# Isothiourea-Catalysed Sequential Kinetic Resolution of Acyclic (±)-1,2-Diols

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**General Experimental**

Reactions involving moisture sensitive reagents were carried out in flame-dried glassware under a nitrogen atmosphere using standard vacuum line techniques, and using anhydrous solvents. Anhydrous solvents (Et₂O, THF, CH₂Cl₂ and toluene) were obtained from an anhydrous solvent system (purified using an alumina column, Mbraun SPS-800). All other reactions were performed in standard glassware with no precautions to exclude air or moisture. Solvents and commercial reagents were used as supplied without further purification unless otherwise stated.

Room temperature (r.t.) refers to 20-25 °C. Temperatures of 0 °C and −78 °C were obtained using ice/water and CO₂(s)/acetone baths respectively.

*In vacuo* refers to the use either a Büchi Rotavapor R-200 with a Büchi V-491 heating bath and Büchi V-800 vacuum controller; a Büchi Rotavapor R-210 with a Büchi V-491 heating bath and Büchi V-850 vacuum controller; a Heidolph Laborota 4001 with vacuum controller; an IKA RV10 rotary evaporator with an IKA HB10 heating bath and ILMVAC vacuum controller; or an IKA RV10 rotary evaporator with an IKA HB10 heating bath and Vacuubrand CVC3000 vacuum controller. Rotary evaporator condensers are fitted to Julabo FL601 Recirculating Coolers filled with ethylene glycol set to −5 °C.

Analytical thin layer chromatography was performed on pre-coated aluminium plates (Kieselgel 60 F₂₅₄ silica). TLC visualisation was carried out with ultraviolet light (254 nm), followed by staining with a 1% aqueous KMnO₄ solution. Flash column chromatography was performed on Kieselgel 60 silica in the solvent system stated. Automated chromatography was performed on a Biotage Isolera Four running Biotage OS578 with a UV/Vis detector using the method stated and cartridges filled with Kieselgel 60 silica.

Melting points were recorded on an Electrothermal 9100 melting point apparatus and are uncorrected.

Optical rotations were measured on a Perkin Elmer Precisely/Model-341 polarimeter operating at the sodium D line with a 100 mm path cell at 20 °C.

HPLC analyses were obtained using either a Shimadzu HPLC consisting of a DGU-20A5 degassing unit, LC-20AT liquid chromatography pump, SIL-20AHT autosampler, CMB-20A communications bus module, SPD-M20A diode array detector and a CTO-20A column oven; or a Shimadzu HPLC consisting of a DGU-20A5R degassing unit, LC-20AD liquid chromatography pump, SIL-20AHT autosampler, SPD-20A UV/Vis detector and a CTO-20A column oven. Separation was achieved using DAICEL CHIRALCEL OD-H and OJ-H columns or DAICEL CHIRALPAK AD-H, AS-H, IC and ID columns using the method stated. HPLC traces of enantiomerically enriched compounds were compared with authentic racemic spectra.

¹H, ¹³C(¹H) and ¹⁹F(¹H) nuclear magnetic resonance (NMR) spectra were acquired on either a Bruker Avance II 400 (¹H 400 MHz; ¹³C(¹H) 101 MHz, ¹⁹F(¹H) 376 MHz) or a Bruker Avance II 500 (¹H 500 MHz; ¹³C(¹H) 126 MHz, ¹⁹F(¹H) 471 MHz) spectrometer at ambient temperature in the deuterated solvent stated. All chemical shifts are quoted in parts per million (ppm) and referenced to the residual solvent peak. All coupling constants, *J*, are quoted in Hz. Multiplicities are indicated by: s
(singlet), d (doublet), t (triplet), q (quartet), dd (doublet of doublets), dt (doublet of triplets), dq (doublet of quartets), tt (triplet of triplets), ddd (doublet of doublet of doublets) and m (multiplet). The abbreviation Ar is used to denote aromatic, Ph to denote phenyl, br to denote broad and app to denote apparent. NMR peak assignments were confirmed using 2D $^1\text{H}-^1\text{C}$ heteronuclear single quantum coherence (HSQC) and 2D $^1\text{H}-^1\text{C}$ heteronuclear multiple-bond correlation spectroscopy (HMBC) where necessary.

Infrared spectra were recorded on a Shimadzu IRAffinity-1 Fourier transform IR spectrophotometer fitted with a Specac Quest ATR accessory (diamond puck). Spectra were recorded of either thin films or solids, with characteristic absorption wavenumbers ($\nu_{\text{max}}$) reported in cm$^{-1}$.

Mass spectrometry ($m/z$) data were acquired by electrospray ionization (ESI), atmospheric pressure chemical ionization (APCI) or nanospray ionization (NSI) either at the University of St Andrews or the EPSRC National Mass Spectrometry Facility, Swansea.
Synthesis of (±)-1,2-Diols

(±)-1,2-Diols 2 and 11–16 were synthesised according to the procedure given in the manuscript (2 and 11), or by following a literature procedure1 (12–16). Spectral data were in accordance with the literature (2, 12 and 14–16,2 11,3 134).

(±)-(E,E)-1,6-Diphenylhexa-1,5-diene-3,4-diol 17

\[
\text{OH} \quad \text{Ph} \quad \text{OH}
\]

\[\text{±}-17\]

THF (1.6 mL, 20 mmol) was added to a solution of TiCl₄ (0.55 mL, 5 mmol) in anhydrous CH₂Cl₂ (10 mL) under an N₂ atmosphere at r.t. and was allowed to stir for 30 seconds. Zinc powder (163 mg, 2.5 mmol) was added, and after a further 30 seconds TMEDA (1.12 mL, 7.5 mmol) was added. After a further 30 seconds, a solution of cinnamaldehyde (0.63 mL, 5 mmol) in CH₂Cl₂ (5 mL) was added and the mixture allowed to stir at r.t. for 1 h. HCl (1 M) was added and the mixture extracted with EtOAc (3 × 20 mL). The combined organic fractions were washed with brine, dried (Na₂SO₄), filtered and concentrated to give a residue, which was purified by Biotage flash silica column chromatography (0→40% EtOAc in hexane) to give a colourless solid, which was further purified by recrystallisation: the material was dissolved hot PhMe:hexane (1:1), allowed to cool to r.t. then cooled in a freezer overnight. The product was filtered and washed with cold hexane to give (±)-(E,E)-1,6-diphenylhexa-1,5-diene-3,4-diol 17 as an colourless crystals (single diastereoisomer, 285 mg, 43%).

\[
\text{mp 90–92 °C; } ^1\text{H NMR (500 MHz, CDCl}_3\text{) } \delta: \text{ 2.44 (2H, br d, } J 2.7, 2 \times \text{OH), 4.24–4.33 (2H, m, 2 } \times \text{HCOH), 6.21–6.34 (2H, 2 } \times \text{PhCH=CH), 6.74 (2H, d, } J 16.0, 2 \times \text{PhCH=CH), 7.21–7.28 (2H, m, PhC(4)H), 7.28–7.36 (4H, m, PhC(3.5)H), 7.36–7.42 (4H, m, PhC(2.6)H). Spectral data in accordance with the literature.}^4
\]

(±)-1,6-Diphenylhexa-1,5-diyne-3,4-diol 18

\[
\text{OH} \quad \text{Ph} \quad \text{OH}
\]

\[\text{±}-18\]

THF (1.4 mL, 17 mmol) was added to a solution of TiCl₄ (0.47 mL, 4.3 mmol) in anhydrous CH₂Cl₂ (8.5 mL) under an N₂ atmosphere at r.t. and was allowed to stir for 30 seconds. Zinc powder (142 mg, 2.18 mmol) was added, and after a further 30 seconds TMEDA (0.95 mL, 6.4 mmol) was added. After a further 30 seconds, a solution of 3-phenylpropionaldehyde (556 mg, 4.3 mmol) in CH₂Cl₂ (4.5 mL) was added and the mixture allowed to stir at r.t. for 1 h. HCl (1 M) was added and the mixture extracted with EtOAc (3 × 20 mL). The combined organic fractions were washed with brine, dried (Na₂SO₄), filtered and concentrated to give a residue, which was purified by Biotage flash silica column chromatography (0→40% EtOAc in hexane) to give a yellow oil, which was further purified by trituration with hexane to give (±)-1,6-diphenylhexa-1,5-diyne-3,4-diol 18 as an off-white solid (single diastereoisomer, 362 mg, 65%).

\[
\text{mp 78–79 °C; } ^1\text{H NMR (500 MHz, CDCl}_3\text{) } \delta: \text{ 2.57 (2H, br s, 2 } \times \text{OH), 4.75 (2H, s, 2 } \times \text{HCOH), 7.26–7.40 (6H, m, ArH), 7.42–7.52 (4H, m, ArH). Spectral data in accordance with the literature.}^4
\]
Synthesis of (±)-Monoalkylated Diols

2-Methoxy-1,2-diphenylethan-1-ol (±)-7 was synthesised according to a literature procedure.1

(±)-2-Isoproxy-1,2-diphenylethan-1-ol 9

A solution of i-PrOH (63 μL, 0.83 mmol) and 2,6-lutidine (105 μL, 0.9 mmol) in anhydrous CH₂Cl₂ (1 mL) was added to a stirred solution of trifluoromethanesulfonic anhydride (151 μL, 0.9 mmol) in anhydrous CH₂Cl₂ (1 mL) at 0 °C under an N₂ atmosphere, and the mixture allowed to warm to r.t. over 30 minutes. This solution was then added dropwise to a solution of (±)-1,2-diphenylethane-1,2-diol 2 (159 mg, 0.75 mmol) and 2,6-lutidine (87 μL, 0.75 mmol) in anhydrous CH₂Cl₂ (2 mL), and the mixture was allowed to stir overnight at r.t. HCl (1 M) was added and the mixture extracted with EtOAc (3 × 20 mL). The combined organic fractions were washed sequentially with HCl (1 M), saturated NaHCO₃ and brine, dried (MgSO₄), filtered and concentrated to give a residue, which was purified by Biotage flash silica column chromatography (0→20% EtOAc in hexane) to give 2-isoproxy-1,2-diphenylethan-1-ol 9 as a colourless oil (71 mg, 37%).

