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Highly diastereoselective crystallization-induced asymmetric transformation of 1,3-disubstituted-tetrahydro-β-carbolines in water†

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A green and highly stereoselective method for the synthesis of *cis* or *trans*-1,3-disubstituted-tetrahydro-β-carbolines has been developed using water as the solvent. A mixture of *cis* and *trans*-1,3-disubstituted-tetrahydro-β-carboline hydrochlorides can be converted to a single *cis* or *trans* isomer *via* a crystallization-induced asymmetric transformation process. It is possible to get both isomers using this method by incorporating different additives in water. This method has advantages such as environmental friendliness, high stereoselectivity, suitability for industrialization, and non-toxicity.

Introduction

Water is one of the most common resources on earth, and has played an important role in humanity's development and engenders life. As we know, enzymatic processes in nature can only occur in an aqueous environment. Water is not only a medium for biochemical reactions, but is also a reagent in some oxidation and reduction reactions in the body. As people pay more and more attention to environmental protection, there is a tendency to implement green chemistry in organic synthesis.[1] Chemists have devoted a lot of research effort to replacing harmful and toxic solvents with more environmentally friendly alternatives in the past two decades. Water is considered a green solvent for organic reactions, having advantages like non-toxicity, non-flammability, non-volatility, cheapness, and ready availability. One of the earliest examples was reported by Diels and Alder in 1931, for the Diels–Alder reaction of furan and maleic anhydride.[2] More and more researchers have carried out various organic reactions using water as a solvent in recent years.[3] Nevertheless, it is still a highly challenging task to find practical reactions which use water as solvent because of the drawbacks such as the insolubility of organic reactants and high surface tension.

Crystallization-induced asymmetric transformations (CIATs), which are also referred to as crystallization-induced stereoisomer transformations (CISTs), are a highly practical means for converting enantiomeric or diastereomeric mixtures into a single stereoisomeric product with a theoretical yield of 100% *via* simple crystallization under appropriate conditions.[4] It can in principle be divided into two types: crystallization-induced enantiomer transformations (CIETs) and crystallization-induced diastereomer transformations (CIDTs). As CIAT processes are consistent with the requirements of "atom-economy" and green chemistry, and as the synthesis of chiral compounds becomes more and more important, there has been an explosion of research activities utilizing CIAT techniques to prepare chiral compounds in the last few decades.[5,4c–e] However, new, efficient and highly stereoselective CIAT processes remain highly desirable.

The tetrahydro-β-carboline scaffold exists widely in natural products and medicinal compounds, so the asymmetric syntheses of chiral tetrahydro-β-carbolines have attracted much attention.[6] 1,3-Disubstituted-tetrahydro-β-carbolines (1,3-disubstituted-THβCs) are an important kind of compound due to their various biological activities[7] including antitumor,[7a] antioxidant,[7b] antiparasitic,[7c] anti-inflammatory,[7d] antithrombotic,[7e] fungicidal,[7f] antiviral[7g] properties and their use in the treatment of type 2 diabetes mellitus.[7h] Several methods have been reported for the preparation of optically pure 1,3-disubstituted-THβCs.[8] However, most of them have drawbacks such as poor stereoselectivity,[8a] the introduction and removal of sterically hindered groups,[8b–e] low yield[8f,8g] and using harmful solvents.[8h] Herein, we describe the first highly stereoselective method for the synthesis of *cis* or *trans* 1,3-disubstituted-THβCs *via* a CIAT process in water.

Results and discussion

Our investigation commenced with the preparation of mixtures of *cis* and *trans* 1,3-disubstituted-THβCs *via* the classical Pictet–Spengler reaction. We obtained the mixtures of *cis* and *trans* 1,3-disubstituted-THβCs hydrochlorides in 85–98% yields *via* the

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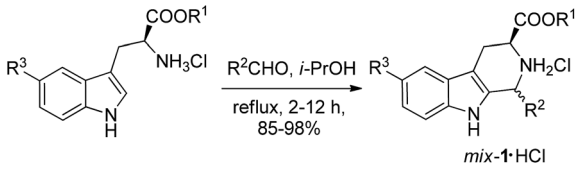


