Horeau Amplification in the Sequential Acylative Kinetic Resolution of (±)-1,2-Diols and (±)-1,3-Diols in Flow

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Experimental Procedures

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

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

Analytical thin layer chromatography was performed on pre-coated aluminium plates (Kieselgel 60 F_{254} silica). TLC visualisation was carried out with ultraviolet light (254 nm), followed by staining with a 1% aqueous $KMnO_4$ solution. Manual column chromatography was performed in glass columns fitted with porosity 3 sintered discs over Kieselgel 60 silica using the solvent system stated. HPLC analyses were obtained using either a Shimadzu HPLC consisting of a DGU-20A5 degassing unit, LC-20AT liquid chromatography pump, SIL-20AHT autosampler, CMB-20A communications bus module, SPD-M20A diode array detector and a CTO-20A column oven; or a Shimadzu HPLC consisting of a DGU-20A5R degassing unit, LC-20AD liquid chromatography pump, SIL-20AHT autosampler, SPD-20A UV/Vis detector and a CTO-20A column oven. Separation was achieved using DAICEL CHIRALCEL OD-H and OJ-H columns or DAICEL CHIRALPAK ID, IC and AS-H columns. All HPLC traces of enantiomerically-enriched compounds were compared with authentic racemic spectra.

^{1}H, ^{13}C, and ^{19}F nuclear magnetic resonance (NMR) spectra were acquired on either a Bruker Avance II 400 (^{1}H 400 MHz; ^{13}C 101 MHz, ^{19}F 377 MHz) or a Bruker Avance II 500 (^{1}H 500 MHz; ^{13}C 126 MHz, ^{19}F 470 MHz) spectrometer at ambient temperature in the deuterated solvent stated. All chemical shifts are quoted in parts per million (ppm) and referenced to the residual solvent peak. All coupling constants, *J*, are quoted in Hz. Multiplicities are indicated by: s (singlet), brs (broad singlet), d (doublet), t (triplet), q (quartet), hept (heptet), dd (doublet of doublets), dt (doublet of triplets), qd (quartet of doublets), td (triplet of doublets), ddd (doublet of doublet of doublets), dp (doublet of pentets) and m (multiplet). The abbreviation Ar is used to denote aromatic, Ph to denote phenyl, Et

to denote ethyl and *i*-Pr to denote isopropyl. NMR peak assignments of monoester **3** were confirmed using 2D $^{1}H-^{13}C$ heteronuclear single quantum coherence (HSQC) and 2D $^{1}H-^{13}C$ heteronuclear multiple-bond correlation spectroscopy (HMBC).

Absolute configuration of monoester **3** were determined following hydrolysis to the corresponding diol and through CSP-HPLC analysis.

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

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

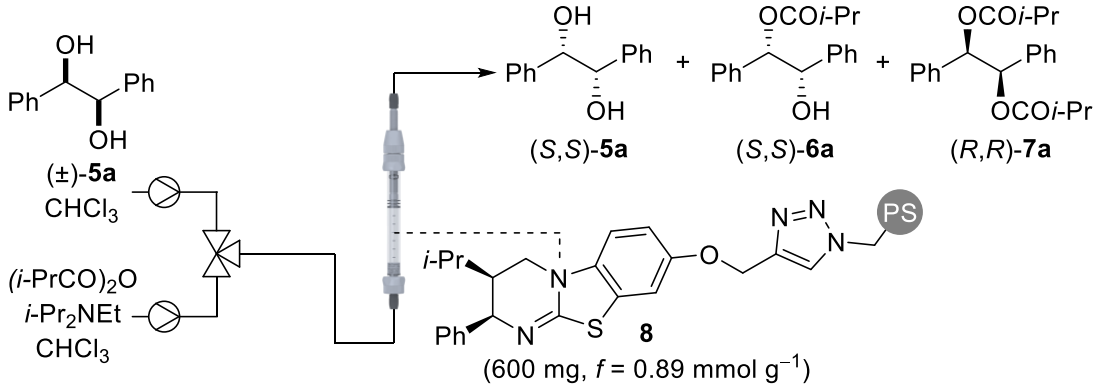
Infrared spectra were recorded on a Shimadzu IRAffinity-1 Fourier transform IR spectrophotometer fitted with a Specac Quest ATR accessory (diamond puck). Spectra were recorded of thin films, with characteristic absorption wave numbers (vmax) reported in cm^{-1}.

Continuous flow experiments were performed using the same polystyrene-supported (2*R*,3*S*)-HyperBTM (**8**) catalyst batch used in previous experiments.[1] The catalyst resin was packed into an Omnifit column [borosilicate glass; length = 100 mm (70 mm adjustable bed height); internal diameter = 10 mm; maximum bed volume 5.6 mL]. A Gilson 305 HPLC pump was used to pump solvent for column equilibration and regeneration. A Legato 200 series syringe pump (World Precision Instruments) was used to deliver solutions of reagents.

