Supporting Information

Acylative Kinetic Resolution of Alcohols Using a Recyclable Polymer-Supported Isothiourea Catalyst in Batch and Flow

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General Information

All anhydrous reactions were carried out in glassware that was flame-dried under vacuum. Anhydrous $CHCl_3$ was purchased from Sigma Aldrich. All other solvents and reagents were used as received without further purification.

Room temperature (r.t.) refers to 20–25 °C. Temperatures of 0 °C and −78 °C were obtained using ice/water and CO_2(s)/acetone baths respectively. Reaction temperatures over 20–25 °C were obtained using a heating mantle equipped with a contact thermometer. *In vacuo* refers to the use of a Büchi Rotavapor R-210 with a vacuum controller V-850, an IKA rotary evaporator IKA RV 10 basic with a vacuum controller Vacubrand CVC 3000 or a Heidolph Laborota 4001 rotary evaporator with a vacuum controller.

Thin layer chromatography (TLC) was carried out using aluminium plates coated with silica (Kieselgel 60 F254 silica) and visualisation was achieved using ultraviolet light (254 nm). Flash chromatography used Kieselgel 60 silica in the solvent system stated. Purifications using an automated Biotage® IsoleraTM 4 were performed using the solvent systems and column volumes (CV) stated.

Melting points were recorded on Electrothermal 100 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 r.t.

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 (ν_{max}) reported in cm^{-1}.

HPLC analyses were obtained on a Shimadzu HPLC consisting of a DGU-20A5 degasser, LC-20AT liquid chromatography, SIL-20AHT autosampler, CMB-20A communications bus module, SPD-M20A diode array 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, and IC columns. All HPLC traces of enantiomerically enriched compounds were compared with authentic racemic spectra.

GC analyses were obtained on a Shimadzu GC consisting of a Shimadzu AOC-20i auto injector and a Shimadzu GC-2025 gas chromatograph. Analysis was performed using Shimadzu GC solution v2.41 software and separation was achieved using Agilent Cycledex-B column (length: 30 m, thickness: 0.250 mm, film thickness: 0.25 μm).

^{1}H, $^{13}C\{^{1}H\}$ NMR spectra were obtained on either a Bruker Avance II 400 (^{1}H 400 MHz) or a Bruker Avance 500 (^{1}H 500 MHz, ^{13}C 126 MHz) spectrometer at r.t. in the deuterated solvent stated. All chemical shifts are in ppm relative to residual solvent peak. All coupling constants, *J*, are quoted in Hz. Multiplicities are indicated by: s (singlet), d (doublet), t (triplet), q (quartet), m (multiplet), dd (doublet of doublets), dt (doublet of triplets), dq (doublet of quartets). The abbreviation Ar is used to denote aromatic, Ph to denote phenyl, br to denote broad and app to denote apparent.

High resolution mass spectrometry (HRMS) data was acquired at the EPSRC UK National Mass Spectrometry Facility at Swansea University. Low resolution nanospray ionization (NSI) MS was carried out on a Micromass Quattro II spectrometer and high resolution NSI MS on a Thermofisher LTQ Orbitrap XL spectrometer.

Flow Chemistry- HPLC analyses was performed on an Agilent Technologies chromatograph (1100 Series), using DAICEL CHIRALCEL OD-H or DAICEL CHIRALPAK AD-H and AS-H. Chiral GC analyses were obtained on an Agilent Technologies apparatus (7890A series) using a BetaDex120 column (length: 30 m, thickness: 0.250 mm, film thickness: 0.25 μm).

Selectivity factors were calculated using the following equations, with all ees determined by chiral HPLC analysis. See reference 1 for the derivation, and alternative forms, of these equations.

$$S = \frac{\ln[(1-conv)(1-ee_{alcohol})]}{\ln[(1-conv)(1+ee_{alcohol})]}$$

where both ee and conv are given as between 0 and 1

$$\text{and,} \quad conv = \frac{ee_{alcohol}}{ee_{alcohol} + ee_{ester}}$$

General Procedures

General Procedure A: *Preparation of racemic alcohols by Grignard reaction*

The appropriate Grignard reagent (1.5 equiv.) was added to a solution of aldehyde (1.0 equiv.) in anhydrous THF (0.3 M) at the specified temperature under a nitrogen atmosphere and the mixture stirred for the specified time. The reaction was quenched with aq. NH_4Cl and the mixture extracted with EtOAc. The organic layer was washed with brine and dried ($MgSO_4$) and concentrated *in vacuo*. The crude product was purified by silica gel column chromatography.

General procedure B: *Preparation of racemic alcohols by $NaBH_4$ reduction*

A solution of the appropriate ketone (1.5 g, 1.0 equiv.) in methanol (15 mL) was cooled to 0 °C. $NaBH_4$ (appropriate amount depending on substrates, 0.6-1.5 equiv.) was added portionwise and stirred for 2 h. After completion of the reaction the solvent was removed *in vacuo* and the residue was dissolved in EtOAc (20 mL). The mixture was washed with 1 M aq. HCl (× 2), sat. aq. $NaHCO_3$, dried ($MgSO_4$), and concentrated *in vacuo*. The crude alcohol was purified by Biotage® Isolera™ 4 [SNAP Ultra 25 g, 75 mL min^{-1}, hexane:EtOAc (100:0 2 CV, 100:0 to 70:40 30 CV, (16 ml each))] to afford pure alcohol.

General Procedure C: *Preparation of racemic esters using DMAP*

A flask was charged with DMAP (10 mol%), appropriate alcohol (1.0 equiv.), *i*-Pr_2NEt (1.1 equiv.), appropriate anhydride (1.0 equiv.) in CH_2Cl_2 (0.2 M) and the reaction mixture was stirred at r.t. for 5 h. The reaction mixture was diluted with CH_2Cl_2 and washed with 1 M HCl, sat. aq. $NaHCO_3$ and brine. The organic layer was dried ($MgSO_4$), filtered and concentrated *in vacuo*. The products did not require further purification.

General Procedure D: *Kinetic resolution of 2° alcohols using PS-HyperBTM catalyst*

The appropriate alcohol (1.0 equiv.) was dissolved in $CHCl_3$ (0.2 M) and the solution cooled to 0 °C. PS-HyperBTM **6** (1 mol%), *i*-Pr_2NEt (0.6 equiv.) and appropriate anhydride (0.55 equiv.) were added and the solution stirred at appropriate temperature for the time given. The catalyst was recovered through filtration, and the filtrate was washed with 1 M HCl, sat. aq. $NaHCO_3$ and brine. The organic layer was dried ($MgSO_4$), filtered and concentrated *in vacuo*. The residue was

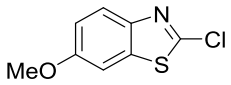
purified by Biotage® Isolera™ 4 [SNAP Ultra 10 g, 75 mL min^{-1}, hexane:EtOAc (100:0 2 CV, 100:0 to 70:30 30 CV, (16 ml each))] to afford the ester and alcohol.

General Procedure E: *Hydrolysis of Esters.*

The appropriate ester (1.0 equiv.) was dissolved in MeOH (0.20 M) and 1 M aq. NaOH (3.0 equiv.) was added and the solution stirred at 50-55°C for 30 min. Upon completion, the reaction was cooled to r.t. and concentrated *in vacuo*. The residue was acidified with 1 M HCl and extracted with EtOAc (× 2). The combined organic fractions were washed with sat. aq. $NaHCO_3$ followed by brine. The organic layer was dried ($MgSO_4$), filtered and concentrated *in vacuo* to give the curresponding alcohol.

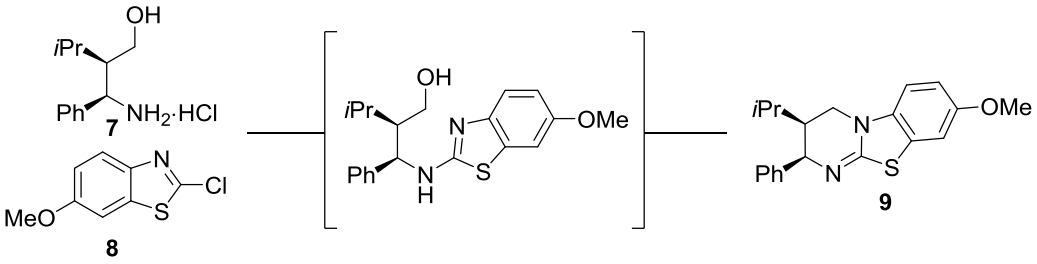
Synthesis of Polymer Supported HyperBTM

2-Chloro-6-methoxybenzo[*d*]thiazole 8



$CuCl_2$ (13.6 g, 100 mmol, 1.2 equiv.) was added to a round bottomed flask and dried under vacuum at 110 °C for 1 h. After cooling to 40-50 °C, MeCN (500 mL) and *tert*-butyl nitrite (15.0 mL, 126 mmol, 1.5 equiv.) were added, followed by a suspension of 6-methoxybenzo[*d*]thiazol-2-amine (15.2 g, 84 mmol, 1.0 equiv.) in MeCN (15 mL), which was added portionwise using a pipette (N_2 gas evolution was observed as a froth). Upon complete addition, the reaction was heated at 65 °C for 3 h. The reaction was cooled to r.t., poured into 4 M HCl (200 mL), and extracted using Et_2O (2 × 200 mL). The combined organic fractions were dried ($MgSO_4$) and concentrated *in vacuo*. Purification by silica column chromatography using EtOAc:Pet. ether (2.5:97.5) afforded 2-chloro-6-methoxybenzo[*d*]thiazole **8** as a colourless solid (11.1 g, 66%). mp: 80-83 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ_H: 3.87 (3H, s, OCH_3), 7.07 (1H, dd, *J* 9.0, 2.6, ArC(5)*H*), 7.22 (1H, dd, *J* 2.5, ArC(7)*H*), 7.82 (1H, dd, *J* 9.0, ArC(4)*H*). Data in accordance with literature.[2]

(2*R*,3*S*)-3-Isopropyl-8-methoxy-2-phenyl-3,4-dihydro-2*H*-benzo[4,5]thiazolo[3,2-*a*]pyrimidine 9

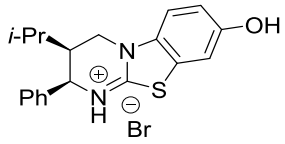


Following a procedure outline by Smith,[3] 2-chloro-6-methoxybenzo[*d*]thiazole **8** (4.0 g, 20 mmol, 1 equiv.) was added to a solution of (*R*)-2-((*R*)-amino(phenyl)methyl)-3-methylbutan-1-ol hydrochloride **7** (4.81 g, 21 mmol, 1.05 equiv.), and *i*-Pr_2NEt (10.45 mL, 60 mmol, 4 equiv.) in *o*-dichlorobenzene (7.5 mL, 2.0 M). The resulting pale yellow suspension was heated at reflux until completion. The resulting mixture was cooled to r.t., H_2O (15 mL) was added and the aqueous phase extracted with CH_2Cl_2 (3 × 20 mL). The combined organic fractions were washed

with brine, dried ($MgSO_4$), and concentrated *in vacuo* to afford the crude product which was triturated with hexane to afford the amino alcohol intermediate as a brown solid which was used in the next step without further purification (5.43 g, 76% yield).

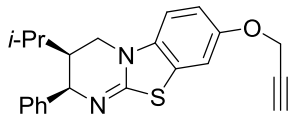
To a slurry of crude amino alcohol (5.43 g, 15.2 mmol, 1 equiv.) in anhydrous CH_2Cl_2 (80 mL, 0.2 M) was added Et_3N (8.47 mL, 60.8 mmol, 4 equiv.) and the reaction mixture was cooled to 0 °C. Methanesulfonyl chloride (1.53 mL, 19.8 mmol, 1.3 equiv.) was added and the reaction mixture was stirred at r.t. for 30 mins. Once complete consumption of the amino alcohol was observed, *i*-PrOH (1 mL) was added and the reaction was heated at reflux for 16 h. The reaction was quenched with 1 M aq. NaOH (20 mL) and the biphasic mixture stirred vigorously for 30 mins. The aqueous layer was extracted with CH_2Cl_2 (3 × 20 mL) and the combined organic fractions washed with brine (50 mL), dried ($MgSO_4$) and concentrated *in vacuo* to afford the crude product which was purified by Biotage® Isolera™ 4 [SNAP Ultra 25 g, 75 mL min^{-1}, CH_2Cl_2:EtOAc (95:5 5 CV, 95:5 to 60:40 10 CV, 60:40 5 CV, 16 mL each)] to afford an off white solid that was recrystallised from 50% EtOAc in Hexane to give (2*R*,3*S*)-3-isopropyl-8-methoxy-2-phenyl-3,4-dihydro-2*H*-benzo[4,5]thiazolo[3,2-*a*]pyrimidine **9** as a colourless solid (2.05 g, 40%); $[\alpha]_D^{20} = -249.1$ (*c* 1.0 $CHCl_3$); mp 139-141 °C; IR ν_{max} (thin film, cm^{-1}) 1614 (C=N), 1487 (C=C); ^{1}H NMR (400 MHz, $CDCl_3$) δ_H: 0.84 (1H, d, *J* 6.6, CH_3), 1.13 (1H, d, *J* 6.6, CH_3), 1.23-1.35 (1H, m, C*H*$(CH_3)_2$), 1.89-2.00 (1H, m, C(3)*H*), 3.33 (1H, app. t, *J* 11.4, C(4)H^AH^B), 3.77-3.88 (4H, m, OCH_3 & C(4)H^AH^B), 4.91 (1H, dd, *J* 4.4, 1.7, C(2)*H*), 6.72 (1H, d, *J* 8.7, ArC(6)*H*), 6.79 (1H, dd, *J* 8.7, 2.5 ArC(7)*H*), 6.95 (1H, d, *J* 2.5, ArC(9)*H*), 7.17-7.34 (5H, m, 5 × PhC*H*); ^{13}C{^{1}H} NMR (126 MHz, $CDCl_3$) δ_C: 20.2 (*C*H_3), 22.1 (*C*H_3), 27.1 (*C*H$(CH_3)_2$), 41.0 (*C*(3)H), 42.1 (*C*(4)H_2), 56.1 (O*C*H_3), 61.1 (*C*(2)H), 108.1 (Ar*C*(6)H), 108.3 (Ar*C*(9)H), 111.8 (Ar*C*(7)H), 124.3 (Ar*C*(5a)), 127.3 (Ph*C*(4)H), 128.1 (Ph*C*(3,5)H), 128.5 (Ph*C*(2,6)H), 134.9 ((Ar*C*(9a)), 140.7 (Ph*C*(1)), 155.5 (Ar*C*(8)), 158.8 (*C*=N); HRMS (ESI^+) $C_{20}H_{23}N_2OS$ $[M+H]^+$, found 339.1516, requires 339.1526 (−2.8 ppm).

(2*R*,3*S*)-8-Hydroxy-3-isopropyl-2-phenyl-3,4-dihydro-2*H*-benzo[4,5]thiazolo[3,2-*a*]pyrimidin-1-ium bromide 10



BBr_3 (15 mL, 8.86 mmol, 1 M in CH_2Cl_2, 10 equiv.) was added dropwise to a solution of (2*R*,3*S*)-3-isopropyl-8-methoxy-2-phenyl-3,4-dihydro-2*H*-benzo[4,5]thiazolo[3,2-*a*]pyrimidine **9** (500 mg, 1.48 mmol, 1 equiv.) in CH_2Cl_2 (10 mL) at 0 °C. The solution was stirred at 0 °C for 2 h then warmed to r.t. and stirred for 16 h. The reaction was carefully quenched with MeOH (10 mL). CH_2Cl_2 (10 mL) was added and the aqueous phase was extracted with CH_2Cl_2 (3 × 20 mL). The organic layers were combined, dried ($MgSO_4$) and concentrated *in vacuo* to afford (2*R*,3*S*)-8-hydroxy-3-isopropyl-2-phenyl-3,4-dihydro-2*H*-benzo[4,5]thiazolo[3,2-*a*]pyrimidin-1-ium bromide **10** as a colourless solid (580 mg, 97%). $[\alpha]_D^{20} = -174.7$ (*c* 1.0, MeOH); mp 194-196 °C; IR ν_{max} (thin film, cm^{-1}) 3147 (O-H), 2929 (C-H), 1597 (C=N), 1450 (C=C); ^{1}H NMR (400 MHz, d_6-DMSO) δ_H: 0.79 (3H, d, *J* 6.7, CH_3), 1.07 (3H, d, *J* 6.5, CH_3), 1.14-1.31 (1H, m, CH(CH_3)$_2$), 2.25-2.38 (1H, m, C(3)*H*), 3.68 (1H, m, C(4)H^AH^B), 4.40 (1H, dd, *J* 13.3, 4.9, C(4)H^AH^B), 5.17 (1H, d, *J* 4.8, C(2)*H*), 6.99 (1H, dd, *J* 8.8, 2.5, C(7)*H*), 7.26-7.34 (2H, m, PhC(2,6)*H*), 7.36-7.43 (3H, m, PhC(3,4,5)*H*), 7.46 (1H, d, *J* 2.5, ArC(9)*H*), 7.65 (1H, d, *J* 8.8, ArC(6)*H*), 9.98 (1H, s, O*H*), 11.14 (1H, s, N*H*); ^{13}C{^{1}H} NMR (126 MHz, d_6-DMSO) δ_C: 19.2 (*C*H_3), 21.5 (*C*H_3), 26.3 (*C*H(CH_3)$_2$), 39.3 (*C*(3)H), 42.5 (*C*(4)H_2), 55.4 (*C*(2)H), 109.7 (Ar*C*(9)H), 114.0 (Ar*C*(6)H), 115.3 (Ar*C*(7)H), 123.4 (Ar*C*(5a)), 127.8 (Ph*C*(2,6)H), 128.5 (Ph*C*(4)H), 128.9 (Ph*C*(3,5)H), 131.3 ((Ar*C*(9a)), 137.7 (Ph*C*(1)), 155.4 (Ar*C*(8)), 162.3 (*C*=N); HRMS (NSI) $C_{19}H_{21}N_2OS$ $[M-Br]^+$ found 325.1369; requires 325.1369 (0.0 ppm).

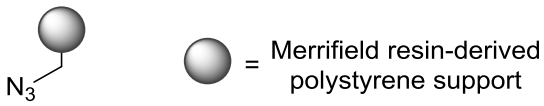
(2*R*,3*S*)-3-Isopropyl-2-phenyl-8-(prop-2-yn-1-yloxy)-3,4-dihydro-2*H*-benzo[4,5]thiazolo[3,2-*a*]pyrimidine 11



KO*t*Bu (395 mg, 3.53 mmol, 2.6 equiv.) was added to a solution of (2*R*,3*S*)-8-hydroxy-3-isopropyl-2-phenyl-3,4-dihydro-2*H*-benzo[4,5]thiazolo[3,2-*a*]pyrimidin-1-ium bromide **10** (0.55

g, 1.36 mmol, 1 equiv.) in THF/DMSO (1:1, 10 mL) at 0 °C, and the reaction mixture was stirred for 2 h. Propargyl bromide (227 µL, 2.04 mmol, 80% in toluene, 1.5 equiv.) was added and the reaction mixture was allowed to warm to r.t. (*ca*. 2 h) and quenched with brine (10 mL). The aqueous phase was extracted with EtOAc (3 × 10 mL). The combined organic fractions were washed with brine, dried ($MgSO_4$) and concentrated *in vacuo*. The residue was purified by Biotage® Isolera^{TM} 4 [SNAP Ultra 25 g, 75 mL min^{-1}, CH_2Cl_2:EtOAc (95:5 5CV, 95:5 to 60:40 10 CV, 60:40 5 CV)] to afford (2*R*,3*S*)-3-isopropyl-2-phenyl-8-(prop-2-yn-1-yloxy)-3,4-dihydro-2*H*-benzo[4,5]thiazolo[3,2-*a*]pyrimidine **11** as a colourless solid (360 mg, 73%); $[\alpha]_D^{20} = -198.7$ (*c* 1.0 $CHCl_3$); mp: 60-63 °C; IR ν_{max} (thin film, cm^{-1}) 2956 (C-H), 1616 (C=N), 1487 (C=C); ^{1}H NMR (400 MHz, $CDCl_3$) δ_H: 0.84 (3H, d, *J* 6.7, C*H*$_3$), 1.13 (3H, d, *J* 6.5, C*H*$_3$), 1.22–1.35 (1H, m, C*H*(CH$_3$)$_2$), 1.94 (1H, app. ddt, *J* 11.3, 9.4, 4.9, C(3)*H*), 2.54 (1H, t, *J* 2.4, C≡C*H*), 3.33 (1H, app. t, *J* 11.5, C(4)*H*^{A}H^B), 3.83 (1H, ddd, *J* 11.5, 5.2, 1.8, C(4)H^A*H*^{B}), 4.68 (2H, d, *J* 2.4, C*H*$_2$O), 4.91 (1H, dd, *J* 4.5, 1.7, C(2)*H*), 6.73 (1H, d, *J* 8.7, ArC(6)*H*), 6.88 (2H, dd, *J* 8.7, 2.5, ArC(7)*H*), 7.04 (1H, d, *J* 2.5, ArC(9)*H*), 7.17-7.33 (5H, m, 5 × PhC*H*); ^{13}C{^{1}H} NMR (126 MHz, $CDCl_3$) δ_C: 20.1 (*C*H$_3$), 22.1 (*C*H$_3$), 27.0 (*C*H(CH$_3$)$_2$), 41.0 (*C*(3)H), 42.1 (*C*(4)H$_2$), 57.0 (*C*H$_2$O), 61.2 (*C*(2)H), 75.9 (C≡*C*H), 78.6 (*C*≡CH), 107.9 (Ar*C*(9)H), 109.9 (Ar*C*(6)H), 113.1 (Ar*C*(7)H), 124.3 (Ar*C*(5)), 127.3 (Ph*C*(4)H), 128.1 (Ph*C*(2,6)H), 128.5 (Ph*C*(3,5)H), 135.8 ((Ar*C*(9a)), 140.7 (Ph*C*(1)), 153.2 (Ar*C*(8)), 158.6 (*C*=N); HRMS (NSI) $C_{22}H_{23}N_2OS$ ([M+H]$^+$, 100%) found 363.1516, requires 363.1526 (−2.7 ppm).

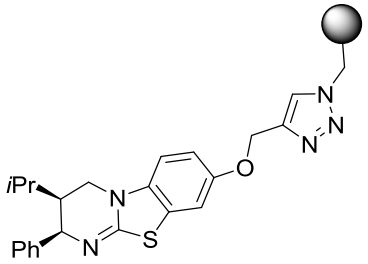
(Azidomethyl)polystyrene S1



Following a procedure outlined by Pericàs *et al*.,[4] (chloromethyl)polystyrene resin (3.0 g, *f* = 1.23 mmol g^{-1}) was added to NaN_3 (780 mg, 51 mmol) in DMSO (30 mL). The mixture was heated at 60 °C (without stirring) for 16 h and then cooled to r.t.. The suspension was filtered and washed sequentially with H_2O (500 mL), THF-MeOH 1:1 (250 mL), MeOH (250 mL) and THF (250 mL). The resulting solid was dried *in vacuo* for 24 h at 40 °C to afford (azidomethyl)polystyrene **S1** (12.6 g). IR ν_{max} (solid, cm^{-1}) 2094 (N_3); Elemental analysis (%) C 85.61, H 6.75, N 5.48; *f* = 1.20 mmol g^{-1}

Functionality (*f*) calculated according to literature method.[5]

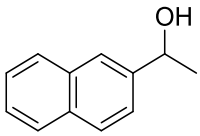
(2*R*,3*S*)-8-((1-Ethyl-1*H*-1,2,3-triazol-4-yl)methoxy)-3-isopropyl-2-phenyl-3,4-dihydro-2*H*-benzo[4,5]thiazolo[3,2-*a*]pyrimidine on polymer support 6



(2*R*,3*S*)-3-Isopropyl-2-phenyl-8-(prop-2-yn-1-yloxy)-3,4-dihydro-2*H*-benzo[4,5]thiazolo[3,2-*a*]pyrimidine **11** (300 mg, 0.828 mmol, 1.1 equiv.), *i*Pr_2NEt (458 µL, 2.63 mmol, 3.5 equiv.) and CuI (7.2 mg, 0.038 mmol, 5 mol%) were added to a suspension of (azidomethyl)polystyrene **S1** (627 mg, 0.752 mmol, f = 1.20 mmol/g, 1 equiv.) in THF:DMF 1:1 (14 mL) with slow stirring (100 rpm). The reaction mixture was stirred until disappearance of the azide band (~2094 cm^{-1}) was confirmed by IR (*ca.* 20 h). The suspension was filtered and washed sequentially with THF (1:1, 130 mL), H_2O (130 mL), H_2O-MeOH (1:1, 130 mL), MeOH (130 mL), MeOH-THF (1:1, 130 mL), THF (130 mL) and CH_2Cl_2 (130 mL) and the resin was dried *in vacuo* at 40 °C for 24 h to afford a pale brown resin (2*R*,3*S*)-8-((1-ethyl-1*H*-1,2,3-triazol-4-yl)methoxy)-3-isopropyl-2-phenyl-3,4-dihydro-2*H*-benzo[4,5]thiazolo[3,2-*a*]pyrimidine on polymer support **6** (980 mg). Elemental analysis (%) C 80.06, H 6.395, N 6.15. f = 0.88 mmol g^{-1} (2^{nd} batch of **6** prepared by same method gave f = 0.89 mmol g^{-1}).

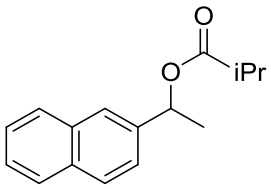
Alcohol and Ester Synthesis and Preparative Kinetic Resolutions

1-(Naphthalen-2-yl)ethan-1-ol 12



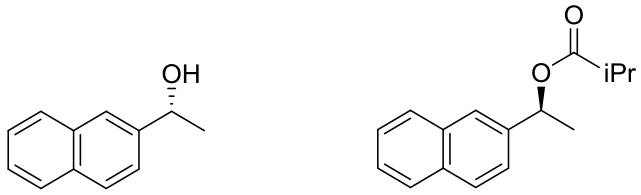
Following General Procedure A, methylmagnesium bromide (29 mL of 3.0 M solution in Et_2O, 88 mmol, 1.5 equiv.) and 2-naphthaldehyde (9.2 g, 59 mmol) in anhydrous THF (105 mL) at –78 °C for 16 h gave, after purification by column chromatography (80:20 Hexane:EtOAc), 1-(naphthalen-2-yl)ethan-1-ol **12** (9.33 g, 92%) as a colourless solid. mp 67-69 °C {Lit.[6] 69-72 °C}. ^{1}H NMR (400 MHz, $CDCl_3$) δ_H: 1.58 (3H, d, *J* 6.5, C*H*$_3$), 2.05 (1H, s, O*H*), 5.06 (1H, d, *J* 6.5, C*H*-OH), 7.43-7.54 (3H, m, 3 × Ar*H*), 7.77-7.88 (4H, m, 4 × Ar*H*). Spectral data in accordance with literature.[6]

1-(Naphthalen-2-yl)ethyl isobutyrate 13



Following General Procedure C, DMAP (3.7 mg, 10 mol%), 1-(naphthalen-2-yl)ethan-1-ol **12** (52 mg, 0.3 mmol), *i*-Pr_2NEt (57 µL, 0.33 mmol, 1.1 equiv.) and (*i*-PrCO)$_2$O (50 µL, 0.3 mmol, 1.0 equiv.) in CH_2Cl_2 (0.2 M) gave 1-(naphthalen-2-yl)ethyl isobutyrate **13** (64 mg, 88%) as a colourless solid. mp 39-41°C; ^{1}H NMR (400 MHz, $CDCl_3$) δ_H: 1.2 (3H, d, *J* 7.0, CH(C*H*$_3$)^A(CH$_3$)^B), 1.23 (3H, d, *J* 7.0, CH(CH$_3$)^A(C*H*$_3$)^B), 1.64 (3H, d, *J* 6.6, C*H*$_3$), 2.57-2.69 (1H, m, C*H*(CH$_3$)$_2$), 6.08 (1H, q, *J* 6.6, C*H*-OCOiPr), 7.45-7.54 (3H, m, 3 × Ar*H*), 7.80-7.90 (4H, m, 4 × Ar*H*). Spectral data in accordance with literature.[7]

Kinetic resolution of 1-(naphthalen-2-yl)ethan-1-ol 12



Following General Procedure D, 1-(naphthalen-2-yl)ethan-1-ol **12** (52 mg, 0.3 mmol), PS-HyperBTM **6** (3.4 mg, 1 mol%), *i*-Pr_2NEt (31 µL, 0.18 mmol, 0.6 equiv.) and $(i\text{-PrCO})_2O$ (27.4 µL, 0.165 mmol, 0.55 equiv.) in $CHCl_3$ (1.5 mL) for 7 h at 0 °C gave the crude product, which was purified by Biotage® Isolera™ 4 to give (*R*)-1-(naphthalen-2-yl)ethan-1-ol **12** (24 mg, 46%) (*S*)-1-(naphthalen-2-yl)ethyl isobutyrate **13** (32 mg, 44%). **(*R*)-1-(Naphthalen-2-yl)ethan-1-ol 12**: mp 68-70 °C {Lit.[6] 69-72 °C}. $[\alpha]_D^{20}$ +42 (*c* 1.0, $CHCl_3$) {Lit.[8] $[\alpha]_D^{20}$ +46.5 (98% ee, *c* 1.2, $CHCl_3$) Chiral HPLC analysis, Chiralcel OJ-H (90:10 hexane:IPA, flow rate 1.0 mL min^{-1}, 254 nm, 30 °C) t_R (*S*): 15.0 min, t_R (*R*): 19.1 min, 7.654:92.346 er. **(*S*)-1-(Naphthalen-2-yl)ethyl isobutyrate 13**: $[\alpha]_D^{20}$ −97 (*c* 1.0, $CHCl_3$) Chiral HPLC analysis, Chiralcel OJ-H (98:2 hexane:IPA, flow rate 1.0 mL min^{-1}, 254 nm, 30 °C) t_R (*R*): 9.4 min, t_R (*S*): 11.6 min, 2.533:97.467 er. *s* = 104

Chiral HPLC data for Table 5, recycle no. 1

(*R*)-1-(Naphthalen-2-yl)ethan-1-ol 12: Chiral HPLC analysis, Chiralcel OJ-H (90:10 hexane:IPA, flow rate 1.0 mL min^{-1}, 254 nm, 30 °C) t_R (*S*): 15.0 min, t_R (*R*): 19.1 min, 3.424:96.576 er. **(*S*)-1-(Naphthalen-2-yl)ethyl isobutyrate 13**: Chiral HPLC analysis, Chiralcel OJ-H (98:2 hexane:IPA, flow rate 1.0 mL min^{-1}, 254 nm, 30 °C) t_R (*R*): 9.4 min, t_R (*S*): 11.6 min, 3.785:96.215 er. *s* = 94

1-Phenylethan-1-ol 15

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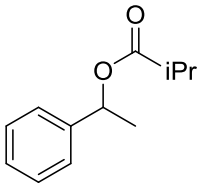
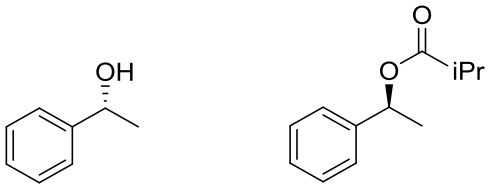
1-Phenylethyl isobutyrate S2



Following General Procedure C, DMAP (3.7 mg, 10 mol%), 1-phenylethan-1-ol **15** (37 mg, 0.3 mmol), *i*-Pr_2NEt (57 µL, 0.33 mmol, 1.1 equiv.) and $(i\text{-PrCO})_2O$ (50 µL, 0.3 mmol, 1.0 equiv.) in CH_2Cl_2 (0.2 M) gave 1-phenylethyl isobutyrate **S2** (45 mg, 78%) as a colourless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ_H: 1.18 (3H, d, *J* 7.0, $CH(CH_3)^A(CH_3)^B$), 1.20 (3H, d, *J* 7.0, $CH(CH_3)^A(CH_3)^B$), 1.54 (3H, d, *J* 6.6, CH_3), 2.50-2.66 (1H, m, $CH(CH_3)^A(CH_3)^B$), 5.90 (1H, q, *J* 6.6, $CHCH_3$), 7.24-7.41 (5H, m, 5 × Ar*H*). Spectral data in accordance with literature.[7]

Kinetic resolution of 1-phenylethan-1-ol 15

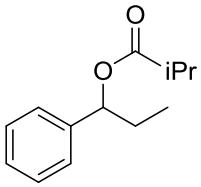


Following General Procedure D, 1-phenylethan-1-ol **15** (37 mg, 0.3 mmol), PS-HyperBTM **6** (3.4 mg, 1 mol%), *i*-Pr_2NEt (31 µL, 0.18 mmol, 0.6 equiv.) and $(i\text{-PrCO})_2O$ (27.4 µL, 0.165 mmol, 0.55 equiv.) in $CHCl_3$ (1.5 mL) for 7 h at 0 °C gave the crude product, which was purified by Biotage® Isolera™ 4 to give (*R*)-phenylethan-1-ol **15** (23 mg, 38%) and (*S*)-1-phenylethyl isobutyrate **S2** (38 mg, 40%). **(*R*)-Phenylethan-1-ol 15:** $[\alpha]_D^{20}$ +42 (*c* 1.0, $CHCl_3$) {Lit.[9] $[\alpha]_D^{20}$ +58 (99 %ee, *c* 1.0, $CHCl_3$)} Chiral HPLC analysis Chiralcel OD-H (99:1 hexane:IPA, flow rate 1.0 mL min^{-1}, 220 nm, 30 °C) t_R (*R*): 22.9 min, t_R (*S*): 28.6 min, 84.6:15.4 er.). **(*S*)-1-Phenylethyl isobutyrate S2:** $[\alpha]_D^{20}$ −128 (*c* 1.0, $CHCl_3$) Chiral HPLC analysis Chiralcel OD-H (99.8:0.2 hexane:IPA, flow rate 1.0 mL min^{-1}, 220 nm, 30 °C) t_R (*R*): 5.0 min, t_R (*S*): 5.4 min, 4.272:95.728 er. *s* = 46

1-Phenylpropan-1-ol 16

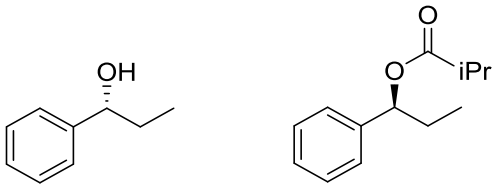
Commercially available

1-Phenylpropyl isobutyrate S3



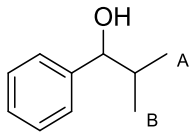
Following General Procedure C, DMAP (3.7 mg, 10 mol%), 1-phenylpropan-1-ol **16** (41 mg, 0.3 mmol), *i*-Pr_2NEt (57 µL, 0.33 mmol, 1.1 equiv.) and $(i\text{-PrCO})_2O$ (50 µL, 0.3 mmol, 1.0 equiv.) in CH_2Cl_2 (0.2 M) gave 1-phenylpropyl isobutyrate **S3** (49 mg, 79%) as a colourless oil. ^1H NMR (400 MHz, $CDCl_3$) δ_H: 0.90 (3H, t, *J* 7.4, CH_2CH_3), 1.16 (3H, d, *J* 7.0, $CH(CH_3)^A(CH_3)^B$), 1.20 (3H, d, *J* 6.7, $(CH_3)^A(CH_3)^B$), 1.75-1.98 (2H, m, CH_2CH_3), 2.48-2.68 (1H, m, $CH(CH_3)_2$), 5.67 (1H, dd, J 7.5,6.1, $CHCH_2$), 7.20-7.42 (5H, m, 5 × Ar*H*). Data in accordance with literature.[7]

Kinetic Resolution of 1-phenylpropan-1-ol 16



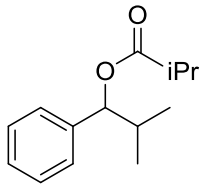
Following General Procedure D, 1-phenylpropan-1-ol **16** (41 mg, 0.3 mmol), PS-HyperBTM **6** (3.4 mg, 1 mol%), *i*-Pr_2NEt (31 µL, 0.18 mmol, 0.6 equiv.) and $(i\text{-PrCO})_2O$ (27.4 µL, 0.165 mmol, 0.55 equiv.) in $CHCl_3$ (1.5 mL) for 7 h at 0 °C gave the crude product, which was purified by Biotage® Isolera™ 4 to give (*R*)-1-phenylpropan-1-ol **16** (15 mg, 37%) and (*S*)-1-phenylpropyl isobutyrate **S3** (22 mg, 36%). **(*R*)-1-Phenylpropan-1-ol 16**: $[\alpha]_D^{20}$ +52 (*c* 1.0, $CHCl_3$) {Lit.[10] $[\alpha]_D^{20}$ +44.5 (97% ee, *c* 1.0, $CHCl_3$) Chiral HPLC analysis Chiralcel OD-H (99.5:0.5 hexane:IPA, flow rate 1.0 mL min^{-1}, 220 nm, 30 °C) t_R (*R*): 28.8 min, t_R (*S*): 31.6 min, 91.705:8.295 er. **(*S*)-1-Phenylpropyl isobutyrate S3:** $[\alpha]_D^{20}$ −111 (*c* 1.0, $CHCl_3$) Chiral HPLC analysis Chiralpak AD-H (99.8:0.2 hexane:IPA, flow rate 1.0 mL min^{-1}, 220 nm, 30 °C) t_R (*R*): 5.1 min, t_R (*S*): 5.6 min, 3.890:96.110 er. *s* = 65

2-Methyl-1-phenylpropan-1-ol 17



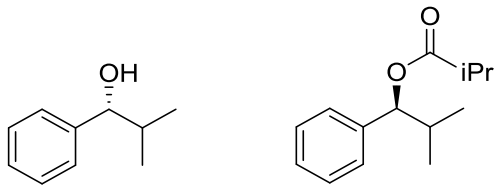
Following General Procedure B, 2-methyl-1-phenylpropan-1-one (1.5 g, 10.12 mmol, 1.0 equiv.) and $NaBH_4$ (383 mg, 10.12 mmol, 1.0 equiv.) in methanol (15 mL) gave, after purification by Biotage® Isolera™ 4, 2-methyl-1-phenylpropan-1-one **17** (1.3 g, 86%) as a colourless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ_H: 0.80 (3H, d, *J* 6.9, CH(C*H*$_3$) ^{A}(CH$_3$)^B), 1.01 (3H, d, *J* 6.7, (CH$_3$) ^{A}(C*H*$_3$)^B), 1.86-2.06 (2H, m, O*H* & C*H*(CH$_3$)$_2$), 4.36 (1H, d, *J* 6.9, C*H*-OH), 7.24-7.37 (5H, m, 5 × Ar*H*). Spectral data in accordance with literature.[11]

2-Methyl-1-phenylpropyl isobutyrate S4



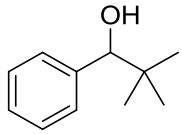
Following General Procedure C, DMAP (3.7 mg, 10 mol%), 2-methyl-1-phenylpropan-1-one **17** (45 mg, 0.3 mmol), *i*-Pr$_2$NEt (57 µL, 0.33 mmol, 1.1 equiv.) and (*i*-PrCO)$_2$O (50 µL, 0.3 mmol, 1.0 equiv.) in CH_2Cl_2 (0.2 M) gave 2-methyl-1-phenylpropyl isobutyrate **S4** (55 mg, 83%) as a colourless oil. IR ν_{max} (thin film, cm^{-1}): 2970 (C-H), 1732 (C=O); ^{1}H NMR (400 MHz, $CDCl_3$) δ_H: 0.82 (3H, d, *J* 6.8, CH(C*H*$_3$)^a), 0.97 (3H, d, *J* 6.7, (C*H*$_3$)^b), 1.16 (3H, d, *J* 7.0, CH(C*H*$_3$)^c), 1.20 (3H, d, *J* 7.0, CH(C*H*$_3$)^d), 2.03-2.17 (1H, m, C*H*(CH$_3$)$_2$), 2.52-2.66 (1H, m, C*H*-C=O), 5.47 (1H, d, *J* 7.3, C*H*-OCOiPr), 7.20-7.39 (5H, m, 5 × ArH); ^{13}C{^{1}H} NMR (126 MHz, $CDCl_3$) δ_C: 18.5 (CH(*C*H$_3$)^C(CH$_3$)^D), 18.9 (CH(CH$_3$)^C(*C*H$_3$)^D), 19.0 (CH(*C*H$_3$)^A(CH$_3$)^B), 19.1 (CH(CH$_3$)^A(*C*H$_3$)^B), 33.8 (*C*H(CH$_3$)^C(CH$_3$)^D), 34.4 (*C*H(CH$_3$)^A(CH$_3$)^B), 80.6 (*C*H-OCOiPr), 127.0 (2 × Ar*C*H), 127.7 (Ar*C*H), 128.2 (2 × Ar*C*H), 140.1 (Ar*C*(1)), 176.3 (C=O); HRMS (ESI) $C_{14}H_{20}O_2Na$ ([M+Na]$^+$, 100%) found 243.1351, requires 243.1356 (+2.1 ppm).

Kinetic resolution of 2-methyl-1-phenylpropan-1-ol 17



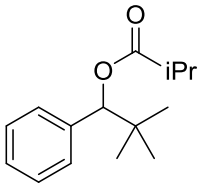
Following General Procedure D, the 2-methyl-1-phenylpropan-1-one **17** (45 mg, 0.3 mmol), PS-HyperBTM **6** (3.4 mg, 1 mol%), *i*-Pr_2NEt (31 µL, 0.18 mmol, 0.6 equiv.) and $(i\text{-PrCO})_2O$ (27.4 µL, 0.165 mmol, 0.55 equiv.) in $CHCl_3$ (1.5 mL) for 7 h at 0 °C gave the crude product, which was purified by Biotage® Isolera™ 4 to give (*R*)-2-methyl-1-phenylpropan-1-one **17** (20 mg, 45%) and (*S*)-2-methyl-1-phenylpropyl isobutyrate **S4** (19 mg, 29%). **(*R*)-2-Methyl-1-phenylpropan-1-one 17**: $[\alpha]_D^{20}$ +18 (*c* 1.0, $CHCl_3$) {Lit.[12] $[\alpha]_D^{20}$, +11.3 (96% ee, *c* 0.42, $CHCl_3$) Chiral HPLC analysis Chiralcel OD-H (95:5 hexane:IPA, flow rate 1.0 mL min^{-1}, 220 nm, 30 °C) t_R (*S*): 6.55 min, t_R (*R*): 7.88 min, 18.107:81.893 er. **(*S*)-2-Methyl-1-phenylpropyl isobutyrate S4**: $[\alpha]_D^{20}$ −108 (*c* 1.0, $CHCl_3$); Chiral HPLC analysis Chiralpak AD-H (99.8:0.2 hexane:IPA, flow rate 1.0 mL min^{-1}, 220 nm, 30 °C) t_R (*R*): 5.18 min, t_R (*S*): 5.97 min, 2.355:97.645 er, *s* = 80

2,2-Dimethyl-1-phenylpropan-1-ol 18



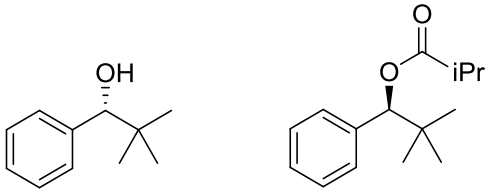
Following General Procedure B, 2,2-dimethyl-1-phenylpropan-1-one (1.0 g, 6.17 mmol, 1.0 equiv.) and $NaBH_4$ (233 mg, 6.17, 1.0 equiv.) in methanol (10 mL) gave, after purification by Biotage® Isolera™ 4 gave 2,2-dimethyl-1-phenylpropan-1-ol **18** (0.85 g, 86%) as a colourless solid. mp 44-46 °C {Lit.[13] 44-45}. ^1H NMR (400 MHz, $CDCl_3$) δ_H: 0.88 (9H, s, 3 × C*H*$_3$), 1.80 (1H, s, O*H*), 4.35 (1H, s, C*H*), 7.18-7.31 (5H, m, 5 × Ar*H*). Spectral data in accordance with literature.[14]

2,2-Dimethyl-1-phenylpropyl isobutyrate S5



Following General Procedure C, DMAP (3.7 mg, 10 mol%), 2,2-dimethyl-1-phenylpropan-1-ol **18** (49 mg, 0.3 mmol), and *i*-Pr_2NEt (57 µL, 0.33 mmol, 1.1 equiv.) and $(i\text{-PrCO})_2O$ (50 µL, 0.3 mmol, 1.0 equiv.) in CH_2Cl_2 (0.2 M) gave 2,2-dimethyl-1-phenylpropyl isobutyrate **S5** (62 mg, 88%) as a colourless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ_H: 0.93 (9H, s, 3×CH_3), 1.18 (3H, d, *J* 7.0, CH (CH_3)^A(CH_3)^B), 1.21 (3H, d, *J* 7.0, CH (CH_3)^A(CH_3)^B), 2.54-2.67 (1H, m, CH(CH_3)$_2$), 5.48 (1H, s, CH-OCOiPr), 7.17-7.38 (5H, m, 5 × ArH). Spectral data in accordance with literature.[15]

Kinetic resolution of 2,2-dimethyl-1-phenylpropan-1-ol 18

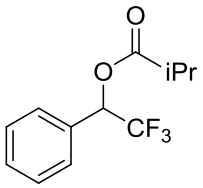


Following General Procedure D, 2,2-dimethyl-1-phenylpropan-1-ol **18** (49 mg, 0.3 mmol), PS-HyperBTM **6** (17 mg, 5 mol%), *i*-Pr_2NEt (31 µL, 0.18 mmol, 0.6 equiv.) and $(i\text{-PrCO})_2O$ (27.4 µL, 0.165 mmol, 0.55 equiv.) in $CHCl_3$ (1.5 mL) for 30 h at r.t. gave the crude product, which was purified by Biotage® Isolera™ 4 to give (*R*)-2,2-dimethyl-1-phenylpropan-1-ol **18** (21 mg, 43%) and (*S*)-2,2-dimethyl-1-phenylpropyl isobutyrate **S5** (23 mg, 33%). **(*R*)-2,2-dimethyl-1-phenylpropan-1-ol 18**: mp 43-45 °C [{Lit.[13] 44-45] $[\alpha]_D^{20}$ +33 (*c* 1.0, $CHCl_3$) {Lit.[16] (*ent*) $[\alpha]_D^{20}$ −22.9 (86% ee, *c* 2.3 in $CHCl_3$) Chiral HPLC analysis Chiralcel OD-H (95:5 hexane:IPA, flow rate 1.0 mL min^{-1}, 220 nm, 30 °C) t_R (*S*): 6.6 min, t_R (*R*): 9.2 min, 18.907:81.093 er. **(*S*)-2,2-Dimethyl-1-phenylpropyl isobutyrate S5:** $[\alpha]_D^{20}$ −62 (*c* 1.0, $CHCl_3$); Chiral HPLC analysis Chiralpak AD-H (99.8:0.2 hexane:IPA, flow rate 1.0 mL min^{-1}, 220 nm, 30 °C) t_R (*R*): 5.3 min, t_R (*S*): 6.6 min, 1.673:98.327 er. *s* = 111

2,2,2-Trifluoro-1-phenylethan-1-ol 19

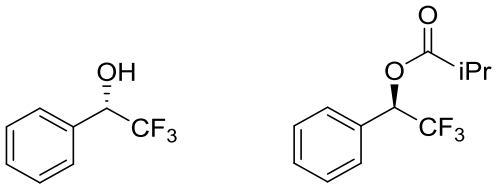
Commercially available

2,2,2-Trifluoro-1-phenylethyl isobutyrate S6



Following General Procedure C, DMAP (3.7 mg, 10 mol%), alcohol 2,2,2-trifluoro-1-phenylethan-1-ol **19** (53 mg, 0.3 mmol), *i*-Pr_2NEt (57 µL, 0.33 mmol, 1.1 equiv.) and $(i\text{-PrCO})_2O$ (50 µL, 0.3 mmol, 1.0 equiv.) in CH_2Cl_2 (0.2 M) gave 2,2,2-trifluoro-1-phenylethyl isobutyrate **S6** (66 mg, 90%) as a colourless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ_H: 1.21 (3H, d, *J* 7.0, CH $(CH_3)^A(CH_3)^B$), 1.24 (3H, d, *J* 7.0, CH $(CH_3)^A(CH_3)^B$), 2.64-2.77 (1H, m, $CH(CH_3)_2$), 6.14 (1H, q, *J* 6.9, $CH\text{-}CF_3$), 7.34-7.54 (5H, m, 5 × Ar*H*). Spectral data in accordance with literature.[17]

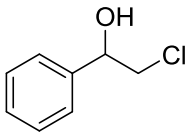
Kinetic resolution of 2,2,2-trifluoro-1-phenylethan-1-ol 19



Following General Procedure D, 2,2,2-trifluoro-1-phenylethan-1-ol **19** (106 mg, 0.6 mmol), PS-HyperBTM **6** (6.8 mg, 1 mol%), *i*-Pr_2NEt (62 µL, 0.36 mmol, 0.6 equiv.) and $(i\text{-PrCO})_2O$ (55 µL, 0.33 mmol, 0.55 equiv.) in $CHCl_3$ (3.0 mL) for 7 h at 0 °C gave the crude product, which was purified by Biotage® Isolera™ 4 to give (*S*)-2,2,2-trifluoro-1-phenylethan-1-ol **19** (48 mg, 45%) and (*R*)-2,2,2-trifluoro-1-phenylethyl isobutyrate **S6** (62 mg, 42%). **(*S*)-2,2,2-trifluoro-1-phenylethan-1-ol 19**: $[\alpha]_D^{20}$ +9 (*c* 1.0, $CHCl_3$) {Lit.[18] $[\alpha]_D^{20}$ +23 (90% ee, *c* 1.56, $CHCl_3$) Chiral HPLC analysis Chiralcel OJ-H (90:10 hexane:IPA, flow rate 1.0 mL min^{-1}, 220 nm, 30 °C) t_R (*R*): 10.4 min, t_R (*S*): 13.1 min, 28.417:71.583 er. **(*R*)-2,2,2-Trifluoro-1-phenylethyl isobutyrate S6**: $[\alpha]_D^{20}$ −47 (*c* 1.0, $CHCl_3$); Chiral HPLC analysis Chiralcel OJ-H (99:1

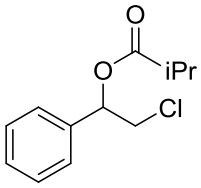
hexane:IPA, flow rate 1.0 mL min^{-1}, 220 nm, 30 °C) t_R (*S*): 4.9 min, t_R (*R*): 5.7 min, 26.554:73.446 er. *s* = 4

2-Chloro-1-phenylethan-1-ol 20



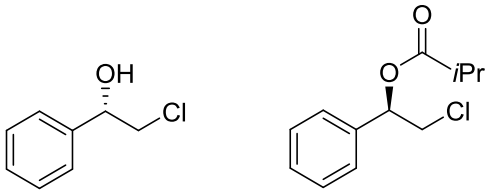
Following General Procedure B, 2-chloro-1-phenylethan-1-one (0.78 g, 5.0 mmol, 1.0 equiv.) and $NaBH_4$ (189 mg, 5.0 mmol, 1 equiv.) in methanol (10 mL) gave, after purification by Biotage® Isolera™ 4, 2-chloro-1-phenylethan-1-ol **20** (0.66 g, 84%) as a colourless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ_H: 2.66 (1H, d, *J* 3.2, O*H*), 3.65 (1H, dd, *J* 11.2, 8.8, CH^AH^B), 3.75 (1H, dd, *J* 11.2, 3.4, CH^AH^B), 4.88-4.94 (1H, m, C*H*-OH), 7.29-7.43 (5H, m, 5 × Ar*H*). Spectral data in accordance with Literature.[19]

2-Chloro-1-phenylethyl isobutyrate S7



Following General Procedure C, DMAP (3.7 mg, 10 mol%), 2-chloro-1-phenylethan-1-ol **20** (47 mg, 0.3 mmol), *i*-Pr_2NEt (57 µL, 0.33 mmol, 1.1 equiv.) and (*i*-PrCO)$_2$O (50 µL, 0.3 mmol, 1.0 equiv.) in CH_2Cl_2 (0.2 M) gave 2-chloro-1-phenylethyl isobutyrate **S7** (57 mg, 84%) as a colourless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ_H: 1.21 (3H, d, *J* 7.0, CH(CH_3)^A(CH_3)^B, 1.25 (3H, d, *J* 7.0, CH(CH_3)^A(CH_3)^B), 2.60-2.74 (1H, m, C*H*(CH_3)$_2$), 3.69-3.85 (2H, m, CH_2Cl), 5.98 (1H, dd, *J* 8.1, 4.4, C*H*-OCOiPr), 7.28-7.46 (5H, m, 5 × Ar*H*). Spectral data in accordance with Literature.[20]

Kinetic resolution of 2-chloro-1-phenylethan-1-ol 20

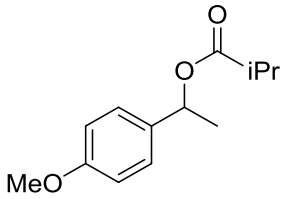


Following General Procedure D, 2-chloro-1-phenylethan-1-ol **20** (47 mg, 0.3 mmol), PS-HyperBTM **6** (3.4 mg, 1 mol%), *i*-Pr_2NEt (31 µL, 0.18 mmol, 0.6 equiv.) and $(i\text{-PrCO})_2O$ (27.4 µL, 0.165 mmol, 0.55 equiv.) in $CHCl_3$ (1.5 mL) for 7 h at 0 °C gave the crude product, which was purified by Biotage® Isolera™ 4 to gave (*S*)-2-chloro-1-phenylethan-1-ol **20** (18 mg, 38%) and (*R*)-2-chloro-1-phenylethyl isobutyrate **S7** (30 mg, 44%). **(*S*)-2-Chloro-1-phenylethan-1-ol 20**: $[\alpha]_D^{20}$ +42 (*c* 1.0, $CHCl_3$) {Lit.[21] $[\alpha]_D^{20}$ +54 (94% ee, *c* 1.0, $CHCl_3$) Chiral HPLC analysis Chiralcel OD-H (98:2 hexane:IPA, flow rate 1.0 mL min^{-1}, 220 nm, 30 °C) t_R (*S*): 19.1 min, t_R (*R*): 21.2 min, 92.875:7.125 er. **(*R*)-2-Chloro-1-phenylethyl isobutyrate S7:** $[\alpha]_D^{20}$ −60 (*c* 1.0, $CHCl_3$). Chiral HPLC analysis Chiralpak AD-H (99.8:0.2 hexane:IPA, flow rate 1.0 mL min^{-1}, 254 nm, 30 °C) t_R (*S*): 7.2 min, t_R (*R*): 8.0 min, 8.020:91.980 er. *s* = 31

1-(4-Methoxyphenyl)ethan-1-ol 21

Commercially available

1-(4-Methoxyphenyl)ethyl isobutyrate S8



Following General Procedure C, DMAP (3.7 mg, 10 mol%), 1-(4-methoxyphenyl)ethan-1-ol **21** (46 mg, 0.3 mmol), *i*-Pr_2NEt (57 µL, 0.33 mmol, 1.1 equiv.) and $(i\text{-PrCO})_2O$ (50 µL, 0.3 mmol, 1.0 equiv.) in CH_2Cl_2 (0.2 M) gave 1-(4-methoxyphenyl)ethyl isobutyrate **S8** (55 mg, 83%) as a colourless oil. ^1H NMR (400 MHz, $CDCl_3$) δ_H: 1.14 (3H, d, *J* 7.0, $CH(CH_3)^A(CH_3)^B$, 1.17 (3H, d, *J* 7.0, $CH(CH_3)^A(CH_3)^B$), 1.51 (3H, d, *J* 6.6, CH_3), 2.47-2.60 (1H, m, $CH(CH_3)_2$), 3.80 (3H, s,

OC*H*$_3$), 5.84 (1H, q, *J* 6.6, C*H*CH$_3$), 6.81-6.95 (2H, m, ArC(3,5)*H*), 7.26-7.31 (2H, m, ArC(2,6)*H*). Spectral data in accordance with Literature.[14]

Kinetic resolution of 1-(4-methoxyphenyl)ethan-1-ol 21

Following General Procedure D, 1-(4-methoxyphenyl)ethan-1-ol **21** (46 mg, 0.3 mmol), PS-HyperBTM **6** (3.4 mg, 1 mol%), *i*-Pr$_2$NEt (31 µL, 0.18 mmol, 0.6 equiv.) and (*i*-PrCO)$_2$O (27.4 µL, 0.165 mmol, 0.55 equiv.) in CHCl$_3$ (1.5 mL) for 7 h at 0 °C gave the crude product, which was purified by Biotage® Isolera™ 4 to gave (*R*)-1-(4-methoxyphenyl)ethan-1-ol **21** (24 mg, 52%) and (*S*)-1-(4-methoxyphenyl)ethyl isobutyrate **S8** (20 mg, 30%). **(*R*)-1-(4-Methoxyphenyl)ethan-1-ol 21**: $[\alpha]_D^{20}$ +29 (*c* 1.0, CHCl$_3$) {Lit.[10] $[\alpha]_D^{20}$ +56.8 (95% ee, *c* 1.0 in CHCl$_3$) Chiral HPLC analysis Chiralcel OD-H (98:2 hexane:IPA, flow rate 1.0 mL min^{-1}, 220 nm, 30 °C) t_R (*R*): 20.8 min, t_R (*S*): 24.7 min, 78.653:21.347 er. **(*S*)-1-(4-Methoxyphenyl)ethyl isobutyrate S8**: $[\alpha]_D^{20}$ −89 (*c* 1.0, CHCl$_3$). Chiral HPLC analysis Chiralcel OJ-H (99.5:0.5 hexane:IPA, flow rate 0.5 mL min^{-1}, 220 nm, 30 °C) t_R (*R*): 19.0 min, t_R (*S*): 23.4 min, 5.414:94.586 er. *s* = 31

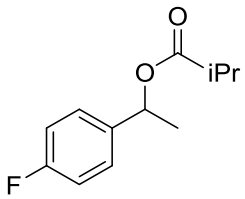
Chiral HPLC data for Table 5, recycle no. 4

(*R*)-1-(4-Methoxyphenyl)ethan-1-ol 21: Chiral HPLC analysis Chiralcel OD-H (98:2 hexane:IPA, flow rate 1.0 mL min^{-1}, 220 nm, 30 °C) t_R (*R*): 20.8 min, t_R (*S*): 24.7 min, 73.377:26.623 er; **(*S*)-1-(4-Methoxyphenyl)ethyl isobutyrate S8**: Chiral HPLC analysis Chiralcel OJ-H (99.5:0.5 hexane:IPA, flow rate 0.5 mL min^{-1}, 220 nm, 30 °C) t_R (*R*): 19.0 min, t_R (*S*): 23.4 min, 5.408:94.592 er. *s* = 28

1-(4-Fluorophenyl)ethan-1-ol 22

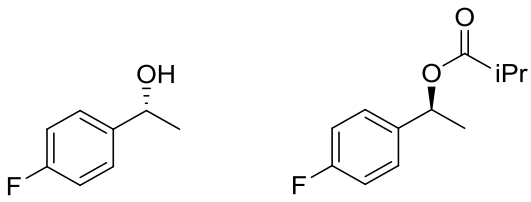
Commercially available.

1-(4-Fluorophenyl)ethyl isobutyrate S9



Following General Procedure C, DMAP (3.7 mg, 10 mol%), 1-(4-fluorophenyl)ethan-1-ol **22** (42 mg, 0.3 mmol), *i*-Pr_2NEt (57 µL, 0.33 mmol, 1.1 equiv.) and $(i\text{-PrCO})_2O$ (50 µL, 0.3 mmol, 1.0 equiv.) in CH_2Cl_2 (0.2 M) gave 1-(4-fluorophenyl)ethyl isobutyrate **S9** (56 mg, 89%) as a colourless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ_H: 1.14 (3H, d, *J* 7.0, $CH(CH_3)^A(CH_3)^B$, 1.17 (3H, d, *J* 7.0, $CH(CH_3)^A(CH_3)^B$), 1.51 (3H, d, *J* 6.6, CH_3), 2.48-2.62 (1H, m, $CH(CH_3)_2$), 5.85 (1H, q, *J* 6.6, C*H*-O-COiPr), 6.96-7.09 (2H, m, ArC(2,6)*H*), 7.26-7.38 (2H, m, ArC(3,5)*H*), Spectral data in accordance with literature.[22]

Kinetic resolution of 1-(4-fluorophenyl)ethan-1-ol 22



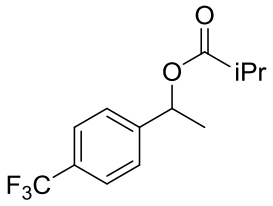
Following General Procedure D, 1-(4-fluorophenyl)ethan-1-ol **22** (42 mg, 0.3 mmol), PS-HyperBTM **6** (3.4 mg, 1 mol%), *i*-Pr_2NEt (31 µL, 0.18 mmol, 0.6 equiv.) and $(i\text{-PrCO})_2O$ (27.4 µL, 0.165 mmol, 0.55 equiv.) in $CHCl_3$ (1.5 mL) for 7 h at 0 °C gave the crude product, which was purified by Biotage® Isolera™ 4 to give (*R*)-1-(4-fluorophenyl)ethan-1-ol **22** (20 mg, 47%) and (*S*)-1-(4-fluorophenyl)ethyl isobutyrate **S9** (23 mg, 36%). **(*R*)-1-(4-Fluorophenyl)ethan-1-ol 22:** $[\alpha]_D^{20}$ +19 (*c* 0.5, $CHCl_3$) {Lit.[23] $[\alpha]_D^{20}$ +39.0 (89% ee, *c* 1.2 in $CHCl_3$). Chiral GC analysis, Agilent Cycledex-B column (length: 30 m, thickness: 0.250 mm, film thickness: 0.25 µm, carrier gas: He, linear velocity: 60 cm sec^{-1}, temperature: 100 °C (10 min), 100 to 110 °C (10 min), 110 °C to 120 °C (10 min)), t_R (*R*): 18.2 min, t_R (*S*): 19.1 min, 83.383:16.617 er. **(*S*)-1-(4-Fluorophenyl)ethyl isobutyrate S9**: $[\alpha]_D^{20}$ −22 (*c* 0.5, $CHCl_3$); Chiral HPLC analysis Chiralpak

AD-H (99.8:0.2 hexane:IPA, flow rate 1.0 mL min^{-1}, 211 nm, 30 °C) t_R (*R*): 5.2 min, t_R (*S*): 6.1 min, 8.478:91.522 er. *s* = 22

1-(4-(Trifluoromethyl)phenyl)ethan-1-ol 23

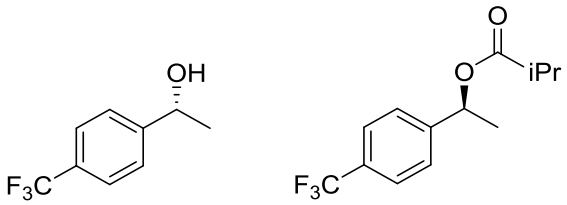
Commercially available

1-(4-(Trifluoromethyl)phenyl)ethyl isobutyrate S10



Following General Procedure C, DMAP (3.7 mg, 10 mol%), 1-(4-(trifluoromethyl)phenyl)ethan-1-ol **23** (57 mg, 0.3 mmol), *i*-Pr_2NEt (57 µL, 0.33 mmol, 1.1 equiv.) and $(i\text{-PrCO})_2O$ (50 µL, 0.3 mmol, 1.0 equiv.) in CH_2Cl_2 (0.2 M) gave 1-(4-(trifluoromethyl)phenyl)ethyl isobutyrate **S10** (67 mg, 86%) as a colourless oil. ^1H NMR (400 MHz, $CDCl_3$) δ_H: 1.17 (3H, d, *J* 7.0, $CH(CH_3)^A(CH_3)^B$), 1.19 (3H, d, *J* 7.0, $CH(CH_3)^A(CH_3)^B$), 1.53 (3H, d, *J* 6.6, CH_3), 2.52-2.65 (1H, m, $CH(CH_3)_2$), 5.89 (1H, q, *J* 6.6, C*H*-OCOiPr), 7.42-7.50 (2H, m, ArC(2,6)*H*),7.56-7.65 (2H, m, ArC(3,5)*H*). Spectral data in accordance with literature.[14]

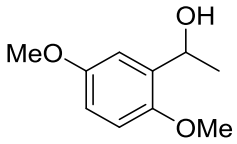
Kinetic resolution of 1-(4-(trifluoromethyl)phenyl)ethan-1-ol 23



Following General Procedure D, 1-(4-(trifluoromethyl)phenyl)ethan-1-ol **23** (57 mg, 0.3 mmol), PS-HyperBTM **6** (3.4 mg, 1 mol%), *i*-Pr_2NEt (31 µL, 0.18 mmol, 0.6 equiv.) and $(i\text{-PrCO})_2O$ (27.4 µL, 0.165 mmol, 0.55 equiv.) in $CHCl_3$ (1.5 mL) for 7 h at 0 °C gave the crude product, which was purified by Biotage® Isolera™ 4 to give (*R*)-1-(4-(trifluoromethyl)phenyl)ethan-1-ol **23** (22 mg, 39%) and 1-(4-(trifluoromethyl)phenyl)ethyl isobutyrate **S10** (36 mg, 46%). **(*R*)-1-(4-(Trifluoromethyl)phenyl)ethan-1-ol 23**: $[\alpha]_D^{20}$ +24 (*c* 1.0, $CHCl_3$) {Lit.[24] $[\alpha]_D^{20}$ +26.1 (97% ee, *c*

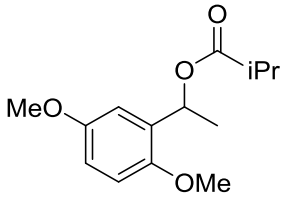
0.9, $CHCl_3$) Chiral HPLC analysis Chiralcel OJ-H (99.8:0.2 hexane:IPA, flow rate 1.0 mL min^{-1}, 220 nm, 30 °C) t_R (*R*): 39.5 min, t_R (*S*): 49.4 min, 90.781:9.219 er. **(*S*)-1-(4-(Trifluoromethyl)phenyl)ethyl isobutyrate S10**: $[\alpha]_D^{20}$ −46 (*c* 1.0, $CHCl_3$) {Lit[14] $[\alpha]_D^{20}$ −32.6 (84% ee, *c* 0.185 in $CHCl_3$) Chiral HPLC analysis Chiralpak AD-H (99.8:0.2 hexane:IPA, flow rate 1.0 mL min^{-1}, 254 nm, 30 °C) t_R (*R*): 4.7 min, t_R (*S*): 6.2 min, 13.844:86.156 er. *s* = 15

1-(2,5-Dimethoxyphenyl)ethan-1-ol 24



Following General Procedure B, 1-(2,5-dimethoxyphenyl)ethan-1-one (0.90 g, 5.0 mmol, 1.0 equiv.) and $NaBH_4$ (113.4 mg, 3.0 mmol, 0.6 equiv.) in methanol (10 mL) gave, after purification by Biotage® Isolera™ 4, 1-(2,5-dimethoxyphenyl)ethan-1-ol **24** (0.70 g, 77%) as a colourless oil ^{1}H NMR (400 MHz, $CDCl_3$) δ_H: 1.49 (3H, d, *J* 6.5, C*H*$_3$), 2.64 (1H, d, *J* 4.5, O*H*), 3.78 (3H, s, OC*H*$_3$), 3.82 (3H, s, OC*H*$_3$), 5.01-5.10 (1H, m, C*H*-OH), 6.72-6.83 (2H, m, ArC(3,4)*H*), 6.94 (1H, d, *J* 3.0, ArC(6)*H*). Spectral data in accordance with Literature.[25]

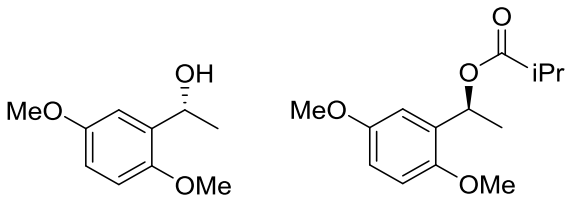
1-(2,5-Dimethoxyphenyl)ethyl isobutyrate S11



Following General Procedure C, DMAP (3.7 mg, 10 mol%), 1-(2,5-dimethoxyphenyl)ethan-1-ol **24** (55 mg, 0.3 mmol), *i*-Pr_2NEt (57 µL, 0.33 mmol, 1.1 equiv.) and (*i*-PrCO)$_2$O (50 µL, 0.3 mmol, 1.0 equiv.) in CH_2Cl_2 (0.2 M) gave 1-(2,5-dimethoxyphenyl)ethyl isobutyrate **S11** (66 mg, 88%) as a colourless oil. IR ν_{max} (thin film, cm^{-1}): 2974 (C-H), 1732 (C=O); ^{1}H NMR (400 MHz, $CDCl_3$) δ_H: 1.16-1.22 (6H, m, CH(C*H*$_3$)$_2$), 1.46 (3H, d, *J* 6.5, C*H*$_3$), 2.53-2.65 (1H, m, C*H*(CH$_3$)$_2$), 3.76 (3H, s, OC*H*$_3$), 3.79 (3H, s, OC*H*$_3$), 6.14-6.23 (1H, m, C*H*CH$_3$), 6.72-6.82 (2H, m, ArC(3,4)*H*), 6.92-6.97 (1H, m, ArC(6)*H*). ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ_C: 19.0 (CH(*C*H$_3$)^A(CH$_3$)^B), 19.1 (CH(CH$_3$)^A(*C*H$_3$)^B), 21.3 (*C*H$_3$), 34.2 (*C*H(CH$_3$)$_2$), 55.7 (ArC(5)O*C*H$_3$),

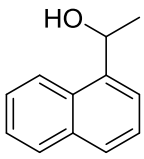
55.7 (ArC(2)O*C*H$_3$), 66.8 (*C*H-OCOiPr), 111.6 (Ar*C*(3)H), 112.1 (Ar*C*(4)H), 112.4 (Ar*C*(6)H), 132.0 (Ar*C*(1)), 150.2 (Ar*C*(2)), 153.7 (Ar*C*(5)), 176.1 (C=O); HRMS (NSI) $C_{14}H_{24}O_4N$ ($[M+NH_4]^+$, 100%) found 270.1702, requires 270.1700 (+0.8 ppm).