Scheme 1 Synthesis of *mix*-**1**·HCl from L-tryptophan ester hydrochloride.

condensation of L-tryptophan ester hydrochlorides with different aldehydes under reflux for 2–12 hours in isopropanol. The diastereomeric ratio of the *cis* and *trans*-isomers ranged from 11 : 89 to 92 : 8 (see Scheme 1). We then tried to convert the mixtures of 1,3-disubstituted-THβCs to a single isomer *via* a CIAT process in water. We began our study with *mix*-**1a**·HCl [mixtures of (1*S*,3*S*) and (1*R*,3*S*)-diastereomers] as the model substrate. The starting diastereomeric ratio of the *cis* and *trans*-isomers was 46 : 54 after the Pictet–Spengler reaction. We initially put *mix*-**1a**·HCl in pure water and heated it to 95 °C. We found that part of the solid dissolved with a quick conversion of the *cis*-isomer to the *trans*-isomer at the beginning. However, the rate of conversion slowed down rapidly and equilibrium between the *cis* and *trans*-isomers was reached after two hours. The diastereomeric ratio of the *cis* and *trans*-isomers changed to 16 : 84.

We thought that the high solubility of the materials probably resulted in the poor stereoselectivity of the CIAT process. Inspired by the salting-out effect and ionic equilibrium theory, we used 5% sodium chloride aqueous solution as the solvent. To our surprise, the diastereomeric ratio of the *cis* and *trans*-isomers changed to 6 : 94 when equilibrium was reached after 6 hours. Experiments were then performed to investigate the influence of the concentration of sodium chloride on the

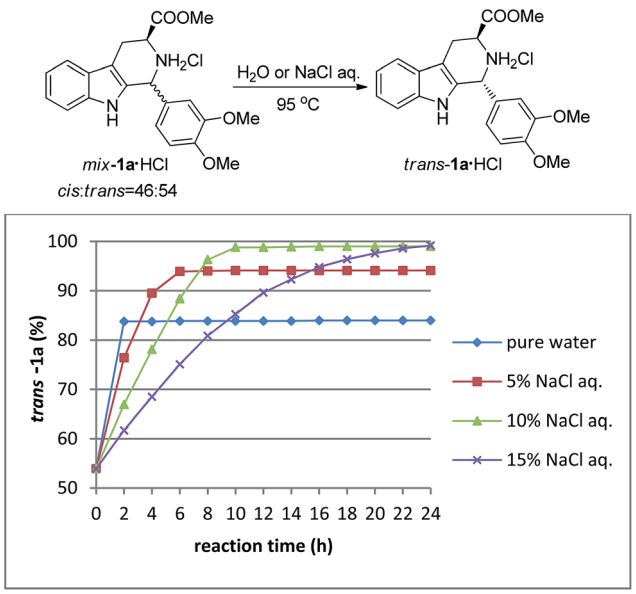
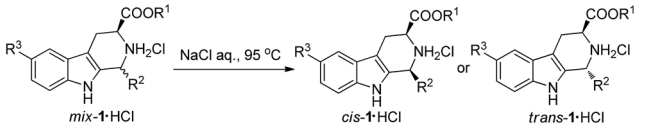
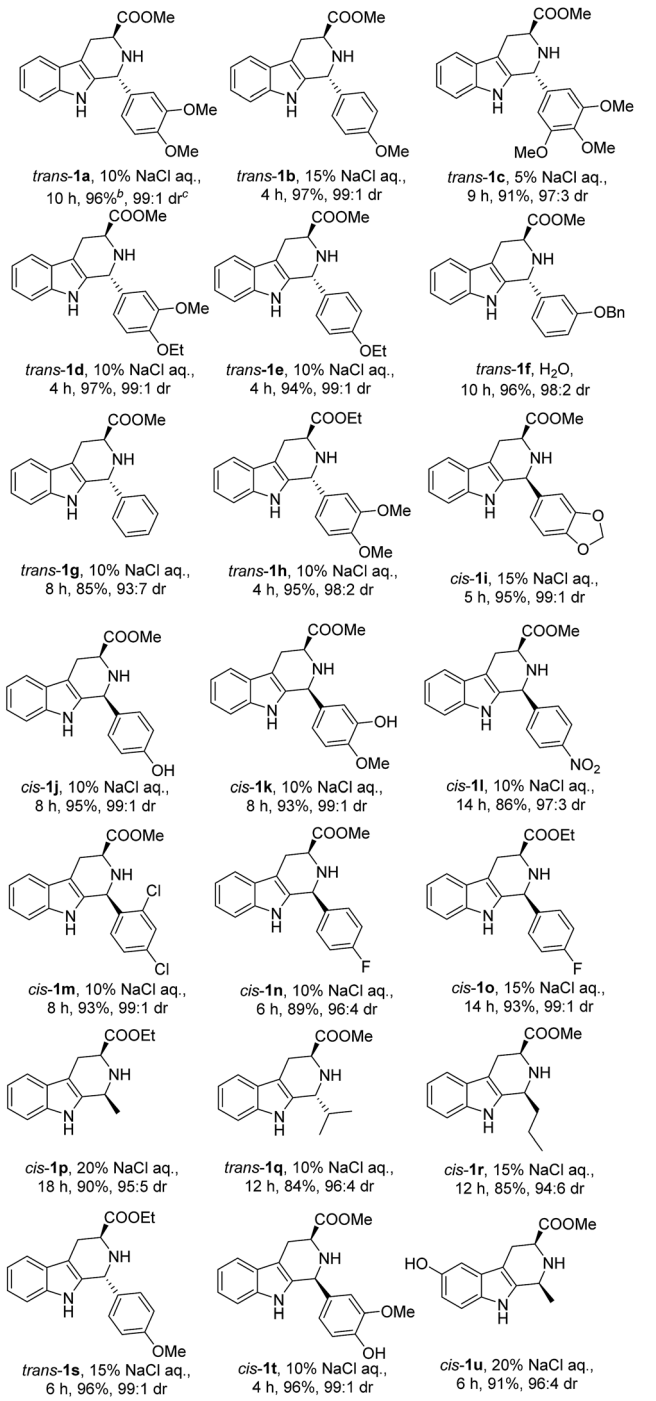