For the kinetic resolution of monoesters, selectivity factors (*s*) were calculated according to Kagan's equation: $s = \ln((1-c)(1-ee_{rsm}))/\ln((1-c)(1+ee_{rsm})) = \ln(1-c(1+ee_{prod}))/\ln(1-c(1-ee_{prod}))$, wherein c is conversion of the reaction, ee_{prod} is the enantiomeric excess of diester product and ee_{rsm} is the enantiomeric excess of the recovered monoester. Conversions were calculated by the following equation: $c = ee_{rsm}/(ee_{prod}+ee_{rsm})$.[2]

Additional results of the optimization study for the Sequential Acylative Kinetic Resolution of (±)-5a (Table S1)

Table S1 Full optimization study for the sequential acylative kinetic resolution of (±)-**5a** in flow-mode conditions.[a]

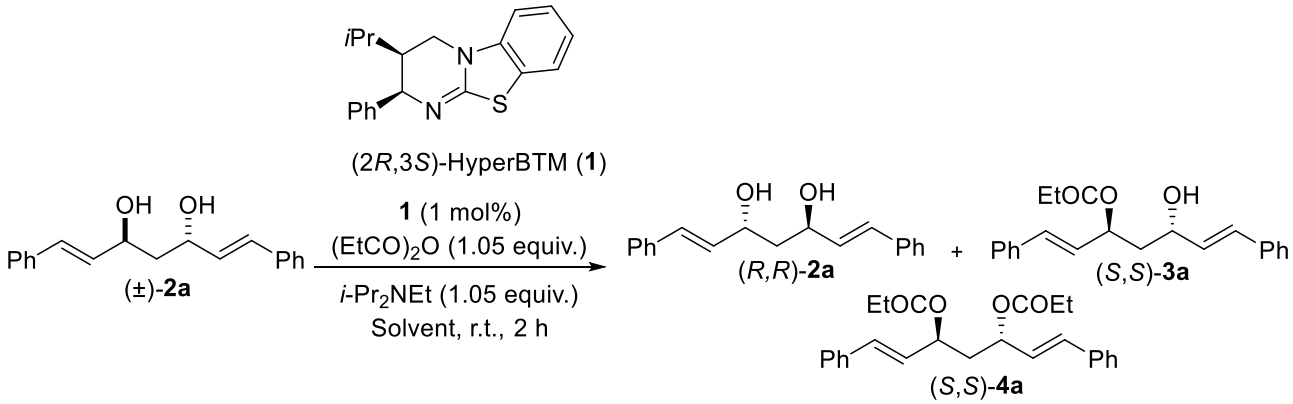


Entry	(±)-**5a** [M]	(*i*-PrCO)$_2$O [M]	*i*-Pr$_2$NEt [M]	Product Ratio (**5a**:**6a**:**7a**)[b]	**5a** er (*S*,*S*):(*R*,*R*)[c]	**6a** er (*S*,*S*):(*R*,*R*)[c]	**7a** er (*R*,*R*):(*S*,*S*)[c]
1	0.2	0.3	0.32	17:39:44	99:1	85:15	95:5
2[d]	0.2	0.3	0.32	12:39:49	>99:1	92:8	97:3
3[e]	0.2	0.3	0.32	4:43:53	-	98:2	93:7
4	0.1	0.15	0.16	10:44:46	>99:1	75:25	94:6
5	0.2	0.4	0.44	1:40:59	-	>99:1	75:25
6	0.2	0.35	0.35	7:41:52	99:1	97:3	90:10
7[f]	0.2	0.3	0.32	14:39:47	99:1	88:12	96:4
8[g]	0.2	0.3	0.32	9:39:52	>99:1	96:4	94:6
9[h]	0.2	0.3	0.32	10:45:45	99:1	89:11	94:6
10[i]	0.2	0.35	0.35	7:41:52	>99:1 (3%)[j]	98:2 (38%)[j]	94:6 (47%)[j]
11[i,f]	**0.2**	**0.35**	**0.35**	**7:40:53**	**>99:1 (3%)**[j]	**98:2 (35%)**[j]	**94:6 (48%)**[j]

[a]Conditions: (±)-**5a** (0.5 mmol), r.t., flow rate 0.1 mL min^{-1}. [b]Determined by ^{1}H NMR spectroscopic analysis of the crude reaction product. [c]Determined by CSP-HPLC analysis. [d]Flow rate 0.05 mL min^{-1}. [e]Flow rate 0.04 mL min^{-1}. [f]Flow set-up: diol and anhydride in one syringe and base in the other one. [g]Flow set-up: all the reagents together in one syringe. [h]Flow set-up: diol and base in one syringe and anhydride in the other one. [i]Conditions: (±)-**5a** (2.0 mmol), r.t., flow rate 0.1 mL min^{-1}. [j]Isolated yield.

Additional results of the optimization study for the Sequential Acylative Kinetic Resolution of (±)-2a in homogeneous conditions (Table S2)

Table S2 Optimization study for the sequential acylative kinetic resolution of (±)-**2a** with homogeneous HyperBTM.[a]

