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Kinetic resolution of sterically hindered secondary alcohols catalyzed by aminophosphinite organocatalyst

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ABSTRACT

Kinetic resolution of secondary alcohols by benzoylation using a phosphinite derivative of (1*S*,2*R*)-1-amino-2-indanol as the catalyst was investigated. The aminophosphinite catalyst is effective for the kinetic resolution of aryl cycloalkyl carbinols with a small number of examples for organocatalytic kinetic resolution to achieve resolution with s = up to 44. Although the benzoylation of phenylalkanols proceeded with a low selectivity, 1-arylalkanols bearing at least one substituent at the *ortho* position on the benzene ring or a branched alkyl group on the carbinol carbon were resolved with acceptable selectivity.

1. Introduction

Kinetic resolution of racemic alcohols is a powerful methodology for obtaining chiral alcohols or their derivatives, versatile synthetic building blocks for organic synthesis. Especially, nonenzymatic acyl transfer organocatalysts are used to achieve kinetic resolution through the concise esterification of alcohols; therefore, the development of more reactive and stereoselective organocatalysts has been performed in the last two decades.[1] Most of them are nucleophilic chiral organocatalysts that activate acylating reagents, and many successful examples using diverse chiral catalysts, e.g., diamine,[2] *N*,*N*-dimethylaminopyridine (DMAP),[3] imidazole,[4] phosphane,[5] *N*-heterocyclic carbene derivatives,[6] amidine-based catalysts,[7] and synthetic peptides,[8] have been reported. Most of these catalysts have been mainly used in the kinetic resolution of racemic secondary alcohols such as aryl alkyl carbinols,[1,9] and allylic[10] or propargylic[11] alcohols to evaluate their catalytic potential. Especially, aryl alkyl carbinols have been used as the most common substrates from the early stage of exploration of acylative kinetic resolution using organocatalysts. In contrast, aryl cycloalkyl carbinols are still rare substrates for the relevant kinetic resolution[6c,12]; to the best of our knowledge, successful acylative kinetic resolution of these substrates by nonenzymatic organocatalysts has not been reported.

We have been studying the nucleophilic chiral acylative organocatalysts and demonstrated that the phosphinite derivatives of cinchona alkaloids **1a,b** or an aminoindanol **2** are effective catalysts for the asymmetric desymmetrization[13] or kinetic resolution[14] of diols (Fig. 1). Especially, the phosphinite derivatives of an aminoindanol are more practical small-molecule catalysts than cinchona alkaloid derivatives in terms of catalytic activity, air stability, and commercial availability of both the enantiomers of (1*S**,2*R**)-aminoindanol. To clarify the applicability of the catalyst to the kinetic resolution of racemic monools, diverse secondary alcohols were subjected to kinetic resolution catalyzed by aminoindanol phosphinite derivative **2**. The catalyst was found to be more effective for the kinetic resolution of aryl cycloalkyl carbinols than aryl acyclic alkyl carbinols such as 1-phenylethanol. Herein, we report the unique substrate specificity of the catalyst, affected by the substitution mode around the carbinol carbon atom of the substrates.

2. Results and discussion

To use aminoindanol-derived phosphinite **2** in the kinetic resolution of monools, asymmetric benzoylation reaction was investigated using benzylic alcohol derivatives as the substrates. Among

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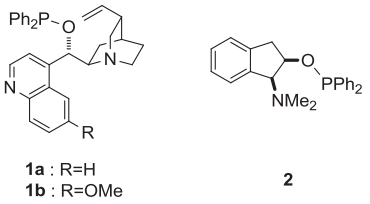


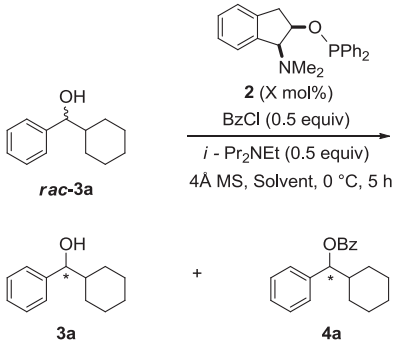
Fig. 1. Aminophosphinite catalysts for asymmetric acylation of diols.

these substrates, cyclohexyl(phenyl)methanol ***rac*-3a** was a promising substrate for the kinetic resolution catalyzed by **2**. The reaction of ***rac*-3a** with benzoyl chloride in the presence of 10 mol% of **2** in $CHCl_3$ proceeded with good selectivity ($s = 25$, Table 1, entry 1). Although similar reactions using acetonitrile as the solvent afforded unreacted alcohol **3a** and benzoate **4a** with a lower enantioselectivity (entry 2), the reactions in CH_2Cl_2 or 1,2-dichloroethane proceeded with a comparable selectivity to that in $CHCl_3$ (entries 3 and 4). On the other hand, the reaction in a less polar solvent, toluene, was very sluggish, providing the benzoate in a low yield under the same reaction conditions (entry 5). Similar reaction under a reduced catalyst loading (5 mol%, entry 6) in $CHCl_3$ achieved the resolution of ***rac*-3a** with a comparable s value, but the conversion slightly decreased.

Next, other aryl cycloalkyl carbinols were subjected to the kinetic resolution catalyzed by **2** to investigate the substrate scope. The reactions were conducted using 0.6 equiv of benzoyl chloride and 10 mol% catalyst **2** in $CHCl_3$ to recover the unreacted alcohols with a high optical purity (Scheme 1). Under these conditions, the reaction of ***rac*-3a** proceeded with resolving efficiency ($s = 22$), similar to the reaction shown in Table 1, and (*S*)-alcohol was recovered with 99% ee. The carbinol bonded to a cycloheptane ring (***rac*-3b**) was also resolved with $s > 20$, indicating that the resolution is practically possible.[1a] The cyclopentyl and cyclopropyl carbinols (***rac*-3c-d**) were also examined. Although cyclopentyl carbinol was resolved with a moderate selectivity ($s = 15$), the selectivity factor for cyclopropyl carbinol apparently decreased ($s = 2.7$) and the configuration of the predominantly recovered alcohol was *R*. These results indicated that the size of cycloalkyl group affected the resolving efficiency and stereochemical outcome for the kinetic resolution catalyzed by **2**. The carbinols having heteroaromatic rings (***rac*-3e–h**) were also subjected to the similar kinetic resolution. Pyridin-2-yl carbinol ***rac*-3e** was resolved with an excellent s value ($s = 44$). In contrast, for the reaction of the isomer, pyridine-3-yl carbinol ***rac*-3f**, the racemic benzoate and unreacted alcohol were obtained. Presumably, the pyridine moiety in the substrate ***rac*-3f** seemed to act as a Lewis base activating benzoyl chloride before the non-stereoselective reaction proceeded. On the other hand, the nucleophilicity of the nitrogen atom in pyridin-2-yl carbinol ***rac*-3e** was thought to be decreased, because of steric repulsion by a bulky substituent at 2-position or intramolecular hydrogen bonding with a hydroxyl group. As a result, the reaction of ***rac*-3e** was assumed to occur without background reaction promoted by the pyridine moiety in the carbinol. The reaction of carbinols having a five-membered heteroaromatic ring (***rac*-3g–h)** was also carried out. The reaction of these substrates proceeded with a good selectivity and the recovered alcohols having *S* configuration similar to that of ***rac*-3a**.

