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SYNTHESIS OF *ROCHE* LACTONE VIA THE ENANTIOSELECTIVE ALCOHOLYSIS OF *meso*-CYCLIC ANHYDRIDE STRATEGY: A PRACTICAL APPROACH EMPLOYING AN EFFICIENT AND REUSABLE ORGANIC MICROGEL AUXILIARIES

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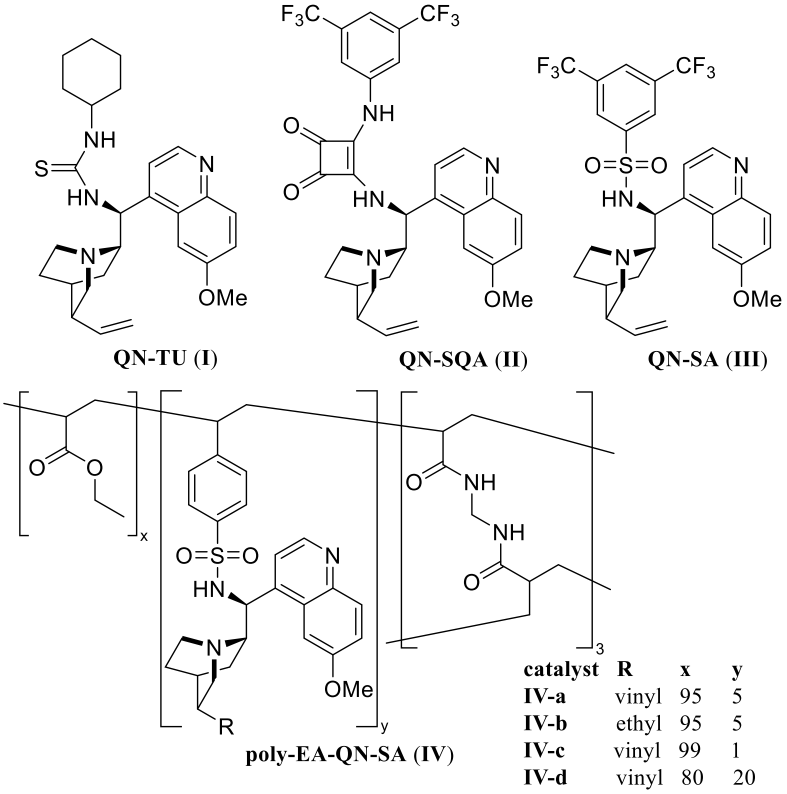
Abstract – *Roche* lactone, a key chiral intermediate of the natural water-soluble B series vitamin (+)-biotin, was synthesized from *meso*-cyclic anhydride in overall yield 78% through three steps, including heterogeneous asymmetric desymmetrization, chemoselective reduction and lactonization.