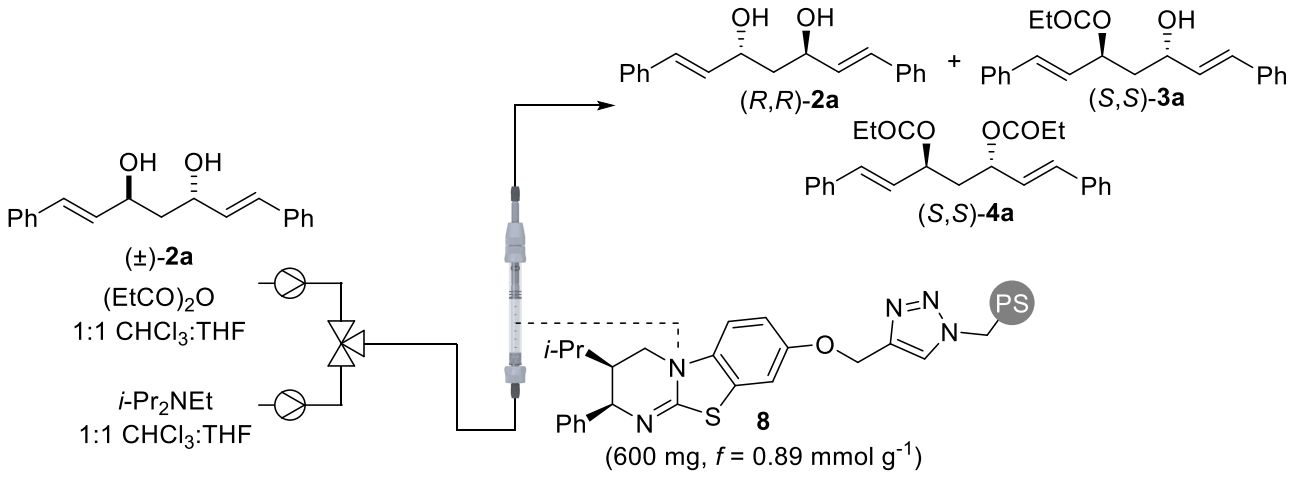


Entry	Solvent	Product Ratio (**2a**:**3a**:**4a**)[b]	**2a** *er* (*R*,*R*):(*S*,*S*)[c]	**4a** *er* (*S*,*S*):(*R*,*R*)[c]
1	CH_2Cl_2	40:8:42	99:1	90:10
2	$CHCl_3$:THF (1:1)	36:6:46	>99:1	87:13

[a]Conditions: (±)-**2a** (0.2 M), $(EtCO)_2O$ (1.05 equiv.), *i*-Pr_2NEt (1.05 equiv), r.t. 2 h. [b]Isolated yield. [c]Determined by CSP-HPLC analysis.

Optimization study for the Sequential Acylative Kinetic Resolution of (±)-2a (Table S3)

Table S3. Optimization study for the sequential acylative kinetic resolution of (±)-**2a** in flow-mode conditions.[a]



Entry	(±)-**2a** [M]	$(EtCO)_2O$ [M]	*i*-Pr_2NEt [M]	Product Ratio (**2a**:**3a**:**4a**)[b]	**2a** er (*R*,*R*):(*S*,*S*)[c]	**4a** er (*S*,*S*):(*R*,*R*)[c]
1	0.2	0.210	0.210	19:7:21[d]	99:1	89:11
2[e]	**0.1**	**0.105**	**0.105**	**39:22:39**	**98:2**	**96:4**
3[f]	0.1	0.105	0.105	39:19:42	98:2	96:4
4[g]	0.1	0.120	0.120	38:21:41	96:4	96:4
5[e,g]	0.1	0.105	0.105	38:27:35	99:1	94:6

[a]Conditions: (±)-**2a** (0.5 mmol), r.t., flow rate 0.1 ml min^{-1}. [b]Determined by ^1H NMR spectroscopic analysis of the crude reaction product. [c]Determined by CSP-HPLC analysis. [d]Isolated yield. [e]Flow rate 0.2 ml min^{-1}. [f]Flow rate 0.16 ml min^{-1}. [g]Flow set-up: diol and base in one syringe and anhydride in the other one.

Synthesis of (±)-*syn*-1,2-diols 5, monoesters (±)-6, and diesters (±)-7

(±)-*syn*-1,2-Diols **5a-5g**, monoesters (±)-**6** and diesters (±)-**7** were synthesized following a literature procedure.[3] Spectral data were in accordance with the literature.[4,5]

Synthesis of (±)-*anti*-1,3-diols 2 and monoesters (±)-3

(±)-*anti*-1,3-Diols **2** and monoester (±)-**3** were synthesised following a literature procedure.[6] Spectral data were in accordance with the literature.[6]

General procedure for the Sequential Acylative Kinetic Resolution of (±)-*syn*-1,2-diols 5 in flow-mode conditions

A packed bed reactor consisting of a vertically-mounted Omnifit glass chromatography column [borosilicate glass; length = 100 mm (70 mm adjustable bed height); internal diameter = 10 mm; maximum bed volume 5.6 mL], with a glass cooling jacket was loaded with PS-HyperBTM resin **8** (600 mg; f = 0.89 mmol g^{-1}). The resin was allowed to swell to its maximum volume by pumping $CHCl_3$ at 1 mL min^{-1} for 30 min at r.t. using a Gilson 305 HPLC pump. Two syringes were used to inject reagents using a Legato 200 series syringe pump by World Precision Instruments. The first syringe was filled with a solution of the appropriate diol (±)-**5** (2.0 mmol, 1.0 equiv.) and $(i\text{-PrCO})_2O$ (3.5 mmol, 1.75 equiv.) in $CHCl_3$ (10 mL total volume) and the second syringe with $i\text{-Pr}_2NEt$ (3.5 mmol, 1.75 equiv.) 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}. After complete addition of the reagents from the syringes, a Gilson 305 HPLC pump was connected, and $CHCl_3$ was pumped at 100 μL min^{-1} for 30 min to ensure elution of the products. A solution of 10% MeOH in $CHCl_3$ was then pumped at 200 μL min^{-1} for 30 min to wash the column and avoid cross contamination. The column was then prepared for the next KR by pumping $CHCl_3$ at 200 μL min^{-1} for 30 min. The mixture was diluted with CH_2Cl_2 and washed sequentially with HCl (1 M), saturated $NaHCO_3$ and brine. The organic layer was dried (Na_2SO_4), filtered and concentrated to give the crude products which were purified by column chromatography.

