Supporting Information

N-Heterocyclic Carbene Catalyzed Monoacylation of Vicinal Diols

Satoru Kuwano*, and Toshinobu Masuda
Typical Procedure for NHC-Catalyzed Mono-selective Acylation of Vicinal Diols.

A solution of 1a (3.4 mg, 0.015 mmol, 0.05 equiv), 2 (0.3 mmol), 4-Me₂NC₆H₄CO₂H (4.9 mg, 0.03 mmol, 0.1 equiv), 1,8-bis(dimethylamino)naphthalene (96 mg, 0.45 mmol, 1.5 equiv), and 2-bromo-3-phenylpropanal (95 mg, 0.45 mmol, 1.5 equiv) in CHCl₃ (3 mL) was stirred for 5 h under argon atmosphere at ambient temperature. The mixture was concentrated in vacuo to give crude reaction mixture. Yields of 2, 3, and 4 were determined by ¹H NMR analysis of the mixture using triphenylmethane as internal standard. To the mixture, EtOAc and 3% HCl solution were added. The aqueous layer was extracted with EtOAc. The combined organic layers were washed with aq. NaHCO₃ and brine, dried over Na₂SO₄, and concentrated in vacuo. The residue was purified by silica gel chromatography (hexane/EtOAc 10:1 to 2:1) to afford mono-acylated product 4.

(±)-(1R,2R)-2-hydroxycyclopentyl 3-phenylpropanoate (4a)

![Structure](image)

Colorless oil. 94% Yield. ¹H and ¹³C NMR were identical to those reported in the literature.¹

HRMS–EI (m/z): (M⁺) calcd for C₁₄H₁₈O₃, 234.1256; found, 234.1254.

(±)-(1R,2R)-2-hydroxycyclohexyl 3-phenylpropanoate (4b)

![Structure](image)

Colorless oil. 93% Yield. ¹H and ¹³C NMR were identical to those reported in the literature.¹

HRMS–EI (m/z): (M⁺) calcd for C₁₅H₂₀O₃, 248.1412; found, 248.1422.

(±)-(1R,2R)-2-hydroxycycloheptyl 3-phenylpropanoate (4c)

![Structure](image)

Colorless oil. 84% Yield. ¹H and ¹³C NMR were identical to those reported in the literature.¹

HRMS–EI (m/z): (M⁺) calcd for C₁₆H₂₂O₃, 262.1569; found, 262.1576.

(±)-(1R,2R)-2-hydroxycyclooctyl 3-phenylpropanoate (4d)

![Structure](image)

Colorless oil. 96% Yield. $^1$H and $^{13}$C NMR were identical to those reported in the literature. $^1$HRMS–EI (m/z): (M$^+$) calcd for C$_{17}$H$_{24}$O$_3$, 276.1725; found, 276.1719.

(±)-(1R,2S)-2-hydroxycyclopentyl 3-phenylpropanoate (4e)

![Structure](image)

Colorless oil. 81% Yield. $^1$H NMR (500 MHz, CDCl$_3$) $\delta$ 1.48–1.97 (m, 7H), 2.69 (t, $J = 8.0$, 2H), 2.97 (t, $J = 8.0$, 2H), 4.10 (s, 1H), 4.93–4.96 (m, 1H), 7.17–7.31 (m, 5H) ppm; $^{13}$C NMR (125 MHz, CDCl$_3$) $\delta$ 19.3, 27.9, 30.6, 30.9, 35.8, 72.9, 76.8, 126.3, 128.2, 128.5, 140.2, 172.7 ppm; HRMS–EI (m/z): (M$^+$) calcd for C$_{14}$H$_{18}$O$_3$, 234.1256; found, 234.1245.

