Supplementary Information

Glycerol conversion to high-value chemicals: Implication of unnatural α-amino acids syntheses using natural resources

Yun Ji Park and Jung Woon Yang*

Department of Energy Science, Sungkyunkwan University, Suwon 16419, Republic of Korea

E-mail: jwyang@skku.edu, Tel: +82-31-299-4276

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General methods

Thin-layer chromatography (TLC) was performed on Merck silica gel 60 F254. $^1$H NMR spectra were recorded on a Varian at 500 MHz in CDCl$_3$ (δ 7.26 ppm) or DMSO-d$_6$ (δ 2.50 ppm) or CD$_3$OD-d$_4$ (δ 2.05 ppm). $^{13}$C NMR spectral measurements were performed at 126 MHz using CDCl$_3$ (δ 77.16 ppm) or DMSO-d$_6$ (δ 39.52 ppm) or CD$_3$OD-d$_4$ (δ 49.00 ppm), $^{19}$F NMR spectral measurements were performed with CDCl$_3$ or CD$_3$OD-d$_4$. The terms m, s, d, t, and q. represent multiplet, singlet, doublet, triplet, and quadruplet, respectively. Commercial grade reagents and solvents were used without further purification. Gas Chromatography High Resolution Mass Spectrometer (GC-HRMS) was obtained on an Agilent 6890 Series and a Bruker Compact mass spectrometer. Infrared (IR) spectra were recorded on a VERTEX 70(Bruker) IR spectrometer in the range of 1000-4000 cm$^{-1}$. HPLC analyses for the enantiomeric excess (ee) were performed with a Varian Pro Star Series HPLC System using chiral columns (Chiralpak AY-H; 250 × 4.6 mm). Melting Point were checked with a M-560(BUCHI). Optical rotation were recorded on PerkinElmer model 343 polarimeter.

General procedure for the synthesis of O-benzylglycerol from glycerol [Method A]

All compounds used in the synthesis of O-benzylglycerol were prepared following literature procedures.$^{[1-3]}$

A mixture of benzaldehyde (5.1 mL, 50 mmol), glycerol (8.06 g, 87.5 mmol), and $p$-
toluenesulfonic acid (0.4 g, 2.3 mmol) in THF (16.5 mL) were placed into a 100 mL flask. The reaction mixture was heated under reflux using a Dean Stark trap until no further water was produced. Thereafter, the reaction was further carried out at room temperature for 1 h. The reaction mixture was quenched with NaHCO$_3$ and diethyl ether. The organic layer was washed with brine, dried over Na$_2$SO$_4$, and concentrated in vacuo to give crude oil. The crude product was purified by column chromatography on silica gel (hexanes:ethyl acetate = 5:1 to 1:1) to give dioxane and dioxolane (S1; 5.5 g, 61 %) as opaque white gel.

NaH (60 % in mineral oil, 1.29 g, 32.2 mmol) was washed with pentane (10 mL × 3) in a 100 mL round bottom flask. Dry THF (10 mL) was added dropwise with argon before a solution of compound S1 (4.85 g, 26.8 mmol) in dry THF was added dropwise over 10 min at 0 °C. After 30 min at 0 °C, tetrabutylammonium iodide (0.1 g, 0.27 mmol) and a solution of benzyl bromide (3.5 mL, 29.5 mmol) in THF (20 mL) were added and the resulting mixture stirred for 3 h at room temperature. The reaction was quenched with cooled water and ethyl acetate. The organic layer was dried over Na$_2$SO$_4$ and concentrated in vacuo. The crude product was purified by column chromatography on silica gel (hexanes:ethyl acetate = 10:1 to 7:1) to give dioxane and dioxolane compound S2 and S3 as-obtained products.

To compound S2 (0.52 g, 1.92 mmol) in MeOH (10.3 mL) was added 5% HCl solution (1.7 mL). The reaction mixture was heated under reflux for 20 min. The reaction mixture was extracted with CH$_2$Cl$_2$ and the combined organic layers washed with water, dried over Na$_2$SO$_4$, and concentrated in vacuo to obtain the crude ester. The crude product was purified by flash chromatography on silica gel (hexanes:ethyl acetate = 3:1 to 1:1) to give 2-O-benzylglycerol 1 as-obtained product.

1-O-Benzylglycerol 1’a was synthesized by the above-mentioned standard procedure with compound S3.

**General procedure for the oxidation of O-benzylglycerol [Method B]**

O-Benzylglycerol (0.5 mmol) and sodium tert-butoxide (2.5 mmol) were added to a flame-dried round 10 mL bottom flask equipped with a magnetic stirring bar. The flask was evacuated in vacuo until the solvent began to bubble and then refilled with oxygen three times. The reaction mixture was stirred and heated at 60 °C for 18 h. 5% HCl solution was slowly added
to acidify (pH 1) the reaction mixture, which was then extracted with ethyl acetate (10 mL × 5). The combined organic layers were dried over Na$_2$SO$_4$, concentrated \textit{in vacuo}, and purified by column chromatography on silica gel (hexanes : ethyl acetate : TFA = 3:1:0.2) to give the corresponding benzoic acid and (benzyloxy)acetic acid.

\textbf{General procedure for the synthesis of aminopropanoate using chiral auxiliary [Method C]}

Each step was carried out according to a literature procedure except for the step using a chiral auxiliary. A 100 mL two-neck round bottom flask was purged with argon and charged of lithium diisopropylamine in heptane/THF/ethylbenzene (2.0 M, 10.2 mL, 20.43 mmol) at -78 °C. A solution of (S)-3-(2-(benzyloxy)acetyl)-4-isopropyloxazolidin-2-one (1.89 g, 6.81 mmol) in THF (16.5 mL) was added dropwise over 5 min to the flask under argon at -78 °C. After stirring for 30 min, benzyl bromide (8.1 mL, 68.1 mmol) in THF (24.7 mL) was added dropwise to the reaction mixture over 5 min at -78 °C and stirring was continued at -45 °C for 3 h. The reaction mixture was quenched with saturated NH$_4$Cl solution and ethyl acetate. The organic layer was washed with brine and NaHCO$_3$, dried over Na$_2$SO$_4$, and concentrated \textit{in vacuo} to give an oil. The crude product was purified by column chromatography on silica gel (hexanes:ethyl acetate = 3:1 to 1:1) to give aminopropanoate (1.84 g, 73.4 %) as transparent brown liquid.

\textbf{General procedure for the esterification of malonic acid [Method D]}

To di-carboxylic acid in MeOH was added carefully 3-4 drops of concentrated sulfuric acid. The reaction mixture was heated under reflux for 20 min. The reaction mixture was extracted in CH$_2$Cl$_2$. The combined organic layers were washed with water, dried over Na$_2$SO$_4$, and concentrated \textit{in vacuo} to give the corresponding ester. The crude product was purified by flash chromatography on silica gel (hexanes:ethyl acetate = 3:1) to give the ester as a white solid.

\textbf{General procedure for removal of oxazolidinone auxiliary [Method E]}

Compound 23 (1.84 g, 5 mmol) in 25 mL of THF-H$_2$O (v/v = 4:1) was placed into 100 mL flask equipped with a magnetic stirring bar. To this solution was added 28% H$_2$O$_2$ (1.7 mL, 20 mmol) over 5 min under argon atmosphere at 0 °C, followed by LiOH (0.24 g, 10 mmol) in 10 mL of water. After stirring at 0 °C for 1 h, Na$_2$SO$_3$ (0.67 g, 20 mmol) in 15 mL of water was added followed by 50 mL of 5% NaHCO$_3$. After removal of the THF on a rotary evaporation, the resulting mixture was extracted with CH$_2$Cl$_2$ (50 mL × 3) to remove the oxazolidinone.
auxiliary. The combined organic layers were dried over Na$_2$SO$_4$, concentrated in *vacuo* to give the oxazolidinone auxiliary as a white solid. This solid was recrystallized from ethyl acetate-hexanes (1:10) to give pure oxazolidinone auxiliary. 38% HCl (*aq*) was slowly added to acidify (pH 1) the aqueous layer, which was then extracted with ethyl acetate (100 mL × 4). The combined organic layers were dried over Na$_2$SO$_4$, concentrated in *vacuo* to give compound 24.
Characterisation data for the products

Yield of product 1A+1B = 75.2% (1A: 25.5%, 1B: 49.7%)

(2R,4R)-4-((Benzyloxy)methyl)-2-phenyl-1,3-dioxolane\(^4\): The title compound was synthesized according to general procedure A. Colorless oil; \(^1\)H NMR (500 MHz, CDCl\(_3\)) \(\delta\) 7.47 (ddd, \(J = 6.3, 3.7, 1.5\) Hz, 2H), 7.39–7.27 (m, 8H), 5.81 (s, 1H), 4.59 (d, \(J = 1.8\) Hz, 2H), 4.43 (dq, \(J = 6.9, 5.6\) Hz, 1H), 4.04 (ddd, \(J = 13.5, 8.2, 6.2\) Hz, 2H), 3.62 (ddd, \(J = 49.0, 9.8, 5.7\) Hz, 2H) ppm; \(^1\)C NMR (126 MHz, CDCl\(_3\)) \(\delta\) 137.91, 137.32, 129.37, 128.44, 128.33, 127.77, 126.68, 104.38, 75.41, 73.55, 70.92, 68.02 ppm.