Hydrolysis of diesters and monoesters

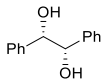
In some instances it was difficult to find conditions to separate the enantiomers of diesters or monoesters by HPLC using a chiral support, and therefore these products were hydrolysed to the diol prior to HPLC analysis: $LiOH{\bullet}H_2O$ (3 equiv.) was added to a solution of the diester or monoester (1 equiv.) in MeOH (0.3 M) and allowed to stir at 50 °C until completion, based on TLC analysis. The

mixture was diluted with EtOAc and washed sequentially with HCl (1 M), saturated $NaHCO_3$ and brine. The organic layer was dried (Na_2SO_4), filtered and concentrated to give the diol product.

Kinetic Resolution of (±)-1,2-diphenylethane-1,2-diol ((±)-5a)

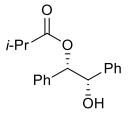
According to the **General Procedure**, (±)-1,2-diphenylethane-1,2-diol (428 mg, 2.0 mmol) and (*i*-PrCO)$_2$O (580 µL, 3.5 mmol, 1.75 equiv.) in $CHCl_3$ (10 mL total volume), and *i*-Pr$_2$NEt (610 µL, 3.5 mmol, 1.75 equiv.) in $CHCl_3$ (10 mL total volume) gave crude products that were purified by column chromatography (80:20 to 60:40 Hexane:EtOAc) to give:

(1*S*,2*S*)-1,2-Diphenylethane-1,2-diol (5a)



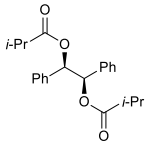
(13 mg, 3%) as a colourless solid with spectroscopic data in accordance with the literature.[3,4] **mp** 118−119 °C {Lit.[3] 121 °C}; $[\alpha]_D^{20}$ −87.1 (*c* 0.55 in $CHCl_3$) {Lit.[3] (*ent*, >99:1 er) $[\alpha]_D^{20}$ +88.0 (*c* 0.15 in $CHCl_3$)}; **Chiral HPLC analysis** Chiralpak ID (90:10 Hexane:IPA, flow rate 1.0 mL min^{-1}, 211 nm, 30 °C) t_R(1*R*,2*R*): 11.6 min, t_R(1*S*,2*S*): 14.9 min, 0.27:99.73 er; **^{1}H NMR** (400 MHz, $CDCl_3$) δ_H: 2.74 – 2.97 (brs, 2H, OH), 4.74 (s, 2H, C*H*OH), 7.10 – 7.20 (m, 4H, ArH), 7.20 – 7.28 (m, 6H, ArH).

(1*S*,2*S*)-2-Hydroxy-1,2-diphenylethyl isobutyrate (6a)



(200 mg, 35%) as a colourless solid with spectroscopic data in accordance with the literature.[5] **mp** 80−82 °C; $[\alpha]_D^{20}$ −6.2 (*c* 0.50 in $CHCl_3$) {Lit.[3] (*ent*, >99:1 er) $[\alpha]_D^{20}$ +2.6 (*c* 0.50 in $CHCl_3$)}; **Chiral HPLC analysis** Chiralcel OD-H (95:5 Hexane:IPA, flow rate 1 mL min^{-1}, 211 nm, 30 °C) t_R (1*S*,2*S*): 11.9 min, t_R (1*R*,2*R*): 17.5 min, 97.90:2.10 er; **^{1}H NMR** (400 MHz, $CDCl_3$) δ_H: 1.18 (d, *J* 7.0 Hz, 3H, CH_3), 1.19 (d, *J* 7.0 Hz, 3H, CH_3), 2.53 (d, *J* 3.6 Hz, 1H, OH), 2.66 (hept, *J* 7.0 Hz, 1H, C*H*CH_3), 4.96 (dd, *J* 7.1, 3.3 Hz, 1H, C*H*OH), 5.88 (d, *J* 7.1 Hz, 1H, C*H*OC(O)*i*-Pr), 7.07 – 7.22 (m, 4H, ArH), 7.22 – 7.28 (m, 6H, ArH).

(1*R*,2*R*)-1,2-Diphenylethane-1,2-diyl bis(2-methylpropanoate) (7a)



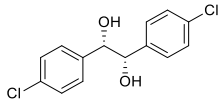
(340 mg, 48%) as a colourless solid with spectroscopic data in accordance with the literature.[5] **mp** 71−72 °C {Lit.[5] 73−74 °C}; $[\alpha]_D^{20}$ −22.9 (*c* 1.0 in $CHCl_3$) {Lit.[3] (*ent*, 97:3 er) $[\alpha]_D^{20}$ +20.2 (*c* 1.0 in $CHCl_3$)}; following hydrolysis to 1,2-diphenylethane-1,2-diol: **Chiral HPLC analysis** Chiralpak ID (90:10 Hexane:IPA, flow rate 1.0 mL min^{-1}, 211 nm, 30 °C) t_R(1*R*,2*R*): 12.7 min, t_R(1*S*,2*S*): 15.4

min, 94.45:5.55 er; **^{1}H NMR** (400 MHz, $CDCl_3$) δ_H: 1.16 (d, *J* 7.0 Hz, 6H, CH_3), 1.17 (d, *J* 7.0 Hz, 6H, CH_3), 2.60 (hept, *J* 7.0 Hz, 2H, C*H*CH_3), 6.07 (s, 2H, C*H*OC(O)*i*-Pr), 7.07 – 7.18 (m, 4H, ArH), 7.17 – 7.27 (m, 6H, ArH).

