Cascade Reactions

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Asymmetric Cascade Reaction to Allylic Sulfonamides from Allylic Alcohols by Palladium(II)/Base-Catalyzed Rearrangement of Allylic Carbamates**

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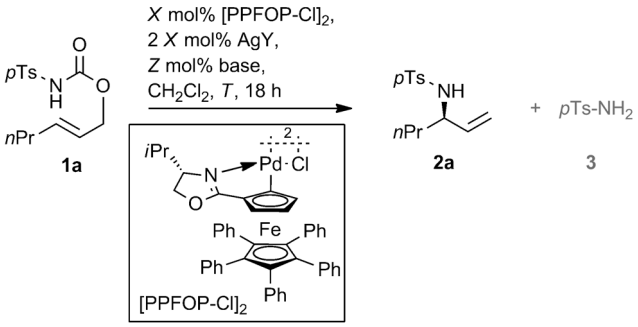
Abstract: *A regio- and enantioselective tandem reaction is reported capable of directly transforming readily accessible achiral allylic alcohols into chiral sulfonyl-protected allylic amines. The reaction is catalyzed by the cooperative action of a chiral ferrocene palladacycle and a tertiary amine base and combines high step-economy with operational simplicity (e.g. no need for inert-gas atmosphere or catalyst activation). Mechanistic studies support a Pd^{II}-catalyzed [3,3] rearrangement of allylic carbamates—generated in situ from the allylic alcohol and an isocyanate—as the key step, which is followed by a decarboxylation.*

Chiral α-branched allylic amines are valuable synthetic building blocks and their catalytic enantioselective synthesis has been intensively studied. Two of the most versatile strategies to form allylic amines with high levels of enantio- and regioselectivity are the Ir-catalyzed allylic amination[1–3] and the Pd^{II}-catalyzed Overman (aza-Claisen) rearrangement of allylic imidates.[4,5] For the latter method the undesired regioisomer is often not detected as a consequence of a rearrangement mechanism via a six-membered cyclic intermediate.[6] The aza-Claisen rearrangement generates carboxamide-protected amines. Unfortunately, only for trichloro- and trifluoroacetamide products the protecting groups could be readily removed.[7,8] Preparation of the trifluoroacetimidate substrates is relatively tedious and expensive, requires the use of CCl_4, and produces large amounts of PPh_3-based waste.[9] In addition, the isolation and storage of trifluoroacetimidates is often hampered by their sensitivity towards hydrolysis.

Herein, we report an alternative, operationally simple tandem reaction, which transforms linear achiral allylic alcohols with high regio- and enantioselectivity into branched chiral sulfonyl protected allylic amines.[10] This reaction proceeds via allylic carbamate intermediates, which undergo a Pd^{II}-catalyzed asymmetric [3,3] rearrangement followed by a decarboxylation step.[11,12]

In our initial studies we examined the possibility of a decarboxylative asymmetric rearrangement with the allylic *N*-tosyl carbamate **1a** (Table 1). The pentaphenylferrocene oxazoline palladacycle $[PPFOP\text{-}Cl]_2$—in the initial studies activated by treatment with $AgNO_3$ for chloride exchange—produced small amounts of rearrangement product **2a** after 18 h in a sealed tube in CH_2Cl_2 at 60 °C (entry 1), yet in almost racemic form. To increase the reactivity of the carbamate N-center, different bases were studied as stoichiometric additives. O-bases resulted in traces of **2a**, at most, but sterically demanding tertiary amines like iPr_2NEt and proton sponge (PS, 1,8-bis(*N*,*N*-dimethylamino)naphthalene) caused a strong increase in reactivity (entries 2 and 3). The product was formed in high yields and with good enantioselectivity, also even when only catalytic amounts of PS were used (entry 4).[13]

Table 1: Development of the enantioselective allylic carbamate rearrangement.



Entry	*X*	Y^-	*Z*	Base	*T* [°C]	Yield [%][a] **3**	**2a**	*ee* [%][b] **2a**
1	5	NO_3^-	0	–	60	17	10	9
2	3	NO_3^-	100	iPr_2NEt	60	6	87	89
3	3	NO_3^-	100	PS	60	3	96	88
4	3	NO_3^-	10	PS	60	5	91	86
5	2	NO_3^-	20	PS	60	6	87	86
6	2	PF_6^-	20	PS	60	14	75	92
7	2	TfO^-	20	PS	60	14	70	91
8	2	$F_3CCO_2^-$	20	PS	60	4	95	87
9	1	$F_3CCO_2^-$	20	PS	40	3	95	88
10	1	–[c]	20	PS	40	4	22	93
11[d]	1	–[c]	25	PS	85	6	88	92

[a] Yield determined by ^1H NMR analysis using an internal standard. [b] Enantiomeric excess determined by HPLC. [c] No silver salt was used for catalyst activation. [d] $CHCl_3$ was used as solvent.