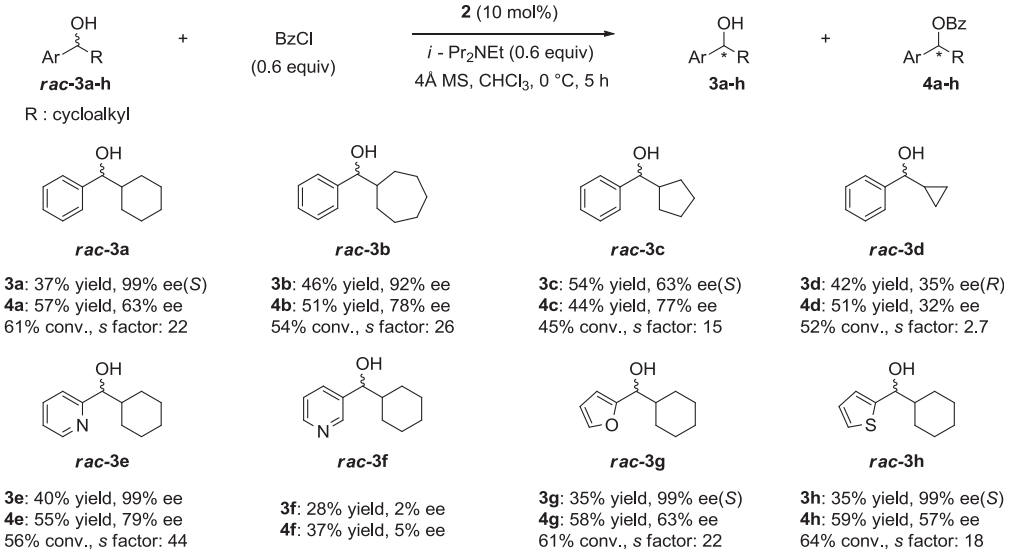
The kinetic resolution of aryl acyclic alkyl carbinols ***rac*-5a–l** containing diverse substituents around the carbinol carbons was also performed to investigate the substrate scope of the acylative kinetic resolution catalyzed by **2** (Scheme 2). First, 1-phenylethanol derivatives ***rac*-5a–e** with an electronically different substituent at the *para* position on the benzene ring were used for the kinetic resolution. When substrates with an electron-withdrawing group such as chloro and trifluoromethyl groups (***rac*-5d, 5e**) were used, the selectivity factors slightly increased compared to the other substrates. However, all these substrates were resolved with a lower selectivity than the cycloalkyl carbinols. In contrast to cycloalkyl carbinols, the *R* isomers of alcohols were preferentially recovered. A similar secondary alcohol with a naphthalene ring (***rac*-5f**) in place of benzene ring was not effective as well.

Table 1
Kinetic resolution of cyclohexyl(phenyl)methanol ***rac*-3a** by benzoylation using aminophosphinite catalyst **2**.



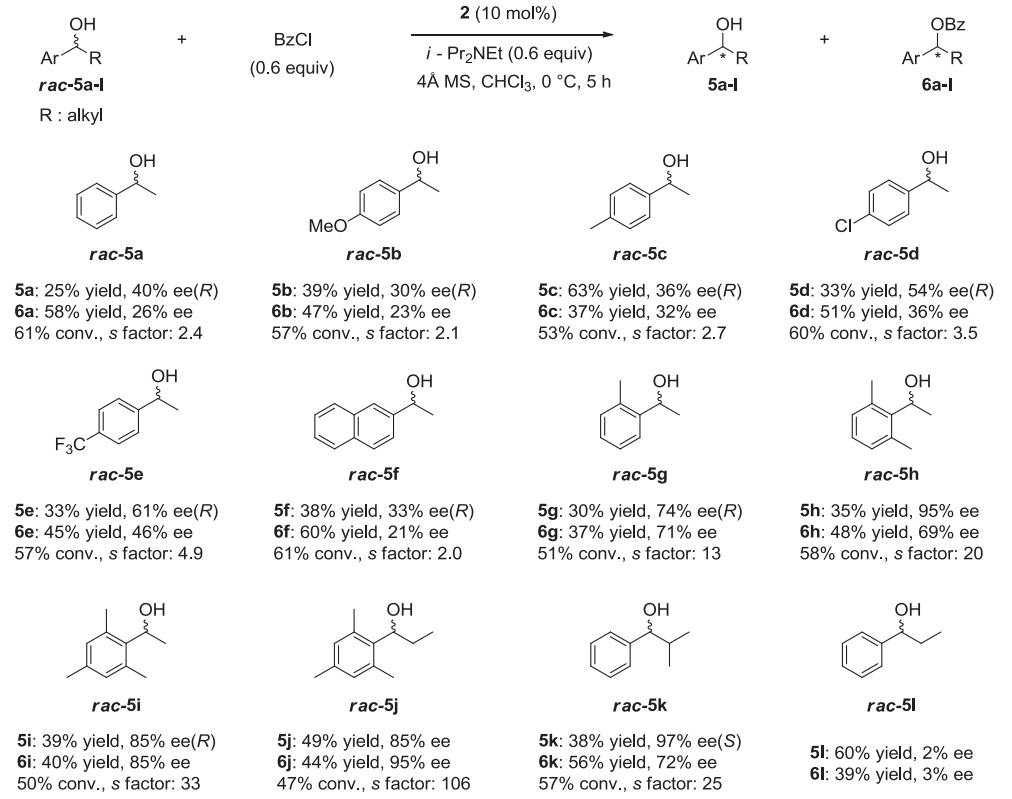
Entry	Solvent	x	Yield (%)[a] (% ee)[b] of 3a	Yield (%)[a] (% ee)[b] of 4a	Conv. (%)[c]	s[d]
1	$CHCl_3$	10	41 (80)	40 (82)	49	25
2	CH_3CN	10	45 (66)	38 (71)	48	12
3	CH_2Cl_2	10	48 (65)	48 (84)	44	22
4	$Cl(CH_2)_2Cl$	10	68 (39)	30 (88)	31	23
5	Toluene	10	88 (4)	10 (80)	5	9
6	$CHCl_3$	5	55 (63)	40 (86)	42	25

[a] Isolated yields.
[b] Determined by chiral HPLC analysis.
[c] Conversion (%) = 100 × ee of unreacted alcohol/(ee of unreacted alcohol + ee of benzoate), see Ref. 15.
[d] Selectivity factor = k (fast-reacting enantiomer)/k (slow-reacting enantiomer), see Ref. 15.



