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Synthesis of β-Amino Acid Derivatives via Enantioselective Lewis Base Catalyzed N-Allylation of Halogenated Amides with Morita-Baylis-Hillman Carbonates

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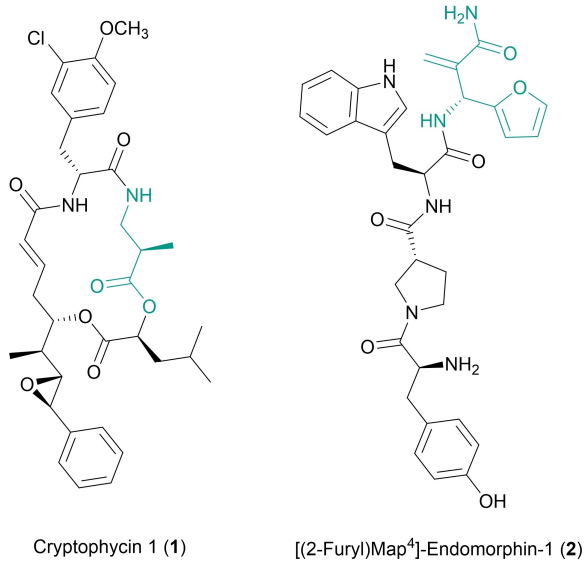
Trifluoro- and trichloroacetamides serving as pronucleophiles undergo enantioselective Lewis base catalyzed *N*-allylation with Morita-Baylis-Hillman carbonates to produce enantioenriched β-amino acid derivatives. The reactions proceed as a kinetic resolution to give the allylation products and the remaining carbonates in good yields and high enantioselectivity. The obtained products are amenable to diastereoselective derivatization to produce a library of spiro-isoxazoline lactams.

Introduction

Nearly 40% of small molecule drugs bear a chiral amine scaffold in their structure.[1] Among them, β-amino acids are of particular interest in organic synthesis. In many cases, this scaffold is essential for the biological activity of various peptides and alkaloids (Figure 1).[1b,2] For example, depsipeptide cryptophycin 1 (**1**) was investigated as potent microtubule binder and inhibitor of cell proliferation,[2c] whereas α-methylene-β-amino propanoic amide **2** has been studied as a potent agonist of the μ-opioid receptor and an antinociceptive agent.[3] With these examples and the growing number of natural products containing β-amino acids,[4] the development of new enantioselective processes for synthesis of β-amino acid building blocks remains a pertinent goal.

Diverse routes for the synthesis of enantioenriched substituted β-amino acids have been reported. Asymmetric carbon-carbon, carbon-nitrogen and carbon-hydrogen bond-forming reactions are the most common approaches for the catalytic synthesis of β-amino acids (Scheme 1a, paths a-d).[5] Selected examples include zirconium catalyzed enantioselective Mannich type reaction of aldoximes with silyl enol ethers (path a),[6] biocatalytic transaminations (path b),[1a] asymmetric C–H insertion of metal carbenoids into *N*-protected methylamines (path c)[7] and enantioselective hydrogenation of β-aminoacrylic acid derivatives (path d).[8] Despite the versatility of these methods, limited substrate scope or poor functional group tolerance leave room for improvement and development of new approaches to these scaffolds.[9]



Cryptophycin 1 (**1**) [(2-Furyl)Map[4]]-Endomorphin-1 (**2**)

Figure 1. Selected examples of natural products and pharmaceuticals bearing β-amino acid moiety

In our previous work we demonstrated that the concept of latent pronucleophiles in Lewis base catalysis can be effectively applied to allylation of N-heterocycles,[10] and we looked to extend this method to other types of *N*-centered nucleophiles and, in particular, to ammonia and alkyl amines as an entry to chiral β-amino acids. The direct application of ammonia or alkyl amine based pronucleophiles with representative allylic electrophiles proved difficult due to the competition of the pronucleophiles with the Lewis base catalysts and formation of the undesired regioisomers (Scheme 1b). This called for a redesign of the reaction partners with emphasis on lowering the nucleophilicity of the N-centered nucleophile while allowing its activation during the reaction and easy removal upon allylation.