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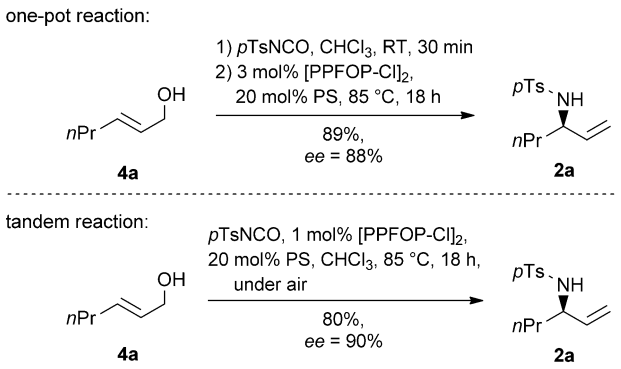
Various silver salts AgY were studied for the catalyst activation.[7e,8,14] Nitrate (entry 5), and $F_3CCO_2^-$ (entry 8) and a number of other anionic ligands Y^- (not shown) resulted in similar yields and *ee* values, employing 2 mol% of precatalyst. With weakly or noncoordinating anions like triflate (entry 7) and PF_6^- (entry 6) more substrate decomposition towards **3** was noticed.[15] With trifluoroacetate the rearrangement was still efficient at 40°C and with 1 mol% of the precatalyst (entry 9). When the precatalyst was not activated by a silver salt, the reactivity was low under these conditions in agreement with our previous investigations (entry 10).[7e,8h,14,16] For the above-mentioned aza-Claisen rearrangement using the same precatalyst, it was even necessary to use an excess of silver salt in order to generate a paramagnetic Pd^{III} species, which offered significantly higher catalytic activity.[8i] This type of catalyst oxidation is not required in the allylic carbamate rearrangement. Also the chloride exchange can be avoided, if the rearrangement is conducted at a higher temperature. At 85°C in $CHCl_3$ using the non-activated $[PPFOP\text{-}Cl]_2$, **2a** was formed in 88% yield and with 92% *ee* (entry 11).

To further improve the operational simplicity, the option of a one-pot procedure was explored, in which the allylic alcohol is used as the substrate (Scheme 1, top).[17] The latter



Scheme 1. Step-economic approaches towards **2a**.

was stirred with 1 equivalent of *p*TsNCO in $CHCl_3$ for 30 min at room temperature before $[PPFOP\text{-}Cl]_2$ (3 mol%) and PS (20 mol%) were added.[18] At 85°C in a sealed tube, the product was again formed in high yield and with high enantioselectivity. This prompted us to inspect a tandem version, in which all reaction components were directly added without separate preformation of the allylic carbamate.[19] This simple procedure led to an almost identical reaction outcome, even in the presence of air (Scheme 1, bottom).

This cascade reaction was investigated for different substrates (Table 2). When the olefin substituent R was an aliphatic group, the product was usually formed in good to high yields and with high regio- and enantioselectivity. The most difficult example in terms of enantioselection, in which R = Me, gave the product with an *ee* of 72% (entry 3), whereas for the other examples the *ee* values ranged from 89–98%. Substrates with α- and β-branched alkyl residues (entries 9 and 10) could also be utilized under the standard

Table 2: Application of the cascade title reaction.

Entry	4/2	R	Yield [%][a]	RS [%][b]	*ee* [%][c]
1	**a**	*n*Pr	80	20:1	90
2	**b**	Et	88	16:1	91
3	**c**	Me	90	40:1	72
4	**d**	*n*Pent	85	22:1	93
5	**e**	$(CH_2)_2Ph$	86	25:1	92
6	**f**	$(CH_2)_2CO_2Me$	80	26:1	90
7	**g**	CH_2OTBS	83	20:1	98
8	**h**	CH_2OBn	90	20:1	98
9	**i**	*i*Pr	52	7:1	92
10	**j**	*i*Bu	79	10:1	89

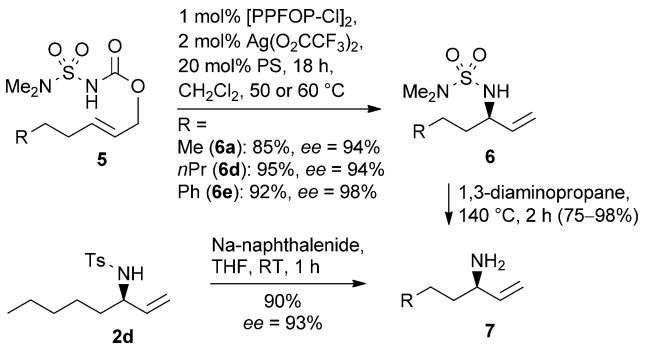
[a] Yield of isolated product. [b] Regioselectivity determined by ^1H NMR analysis of the crude product. [c] Enantiomeric excess determined by HPLC.