(±)-(1R,2S)-2-hydroxycyclohexyl 3-phenylpropanoate (4f)

![Structure](image)

Colorless oil. 81% Yield. $^1$H NMR (500 MHz, CDCl$_3$) $\delta$ 1.31–1.36 (m, 2H), 1.52–1.81 (m, 7H), 2.69 (t, $J = 7.5$, 2H), 2.97 (t, $J = 7.5$, 2H), 3.77 (s, 1H), 4.90 (d, $J = 8.0$, 1H), 7.20–7.31 (m, 5H) ppm; $^{13}$C NMR (125 MHz, CDCl$_3$) $\delta$ 20.9, 21.9, 26.8, 30.2, 31.0, 36.0, 69.0, 74.2, 126.3, 128.2, 128.5, 140.3, 172.5 ppm; HRMS–EI (m/z): (M$^+$) calcd for C$_{15}$H$_{20}$O$_3$, 248.1412; found, 248.1401.

(±)-(1R,2S)-2-hydroxycycloheptyl 3-phenylpropanoate (4g)

![Structure](image)

Colorless oil. 83% Yield. $^1$H NMR (500 MHz, CDCl$_3$) $\delta$ 1.25–1.72 (m, 10H), 1.86–1.92 (m, 1H), 2.69 (t, $J = 7.5$, 2H), 2.98 (t, $J = 7.5$, 2H), 3.87 (s, 1H), 4.92–4.95 (m, 1H), 7.20–7.30 (m, 5H) ppm; $^{13}$C NMR (125 MHz, CDCl$_3$) $\delta$ 21.7, 22.6, 27.0, 27.6, 31.0, 31.4, 36.0, 72.2, 77.9, 126.3, 128.2, 128.5, 140.2, 172.5 ppm; HRMS–EI (m/z): (M$^+$) calcd for C$_{16}$H$_{22}$O$_3$, 262.1569; found, 262.1578.
(±)-(1R,2S)-2-hydroxycyclooctyl 3-phenylpropanoate (4h)

Colorless oil. 87% Yield. $^1$H NMR (500 MHz, CDCl$_3$) δ 1.49–1.81 (m, 11H), 1.97–2.04 (m, 2H), 2.67 (t, $J$ = 7.5, 2H), 2.96 (t, $J$ = 7.5, 2H), 3.85 (s, 1H), 5.02 (d, $J$ = 9.5), 7.19–7.30 (m, 5H) ppm; $^{13}$C NMR (125 MHz, CDCl$_3$) δ 21.7, 24.4, 25.3, 26.9, 27.5, 30.1, 31.0, 35.9, 71.2, 76.9, 126.3, 128.2, 128.5, 140.2, 172.3 ppm; HRMS-ESI (m/z): [M+Na]$^+$ calcd for C$_{17}$H$_{24}$O$_3$, 276.1725; found, 276.1719.

(±)-(1S,2S)-2-hydroxy-1,2-diphenylethyl 3-phenylpropanoate (4i)

White solids of mp 133–134 °C. 89% Yield. $^1$H NMR (500 MHz, CDCl$_3$) δ 2.49 (s, 1H), 2.68–2.71 (m, 2H), 2.92 (t, $J$ = 7.5, 2H), 4.84 (d, $J$ = 7.5, 1H), 5.82 (d, $J$ = 7.5, 1H), 7.04–7.28 (m, 15H) ppm; $^{13}$C NMR (125 MHz, CDCl$_3$) δ 30.7, 35.8, 76.2, 78.9, 126.2, 126.9, 127.6, 127.97, 128.01, 128.10, 128.12, 128.13, 128.3, 136.3, 139.5, 140.2, 171.6 ppm; HRMS-ESI (m/z): [M+Na]$^+$ calcd for C$_{23}$H$_{22}$NaO$_3$, 369.1461; found, 369.1461.

(±)-(1R,2S)-2-hydroxy-1,2-diphenylethyl 3-phenylpropanoate (4j)

White solids of mp 151–153 °C. 85% Yield. $^1$H NMR (500 MHz, CDCl$_3$) δ 2.19 (d, $J$ = 11.5, 1H), 2.56 (t, $J$ = 7.5, 2H), 2.80 (t, $J$ = 7.5, 2H), 4.84–4.90 (m, 1H), 5.88 (d, $J$ = 5.0, 1H), 7.08–7.26 (m, 15H) ppm; $^{13}$C NMR (125 MHz, CDCl$_3$) δ 30.7, 35.8, 76.2, 78.9, 126.2, 126.9, 127.6, 127.97, 128.01, 128.10, 128.13, 128.3, 136.3, 139.5, 140.2, 171.6 ppm; HRMS-ESI (m/z): [M+Na]$^+$ calcd for C$_{23}$H$_{23}$O$_3$, 346.1569; found, 346.1577.