INTRODUCTION

Development of efficient synthetic methods for *Roche* lactone, chemically known as (3a*S*, 6a*R*)-1, 3-dibenzyl-tetrahydro-4*H*-furo[3,4-*d*]imidazole-2,4(1*H*)-dione (**1**), is an important research area in synthetic chemistry[1-5] because oxygen heterocyclic compounds are not only valuable key synthetic intermediate for (+)-biotin but also important structural units existing in various useful pharmaceutical molecules. Pioneering classical enantioselective alcoholysis of *meso*-cyclic anhydride **2** have been demonstrated as a useful synthetic strategy for the synthesis of *Roche* lactone **1** as the differentiation of two enantiotopic groups facilitates the formation of two contiguous stereocentres at C-3a and C-6a of the biotin skeleton in a single transformation.[5] Previously, we developed a series of bifunctional thiourea,[6] squaramide[7] and sulfonamide[8] organocatalysts (**I-III**, **Figure 1**) derived from quinine, a naturally-occurring cinchona alkaloid, as the chiral scaffold for the homogeneous asymmetric alcoholysis of *meso*-cyclic anhydride **2** with excellent enantioselectivities (up to 97% ee) and yields (up to 99%). Although these homogeneous organocatalysts demonstrated excellent enantioselectivities and yields at room temperature, the syntheses might suffer from tedious work-up procedure to purify the product at the end of the reaction.[6-8] Furthermore, the possible contamination of the product by these organocatalysts may also restrict their further use in industrial scale applications.[9,10] Obviously, immobilization of these soluble organocatalysts could simplify the work-up procedure required for purification of the product, and allow for easy recovery of the organocatalyst and its potential recycling.[11] The group of Pich and Rueping have recently reported the use of various new temperature-responsive organic polymer microgel as heterogeneous organocatalysts (**IV**, **Figure 1**) for the highly enantioselective methanolysis of *cis*-tetrahydrophthalic anhydride.[12] By a simple change in the temperature of reaction solution, these adaptable microgelzymes catalysts can be reversible switched into its soluble or precipitated form, which combined the advantages of homogeneous and heterogeneous catalysis. Although this new adaptable microgel-based colloidal catalyst system have been shown to be successful in the asymmetric desymmetrization with *cis*-tetrahydrophthalic anhydride as the only substrate, their use in desymmetrization with other anhydrides has not yet been explored. Following from this work and coupled with our own experience in asymmetric synthesis of *Roche* lactone **1** via the desymmetrization strategy with *meso*-cyclic anhydride **2** as substrate,[8,13,14] we speculated that organic polymer microgel catalyst could be excellent candidate to be used as heterogeneous catalyst for synthesizing *Roche* lactone by utilizing the enantioselective alcoholysis of *meso*-cyclic anhydride **2** strategy. Herein we described their application in the asymmetric desymmetrization of *meso*-cyclic anhydride **2** to prepare (4*S*, 5*R*)-hemiester **3**, the direct precursor to *Roche* lactone **1**, to complete an efficient asymmetric synthesis of **1**.



QN-TU (I) **QN-SQA (II)** **QN-SA (III)**

poly-EA-QN-SA (IV)

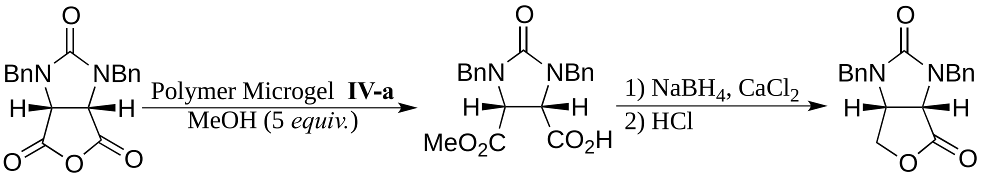
catalyst	**R**	**x**	**y**
IV-a	vinyl	95	5
IV-b	ethyl	95	5
IV-c	vinyl	99	1
IV-d	vinyl	80	20

Figure 1. Structure of chiral bifunctional cinchona alkaloid-derived organocatalysts

RESULTS AND DISCUSSION

Initial screening of various polymer microgel catalysts developed by Pich and Rueping group for the enantioselective methanolysis of *cis*-tetrahydrophthalic anhydride[12] led us to select poly-EA-QN-SA (**IV-a**) as a promising catalyst for desymmetrization of *meso*-cyclic anhydride **2** into (4*S*, 5*R*)-hemiester **3a**. To validate this assumption, we examined the asymmetric organocatalytic desymmetrization of **2** upon treatment with 5 equiv. methanol in the presence of 0.9 equiv. catalyst **IV-a** in Et_2O at room temperature and the desired (4*S*, 5*R*)-methyl hemiester **3a** was obtained with poor enantioselectivity (23% ee, **Table 1**, entry 1). To our delight, further increasing the catalyst loading from 0.9 equiv. to 1.2 equiv. led to an increase in the enantioselectivity of **3a** up to 62% (**Table 1**, entries 1-4), and no significant improvement in the enantioselectivity was noticed on increasing the catalyst loading from 1.2 equiv. to 1.5 equiv. (**Table 1**, entries 4-5). As described in **Scheme 1**, the use of stoichiometric polymer microgel catalyst **IV-a** can be explained by the formation of a specific product-base complex salt **C**, resulting from a plausible catalyst-transition state analogue complex **B**. The proposed catalytic cycle reaction mechanism suggests that the use of superstoichiometric loading amount of catalyst than usual substoichiometric catalyst loading is necessary to increase the reaction rate and the enantiocontrol of the desired product methyl hemiester **3a**.