(2S,4R)-4-((Benzyloxy)methyl)-2-phenyl-1,3-dioxolane\(^4\): The title compound was synthesized according to general procedure A. Colorless oil; \(^1\)H NMR (500 MHz, CDCl\(_3\)) \(\delta\) 7.47 (dt, \(J = 4.5, 2.5\) Hz, 2H), 7.40–7.28 (m, 8H), 5.94 (s, 1H), 4.61 (s, 2H), 4.45 (tt, \(J = 6.6, 5.2\) Hz, 1H), 4.23 (dd, \(J = 8.2, 6.7\) Hz, 1H), 3.86 (dd, \(J = 8.3, 6.7\) Hz, 1H), 3.64 (ddd, \(J = 29.9, 10.2, 5.2\) Hz, 2H) ppm; \(^1\)C NMR (126 MHz, CDCl\(_3\)) \(\delta\) 137.92, 137.91, 129.18, 128.46, 128.33, 127.79, 127.73, 126.44, 103.82, 76.06, 73.58, 70.45, 67.75 ppm.

(2R,5R)-5-(Benzyloxy)-2-phenyl-1,3-dioxane\(^3\): The title compound was synthesized according to general procedure A. White solid; \(^1\)H NMR (500 MHz, CDCl\(_3\)) \(\delta\) 7.50–7.28 (m, 10H), 5.40 (s, 1H), 4.60 (s, 2H), 4.36 (dd, \(J = 11.1, 4.9\) Hz, 2H), 3.80 (ddd, \(J = 15.0, 9.8, 4.9\) Hz, 1H), 3.70–3.61 (m, 2H) ppm; \(^1\)C NMR (126 MHz, CDCl\(_3\)) \(\delta\) 138.13, 138.11, 128.92, 128.47, 128.21, 128.06, 127.77, 126.05, 101.29, 71.86, 70.21, 67.92 ppm.

(2S,5R)-5-(Benzyloxy)-2-phenyl-1,3-dioxane\(^3\): The title compound was synthesized according to general procedure A. White solid; \(^1\)H NMR (500 MHz, CDCl\(_3\)) \(\delta\) 7.53 (ddd, \(J = 8.3, 2.8, 1.5\) Hz, 2H), 7.42–7.26 (m, 8H), 5.55 (s, 1H), 4.70 (s, 2H), 4.40–4.32 (m, 2H), 4.02 (dq, \(J = 12.6, 1.4\) Hz, 2H), 3.35–3.31 (m, 1H) ppm; \(^1\)C NMR (126 MHz, CDCl\(_3\)) \(\delta\) 138.13, 138.11, 128.92, 128.47, 128.21, 127.77, 127.72, 126.21, 101.42, 70.32, 69.22, 69.0 ppm.

2-(Benzyloxy)propane-1,3-diol (1a)\(^3\): The title compound was synthesized according to general procedure A. White solid; Yield = 75%; \(^1\)H NMR (500 MHz, CDCl\(_3\)) \(\delta\) 7.40–7.29 (m, 5H), 4.66 (s, 2H), 3.76 (ddd, \(J = 37.3, 11.7, 4.7\) Hz, 4H), 3.60 (p, \(J =
4.7 Hz, 1H), 2.04 (s, 2H) ppm; $^{13}$C NMR (126 MHz, CDCl$_3$) $\delta$ 137.99, 128.63, 128.05, 127.85, 79.19, 72.01, 62.38, 62.29 ppm.

3-(Benzyloxy)propane-1,2-diol (1'a)$^{[5]}$: The title compound was synthesized according to general procedure A. White solid; Yield = 60.2%; $^1$H NMR (500 MHz, CDCl$_3$) $\delta$ 7.40–7.28 (m, 5H), 4.55 (s, 2H), 3.89 (dq, $J$ = 9.9, 5.1 Hz, 1H), 3.73–3.60 (m, 2H), 3.56 (qd, $J$ = 9.7, 5.1 Hz, 2H), 2.74 (s, 1H), 2.26 (s, 1H) ppm; $^{13}$C NMR (126 MHz, CDCl$_3$) $\delta$ 137.64, 128.54, 127.96, 127.81, 73.63, 71.82, 70.61, 64.10 ppm.

Yield of product 2A+2B = 68.6% (2A: 24.6%, 2B: 44.0%)

$^{(2R,4R)}$-4-((Naphthalen-2-ylmethoxy)methyl)-2-phenyl-1,3-dioxolane: The title compound was synthesized according to general procedure A. Slightly yellow-colored semisolid; $^1$H NMR (500 MHz, CDCl$_3$) $\delta$ 7.85–7.76 (m, 4H), 7.51–7.44 (m, 5H), 7.38–7.32 (m, 3H), 5.81 (s, 1H), 4.74 (s, 2H), 4.45 (dq, $J$ = 11.2, 5.6 Hz, 1H), 4.13–4.07 (m, 1H), 3.98 (dd, $J$ = 8.2, 6.7 Hz, 1H), 3.67 (ddd, $J$ = 30.5, 10.2, 5.2 Hz, 2H) ppm; $^{13}$C NMR (126 MHz, CDCl$_3$) $\delta$ 137.30, 135.36, 133.25, 133.04, 129.18, 128.34, 128.30, 127.88, 127.72, 126.59, 126.45, 125.97, 125.70, 103.83, 75.10, 73.69, 70.44, 67.74 ppm; HRMS (EI+) m/z: [M]$^+$ calcd for C$_{21}$H$_{20}$O$_3$ 320.1412, found 320.1411; IR: 3053, 2868, 1601, 1506, 1458, 1394, 1344, 1267, 1218, 1095, 1063, 1021 cm$^{-1}$.

$^{(2S,4R)}$-4-((Naphthalen-2-ylmethoxy)methyl)-2-phenyl-1,3-dioxolane: The title compound was synthesized according to general procedure A. Slightly yellow-colored solid; $^1$H NMR (500 MHz, CDCl$_3$) $\delta$ 7.87–7.78 (m, 4H), 7.52–7.45 (m, 5H), 7.40–7.33 (m, 3H), 5.95 (s, 1H), 4.77 (s, 2H), 4.49–4.44 (m, 1H), 4.23 (dd, $J$ = 8.2, 6.7 Hz, 1H), 3.86 (dd, $J$ = 8.2, 6.7 Hz, 1H), 3.67 (ddd, $J$ = 30.5, 10.2, 5.2 Hz, 2H) ppm; $^{13}$C NMR (126 MHz, CDCl$_3$) $\delta$ 137.89, 135.37, 133.24, 133.05, 129.18, 128.34, 128.30, 127.88, 127.72, 126.59, 126.45, 125.97, 125.70, 103.83, 75.10, 73.69, 70.44, 67.74 ppm. HRMS (EI+) m/z: [M]$^+$ calcd for C$_{21}$H$_{20}$O$_3$ 320.1412, found 320.1411; IR: 3053, 2868, 1601, 1506, 1458, 1394, 1344, 1267, 1218, 1095, 1063, 1021 cm$^{-1}$.

$^{(2R,5R)}$-5-((Naphthalen-2-ylmethoxy)-2-phenyl-1,3-dioxane$^{[6]}$: The title compound was synthesized according to general procedure A.
yellow-colored solid; $^1$H NMR (500 MHz, CDCl$_3$) $\delta$ 7.82 (dd, $J = 8.8, 5.5$ Hz, 3H), 7.76 (s, 1H), 7.49–7.42 (m, 5H), 7.36–7.30 (m, 3H), 5.39 (s, 1H), 4.73 (s, 2H), 4.37 (dd, $J = 11.1, 4.9$ Hz, 2H), 3.83 (tt, $J = 9.8, 5.0$ Hz, 1H), 3.66 (dd, $J = 15.2, 6.2$ Hz, 2H) ppm; $^{13}$C NMR (126 MHz, CDCl$_3$) $\delta$ 137.63, 135.33, 133.24, 133.14, 128.98, 128.43, 128.28, 127.91, 127.72, 126.68, 126.28, 126.12, 126.04, 125.61, 101.30, 72.02, 70.24, 67.95 ppm.

(2S,5R)-5-(Naphthalen-2-ylmethoxy)-2-phenyl-1,3-dioxane$^{[6]}$: The title compound was synthesized according to general procedure A. Slightly yellow-colored solid; $^1$H NMR (500 MHz, CDCl$_3$) $\delta$ 7.83 (dd, $J = 10.9, 5.9$ Hz, 4H), 7.55 (dd, $J = 8.1, 1.5$ Hz, 3H), 7.50–7.44 (m, 2H), 7.40–7.31 (m, 3H), 5.57 (s, 1H), 4.87 (s, 2H), 4.40 (dd, $J = 12.5, 1.3$ Hz, 2H), 4.04 (dd, $J = 12.6, 1.6$ Hz, 2H), 3.40–3.36 (m, 1H) ppm; $^{13}$C NMR (126 MHz, CDCl$_3$) $\delta$ 138.13, 135.62, 133.25, 133.05, 128.92, 128.32, 128.22, 127.86, 127.73, 126.55, 126.22, 126.18, 125.94, 125.77, 101.42, 70.48, 69.21, 69.08 ppm.

2-(Naphthalen-2-ylmethoxy)propane-1,3-diol (1b): The title compound was synthesized according to general procedure A. White solid; Yield = 60.5%; $^1$H NMR (500 MHz, CDCl$_3$) $\delta$ 7.89–7.78 (m, 4H), 7.53–7.45 (m, 3H), 4.83 (s, 2H), 3.79 (ddd, $J = 39.0, 11.7, 4.6$ Hz, 4H), 3.68–3.64 (m, 1H), 1.93 (s, 2H) ppm; $^{13}$C NMR (126 MHz, CDCl$_3$) $\delta$ 135.42, 133.27, 133.10, 128.52, 127.90, 127.76, 126.71, 126.32, 126.14, 125.68, 79.15, 72.14, 62.49 ppm.