Kinetic Resolution of (±)-1,2-bis(4-chlorophenyl)ethane-1,2-diol ((±)-5b)

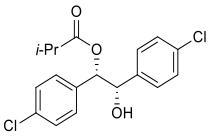
According to the **General Procedure**, (±)-1,2-bis(4-chlorophenyl)ethane-1,2-diol (567 mg, 2.0 mmol) and (*i*-PrCO)$_2$O (580 µL, 3.5 mmol, 1.75 equiv.) in $CHCl_3$ (10 mL total volume) and *i*-Pr$_2$NEt (610 µL, 3.5 mmol, 1.75 equiv.) in $CHCl_3$ (10 mL total volume) gave crude products that were purified by column chromatography (80:20 to 60:40 Hexane:EtOAc) to give:

(1*S*,2*S*)-1,2-bis(4-chlorophenyl)ethane-1,2-diol (5b)



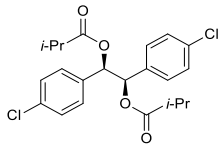
(45 mg, 8%) as a colourless solid with spectroscopic data in accordance with the literature.[7] **mp** 131−133 °C {Lit.[8] 126−128 °C}; $[\alpha]_D^{20}$ −183.0 (*c* 0.25 in $CHCl_3$) {Lit.[3] (*ent*, >99:1 er) $[\alpha]_D^{20}$ +112 (*c* 0.25 in $CHCl_3$)}; **Chiral HPLC analysis** Chiralpak AS-H (98:2 Hexane:IPA, flow rate 1 mL min^{-1}, 220 nm, 30 °C) t_R (1*S*,2*S*): 41.5 min, 0:100 er; **^{1}H NMR** (500 MHz, $CDCl_3$) δ_H: 2.91 (q, *J* 1.2 Hz, 2H, OH), 4.65 (t, *J* 1.2 Hz, 2H, C*H*OH), 7.00 – 7.09 (m, 4H, ArH), 7.18 – 7.27 (m, 4H, ArH).

(1*S*,2*S*)-1,2-bis(4-chlorophenyl)-2-hydroxyethyl isobutyrate (6b)



(240 mg, 34%) as a colourless solid with spectroscopic data in accordance with the literature.[3] **mp** 141−142 °C {Lit.[3] 143 °C}; $[\alpha]_D^{20}$ −34.5 (*c* 1.0 in $CHCl_3$) {Lit.[3] (*ent*, 99:1 er) $[\alpha]_D^{20}$ +35.0 (*c* 1.0 in $CHCl_3$)}; **Chiral HPLC analysis** Chiralpak AS-H (99.5:0.5 Hexane:IPA, flow rate 0.7 mL min^{-1}, 220 nm, 30 °C) t_R (1*S*,2*S*): 35.9 min, t_R (1*R*,2*R*): 46.7 min, 95.53:4.47 er; **^{1}H NMR** (400 MHz, $CDCl_3$) δ_H: 1.18 (d, *J* 7.0 Hz, 3H, CH_3), 1.20 (d, *J* 7.0 Hz, 3H, CH_3), 2.54 (d, *J* 3.4 Hz, 1H, OH), 2.65 (hept, *J* 7.0 Hz, 1H, C*H*CH_3), 4.91 (dd, *J* 7.3, 3.3 Hz, 1H, C*H*OH), 5.76 (d, *J* 7.3 Hz, 1H, C*H*OC(O)*i*-Pr), 6.98 – 7.13 (m, 4H, ArH), 7.19 – 7.28 (m, 4H, ArH).

(1*R*,2*R*)-1,2-bis(4-chlorophenyl)ethane-1,2-diyl bis(2-methylpropanoate) (7b)

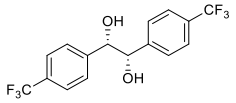


(381 mg, 45%) as a colourless oil with spectroscopic data in accordance with the literature.[3] $[\alpha]_D^{20}$ −5.3 (*c* 1.0 in $CHCl_3$) {Lit.[3] (*ent*, 95:5 er) $[\alpha]_D^{20}$ +4.8 (*c* 0.5 in $CHCl_3$)}; following hydrolysis to 1,2-bis(4-chlorophenyl)ethane-1,2-diol: **Chiral HPLC analysis** Chiralpak AS-H (98:2 Hexane:IPA, flow rate 1 mL min^{-1}, 220 nm, 30 °C) t_R (1*R*,2*R*): 31.0 min, t_R (1*S*,2*S*): 43.8 min, 93.62:6.38 er; **^{1}H NMR** (400 MHz, $CDCl_3$) δ_H: 1.15 (d, *J* 7.0 Hz, 6H, CH_3), 1.17 (d, *J* 7.0 Hz, 6H, CH_3), 2.59 (hept, *J* 7.0 Hz, 2H, C*H*CH_3), 5.99 (s, 2H, C*H*OC(O)*i*-Pr), 7.02 – 7.12 (m, 4H, ArH), 7.17 – 7.27 (m, 4H, ArH).