Scheme 1. Kinetic resolution of aryl cycloalkyl carbinols.

The steric effect of substituents located close to a hydroxyl group in secondary alcohols was also investigated. Similar reaction of a 1-arylethanol bearing a methyl group at the *ortho* position on the benzene ring (***rac*-5g**) afforded the corresponding benzoate and unreacted alcohol with a significantly increased selectivity factor ($s = 13$). Moreover, the selectivity factors increased with an increase in the number of methyl groups on the benzene ring, and 2,6-dimethylphenyl- and 2,4,6-trimethylphenyl carbinols (***rac*-5h, 5i**) could be resolved with $s = 20$ and 33, respectively. Similar reaction of a more sterically demanding substrate (***rac*-5j**) proceeded with an excellent selectivity ($s = 106$). The absolute configuration of the main alcohols recovered from the reaction of these sterically hindered carbinols was assumed to be *R*, similar to the reactions of 1-arylethanols ***rac*-5a–f**, because the optical rotation data showed that the *R* isomers were enriched in the recovered alcohols obtained from the reaction of ***rac*-5g** and ***rac*-5i**. On the other hand, carbinol with a branched alkyl group, but not a sterically demanding aryl group was also an effective substrate for the acylative kinetic resolution catalyzed by **2**. The reaction of 2-methyl-1-phenylpropan-1-ol ***rac*-5k** was achieved with an acceptable



Scheme 2. Kinetic resolution of aryl acyclic alkyl carbinols.

stereoselectivity ($s = 25$). In contrast to the result, a similar substrate in which the alkyl group was not branched (***rac*-5l**) was not resolved with stereoselectivity. Interestingly, *S* isomer was the preferentially recovered alcohol from the reaction of ***rac*-5k** opposite to the reaction of other aryl acyclic alkyl carbinols. These results obtained from the reactions of ***rac*-5k** indicate that branching at the carbon atom adjacent to the carbinol carbon atom significantly affected the recognition of substrates using catalyst **2**. Given that absolute configuration of recovered alcohols was inverse, a reactive intermediate seemed to recognize a substituent around the carbinol carbon as the largest one in the following order; a branched sp^3 carbon (except for cyclopropyl) > a branched sp^2 carbon > a methyl group. The reactive intermediate could not be unambiguously identified yet, but it is possible that the benzoyl group was transferred to the hydroxyl group from the benzoyl phosphonium salt generated from the catalyst **2** and benzoyl chloride. The benzoyl phosphonium salt was a very bulky group. Therefore, the present kinetic resolution seemed to be strictly influenced by the steric interaction between the largest substituent in the carbinol and benzoyl phosphonium moiety in the intermediate.

3. Conclusions

In this work, kinetic resolution of diverse secondary alcohols was attempted by benzoylation reaction catalyzed by aminophosphinite organocatalyst **2**. Although phenyl- or *para*-substituted phenyl alkanols were not effective substrates, bulky aryl cycloalkyl carbinols could be resolved with a practical level of selectivity. The steric effect of the substituents located around carbinols significantly affected the efficiency of the resolution, and (2,4,6-trimethylphenyl)propan-1-ol was resolved with $s = 106$. Especially, branching of the alkyl group at the position adjacent to the carbinol carbon atom of aryl alkyl carbinols significantly affect the stereoselectivity and absolute configuration of the products.

4. Experimental section

4.1. General procedures

^{1}H NMR spectra were performed using a Bruker Avance 400 spectrometer. Proton chemical shifts are reported in parts per million (δ, ppm) relative to the internal standard tetramethylsilane (TMS, δ 0.0 ppm). High-resolution mass spectra were obtained using a Bruker micrOTOF II LC-MS spectrometer equipped with an electrospray ionization source (ESI). All the high-resolution mass spectra were performed using anhydrous CH_3CN as the solvent. Enantiomeric excess was determined by HPLC analysis using a Jasco Gulliver series HPLC equipped with a Daicel Chiralcel column as indicated below. All the peaks of HPLC were detected with 254-nm UV light. Specific rotation was measured using a Jasco DIP-370 digital polarimeter. Flash column chromatography was performed using Cica-Merck Silica Gel 60 N (40–50 μm). All the reactions were performed using dried glassware under nitrogen atmosphere. All the solvents were purified by conventional methods before use. Catalyst **2** was prepared as previously described.[13b]

4.2. General procedure for the kinetic resolution of secondary alcohols by catalytic benzoylation

A two-neck round-bottom flask containing powdered 4 Å molecular sieves (0.6 g) was dried under nitrogen atmosphere. A solution of secondary alcohol (3 mmol) in $CHCl_3$ (6 mL) was added to the flask. Subsequently, catalyst **2** (108 mg, 0.3 mmol) and *i*-Pr_2NEt (233 mg, 1.8 mmol) were added to the solution, and benzoyl chloride (253 mg, 1.8 mmol) was added dropwise to the mixture at 0 °C. After the resulting mixture was stirred at 0 °C for 5 h, the molecular sieves were filtered with suction filtration. To the filtrate, water was added, and the mixture was extracted with $CHCl_3$. The combined organic layers were washed with brine, dried over Na_2SO_4, and concentrated under reduced pressure. The crude reaction mixture was purified by flash column chromatography using a mixture of ethyl acetate/*n*-hexane, affording the corresponding benzoate and recovering the alcohol.

4.2.1. Cyclohexyl(phenyl)methanol (***rac*-3a**)

Recovered alcohol (***3a***)[16]: 214 mg (37% yield), colorless solid; $[\alpha]_D^{28}$ −30.9 (c 1.05, CH_2Cl_2) [lit. $[\alpha]_D^{25}$ +28.0 (c 1.0, CH_2Cl_2), 92% ee (*R*)]; ^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.36–7.24 (m, 5H), 4.37 (dd, $J = 3.3$, 7.2 Hz, 1H), 2.02–1.95 (m, 1H), 1.80–1.73 (m, 2H), 1.70–1.57 (m, 3H), 1.41–1.35 (m, 1H), 1.28–0.88 (m, 5H); HPLC (AD-H, hexane/*i*-PrOH = 99/1, flow rate = 1.0 mL/min), retention times: 24.4 min (major), 28.5 min (minor) (99% ee). *Benzoate* (***4a***): 501 mg (57% yield), colorless solid; $[\alpha]_D^{29}$ −23.9 (c 1.00, CH_2Cl_2); ^{1}H NMR ($CDCl_3$, 400 MHz) δ 8.10–8.07 (m, 2H), 7.58–7.53 (m, 1H), 7.47–7.42 (m, 2H), 7.38–7.30 (m, 4H), 7.28–7.24 (m, 1H), 5.74 (d, $J = 7.4$ Hz, 1H), 1.98–1.87 (m, 2H), 1.79–1.61 (m, 3H), 1.54–1.47 (m, 1H), 1.30–0.98 (m, 5H); HRMS (ESI-TOF) *m/z* $[M + Na]^+$, calcd for $C_{20}H_{22}NaO_2$ 317.1512, found 317.1516; HPLC (AD-H, hexane/*i*-PrOH = 98/2, flow rate = 1.0 mL/min), retention times: 8.5 min (major), 11.1 min (minor) (63% ee).