Fig. 1 Influence of the concentration of NaCl on the diastereoselectivity and reaction rate of CIAT processes for *mix*-**1a**·HCl.

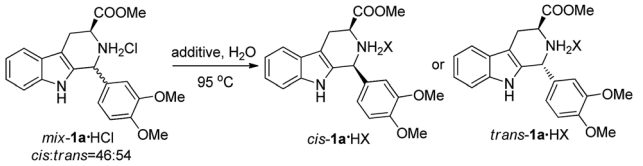
Table 1 Highly stereoselective synthesis of *cis* or *trans* 1,3-disubstituted-THβCs *via* a CIAT process in water[a]





[a] Reaction conditions: *mix*-**1a–u**·HCl (1 g), NaCl aqueous solution (8 mL), 95 °C. [b] Isolated yields. [c] *Cis*/*trans* ratios were determined by HPLC, the relative configurations of products were assigned by ^{1}H–^{1}H NOESY.[8h]

Table 2 The effect of additives on the CIAT stereoselectivity of *mix*-**1a**·HCl



Entry	Additive[a]	Time (h)	Products[b] (*cis* : *trans*)	Yield[c] (%)
1	NaCl	10	1 : 99	96
2	KCl	12	3 : 97	92
3	LiCl	10	1 : 99	96
4	$CaCl_2$	12	2 : 98	94
5	$MgCl_2$	11	4 : 96	92
6	$BaCl_2$	10	2 : 98	93
7	$ZnCl_2$	14	10 : 90	81
8	$AlCl_3$	10	3 : 97	93
9	NaBr	6	98 : 2	94
10	KBr	6	98 : 2	95
11	Na_2SO_4	6	5 : 95	83
12	K_2SO_4	10	9 : 91	81
13	Mg_2SO_4	8	3 : 97	80
14	$NaNO_3$	4	99 : 1	96
15	NaI	5	96 : 4	91
16	KI	4	97 : 3	94
17	$NaBF_4$	3	97 : 3	93
18	Na_2SO_3	12	13 : 87	73
19	CH_3CO_2Na	10	6 : 94	84
20	HCO_2Na	10	5 : 95	86