For development of the enantioselective Lewis base catalyzed allylation, the *N*-centered nucleophiles should meet

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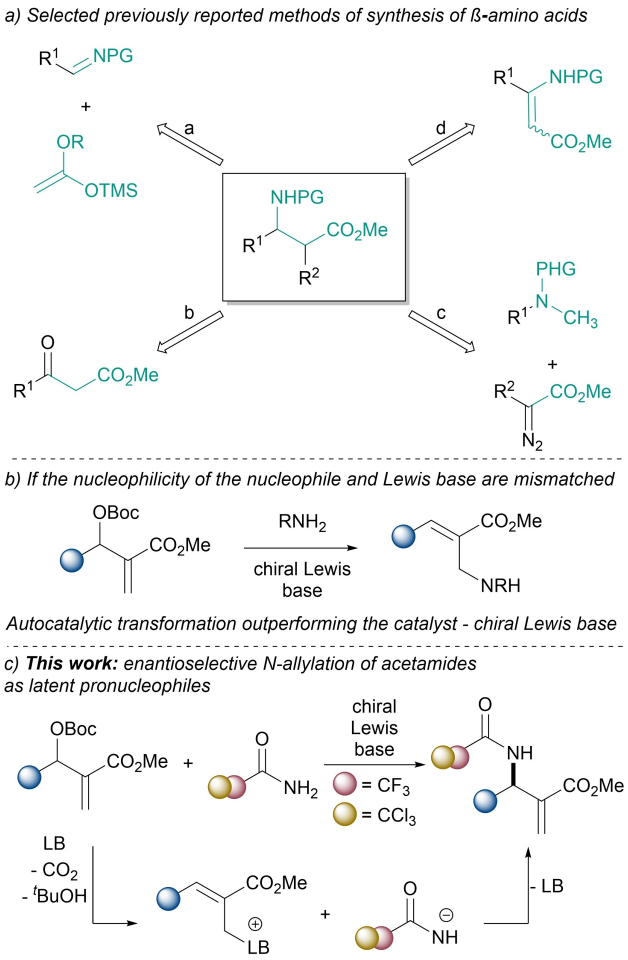


Scheme 1. a) Examples of previously reported catalytic methods of synthesis of β-amino acids. b) Reaction outcome dependent on nucleophilicity of the nucleophile and the Lewis base catalysts. c) Proposed pathway of the enantioselective Lewis base catalyzed N-allylation of halogenated acetamides with MBH carbonates.

several requirements.[11] We envisioned that using the halogenated acetamides as pronucleophiles and surrogates of ammonia would be effective. The nucleophilicity of these compounds would be lower than that of the parent nucleophile, ammonia, which would render them less reactive than the Lewis base catalyst (Scheme 1c). The acidity of these compounds should be sufficient to allow for their activation by deprotonation using a suitable leaving group from the allylic electrophile making allylic carbonates perfect reaction partners. We hypothesized that, in this scenario, the addition of Lewis base catalyst would produce the activated electrophile and basic leaving group which would activate the pronucleophile via deprotonation, a common strategy in substitution of Morita-Baylis-Hillman (MBH) derivatives.[11–12,13a,c] The subsequent addition of the activated pronucleophile to the activated electrophile would form the desired C–N bond and release the Lewis base catalysts thus closing the catalytic cycle. The resulting products, halogenated derivatives of chiral β-amino acids, should allow for cleavage of halogenated acetamides under mild conditions and afford chiral β-amino acids making halogenated acetamides excellent surrogates of ammonia in these reactions. Herein, we report the Lewis base catalyzed synthesis of chiral β-amino acids and their derivatives employing an enantioselective *N*-allylation of trifluoro- and trichloroacetamides as latent pronucleophiles with Morita-Baylis-Hillman carbonates.