conditions and provided high enantioselectivity, albeit with reduced reactivity in the first case. In addition, several functional groups were compatible with the cascade reaction conditions as shown in Table 2 for an ether moiety (entry 8), a silyl ether (entry 7), and an ester residue (entry 6), thus providing rapid access to protected chiral β-amino alcohol and γ-amino acid derivatives possessing an olefin moiety for further synthetic manipulations. In contrast, aromatic residues R were not well tolerated. The observed substrate preference seems to be complementary to that of the Ir-catalyzed allylic aminations, for which aromatic residues R often led to higher regioselectivities than aliphatic ones.[1]

Like the Pd^{II}-catalyzed allylic imidate rearrangement, the title reaction is slower for *Z*-configured substrates. Under the conditions listed in Table 2, the product **2a** was formed in only 29% yield starting from (*Z*)-**4a** and with opposite absolute configuration (75% *ee*, not shown in Table 2). The allylic alcohols should thus be nearly geometrically pure for the best possible enantioselectivity (see also the Supporting Information).

The scalability of the cascade reaction has been examined for substrate **4d** on a gram scale. Repeating the reaction of Table 2, entry 4 with 5.24 mmol of substrate provided 1.366 g of **2d** (92% yield) with 91% *ee*.

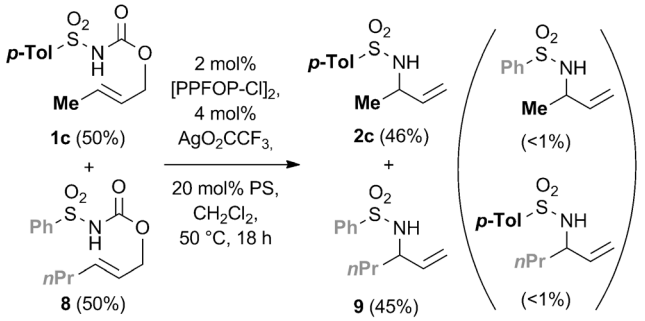
Tosyl protecting groups on allylic amines can often be removed in good yields under reductive conditions, even in large-scale industrial processes.[20] To showcase the utility of the products, **2d** was deprotected under standard conditions[20c] in high yield and with no loss of optical purity (Scheme 2). The decarboxylative allylic carbamate rearrangement also offers the opportunity of altering the *N*-protecting group by the formation of different allylic carbamates. This has been demonstrated for carbamates **5** carrying a dimethylaminosulfonyl protecting group (Scheme 2). In this case the rearrangement proceeded with high yields and regioselectivities (**6a**/**d**: 24:1; **6e**: 39:1) and gave the sulfonamides **6** with *ee* values of 94–98%. Deprotection of **6** under standard con-



Scheme 2. Application of a dimethylaminosulfonyl protecting group.

ditions[21] using 1,3-diaminopropane furnished the free amines in high yields. For substrate **5e** we have also studied the use of the non-activated catalyst $[PPFOP\text{-}Cl]_2$ (1.0 mol%) in the presence of PS (20 mol%). Gratifyingly, in $CHCl_3$ the product was again formed in good yield (93%) with a high *ee* value (96%) and good regioselectivity (24:1) after 24 h at 80 °C (not shown).[22]

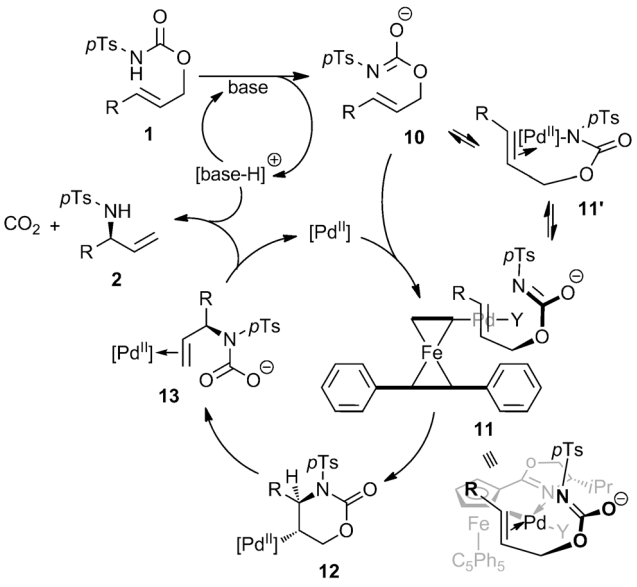
Crossover experiments have been performed to determine whether the sulfonamide formation proceeds by a rearrangement mechanism. A 1:1 mixture of allylic carbamates **1c** and **8**, which differ in both the N-sulfonyl moiety and the olefin substituent, was treated with the palladacycle catalyst (Scheme 3). The two products that are expected for an



Scheme 3. Crossover experiment confirming an intramolecular reaction pathway.

intramolecular pathway were formed in equally high quantities, whereas crossover products were present in only trace quantities (< 1% in GC–MS). For that reason the reaction most likely occurs by means of an intramolecular carbamate rearrangement, and not by an allylic substitution pathway.