(±)-(3S,4S)-4-hydroxyhexan-3-yl 3-phenylpropanoate (4k)

Colorless oil. 90% Yield. $^1$H NMR (500 MHz, CDCl$_3$) δ 0.85 (t, $J$ = 7.5, 3H), 0.92 (t, $J$ = 7.5, 3H), 1.26–1.42 (m, 2H), 1.57–1.68 (m, 2H), 2.03 (s, 1H), 2.69 (t, $J$ = 7.5, 2H), 2.97 (t, $J$ = 7.5, 2H), 3.45–3.48 (m, 1H), 4.76–4.79 (m, 1H), 7.19–7.29 (m, 5H) ppm; $^{13}$C NMR (125 MHz, CDCl$_3$) δ 9.7, 9.9,
23.6, 26.4, 30.9, 35.8, 73.4, 77.5, 126.2, 128.2, 128.4, 140.3, 172.8 ppm; HRMS–EI (m/z): (M') calcd for C_{15}H_{22}O_{3}, 250.1569; found, 250.1571.

(±)-(3R,4S)-4-hydroxyhexan-3-yl 3-phenylpropanoate (4l)

![Chemical structure](image)

Colorless oil. 93% Yield. \(^1\)H NMR (500 MHz, CDCl\(_3\)) \(\delta\) 0.84 (t, \(J = 7.5, 3\)H), 0.95 (t, \(J = 7.5, 3\)H), 1.29–1.35 (m, 1H), 1.41–1.46 (m, 1H), 1.57–1.62 (m, 2H), 1.94 (s, 1H), 2.68 (t, \(J = 7.5, 2\)H), 2.96 (t, \(J = 7.5, 2\)H), 3.46–3.53 (m, 1H), 4.77–4.80 (m, 1H), 7.20–7.30 (m, 5H) ppm; \(^{13}\)C NMR (125 MHz, CDCl\(_3\)) \(\delta\) 9.9, 10.2, 21.8, 25.0, 31.0, 35.9, 74.2, 78.7, 126.3, 128.2, 128.4, 140.2, 173.0 ppm; HRMS–EI (m/z): (M') calcd for C_{15}H_{22}O_{3}, 250.1569; found, 250.1571.

(±)-(1R,3R)-3-hydroxy-1,3-diphenylpropyl 3-phenylpropanoate (4m)

![Chemical structure](image)

Colorless oil. 87% Yield. \(^1\)H NMR (500 MHz, CDCl\(_3\)) \(\delta\) 2.05–2.21 (m, 2H), 2.63 (s, 1H), 2.70 (t, \(J = 8.0, 2\)H), 2.96 (t, \(J = 8.0, 2\)H), 4.48–4.50 (m, 1H), 6.05 (dd, \(J = 10.5, 3.0, 1\)H), 7.17–7.32 (m, 15H) ppm; \(^{13}\)C NMR (125 MHz, CDCl\(_3\)) \(\delta\) 9.9, 10.2, 21.8, 25.0, 31.0, 35.9, 74.2, 78.7, 126.3, 128.2, 128.4, 128.50, 128.52, 140.2, 140.3, 143.7, 172.9 ppm; HRMS–EI (m/z): (M') calcd for C_{24}H_{24}O_{3}, 360.1725; found, 360.1750.