4.2.2. Cycloheptyl(phenyl)methanol (***rac*-3b**)

Recovered alcohol (***3b***)[17]: 279 mg (46% yield), colorless oil; $[\alpha]_D^{30}$ −13.8 (c 1.20, $CHCl_3$); ^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.36–7.24 (m, 5H), 4.47 (dd, $J = 3.3$, 6.4 Hz, 1H), 1.92–1.81 (m, 2H), 1.78 (d, $J = 3.3$ Hz, 1H), 1.72–1.30 (m, 10H), 1.22–1.12 (m, 1H); HPLC (AD-H, hexane/*i*-PrOH = 99/1, flow rate = 0.6 mL/min), retention times: 36.1 min (major), 41.1 min (minor) (92% ee). *Benzoate* (***4b***): 473 mg (51% yield), colorless solid; $[\alpha]_D^{31}$ −27.0 (c 1.32, $CHCl_3$); ^{1}H NMR ($CDCl_3$, 400 MHz) δ 8.10–8.07 (m, 2H), 7.57–7.52 (m, 1H), 7.46–7.42 (m, 2H), 7.39–7.37 (m, 2H), 7.34–7.29 (m, 2H), 7.27–7.22 (m, 1H), 5.84 (d, $J = 7.2$ Hz, 1H), 2.18–2.10 (m, 1H), 1.94–1.86 (m, 1H), 1.72–1.34 (m, 10H), 1.31–1.21 (m, 1H); HRMS (ESI-TOF) *m/z* $[M + Na]^+$, calcd for $C_{21}H_{24}NaO_2$ 331.1669, found 331.1665; HPLC (AD-H, hexane/*i*-PrOH = 98/2, flow rate = 1.0 mL/min), retention times: 10.3 min (major), 12.9 min (minor) (78% ee).

4.2.3. Cyclopentyl(phenyl)methanol (***rac*-3c**)

Recovered alcohol (***3c***)[18]: 286 mg (54% yield), colorless oil; $[\alpha]_D^{25}$ −34.2 (c 1.06, $CHCl_3$) [lit. $[\alpha]_D^{20}$ −44.4 (c 0.9, $CHCl_3$), >99% ee (*S*)]; ^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.35–7.24 (m, 5H), 4.40 (dd, $J = 3.2$, 8.4 Hz, 1H), 2.27–2.17 (m, 1H), 1.92–1.85 (m, 2H), 1.72–1.43 (m, 5H), 1.41–1.34 (m, 1H), 1.20–1.11 (m, 1H); HPLC (OB-H, hexane/*i*-PrOH = 95/5, flow rate = 0.2 mL/min), retention times: 33.3 min (major), 37.3 min (minor) (63% ee). *Benzoate* (***4c***): 372 mg (44% yield), colorless oil; $[\alpha]_D^{26}$ −16.4 (c 1.06, $CHCl_3$); ^{1}H NMR ($CDCl_3$, 400 MHz) δ 8.09–8.06 (m, 2H), 7.57–7.53 (m, 1H), 7.46–7.41 (m, 4H), 7.34–7.31 (m, 2H), 7.28–7.24 (m, 1H), 5.79 (d, $J = 8.7$ Hz, 1H), 2.57–2.47 (m, 1H), 1.90–1.84 (m, 1H), 1.72–1.46 (m, 6H), 1.32–1.22 (m, 1H); HRMS (ESI-TOF) *m/z* $[M + Na]^+$, calcd for $C_{19}H_{20}NaO_2$ 303.1356, found 303.1351; HPLC (AD-H, hexane/*i*-PrOH = 90/10, flow rate = 1.0 mL/min), retention times: 5.6 min (major), 6.8 min (minor) (77% ee).

4.2.4. Cyclopropyl(phenyl)methanol (***rac*-3d**)

Recovered alcohol (***3d***)[19]: 188 mg (42% yield), colorless oil; $[\alpha]_D^{26}$ −10.1 (c 1.01, CH_2Cl_2) [lit. $[\alpha]_D^{28}$ −12.89 (c 0.57, CH_2Cl_2), 43% ee (*R*)]; ^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.44–7.41 (m, 2H), 7.38–7.33 (m, 2H), 7.31–7.26 (m, 1H), 4.02 (d, $J = 8.3$ Hz, 1H), 1.94 (s, 1H), 1.27–1.18

(m, 1H), 0.68–0.61 (m, 1H), 0.59–0.53 (m, 1H), 0.51–0.45 (m, 1H), 0.41–0.35 (m, 1H); HPLC (OJ-H, hexane/*i*-PrOH = 90/10, flow rate = 1.0 mL/min), retention times: 10.6 min (major), 9.5 min (minor) (35% ee). *Benzoate (**4d**)*[20]: 385 mg (51% yield), colorless oil; $[\alpha]_D^{26}$ +15.5 (c 1.13, $CHCl_3$); ^1H NMR ($CDCl_3$, 400 MHz) δ 8.12–8.09 (m, 2H), 7.58–7.54 (m, 1H), 7.48–7.43 (m, 4H), 7.38–7.34 (m, 2H), 7.32–7.28 (m, 1H), 5.51 (d, J = 8.5 Hz, 1H), 1.48–1.40 (m, 1H), 0.69–0.59 (m, 3H), 0.50–0.46 (m, 1H); HPLC (AD-H, hexane/*i*-PrOH = 90/10, flow rate = 1.0 mL/min), retention times: 6.9 min (major), 5.8 min (minor) (32% ee).