¹H NMR (500 MHz, CDCl₃) δH: 1.12 (3H, d, J 6.2, CH₃), 1.21 (3H, d, J 6.1, CH₃), 3.58 (1H, app sept., J 6.1, CH(CH₃)₂), 3.59 (1H, br s, OH), 4.31 (1H, d, J 8.2, HCO-i-Pr), 4.59 (1H, d, J 8.2, HCOH), 6.97–7.06 (4H, m, ArH), 7.14–7.23 (6H, m, ArH); ¹³C NMR (126 MHz, CDCl₃) δC: 21.4 (CH₃), 23.6 (CH₃), 69.8 (CH(CH₃)₂), 78.7 (HCOH), 84.9 (HCO-i-Pr), 127.4 (2 × ArCH), 127.75 (ArCH), 127.84 (2 × ArCH), 127.9 (3 × ArCH), 128.0 (2 × ArCH), 138.8 (ArC), 139.4 (ArC); IR (neat) νmax cm⁻¹: 3482 (OH), 3030, 2970, 2887, 1454, 1383, 1196, 1123, 1057; HRMS (ESI⁺) C₁₇H₂₀O₂Na [M+Na]⁺, found 279.1349, requires 279.1356 (−2.5 ppm).
Synthesis of (±)-Monoesters and (±)-Diesters

General Procedure

(i-PrCO)₂O (1–1.5 equiv.), i-Pr₂NEt (1–1.5 equiv.) and DMAP (10 mol%) were added to a solution of the appropriate (±)-1,2-diol (1 equiv) in CH₂Cl₂ (0.2 M) at r.t., and the was allowed to stir for 5 h. The mixture was then diluted with EtOAc and washed sequentially with HCl (1 M), saturated NaHCO₃ and brine, dried (Na₂SO₄), filtered and concentrated to give a residue, which was purified by Biotage flash silica column chromatography (0→40% EtOAc in hexane) to give the diester and monoester products, and recovered diol.

The monoester (and diester) products derived from (±)-1,2-diols 2 12–16 and 2-methoxy-1,2-diphenylethanol-1-ol (±)-7 provided spectral data consistent with those previously reported in the literature.¹

(±)-2-Hydroxy-1,2-bis(4-methoxyphenyl)ethyl isobutyrate S1 and (±)-1,2-bis(4-methoxyphenyl)ethane-1,2-diyl bis(2-methylpropanoate) S2

According to the General Procedure, (±)-1,2-bis(4-methoxyphenyl)ethane-1,2-diol 11 (137 mg, 0.5 mmol), (i-PrCO)₂O (83 µL, 0.5 mmol), i-Pr₂NEt (87 µL, 0.5 mmol) and DMAP (6 mg, 0.05 mmol) in CH₂Cl₂ (2.5 ml) gave (±)-2-hydroxy-1,2-bis(4-methoxyphenyl)ethyl isobutyrate S1 as a colourless solid (125 mg, 73%) and (±)-1,2-bis(4-methoxyphenyl)ethane-1,2-diyl bis(2-methylpropanoate) S2 as a colourless solid (21 mg, 10%).

(±)-2-Hydroxy-1,2-bis(4-methoxyphenyl)ethyl isobutyrate S1: mp 88 °C; ¹H NMR (400 MHz, CDCl₃) δp: 1.16 (3H, d, J 7.0, CH₃), 1.18 (3H, d, J 7.0, CH₃), 2.49 (1H, br s, OH), 2.63 (1H, app sept., J 7.0, CH(CH₃)₂), 3.75 (3H, s, OCH₃), 1.36 (3H, s, OCH₃), 4.86 (1H, d, J 7.8, CHOH), 5.75 (1H, d, J 7.8, CHOCH(O)i-Pr), 6.72–6.79 (4H, m, ArH), 7.00–7.08 (4H, m, ArH); ¹³C(¹H) NMR (126 MHz, CDCl₃) δC: 18.98 (CH₃), 19.01 (CH₂), 34.3 (CH₂(CH₃)), 55.3 (OCH₃), 55.34 (OCH₃), 76.9 (CHOH), 79.8 (CHOCH(O)i-Pr), 113.6 (2 × ArH), 113.7 (2 × ArCH), 128.4 (2 × ArCH), 128.6 (2 × ArCH), 128.9 (2 × ArC), 131.4 (ArC), 159.36 (ArCOMe), 159.38 (ArCOMe), 176.3 (C=O); IR (neat) νmax cm⁻¹: 3482 (OH), 2970, 1732 (C=O), 1612, 1514, 1466, 1304, 1250, 1176, 1155, 1034; HRMS (ESI⁺) C₂₀H₂₄O₄Na [M+Na]⁺, found 367.1518, requires 367.1516 (+0.6 ppm).

(±)-1,2-Bis(4-methoxyphenyl)ethane-1,2-diyl bis(2-methylpropanoate) S2: mp 126–128 °C; ¹H NMR (400 MHz, CDCl₃) δp: 1.13 (6H, d, J 7.0, 2 × CH₃), 1.15 (6H, d, J 7.0, 2 × CH₃), 2.55 (2H, app sept., J 7.0, 2 × CH(CH₃)₂), 3.74 (6H, s, 2 × OCH₃), 5.97 (2H, s, 2 × CHOC(O)i-Pr), 6.69–6.75 (4H, m, ArH), 7.00–7.07 (4H, m, ArH); ¹³C(¹H) NMR (126 MHz, CDCl₃) δC: 18.98 (2 × CH₃), 19.01 (2 × CH₂), 34.3 (2 × CH(CH₃)₂), 55.3 (2 × OCH₃), 76.8 (2 × CHOC(O)i-Pr), 113.7 (4 × ArCH), 128.8 (2 × ArC), 128.9 (4 × ArCH), 159.4 (2 × ArCOMe), 175.9 (2 × C=O); IR (neat) νmax cm⁻¹: 2972, 1734 (C=O), 1614, 1514, 1470, 1248, 1177, 1153; HRMS (NSI⁺) C₂₀H₂₄O₄Na [M+Na]⁺, found 437.1931, requires 437.1935 (−0.8 ppm).
(±)-1,2-Bis(4-chlorophenyl)-2-hydroxyethyl isobutyrate **S3** and (±)-1,2-bis(4-chlorophenyl)ethane-1,2-diyl bis(2-methylpropanoate) **S4**

According to the **General Procedure**, (±)-1,2-bis(4-chlorophenyl)ethane-1,2-diol **13** (85 mg, 0.3 mmol), (i-PrCO)₂O (50 μL, 0.3 mmol), i-Pr₂NET (52 μL, 0.3 mmol) and DMAP (4 mg, 0.03 mmol) in CH₂Cl₂ (1.5 mL) gave (±)-2-hydroxy-1,2-bis(4-chlorophenyl)ethyl isobutyrate **S3** as a colourless solid (45 mg, 43%) and (±)-1,2-bis(4-chlorophenyl)ethane-1,2-diyl bis(2-methylpropanoate) **S4** as a colourless oil (30 mg, 23%).

(±)-2-Hydroxy-1,2-bis(4-chlorophenyl)ethyl isobutyrate **S3**: mp 143 °C; ¹H NMR (500 MHz, CDCl₃) δppm: 1.16 (3H, d, J 7.0, CH₃), 1.17 (3H, d, J 7.0, CH₃), 2.55 (1H, d, J 2.5, OH), 2.63 (1H, app sept., J 7.0, CH(CH₃)₂), 4.88 (1H, dd, J 7.4, 2.5, CHOH), 5.74 (1H, d, J 7.4, CH(O)CH₂), 6.99–7.08 (4H, m, ArH), 7.19–7.24 (4H, m, ArH); ¹³C(¹H) NMR (126 MHz, CDCl₃) δppm: 18.95 (CH₃), 19.04 (CH₃), 34.2 (CH(CH₃)₂), 79.0 (CHOH), 76.4 (CH(O)O-Pr), 128.5 (4 × ArCH), 128.7 (4 × ArCH), 134.1 (ArC), 134.3 (ArC), 135.3 (ArCCI), 137.3 (ArCCI), 176.1 (C=O); IR (neat) νmax cm⁻¹: 3460 (O-H), 2972 (C-H), 1708 (C=O); HRMS (ESI⁺) C₁₈H₁₈O₂Cl₃Na [M+Na]+, found 375.0531, requires 375.0525 (+1.6 ppm).