Kinetic resolution of 1-(2,5-dimethoxyphenyl)ethan-1-ol 24



Following General Procedure D, 1-(2,5-dimethoxyphenyl)ethan-1-ol **24** (55 mg, 0.3 mmol), PS-HyperBTM **6** (3.4 mg, 1 mol%), *i*-Pr$_2$NEt (31 µL, 0.18 mmol, 0.6 equiv.) and (*i*-PrCO)$_2$O (27.4 µL, 0.165 mmol, 0.55 equiv.) in $CHCl_3$ (1.5 mL) for 7 h at 0 °C gave the crude product, which was purified by Biotage® Isolera™ 4 to give (*R*)-1-(2,5-dimethoxyphenyl)ethan-1-ol **24** (26 mg, 48%) and (*S*)-1-(2,5-dimethoxyphenyl)ethyl isobutyrate **S11** (30 mg, 40%). **(*R*)-1-(2,5-dimethoxyphenyl)ethan-1-ol 24**: $[\alpha]_D^{20}$ +15 (*c* 1.0, $CHCl_3$) {Lit.[26] $[\alpha]_D^{20}$ +18.6 (71% ee, *c* 0.5, $CHCl_3$) Chiral HPLC analysis Chiralcel OD-H (99:1 hexane:IPA, flow rate 0.8 mL min^{-1}, 220 nm, 30 °C) t_R (*S*): 50.0 min, t_R (*R*): 53.2 min, 21.699:78.301 er. **(*S*)-1-(2,5-Dimethoxyphenyl)ethyl isobutyrate S11**: $[\alpha]_D^{20}$ −20 (*c* 1.0, $CHCl_3$); following General Procedure E, (*S*)-1-(2,5-dimethoxyphenyl)ethyl isobutyrate **S11** hydrolysed to corresponding alcohol (*S*)-1-(2,5-dimethoxyphenyl)ethan-1-ol **24** for HPLC analysis. Chiral HPLC analysis Chiralcel OD-H (99:1 hexane:IPA, flow rate 1.0 mL min^{-1}, 220 nm, 30 °C) t_R (*S*): 50.1 min, t_R (*R*): 54.6 min, 88.919:11.081 er. *s* = 14

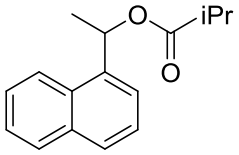
1-(Naphthalen-1-yl)ethan-1-ol 25



Following General Procedure A, methylmagnesium bromide (7.25 mL of 3.0 M solution in Et_2O, 22.13 mmol) and 1-naphthaldehyde (2.0 mL, 14.75 mmol, 1.0 equiv.) in anhydrous THF (20

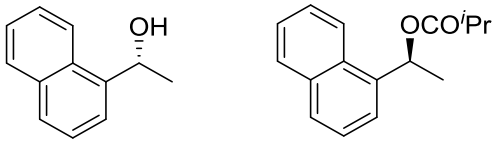
mL) at −78 °C for 16 h gave, after purification by column chromatography (80:20 hexane:EtOAc), 1-(naphthalen-1-yl)ethan-1-ol **25** (2.10 g, 80%) as a colourless solid. mp 60-62 °C {Lit.[27] mp 63-64 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ_H: 1.68 (3H, d, *J* 6.5, C*H*$_3$), 1.98 (1H, s, O*H*), 5.63-5.73 (1H, m, C*H*-OH), 7.45-7.57 (3H, m, 3 × Ar*H*), 7.66-7.71 (1H, m, Ar*H*), 7.76-7.81 (1H, m, Ar*H*), 7.86-7.90 (1H, m, Ar*H*), 8.09-8.15 (1H, m, Ar*H*). Spectral data in accordance with literature.[28]

1-(Naphthalen-1-yl)ethyl isobutyrate S12



Following General Procedure C, DMAP (3.7 mg, 10 mol%), 1-(naphthalen-1-yl)ethan-1-ol **25** (52 mg, 0.3 mmol), *i*-Pr$_2$NEt (57 µL, 0.33 mmol, 1.1 equiv.) and (*i*-PrCO)$_2$O (50 µL, 0.3 mmol, 1.0 equiv.) in CH_2Cl_2 (0.2 M) gave 1-(naphthalen-1-yl)ethyl isobutyrate **S12** (61 mg, 84%) as a colourless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ_H: 1.22 (3H, d, *J* 7.0, CH(C*H*$_3$)(CH$_3$)), 1.25 (3H, d, *J* 7.0, CH(CH$_3$)(C*H*$_3$)), 1.74 (3H, d, *J* 6.6, C*H*$_3$), 2.58-2.72 (1H, m, C*H*(CH$_3$)$_2$), 6.69 (1H, q, *J* 6.6, C*H*-OCOiPr), 7.46-7.60 (3H, m, 3 × Ar*H*), 7.64 (1H, ddd, *J* 7.2, 1.2, 0.7, Ar*H*), 7.82 (1H, app dt, *J* 8.3, 1.1, Ar*H*), 7.87-7.93 (1H, m, Ar*H*), 8.13 (1H, m, Ar*H*). Data in accordance with literature.[29]

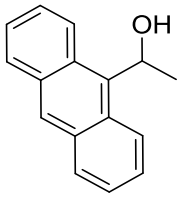
Kinetic resolution of 1-(naphthalen-1-yl)ethan-1-ol 25



Following General Procedure D, 1-(naphthalen-1-yl)ethan-1-ol **25** (52 mg, 0.3 mmol), PS-HyperBTM **6** (3.4 mg, 1 mol%), *i*-Pr$_2$NEt (31 µL, 0.18 mmol, 0.6 equiv.) and (*i*-PrCO)$_2$O (27.4 µL, 0.165 mmol, 0.55 equiv.) in $CHCl_3$ (1.5 mL) for 7 h at 0 °C gave the crude product, which was purified by Biotage® Isolera™ 4 to give (*R*)-1-(naphthalen-1-yl)ethan-1-ol **25** (25 mg, 48%) and (*S*)-1-(naphthalen-1-yl)ethyl isobutyrate **S12** (31 mg, 43%). **(*R*)-1-(naphthalen-1-yl)ethan-1-ol 25**: mp 60-62 °C {Lit.[27] mp 63-64 °C. $[\alpha]_D^{20}$ +57 (*c* 1.0, $CHCl_3$) {Lit.[14] $[\alpha]_D^{20}$ +32.5 (46% ee, *c*

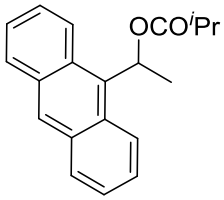
0.47, $CHCl_3$) Chiral HPLC analysis Chiralcel OD-H (95:5 hexane:IPA, flow rate 1.0 mL min^{-1}, 254 nm, 30 °C) t_R (*S*): 14.3 min, t_R (*R*): 23.5 min, 10.166:89.834 er. **(*S*)-1-(Naphthalen-1-yl)ethyl isobutyrate S12**: $[\alpha]_D^{20}$ −39 (*c* 1.0, $CHCl_3$) {Lit.[14] $[\alpha]_D^{20}$ −17.2 (85% ee, *c* 0.41, $CHCl_3$) Chiral HPLC analysis Chiralcel OD-H (99.5:0.5 hexane:IPA, flow rate 1.0 mL min^{-1}, 254 nm, 30 °C) t_R (*R*): 6.0 min, t_R (*S*): 8.5 min, 5.667:94.333 er. *s* = 41

1-(Anthracen-9-yl)ethan-1-ol 26



Following General Procedure B, 1-(anthracen-9-yl)ethan-1-one (1.0 g, 4.54 mmol, 1.0 equiv.) and $NaBH_4$ (260 mg, 6.82 mmol, 1.2 equiv.) in methanol (10 mL) gave, after purification by Biotage® Isolera™ 4, 1-(anthracen-9-yl)ethan-1-ol **26** (0.91g, 91%) as an orange-yellow solid. mp: 110-112°C {Lit.[30] 116-117 °C}. ^{1}H NMR (400 MHz, $CDCl_3$) δ_H: 1.94 (3H, d, *J* 6.8, C*H*$_3$), 6.49 (1H, q, *J* 6.8, C*H*-OH), 7.42-7.54 (4H, m, 4 × Ar*H*), 7.97-8.05 (2H, m, 2 × Ar*H*), 8.40 (1H, s, ArC(10)*H*), 8.68 (2H, d, *J* 8.7, 2 × Ar*H*). Spectroscopic data in accordance with the literature.[31]

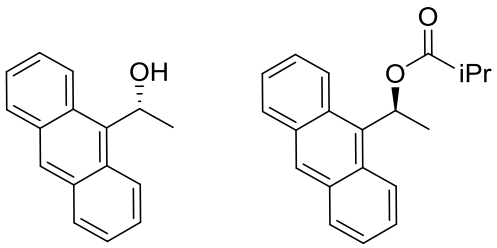
1-(Anthracen-9-yl)ethyl isobutyrate S13



Following General Procedure C, DMAP (3.7 mg, 10 mol%), 1-(anthracen-9-yl)ethan-1-ol **26** (67 mg, 0.3 mmol), *i*-Pr_2NEt (57 µL, 0.33 mmol, 1.1 equiv.) and (*i*-PrCO)$_2$O (50 µL, 0.3 mmol, 1.0 equiv.) in CH_2Cl_2 (0.2 M) gave 1-(anthracen-9-yl)ethyl isobutyrate **S13** (76 mg, 87%) as a yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ_H: 1.08 (3H, d, *J* 7.0, CH(C*H*$_3$)^A(CH$_3$)^B, 1.19 (3H, d, *J* 7.0, CH(CH$_3$)^A(C*H*$_3$)^B, 1.94 (3H, d, *J* 7.0, CHC*H*$_3$), 2.54-2.67 (1H, m, C*H*(CH$_3$)$_2$), 7.38-7.58 (5H, m,

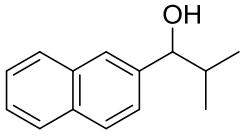
4 × Ar*H* & C*H*-OCOiPr), 7.97-8.05 (2H, m, 2 × Ar*H*), 8.42 (1H, s, ArC(10)*H*), 8.61-8.65 (2H, m, 2 × Ar*H*). Spectroscopic data in accordance with the literature.[14]

Kinetic resolution of 1-(anthracen-9-yl)ethan-1-ol 26



Following General Procedure D, 1-(anthracen-9-yl)ethan-1-ol **26** (67 mg, 0.3 mmol), PS-HyperBTM **6** (3.4 mg, 1 mol%), *i*-Pr_2NEt (31 µL, 0.18 mmol, 0.6 equiv.) and $(i\text{-PrCO})_2O$ (27.4 µL, 0.165 mmol, 0.55 equiv.) in $CHCl_3$ (1.5 mL) for 7 h at r.t. gave the crude product, which was purified by Biotage® Isolera™ 4 to gave (*R*)-1-(anthracen-9-yl)ethan-1-ol **26** (39 mg, 59%) and (*S*)-1-(anthracen-9-yl)ethyl isobutyrate **S13** (20 mg, 23%). **(*R*)-1-(Anthracen-9-yl)ethan-1-ol 26**: mp: 110-112°C {Lit.[30] 116-117 °C}. $[\alpha]_D^{20}$ +15.2 (*c* 1.0, $CHCl_3$) {Lit.[32] $[\alpha]_D^{20}$ +18.5 (99% ee, *c* 1.0, $CHCl_3$), Chiral HPLC analysis Chiralpak AD-H (90:10 hexane:IPA, flow rate 1.0 mL min^{-1}, 220 nm, 30 °C) t_R (*R*): 11.3 min, t_R (*S*): 14.9 min, 87.7687:12.233 er. **(*S*)-1-(Anthracen-9-yl)ethyl isobutyrate S13**: $[\alpha]_D^{20}$ −27.3 (*c* 1.0, $CHCl_3$). Chiral HPLC analysis Chiralpak AD-H (99.8:0.2 hexane:IPA, flow rate 1.0 mL min^{-1}, 254 nm, 30 °C) t_R (*R*): 6.6 min, t_R (*S*): 10.1 min, 2.887:97.113 er. *s* = 77

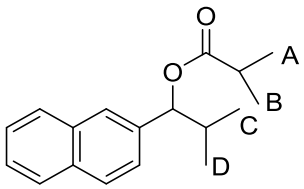
2-Methyl-1-(naphthalen-2-yl)propan-1-ol 27



Following General Procedure A, isopropylmagnesium bromide (7.50 mL of 1.0 M solution in THF, 7.50 mmol) and 2-naphthaldehyde (0.78 g, 5.00 mmol) in anhydrous THF (10 mL) at –78 °C for 16 h gave, after purification by column chromatography (80:20 hexane:EtOAc), 2-methyl-1-(naphthalen-2-yl)propan-1-ol **27** (0.74 mg, 74%) as a yellow oil. ^1H NMR (400 MHz, $CDCl_3$) δ_H: 0.84 (3H, d, *J* 6.9, CH(C*H*$_3$)(CH_3), 1.05 (3H, d, *J* 6.6, CH(CH_3)(C*H*$_3$)), 1.94 (1H, s, O*H*),

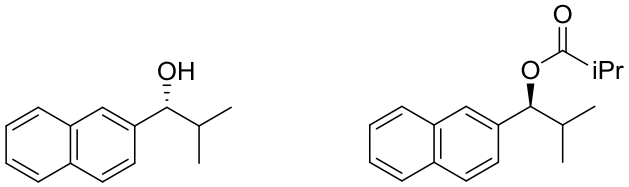
2.00-2.15 (1H, m, C*H*(CH_3)$_2$), 4.54 (1H, d, *J* 6.9, C*H*-OH), 7.43-7.51 (3H, m, 3 × Ar*H*), 7.74-7.77 (1H, m, Ar*H*), 7.80-7.86 (3H, m, 3 × Ar*H*). Spectral data in accordance with literature.[33]

2-Methyl-1-(naphthalen-2-yl)propyl isobutyrate S14



Following General Procedure C, DMAP (3.7 mg, 10 mol%) 2-methyl-1-(naphthalen-2-yl)propan-1-ol **27** (60 mg, 0.3 mmol), *i*-Pr_2NEt (57 µL, 0.33 mmol, 1.1 equiv.) and (*i*-PrCO)$_2$O (50 µL, 0.3 mmol, 1.0 equiv.) in CH_2Cl_2 (0.2 M) gave 2-methyl-1-(naphthalen-2-yl)propyl isobutyrate **S14** (73 mg, 90%) as a colourless oil. IR ν_{max} (thin film, cm^{-1}): 2968 (C-H), 1732 (C=O); ^{1}H NMR (400 MHz, $CDCl_3$) δ_H: 0.89 (3H, d, *J* 6.8, CH(C*H*$_3$)^C(CH_3)^D, 1.07 (3H, d, *J* 6.8, CH(CH_3)^C(C*H*$_3$)^D, 1.21 (3H, d, *J* 7.0, CH(C*H*$_3$)^A(CH_3)^B, 1.26 (3H, d, *J* 7.0, CH(CH_3)^A(C*H*$_3$)^B, 2.19-2.32 (1H, m, C*H*(CH_3)^C(CH_3)^D, 2.59-2.72 C*H*(CH_3)^A(CH_3)^B, 5.68 (1H, d, *J* 7.6, C*H*-OCOiPr), 7.43-7.55 (3H, m, 3 × Ar*H*), 7.76-7.91 (4H, m, 4 × Ar*H*); ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ_C: 18.6 (*C*H_3), 19.0 (2 × *C*H_3), 19.1 (*C*H_3), 33.7 (*C*H(CH_3)$_2$), 34.4 (*C*H(CH_3)$_2$), 80.8 (*C*H-OCOiPr), 124.8 (Ar*C*H), 126.0 (Ar*C*H), 126.1 (Ar*C*H), 126.3 (Ar*C*H), 127.7 (Ar*C*H), 128.0 (Ar*C*H), 128.1 (Ar*C*H), 133.0 (Ar*C*), 133.1 (Ar*C*), 137.5 (Ar*C*), 176.3 (C=O); HRMS (NSI) $C_{18}H_{26}O_2N$ ([M+NH_4]$^+$, 100%) found 288.1960, requires 288.1958 (+0.7 ppm).

Kinetic resolution of 2-methyl-1-(naphthalen-2-yl)propan-1-ol 27



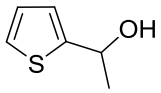
Following General Procedure D, 2-methyl-1-(naphthalen-2-yl)propan-1-ol **27** (60 mg, 0.3 mmol), PS-HyperBTM **6** (3.4 mg, 1 mol%), *i*-Pr_2NEt (31 µL, 0.18 mmol, 0.6 equiv.) and (*i*-PrCO)$_2$O (27.4 µL, 0.165 mmol, 0.55 equiv.) in $CHCl_3$ (1.5 mL) for 7 h at 0 °C gave the crude product, which was purified by Biotage® Isolera™ 4 to give (*R*)-2-methyl-1-(naphthalen-2-yl)propan-1-ol **27** (31 mg, 52%) and (*S*)-2-methyl-1-(naphthalen-2-yl)propyl isobutyrate **S14** (34

mg, 42%). **(*R*)-2-Methyl-1-(naphthalen-2-yl)propan-1-ol 27**: $[\alpha]_D^{20}$ +25 (*c* 1.0, $CHCl_3$); Chiral HPLC analysis Chiralcel OD-H (95:5 hexane:IPA, flow rate 1.0 mL min^{-1}, 254 nm, 30 °C) t_R (*S*): 13.9 min, t_R (*R*): 15.6 min, 8.741:91.259 er. **(*S*)-2-Methyl-1-(naphthalen-2-yl)propyl isobutyrate S14**: $[\alpha]_D^{20}$ −78 (*c* 1.0, $CHCl_3$); Chiral HPLC analysis Chiralpak AD-H (99.8:0.2 hexane:IPA, flow rate 1.0 mL min^{-1}, 270 nm, 30 °C) t_R (*R*): 7.0 min, t_R (*S*): 8.9 min, 0.415:99.585 er. *s* = 622

Chiral HPLC data for Table 5, recycle no. 7

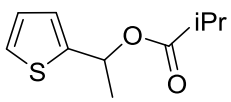
(*R*)-2-Methyl-1-(naphthalen-2-yl)propan-1-ol 27: Chiral HPLC analysis Chiralcel OD-H (95:5 hexane:IPA, flow rate 1.0 mL min^{-1}, 254 nm, 30 °C) t_R (*S*): 13.9 min, t_R (*R*): 15.6 min, 19.293:80.707 er. **(*S*)-2-Methyl-1-(naphthalen-2-yl)propyl isobutyrate S14**: Chiral HPLC analysis Chiralpak AD-H (99.8:0.2 hexane : IPA, flow rate 1.0 mL min^{-1}, 270 nm, 30 °C) t_R (*R*): 7.0 min, t_R (*S*): 8.9 min, 0.783:99.217 er. *s* = 238

1-(Thiophen-2-yl)ethan-1-ol 28



Following General Procedure B, 1-(thiophen-2-yl)ethan-1-one (0.94 g, 7.5 mmol, 1.0 equiv.) and $NaBH_4$ (283 mg, 7.5 mmol, 1.0 equiv.) in methanol (15 mL) gave, after purification by Biotage® Isolera™ 4, 1-(thiophen-2-yl)ethan-1-ol **28** (0.75 g, 79%) as a colourless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ_H: 1.61 (3H, d, *J* 6.4, C*H*$_3$), 1.99 (1H, d, *J* 4.3, O*H*), 5.07-5.19 (1H, m, C*H*-OH), 6.94- 7.00 (2H, m, ArC(3,4)*H*), 7.24 (1H, dd, *J* 4.8, 1.4, ArC(5)*H*). Spectroscopic data in accordance with the literature.[34]

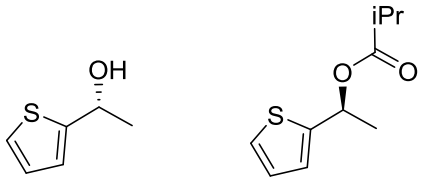
1-(Thiophen-2-yl)ethyl isobutyrate S15



Following General Procedure C, DMAP (3.7 mg, 10 mol%), 1-(thiophen-2-yl)ethan-1-ol **28** (38 mg, 0.3 mmol), *i*-Pr_2NEt (57 µL, 0.33 mmol, 1.1 equiv.) and (*i*-PrCO)$_2$O (50 µL, 0.3 mmol, 1.0 equiv.) in CH_2Cl_2 (0.2 M) gave 1-(thiophen-2-yl)ethyl isobutyrate **S15** (47 mg, 79%) as a

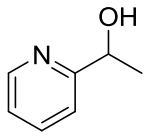
colourless oil. IR ν_{max} (thin film, cm^{-1}): 2976 (C-H), 1732 (C=O); ^{1}H NMR (400 MHz, $CDCl_3$) δ_H: 1.15 (3H, d, *J* 7.0, CH(C*H*$_3$)^A(CH$_3$)^B, 1.18 (3H, d, *J* 7.0, CH(CH$_3$)^A(C*H*$_3$)^B, 1.63 (3H, d, *J* 6.6, C*H*$_3$), 2.48-2.62 (1H, m, C*H*(CH$_3$)$_2$), 6.17 (1H, dd, *J* 6.6, 0.8, C*H*-CH$_3$), 6.95 (1H, dd, *J* 5.1, 3.5, ArC(4)*H*), 7.01-7.06 (1H, m, ArC(3)*H*), 7.24 (1H, dd, *J* 5.1, 1.2, ArC(5)*H*); $^{13}C\{^{1}H\}$ NMR (101 MHz, $CDCl_3$) δ_C: 18.9 (2 × *C*H$_3$), 22.2 (*C*H$_3$), 34.2 (*C*H(CH$_3$CH$_3$)), 67.5 (*C*H-OCOiPr), 125.0 (Ar*C*H), 125.1 (Ar*C*H), 126.6 (Ar*C*H), 144.9 (Ar*C*), 176.4 (C=O). HRMS (NSI) $C_{10}H_{14}O_2SNa$ ($[M+Na]^+$, 100%) found 221.0604, requires 211.0607 (−1.3 ppm).

Kinetic resolution of 1-(thiophen-2-yl)ethan-1-ol 28



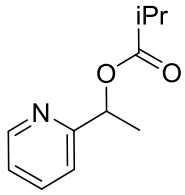
Following General Procedure D, 1-(thiophen-2-yl)ethan-1-ol **28** (64 mg, 0.5 mmol), PS-HyperBTM **6** (5.7 mg, 1 mol%), *i*-Pr$_2$NEt (51 µL, 0.3 mmol, 0.6 equiv.) and (*i*-PrCO)$_2$O (46 µL, 0.28 mmol, 0.55 equiv.) in $CHCl_3$ (2.5 mL) for 7 h at 0 °C gave the crude product, which was purified by Biotage® Isolera™ 4 to give (*R*)-1-(thiophen-2-yl)ethan-1-ol **28** (26 mg, 41%) and (*S*)-1-(thiophen-2-yl)ethyl isobutyrate **S15** (39 mg, 39%). **(*R*)-1-(Thiophen-2-yl)ethan-1-ol 28:** $[\alpha]_D^{20}$ +31 (*c* 1.0, $CHCl_3$) {Lit.[35] $[\alpha]_D^{20}$ +26 (98% ee, *c* 2.0, $CHCl_3$) Chiral GC analysis, Agilent Cycledex-B column (length: 30 m, thickness: 0.250 mm, film thickness: 0.25 µm, carrier gas: He, linear velocity: 60 cm sec^{-1}, temperature: 100 °C (10 min), 100 °C to 110 °C (10 min), 110 °C to 120 °C (10 min)) t_R (*R*): 16.7 min, t_R (*S*): 17.4 min, 89.649:10.351 er. **(*S*)-1-(Thiophen-2-yl)ethyl isobutyrate S15**: $[\alpha]_D^{20}$ −121 (*c* 1.0, $CHCl_3$); Chiral HPLC analysis Chiralpak AD-H (99.8:0.2 hexane:IPA, flow rate 1.0 mL min^{-1}, 254 nm, 30 °C) t_R (*R*): 5.3 min, t_R (*S*): 5.8 min, 8.865:91.135 er. *s* = 25

1-(Pyridin-2-yl)ethan-1-ol 29



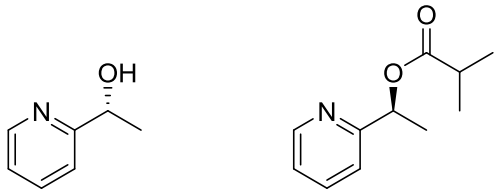
Following General Procedure B, 1-(pyridin-2-yl)ethan-1-one (0.91 g, 7.5 mmol, 1.0 equiv.) and $NaBH_4$ (283 mg, 7.50 mmol, 1.0 equiv.) in methanol (15 mL) gave, after purification by Biotage® Isolera™ 4, 1-(pyridin-2-yl)ethan-1-ol **29** (0.78 g, 84%) as a colourless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ_H: 1.50 (3H, d, *J* 6.6, C*H*$_3$), 4.30 (1H, s, O*H*), 4.83-4.93 (1H, m, C*H*-OH), 7.16-7.22 (1H, m, ArC(5)*H*), 7.25-7.29 (1H, m, ArC(3)*H*), 7.68 (1H, app. td, *J* 7.7, 1.8, ArC(4)*H*), 8.51-8.55 (1H, m, ArC(6)*H*). Spectroscopic data in accordance with the literature.[36]

1-(Pyridin-2-yl)ethyl isobutyrate S16



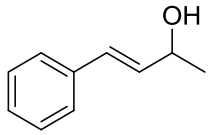
Following General Procedure C, DMAP (3.7 mg, 10 mol%), 1-(pyridin-2-yl)ethan-1-ol **29** (37 mg, 0.3 mmol), *i*-Pr_2NEt (57 µL, 0.33 mmol, 1.1 equiv.) and (*i*-PrCO)$_2$O (50 µL, 0.3 mmol, 1.0 equiv.) in CH_2Cl_2 (0.2 M) gave 1-(pyridin-2-yl)ethyl isobutyrate **S16** (45 mg, 78%) as a colourless oil. IR ν_{max} (thin film, cm^{-1}): 2976 (C-H), 1732 (C=O); ^{1}H NMR (400 MHz, $CDCl_3$) δ_H: 1.09-1.17 (6H, m, CH(C*H*$_3$)$_2$), 1.52 (3H, d, *J* 6.7, CH-C*H*$_3$), 2.47-2.62 (1H, m, C*H*(CH$_3$)$_2$), 5.86 (1H, q, *J* 6.6, C*H*CH$_3$), 7.14 (1H, ddd, *J* 7.6, 4.9, 1.2, ArC(5)*H*), 7.24-7.31 (1H, m, ArC(3)*H*), 7.62 (1H, app td, *J* 7.7, 1.8, ArC(4)*H*), 8.50-8.55 (1H, m, ArC(6)*H*); ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ_C: 18.8 (CH(*C*H$_3$)^A(CH$_3$)^B), 18.9 (CH(CH$_3$)^A(*C*H$_3$)^B), 20.7 (*C*H$_3$), 34.2 (*C*H(CH$_3$)$_2$), 72.5 (*C*H-OCOiPr), 120.1 (Ar*C*(5)H), 122.6 (Ar*C*(3)H), 136.9 (Ar*C*(4)H), 149.0 (Ar*C*(6)H), 160.5 (Ar*C*(2)), 176.2 (C=O); HRMS (NSI) $C_{11}H_{16}O_2N$ ([M+H]$^+$, 100%) found 194.1175, requires 194.1176 (−0.3 ppm).

Kinetic resolution of 1-(pyridin-2-yl)ethan-1-ol 29



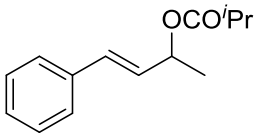
Following General Procedure D, 1-(pyridin-2-yl)ethan-1-ol **29** (62 mg, 0.5 mmol), PS-HyperBTM **6** (5.7 mg, 1 mol%), *i*-Pr_2NEt (51 µL, 0.3 mmol, 0.6 equiv.) and $(i\text{-PrCO})_2O$ (46 µL, 0.28 mmol, 0.55 equiv.) in $CHCl_3$ (2.5 mL) for 7 h at 0 °C gave the crude product, which was purified by Biotage® Isolera™ 4 to give (*R*)-1-(pyridin-2-yl)ethan-1-ol **29** (33 mg, 54%) and (*S*)-1-(pyridin-2-yl)ethyl isobutyrate **S16** (20 mg, 21%). **(*R*)-1-(Pyridin-2-yl)ethan-1-ol 29**: $[\alpha]_D^{20}$ +6 (*c* 1.0, $CHCl_3$) {Lit.[37] $[\alpha]_D^{20}$ +12.4 (67% ee, *c* 0.5, $CHCl_3$) Chiral HPLC analysis Chiralcel OD-H (99:1 hexane:IPA, flow rate 1.0 mL min^{-1}, 254 nm, 30 °C) t_R (*R*): 18.2 min, t_R (*S*): 21.4 min, 55.705:44.295 er. **(*S*)-1-(Pyridin-2-yl)ethyl isobutyrate S16**: $[\alpha]_D^{20}$ −30 (*c* 1.0, $CHCl_3$); following General Procedure E, (*S*)-1-(pyridin-2-yl)ethyl isobutyrate **S16** hydrolysed to corresponding alcohol (*S*)-1-(pyridin-2-yl)ethan-1-ol **29** for HPLC analysis. Chiral HPLC analysis Chiralcel OD-H (99:1 hexane:IPA, flow rate 1.0 mL min^{-1}, 254 nm, 30 °C) t_R (*R*): 18.5 min, t_R (*S*): 21.6 min, 36.632:63.368 er. *s*=1.9.

(*E*)-4-Phenylbut-3-en-2-ol 30



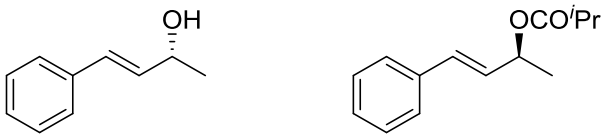
Following General Procedure B, (*E*)-4-phenylbut-3-en-2-one (1.5 g, 10.27 mmol, 1.0 equiv.) and $NaBH_4$ (389 mg, 10.27, 1.0 equiv.) in methanol (15 mL) gave, after purification by Biotage® Isolera™ 4, (*E*)-4-phenylbut-3-en-2-ol **30** (1.2 g, 79%) as a colourless oil. ^1H NMR (400 MHz, $CDCl_3$) δ_H: 1.39 (3H, d, *J* 6.4, C*H*$_3$), 1.81 (1H, s, O*H*), 4.45-4.54 (1H, m, C(2)*H*-OH), 6.28 (1H, dd, *J* 15.9, 6.4, C(3)*H*), 6.53-6.62 (1H, dd, *J* 15.9, 0.7, C(4)*H*), 7.21-7.43 (5H, m, 5 × Ar*H*). Spectroscopic data in accordance with the literature.[38]

(*E*)-4-Phenylbut-3-en-2-yl isobutyrate S17



Following General Procedure C, DMAP (3.7 mg, 10 mol%), (*E*)-4-phenylbut-3-en-2-ol **30** (44 mg, 0.3 mmol), *i*-Pr$_2$NEt (57 µL, 0.33 mmol, 1.1 equiv.) and (*i*-PrCO)$_2$O (50 µL, 0.3 mmol, 1.0 equiv.) in CH_2Cl_2 (0.2 M) gave (*E*)-4-phenylbut-3-en-2-yl isobutyrate **S17** (57 mg, 87%) as a colourless oil. IR ν_{max} (thin film, cm^{-1}): 2976 (C-H), 1726 (C=O);^{1}H NMR (500 MHz, $CDCl_3$) δ_H: 1.18 (3H, d, *J* 7.0, CH(C*H*$_3$)^A(CH$_3$)^B, 1.19 (3H, d, *J* 7.0, CH(CH$_3$)^A(C*H*$_3$)^B, 1.40 (3H, d, *J* 6.5, C*H*$_3$), 2.50-2.61 (1H, m, C*H*(CH$_3$)$_2$), 5.48-5.57 (1H, m, C*H*-OCOiPr), 6.21 (1H, dd, *J* 16.0, 6.6, C(3)*H*), 6.60 (1H, d, *J* 16.0, C(4)*H*), 7.21-7.42 (5H, m, 5 × Ar*H*); ^{13}C{^{1}H} NMR (126 MHz, $CDCl_3$) δ_C: 19.01 (CH(*C*H$_3$)^A(CH$_3$)^B), 19.04 (CH(CH$_3$)^A(*C*H$_3$)^B), 20.4 (*C*H$_3$), 34.2 (*C*H(CH$_3$)$_2$), 70.6 (*C*H-OCOiPr), 126.6 (2 × Ar*C*H), 127.9 (PhCH=*C*H), 128.6 (2 × Ar*C*H), 129.0 (Ar*C*H), 131.3 (Ph*C*H=CH), 136.4 (Ar*C*(1)), 176.4 (C=O); HRMS(ESI) $C_{14}H_{18}O_2Na$([M+Na]$^+$, 100%) found 241.1196, requires 241.1199 (+1.2 ppm).

Kinetic resolution of (*E*)-4-phenylbut-3-en-2-ol 30



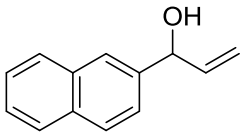
Following General Procedure D, (*E*)-4-phenylbut-3-en-2-ol **30** (44 mg, 0.3 mmol), PS-HyperBTM **6** (3.4 mg, 1 mol%), *i*-Pr$_2$NEt (31 µL, 0.18 mmol, 0.6 equiv.) and (*i*-PrCO)$_2$O (27.4 µL, 0.165 mmol, 0.55 equiv.) in $CHCl_3$ (1.5 mL) for 7 h at 0 °C gave the crude product, which was purified by Biotage® Isolera™ 4, to give (*R*)-(*E*)-4-phenylbut-3-en-2-ol **30** (22 mg, 50%) and (*S*)-(*E*)-4-phenylbut-3-en-2-yl isobutyrate **S17** (27 mg, 41%). **(*R*)-(*E*)-4-Phenylbut-3-en-2-ol 30**: $[\alpha]_D^{20}$ +20 (*c* 1.0, $CHCl_3$) {Lit.[39] $[\alpha]_D^{20}$ +27.8 (90% ee, *c* 0.92, $CHCl_3$) Chiral HPLC analysis Chiralcel OD-H (95:5 hexane:IPA, flow rate 1.0 mL min^{-1}, 220 nm, 30 °C) t_R (*R*): 14.7 min, t_R (*S*): 24.1 min, 84.504:15.496 er. **(*S*)-(*E*)-4-Phenylbut-3-en-2-yl isobutyrate S17**: $[\alpha]_D^{20}$ −87 (*c*

1.0, $CHCl_3$); Chiral HPLC analysis Chiralpak AD-H (99.8:0.2 hexane:IPA, flow rate 1.0 mL min^{-1}, 270 nm, 30 °C) t_R (*R*): 5.7 min, t_R (*S*): 6.5 min, 10.693:89.307 er. *s* = 17

Chiral HPLC data for Table 5, recycle no. 10

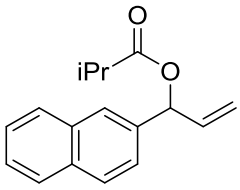
(*R*)-(*E*)-4-Phenylbut-3-en-2-ol 30: Chiral HPLC analysis Chiralcel OD-H (95:5 hexane:IPA, flow rate 1.0 mL min^{-1}, 220 nm, 30 °C) t_R (*R*): 14.7 min, t_R (*S*): 24.1 min, 77.487:22.513 er. **(*S*)-(*E*)-4-Phenylbut-3-en-2-yl isobutyrate S17**: Chiral HPLC analysis Chiralpak AD-H (99.8:0.2 hexane:IPA, flow rate 1.0 mL min^{-1}, 270 nm, 30 °C) t_R (*R*): 5.7 min, t_R (*S*): 6.5 min, 8.975:91.025 er. *s* = 18

1-(Naphthalen-2-yl)prop-2-en-1-ol 31



Following General Procedure A, vinylmagnesium bromide (5.5 mL of 0.7 M solution in THF, 3.84 mmol) and 2-naphthaldehyde (400 mg, 2.56 mmol) in anhydrous THF (13 mL) at −78 °C for 16 h gave, after purification by column chromatography (80:20 Hexane:EtOAc), 1-(naphthalen-2-yl)prop-2-en-1-ol 1-(naphthalen-2-yl)prop-2-en-1-ol **31** (439 mg, 93%) as a yellow oil. ^{1}H NMR (500 MHz, $CDCl_3$): 2.04 (1H, d, *J* 5, O*H*), 5.25 (1H, m, C(3)H_{trans}), 5.37-5.45 (2H, m, C(1)*H* &C(3)H_{cis}), 6.13 (1H, ddd, *J* 17.0, 10.3, 6.0, C(2)*H*), 7.43-7.52 (3H, m, 3 × Ar*H*), 7.79-7.88 (4H, m, 4 × Ar*H*). Spectroscopic data in accordance with the literature.[40]

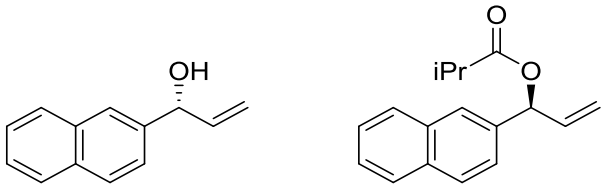
1-(Naphthalen-2-yl)allyl isobutyrate S18



Following General Procedure C, DMAP (3.7 mg, 10 mol%), 1-(naphthalen-2-yl)prop-2-en-1-ol **31** (55 mg, 0.3 mmol), *i*-Pr_2NEt (57 µL, 0.33 mmol, 1.1 equiv.) and (*i*-PrCO)$_2$O (50 µL, 0.3 mmol, 1.0 equiv.) in CH_2Cl_2 (0.2 M) gave 1-(naphthalen-2-yl)allyl isobutyrate **S18** (65 mg, 85%) as a colourless oil. ^{1}H NMR (500 MHz, $CDCl_3$) δ_H: 1.19 (3H, d, *J* 7.0, CH(CH_3)^A(CH_3)^B), 1.22

(3H, d, *J* 7.0, CH(CH_3)^A(CH_3)^B), 2.65 (1H, m, C*H*(CH_3)$_2$), 5.28 (1H, app dt, *J* 10.5, 1.3, C(3)H_{trans}), 5.35 (1H, app dt, *J* 17.2, 1.3, C(3)H_{cis}), 6.09 (1H, ddd, *J* 17.2, 10.5, 5.7, C(2)*H*), 6.42 (1H, d, *J* 5.7, C(1)*H*), 7.43-7.53 (3H, m, 3 × Ar*H*), 7.79–7.88 (4H, m, 4 × Ar*H*). Spectroscopic data in accordance with the literature.[40]

Kinetic resolution of 1-(naphthalen-2-yl)prop-2-en-1-ol 31

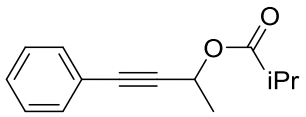


Following General Procedure D, 1-(naphthalen-2-yl)prop-2-en-1-ol **31** (55 mg, 0.3 mmol), PS-HyperBTM **6** (3.4 mg, 1 mol%), *i*-Pr_2NEt (31 µL, 0.18 mmol, 0.6 equiv.) and (*i*-PrCO)$_2$O (27.4 µL, 0.165 mmol, 0.55 equiv.) in $CHCl_3$ (1.5 mL) for 7 h at 0 °C gave the crude product, which was purified by Biotage® Isolera™ 4 to give (*R*)-1-(naphthalen-2-yl)prop-2-en-1-ol **31** (23 mg, 42%) and (*S*)-1-(naphthalen-2-yl)allyl isobutyrate **S18** (34 mg, 45%). **(*R*)-1-(Naphthalen-2-yl)prop-2-en-1-ol 31:** $[\alpha]_D^{20}$ +9.6 (*c* 1.0, $CHCl_3$) {Lit.[40] $[\alpha]_D^{20}$ −6.3 (ent) (99% ee, *c* 0.7, $CHCl_3$) Chiral HPLC analysis Chiralcel OJ-H (80:20 hexane:IPA, flow rate 1.0 mL min^{-1}, 270 nm, 30 °C) t_R (*S*): 11.0 min, t_R (*R*): 13.2 min, 3.332:96.668 er. **(*S*)-1-(Naphthalen-2-yl)allyl isobutyrate S18:** $[\alpha]_D^{20}$ −42.3 (*c* 1.0, $CHCl_3$) {Lit.[40] $[\alpha]_D^{20}$ +54.0 (ent) (99% ee, *c* 1.0, $CHCl_3$) Chiral HPLC analysis Chiralcel OJ-H (95:5 hexane:IPA, flow rate 0.5 mL min^{-1}, 270 nm, 30 °C) t_R (*R*): 15.2 min, t_R (*S*): 22.0 min, 11.943:88.057 er. *s* = 25

4-Phenylbut-3-yn-2-ol 32

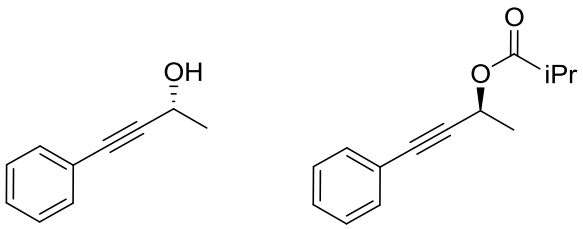
Commercially available

4-Phenylbut-3-yn-2-yl isobutyrate S19



Following General Procedure C, DMAP (3.7 mg, 10 mol%), 4-phenylbut-3-yn-2-ol **32** (44 mg, 0.3 mmol), *i*-Pr_2NEt (57 µL, 0.33 mmol, 1.1 equiv.) and $(i\text{-PrCO})_2O$ (50 µL, 0.3 mmol, 1.0 equiv.) in CH_2Cl_2 (0.2 M) gave 4-phenylbut-3-yn-2-yl isobutyrate **S19** (57 mg, 88%) as a colourless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ_H: 1.19 (3H, d, *J* 7.0, $CH(CH_3)^A(CH_3)^B$), 1.20 (3H, d, *J* 7.0, $CH(CH_3)^A(CH_3)^B$, 1.57 (3H, d, *J* 6.7, CH_3), 2.52-2.65 (1H, m, $CH(CH_3)_2$), 5.70 (1H, q, *J* 6.7, C*H*-OCOiPr), 7.27-7.34 (3H, m, 3 × Ar*H*), 7.41-7.47 (2H, m, 2 × Ar*H*). Spectral data in accordance with Literature.[7]

Kinetic resolution of 4-phenylbut-3-yn-2-ol 27



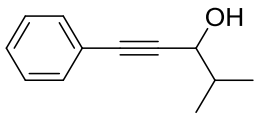
Following General Procedure D, (*R*)-4-phenylbut-3-yn-2-ol **32** (44 mg, 0.3 mmol), PS-HyperBTM **6** (3.4 mg, 1 mol%), *i*-Pr_2NEt (31 µL, 0.18 mmol, 0.6 equiv.) and $(i\text{-PrCO})_2O$ (27.4 µL, 0.165 mmol, 0.55 equiv.) in $CHCl_3$ (1.5 mL) for 7 h at 0 °C gave the crude product, which was purified by Biotage® Isolera™ 4 to give (*R*)-4-phenylbut-3-yn-2-ol **32** (17 mg, 39%) and (*S*)-4-phenylbut-3-yn-2-yl isobutyrate **S19** (29 mg, 45%). **(*R*)-4-Phenylbut-3-yn-2-ol 32**: $[\alpha]_D^{20}$ +25 (*c* 1.0, $CHCl_3$) {Lit.[41] $[\alpha]_D^{20}$ +32.4 (96% ee, *c* 0.966, $CHCl_3$) Chiral HPLC analysis Chiralcel OD-H (95:5 hexane:IPA, flow rate 1.0 mL min^{-1}, 220 nm, 30 °C) t_R (*R*): 11.0 min, t_R (*S*): 27.3 min, 94.914:5.086 er. **(*S*)-4-Phenylbut-3-yn-2-yl isobutyrate S19**: $[\alpha]_D^{20}$ −23 (*c* 1.0, $CHCl_3$)

Following General Procedure E, (*S*)-4-phenylbut-3-yn-2-yl isobutyrate **S19** hydrolysed to corresponding (*S*)-4-phenylbut-3-yn-2-ol **32** for HPLC analysis. Chiral HPLC analysis Chiralcel OD-H (95:5 hexane:IPA, flow rate 1.0 mL min^{-1}, 220 nm, 30 °C) t_R (*R*): 10.9 min, t_R (*S*): 27.2 min, 11.457:88.543 er. *s*=23.2

Chiral HPLC data for Table 5, recycle no. 2

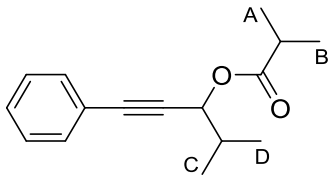
(*R*)-4-Phenylbut-3-yn-2-ol 32: Chiral HPLC analysis Chiralcel OD-H (95:5 hexane:IPA, flow rate 1.0 mL min^{-1}, 220 nm, 30 °C) t_R (*R*): 11.0 min, t_R (*S*): 27.3 min, 93.311:6.689 er. **(*S*)-4-Phenylbut-3-yn-2-yl isobutyrate S19**: Following General Procedure E, (*S*)-4-phenylbut-3-yn-2-yl isobutyrate **S19** hydrolysed to corresponding (*S*)-4-phenylbut-3-yn-2-ol **32** for HPLC analysis. Chiral HPLC analysis Chiralcel OD-H (95:5 hexane:IPA, flow rate 1.0 mL min^{-1}, 220 nm, 30 °C) t_R (*R*): 10.9 min, t_R (*S*): 27.2 min, 14.648:85.352 er. *s* =16

4-Methyl-1-phenylpent-1-yn-3-ol 33



n-Butyllithium (2.6 mL, 6.60 mmol, 1.2 equiv.) was added to a solution of phenyl acetylene (0.56 g, 5.50 mmol, 1.0 equiv.) in anhydrous THF (10 mL) at –78 °C, and stirred for 30 min. Isobutyraldehyde (0.50 mL, 5.50 mmol, 1.0 equiv.) was added and stirred at −78 °C for 1 h, then r.t. for 16 h. Sat. aq. NH_4Cl (10 mL) was added, and the mixture extracted with Et_2O (2 × 25 mL). The combined organic fractions were dried ($MgSO_4$) and evaporated *in vacuo*. The crude product was purified by Biotage® Isolera™ 4 [SNAP UltRa 25 g, 75 mL min^{-1}, CH_2Cl_2:EtOAc (95:5 5 CV, 95:5 to 60:40 10 CV, 60:40 5 CV, 16 mL each)] to give 4-methyl-1-phenylpent-1-yn-3-ol **33** (0.71 mg, 74%) as colourless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ_H: 1.06 (3H, d, *J* 6.8, $CH(CH_3)^A(CH_3)^B$, 1.20 (3H, d, *J* 6.8, $CH(CH_3)^A(CH_3)^B$, 1.92-2.05 (1H, m, $CH(CH_3)^A(CH_3)^B$), 2.49 (1H, s, O*H*), 4.41 (1H, d, *J* 5.7, C*H*-OH), 7.28-7.33 (3H, m, Ar*H*), 7.41-7.45 (2H, m, 2 × Ar*H*). Spectral data in accordance with literature.[42]

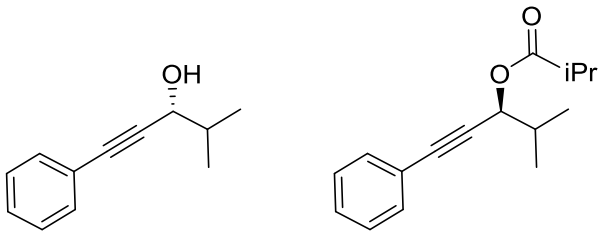
4-Methyl-1-phenylpent-1-yn-3-yl isobutyrate S20



Following General Procedure C, DMAP (3.7 mg, 10 mol%), 4-methyl-1-phenylpent-1-yn-3-ol **33** (52 mg, 0.3 mmol), *i*-Pr_2NEt (57 µL, 0.33 mmol, 1.1 equiv.) and $(i\text{-PrCO})_2O$ (50 µL, 0.3

mmol, 1.0 equiv.) in CH_2Cl_2 (0.2 M) gave 4-methyl-1-phenylpent-1-yn-3-yl isobutyrate **S20** (63 mg, 86%) as a colourless oil. IR ν_{max} (thin film, cm^{-1}): 2968 (C-H), 1736 (C=O); ^1H NMR (400 MHz, $CDCl_3$) δ_H: 0.89 (3H, d, *J* 6.8, CH(C*H*$_3$)^C(CH$_3$)^D), 1.07 (3H, d, *J* 6.8, CH(CH$_3$)^C(C*H*$_3$)^D), 1.20 (3H, d, *J* 7.0, CH(C*H*$_3$)^A(CH$_3$)^B), 1.21 (3H, d, *J* 7.0, CH(CH$_3$)^A(C*H*$_3$)^B), 2.19-2.32 (1H, m, C*H*(CH$_3$)^C(CH$_3$)^D), 2.01-2.16 (1H, m, C*H*(CH$_3$)^A(CH$_3$)^B), 2.55-2.68 (1H, m, C*H*(CH$_3$)^A(CH$_3$)^B), 5.46 (1H, d, *J* 5.7, C*H*-OCOiPr), 7.26-7.33 (3H, m, 3 × Ar*H*), 7.40-7.47 (2H, m, 2 × Ar*H*); $^{13}C\{^1H\}$ NMR (101 MHz, $CDCl_3$) δ_C: 17.8 (CH(*C*H$_3$)^C(CH$_3$)^D), 18.4 (CH(CH$_3$)^C(*C*H$_3$)^D), 18.9 (CH(*C*H$_3$)^A(CH$_3$)^B), 19.2 (CH(CH$_3$)^A(*C*H$_3$)^B), 32.8 (*C*H(CH$_3$)^C(CH$_3$)^D), 34.2 (*C*H(CH$_3$)^A(CH$_3$)^B), 69.2 (*C*H-OCOiPr), 85.66 (*C*≡C), 85.69 (C≡*C*), 122.7 (Ar*C*), 128.3 (Ar*C*(2,6)H), 128.6 (Ar*C*(4)H), 132.0 (Ar*C*(3,5)H), 176.2 (C=O); HRMS (NSI) $C_{16}H_{21}O_2$ ($[M+H]^+$, 100%) found 245.1538, requires 245.1536 (+0.8 ppm).

Kinetic resolution of 4-methyl-1-phenylpent-1-yn-3-ol 33



Following General Procedure D, 4-methyl-1-phenylpent-1-yn-3-ol **33** (52 mg, 0.3 mmol), PS-HyperBTM **6** (3.4 mg, 1 mol%), *i*-Pr$_2$NEt (31 µL, 0.18 mmol, 0.6 equiv.) and (*i*-PrCO)$_2$O (27.4 µL, 0.165 mmol, 0.55 equiv.) in $CHCl_3$ (1.5 mL) for 7 h at 0 °C gave the crude product, which was purified by Biotage® Isolera™ 4 to give (*R*)-4-methyl-1-phenylpent-1-yn-3-ol **33** (21 mg, 40%) and (*S*)-4-methyl-1-phenylpent-1-yn-3-yl isobutyrate **S20** (35 mg, 48%). **(*R*)-4-Methyl-1-phenylpent-1-yn-3-ol 33**: $[\alpha]_D^{20}$ +1.2 (*c* 1.0, $CHCl_3$) {Lit.[43] $[\alpha]_D^{20}$ 5.06 (75% ee, *c* 10.0, $CHCl_3$)} Chiral HPLC analysis Chiralcel OD-H (95:5 hexane:IPA, flow rate 1.0 mL min^{-1}, 220 nm, 30 °C) t_R (*R*): 7.8 min, t_R (*S*): 15.1 min, 95.367:4.633 er. **(*S*)-4-Methyl-1-phenylpent-1-yn-3-yl isobutyrate S20**: $[\alpha]_D^{20}$ −98 (*c* 1.0, $CHCl_3$); Chiral HPLC analysis Chiralpak AD-H (100:0 hexane:IPA, flow rate 1.0 mL min^{-1}, 254 nm, 30 °C) t_R (*S*): 9.0 min, t_R (*R*): 11.6 min, 89.283:10.717 er. *s* = 26

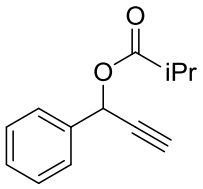
Chiral HPLC data for Table 5, recycle no. 5

(*R*)-4-Methyl-1-phenylpent-1-yn-3-ol 33: Chiral HPLC analysis Chiralcel OD-H (95:5 hexane:IPA, flow rate 1.0 mL min^{-1}, 220 nm, 30 °C) t_R (*R*): 7.8 min, t_R (*S*): 15.1 min, 91.052:8.948 er. **(*S*)-4-Methyl-1-phenylpent-1-yn-3-yl isobutyrate S20**: Chiral HPLC analysis Chiralpak AD-H (100:0 hexane:IPA, flow rate 1.0 mL min^{-1}, 254 nm, 30 °C) t_R (*S*): 9.0 min, t_R (*R*): 11.6 min, 87.781:12.220 er. *s* = 18

1-Phenylprop-2-yn-1-ol 34

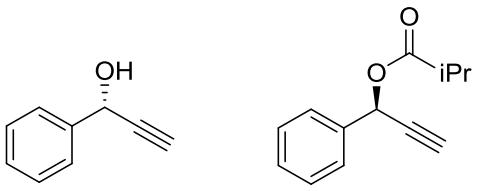
Commercially available

1-Phenylprop-2-yn-1-yl isobutyrate S21



Following General Procedure C, DMAP (3.7 mg, 10 mol%), 1-phenylprop-2-yn-1-ol **34** (40 mg, 0.3 mmol), *i*-Pr_2NEt (57 µL, 0.33 mmol, 1.1 equiv.) and $(i\text{-PrCO})_2O$ (50 µL, 0.3 mmol, 1.0 equiv.) in CH_2Cl_2 (0.2 M) gave 1-phenylprop-2-yn-1-yl isobutyrate **S21** (50 mg, 82%) as a colourless oil. IR ν_{max} (thin film, cm^{-1}): 3288 (CC-H), 2976 (C-H), 1735 (C=O); ^{1}H NMR (400 MHz, $CDCl_3$) δ_H: 1.17 (3H, d, *J* 7.0, $CH(CH_3)^A(CH_3)^B$, 1.21 (3H, d, *J* 7.0, $CH(CH_3)^A(CH_3)^B$, 2.54-2.69 (2H, m, $CH(CH_3)^A(CH_3)^B$ & C≡C*H*), 6.46 (1H, d, *J* 2.3, C*H*-OCO^{i}Pr), 7.32-7.44 (3H, m, ArC(3,4,5)*H*), 7.48-7.57 (2H, m, ArC(2,6)*H*); ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ_C: 18.8 (2 × CH_3), 34.0 ($CH(CH_3)_2$), 65.1 (*C*H-OCOiPr), 75.4 (*C*CH), 80.5 (C*C*H), 127.6 (Ar*C*(2,6)H), 128.8 (Ar*C*(3,5)H), 129.0 (Ar*C*(4)H), 136.8 (Ar*C*(1)), 175.8 (C=O); HRMS (EI) $C_{13}H_{14}O_2$ ([M], 100%) found 202.1000, requires 202.0994 (+3.0 ppm).

Kinetic resolution of 1-phenylprop-2-yn-1-ol 34



Following General Procedure D, 1-phenylprop-2-yn-1-ol **34** (66 mg, 0.5 mmol), PS-HyperBTM **6** (5.7 mg, 1 mol%), *i*-Pr_2NEt (51 µL, 0.3 mmol, 0.6 equiv.) and $(i\text{-PrCO})_2O$ (46 µL, 0.28 mmol, 0.55 equiv.) in $CHCl_3$ (2.5 mL) for 7 h at 0 °C gave the crude product, which was purified by Biotage® Isolera™ 4 to give (*R*)-1-phenylprop-2-yn-1-ol **34** (28 mg, 42%) and (*S*)-1-phenylprop-2-yn-1-yl isobutyrate **S21** (46 mg, 46%). **(*R*)-1-Phenylprop-2-yn-1-ol 34**: $[\alpha]_D^{20}$ −11.5 (*c* 1.0, $CHCl_3$) {Lit.[44] $[\alpha]_D^{20}$ −26.8 (*c* 3.5, $CHCl_3$) Chiral HPLC analysis Chiralcel OD-H (95:5 hexane:IPA, flow rate 1.0 mL min^{-1}, 220 nm, 30 °C) t_R (*R*): 12.1 min, t_R (*S*) : 19.0 min, 67.334:32.666 er. **(*S*)-1-Phenylprop-2-yn-1-yl isobutyrate S21:** $[\alpha]_D^{20}$ +8.8 (*c* 1.0, $CHCl_3$); Chiral HPLC or GC conditions could not be found to separate ester enantiomers, and hydrolysis of ester according to General Procedure E led to a mixture of products. The selectivity factor, was therefore calculated using er of the alcohol and reaction conversion, which was determined by ^{1}H NMR spectroscopic analysis of the crude reaction (conversion = 52%, *s* = 3).

***trans*-2-Phenylcyclohexan-1-ol 35 and *cis*-2-phenylcyclohexan-1-ol 37**

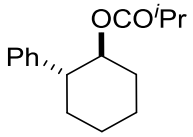
Following General Procedure B, 2-phenylcyclohexan-1-one (1.39 g, 8.00 mmol, 1.0 equiv.) and $NaBH_4$ (302 mg, 8.00 mmol, 1.0 equiv.) in methanol (20 mL) gave, after purification by Biotage® Isolera™ 4, *trans*-2-phenylcyclohexan-1-ol **35** (0.65 g, 46%) and *cis*-2-phenylcyclohexan-1-ol **37** (0.58 g, 41%).