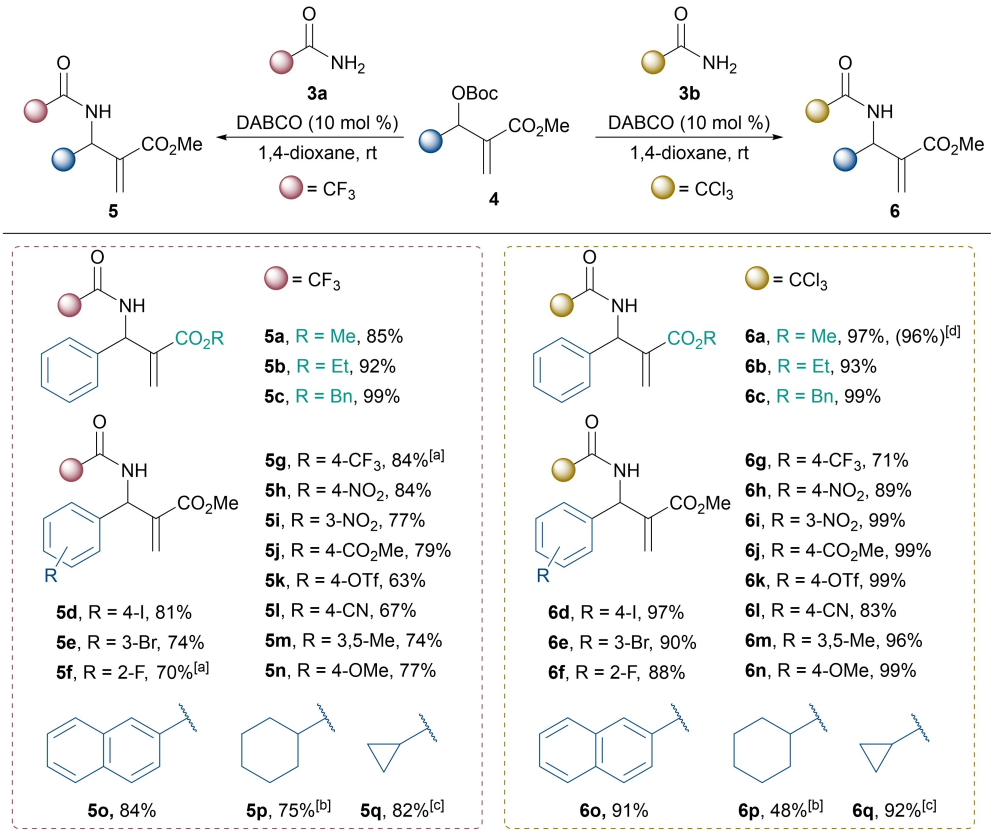
Results and Discussion

Our work commenced with optimization of the reaction conditions for the model reaction between trifluoroacetic amide **3a** and allylic carbonate **4a**, derived from MBH alcohol adduct of methyl acrylate and benzaldehyde (Scheme 2). The influence of various solvents and Lewis bases, as well as catalyst loading and stoichiometry of the reactants were evaluated during the optimization studies. For practical reason (short reaction time) the concentration of the solvent was not altered (please see the Supporting information for details on the optimization studies). The optimized conditions included reactions with slight excess of trifluoroacetamide **3a** and 10 mol% of DABCO as a catalyst in 1,4-dioxane at room temperature. Under optimized conditions, the protected β-amino acid derivative **5a** was obtained with 85% yield within 30 min. We hypothesized that this transformation could be applied to similar *N*-centered nucleophiles. Indeed, DABCO catalyzed alkylation of MBH carbonate **4a** with trichloroacetamide **3b** resulted in the desired β-amino acid derivative **6a** with 97% yield.

Both trifluoroacetamide **3a** (Scheme 2, left) and trichloroacetamide **3b** (Scheme 2, right) underwent an efficient DABCO catalyzed allylation with various MBH carbonates **4**. The reaction showed generally broad tolerance for structural changes within the MBH carbonates. The exchange of methyl ester with ethyl or benzyl analogues did not affect the reaction outcome (**5b–c**, **6b–c**). Halogen-substituted MBH carbonates gave corresponding allylation products (**5d–f**, **6d–f**) in good yields regardless of the substitution pattern although higher catalyst loading was required for *ortho*-substituted electrophile (**5f**) which is consistent with the increased steric bulk close to the reactive center. Substrates with both electron-withdrawing (**5g–l**, **6g–l**) and electron-donating (**5m–n**, **6m–n**) substituents afforded the desired products in good to excellent yields. Extended π-system of the electrophile was also well tolerated (**5o** and **6o**). Alkyl carbonates proved to be reactive under the reaction conditions as well, albeit yield deterioration and extended reaction time were observed for secondary alkyl substituents (**5p–q**, **6p–q**).