(±)-1,2-Bis(4-chlorophenyl)ethane-1,2-diyl bis(2-methylpropanoate) **S4**: ¹H NMR (400 MHz, CDCl₃) δppm: 1.13 (6H, d, J 7.0, 2 × CH₃), 1.15 (6H, d, J 7.0, 2 × CH₃), 2.57 (2H, app sept., J 7.0, 2 × CH(CH₃)₂), 5.97 (2H, s, 2 × CHOC(O)Pr), 7.01–7.07 (4H, m, ArH), 7.17–7.23 (4H, m, ArH); ¹³C(¹H) NMR (126 MHz, CDCl₃) δppm: 18.92 (2 × CH₃), 18.95 (2 × CH₃), 34.2 (2 × CH(CH₃)₂), 76.1 (2 × HCOCH(O)–Pr), 128.7 (4 × ArCH), 128.8 (4 × ArCH), 134.5 (2 × ArC), 134.8 (2 × ArCCI), 175.6 (2 × C=O); IR (neat) νmax cm⁻¹: 2974 (C-H), 1735 (C=O); HRMS (ESI⁺) C₂₅H₂₅O₄Cl₂Na [M+Na]+, found 445.0940, requires 445.0940 (−0.9 ppm).

(±)-(E,E)-4-Hydroxy-1,6-diphenylhexa-1,5-dien-3-yl isobutyrate **S5** and (±)-(E,E)-1,6-diphenylhexa-1,5-diene-3,4-diyl bis(2-methylpropanoate) **S6**

According to the **General Procedure**, (±)-(E,E)-1,6-diphenylhexa-1,5-diene-3,4-diol **17** (53 mg, 0.2 mmol), (i-PrCO)₂O (50 μL, 0.3 mmol), i-Pr₂NET (52 μL, 0.3 mmol) and DMAP (2 mg, 0.02 mmol) in CH₂Cl₂ (1 mL) gave (±)-(E,E)-4-hydroxy-1,6-diphenylhexa-1,5-dien-3-yl isobutyrate **S5** as a colourless solid (24 mg, 36%) and (±)-(E,E)-1,6-diphenylhexa-1,5-diene-3,4-diyl bis(2-methylpropanoate) **S6** as a colourless solid (44 mg, 54%).

S7
(±)-(E,E)-4-Hydroxy-1,6-diphenylhexa-1,5-dien-3-yl isobutyrate  S5: mp 95–96 °C; 1H NMR (400 MHz, CDCl3) δH: 1.20 (3H, d, J 7.1, CH3), 1.20 (3H, d, J 7.1, CH3), 2.14 (1H, br s, OH), 2.64 (1H, app sept., J 7.1, CH(CH3)2), 4.46–4.54 (1H, m, CHO), 5.49–5.54 (1H, m, CHO(C=O)i-Pr), 6.19–6.29 (2H, m, 2 × PhCH=CH), 6.68–6.77 (2H, m, 2 × PhCH=CH), 7.22–7.28 (2H, m, ArH), 7.29–7.35 (4H, m, ArH), 7.35–7.42 (4H, m, ArH); 13C[1H] NMR (126 MHz, CDCl3) δC: 19.1 (CH3), 19.3 (CH3), 34.3 (CH(CH3)2), 74.1 (HCOH), 76.8 (HCOC(O)i-Pr), 123.8 (PhCH=CH), 126.7 (2 × PhCH=CH), 126.9 (2 × ArCH), 127.4 (PhCH=CH), 128.0 (ArCH), 128.3 (ArCH), 128.7 (4 × ArCH), 132.5 (PhCH=CH), 134.3 (PhCH=CH), 136.2 (ArC), 136.2 (ArC), 176.4 (C=O); IR (neat) νmax cm⁻¹: 3445 (O-H), 3026, 2974, 1732 (C=O), 1495, 1470, 1449, 1387, 1192, 1156; HRMS (NSI⁺) C29H24ONa [M+Na]⁺, found 359.1621, requires 359.1621 (+0.9 ppm).

(±)-(E,E)-1,6-Diphenylhexa-1,5-diene-3,4-diyl bis(2-methylpropanoate) S6: mp 84–85 °C; 1H NMR (400 MHz, CDCl3) δH: 1.19 (6H, d, J 7.0, 2 × CH3), 1.19 (6H, d, J 7.0, 2 × CH3), 2.60 (2H, app sept., J 7.0, 2 × CH(CH3)2), 5.64–5.70 (2H, m, 2 × CHOH(i-Pr)), 6.08–6.18 (2H, m, 2 × PhCH=CH), 6.69 (2H, d, J 16.0, 2 × PhCH=CH), 7.21–7.39 (10H, m, ArH); 13C[1H] NMR (126 MHz, CDCl3) δC: 19.1 (2 × CH3), 19.2 (2 × CH3), 34.3 (2 × CH(CH3)2), 74.3 (2 × CHOC(O)i-Pr), 123.4 (2 × PhCH=CH), 126.8 (4 × ArCH), 128.3 (2 × ArCH), 128.7 (4 × ArCH), 134.7 (2 × PhCH=CH), 136.2 (2 × ArC), 176.0 (2 × C=O); IR (neat) νmax cm⁻¹: 2974 (C-H), 1736 (C=O), 1188, 1150; HRMS (NSI⁺) C26H36O4N [M+NH4]⁺, found 424.2482, requires 424.2482 (–0.1 ppm).

According to the General Procedure, (±)-1,6-diphenylhexa-1,5-diene-3,4-diy-3,4-diylibis(2-methylpropanoate) S7 as a colourless solid (31 mg, 47%) and (±)-1,6-diphenylhexa-1,5-diene-3,4-diylibis(2-methylpropanoate) S8 as a colourless oil (39 mg, 49%).

(±)-4-Hydroxy-1,6-diphenylhexa-1,5-diy-3-yl isobutyrate  S7 and (±)-1,6-diphenylhexa-1,5-diyn-3-yl isobutyrate  S8

According to the General Procedure, (±)-1,6-diphenylhexa-1,5-diyne-3,4-diylibis(2-methylpropanoate) S8: 1H NMR (400 MHz, CDCl3) δH: 1.24 (3H, d, J 7.0, CH3), 1.25 (3H, d, J 7.0, CH3), 2.41 (1H, br d, J 6.7, OH), 2.69 (1H, app sept., J 7.0, CH(CH3)2), 4.81–4.89 (1H, m, CHO), 5.84 (1H, d, J 5.7, CHOCHO(i-Pr), 7.28–7.38 (6H, m, ArH), 7.43–7.51 (4H, m, ArH); 13C[1H] NMR (126 MHz, CDCl3) δC: 18.9 (CH3), 19.3 (CH3), 34.1 (CH(CH3)2), 65.1 (CHOH), 67.1 (CHOCHO(i-Pr), 83.1 (C=O), 85.6 (C=C), 86.9 (C=C), 87.3 (C=C), 121.9 (ArC), 122.1 (ArC), 128.4 (2 × ArCH), 128.5 (2 × ArCH), 129.0 (ArCH), 129.1 (ArCH), 132.0 (2 × ArCH), 132.2 (2 × ArCH), 176.0 (C=O); IR (neat) νmax cm⁻¹: 3447 (O-H), 2974 (C-H), 2236 (C=C), 1740 (C=O), 1491, 1188, 1148; HRMS (ASAP⁺) C28H22O2 [M+H]⁺, found 333.1490, requires 333.1485 (+0.9 ppm).

(±)-1,6-Diphenylhexa-1,5-diyn-3-4-diylibis(2-methylpropanoate) S8: 1H NMR (400 MHz, CDCl3) δH: 1.22 (6H, d, J 7.0, 2 × CH3), 1.22 (6H, d, J 7.0, 2 × CH3), 2.66 (2H, app sept., J 7.0, 2 × CH(CH3)2), 5.97 (2H, s, 2 × CHOC(O)i-Pr), 7.27–7.36 (6H, m, ArH), 7.42–7.47 (4H, m, ArH); 13C[1H] NMR (126 MHz,
CDCl₃ δC: 18.8 (2 × CH₃), 19.2 (2 × CH₃), 34.1 (2 × CH(CH₃)₂), 64.8 (2 × CHOC(O)i-Pr), 82.7 (2 × C≡C), 87.1 (2 × C≡C), 122.0 (2 × ArC), 128.4 (4 × ArCH), 129.1 (2 × ArCH), 132.1 (4 × ArCH), 175.6 (2 × C=O);

IR (neat) νmax cm⁻¹: 2974 (C-H), 2239 (C≡C), 1746 (C=O), 1491, 1194;

HRMS (ESI⁺) C₂₆H₃₄O₄N [M+NH₄]⁺, found 424.2482, requires 424.2482 (+0.1 ppm).