***trans*-2-Phenylcyclohexan-1-ol 35:** mp 56-58°C {Lit.[45] mp 60-61 °C.} ^{1}H NMR (400 MHz, $CDCl_3$) δ_H: 1.27-1.61 (5H, m, C*H*$_2$, C*H*$_2$ & CH-O*H*), 1.72-1.80 (1H, m, C*H*^{A}H^B), 1.82-1.91 (2H, m, C*H*$_2$), 2.08-2.16 (1H, m, CH^A*H*^{B}), 2.38-2.48 (1H, m, C(2)*H*), 3.62-3.72 (1H, m, C(1)*H*), 7.21-

7.28 (3H, m, ArC(3,4,5)*H*), 7.30-7.37 (2H, m, ArC(2,6)*H*). Spectral data in accordance with literature.[46]

***cis*-2-Phenylcyclohexan-1-ol 37:** mp 39-41°C {Lit.[47] mp 40-42 °C} ^{1}H NMR (400 MHz, $CDCl_3$) δ_H: 1.24-1.78 (6H, m, CH_2, CH_2 & CH_2), 1.83-2.15 (3H, m, CH_2 & O*H*), 2.71-2.79 (1H, m, C(2)*H*), 4.03 (1H, m, C(1)*H*), 7.19-7.30 (3H, m, ArC(3,4,5)*H*), 7.30-7.39 (2H, m, ArC(2,6)*H*). Spectral data in accordance with literature.[48]

***trans*-2-Phenylcyclohexyl isobutyrate S22**



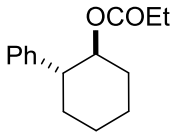
Following General Procedure C, DMAP (3.7 mg, 10 mol%), *trans*-2-phenylcyclohexan-1-ol **35** (53 mg, 0.3 mmol), *i*-Pr_2NEt (57 µL, 0.33 mmol, 1.1 equiv.) and (*i*-$PrCO)_2O$ (50 µL, 0.3 mmol, 1.0 equiv.) in CH_2Cl_2 (0.2 M) gave *trans*-2-phenylcyclohexyl isobutyrate **S22** (60 mg, 81%) as a colourless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ_H: 0.74 (3H, d, *J* 7.0, CH(CH_3)^A(CH_3)^B), 0.89 (3H, d, *J* 7.0, CH(CH_3)^A(CH_3)^B), 1.27-1.65 (4H, m, CH_2 & CH_2), 1.73-1.98 (3H, m, CH_2 & CH^AH^B), 2.05-2.15 (1H, m, CH^AH^B), 2.17-2.3 (1H, m, C*H*(CH_3)^A(CH_3)^B), 2.61-2.71 (1H, m, C(2)*H*), 4.97 (1H, td, *J* 10.7, 4.3, C(1)*H*), 7.12-7.21 (3H, m, ArC(3,4,5)*H*), 7.21-7.29 (2H, m, ArC(2,6)*H*). Spectral data in accordance with literature.[14]

Kinetic resolution of *trans*-2-phenylcyclohexan-1-ol 35

Following General Procedure D, *trans*-2-phenylcyclohexan-1-ol **35** (53 mg, 0.3 mmol), PS-HyperBTM **6** (3.4 mg, 1 mol%), *i*-Pr_2NEt (31 µL, 0.18 mmol, 0.6 equiv.) and (*i*-$PrCO)_2O$ (27.4 µL, 0.165 mmol, 0.55 equiv.) in $CHCl_3$ (1.5 mL) for 7 h at r.t. gave the crude product, which was purified by Biotage® Isolera™ 4 to (1*R*,2*S*)-2-phenylcyclohexan-1-ol **35** (31 mg, 59%) and (1*S*, 2*R*)-2-phenylcyclohexyl isobutyrate **S22** (16 mg, 22%). **(1*R*,2*S*)-2-Phenylcyclohexan-1-ol 35**: mp 55-57 °C {Lit.[45] mp 60-61 °C}. $[\alpha]_D^{20}$ −10.5 (*c* 1.0, $CHCl_3$) {Lit.[49] (*ent*) $[\alpha]_D^{20}$ +11.3 (66 %ee, *c*

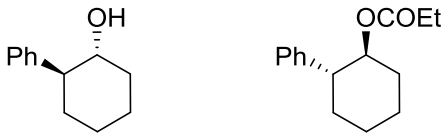
0.4, $CHCl_3$} Chiral HPLC analysis, Chiralcel OD-H (99:1 hexane:IPA, flow rate 1.0 mL min^{-1}, 220 nm, 30 °C) t_R (1*S*,2*R*): 11.4 min, t_R (1*R*,2*S*): 12.6 min, 31.095:68.905 er. **(1*S*,2*R*)-2-Phenylcyclohexyl isobutyrate S22**: $[\alpha]_D^{20}$ +21 (*c* 1.0, $CHCl_3$) {Lit.[50] $[\alpha]_D^{20}$ +11.3 (82% ee, *c* 1.5, $CHCl_3$), Following General Procedure E, (1*S*,2*R*)-2-phenylcyclohexyl isobutyrate **S22** hydrolysed to corresponding (1*S*,2*R*)-2-phenylcyclohexan-1-ol **35** for HPLC analysis. Chiral HPLC analysis Chiralcel OD-H (99:1 hexane:IPA, flow rate 1.0 mL min^{-1}, 220 nm, 30 °C) t_R (1*S*,2*R*): 11.4 min, t_R (1*R*,2*S*): 12.7 min, 96.486:3.514 er. *s* = 40

***trans*-2-Phenylcyclohexyl propionate 36**



Following General Procedure C, DMAP (3.7 mg, 10 mol%), *trans*-2-phenylcyclohexan-1-ol **35** (53 mg, 0.30 mmol), *i*-Pr_2NEt (57 µL, 0.33 mmol, 1.1 equiv.) and $(EtCO)_2O$ (38 µL, 0.30 mmol, 1.0 equiv.) in CH_2Cl_2 (0.2 M) gave *trans*-2-phenylcyclohexyl propionate **36** (58 mg, 83%) as a colourless oil. ^1H NMR (400 MHz, $CDCl_3$) δ_H: 0.85 (3H, t, *J* 7.6, C*H*$_3$), 1.28-1.65 (4H, m, C*H*$_2$ & C*H*$_2$), 1.74-2.16 (6H, m, C*H*$_2$, C*H*$_2$ & C*H*$_2$CH$_3$), 2.62-2.72 (1H, m, C(2)*H*), 4.94-5.03 (1H, m, C(1)*H*), 7.14-7.22 (3H, m, ArC(3,4,5)*H*), 7.23-7.30 (2H, m, ArC(2,6)*H*). Spectral data in accordance with literature.[51]

Kinetic resolution of *trans*-2-phenylcyclohexan-1-ol 35



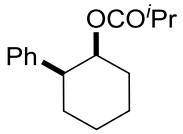
Following General Procedure D, *trans*-2-phenylcyclohexan-1-ol **35** (53 mg, 0.3 mmol), PS-HyperBTM **6** (3.4 mg, 1 mol%), *i*-Pr_2NEt (31 µL, 0.18 mmol, 0.6 equiv.) and $(EtCO)_2O$ (27.4 µL, 0.165 mmol, 0.55 equiv.) in $CHCl_3$ (1.5 mL) for 16 h at r.t. gave the crude product, which was purified by Biotage® Isolera™ 4 to give (1*R*,2*S*)-2-phenylcyclohexan-1-ol **35** (22 mg, 42%) and (1*S*,2*R*)-2-phenylcyclohexyl propionate **36** (35 mg, 50%). **(1*R*,2*S*)-2-Phenylcyclohexan-1-ol 35**: mp 56-58°C {Lit.[45] mp 60-61 °C}. $[\alpha]_D^{20}$ −30.1 (*c* 1.0, $CHCl_3$)) {Lit.[49] (*ent*) $[\alpha]_D^{20}$ +11.3 (66

%ee, *c* 0.4, $CHCl_3$} Chiral HPLC analysis, Chiralcel OD-H (99:1 hexane:IPA, flow rate 1.0 mL min^{-1}, 220 nm, 30 °C) t_R (1*S*,2*R*): 11.7 min, t_R (1*R*,2*S*): 12.9 min, 0.714:99.286 er. **(1*S*,2*R*)-2-Phenylcyclohexyl propionate 36**: $[\alpha]_D^{20}$ +18.4 (*c* 1.0, $CHCl_3$); Chiral HPLC analysis, Chiralpak AD-H (100:0 hexane:IPA, flow rate 1.0 mL min^{-1}, 211 nm, 30 °C) t_R (1*S*,2*R*): 7.7 min, t_R (1*R*,2*S*): 8.2 min 91.380:8.620 er. *s* =51

Chiral HPLC data for Table 5, recycle no. 3

(1*R*,2*S*)-2-Phenylcyclohexan-1-ol 35: Chiral HPLC analysis, Chiralcel OD-H (99:1 hexane:IPA, flow rate 1.0 mLmin^{-1}, 220 nm, 30 °C) t_R (1*S*,2*R*): 11.7 min, t_R (1*R*,2*S*): 12.9 min, 2.005:97.995 er. **(1*S*,2*R*)-2-Phenylcyclohexyl propionate 36**: Chiral HPLC analysis, Chiralpak AD-H (100:0 hexane:IPA, flow rate 1.0 mLmin^{-1}, 211 nm, 30 °C) t_R (1*S*,2*R*): 7.7 min, t_R (1*R*,2*S*): 8.2 min 90.534:9.466 er. *s* = 37

***cis*-2-Phenylcyclohexyl isobutyrate S23**

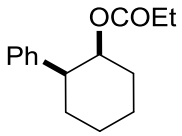


Following General Procedure C, DMAP (3.7 mg, 10 mol%), *cis*-2-phenylcyclohexyl isobutyrate **37** (53 mg, 0.3 mmol), *i*-Pr_2NEt (57 µL, 0.33 mmol, 1.1 equiv.) and (*i*-$PrCO)_2O$ (50 µL, 0.3 mmol, 1.0 equiv.) in CH_2Cl_2 (0.2 M) gave *cis*-2-phenylcyclohexyl isobutyrate **S23** (48 mg, 65%) as a colourless oil. ^1H NMR (400 MHz, $CDCl_3$) δ_H: 1.00 (3H, d, *J* 7.0, $CH(CH_3)^A(CH_3)^B$), 1.06 (3H, d, *J* 7.0, $CH(CH_3)^A(CH_3)^B$), 1.37-1.68 (4H, m, CH_2 & CH_2), 1.74-1.82 (1H, m, CH^AH^B), 1.87-1.96 (1H, m, CH^AH^B), 1.99-2.15 (2H, m, CH_2), 2.35-2.48 (1H, $CH(CH_3)^A(CH_3)^B$), 2.80 (1H, app dt, *J* 12.9, 3.0, C(2)*H*), 5.15 (1H, m, C(1)*H*), 7.12-7.30 (5H, m, 5 × Ar*H*). Spectral data in accordance with literature.[52]

Kinetic resolution of *cis*-2-phenylcyclohexan-1-ol 37

Following General Procedure D, *cis*-2-phenylcyclohexyl isobutyrate **37** (53 mg, 0.3 mmol), PS-HyperBTM **6** (34 mg, 10 mol%), *i*-Pr_2NEt (31 µL, 0.18 mmol, 0.6 equiv.) and $(i\text{-PrCO})_2O$ (27.4 µL, 0.165 mmol, 0.55 equiv.) in $CHCl_3$ (1.5 mL) for 48 h at r.t. gave the crude product, which was purified by Biotage® Isolera™ 4 to give (1*R*,2*R*)-2-phenylcyclohexan-1-ol **37** (40 mg, 75%) and (1*S*,2*S*)-2-phenylcyclohexyl isobutyrate **S23** (8 mg, 11%). **(1*R*,2*R*)-2-Phenylcyclohexan-1-ol 37**: mp 40-42 °C {Lit.[47] mp 40-42 °C} $[\alpha]_D^{20}$ −18.4 (*c* 1.0, $CHCl_3$) Chiralcel OD-H (99:1 hexane:IPA, flow rate 1.0 mL min^{-1}, 211 nm, 30 °C) t_R (1*S*,2*S*): 10.6 min, t_R (1*R*,2*R*): 12.9 min, 43.995:56.005 er. **(1*S*,2*S*)-2-Phenylcyclohexyl isobutyrate S23**: $[\alpha]_D^{20}$ +60.1 (*c* 1.0, $CHCl_3$) {Lit.[50] $[\alpha]_D^{20}$ +71.9 (90% ee, *c* 1.02, $CHCl_3$). Following General Procedure E, (1*S*,2*S*)-2-phenylcyclohexyl isobutyrate **S23** hydrolysed to corresponding (1*S*,2*S*)-2-phenylcyclohexan-1-ol **37** for HPLC analysis. Chiralcel OD-H (99:1 hexane:IPA, flow rate 1.0 mL min^{-1}, 211 nm, 30 °C) t_R (1*S*,2*S*): 10.6 min, t_R (1*R*,2*R*): 12.9 min, 84.870:15.130 er. $s = 6$

***cis*-2-Phenylcyclohexyl propionate 38**

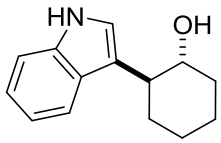


Following General Procedure C, DMAP (3.7 mg, 10 mol%), *cis*-2-phenylcyclohexyl isobutyrate **37** (53 mg, 0.30 mmol), *i*-Pr_2NEt (57 µL, 0.33 mmol, 1.1 equiv.) and $(EtCO)_2O$ (38 µL, 0.30 mmol, 1.0 equiv.) in CH_2Cl_2 (0.2 M) gave *cis*-2-phenylcyclohexyl propionate **38** (60 mg, 86%) as a colourless oil. ^1H NMR (400 MHz, $CDCl_3$) δ_H: 1.02 (3H, t, *J* 7.6, CH_3), 1.36-1.70 (4H, m, CH_2 & CH_2), 1.71-1.81 (1H, m, CH^AH^B), 1.86-1.96 (1H, m, CH^AH^B), 1.99-2.30 (4H, m, CH_2 & CH_2CH$_3$), 2.73-2.84 (1H, C(2)*H*), 5.12-5.20 (1H, m, C(1)*H*), 7.14-7.31 (5H, m, 5 × Ar*H*). Spectral data in accordance with literature.[51]

Kinetic resolution of *cis*-2-phenylcyclohexan-1-ol 37

Following General Procedure D, *cis*-2-phenylcyclohexan-1-ol **37** (53 mg, 0.3 mmol), PS-HyperBTM **6** (3.4 mg, 1 mol%), *i*-Pr_2NEt (31 µL, 0.18 mmol, 0.6 equiv.) and $(EtCO)_2O$ (21 µL, 0.165 mmol, 0.55 equiv.) in $CHCl_3$ (1.5 mL) for 16 h at r.t. gave the crude product, which was purified by Biotage® Isolera™ 4 to give (1*R*,2*R*)-2-phenylcyclohexan-1-ol **37** (20 mg, 38%) and (1*S*,2*S*)-2-phenylcyclohexyl propionate **38** (32 mg, 46%). **(1*R*,2*R*)-2-Phenylcyclohexan-1-ol 37**: mp 39-41 °C {Lit.[47] mp 40-42 °C}. $[\alpha]_D^{20}$ −26.8 (*c* 1.0, $CHCl_3$) Chiral HPLC analysis Chiralcel OD-H (99:1 hexane:IPA, flow rate 1.0 mL min^{-1}, 220 nm, 30 °C) t_R (1*S*,2*S*): 10.2 min, t_R (1*R*,2*R*): 12.5 min, 16.114:83.886 er. **(1*S*,2*S*)-2-Phenylcyclohexyl propionate 38**: $[\alpha]_D^{20}$ +56.3 (*c* 1.0, $CHCl_3$); following General Procedure E, (1*S*,2*S*)-2-phenylcyclohexyl propionate **38** hydrolysed to corresponding (1*S*,2*S*)-2-phenylcyclohexan-1-ol **37** for HPLC analysis. Chiral HPLC analysis Chiralcel OD-H (99:1 hexane:IPA, flow rate 1.0 mL min^{-1}, 220 nm, 30 °C) t_R (1*S*,2*S*): 10.2 min, t_R (1*R*,2*R*): 12.5 min, 84.420:15.580 er. *s* = 36

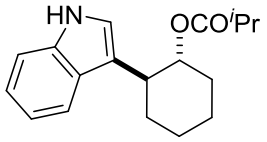
***trans*-2-(1*H*-Indol-3-yl)cyclohexan-1-ol 39**



According to a literature procedure,[51] n-butyllithium (4.0 mL, 7.6 mmol, 2.5 M) was added dropwise to a solution of indole (900 mg, 7.6 mmol, 1.0 equiv.) in THF (20 mL) at −78 °C under nitrogen atmosphere, and stirred for 30 min. Boron trifluoride etherate complex in ether (1.25 mL, 4.1 mmol) was added and the reaction mixture was stired for another 15 min. Cyclohexene oxide (0.95 mL, 9.4 mmol, 1.25 equiv.) was added dropwise at −78 °C and stirred at r.t. for 2.5 h. Sat. aq. NH_4Cl (10 mL) was added, and the mixture extracted with Et_2O (2 × 25 mL). The combined organic fractions were dried ($MgSO_4$) and evaporated *in vacuo*. The crude product was purified by Biotage® Isolera™ 4 [SNAP UltRa 25 g, 75 mL min^{-1}, hexane:EtOAc (100:0 2 CV,

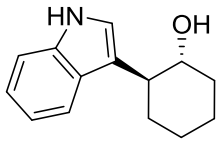
100:0 to 70:40 40 CV, (16 ml each))] to give *trans*-2-(1*H*-indol-3-yl)cyclohexan-1-ol **39** (1.1 g, 67%) as a colourless solid. mp 152-154°C {Lit.[51] 154-156 °C}. ^{1}H NMR (400 MHz, $CDCl_3$) δ_H: 1.34-1.53 (3H, m, CH_2, CH^AH^B), 1.65-2.22 (6H, m, CH_2, CH_2, CH^AH^B, O*H*), 2.71-2.81 (1H, m, C(2)*H*), 3.74-3.83 (1H, m, C(1)*H*), 7.05-7.16 (2H, m, 2 × Ar*H*), 7.17-7.25 (1H, m, Ar*H*), 7.35-7.42 (1H, m, Ar*H*), 7.70-7.77 (1H, m, Ar*H*), 8.10 (1H, s, N*H*). Spectral data in accordance with literature.[51]

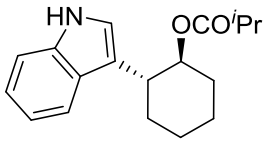
trans-2-(1*H*-Indol-3-yl)cyclohexyl isobutyrate S24



Following General Procedure C, DMAP (3.7 mg, 10 mol%), *trans*-2-(1*H*-indol-3-yl)cyclohexan-1-ol **39** (65 mg, 0.30 mmol), *i*-Pr_2NEt (57 µL, 0.33 mmol, 1.1 equiv.) and (*i*-PrCO)$_2$O (50 µL, 0.30 mmol, 1.0 equiv.) in CH_2Cl_2 (0.2 M) gave *trans*-2-(1*H*-indol-3-yl)cyclohexyl isobutyrate **S24** (70 mg, 82%) as a colourless oil. IR ν_{max} (thin film, cm^{-1}): 2931 (C-H), 1708 (C=O); ^{1}H NMR (400 MHz, $CDCl_3$) δ_H: 0.67 (3H, d, *J* 7.0, CH(CH_3)^A(CH_3)^B), 0.91 (3H, d, *J* 7.0, CH(CH_3)^A(CH_3)^B), 1.35-1.70 (4H, m, C(3)H_AH$_B$, C(4)H_AH$_B$, C(5)H_AH$_B$, C(6)H_AH$_B$), 1.75-1.95 (2H, m, C(4)H$_A$$H_B$, C(5)H$_A$$H_B$), 2.04-2.18 (2H, m, C(3)H$_A$$H_B$, C(6)H$_A$$H_B$), 2.22 (1H, app. sept., *J* 7.0, C*H*(CH_3)$_2$), 3.00-3.10 (1H, m, C(2)*H*), 4.97-5.07 (1H, m, C(1)*H*), 6.96 (1H, d, *J* 2.4, Ind-C(2)*H*), 7.07-7.12 (1H, m, Ind-C(5)*H*), 7.13-7.19 (1H, m, Ind-C(6)*H*), 7.31 (1H, app d, *J* 8.0, Ind-C(7)*H*), 7.68 (1H, app d, *J* 7.8, Ind-C(4)*H*), 8.03 (1H, s, N*H*); ^{13}C{^{1}H} NMR (126 MHz, $CDCl_3$) δ_C: 18.6 (*C*H$_3$^{A}), 18.9 (*C*H$_3$^{B}), 24.9 (*C*(5)H$_2$), 26.0 (*C*(4)H$_2$), 32.5 (*C*(6)H$_2$), 33.4 (*C*(3)H$_2$), 34.1 (*C*H(CH_3)$_2$), 40.2 (*C*(2)H), 76.5 (*C*(1)H), 111.1 (Ind-*C*(7)H), 118.5 (Ind-*C*(3)), 119.1 (Ind-*C*(5)H), 119.3 (Ind-*C*(4)H), 120.3 (Ind-*C*(2)H), 121.8 (Ind-*C*(6)H), 127.6 (Ind-*C*(3a)), 136.1 (Ind-*C*(7a)), 177.0 (C=O); HRMS (NSI) $C_{18}H_{23}NO_2Na$ ([M+Na]$^+$, 100%) found 308.1616, requires 308.1621 (−1.6 ppm).

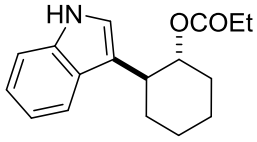
Kinetic resolution of *trans*-2-(1*H*-indol-3-yl)cyclohexan-1-ol 39





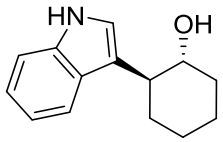
Following General Procedure D, *trans*-2-(1*H*-indol-3-yl)cyclohexan-1-ol **39** (65 mg, 0.3 mmol), PS-HyperBTM **6** (3.4 mg, 1 mol%), *i*-Pr_2NEt (31 µL, 0.18 mmol, 0.6 equiv.) and $(EtCO)_2O$ (27.4 µL, 0.165 mmol, 0.55 equiv.) in $CHCl_3$ (1.5 mL) for 7 h at r.t. gave the crude product, which was purified by Biotage® Isolera™ 4 to give (1*R*,2*S*)-2-(1*H*-indol-3-yl)cyclohexan-1-ol **39** (40 mg, 62%) and (1*S*,2*R*)-2-(1*H*-indol-3-yl)cyclohexyl isobutyrate **S24** (19 mg, 22%). **(1*R*,2*S*)-2-(1*H*-Indol-3-yl)cyclohexan-1-ol 39:** mp 153-155°C {Lit.[51] 154-156 °C}. $[\alpha]_D^{20}$ −1.4 (*c* 1.0, $CHCl_3$); Chiral HPLC analysis, Chiralcel OD-H (90:10 hexane:IPA, flow rate 1.0 mL min^{-1}, 270 nm, 30 °C) t_R (1*S*,2*R*): 16.9 min, t_R (1*R*,2*S*): 21.3 min, 45.244:54.756 er. **(1*S*,2*R*)-2-(1*H*-Indol-3-yl)cyclohexyl isobutyrate S24** $[\alpha]_D^{20}$ +3.1 (*c* 1.0, $CHCl_3$); Chiral HPLC analysis, Chiralpak AD-H (96:4 hexane:IPA, flow rate 1.0 mL min^{-1}, 220 nm, 30 °C) t_R (1*R*,2*S*): 12.2 min, t_R (1*S*,2*R*): 14.1 min 37.710:62.290 er. $s = 2$

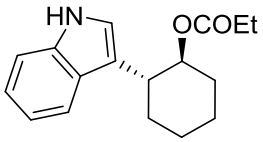
***trans*-2-(1*H*-Indol-3-yl)cyclohexyl propionate 40**



Following General Procedure C, DMAP (3.7 mg, 10 mol%), *trans*-2-(1*H*-indol-3-yl)cyclohexan-1-ol **39** (65 mg, 0.30 mmol), *i*-Pr_2NEt (57 µL, 0.33 mmol, 1.1 equiv.) and $(EtCO)_2O$ (38 µL, 0.30 mmol, 1.0 equiv.) in CH_2Cl_2 (0.2 M) gave *trans*-2-(1*H*-indol-3-yl)cyclohexyl propionate **40** (74 mg, 91%) as a colourless oil. ^1H NMR (400 MHz, $CDCl_3$) δ_H: 0.87 (3H, t, *J* 7.6, C*H*$_3$), 1.42-1.80 (4H, m, C*H*$_2$ & C*H*$_2$), 1.83-2.29 (6H, m, C*H*$_2$, C*H*$_2$, C*H*$_2$CH$_3$), 3.14 (1H, ddd, J 12.0, 10.4, 3.7, C(2)*H*), 5.05-5.18 (1H, m, C(1)*H*), 6.97 (1H, d, *J* 2.4, Ar*H*), 7.14-7.35 (3H, m, 3 × Ar*H*), 7.79 (1H, dd, *J* 7.7, 1.5, Ar*H*), 8.30 (1H, s, N*H*). Spectral data in accordance with Literature.[51]

Kinetic resolution of *trans*-2-(1*H*-indol-3-yl)cyclohexan-1-ol 39



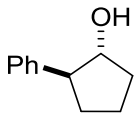


Following General Procedure D, *trans*-2-(1*H*-indol-3-yl)cyclohexan-1-ol **39** (65 mg, 0.3 mmol), PS-HyperBTM **6** (3.4 mg, 1 mol%), *i*-Pr_2NEt (31 µL, 0.18 mmol, 0.6 equiv.) and $(EtCO)_2O$ (27.4 µL, 0.165 mmol, 0.55 equiv.) in $CHCl_3$ (1.5 mL) for 16 h at r.t. gave the crude product, which was purified by Biotage® Isolera™ 4 to give (1*R*,2*S*)-2-(1*H*-indol-3-yl)cyclohexan-1-ol **39** (31 mg, 48%) and (1*S*,2*R*)-2-(1*H*-indol-3-yl)cyclohexyl propionate **40** (32 mg, 39%). **(1*R*,2*S*)-2-(1*H*-Indol-3-yl)cyclohexan-1-ol 39:** mp 156-158 °C {Lit.[51] 154-156 °C}. $[\alpha]_D^{20}$ −23.2 (*c* 1.0, $CHCl_3$); Chiral HPLC analysis, Chiralcel OD-H (90:10 hexane:IPA, flow rate 1.0 mL min^{-1}, 270 nm, 30 °C) t_R (1*S*,2*R*): 16.9 min, t_R (1*R*,2*S*): 21.3 min, 11.491:88.509 er. **(1*S*,2*R*)-2-(1*H*-Indol-3-yl)cyclohexyl propionate 40**: $[\alpha]_D^{20}$ +18.8 (*c* 1.0, $CHCl_3$); Chiral HPLC analysis, Chiralpak AD-H (97:3 hexane:IPA, flow rate 1.0 mL min^{-1}, 220 nm, 30 °C) t_R (1*R*,2*S*): 12.2 min, t_R (1*S*,2*R*): 26.3 min 4.345:95.655 er. *s* = 51

Chiral HPLC data for Table 5, recycle no. 6

(1*R*,2*S*)-2-(1*H*-Indol-3-yl)cyclohexan-1-ol 39: Chiral HPLC analysis, Chiralcel OD-H (90:10 hexane:IPA, flow rate 1.0 mLmin^{-1}, 270 nm, 30 °C) t_R (1*S*,2*R*): 16.9 min, t_R (1*R*,2*S*): 21.3 min, 7.011:92.989 er. **(1*S*,2*R*)-2-(1*H*-Indol-3-yl)cyclohexyl propionate 40**: Chiral HPLC analysis, Chiralpak AD-H (97:3 hexane:IPA, flow rate 1.0 mLmin^{-1}, 220 nm, 30 °C) t_R (1*R*,2*S*): 12.2 min, t_R (1*S*,2*R*): 26.3 min 7.184:92.816 er. *s* = 36

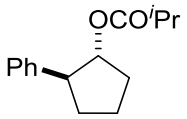
***trans*-2-Phenylcyclopentan-1-ol 41**



Following a literature procedure,[53] a solution of cyclopentene oxide (0.75 g, 8.92 mmol) in THF (5.0 mL) was added dropwise (over 1 h) to a solution of phenylmagnesium bromide (3 M, 5.95 mL, 17.84 mmol) and copper iodide (113 mg, 0.59 mmol) in THF (10 ml) at 0 °C under N_2-atm.

After 2 h at r.t., a 25% solution of ammonium chloride (15 mL) was added, and the mixture extracted with Et_2O (10 mL × 2). The combined organic fractions were washed with 25% ammonium chloride solution, dried ($MgSO_4$), filtered, and evaporated *in vacuo*. The crude product was purified by Biotage® Isolera™ 4 [SNAP UltRa 25 g, 75 mL min^{-1}, hexane:EtOAc (100:0 2 CV, 100:0 to 70:40 40 CV, (16 ml each))] to give *trans*-2-phenylcyclopentan-1-ol **41** (0.92g, 64%) as a colourless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ_H: 1.62-1.96 (5H, m, 2 × C*H*$_2$, O*H)*, 2.04-2.23 (2H, m, C*H*$_2$), 2.81-2.93 (1H, m, C(2)*H*), 4.11-4.21 (1H, m, C(1)*H*), 7.19-7.28 (3H, m, ArC(2,4,6)*H*), 7.29-7.36 (2H, m, ArC(3,5)*H*). Spectral data in accordance with literature.[53]

trans-2-Phenylcyclopentyl isobutyrate 42



Following General Procedure C, DMAP (3.7 mg, 10 mol%), *trans*-2-phenylcyclopentan-1-ol **41** (49 mg, 0.3 mmol, 1.0 equiv.), *i*-Pr_2NEt (57 µL, 0.33 mmol, 1.1 equiv.) and (*i*-PrCO)$_2$O (50 µL, 0.3 mmol, 1.0 equiv.) in CH_2Cl_2 (0.2 M) gave *trans*-2-phenylcyclopentyl isobutyrate **42** (64 mg, 92%) as a colourless oil. IR ν_{max} (thin film, cm^{-1}): 2970 (C-H), 1728 (C=O); ^{1}H NMR (400 MHz, $CDCl_3$) δ_H: 1.11 (3H, d, *J* 7.0, CH(C*H*$_3$)^A(CH$_3$)^B), 1.14 (3H, d, *J* 7.0, CH(CH$_3$)^A(C*H*$_3$)^B), 1.63-1.92 (4H, m, 2 × C*H*$_2$), 2.12-2.25 (2H, m, C*H*$_2$), 2.44-2.56 (1H, m, C*H*(CH$_3$)^A(CH$_3$)^B), 3.12-3.22 (1H, m, C(2)*H*), 5.08-5.14 (1H, m, C(1)*H*), 7.17-7.33 (5H, m, 5 × Ar*H*); ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ_C: 18.99 (CH(*C*H$_3$)^A(CH$_3$)^B), 19.05 (CH(CH$_3$)^A(*C*H$_3$)^B), 23.0 (*C*(4)H$_2$), 31.8 (*C*(3)H$_2$), 32.0 (*C*(5)H$_2$), 34.1 (*C*H(CH$_3$)$_2$), 51.1 (*C*(2)H), 81.7 (*C*(1)H), 126.4 (Ar*C*H), 127.4 (2 × Ar*C*H), 128.5 (2 × Ar*C*H), 142.9 (Ar*C*), 177.0 (C=O); HRMS (NSI) $C_{15}H_{21}O_2$ ([M+H]$^+$, 100%) found 233.1537, requires 233.1536 (+0.4 ppm).

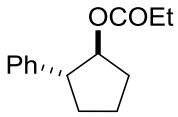
Kinetic resolution of *trans*-2-phenylcyclopentan-1-ol 41

Following General Procedure D, *trans*-2-phenylcyclopentan-1-ol **41** (49 mg, 0.3 mmol), PS-HyperBTM **6** (3.4 mg, 1 mol%), *i*-Pr_2NEt (31 µL, 0.18 mmol, 0.6 equiv.) and $(i\text{-PrCO})_2O$ (27.4 µL, 0.165 mmol, 0.55 equiv.) in $CHCl_3$ (1.5 mL) for 7 h at r.t. gave the crude product, which was purified by Biotage® Isolera™ 4 to give (1*R*,2*S*)-2-phenylcyclopentan-1-ol **41** (22 mg, 45%) and (1*S*,2*R*)-2-phenylcyclopentyl isobutyrate **42** (30 mg, 43%). **(1*R*,2*S*)-2-Phenylcyclopentan-1-ol 41**: $[\alpha]_D^{20}$ −60.7 (*c* 1.0, $CHCl_3$) Chiral HPLC analysis, Chiralcel OD-H (98:2 hexane:IPA, flow rate 1.0 mL min^{-1}, 211 nm, 30 °C) t_R (1*S*,2*R*): 14.4 min, t_R (1*R*,2*S*): 16.0 min, 2.445:97.555 er. **(1*S*,2*R*)-2-Phenylcyclopentyl isobutyrate 42**: $[\alpha]_D^{20}$ +67.2 (*c* 1.0, $CHCl_3$); Following General Procedure E, (1*S*,2*R*)-2-phenylcyclopentyl isobutyrate **42** hydrolysed to corresponding alcohol (1*S*,2*R*)-2-phenylcyclopentan-1-ol **41** for HPLC analysis. Chiral HPLC analysis Chiralcel OD-H (98:2 hexane:IPA, flow rate 1.0 mL min^{-1}, 211 nm, 30 °C) t_R (1*S*,2*R*): 14.4 min, t_R (1*R*,2*S*): 16.0 min, 95.093:4.907 er. *s* = 72

Chiral HPLC data for Table 5, recycle no. 8

(1*R*,2*S*)-2-Phenylcyclopentan-1-ol 41: Chiral HPLC analysis, Chiralcel OD-H (98:2 hexane:IPA, flow rate 1.0 mLmin^{-1}, 211 nm, 30 °C) t_R (1*S*,2*R*): 14.4 min, t_R (1*R*,2*S*): 16.0 min, 12.418:87.522 er. **(1*S*,2*R*)-2-Phenylcyclopentyl isobutyrate 42**: Following General Procedure E, (1*S*,2*R*)-2-phenylcyclopentyl isobutyrate **42** hydrolysed to corresponding alcohol, (1*S*,2*R*)-2-phenylcyclopentan-1-ol **41**, for HPLC analysis. Chiral HPLC analysis Chiralcel OD-H (98:2 hexane:IPA, flow rate 1.0 mL min^{-1}, 211 nm, 30 °C) t_R (1*S*,2*R*): 14.4 min, t_R (1*R*,2*S*): 16.0 min, 96.412:3.588 er. *s* = 61

***trans*-2-Phenylcyclopentyl propionate S25**



Following General Procedure C, DMAP (3.7 mg, 10 mol%), *trans*-2-phenylcyclopentan-1-ol **41** (49 mg, 0.3 mmol, 1.0 equiv.), *i*-Pr_2NEt (57 µL, 0.33 mmol, 1.1 equiv.) and $(EtCO)_2O$ (38 µL, 0.30 mmol, 1.0 equiv.) in CH_2Cl_2 (0.2 M) gave *trans*-2-phenylcyclopentyl propionate **S25** (57 mg, 86%) as a colourless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ_H: 1.11 (3H, t, *J* 7.6, CH_2CH_3), 1.66-1.92 (4H, m, C(3)H_2 & C(4)H_2), 2.14-2.25 (2H, m, C(5)H_2), 2.29 (2H, q, *J* 7.6, CH_2CH_3), 3.13-3.25 (1H, m, C(2)*H*), 5.10-5.18 (1H, m, C(1)*H*), 7.18-7.35 (5H, m, 5 × Ar*H*). Spectral data in accordance with literature.[51]

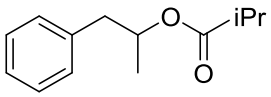
Kinetic resolution of *trans*-2-phenylcyclopentan-1-ol 41

Following General Procedure D, *trans*-2-phenylcyclopentan-1-ol **41** (49 mg, 0.3 mmol), PS-HyperBTM **6** (3.4 mg, 1 mol%), *i*-Pr_2NEt (31 µL, 0.18 mmol, 0.6 equiv.) and $(EtCO)_2O$ (21 µL, 0.165 mmol, 0.55 equiv.) in $CHCl_3$ (1.5 mL) for 7 h at r.t. gave the crude product, which was purified by Biotage® Isolera™ 4 to give (1*R*,2*S*)-2-phenylcyclopentan-1-ol **41** (21 mg, 43%) and (1*S*,2*R*)-2-phenylcyclopentyl propionate **S25** (28 mg, 42%). **(1*R*,2*S*)-2-Phenylcyclopentan-1-ol 41**: $[\alpha]_D^{20}$ −51.6 (*c* 1.0, $CHCl_3$) Chiral HPLC analysis, Chiralcel OD-H (98:2 hexane:IPA, flow rate 1.0 mL min^{-1}, 211 nm, 30 °C) t_R (1*S*,2*R*): 14.6 min, t_R (1*R*,2*S*): 16.3 min, 5.905:94.095 er. **(1*S*,2*R*)-2-Phenylcyclopentyl propionate S25**: $[\alpha]_D^{20}$ +59.1 (*c* 1.0, $CHCl_3$); Following General Procedure E, (1*S*,2*R*)-2-phenylcyclopentyl propionate **S25** hydrolysed to corresponding alcohol (1*S*,2*R*)-2-phenylcyclopentan-1-ol **41** for HPLC analysis. Chiral HPLC analysis, Chiralcel OD-H (98:2 hexane:IPA, flow rate 1.0 mL min^{-1}, 211 nm, 30 °C) t_R (1*S*,2*R*): 14.6 min, t_R (1*R*,2*S*): 16.3 min, 92.458:7.542 er. *s* = 36

1-Phenylpropan-2-ol 43

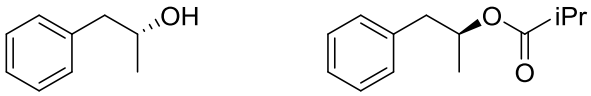
Commercially available

1-Phenylpropan-2-yl isobutyrate S26



Following General Procedure C, DMAP (3.7 mg, 10 mol%), 1-phenylpropan-2-ol **43** (41 mg, 0.3 mmol), *i*-Pr_2NEt (57 µL, 0.33 mmol, 1.1 equiv.) and $(i\text{-PrCO})_2O$ (50 µL, 0.3 mmol, 1.0 equiv.) in CH_2Cl_2 (0.2 M) gave 1-phenylpropan-2-yl isobutyrate **S26** (50 mg, 81%) as a colourless oil. IR ν_{max} (thin film, cm^{-1}): 2974 (C-H), 1728 (C=O); ^1H NMR (400 MHz, $CDCl_3$) δ_H: 1.07 (3H, d, *J* 7.0, $CH(CH_3)^A(CH_3)^B$, 1.11 (3H, d, *J* 7.0, $CH(CH_3)^A(CH_3)^B$), 1.22 (3H, d, *J* 6.2, CH_3), 2.39-2.53 (1H, m, $CH(CH_3)_2$), 2.77 (1H, dd, *J* 13.7, 6.2, CH^AH^B), 2.91 (1H, dd, *J* 13.7, 7.1, CH^AH^B), 5.06-5.18 (1H, m, C*H*-OCOiPr), 7.17-7.24 (3H, m, ArC(2,4,6)*H*), 7.24-7.32 (2H, m, ArC(3,5)*H*); $^{13}C\{^1H\}$ NMR (101 MHz, $CDCl_3$) δ_C: 18.97 $(CH(CH_3)^A(CH_3)^B)$, 19.04 $(CH(CH_3)^A(CH_3)^B)$, 19.6 (CH_3), 34.3 ($CH(CH_3)_2$), 42.4 ($ArCH_2$), 71.1 (*C*H-OCOiPr), 126.5 (Ar*C*(4)H), 128.4 (Ar*C*(2,6)H), 129.6 (Ar*C*(3,5)H), 137.8 (Ar*C*(1)), 176.7 (C=O); HRMS (NSI) $C_{13}H_{19}O_2$ ($[M+H]^+$, 100%) found 207.1381, requires 207.1380 (+0.7 ppm).

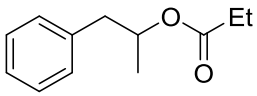
Kinetic resolution of 1-phenylpropan-2-ol 43



Following General Procedure D, 1-phenylpropan-2-ol **43** (41 mg, 0.3 mmol), PS-HyperBTM **6** (3.4 mg, 1 mol%), *i*-Pr_2NEt (31 µL, 0.18 mmol, 0.6 equiv.) and $(i\text{-PrCO})_2O$ (27.4 µL, 0.165 mmol, 0.55 equiv.) in $CHCl_3$ (1.5 mL) for 7 h at 0 °C gave the crude product, which was purified by Biotage® Isolera™ 4 to give (*R*)-1-phenylpropan-2-ol **43** (14 mg, 35%) and (*S*)-1-phenylpropan-2-yl isobutyrate **S26** (23 mg, 37%). **(*R*)-1-Phenylpropan-2-ol 43:** $[\alpha]_D^{20}$ −10 (*c* 1.0, $CHCl_3$) {Lit.[54] $[\alpha]_D^{20}$ −20.7 (50.7% ee, *c* 1.08, $CHCl_3$). Chiral HPLC analysis Chiralcel OD-H (99.5:0.5 hexane:IPA, flow rate 1.0 mL min^{-1}, 220 nm, 30 °C) t_R (*S*): 20.3 min, t_R (*R*): 23.1 min, 36.823:63.177 er. **(*S*)-1-Phenylpropan-2-yl isobutyrate S26:** $[\alpha]_D^{20}$ +8 (*c* 1.0, $CHCl_3$); Following

general procedure E, ester hydrolysed to corresponding alcohol for HPLC analysis. Chiral HPLC analysis Chiralcel OD-H (99.5:0.5 hexane:IPA, flow rate 1.0 mL min^{-1}, 220 nm, 30 °C) t_R (*S*): 20.4 min, t_R (*R*): 23.5 min, 65.391:34.609 er. *s* = 2

1-Phenylpropan-2-yl propionate 44



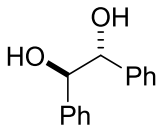
Following General Procedure C, DMAP (3.7 mg, 10 mol%), 1-phenylpropan-2-ol **43** (41 mg, 0.3 mmol), *i*-Pr_2NEt (57 µL, 0.33 mmol, 1.1 equiv.) and $(EtCO)_2O$ (38 µL, 0.3 mmol, 1.0 equiv.) in CH_2Cl_2 (0.2 M) gave 1-phenylpropan-2-yl propionate **44** (48mg, 77%) as a colourless oil. ^1H NMR (400 MHz, $CDCl_3$) δ_H: 1.08 (3H, t, *J* 7.6, CH_2CH_3), 1.22 (3H, d, *J* 6.3, $CHCH_3$), 2.27 (2H, m, CH_2CH_3), 2.76 (1H, dd, *J* 13.6, 6.4, Ph-CH^AH^B), 2.92 (1H, dd, *J* 13.6, 6.8, Ph-CH^AH^B), 5.08-5.18 (1H, m, C*H*-OCOiPr), 7.17-7.24 (3H, m, ArC(2,4,6)*H*), 7.25-7.32 (2H, m, Ar(3,5)*H*). Spectral data in accordance with literature.[55]

Kinetic resolution of 1-phenylpropan-2-ol 43

Following General Procedure D, 1-phenylpropan-2-ol **43** (82 mg, 0.6 mmol), PS-HyperBTM **6** (6.8 mg, 1 mol%), *i*-Pr_2NEt (62 µL, 0.36 mmol, 0.6 equiv.) and $(EtCO)_2O$ (42 µL, 0.33 mmol, 0.55 equiv.) in $CHCl_3$ (3.0 mL) for 7 h at r.t. gave the crude product, which was purified by Biotage® Isolera™ 4 to give (*R*)-1-phenylpropan-2-ol **43** (37 mg, 45%) and (*S*)-1-phenylpropan-2-yl propionate **44** (44 mg, 38%). **(*R*)-1-Phenylpropan-2-ol 43**: $[\alpha]_D^{20}$ −19 (*c* 1.0, $CHCl_3$) {Lit.[54] $[\alpha]_D^{20}$ −20.7 (50.7% ee, *c* 1.08, $CHCl_3$). Chiral HPLC analysis Chiralcel OD-H (99.5:0.5 hexane:IPA, flow rate 1.0 mL min^{-1}, 220 nm, 30 °C) t_R (*S*): 20.5 min, t_R (*R*): 23.3 min, 22.909:77.091 er. **(*S*)-1-Phenylpropan-2-yl propionate 44**: (44 mg, 38%)**:** $[\alpha]_D^{20}$ +14 (*c* 1.0, $CHCl_3$); following General Procedure E, (*S*)-1-phenylpropan-2-yl propionate **44** hydrolysed to corresponding alcohol (*S*)-1-phenylpropan-2-ol **43** for HPLC analysis. Chiral HPLC analysis

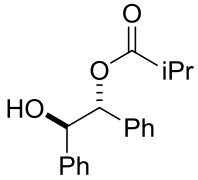
Chiral HPLC analysis Chiralcel OD-H (99.5:0.5 hexane:IPA, flow rate 1.0 mL min^{-1}, 220 nm, 30 °C) t_R (*S*): 20.3 min, t_R (*R*): 23.2 min, 80.421:19.579 er. *s* =7

(±)-1,2-Diphenylethane-1,2-diol 45



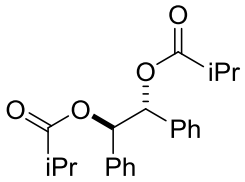
According to the procedure,[56] OsO_4 (3.75 mg, 0.5 mol%) and NMO·H_2O (567 mg, 3.9 mmol, 1.3 equiv.) were added to a solution of *trans*-stilbene (540 mg, 3 mmol, 1.0 equiv.) in poly(ethylene glycol) (400 MW, 3 g) under N_2 atmosphere and stirred at r.t. for 2 h. Et_2O (7.5 mL) was added and stirred for 5 min. Ether layer was decanted and the procedure was repeated (7.5 mL). The combined Et_2O fractions were washed sequentially with 5% $Na_2S_2O_5$ aq. solution (10 mL), water (5 mL) and brine (5 mL), dried ($MgSO_4$), and concentrated *in vacuo* to give the crude product, which was purified by Biotage® Isolera™ 4 to give (±)-1,2-diphenylethane-1,2-diol **45** (270 mg, 42%) as a colourless solid. mp 142-144 °C {Lit.[57] 146-147 °C}. ^{1}H NMR (400 MHz, $CDCl_3$) δ_H: 4.71 (2H, s, 2 × C*H*-OH), 7.09-7.16 (4H, m, 4 × Ar*H*), 7.20-7.25 (6H, m, 6 × Ar*H*). Spectral data in accordance with literature.[58]

(±)-2-Hydroxy-1,2-diphenylethyl isobutyrate 46



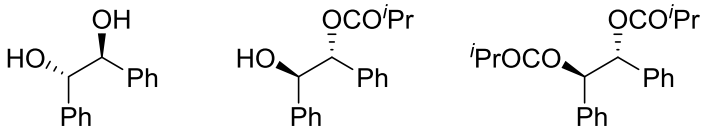
Following General Procedure C, DMAP (3.7 mg, 10 mol%), (±)-1,2-diphenylethane-1,2-diol **45** (64 mg, 0.3 mmol), *i*-Pr_2NEt (57 µL, 0.33 mmol, 1.1 equiv.) and (*i*-$PrCO)_2O$ (50 µL, 0.3 mmol, 1.0 equiv.) in CH_2Cl_2 (0.2 M) gave (±)-2-hydroxy-1,2-diphenylethyl isobutyrate **46** (57 mg, 67%) as a colourless solid. mp 80-82 °C {Lit.[59] 105-106 °C}. ^{1}H NMR (400 MHz, $CDCl_3$) δ_H: 1.16 (3H, d, *J* 7.0, CH(CH_3)^A(CH_3)^B), 1.17 (3H, d, *J* 7.0, CH(CH_3)^A(CH_3)^B), 2.45-2.70 (2H, m, O*H* & C*H*(CH_3)$_2$), 4.94 (1H, d, *J* 7.1, C*H*-OH), 5.86 (1H, d, *J* 7.1, C*H*-OCOiPr), 7.09-7.18 (4H, m, 4 × Ar*H*), 7.19-7.25 (6H, m, 6 × Ar*H*). Spectral data in accordance with literature.[14]

(±)-1,2-Diphenylethane-1,2-diyl bis(2-methylpropanoate) 47



Following General Procedure C, DMAP (3.7 mg, 10 mol%), (±)-1,2-diphenylethane-1,2-diol **45** (64 mg, 0.3 mmol), *i*-Pr_2NEt (155 µL, 0.90 mmol, 3.0 equiv.) and (*i*-$PrCO)_2O$ (150 µL, 0.9 mmol, 3.0 equiv.) in CH_2Cl_2 (0.2 M) gave (±)-1,2-diphenylethane-1,2-diyl bis(2-methylpropanoate) **47** (90 mg, 85%) as a colourless solid. mp 86-88 °C {Lit.[14] 73.1-74.0 °C}. ^{1}H NMR (400 MHz, $CDCl_3$) δ_H: 1.14 (6H, d, *J* 7.0, 2 × C*H*$_3$), 1.15 (6H, d, *J* 7.0, 2 × C*H*$_3$), 2.48-2.68 (2H, m, 2 × C*H*$(CH_3)_2$), 6.05 (2H, s, 2 × C*H*-OCOiPr), 7.10-7.15 (4H, m, 4 × Ar*H*), 7.17-7.23 (6H, m, 6 × Ar*H*). Spectral data in accordance with literature.[14]

Kinetic resolution of (±)-1,2-diphenylethane-1,2-diol 45

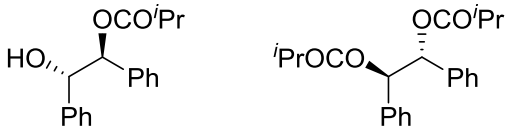


Following General Procedure D, (±)-1,2-diphenylethane-1,2-diol **45** (64 mg, 0.3 mmol), PS-HyperBTM **6** (3.4 mg, 1 mol%), *i*-Pr_2NEt (31 µL, 0.18 mmol, 0.6 equiv.) and (*i*-$PrCO)_2O$ (27.4 µL, 0.165 mmol, 0.55 equiv.) in $CHCl_3$ (1.5 mL) for 7 h at 0 °C gave the crude product (from ^{1}H NMR spectroscopy, crude ratio of 56:38:6 (**45:46:47**) determined) which was purified by Biotage® Isolera™ 4 to give (*S*,*S*)-1,2-diphenylethane-1,2-diol **45** (35 mg, 54%), (*R*,*R*)-2-hydroxy-1,2-diphenylethyl isobutyrate **46** (29 mg, 34%), and (*R*,*R*)-1,2-diphenylethane-1,2-diyl bis(2-methylpropanoate) **47** (7 mg, 7%). **(*S*,*S*)-1,2-Diphenylethane-1,2-diol 45**: mp 140-142 °C {Lit.[57] 146-147 °C}. $[\alpha]_D^{20}$ −70.6 (*c* 1.0, $CHCl_3$) Chiral HPLC analysis, Chiralcel OJ-H (93:7 hexane:IPA, flow rate 1.0 mL min^{-1}, 220 nm, 30 °C) t_R (*S*,*S*): 20.0 min, t_R (*R*,*R*): 23.3 min, 83.768:16.232 er. **(*R*,*R*)-2-Hydroxy-1,2-diphenylethyl isobutyrate 46**: mp 79-81 °C {Lit.[59] 105-106 °C}. $[\alpha]_D^{20}$ +1.6 (*c* 1.0, $CHCl_3$) Chiral HPLC analysis, Chiralcel OD-H (95:5 hexane:IPA, flow rate 1.0 mL min^{-1}, 220 nm, 30 °C) t_R (*S*,*S*): 12.4 min, t_R (*R*,*R*): 19.3 min, 10.671:89.329 er. **(*R*,*R*)-1,2-Diphenylethane-1,2-diyl bis(2-methylpropanoate) 47:** mp 87-89 °C {Lit.[14] 73.1-

74.0 °C}. $[\alpha]_D^{20}$ −30 (*c* 0.5, $CHCl_3$); Following General Procedure E, (*R*,*R*)-1,2-diphenylethane-1,2-diyl bis(2-methylpropanoate) **47** hydrolysed to corresponding alcohol, (*R*,*R*)-1,2-diphenylethane-1,2-diol **45**, for HPLC analysis. Chiral HPLC analysis, Chiralcel OJ-H (93:7 hexane:IPA, flow rate 1.0 mL min^{-1}, 220 nm, 30 °C) t_R (*S*,*S*): 20.7 min, t_R (*R*,*R*): 23.6 min, 0.708:99.292 er.

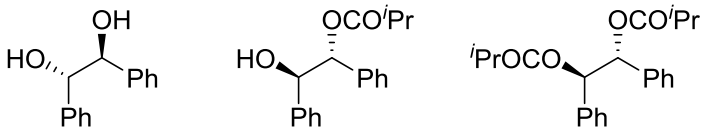
s (for 1st KR) = 27. Calculated using er of diol, (*S*,*S*)-**45** (83.768:16.232 *S*,*S*:*R*,*R*) and reaction conversion (44%), which was determined by ^{1}H NMR spectroscopic analysis of the crude recation product.

Kinetic resolution of (±)-2-hydroxy-1,2-diphenylethyl isobutyrate 46



Following General Procedure D, (±)-2-hydroxy-1,2-diphenylethyl isobutyrate **46** (85 mg, 0.3 mmol), PS-HyperBTM **6** (3.4 mg, 1 mol%), *i*-Pr_2NEt (31 µL, 0.18 mmol, 0.6 equiv.) and (*i*-$PrCO)_2O$ (27.4 µL, 0.165 mmol, 0.55 equiv.) in $CHCl_3$ (1.5 mL) for 7 h at 0 °C gave the crude product, which was purified by Biotage® Isolera™ 4 to give (*S*,*S*)-2-hydroxy-1,2-diphenylethyl isobutyrate **46** (45 mg, 53%), and (*R*,*R*)-1,2-diphenylethane-1,2-diyl bis(2-methylpropanoate) **47** (40 mg, 38%). **(*S*,*S*)-2-Hydroxy-1,2-diphenylethyl isobutyrate 46**: $[\alpha]_D^{20}$ −2.0 (*c* 1.0, $CHCl_3$); Chiral HPLC analysis, Chiralcel OD-H (95:5 hexane:IPA, flow rate 1.0 mL min^{-1}, 220 nm, 30 °C) t_R (*S*,*S*): 12.6 min, t_R (*R*,*R*): 19.6 min, 91.580:8.420 er. **(*R*,*R*)-1,2-Diphenylethane-1,2-diyl bis(2-methylpropanoate) 47:** $[\alpha]_D^{20}$ −26.0 (*c* 1.0, $CHCl_3$); following General Procedure E, (*R*,*R*)-1,2-diphenylethane-1,2-diyl bis(2-methylpropanoate) **47** hydrolysed to corresponding alcohol (*R*,*R*)-1,2-diphenylethane-1,2-diol **45** for HPLC analysis. Chiral HPLC analysis, Chiralcel OJ-H (93:7 hexane:IPA, flow rate 1.0 mL min^{-1}, 220 nm, 30 °C) t_R (*S*,*S*): 20.5 min, t_R (*R*,*R*): 23.4 min, 2.364:97.636 er. *s* = 108

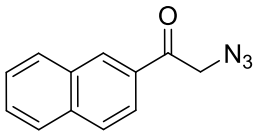
Kinetic resolution of (±)-1,2-diphenylethane-1,2-diol 45 [using 1.5 equiv. (*i*-PrCO)$_2$O)]



Following General Procedure D, (±)-1,2-diphenylethane-1,2-diol **45** (64 mg, 0.3 mmol), PS-HyperBTM **6** (3.4 mg, 1 mol%), *i*-Pr$_2$NEt (84 µL, 0.48 mmol, 1.6 equiv.) and (*i*-PrCO)$_2$O (75 µL, 0.45 mmol, 1.5 equiv.) in $CHCl_3$ (1.5 mL) for 7 h at 0 °C gave the crude product (from ^{1}H NMR spectroscopy, crude ratio of 27:28:45 (**45:46:47**) determined) which was purified by Biotage® Isolera™ 4 to give (*S*,*S*)-1,2-diphenylethane-1,2-diol **45** (13 mg, 20%), (*S*,*S*)-2-hydroxy-1,2-diphenylethyl isobutyrate **46** (23 mg, 27%), and (*R*,*R*)-1,2-diphenylethane-1,2-diyl bis(2-methylpropanoate) **47** (46 mg, 43%). **(*S*,*S*)-1,2-Diphenylethane-1,2-diol 45**: Chiral HPLC analysis, Chiralcel OJ-H (93:7 hexane:IPA, flow rate 1.0 mL min^{-1}, 220 nm, 30 °C) t_R (*S*,*S*): 19.2 min, t_R (*R*,*R*): 21.7 min, 99.922:0.078 er. **(*S*,*S*)-2-Hydroxy-1,2-diphenylethyl isobutyrate 46**: Chiral HPLC analysis, Chiralcel OD-H (95:5 hexane:IPA, flow rate 1.0 mL min^{-1}, 220 nm, 30 °C) t_R (*S*,*S*): 12.4 min, t_R (*R*,*R*): 18.7 min, 81.656:18.344 er. **(*R*,*R*)-1,2-Diphenylethane-1,2-diyl bis(2-methylpropanoate) 47:** Following General Procedure E, (*R*,*R*)-1,2-diphenylethane-1,2-diyl bis(2-methylpropanoate) **47** hydrolysed to corresponding alcohol, (*R*,*R*)-1,2-diphenylethane-1,2-diol **45**, for HPLC analysis. Chiral HPLC analysis, Chiralcel OJ-H (93:7 hexane:IPA, flow rate 1.0 mL min^{-1}, 220 nm, 30 °C) t_R (*S*,*S*): 18.2 min, t_R (*R*,*R*): 20.0 min, 0.012:99.988 er.

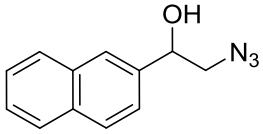
Synthesis of (*S*)-Pronethalol

2-Azido-1-(naphthalen-2-yl)ethan-1-one S27



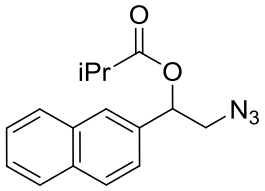
Sodium azide (227 mg, 6.02 mmol, 1.0 equiv.) was added in one portion to a solution of 2-(bromoacetyl)naphthalene (1.5 g, 6.02 mmol, 1.0 equiv.) in DMSO (15 mL) at 5-10 °C under argon, and the reaction was stirred, warming to r.t. over 1.5 h. H_2O (20 mL) was added and the mixture extracted with EtOAc (3 × 30 mL). The combined organic fractions were washed with H_2O, dried ($MgSO_4$), filtered, and concentrated *in vacuo* to give 2-azido-1-(naphthalen-2-yl)ethan-1-one **S27** as a pale yellow solid (0.71 g, 56%). mp 66-68 °C {Lit.[60] 66-67 °C}. ^{1}H NMR (400 MHz, $CDCl_3$) δ_H: 4.71 (2H, s, C*H*$_2$), 7.55-7.61 (1H, m, Ar*H*), 7.63-7.67 (1H, m, Ar*H*), 7.87-8.01 (4H, m, 4 × Ar*H*), 8.41 (1H, m, Ar*H*). Spectral data in accordance with literature.[61]

2-Azido-1-(naphthalen-2-yl)ethan-1-ol 48



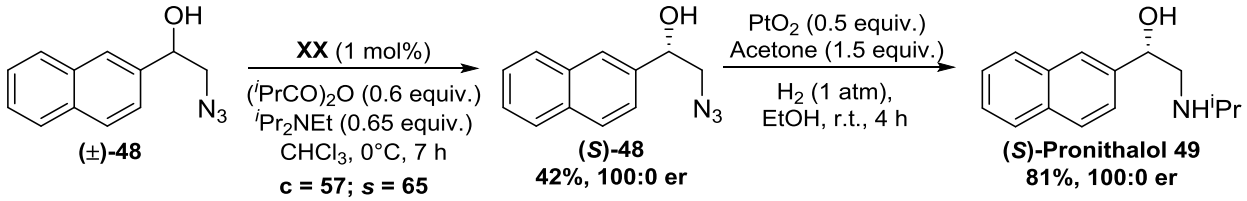
Following General Procedure B, 2-azido-1-(naphthalen-2-yl)ethan-1-one **S27** (0.60 g, 2.84 mmol 1.0 equiv.) and $NaBH_4$ (107 mg, 2.84 mmol, 1.0 equiv.) in methanol (10 mL) gave, after purification by Biotage® Isolera™ 4, 2-azido-1-(naphthalen-2-yl)ethan-1-ol **48** as a colourless solid (0.56 g, 92%). mp 87-89 °C {Lit.[62] 82-83 °C}. ^{1}H NMR (400 MHz, $CDCl_3$) δ_H: 3.50-3.62 (2H, m, C*H*$_2$), 5.06 (1H, dd, *J* 7.9, 4.1, C*H*-OH), 7.43-7.55 (3H, m, 3 × Ar*H*), 7.80-7.90 (4H, m, 4 × Ar*H*). Spectral data in accordance with literature.[63]

2-Azido-1-(naphthalen-2-yl)ethyl isobutyrate S28



Following General Procedure C, DMAP (3.7 mg, 10 mol%), 2-azido-1-(naphthalen-2-yl)ethan-1-ol **48** (64 mg, 0.3 mmol), *i*-Pr_2NEt (57 µL, 0.33 mmol, 1.1 equiv.) and (*i*-$PrCO)_2O$ (50 µL, 0.3 mmol, 1.0 equiv.) in CH_2Cl_2 (0.2 M) gave 2-azido-1-(naphthalen-2-yl)ethyl isobutyrate **S28** (75 mg, 88%) as a colourless solid. mp 36-38 °C; IR ν_{max} (thin film, cm^{-1}): 2974 (C-H), 2098 (N_3), 1736 (C=O); ^1H NMR (400 MHz, $CDCl_3$) δ_H: 1.23 (3H, d, *J* 7.0, $CH(CH_3)^A(CH_3)^B$, 1.26 (3H, d, *J* 7.0, $CH(CH_3)^A(CH_3)^B$), 2.64-2.76 (1H, m, $CH(CH_3)_2$), 3.53 (1H, dd, *J* 13.2, 3.9, CH^AH^B), 3.73 (1H, dd, *J* 13.2, 8.2, CH^AH^B), 6.05-6.11 (1H, m, C*H*-OCOiPr), 7.42-7.55 (3H, m, 3 × Ar*H*), 7.80-7.89 (4H, m, 4 × Ar*H*); $^{13}C\{^1H\}$ NMR (101 MHz, $CDCl_3$) δ_C: 18.9 ($CH(CH_3)^A(CH_3)^B$), 19.0 ($CH(CH_3)^A(CH_3)^B$), 34.2 ($CH(CH_3)_2$), 55.4 (CH_2N_3), 74.7 (*C*H-OC(O)iPr), 123.8 (Ar*C*H), 125.9 (Ar*C*H), 126.60 (Ar*C*H), 126.62 (Ar*C*H), 127.9 (Ar*C*H), 128.2 (Ar*C*H), 128.8 (Ar*C*H), 133.2 (Ar*C*), 133.4 (Ar*C*), 134.9 (Ar*C*), 176.1 (C=O); HRMS (NSI) $C_{16}H_{21}O_2N_4$ ($[M+NH_4]^+$, 100%) found 301.1662, requires 301.1659 (+1.0 ppm).

Kinetic resolution of 2-azido-1-(naphthalen-2-yl)ethan-1-ol 48 followed by synthesis of (*S*)-Pronithalol 49



Following General Procedure D, 2-azido-1-(naphthalen-2-yl)ethan-1-ol **48** (213 mg, 1.0 mmol), PS-HyperBTM **6** (11.3 mg, 1 mol%), *i*-Pr_2NEt (112 µL, 0.65 mmol, 0.65 equiv.) and (*i*-$PrCO)_2O$ (100 µL, 0.6 mmol, 0.6 equiv.) in $CHCl_3$ (5.0 mL) for 7 h at 0 °C gave the crude product, which was purified by Biotage® Isolera™ 4 to give (*S*)-2-azido-1-(naphthalen-2-yl)ethan-1-ol **48** (91 mg, 42%) and (*R*)-2-azido-1-(naphthalen-2-yl)ethyl isobutyrate **S28** (158 mg, 55%). **(*S*)-2-**

Azido-1-(naphthalen-2-yl)ethan-1-ol 48: mp 86-88 °C {Lit.[62] 82-83 °C}. $[\alpha]_D^{20}$ +115 (*c* 1.0, $CHCl_3$); {Lit.[64] $[\alpha]_D^{20}$ +125.2 (99 %ee, *c* 1.3, $CHCl_3$), HPLC analysis, Chiralcel OD-H (95:5 hexane:IPA, flow rate 1.0 mL min^{-1}, 220 nm, 30 °C) t_R (*R*): 30.9 min, t_R (*S*): 35.2 min, 0.014:99.986 er. (***R*)-2-Azido-1-(naphthalen-2-yl)ethyl isobutyrate S28**: mp 37-39 °C; $[\alpha]_D^{20}$ −78 (*c* 1.0, $CHCl_3$); Chiral HPLC analysis, Chiralpak AD-H (99.8:0.2 hexane:IPA, flow rate 1.0 mL min^{-1}, 220 nm, 30 °C) t_R (*S*): 15.4 min, t_R (*R*): 25.9 min, 12.445:87.555 er. *s* = 59.

Following a literature procedure,[62] PtO_2 (33 mg, 0.15 mmol, 0.5 equiv.) was added to a solution of (*S*)-2-azido-1-(naphthalen-2-yl)ethan-1-ol **48** (64 mg, 0.3 mmol, 1.0 equiv.) and acetone (35 µL, 0.45 mmol, 1.5 equiv.) in ethanol (3.5 mL) under an atmosphere of N_2. The reaction was stirred vigorously, and the atmosphere of the flask was purged using a balloon of H_2. A second balloon of H_2 was attached, and the reaction stirred vigorously for 4 h at r.t.. The reaction was filtered through celite and washed with ethanol (2 mL × 2). The filtrate was concentrated *in vacuo* to give (*S*)-pronethalol **49** as a colourless solid (56 mg, 81%). mp: 101-103 °C {Lit.[65] 108-109 °C}. $[\alpha]_D^{20}$ +20.2 (*c* 1.0, $CHCl_3$); ^{1}H NMR (400 MHz, $CDCl_3$) δ_H: 1.08 (3H, d, *J* 6.3, $CH(CH_3)^A(CH3)^B$), 1.09 (3H, d, *J* 6.3, $CH(CH_3)^A(CH3)^B$), 2.75 (1H, dd, *J* 12.1, 8.7, CH^AH^B), 2.80-2.91 (1H, m, $CH(CH_3)_2$), 3.03 (1H, dd, *J* 12.1, 3.8, CH^AH^B), 4.83 (1H, dd, *J* 8.7, 3.8, Ph-C*H*), 7.43-7.51 (3H, m, 3 × Ar*H*), 7.79-7.89 (4H, m, 4 × Ar*H*). Chiral HPLC analysis, Chiralpak IC (94.9:5.0:0.1 hexane:IPA:diethylamine, flow rate 0.7 mL min^{-1}, 254 nm, 30 °C) t_R (*R*): 15.9 min, t_R (*S*): 24.5 min, 0.310:99.690 er.

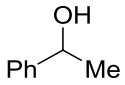
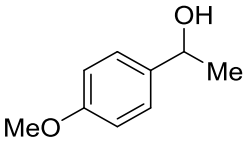
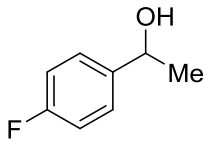
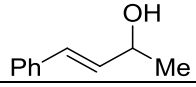
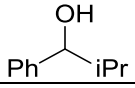
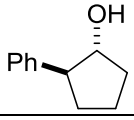
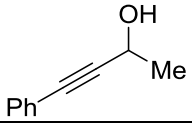
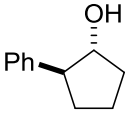
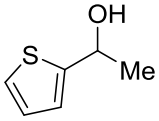
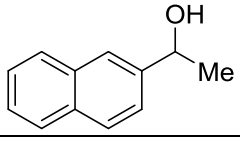
Chiral HPLC data for Table 5, recycle no. 9

(*S*)-2-Azido-1-(naphthalen-2-yl)ethan-1-ol 48: Chiral HPLC analysis, Chiralcel OD-H (95:5 hexane:IPA, flow rate 1.0 mL min^{-1}, 220 nm, 30 °C) t_R (*S*): 30.9 min, t_R (*R*): 35.2 min, 4.835:95.165 er. (***R*)-2-Azido-1-(naphthalen-2-yl)ethyl isobutyrate S28**: Chiral HPLC analysis, Chiralpak AD-H (99.8:0.2 hexane:IPA, flow rate 1.0 mL min^{-1}, 220 nm, 30 °C) t_R (*R*): 15.4 min, t_R (*S*): 25.9 min, 4.743:95.257 er. *s* = 62

Application in Continuous Flow

Description of Kinetic Resolution in Continuous Flow

A packed bed reactor consisting of a vertically-mounted Omnifit glass chromatography column (10 mm pore size and up to maximal 70 mm of adjustable bed height), with a glass cooling jacket was loaded with PS-HyperBTM **6** resin (600 mg; f = 0.86 mmol g^{-1}). The resin was allowed to swell to its maximum volume by pumping $CHCl_3$ at 1 µL min^{-1} for 30 min at r.t. using a P 4.1S Azura pump developed by Knauer. The column was then cooled by circulting ethylene glycol (−5 °C) using a Huber Ministat over 10 min, during which time $CHCl_3$ was pumped through the packed bed reactor at 100 µL min^{-1}. Two syringes were used to inject reagents using a Legato 200 series syringe pump by KD Scientific. The first syringe was filled with a solution of the appropriate alcohol (4 mmol) in $CHCl_3$ (10 mL) and the second syringe with a mixture of the appropiate anhydride (2.4 mmol) and *i*-Pr_2NEt (449 µL, 2.6 mmol) in $CHCl_3$ (10 mL total volume). Both solutions were injected at 50 µL min^{-1}, mixed in a T-type mixing chamber, and passed through the reactor at a combined flow rate of 100 µL min^{-1} (total flow time, 3 h 20 min). After complete addition of the reagents from the syringes, a P 4.1S Azura pump developed by Knauer was connected, and pumped $CHCl_3$ was pumped at 100 µL min^{-1} for 45 min to ensure elution of the products and achieve regeneration of the column for the next reaction. When different anhydrides were used in consecutive kinetic resolutions, an additional solution of 10% MeOH in $CHCl_3$ was pumped at 200 µL min^{-1} for 30 min to avoid cross contamination.