4.2.5. Cyclohexyl(pyridin-2-yl)methanol (**rac-3e**)

*Recovered alcohol (**3e**)*[21]: 154 mg (40% yield, reaction using 2 mmol of rac-3e), colorless solid; $[\alpha]_D^{24}$ −19.7 (c 0.99, $CHCl_3$) [lit. $[\alpha]_D^{20}$ −3.7 (c 0.42, $CHCl_3$), 63% ee]; ^1H NMR ($CDCl_3$, 400 MHz) δ 8.55–8.53 (m, 1H), 7.68–7.64 (m, 1H), 7.23–7.17 (m, 2H), 4.52 (d, J = 4.6 Hz, 1H), 4.09 (brs, 1H), 1.78–1.58 (m, 5H), 1.50–1.43 (m, 1H), 1.29–1.06 (m, 5H); HPLC (AD-H, hexane/*i*-PrOH = 99/1, flow rate = 1.0 mL/min), retention times: 24.4 min (major), 27.9 min (minor) (99% ee). *Benzoate (**4e**)*: 327 mg (55% yield), colorless oil; $[\alpha]_D^{24}$ −32.1 (c 0.91, $CHCl_3$); ^1H NMR ($CDCl_3$, 400 MHz) δ 8.62–8.60 (m, 1H), 8.14–8.11 (m, 2H), 7.66–7.62 (m, 1H), 7.60–7.55 (m, 1H), 7.48–7.44 (m, 2H), 7.33 (d, J = 7.8 Hz, 1H), 7.20–7.16 (m, 1H), 5.85 (d, J = 6.4 Hz, 1H), 2.23–2.14 (m, 1H), 1.86–1.63 (m, 4H), 1.59–1.52 (m, 1H), 1.32–1.11 (m, 5H); HRMS (ESI-TOF) *m/z* $[M + Na]^+$, calcd for $C_{19}H_{21}NNaO_2$ 318.1465, found 318.1473; HPLC (AD-H, hexane/*i*-PrOH = 99/1, flow rate = 1.0 mL/min), retention times: 31.9 min (major), 61.0 min (minor) (79% ee).

4.2.6. Cyclohexyl(pyridin-3-yl)methanol (**rac-3f**)

*Recovered alcohol (**3f**)*[22]: 106 mg (28% yield, reaction using 2 mmol of rac-3f), colorless solid; ^1H NMR ($CDCl_3$, 400 MHz) δ 8.45–8.44 (m, 2H), 7.67–7.64 (m, 1H), 7.27–7.24 (m, 1H), 4.42 (d, J = 6.9 Hz, 1H), 2.81 (brs, 1H), 1.96–1.93 (m, 1H), 1.79–1.75 (m, 1H), 1.71–1.57 (m, 3H), 1.42–1.38 (m, 1H), 1.28–0.90 (m, 5H); HPLC (AD-H, hexane/*i*-PrOH = 90/10, flow rate = 1.0 mL/min), retention times: 14.0 min (major), 10.0 min (minor) (2% ee). *Benzoate (**4f**)*: 216 mg (37% yield), colorless solid; ^1H NMR ($CDCl_3$, 400 MHz) δ 8.65 (d, J = 2.0 Hz, 1H), 8.54–8.52 (m, 1H), 8.08–8.06 (m, 2H), 7.69–7.66 (m, 1H), 7.60–7.55 (m, 1H), 7.48–7.44 (m, 2H), 7.28–7.25 (m, 1H), 5.77 (d, J = 7.4 Hz, 1H), 1.98–1.90 (m, 2H), 1.80–1.66 (m, 3H), 1.53–1.50 (m, 1H), 1.31–0.99 (m, 5H); HRMS (ESI-TOF) *m/z* $[M + Na]^+$, calcd for $C_{19}H_{21}NNaO_2$ 318.1465, found 318.1437; HPLC (AD-H, hexane/*i*-PrOH = 90/10, flow rate = 1.0 mL/min), retention times: 10.7 min (major), 17.0 min (minor) (5% ee).

4.2.7. Cyclohexyl(furan-2-yl)methanol (**rac-3g**)

*Recovered alcohol (**3g**)*[23]: 189 mg (35% yield), yellow oil; $[\alpha]_D^{24}$ −21.1 (c 1.03, $CHCl_3$) [lit. $[\alpha]_D^{20}$ −16.4 (c 1.1, $CHCl_3$), 84% ee (*S*)]; ^1H NMR ($CDCl_3$, 400 MHz) δ 7.37–7.36 (m, 1H), 6.33–6.32 (m, 1H), 6.21 (d, J = 3.2 Hz, 1H), 4.38 (d, J = 7.0 Hz, 1H), 2.01–1.94 (m, 1H), 1.83–1.74 (m, 3H), 1.72–1.63 (m, 2H), 1.47–1.41 (m, 1H), 1.32–0.92 (m, 5H); HPLC of benzoate derivative of **3g** which was obtained by kinetic resolution. (OJ-H, hexane/*i*-PrOH = 99/1, flow rate = 0.4 mL/min), retention times: 17.6 min (major), 20.7 min (minor) (99% ee). *Benzoate (**4g**)*: 498 mg (58% yield), yellow oil; $[\alpha]_D^{24}$ +43.7 (c 1.15, $CHCl_3$); ^1H NMR ($CDCl_3$, 400 MHz) δ 8.08–8.05 (m, 2H), 7.57–7.52 (m, 1H), 7.45–7.38 (m, 3H), 6.36–6.31 (m, 2H), 5.84 (d, J = 8.5 Hz, 1H), 2.20–2.10 (m, 1H), 1.98–1.91 (m, 1H), 1.79–1.64 (m, 3H), 1.55–1.48 (m, 1H), 1.34–0.99 (m, 5H); HRMS (ESI-TOF) *m/z* $[M + Na]^+$, calcd for $C_{18}H_{20}NaO_3$ 307.1305, found 307.1307; HPLC (OJ-H, hexane/*i*-PrOH = 99/1, flow rate = 0.4 mL/min), retention times: 17.7 min (minor), 19.9 min (major) (63% ee).

4.2.8. Cyclohexyl(thiophen-2-yl)methanol (**rac-3h**)

*Recovered alcohol (**3h**)*[24]: 209 mg (35% yield), colorless oil; $[\alpha]_D^{28}$ −13.8 (c 1.22, $CHCl_3$) [lit. $[\alpha]_D^{23}$ −2.3 (c 1.2, $CHCl_3$), 20% ee (*S*)]; ^1H NMR ($CDCl_3$, 400 MHz) δ 7.26–7.23 (m, 1H), 6.98–6.93 (m, 2H), 4.63 (d, J = 7.2 Hz, 1H), 2.06–1.97 (m, 2H), 1.81–1.75 (m, 1H), 1.73–1.61 (m, 3H), 1.51–1.46 (m, 1H), 1.31–0.92 (m, 5H); HPLC (OB-H, hexane/*i*-PrOH = 98/2, flow rate = 0.7 mL/min), retention times: 17.7 min (major), 24.6 min (minor) (99% ee). *Benzoate (**4h**)*: 535 mg (59% yield), colorless oil; $[\alpha]_D^{29}$ +5.5 (c 1.20, $CHCl_3$); ^1H NMR ($CDCl_3$, 400 MHz) δ 8.08–8.06 (m, 2H), 7.57–7.53 (m, 1H), 7.46–7.42 (m, 2H), 7.26–7.24 (m, 1H), 7.08–7.06 (m, 1H), 6.97–6.95 (m, 1H), 6.06 (d, J = 8.0 Hz, 1H), 2.02–1.93 (m, 2H), 1.80–1.55 (m, 4H), 1.32–0.99 (m, 5H); HRMS (ESI-TOF) *m/z* $[M + Na]^+$, calcd for $C_{18}H_{20}NaO_2S$ 323.1076, found 323.1076; HPLC (AD-H, hexane/*i*-PrOH = 99/1, flow rate = 0.4 mL/min), retention times: 24.8 min (major), 30.5 min (minor) (57% ee).

