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Enzyme-catalyzed resolution of aromatic ring fused cyclic tertiary alcohols

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ABSTRACT

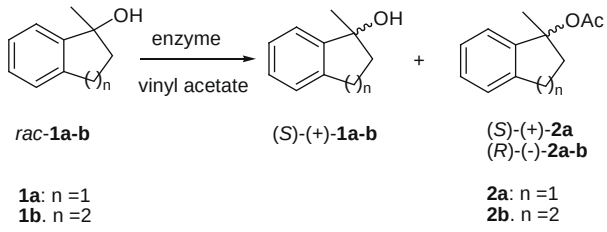
An efficient chemoenzymatic route for the synthesis of optically active aromatic ring fused cyclic tertiary alcohols (*S*)-(−)-1-methyl-1,2,3,4-tetrahydronaphthalen-1-ol (−)-**1b** and (*S*)-(+)-1-methyl-2,3-dihydro-1*H*-inden-1-ol (+)-**1a** has been developed. Different lipases have been tested in the transesterification of these tertiary alcohols; CAL-A (*Candida antarctica* Lipase A) was found to be the best biocatalyst for **1b** and CAL-A-CLEA (Lipase A, *C. antarctica*, cross-linked enzyme aggregate) for **1a**, obtained with ee values of 20% and 45%, respectively, and the corresponding esters **2b** and **2a** with the ee values of 99% and 71%, respectively.

1. Introduction

The construction of building blocks that contain chiral tertiary alcohol moieties is of great importance in the field of natural product synthesis and pharmaceuticals. Therefore, the synthesis of enantiopure tertiary alcohols and their esters has attracted considerable attention.[1] In particular, chiral aromatic fused hydrocarbon compounds, such as tetralin and indane have been classified as biomarkers since their structure is similar to that of the structural subunits of biological precursors, such as lipids and steroids.[2] Moreover, oxygen-containing derivatives of indane and tetralin are valuable intermediates for various chiral organic compounds,[3] for example, α-tetralone has been used to synthesize β-keto esters and thermorubin, an influential antibiotic substance.[4]

To the best of our knowledge, the synthesis of optically active tertiary alcohols of ketones (e.g., α-indanone and α-tetralone) is generally limited. One type is the synthesis of (*R*)-(−)-1-methyl-1,2,3,4-tetrahydronaphthalen-1-ol (−)-**1b** and (*S*)-(+)-1-methyl-2,3-dihydro-1*H*-inden-1-ol (+)-**1a** via their chiral trichromium complexes.[5] The other involves the catalytic asymmetric addition of alkylzinc reagents to ketones.[6] Furthermore, the enzymatic resolution of aromatic ring fused tertiary alcohols is also limited in the literature since there is great steric hindrance with regard to these molecules accepting the active site of the enzyme. It is well known that the structure of the substrate strongly affects the enantioselectivity. It has been reported that various hydrolases, PLE (Pig liver esterase), PPL (Porcine pancreatic lipase), CRL (*Candida rugosa* lipase), and CAL-A (Lipase A from *Candida antarctica*), are active biocatalysts toward esters of tertiary alcohols.[7] In particular, PLE has been shown to be an active biocatalyst in the resolution of (±)-3-ethynyl-3-oxbutyryl quiniclidine which afforded (*R*)-(+)-3-ethynyl-3-hydroxy quiniclidine and (*S*)-(−)-3-ethynyl-3-oxbutyryl quiniclidine in 97% and 99% ee, respectively.[8] Moreover, CAL-A was found to be the most efficient enzyme for the resolution of (±)-2-phenylbut-3-yn-2-ol while CRL showed low enantioselectivity.[9]

Herein, we report the results of the enzymatic resolution of (±)-1-methyl-2,3-dihydro-1*H*-inden-1-ol, (±)-**1a** and (±)-1-methyl-1,2,3,4-tetrahydronaphthalen-1-ol, (±)-**1b** (Scheme 1).