3-(Naphthalen-2-ylmethoxy)propane-1,2-diol (1’b): The title compound was synthesized according to general procedure A. White solid; Yield = 65.3%; $^1$H NMR (500 MHz, CDCl$_3$) $\delta$ 7.89–7.74 (m, 4H), 7.53–7.42 (m, 3H), 4.72 (s, 2H), 3.92 (d, $J = 3.5$ Hz, 1H), 3.76–3.56 (m, 4H), 2.62 (d, $J = 3.9$ Hz, 1H), 2.09 (s, 1H) ppm; $^{13}$C NMR (126 MHz, CDCl$_3$) $\delta$ 135.09, 133.23, 133.08, 128.40, 127.88, 127.73, 126.70, 126.26, 126.08, 125.65, 73.75, 71.85, 70.58, 64.13 ppm.

1-(Chloromethyl)-3-methoxybenzene: The title compound was synthesized according to reference$^{[7]}$. Brown-colored liquid; Yield = 99%; $^1$H NMR (500 MHz, CDCl$_3$) $\delta$ 7.31–7.24 (m, 1H), 7.00–6.92 (m, 2H), 6.89–6.83 (m, 1H), 4.56 (s, 2H), 3.82 (s, 3H) ppm; $^{13}$C NMR (126 MHz, CDCl$_3$) $\delta$ 159.81, 138.90, 129.77, 120.80, 114.10, 114.01, 55.28, 46.21 ppm.
Yield of product $3A+3B = 80\%$ ($3A$: 27.7\%, $3B$: 52.3\%)

4-(((3-Methoxybenzyl)oxy)methyl)-2-phenyl-1,3-dioxolane: The title compound was synthesized according to general procedure A. Colorless oil; No attempt was made to separate the diastereomers mixture; $^1$H NMR (500 MHz, CDCl$_3$) $\delta$ 7.48 (dd, $J = 5.3$, 2.0 Hz, 2H both diastereomers), 7.40–7.35 (m, 3H both diastereomers), 7.29–7.23 (m, 1H both diastereomers), 6.92 (t, $J = 7.6$ Hz, 2H both diastereomers), 6.86–6.82 (m, 1H both diastereomers), 4.48–4.39 (m, 1H both diastereomers), 3.72–3.54 (m, 2H both diastereomers) ppm; Cis: $\delta$ 5.95 (s, 1H), 4.59 (s, 2H), 4.24 (dd, $J = 8.2$, 6.7 Hz, 1H), 3.86 (dd, $J = 8.2$, 6.7 Hz, 1H), 3.81 (s, 3H) ppm; Trans: $\delta$ 5.81 (s, 1H), 4.57 (s, 2H), 4.14–4.07 (m, 1H), 3.98 (dd, $J = 8.2$, 5.3 Hz, 1H) ppm.

$^{13}$C NMR (126 MHz, CDCl$_3$) Cis: $\delta$ 159.79, 139.54, 137.89, 129.48, 129.18, 128.33, 126.44, 119.93, 113.34, 113.05, 103.82, 75.07, 73.42, 70.44, 67.73 ppm; Trans: $\delta$ 159.79, 139.56, 137.29, 129.46, 129.38, 128.33, 126.69, 119.97, 113.41, 113.02, 104.40, 75.43, 73.40, 70.89, 67.99 ppm; HRMS (EI+) $m/z$: [M]$^+$ calcd for C$_{18}$H$_{20}$O$_4$ 300.1362, found 300.1357; IR: 3041, 2873, 1596, 1486, 1458, 1396, 1265, 1219, 1155, 1096, 1052 cm$^{-1}$.

$(2R,5R)$-5-((3-Methoxybenzyl)oxy)-2-phenyl-1,3-dioxane:
The title compound was synthesized according to general procedure A. White solid; mp = 52.6–53.4 °C; $^1$H NMR (500 MHz, CDCl$_3$) $\delta$ 7.45 (dd, $J = 7.6$, 1.7 Hz, 2H), 7.38–7.32 (m, 3H), 7.27 (dd, $J = 12.8$, 4.9 Hz, 1H), 6.87 (dddd, $J = 11.7$, 10.5, 5.0 Hz, 3H), 5.40 (s, 1H), 4.58 (s, 2H), 4.39–4.33 (m, 2H), 3.84–3.76 (m, 4H), 3.69–3.62 (m, 2H) ppm; $^{13}$C NMR (126 MHz, CDCl$_3$) $\delta$ 159.82, 139.51, 137.64, 129.62, 128.99, 128.29, 126.04, 119.96, 113.50, 113.21, 101.29, 71.73, 70.21, 67.94, 55.24 ppm; HRMS (EI+) $m/z$: [M]$^+$ calcd for C$_{18}$H$_{20}$O$_4$ 300.1362, found 300.1362; IR: 3029, 2968, 2903, 1613, 1583, 1489, 1453, 1388, 1336, 1150, 1092, 1045, 1013 cm$^{-1}$.

$(2S,5R)$-5-((3-Methoxybenzyl)oxy)-2-phenyl-1,3-dioxane:
The title compound was synthesized according to general procedure A. White solid; mp = 77.1–78.0 °C; $^1$H NMR (500 MHz, CDCl$_3$) $\delta$ 7.54–7.49 (m, 2H), 7.36–7.20 (m, 4H), 6.99–6.91 (m, 2H), 6.80 (dd, $J = 8.2$, 2.3 Hz, 1H), 5.52 (s, 1H), 4.64 (s, 2H), 4.32 (dd, $J = 12.4$, 1.0 Hz, 2H), 3.98 (dd, $J = 12.5$, 1.4 Hz, 2H), 3.75 (s, 3H), 3.31–3.27 (m, 1H) ppm; $^{13}$C NMR (126 MHz, CDCl$_3$) $\delta$ 159.82, 139.84, 138.11, 129.45, 128.90, 128.19, 126.19, 119.90, 113.42, 112.97, 101.38, 70.20, 69.23, 69.03, 55.23 ppm; HRMS (EI+): $m/z$ calcd for C$_{18}$H$_{20}$O$_4$ ([M]$^+$): 300.1362; observed: 300.1362; IR: 3029, 2968, 2903, 1613, 1583, 1489, 1453, 1388, 1336, 1150, 1092, 1045, 1013 cm$^{-1}$.
2-((3-Methoxybenzyl)oxy)propane-1,3-diol (1c): The title compound was synthesized according to general procedure A. Colorless oil; Yield = 70.1%; $^1$H NMR (500 MHz, CDCl$_3$) δ 7.27 (dd, J = 10.7, 5.0 Hz, 1H), 6.96–6.89 (m, 2H), 6.85 (dd, J = 8.0, 2.3 Hz, 1H), 4.63 (s, 2H), 3.81 (s, 3H), 3.78 (dd, J = 11.7, 4.5 Hz, 2H), 3.71 (dd, J = 11.7, 4.9 Hz, 2H), 3.58 (p, J = 4.7 Hz, 1H), 2.27 (s, 2H) ppm; $^{13}$C NMR (126 MHz, CDCl$_3$) δ 159.83, 139.64, 129.67, 120.01, 113.39, 113.37, 79.21, 71.83, 62.31, 55.25 ppm; HRMS (EI+) m/z: [M]$^+$ calcd for C$_{11}$H$_{16}$O$_4$ 212.1049, found 212.1046; IR: 3390, 2936, 2880, 1594, 1490, 1345, 1315, 1265, 1158, 1111 cm$^{-1}$.

3-((3-Methoxybenzyl)oxy)propane-1,2-diol (1'c)$^{[8]}$: The title compound was synthesized according to general procedure A. Colorless oil; Yield = 65.5%; $^1$H NMR (500 MHz, CDCl$_3$) δ 7.27 (dd, J = 9.9, 5.7 Hz, 1H), 6.93–6.82 (m, 3H), 4.53 (s, 2H), 3.93–3.87 (m, 1H), 3.81 (s, 3H), 3.74–3.52 (m, 4H), 2.64 (s, 1H), 2.15 (s, 1H) ppm; $^{13}$C NMR (126 MHz, CDCl$_3$) δ 159.79, 139.31, 129.56, 119.99, 113.35, 113.24, 73.44, 71.78, 70.66, 64.05, 55.23 ppm.

4-Chlorobenzyl methanesulfonate: The title compound was synthesized according to reference $^{[9]}$. White solid; Yield = 95%; $^1$H NMR (500 MHz, CDCl$_3$) δ 7.42–7.34 (m, 4H), 5.21 (s, 2H), 2.95 (s, 3H) ppm; $^{13}$C NMR (126 MHz, CDCl$_3$) δ 135.48, 131.95, 130.19, 129.17, 70.36, 38.41 ppm.

4-(4-Chlorobenzyl)oxy)methyl)-2-phenyl-1,3-dioxolane: The title compound was synthesized according to general procedure A. White solid; No attempt was made to separate the diastereomers mixture; $^1$H NMR (500 MHz, CDCl$_3$) δ 7.47 (dd, J = 7.5, 2.0 Hz, 2H both diastereomers), 7.40–7.26 (m, 7H both diastereomers), 4.48–4.41 (m, 1H both diastereomers), 3.63 (ddd, J = 25.8, 10.2, 5.1 Hz, 2H both diastereomers) ppm; Cis: δ 5.94 (s, 1H), 4.57 (s, 2H), 4.24 (dd, J = 8.3, 6.7 Hz, 1H), 3.85 (dd, J = 8.3, 6.7 Hz, 1H) ppm; Trans: δ 5.81 (s, 1H), 4.54 (s, 2H), 4.10 (dd, J = 8.1, 7.1 Hz, 1H), 3.97 (dd, J = 8.2, 5.3 Hz, 1H) ppm; $^{13}$C NMR (126
MHz, CDCl₃) **Cis:** δ 137.24, 136.42, 133.52, 129.42, 129.04, 128.60, 128.35, 126.66, 104.42, 75.38, 72.74, 71.01, 67.90 ppm; **Trans:** δ 137.82, 136.42, 129.22, 128.99, 128.62, 126.43, 103.87, 75.03, 72.78, 70.58, 67.64 ppm.