(±)-2-Isopropoxy-1,2-diphenylethyl isobutyrate 10

According to the General Procedure, (±)-2-isopropoxy-1,2-diphenylethan-1-ol 9 (25 mg, 0.1 mmol), (i-PrCO)₂O (17 µL, 0.1 mmol), i-Pr₂NEt (18 µL, 0.1 mmol) and DMAP (1 mg, 0.01 mmol) in CH₂Cl₂ (0.5 mL) gave (±)-2-isopropoxy-1,2-diphenylethyl isobutyrate 10 as a colourless solid (28 mg, 86%).

mp 67 °C; ¹H NMR (500 MHz, CDCl₃) δH: 1.04 (3H, d, J 6.1, CH₃), 1.10 (3H, d, J 6.1, CH₃), 1.16 (3H, d, J 7.0, CH₃), 1.18 (3H, d, J 7.0, CH₃), 2.60 (1H, app sept., J 7.0, C(O)CH(CH₃)₂), 3.50 (1H, app sept., J 6.1, OCH(CH₃)₂), 4.57 (1H, d, J 7.0, CHO-i-Pr), 5.86 (1H, d, J 7.0, CHOC(O)i-Pr), 7.04–7.10 (4H, m, ArH), 7.14–7.20 (6H, m, ArH); ¹³C{¹H} NMR (500 MHz, CDCl₃) δC: 19.0 (CH₃), 19.1 (CH₃), 21.5 (CH₃), 23.4 (CH₃), 34.3 (C(O)CH₃CH₃), 70.4 (OCH₃CH₃), 78.5 (CHOC(O)i-Pr), 82.0 (CHOi-Pr), 127.6 (2 × ArCH), 127.7 (ArCH), 127.8 (ArCH), 127.85 (2 × ArCH), 127.88 (4 × ArCH), 137.7 (ArC), 139.2 (ArC), 176.0 (C=O); IR (neat) νmax cm⁻¹: 2970 (C-H), 1726 (C=O), 1454, 1202, 1159; HRMS (ESI⁺) C₂₆H₃₄O₄Na [M+Na]⁺, found 439.1779, requires 439.1774 (+1.4 ppm).
Sequential Kinetic Resolution of (±)-1,2-Diols

General KR Procedure

(i-PrCO)₂O (1.5 equiv.), i-Pr₂NET (1.6 equiv.) and HyperBTM (1 mol%) were added to a solution of the appropriate (±)-1,2-diol (1 equiv) in CHCl₃ (0.2 m) at 0 °C, and the was allowed to stir for 7 h at 0 °C. The mixture was then diluted with EtOAc and washed sequentially with HCl (1 m), saturated NaHCO₃ and brine, dried (Na₂SO₄), filtered and concentrated to give a residue, which was purified by Biotage flash silica column chromatography (0→40% EtOAc in hexane) to give the diester and monoester products, and recovered diol.

Hydrolysis of diesters

In some instances it was difficult to find conditions to separate the enantiomers of diesters by HPLC using a chiral support, and therefore these products were hydrolysed to the diol prior to HPLC analysis: LiOH+H₂O (3 equiv.) was added to a solution of the diester (1 equiv.) in MeOH (0.3 m) and allowed to stir at 50 °C until completion, based on TLC analysis. The mixture was with EtOAc and washed sequentially with HCl (1 m), saturated NaHCO₃ and brine. The organic layer was dried (Na₂SO₄), filtered and concentrated to give the diol product.

Kinetic Resolution of (±)-1,2-diphenylethane-1,2-diol 2

According to the General Procedure, (±)-1,2-diphenylethane-1,2-diol (64 mg, 0.3 mmol), (i-PrCO)₂O (75 μL, 0.45 mmol), i-Pr₂NET (84 μL, 0.48 mmol) and HyperBTM (1.0 mg, 0.003 mmol) in CHCl₃ (1.5 mL) gave (1R,2R)-1,2-diphenylethane-1,2-diol (5 mg, 8%), (1R,2R)-2-hydroxy-1,2-diphenylethyl isobutyrate (27 mg, 31%) and (1S,2S)-1,2-diphenylethane-1,2-diyl bis(2-methylpropanoate) (53 mg, 50%).

1,2-Diphenylethane-1,2-diol: α²⁰ = +88.0 (c 0.15 in CHCl₃) {Lit.¹ > 99:1 er} α₀²⁰ = +91.2 (c 0.5 in CHCl₃); Chiral HPLC analysis Chiralpak ID (90:10 Hexane:IPA, flow rate 1.0 mLmin⁻¹, 211 nm, 30 °C) tₐ(1R,2R): 13.7 min, tₐ(1S,2S): 16.7 min; 99.94:0.06 er

2-Hydroxy-1,2-diphenylethyl isobutyrate: α₀²⁰ = +2.6 (c 0.5 in CHCl₃); Chiral HPLC analysis Chiralcel OD-H (95:5 hexane:IPA, flow rate 1 mLmin⁻¹, 211 nm, 30 °C) tₐ(1S,2S): 12.3 min, tₐ(1R,2R): 18.5 min, 0.16:99.84 er.

1,2-Diphenylethane-1,2-diyl bis(2-methylpropanoate): α₀²⁰ = +20.2 (c 1.0 in CHCl₃); Following hydrolysis to 1,2-diphenylethene-1,2-diol: Chiral HPLC analysis Chiralpak ID (90:10 Hexane:IPA, flow rate 1.0 mLmin⁻¹, 211 nm, 30 °C) tₐ(1R,2R): 13.8 min, tₐ(1S,2S): 17.2 min; 3.48:96.52 er. 20

Kinetic Resolution of (±)-1,2-bis(4-methoxyphenyl)ethene-1,2-diol 11

According to the General Procedure, (±)-1,2-bis(4-methoxyphenyl)ethane-1,2-diol (82 mg, 0.3 mmol), (i-PrCO)₂O (75 μL, 0.45 mmol), i-Pr₂NET (84 μL, 0.48 mmol) and HyperBTM (1.0 mg, 0.003 mmol) in CHCl₃ (1.5 mL) gave (1R,2R)-1,2-bis(4-methoxyphenyl)ethane-1,2-diol (12 mg, 14%), (1R,2R)-2-hydroxy-1,2-bis(4-methoxyphenyl)ethyl isobutyrate (29 mg, 28%) and (1S,2S)-1,2-bis(4-methoxyphenyl)ethane-1,2-diyl bis(2-methylpropanoate) (55 mg, 44%).
1,2-Bis(4-methoxyphenyl)ethane-1,2-diol: $\alpha_{D}^{20} = +126$ (c 0.25 in CHCl$_3$); Chiral HPLC analysis Chiralpak ID (85:15 hexane:IPA, flow rate 1 mLmin$^{-1}$, 211 nm, 30 °C) $t_{R}$ (1R,2R): 22.2 min, 100:0 er.

2-Hydroxy-1,2-bis(4-methoxyphenyl)ethyl isobutyrate: $\alpha_{D}^{20} = +23.6$ (c 0.5 in CHCl$_3$); Chiral HPLC analysis Chiralpak AD-H (85:15 hexane:IPA, flow rate 1 mLmin$^{-1}$, 211 nm, 30 °C) $t_{R}$ (1R,2R): 15.1 min, $t_{S}$ (1S,2S): 21.9 min, 98.73:1.27 er.

1,2-Bis(4-methoxyphenyl)ethane-1,2-diyi bis(2-methylpropanoate): $\alpha_{D}^{20} = +19.3$ (c 1.0 in CHCl$_3$); Chiral HPLC analysis Chiralpak AD-H (90:10 hexane:IPA, flow rate 1 mLmin$^{-1}$, 211 nm, 30 °C) $t_{R}$ (1S,2S): 9.3 min, $t_{S}$ (1R,2R): 11.5 min, 97.94:2.06 er.

Kinetic Resolution of (±)-1,2-di-p-tolylethane-1,2-diol 12

According to the General Procedure, (±)-1,2-di-p-tolylethane-1,2-diol (73 mg, 0.3 mmol), (i-PrCO)$_2$O (75 μL, 0.45 mmol), i-Pr$_2$NEt (84 μL, 0.48 mmol) and HyperBTM (1.0 mg, 0.003 mmol) in CHCl$_3$ (1.5 mL) gave (1R,2R)-1,2-di-p-tolylethane-1,2-diol (6 mg, 9%), (1R,2R)-2-hydroxy-1,2-di-p-tolylethyl isobutyrate (34 mg, 36%) and (1S,2S)-1,2-di-p-tolylethane-1,2-diyi bis(2-methylpropanoate) (55 mg, 48%).

1,2-Di-p-tolylethane-1,2-diol: $\alpha_{D}^{20} = +120$ (c 0.25 in CHCl$_3$); Chiral HPLC analysis Chiralpak ID (95:5 hexane:IPA, flow rate 1 mLmin$^{-1}$, 211 nm, 30 °C) $t_{R}$ (1R,2R): 24.0 min, 100:0 er.

2-Hydroxy-1,2-di-p-tolylethyl isobutyrate: $\alpha_{D}^{20} = +20.4$ (c 1.0 in CHCl$_3$); Chiral HPLC analysis Chiralpak AS-H (99.5:0.05 hexane:IPA, flow rate 0.7 mLmin$^{-1}$, 211 nm, 30 °C) $t_{R}$ (1S,2S): 18.3 min, $t_{S}$ (1R,2R): 23.6 min, 0.43:99.57 er.

1,2-Di-p-tolylethane-1,2-diyi bis(2-methylpropanoate): $\alpha_{D}^{20} = +14.8$ (c 0.5 in CHCl$_3$); Following hydrolysis to 1,2-di-p-tolylethane-1,2-diol: Chiral HPLC analysis Chiralpak ID (95:5 hexane:IPA, flow rate 1 mLmin$^{-1}$, 211 nm, 30 °C) $t_{R}$ (1R,2R): 24.3 min, $t_{S}$ (1S,2S): 35.5 min, 3.92:96.08 er.

Kinetic Resolution of (±)-1,2-bis(4-chlorophenyl)ethane-1,2-diol 13

According to the General Procedure, (±)-1,2-bis(4-chlorophenyl)ethane-1,2-diol (85 mg, 0.3 mmol), (i-PrCO)$_2$O (75 μL, 0.45 mmol), i-Pr$_2$NEt (84 μL, 0.48 mmol) and HyperBTM (1.0 mg, 0.003 mmol) in CHCl$_3$ (1.5 mL) gave (1R,2R)-1,2-bis(4-chlorophenyl)ethane-1,2-diol (6 mg, 7%), (1R,2R)-2-hydroxy-1,2-bis(4-chlorophenyl)ethyl isobutyrate (38 mg, 36%) and (1S,2S)-1,2-bis(4-chlorophenyl)ethane-1,2-diyi bis(2-methylpropanoate) (61 mg, 48%).