For substrates that required longer reactions times, reaction rates could be effectively addressed by increasing the catalyst loading (**5g**). Notably, the synthesis of β-amino acid **6a** could be performed on a larger scale (1.4 mmol) with a lower catalyst loading (1 mol% DABCO) and without significant deterioration of yield. The product of formal SN2′ substitution (Scheme 1b) was not observed in any of the performed reactions.

Having established that the reaction of MBH carbonates with the halogenated acetamides is highly regioselective and features a broad substrate scope, we focused on the development of the enantioselective allylation. Since DABCO showed the highest efficacy as the catalyst, cinchona-alkaloid-based



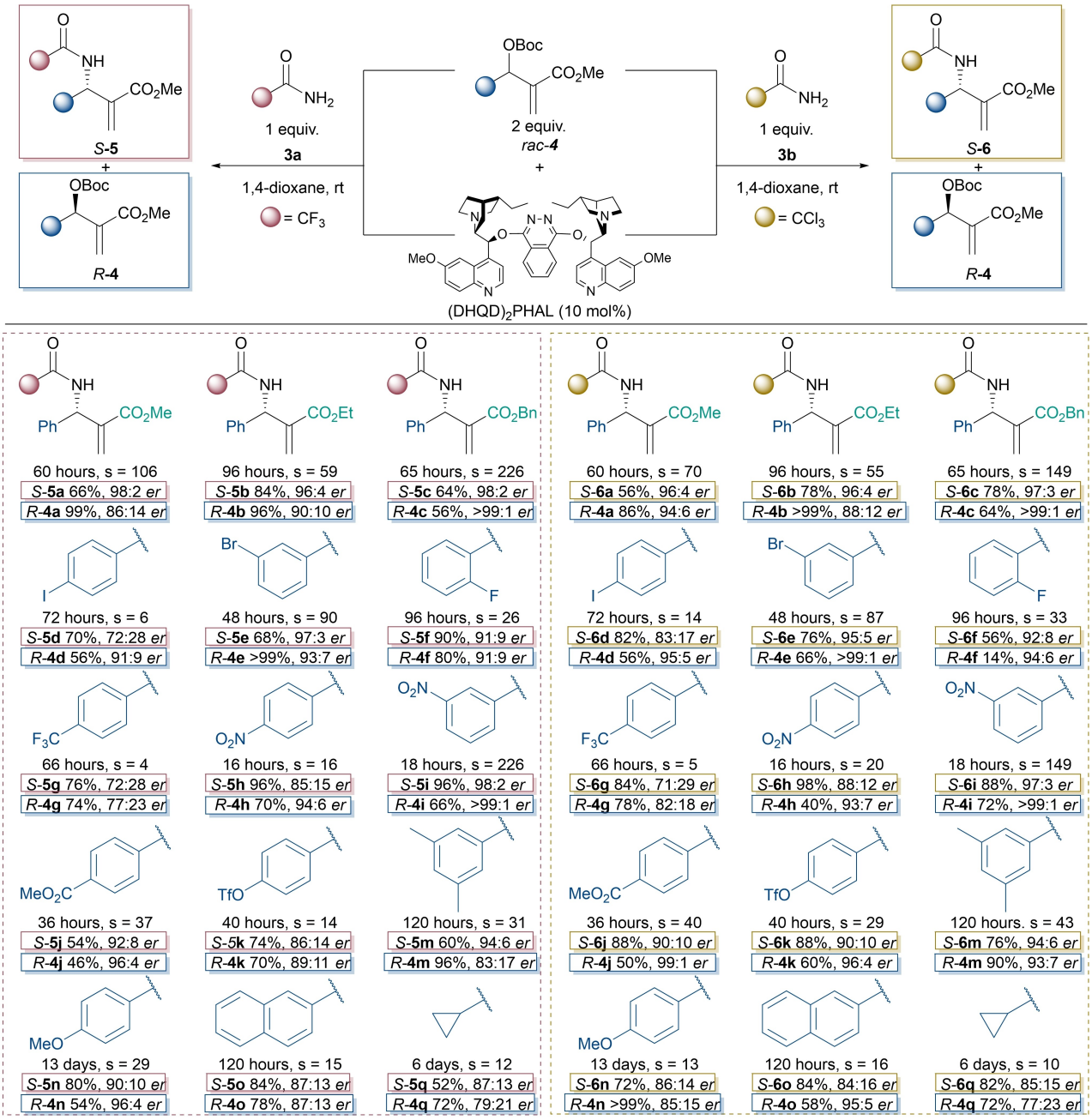
Scheme 2. DABCO catalyzed allylation of trifluoroacetamide **3a** and trichloroacetamide **3b** with MBH carbonates **4**. Allylic carbonate (1 equiv.), halogenated acetamide (1.5 equiv.) and DABCO (10 mol%) as catalyst in 1,4-dioxane (0.06 M) at room temperature. [a]DABCO (20 mol%) was used. [b]Reaction was performed with 3 equiv. of **4p** and 20 mol% DABCO. Reaction time was 4 h. [c]DABCO (30 mol%) was used. [d]Reaction was performed on 1.4 mmol scale using 1 mol% DABCO.