Kinetic Resolution of (±)-1,2-bis(4-chlorophenyl)ethane-1,2-diol ((±)-5c)

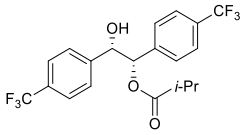
According to the **General Procedure**, (±)-1,2-bis(4-(trifluoromethyl)phenyl)ethane-1,2-diol (700 mg, 2.0 mmol) and (*i*-PrCO)$_2$O (580 µL, 3.5 mmol, 1.75 equiv.) in $CHCl_3$ (10 mL total volume) and *i*-Pr$_2$NEt (610 µL, 3.5 mmol, 1.75 equiv.) in $CHCl_3$ (10 mL total volume) gave crude products that were purified by column chromatography (80:20 to 60:40 Hexane:EtOAc) to give:

(1*S*,2*S*)-1,2-bis(4-(trifluoromethyl)phenyl)ethane-1,2-diol (5c)



(8 mg, 1%) as a colourless solid with spectroscopic data in accordance with the literature.[5] **mp** 135−137 °C {Lit.[9] 128−130 °C}; $[\alpha]_D^{20}$ −10.7 (*c* 0.7 in $CHCl_3$) {Lit.[3] (*ent*, >99:1 er) $[\alpha]_D^{20}$ +41.1 (*c* 1.0 in $CHCl_3$)}; **Chiral HPLC analysis** Chiralpak AS-H (97:3 Hexane:IPA, flow rate 1 mL min^{-1}, 220 nm, 30 °C) t_R (1*R*,2*R*): 11.3 min, t_R (1*S*,2*S*): 15.6 min, 0.27:99.73 er; **^{1}H NMR** (500 MHz, $CDCl_3$) δ_H: 2.98 (t, *J* 1.4 Hz, 2H, OH), 4.73 – 4.81 (m, 2H, C*H*OH), 7.26 (d, *J* 8.0 Hz, 4H, ArH), 7.54 (d, *J* 8.1 Hz, 4H, ArH); **^{19}F NMR** (377 MHz, $CDCl_3$) δ_F: −62.58.

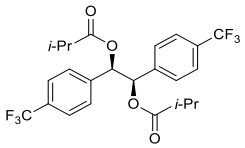
(1*S*,2*S*)-2-hydroxy-1,2-bis(4-(trifluoromethyl)phenyl)ethyl isobutyrate (6c)



(285 mg, 34%) as a colourless solid with spectroscopic data in accordance with the literature.[5] **mp** 147−148 °C {Lit.[5] 148 °C}; $[\alpha]_D^{20}$ −4.4 (*c* 1.0 in $CHCl_3$) {Lit.[5] (96:4 er) $[\alpha]_D^{23}$ −1.06 (*c* 1.01 in

$CHCl_3$)}; **Chiral HPLC analysis** Chiralcel OJ-H (95:5 Hexane:IPA, flow rate 1 mL min^{-1}, 220 nm, 30 °C) t_R (1*S*,2*S*): 7.7 min, 100:0 er; **^{1}H NMR** (400 MHz, $CDCl_3$) δ_H: 1.19 (d, *J* 7.1, 6H, CH_3), 2.58 (d, *J* 3.7 Hz, 1H, OH), 2.67 (hept, *J* 7.0 Hz, 1H, C*H*CH_3), 5.05 (dd, *J* 6.7, 3.7 Hz, 1H, C*H*OH), 5.91 (d, *J* 6.6 Hz, 1H, C*H*OC(O)*i*-Pr), 7.23 – 7.34 (m, 4H, ArH), 7.55 (dd, *J* 8.3, 2.1 Hz, 4H, ArH); **^{19}F NMR** (377 MHz, $CDCl_3$) δ_F: −62.61, −62.68.

(1*R*,2*R*)-1,2-bis(4-(trifluoromethyl)phenyl)ethane-1,2-diyl bis(2-methylpropanoate) (7c)

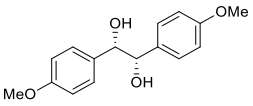


(520 mg, 53%) as a colourless solid with spectroscopic data in accordance with the literature.[5] **mp** 109−110 °C {Lit.[4] 108 °C}; $[\alpha]_D^{20}$ −9.0 (*c* 1.0 in $CHCl_3$) {Lit.[3] (*ent*, 97:3 er) $[\alpha]_D^{20}$ +11.0 (*c* 0.10 in $CHCl_3$)}; following hydrolysis to 1,2-bis(4-(trifluoromethyl)phenyl)ethane-1,2-diol: **Chiral HPLC analysis** Chiralpak AS-H (97:3 Hexane:IPA, flow rate 1 mL min^{-1}, 211 nm, 30 °C) t_R (1*R*,2*R*): 11.5 min, t_R (1*S*,2*S*): 15.8 min, 81.67:18.33 er; **^{1}H NMR** (400 MHz, $CDCl_3$) δ_H: 1.16 (d, *J* 7.0 Hz, 12H, CH_3), 2.55 – 2.69 (m, *J* 6.9 Hz, 2H, C*H*CH_3), 6.12 (s, 2H, C*H*OC(O)*i*-Pr), 7.34 – 7.22 (m, 4H, ArH), 7.46 – 7.62 (m, 4H, ArH); **^{19}F NMR** (377 MHz, $CDCl_3$) δ_F: −62.72.

Kinetic Resolution of (±)-1,2-bis(4-methoxyphenyl)ethane-1,2-diol ((±)-5d)

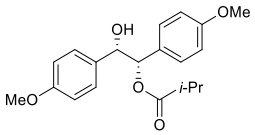
According to the **General Procedure**, (±)-1,2-diphenylethane-1,2-diol (548 mg, 2.0 mmol) and (*i*-PrCO)$_2$O (580 µL, 3.5 mmol, 1.75 equiv.), in $CHCl_3$ (10 mL total volume), and *i*-Pr_2NEt (610 µL, 3.5 mmol, 1.75 equiv.) in $CHCl_3$ (10 mL total volume) gave crude products that were purified by column chromatography (80:20 to 60:40 Hexane:EtOAc) to give:

(1*S*,2*S*)-1,2-bis(4-methoxyphenyl)ethane-1,2-diol (5d)