1,2-Bis(4-chlorophenyl)ethane-1,2-diol: $\alpha_{D}^{20} = +112$ (c 0.25 in CHCl$_3$); Chiral HPLC analysis Chiralpak AS-H (98:2 hexane:IPA, flow rate 1 mLmin$^{-1}$, 211 nm, 30 °C) $t_{R}$ (1R,2R): 32.5 min, 100:0 er.

2-Hydroxy-1,2-bis(4-chlorophenyl)ethyl isobutyrate: $\alpha_{D}^{20} = +35.0$ (c 1.0 in CHCl$_3$); Chiral HPLC analysis Chiralpak AS-H (99.5:0.5 hexane:IPA, flow rate 0.7 mLmin$^{-1}$, 211 nm, 30 °C) $t_{R}$ (1R,2R): 33.4 min, $t_{S}$ (1S,2S): 39.7 min, 0.81:99.19 er.

1,2-Bis(4-chlorophenyl)ethane-1,2-diyi bis(2-methylpropanoate): $\alpha_{D}^{20} = +4.8$ (c 0.5 in CHCl$_3$); Following hydrolysis to 1,2-bis(4-chlorophenyl)ethane-1,2-diol: Chiral HPLC analysis Chiralpak AS-H
Kinetische Auflösung des (±)-4,4'-[1,2-dihydroxyethan-1,2-diyl]dibenzoesäure 14

Nach der allgemeinen Verfahrensbeschreibung, (±)-4,4'-[1,2-dihydroxyethan-1,2-diyl]dibenzoesäure (99 mg, 0,3 mmol), (i-PrCO)2O (75 µL, 0,45 mmol), i-Pr2NEt (84 µL, 0,48 mmol) und HyperBTM (1,0 mg, 0,003 mmol) in CHCl3 (1,5 mL) gaben (1R,2R)-4,4'-[1,2-dihydroxyethan-1,2-diyl]dibenzoesäure (10 mg, 10%), (1R,2R)-4,4'-[1,2-dihydroxyethan-1,2-diyl]dibenzoesäure (41 mg, 34%) und (1S,2S)-4,4'-[1,2-dihydroxyethan-1,2-diyl]dibenzoesäure (67 mg, 48%).

Dimethyl 4,4'-[1,2-dihydroxyethan-1,2-diyl]dibenzoesäure: \( \alpha_{D}^{20} = +106 \) (c 0,05 in CHCl3); Chiral HPLC Analysis Chiralpak AS-H (85:15 hexane:IPA, flow rate 1 mLmin\(^{-1} \), 211 nm, 30 °C) \( t_{R} \) (1S,2S): 18,0 min, \( t_{R} \) (1R,2R): 26,8 min, 12,84:87,16 er.

Dimethyl 4,4'-[1,2-dihydroxyethan-1,2-diyl]dibenzoesäure: \( \alpha_{D}^{20} = +33,4 \) (c 1,0 in CHCl3); Chiral HPLC Analysis Chiralpak AD-H (90:10 hexane:IPA, flow rate 1 mLmin\(^{-1} \), 211 nm, 30 °C) \( t_{R} \) (1R,2R): 28,6 min, \( t_{R} \) (1S,2S): 41,1 min, 93,98:6,02 er.

Dimethyl 4,4'-[1,2-dihydroxyethan-1,2-diyl]dibenzoesäure: \( \alpha_{D}^{20} = -1,5 \) (c 1,0 in CHCl3); Chiral HPLC Analysis Chiralpak ID (95:5 hexane:IPA, flow rate 1 mLmin\(^{-1} \), 211 nm, 30 °C) \( t_{R} \) (1S,2S): 24,8 min, \( t_{R} \) (1R,2R): 44,6 min, 90,11:9,89 er.

Kinetische Auflösung des (±)-1,2-bis-[4-(trifluoromethyl)phenyl]ethan-1,2-diol 15

Nach der allgemeinen Verfahrensbeschreibung, (±)-1,2-bis-[4-(trifluoromethyl)phenyl]ethan-1,2-diol (105 mg, 0,3 mmol), (i-PrCO)2O (75 µL, 0,45 mmol), i-Pr2NEt (84 µL, 0,48 mmol) und HyperBTM (1,0 mg, 0,003 mmol) in CHCl3 (1,5 mL) gaben (1R,2R)-1,2-bis-[4-(trifluoromethyl)phenyl]ethan-1,2-diol (7 mg, 7%), (1R,2R)-2-hydroxy-1,2-bis-[4-(trifluoromethyl)phenyl]ethylester (48 mg, 38%) und (1S,2S)-1,2-bis-[4-(trifluoromethyl)phenyl]ethan-1,2-diyl bis[2-methylpropanoate] (74 mg, 51%).

1,2-Bis-[4-(trifluoromethyl)phenyl]ethan-1,2-diol: \( \alpha_{D}^{20} = +41,1 \) (c 1,0 in CHCl3) (Lit.1 (99:1 er) \( \alpha_{D}^{20} = +43,6 \) (c 1,01 in CHCl3)); Chiral HPLC Analysis Chiralpak AS-H (97:3 hexane:IPA, flow rate 1 mLmin\(^{-1} \), 211 nm, 30 °C) \( t_{R} \) (1R,2R): 11,6 min, 100:0 er.

2-Hydroxy-1,2-bis-[4-(trifluoromethyl)phenyl]ethylester isobutyrate: \( \alpha_{D}^{20} = -1,5 \) (c 1,0 in CHCl3); Chiral HPLC Analysis Chiralcel OJ-H (95:5 hexane:IPA, flow rate 1 mLmin\(^{-1} \), 211 nm, 30 °C) \( t_{R} \) (1R,2R): 8,2 min, \( t_{R} \) (1S,2S): 22,9 min, 1,47:98,53 er.

1,2-Bis-[4-(trifluoromethyl)phenyl]ethan-1,2-diyl bis[2-methylpropanoate]: \( \alpha_{D}^{20} = +11,0 \) (c 0,1 in CHCl3); Following hydrolysis to 1,2-bis-[4-(trifluoromethyl)phenyl]ethan-1,2-diol: Chiral HPLC Analysis Chiralpak AS-H (97:3 hexane:IPA, flow rate 1 mLmin\(^{-1} \), 211 nm, 30 °C) \( t_{R} \) (1R,2R): 12,0 min, \( t_{R} \) (1S,2S): 16,1 min, 4,00:96,00 er.
Kinetic Resolution of (±)-1,2-di(naphthalen-1-yl)ethane-1,2-diol 16

According to the General Procedure, (±)-1,2-di(naphthalen-1-yl)ethane-1,2-diol (94 mg, 0.3 mmol), (i-PrCO)₂O (75 µL, 0.45 mmol), i-Pr₂NEt (84 µL, 0.48 mmol) and HyperBTM (1.0 mg, 0.003 mmol) in CHCl₃ (1.5 mL) gave (1R,2R)-1,2-di(naphthalen-1-yl)ethane-1,2-diol (12 mg, 13%), (1R,2R)-2-hydroxy-1,2-di(naphthalen-1-yl)ethyl isobutyrate (41 mg, 35%) and (1S,2S)-1,2-di(naphthalen-1-yl)ethane-1,2-diyl bis(2-methylpropanoate) (68 mg, 50%).

1,2-Di(naphthalen-1-yl)ethane-1,2-diol: α_D^{20} = −9.6 (c 0.25 in CHCl₃); Chiral HPLC analysis Chiralcel OJ-H (80:20 hexane:IPA, flow rate 1.5 mL min⁻¹, 211 nm, 30 °C) t_R (1S,2S): 16.2 min, t_R (1R,2R): 21.5 min, 0.33:99.67 er.

2-Hydroxy-1,2-di(naphthalen-1-yl)ethyl isobutyrate: α_D^{20} = +57.2 (c 1.0 in CHCl₃); Chiral HPLC analysis Chiralcel OD-H (90:10 hexane:IPA, flow rate 1.5 mL min⁻¹, 211 nm, 30 °C) t_R (1S,2S): 9.6 min, t_R (1R,2R): 14.9 min, 0.49:99.51 er.

1,2-Di(naphthalen-1-yl)ethane-1,2-diyl bis(2-methylpropanoate): α_D^{20} = −67.5 (c 1.0 in CHCl₃); Chiral HPLC analysis Chiralcel OD-H (99:1 hexane:IPA, flow rate 1 mL min⁻¹, 211 nm, 30 °C) t_R (1S,2S): 6.4 min, t_R (1R,2R): 9.8 min, 95.09:4.91 er.

Kinetic Resolution of (±)-(E,E)-1,6-diphenylhexa-1,5-diene-3,4-diol 17

According to the General Procedure, (±)-(E,E)-1,6-diphenylhexa-1,5-diene-3,4-diol (79 mg, 0.3 mmol), (i-PrCO)₂O (75 µL, 0.45 mmol), i-Pr₂NEt (84 µL, 0.48 mmol) and HyperBTM (1.0 mg, 0.003 mmol) in CHCl₃ (1.5 mL) gave (3R,4R)-(E,E)-4-hydroxy-1,6-diphenylhexa-1,5-dien-3-yl isobutyrate (49 mg, 49%) and (3S,4S)-(E,E)-1,6-diphenylhexa-1,5-diene-3,4-diyl bis(2-methylpropanoate) (58 mg, 48%).

(E,E)-4-Hydroxy-1,6-diphenylhexa-1,5-dien-3-yl isobutyrate: α_D^{20} = +0.9 (c 1.0 in CHCl₃); Chiral HPLC analysis Chiralpak IC (95:5 hexane:IPA, flow rate 1 mL min⁻¹, 211 nm, 30 °C) t_R (3S,4S): 18.8 min, t_R (3R,4R): 23.6 min, 22.74:77.26 er.