Lewis base catalysts were selected for further investigation. Multiple reported studies employing these chiral amines for asymmetric alkylation of diverse MBH derivatives strongly supported the choice of the type of chiral catalyst.[10–13] During the optimization studies we evaluated influence of the catalyst and solvent, concentration, catalyst loading, stoichiometry of the reactants and the reaction temperature (please see the Supporting information for details of optimization studies). In the presence of $(DHQD)_2PHAL$, the allylation of trifluoroacetamide **3a** with MBH carbonate **4a** yielded the desired product *S*-**5a** with good level of stereoselectivity. However, despite the excess of the nucleophile, the yields remained below 50%. This observation was indicative of a kinetic resolution scenario with significant difference in reaction rates for the two enantiomers of MBH carbonate.[13d,f] Hence, the optimization was directed towards the use of two-fold excess of the MBH carbonate and included the transformation of carbonate (2 equiv.), halogenated acetamide (1 equiv.) with $(DHQD)_2PHAL$ (10 mol%) as the catalyst in 1,4-dioxane at room temperature.

With the optimized conditions in hand, we prepared a library of enantioenriched protected β-amino acid derivatives (Scheme 3) to test the reaction scope. The reactions were monitored by GC-analysis and were allowed to run until acetamide **3a** or **3b** were fully consumed. Alternatively, the reaction progress could be monitored by HPLC analysis on a chiral stationary phase. In this case, the reactions were stopped when no further changes in the enantiomeric ratio of the remaining carbonate were detected. The extended reaction time for asymmetric allylations of both halogenated acetamides comparing to the DABCO catalyzed ones agrees with the observations for similar cinchona alkaloid catalyzed transformations.[13e]

The enantioselective allylation of trifluoroacetamide **3a** (Scheme 3, left) and trichloroacetamide **3b** (Scheme 3, right) with the parent carbonate **4a** delivered corresponding protected β-amino acids in moderate yield and enantioselectivity of above 90:10 *er*. The allylation proceeded with good yields of roughly 80% and enantiomeric ratio of 96:4 to 98:2 regardless of the identity of the ester within the allylic carbonate (*S*-**5b–c**, *S*-**6b–c**).

Halogen substituted MBH carbonates were well tolerated, and all gave the desired products (*S*-**5d–f**, *S*-**6d–f**) in good yields (56-90%) with moderate to high enantioselectivity (72:28 to 97:3 *er*). Electron-deficient allylic carbonates predictably demonstrated high reaction rates and required only few hours to reach the full conversion of the acetamides (*S*-**5h–k**, *S*-**6h–k**). The desired products were delivered with good yields (54-98%) and good degree of stereocontrol (85:15 to 98:2 *er*). The 4-CF_3-substituted substrates afforded corresponding products (*S*-**5g** and *S*-**6g**) in good yields and moderate enantioselectivity