Table 1. Optimization of the reaction conditions in the enantioselective methanolysis of *meso*-cyclic anhydride **2** by chiral heterogeneous polymer microgel **IV-a**



2 **3a** ***Roche* lactone, 1**

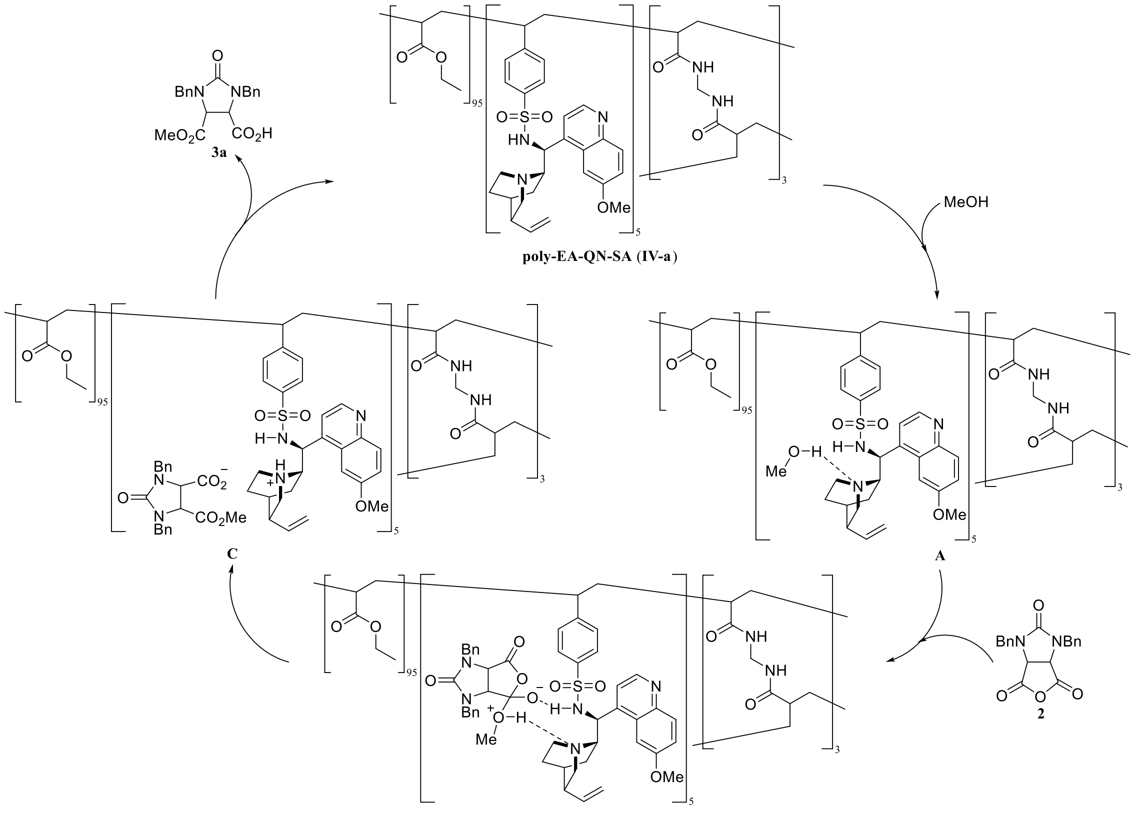
Entry	Catalyst loading[a]	Solvent	T (°C)	Time (h)	Yield[b] (%)	ee[c] (%)
1	90 mol%	Et_2O	25	3.5	97	23
2	100 mol%	Et_2O	25	3	96	30
3	110 mol%	Et_2O	25	2.5	97	49
4	120 mol%	Et_2O	25	2	97	62
5	150 mol%	Et_2O	25	2	98	62
6	120 mol%	Et_2O	10	6	97	38
7	120 mol%	Et_2O	40	1	97	43
8	120 mol%	acetone	25	2	98	50
9	120 mol%	CH_2Cl_2	25	2	97	34

10	120 mol%	MTBE	25	2	98	41
11	120 mol%	toluene	25	2	39	39

[a] Reaction conditions: *meso*-cyclic anhydride **2** (0.15 mmol), MeOH (0.75 mmol) and chiral heterogeneous polymer microgel **IV-a** in different solvent (8 mL) stirred at specified reaction temperature.

