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# Synthesis of Allylarenes *via* Catalytic Decarboxylation of Allyl Benzoates

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**Abstract:** A catalyst system consisting of the palladium(0) complex  $Pd_2(dba)_3$  and tri(p-tolyl)phosphine was found to efficiently promote the decarboxylation of allyl benzoates with formation ofallylarenes. This catalytic C–O activation followedby extrusion of carbon dioxide and C–C bond formation represents a sustainable alternative to traditional waste-intensive cross-couplings. The scope ofthe transformation includes allyl and cinnamylesters of various*ortho*-substituted benzoic acids.For particularly activated substrates, the palladiumcatalyst can optionally be replaced by an inexpensive nickel complex.

**Keywords:** allylation; decarboxylation; homogeneous catalysis; nickel; palladium

Over the past decades, transition metal-catalyzed cross-coupling reactions have become established as powerful tools for the regioselective formation of C– C bonds.<sup>[1]</sup> In redox-neutral transformations, coupling occurs between a carbon electrophile and a carbon nucleophile, usually an organometallic reagent. However, the synthetic value of such transformations depends on the availability and stability of the starting materials, which are normally synthesized in additional, waste-intensive steps. In recent years, the interest in sustainable and salt-free alternatives has grown considerably, and the use of cheap and easily available carboxylic acids as coupling partners has received tremendous attention.<sup>[2]</sup>

In decarboxylative couplings, organometallic reagents are replaced by simple carboxylates as the nucleophilic coupling partner. This concept has found application in the synthesis of biaryls<sup>[3]</sup> and aryl ketones,<sup>[4]</sup> and for introducing allyl and benzyl groups.<sup>[5]</sup> The catalytic activation of C–O bonds has allowed the replacement of ecologically questionable organohalides by ethers or carboxylates as the carbon electrophiles. Prominent examples include Tsuji–Trost allylations of allyl esters,<sup>[6]</sup> as well as Ni-catalyzed crosscouplings of anisoles or aryl pivalates with organometallic reagents.<sup>[7]</sup>

The two innovative concepts of C-O activation and decarboxylative coupling are combined in the catalytic decarboxylation of allyl carboxylates (Scheme 1, top). This reaction type was pioneered by the groups of Tsuji<sup>[8]</sup> and Saegusa,<sup>[9]</sup> and advanced to synthetic maturity by Tunge,<sup>[2c,10]</sup> Stoltz,<sup>[11]</sup> and others. However, its scope has long remained limited to activated structures that, upon decarboxylation, lead to stabilized carbanions, for example, enolates.<sup>[9]</sup> Such carboxylate substrates, for example,  $\alpha$ -oxo esters or dialkyl malonates, readily extrude CO<sub>2</sub> even without a catalyst. Just recently, the reaction concept was extended to a class of non-activated carboxylates. Thus, a combination of palladium and nucleophilic organocatalysts was shown to catalyze the conversion of allyl  $\alpha$ -oxocarboxylates into the corresponding  $\alpha,\beta$ -unsaturated ketones in a decarboxylation/isomerization sequence (Scheme 1, center).<sup>[12]</sup>

It would be highly desirable to use a related reaction concept also for the decarboxylative allylation of non-activated carboxylic acids, for example, simple benzoic acids. The allylbenzene scaffold is a common structural motif,<sup>[13]</sup> and the allyl moiety is a versatile anchor for further derivatization.<sup>[14]</sup> However, the only example of an allylarene synthesis *via* decarboxylative coupling is the reaction of dimethoxybenzoates with allyl halides reported by Liu et al., which calls for an elaborate palladium/copper catalyst system and three equivalents of silver carbonate.<sup>[5a]</sup>

We herein present the first example of a decarboxylative allylation of benzoates that requires only a catalytic amount of metal and no stoichiometric additive, thus generating volatile  $CO_2$  as the only by-product.

We started the catalyst development by using allyl pentafluorobenzoate as a model substrate, since this benzoic acid decarboxylates easily in the presence of

previous work:

Tsuji, Saegusa, Tunge, Stoltz and others:



O ↓\_Pd(II)-

**Scheme 1.** Decarboxylative allylation of allyl esters.

palladium catalysts that are also known to efficiently insert into allyl-C–O bonds.<sup>[15]</sup> With Liu's catalyst system, the allylbenzene was observed in only 31% yield, along with pentafluorobenzene. The monometallic palladium catalyst that had been employed in the decarboxylative allylation of  $\alpha$ -oxocarboxylates [2.5 mol% Pd<sub>2</sub>(dba)<sub>3</sub>/25 mol% P(*p*-Tol)<sub>3</sub>] gave only 7% yield (Table 1, entry 1).