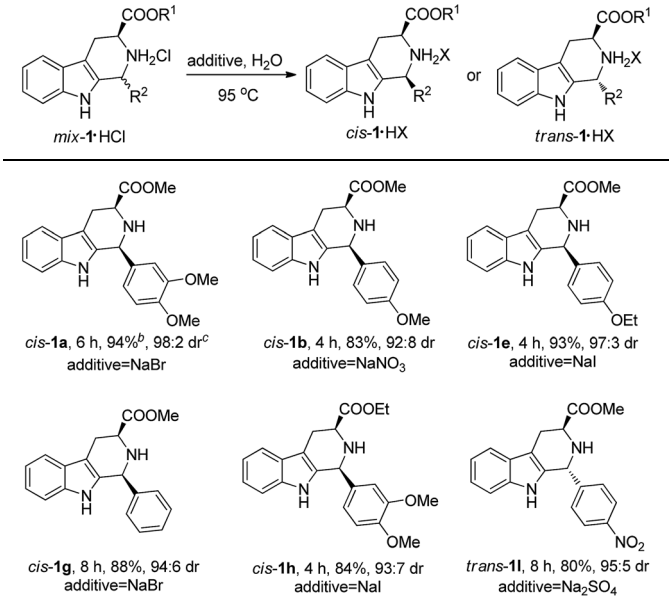
[a] Reaction conditions: 10% (w/w) aqueous solutions were used for various salts. [b] *Cis*/*trans* ratios were determined by HPLC. [c] Isolated yields.

diastereomeric ratio and reaction rate (see Fig. 1). As can be seen from Fig. 1, when the concentration of sodium chloride was increased from 0% to 15%, the purity of the *trans*-isomer was improved from 84% to >99% after the equilibrium was finally reached. On the other hand, increasing the concentration of sodium chloride significantly decreased reaction rate. When a 10% sodium chloride aqueous solution was used as the solvent, a great diastereoselectivity (99 : 1) was obtained after 10 hours.

Next, we attempted to extend the scope of the above CIAT process (see Table 1). We found that the above CIAT process could be applied to almost all substrates; the CIAT of some substrates produced *trans*-isomers with high diastereoselectivities, while the CIAT of other substrates produced *cis*-isomers with high diastereoselectivities. We also found that the optimized concentrations of sodium chloride were different for various substrates, ranging from 0% to 20%.

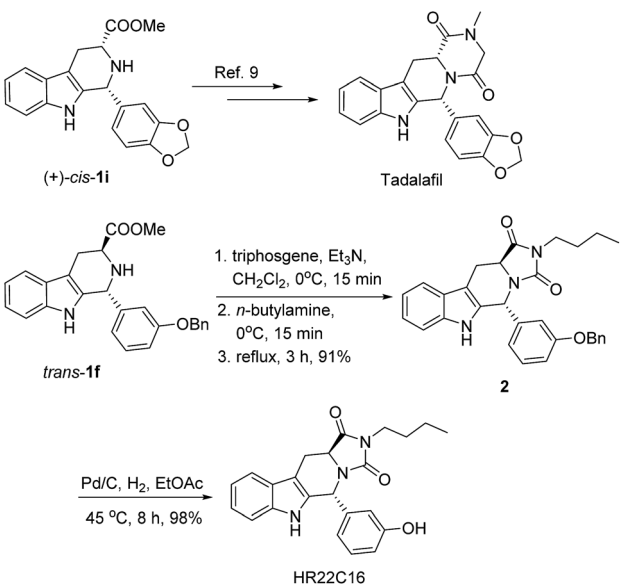
We have also investigated the influence of various salts on the above CIAT process using *mix*-**1a**·HCl; a total of twenty salts were examined and the results are summarized in Table 2. To our surprise, the addition of some salts produced *trans*-**1a**·HX as the major product (entries 1–8, 11–13 and 18–20), while the addition of other salts produced *cis*-**1a**·HX as the major product (entries 9, 10 and 14–17). We found that the different anions of

Table 3 The effect of different additive salts on the diastereoselectivities of CIATs[a]