(±)-(1R,3S)-3-hydroxy-1,3-diphenylpropyl 3-phenylpropanoate (4n)

![Chemical structure](image)

Colorless oil. 68% Yield. \(^1\)H NMR (500 MHz, CDCl\(_3\)) \(\delta\) 2.07–2.12 (m, 2H), 2.40–2.46 (m, 1H), 2.60 (t, \(J = 8.0, 2\)H), 2.90 (t, \(J = 8.0, 2\)H), 4.53 (br s, 1H), 5.85 (t, \(J = 7.0, 1\)H), 7.15–7.32 (m, 15H) ppm; \(^{13}\)C NMR (125 MHz, CDCl\(_3\)) \(\delta\) 30.8, 35.9, 45.2, 71.5, 74.1, 125.7, 126.2, 126.6, 127.8, 128.1, 128.3, 128.4, 128.53, 128.54, 140.0, 140.4, 143.8, 171.9 ppm; HRMS–EI (m/z): (M') calcd for C_{24}H_{24}O_{3}, 360.1725; found, 360.1735.

(±)-(2R,4S)-4-hydroxypentan-2-yl 3-phenylpropanoate (4o)

![Chemical structure](image)
Colorless oil. 92% Yield. $^1H$ NMR (500 MHz, CDCl$_3$) $\delta$ 1.10 (d, $J = 6.5$, 3H), 1.21 (d, $J = 6.5$, 3H), 1.49–1.58 (m, 2H), 2.65 (t, $J = 7.5$, 2H), 2.79 (br s, 1H), 2.95 (t, $J = 7.5$, 2H), 5.13–5.15 (m, 1H), 7.20–7.30 (m, 5H) ppm; $^{13}C$ NMR (125 MHz, CDCl$_3$) $\delta$ 20.6, 22.9, 30.9, 36.0, 45.9, 63.3, 68.3, 126.2, 128.3, 128.4, 140.2, 173.6 ppm; HRMS–ESI ($m/z$): [M+Na]$^+$ calcd for C$_{14}$H$_{20}$NaO$_3$, 259.1305; found, 259.1300.

$(\pm$)-(2R,5R)-5-hydroxyhexan-2-yl 3-phenylpropanoate (4p)

$(\pm)$-5-hydroxyhexan-2-yl 3-phenylpropanoate (4p)

Colorless oil. 61% Yield. $^1H$ NMR (500 MHz, CDCl$_3$) $\delta$ 1.16 (d, $J = 6.5$, 3H), 1.19 (d, $J = 6.5$, 3H), 1.35–1.40 (m, 2H), 1.48–1.55 (m, 1H), 1.62–1.69 (m, 2H), 2.08 (br s, 1H), 2.61 (t, $J = 7.5$, 2H), 2.94 (t, $J = 7.5$, 2H), 3.72–3.78 (m, 1H), 4.89–4.95 (m, 1H), 7.19–7.29 (m, 5H) ppm; $^{13}C$ NMR (125 MHz, CDCl$_3$) $\delta$ 19.9, 23.4, 31.0, 31.9, 34.6, 36.1, 67.6, 70.8, 126.2, 128.3, 128.4, 140.5, 172.6 ppm; HRMS–ESI ($m/z$): [M+Na]$^+$ calcd for C$_{15}$H$_{22}$NaO$_3$, 273.1461; found, 273.1461.
Typical Procedure for DMAP-Catalyzed Acylation of Vicinal Diols.

A solution of DMAP (3.1 mg, 0.025 mmol, 0.5 equiv), 2 (0.5 mmol), pyridine (0.06 mL, 0.75 mmol, 1.5 equiv), and hydrocinnamoyl chloride (74 μL, 0.5 mmol, 1.0 equiv) in CH₂Cl₂ (0.5 mL) was stirred for 1 h at room temperature. The mixture was concentrated *in vacuo* to give crude reaction mixture. Yields of 2, 3, and 4 were determined by ¹H NMR analysis of the mixture using triphenylmethane as internal standard. The mixture was purified by silica gel chromatography (hexane–EtOAc) to give 3 and 4.