4.2.9. 1-Phenylethanol (**rac-5a**)

*Recovered alcohol (**5a**)*[25]: 93.2 mg (25% yield), colorless oil; $[\alpha]_D^{24}$ +18.1 (c 1.07, CH_2Cl_2) [lit. $[\alpha]_D^{20}$ +48.3 (c 1.0, CH_2Cl_2), 97% ee (*R*)]; ^1H NMR ($CDCl_3$, 400 MHz) δ 7.39–7.33 (m, 4H), 7.30–7.25 (m, 1H), 4.90 (q, J = 6.4 Hz, 1H), 1.80 (brs, 1H), 1.53 (d, J = 6.4 Hz, 3H); HPLC (OJ-H, hexane/*i*-PrOH = 95/5, flow rate = 1.0 mL/min), retention times: 11.2 min (minor), 13.0 min (major) (40% ee). *Benzoate (**6a**)*[26]: 394 mg (58% yield), colorless oil; $[\alpha]_D^{24}$+8.85 (c 1.03, EtOH) [lit. $[\alpha]_D^{23}$ −23 (c 1.2, EtOH), 92% ee (*R*)]; ^1H NMR ($CDCl_3$, 400 MHz) δ 8.08 (brd, J = 7.4 Hz, 2H), 7.58–7.53 (m, 1H), 7.46–7.42 (m, 4H), 7.39–7.35 (m, 2H), 7.32–7.27 (m, 1H), 6.14 (q, J = 6.6 Hz, 1H), 1.67 (d, J = 6.6 Hz, 3H); HPLC (AD-H, hexane/*i*-PrOH = 98/2, flow rate = 1.0 mL/min), retention times: 7.2 min (minor), 9.4 min (major) (26% ee).

4.2.10. 1-(4-Methoxyphenyl)ethanol (**rac-5b**)

*Recovered alcohol (**5b**)*[27]: 177 mg (39% yield), colorless oil; $[\alpha]_D^{30}$ +12.1 (c 1.05, $CHCl_3$) [lit. $[\alpha]_D^{20}$ +47.8 (c 1.60, $CHCl_3$), 97% ee (*R*)]; ^1H NMR ($CDCl_3$, 400 MHz) δ 7.32–7.28 (m, 2H), 6.90–6.86 (m, 2H), 4.86 (q, J = 6.4 Hz, 1H), 3.80 (s, 3H), 1.88 (brs, 1H), 1.48 (d, J = 6.4 Hz, 3H); HPLC (OJ-H, hexane/*i*-PrOH = 98/2, flow rate = 1.0 mL/min), retention times: 54.2 min (minor), 57.4 min (major) (30% ee). *Benzoate (**6b**)*[28]: 360 mg (47% yield), colorless oil; $[\alpha]_D^{24}$ +2.2 (c 1.22, $CHCl_3$); ^1H NMR ($CDCl_3$, 400 MHz) δ 8.07–8.05 (m, 2H), 7.56–7.52 (m, 1H), 7.44–7.37 (m, 4H), 6.91–6.88 (m, 2H), 6.10 (q, J = 6.6 Hz, 1H), 3.80 (s, 3H), 1.66 (d, J = 6.6 Hz, 3H); HPLC (OJ-H, hexane/*i*-PrOH = 95/5, flow rate = 0.8 mL/min), retention times: 9.4 min (minor), 10.5 min (major) (23% ee).

4.2.11. 1-(4-Methylphenyl)ethanol (**rac-5c**)

*Recovered alcohol (**5c**)*[29]: 256 mg (63% yield), colorless oil; $[\alpha]_D^{27}$ +12.6 (c 0.99, CH_2Cl_2) [lit. $[\alpha]_D^{20}$ +18.5 (c 4.0, CH_2Cl_2), 90% ee (*R*)]; ^1H NMR ($CDCl_3$, 400 MHz) δ 7.26 (d, J = 8.1 Hz, 2H), 7.16 (d, J = 8.1 Hz, 2H), 4.86 (q, J = 6.4 Hz,1H), 2.34 (s, 3H), 1.79 (brs, 1H), 1.48 (d, J = 6.4 Hz, 3H); HPLC (OB-H, hexane/*i*-PrOH = 95/5, flow rate = 1.0 mL/min), retention times: 7.9 min (minor), 8.7 min (major) (36% ee). *Benzoate (**6c**)*[30]: 266 mg (37% yield), colorless oil; $[\alpha]_D^{25}$ +5.2 (c 0.92, $CHCl_3$); ^1H NMR ($CDCl_3$, 400 MHz) δ 8.09–8.06 (m, 2H), 7.56–7.52 (m, 1H), 7.45–7.41 (m, 2H), 7.34 (d, J = 8.0 Hz, 2H), 7.17 (d, J = 8.0 Hz, 2H), 6.10 (q, J = 6.6 Hz, 1H), 2.34 (s, 3H), 1.66 (d, J = 6.6 Hz, 3H); HPLC (AD-H, hexane/*i*-PrOH = 99/1, flow rate = 1.0 mL/min), retention times: 7.1 min (minor), 11.5 min (major) (32% ee).

4.2.12. 1-(4-Chlorophenyl)ethanol (**rac-5d**)

*Recovered alcohol (**5d**)*[31]: 155 mg (33% yield), pale yellow oil; $[\alpha]_D^{30}$ +28.1 (c 0.97, Et_2O) [lit. $[\alpha]_D^{25}$ −36.0 (c 1.0, Et_2O), 72% ee (*S*)]; ^1H NMR ($CDCl_3$, 400 MHz) δ 7.31 (s, 4H), 4.88 (q, J = 6.4 Hz, 1H), 1.82

(brs, 1H), 1.48 (d, J = 6.4 Hz, 3H); HPLC (OJ-H, hexane/*i*-PrOH = 97/3, flow rate = 1.0 mL/min), retention times: 15.5 min (minor), 16.5 min (major) (54% ee). *Benzoate* (***6d***)[32]: 401 mg (51% yield), colorless oil; $[\alpha]_D^{25}$ +12.0 (c 1.01, $CHCl_3$); ^{1}H NMR ($CDCl_3$, 400 MHz) δ 8.08–8.05 (m, 2H), 7.59–7.54 (m, 1H), 7.47–7.42 (m, 2H), 7.40–7.32 (m, 4H), 6.09 (q, J = 6.6 Hz, 1H), 1.65 (d, J = 6.6 Hz, 3H); HPLC (AD-H, hexane/*i*-PrOH = 98/2, flow rate = 0.5 mL/min), retention times: 10.5 min (minor), 15.7 min (major) (36% ee).