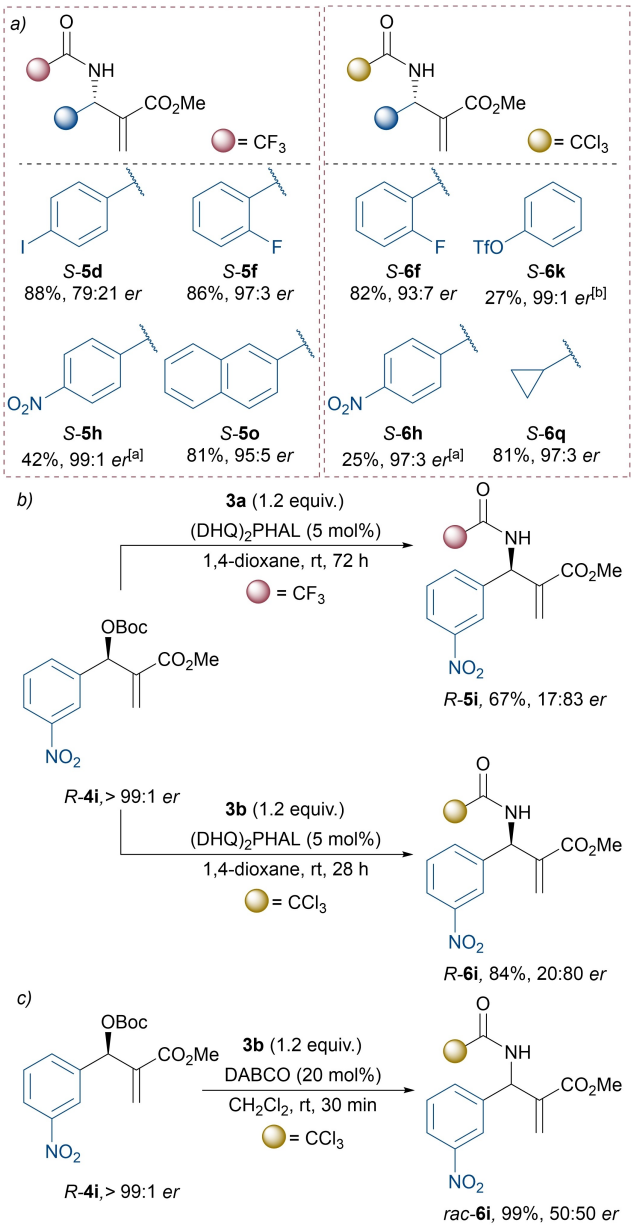
Scheme 3. Enantioselective allylation of trifluroacetamide **3a** and trichloroacetamide **3b** using MBH carbonates **4**. The reactions were performed using allylic carbonate (2 equiv.), halogenated acetamide (1 equiv.) and $(DQHD)_2PHAL$ (10 mol%) as catalyst in 1,4-dioxane (0.4 M) at room temperature.

(72:28 and 71:29 *er*). It is worth mentioning here, that substrates bearing *meta*-substituents (*S*-**5e**, *S*-**5i**, *S*-**6e** and *S*-**6i**) were transformed to the products with high degree of stereocontrol (94:6 to 98:2 *er*). Furthermore, the recovered starting material was highly enantioenriched (>99:1 *er*). The presence of electron donating groups noticeably decreased the reaction rates resulting in slow conversion on a time scale of several days (*S*-**5m** and *S*-**6m**) or even weeks (*S*-**5n** and *S*-**6n**). Nevertheless, the products were isolated with good yields (60-80%) and satisfactory level of enantioenrichment (86:14 to 90:10 *er*). Extended π-system of the carbonate performed well in these reactions too (*S*-**5o** and *S*-**6o**). MBH carbonate featuring a cyclopropyl scaffold delivered the desired products (*S*-**5q** and *S*-**6q**) with satisfactory yields and slightly lower stereocontrol (87:13 and 84:16 *er*).

The absolute configuration of the stereogenic center of the major product enantiomer when $(DHQD)_2PHAL$ was used as catalyst was assigned as *S* by comparison to previously reported data for allylic trichloroacetamide *S*-**6a**.[14] To exclude the possibility of the self-disproportion of enantiomers,[15] all enantiomeric ratios were determined by analysis of both the crude reaction mixtures and the isolated compounds using

HPLC with chiral stationary phase. The analysis was performed for the recovered starting materials (*R*-**4**) along with the isolated products (*S*-**5** and *S*-**6**). To access products with higher enantiomeric purity, recrystallization experiments were performed with selected acetamides to show that highly enantioenriched β-amino acids derivatives with *er* up to 99:1 can be obtained (Scheme 4a).