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(±)-(1R,2R)-cyclopentane-1,2-diyl bis(3-phenylpropanoate) (3a)

Colorless oil. \( ^1H \text{ NMR} \) (500 MHz, CDCl\(_3\)) \( \delta \) 1.52–1.57 (m, 2H), 1.65–1.71 (m, 2H), 1.99–2.06 (m, 2H), 2.60 (t, \( J = 7.5, 4H \)), 2.93 (t, \( J = 7.5, 4H \)), 5.01–5.03 (m, 2H), 7.18–7.29 (m, 10H) ppm; \( ^{13}C \text{ NMR} \) (125 MHz, CDCl\(_3\)) \( \delta \) 21.4, 30.3, 30.9, 35.8, 78.9, 126.2, 128.2, 128.4, 140.3, 172.1 ppm; \( \text{HRMS–EI (m/z): (M}^+ \text{) calcd for C\(_{23}\)H\(_{26}\)O\(_4\), 366.1831; found, 366.1838.}

(±)-(1R,2R)-cyclohexane-1,2-diyl bis(3-phenylpropanoate) (3b)

Colorless oil. \( ^1H \text{ NMR} \) (500 MHz, CDCl\(_3\)) \( \delta \) 1.22–1.42 (m, 4H), 1.58–1.78 (m, 2H), 1.91–2.01 (m, 2H), 2.47–2.58 (m, 4H), 2.88 (t, \( J = 8.0, 4H \)), 4.79–4.81 (m, 2H), 7.14–7.27 (m, 10H) ppm; \( ^{13}C \text{ NMR} \) (125 MHz, CDCl\(_3\)) \( \delta \) 23.3, 30.0, 30.8, 35.8, 73.6, 126.2, 128.2, 128.4, 140.3, 172.2 ppm; \( \text{HRMS–EI (m/z): (M}^+ \text{) calcd for C\(_{24}\)H\(_{28}\)O\(_4\), 380.1988; found, 380.1985.}

(±)-(1R,2R)-cycloheptane-1,2-diyl bis(3-phenylpropanoate) (3c)

Colorless oil. \( ^1H \text{ NMR} \) (500 MHz, CDCl\(_3\)) \( \delta \) 1.58–1.78 (m, 10H), 2.48–2.55 (m, 4H), 2.89 (t, \( J = 7.5, 4H \)), 4.94–4.97 (m, 2H), 7.15–7.27 (m, 10H) ppm; \( ^{13}C \text{ NMR} \) (125 MHz, CDCl\(_3\)) \( \delta \) 22.7, 28.2, 30.3, 30.8, 35.9, 76.8, 126.2, 128.2, 128.4, 172.1 ppm; \( \text{HRMS–EI (m/z): (M}^+ \text{) calcd for C\(_{25}\)H\(_{30}\)O\(_4\), 394.2144; found, 394.2143.}

(±)-(1R,2R)-cyclooctane-1,2-diyl bis(3-phenylpropanoate) (3d)

Colorless oil. \( ^1H \text{ NMR} \) (500 MHz, CDCl\(_3\)) \( \delta \) 1.40–1.74 (m, 12H), 2.48–2.55 (m, 4H), 2.88 (t, \( J = 8.0, \))
4H), 5.07–5.08 (m, 2H), 7.15–7.27 (m, 10H) ppm; $^{13}$C NMR (125 MHz, CDCl$_3$) δ 24.1, 25.5, 28.9, 30.9, 35.9, 75.7, 126.2, 128.2, 128.4, 140.4, 172.2 ppm; HRMS–EI (m/z): (M$^+$) calcd for C$_{26}$H$_{32}$O$_4$, 408.2301; found, 408.2299.

(1R,2S)-cyclopentane-1,2-diyl bis(3-phenylpropanoate) (3e)

![Cyclopentane-1,2-diyl bis(3-phenylpropanoate) (3e)](image)

Colorless oil. $^1$H NMR (500 MHz, CDCl$_3$) δ 1.54–1.62 (m, 1H), 1.67–1.74 (m, 2H), 1.78–1.84 (m, 1H), 1.91–1.96 (m, 2H), 2.56 (t, $J$ = 8.0, 4H), 2.92 (t, $J$ = 8.0, 4H), 5.13–5.15 (m, 2H), 7.15–7.27 (m, 10H) ppm; $^{13}$C NMR (125 MHz, CDCl$_3$) δ 19.0, 28.1, 30.8, 35.7, 74.1, 126.2, 128.2, 128.4, 140.4, 172.2 ppm; HRMS–EI (m/z): (M$^+$) calcd for C$_{23}$H$_{26}$O$_4$, 366.1831; found, 366.1834.