The title compound was synthesized according to general procedure A. White solid; mp = 111.1-111.6 °C; ¹H NMR (500 MHz, CDCl₃) δ 7.45 (dd, J = 7.5, 1.8 Hz, 2H), 7.38–7.25 (m, 7H), 5.40 (s, 1H), 4.56 (s, 2H), 4.35 (dd, J = 11.0, 4.9 Hz, 2H), 3.77 (dt, J = 14.9, 4.8 Hz, 1H), 3.69–3.61 (m, 2H) ppm; ¹³C NMR (126 MHz, CDCl₃) δ 137.56, 136.40, 133.85, 129.03, 129.02, 128.74, 128.31, 126.04, 101.33, 71.03, 71.12, 68.12 ppm; HRMS (EI+) m/z: [M]⁺ calcd for C₁₇H₁₇ClO₃ 304.0866, found 304.0860; IR: 2976, 2866, 1492, 1456, 1386, 1337, 1152, 1090, 1011 cm⁻¹.

(2S,5R)-5-((4-Chlorobenzyl)oxy)-2-phenyl-1,3-dioxane: The title compound was synthesized according to general procedure A. White solid; Yield = 67.1%; ¹H NMR (500 MHz, CDCl₃) δ 7.52 (dd, J = 7.7, 1.5 Hz, 2H), 7.40–7.29 (m, 7H), 5.55 (s, 1H), 4.64 (s, 2H), 4.34 (dd, J = 12.6, 1.4 Hz, 2H), 4.04 (dd, J = 12.6, 1.6 Hz, 2H), 3.36–3.28 (m, 1H) ppm; ¹³C NMR (126 MHz, CDCl₃) δ 138.05, 136.71, 133.37, 128.96, 128.92, 128.56, 128.20, 126.15, 101.36, 69.68, 69.59, 68.92 ppm.

2-((4-Chlorobenzyl)oxy)propane-1,3-diol (1d): The title compound was synthesized according to general procedure A. White solid; Yield = 67.1%; ¹H NMR (500 MHz, CDCl₃) δ 7.37–7.26 (m, 4H), 4.63 (s, 2H), 3.84–3.70 (m, 4H), 3.59 (p, J = 4.7 Hz, 1H), 2.04 (s, 2H) ppm; ¹³C NMR (126 MHz, CDCl₃) δ 136.51, 133.80, 129.11, 128.77, 79.29, 71.19, 62.45 ppm.

3-((4-Chlorobenzyl)oxy)propane-1,2-diol (1’d): The title compound was synthesized according to general procedure A. White solid; Yield = 55.8%; ¹H NMR (500 MHz, CDCl₃) δ 7.35–7.22 (m, 4H), 4.51 (s, 2H), 3.93–3.86 (m, 1H), 3.74–3.59 (m,
2H), 3.54 (qd, J = 9.6, 5.2 Hz, 2H), 2.76 (d, J = 4.7 Hz, 1H), 2.29 (t, J = 5.7 Hz, 1H) ppm; $^{13}$C NMR (126 MHz, CDCl$_3$) $\delta$ 136.17, 133.72, 129.08, 128.70, 72.82, 71.89, 70.59, 64.03 ppm.

**4-Bromobenzyl methanesulfonate**: The title compound was synthesized according to reference$^{[10]}$. White solid; Yield = 90.9%; $^1$H NMR (500 MHz, CDCl$_3$) $\delta$ 7.57–7.53 (m, 2H), 7.32–7.28 (m, 2H), 5.19 (s, 2H), 2.95 (s, 3H) ppm; $^{13}$C NMR (126 MHz, CDCl$_3$) $\delta$ 132.47, 132.15, 130.42, 123.66, 70.36, 38.42 ppm.

Yield of product $5A + 5B = 87.7\%$ ($5A$: 20.5%, $5B$: 67.1%)

**4-(((4-Bromobenzyl)oxy)methyl)-2-phenyl-1,3-dioxolane**: The title compound was synthesized according to general procedure A. Opaque white gel type; No attempt was made to separate the diastereomers mixture; $^1$H NMR (500 MHz, CDCl$_3$) $\delta$ 7.47 (ddd, $J$ = 6.4, 4.7, 2.2 Hz, 4H both diastereomers), 7.39–7.35 (m, 3H both diastereomers), 7.22 (dd, $J$ = 11.3, 8.4 Hz, 2H both diastereomers), 4.47–4.38 (m, 2H both diastereomers), 3.62 (ddd, $J$ = 15.6, 11.9, 10.0, 6.1 Hz, 2H both diastereomers) ppm; **Cis**: $\delta$ 5.94 (s, 1H), 4.56 (s, 2H), 4.24 (dd, $J$ = 8.3, 6.7 Hz, 1H), 3.85 (dd, $J$ = 8.3, 6.7 Hz, 1H) ppm; **Trans**: $\delta$ 5.81 (s, 1H), 4.53 (s, 2H), 4.10 (dd, $J$ = 8.1, 7.1 Hz, 1H), 3.97 (dd, $J$ = 8.2, 5.3 Hz, 1H) ppm; $^{13}$C NMR (126 MHz, CDCl$_3$) $\delta$ 129.42, 129.36, 129.31, 129.22 (aromatic peaks of diastereomers) ppm; **Cis**: $\delta$ 137.83, 136.96, 131.58, 128.35, 126.43, 121.65, 103.87, 75.03, 72.81, 70.60, 67.64 ppm; **Trans**: $\delta$ 137.26, 136.95, 131.56, 128.35, 126.66, 121.64, 104.43, 75.39, 72.77, 71.03, 67.90 ppm; HRMS (EI+) $m/z$: [M]$^+$ calcld for Chemical C$_{17}$H$_{17}$BrO$_3$ 348.0361, found 348.0356; IR: 2908, 2867, 1592, 1485, 1400, 1221, 1099, 1057 cm$^{-1}$.

**$(2R,5R)$-5-(((4-Bromobenzyl)oxy)-2-phenyl-1,3-dioxane$^{[11]}$**: The title compound was synthesized according to general procedure A. White solid; $^1$H NMR (500 MHz, CDCl$_3$) $\delta$ 7.51–7.43 (m, 4H), 7.38–7.32 (m, 3H), 7.20 (d, $J$ = 8.3 Hz, 2H), 5.39 (s, 1H), 4.54 (s, 2H), 4.35 (dd, $J$ = 11.0, 4.9 Hz, 2H), 3.77 (dd, $J$ = 14.9, 9.7, 4.9 Hz, 1H), 3.64 (t, $J$ = 10.7 Hz, 2H) ppm; $^{13}$C NMR (126 MHz, CDCl$_3$) $\delta$ 137.56, 136.92, 131.67, 129.31, 129.02, 128.29, 126.04, 121.93, 101.31, 71.03, 70.09, 68.13 ppm.

**$(2S,5R)$-5-(((4-Bromobenzyl)oxy)-2-phenyl-1,3-dioxane$^{[11]}$**: The title compound was
synthesized according to general procedure A. White solid; \(^1\)H NMR (500 MHz, CDCl\(_3\)) \(\delta\) 7.49 (ddt, \(J = 10.7, 8.9, 2.0\) Hz, 4H), 7.39–7.27 (m, 5H), 5.56 (s, 1H), 4.65 (s, 2H), 4.35 (dd, \(J = 12.6, 1.4\) Hz, 2H), 4.10–4.02 (m, 2H), 3.37–3.32 (m, 1H) ppm; \(^{13}\)C NMR (126 MHz, CDCl\(_3\)) \(\delta\) 138.04, 137.24, 131.55, 129.30, 128.95, 128.23, 126.16, 121.54, 101.41, 69.70, 69.65, 68.95 ppm.

### 2-((4-Bromobenzyl)oxy)propane-1,3-diol (1e)

The title compound was synthesized according to general procedure A. White solid; Yield = 99%; \(^1\)H NMR (500 MHz, CDCl\(_3\)) \(\delta\) 7.49 (d, \(J = 8.2\) Hz, 2H), 7.24 (d, \(J = 8.1\) Hz, 2H), 4.62 (s, 2H), 3.77 (td, \(J = 11.6, 7.2\) Hz, 2H), 3.62–3.56 (m, 1H), 1.94 (s, 2H) ppm; \(^{13}\)C NMR (126 MHz, CDCl\(_3\)) \(\delta\) 137.05, 131.72, 129.41, 121.90, 79.33, 71.22, 62.47 ppm.

### 3-((4-Bromobenzyl)oxy)propane-1,2-diol (1'e)

The title compound was synthesized according to general procedure A. White solid; Yield = 95%; \(^1\)H NMR (500 MHz, CDCl\(_3\)) \(\delta\) 7.45 (d, \(J = 8.3\) Hz, 2H), 7.17 (d, \(J = 8.2\) Hz, 2H), 4.46 (s, 2H), 3.91–3.82 (m, 1H), 3.69–3.43 (m, 5H), 3.20 (s, 1H) ppm; \(^{13}\)C NMR (126 MHz, CDCl\(_3\)) \(\delta\) 136.74, 131.58, 129.37, 121.72, 72.71, 71.69, 70.81, 63.91 ppm.