4.2.13. 1-(4-(Trifluoromethyl)phenyl)ethanol (*rac-5e*)

Recovered alcohol (***5e***)[33]: 186 mg (33% yield), colorless oil; $[\alpha]_D^{28}$ +14.9 (c 1.07, $CHCl_3$) [lit. $[\alpha]_D$ +24.14 (c 1.41, $CHCl_3$), >99% ee (*R*)]; ^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.61 (d, J = 8.2 Hz, 2H), 7.49 (d, J = 8.2 Hz, 2H), 5.00–4.94 (m, 1H), 1.87 (d, J = 3.6 Hz, 1H), 1.51 (d, J = 6.5 Hz, 3H); HPLC of a benzoate derivative of **5e** which was recovered after kinetic resolution. (AD-H, hexane/*i*-PrOH = 95/5, flow rate = 0.8 mL/min), retention times: 5.5 min (major), 6.9 min (minor) (61% ee). *Benzoate* (***6e***)[30]: 397 mg (45% yield), colorless oil; $[\alpha]_D^{24}$ +19.6 (c 1.08, $CHCl_3$); ^{1}H NMR ($CDCl_3$, 400 MHz) δ 8.10–8.07 (m, 2H), 7.64–7.54 (m, 5H), 7.48–7.44 (m, 2H), 6.16 (q, J = 6.6 Hz, 1H), 1.68 (d, J = 6.6 Hz, 3H); HPLC (AD-H, hexane/*i*-PrOH = 95/5, flow rate = 0.8 mL/min), retention times: 5.2 min (minor), 6.2 min (major) (46% ee).

4.2.14. 1-(Naphtalen-2-yl)ethanol (*rac-5f*)

Recovered alcohol (***5f***)[34]: 197 mg (38% yield), colorless solid; $[\alpha]_D^{26}$ +12.7 (c 1.27, MeOH) [lit. $[\alpha]_D^{25}$ −40.8 (c 0.90, MeOH), 79% ee (*S*)]; ^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.85–7.81 (m, 4H), 7.52–7.44 (m, 3H), 5.07 (q, J = 6.4 Hz, 1H), 1.91 (s, 1H), 1.58 (d, J = 6.4 Hz, 3H); HPLC (OJ-H, hexane/*i*-PrOH = 95/5, flow rate = 1.0 mL/min), retention times: 20.4 min (minor), 27.1 min (major) (33% ee). *Benzoate* (***6f***)[35]: 498 mg (60% yield), colorless solid; $[\alpha]_D^{27}$ +13.6 (c 1.02, MeOH); ^{1}H NMR ($CDCl_3$, 400 MHz) δ 8.12–8.09 (m, 2H), 7.89–7.80 (m, 4H), 7.59–7.53 (m, 2H), 7.50–7.42 (m, 4H), 6.30 (q, J = 6.6 Hz, 1H), 1.76 (d, J = 6.6 Hz, 3H); HPLC (AD-H, hexane/*i*-PrOH = 98/2, flow rate = 1.0 mL/min), retention times: 11.8 min (minor), 16.6 min (major) (21% ee).

4.2.15. 1-(2-Methylphenyl)ethanol (*rac-5g*)

Recovered alcohol (***5g***)[36]: 122 mg (30% yield), colorless oil; $[\alpha]_D^{26}$ +46.4 (c 1.00, EtOH) [lit. $[\alpha]_D$ +61.9 (c 1.01, EtOH), 96% ee (*R*)]; ^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.52–7.50 (m, 1H), 7.23–7.21 (m, 1H), 7.19–7.11 (m, 2H), 5.13 (q, J = 6.4 Hz, 1H), 2.34 (s, 3H), 1.75 (s, 1H), 1.46 (d, J = 6.4 Hz, 3H); HPLC (AD-H, hexane/*i*-PrOH = 95/5, flow rate = 0.5 mL/min), retention times: 18.7 min (major), 21.0 min (minor) (74% ee). *Benzoate* (***6g***)[37]: 268 mg (37% yield), colorless oil; $[\alpha]_D^{25}$ +38.7 (c 0.99, $CHCl_3$); ^{1}H NMR ($CDCl_3$, 400 MHz) δ 8.10–8.07 (m, 2H), 7.57–7.49 (m, 2H), 7.46–7.42 (m, 2H), 7.25–7.15 (m, 3H), 6.32 (q, J = 6.6 Hz, 1H), 2.44 (s, 3H), 1.64 (d, J = 6.6 Hz, 3H); HPLC (OJ-H, hexane/*i*-PrOH = 97/3, flow rate = 0.5 mL/min), retention times: 13.7 min (minor), 16.4 min (major) (71% ee).

4.2.16. 1-(2,6-Dimethylphenyl)ethanol (*rac-5h*)

Recovered alcohol (***5h***)[3g]: 157 mg (35% yield), colorless solid; $[\alpha]_D^{24}$ +66.8 (c 0.98, $CHCl_3$); ^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.05–6.98 (m, 3H), 5.39 (dq, J = 2.2, 6.8 Hz, 1H), 2.45 (s, 6H), 1.71 (d, J = 2.2 Hz, 1H), 1.54 (d, J = 6.8 Hz, 3H); HPLC (OD-H, hexane/*i*-PrOH = 95/5, flow rate = 1.0 mL/min), retention times: 7.7 min (minor), 9.4 min (major) (95% ee). *Benzoate* (***6h***): 369 mg (48% yield), colorless oil; $[\alpha]_D^{24}$ +98.0 (c 0.98, $CHCl_3$); ^{1}H NMR ($CDCl_3$, 400 MHz) δ 8.07–8.05 (m, 2H), 7.56–7.53 (m, 1H), 7.45–7.41 (m, 2H), 7.08–6.99 (m, 3H), 6.55 (q, J = 7.0 Hz, 1H), 2.55 (s, 6H), 1.71 (d, J = 7.0 Hz, 3H); HRMS (ESI-TOF) m/z $[M + Na]^+$, calcd for $C_{17}H_{18}NaO_2$ 277.1199, found 277.1200; HPLC (OD-H, hexane/*i*-PrOH = 90/10, flow rate = 0.3 mL/min), retention times: 13.8 min (minor), 14.9 min (major) (69% ee).