Scheme 1. Transesterification of (±)-1-methyl-2,3-dihydro-1*H*-inden-1-ol **1a** and (±)-1-methyl-1,2,3,4-tetrahydronaphthalen-1-ol **1b**.

2. Results and discussion

It is well known that the enantioselectivity of enzymatic reactions depends upon certain parameters, that is, temperature, cosolvent, and pH.[10] All reactions were performed by changing these parameters to determine the optimum conditions. Firstly, various hydrolases were tested, for example, CAL-A-CLEA, CRL, CAL-A, CAL-B, and Amano PS-C II, on (±)-1-methyl-1,2,3,4-tetrahydronaphthalen-1-ol (±)-**1b** by changing the substrate:enzyme ratio (w/w) from 1:1 to 1:0.25. Compound (±)-**1b** (100 mg) was subjected to transesterification with CAL-A (25 mg) with 1 mL of vinyl acetate (16 mmol equiv) and 1 mL of isooctane at room temperature

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(25 °C) for 168 h. No conversion was observed. Several cosolvents, for example, THF, hexane, and 1,4-dioxane, were also used but we did not obtain any results. Without cosolvent under the same conditions, no conversion was observed. Temperature effects were then tested. The experiment was screened at the temperatures ranging from 25 to 32 °C. (*R*)-(−)-1-Methyl-1,2,3,4-tetrahydronaphthalen-1-yl acetate, (−)-**2b** was obtained with 20% conversion in 99% ee beside compound (*S*)-(+)-**1b** in 20% ee after shaking for 144 h at 32 °C. Some unidentified decomposed products were also observed at higher temperatures.

The results obtained from CAL-A prompted us to test the other enzymes, that is, CAL-A-CLEA, in the transesterification of substrate (±)-**1b**. In the case of CAL-A-CLEA with the substrate:enzyme ratio (w/w) 1:0.5, (*R*)-(−)-1-methyl-1,2,3,4-tetrahydronaphthalen-1-yl acetate, (−)-**2b**, and (*S*)-(+)-1-methyl-1,2,3,4-tetrahydronaphthalen-1-ol, (+)-**1b** were isolated in 85% and 35% ee, respectively, in 20% conversion for 48 h. By changing the substrate:enzyme ratio (w/w) to 1:0.75, the ee value of (*R*)-(−)-1-methyl-1,2,3,4-tetrahydronaphthalen-1-yl acetate, (−)-**2b** was increased to 91% ee while that of the alcohol (*S*)-(+)-**1b** to 38% ee, at 25% conversion. The enzymatic resolution with Amano PS-C II of (±)-**1b** was carried out by changing the substrate:enzyme ratio (w/w) from 1:0.25 to 1:1. The best substrate:enzyme ratio (w/w) 1:1 afforded (*R*)-(−)-**2b** and (*S*)-(+)-**1b** in 80% ee and 15% ee, respectively, after 15% conversion for 96 h. CRL has been used in the enantioselective resolution of various esters of tertiary alcohols.[11] It was also used in the resolution of compound (±)-**1b**. The same optimization reactions were retried and the best result was obtained with 10% conversion and 90% ee of (*R*)-(−)-1-methyl-1,2,3,4-tetrahydronaphthalen-1-yl acetate, (−)-**2b** at 32 °C for 145 h with a 1:1 substrate:enzyme ratio (w/w) (Table 1).