Alcohol		Alc		Est		C	s
		er	Yield	er	Yield		
	Run 1	97.152:2.848	190 mg 39%	10.824:89.176	362 mg 47 %	54.62	29.0
	Run 2	96.731:3.269	207 mg 42 %	10.319:89.681	376 mg 49%	54.08	29.6
	Run 3	98.126:1.874	184 mg 38 %	12.443:87.557	383 mg 50 %	56.17	27.3
	Run 4	96.523:3.477	200 mg 41%	11.104:88.896	374 mg 47 %	54.46	26.8
	Run 5	97.846:2.153	186 mg 38 %	11.281:88.718	391 mg 51 %	55.27	29.7
		96.950:3.050	234 mg (38)	11.422:88.578	442 mg (50%)	54.88	26.9
		95.316:4.684	198 mg (35)	17.758:82.242	429 mg (51%)	58.43	14.0
		92.492:7.508	248 mg (42%)	14.636:85.364	445 mg (51%)	54.58	15.4
		96.188:3.811	278 mg (46%)	1.875:98.125	382 mg (43%)	48.97	175.2
		98.894:1.106	255 mg (39%)	14.950:85.050	503 mg (54%)	58.25	24.5
		96.297:3.703	151 mg (26%)	23.271:76.729	440 mg (51%)	63.00	10.6
		98.015:1.985	230 mg (35%)	18.901:81.099	477 mg (55%)	60.69	16.0
		96.892:3.108	186 mg (36%)	14.140:85.860	384 mg (48%)	56.67	20.7
		94.097:5.903	315 mg (46%)	9.866:90.134	473 mg (49%)	52.35	26.4

Estimation of Analytical Error in *s*

The analytical error present in the calculation of *s* was estimated. The inherent error of HPLC analysis is very low (±0.004), and therefore the largest error in calculation of %ee is likely introduced by the ability of the user to accurately and reproducibility integrate the HPLC spectra. The corresponding error in *s* was therefore estimated as follows:

i) The error in the %ee was estimated by reintegrating the same HPLC traces (one alcohol and one ester) 11 times each to quantify the reproducibility of integration:

Substrate 15	**%ee$_{alc}$**	**%ee$_{est}$**
	69.146	91.456
	69.016	91.372
	69.094	91.29
	68.698	91.188
	69.098	91.354
	69.066	91.534
	69.036	91.472
	68.982	91.448
	69.024	91.418
	69.004	91.472
	69.23	91.446
Mean	69.036	91.405
Standard deviation	0.133	0.098

2 standard deviations of the mean (95.4% of the data) suggests the %ee values obtained are accurate to ~ ±0.25.

ii) Using this %ee error value of ±0.25, the error in *s* can be estimated by taking the measured data and systematically adjusting the %ee values by ±0.25 and recalculating *s* in each case. The largest deviations from the reported values are obtained when both %ee values are either reduced or increased by 0.25. This gives the lowest and highest possible values of *s* based on the error in %ee. This process was undertaken for every KR presented in this paper and the data provided below:

NB: for each substrate: the 1st row is the measured data; the 2nd row is both %ee + 0.25 (higher bound for *s* value); the 3rd row is both %ee − 0.25 (lower bound for *s* value)

Substrate	$\%ee_{alc}$	$\%ee_{est}$	c	s	Difference
15	69.146	91.456	43.05	46.4	
	69.396	91.706	43.08	48.0	1.6
	68.896	91.206	43.03	44.9	-1.5
16	83.41	92.22	47.49	64.8	
	83.66	92.47	47.50	67.4	2.6
	83.16	91.97	47.48	62.4	-2.4
17	63.786	95.29	40.10	80.1	
	64.036	95.54	40.13	85.0	4.9
	63.536	95.04	40.07	75.8	-4.4
18	62.186	96.654	39.15	111.4	
	62.436	96.904	39.18	121.0	9.5
	61.936	96.404	39.12	103.2	-8.2
19	43.166	46.892	47.93	4.1	
	43.416	47.142	47.94	4.2	0.0
	42.916	46.642	47.92	4.1	0.0
20	85.75	83.96	50.53	31.4	
	86	84.21	50.53	32.1	0.7
	85.5	83.71	50.53	30.7	-0.7
21	57.306	89.172	39.12	31.1	
	57.556	89.422	39.16	32.0	0.9
	57.056	88.922	39.09	30.3	-0.8
22	66.766	83.044	44.57	21.5	
	67.016	83.294	44.59	22.0	0.4
	66.516	82.794	44.55	21.1	-0.4
23	81.562	72.312	53.01	15.4	
	81.812	72.562	53.00	15.7	0.2
	81.312	72.062	53.02	15.2	-0.2
24	56.602	77.838	42.10	14.1	
	56.852	78.088	42.13	14.3	0.2
	56.352	77.588	42.07	13.9	-0.2
25	79.668	88.666	47.33	40.5	
	79.918	88.916	47.34	41.7	1.2
	79.418	88.416	47.32	39.4	-1.1
26	75.534	94.226	44.49	76.7	
	75.784	94.476	44.51	80.6	3.9
	75.284	93.976	44.48	73.1	-3.6
27	82.518	99.17	45.42	622.1	
	82.768	99.42	45.43	896.0	273.9
	82.268	98.92	45.40	475.0	-147.1

28	79.298	82.27	49.08	24.7	
	79.548	82.52	49.08	25.2	0.5
	79.048	82.02	49.08	24.2	-0.5
29	11.41	26.736	29.91	1.9	
	11.66	26.986	30.17	1.9	0.0
	11.16	26.486	29.64	1.9	0.0
30	69.008	78.614	46.75	17.1	
	69.258	78.864	46.76	17.4	0.3
	68.758	78.364	46.74	16.8	-0.3
31	93.336	76.114	55.08	24.9	
	93.586	76.364	55.07	25.4	0.6
	93.086	75.864	55.10	24.3	-0.5
32	89.828	77.086	53.82	23.2	
	90.078	77.336	53.81	23.7	0.5
	89.578	76.836	53.83	22.8	-0.5
33	90.734	78.566	53.59	25.8	
	90.984	78.816	53.58	26.3	0.6
	90.484	78.316	53.60	25.3	-0.5
34	34.668		52.00	2.7	
	34.918		51.50	2.7	0.1
	34.418		52.50	2.6	-0.1
35	98.572	82.76	54.36	51.1	
	98.822	83.01	54.35	53.9	2.8
	98.322	82.51	54.37	48.8	-2.4
37	67.772	68.84	49.61	10.8	
	68.022	69.09	49.61	11.0	0.1
	67.522	68.59	49.61	10.7	-0.1
39	77.018	91.31	45.75	51.3	
	77.268	91.56	45.77	53.1	1.8
	76.768	91.06	45.74	49.6	-1.7
41	95.11	90.186	51.33	72.3	
	95.36	90.436	51.33	75.3	3.0
	94.86	89.936	51.33	69.5	-2.8
43	54.182	60.842	47.10	7.0	
	54.432	61.092	47.12	7.0	0.1
	53.932	60.592	47.09	6.9	-0.1
45	67.536		44.00	26.7	
	67.786		43.50	31.9	5.2
	67.286		44.50	23.0	-3.7
46	83.16	95.272	46.61	108.1	
	83.41	95.522	46.62	114.9	6.8
	82.91	95.022	46.60	102.0	-6.1

48	99.972	75.11	57.10	58.9	
	99.99	75.36	57.02	66.4	7.5
	99.722	74.86	57.12	43.4	-15.5

Table 6					
12	92.43	93.152	49.81	94.2	
	92.68	93.402	49.81	98.9	4.6
	92.18	92.902	49.80	90.0	-4.3
32	86.622	70.704	55.06	16.0	
	86.872	70.954	55.04	16.2	0.3
	86.372	70.454	55.08	15.7	-0.3
35	95.99	91.068	51.32	83.9	
	96.24	91.318	51.31	87.8	3.9
	95.74	90.818	51.32	80.3	-3.6
21	46.754	89.184	34.39	27.7	
	47.004	89.434	34.45	28.5	0.8
	46.504	88.934	34.34	27.0	-0.7
33	82.104	75.56	52.00	18.2	
	82.354	75.81	52.00	18.5	0.3
	81.854	75.31	52.00	18.0	-0.3
39	85.978	85.632	50.10	35.6	
	86.228	85.882	50.10	36.5	0.9
	85.728	85.382	50.10	34.7	-0.9
27	61.414	98.434	38.42	238.1	
	61.664	98.684	38.46	284.6	46.5
	61.164	98.184	38.38	204.4	-33.7
41	75.164	92.824	44.74	60.9	
	75.414	93.074	44.76	63.4	2.5
	74.914	92.574	44.73	58.5	-2.4
48	90.33	90.514	49.95	62.4	
	90.58	90.764	49.95	64.7	2.3
	90.08	90.264	49.95	60.2	-2.2
30	54.974	82.05	40.12	17.5	
	55.224	82.3	40.16	17.9	0.3
	54.724	81.8	40.08	17.2	-0.3

Flow					
15	94.79	76.859	55.22	27.5	
	95.04	77.109	55.21	28.2	0.7
	94.54	76.609	55.24	26.8	-0.7

21	93.9	77.21	54.88	26.9	
	94.15	77.46	54.86	27.5	0.6
	93.65	76.96	54.89	26.3	-0.6
22	90.63	64.484	58.43	14.0	
	90.88	64.734	58.40	14.2	0.2
	90.38	64.234	58.46	13.7	-0.2
30	84.98	70.728	52.00	22.1	
	85.23	70.978	52.00	22.5	0.4
	84.73	70.478	52.00	21.7	-0.4
17	92.38	96.25	48.97	175.2	
	92.63	96.5	48.98	189.7	14.6
	92.13	96	48.97	162.5	-12.7
41	97.79	76.859	55.99	33.4	
	98.04	77.109	55.98	34.7	1.3
	97.54	76.609	56.01	32.2	-1.2
32	92.59	54.38	63.00	10.6	
	92.84	54.63	62.96	10.8	0.2
	92.34	54.13	63.04	10.4	-0.2
41	96.03	62.197	60.69	16.0	
	96.28	62.447	60.66	16.3	0.4
	95.78	61.947	60.73	15.6	-0.4
28	93.78	71.72	56.66	20.7	
	94.03	71.97	56.64	21.1	0.4
	93.53	71.47	56.68	20.3	-0.4
12	88.19	80.268	52.35	26.4	
	88.44	80.518	52.34	27.0	0.6
	87.94	80.018	52.36	25.9	-0.5

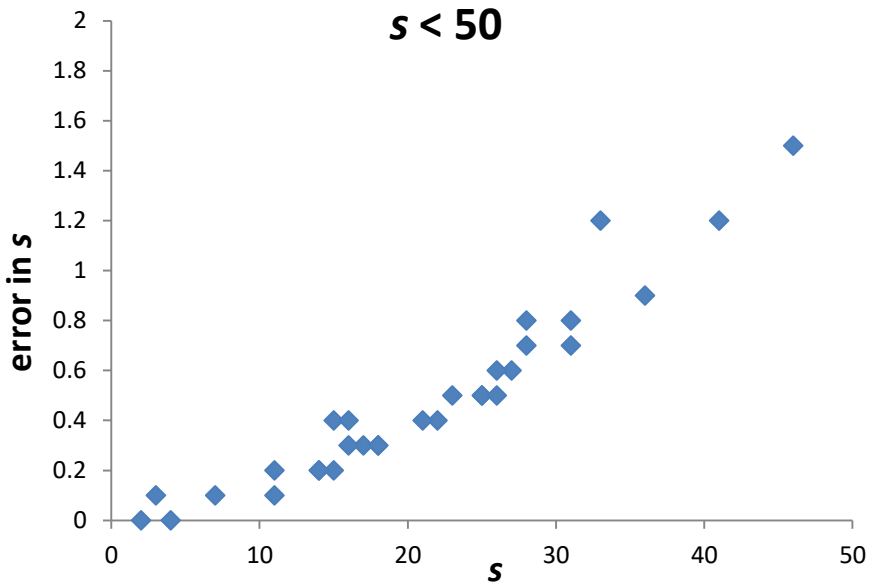
Notes:

- The *s* values for the KR of **34** and **45** were determined by using the %ee of the recovered alcohol and conversion determined by ^{1}H NMR analysis of the crude reaction products (an error of ~ ±0.5% conversion was assumed in these cases)
- The KR of **48** was taken to high conversion to obtain highly enantioenriched recovered alcohol. This inherently results in much greater error in *s* due difficultly in accurately integrating the very small quantiy of the minor enantiomer of alcohol remaining.

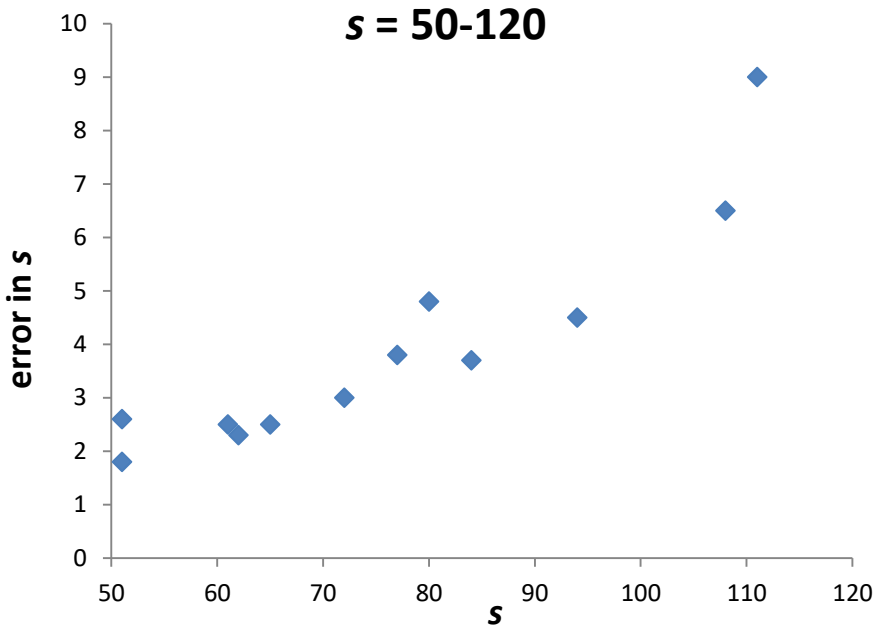
General trends:

The error in *s* increases with increasing magnitude of *s*.

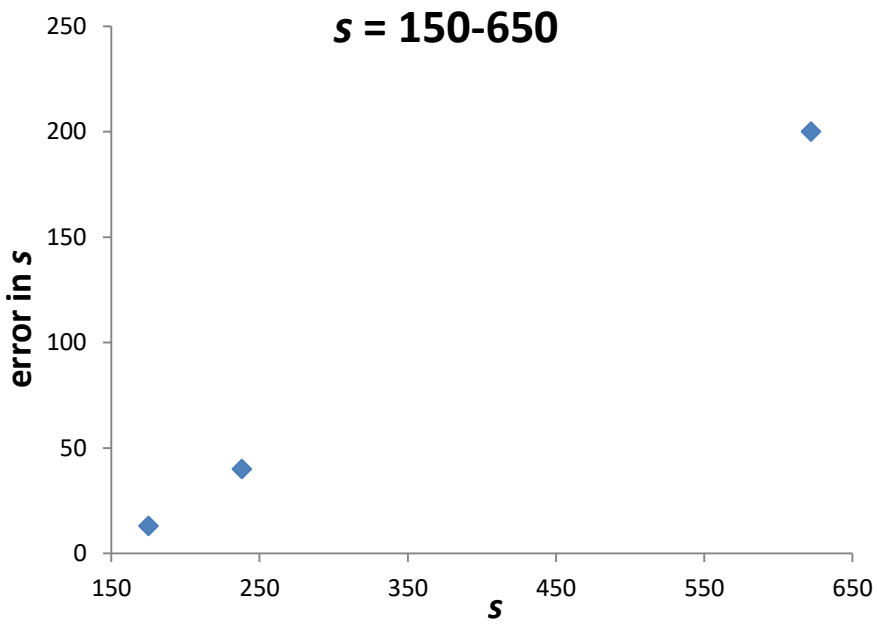
For *s* < 50, the associated errors are generally low (≤ 1). The error associated with the KR of **45** (±5) is larger due to the use of NMR conversion to determine *s*.



For *s* = 50–120, the errors are larger (2–9).



For *s* > 150, the errors become increasingly large.



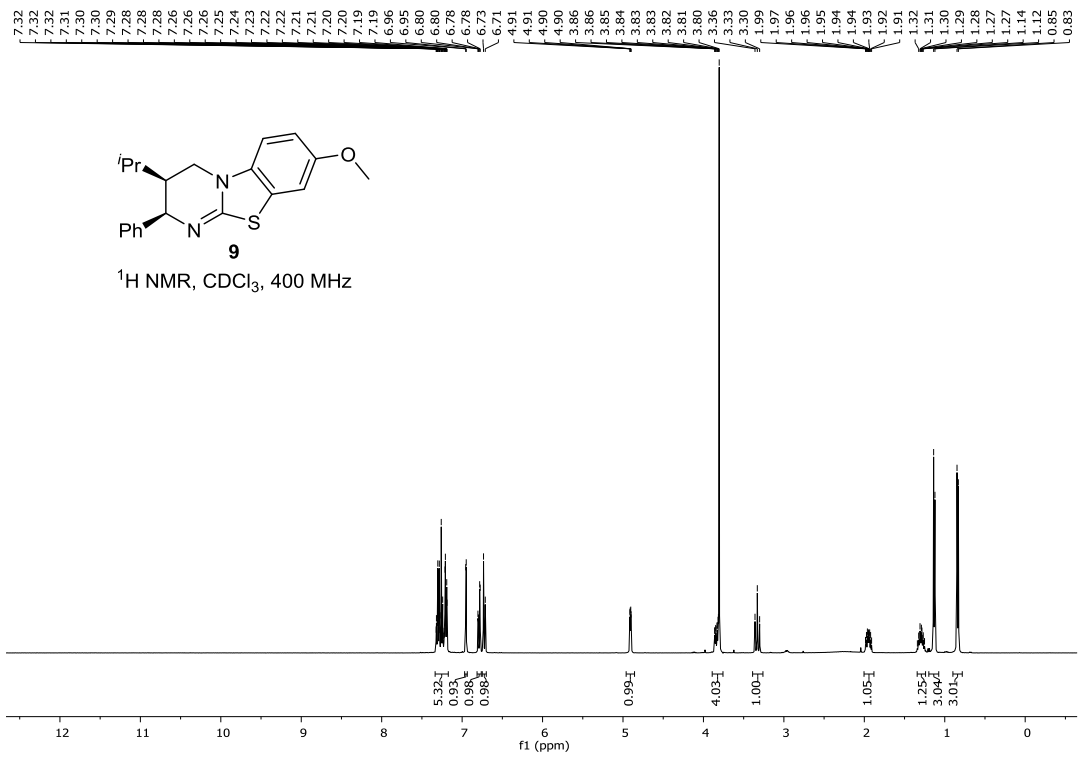
Conclusions:

The *s* values reported in the manuscript have therefore been adjusted to account for the likely associated error. This has led to the following general rules which have been applied:

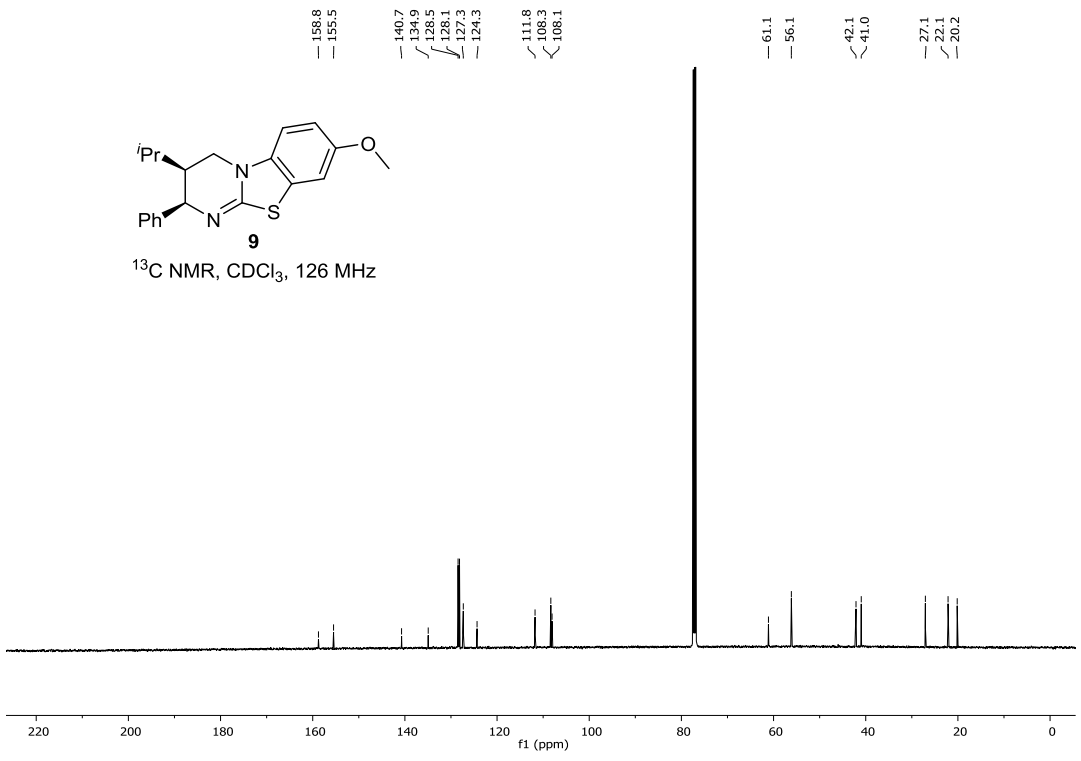
- For *s* < 50, the value is given to the closest integer
- For *s* 50–120, the value is given to the closest 10
- For *s* > 150, the values are given to the closest 100

In addition to the analytical error associated with *s* estimated here, there will be experimental between each KR. An indication of this error is represented in the reproducibility studies given in Figure 1, where the calculated error of ±12 is greater than the analytical error alone. This experimental error is not specific to KR processes however, with reaction yields, drs and ers reported in all synthetic methods inherently liable to include experimental error.

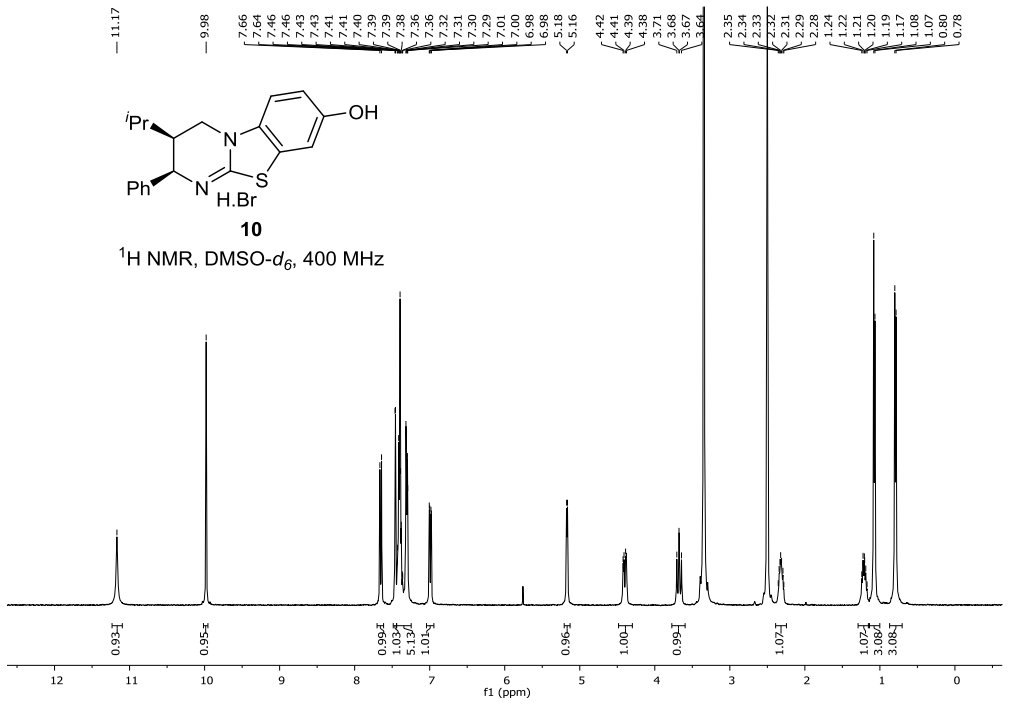
^{1}H & ^{13}C NMR Spectra

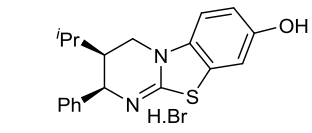


^{1}H NMR, $CDCl_3$, 400 MHz



^{13}C NMR, $CDCl_3$, 126 MHz



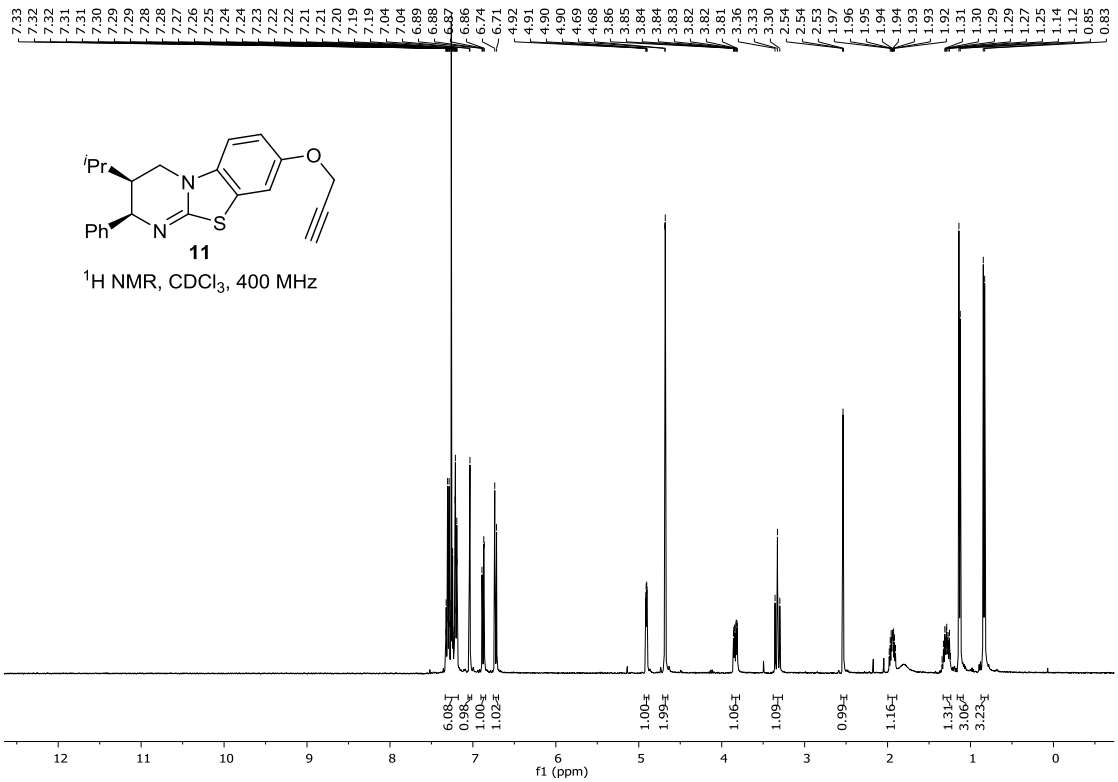


10

^{1}H NMR, DMSO-d_6, 400 MHz

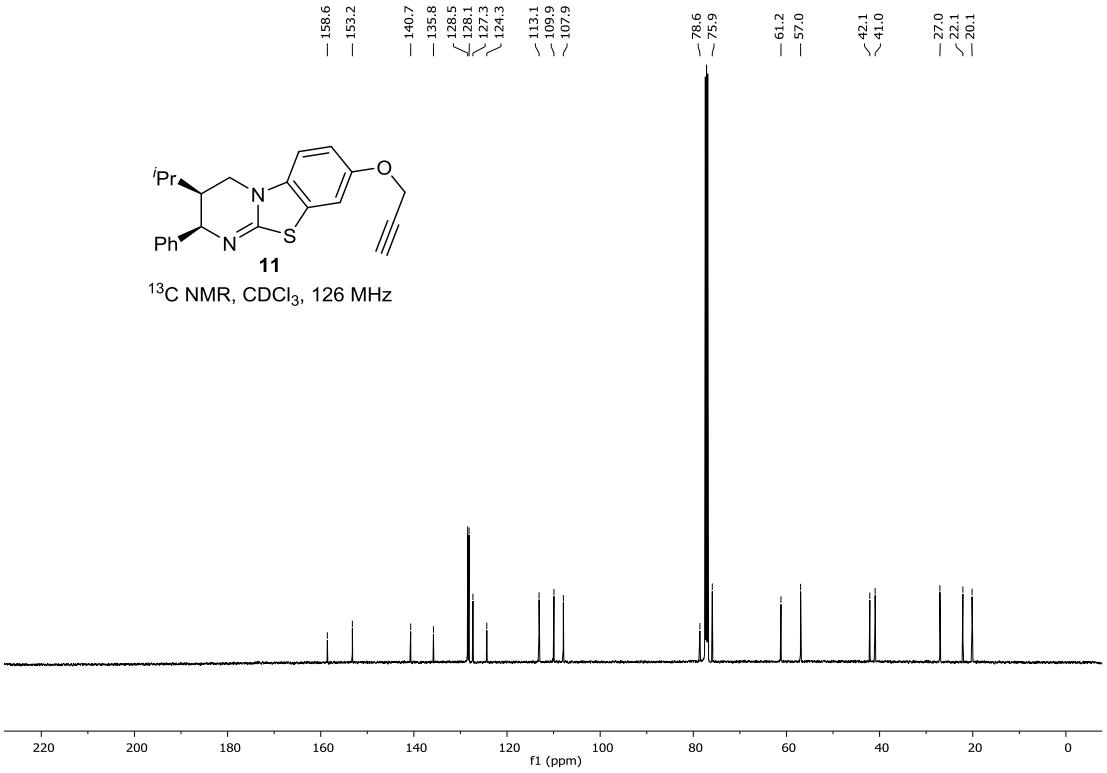
10

^{13}C NMR, DMSO-d_6, 126 MHz



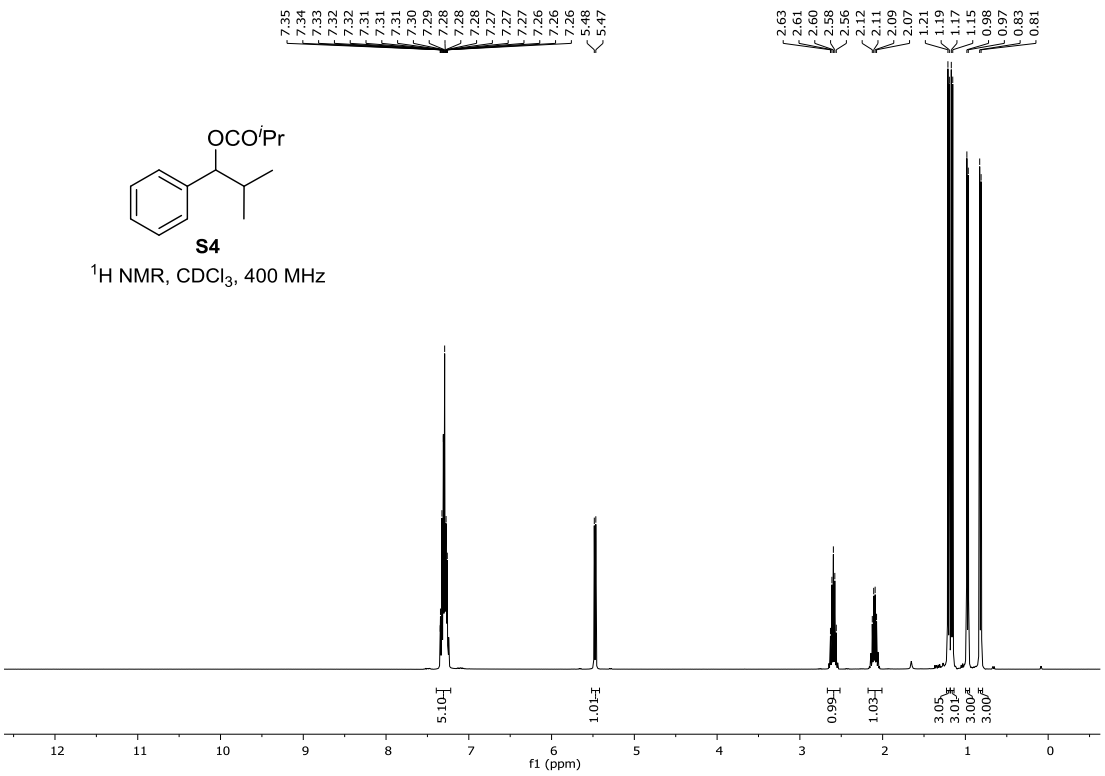
11

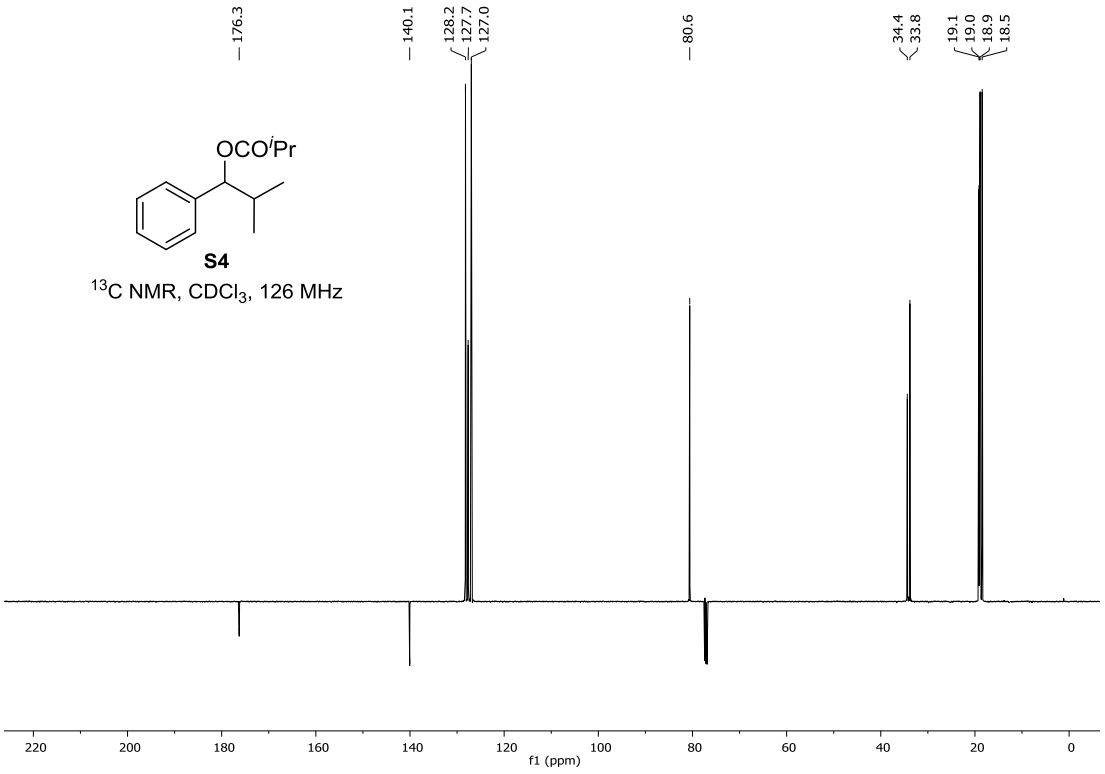
^{1}H NMR, $CDCl_3$, 400 MHz

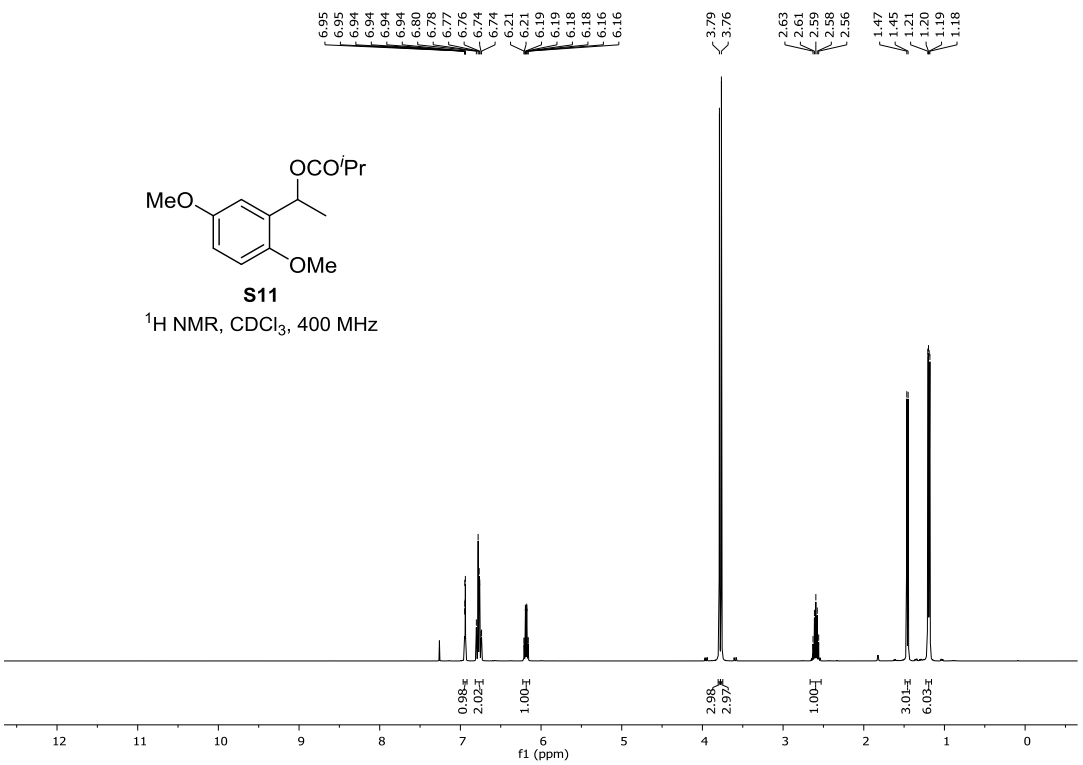


11

^{13}C NMR, $CDCl_3$, 126 MHz

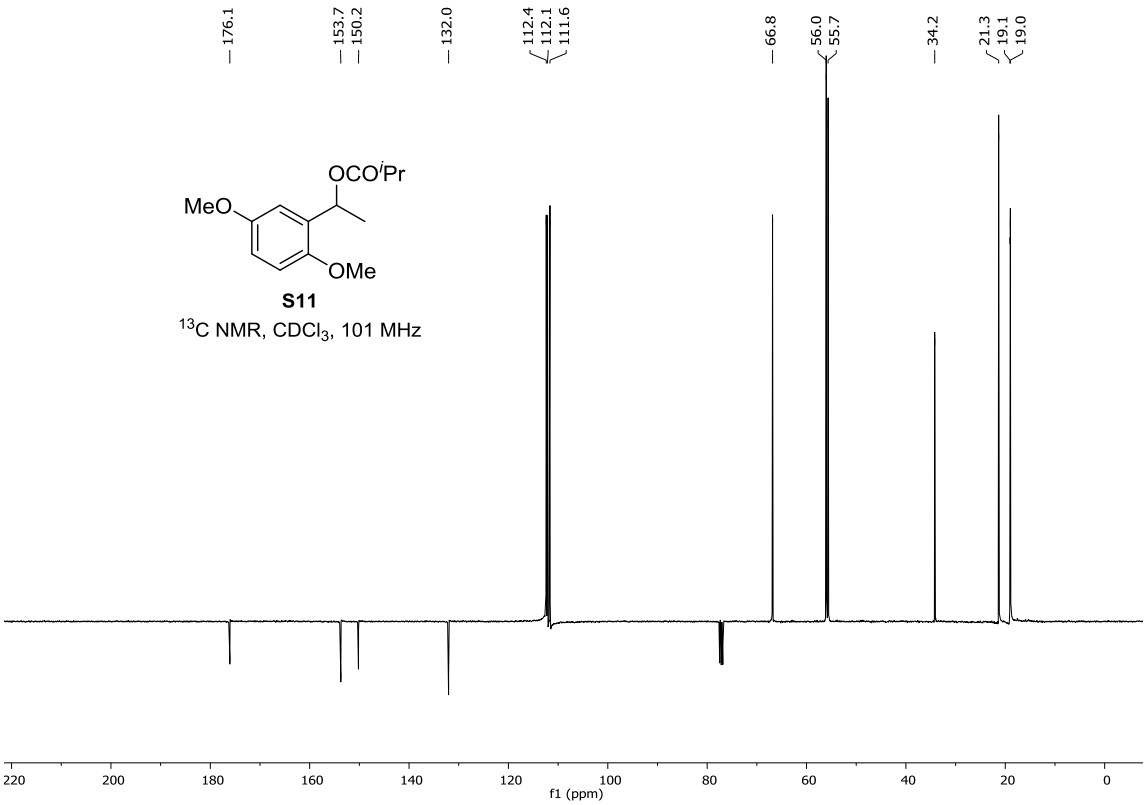

OCO^{i}Pr
S4
^{1}H NMR, CDCl$_3$, 400 MHz
7.35 7.34 7.33 7.32 7.32 7.31 7.31 7.31 7.30 7.29 7.28 7.28 7.28 7.27 7.27 7.27 7.26 7.26 7.26 5.48 5.47
2.63 2.61 2.60 2.58 2.56 2.12 2.11 2.09 2.07 1.21 1.19 1.17 1.15 0.98 0.97 0.83 0.81
5.10 1.01 0.99 1.03 3.05 3.01 3.00 3.00
f1 (ppm)


OCO^{i}Pr
S4
^{13}C NMR, CDCl$_3$, 126 MHz
176.3 140.1 128.2 127.7 127.0 80.6 34.4 33.8 19.1 19.0 18.9 18.5
f1 (ppm)



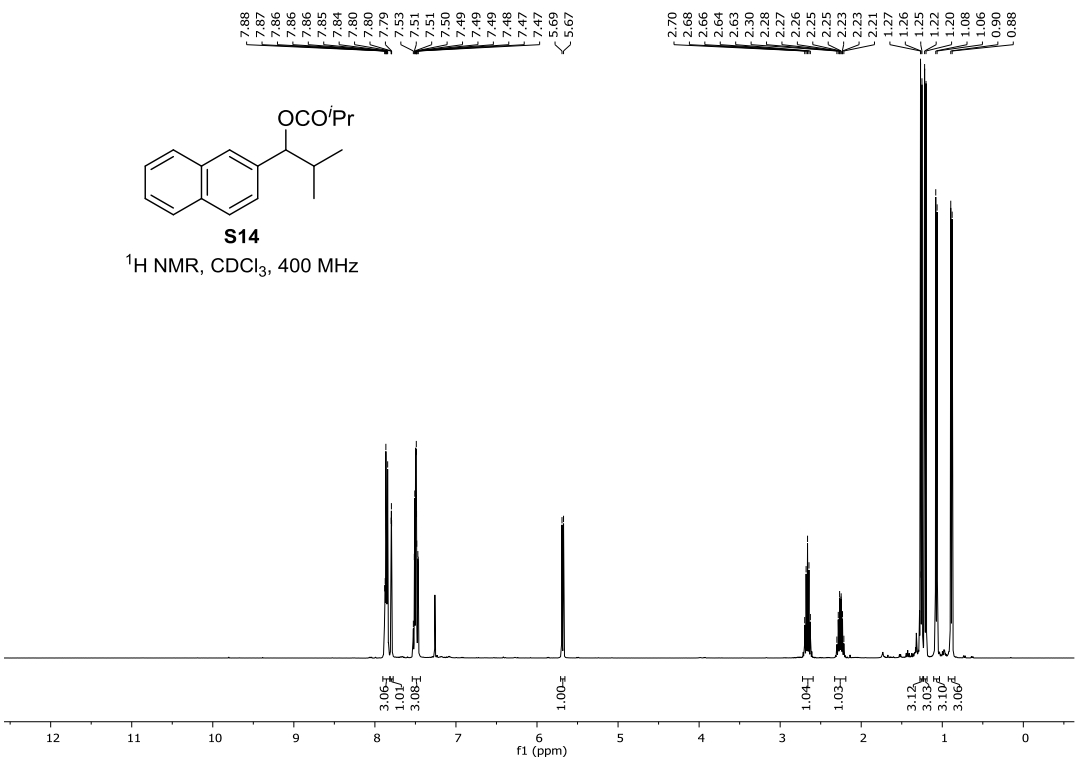
S11

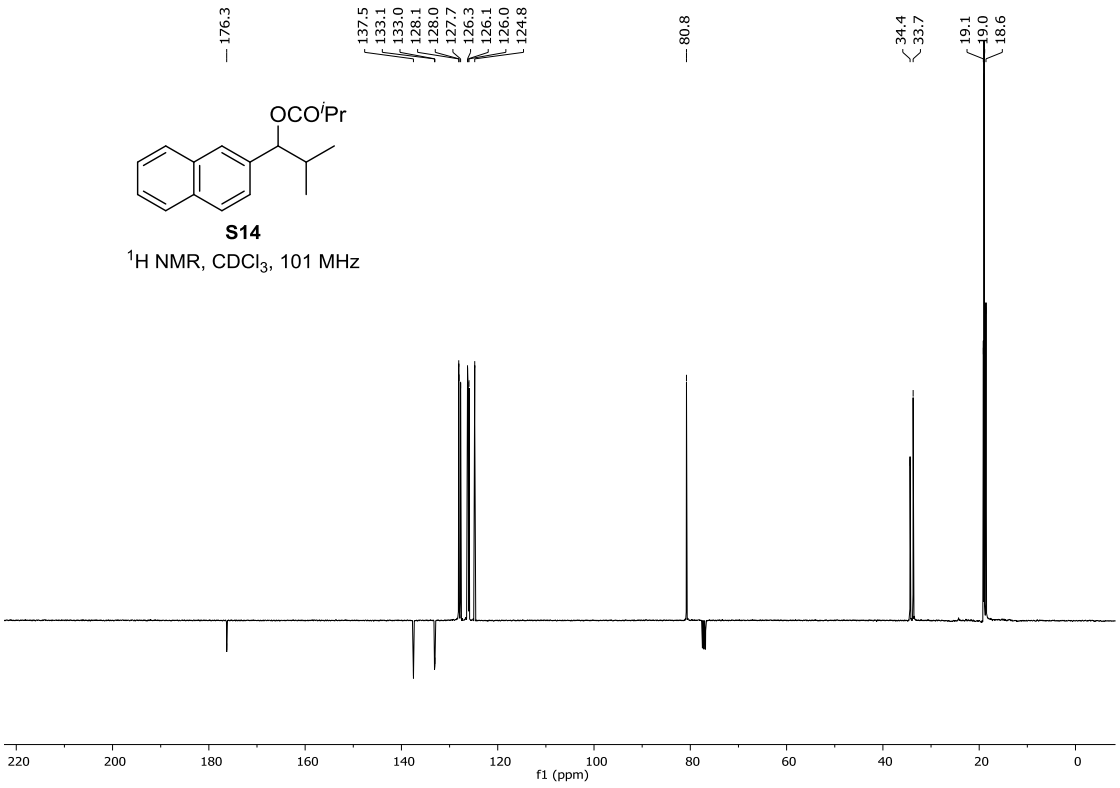
^{1}H NMR, $CDCl_3$, 400 MHz

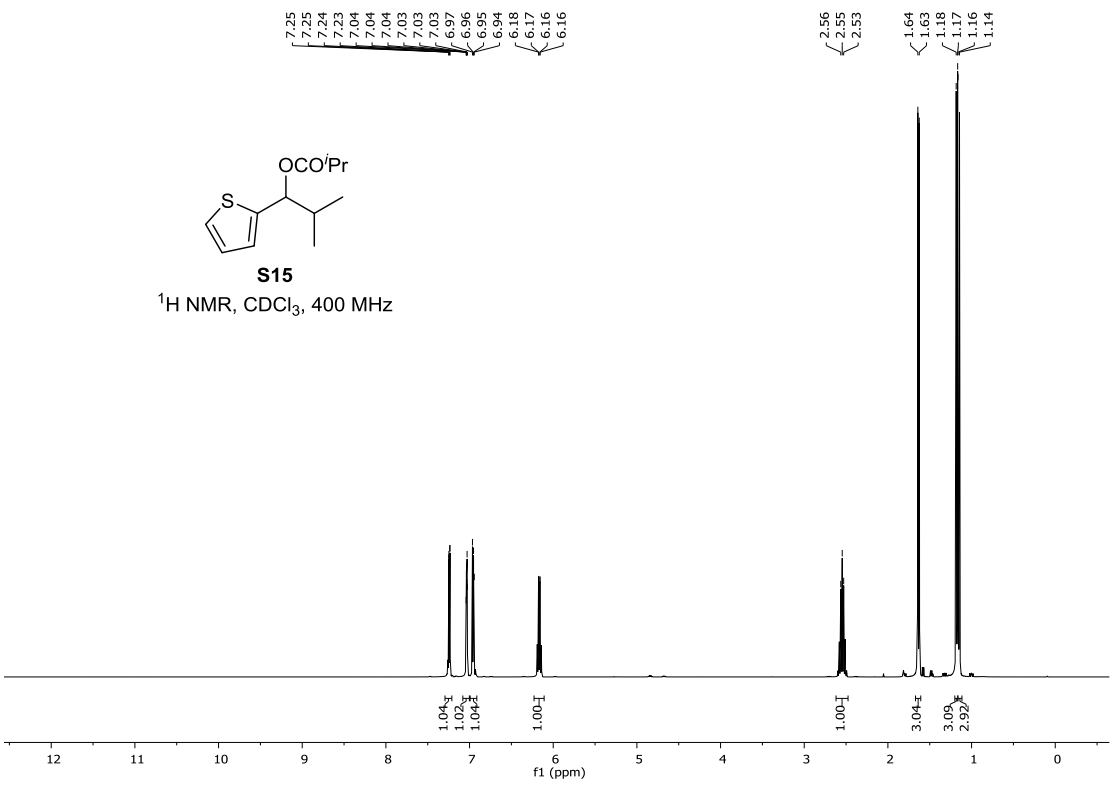


S11

^{13}C NMR, $CDCl_3$, 101 MHz

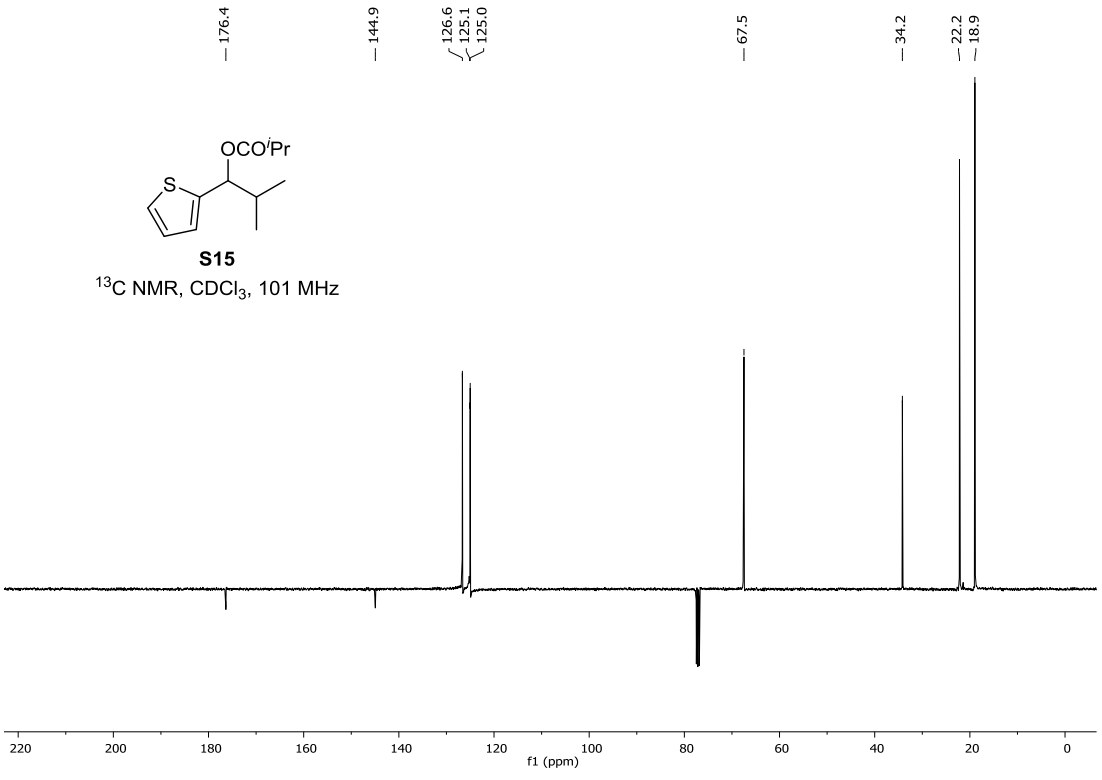

OCO^{i}Pr
S14
^{1}H NMR, CDCl$_3$, 400 MHz
f1 (ppm)


OCO^{i}Pr
S14
^{1}H NMR, CDCl$_3$, 101 MHz
f1 (ppm)



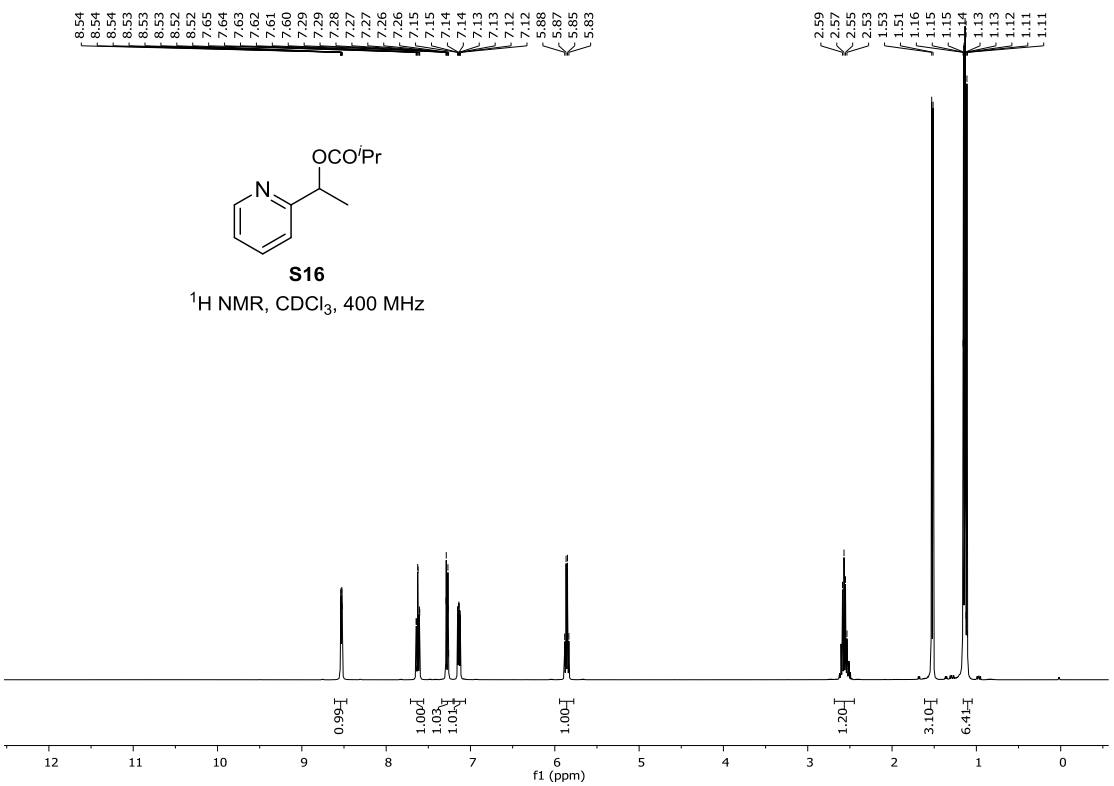
S15

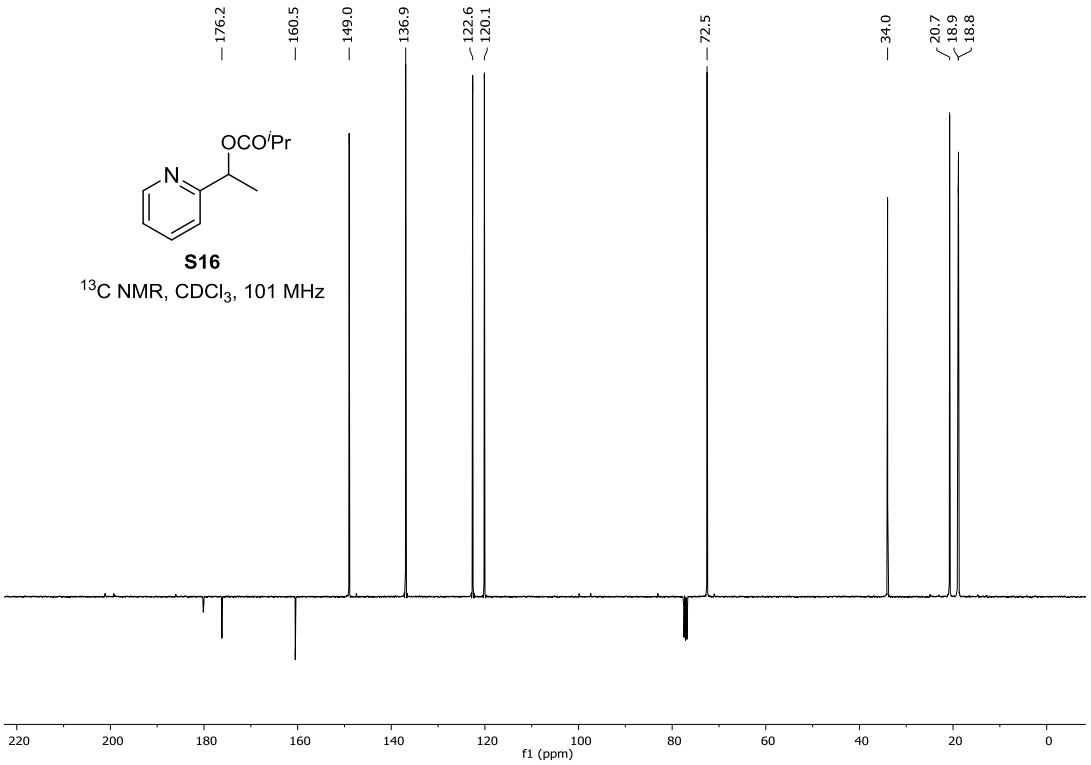
^{1}H NMR, $CDCl_3$, 400 MHz

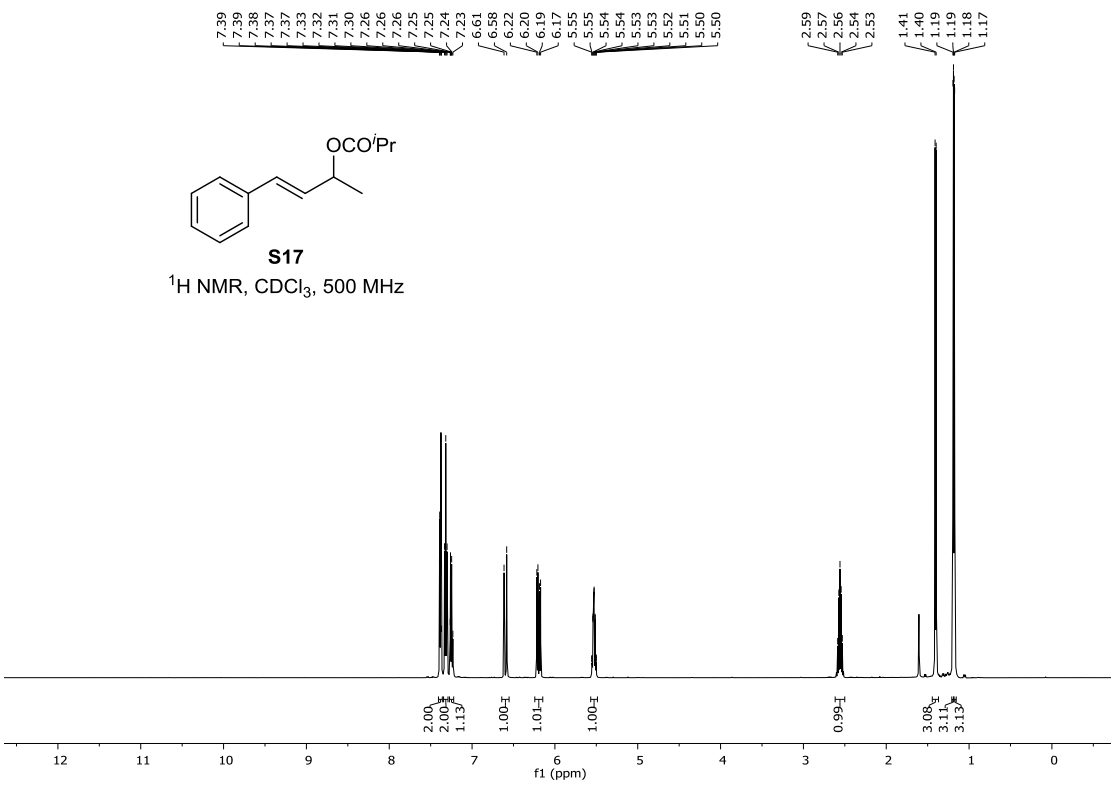


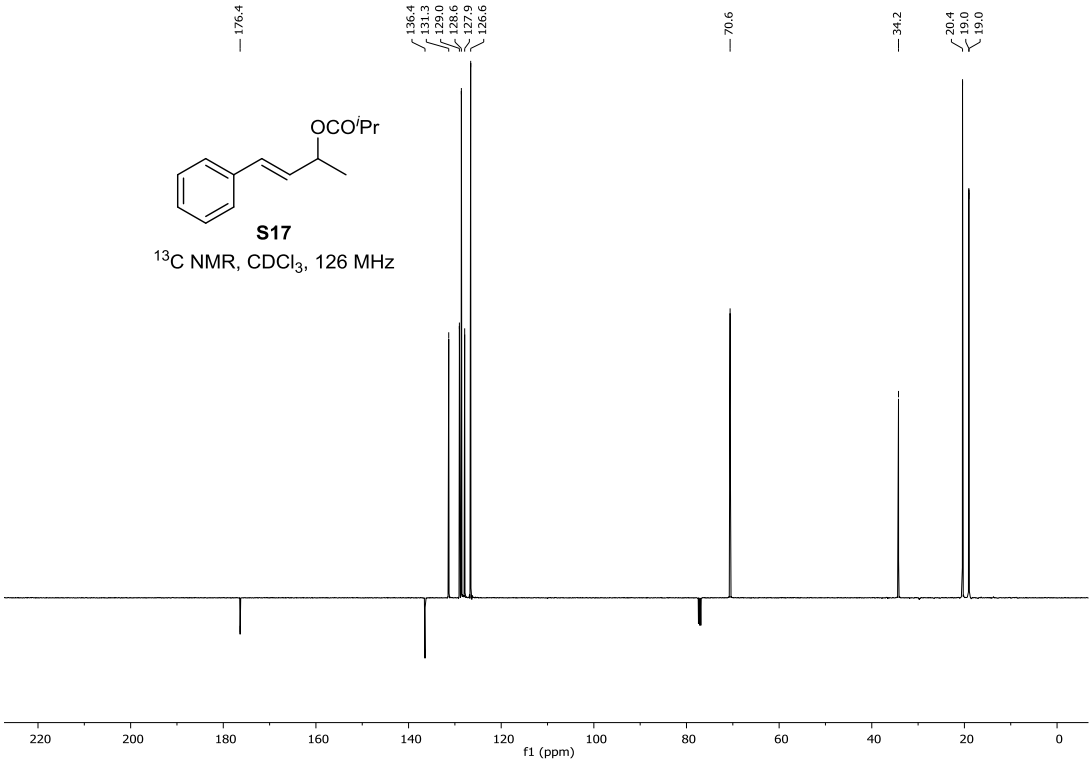
S15

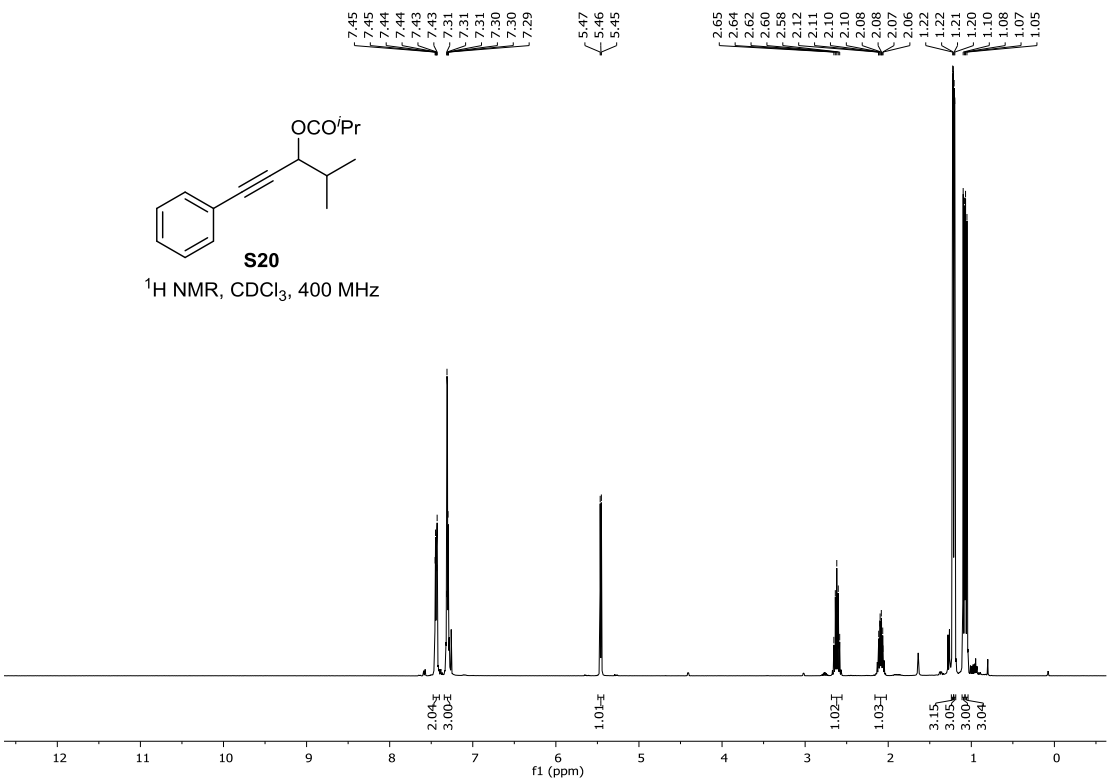
^{13}C NMR, $CDCl_3$, 101 MHz

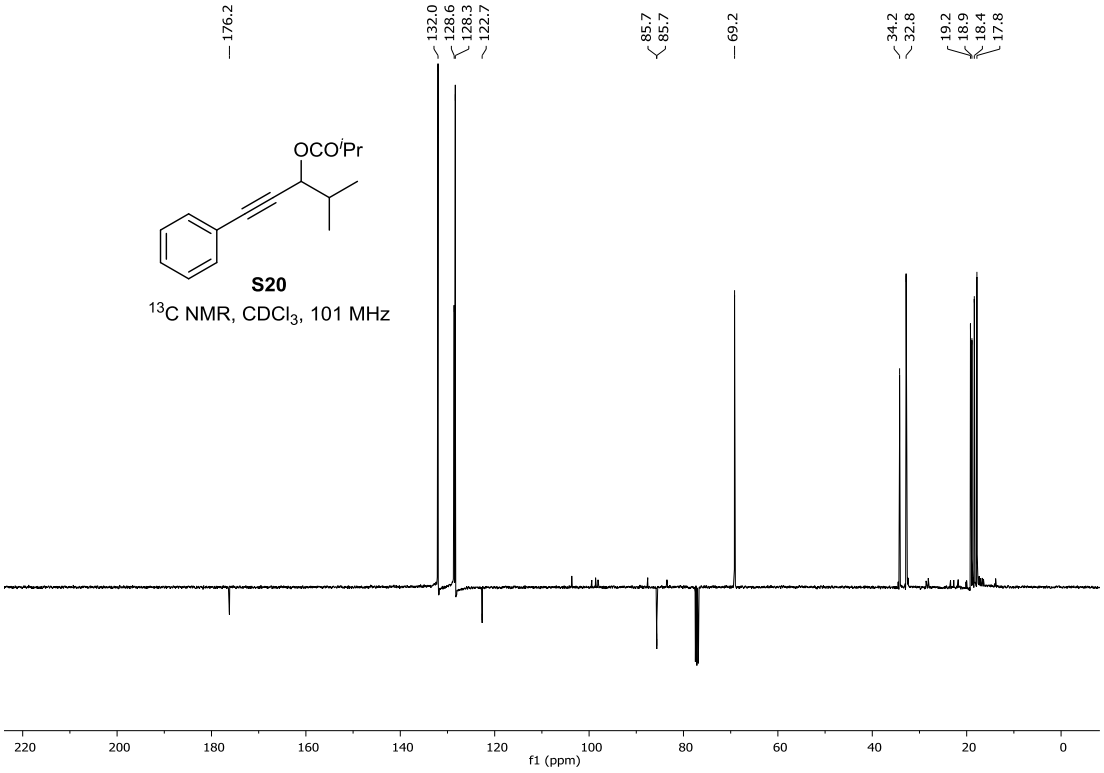

OCO^{i}Pr
N
S16
^{1}H NMR, CDCl$_3$, 400 MHz
f1 (ppm)

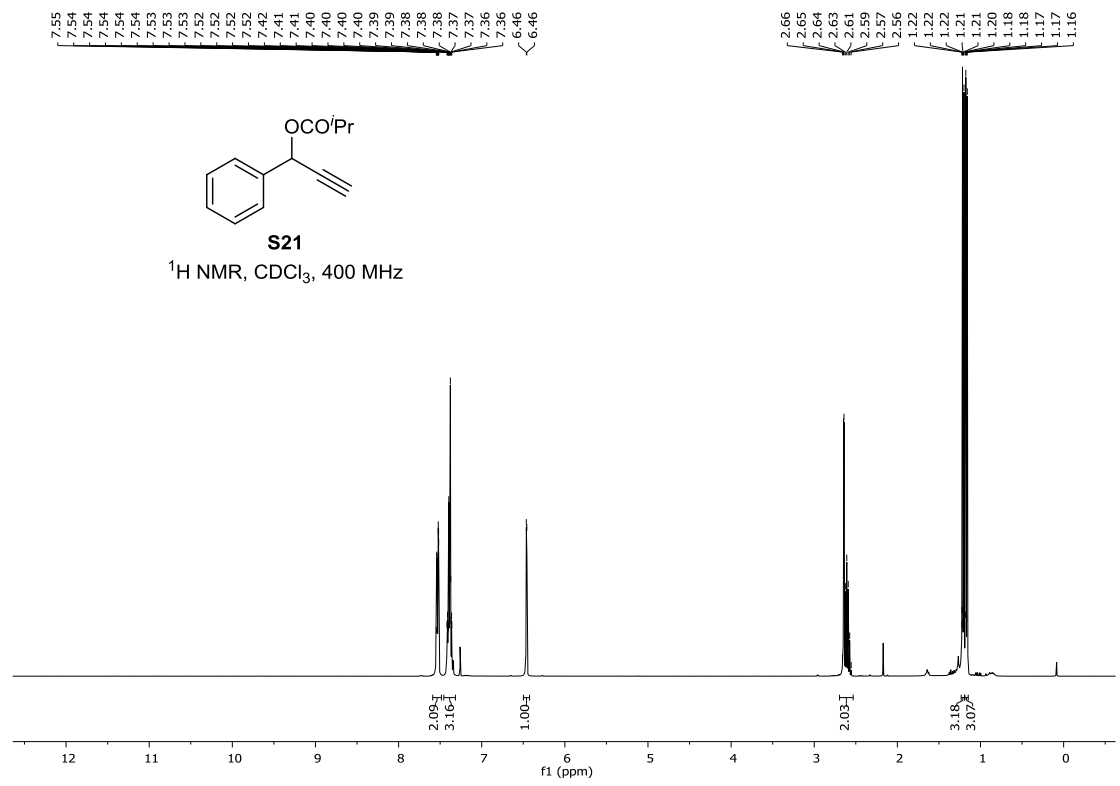

OCO^{i}Pr
N
S16
^{13}C NMR, CDCl$_3$, 101 MHz
f1 (ppm)

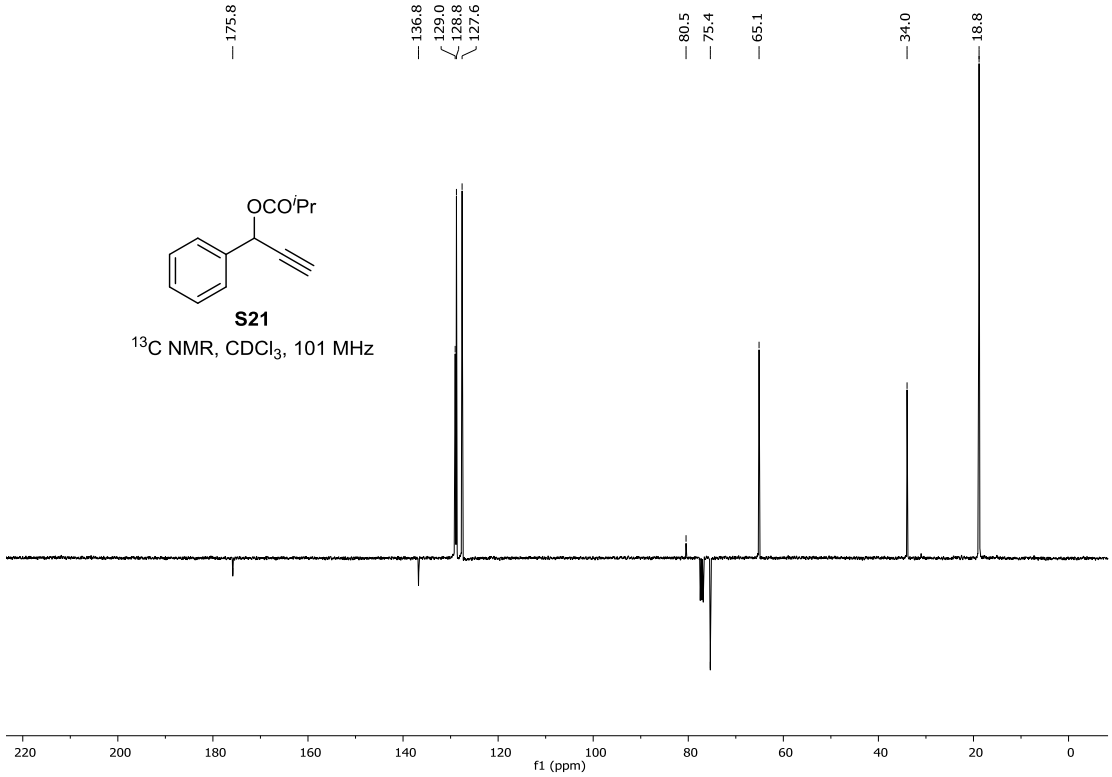

OCO^{i}Pr
S17
^{1}H NMR, CDCl$_3$, 500 MHz
f1 (ppm)