(E,E)-1,6-Diphenylhexa-1,5-diene-3,4-diyl bis(2-methylpropanoate): α_D^{20} = +10.8 (c 1.0 in CHCl₃); Chiral HPLC analysis Following hydrolysis to (E,E)-1,6-diphenylhexa-1,5-diene-3,4-diol: Chiralpak ID (85:15 hexane:IPA, flow rate 1 mL min⁻¹, 211 nm, 30 °C) t_R (3S,4S): 13.2 min, t_R (3R,4R): 15.6 min, 79.21:20.79 er.

Kinetic Resolution of (±)-1,6-diphenylhexa-1,5-diyne-3,4-diol 18

According to the General Procedure, (±)-1,6-diphenylhexa-1,5-diyne-3,4-diol (78 mg, 0.3 mmol), (i-PrCO)₂O (75 µL, 0.45 mmol), i-Pr₂NEt (84 µL, 0.48 mmol) and HyperBTM (1.0 mg, 0.003 mmol) in CHCl₃ (1.5 mL) gave (3S,4S)-1,6-diphenylhexa-1,5-diyne-3-yl isobutyrate (41 mg, 41%) and (3S,4S)-1,6-diphenylhexa-1,5-diyne-3,4-diyl bis(2-methylpropanoate) (60 mg, 50%).

1,6-Diphenylhexa-1,5-diyne-3,4-diol: α_D^{20} = +97.2 (c 0.25 in CHCl₃); Chiral HPLC analysis Chiralpak IC (90:10 hexane:IPA, flow rate 1 mL min⁻¹, 211 nm, 30 °C) t_R (3R,4R): 11.3 min, t_R (3S,4S): 18.1 min, 99.74:0.26 er.
4-Hydroxy-1,6-diphenylhexa-1,5-diyne-3-yl isobutyrate: $\alpha_D^{20} = -12.2$ (c 1.0 in CHCl$_3$); Chiral HPLC analysis Chiralpak IC (95:5 hexane:IPA, flow rate 1 mL/min$^{-1}$, 211 nm, 30 °C) $t_\text{R}$ (3$R$,4$R$): 13.8 min, $t_\text{S}$ (3$S$,4$S$): 38.9 min, 85.44:14.56 er.

1,6-Diphenylhexa-1,5-diyn-3,4-diyl bis(2-methylpropanoate): $\alpha_D^{20} = +39.8$ (c 0.5 in CHCl$_3$); Following hydrolysis to (E,E)-1,6-diphenylhexa-1,5-diene-3,4-diol: Chiral HPLC analysis Chiralpak IC (90:10 hexane:IPA, flow rate 1 mL/min$^{-1}$, 211 nm, 30 °C) $t_\text{R}$ (3$R$,4$R$): 11.3 min, $t_\text{S}$ (3$S$,4$S$): 18.0 min, 16.55:83.45 er.

Kinetic Resolution of (±)-2-methoxy-1,2-diphenylethanol-1-ol 7

According to a modification of the General Procedure, (±)-2-methoxy-1,2-diphenylethan-1-ol (68 mg, 0.3 mmol), (i-PrCO)$_2$O (27 µL, 0.165 mmol), i-Pr$_3$NEt (31 µL, 0.18 mmol) and HyperBTM (1.0 mg, 0.003 mmol) in CHCl$_3$ (1.5 mL) gave (1$R$,2$R$)-2-methoxy-1,2-diphenylethanol-1-ol (35 mg, 52%), (1$S$,2$S$)-2-methoxy-1,2-diphenylethyl isobutyrate (36 mg, 41%).

2-Methoxy-1,2-diphenylethanol-1-ol: $\alpha_D^{20} = +29.5$ (c 1.0 in CHCl$_3$) {Lit.$^{10}$ (96:4 er) $\alpha_D^{21} = +53.3$ (c 1.5 in CHCl$_3$)}; Chiral HPLC analysis Chiralcel OD-H (95:5 hexane:IPA, flow rate 1 mL/min$^{-1}$, 211 nm, 30 °C) $t_\text{R}$ (1$S$,2$S$): 7.7 min, $t_\text{R}$ (1$R$,2$R$): 8.7 min, 21.73:78.27 er.

2-Methoxy-1,2-diphenylethyl isobutyrate: $\alpha_D^{20} = +27.0$ (c 1.0 in CHCl$_3$) {Lit.$^{1}$ (54:46 er) $\alpha_D^{21} = +1.6$ (c 0.47 in CHCl$_3$)}; Chiral HPLC analysis Chiralcel OD-H (98:2 hexane:IPA, flow rate 1 mL/min$^{-1}$, 211 nm, 30 °C) $t_\text{R}$ (1$S$,2$S$): 4.4 min, $t_\text{R}$ (1$R$,2$R$): 4.9 min, 81.95:18.05 er.

Kinetic Resolution of (±)-2-isopropoxy-1,2-diphenylethanol-1-ol 9

According to a modification of the General Procedure, (±)-2-isopropoxy-1,2-diphenylethanol-1-ol (56 mg, 0.22 mmol), (i-PrCO)$_2$O (20 µL, 0.12 mmol), i-Pr$_3$NET (23 µL, 0.13 mmol) and HyperBTM (0.7 mg, 0.002 mmol) in CHCl$_3$ (1.1 mL) gave (1$R$,2$R$)-2-isopropoxy-1,2-diphenylethanol-1-ol (43 mg, 77%), (1$S$,2$S$)-2-isopropoxy-1,2-diphenylethyl isobutyrate (13 mg, 18%).

2-Isopropoxy-1,2-diphenylethanol-1-ol: $\alpha_D^{20} = +4.8$ (c 1.0 in CHCl$_3$); Chiral HPLC analysis Chiralcel OD-H (95:5 hexane:IPA, flow rate 1 mL/min$^{-1}$, 211 nm, 30 °C) $t_\text{R}$ (1$S$,2$S$): 4.9 min, $t_\text{R}$ (1$R$,2$R$): 5.5 min, 38.58:61.42 er.

2-Isopropoxy-1,2-diphenylethyl isobutyrate: $\alpha_D^{20} = +39.2$ (c 1.0 in CHCl$_3$); Following hydrolysis to 2-isopropoxy-1,2-diphenylethanol-1-ol: Chiral HPLC analysis Chiralcel OD-H (95:5 hexane:IPA, flow rate 1 mL/min$^{-1}$, 211 nm, 30 °C) $t_\text{R}$ (1$S$,2$S$): 4.9 min, $t_\text{R}$ (1$R$,2$R$): 5.5 min, 96.41:3.59 er.
Kinetic Resolutions To Obtain \( s \) Values For Each KR Step

Kinetic Resolution of \((\pm)-1,2\)-diphenylethane-1,2-diol to low conv. using 5 mol% catalyst at r.t.

According to a modification of the General Procedure, \((\pm)-1,2\)-diphenylethane-1,2-diol (64 mg, 0.3 mmol), \((i-PrCO)\_2\)O (27 \( \mu \)L, 0.165 mmol), \(i-Pr\_2\)NET (29 \( \mu \)L, 0.165 mmol) and HyperBTM (4.6 mg, 0.015 mmol) in CHCl\(_3\) (1.5 mL) at room temperature gave \((1R,2R)-1,2\)-diphenylethane-1,2-diol (33 mg, 52%), \((1S,2S)-2\)-hydroxy-1,2-diphenylethyl isobutyrate (34 mg, 40%) and \((1S,2S)-1,2\)-diphenylethane-1,2-diyl bis(2-methylpropanoate) (4 mg, 4%).

\(^1\)H NMR spectroscopic analysis of crude reaction product mixture indicated a ratio of dipi:monoester:diester of 34:16:5.

1,2-Diphenylethane-1,2-diol: Chiral HPLC analysis Chiralpak ID (90:10 Hexane:IPA, flow rate 1.0 mLmin\(^{-1}\), 211 nm, 30 °C) \( t\_A(1R,2R) \): 13.7 min, \( t\_A(1S,2S) \): 17.0 min; 83.41:16.59 er.

2-Hydroxy-1,2-diphenylethyl isobutyrate: Chiral HPLC analysis Chiralcel OD-H (95:5 hexane:IPA, flow rate 1 mLmin\(^{-1}\), 211 nm, 30 °C) \( t\_A(1S,2S) \): 12.2 min, \( t\_A(1R,2R) \): 17.8 min, 87.57:12.43 er.

1,2-Diphenylethane-1,2-diyl bis(2-methylpropanoate): Following hydrolysis to 1,2-diphenylethane-1,2-diol: Chiral HPLC analysis Chiralpak ID (90:10 Hexane:IPA, flow rate 1.0 mLmin\(^{-1}\), 211 nm, 30 °C) \( t\_A(1R,2R) \): 13.8 min, \( t\_A(1S,2S) \): 17.3 min; 0.11:99.89 er.

\( s \) Value Calculation: Using the er of the diol (83.41:16.59) and conversion by \(^1\)H NMR spectroscopy (46%) gives an \( s \) value of 16.

Kinetic Resolution of \((\pm)-1,2\)-diphenylethane-1,2-diol to low conv. using 1 mol% catalyst at 0 °C

According to a modification of the General Procedure, \((\pm)-1,2\)-diphenylethane-1,2-diol (64 mg, 0.3 mmol), \((i-PrCO)\_2\)O (27 \( \mu \)L, 0.165 mmol), \(i-Pr\_2\)NET (29 \( \mu \)L, 0.165 mmol) and HyperBTM (1.0 mg, 0.03 mmol) in CHCl\(_3\) (1.5 mL) gave \((1R,2R)-1,2\)-diphenylethane-1,2-diol (34 mg, 53%), \((1S,2S)-2\)-hydroxy-1,2-diphenylethyl isobutyrate (30 mg, 35%) and \((1S,2S)-1,2\)-diphenylethane-1,2-diyl bis(2-methylpropanoate) (4 mg, 4%).