The enzymatic resolution of (±)-1-methyl-2,3-dihydro-1*H*-inden-1-ol, (±)-**1a** was also examined with the same enzymes. The optimization reactions of (±)-**1a** were carried out by using CAL-A-CLEA with the substrate:enzyme ratio (w/w) 1:0.25 to afford (*R*)-(−)-**2a** in 71% ee with (*S*)-(+)-**1a** in 45% ee, respectively, after 20% conversion for 72 h. Amano PS-C II with substrate:enzyme ratio (w/w) 1:1 yielded (*S*)-(+)-**2a** in 15% ee after 7% conversion for 140 h. CRL did not work efficiently with compound (±)-**1a** and yielded (*R*)-(−)-**2a** in 15% ee after 5% conversion. Although CAL-A (Lipase A *C. antarctica*) worked well in the enzymatic resolution of compound (±)-**1b,** substrate **1a** was isolated as a racemic mixture. The results of the enzyme-catalyzed reaction of compound (±)-**1a** with various hydrolases are given in Table 2.

The absolute configurations of both (+)-**1a** and (+)-**1b** were assigned as (*S*), by comparison of their specific rotation values with the literature data.[5,12] Related to these results, the absolute configurations of (+)-**2a** and (−)-**2a** were assigned as (*S*) and (*R*), respectively, whereas those of (+)-**2b** and (−)-**2b** were assigned as (*S*) and (*R*), respectively.

3. Conclusion

In this report, we have demonstrated the first enzyme-catalyzed resolution of (±)-1-methyl-2,3-dihydro-1*H*-inden-1-ol, (±)-**1a** and (±)-1-methyl-1,2,3,4-tetrahydronaphthalen-1-ol, (±)-**1b** by using various hydrolases, that is, CAL-A-CLEA, CAL-A, CRL, and Amano PS-C II. According to the results obtained, CAL-A-CLEA was the best biocatalyst for (±)-1-methyl-2,3-dihydro-1*H*-inden-1-ol, (±)-**1a** in 45% ee and corresponding ester with 71% ee. Furthermore, CAL-A was the best for (±)-1-methyl-1,2,3,4-tetrahydronaphthalen-1-ol, (±)-**1b** in 20% ee and the corresponding ester with 99% ee. Temperature is an important parameter in reactions catalyzed by enzymes.[13,14] In this study, 32 °C was the optimum temperature. Compounds (±)-**2a**–**b** were easily decomposed at higher temperatures. In particular, the methodology should be useful in the synthesis of chiral drugs and their building blocks. We are currently studying the enzymatic resolution of various tertiary alcohols synthesized from α,β-unsaturated cyclic ketones.

4. Experimental

^{1}H NMR and ^{13}C NMR spectra were recorded in $CDCl_3$ on a Bruker Spectrospin Avance DPX-400 spectrometer and the chemical shifts are given in ppm downfield from tetramethylsilane. Infrared spectra were recorded on a Shimadzu 8400S FT-IR spectrophotometer. Optical rotations were measured in a 1 dm cell Rudolph Research Analytical Autopol III polarimeter. Melting points were determined on an Electrothermal apparatus (Model No.: 9200) and are uncorrected. High performance liquid chromatography was performed with a Thermo Separation Products, P1500-SN-4000-UV2000 instrument using Daicel Chiralcel OD-H and OJ-H chiral columns. Column chromatography was performed on Silica Gel (60-Mesh, Merck Silica). TLC was performed on Merck 0.2-mm Silica Gel 60 F_{254} aluminum sheets. CAL-A (Lipase A, *C. antarctica*, recombinant from Aspergillus oryzae) and CAL-A-CLEA (Lipase A, *C. antarctica*, cross-linked enzyme aggregate) were purchased from Fluka, CRL (Lipase from *C. rugosa*, Type VII, ⩾700 unit/mg solid) was purchased from Sigma. Amano Lipase PS-C II (immobilized on ceramic) was purchased from Aldrich.