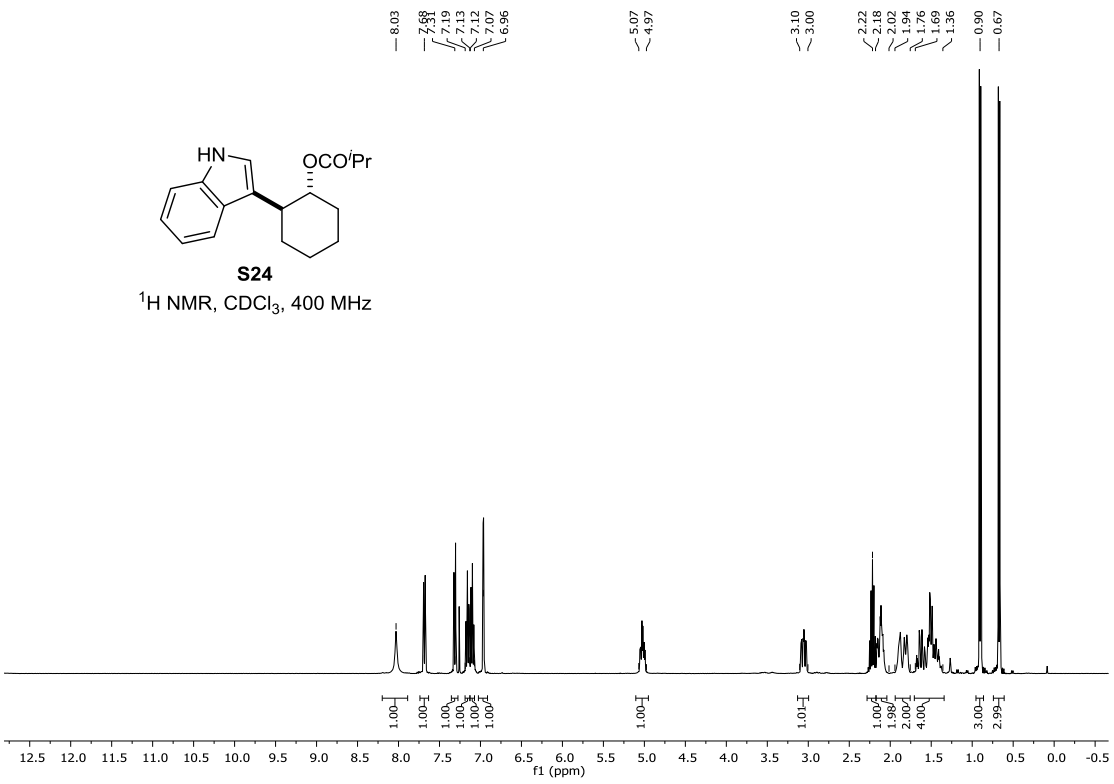

OCO^{i}Pr
S17
^{13}C NMR, CDCl$_3$, 126 MHz
f1 (ppm)


OCO^{i}Pr
S20
^{1}H NMR, CDCl$_3$, 400 MHz
f1 (ppm)


OCO^{i}Pr
S20
^{13}C NMR, CDCl$_3$, 101 MHz
f1 (ppm)

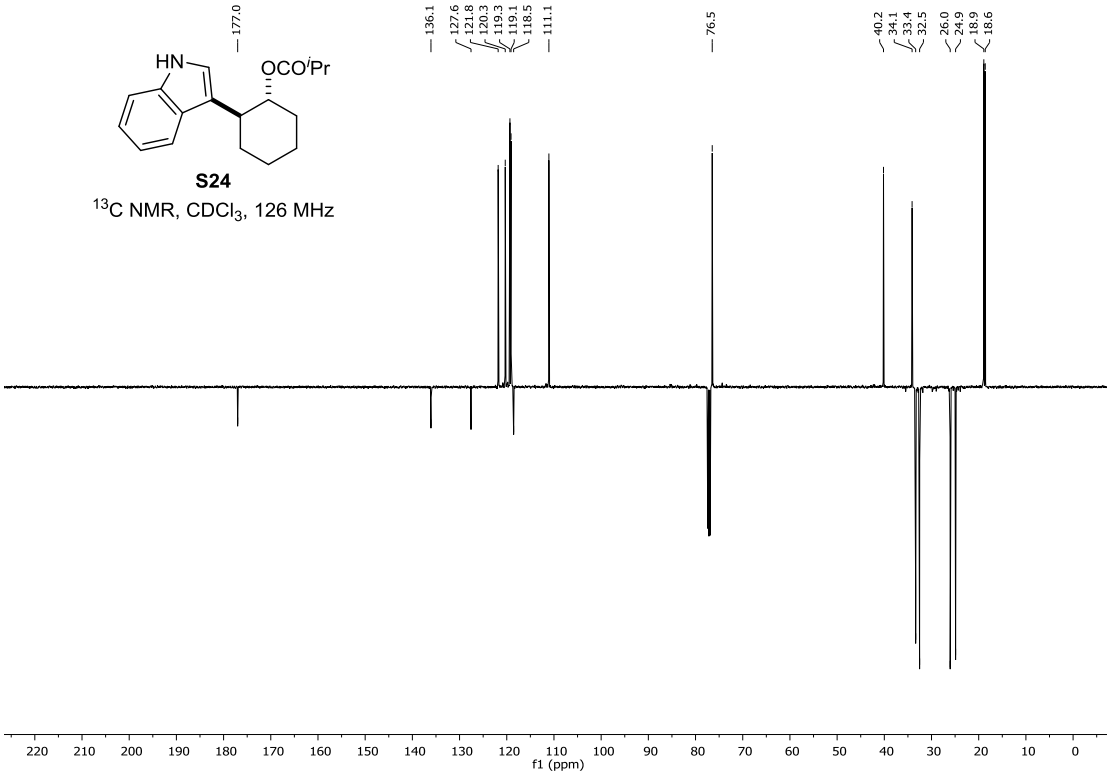

OCO^{i}Pr
S21
^{1}H NMR, CDCl$_3$, 400 MHz
f1 (ppm)


OCO^{i}Pr
S21
^{13}C NMR, CDCl$_3$, 101 MHz
f1 (ppm)



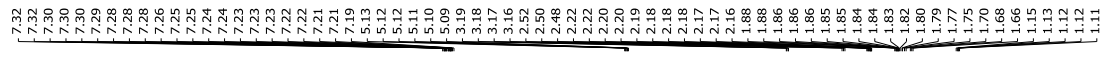
S24

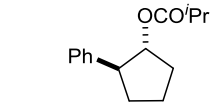
^{1}H NMR, $CDCl_3$, 400 MHz



S24

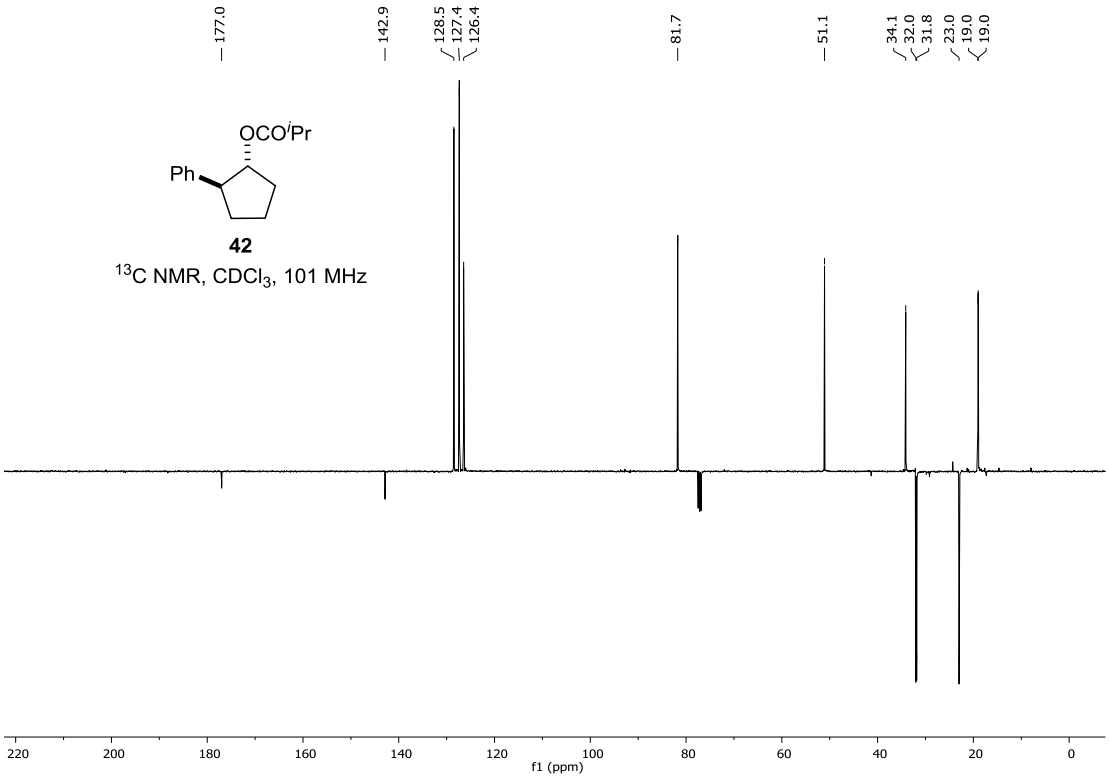
^{13}C NMR, $CDCl_3$, 126 MHz





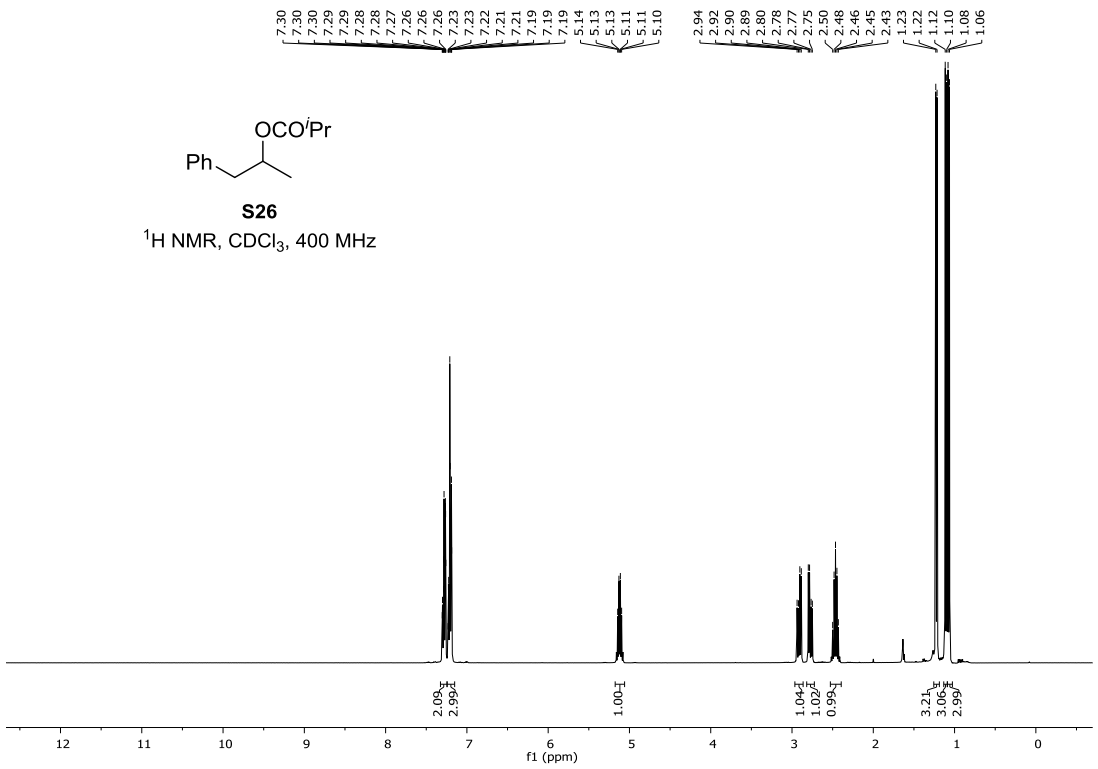
42

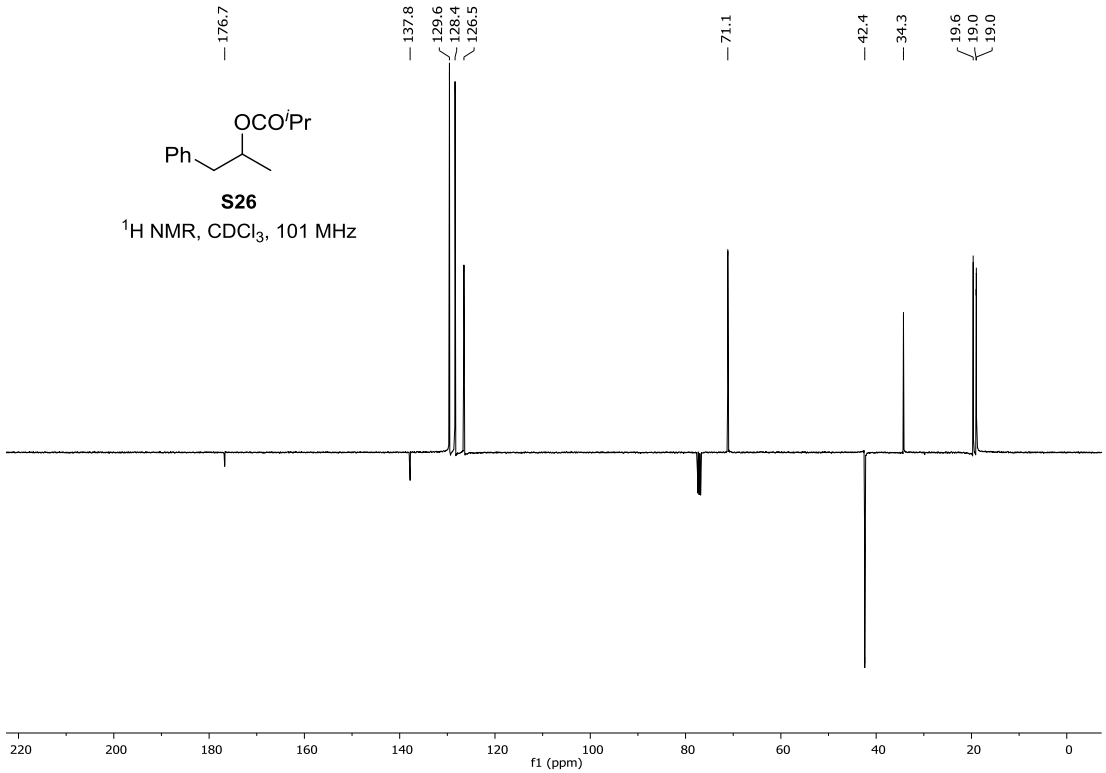
^{1}H NMR, $CDCl_3$, 400 MHz

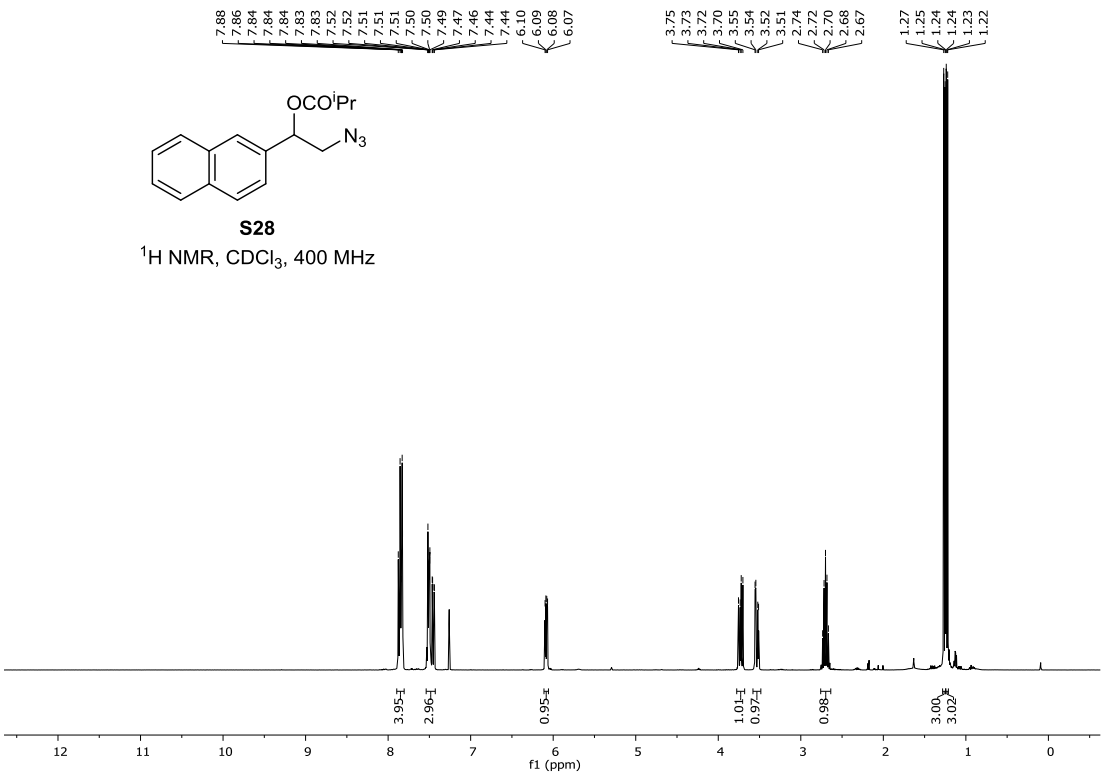


42

^{13}C NMR, $CDCl_3$, 101 MHz

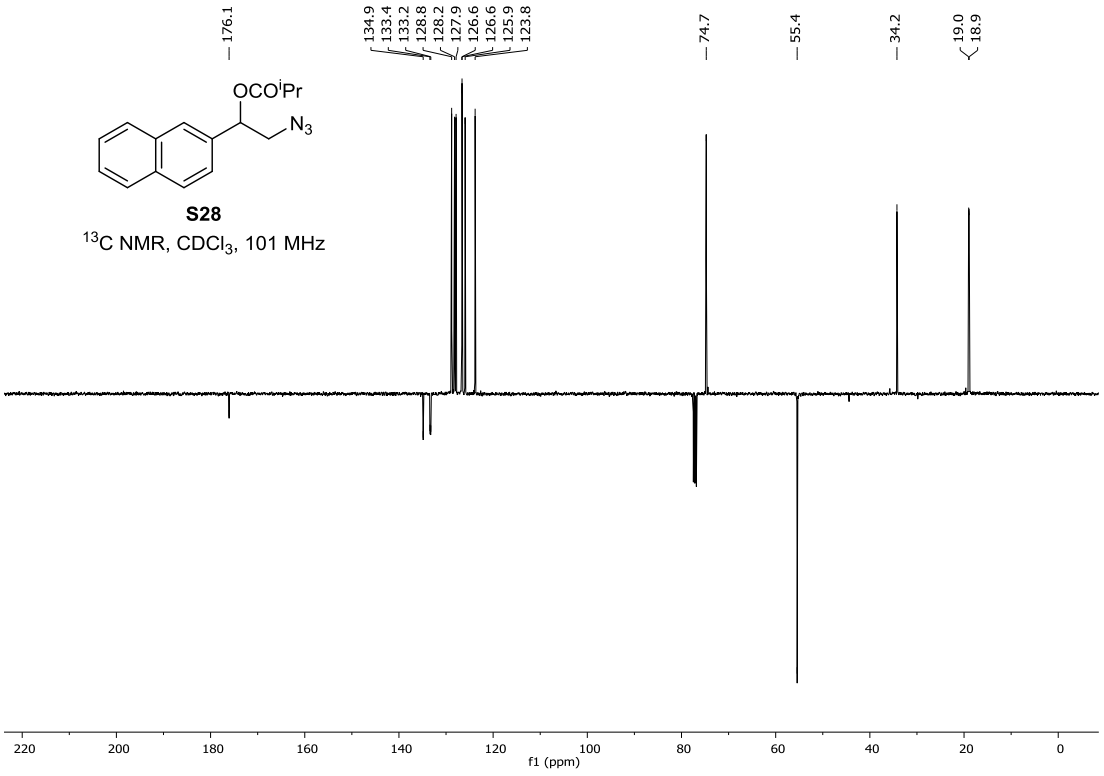

7.30
7.30
7.30
7.29
7.29
7.28
7.28
7.27
7.26
7.26
7.26
7.23
7.23
7.22
7.21
7.21
7.19
7.19
7.19
5.14
5.13
5.13
5.11
5.11
5.10
2.94
2.92
2.90
2.89
2.80
2.78
2.77
2.75
2.50
2.48
2.46
2.45
2.43
1.23
1.22
1.12
1.10
1.08
1.06
OCO^{i}Pr
Ph
S26
^{1}H NMR, CDCl$_3$, 400 MHz
2.09
2.99
1.00
1.04
1.02
0.99
3.21
3.06
2.99
f1 (ppm)


176.7
137.8
129.6
128.4
126.5
71.1
42.4
34.3
19.6
19.0
19.0
OCO^{i}Pr
Ph
S26
^{1}H NMR, CDCl$_3$, 101 MHz
f1 (ppm)



S28

^{1}H NMR, $CDCl_3$, 400 MHz

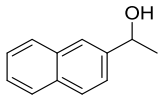


S28

^{13}C NMR, $CDCl_3$, 101 MHz

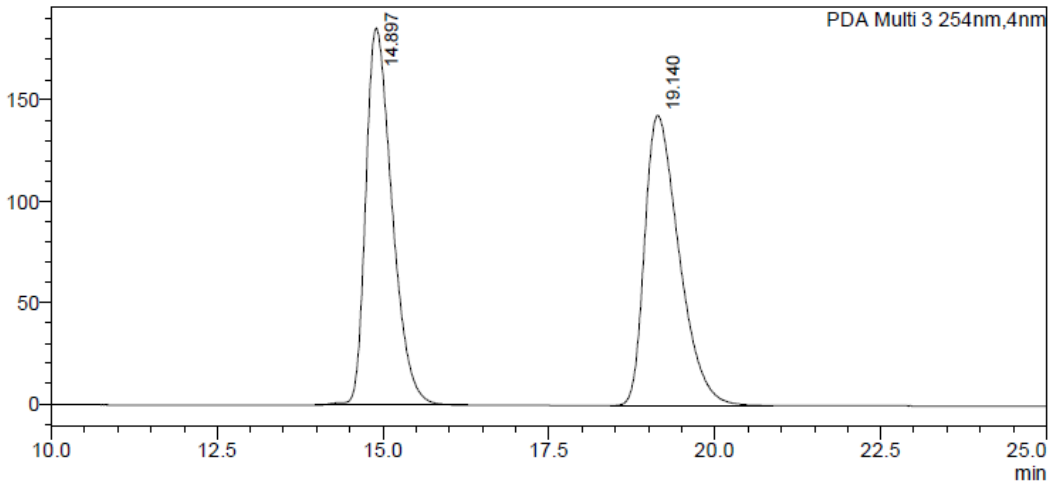
HPLC Spectra

HPLC Data for 12: Chiralcel OJ-H (90:10 hexane : IPA, flow rate 1.0 mLmin^{-1}, 254 nm, 30 °C) t_R (*S*): 15.0 min, t_R (*R*): 19.1 min, 7.654:92.346 er. *S* = 104



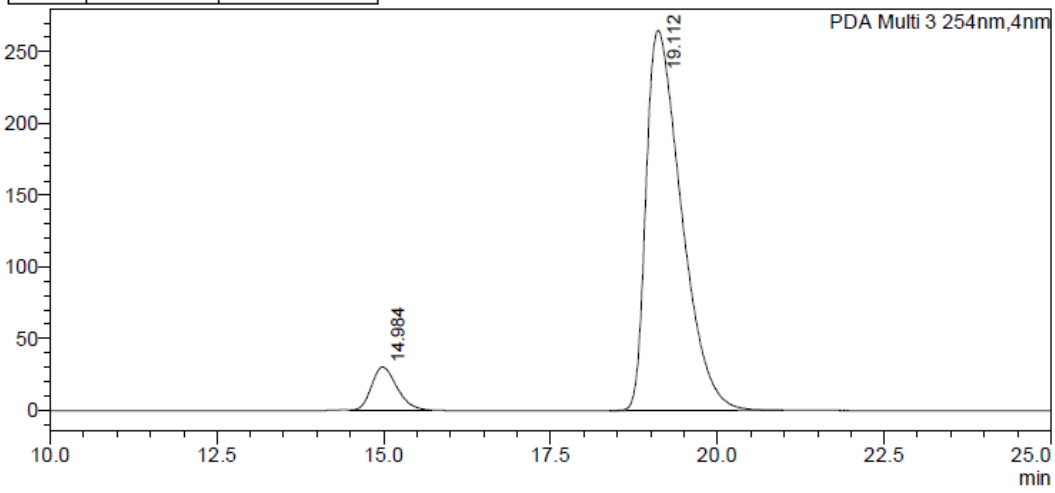
PDA Ch3 254nm

Peak#	Ret. Time	Area%
1	14.897	49.923
2	19.140	50.077
Total		100.000

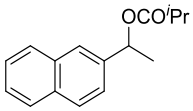


PDA Ch3 254nm

Peak#	Ret. Time	Area%
1	14.984	7.654
2	19.112	92.346
Total		100.000

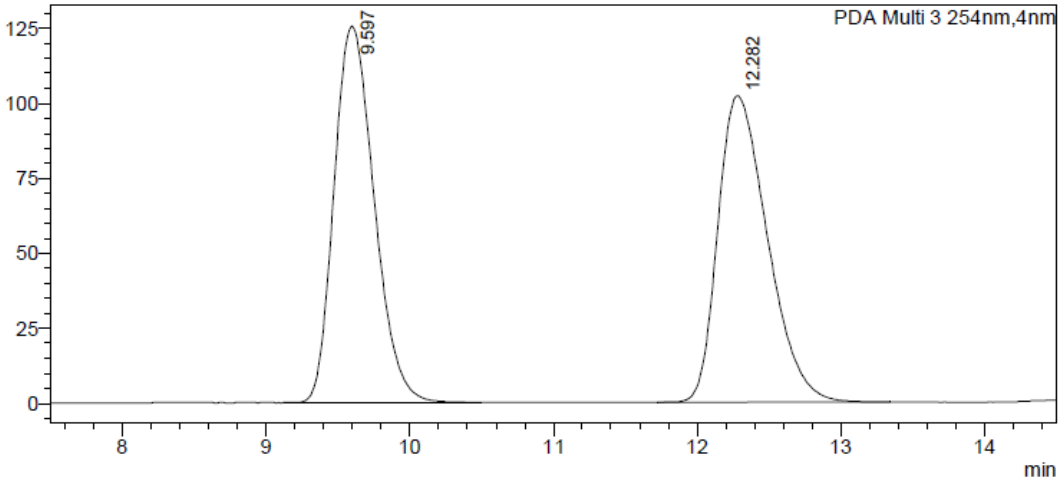


HPLC Data for 13: Chiralcel OJ-H (98:2 hexane : IPA, flow rate 1.0 mLmin^{-1}, 254 nm, 30 °C) t_R (*R*): 9.4 min, t_R (*S*): 11.6 min, 2.533:97.467 er.



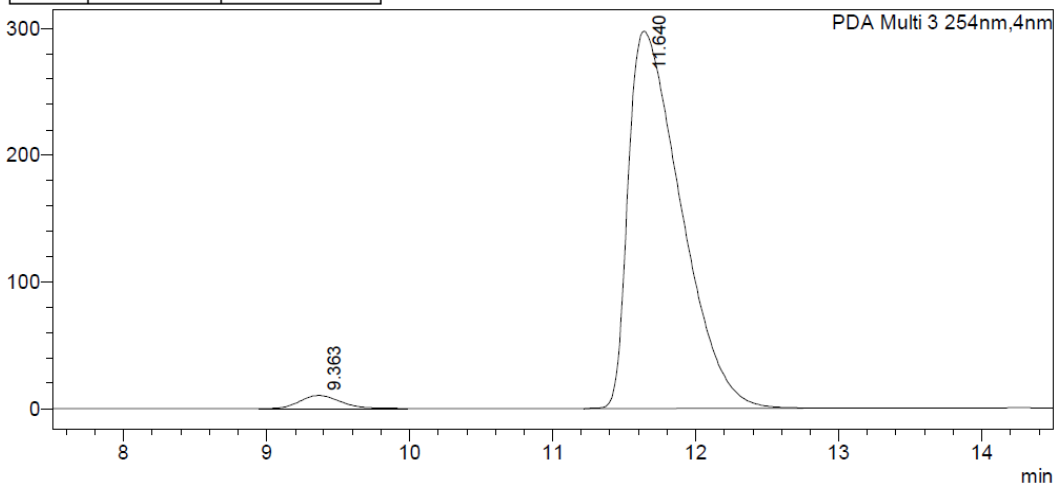
PDA Ch3 254nm

Peak#	Ret. Time	Area%
1	9.597	49.837
2	12.282	50.163
Total		100.000

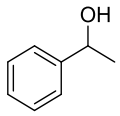


PDA Ch3 254nm

Peak#	Ret. Time	Area%
1	9.363	2.533
2	11.640	97.467
Total		100.000

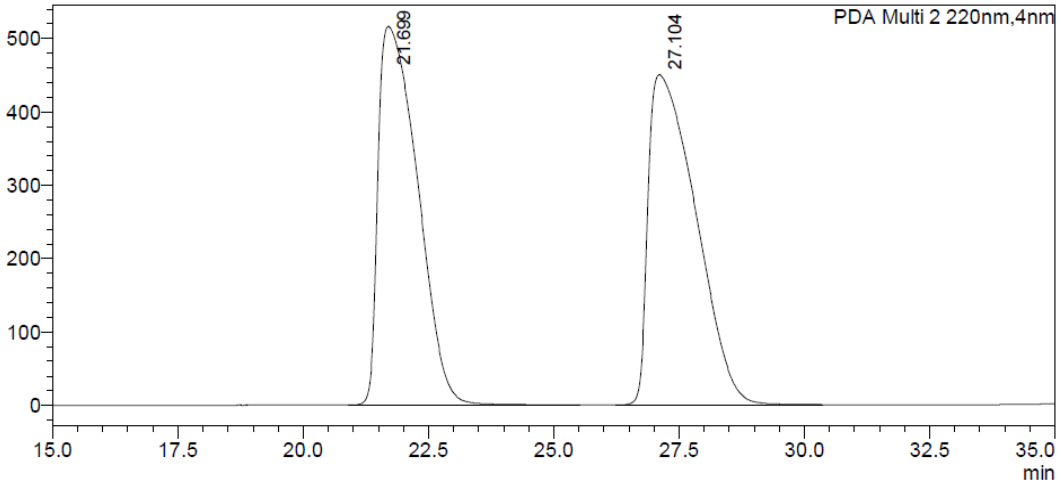


HPLC Data for 15: Chiralcel OD-H (99:1 hexane : IPA, flow rate 1.00 mLmin^{-1}, 220 nm, 30 °C) t_R (*R*): 22.9 min, t_R (*S*) : 28.6 min, 84.573:15.427 er. S = 46.4



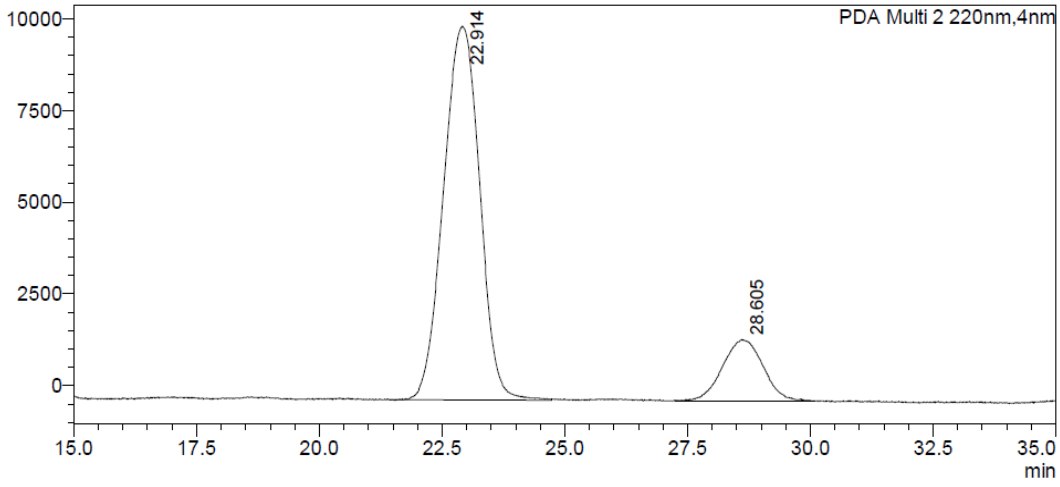
PDA Ch2 220nm

Peak#	Ret. Time	Area%
1	21.699	48.749
2	27.104	51.251
Total		100.000

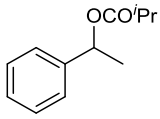


PDA Ch2 220nm

Peak#	Ret. Time	Area%
1	22.914	84.573
2	28.605	15.427
Total		100.000

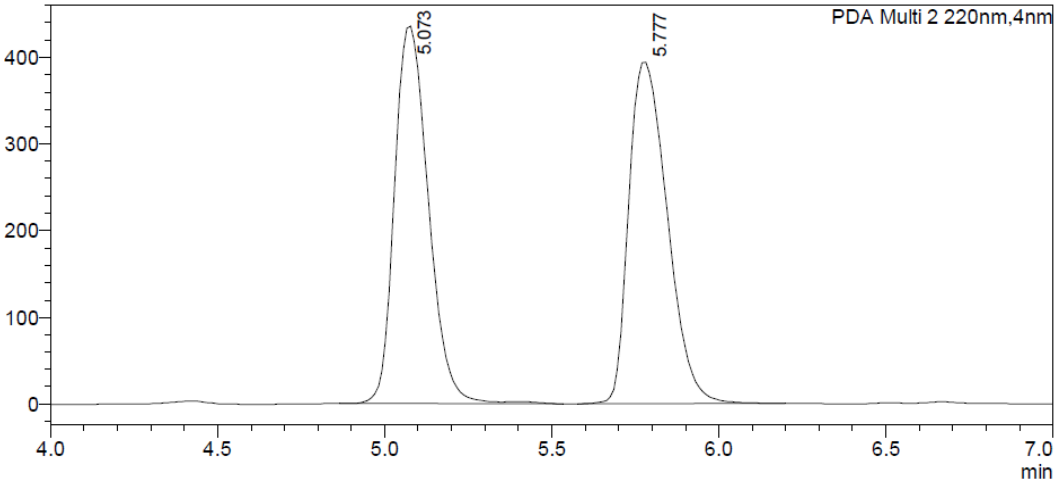


HPLC Data for S2: Chiralpak AD-H (99.8:0.2 hexane : IPA, flow rate 1.00 mLmin^{-1}, 220 nm, 30 °C) t_R (*R*): 4.9 min, t_R (*S*) : 5.6 min, 4.272:95.728 er.



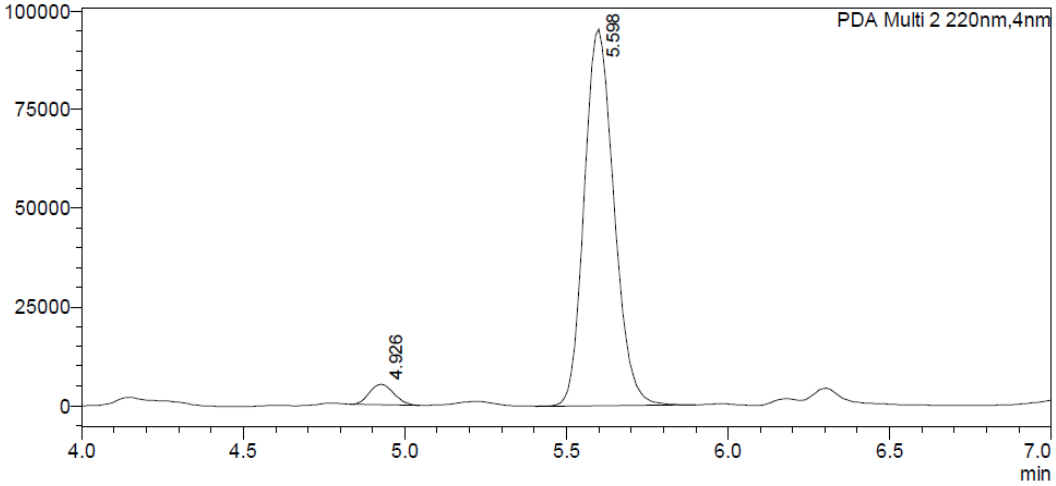
PDA Ch2 220nm

Peak#	Ret. Time	Area%
1	5.073	49.320
2	5.777	50.680
Total		100.000

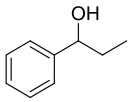


PDA Ch2 220nm

Peak#	Ret. Time	Area%
1	4.926	4.272
2	5.598	95.728
Total		100.000

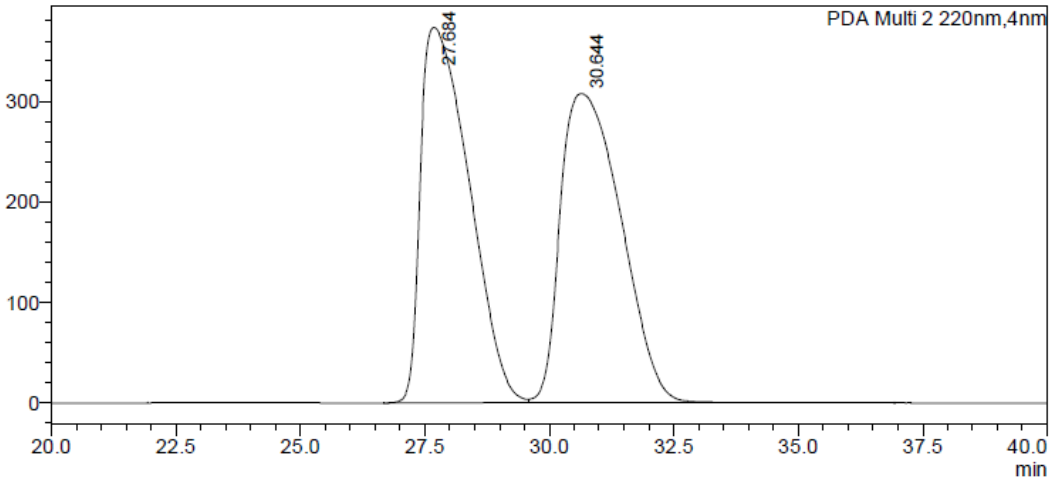


HPLC Data for 16: Chiralcel OD-H (99.5:0.5 hexane : IPA, flow rate 1.00 mLmin^{-1}, 220 nm, 30 °C) t_R (*R*): 28.8 min, t_R (*S*): 31.6 min, 91.705:8.295 er. *s*=64.8



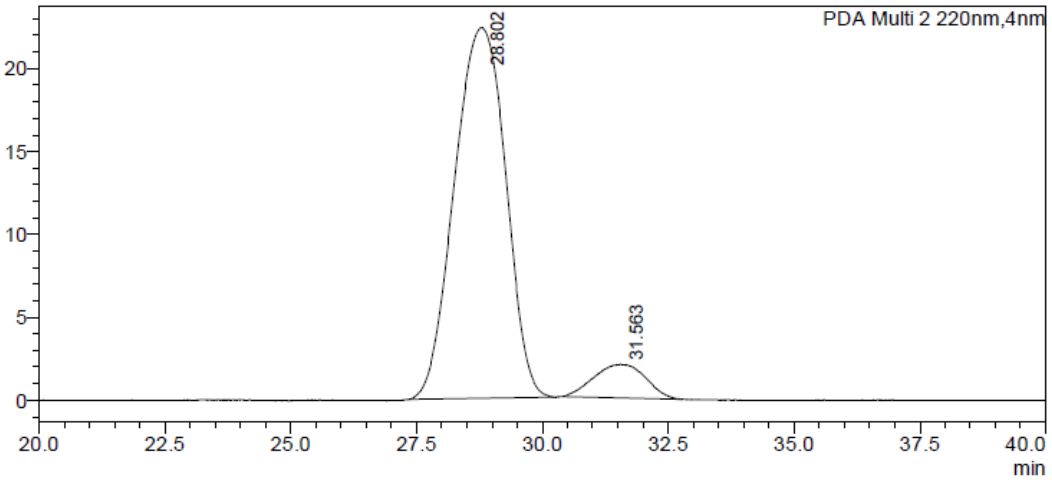
PDA Ch2 220nm

Peak#	Ret. Time	Area%
1	27.684	49.379
2	30.644	50.621
Total		100.000

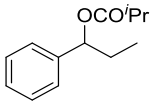


PDA Ch2 220nm

Peak#	Ret. Time	Area%
1	28.802	91.705
2	31.563	8.295
Total		100.000

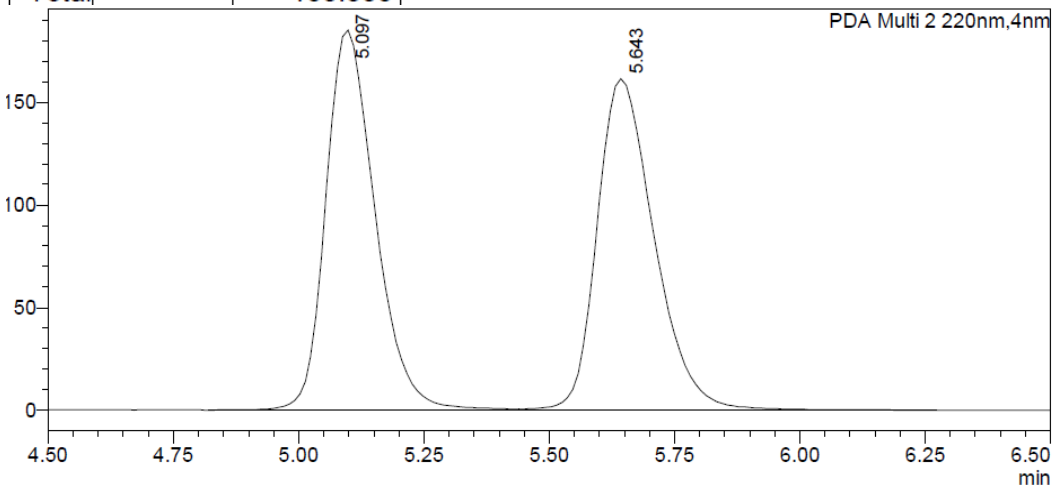


HPLC Data for S3: Chiralpak AD-H (99.8:0.2 hexane : IPA, flow rate 1.00 mLmin^{-1}, 220 nm, 30 °C) t_R (*R*): 5.1 min, t_R (*S*): 5.6 min, 3.890:96.110 er.



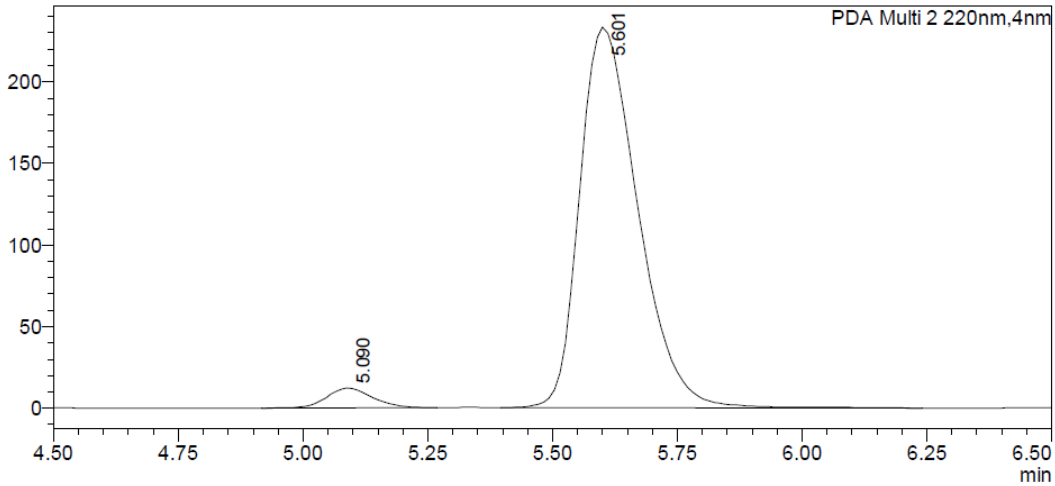
PDA Ch2 220nm

Peak#	Ret. Time	Area%
1	5.097	49.762
2	5.643	50.238
Total		100.000

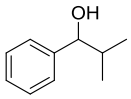


PDA Ch2 220nm

Peak#	Ret. Time	Area%
1	5.090	3.890
2	5.601	96.110
Total		100.000

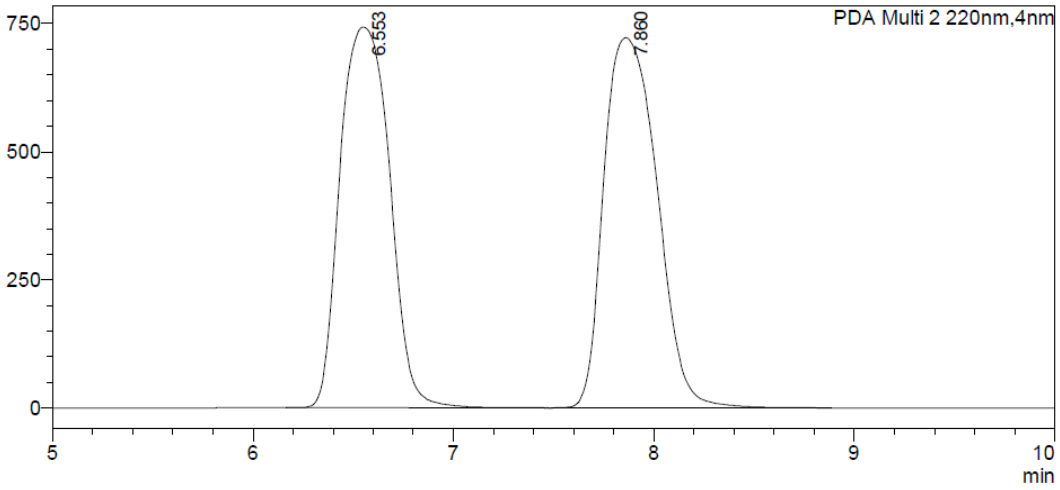


HPLC Data for 17: Chiralcel OD-H (95:5 hexane : IPA, flow rate 1.00 mLmin^{-1}, 220 nm, 30 °C) t_R (*S*): 6.6 min, t_R (*R*): 7.9 min, 18.107:81.893 er. *s*=80.1



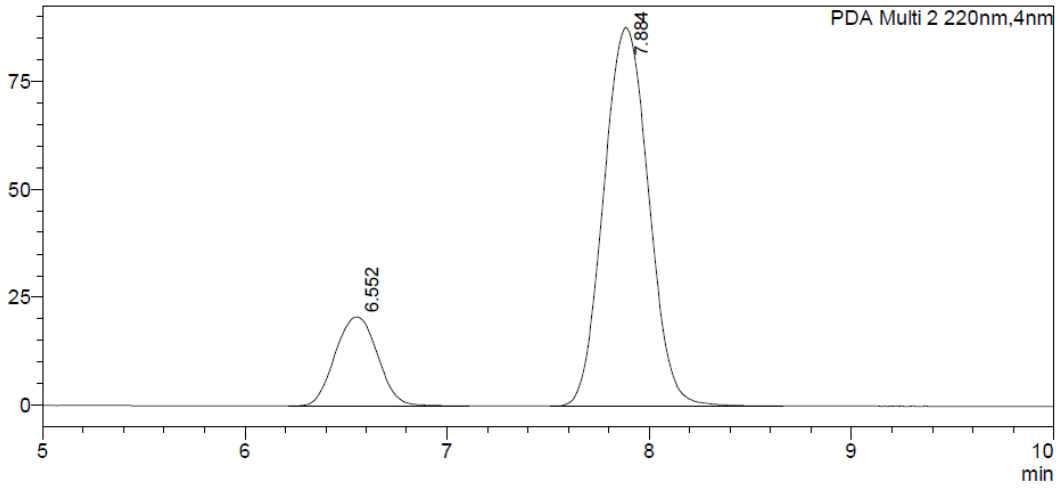
PDA Ch2 220nm

Peak#	Ret. Time	Area%
1	6.553	48.949
2	7.860	51.051
Total		100.000

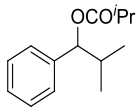


PDA Ch2 220nm

Peak#	Ret. Time	Area%
1	6.552	18.107
2	7.884	81.893
Total		100.000

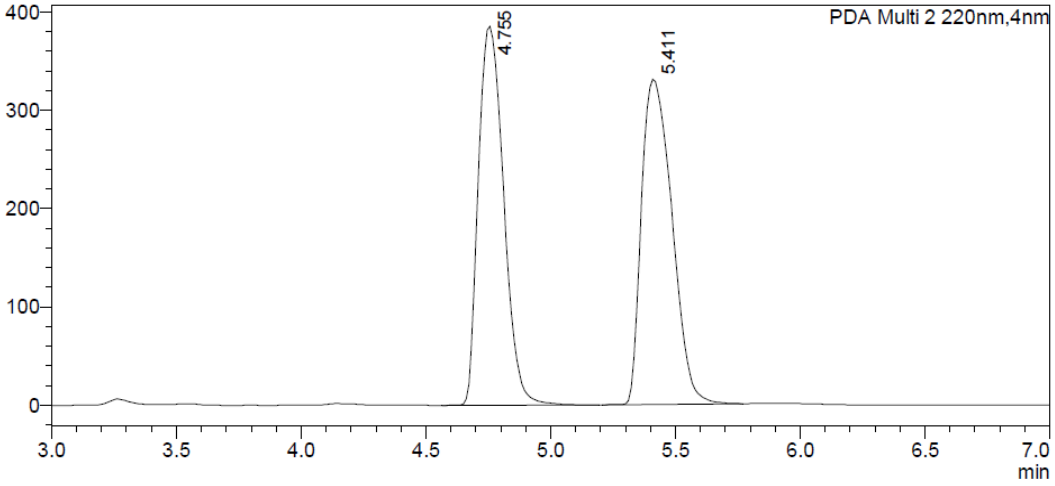


HPLC Data for S4: Chiralpak AD-H (99.8:0.2 hexane : IPA, flow rate 1.00 mLmin^{-1}, 220 nm, 30 °C) t_R (*R*): 5.2 min, t_R (*S*): 6.0 min, 2.355:97.645 er



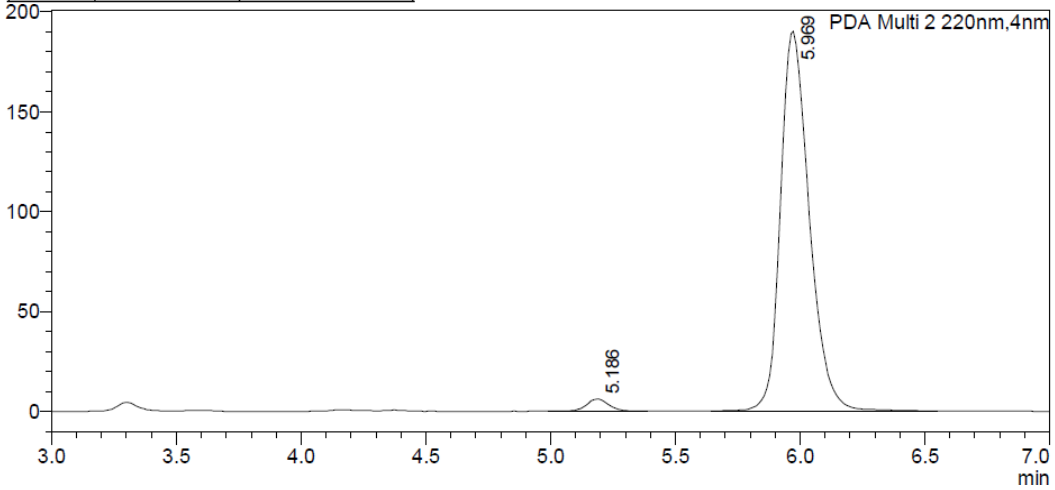
PDA Ch2 220nm

Peak#	Ret. Time	Area%
1	4.755	49.087
2	5.411	50.913
Total		100.000

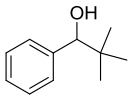


PDA Ch2 220nm

Peak#	Ret. Time	Area%
1	5.186	2.355
2	5.969	97.645
Total		100.000

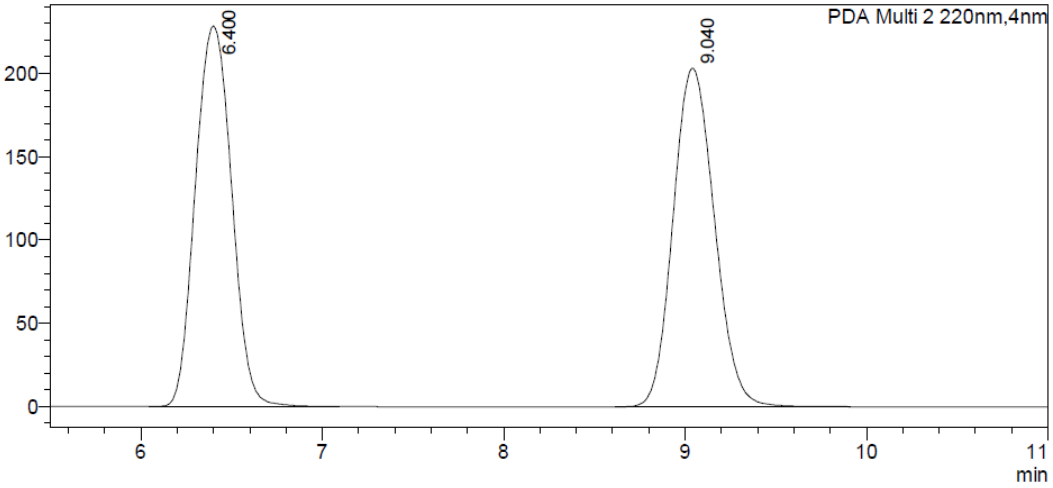


HPLC Data for 18: Chiralcel OD-H (95:5 hexane : IPA, flow rate 1.00 mLmin^{-1}, 220 nm, 30 °C) t_R (*S*): 6.6 min, t_R (*R*): 9.2 min, 18.907:81.093 er. *s*=111.4



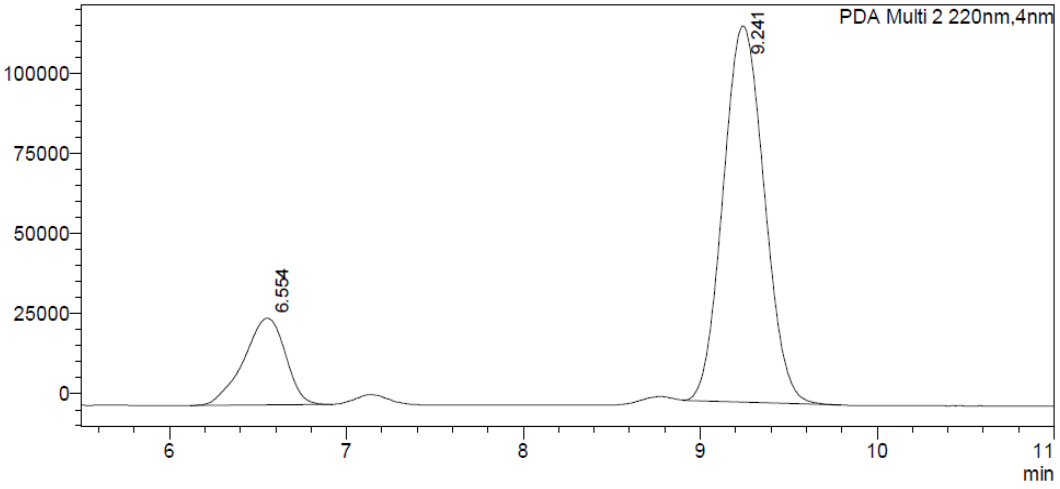
PDA Ch2 220nm

Peak#	Ret. Time	Area%
1	6.400	49.677
2	9.040	50.323
Total		100.000

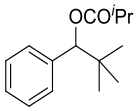


PDA Ch2 220nm

Peak#	Ret. Time	Area%
1	6.554	18.907
2	9.241	81.093
Total		100.000

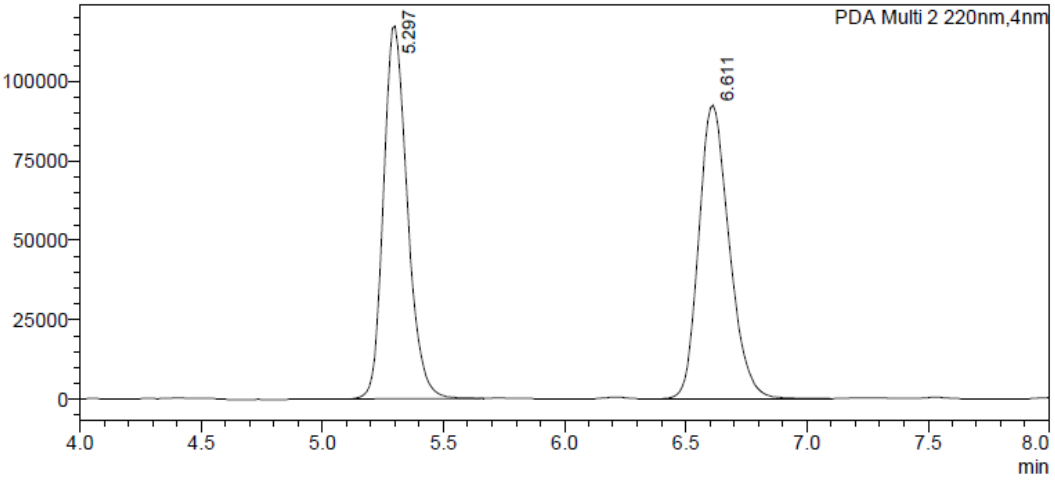


HPLC Data for S5: Chiralpak AD-H (99.8:0.2 hexane : IPA, flow rate 1.00 mLmin^{-1}, 220 nm, 30 °C) t_R (*R*): 5.3 min, t_R (*S*): 6.6 min, 1.673:98.327 er.



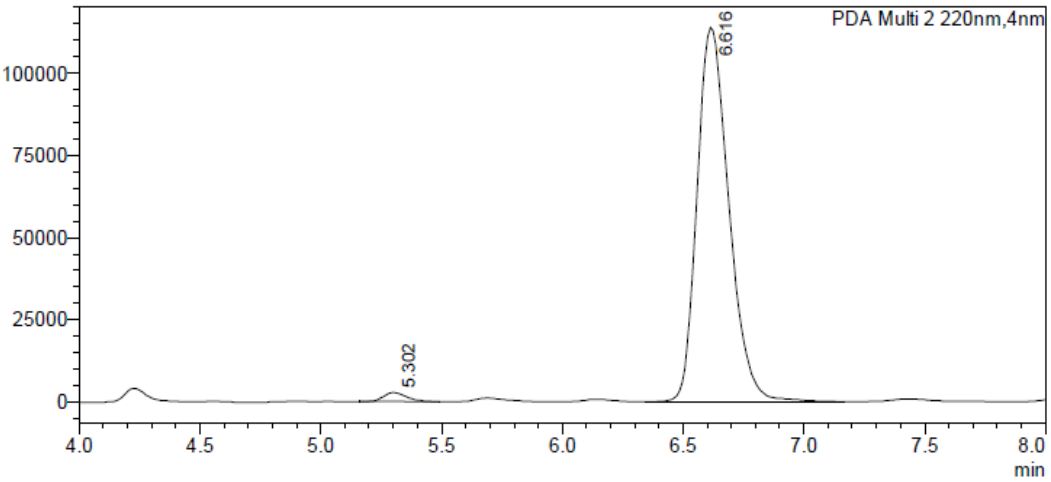
PDA Ch2 220nm

Peak#	Ret. Time	Area%
1	5.297	49.734
2	6.611	50.266
Total		100.000

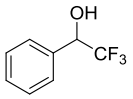


PDA Ch2 220nm

Peak#	Ret. Time	Area%
1	5.302	1.673
2	6.616	98.327
Total		100.000

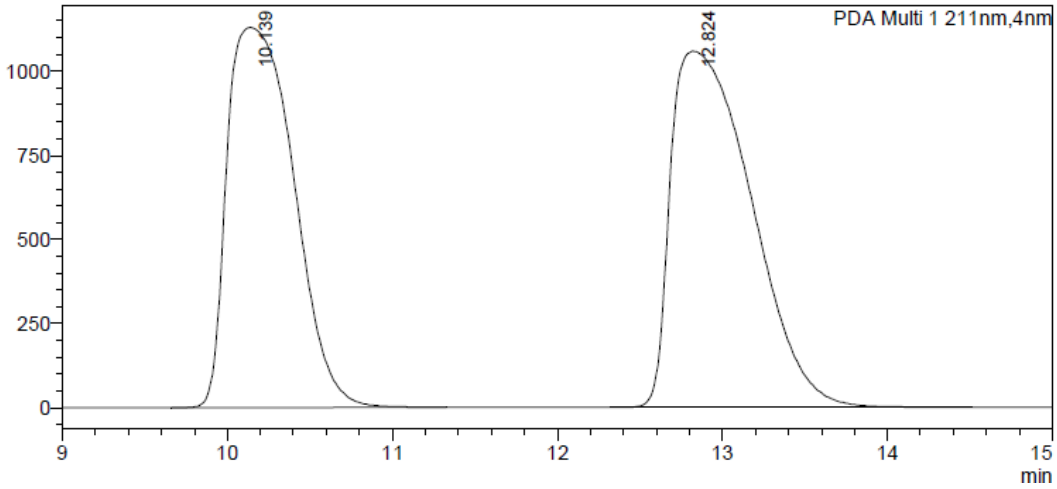


HPLC Data for 19: Chiralcel OJ-H (90:10 hexane : IPA, flow rate 1.00 mLmin^{-1}, 211 nm, 30 °C) t_R (*R*): 10.4 min, t_R (*S*): 13.1 min, 28.417:71.583 er. s=4.1



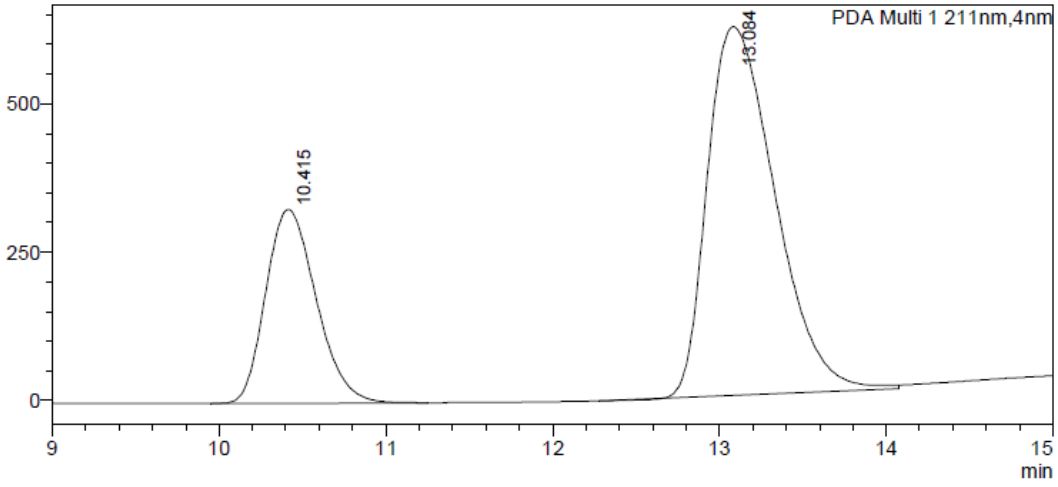
PDA Ch1 211nm

Peak#	Ret. Time	Area%
1	10.139	46.870
2	12.824	53.130
Total		100.000

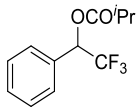


PDA Ch1 211nm

Peak#	Ret. Time	Area%
1	10.415	28.417
2	13.084	71.583
Total		100.000

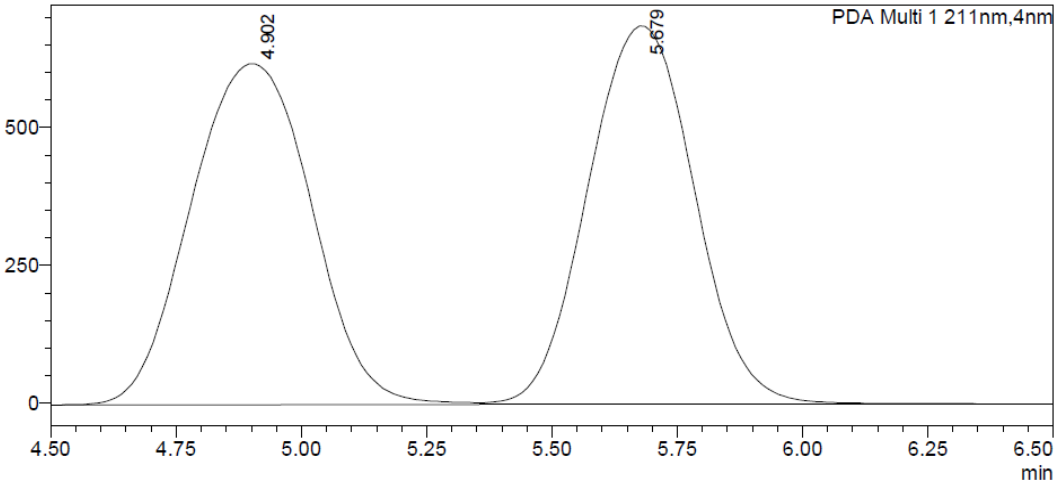


HPLC Data for S6: Chiralcel OJ-H (99:1 hexane : IPA, flow rate 1.00 mLmin^{-1}, 211 nm, 30 °C) t_R (*S*): 4.9 min, t_R (*R*): 5.7 min, 26.554:73.446 er.