Simply switching the catalyst to $(DHQ)_2PHAL$, the pseudoenantiomer of $(HQD)_2PHAL$, allowed the synthesis of the corresponding protected *R*-β-amino acids in moderate yields and with slightly lower stereoselectivity (17:83 *er* for *R*-**5i** and 20:80 *er* for *R*-**6i**). The enantioenriched carbonates *R*-**4i** recovered from kinetic resolution reactions using $(DHQD)_2PHAL$ could be used as starting materials, with comparable reaction rates, for synthesis of the other enantiomers of β-amino acids (Scheme 4b) showing the preference of $(DHQD)_2PHAL$ and $(DHQ)_2PHAL$ for the opposite enantiomers of the allylic carbonate as well. In turn, the DABCO-catalyzed allylation of trichloroacetamide **3b** with *R*-**4i** resulted in formation of racemic product (Scheme 4c) clearly demonstrating that the stereochemical outcome of the reactions is under full catalyst control.

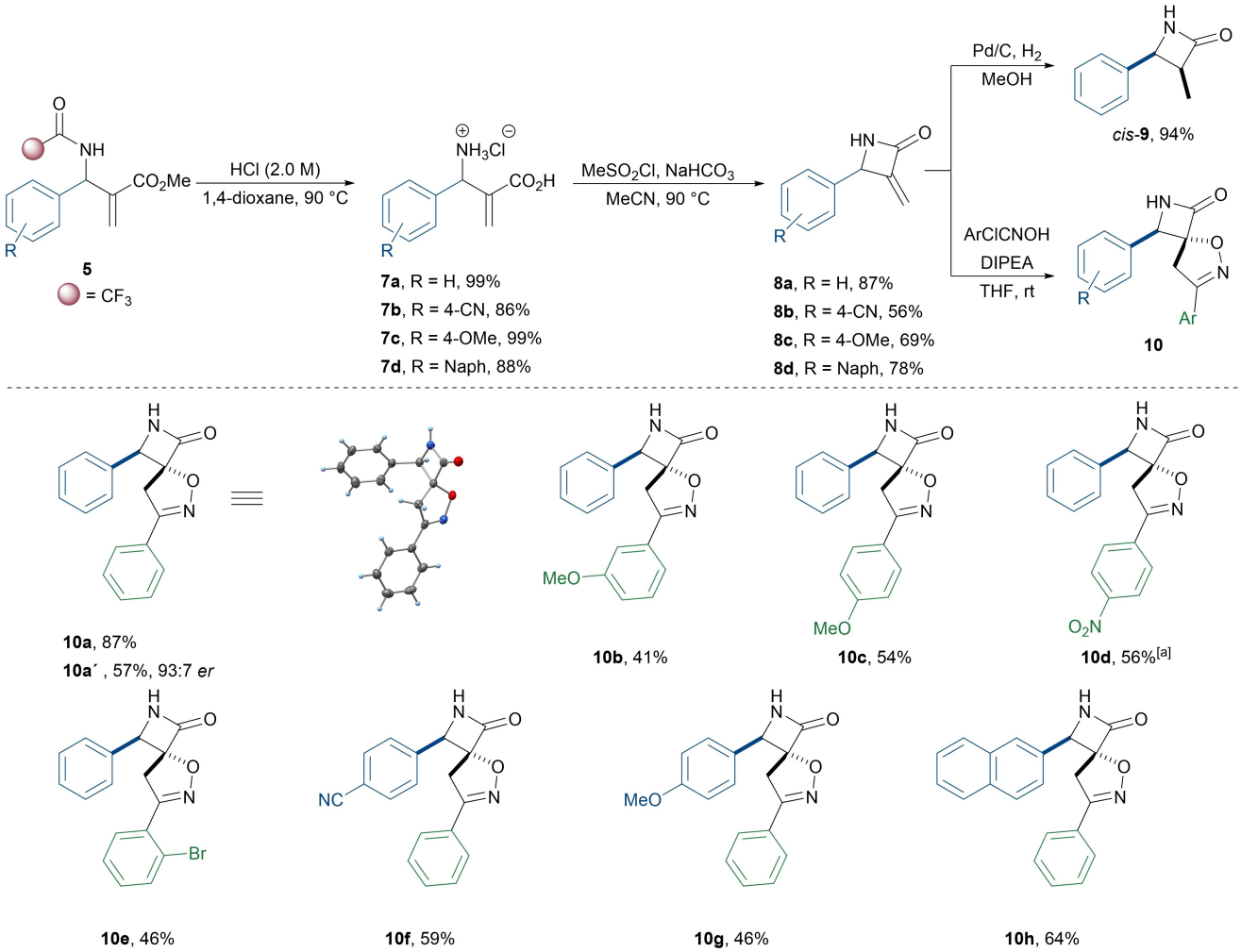


Scheme 4. a) Enantioenriched products after recrystallization from heptane. The structures are given with the yield of the recrystallization process and the *er* of the filtered precipitate. [a]Mixture of heptane and diethyl ether was used; enantioenriched product was isolated from the mother liquor. [b]Enantioenriched product was isolated from the mother liquor. b) Synthesis of *R*-**5i** and *R*-**6i** from enantioenriched carbonate. c) A comparative test with DABCO instead of $(DHQ)_2PHAL$ as a catalyst.

The synthetic utility of the prepared β-amino esters was demonstrated by converting them to the corresponding amino acid hydrochlorides **7** and β-lactams **8** (Scheme 5). The desired products were obtained with excellent yields regardless of the electronic properties of the starting materials. The presence of an *exo*-methylene group in the resulting lactam **8** was seen as an excellent opportunity for further modifications of the scaffold in a diastereoselective manner. Simple heterogeneous hydrogenation of *exo*-methylene lactam **8a** over the palladium catalyst provided the desired *cis*-β-lactam **9** in excellent yield.[16] Upon treatment of lactams **8** with α-chlorobenzaldoximes, a single diastereomer of a spiro-isoxazoline β-lactam **10** was obtained.[17] The relative stereochemistry of product **10a** was assigned based on a single-crystal X-ray diffraction analysis. Notably, during the [3+2] cycloaddition the level of stereoselectivity set in the previous steps was retained (**10a'**). Various substituted aryl chlorides performed well and delivered corresponding products with good yields without influencing the diastereoselectivity (**10b–e**).