4.1. General procedure for the synthesis of (±)-1a–b

To a stirred solution of Mg turnings (15 mmol) and iodine (2 pieces) in dry diethyl ether (7 mL) at 25 °C equipped with a reflux condenser was added dropwise a mixture of iodomethane (11 mmol) in anhydrous diethyl ether (5 mL). The mixture was allowed to reflux for 30 min. The mixture was cooled down to 0 °C and then corresponding ketone (1-indanone or α-tetralone) (10 mmol) in dry diethylether (3 mL) was added dropwise. The resultant mixture was stirred for 2 h. The reaction mixture was hydrolyzed with saturated ammonium chloride solution (10 mL) and then with 1 M HCl (2 mL). The resultant mixture was extracted with diethylether (3 × 30 mL). The combined organic phase was washed with brine (20 mL) and dried over $MgSO_4$ and evaporated in vacuo. The crude products were purified by flash column chromatography with a mixture of solvents of ethyl acetate and hexane at a ratio of 1:8 for **1a** and 1:7 for **1b**.

Table 1
The results of enzymatic resolution of (±)-1-methyl-1,2,3,4-tetrahydronaphthalen-1-ol **1b**

Enzyme	Time (h)	Conversion[a]	ee_p[b] (%)	ee_s (%)	E[d]	$[\alpha]_D^{26}$ (prod.)/abs. conf.	$[\alpha]_D^{26}$ (subs.)/abs. conf.
CAL-A-CLEA	48	25	91	38	29	$[\alpha]_D^{26} = -5.1$ (*c* 0.60, $CHCl_3$) (*R*)	$[\alpha]_D^{26} = +7.2$ (*c* 1, $CHCl_3$) (*S*)
CAL-A	144	20	99	20	253	$[\alpha]_D^{26} = -14.3$ (*c* 0.60, $CHCl_3$) (*R*)	$[\alpha]_D^{26} = +4.5$ (*c* 1, $CHCl_3$) (*S*)
Amano PS-C II	96	15	80	15	10	$[\alpha]_D^{26} = -4.0$ (*c* 1.30, $CHCl_3$) (*R*)	$[\alpha]_D^{26} = +2.1$ (*c* 1, $CHCl_3$) (*S*)
CRL	145	10	90	—[c]	21	$[\alpha]_D^{26} = -4.5$ (*c* 0.25, $CHCl_3$) (*R*)	—[c]

[a] Conversions were determined using HPLC analysis.
[b] Enantiomeric excesses were determined by Daicel Chiralcel OD-H and OJ-H column HPLC analysis.
[c] Isolated as a racemate.
[d] *E* values were calculated according to the literature.[9]

Table 2
The results of enzymatic resolution of (±)-1-methyl-2,3-dihydro-1*H*-inden-1-ol **1a**

Enzyme	Time (h)	Conversion[a]	ee_p[b] (%)	ee_s (%)	E[d]	$[\alpha]_D^{26}$ (prod.)/abs. conf.	$[\alpha]_D^{26}$ (subs.)/abs. conf.
CAL-A-CLEA	72	20	71	45	7	$[\alpha]_D^{26} = -5.0$ (*c* 0.25, $CHCl_3$) (*R*)	$[\alpha]_D^{26} = +7.9$ (*c* 1, $CHCl_3$) (*S*)
Amano PS-C II	140	7	55	—[c]	4	$[\alpha]_D^{26} = +4.6$ (*c* 0.25, $CHCl_3$) (*S*)	—[c]
CRL	168	5	15	—[c]	1.4	$[\alpha]_D^{26} = -1.1$ (*c* 0.25, $CHCl_3$) (*R*)	—[c]

[a] Conversions were determined using HPLC analysis.
[b] Enantiomeric excesses were determined by Daicel Chiralcel OD-H and OJ-H column by HPLC analysis.
[c] Isolated as a racemate.
[d] *E* values were calculated according to the literature.[9]