(1R,2S)-cyclohexane-1,2-diyl bis(3-phenylpropanoate) (3f)

![Cyclohexane-1,2-diyl bis(3-phenylpropanoate) (3f)](image)

Colorless oil. $^1$H NMR (500 MHz, CDCl$_3$) δ 1.33–1.44 (m, 2H), 1.53–1.64 (m, 4H), 1.74–1.76 (m, 2H), 2.59 (t, $J$ = 8.0, 4H), 2.93 (t, $J$ = 8.0, 4H), 5.01–5.03 (m, 2H), 7.16–7.28 (m, 10H) ppm; $^{13}$C NMR (125 MHz, CDCl$_3$) δ 21.6, 27.6, 30.8, 35.8, 70.9, 126.1, 128.2, 128.4, 140.4, 172.1 ppm; HRMS–EI (m/z): (M$^+$) calcd for C$_{24}$H$_{28}$O$_4$, 380.1988; found, 380.2001.

(1R,2S)-cycloheptane-1,2-diyl bis(3-phenylpropanoate) (3g)

![Cycloheptane-1,2-diyl bis(3-phenylpropanoate) (3g)](image)

Colorless oil. $^1$H NMR (500 MHz, CDCl$_3$) δ 1.48–1.69 (m, 8H), 1.82–1.87 (m, 2H), 2.59–2.62 (m, 4H), 2.94 (t, $J$ = 7.5, 4H), 5.07–5.09 (m, 2H), 7.16–7.28 (m, 10H) ppm; $^{13}$C NMR (125 MHz, CDCl$_3$) δ 22.4, 26.5, 28.6, 30.8, 35.9, 74.1, 126.2, 128.2, 128.4, 172.0 ppm; HRMS–EI (m/z): (M$^+$) calcd for C$_{25}$H$_{30}$O$_4$, 394.2144; found, 394.2141.
(1R,2S)-cyclooctane-1,2-diyl bis(3-phenylpropanoate) (3h)

![Structure image]

Colorless oil. $^1$H NMR (500 MHz, CDCl$_3$) $\delta$ 1.56–1.66 (m, 10H), 1.88–1.94 (m, 2H), 2.54–2.64 (m, 4H), 2.93 (t, $J$ = 8.0, 4H), 5.14 (d, $J$ = 9.0, 2H), 7.17–7.28 (m, 10H) ppm; $^{13}$C NMR (125 MHz, CDCl$_3$) $\delta$ 22.8, 26.2, 28.3, 30.8, 35.8, 73.5, 126.1, 128.2, 128.4, 140.4, 172.1 ppm; HRMS–EI ($m/z$): ([M$^+$]) calcd for C$_{26}$H$_{32}$O$_4$, 408.2301; found, 408.2309.

(±)-(1S,2S)-1,2-diphenylethane-1,2-diyl bis(3-phenylpropanoate) (3i)

![Structure image]

White solids of mp 79–81 °C. $^1$H NMR (500 MHz, CDCl$_3$) $\delta$ 2.60 (t, $J$ = 7.5, 4H), 2.88 (t, $J$ = 7.5, 4H), 6.05 (s, 2H), 7.06–7.25 (m, 20H) ppm; $^{13}$C NMR (125 MHz, CDCl$_3$) $\delta$ 30.7, 35.7, 77.1, 126.2, 127.4, 128.1, 128.2, 128.3, 128.5, 136.0, 140.2, 171.6 ppm; HRMS–ESI ($m/z$): [M+Na]$^+$ calcd for C$_{32}$H$_{30}$NaO$_4$, 501.2036; found, 501.2036.