[b] Isolated yield of **3a**.

[c] Determined by chiral HPLC analysis of the corresponding chiral *Roche* lactone **1**, which were obtained by selective reduction of the ester group with *in situ* prepared $Ca(BH_4)_2$, followed by acid-catalyzed lactonization.

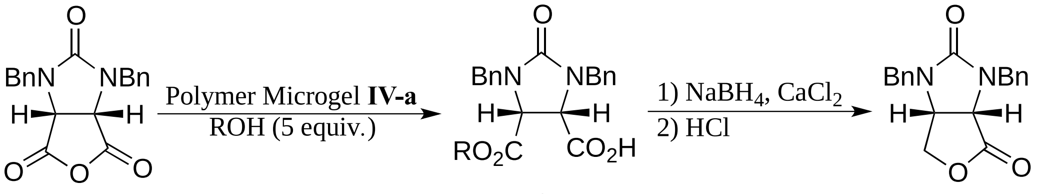


Scheme 1. The proposed mechanism for the chiral heterogeneous polymer microgel **IV-a** mediated enantioselective methanolysis of *meso*-cyclic anhydride **2** to afford (4*S*, 5*R*)-methyl hemiester **3a**

Encouraged by these experimental observations, we then screened the effect of reaction temperature and various solvents for this desymmetrization reaction. Switching the reaction temperature to 40 °C or 10 °C failed to give better results (**Table 1**, entries 4, 6-7). Among the other solvents test, low enantioselectivity of **3a** was observed in the cases of acetone, CH_2Cl_2, methyl *tert*-butyl ether (MTBE) and toluene (**Table 1**, entries 4, 8-11).

After identification of the optimized reaction conditions, the scope and limitations of this desymmetrization process were examined by varying the structure of nucleophile. As indicated in **Table 2**, treatment of **2** with different alcohol, using as the test nucleophile, generated the corresponding (4*S*, 5*R*)-hemiester **3b**-**3e** in excellent isolated yield. These experimental results also illustrated that the steric hindrance of nucleophile might influence the desymmetrization alcoholysis reaction. The *trans*-cinnamyl alcohol turned out to be the best nucleophile as it gave the resultant chiral cinnamyl hemiester **3d** with 72% ee. The enantiopure **3d** could be easily obtained after a single recrystallization from EtOAc and the structure and absolute configuration of **3d** was confirmed to be 4*S* and 5*R* by its subsequent conversion into the known *Roche* lactone **1** which was obtained in 98% isolated yield by chemoselective reduction of cinnamyl ester group of **3d** with *in situ* prepared $Ca(BH_4)_2$ followed by acid-catalyzed lactonization using our previous reported procedure.[8] Thus, we concluded that poly-EA-QN-SA (**IV-a**) as the chiral heterogeneous polymer microgel auxiliaries, *meso*-cyclic anhydride **2** as the substrate, *trans*-cinnamyl alcohol as the nucleophile and Et_2O as the solvent is an optimized combination for this asymmetric desymmetrization process, which afforded an efficient process for the preparation of *Roche* lactone **1** with high levels of enantioselectivity.

Table 2. Asymmetric alcoholysis of *meso*-cyclic anhydride **2** with different alcohols



2 **3** ***Roche* lactone, 1**

Entry	ROH[a]	Time (h)	Product	Yield[b] (%)	ee[c] (%)
1	benzyl alcohol	2	**3b**	97	45
2	cyclohexanol	2	**3c**	95	42
3	*trans*-cinnamyl alcohol	2	**3d**	97	72
4	2-propanol	2	**3e**	96	40

[a] Reaction conditions: *meso*-cyclic anhydride **2** (0.15 mmol), alcohol (0.75 mmol) and the chiral heterogeneous polymer microgel auxiliaries **IV-a** (0.18 mmol) in Et_2O (8 mL) stirred at room temperature (25 °C).

[b] Isolated yield.

[c] Determined by chiral HPLC analysis of the corresponding chiral *Roche* lactone **1**, which were obtained by selective reduction of the ester group with *in situ* prepared $Ca(BH_4)_2$, followed by acid-catalyzed lactonization.