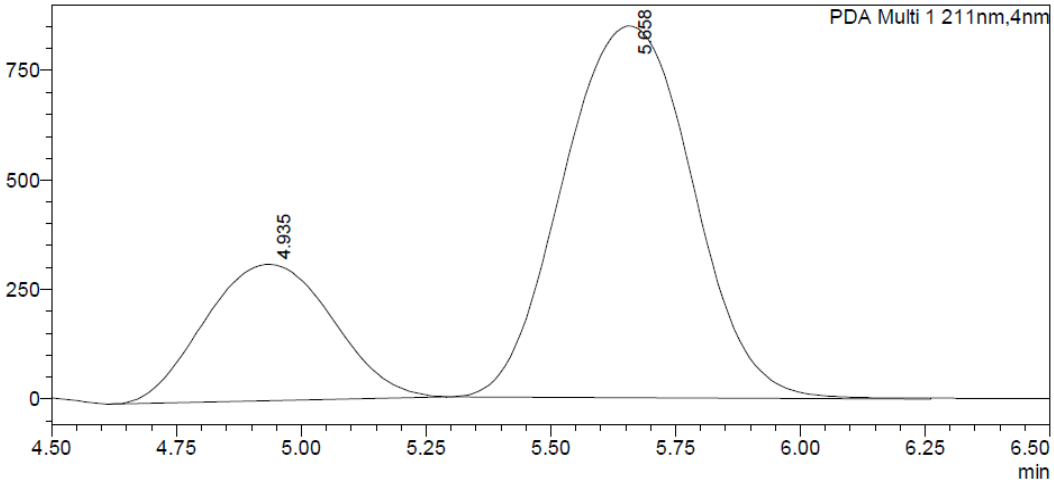
PDA Ch1 211nm

Peak#	Ret. Time	Area%
1	4.902	50.335
2	5.679	49.665
Total		100.000

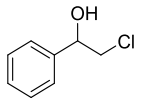


PDA Ch1 211nm

Peak#	Ret. Time	Area%
1	4.935	26.554
2	5.658	73.446
Total		100.000

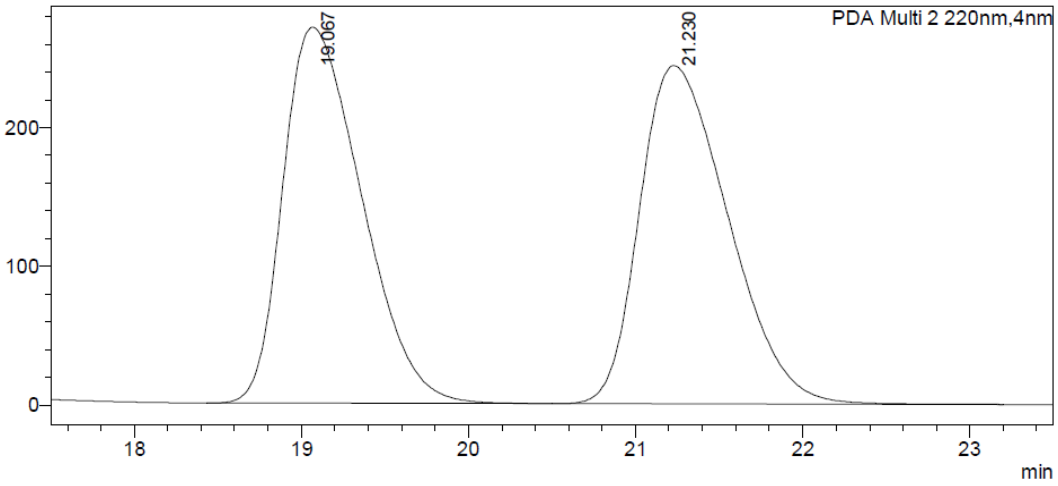


HPLC Data for 20: Chiralcel OD-H (98:2 hexane : IPA, flow rate 1.00 mLmin^{-1}, 220 nm, 30 °C) t_R (*S*): 19.1 min, t_R (*R*): 21.2 min, 92.875:7.125 er. s=31.4



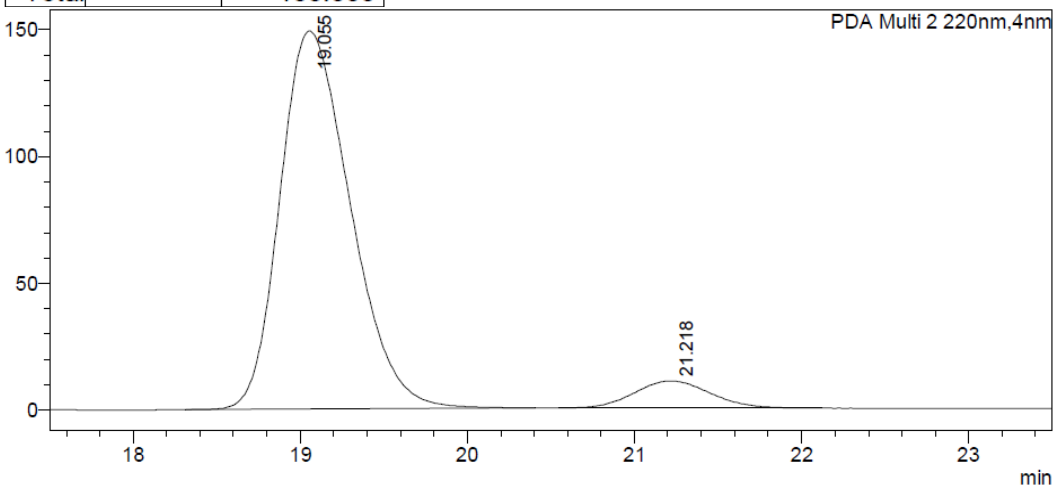
PDA Ch2 220nm

Peak#	Ret. Time	Area%
1	19.067	49.709
2	21.230	50.291
Total		100.000

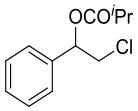


PDA Ch2 220nm

Peak#	Ret. Time	Area%
1	19.055	92.875
2	21.218	7.125
Total		100.000

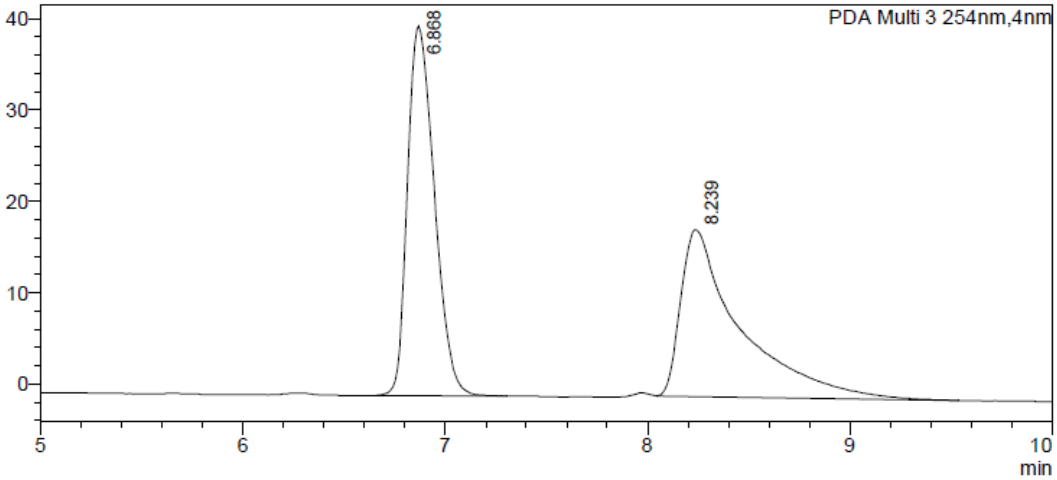


HPLC Data for S7: Chiralpak AD-H (99.8:0.2 hexane : IPA, flow rate 1.00 mLmin^{-1}, 254 nm, 30 °C) t_R (*S*): 7.2 min, t_R (*R*): 8.0 min, 8.020:91.980 er.



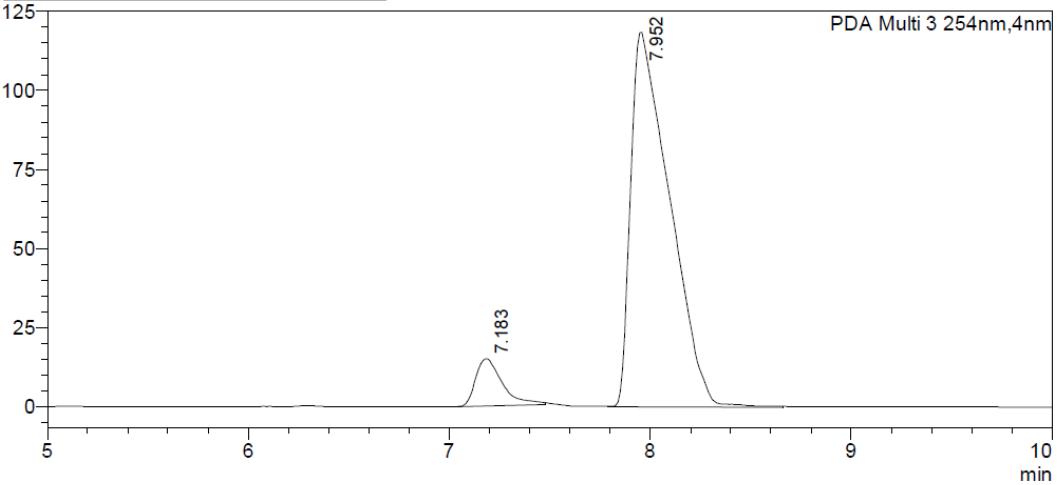
PDA Ch3 254nm

Peak#	Ret. Time	Area%
1	6.868	50.180
2	8.239	49.820
Total		100.000

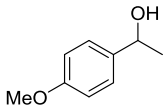


PDA Ch3 254nm

Peak#	Ret. Time	Area%
1	7.183	8.020
2	7.952	91.980
Total		100.000

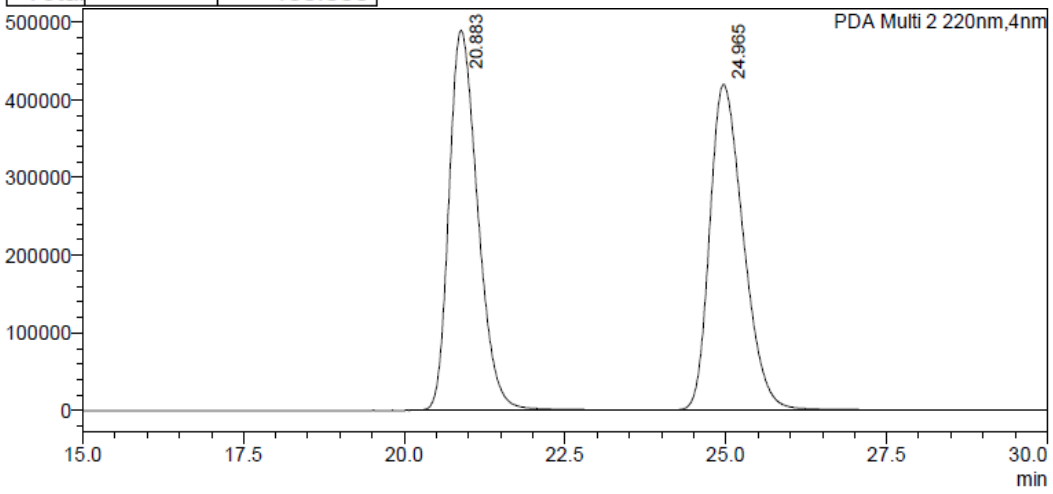


HPLC Data for 21: Chiralcel OD-H (98:2 hexane : IPA, flow rate 1.0 mLmin^{-1}, 220 nm, 30 °C) t_R (*R*): 20.8 min, t_R (S): 24.7 min, 78.653:21.347 er. *s*=31.1



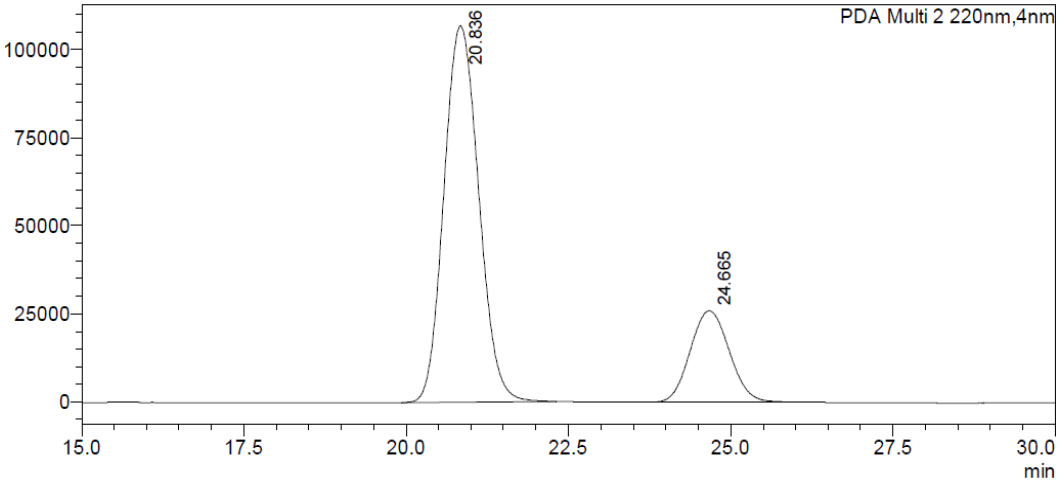
PDA Ch2 220nm

Peak#	Ret. Time	Area%
1	20.883	49.892
2	24.965	50.108
Total		100.000

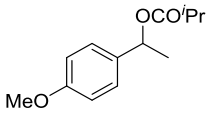


PDA Ch2 220nm

Peak#	Ret. Time	Area%
1	20.836	78.653
2	24.665	21.347
Total		100.000

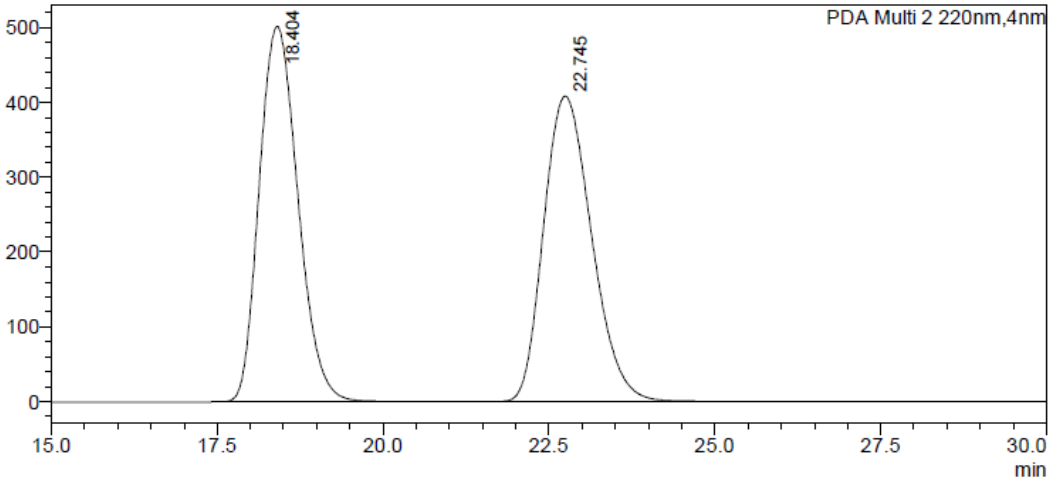


HPLC Data for S3: Chiralcel OJ-H (99.5:0.5 hexane : IPA, flow rate 0.5 mLmin^{-1}, 220 nm, 30 °C) t_R (*R*): 19.0 min, t_R (*S*): 23.4 min, 5.414:94.586 er.



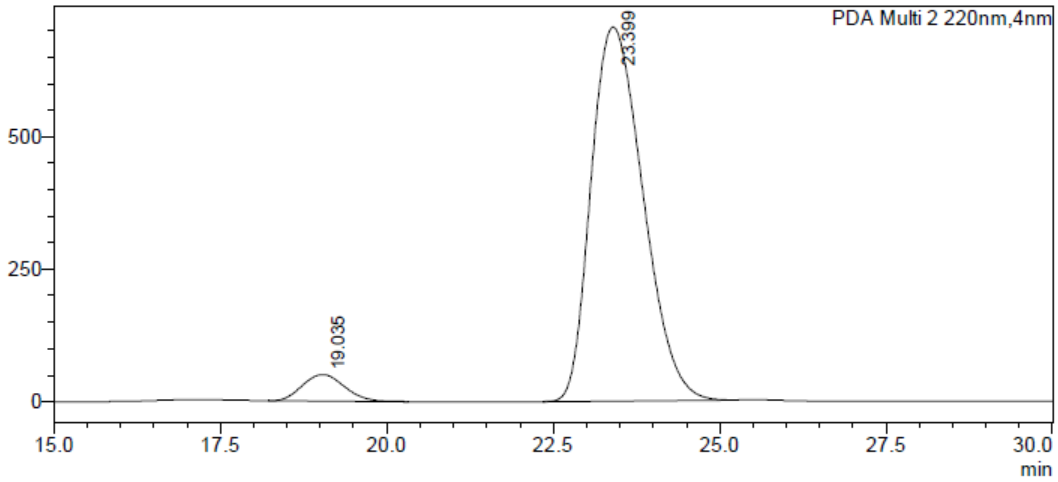
PDA Ch2 220nm

Peak#	Ret. Time	Area%
1	18.404	49.791
2	22.745	50.209
Total		100.000

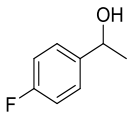


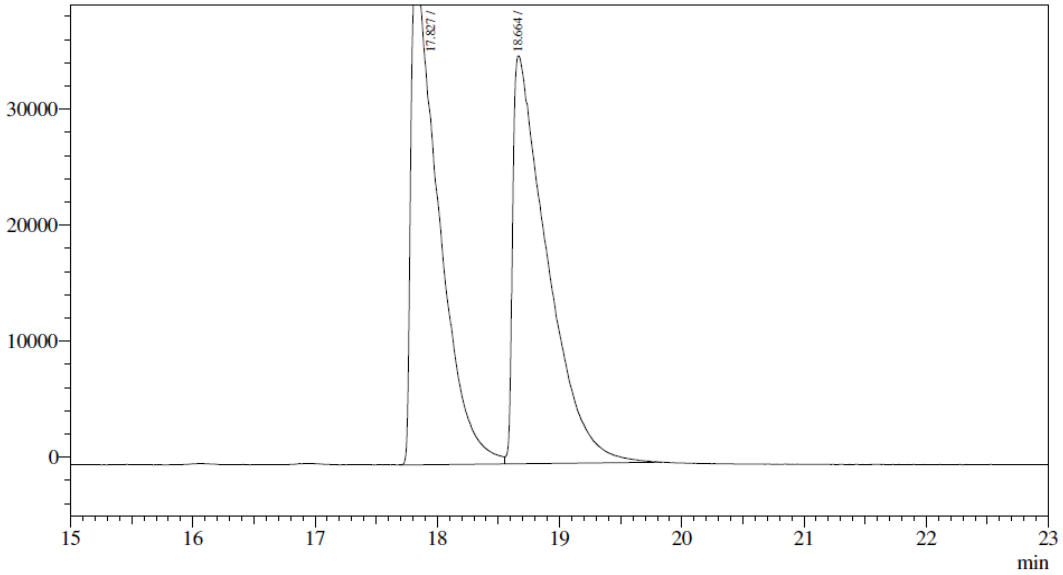
PDA Ch2 220nm

Peak#	Ret. Time	Area%
1	19.035	5.414
2	23.399	94.586
Total		100.000

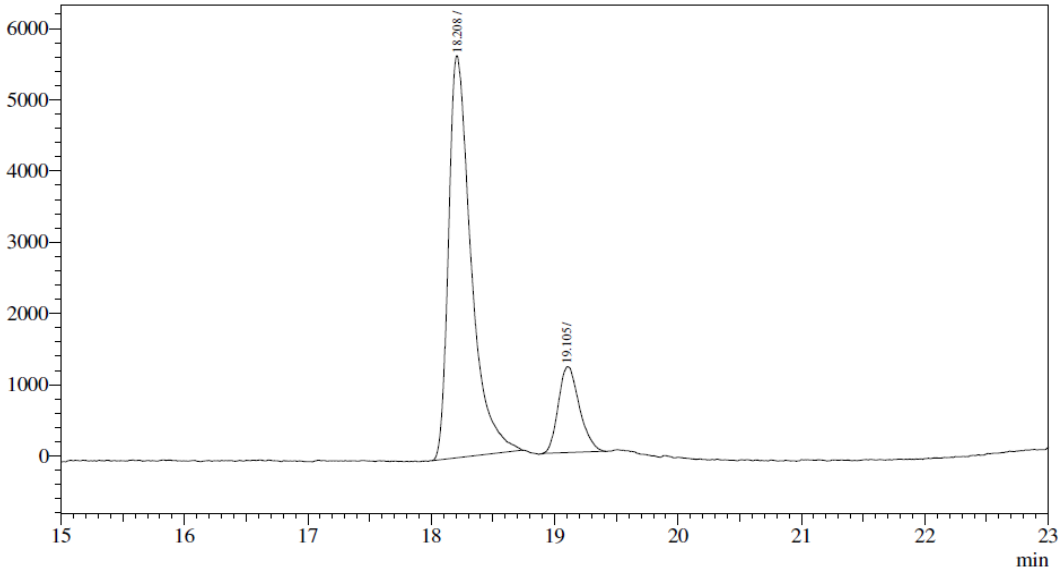


GC Data for 22: Cyclodex-B (length: 30 m, thickness: 0.250mm, film thickness: 0.25 um, carrier gas: He, linear velocity: 60 cm/sec, temperature: 100 °C (10 min), 100 – 110 °C (10 min), 110 °C (10 min), 110 – 120 °C (10 min)). t_R (*R*): 18.2 min, t_R (*S*): 19.1 min, 83.383:16.617 er. *s*=21.5



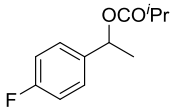


Peak#	Ret.Time	Area	Height	Conc.	Unit	Mark	ID#	Cmpd Name
1	17.827	676110	41958	49.631				
2	18.664	686164	35171	50.369		SV		
Total		1362274	77129					



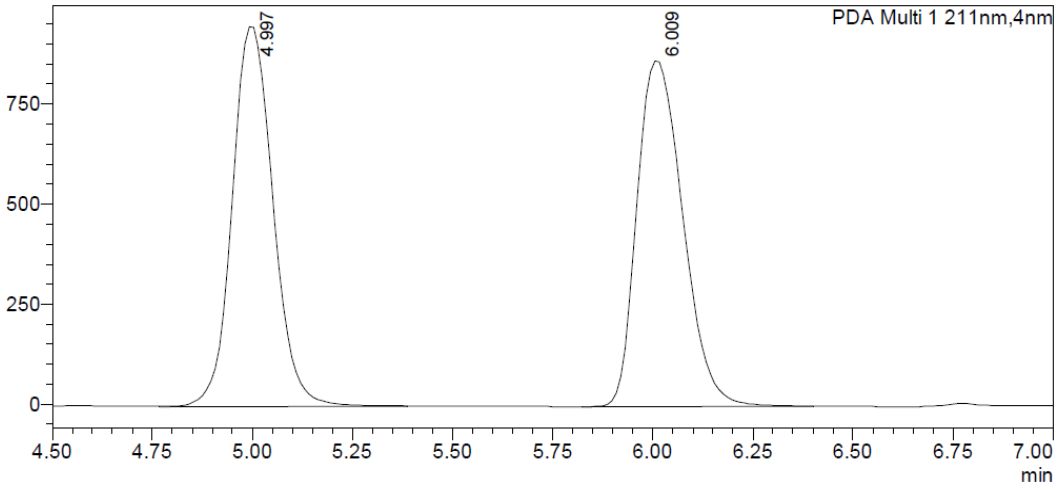
Peak#	Ret.Time	Area	Height	Conc.	Unit	Mark	ID#	Cmpd Name
1	18.208	71743	5649	83.383				
2	19.105	14298	1206	16.617				
Total		86041	6855					

HPLC Data for S9: Chiralpak AD-H (99.8:0.2 hexane : IPA, flow rate 1.00 mLmin^{-1}, 211 nm, 30 °C) t_R (*R*): 5.2 min, t_R (*S*): 6.1 min, 8.478:91.522 er.



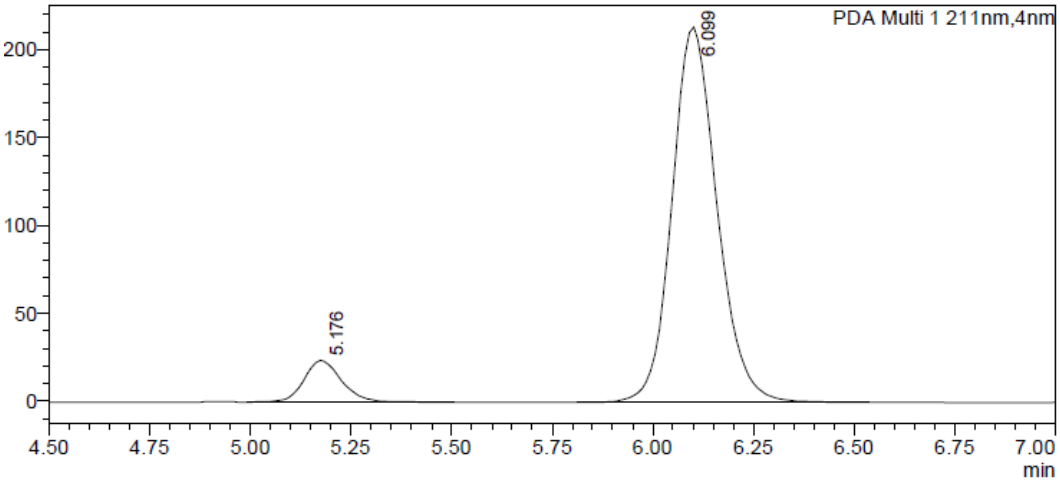
PDA Ch1 211nm

Peak#	Ret. Time	Area%
1	4.997	49.447
2	6.009	50.553
Total		100.000

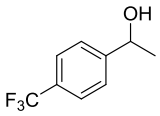


PDA Ch1 211nm

Peak#	Ret. Time	Area%
1	5.176	8.478
2	6.099	91.522
Total		100.000

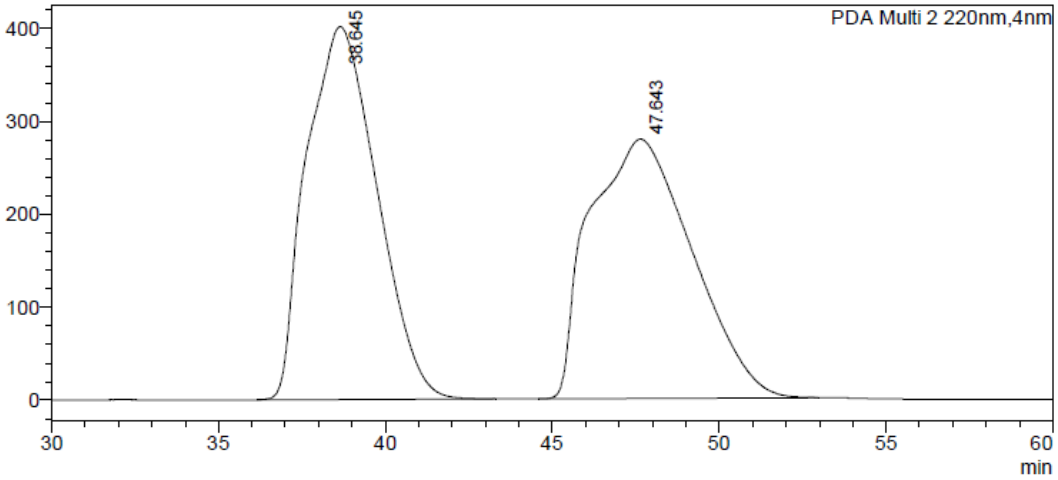


HPLC Data for 23: Chiralcel OJ-H (99.8:0.2 hexane : IPA, flow rate 1.00 mLmin^{-1}, 220 nm, 30 °C) t_R (*R*): 39.6 min, tR (*S*): 49.4 min, 90.781:9.219 er. *s*=15.4



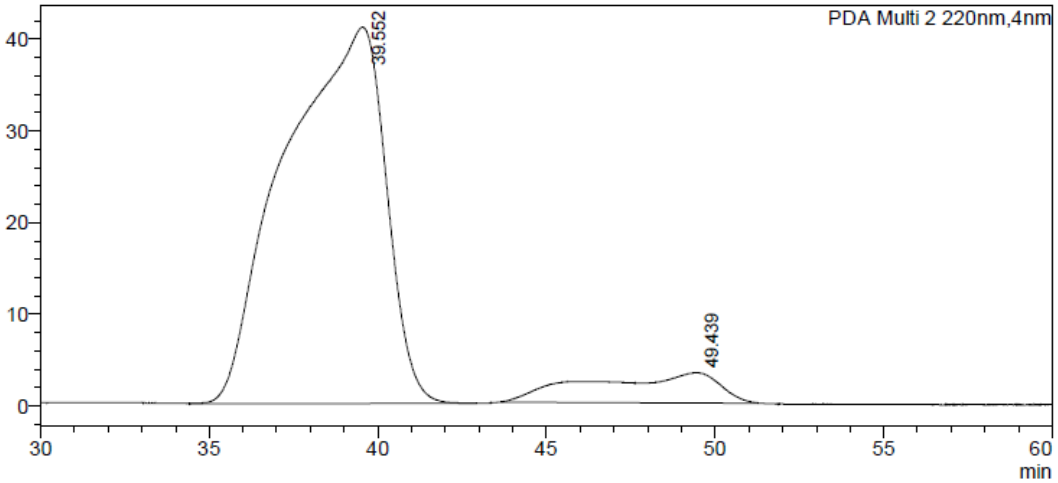
PDA Ch2 220nm

Peak#	Ret. Time	Area%
1	38.645	49.787
2	47.643	50.213
Total		100.000

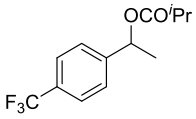


PDA Ch2 220nm

Peak#	Ret. Time	Area%
1	39.552	90.781
2	49.439	9.219
Total		100.000

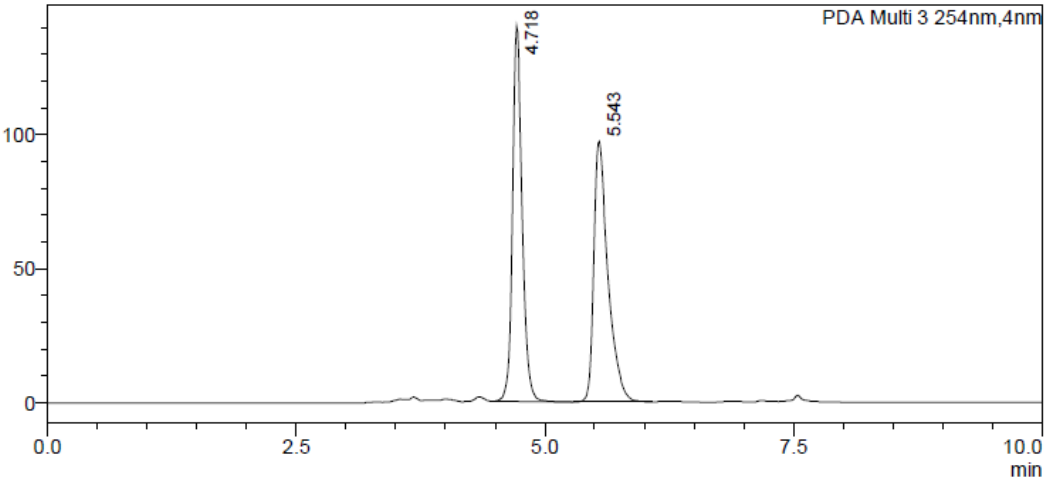


HPLC Data for S10: Chiralpak AD-H (99.8:0.2 hexane : IPA, flow rate 1.00 mLmin^{-1}, 254 nm, 30 °C) t_R (*R*): 4.7 min, t_R (*S*): 6.2 min, 13.844:86.156 er.



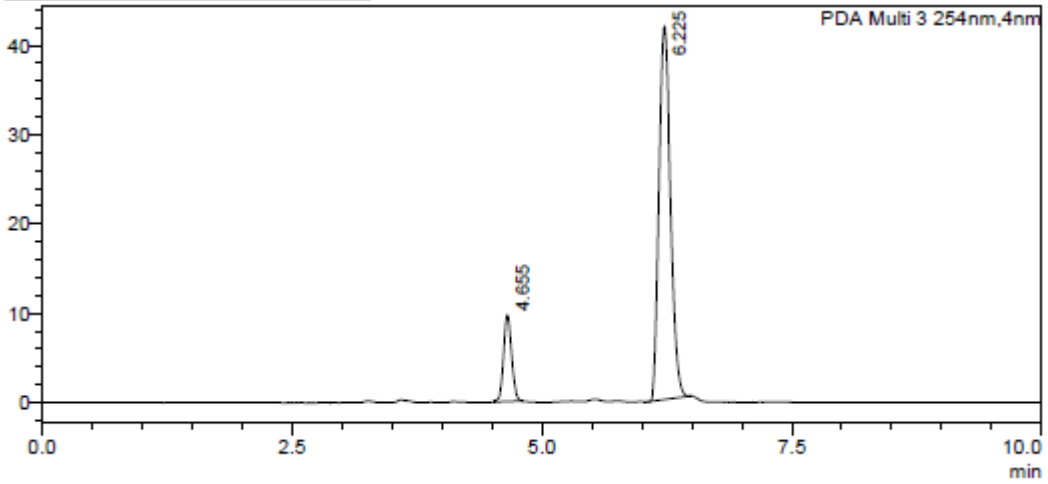
PDA Ch3 254nm

Peak#	Ret. Time	Area%
1	4.718	49.820
2	5.543	50.180
Total		100.000

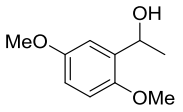


PDA Ch3 254nm

Peak#	Ret. Time	Area%
1	4.655	13.844
2	6.225	86.156
Total		100.000

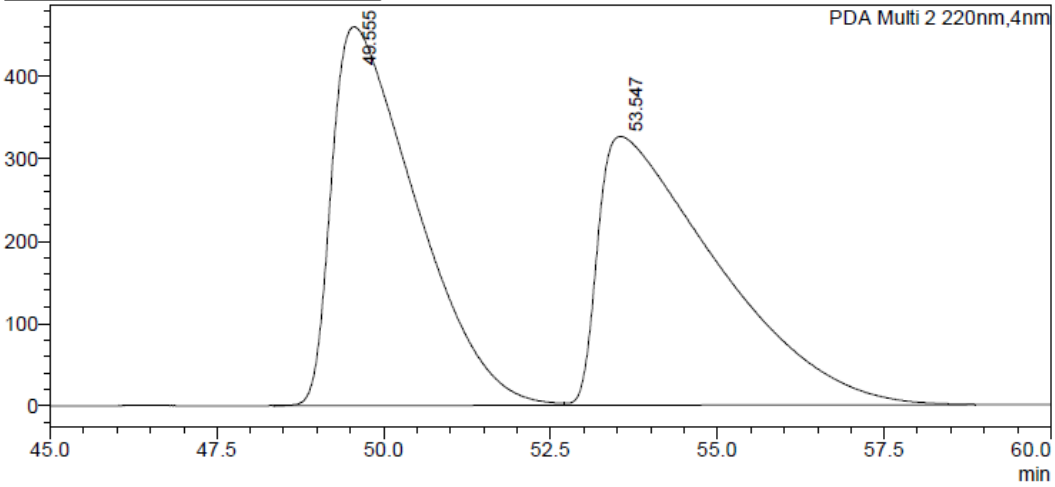


HPLC Data for 24: Chiralcel OD-H (99:1 hexane : IPA, flow rate 0.8 mLmin^{-1}, 220 nm, 30 °C) t_R (*S*): 50.0 min, t_R (*R*): 53.2 min, 21.699:78.301 er. *s*=14.1



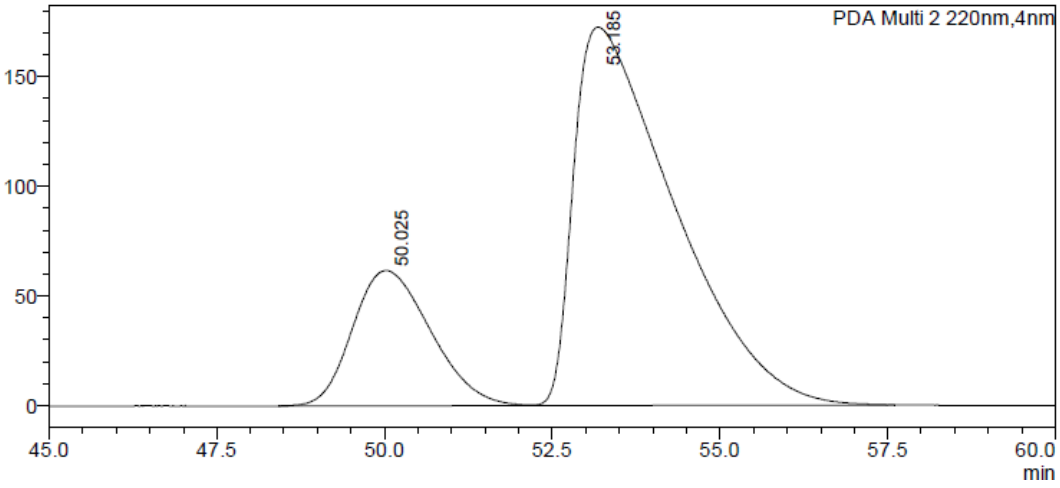
PDA Ch2 220nm

Peak#	Ret. Time	Area%
1	49.555	49.909
2	53.547	50.091
Total		100.000



PDA Ch2 220nm

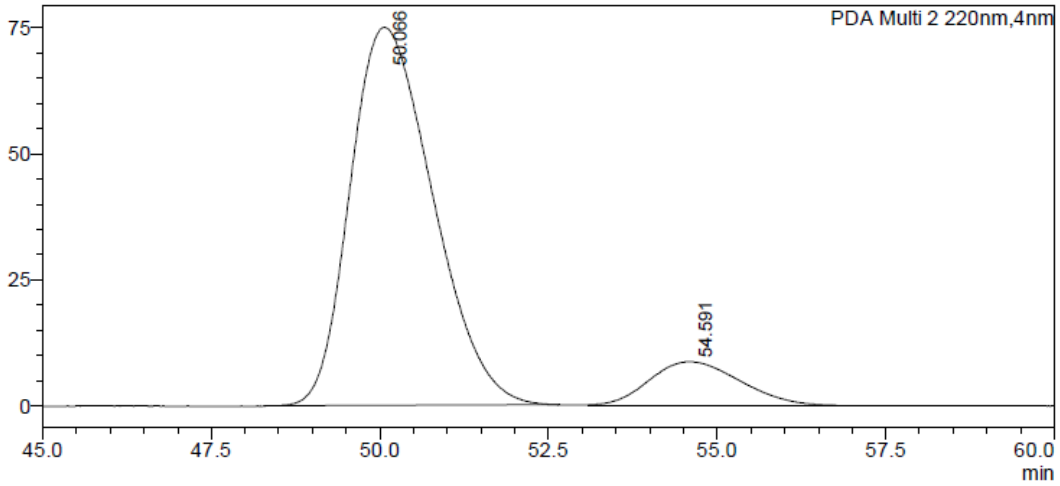
Peak#	Ret. Time	Area%
1	50.025	21.699
2	53.185	78.301
Total		100.000



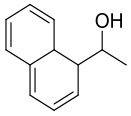
HPLC Data for S11 (following hydrolysis to give alcohol 24): Chiralcel OD-H (99:1 hexane : IPA, flow rate 0.8 mLmin^{-1}, 220 nm, 30 °C) t_R (*S*): 50.1 min, t_R (*R*): 54.6 min, 88.919:11.081 er.

PDA Ch2 220nm

Peak#	Ret. Time	Area%
1	50.066	88.919
2	54.591	11.081
Total		100.000

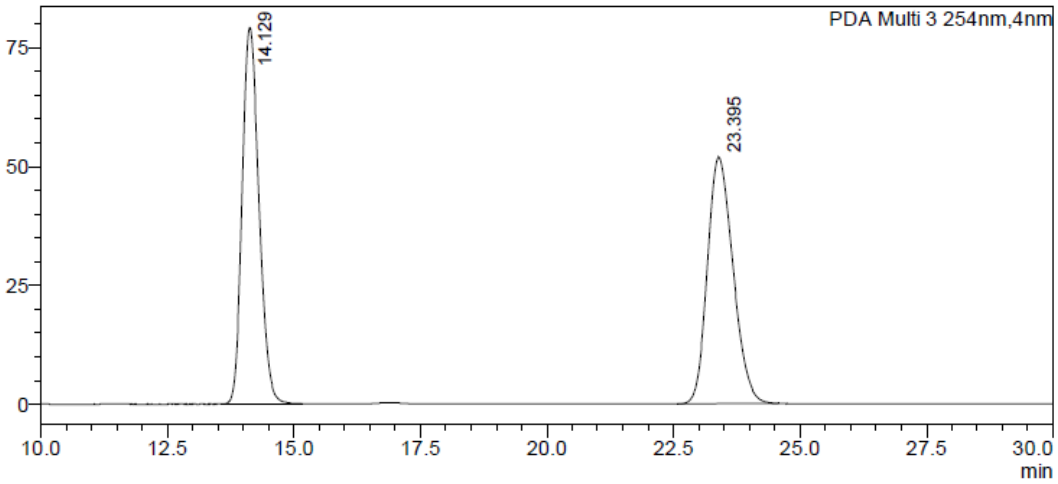


HPLC Data for 25: Chiralcel OD-H (95:5 hexane : IPA, flow rate 1.0 mLmin^{-1}, 254 nm, 30 °C) t_R (*S*): 14.3 min, t_R (*R*): 23.5 min, 10.166:89.834 er. *s*=40.5



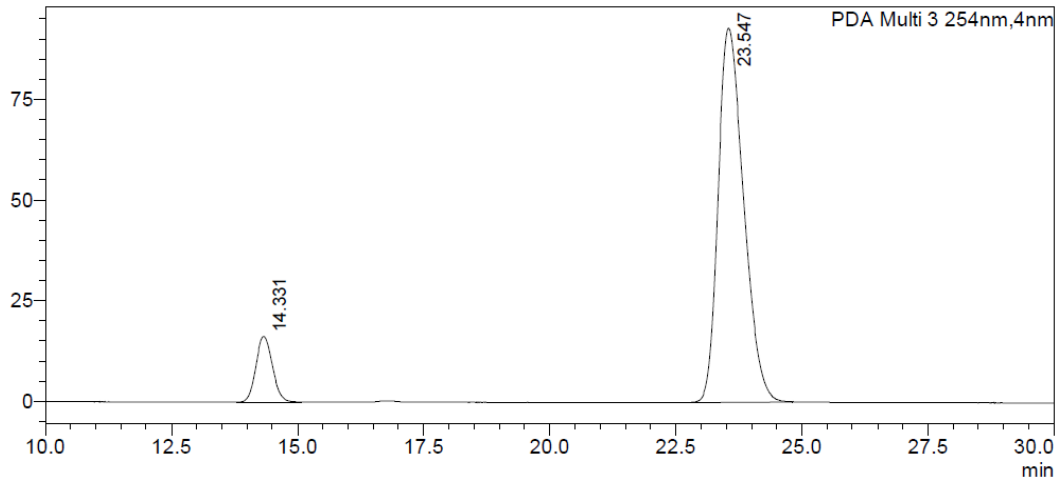
PDA Ch3 254nm

Peak#	Ret. Time	Area%
1	14.129	49.965
2	23.395	50.035
Total		100.000

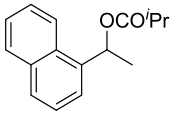


PDA Ch3 254nm

Peak#	Ret. Time	Area%
1	14.331	10.166
2	23.547	89.834
Total		100.000

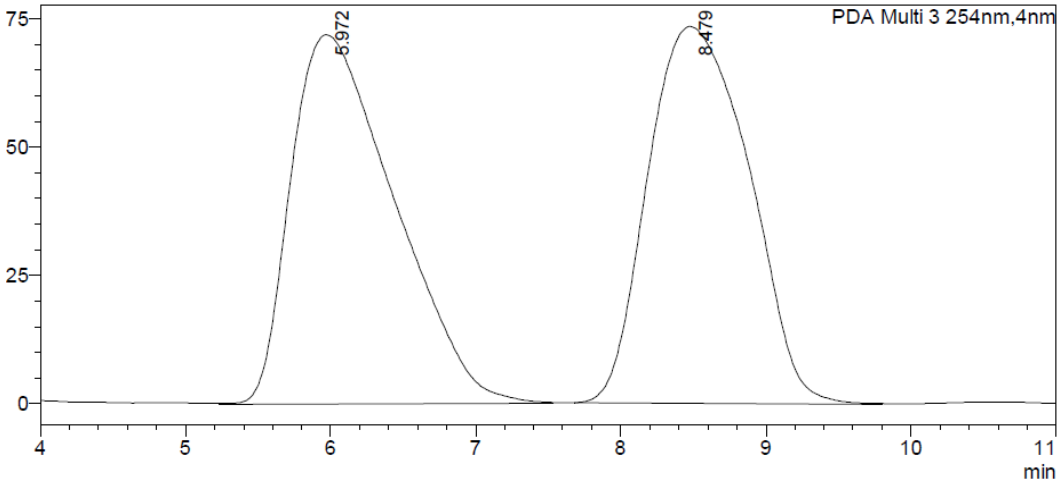


HPLC Data for S12: Chiralcel OD-H (99.5:0.5 hexane : IPA, flow rate 1.0 mLmin^{-1}, 254 nm, 30 °C) t_R (*S*): 6.0 min, t_R (*R*): 8.5 min, 5.667:94.333 er



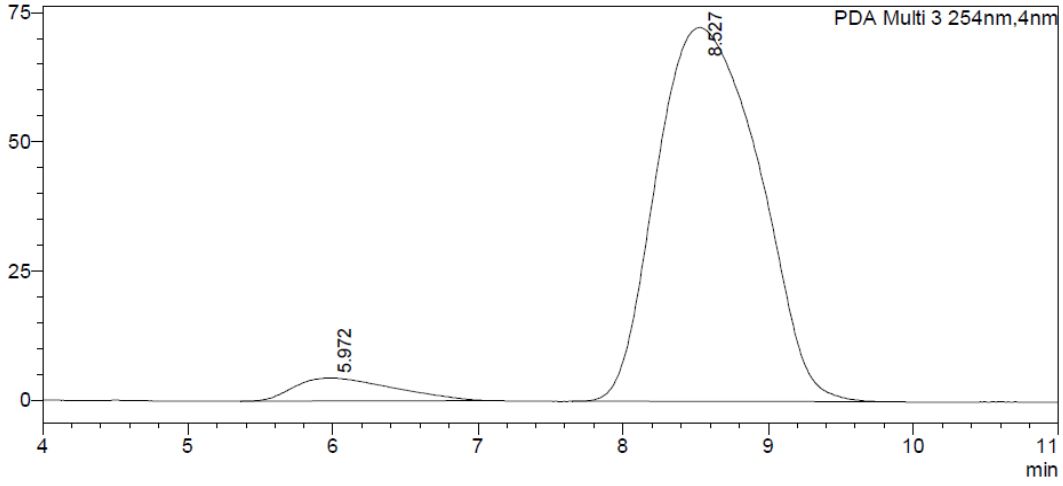
PDA Ch3 254nm

Peak#	Ret. Time	Area%
1	5.972	50.092
2	8.479	49.908
Total		100.000

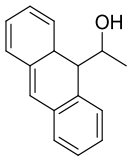


PDA Ch3 254nm

Peak#	Ret. Time	Area%
1	5.972	5.667
2	8.527	94.333
Total		100.000

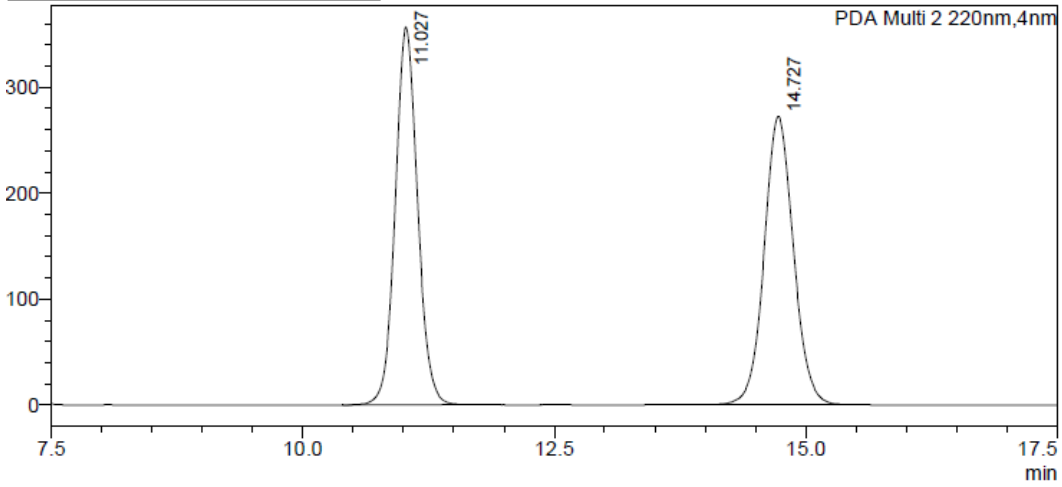


HPLC Data for 26: Chiralpak AD-H (90:10 hexane : IPA, flow rate 1.0 mLmin^{-1}, 220 nm, 30 °C) t_R (*R*): 11.3 min, t_R (*S*): 14.9 min, 87.767:12.233 er. *s*=76.7



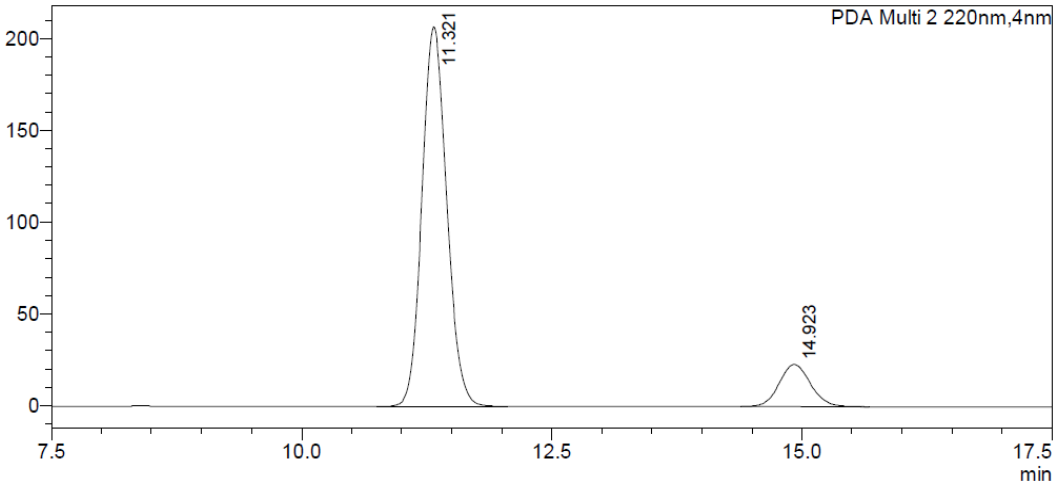
PDA Ch2 220nm

Peak#	Ret. Time	Area%
1	11.027	49.965
2	14.727	50.035
Total		100.000

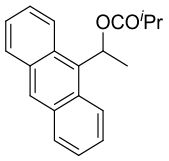


PDA Ch2 220nm

Peak#	Ret. Time	Area%
1	11.321	87.767
2	14.923	12.233
Total		100.000

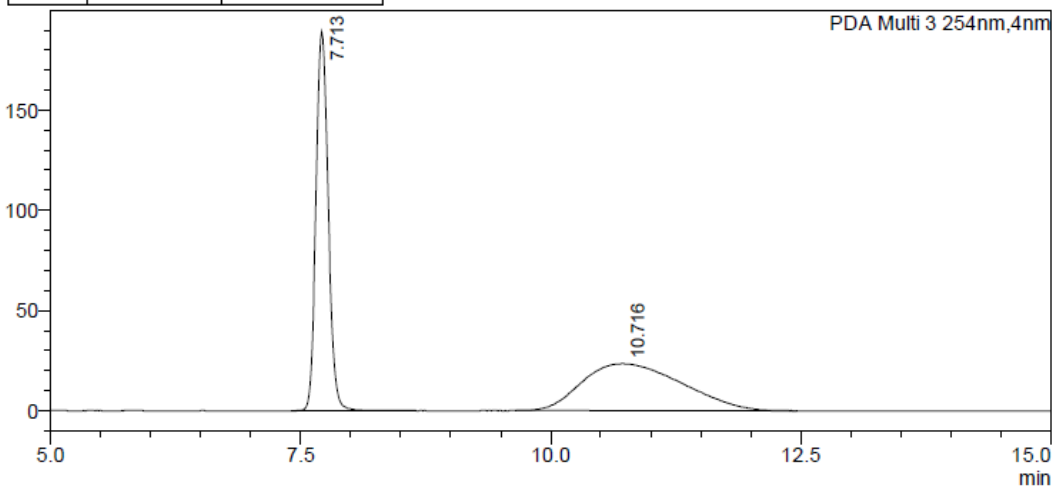


HPLC Data for S13: Chiralpak AD-H (99.9:0.1 hexane : IPA, flow rate 1.0 mLmin^{-1}, 254 nm, 30 °C) t_R (*R*): 6.6 min, t_R (*S*): 10.1 min, 2.887:97.113 er.



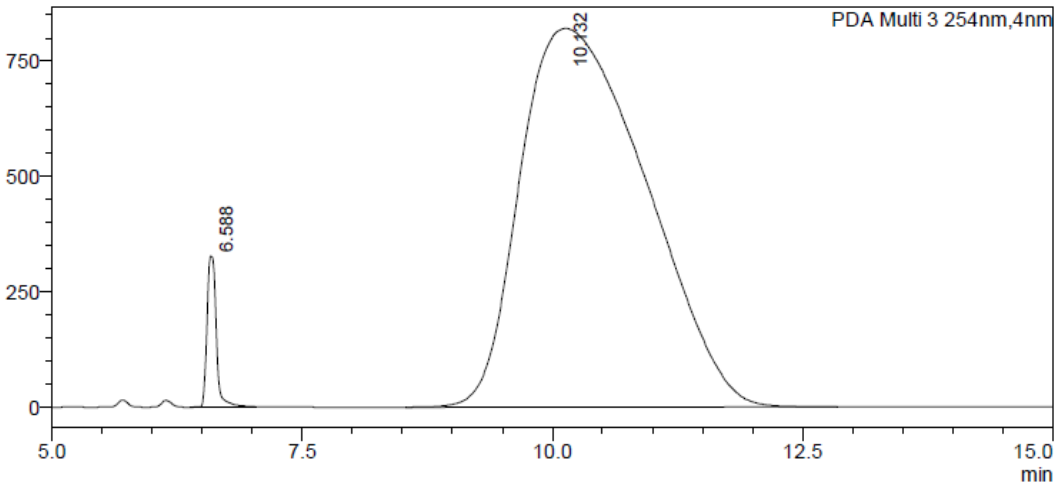
PDA Ch3 254nm

Peak#	Ret. Time	Area%
1	7.713	49.843
2	10.716	50.157
Total		100.000

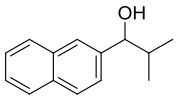


PDA Ch3 254nm

Peak#	Ret. Time	Area%
1	6.588	2.887
2	10.132	97.113
Total		100.000

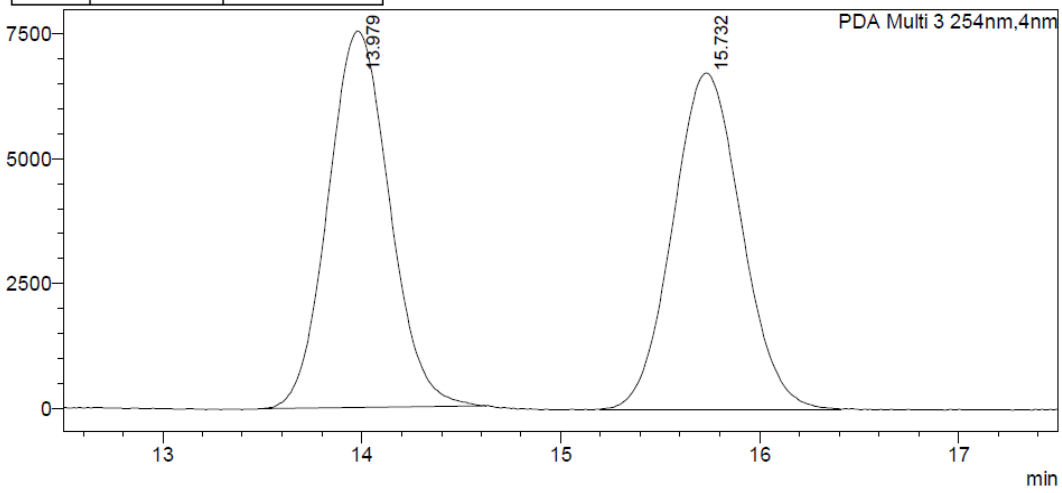


HPLC Data for 27: Chiralcel OD-H (95:5 hexane : IPA, flow rate 1.0 mLmin^{-1}, 254 nm, 30 °C) t_R (*S*): 13.9 min, t_R (*R*): 15.6 min, 8.741:91.25 er. *s*=622.1



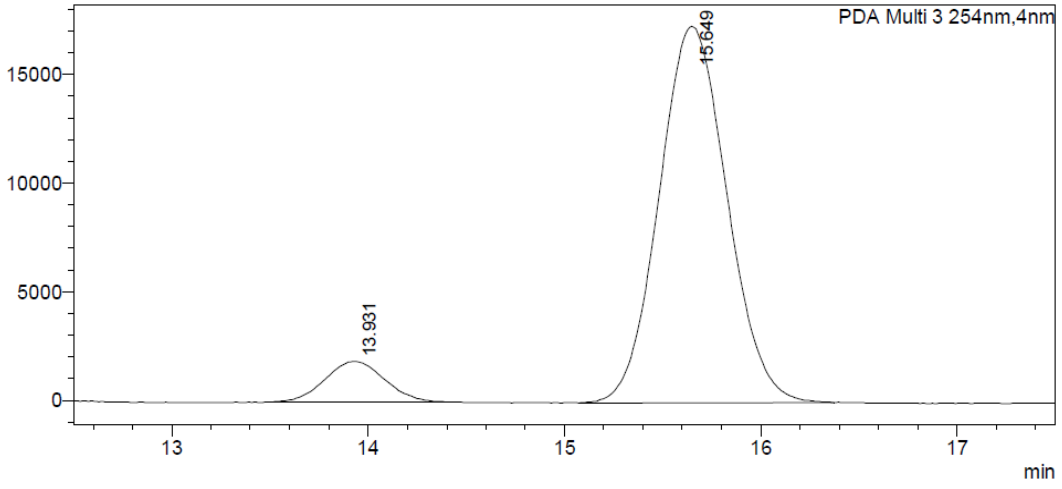
PDA Ch3 254nm

Peak#	Ret. Time	Area%
1	13.979	50.077
2	15.732	49.923
Total		100.000

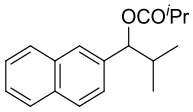


PDA Ch3 254nm

Peak#	Ret. Time	Area%
1	13.931	8.741
2	15.649	91.259
Total		100.000

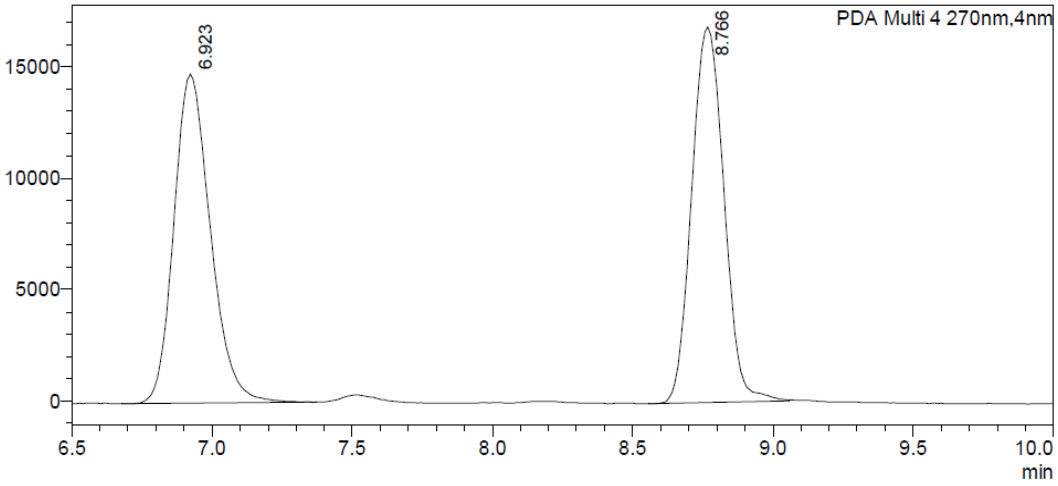


HPLC Data for 14: Chiralpak AD-H (99.8:0.2 hexane : IPA, flow rate 1.0 mLmin^{-1}, 270 nm, 30 °C) t_R (*R*): 7.0 min, t_R (*S*): 8.9 min, 0.415:99.585 er.



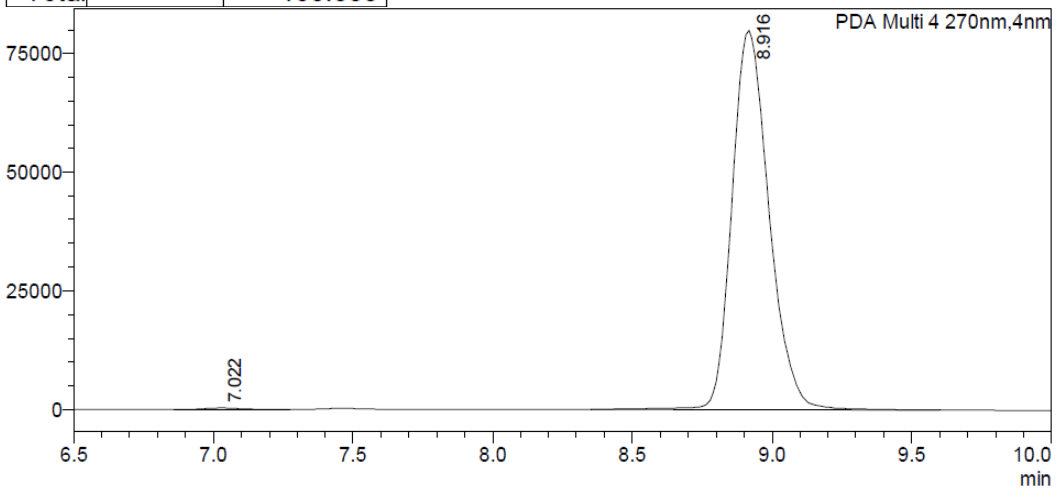
PDA Ch4 270nm

Peak#	Ret. Time	Area%
1	6.923	49.833
2	8.766	50.167
Total		100.000

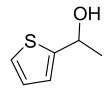


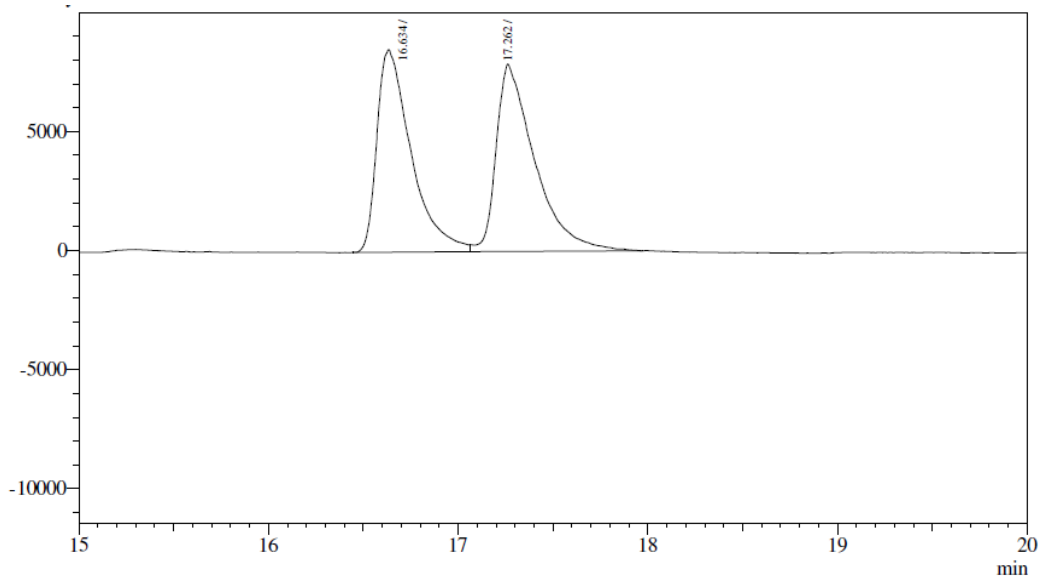
PDA Ch4 270nm

Peak#	Ret. Time	Area%
1	7.022	0.415
2	8.916	99.585
Total		100.000

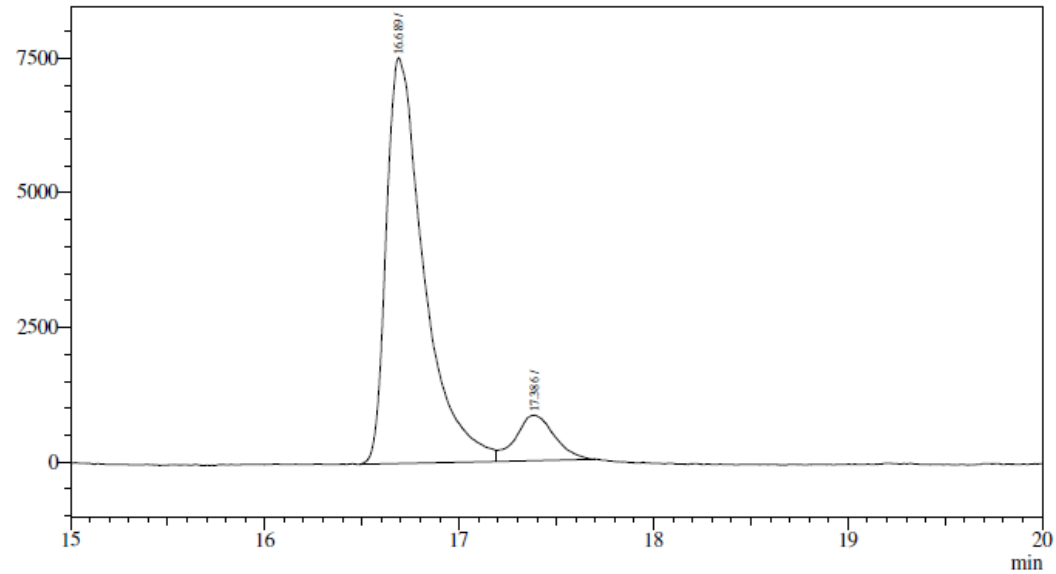


GC Data for 28: Cyclodex-B (length: 30 m, thickness: 0.250mm, film thickness: 0.25 um, carrier gas: He, linear velocity: 60 cm/sec, temperature: 100 °C (10 min), 100 – 110 °C (10 min), 110 °C (10 min), 110 – 120 °C (10 min)). t_R (*R*): 16.7 min, t_R (*S*): 17.4 min, 89.649:10.351 er. *s*=24.7



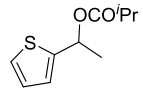


Peak#	Ret.Time	Area	Height	Conc.	Unit	Mark	ID#	Cmpd Name
1	16.634	106558	8504	48.983				
2	17.262	110984	7860	51.017		SV		
Total		217542	16364					



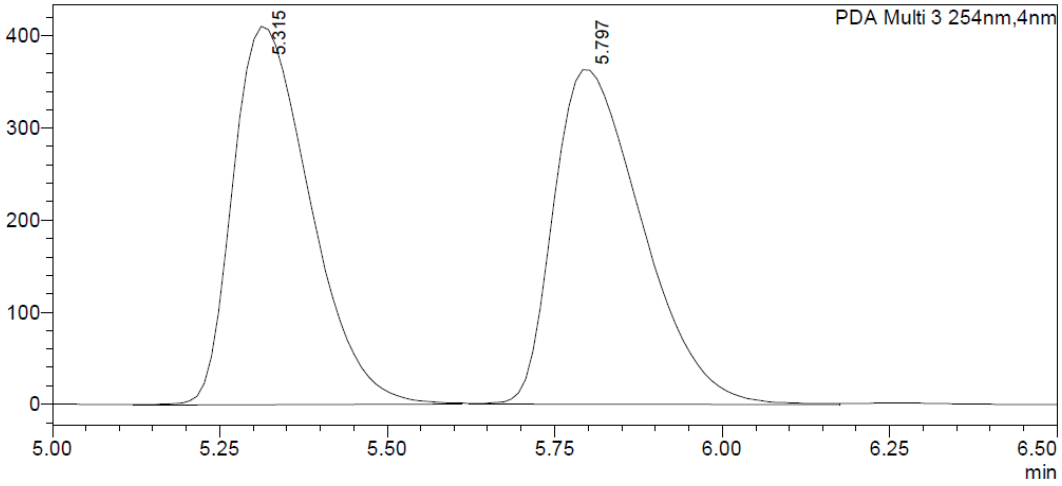
Peak#	Ret.Time	Area	Height	Conc.	Unit	Mark	ID#	Cmpd Name
1	16.689	101313	7528	89.649				
2	17.386	11698	843	10.351		V		
Total		113011	8371					

HPLC Data for S15: Chiralpak AD-H (99.8:0.2 hexane : IPA, flow rate 1.0 mLmin^{-1}, 254 nm, 30 °C) t_R (*R*): 5.3 min, t_R (*S*): 5.8 min, 8.865:91.135 er.