(1R,2S)-1,2-diphenylethane-1,2-diyl bis(3-phenylpropanoate) (3j)

![Structure image]

White solids of mp 180–182 °C. $^1$H NMR (500 MHz, CDCl$_3$) $\delta$ 2.57 (t, $J$ = 7.5, 4H), 2.82 (t, $J$ = 7.5, 4H), 6.09 (s, 2H), 7.09–7.27 (m, 20H) ppm; $^{13}$C NMR (125 MHz, CDCl$_3$) $\delta$ 30.9, 36.0, 76.8, 126.5, 127.8, 128.3, 128.4, 128.6, 128.7, 136.3, 140.5, 171.7 ppm; HRMS–ESI ($m/z$): [M+Na]$^+$ calcd for C$_{32}$H$_{30}$NaO$_4$, 501.2036; found, 501.2036.

(±)-(3S,4S)-hexane-3,4-diyl bis(3-phenylpropanoate) (3k)

![Structure image]
Colorless oil. $^1$H NMR (500 MHz, CDCl$_3$) $\delta$ 0.89 (t, $J = 7.5$, 6H), 1.42–1.47 (m, 4H), 2.62–2.67 (m, 4H), 2.94 (t, $J = 7.5$, 4H), 4.92–4.94 (m, 2H), 7.16–7.28 (m, 10H) ppm; $^{13}$C NMR (125 MHz, CDCl$_3$) $\delta$ 9.5, 23.7, 30.9, 35.7, 74.7, 126.2, 128.3, 128.4, 140.3, 172.4 ppm; HRMS –EI ($m/z$): (M$^+$) calcd for C$_{24}$H$_{30}$O$_4$, 382.2144; found, 382.2146.

(3R,4S)-hexane-3,4-diy l bis(3-phenylpropanoate) (3I)

Colorless oil. $^1$H NMR (500 MHz, CDCl$_3$) $\delta$ 0.82 (t, $J = 7.5$, 6H), 1.48–1.53 (m, 4H), 2.61 (t, $J = 7.5$, 4H), 2.94 (t, $J = 7.5$, 4H), 4.91–4.93 (m, 2H), 7.17–7.28 (m, 10H) ppm; $^{13}$C NMR (125 MHz, CDCl$_3$) $\delta$ 9.8, 22.5, 30.9, 35.7, 75.2, 126.2, 128.2, 128.4, 140.4, 172.5 ppm; HRMS –EI ($m/z$): (M$^+$) calcd for C$_{24}$H$_{30}$O$_4$, 382.2146; found, 382.2146.

(±)-(1R,3R)-1,3-diphenylpropane-1,3-diy l bis(3-phenylpropanoate) (3m)

Colorless oil. $^1$H NMR (500 MHz, CDCl$_3$) $\delta$ 2.31–2.34 (m, 2H), 2.61–2.64 (m, 4H), 2.89–2.92 (m, 4H), 4.81–4.88 (m, 2H), 7.16–7.31 (m, 20H) ppm; $^{13}$C NMR (125 MHz, CDCl$_3$) $\delta$ 30.8, 35.8, 42.9, 72.3, 126.2, 126.4, 128.1, 128.2, 128.5, 128.6, 139.9, 140.4, 172.0 ppm; HRMS –ESI ($m/z$): [M+Na]$^+$ calcd for C$_{33}$H$_{32}$NaO$_4$, 515.2193; found, 515.2191.

(1R,3S)-1,3-diphenylpropane-1,3-diy l bis(3-phenylpropanoate) (3n)

Colorless oil. $^1$H NMR (500 MHz, CDCl$_3$) $\delta$ 2.12–2.18 (m, 1H), 2.52–2.63 (m, 5H), 2.91 (t, $J = 7.5$, 4H), 5.61 (t, $J = 7.0$, 4H), 7.15–7.29 (m, 20H) ppm; $^{13}$C NMR (125 MHz, CDCl$_3$) $\delta$ 30.7, 35.8, 42.6, 73.0, 126.2, 126.4, 128.1, 128.3, 128.5, 128.6, 139.6, 140.3, 171.8 ppm; HRMS –ESI ($m/z$): [M+Na]$^+$ calcd for C$_{33}$H$_{32}$NaO$_4$, 515.2193; found, 515.2199.