However, the yields increased when lowering the amount of  $P(p-Tol)_3$ , so that with 2.5 mol%, 42% of **2a** were obtained. Even lower phosphine amounts gave inferior results (Table 1, entries 1–5). Systematic variation of the Pd catalyst revealed that  $Pd_2(dba)_3$  is optimal, although  $Pd(PPh_3)_4$  and  $Pd(OAc)_2$  are also active (Table 1, entries 6–9). Control experiments confirmed that the reaction does not take place in the absence of palladium (Table 1, entry 23).

Changing the solvent to 1,4-dioxane gave a decisive step-up in the yields, whereas other solvents were less effective (Table 1, entries 10–14). In comparison to other triarylphosphines (Table 1, entries 15–18) and trialkylphosphines (Table 1, entries 19 and 20), P(p-Tol)<sub>3</sub> gave the best results. The reaction temperature also has a profound influence on the reaction outcome. At the optimum temperature of 110 °C, **2a** was obtained in near quantitative yield (Table 1, entry 21). It is remarkable that double bond migration, as occurs quantitatively in the case of  $\alpha$ -oxocarboxylates, is not observed.

Having thus identified an efficient catalytic system, we next investigated the scope of the new reaction. As can be seen from Table 2, various substituted allyl benzoates could be converted to the corresponding allylbenzenes in good to excellent yields. It is not surprising that the scope with regard to the benzoic acids remains limited to substrates that can be easily decarboxylated with palladium catalysts.<sup>[15]</sup> ortho-Nitroben-

Table 1. Optimization of the reaction conditions.<sup>[a]</sup>

– CO<sub>2</sub> – [Pd]



#	Catalyst	$PR_3 (mol\%)$	Solvent	Yield [%] <sup>[b]</sup>
1	$Pd_2(dba)_3$	P( <i>p</i> -Tol) <sub>3</sub> (25)	toluene	7
2	$Pd_2(dba)_3$	$P(p-Tol)_{3}(10)$	toluene	29
3	$Pd_2(dba)_3$	$P(p-Tol)_3(5)$	toluene	34
4	$Pd_2(dba)_3$	$P(p-Tol)_{3}$ (2.5)	toluene	42
5	$Pd_2(dba)_3$	-	toluene	29
6	$Pd(PPh_3)_4$	-	toluene	7
7	$Pd(OAc)_2$	$P(p-Tol)_3$	toluene	28
8	PdCl <sub>2</sub>	$P(p-Tol)_3$	toluene	0
9	$Pd(acac)_2$	$P(p-Tol)_3$	toluene	0
10	$Pd_2(dba)_3$	$P(p-Tol)_3$	anisole	50
11	$Pd_2(dba)_3$	$P(p-Tol)_3$	1,4-dioxane	73
12	$Pd_2(dba)_3$	$P(p-Tol)_3$	diglyme	34
13	$Pd_2(dba)_3$	$P(p-Tol)_3$	NMP	43
14	$Pd_2(dba)_3$	$P(p-Tol)_3$	DMSO	21
15	$Pd_2(dba)_3$	PPh <sub>3</sub>	1,4-dioxane	35
16	$Pd_2(dba)_3$	$P(p-F-C_6H_4)_3$	1,4-dioxane	27
17	$Pd_2(dba)_3$	$P(p-MeO-C_6H_4)_3$	1,4-dioxane	25
18	$Pd_2(dba)_3$	$P(2-furyl)_3$	1,4-dioxane	58
19	$Pd_2(dba)_3$	PCy <sub>3</sub>	1,4-dioxane	15
20	$Pd_2(dba)_3$	$P(t-Bu)_3$	1,4-dioxane	9
21 <sup>[c]</sup>	$Pd_2(dba)_3$	$P(p-Tol)_3$	1,4-dioxane	99
22 <sup>[d]</sup>	$Pd_2(dba)_3$	$P(p-Tol)_3$	1,4-dioxane	13
23	_	$P(p-Tol)_3$	1,4-dioxane	0

[a] Reaction conditions: 1a (0.50 mmol), palladium (5 mol%), PR<sub>3</sub> (2.5 mol% unless otherwise specified), solvent (2.5 mL), N<sub>2</sub> atmosphere, 100 °C, 24 h.