Conclusions

In summary, we developed an efficient enantioselective synthesis of β-amino acids and their derivatives via enantioselective Lewis based catalyzed allylation of trifluoro- and trichloroacetamides with MBH carbonates. We demonstrated that the concept of latent pronucleophiles can be successfully applied for enantioselective *N*-allylation reactions. We introduced halogenated acetamides as suitable pronucleophile surrogates of ammonia where the electron-withdrawing group lowers the nucleophilicity of the parent nucleophile and sufficiently lowers the acidity of the N–H protons which allows for nucleophile activation via deprotonation during the course of the reactions. The approach features a broad substrate scope, good enantioselectivity and mild reaction conditions. The synthesized products can be transformed into desired amino acids via easy cleavage of the halogenated acetamides and further derivatives like β-lactams and spiro-isoxazolines can be accessed in a single step via highly diastereoselective transformations of the reaction products.



Scheme 5. Subsequent transformations towards synthesis of β-amino acids, lactams, and diastereoselective synthesis of spiro-isoxazoline lactams. [a]Yield is calculated based on the reisolated starting material. Triethylamine was used instead of DIPEA.

Supporting Information

Deposition Number 2310840 (for **10a**) contains the supplementary crystallographic data for this paper. These data are provided free of charge by the joint Cambridge Crystallographic Data Centre and Fachinformationszentrum Karlsruhe (http://www.ccdc.cam.ac.uk/structures).

Details of optimization studies, characterization data for new compounds and crystallographic data are provided in the supporting information document. The authors have cited additional references within the Supporting Information.

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Conflict of Interests

The authors declare no conflict of interest.

Data Availability Statement

The data that support the findings of this study are available from the corresponding author upon reasonable request.

Keywords: β-amino acid · Lewis base catalysis · Morita Baylis Hillman adducts · enantioselective synthesis · spiro compounds

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