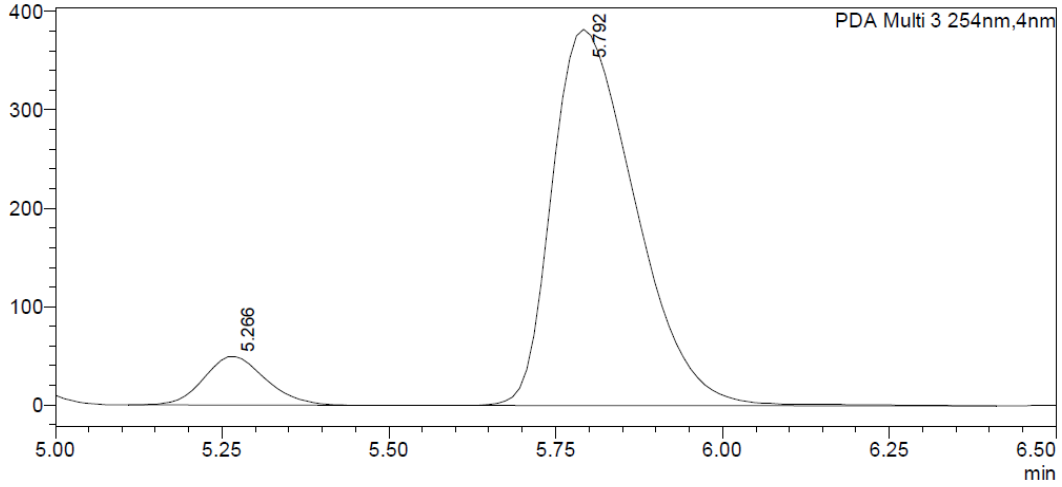
PDA Ch3 254nm

Peak#	Ret. Time	Area%
1	5.315	49.657
2	5.797	50.343
Total		100.000

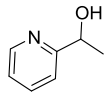


PDA Ch3 254nm

Peak#	Ret. Time	Area%
1	5.266	8.865
2	5.792	91.135
Total		100.000

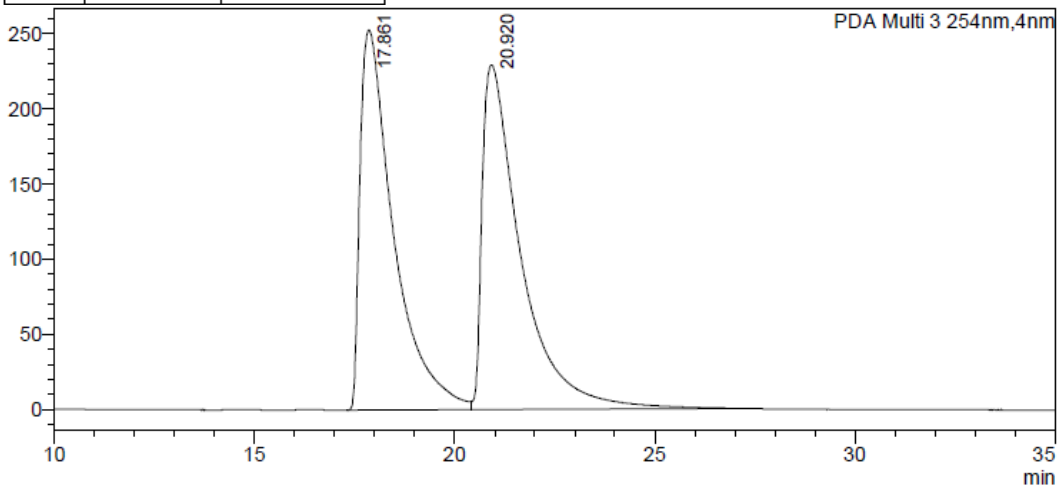


HPLC Data for 29: Chiralcel OD-H (99:1 hexane : IPA, flow rate 1.0 mLmin^{-1}, 254 nm, 30 °C) t_R (*R*): 18.2 min, t_R (*S*): 21.4 min, 55.705:44.295 er. *s*=1.9



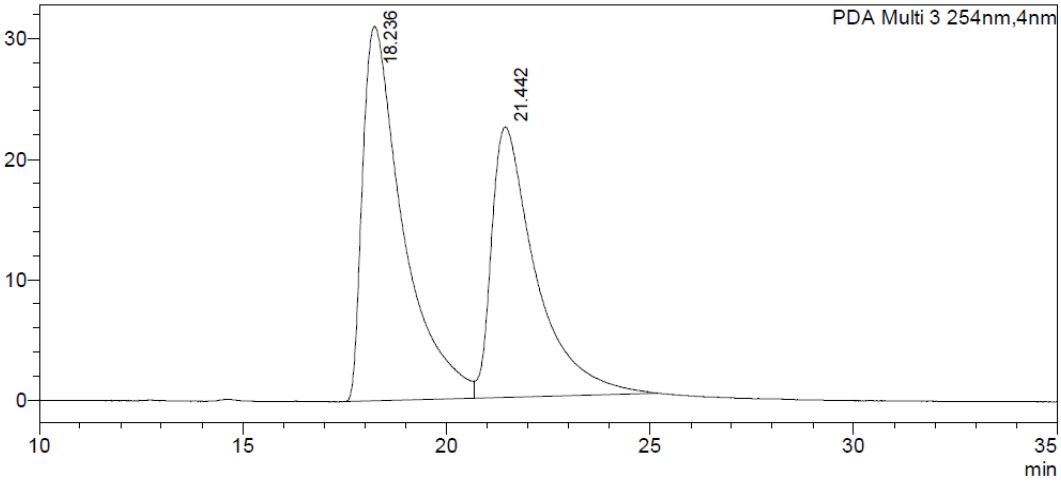
PDA Ch3 254nm

Peak#	Ret. Time	Area%
1	17.861	48.900
2	20.920	51.100
Total		100.000

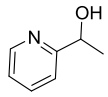


PDA Ch3 254nm

Peak#	Ret. Time	Area%
1	18.236	55.705
2	21.442	44.295
Total		100.000

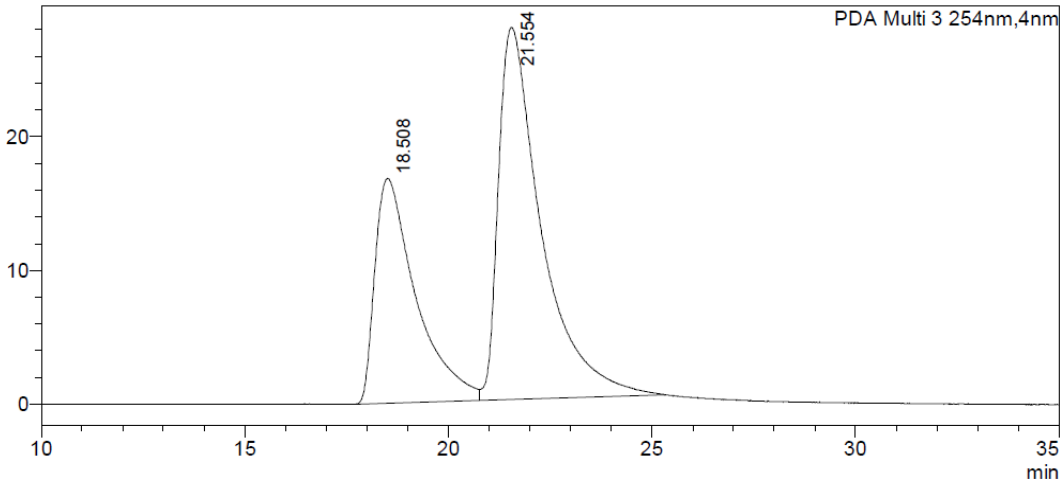


HPLC Data for S16 (following hydrolysis to alcohol 29): Chiralcel OD-H (99:1 hexane : IPA, flow rate 1.0 mLmin^{-1}, 254 nm, 30 °C) t_R (*R*): 18.5 min, t_R (*S*): 21.6 min, 36.632:63.368 er.

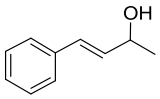


PDA Ch3 254nm

Peak#	Ret. Time	Area%
1	18.508	36.632
2	21.554	63.368
Total		100.000

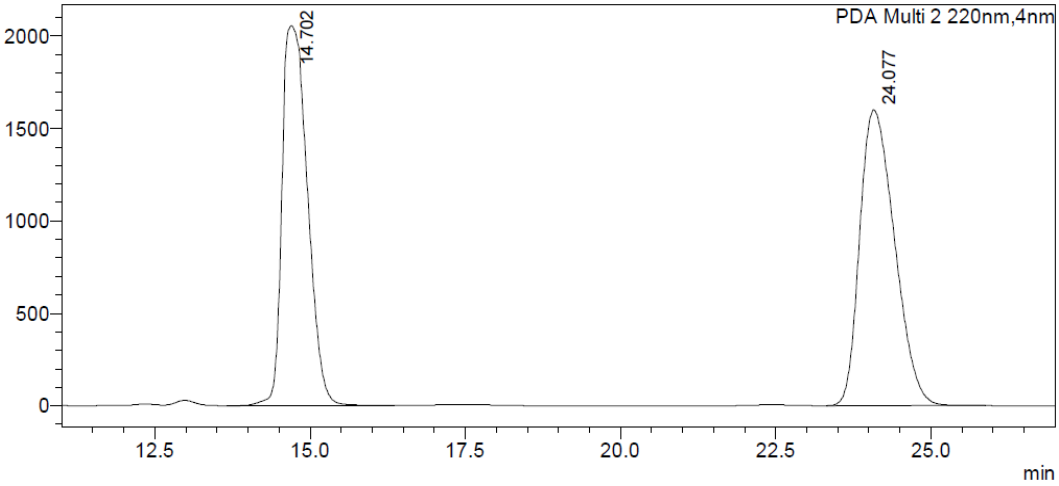


HPLC Data for 30: Chiralcel OD-H (95:5 hexane : IPA, flow rate 1.0 mLmin^{-1}, 220 nm, 30 °C) t_R (*R*): 14.7 min, t_R (*S*): 24.1 min, 84.504:15.496 er. *s*=17.1



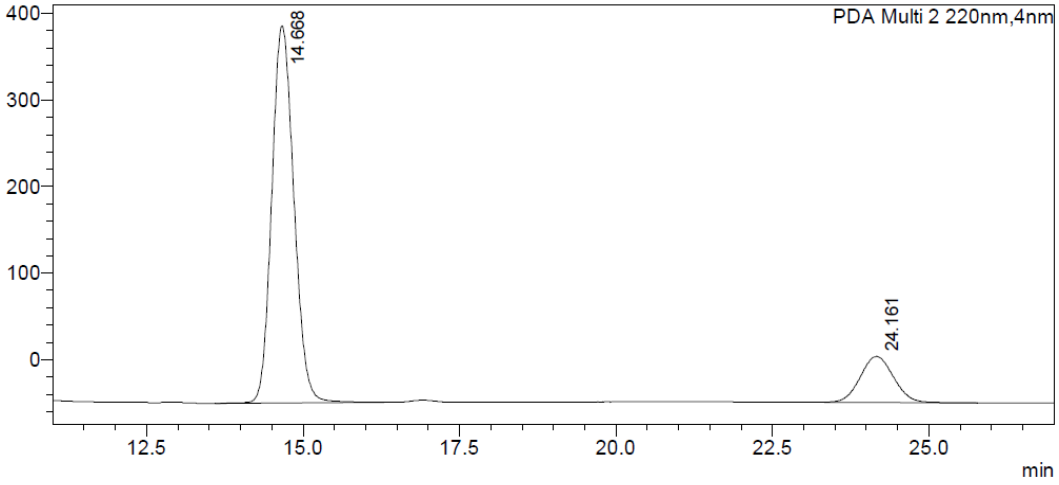
PDA Ch2 220nm

Peak#	Ret. Time	Area%
1	14.702	48.459
2	24.077	51.541
Total		100.000

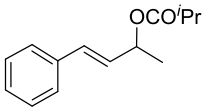


PDA Ch2 220nm

Peak#	Ret. Time	Area%
1	14.668	84.504
2	24.161	15.496
Total		100.000

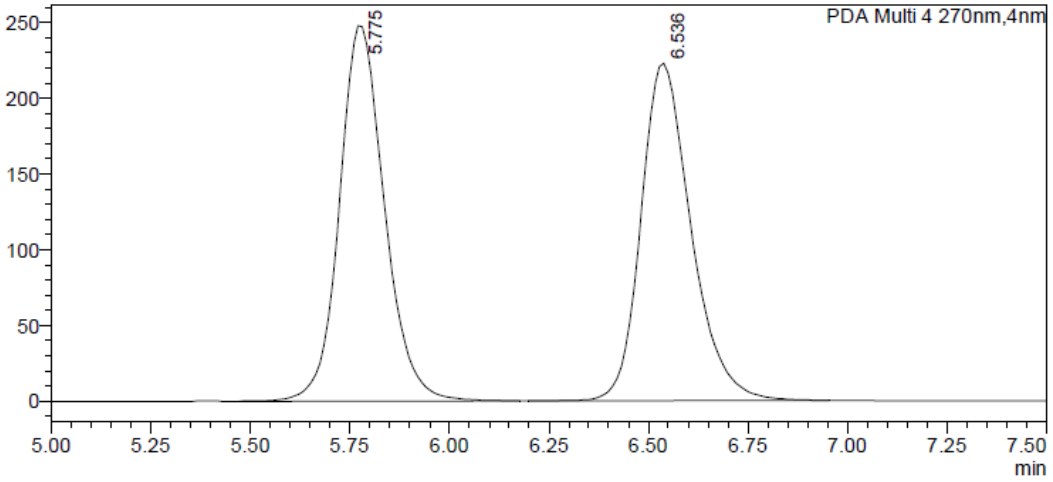


HPLC Data for S17: Chiralpak AD-H (99.8:0.2 hexane : IPA, flow rate 1.0 mLmin^{-1}, 270 nm, 30 °C) t_R (*R*): 5.7 min, t_R (*S*): 6.5 min, 10.693:89.307 er.



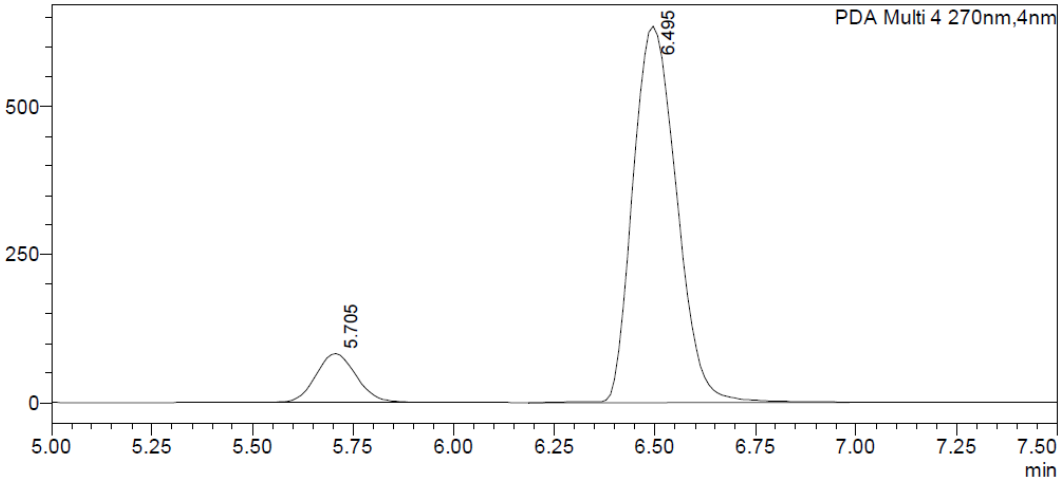
PDA Ch4 270nm

Peak#	Ret. Time	Area%
1	5.775	50.040
2	6.536	49.960
Total		100.000

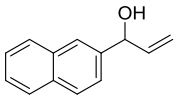


PDA Ch4 270nm

Peak#	Ret. Time	Area%
1	5.705	10.693
2	6.495	89.307
Total		100.000

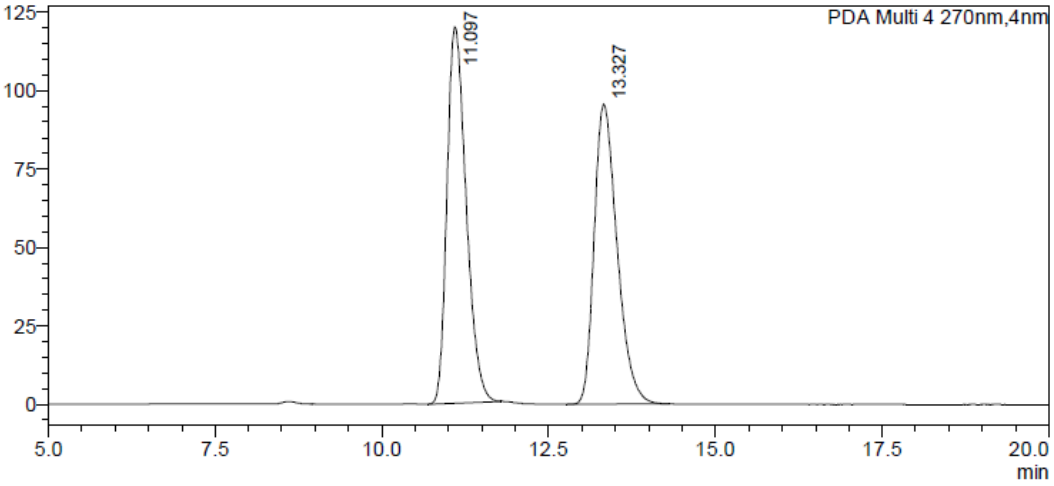


HPLC Data for 31: Chiralcel OJ-H (80:20 hexane : IPA, flow rate 1.0 mLmin^{-1}, 270 nm, 30 °C) t_R (*S*): 11.0 min, t_R (*R*): 13.2 min, 3.332:96.668 er. *s*=24.9



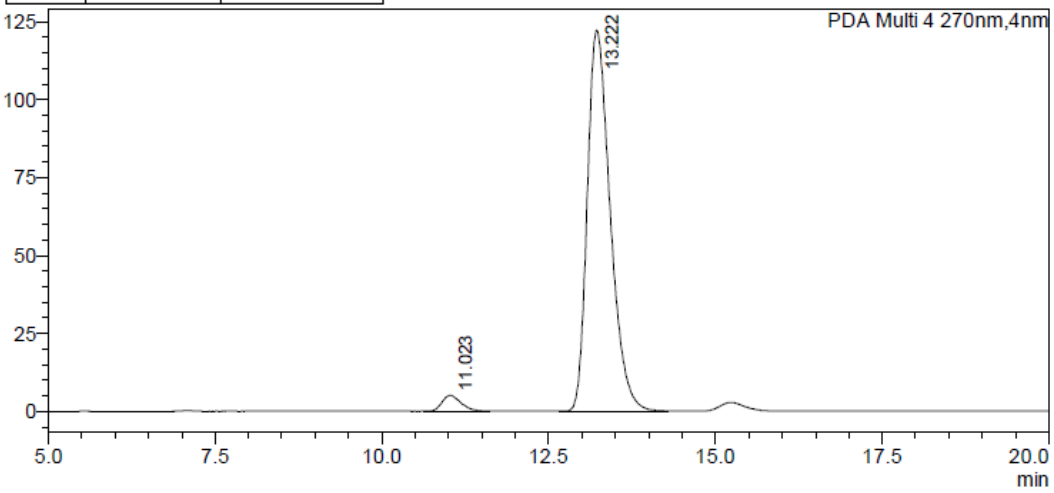
PDA Ch4 270nm

Peak#	Ret. Time	Area%
1	11.097	51.166
2	13.327	48.834
Total		100.000

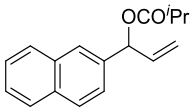


PDA Ch4 270nm

Peak#	Ret. Time	Area%
1	11.023	3.332
2	13.222	96.668
Total		100.000

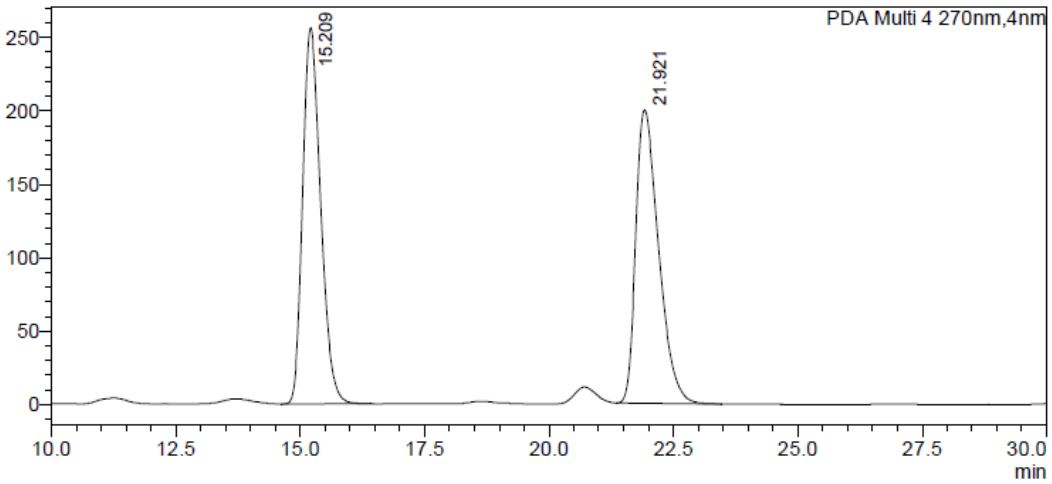


HPLC Data for S18: Chiralcel OJ-H (95:5 hexane : IPA, flow rate 0.5 mLmin^{-1}, 270 nm, 30 °C) t_R (*R*): 15.2 min, t_R (*S*): 22.0 min, 11.943:88.057 er.



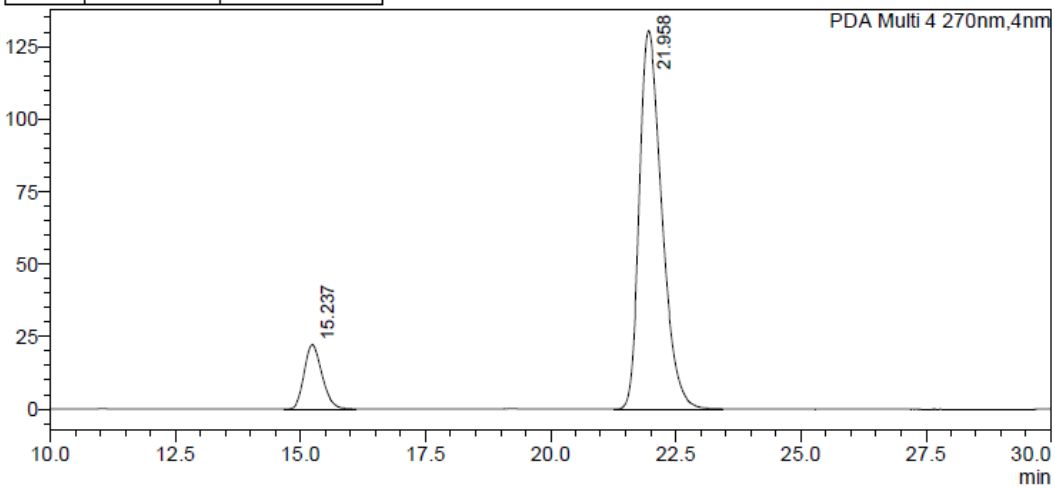
PDA Ch4 270nm

Peak#	Ret. Time	Area%
1	15.209	50.292
2	21.921	49.708
Total		100.000

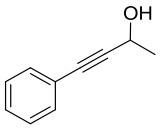


PDA Ch4 270nm

Peak#	Ret. Time	Area%
1	15.237	11.943
2	21.958	88.057
Total		100.000

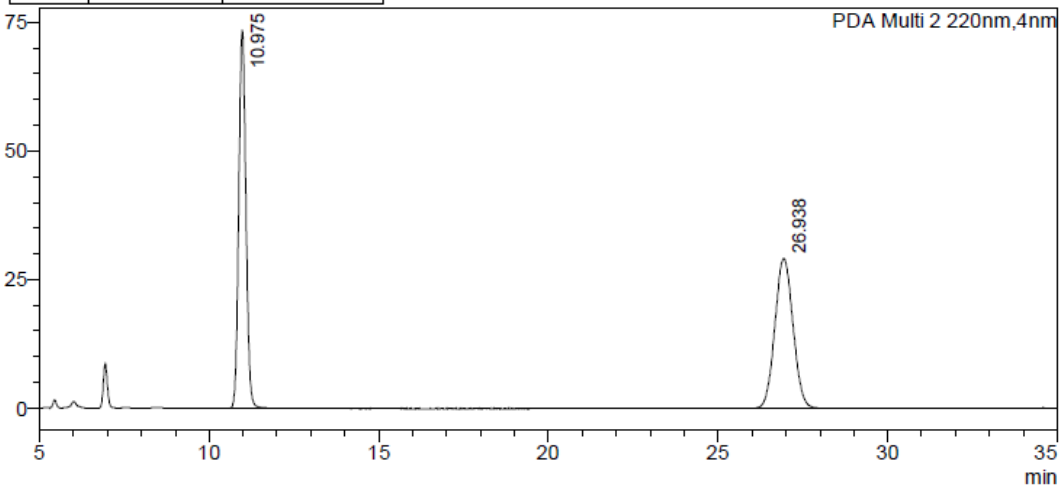


HPLC Data for 32: Chiralcel OD-H (95:5 hexane : IPA, flow rate 1.0 mLmin^{-1}, 220 nm, 30 °C) t_R (*R*): 11.0 min, t_R (*S*): 27.3 min, 94.914:5.086 er. *s*=23.2



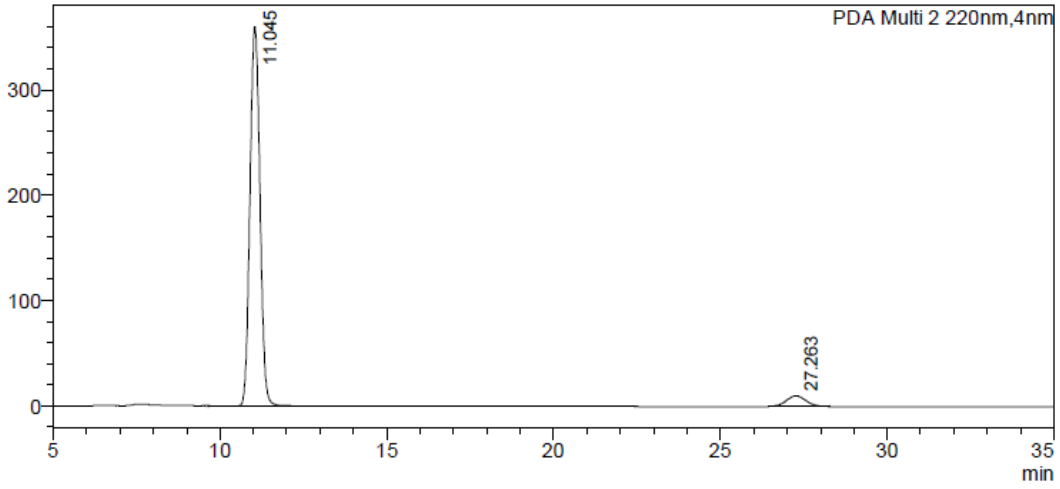
PDA Ch2 220nm

Peak#	Ret. Time	Area%
1	10.975	49.954
2	26.938	50.046
Total		100.000

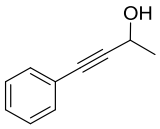


PDA Ch2 220nm

Peak#	Ret. Time	Area%
1	11.045	94.914
2	27.263	5.086
Total		100.000

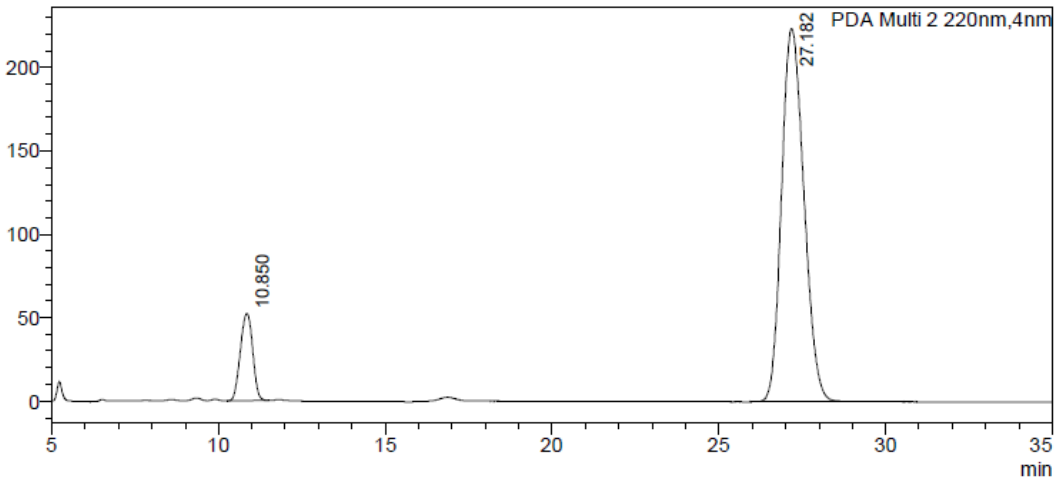


HPLC Data for S19 (following hydrolysis to alcohol 32): Chiralcel OD-H (95:5 hexane : IPA, flow rate 1.0 mLmin^{-1}, 220 nm, 30 °C) t_R (*R*): 10.9 min, t_R (*S*): 27.2 min, 11.457:88.543 er.

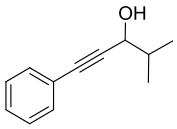


PDA Ch2 220nm

Peak#	Ret. Time	Area%
1	10.850	11.457
2	27.182	88.543
Total		100.000

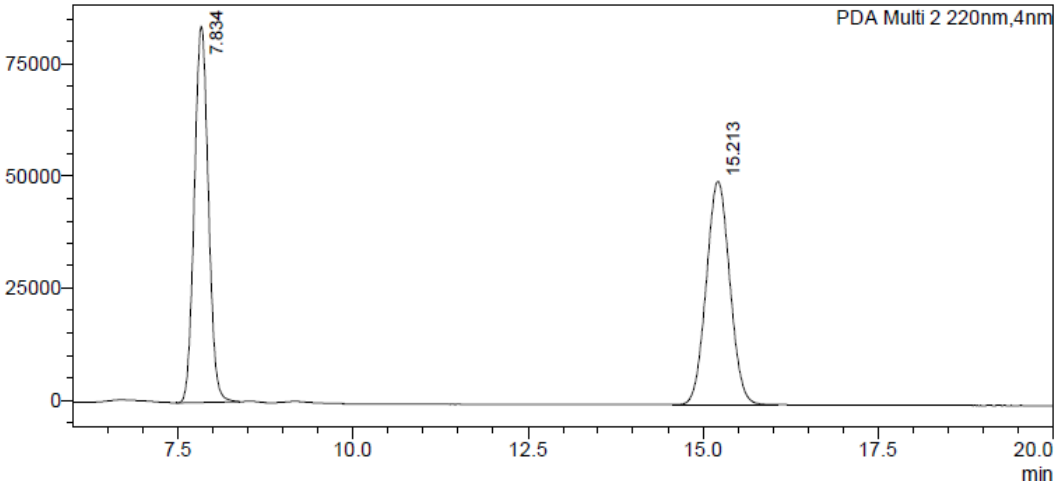


HPLC Data for 33: Chiralcel OD-H (95:5 hexane : IPA, flow rate 1.0 mLmin^{-1}, 220 nm, 30 °C) t_R (*R*): 7.8 min, t_R (*S*): 15.1 min, 95.367:4.633 er. *s*=25.8



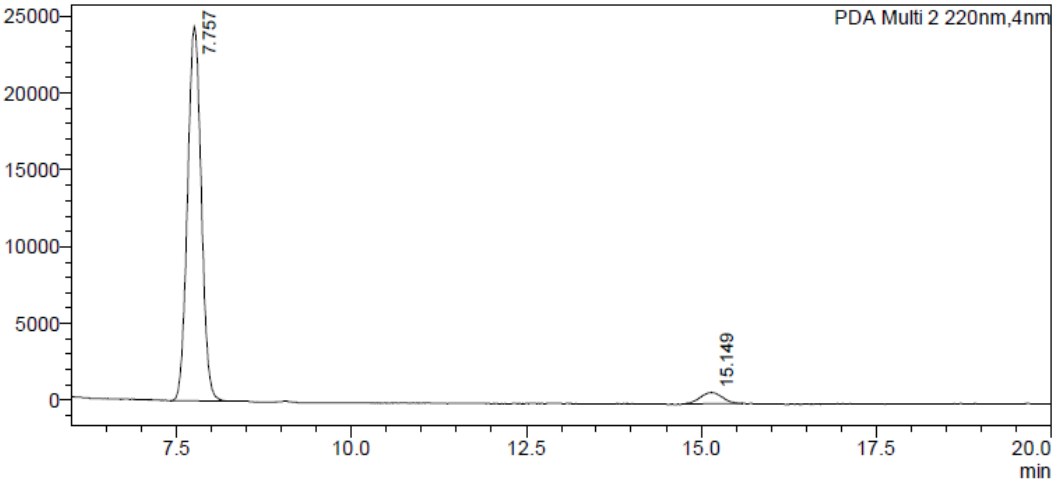
PDA Ch2 220nm

Peak#	Ret. Time	Area%
1	7.834	50.121
2	15.213	49.879
Total		100.000

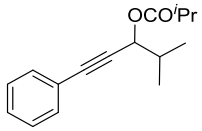


PDA Ch2 220nm

Peak#	Ret. Time	Area%
1	7.757	95.367
2	15.149	4.633
Total		100.000

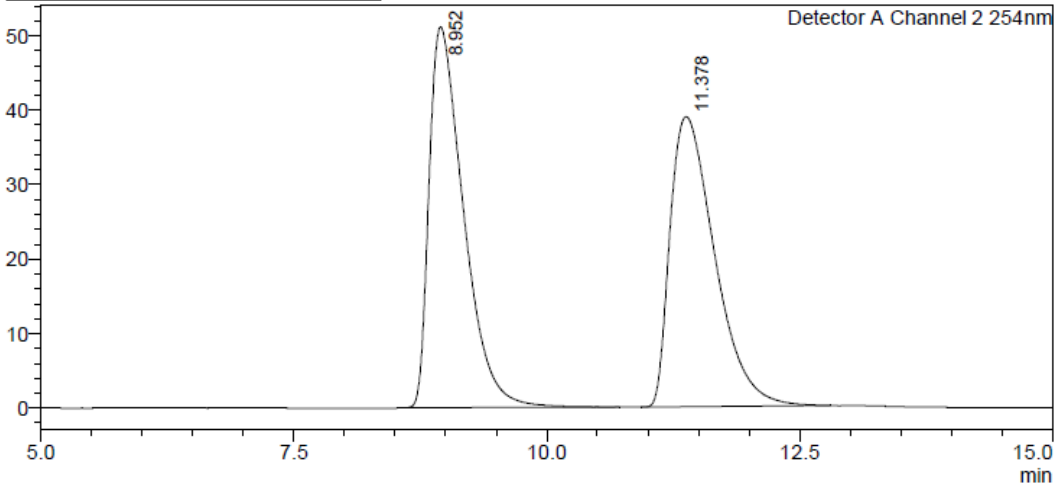


HPLC Data for S20: Chiralpak AD-H (100:0 hexane : IPA, flow rate 1.0 mLmin^{-1}, 254 nm, 30 °C) t_R (*S*): 9.0 min, t_R (*R*): 11.6 min, 89.283:10.717 er.



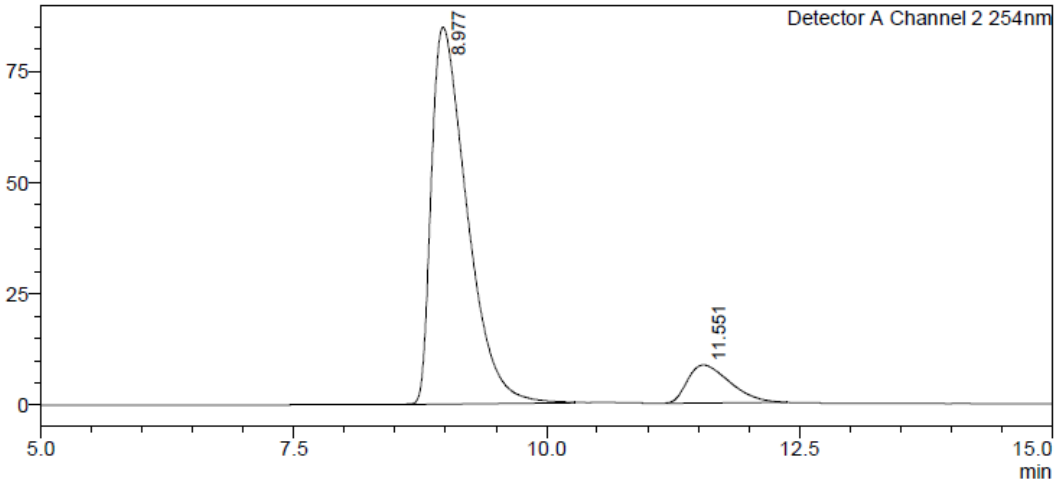
Detector A Channel 2 254nm

Peak#	Ret. Time	Area%
1	8.952	50.356
2	11.378	49.644
Total		100.000

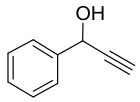


Detector A Channel 2 254nm

Peak#	Ret. Time	Area%
1	8.977	89.283
2	11.551	10.717
Total		100.000

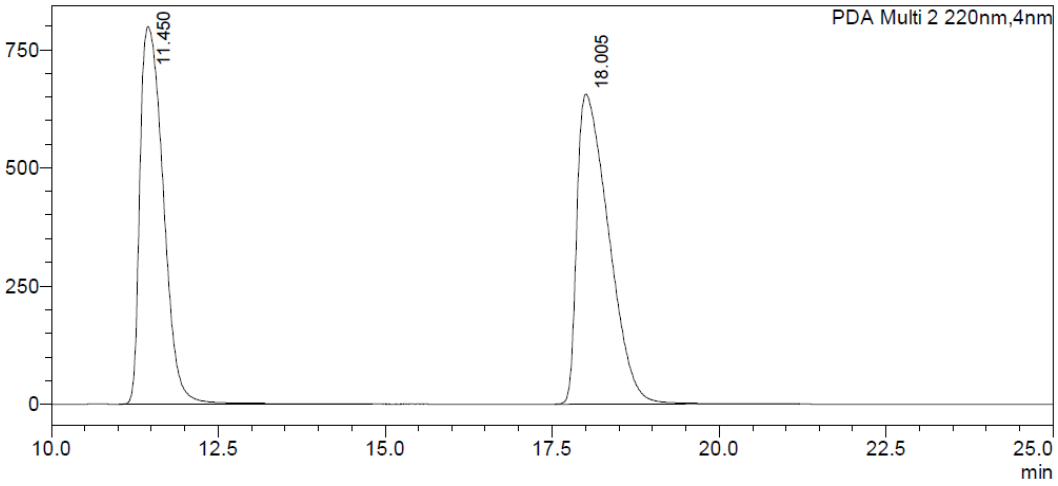


HPLC Data for 34: Chiralcel OD-H (95:5 hexane : IPA, flow rate 1.0 mLmin^{-1}, 220 nm, 30 °C) t_R (*R*): 12.1 min, t_R (*S*) : 19.0 min, 67.334:32.666 er. *s*=2.7



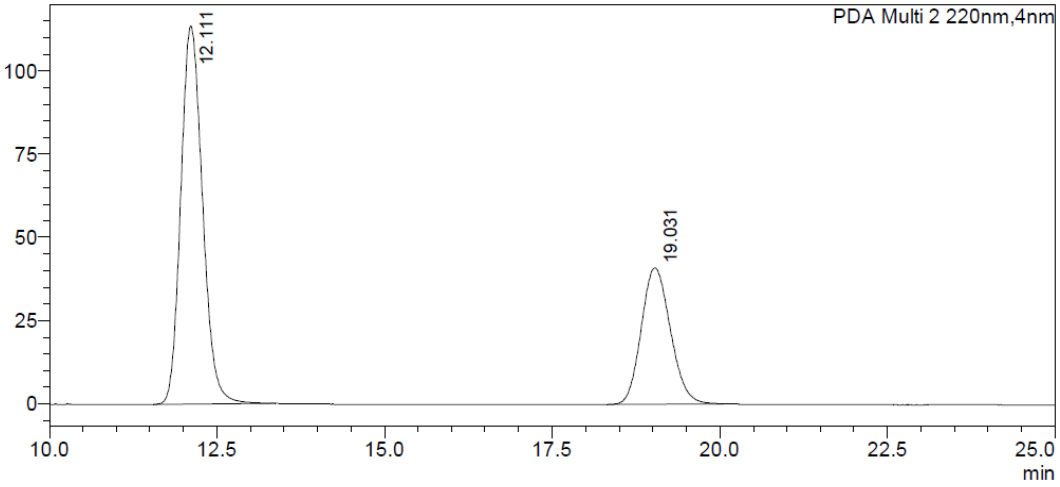
PDA Ch2 220nm

Peak#	Ret. Time	Area%
1	11.450	47.359
2	18.005	52.641
Total		100.000

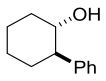


PDA Ch2 220nm

Peak#	Ret. Time	Area%
1	12.111	67.334
2	19.031	32.666
Total		100.000

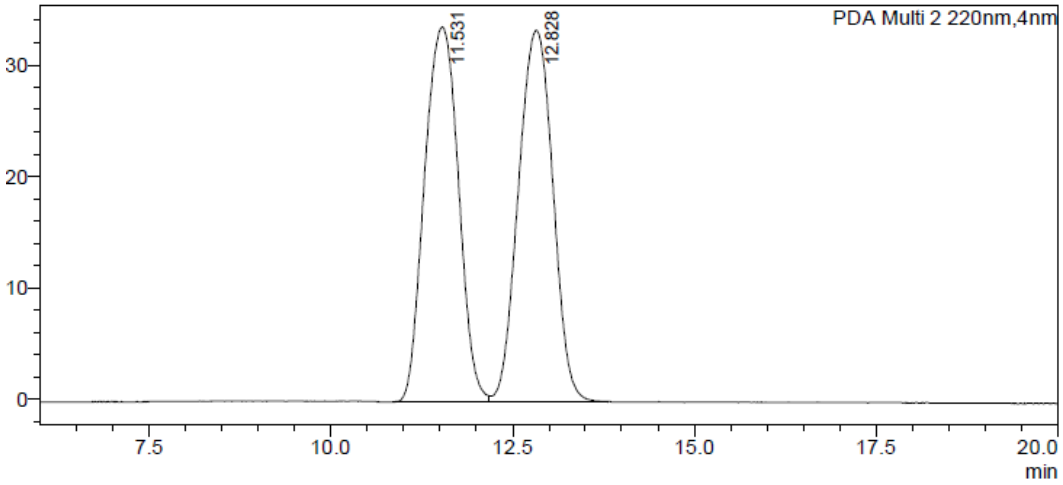


HPLC Data for 35 (Using Isobutyric anhydride): Chiralcel OD-H (99:1 hexane : IPA, flow rate 1.0 mLmin^{-1}, 220 nm, 30 °C) t_R (1*S*, 2*R*): 11.4 min, t_R (1*R*,2*S*) : 12.6 min, 31.095:68.905 er. *s*=39.9



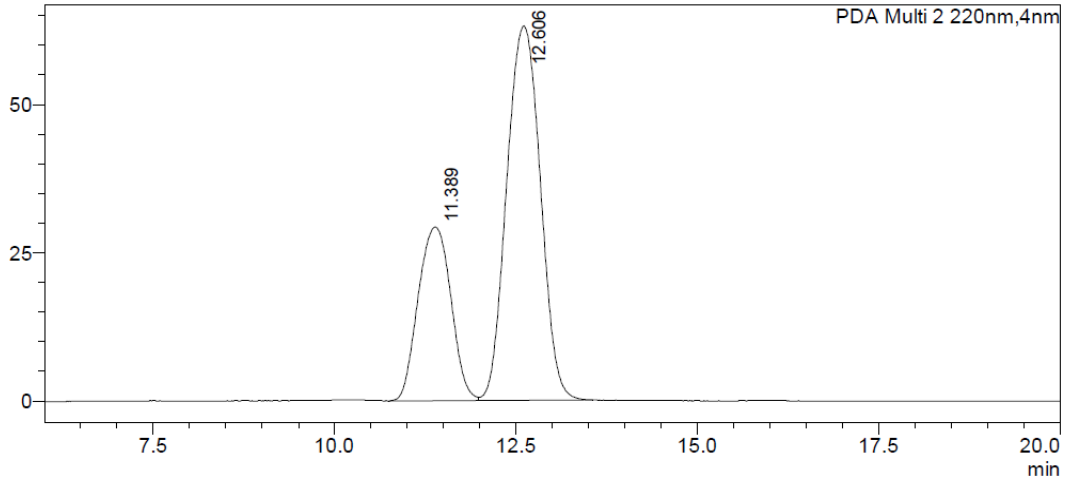
PDA Ch2 220nm

Peak#	Ret. Time	Area%
1	11.531	49.850
2	12.828	50.150
Total		100.000

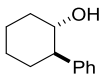


PDA Ch2 220nm

Peak#	Ret. Time	Area%
1	11.389	31.095
2	12.606	68.905
Total		100.000

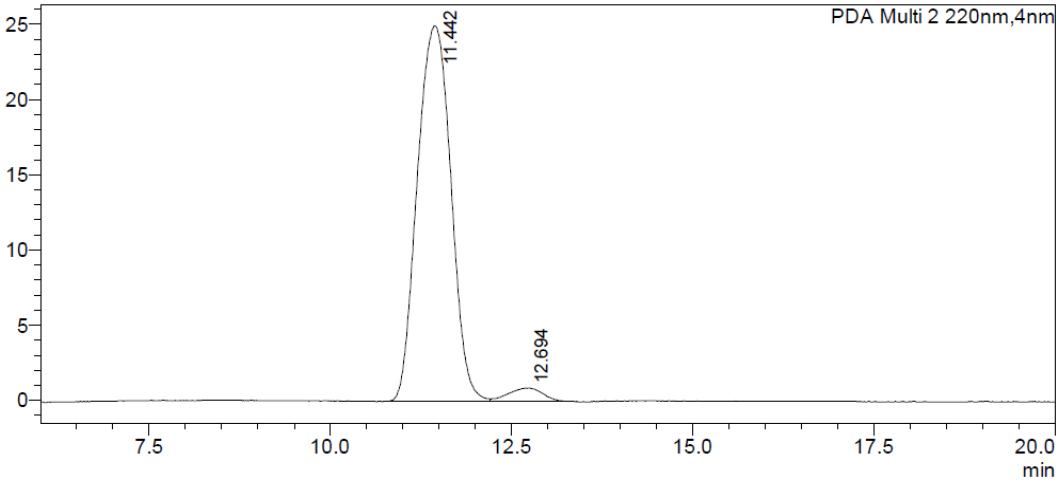


HPLC Data for S22 (following hydrolysis to alcohol 35): Chiralcel OD-H (99:1 hexane : IPA, flow rate 1.0 mLmin^{-1}, 220 nm, 30 °C) t_R (1*S*,2*R*): 11.4 min, t_R (1*R*,2*S*) : 12.7 min, 96.486:3.514 er.

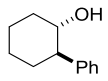


PDA Ch2 220nm

Peak#	Ret. Time	Area%
1	11.442	96.486
2	12.694	3.514
Total		100.000

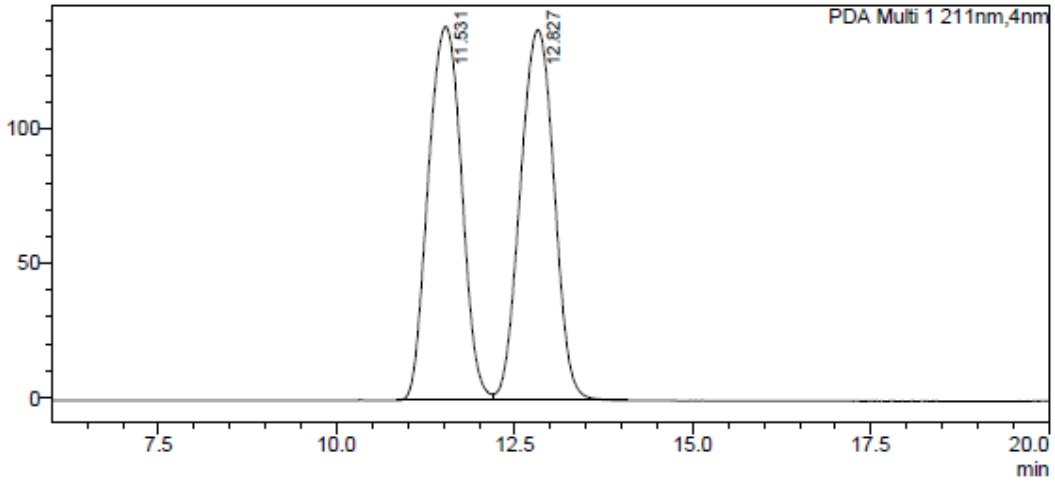


HPLC Data for 35 (Using Propionic anhydride): Chiralcel OD-H (99:1 hexane : IPA, flow rate 1.0 mLmin^{-1}, 211 nm, 30 °C) t_R (1*S*, 2*R*): 11.7 min, t_R (1*R*,2*S*) : 12.9 min, 0.714:99.286 er. *s*=51.1



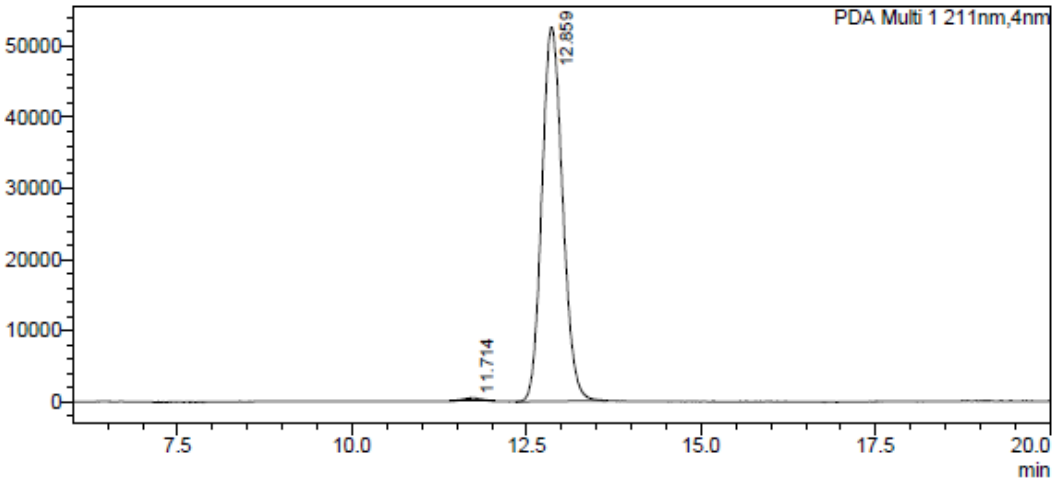
PDA Ch1 211nm

Peak#	Ret. Time	Area%
1	11.531	49.841
2	12.827	50.159
Total		100.000

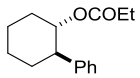


PDA Ch1 211nm

Peak#	Ret. Time	Area%
1	11.714	0.714
2	12.859	99.286
Total		100.000

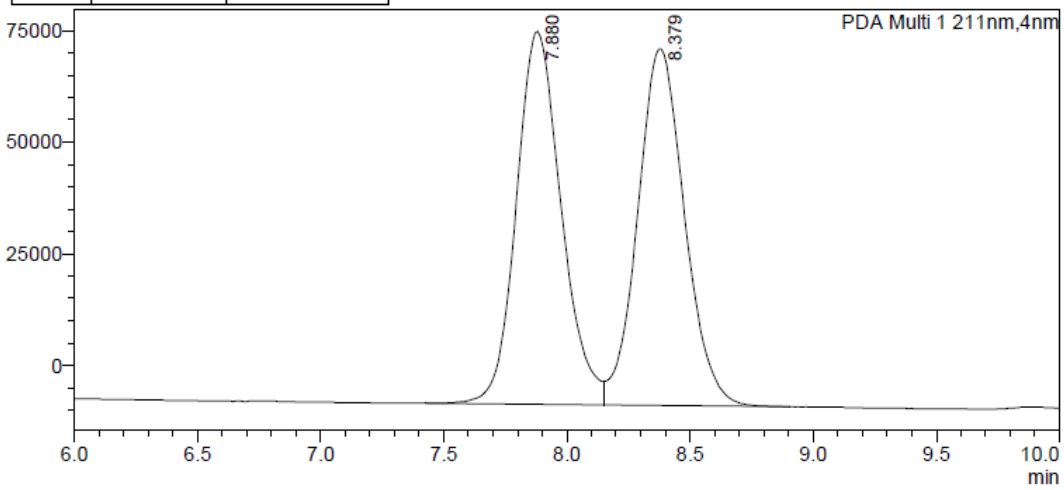


HPLC Data for 36: Chiralpak AD-H (100:0 hexane : IPA, flow rate 1.0 mLmin^{-1}, 211 nm, 30 °C) t_R (1*S*, 2*R*): 7.7 min, t_R (1*R*,2*S*) : 8.2 min 91.380:8.620 er.



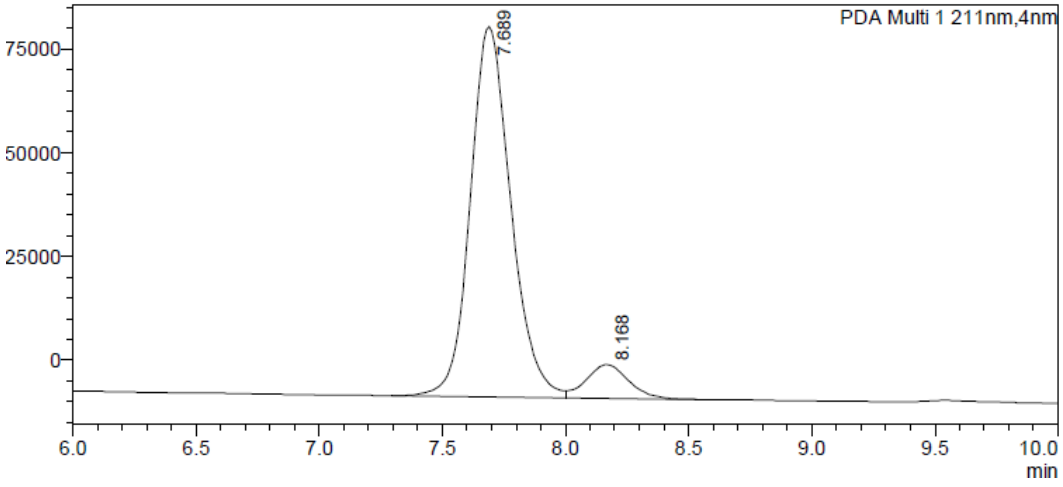
PDA Ch1 211nm

Peak#	Ret. Time	Area%
1	7.880	50.061
2	8.379	49.939
Total		100.000

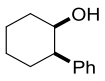


PDA Ch1 211nm

Peak#	Ret. Time	Area%
1	7.689	91.380
2	8.168	8.620
Total		100.000

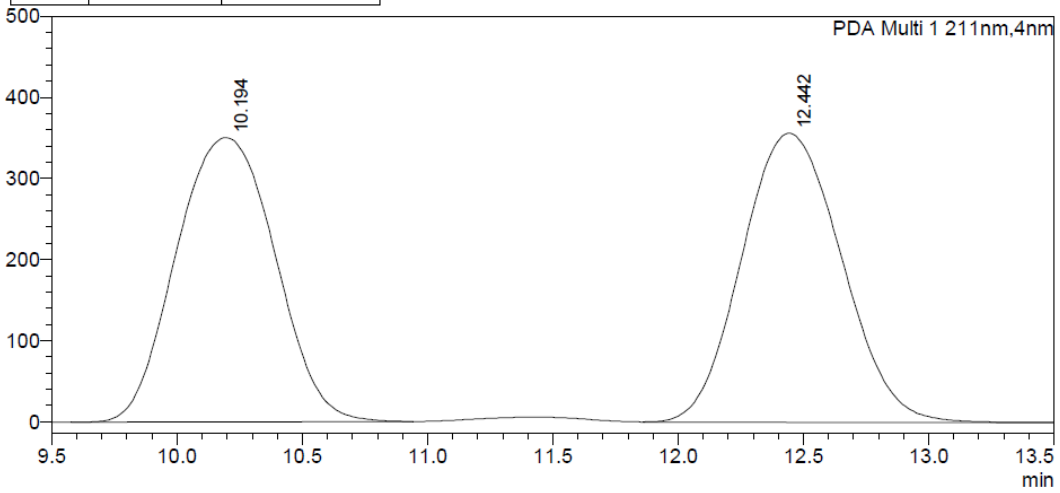


HPLC Data for 37 (Using Isobutyric anhydride): Chiralcel OD-H (99:1 hexane : IPA, flow rate 1.0 mLmin^{-1}, 211 nm, 30 °C) t_R (1*S*,2*S*): 10.6 min, t_R (1*R*,2*R*): 12.9 min, 43.995:56.005 er. *s*=6.3



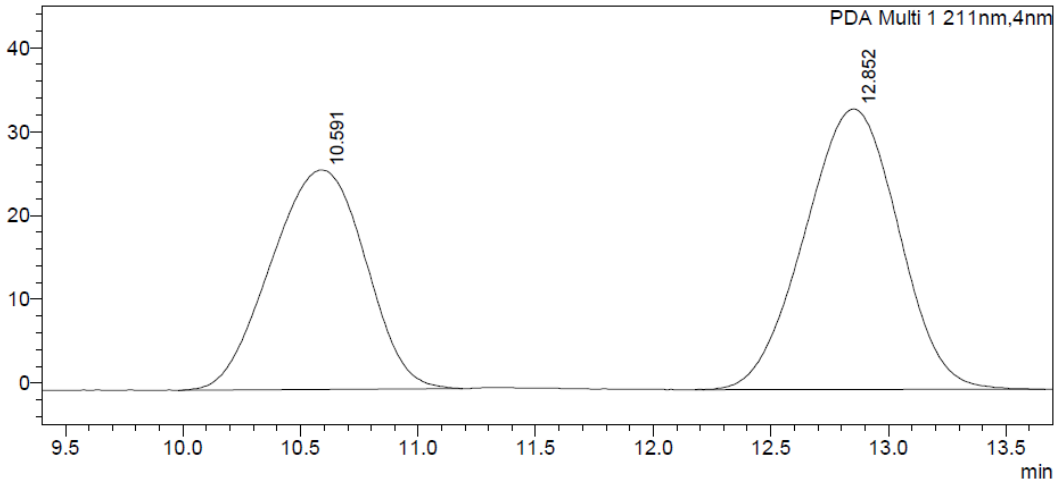
PDA Ch1 211nm

Peak#	Ret. Time	Area%
1	10.194	49.370
2	12.442	50.630
Total		100.000

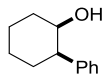


PDA Ch1 211nm

Peak#	Ret. Time	Area%
1	10.591	43.995
2	12.852	56.005
Total		100.000

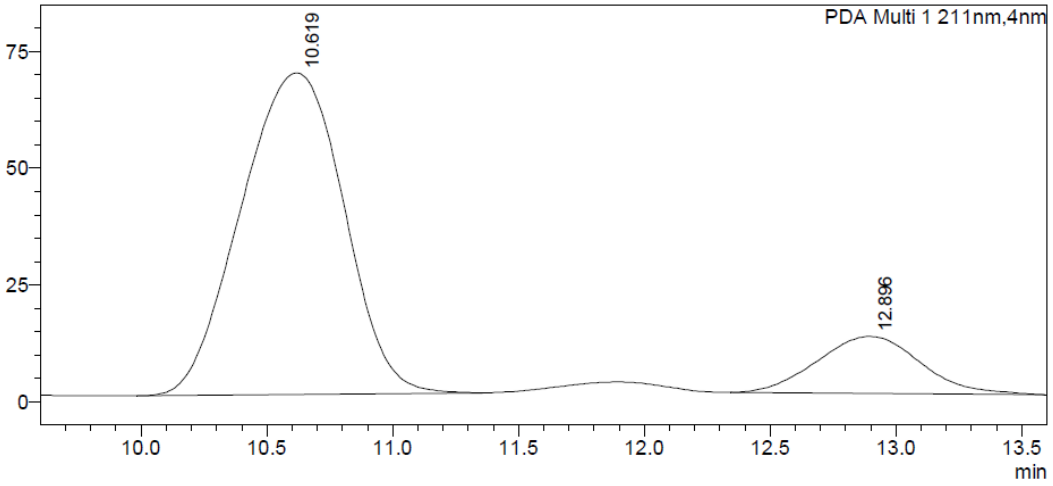


HPLC Data for S23 (following hydrolysis to alcohol 37): Chiralcel OD-H (99:1 hexane : IPA, flow rate 1.0 mLmin^{-1}, 211 nm, 30 °C) t_R (1*S*,2*S*): 10.6 min, t_R (1*R*,2*R*): 12.9 min, 84.870:15.130 er.

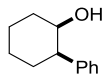


PDA Ch1 211nm

Peak#	Ret. Time	Area%
1	10.619	84.870
2	12.896	15.130
Total		100.000

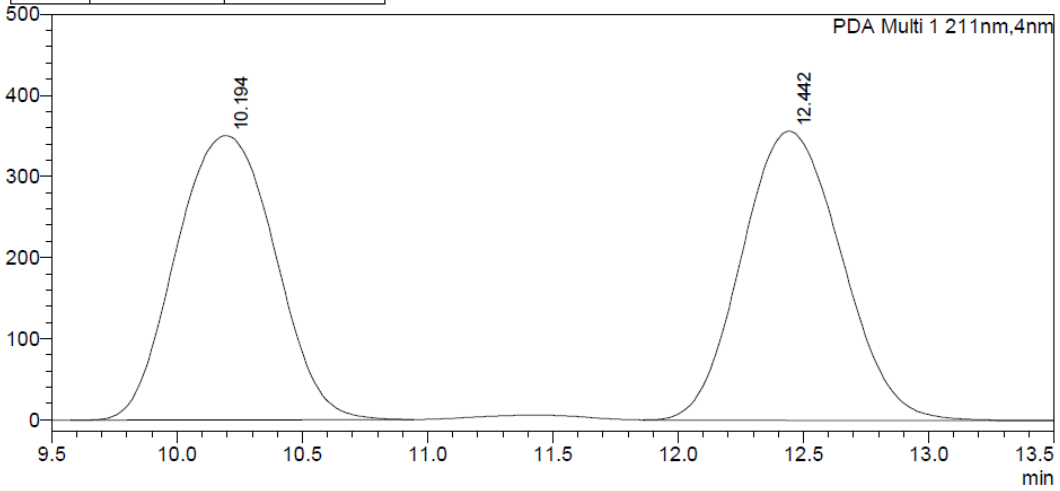


HPLC Data for 37 (Using Propionic anhydride): Chiralcel OD-H (99:1 hexane : IPA, flow rate 1.0 mLmin^{-1}, 211 nm, 30 °C) t_R (1*S*,2*S*): 10.2 min, t_R (1*R*,2*R*): 12.5 min, 16.114:83.886 er. *s*=35.6



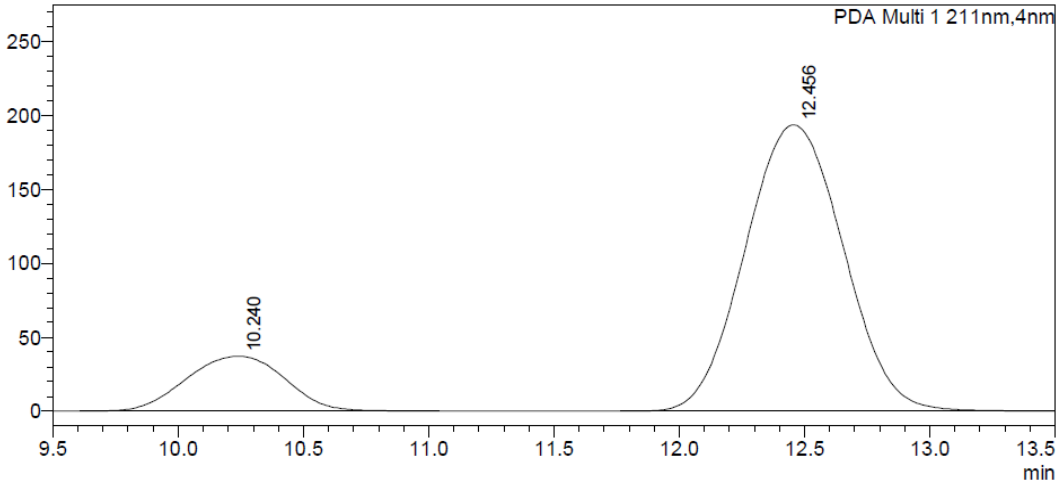
PDA Ch1 211nm

Peak#	Ret. Time	Area%
1	10.194	49.370
2	12.442	50.630
Total		100.000

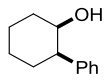


PDA Ch1 211nm

Peak#	Ret. Time	Area%
1	10.240	16.114
2	12.456	83.886
Total		100.000

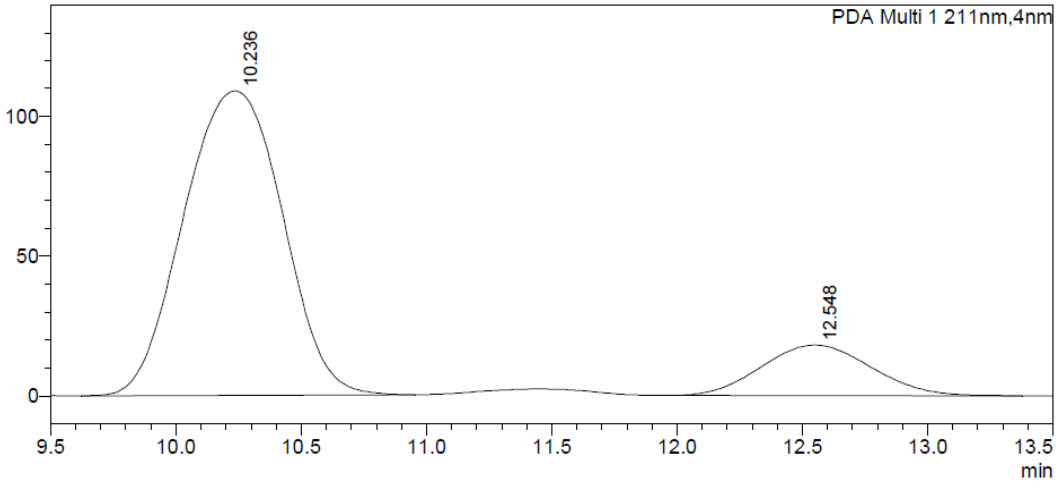


HPLC Data for 38 (following hydrolysis to alcohol 38): Chiralcel OD-H (99:1 hexane : IPA, flow rate 1.0 mLmin^{-1}, 211 nm, 30 °C) t_R (1*S*,2*S*): 10.2 min, t_R (1*R*,2*R*): 12.5 min, 84.420:15.580 er.