In addition, it is noteworthy that the temperature sensitivity property of organic polymer microgel auxiliaries **IV-a** can be used to obtain the pure form hemiester product **3** after simple centrifugal treatment of the cooled reaction mixture, followed by evaporation of the volatile solvent. The organic polymer microgel **IV-a** obtained by centrifugation can be refreshed after washed with Et_2O and no significant loss in the catalysis activity and enantioselectivity was observed when it was reused up to ten times (**Table 3**).

Table 3. Recycling of the polymer microgel auxiliaries **IV-a** in the asymmetric desymmetrization of *meso*-cyclic anhydride **2** with *trans*-cinnamyl alcohol[a]

Run	Time (h)	Yield[b] (%)	ee[c] (%)
1	2	97	72
2	2	97	72
3	2	97	72
4	2	96	71
5	2	96	71
6	2	98	70
7	2	98	70
8	2	98	70
9	2	97	71
10	2	98	70

[a] Reaction conditions: *meso*-cyclic anhydride **2** (0.15 mmol), *trans*-cinnamyl alcohol (0.75 mmol) and the organic polymer microgel **IV-a** (0.18 mmol) in Et_2O (8 mL) stirred at room temperature (25 °C).

[b] Isolated yield of **3d**.

[c] Determined by chiral HPLC analysis of the corresponding chiral *Roche* lactone **1**, which were obtained by selective reduction of the ester group with *in situ* prepared $Ca(BH_4)_2$, followed by acid-catalyzed lactonization.

In summary, an improved and convenient method for the asymmetric synthesis of *Roche* lactone, starting from the commercially available *cis*-1,3-dibenzyl-tetrahydro-2*H*-furo[3,4-*d*]imidazole-2,4,6-trione (**2**), has been established in our laboratory by ultilizing stereoselective heterogeneous asymmetric desymmetrization strategy. This process relies on a new type of temperature-responsive polymer microgel **IV-a** as chiral heterogeneous auxiliaries for the desymmetrization of *meso*-cyclic anhydride **2**. With respect to economic and environmental consideration, highly effective catalytic activity and its recyclability and reusability made this protocol more attractive from green chemistry perspective. We are currently investigating the development of this temperature-responsive organic polymer microgel heterogeneous organocatalyst with other chiral scaffold source and more details of this extended studies will be reported in due course.

EXPERIMENTAL

Unless otherwise noted, all reagents and solvents were commercially available with analytical grade from Adamas-Beta and used as received. Further purification and drying by standard method were employed and distilled prior to use when necessary. All evaporations of organic solvents were carried out with a rotary evaporator in conjunction with a water aspirator. The *cis*-1,3-dibenzyl-tetrahydro-2*H*-furo[3,4-*d*]imidazole-2,4,6-trione (**2**) and heterogeneous organic polymer microgel auxiliaries **poly-EA-QN-SA** (**IV-a**) were freshly prepared according to the published methods.[8,12] Melting points were determined with a *WRS*-1B digital melting point apparatus and are uncorrected. NMR spectra were recorded on a Bruker Avance 400 spectrometer using TMS as an internal standard. Chemical shifts (δ) are expressed in ppm. Optical rotations were measured with a *SGW*-2 digital automatic polarimeter. The enantiomeric excesses of product **3** was determined by chiral HPLC analysis of the corresponding chiral *Roche* lactone **1**, which were obtained by selective reduction of the ester group with *in situ* prepared $Ca(BH_4)_2$, followed by acid-catalyzed lactonization. HPLC analysis of the enantiomeric excesses of monoester **3** was performed using chiralcel OD column (*n*-hexane / *i*-PrOH = 70 / 30). Mass spectra were recorded on a Waters Quattro-Micromass instrument by using electrospray ionization (ESI) techniques.