\(^1\)H NMR spectroscopic analysis of crude reaction product mixture indicated a ratio of dipi:monoester:diester of 56:38:5.

1,2-Diphenylethane-1,2-diol: Chiral HPLC analysis Chiralpak ID (90:10 Hexane:IPA, flow rate 1.0 mLmin\(^{-1}\), 211 nm, 30 °C) \( t\_A(1R,2R) \): 13.7 min, \( t\_A(1S,2S) \): 17.0 min; 84.94:15.06 er.

2-Hydroxy-1,2-diphenylethyl isobutyrate: Chiral HPLC analysis Chiralcel OD-H (95:5 hexane:IPA, flow rate 1 mLmin\(^{-1}\), 211 nm, 30 °C) \( t\_A(1S,2S) \): 12.1 min, \( t\_A(1R,2R) \): 17.9 min, 93.62:6.38 er.

1,2-Diphenylethane-1,2-diyl bis(2-methylpropanoate): Following hydrolysis to 1,2-diphenylethane-1,2-diol: Chiral HPLC analysis Chiralpak ID (90:10 Hexane:IPA, flow rate 1.0 mLmin\(^{-1}\), 211 nm, 30 °C) \( t\_A(1R,2R) \): 13.7 min, \( t\_A(1S,2S) \): 17.1 min; 0.08:99.92 er.

\( s \) Value Calculation: Using the er of the diol (84.94:15.06) and conversion by \(^1\)H NMR spectroscopy (44%) gives an \( s \) value of 36.
Kinetic Resolution of (±)-2-hydroxy-1,2-diphenylethyl isobutyrate using 5 mol% catalyst at r.t.

According to a modification of the General Procedure, (±)-2-hydroxy-1,2-diphenylethyl isobutyrate (56 mg, 0.2 mmol), (i-PrCO)₂O (18 μL, 0.11 mmol), i-Pr₂NEt (19 μL, 0.11 mmol) and HyperBTM (3.1 mg, 0.01 mmol) in CHCl₃ (1 mL) at room temperature gave (1R,2R)-2-hydroxy-1,2-diphenylethyl isobutyrate (29 mg, 51%) and (1S,2R)-1,2-diphenylethane-1,2-diyl bis(2-methylpropanoate) (28 mg, 40%).

¹H NMR spectroscopic analysis of crude reaction product mixture indicated a ratio of monoester:diester of 55:45.

2-Hydroxy-1,2-diphenylethyl isobutyrate: Chiral HPLC analysis Chiralcel OD-H (95:5 hexane:IPA, flow rate 1 mLmin⁻¹, 211 nm, 30 °C) tᵣ (15,2S): 12.2 min, tₛ (1R,2R): 18.2 min, 12.17:87.83 er.

1,2-Diphenylethane-1,2-diyl bis(2-methylpropanoate): Following hydrolysis to 1,2-diphenylethane-1,2-diol: Chiral HPLC analysis Chiralpak ID (90:10 Hexane:IPA, flow rate 1.0 mLmin⁻¹, 211 nm, 30 °C) tᵣ (1R,2R): 13.8 min, tₛ (15,2S): 17.3 min; 3.61:96.39 er.

s Value Calculation: Using the er of the monoester (12.17:87.83) and diester (3.61:96.39) gives an s value of 61, which was rounded to 60 to account for analytical error associated with large s values.⁸

Kinetic Resolution of (±)-2-hydroxy-1,2-diphenylethyl isobutyrate using 1 mol% catalyst at 0 °C

According to a modification of the General Procedure, (±)-2-hydroxy-1,2-diphenylethyl isobutyrate (85 mg, 0.3 mmol), (i-PrCO)₂O (27 μL, 0.165 mmol), i-Pr₂NEt (29 μL, 0.165 mmol) and HyperBTM (1.0 mg, 0.015 mmol) in CHCl₃ (1.5 mL) give (1R,2R)-2-hydroxy-1,2-diphenylethyl isobutyrate (41 mg, 48%) and (1S,2S)-1,2-diphenylethane-1,2-diyl bis(2-methylpropanoate) (45 mg, 43%).

¹H NMR spectroscopic analysis of crude reaction product mixture indicated a ratio of monoester:diester of 50:50.

2-Hydroxy-1,2-diphenylethyl isobutyrate: Chiral HPLC analysis Chiralcel OD-H (95:5 hexane:IPA, flow rate 1 mLmin⁻¹, 211 nm, 30 °C) tᵣ (15,2S): 12.2 min, tₛ (1R,2R): 18.4 min, 4.85:95.15 er.

1,2-Diphenylethane-1,2-diyl bis(2-methylpropanoate): Following hydrolysis to 1,2-diphenylethane-1,2-diol: Chiral HPLC analysis Chiralpak ID (90:10 Hexane:IPA, flow rate 1.0 mLmin⁻¹, 211 nm, 30 °C) tᵣ (1R,2R): 13.7 min, tₛ (15,2S): 17.2 min; 3.95:96.05 er.

s Value Calculation: Using the er of the monoester (4.85:95.15) and diester (3.95:96.05) gives an s value of 76, which was rounded to 80 to account for analytical error associated with large s values.⁸
Simulated Data for Sequential Kinetic Resolution

Using the SeKiRe software developed by Faber,\(^9\) values for each rate constant \((k_1-k_4, \text{ see Scheme 1 of manuscript})\) were input, with the \(s\) values calculated for each KR step at room temperature used to set the values of \(k_1/k_3\) as 16 and \(k_2/k_4\) as 60. The magnitude of \(k_3/k_2\) was then varied until the simulated data provided a good match for the relative proportions and \(er\) values of the diol, monoester and diester obtained in the three KRs conducted at room temperature (see data in Table 1 of manuscript). The best overall match was obtained when \(k_1 = 24; k_3 = 1.5; k_2 = 3.5; k_4 = 0.0583\), indicating the first KR process is significantly faster than the second. The following data was used to generate Figure 1 of the manuscript:

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(1R,2R)-1,2-Diphenylethane-1,2-diol

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(1R,2R)-2-Hydroxy-1,2-diphenylethyl isobutyrate

$\text{Ph} \quad \text{OH}$

Chiralcel OD-H (95:5 hexane:IPA, flow rate 1 mLmin$^{-1}$, 211 nm, 30 °C) $t_R(1S,2S)$: 12.3 min, $t_R(1R,2R)$: 18.5 min, 0.16:99.84 er.
(1S,2S)-1,2-Diphenylethane-1,2-diol

1,2-Diphenylethane-1,2-diyl bis(2-methylpropanoate) could not be separated by HPLC so was hydrolysed to give 1,2-diphenylethane-1,2-diol. Chiralpak ID (90:10 Hexane:IPA, flow rate 1.0 mLmin\(^{-1}\), 211 nm, 30 °C) \(t_{R}(1R,2R): 13.8\) min, \(t_{R}(1S,2S): 17.2\) min; 3.48:96.52 er.
(1R,2R)-1,2-Bis(4-methoxyphenyl)ethane-1,2-diol

Chiralpak ID (85:15 hexane:IPA, flow rate 1 mL/min, 211 nm, 30 °C) \( t_R (1R,2R) \): 22.2 min, 100:0 er.

(1R,2R)-2-Hydroxy-1,2-bis(4-methoxyphenyl)ethyl isobutyrate

Chiralpak AD-H (85:15 hexane:IPA, flow rate 1 mL/min, 211 nm, 30 °C) \( t_R (1R,2R) \): 15.1 min, \( t_R (1S,2S) \): 21.9 min, 98.73:1.27 er.
(1S,2S)-1,2-Bis(4-methoxyphenyl)ethane-1,2-diyl bis(2-methylpropanoate)

Chiralpak AD-H (90:10 hexane:IPA, flow rate 1 mL/min, 211 nm, 30 °C) $t_R(1S,2S)$: 9.3 min, $t_R(1R,2R)$: 11.5 min, 97.94:2.06 er.
(1R,2R)-1,2-Di-p-tolylethane-1,2-diol

Chiralpak ID (95:5 hexane:IPA, flow rate 1 mL min⁻¹, 211 nm, 30 °C)

$t_R$ (1R,2R): 24.0 min, 100:0 er.

(1R,2R)-2-Hydroxy-1,2-di-p-tolylethyl isobutyrate

Chiralpak AS-H (99.5:0.05 hexane:IPA, flow rate 0.7 mL min⁻¹, 211 nm, 30 °C)

$t_R$ (1S,2S): 18.3 min, $t_R$ (1R,2R): 23.6 min, 0.43:99.57 er.
(1S,2S)-1,2-Di-p-tolylethane-1,2-diol

1,2-Di-p-tolylethane-1,2-diyl bis(2-methylpropanoate) could not be separated by HPLC so was hydrolysed to give di-p-tolylethane-1,2-diol

Chiralpak ID (95:5 hexane:IPA, flow rate 1 mLmin⁻¹, 211 nm, 30 °C)

$E(1R,2R): 24.3 \text{ min, } E(1S,2S): 35.5 \text{ min, } 3.92:96.08 \text{ er.}$
(1R,2R)-1,2-Bis(4-chlorophenyl)ethane-1,2-diol

Chiralpak AS-H (98:2 hexane:IPA, flow rate 1 mLmin⁻¹, 211 nm, 30 °C) 

$t_r (1R,2R)$: 32.5 min, 100:0 er.