4.1.1. (±)-1-Methyl-2,3-dihydro-1*H*-inden-1-ol 1a

Yellow solid, mp 52–55 °C, 1.39 g, 83% yield. IR ν_{max} (neat, cm^{-1}): 3327, 2964, 2378, 1485. ^1H NMR (400 MHz, $CDCl_3$): δ 7.34–7.37 (m, 1H), 7.23–7.26 (m, 3H), 2.99–3.07 (m, 1H), 2.80–2.87 (m, 1H), 2.14–2.27 (m, 2H), 1.78 (br s, 1H) 1.57 (s, 3H); ^{13}C NMR (100.6 MHz, $CDCl_3$): δ 148.3, 142.6, 128.2, 126.9, 125.0, 122.2, 81.3, 42.4, 29.4, 27.3. Anal. Calcd for $C_{10}H_{12}O$: C, 81.04; H, 8.16. Found: C, 80.82; H, 8.15.

4.1.2. (±)-1-Methyl-1,2,3,4-tetrahydronaphthalen-1-ol 1b

White solid, mp 68–70 °C, [lit.[12] mp 69 °C], (1.45 g, 87%) IR ν_{max} (neat, cm^{-1}): 3323, 2931, 2314, 1441, 1310. ^1H NMR (400 MHz, $CDCl_3$): δ 7.59 (dd, J = 1.6 and 7.6 Hz, 1H), 7.14–7.25 (m, 2H), 7.07 (d, J = 7.2 Hz, 1H), 2.72–2.86 (m, 2H), 1.90–1.98 (m, 3H), 1.77–1.88 (m, 1H), 1.75 (br s, 1H), 1.56 (s, 3H); ^{13}C NMR (100.6 MHz, $CDCl_3$): δ 142.9, 136.3, 128.8, 127.1, 126.4, 126.3, 70.6, 39.8, 30.7, 29.9, 20.4. Anal. Calcd for $C_{11}H_{14}O$: C, 81.44; H, 8.70. Found: C, 81.29; H, 8.65.

4.2. General procedure for the acetylation of (±)-1a–b

A 35% KH (4 mmol) suspension was washed with dry hexane three times, then 5 mL of dry THF was added. Compounds (±)-**1a**–**b** (2 mmol) were dissolved in 5 mL of dry diethyl ether and then put into the reaction mixture. After 2 h, the dropwise addition of acetic anhydride (4 mmol) with tetrabutylammonium iodide (0.2 mmol) was performed. The reaction mixture was stirred for an additional 2 h, then filtered by washing with CH_2Cl_2, dried over $MgSO_4$, and finally evaporated in vacuo. The crude products were purified by flash column chromatography with a mixture of solvents of ethyl acetate and hexane at a ratio of 1:8 for **2a** and 1:7 for **2b**.[15]

4.2.1. (±)-1-Methyl-2,3-dihydro-1*H*-inden-1-yl acetate 2a

Yellow oil, (0.71 g, 55%) IR ν_{max} (neat, cm^{-1}): 2957, 2860, 1746, 1452, 1238. ^1H NMR (400 MHz, $CDCl_3$): δ 7.35–7.37 (m, 1H), 7.16–7.25 (m, 3H), 2.95–3.02 (m, 1H), 2.74–2.81 (m, 1H), 2.45–2.52 (m, 1H), 2.26–2.33 (m, 1H), 2.01 (s, 3H), 1.70 (s, 3H); ^{13}C NMR (100.6 MHz, $CDCl_3$): δ 170.8, 145.4, 143.5, 128.4, 126.0, 124.7, 123.9, 90.2, 37.9, 30.2, 25.1, 22.5. Anal. Calcd for $C_{12}H_{14}O_2$: C, 75.76; H, 7.42. Found: C, 75.58; H, 7.35.