Yield of product 6A+6B = 90.6% (6A: 30.1%, 6B: 60.5%)

### (2R,4R)-2-Phenyl-4-(((4-(trifluoromethyl)benzyl)oxy)methyl)-1,3-dioxolane

The title compound was synthesized according to general procedure A. White solid; mp = 57.2–58.1 °C; \(^1\)H NMR (500 MHz, CDCl\(_3\)) \(\delta\) 7.59 (d, \(J = 8.1\) Hz, 2H), 7.52–7.42 (m, 4H), 7.40–7.34 (m, 3H), 5.82 (s, 1H), 4.64 (s, 2H), 4.45 (dq, \(J = 6.9, 5.5\) Hz, 1H), 4.12 (dd, \(J = 8.1, 7.1\) Hz, 1H), 4.00 (dd, \(J = 8.2, 5.3\) Hz, 1H), 3.65 (dd, \(J = 33.2, 9.9, 5.6\) Hz, 2H) ppm; \(^{13}\)C NMR (126 MHz, CDCl\(_3\)) \(\delta\) 142.08 (s), 137.25 (s), 129.90 (q, \(J = 32.4\) Hz), 129.44 (s), 128.36 (s), 127.61 (s), 126.66 (s), 125.38 (q, \(J = 3.8\) Hz), 124.16 (q, \(J = 272.0\) Hz), 104.46 (s), 75.39 (s), 72.69 (s), 71.29 (s), 67.83 (s) ppm; \(^{19}\)F NMR (471 MHz, CDCl\(_3\)) \(\delta\) -62.50 ppm; HRMS (EI+) \(m/z\): [M]+ calcd for C\(_{18}\)H\(_{17}\)F\(_3\)O\(_3\) 338.1130, found 338.1125; IR: 2878, 1460, 1402, 1322, 1232, 1153, 1107, 1057, 1023 cm\(^{-1}\).

### (2S,4R)-2-Phenyl-4-(((4-(trifluoromethyl)benzyl)oxy)methyl)-1,3-dioxolane

The title compound was synthesized according to general procedure A. White solid; mp = 53.9–54.4 °C;
1H NMR (500 MHz, CDCl$_3$) $\delta$ 7.62 (d, $J = 8.1$ Hz, 2H), 7.52–7.44 (m, 4H), 7.42–7.35 (m, 3H), 5.96 (s, 1H), 4.67 (s, 2H), 4.50–4.44 (m, 1H), 4.29–4.23 (m, 1H), 3.87 (dd, $J = 8.1$, 6.9 Hz, 1H), 3.68 (qd, $J = 10.2$, 5.0 Hz, 2H) ppm; $^{13}$C NMR (126 MHz, CDCl$_3$) $\delta$ 142.07, 137.80, 129.94 (dd, $J = 64.7$, 32.4 Hz), 129.26, 128.38, 127.57, 126.44, 125.41 (q, $J = 3.7$ Hz), 124.15 (dd, $J = 544.0$, 272.0 Hz), 103.93, 75.04, 72.75, 70.89, 67.57 (s) ppm; $^{19}$F NMR (471 MHz, CDCl$_3$) $\delta$ -62.49 ppm; HRMS (EI+) $m/z$: [M]+ calcd for C$_{18}$H$_{17}$F$_3$O$_3$ 338.1130, found 338.1135; IR: 2878, 1463, 1410, 1330, 1221, 1158, 1101, 1062, 1021 cm$^{-1}$.

(2R,5R)-2-Phenyl-5-((4-(trifluoromethyl)benzyl)oxy)-1,3-dioxane: The title compound was synthesized according to general procedure A. White solid; mp = 118.5-119.4 °C; $^1$H NMR (500 MHz, CDCl$_3$) $\delta$ 7.62 (d, $J = 8.1$ Hz, 2H), 7.49–7.43 (m, 4H), 7.39–7.33 (m, 3H), 5.41 (s, 1H), 4.66 (s, 2H), 4.39 (dd, $J = 11.0$, 4.9 Hz, 2H), 3.81 (ddd, $J = 14.9$, 9.7, 4.9 Hz, 1H), 3.68 (qd, $J = 11.2$, 10.1 Hz, 2H) ppm; $^{13}$C NMR (126 MHz, CDCl$_3$) $\delta$ 142.40, 138.03, 129.85 (q, $J = 32.4$ Hz), 129.65, 128.32, 127.61, 125.50 (q, $J = 3.8$ Hz), 124.07 (dd, $J = 544.1$, 272.0 Hz), 120.93, 70.14, 69.65, 68.91 ppm; $^{19}$F NMR (471 MHz, CDCl$_3$) $\delta$ -62.58 ppm; HRMS (EI+) $m/z$: [M]+ calcd for C$_{18}$H$_{17}$F$_3$O$_3$ 338.1130, found 338.1136; IR: 2982, 2869, 1457, 1400, 1330, 1217, 1146, 1097, 1066, 1019 cm$^{-1}$.

(2S,5R)-2-Phenyl-5-((4-(trifluoromethyl)benzyl)oxy)-1,3-dioxane: The title compound was synthesized according to general procedure A. White solid; mp = 92.9-93.5 °C; $^1$H NMR (500 MHz, CDCl$_3$) $\delta$ 7.57 (dd, $J = 39.1$, 8.3 Hz, 6H), 7.41–7.34 (m, 3H), 5.58 (s, 1H), 4.75 (s, 2H), 4.38 (dd, $J = 12.6$, 1.2 Hz, 2H), 4.08 (dd, $J = 12.5$, 1.0 Hz, 2H), 3.37 (s, 1H) ppm; $^{13}$C NMR (126 MHz, CDCl$_3$) $\delta$ 142.40, 138.03, 129.85 (q, $J = 32.4$ Hz), 128.99, 128.26, 127.57, 126.16, 125.38 (q, $J = 3.8$ Hz), 124.17 (q, $J = 271.9$ Hz), 101.43, 70.14, 69.65, 68.91 ppm; $^{19}$F NMR (471 MHz, CDCl$_3$) $\delta$ -62.48 ppm; HRMS (EI+) $m/z$: [M]+ calcd for C$_{18}$H$_{17}$F$_3$O$_3$ 338.1130, found 338.1136; IR: 2982, 2869, 1618, 1453, 1394, 1323, 1280, 1217, 1146, 1097, 1065, 1014 cm$^{-1}$.

2-((4-(Trifluoromethyl)benzyl)oxy)propane-1,3-diol (1f)$^{[13]}$: The title compound was synthesized according to general procedure A. White solid; Yield = 84.1%; $^1$H NMR (500 MHz, CDCl$_3$) $\delta$ 7.62 (d, $J = 8.1$ Hz, 2H), 7.47 (d, $J = 8.0$ Hz, 2H), 4.72 (s, 2H), 3.86–3.72 (m, 4H), 3.60 (p, $J = 4.7$ Hz, 1H), 2.18 (s, 2H) ppm; $^{13}$C NMR (126 MHz, CDCl$_3$) $\delta$
142.10, 130.12 (dd, J = 64.8, 32.3 Hz), 127.71, 125.51 (q, J = 3.7 Hz), 124.08 (dd, J = 544,0, 272.0 Hz), 79.56, 71.12, 62.45 ppm; \(^{19}\)F NMR (471 MHz, CDCl\(_3\)) \(\delta\) -62.55 ppm.

3-((4-(Trifluoromethyl)benzyl)oxy)propane-1,2-diol (1'f): The title compound was synthesized according to general procedure A. Colorless oil; Yield = 83.1%; \(^1\)H NMR (500 MHz, CDCl\(_3\)) \(\delta\) 7.61 (d, J = 8.1 Hz, 2H), 7.44 (d, J = 8.0 Hz, 2H), 4.62 (s, 2H), 3.97–3.90 (m, 1H), 3.77–3.55 (m, 4H), 2.67 (d, J = 4.4 Hz, 1H), 2.17 (s, 1H) ppm; \(^{13}\)C NMR (126 MHz, CDCl\(_3\)) \(\delta\) 141.81, 130.08 (q, J = 32.4 Hz), 127.66, 125.47 (q, J = 3.7 Hz), 124.10 (q, J = 272.0 Hz), 72.76, 72.09, 70.69, 70.97 ppm; \(^{19}\)F NMR (471 MHz, CDCl\(_3\)) \(\delta\) -62.56 ppm; HRMS (EI+) \(m/z\): [M]\(^+\) calcd for C\(_{11}\)H\(_{13}\)F\(_3\)O\(_3\) 250.0817, found 250.0817; IR: 3377, 2926, 2873, 1718, 1621, 1456, 1417, 1325, 1163, 1118, 1065, 1020 cm\(^{-1}\).