(1R,2R)-1,2-Bis(4-chlorophenyl)ethane-1,2-diol

Chiralpak AS-H (99.5:0.5 hexane:IPA, flow rate 0.7 mLmin⁻¹, 211 nm, 30 °C) 

$t_r (1R,2R)$: 33.4 min, $t_r (1S,2S)$: 39.7 min, 0.81:99.19 er.
(1S,2S)-1,2-Bis(4-chlorophenyl)ethane-1,2-diol

1,2-Bis(4-chlorophenyl)ethane-1,2-diyl bis(2-methylpropanoate) could not be separated by HPLC so was hydrolysed to give 1,2-bis(4-chlorophenyl)ethane-1,2-diol

Chiralpak AS-H (98:2 hexane:IPA, flow rate 1 mL min⁻¹, 211 nm, 30 °C)

$t_R (1R,2R)$: 33.5 min, $t_R (1S,2S)$: 43.0 min, 5.29:94.71 er.
(1R,2R)-Dimethyl 4,4′-(1,2-dihydroxyethane-1,2-diyl)dibenzoate

Chiralpak AS-H (85:15 hexane:IPA, flow rate 1 mL min⁻¹, 211 nm, 30 °C) tᵣ (1S,2S): 18.0 min, tᵣ (1R,2R): 26.8 min, 12.84:87.16 er.

(1R,2R)-Dimethyl 4,4′-(1,2-dihydroxyethane-1,2-diyl)dibenzoate

Chiralpak AD-H (90:10 hexane:IPA, flow rate 1 mL min⁻¹, 211 nm, 30 °C) tᵣ (1R,2R): 28.6 min, tᵣ (1S,2S): 41.1 min, 93.98:6.02 er.
(15,2S)-Dimethyl 4,4'-[(1,2-dihydroxyethane-1,2-diyl)dibenzoate

Chiralpak ID (95:5 hexane:IPA, flow rate 1 mLmin⁻¹, 211 nm, 30 °C) $t_R$ (15,2S): 24.8 min, $t_R$ (1R,2R): 44.6 min, 90.11:9.89 er.
(1R,2R)-1,2-Bis(4-(trifluoromethyl)phenyl)ethane-1,2-diol

Chiralpak AS-H (97:3 hexane:IPA, flow rate 1 mLmin⁻¹, 211 nm, 30 °C) tᵣ (1R,2R): 11.6 min, 100:0 er.

(1R,2R)-2-Hydroxy-1,2-bis(4-(trifluoromethyl)phenyl)ethyl isobutyrate

Chiralcel OJ-H (95:5 hexane:IPA, flow rate 1 mLmin⁻¹, 211 nm, 30 °C) tᵣ (1R,2R): 8.2 min, tᵣ (1S,2S): 22.9 min, 1.47:98.53 er.
(1S,2S)-1,2-Bis(4-(trifluoromethyl)phenyl)ethane-1,2-diol

1,2-Bis(4-(trifluoromethyl)phenyl)ethane-1,2-diol bis(2-methylpropanoate) could not be separated by HPLC so was hydrolysed to 1,2-bis(4-(trifluoromethyl)phenyl)ethane-1,2-diol Chiralpak AS-H (97:3 hexane:IPA, flow rate 1 mLmin⁻¹, 211 nm, 30 °C) t₁R (1R,2R): 12.0 min, t₁S (1S,2S): 16.1 min, 4.00:96.00 er.
(1R,2R)-1,2-Bis(4-(trifluoromethyl)phenyl)ethane-1,2-diol

Chiral OJ-H (80:20 hexane:IPA, flow rate 1.5 mLmin⁻¹, 211 nm, 30 °C) t_R (1S,2S): 16.2 min, t_R (1R,2R): 21.5 min, 0.33:99.67 er.

(1R,2R)-2-Hydroxy-1,2-di(naphthalen-1-yl)ethyl isobutyrate

Chiral OD-H (90:10 hexane:IPA, flow rate 1.5 mLmin⁻¹, 211 nm, 30 °C) t_R (1S,2S): 9.6 min, t_R (1R,2R): 14.9 min, 0.49:99.51 er.
(1S,2S)-1,2-Di(naphthalen-1-yl)ethane-1,2-diyl bis(2-methylpropanoate)

Chiralcel OD-H (99:1 hexane:IPA, flow rate 1 mL min⁻¹, 211 nm, 30 °C) tᵣ (1S,2S): 6.4 min, tᵣ (1R,2R): 9.8 min, 95.09:4.91 er.
(3R,4R)-(E,E)-4-Hydroxy-1,6-diphenylhexa-1,5-dien-3-yl isobutyrate

Chiralpak IC (95:5 hexane:IPA, flow rate 1 mL min⁻¹, 211 nm, 30 °C) \( t_R \) (3S,4S): 18.8 min, \( t_R \) (3R,4R): 23.6 min, 22.74:77.26 er.

(3S,4S)-(E,E)-1,6-Diphenylhexa-1,5-diene-3,4-diol

(E,E)-1,6-Diphenylhexa-1,5-diene-3,4-diyl bis(2-methylpropanoate) could not be separated by HPLC so was hydrolysed to (E,E)-1,6-diphenylhexa-1,5-diene-3,4-diol: Chiralpak ID (85:15 hexane:IPA, flow rate 1 mL min⁻¹, 211 nm, 30 °C) \( t_R \) (3S,4S): 13.2 min, \( t_R \) (3R,4R): 15.6 min, 79.21:20.79 er.
(3R,4R)-1,6-Diphenylhexa-1,5-diyne-3,4-diol

Chiralpak IC (90:10 hexane:IPA, flow rate 1 mL min⁻¹, 211 nm, 30 °C) tₚₐ (3R,4R): 11.3 min, tₚₐ (3S,4S): 18.1 min, 99.74:0.26 er.
(3R,4R)-1,6-Diphenylhexa-1,5-diyne-3,4-diol

\[
\begin{align*}
\text{Chiralpak IC (95:5 hexane:IPA, flow rate 1 mLmin}^{-1}, 211 \text{ nm, } 30 \text{ °C) } t_R \\
(3R,4R): 13.8 \text{ min, } t_R (3S,4S): 38.9 \text{ min, } 85.44:14.56 \text{ er.}
\end{align*}
\]

(3S,4S)-1,6-Diphenylhexa-1,5-diyne-3,4-diol

1,6-Diphenylhexa-1,5-diyne-3,4-diyl bis(2-methylpropanoate) could not be separated by HPLC so was hydrolysed to (E,E)-1,6-diphenylhexa-1,5-diene-3,4-diol: Chiralpak IC (90:10 hexane:IPA, flow rate 1 mLmin\(^{-1}\), 211 nm, 30 °C) \( t_R (3R,4R): 11.3 \text{ min, } t_R (3S,4S): 18.0 \text{ min, } 16.55:83.45 \text{ er.} \)

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(1R,2R)-2-Methoxy-1,2-diphenylethan-1-ol

Chiralcel OD-H (95:5 hexane:IPA, flow rate 1 mL min⁻¹, 211 nm, 30 °C) tᵣ (1S,2S): 7.7 min, tᵣ (1R,2R): 8.7 min, 21.73:78.27 er.
(1S,2S)-2-Methoxy-1,2-diphenylethyl isobutyrate

\[
\begin{align*}
\text{Chiralcel OD-H (98:2 hexane:IPA, flow rate 1 mL min}^{-1}, 211 \text{ nm, 30 °C})
\end{align*}
\]
\[t_R (1S,2S): 4.4 \text{ min, } t_R (1R,2R): 4.9 \text{ min, 81.95:18.05 er.}\]

(1R,2R)-2-Isopropoxy-1,2-diphenylethan-1-ol

\[
\begin{align*}
\text{Chiralcel OD-H (95:5 hexane:IPA, flow rate 1 mL min}^{-1}, 211 \text{ nm, 30 °C})
\end{align*}
\]
\[t_R (1S,2S): 4.9 \text{ min, } t_R (1R,2R): 5.5 \text{ min, 38.58:61.42 er.}\]
(1S,2S)-2-Isoproxy-1,2-diphenylethan-1-ol

2-Isoproxy-1,2-diphenylethyl isobutyrate could not be separated by HPLC so was hydrolysed to 2-isoproxy-1,2-diphenylethan-1-ol:

Chiralcel OD-H (95:5 hexane:IPA, flow rate 1 mLmin⁻¹, 211 nm, 30 °C) tᵣ (1S,2S): 4.9 min, tᵣ (1R,2R): 5.5 min, 96.41:3.59 er.
NMR Spectra

$^1$H, CDCl$_3$, 500 MHz

$^{13}$C, CDCl$_3$, 126 MHz
$^1$H, CDCl$_3$, 400 MHz

$^{13}$C, CDCl$_3$, 126 MHz
$^1$H, CDCl$_3$, 500 MHz

$^{13}$C, CDCl$_3$, 126 MHz
$^1$H, CDCl$_3$, 400 MHz

$^{13}$C, CDCl$_3$, 126 MHz
S6

$^1$H, CDCl$_3$, 400 MHz

$^{13}$C, CDCl$_3$, 126 MHz
$^1$H, CDCl$_3$, 400 MHz

$^{13}$C, CDCl$_3$, 126 MHz
$^{1}H$, CDCl$_3$, 500 MHz

$^{13}C$, CDCl$_3$, 126 MHz
References