4.2.2. (±)-1-Methyl-1,2,3,4-tetrahydronaphthalen-1-yl acetate 2b[16]

Yellow oil, (0.77 g, 61%) IR ν_{max} (neat, cm^{-1}): 2924, 2850, 2384, 1730, 1475, 1435, 1236. ^1H NMR (400 MHz, $CDCl_3$): δ 7.37–7.39 (m, 1H), 7.15–7.18 (m, 2H), 7.06–7.08 (m, 1H), 2.84–2.92 (m, 1H), 2.73 (td, J = 4.8 and 16.4 Hz, 1H), 2.55 (dt, J = 3.2 and 12.0 Hz, 1H), 2.09–2.15 (m, 1H), 2.00 (s, 3H), 1.93–1.99 (m, 1H), 1.75–1.80 (m, 1H), 1.74 (s, 3H); ^{13}C NMR (100.6 MHz, $CDCl_3$): δ 169.9, 140.3, 136.5, 128.7, 127.0, 126.1, 125.9, 81.4, 34.5, 29.7, 29.6, 22.5, 20.8. Anal. Calcd for $C_{13}H_{16}O_2$: C, 76.44; H, 7.90. Found: C, 76.32; H, 7.85.

4.3. General procedure for the transesterification reaction of (±)-1a–b

To 100 mg of substrate (±)-**1a**–**b** and 1 mL (16 mmol equiv) of vinyl acetate, the corresponding enzyme (i.e., 25 mg of CAL-A, 50 mg of CAL-A-CLEA, 100 mg of Amano PSC-II, 100 mg of CRL) was added, followed by shaking at 32 °C. The mixture was then monitored by TLC. When the reaction was completed, the mixture was filtered and the filtrate concentrated in vacuo. Purification was done by flash column chromatography using ethyl acetate/hexane as an eluent.

4.3.1. (*S*)-(+)-1-Methyl-2,3-dihydro-1*H*-inden-1-ol, (*S*)-(+)-1a

Yellow solid, 52 mg, (52% yield), $[\alpha]_D^{26} = +7.9$ (*c* 1, $CHCl_3$), 45% ee. The enantiomeric excess of the product was determined by HPLC analysis (Daicel Chiralcel OD-H, *n*-hexane/2-propanol 98:2, flow rate 0.5 mL/min, λ = 214 nm, $t_{1(S)}$ = 19.8 min, $t_{2(R)}$ = 23.8 min).

4.3.2. (*S*)-(+)-1-Methyl-1,2,3,4-tetrahydronaphthalen-1-ol, (*S*)-(+)-1b

White solid, 50 mg, (50% yield), $[\alpha]_D^{26} = +7.2$ (*c* 1, $CHCl_3$), 38% ee. The enantiomeric excess of the chiral product was determined by HPLC analysis (Daicel Chiralcel OJ-H, *n*-hexane/2-propanol 98:2, flow rate 0.5 mL/min, λ = 214 nm, $t_{1(R)}$ = 18.2 min, $t_{2(S)}$ = 21.6 min).

4.3.3. (*R*)-(−)-1-Methyl-2,3-dihydro-1*H*-inden-1-yl acetate, (*R*)-(−)-2a

Yellow oil, 17 mg, (13% yield), $[\alpha]_D^{26} = -5.0$ (*c* 0.25, $CHCl_3$), 45% ee. The enantiomeric excess of the product was determined by HPLC analysis (Daicel Chiralcel OD-H, *n*-hexane/2-propanol 98:2, flow rate 0.5 mL/min, λ = 214 nm, $t_{1(R)}$ = 9.5 min, $t_{2(S)}$ = 10.9 min).

4.3.4. (*R*)-(−)-1-Methyl-1,2,3,4-tetrahydronaphthalen-1-yl acetate, (*R*)-(−)-2b

Yellow oil, 20 mg, (16% yield), $[\alpha]_D^{26} = -14.3$ (*c* 0.6, $CHCl_3$), 99% ee. The enantiomeric excess of the product was determined by HPLC analysis (Daicel Chiralcel OD-H, *n*-hexane/2-propanol 98:2, flow rate 0.5 mL/min, λ = 214 nm, $t_{1(S)}$ = 10.0 min, $t_{2(R)}$ = 10.6 min).

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