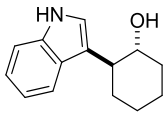


PDA Ch1 211nm

Peak#	Ret. Time	Area%
1	10.236	84.420
2	12.548	15.580
Total		100.000

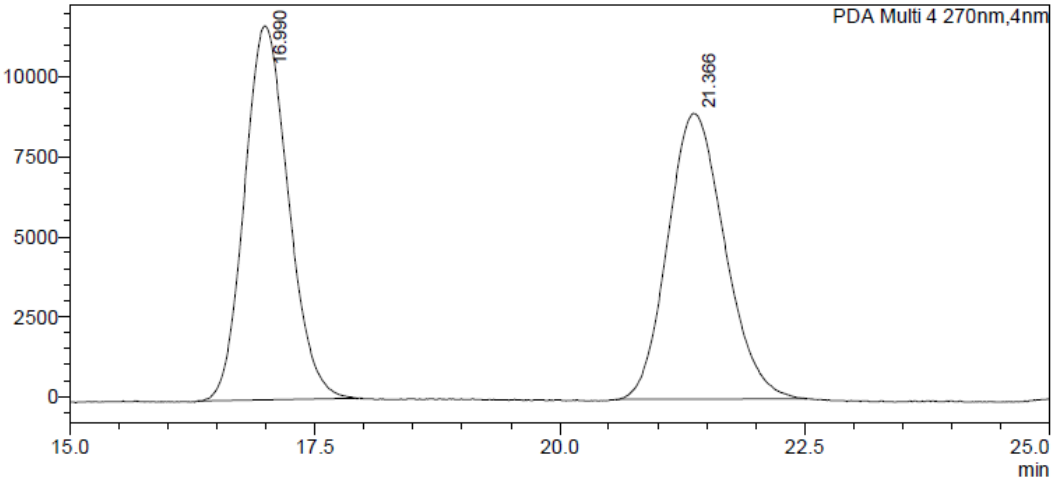


HPLC Data for 39 (Using Isobutyric anhydride): Chiralcel OD-H (90:10 hexane : IPA, flow rate 1.0 mLmin^{-1}, 270 nm, 30 °C) t_R (1*S*,2*R*): 16.9 min, t_R (1*R*,2*S*) : 21.3 min, 45.244:54.756 er. *s*=1.8



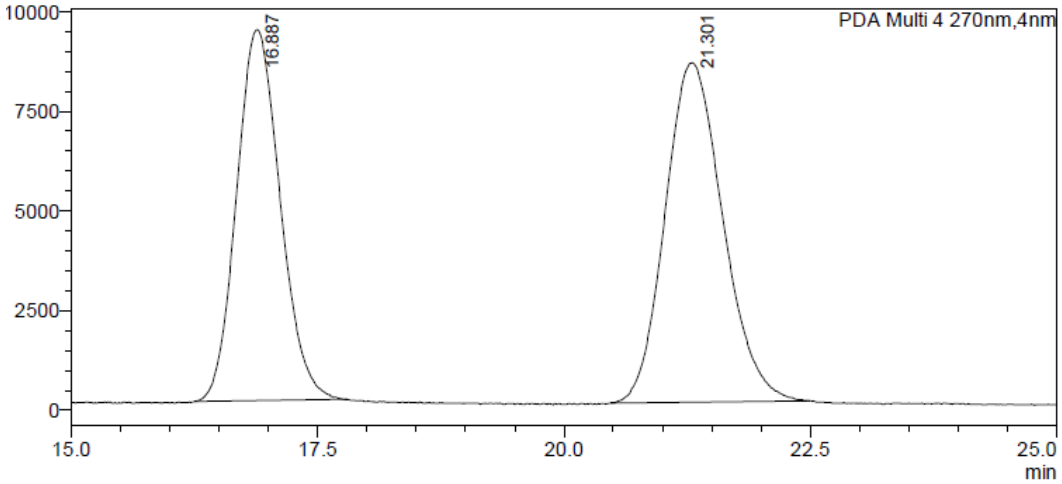
PDA Ch4 270nm

Peak#	Ret. Time	Area%
1	16.990	50.193
2	21.366	49.807
Total		100.000

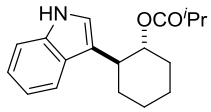


PDA Ch4 270nm

Peak#	Ret. Time	Area%
1	16.887	45.244
2	21.301	54.756
Total		100.000

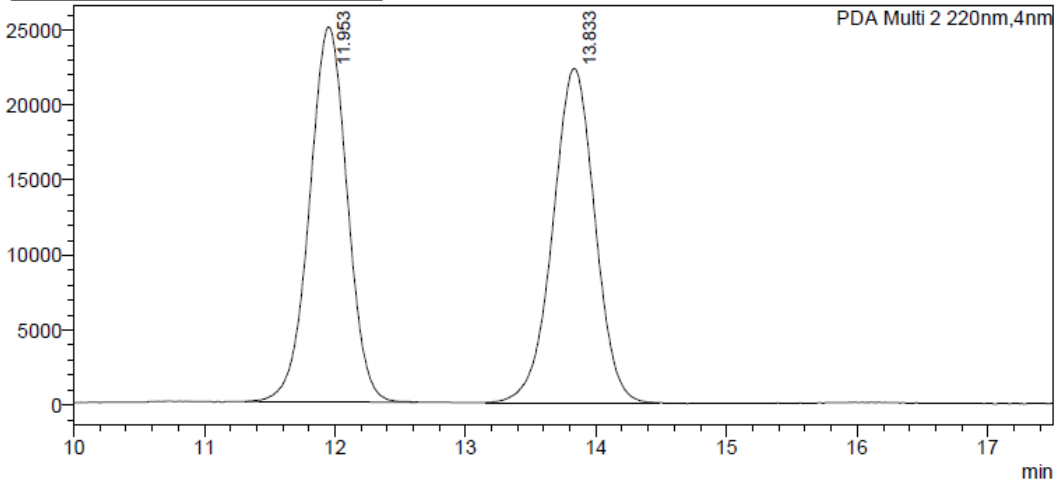


HPLC Data for S24: Chiralpak AD-H (96:4 hexane : IPA, flow rate 1.0 mLmin^{-1}, 220 nm, 30 °C) t_R (1*R*,2*S*): 12.2 min, t_R (1*S*,2*R*) : 14.1 min 37.710:62.290 er.



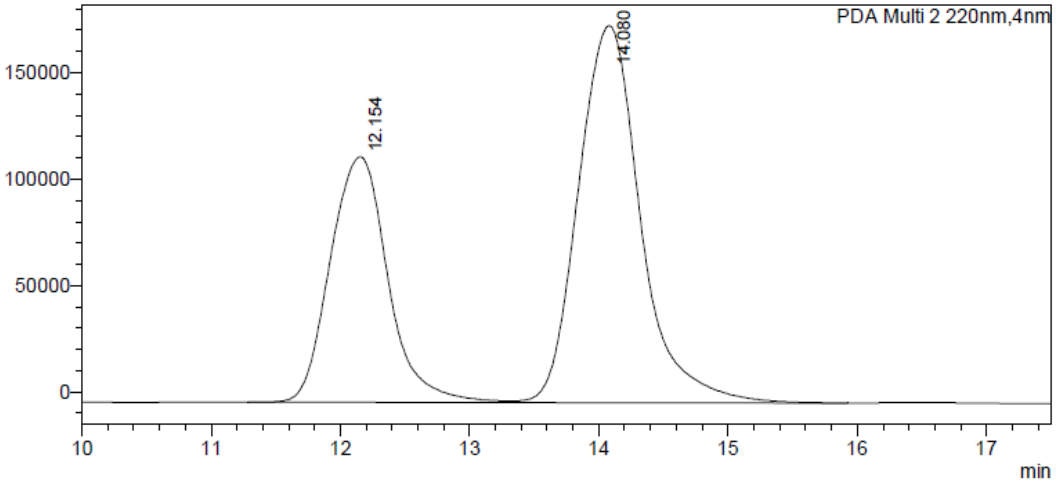
PDA Ch2 220nm

Peak#	Ret. Time	Area%
1	11.953	50.085
2	13.833	49.915
Total		100.000

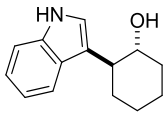


PDA Ch2 220nm

Peak#	Ret. Time	Area%
1	12.154	37.710
2	14.080	62.290
Total		100.000

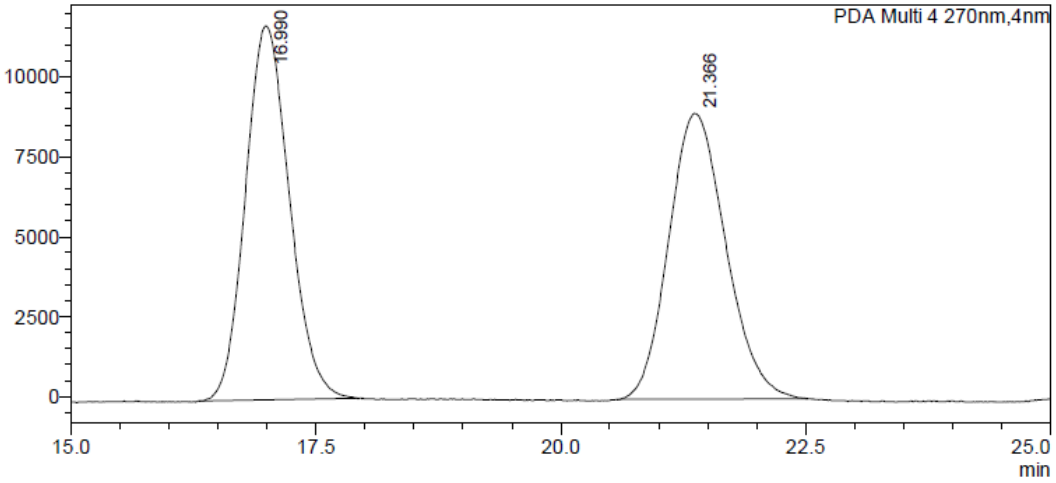


HPLC Data for 39 (Using Propionic anhydride): Chiralcel OD-H (90:10 hexane : IPA, flow rate 1.0 mLmin^{-1}, 270 nm, 30 °C) t_R (1*S*,2*R*): 17.0 min, t_R (1*R*,2*S*) : 21.3 min, 11.491:88.509 er. *s*=51.3



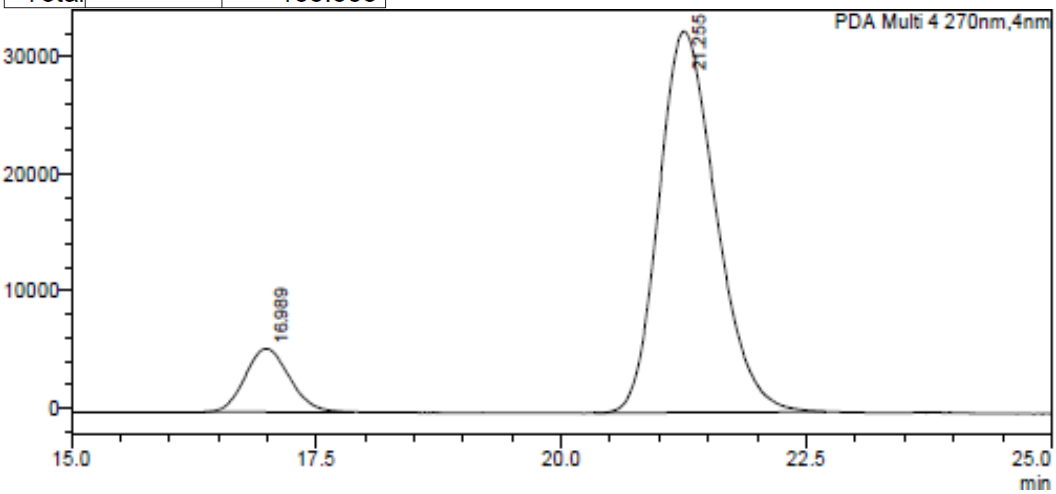
PDA Ch4 270nm

Peak#	Ret. Time	Area%
1	16.990	50.193
2	21.366	49.807
Total		100.000

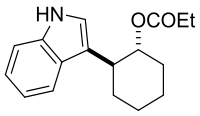


PDA Ch4 270nm

Peak#	Ret. Time	Area%
1	16.989	11.491
2	21.255	88.509
Total		100.000

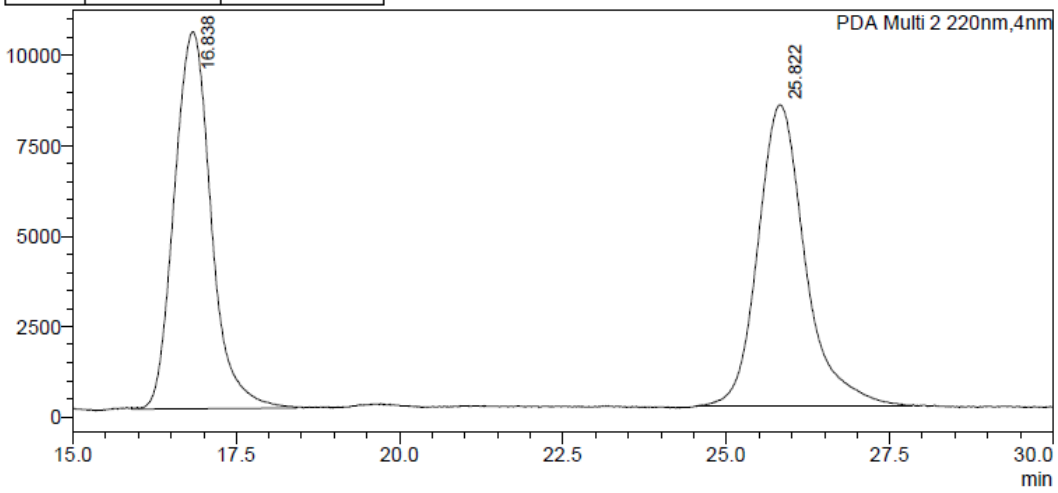


HPLC Data for 40: Chiralpak AD-H (97:3 hexane : IPA, flow rate 1.0 mLmin^{-1}, 220 nm, 30 °C) t_R (1*R*,2*S*): 17.2 min, t_R (1*S*,2*R*) : 26.3 min, 4.345:95.655 er.



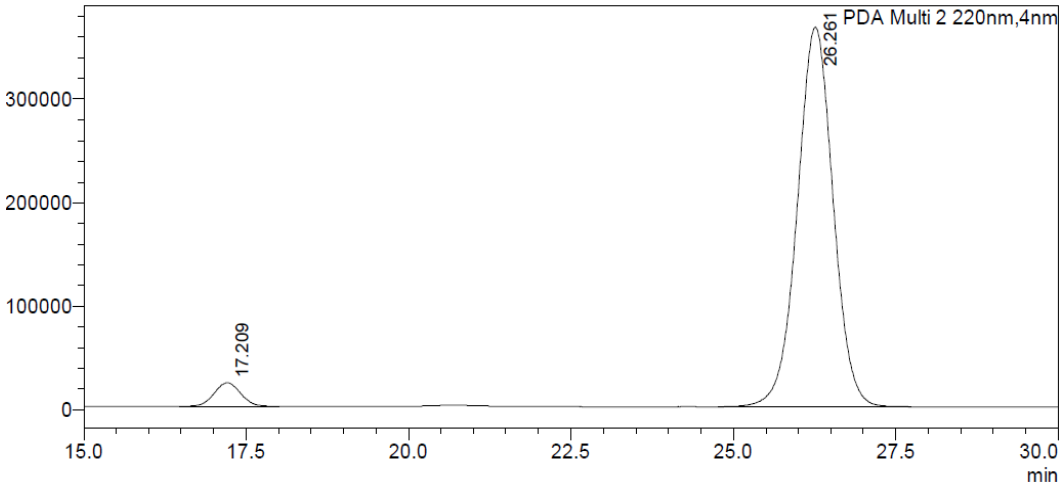
PDA Ch2 220nm

Peak#	Ret. Time	Area%
1	16.838	49.461
2	25.822	50.539
Total		100.000

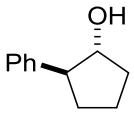


PDA Ch2 220nm

Peak#	Ret. Time	Area%
1	17.209	4.345
2	26.261	95.655
Total		100.000

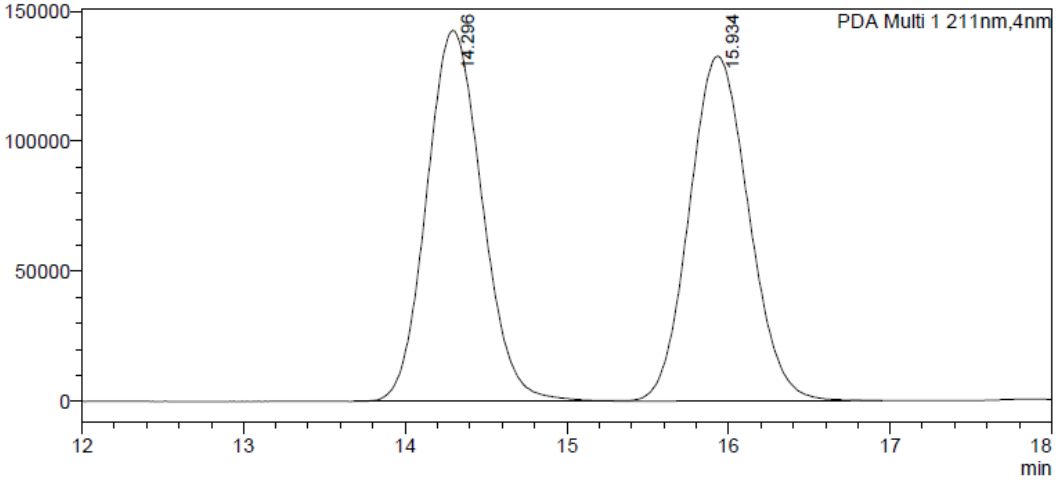


HPLC Data for 41 (Using Isobutyric anhydride):: Chiralcel OD-H (98:2 hexane : IPA, flow rate 1.0 mLmin^{-1}, 211 nm, 30 °C) t_R (1*S*, 2*R*): 14.4 min, t_R (1*R*,2*S*) : 16.0 min, 2.445:97.555 er. *s*=72.3



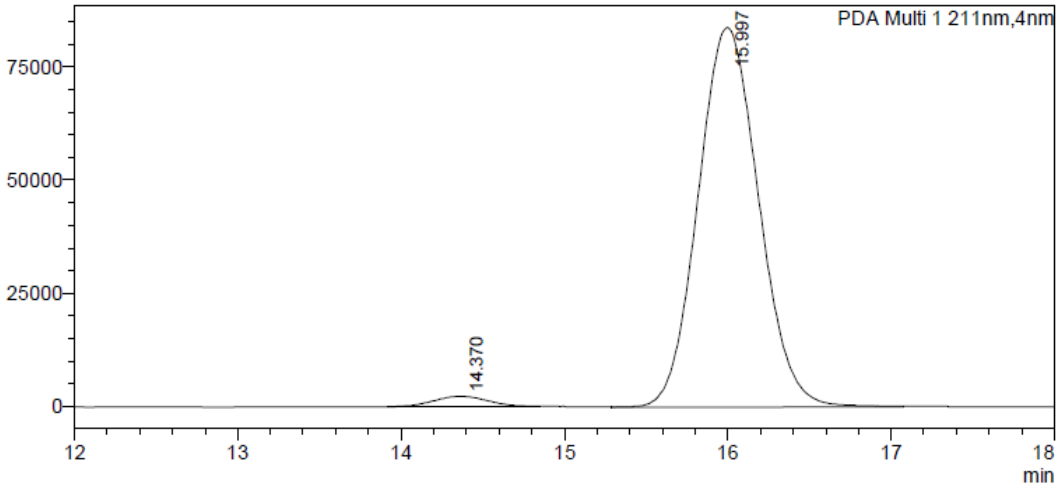
PDA Ch1 211nm

Peak#	Ret. Time	Area%
1	14.296	50.153
2	15.934	49.847
Total		100.000

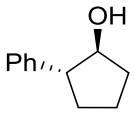


PDA Ch1 211nm

Peak#	Ret. Time	Area%
1	14.370	2.445
2	15.997	97.555
Total		100.000

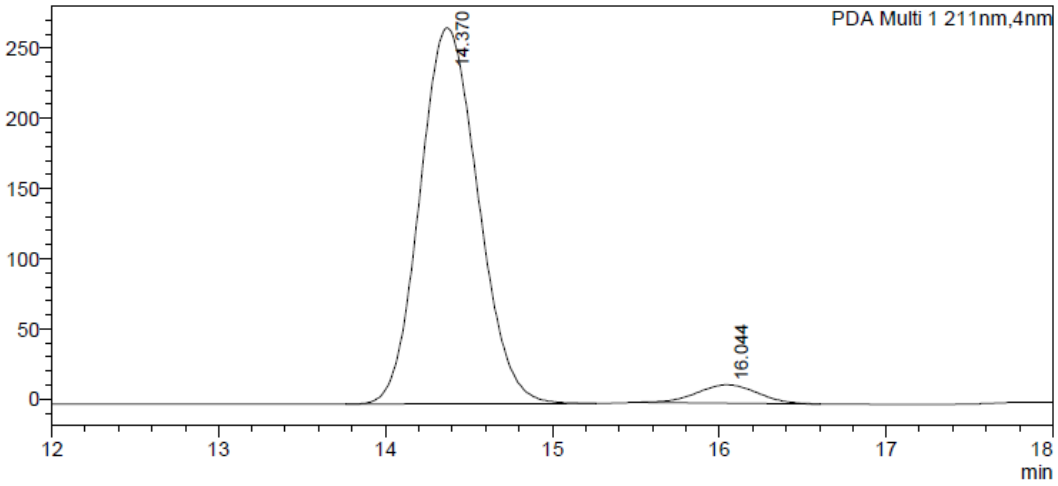


HPLC Data for 42 (following hydrolysis to alcohol 41): Chiralcel OD-H (98:2 hexane : IPA, flow rate 1.0 mLmin^{-1}, 211 nm, 30 °C) t_R (1*S*, 2*R*): 14.4 min, t_R (1*R*,2*S*) : 16.0 min, 95.093:4.907 er.

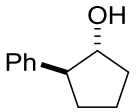


PDA Ch1 211nm

Peak#	Ret. Time	Area%
1	14.370	95.093
2	16.044	4.907
Total		100.000

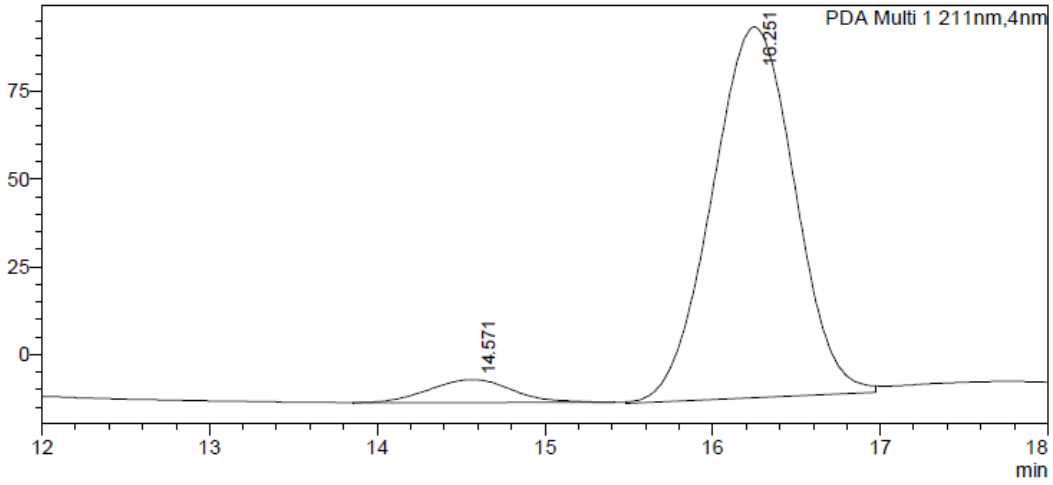


HPLC Data for 42 (Propionic anhydride): Chiralcel OD-H (98:2 hexane : IPA, flow rate 1.0 mLmin^{-1}, 211 nm, 30 °C) t_R (1*S*, 2*R*): 14.6 min, t_R (1*R*,2*S*) : 16.3 min, 5.905:94.095 er. *s*=35.6



PDA Ch1 211nm

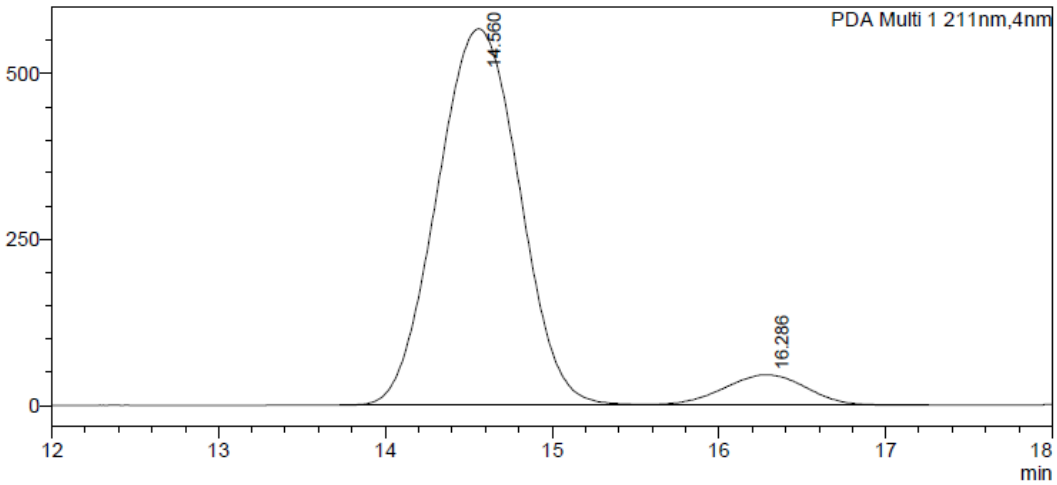
Peak#	Ret. Time	Area%
1	14.571	5.905
2	16.251	94.095
Total		100.000



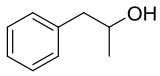
HPLC Data for S25 (following hydrolysis to alcohol 42: Chiralcel OD-H (98:2 hexane : IPA, flow rate 1.0 mLmin^{-1}, 211 nm, 30 °C) t_R (1*S*, 2*R*): 14.6 min, t_R (1*R*,2*S*) : 16.3 min, 92.458:7.542 er.

PDA Ch1 211nm

Peak#	Ret. Time	Area%
1	14.560	92.458
2	16.286	7.542
Total		100.000

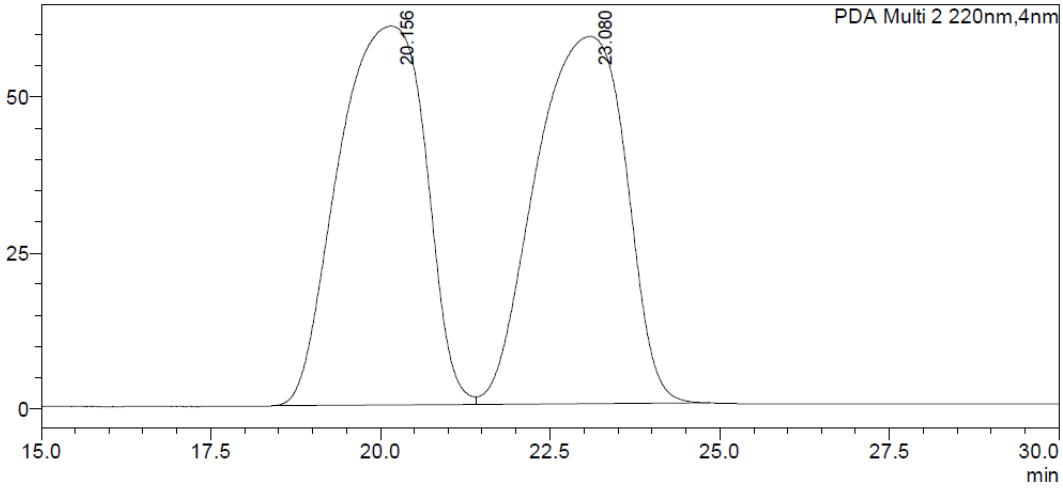


HPLC Data for 43 (Using Isobutyric anhydride): Chiralcel OD-H (99.5:0.5 hexane : IPA, flow rate 1.0 mLmin^{-1}, 220 nm, 30 °C) t_R (*S*): 20.3 min, t_R (*R*): 23.1 min, 36.823:63.177 er. *s*=2.4



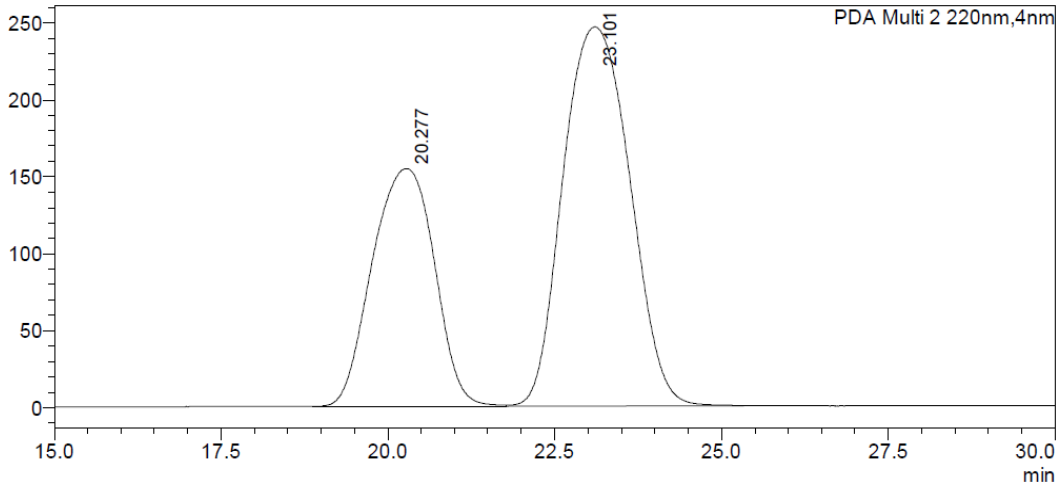
PDA Ch2 220nm

Peak#	Ret. Time	Area%
1	20.156	49.952
2	23.080	50.048
Total		100.000

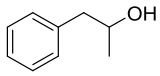


PDA Ch2 220nm

Peak#	Ret. Time	Area%
1	20.277	36.823
2	23.101	63.177
Total		100.000

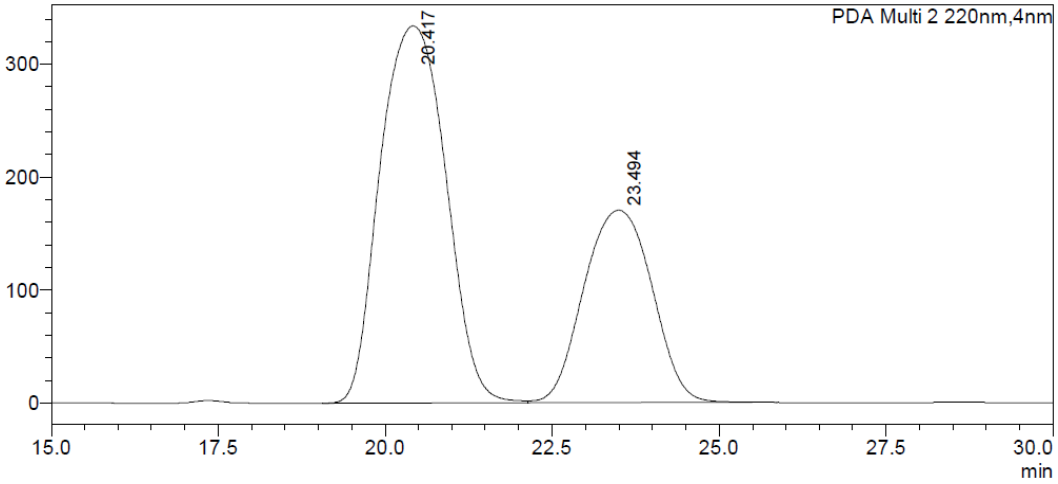


HPLC Data for S26 (following hydrolysis to alcohol 43): Chiralcel OD-H (99.5:0.5 hexane : IPA, flow rate 1.0 mLmin^{-1}, 220 nm, 30 °C) t_R (*S*): 20.4 min, t_R (*R*): 23.5 min, 65.391:34.609 er

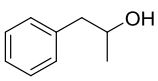


PDA Ch2 220nm

Peak#	Ret. Time	Area%
1	20.417	65.391
2	23.494	34.609
Total		100.000

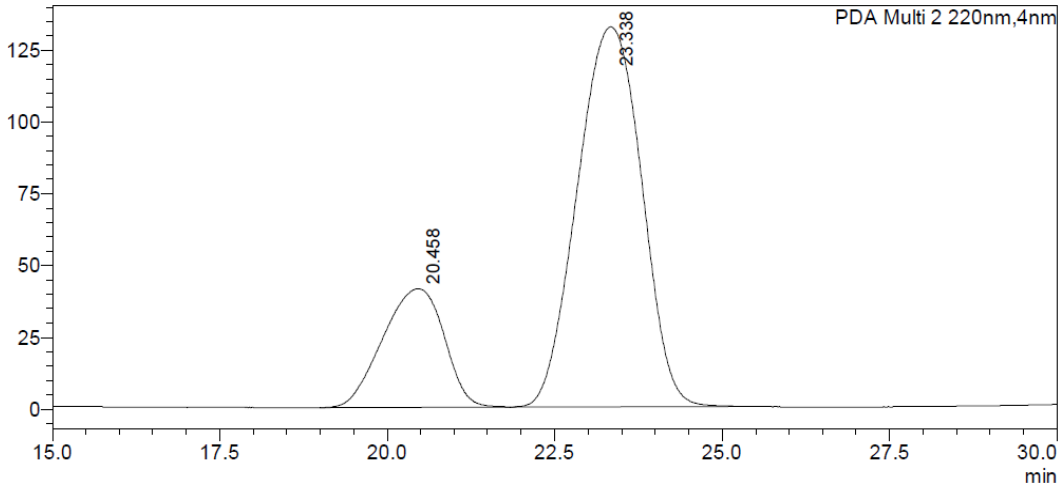


HPLC Data for 43 (Using Propionic anhydride): Chiralcel OD-H (99.5:0.5 hexane : IPA, flow rate 1.0 mLmin^{-1}, 220 nm, 30 °C) t_R (*S*): 20.5 min, t_R (*R*): 23.3 min, 22.909:77.091 er. *s*=7.0



PDA Ch2 220nm

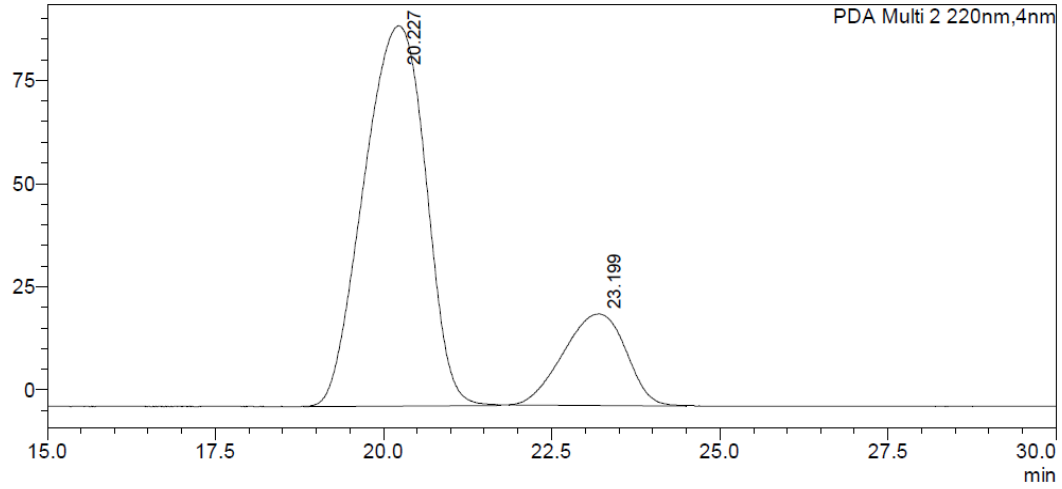
Peak#	Ret. Time	Area%
1	20.458	22.909
2	23.338	77.091
Total		100.000



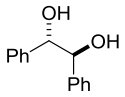
HPLC Data for 44 (following hydrolysis to alcohol 43): Chiralcel OD-H (99.5:0.5 hexane : IPA, flow rate 1.0 mLmin^{-1}, 220 nm, 30 °C) t_R (*S*): 20.3 min, t_R (*R*): 23.2 min, 80.421:19.579 er.

PDA Ch2 220nm

Peak#	Ret. Time	Area%
1	20.227	80.421
2	23.199	19.579
Total		100.000

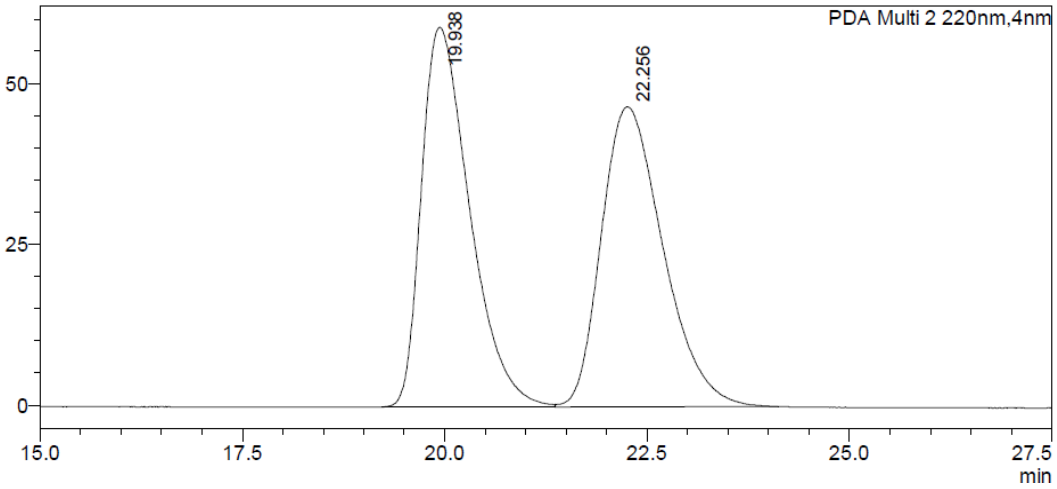


HPLC Data for 45: Chiralcel OJ-H (93:7 hexane : IPA, flow rate 1.0 mLmin^{-1}, 220 nm, 30 °C) t_R (*S*,*S*): 20.7 min, t_R (*R*,*R*) : 23.6 min, 83.768:16.232 er



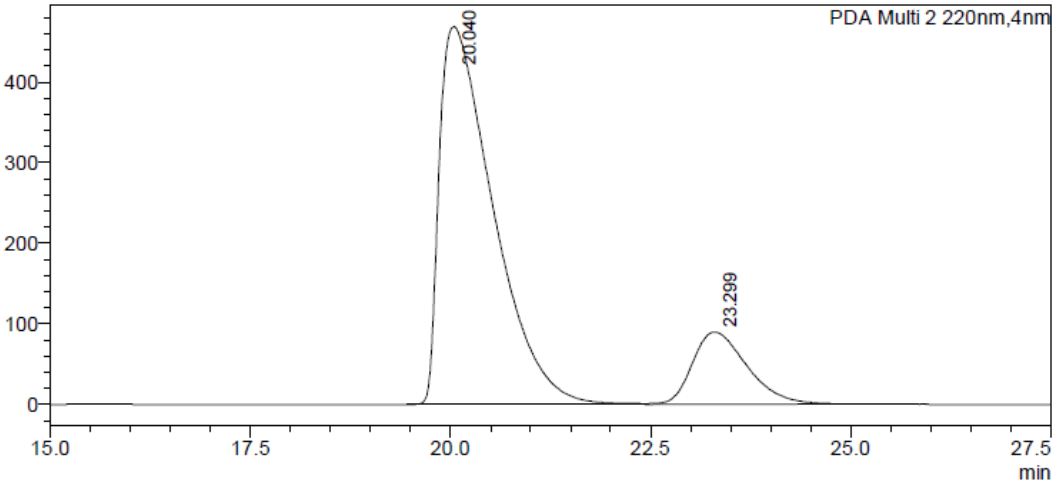
PDA Ch2 220nm

Peak#	Ret. Time	Area%
1	19.938	49.764
2	22.256	50.236
Total		100.000

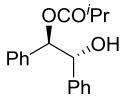


PDA Ch2 220nm

Peak#	Ret. Time	Area%
1	20.040	83.768
2	23.299	16.232
Total		100.000

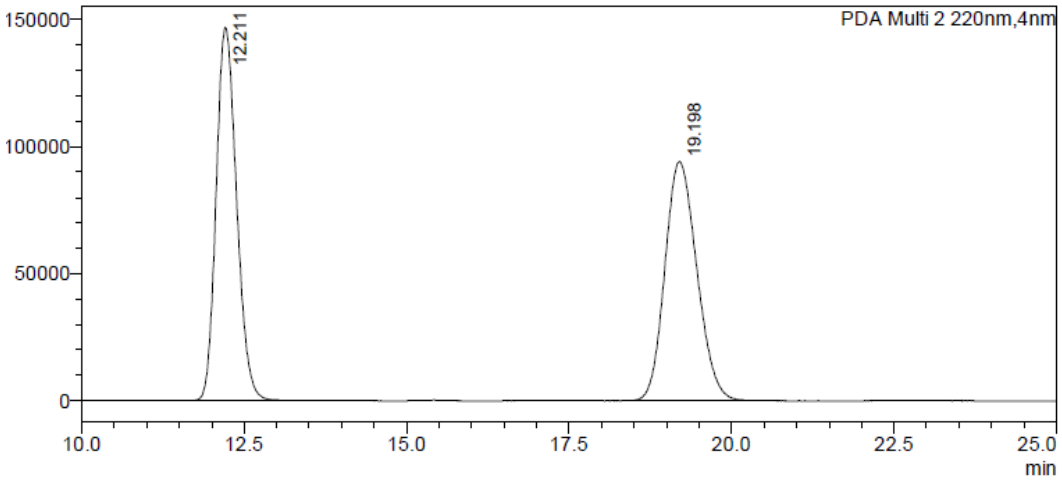


HPLC Data for 46: Chiralcel OD-H (95:5 hexane : IPA, flow rate 1.0 mLmin^{-1}, 220 nm, 30 °C) t_R (*S*,*S*): 12.4 min, t_R (*R*,*R*) : 19.3 min, 10.671:89.329 er. s=16.8



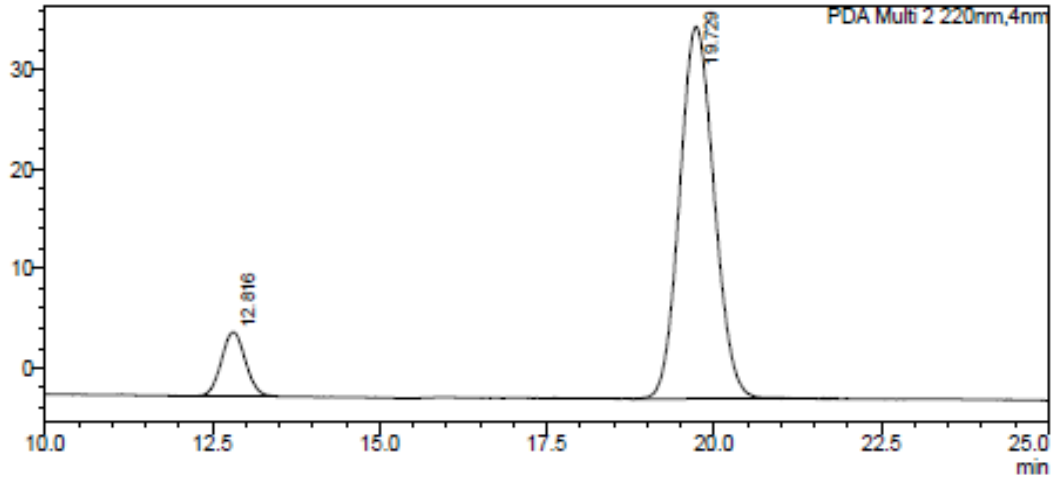
PDA Ch2 220nm

Peak#	Ret. Time	Area%
1	12.211	49.986
2	19.198	50.014
Total		100.000

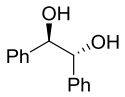


PDA Ch2 220nm

Peak#	Ret. Time	Area%
1	12.816	10.671
2	19.729	89.329
Total		100.000

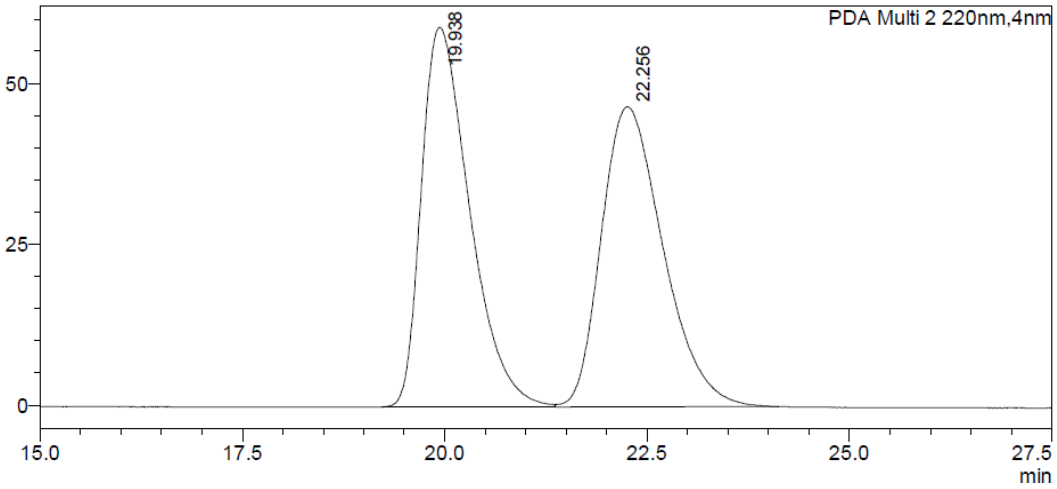


HPLC Data for 47 (following hydrolysis to alcohol 45): Chiralcel OJ-H (93:7 hexane : IPA, flow rate 1.0 mLmin^{-1}, 220 nm, 30 °C) t_R (*S*,*S*): 20.7 min, t_R (*R*,*R*): 23.6 min, 0.708:99.292 er.



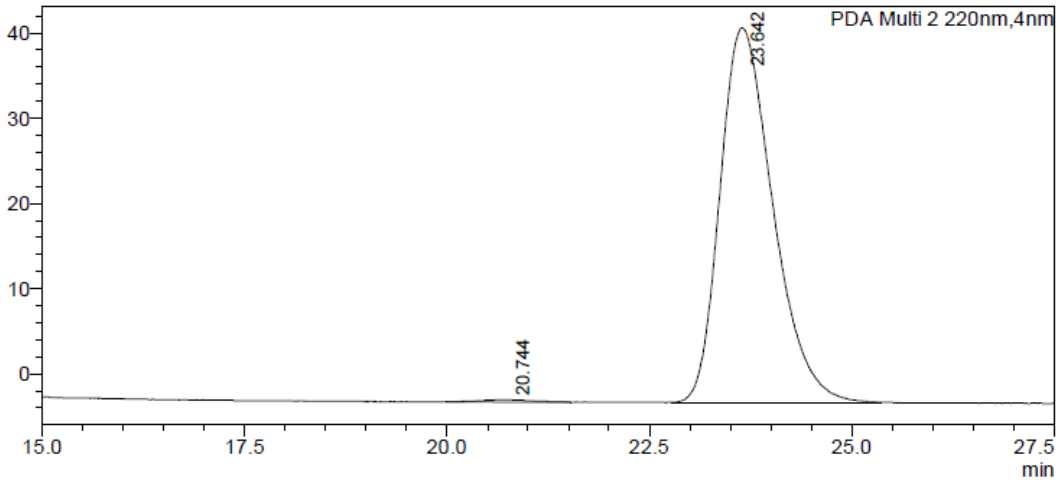
PDA Ch2 220nm

Peak#	Ret. Time	Area%
1	19.938	49.764
2	22.256	50.236
Total		100.000



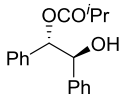
PDA Ch2 220nm

Peak#	Ret. Time	Area%
1	20.744	0.708
2	23.642	99.292
Total		100.000



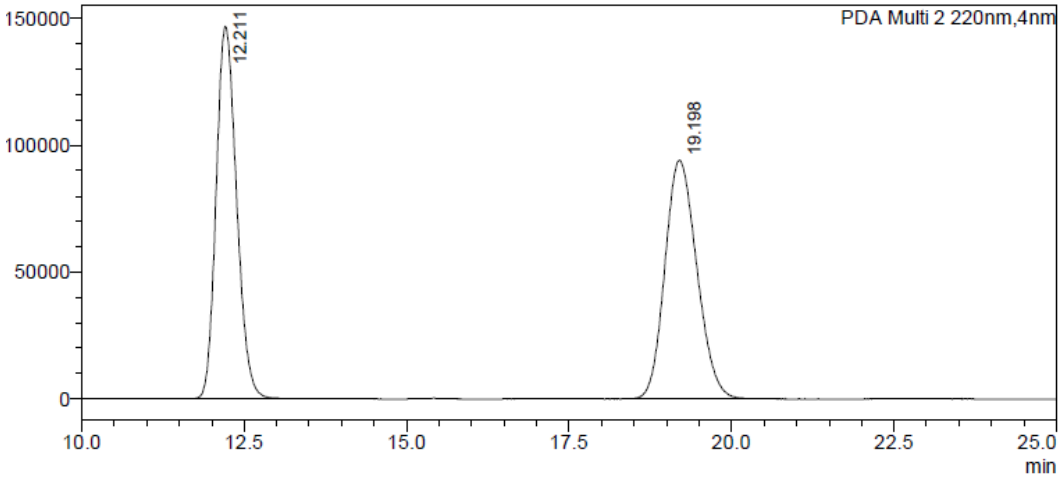
KR of (±)-46

HPLC Data for 46: Chiralcel OD-H (95:5 hexane : IPA, flow rate 1.0 mLmin^{-1}, 220 nm, 30 °C) t_R (*S*,*S*): 12.6 min, t_R (*R*,*R*): 19.6 min, 91.580:8.420 er. *s*=108.1



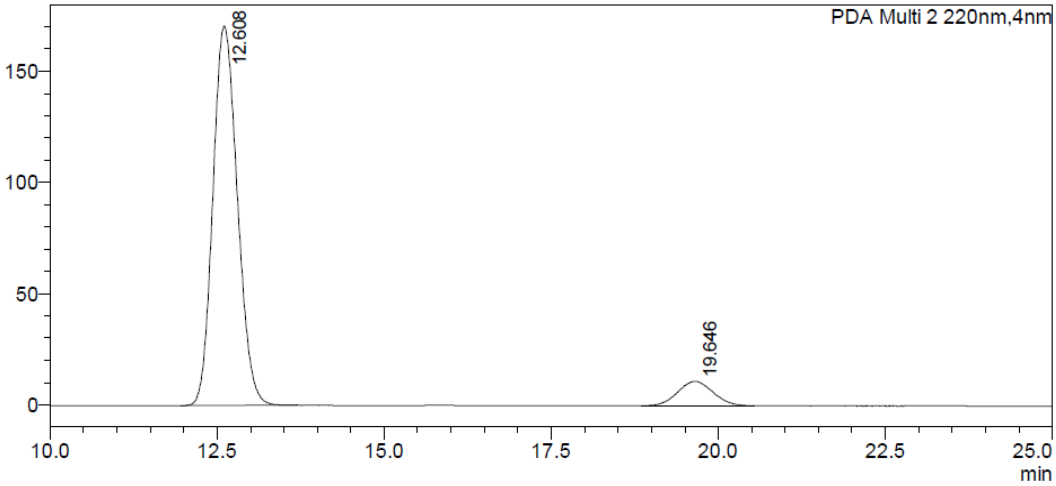
PDA Ch2 220nm

Peak#	Ret. Time	Area%
1	12.211	49.986
2	19.198	50.014
Total		100.000

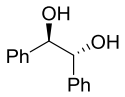


PDA Ch2 220nm

Peak#	Ret. Time	Area%
1	12.608	91.580
2	19.646	8.420
Total		100.000

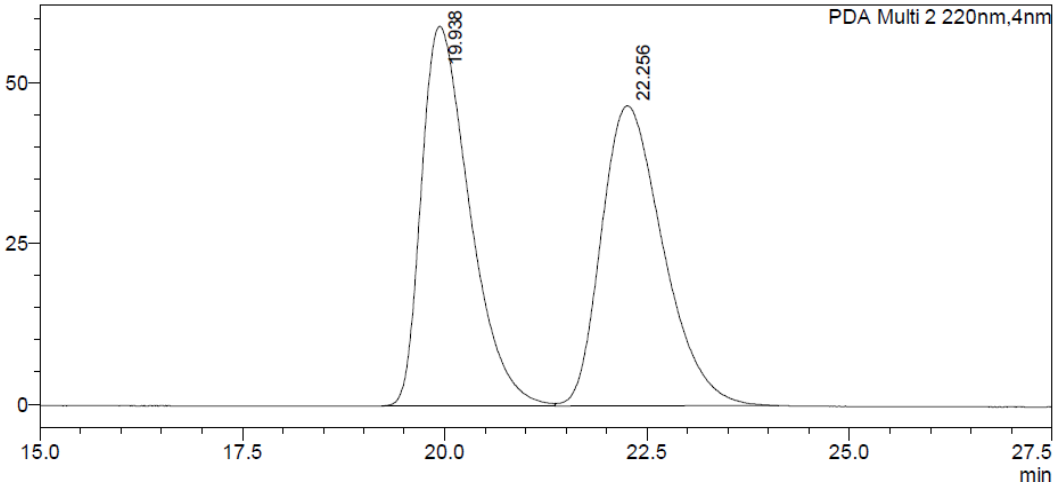


HPLC Data for 47 (following hydrolysis to alcohol 45): Chiralcel OJ-H (93:7 hexane : IPA, flow rate 1.0 mLmin^{-1}, 220 nm, 30 °C) t_R (*S*,*S*): 20.5 min, t_R (*R*,*R*) : 23.4 min, 2.364:97.636 er.



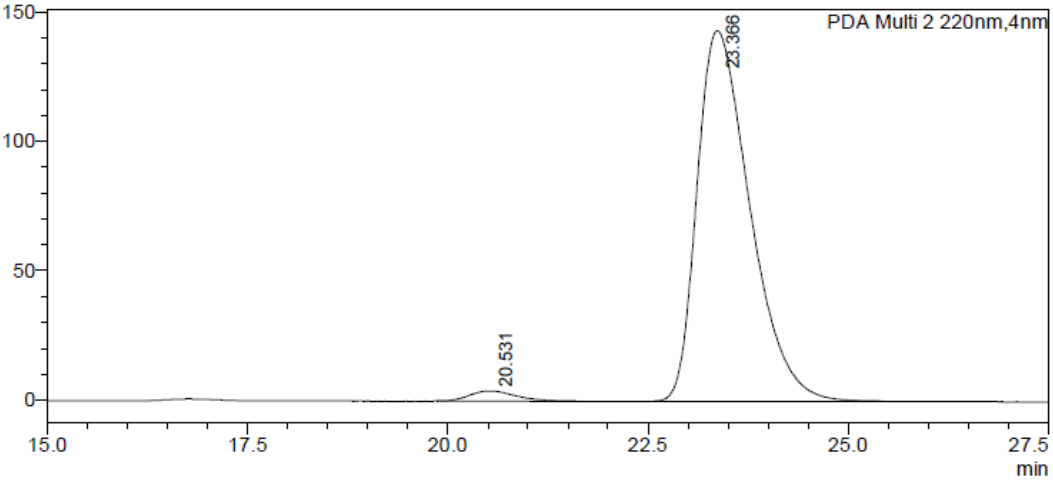
PDA Ch2 220nm

Peak#	Ret. Time	Area%
1	19.938	49.764
2	22.256	50.236
Total		100.000



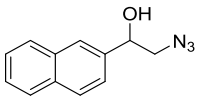
PDA Ch2 220nm

Peak#	Ret. Time	Area%
1	20.531	2.364
2	23.366	97.636
Total		100.000



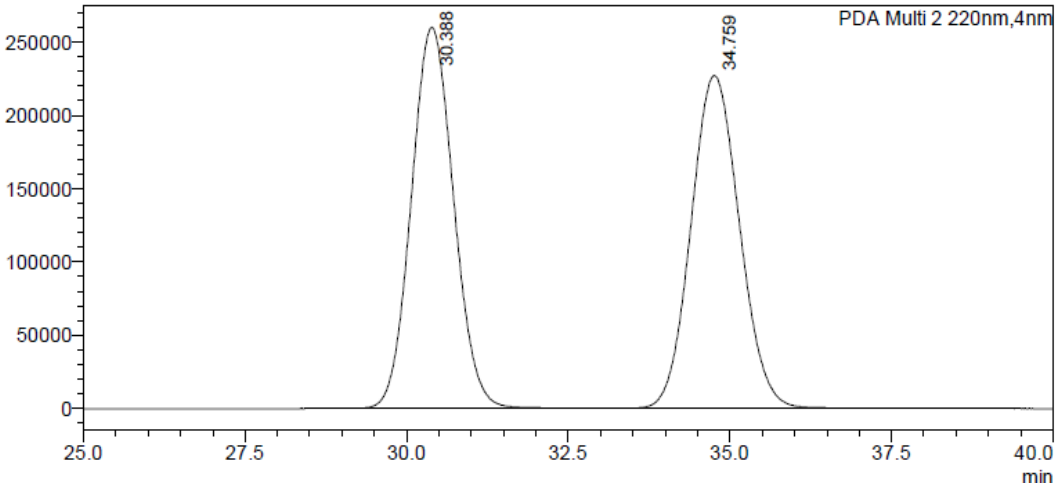
(*S*)-Pronithalol KR

HPLC Data for 48: Chiralcel OD-H (95:5 hexane : IPA, flow rate 1.0 mLmin^{-1}, 220 nm, 30 °C) t_R (*R*): 30.9 min, t_R (*S*): 35.2 min, 0.014:99.986 er. s=58.9



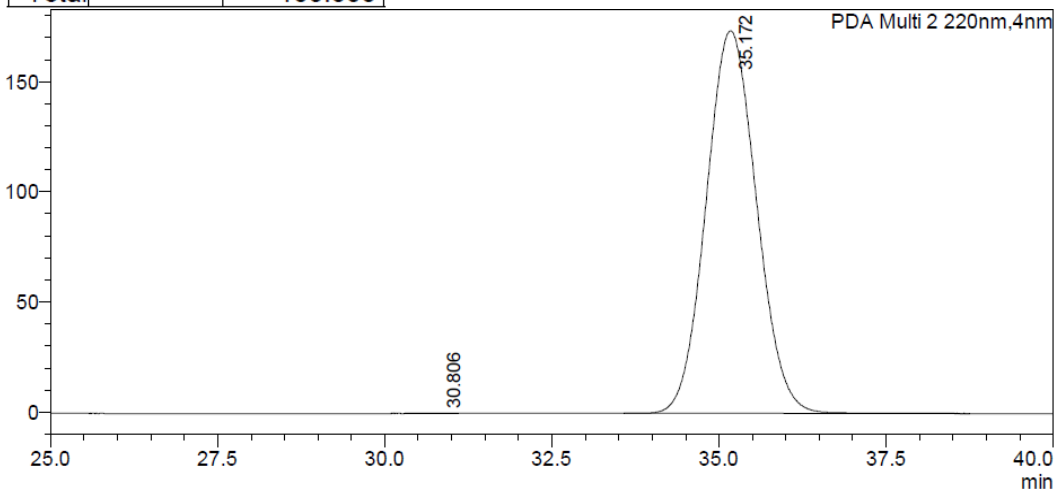
PDA Ch2 220nm

Peak#	Ret. Time	Area%
1	30.388	50.015
2	34.759	49.985
Total		100.000

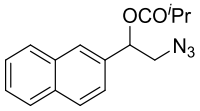


PDA Ch2 220nm

Peak#	Ret. Time	Area%
1	30.806	0.014
2	35.172	99.986
Total		100.000

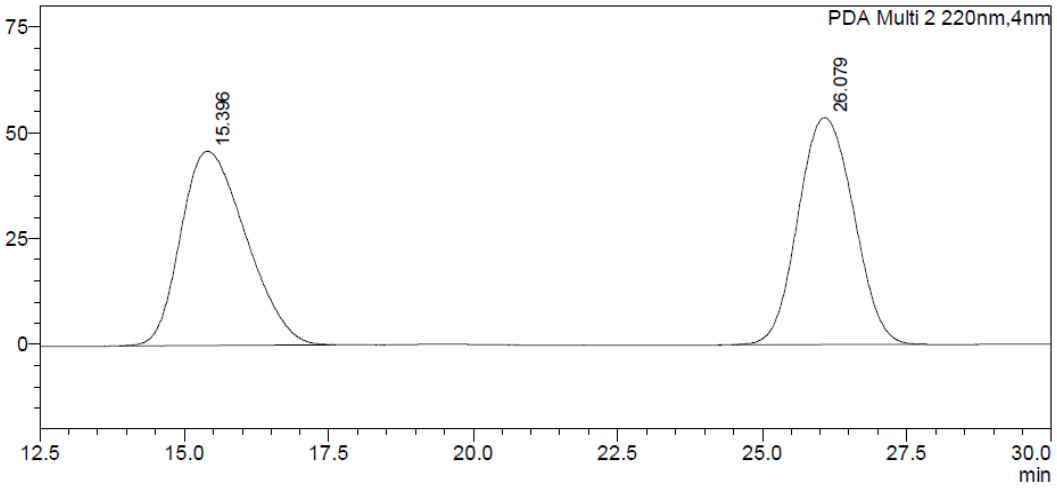


HPLC Data for S28: Chiralpak AD-H (99.8:0.2 hexane : IPA, flow rate 1.0 mLmin^{-1}, 220 nm, 30 °C) t_R (*S*): 15.4 min, t_R (*R*): 25.9 min, 12.445:87.555 er.



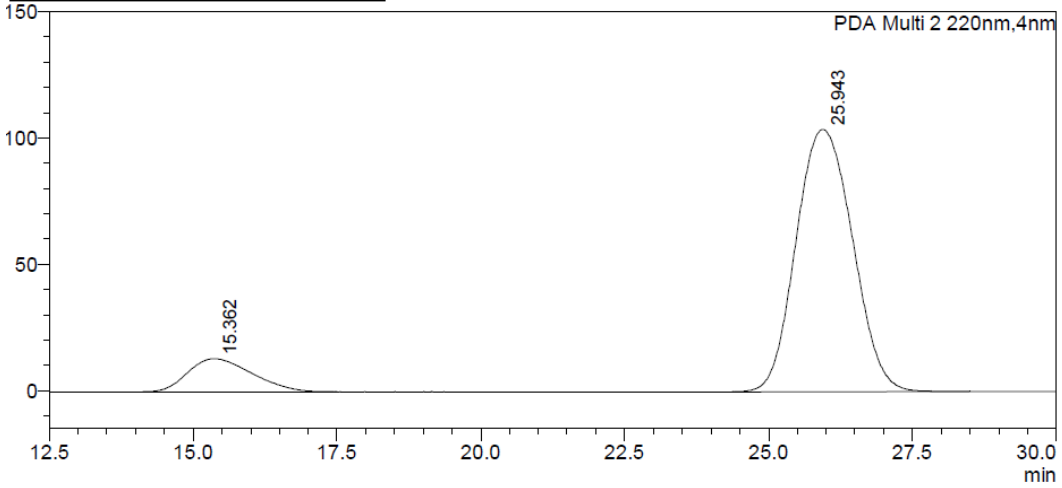
PDA Ch2 220nm

Peak#	Ret. Time	Area%
1	15.396	50.105
2	26.079	49.895
Total		100.000

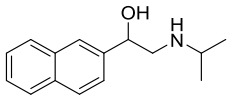


PDA Ch2 220nm

Peak#	Ret. Time	Area%
1	15.362	12.445
2	25.943	87.555
Total		100.000

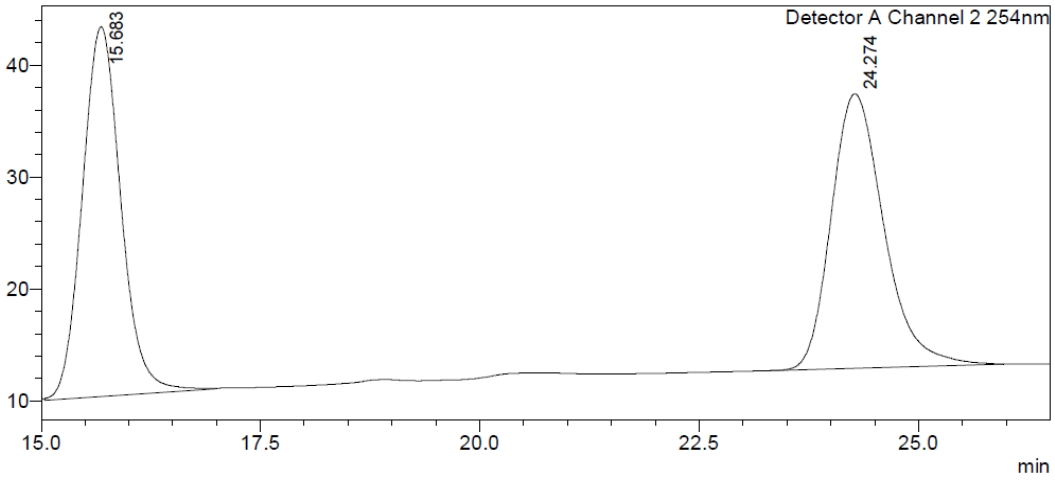


HPLC Data for 49: Chiralpak IC (94.9:5.0:0.1 hexane : IPA : diethylamine, flow rate 0.7 mL min^{-1} , 254 nm, 30 °C) t_R (*R*): 15.9 min, t_R (*S*): 24.5 min, 0.310:99.690 er



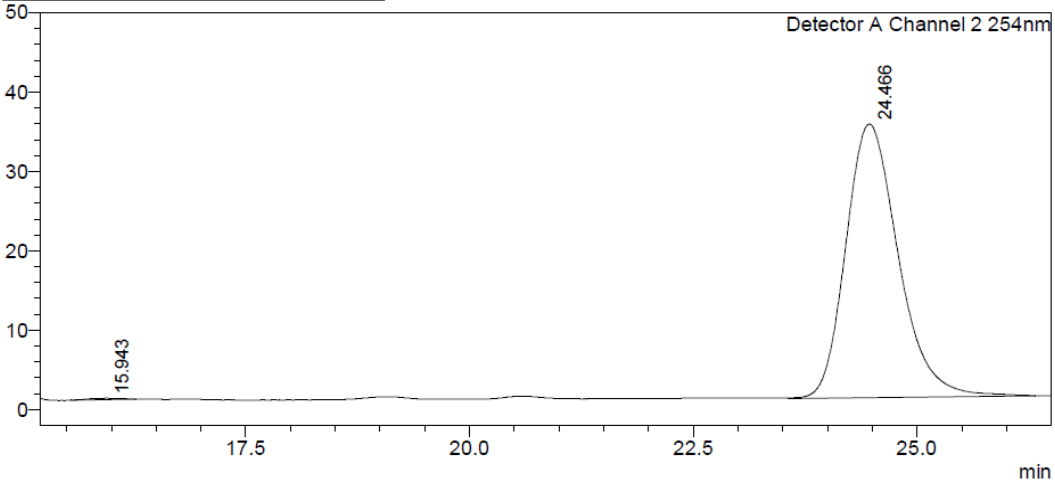
Detector A Channel 2 254nm

Peak#	Ret. Time	Area%
1	15.683	49.700
2	24.274	50.300
Total		100.000



Detector A Channel 2 254nm

Peak#	Ret. Time	Area%
1	15.943	0.310
2	24.466	99.690
Total		100.000



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