a** – **4d**. Using commercially available perfluoroisopropyl iodide, the perfluoroisopropylated 4-phenylbutene **4e** was obtained in quantitative yield selectively as *E*-isomer. Notably, perfluoroalkyl bromides underwent similar perfluoroalkylations in the presence of this Ni catalyst. For example, 4-bromo-perfluorobutyl bromide was successfully used to functionalize 3,3-dimethylbutene in good yield to give **4f**.

Among the possible fluoroalkylation reactions, trifluoromethylations are synthetically amongst the most interesting ones. Hence, we studied such reactions using either CF_3Br or CF_3I. In general, both reagents can be used, but CF_3I is more active than CF_3Br in the presence of the Ni catalyst.

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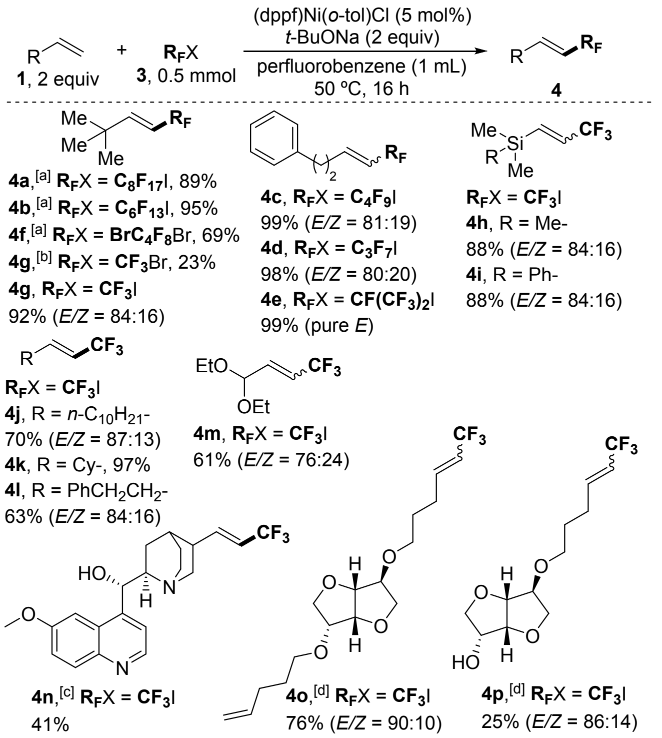
Trifluoromethylations of structurally diverse alkenes including the anti-arrhythmia drug quinidine and isosorbide gave the corresponding products **4g**–**4p** in up to 97% yield.

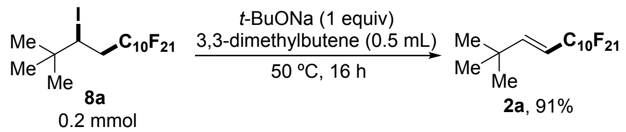


Scheme 2. Nickel-catalyzed fluoroalkylation of alkenes with different perfluoro-alkyl reagents. Yields were determined by ^{19}F NMR. [a] 1 mL 3,3-dimethylbutene, no perfluorobenzene. [b] 0.5 mmol 3,3-dimethylbutene, 5 bar CF_3Br, 40 bar N_2. [c] 0.5 mmol alkene, 2 equiv CF_3I. [d] DCE as the solvent.

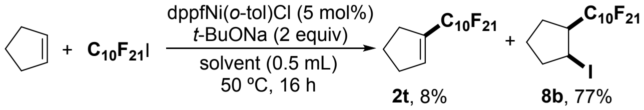
To understand this novel methodology and get mechanistic insights, a kinetic profile of the perfluorodecylation of 3,3-dimethylbutene was performed under standard reaction conditions (5 mol% dppfNi(o-tol)Cl, 2 equiv *t*-BuONa, 50 °C; see SI, Figure S1). Notably, the activation of $C_{10}F_{21}I$ is very fast and full conversion is observed within 10 min. The initially formed product was identified as the corresponding 1,2-adduct 8a, which slowly underwent base induced elimination of HI to give the final product 2a.

Control experiments revealed that this E2-elimination process is solely mediated by the strong base and does not need the presence of the nickel catalyst (Scheme 3a). To understand the low product yields in case of cyclic olefins, the perfluorodecylation of cyclopentene was re-investigated in more detail. In fact, fast conversion of the perfluoroalkylation reagent occurred, too. However, only small amounts of the desired olefin **2t** (8%) were obtained. Instead, the corresponding 1,2-adduct 8b could be isolated as the major product (77%). Notably, 8b failed to undergo further transformation under standard reaction conditions, which is explained by the lower acidity of the secondary alklyl carbon substituted with the perfluoroalkyl group (Schemes 3b and 3c). Next, the benchmark reaction was completed in the presence of 10 mol% TEMPO to study the importance of radical intermediates in this process. As shown in Scheme 3d neither the desired product nor the 1,2-adduct **8a** are observed after adding TEMPO. Thus, the following catalytic cycle is proposed (Scheme 4): Initially, the air- and water-stable Ni pre-catalyst forms an active Ni(I) species **A**, which is detected by *in situ* EPR spectroscopy (see SI Figure S2). A control experiment (50 mg catalyst with 0.4 mmol *t*-BuONa in 0.5 mL perfluorobenzene) revealed at the same time the formation of 2,2'-dimethylbiphenyl (GCMS). Then, the Ni(I) species **A** reacts with R_FX to give the more stable complex **B** and a perfluoroalkyl radical, which is again supported by *in situ* EPR studies (see SI, Figure S3). After addition of this radical to the alkene substrate, a new radical species **C** is formed, this is supposed to have a very short living time.[20] As a major pathway **C** leads to the respective 1,2-addition product, which eliminates HX via an E2 process. However, based on the minor formation of **2t**, we cannot exclude a direct perfluoroalkylation pathway, in which **C** directly forms the desired product. Finally, the resulting Ni^{III} complex regenerates the active nickel(I) complex **A**.[21]