(±)-(2S,4S)-pentane-2,4-diy l bis(3-phenylpropanoate) (3o)
Colorless oil. $^1$H NMR (500 MHz, CDCl$_3$) $\delta$ 1.17 (d, $J = 6.0$, 6H), 1.71 (t, $J = 6.5$, 2H), 2.55–2.63 (m, 4H), 2.93 (t, $J = 8.0$, 4H), 4.92–4.98 (m, 2H), 7.17–7.29 (m, 10H) ppm; $^{13}$C NMR (125 MHz, CDCl$_3$) $\delta$ 20.4, 30.9, 36.0, 42.2, 67.2, 126.1, 128.2, 128.4, 140.5, 172.3 ppm; HRMS–ESI ($m/z$): [M+Na]$^+$ calcd for C$_{23}$H$_{28}$NaO$_4$, 391.1880; found, 391.1880.

($\alpha$)-(2R,5R)-hexane-2,5-diyl bis(3-phenylpropanoate) (3p)

Colorless oil. $^1$H NMR (500 MHz, CDCl$_3$) $\delta$ 1.15 (d, $J = 6.5$, 6H), 1.38–1.51 (m, 4H), 2.60 (t, $J = 7.5$, 4H), 2.94 (t, $J = 7.5$, 4H), 4.82–4.91 (m, 2H), 7.17–7.29 (m, 10H) ppm; $^{13}$C NMR (125 MHz, CDCl$_3$) $\delta$ 19.9, 31.0, 31.5, 36.1, 70.5, 126.2, 128.3, 128.4, 140.5, 172.4 ppm; HRMS–ESI ($m/z$): [M+Na]$^+$ calcd for C$_{24}$H$_{30}$NaO$_4$, 405.2036; found, 405.2036.
$4e \ (^1H \text{NMR}, \ 500 \text{MHz}, \text{CDCl}_3)$
\( \text{4e (}^{13}\text{C NMR, 125 MHz, CDCl}_3) \)
$^{1}H$ NMR, 500 MHz, CDCl$_3$
$^{13}$C NMR, 125 MHz, CDCl$_3$
$4g \left({}^1H \text{ NMR, } 500 \text{ MHz, CDCl}_3\right)$
$^{13}$C NMR, 125 MHz, CDCl$_3$
$4h$ ($^1$H NMR, 500 MHz, CDCl$_3$)
$^{13}$C NMR, 125 MHz, CDCl$_3$
$^{1}$H NMR, 500 MHz, CDCl$_3$
$4i \left( ^{13}C \text{NMR, 125 MHz, CDCl}_3 \right)$
$4j$ ($^1$H NMR, 500 MHz, CDCl$_3$)
$^4j$ ($^{13}$C NMR, 125 MHz, CDCl$_3$)
$4k$ ($^1\text{H NMR, 500 MHz, CDCl}_3$)
4k (\textsuperscript{13}C NMR, 125 MHz, CDCl\textsubscript{3})
41 (¹H NMR, 500 MHz, CDCl₃)
$^{13}$C NMR, 125 MHz, CDCl$_3$
$^{1}H$ NMR, 500 MHz, CDCl$_3$
$4m$ ($^{13}$C NMR, 125 MHz, CDCl$_3$)
$4n$ ($^1$H NMR, 500 MHz, CDCl$_3$)
$4n$ ($^{13}$C NMR, 125 MHz, CDCl$_3$)
$40$ ($^1$H NMR, 500 MHz, CDCl$_3$)
$^{13}$C NMR, 125 MHz, CDCl$_3$
4p (\textsuperscript{1}H NMR, 500 MHz, CDCl\textsubscript{3})
$4p$ ($^{13}$C NMR, 125 MHz, CDCl$_3$)