The general procedure for the asymmetric alcoholysis of 2 with various alcohols: A mixture of **2** (0.15 mmol), **IV-a** (0.18 mmol) and anhydrous Et_2O (8 mL) was stirred at room temperature (25 °C) for 10 min, and then alcohol (0.75 mmol) was added in dropwise. The resulting suspension was kept stirring at room temperature for 2 h and then cooled to 0 °C. The precipitated microgel auxiliaries **IV-a** was separated from the supernatant solvent by centrifugation, and the recovered catalyst was then washed by repeating dissolving with anhydrous Et_2O / cooling to 0 °C / centrifugation procedure in order to remove the possible residue of the product (progress of the washing process was monitored by TLC control, EtOH / acetone = 9 : 1, I_2). The combined supernatant solution was concentrated under reduced pressure to give the desired chiral hemiester **3**, which was used directly in the next step.

(4*S*,5*R*)-1,3-Dibenzyl-5-(methoxycarbonyl)-2-oxoimidazolidine-4-carboxylic acid (3a): White solid; yield: 53.4 mg (97%); ee = 62%; mp 144.9-147.5 °C; ^{1}H NMR ($CDCl_3$) δ: 3.63 (s, 3H), 4.13-4.04 (m, 4H), 4.98 (d, 1H, *J* = 14.8 Hz), 5.09 (d, 1H, *J* = 14.8 Hz), 6.85 (br s, 1H), 7.36-7.19 (m, 10H); ^{13}C NMR($CDCl_3$) δ: 46.80, 46.89, 52.6, 56.7, 57.3, 127.93, 127.98, 128.56, 128.64, 128.79, 128.85, 135.52, 135.57, 159.5, 168.5, 171.7; IR (KBr): ν = 3258, 2943, 1756, 1713, 1449, 1411, 1252, 968, 799, 598, 458 cm^{-1}; MS (ESI): *m/z* = 391.1 $(M+Na)^+$.

(4*S*,5*R*)-1,3-Dibenzyl-5-(benzyloxycarbonyl)-2-oxoimidazolidine-4-carboxylic acid (3b): White solid; yield: 64.7 mg (97%); ee = 45%; mp 56.9-60.5 °C; ^{1}H NMR ($CDCl_3$) δ: 3.70 (br s, 1H), 4.11-4.00 (m, 4H), 5.12-4.97 (m, 4H), 7.34-7.10 (m, 15H); ^{13}C NMR ($CDCl_3$) δ: 46.79, 46.80, 56.5, 57.2, 67.7, 127.89, 127.94, 128.58, 128.61, 128.63, 128.70, 128.76, 128.83, 135.53, 135.65 159.5, 167.8, 170.8; IR (KBr): ν = 3031, 2945, 1752, 1664, 1454, 1236, 1201, 967, 741, 700 cm^{-1}; MS (ESI): *m/z* = 467.2 $(M+Na)^+$.

(4*S*,5*R*)-1,3-Dibenzyl-5-(cyclohexyloxycarbonyl)-2-oxoimidazolidine-4-carboxylic acid (3c): Yellow oil; yield: 62.3 mg (95%); ee = 42%; ^{1}H NMR ($CDCl_3$) δ: 1.13-1.32 (m, 5H), 1.52-1.55 (m, 1H), 1.70-1.72 (m, 2H), 1.87-1.90 (m, 2H), 3.61-3.66 (m, 1H), 3.99-4.08 (m, 4H), 5.04 (d, 2H, *J* = 15.2 Hz), 7.20-7.32 (m, 10H); ^{13}C NMR ($CDCl_3$) δ: 8.8, 21.7, 25.9, 39.4, 46.3, 52.1, 58.2, 62.5, 68.7, 127.6, 137.2, 160.2, 168.8; IR (KBr): ν = 3462, 2933, 2518, 1649, 1453, 1228, 765, 741, 700 cm^{-1}; MS (ESI): *m/z* = 459 (M+Na)$^+$.