Based on the result of these crossover experiments and the absolute configuration of the products[23] we suggest the mechanism depicted in Scheme 4. We propose—like in the allylic imidate rearrangement[7e]—a face-selective coordination of the olefin moiety to the Pd^{II} center in **11**. Based on the typical coordination properties of ferrocene palladacycles,[7e,24] the neutral olefin is expected to coordinate *trans* to the oxazoline N atom. A stereoelectronically preferred orientation of the olefin part parallel to the ferrocene axis, that is, perpendicular to the Pd square plane,[25] is likely for



Scheme 4. Possible simplified mechanism of the Pd^{II}/base-catalyzed decarboxylative allylic carbamate rearrangement.

the ground-state conformation in which the allylic C-1 methylene moiety with its relatively low steric demand points towards the massive C_5Ph_5 ligand, while the sulfonyl moiety avoids the ferrocene core to minimize repulsive interactions (Scheme 4). Coordination of the enantiotopic olefin face is assumed to be less favorable for steric reasons. In one possible scenario, the deprotonated carbamate N atom could bind to the Pd atom (*cis* to the oxazoline N) and could attack the olefin within this chelate **11′** by an inner-sphere mechanism. However, since olefin insertions of this type are usually concerted processes, the reactive conformation would require the olefin double bond to be in the Pd^{II} square plane, thus entailing a rotation of the coordinated olefin by roughly 90°.[25] This should cause considerable repulsion between the C_5Ph_5 ligand and an olefin substituent. In the alternative case of an outer-sphere mechanism via olefin complex **11** the anionic N center would attack from the face remote to the Pd center. Inner- and outer-sphere mechanisms are thus expected to provide different enantiomers. For the proposed face selectivity of the substrate coordination only the suggested outer-sphere attack would be in agreement with the absolute configuration of the major product enantiomers.

Kinetic investigations by ^1H NMR spectroscopy showed a nearly linear relationship of the product yield and the reaction time for yields up to about 80% (see the Supporting Information) indicating a pseudo-zero-order kinetic dependence on the carbamate. This suggests 1) a substrate saturation and thus a high tendency of the substrate to coordinate, 2) C–N bond formation to be the probable rate-limiting step, and 3) that catalyst deactivation or decomposition play only a minor role. The high affinity for the substrate coordination, which is not observed in the aza-Claisen rearrangement for the same catalyst,[7e] might be explained by an initial formation of chelate complex **11′**, in which both the olefin and the anionic carbamate moiety bind to the metal center. This would be in agreement with the observation that the type

of anionic ligand in the activated catalysts is less critical than usually found and that even the often inert chloride-bridged dimer [PPFOP-Cl]$_2$ is a competent catalyst at more higher temperatures, as Y^- or chloride would be replaced by the deprotonated substrate. The suggested outer-sphere mechanism would require a dissociation of the anionic carbamate moiety prior to the rearrangement.

To confirm substrate saturation, the reaction mixture was examined by ESI-HRMS after 0.5 h at 35 °C using 5 mol % of [PPFOP-O_2CCF_3]$_2$. In the dominating ferrocene palladacycle detected (carrying not only the C,N ligand), one substrate molecule is bound to a monomeric palladacycle (see the Supporting Information). Heavier species, in which, for example, two substrate molecules bind to one palladacycle, provided smaller peaks. ^{19}F NMR spectroscopy confirmed that nearly none ($<2\%$) of the initially generated activated catalyst species [PPFOP-O_2CCF_3]$_2$ is still present under the reaction conditions. The large majority of $^-O_2CCF_3$ seems not to bind any more to the catalyst (broad signals, see the Supporting Information).

In conclusion, we have described a step-economic catalytic asymmetric methodology capable of transforming achiral allylic alcohols in a single step and with high enantio- and regioselectivity into sulfonyl-protected chiral allylic amines. These reactions have been demonstrated to proceed through a decarboxylative rearrangement of allylic carbamates, which explains the preference for the branched allylic product regioisomers. The allylic carbamates can be generated in situ by addition of the corresponding allylic alcohol to an isocyanate. The title reaction offers the additional practical advantages that it can be performed under air and that catalyst activation by a silver salt, which is necessary for many other chiral palladacycle-catalyzed asymmetric reactions, is not required in the present case.

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[23] The absolute configuration of **2a** was determined by X-ray analysis. CCDC 990155 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif. For products **2a–d**,**h–j** the absolute configurations have already been assigned (see the Supporting Information).

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