Yield of product 7A+7B = 82% (7A: 19.3%, 7B: 62.7%)

4-(((3-Nitrobenzyl)oxy)methyl)-2-phenyl-1,3-dioxolane: The title compound was synthesized according to general procedure A. Slightly orange-colored liquid; Yield = 19.3%; No attempt was made to separate the diastereomers mixture; \(^1\)H NMR (500 MHz, CDCl\(_3\)) \(\delta\) 8.28–8.08 (m, 2H both diastereomers), 7.74–7.62 (m, 1H both diastereomers), 7.57–7.45 (m, 3H both diastereomers), 7.42–7.31 (m, 3H, both diastereomers), 4.52–4.42 (m, 1H both diastereomers), 3.76–3.62 (m, 2H both diastereomers) ppm; \(^{13}\)C NMR (126 MHz, CDCl\(_3\)) \(\delta\) 29.42, 129.46, 128.39, 129.28 (aromatic peaks of diastereomers); \(\text{Cis:}\ \delta\ 5.96 \text{ (s, 1H), 4.70 (s, 2H), 4.26 \text{ (dd, J = 8.2, 6.9 Hz, 1H), 3.87 \text{ (dd, J = 8.3, 6.8 Hz, 1H) ppm; Trans:}\ \delta\ 5.82 \text{ (s, 1H), 4.67 \text{ (s, 2H), 4.16–4.10 (m, 1H), 4.00 \text{ (dd, J = 8.2, 5.3 Hz, 1H) ppm;}^{13}\)C NMR (126 MHz, CDCl\(_3\)) \(\delta\) 29.42, 129.46, 128.39, 129.28 (aromatic peaks of diastereomers); \(\text{Cis:}\ \delta\ 148.37, 137.80, 133.32, 126.47, 122.70, 122.23, 103.97, 75.05, 72.28, 71.13, 67.52 ppm; \(\text{Trans:}\ \delta\ 140.27, 137.38, 133.38, 126.66, 122.69, 122.26, 104.48, 75.39, 72.25, 71.49, 67.76 ppm; HRMS (FAB+) \(m/z\): [M+H]\(^+\) calcd for C\(_{17}\)H\(_{17}\)NO\(_5\) 316.1185, found 316.1179; IR: 2874, 1734, 1528, 1216, 1096, 1022 cm\(^{-1}\).

(2R,5R)-5-(((3-Nitrobenzyl)oxy)-2-phenyl-1,3-dioxane: The title compound was synthesized according to general procedure A. Light yellow-colored solid; \(m/p = 87.8-88.6 \degree C; \(^1\)H NMR (500 MHz, CDCl\(_3\)) \(\delta\) 8.27–8.14 (m, 2H), 7.65 (d, J = 7.6 Hz, 1H),
7.55–7.33 (m, 6H), 5.43 (s, 1H), 4.69 (s, 2H), 4.41 (dd, \(J = 11.0, 4.9\) Hz, 2H), 3.87–3.79 (m, 1H), 3.70 (t, \(J = 10.6\) Hz, 2H) ppm; \(^{13}\)C NMR (126 MHz, CDCl\(_3\)) \(\delta\) 148.41, 140.11, 137.47, 133.19, 129.50, 129.07, 128.32, 126.06, 122.90, 122.19, 101.36, 70.36, 69.93, 68.62 ppm; HRMS (FAB+) \(m/z\): [M+H]\(^+\) calcd for C\(_{17}\)H\(_{17}\)NO\(_3\) 316.1185, found 316.1181; IR: 2979, 2914, 2875, 1619, 1583, 1527, 1478, 1452, 1397, 1349, 1311, 1269, 1219, 1105 cm\(^{-1}\).

**\(2S,5R\)-5-(3-Nitrobenzyl)oxy)-2-phenyl-1,3-dioxane:** The title compound was synthesized according to general procedure A. Thick yellow-colored solid; mp = 113.1–113.9 °C; \(^1\)H NMR (500 MHz, CDCl\(_3\)) \(\delta\) 8.28 (s, 1H), 8.14 (dd, \(J = 8.2, 1.4\) Hz, 1H), 7.77 (d, \(J = 7.6\) Hz, 1H), 7.52 (t, \(J = 7.9\) Hz, 3H), 7.41–7.33 (m, 3H), 5.59 (s, 1H), 4.78 (s, 2H), 4.40 (dd, \(J = 12.7, 1.3\) Hz, 2H), 4.11 (dd, \(J = 12.7, 1.5\) Hz, 2H), 3.45–3.41 (m, 1H) ppm; \(^{13}\)C NMR (126 MHz, CDCl\(_3\)) \(\delta\) 148.33, 140.59, 137.98, 133.36, 129.40, 129.00, 128.26, 126.15, 122.61, 122.18, 101.45, 70.63, 69.25, 68.86 ppm; HRMS (FAB+) \(m/z\): [M+H]\(^+\) calcd for C\(_{17}\)H\(_{17}\)NO\(_3\) 316.1185, found 316.1181; IR: 2979, 2918, 2860, 1526, 1456, 1389, 1345, 1152, 1090 cm\(^{-1}\).

**\(2\)-(3-Nitrobenzyl)oxy)propane-1,3-diol (1g):** The title compound was synthesized according to general procedure A. Orange-colored solid; mp = 61.2–62.0 °C; Yield = 85.2%; \(^1\)H NMR (500 MHz, CDCl\(_3\)) \(\delta\) 8.24 (s, 1H), 8.16 (dd, \(J = 8.2, 1.3\) Hz, 1H), 7.70 (dd, \(J = 7.6, 0.5\) Hz, 1H), 7.54 (t, \(J = 7.9\) Hz, 1H), 4.78 (s, 2H), 3.83 (ddd, \(J = 34.4, 11.7, 4.5\) Hz, 4H), 3.65 (p, \(J = 4.7\) Hz, 1H), 2.14 (s, 2H) ppm; \(^{13}\)C NMR (126 MHz, CDCl\(_3\)) \(\delta\) 148.33, 140.59, 137.98, 133.36, 129.40, 129.00, 128.26, 126.15, 122.61, 122.18, 101.45, 70.63, 69.25, 68.86 ppm; HRMS (Cl+) \(m/z\): [M]\(^+\) calcd for C\(_{10}\)H\(_{13}\)NO\(_5\) 228.0872, found 228.0879; IR: 3353, 2949, 2888, 1518, 1482, 1447, 1349, 1246, 1212, 1086, 1042 cm\(^{-1}\).

**\(3\)-(3-Nitrobenzyl)oxy)propane-1,2-diol (1′g):** The title compound was synthesized according to general procedure A. Pale yellow-colored solid; Yield = 76.5%; \(^1\)H NMR (500 MHz, CDCl\(_3\)) \(\delta\) 8.25–8.13 (m, 2H), 7.67 (d, \(J = 7.6\) Hz, 1H), 7.54 (t, \(J = 7.9\) Hz, 1H), 4.66 (s, 2H), 3.99–3.93 (m, 1H), 3.79–3.59 (m, 4H), 2.57 (s, 1H), 2.04 (s, 1H) ppm; \(^{13}\)C NMR (126 MHz, CDCl\(_3\)) \(\delta\) 148.41, 140.00, 133.36, 129.50, 122.83, 122.30, 72.30, 72.21, 70.67, 63.87 ppm.
**Benzoic acid (2a)**\(^{[1]}\): The title compound was synthesized according to general procedure B. White solid; \(^1\)H NMR (500 MHz, CDCl\(_3\)) \(\delta\) 12.74 (s, 1H), 8.27–8.06 (m, 2H), 7.71–7.43 (m, 3H) ppm; \(^{13}\)C NMR (126 MHz, CDCl\(_3\)) \(\delta\) 172.55, 133.85, 130.24, 129.35, 128.50 ppm.

**2-Naphthoic acid (2b)**\(^{[14]}\): The title compound was synthesized according to general procedure B. White solid; \(^1\)H NMR (500 MHz, CDCl\(_3\)) \(\delta\) 8.73 (s, 1H), 8.13 (dd, \(J = 8.6, 1.5\) Hz, 1H), 8.04–7.87 (m, 3H), 7.67–7.54 (m, 2H) ppm; \(^{13}\)C NMR (126 MHz, CDCl\(_3\)) \(\delta\) 171.73, 135.99, 132.47, 132.18, 129.56, 128.69, 128.36, 127.84, 126.81, 126.46, 125.41 ppm.

**3-Methoxybenzoic acid (2c)**\(^{[15]}\): The title compound was synthesized according to general procedure B. White solid; \(^1\)H NMR (500 MHz, CDCl\(_3\)) \(\delta\) 7.73 (d, \(J = 7.8\) Hz, 1H), 7.63 (dd, \(J = 2.5, 1.5\) Hz, 1H), 7.39 (t, \(J = 8.0\) Hz, 1H), 7.17 (ddd, \(J = 8.3, 2.6, 0.8\) Hz, 1H), 3.87 (s, 3H) ppm; \(^{13}\)C NMR (126 MHz, CDCl\(_3\)) \(\delta\) 171.85, 159.64, 130.53, 129.55, 122.71, 120.52, 114.41, 55.48 ppm.

**4-Chlorobenzoic acid (2d)**\(^{[15]}\): The title compound was synthesized according to general procedure B. White solid; \(^1\)H NMR (500 MHz, DMSO-\(d_6\)) \(\delta\) 13.22 (s, 1H), 7.97 (d, \(J = 8.5\) Hz, 2H), 7.58 (d, \(J = 8.5\) Hz, 2H) ppm; \(^{13}\)C NMR (126 MHz, DMSO-\(d_6\)) \(\delta\) 166.93, 138.25, 131.59, 130.14, 129.16 ppm.