<sup>[b]</sup> Yields were determined by GC analysis using *n*-tetradecane as internal standard.

<sup>[c]</sup> 110°C.

<sup>[d]</sup> 90 °C.

 Table 2. Scope of the decarboxylation.<sup>[a]</sup>

	$Ar \xrightarrow{O} R^{2} R^{2} \frac{Pd_{2}(dba)_{3} (2.5 \text{ m})}{11 \text{ m}}$	$\frac{1}{100} \frac{1}{100} Ar \frac{1}{100} R^2 Ar \frac{1}{100} R^2$	2
	<b>1a-t</b>	C, 24 h K R' <b>2a-t 3a-t</b>	
Product	Yield [%] (2:3)	Product	Yield [%] (2:3)
F + F + F + F + F + F + F + F + F + F +	88 (99:1)	$F \\ F \\ 2k$	99 (99:1)
F F F 2b	68 <sup>[b]</sup> (99:1)		97 (99:1)
F F 2c	76 <sup>[c]</sup> (97:3)	$F \rightarrow F \rightarrow$	92 (99:1)
F F 2d	92 (98:2)	$F \rightarrow F \rightarrow$	22 (96:4)
$H_{3}C$ $F$ $F$ $F$ $F$ $F$ $2e$	61 <sup>[d]</sup> (99:1)		87 (99:1)
MeO 2f	99 <sup>[b]</sup> (97:3)		99 <sup>[f]</sup> (98:2)
F OMe 2g	66 <sup>[e]</sup> (97:3)		97 <sup>[f]</sup> (98:2)
$F \xrightarrow{F} CH_3$ F F 2h	87 (99:1)	F F F 2r	trace <sup>[f]</sup>
F 2i	0	$F \\ F \\ F \\ 2s$	90 (99:1)
	95 (98:2)	F F 2t	84 (99:1)

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zoates and other substrates bearing only one substituent in the *ortho*-position were unreactive. Polyfluorinated arenes gave particularly high yields, and methoxy-substituted arenes were also smoothly converted (2f, g).

On the allyl side, several substituents were tolerated both in the 2- and 3-positions. Bulky substituents such as 3-cyclohexyl groups (2n, r) seem to hamper the reaction, whereas 3-phenyl groups lead to even better yields.

Double bond migration with formation of **3** was observed in less than 4% yield for substrate **2n**, which bears a cyclohexyl-substituted allyl group, and in even smaller amounts for all other products.

We next investigated whether this transformation is restricted to palladium catalysts only. Nickel(0) complexes appeared to be promising candidates, since they had successfully been used in C–O activation.<sup>[7]</sup> Indeed, a systematic survey revealed that allyl pentafluorobenzoate can efficiently be decarboxylated by a Ni(0) catalyst generated *in situ* from nickel(II) chloride, BINAP, and zinc powder (see the Supporting Information, Table S1). With this system, **2a** was obtained in 82% yield at 100 °C in NMP (Scheme 2).

For the 2,6-difluorobenzoate, the allylarene **2d** was obtained in 39% yield after 16 h at 150 °C using a dppe ligand. For even less activated substrates such as **1g** or **1k**, the product was obtained only in traces using this prototypical nickel catalyst.

In conclusion, the advantageous concept of forming C–C bonds by catalytic ester decarboxylation was successfully employed for benzoic acid substrates. In the presence of a  $Pd_2(dba)_3/P(p-Tol)_3$  catalyst, various allylarenes were synthesized in good yields, along with CO<sub>2</sub> as the only by-product. Particularly activated substrates can also be converted by inexpensive nickel catalysts. Further catalyst development aiming at extending this sustainable transformation to the entire range of allyl benzoates is currently underway.

# **Experimental Section**

# General Procedure for the Palladium-Catalyzed Decarboxylative Allylation

A 20-mL vessel was charged with  $Pd_2(dba)_3$  (22.9 mg, 0.025 mmol) and tri-*p*-tolylphosphine (7.76 mg, 0.025 mmol) and the vessel was brought under an atmosphere of dry nitrogen. 1,4-Dioxane (4 mL) and allyl 2,3,4,5,6-pentafluorobenzoate (**1a**, 252 mg, 1.00 mmol) were added *via* syringe and the mixture was stirred at 110 °C for 24 h. After cooling to room temperature, the mixture was diluted with *n*-pentane (20 mL), washed with aqueous 1N NaOH (3×20 mL), water (20 mL) and brine (20 mL), dried over MgSO<sub>4</sub> and filtered. The solvent was removed at ambient pressure and the products **2a–t** were isolated from the residue by flash column chromatography (SiO<sub>2</sub>, Et<sub>2</sub>O/*n*-pentane gradient).