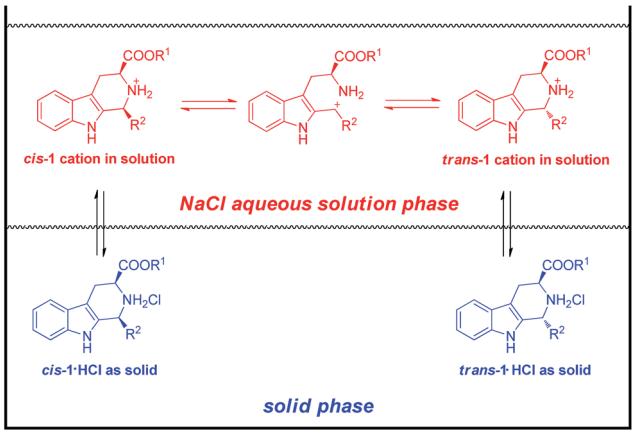


[a] Reaction conditions: *mix*-**1**·HCl (1 g), 10% additive salt aqueous solution (8 mL), 95 °C. [b] Isolated yields. [c] *Cis*/*trans* ratios were determined by HPLC, the relative configurations of the products were assigned by ^{1}H–^{1}H NOESY.[8h]

the additive salts could change the configuration of the major product (comparing entries 1, 9, 11, 14, 15 and 17–20), whereas the different cations of the additive salts could not change the configuration of the major product (comparing entries 1–8 or 11–13). This is probably due to the fact that the different anions



Scheme 2 Syntheses of Tadalafil and HR22C16 from the 1,3-disubstituted-THβCs *cis*-**1i** and *trans*-**1f**, respectively.



Scheme 3 Proposed mechanism for the CIAT process of *mix*-1a·HCl in aqueous NaCl solution.

(X^-) of the additive salts might exchange with the Cl^- of *mix*-**1a**·HCl to crystallize out less soluble *trans* (or *cis*)-**1a**·HX.

We then investigated the CIAT processes of other substrates by adding different salts. We examined the CIAT processes of all twenty-one substrates shown in Table 1. It was found that the configuration of the major products from the CIAT processes of six substrates could be changed by adding different salts (see Table 3). Therefore we can obtain both the *cis* and *trans*-isomers of these six substrates by incorporating different additive salts in water.

It is worth noting that *cis* or *trans* 1,3-disubstituted-THβCs, which can be readily obtained by the above CIAT process, are very useful for drug synthesis. For example, (+)-*cis*-**1i** was synthesized from D-tryptophan methyl ester hydrochloride *via* the same CIAT process, which is an important intermediate for the synthesis of Tadalafil (see Scheme 2),[9] a selective PDE5 inhibitor. Additionally, *trans*-**1f** was successfully used herein for the synthesis of HR22C16 (see Scheme 2), which is a potent mitotic kinesin Eg5 inhibitor.[10]

Some people have proposed that the least soluble diastereomer will crystallize preferentially during a CIAT process.[5b,11] Based on this assumption, a plausible mechanism for the above CIAT process of *mix*-**1**·HCl in aqueous NaCl solution is proposed in Scheme 3. Both the solid *cis* and *trans*-**1**·HCl would partially dissolve into the aqueous solution phase by dissociating into *cis* and *trans*-**1** cations and chloride anions, the *cis* and *trans*-**1** cations would interchange *via* cleavage and formation of the C–N bond.[12] Crystallization of the less soluble isomer would shift the equilibrium to afford the less soluble isomer as the major product of the CIAT process.

Conclusions

In conclusion, we have developed the first protocol for a highly diastereoselective CIAT process of 1,3-disubstituted-THβCs in water. This protocol provides efficient access to a single isomer of either *cis* or *trans*-1,3-disubstituted-THβCs, which are very useful for drug synthesis. It was also observed that addition of different salts in water exhibits an obvious influence on the diastereoselectivities and rate of the CIAT process. The above CIAT protocol has the following advantages: (1) it was successfully carried out in pure water, so it is environmentally benign; (2) all the products have good to excellent diastereoselectivities; (3) the scope of the CIAT process is very wide, it was applicable to all the tested substrates; (4) the experiments were carried out on a gram scale; (5) it is practical because the starting materials are easy to obtain and operation is very simple.

Conflicts of interest

There are no conflicts to declare.

Acknowledgements

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