Enantioselective Resolution of (R,S)-1-Phenylethanol Catalyzed by Lipases Immobilized in Starch Films

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Characterization by optical microscopy of grains extracted from ginger starch

Chemical synthesis of racemic (R,S)-1-phenylethyl acetate (3)

The racemic (R,S)-1-phenylethyl acetate was prepared by chemical acetylation of the precursor alcohol (R,S)-1 (4.9 mL, 4.1 mmol) employing acetic anhydride (19.4 mL, 20.5 mmol) in dichloromethane (30 mL) and acetic acid as the catalyst, as described in the literature. A yellow oil was obtained in 70% yield after purification by column chromatography on silica gel using a mixture of n-hexane and ethyl acetate (9:1 v:v) as the eluent. This compound was used as a standard in the chiral gas chromatography analysis and was analyzed by $^1$H NMR, IR, chiral GC and specific rotation. $^1$H NMR (CDCl$_3$, 400 MHz) δ 7.23 (m, 5H), 5.82 (q, 1H, J 6.8), 2.10 (s, 3H), 1.50 (d, 3H, J 6.8 Hz); IR (KBr) ν$_{max}$/cm$^{-1}$: 3064-2872, 1734, 1242; chiral GC tR/min: 5.0 and 5.4; [α]$_D$ 0.00 (2.56 × 10$^{-2}$ CHCl$_3$).

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Figure S2. $^1$H NMR spectrum of (R,S)-3 (CDCl$_3$, 400 MHz).

Figure S3. IR spectrum of compound (R,S)-3 (film).
Enzymatic resolution of (R,S)-I

Figure S4. Chromatogram of the racemic mixture of (R,S)-3,(S)-3 t_R 5.0 and (R)-3 t_R 5.4 min.

Figure S5. Chromatogram of (R,S)-1-phenylethanol resolution catalyzed by LBC immobilized in ginger starch film. (R)-3 t_R 5.4 min. (R)-1 t_R 5.8 min, (S)-1 t_R 6.1 min.
Spectral data of (R)-1-phenylethyl acetate (3)

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\text{(R)-3} \\
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\(^1\)H NMR (CDCl\textsubscript{3}, 400 MHz) \(\delta\) 7.23 (m, 5H), 5.82 (q, 1H, \(J\) 6.8), 2.10 (s, 3H), 1.50 (d, 3H, \(J\) 6.8 Hz); IR (KBr) \(\nu_{\text{max}}/\text{cm}^{-1}\): 3064-2872, 1734, 1242; chiral GC \(t_{R}\)/min: 5.4; \([\alpha]_D^{10} +36\) (0.03 CHCl\textsubscript{3}), \([\alpha]_D^{10} +43\) (2.1 CHCl\textsubscript{3}).

References