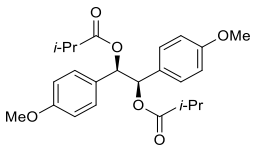
(33 mg, 6%) as a colourless solid with spectroscopic data in accordance with the literature.[10] **mp** 106−107 °C {Lit.[8] 118−119 °C}; $[\alpha]_D^{20}$ −102 (*c* 0.25 in $CHCl_3$) {Lit.[3] $[\alpha]_D^{20}$ (*ent*, >99:1 er) $[\alpha]_D^{20}$ +126 (*c* 0.25 in $CHCl_3$)}; **Chiral HPLC analysis** Chiralpak ID (80:20 Hexane:IPA, flow rate 1 mL min^{-1}, 211 nm, 30 °C) t_R (1*S*,2*S*): 24.09 min, 0:100 er; **^{1}H NMR** (400 MHz, $CDCl_3$) δ_H: 3.79 (s, 6H, OCH_3), 4.66 (s, 2H, C*H*OH), 6.74 – 6.84 (m, 4H, ArH), 7.02 – 7.11 (m, 4H, ArH).

(1*S*,2*S*)-2-hydroxy-1,2-bis(4-methoxyphenyl)ethyl isobutyrate (6d)



(254 mg, 37%) as a colourless solid with spectroscopic data in accordance with the literature.[3] **mp** 88−89 °C {Lit.[3] 88 °C}; $[\alpha]_D^{20}$ −26.7 (*c* 0.5 in $CHCl_3$) {Lit.[3] $[\alpha]_D^{20}$ (*ent*, 99:1 er) +23.6 (*c* 0.5 in $CHCl_3$)}; following hydrolysis to 1,2-bis(4-methoxyphenyl)ethane-1,2-diol: **Chiral HPLC analysis** Chiralpak ID (85:15 Hexane:IPA, flow rate 1 mL min^{-1}, 211 nm, 30 °C) t_R (1*R*,2*R*): 21.91 min, t_R (1*S*,2*S*): 36.53 min, 1.56:98.44 er; **^{1}H NMR** (400 MHz, $CDCl_3$) δ_H: 1.18 (d, *J* 7.0 Hz, 3H, CH_3), 1.20 (d, *J* 7.0 Hz, 3H, CH_3), 2.65 (hept, *J* 7.0 Hz, 1H, C*H*CH_3), 3.78 (d, *J* 3.8 Hz, 6H, OCH_3), 4.89 (d, *J* 7.8 Hz, 1H, C*H*OH), 5.78 (d, *J* 7.7 Hz, 1H, C*H*OC(O)*i*-Pr), 6.71 – 6.84 (m, 4H, ArH), 6.98 – 7.13 (m, 4H, ArH).

(1*R*,2*R*)-1,2-bis(4-methoxyphenyl)ethane-1,2-diyl bis(2-methylpropanoate) (7d)

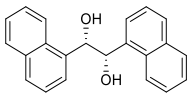


(380 mg, 45%) as a colourless oil with spectroscopic data in accordance with the literature.[5] **mp** 122−124 °C {Lit.[3] 126−128 °C}; $[\alpha]_D^{20}$ −25.0 (*c* 1.0 in $CHCl_3$) {Lit.[3] $[\alpha]_D^{20}$ (*ent*, 98:2 er) +19.3 (*c* 1.0 in $CHCl_3$)}; following hydrolysis to 1,2-bis(4-methoxyphenyl)ethane-1,2-diol: **Chiral HPLC analysis** Chiralpak ID (85:15 Hexane:IPA, flow rate 1 mL min^{-1}, 211 nm, 30 °C) t_R (1*R*,2*R*): 21.81 min, t_R (1*S*,2*S*): 38.61 min, 94.66:5.34 er; **^{1}H NMR** (400 MHz, $CDCl_3$) δ_H: 1.15 (d, *J* 7.0 Hz, 6H, CH_3), 1.18 (d, *J* 7.0 Hz, 6H, CH_3), 2.58 (hept, *J* 7.0 Hz, 2H, C*H*CH_3), 3.76 (s, 6H, OCH_3), 6.00 (s, 2H, C*H*OC(O)*i*-Pr), 6.68 – 6.83 (m, 4H, ArH), 7.00 – 7.11 (m, 4H, ArH).

Kinetic Resolution of (±)-1,2-di(naphthalen-1-yl)ethane-1,2-diol ((±)-5e)

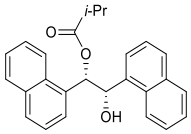
According to the **General Procedure**, (±)-1,2-di(naphthalen-1-yl)ethane-1,2-diol (628 mg, 2.0 mmol) and (*i*-PrCO)$_2$O (580 µL, 3.5 mmol, 1.75 equiv.), in 1:1 $CHCl_3$:THF mixture (10 mL total volume), and *i*-Pr_2NEt (610 µL, 3.5 mmol, 1.75 equiv.) in 1:1 $CHCl_3$:THF mixture (10 mL total volume) gave crude products that by column chromatography (80:20 to 60:40 Hexane:EtOAc) to give:

(1*S*,2*S*)-1,2-di(naphthalen-1-yl)ethane-1,2-diol (5e)



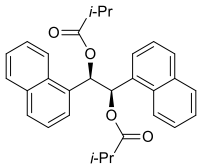
(125 mg, 20%) as a colourless solid with spectroscopic data in accordance with the literature.[5] **mp** 128−130 °C {Lit.[11] 122−124 °C}; $[\alpha]_D^{20}$ −28.6 (*c* 0.25 in $CHCl_3$) {Lit.[5] $[\alpha]_D^{24}$ (*ent*, 88:12 er) +40.7 (*c* 0.99 in THF)}; **Chiral HPLC** Chiralpak OJ-H (80:20 Hexane:IPA, flow rate 1 mL min^{-1}, 220 nm, 30 °C) t_R (1*S*,2*S*): 22.6 min, t_R (1*R*,2*R*): 33.7 min, 97.49:2.51 er; **^{1}H NMR** (500 MHz, $CDCl_3$) δ_H: 3.00 (dd, *J* 2.1, 1.1 Hz, 2H, OH), 5.76 – 5.86 (m, 2H, C*H*OH), 7.31 (ddd, *J* 8.3, 6.8, 1.4 Hz, 2H, ArH), 7.35 – 7.47 (m, 4H, ArH), 7.65 – 7.87 (m, 6H, ArH), 7.90 (dd, *J* 8.6, 1.2 Hz, 2H, ArH).