(4*S*,5*R*)-1,3-Dibenzyl-2-oxo-5-[(3-phenylallyloxy)carbonyl]imidazolidine-4-carboxylic acid (3d): White solid; yield: 68.7 mg (97%); ee = 72%; after recrystallization from EtOAc, **3d** was obtained in 80% yield with 98% ee; mp 121.9-122.5 °C; $[\alpha]_D^{22}$: +7.51 (*c* 1.0, CH_3OH) [Lit.[15] mp 122.0-122.8 °C; $[\alpha]_D^{21.9}$: +7.58 (*c* 1.0, MeOH)]; ^{1}H NMR ($CDCl_3$) δ: 4.02-4.13 (m, 4H), 4.62-4.72 (m, 2H), 5.01 (d, *J* = 14.8 Hz, 1H), 5.09 (d, *J* = 14.8 Hz, 1H), 6.13-6.21 (dt, *J* = 16.0, 6.4 Hz, 1H), 6.59 (d, *J* = 16.0 Hz, 1H), 7.18-7.39 (m, 15H), 10.53 (br s, 1H); IR (KBr): ν = 3449, 3028, 2930, 1751, 1711, 1664, 1450, 1357, 1238, 1196, 748, 701 cm^{-1}; MS (ESI): *m/z* = 470.3 (M+1)$^+$.

(4*S*, 5*R*)-1,3-Dibenzyl-5-(isopropoxycarbonyl)-2-oxoimidazolidine-4-carboxylic acid (3e): Yellow oil; yield: 57.2 g (96%); ee = 40%; ^{1}H NMR ($CDCl_3$) δ: 1.04 (d, 6H, *J* = 6.0 Hz), 3.50 (m, 1H), 3.69-4.07 (m, 4H), 4.77 (d, 1H, *J* = 14.4 Hz), 4.79 (d, 1H, *J* = 14.4 Hz), 7.18-7.32 (m, 10H); ^{13}C NMR ($CDCl_3$) δ: 8.8, 21.7, 25.9, 39.4, 46.3, 52.1, 58.2, 62.5, 68.7, 127.6, 137.2, 160.2, 168.8; IR (KBr): ν = 3438, 3028, 2979, 2494, 1956, 1697, 1446, 1235, 1108, 957, 836, 753, 702, 458 cm^{-1}; MS (ESI): *m/z* = 419 (M+Na)$^+$.

(3a*S*,6a*R*)-1,3-Dibenzyl-tetrahydro-4*H*-furo[3,4-*d*]imidazole-2,4(1*H*)-dione (*Roche* lactone, 1): Chiral cinnamyl hemiester **3d** (1.41 g, 3 mmol) and granulated anhydrous $CaCl_2$ (0.33 g, 3 mmol) was suspended in anhydrous EtOH (16 mL) at 0 °C. $NaBH_4$ (0.34 g, 9 mmol) was added to the reaction mass in three portions at 0 °C. The resultant mixture was warmed up to room temperature and keep stirring for another 18 h at 25 °C. Subsequently, the resulting solution was concentrated in vacuo to dryness, and the residue was dissolved in EtOAc (10 mL). The solution was extracted with 10% aqueous Na_2CO_3 (3×10 mL) and the combined aqueous phases was neutralized with concentrated aqueous HCl, then treated with 5% aqueous HCl (10 mL) at 55-60 °C. After 0.5 h at this temperatue, the solution was extracted with CH_2Cl_2 (3×10 mL). The combined organic phases were dried over $MgSO_4$ and concentrated in vacuo to afford **1** as a white solid; yield: 0.95 g (98%); mp 119.0-119.6 °C; $[\alpha]_D^{25}$: +61.1 (*c* 2.0, $CHCl_3$) [Lit.[16] mp 119.8-120.5 °C; $[\alpha]_D^{25}$: +61.0 (*c* 2.0, $CHCl_3$)]; ^{1}H NMR ($CDCl_3$) δ: 3.92 (d, 1H, *J* = 8.0Hz), 4.09-4.16 (m, 3H), 4.37 (dd, 2H, *J* = 10.4 Hz, 15.2 Hz), 4.63 (d, 1H, *J* = 15.2 Hz), 5.05 (d, 1H, *J* = 15.2 Hz), 7.24-7.36 (m, 10H); ^{13}C NMR ($CDCl_3$) δ: 45.2, 46.9, 52.4, 54.3, 70.1, 127.8, 128.0, 128.2, 128.7, 128.8, 129.0, 135.9, 136.0, 158.1, 172.7; IR (KBr): ν = 3031, 2919, 1775, 1706, 1415, 1365, 1237, 1209, 1146, 970, 754, 700, 639, 527 cm^{-1}; MS (ESI): *m/z* = 345.2 (M+Na)$^+$.

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