**4-Bromobenzoic acid (2e)**\(^{[16]}\): The title compound was synthesized according to general procedure B. White solid; \(^1\)H NMR (500 MHz, Acetone-\(d_6\)) \(\delta\) 11.45 (s,
1H), 8.00–7.95 (m, 2H), 7.75–7.69 (m, 2H) ppm; $^{13}$C NMR (126 MHz, DMSO-$d_6$) $\delta$ 166.52, 131.61, 131.20, 129.92, 126.79 ppm.

\[
\text{2f} \quad 
\begin{array}{c}
\text{F}_3\text{C} \\
\text{O} \\
\text{H}
\end{array}
\]

4-(Trifluoromethyl)benzoic acid (2f)\textsuperscript{[16]}: The title compound was synthesized according to general procedure B. White solid; $^1$H NMR (500 MHz, Acetone) $\delta$ 11.72 (s, 1H), 8.30–8.22 (m, 2H), 7.89 (d, $J$ = 8.1 Hz, 2H) ppm; $^{13}$C NMR (126 MHz, DMSO-$d_6$) $\delta$ 166.68, 135.14, 132.92 (q, $J$ = 31.8 Hz), 130.57, 126.07 (q, $J$ = 3.7 Hz), 124.29 (q, $J$ = 272.7 Hz) ppm; $^{19}$F NMR (471 MHz, MeOD) $\delta$ -63.62 ppm.

\[
\text{2g} \quad 
\begin{array}{c}
\text{O} \\
\text{N} \\
\text{O} \\
\text{H}
\end{array}
\]

3-Nitrobenzoic acid (2g)\textsuperscript{[16]}: The title compound was synthesized according to general procedure B. White solid; $^1$H NMR (500 MHz, DMSO-$d_6$) $\delta$ 13.70 (s, 1H), 8.65–8.59 (m, 1H), 8.47 (ddd, $J$ = 8.2, 2.3, 0.9 Hz, 1H), 8.38–8.33 (m, 1H), 7.82 (t, $J$ = 8.0 Hz, 1H); $^{13}$C NMR (126 MHz, DMSO-$d_6$) $\delta$ 165.44, 147.82, 135.29, 132.39, 130.46, 127.26, 123.61 ppm.

\[
\text{3a} \quad 
\begin{array}{c}
\text{O} \\
\text{O} \\
\text{C}
\end{array}
\]

2-(Benzyl oxy)acetic acid (3a)\textsuperscript{[17]}: The title compound was synthesized according to general procedure B. Slightly yellow-colored oil; Yield = 92%; $^1$H NMR (500 MHz, CDCl$_3$) $\delta$ 7.42–7.29 (m, 5H), 4.65 (s, 2H), 4.15 (s, 2H) ppm; $^{13}$C NMR (126 MHz, CDCl$_3$) $\delta$ 174.55, 136.49, 128.64, 128.32, 128.14, 73.53, 66.59 ppm.

\[
\text{3b} \quad 
\begin{array}{c}
\text{O} \\
\text{O} \\
\text{C}
\end{array}
\]

2-(Naphthalen-2-ylmethoxy)acetic acid (3b): The title compound was synthesized according to general procedure B. Yellow-colored solid; $^1$H NMR (500 MHz, CDCl$_3$) $\delta$ 7.89–7.76 (m, 4H), 7.54–7.45 (m, 3H), 4.81 (s, 2H), 4.18 (s, 2H) ppm; $^{13}$C NMR (126 MHz, CDCl$_3$) $\delta$ 174.79, 133.96, 133.21, 133.19, 128.53, 127.94, 127.74, 127.16, 126.32, 126.23, 125.76, 73.60, 66.59 ppm.
2-((3-Methoxybenzyl)oxy)acetic acid (3c): The title compound was synthesized according to general procedure B. White solid; $^1$H NMR (500 MHz, CDCl$_3$) $\delta$ 7.28 (dd, $J =$ 13.4, 5.6 Hz, 1H), 6.98–6.84 (m, 3H), 4.63 (s, 2H), 4.14 (s, 2H), 3.82 (s, 3H) ppm; $^{13}$C NMR (126 MHz, CDCl$_3$) $\delta$ 174.24, 159.89, 138.04, 129.70, 120.31, 113.95, 113.40, 73.40, 66.56, 55.27 ppm.

2-((4-Chlorobenzyl)oxy)acetic acid (3d): The title compound was synthesized according to general procedure B. White solid; $^1$H NMR (500 MHz, CDCl$_3$) $\delta$ 7.38–7.28 (m, 4H), 4.62 (s, 2H), 4.15 (s, 2H) ppm; $^{13}$C NMR (126 MHz, CDCl$_3$) $\delta$ 173.76, 135.03, 134.17, 129.39, 128.83, 72.75, 66.70 ppm.

2-((4-Bromobenzyl)oxy)acetic acid (3e): The title compound was synthesized according to general procedure B. White solid; $^1$H NMR (500 MHz, CDCl$_3$) $\delta$ 7.54–7.47 (m, 2H), 7.29–7.22 (m, 2H), 4.60 (s, 2H), 4.16 (s, 2H) ppm; $^{13}$C NMR (126 MHz, CDCl$_3$) $\delta$ 174.89, 135.61, 131.76, 129.67, 122.25, 72.75, 66.71 ppm.

2-((4-(Trifluoromethyl)benzyl)oxy)acetic acid (3f)\cite{18}: The title compound was synthesized according to general procedure B. White solid; $^1$H NMR (500 MHz, CDCl$_3$) $\delta$ 7.63 (d, $J =$ 8.1 Hz, 2H), 7.49 (d, $J =$ 8.0 Hz, 2H), 4.71 (s, 2H), 4.20 (s, 2H) ppm; $^{13}$C NMR (126 MHz, CDCl$_3$) $\delta$ 175.11, 140.78, 130.40 (q, $J =$ 32.4 Hz), 127.94, 125.56 (q, $J =$ 3.8 Hz), 124.05 (dd, $J =$ 544.1, 272.1 Hz), 72.69, 66.99 ppm; $^{19}$F NMR (471 MHz, CDCl$_3$) $\delta$ -62.61 ppm.

2-((3-Nitrobenzyl)oxy)acetic acid (3g): The title compound was synthesized according to general procedure B. Yellow solid; $^1$H NMR (500 MHz, CDCl$_3$) $\delta$ 8.25 (s, 1H), 8.18 (dd, $J =$ 8.2, 1.3 Hz, 1H), 7.72 (d, $J =$ 7.9 Hz, 1H), 7.56 (t, $J =$ 7.9 Hz, 1H), 4.75 (s, 2H), 4.26 (s, 2H); $^{13}$C NMR (126 MHz, CDCl$_3$) $\delta$ 175.14, 148.42, 139.05, 133.61, 129.60, 123.11, 122.57, 72.24, 67.20 ppm.
Dimethyl 2-(benzyloxy)malonate\(^{[19]}\): The title compound was synthesized according to general procedure D. Colorless oil; \(^1\)H NMR (500 MHz, CDCl\(_3\)) \(\delta\) 7.41–7.30 (m, 5H), 4.71 (s, 2H), 4.57 (s, 1H), 3.80 (s, 6H) ppm; \(^{13}\)C NMR (126 MHz, CDCl\(_3\)) \(\delta\) 166.92, 135.95, 128.58, 128.42, 128.41, 77.51, 72.89, 52.92 ppm.

2-(Benzyloxy)malonic acid (18): The title compound was synthesized according to general procedure B. White solid; \(^1\)H NMR (500 MHz, MeOD) \(\delta\) 11.62 (s, 2H), 7.48–7.29 (m, 5H), 4.73 (s, 2H), 4.69 (s, 1H) ppm; \(^{13}\)C NMR (126 MHz, MeOD) \(\delta\) 170.15, 138.15, 129.52, 129.47, 129.25, 79.01, 73.52 ppm.

2-(Benzyloxy)acetyl chloride (21): The title compound was synthesized according to reference\(^{[17]}\). Transparent brown-colored liquid; Yield = 99%; \(^1\)H NMR (500 MHz, CDCl\(_3\)) \(\delta\) 7.40–7.29 (m, 5H), 4.66 (s, 2H), 4.43 (s, 2H) ppm; \(^{13}\)C NMR (126 MHz, CDCl\(_3\)) \(\delta\) 171.88, 136.07, 128.68, 128.46, 128.15, 74.82, 73.59 ppm.

(S)-3-(2-(Benzyloxy)acetyl)-4-isopropyloxazolidin-2-one (22): The title compound was synthesized according to reference\(^{[20]}\). White solid; Yield = 70%; \(^1\)H NMR (500 MHz, CDCl\(_3\)) \(\delta\) 7.45–7.25 (m, 5H), 4.70 (s, 2H), 4.66 (s, 2H), 4.47–4.42 (m, 1H), 4.32 (t, \(J = 8.8\) Hz, 1H), 4.25 (dd, \(J = 9.2, 3.1\) Hz, 1H), 2.48–2.38 (m, 1H), 0.90 (dd, \(J = 24.6, 7.0\) Hz, 6H) ppm; \(^{13}\)C NMR (126 MHz, CDCl\(_3\)) \(\delta\) 170.10, 154.02, 137.22, 128.50, 128.10, 128.01, 73.51, 69.63, 64.44, 58.24, 28.28, 17.92, 14.67 ppm.
(S)-3-((R)-2-(Benzyloxy)-3-phenylpropanoyl)-4-isopropyl-oxazolidin-2-one (23): The title compound was synthesized according to general procedure C. Transparent brown-colored liquid; Yield = 73.4%; $^1$H NMR (500 MHz, CDCl$_3$) δ 7.36 (d, $J = 7.2$ Hz, 2H), 7.31–7.20 (m, 6H), 7.12 (dd, $J = 6.6$, 2.9 Hz, 2H), 5.32 (dd, $J = 9.2$, 3.6 Hz, 1H), 4.50 (d, $J = 11.7$ Hz, 1H), 4.36 (td, $J = 8.5$, 4.2 Hz, 2H), 4.23–4.14 (m, 2H), 3.17 (dd, $J = 13.5$, 3.6 Hz, 1H), 2.29–2.21 (m, 1H), 0.87 (d, $J = 7.0$ Hz, 3H), 0.76 (d, $J = 6.9$ Hz, 3H) ppm; $^{13}$C NMR (126 MHz, CDCl$_3$) δ 172.45, 153.65, 137.42, 137.09, 129.76, 128.14, 128.07, 127.66, 126.63, 78.45, 72.81, 64.02, 58.27, 59.32, 28.42, 17.86, 14.76 ppm; HRMS (FAB+) $m/z$: [M+H]$^+$ calcd for C$_{22}$H$_{25}$NO$_3$ 368.1862, found 368.1863; IR: 2964, 2875, 1776, 1709, 1495, 1454, 1388, 1300, 1241, 1204, 1110 cm$^{-1}$.