#### Synthesis of Allyl 2,3,4,5,6-Pentafluorobenzene (2a)

Following the general procedure, compound **2a** [CAS: 1736-60-3] was synthesized from allyl 2,3,4,5,6-pentafluorobenzoate (**1a**, 252 mg, 1.00 mmol). The product was obtained as a colorless liquid; yield: 183 mg (879 µmol, 88%). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>):  $\delta$ =5.82–5.96 (m, 1H), 5.03–5.16 (m, 2H), 3.45 (dt, *J*=6.4, 1.7 Hz, 2H); <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>):  $\delta$ =145.1 (m), 139.8 (m), 137.2 (m), 132.9 (s), 116.8– 117.2 (m), 112.8–113.4 (m), 26.3 (d, *J*=1.47 Hz); <sup>19</sup>F NMR (376 MHz, CDCl<sub>3</sub>):  $\delta$ =-144.23 to -144.05 (m, 1F), -157.73 to -157.38 (m, 1F), -163.04 to -162.75 (m, 1F); IR (NaCl): *v*=1655, 1643, 1503, 1443, 1415, 1315, 1299, 1219, 1123, 1011, 983, 911, 895, 752, 692, 624 cm<sup>-1</sup>; HR-MS-EI (TOF): *m*/*z*=208.0319 [M<sup>+</sup>], calcd. for C<sub>9</sub>H<sub>3</sub>F<sub>5</sub>: 208.0311; anal. calcd. for C<sub>9</sub>H<sub>3</sub>F<sub>5</sub>: C 51.94, H 2.42; found: C 52.20, H 2.59.

#### Nickel-Catalyzed Decarboxylative Allylation of 1a

A crimp-cap reaction vessel was charged with nickel(II) chloride (13.0 mg, 0.10 mmol), BINAP (76.2 mg, 0.12 mmol) and zinc powder (65.4 mg, 1.00 mmol). Under an inert atmosphere, degassed NMP (2 mL) and **1a** (252 mg, 1.00 mmol, 183  $\mu$ L) were added *via* syringe. The reaction





<sup>&</sup>lt;sup>[a]</sup> *Reaction conditions:* Allyl ester **1a-t** (1.00 mmol), Pd<sub>2</sub>(dba)<sub>3</sub> (2.5 mol%), P(*p*-Tol)<sub>3</sub> (2.5 mol%), 1,4-dioxane (4 mL), N<sub>2</sub> atmosphere, 110 °C, 24 h, isolated yields, product ratios were determined by <sup>19</sup>F NMR and GC analysis.

<sup>[b]</sup> 4 mol% P(*p*-Tol)<sub>3</sub>.

<sup>[e]</sup> 5 mol%  $Pd_2(dba)_3$ , 5 mol%  $P(p-Tol)_3$ .

<sup>&</sup>lt;sup>[c]</sup> 5 mol%  $P(p-Tol)_3$ .

<sup>&</sup>lt;sup>[d]</sup> 3.75 mol%  $P(p-Tol)_3$ .

<sup>&</sup>lt;sup>[f]</sup> 130°C.

mixture was stirred at 100 °C for 16 h and then cooled to room temperature. The slight pressure build-up caused by the partially dissolved CO<sub>2</sub> was carefully released by piercing the septum with a syringe needle before uncapping. Pentane (20 mL) was added, and the mixture was washed with water (2 × 20 mL) and brine (20 mL), dried over MgSO<sub>4</sub>, filtered and concentrated (40 °C). The crude product was further purified by flash chromatography (SiO<sub>2</sub>, pentane), yielding the allylbenzene **2a** as colorless liquid; yield: 171 mg (0.82 mmol, 82%). The analytical data matched those described for allyl 2,3,4,5,6-pentafluorobenzene [CAS: 1736-60-3].

**Note Added in Proof:** A related reaction stoichiometric in silver was simultaneously disclosed by the Jana group.<sup>[16]</sup>

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