4.2.17. 1-(2,4,6-Trimethylphenyl)ethanol (*rac-5i*)

Recovered alcohol (***5i***)[38]: 194 mg (39% yield), colorless solid; $[\alpha]_D^{28}$ +55.0 (c 0.95, $CHCl_3$) [lit. $[\alpha]_D^{27}$ −60.6 (c 6.06, $CHCl_3$), 95% ee (*S*)]; ^{1}H NMR ($CDCl_3$, 400 MHz) δ 6.82 (s, 2H), 5.36 (q, J = 6.8 Hz, 1H), 2.41 (s, 6H), 2.24 (s, 3H), 1.67 (s, 1H), 1.52 (d, J = 6.8 Hz, 3H); HPLC (AD-H, hexane/*i*-PrOH = 98/2, flow rate = 1.0 mL/min), retention times: 7.5 min (major), 8.3 min (minor) (85% ee). *Benzoate* (***6i***): 324 mg (40% yield), colorless oil; $[\alpha]_D^{24}$ +80.9 (c 0.94, $CHCl_3$); ^{1}H NMR ($CDCl_3$, 400 MHz) δ 8.07–8.05 (m, 2H), 7.56–7.52 (m, 1H), 7.45–7.41 (m, 2H), 6.83 (s, 2H), 6.51 (q, J = 6.9 Hz, 1H), 2.51 (s, 6H), 2.24 (s, 3H), 1.69 (d, J = 6.9 Hz, 3H); HRMS (ESI-TOF) m/z $[M + Na]^+$, calcd for $C_{18}H_{20}NaO_2$ 291.1356, found 291.1378; HPLC (AD-H, hexane/*i*-PrOH = 99/1, flow rate = 0.4 mL/min), retention times: 11.0 min (major), 12.7 min (minor) (85% ee).

4.2.18. 1-(2,4,6-Trimethylphenyl)-1-propanol (*rac-5j*)

Recovered alcohol (***5j***)[39]: 263 mg (49% yield), colorless solid; $[\alpha]_D^{24}$ +37.8 (c 1.0, $CHCl_3$); ^{1}H NMR ($CDCl_3$, 400 MHz) δ 6.81 (s, 2H), 5.05 (dd, J = 6.1, 8.4 Hz, 1H), 2.40 (s, 6H), 2.24 (s, 3H), 2.04–1.93 (m, 1H), 1.83–1.63 (m, 2H), 0.98 (t, J = 7.4 Hz, 3H); HPLC (AD-H, hexane/*i*-PrOH = 95/5, flow rate = 0.8 mL/min), retention times: 7.4 min (major), 8.3 min (minor) (85% ee). *Benzoate* (***6j***): 376 mg (44% yield), colorless oil; $[\alpha]_D^{23}$ +101.4 (c 1.07, $CHCl_3$); ^{1}H NMR ($CDCl_3$, 400 MHz) δ 8.08–8.05 (m, 2H), 7.56–7.52 (m, 1H), 7.45–7.41 (m, 2H), 6.82 (s, 2H), 6.29 (dd, J = 6.4, 8.9 Hz, 1H), 2.50 (s, 6H), 2.30–2.18 (m, 4H), 2.01–1.90 (m, 1H), 1.01 (t, J = 7.4 Hz, 3H); HRMS (ESI-TOF) m/z $[M + Na]^+$, calcd for $C_{19}H_{22}NaO_2$ 305.1512, found 305.1513; HPLC (AD-H, hexane/*i*-PrOH = 200/1, flow rate = 0.8 mL/min), retention times: 7.6 min (major), 12.4 min (minor) (95% ee).

4.2.19. 2-Methyl-1-phenyl-1-propanol (*rac-5k*)

Recovered alcohol (***5k***)[40]: 172 mg (38% yield), colorless oil; $[\alpha]_D^{27}$ −42.4 (c 1.13, $CHCl_3$) [lit. $[\alpha]_D^{23}$ +12.3 (c 1.2, $CHCl_3$), 38.3% ee (*R*)]; ^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.36–7.24 (m, 5H), 4.37 (dd, J = 3.2, 6.8 Hz, 1H), 2.02–1.90 (m,1H), 1.80 (d, J = 3.2 Hz, 1H), 1.00 (d, J = 6.7 Hz, 3H), 0.80 (d, J = 6.8 Hz, 3H); HPLC (OD-H, hexane/*i*-PrOH = 90/10, flow rate = 0.5 mL/min), retention times: 11.4 min (major), 12.6 min (minor) (97% ee). *Benzoate* (***6k***)[41]: 424 mg (56% yield), colorless oil; $[\alpha]_D^{28}$ −32.9 (c 1.36, $CHCl_3$); ^{1}H NMR ($CDCl_3$, 400 MHz) δ 8.11–8.08 (m, 2H), 7.58–7.54 (m, 1H), 7.47–7.43 (m, 2H), 7.39–7.31 (m, 4H), 7.29–7.24 (m, 1H), 5.73 (d, J = 7.2 Hz, 1H), 2.31–2.19 (m, 1H), 1.05 (d, J = 6.7 Hz, 3H), 0.90 (d, J = 6.8 Hz, 3H); HPLC (AD-H, hexane/*i*-PrOH = 98/2, flow rate = 1.0 mL/min), retention times: 7.9 min (major), 10.9 min (minor) (72% ee).

4.2.20. 1-Phenyl-1-propanol (*rac-5l*)

Recovered alcohol (***5l***)[25]: 245 mg (60% yield), colorless oil; ^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.37–7.33 (m, 4H), 7.31–7.25 (m, 1H), 4.60 (t, J = 6.6 Hz, 1H), 1.88–1.70 (m, 3H), 0.92 (t, J = 7.4 Hz, 3H); HPLC (OJ-H, hexane/*i*-PrOH = 98/2, flow rate = 1.0 mL/min), retention times: 16.6 min, 17.7 min (2% ee). *Benzoate* (***6l***)[32]: 280 mg (39% yield), colorless oil; ^{1}H NMR ($CDCl_3$, 400 MHz) δ 8.10–8.08 (m, 2H), 7.55–7.50 (m, 1H), 7.44–7.40 (m, 4H), 7.35–7.32 (m, 2H), 7.29–7.24 (m, 1H), 5.93 (t, J = 6.8 Hz, 1H), 2.12–1.89 (m, 2H), 0.96 (t, J = 7.4 Hz, 3H); HPLC (AD-H, hexane/*i*-PrOH = 99/1, flow rate = 1.0 mL/min), retention times: 9.8 min, 15.2 min (3% ee).

Appendix A. Supplementary data

Supplementary data related to this article can be found at https://doi.org/10.1016/j.tet.2017.11.062.

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