(R)-2-(Benzyloxy)-3-phenylpropanoic acid (24): The title compound was synthesized according to reference$^{[21]}$. White solid; $[\alpha]_D^{20}$ +78.25° (c 0.8, EtOH). (lit. $[\alpha]_D^{20}$ +72.5° (c 0.8, EtOH)$^{[22]}$; Yield = 83.4%; $^1$H NMR (500 MHz, CDCl$_3$) δ 7.51–7.10 (m, 10H), 4.63 (dd, $J = 11.6$, 4.5 Hz, 1H), 4.43 (d, $J = 11.7$ Hz, 1H), 4.20 (dd, $J = 8.3$, 4.1 Hz, 1H), 3.12 (ddd, $J = 22.4$, 14.1, 6.2 Hz, 2H) ppm; $^{13}$C NMR (126 MHz, CDCl$_3$) δ 172.45, 153.65, 137.42, 137.09, 129.76, 128.14, 128.07, 127.66, 126.63, 78.45, 72.81, 64.02, 58.27, 39.52, 28.42, 17.86, 14.76 ppm.

(R)-2-Hydroxy-3-phenylpropanoic acid (25): The title compound was synthesized according to reference$^{[23]}$. White solid; $[\alpha]_D^{20}$ +26.7° (c 1.0, (CH$_3$)$_2$CO). (lit. $[\alpha]_D^{20}$ +26.95° (c 1.0, (CH$_3$)$_2$CO)$^{[24]}$; Yield = 83.4%; $^1$H NMR (500 MHz, DMSO-$d_6$) δ 7.43–7.00 (m, 5H), 4.13 (dd, $J = 7.8$, 4.2 Hz, 1H), 2.86 (ddd, $J = 22.0$, 13.7, 6.2 Hz, 2H) ppm; $^{13}$C NMR (126 MHz, DMSO-$d_6$) δ 175.01, 138.03, 129.28, 127.85, 125.99, 70.94, 39.89 ppm.
**S22**

(R)-Methyl 2-hydroxy-3-phenylpropanoate (26): The title compound was synthesized according to reference\(^{[25]}\). White solid; \([\alpha]^{20}_{D} +7.2^\circ\) (c 1.0, CHCl\(_3\)). (lit. \([\alpha]^{22}_{D} +6.1^\circ\) (c 1.0, CHCl\(_3\))\(^{[26]}\); Yield = 86.1%; \(^1\)H NMR (500 MHz, CDCl\(_3\)) \(\delta 7.38–7.18\) (m, 5H), 4.45 (td, \(J = 6.5, 4.5\) Hz, 1H), 3.77 (s, 3H), 3.12 (dd, \(J = 13.9, 4.4\) Hz, 1H), 2.96 (dd, \(J = 13.9, 6.8\) Hz, 1H), 2.72 (dd, \(J = 11.4, 6.2\) Hz, 1H) ppm; \(^{13}\)C NMR (126 MHz, CDCl\(_3\)) \(\delta 174.58, 136.42, 129.47, 128.41, 126.87, 71.34, 52.39, 40.55\) ppm.

(S)-Methyl 2-bromo-3-phenylpropanoate: The title compound was synthesized according to reference\(^{[27]}\). Colorless oil; \([\alpha]^{20}_{D} -21.6^\circ\) (c 1.28, CHCl\(_3\)). (lit. \([\alpha]^{24}_{D} -21.8^\circ\) (c 1.28, CHCl\(_3\))\(^{[27]}\); Yield = 86.1%; \(^1\)H NMR (500 MHz, CDCl\(_3\)) \(\delta 7.34–7.18\) (m, 5H), 4.40 (dd, \(J = 8.4, 7.1\) Hz, 1H), 3.72 (s, 3H), 3.46 (dd, \(J = 14.1, 8.4\) Hz, 1H), 3.24 (dd, \(J = 14.1, 7.1\) Hz, 1H) ppm; \(^{13}\)C NMR (126 MHz, CDCl\(_3\)) \(\delta 169.89, 136.73, 129.17, 128.69, 127.37, 52.93, 45.10, 41.14\) ppm.

(R)-Methyl 2-azido-3-phenylpropanoate (27): The title compound was synthesized according to reference\(^{[28]}\). Colorless oil; \([\alpha]^{20}_{D} +45.4^\circ\) (c 1.0, CHCl\(_3\)). (lit. \([\alpha]^{28}_{D} +47.8^\circ\) (c 1.0, CHCl\(_3\))\(^{[29]}\); Yield = 92%; \(^1\)H NMR (500 MHz, CDCl\(_3\)) \(\delta 7.36–7.21\) (m, 5H), 4.07 (dd, \(J = 8.4, 5.4\) Hz, 1H), 3.77 (s, 3H), 3.46 (dd, \(J = 14.1, 8.4\) Hz, 1H), 3.01 (dd, \(J = 14.1, 7.1\) Hz, 1H) ppm; \(^{13}\)C NMR (126 MHz, CDCl\(_3\)) \(\delta 169.89, 136.73, 129.17, 128.69, 127.37, 52.93, 45.10, 41.14\) ppm.

(R)-Methyl 2-amino-3-phenylpropanoate (28): The title compound was synthesized according to reference\(^{[30]}\). Colorless oil; \([\alpha]^{20}_{D} -26.2^\circ\) (c 4.0, EtOH). (lit. \([\alpha]^{23}_{D} -25.0^\circ\) (c 4.04, EtOH)\(^{[31]}\); Yield = 85%; \(^1\)H NMR (500 MHz, CDCl\(_3\)) \(\delta 7.36–7.16\) (m, 5H), 3.80–3.68 (m, 4H), 3.09 (dd, \(J = 13.5, 5.2\) Hz, 1H), 2.86 (dd, \(J = 13.5, 7.9\) Hz, 1H), 1.52 (s, 2H); \(^{13}\)C NMR (126 MHz, CDCl\(_3\)) \(\delta 175.44, 137.24, 129.27, 128.57, 126.84, 55.84, 51.96, 41.11\) ppm; HPLC
analysis: Chiralpak AY-H, (hexane/EtOH/2-aminoethanol = 1/1/0.1 %, wavelength = 257 nm, flow rate = 1.0 mL/min), \( t_R = 6.2 \text{ min (major)}, t_R = 7.5 \text{ min (minor)}. \)

\[
\begin{align*}
\text{(S)-Methyl 2-azido-3-phenylpropanoate (29): } & \text{The title compound was synthesized according to reference}^{[32]} . \text{ Colorless oil; } [\alpha]_D^{20} -45.2^\circ \text{ (c 1.0, CHCl}_3). \text{ (lit. } [\alpha]_D^{28} -47.8^\circ \text{ (c 1.0, CHCl}_3)^{[29]}; \text{ Yield = 88.6\%}; \text{ } ^1\text{H NMR (500 MHz, CDCl}_3) \delta 7.36–7.21 \text{ (m, 5H), 4.07 (dd, } J = 8.8, 5.4 \text{ Hz, 1H), 3.77 (s, 3H), 3.18 (dd, } J = 14.0, 5.4 \text{ Hz, 1H), 3.01 (dd, } J = 14.0, 8.8 \text{ Hz, 1H) ppm; } ^{13}\text{C NMR (126 MHz, CDCl}_3) \delta 170.40, 135.93, 129.18, 128.71, 127.29, 63.28, 52.64, 37.67 \text{ ppm.} \\
\end{align*}
\]

\[
\begin{align*}
\text{(S)-Methyl 2-amino-3-phenylpropanoate (30): } & \text{The title compound was synthesized according to reference}^{[30]} . \text{ Colorless oil; } [\alpha]_D^{20} +26.5^\circ \text{ (c 4.0, EtOH). (lit. } [\alpha]_D^{23} +25.0^\circ \text{ (c 4.04, EtOH)}^{[31]}; \text{ Yield = 85\%; } ^1\text{H NMR (500 MHz, CDCl}_3) \delta 7.36–7.16 \text{ (m, 5H), 3.80–3.68 (m, 4H), 3.09 (dd, } J = 13.5, 5.2 \text{ Hz, 1H), 2.86 (dd, } J = 13.5, 7.9 \text{ Hz, 1H), 1.52 (s, 2H); } ^{13}\text{C NMR (126 MHz, CDCl}_3) \delta 175.44, 137.24, 129.27, 128.57, 126.84, 55.84, 51.96, 41.11 \text{ ppm; HPLC analysis: Chiralpak AY-H (hexane/EtOH/2-aminoethanol = 1/1/0.1 \%, wavelength = 257 nm, flow rate = 1.0 mL/min), } t_R = 6.2 \text{ min (minor), } t_R = 7.3 \text{ min (major).} \\
\end{align*}
\]
References

$^1$H NMR and $^{13}$C NMR spectra of the products
6B (anti)
6B (syn)
21

1H NMR (400 MHz, CDCl₃) δ ppm:
- 7.45 (m, 5H, Ar-H)
- 4.03 (s, 2H, CH₂)
- 2.02 (s, 3H, CH₃)

13C NMR (100 MHz, CDCl₃) δ ppm:
- 168.1 (s, C=O)
- 135.1 (s, Ar-C)
- 119.6 (d, Ar-C)
- 40.8 (t, CH₂)
- 22.1 (s, CH₃)
NMR spectra of recovering chiral auxiliary
HPLC spectra of chiral products

HPLC spectrum of racemic 28

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rac-28

HPLC spectrum of chiral product 28

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30, ee = 92%