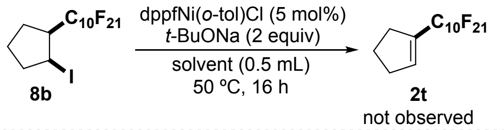
a) Conversion of 1,2-adduct to the Heck-type product with *t*-BuONa



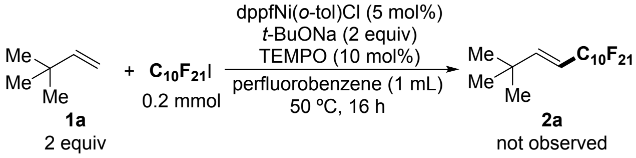
b) Perfluoroalkylation of cyclopentene



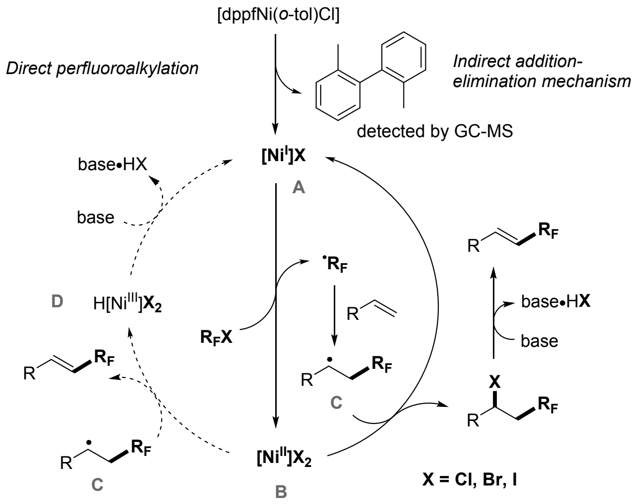
c) Conversion of 1,2-adduct to the Heck-type product



d) Radical trap experiment with TEMPO



Scheme 3. Mechanistic control experiments



Scheme 4. Proposed catalytic mechanism

In conclusion, we have developed a mild and versatile protocol for the fluoroalkylation of alkenes including synthetically interesting trifluoromethylations. Using terminal olefins including a variety of functionalized ones, the corresponding fluoroalkylated alkenes are obtained in good yields and E/Z-selectivity. Advantageously, the presented nickel catalyst is comparatively inexpensive as well as air- and moisture-stable. Mechanistic investigations reveal that this transformation mainly proceeds via fast formation of the 1,2-adduct, which subsequently undergoes slow base-catalyzed elimination. EPR measurements also provide evidence for the importance of radical intermediates.

We are grateful for the financial support from the State Mecklenburg-Western Pomerania, the federal German Ministry BMBF and LONZA.

Conflicts of interest

There are no conflicts to declare.

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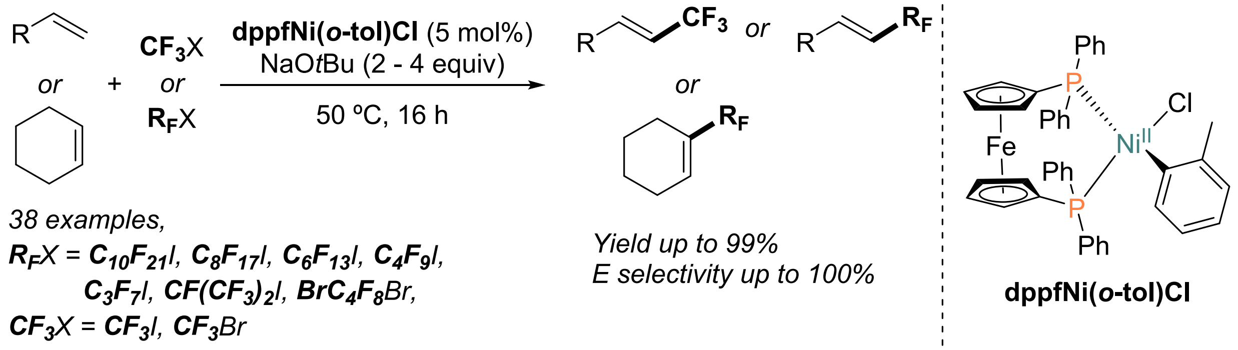

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