(1*S*,2*S*)-2-hydroxy-1,2-di(naphthalen-1-yl)ethyl isobutyrate (6e)



(177 mg, 23%) as a colourless oil with spectroscopic data in accordance with the literature.[5] **mp** 102−104 °C {Lit.[5] 110−111 °C}; $[\alpha]_D^{20}$ −49.4 (*c* 1.2 in $CHCl_3$) {Lit.[3] $[\alpha]_D^{20}$ (*ent*, >99:1 er) +57.2 (*c* 1.0 in $CHCl_3$)}; following hydrolysis to 1,2-di(naphthalen-1-yl)ethane-1,2-diol **Chiral HPLC** Chiralpak OJ-H (80:20 Hexane:IPA, flow rate 1 mL min^{-1}, 220 nm, 30 °C) t_R (1*S*,2*S*): 23.1 min, t_R (1*R*,2*R*): 33.5 min, 70.30:29.70 er; **^{1}H NMR** (400 MHz, $CDCl_3$) δ_H:. 1.18 (d, *J* 7.0 Hz, 3H, CH_3), 1.20 (d, *J* 7.0 Hz, 3H, CH_3), 2.66 (dp, *J* 31.8, 7.0 Hz, 1H, C*H*CH_3), 5.95 (d, *J* 6.5 Hz, 1H, C*H*OH), 6.94 (d, *J* 6.5 Hz, 1H, C*H*OC(O)*i*-Pr), 7.31 – 7.47 (m, 6H, ArH), 7.48 – 7.63 (m, 2H, ArH), 7.66 – 7.80 (m, 4H, ArH), 8.01 – 8.14 (m, 2H, ArH).

(1*R*,2*R*)-1,2-di(naphthalen-1-yl)ethane-1,2-diyl bis(2-methylpropanoate) (7e)

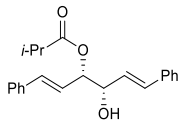


(372 mg, 41%) as a colourless solid with spectroscopic data in accordance with the literature.[5] **mp** 115−117 °C {Lit.[5] 113−114 °C}; $[\alpha]_D^{20}$ +71.2 (*c* 1.0 in $CHCl_3$) {Lit.[3] $[\alpha]_D^{20}$ (*ent*, 95:5 er) −67.5 (*c* 1.0 in $CHCl_3$)}; following hydrolysis to 1,2-di(naphthalen-1-yl)ethane-1,2-diol **Chiral HPLC analysis** Chiralpak OJ-H (80:20 Hexane:IPA, flow rate 1 mL min^{-1}, 220 nm, 30 °C) t_R (1*S*,2*S*): 23.9 min, t_R (1*R*,2*R*): 32.4 min, 6.41:93.59 er; **^{1}H NMR** (400 MHz, $CDCl_3$) δ_H: 1.17 (d, *J* 7.0 Hz, 6H, CH_3), 1.22 (d, *J* 7.0 Hz, 6H, CH_3), 2.55 – 2.75 (m, 2H, C*H*CH_3), 7.11 (s, 2H, C*H*OC(O)*i*-Pr), 7.24 (t, *J* 7.7 Hz, 2H, ArH), 7.39 (td, *J* 6.8, 5.9, 2.9 Hz, 6H, ArH), 7.60 – 7.77 (m, 4H, ArH), 8.11 – 8.31 (m, 2H, ArH).

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

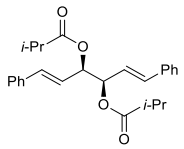
According to the **General Procedure**, (±)-(1*E*,5*E*)-1,6-diphenylhexa-1,5-diene-3,4-diol (532 mg, 2.0 mmol) and (*i*-PrCO)$_2$O (580 µL, 3.5 mmol, 1.75 equiv.), in $CHCl_3$ (10 mL total volume), and *i*-Pr$_2$NEt (610 µL, 3.5 mmol, 1.75 equiv.) in $CHCl_3$ (10 mL total volume) gave crude products that were purified by column chromatography (80:20 to 60:40 Hexane:EtOAc) to give:

(1*E*,3*S*,4*S*,5*E*)-4-hydroxy-1,6-diphenylhexa-1,5-dien-3-yl isobutyrate (6f)



(208 mg, 31%) as a colourless solid with spectroscopic data in accordance with the literature.[3] **mp** 102−103 °C {Lit.[3] 95−96 °C}; $[\alpha]_D^{20}$ −0.6 (*c* 0.4 in $CHCl_3$) {Lit.[3] $[\alpha]_D^{20}$ (*ent*, 77:23 er) +0.9 (*c* 1.0 in $CHCl_3$)}; **Chiral HPLC analysis** Chiralpak IC (95:5 Hexane:IPA, flow rate 1 mL min^{-1}, 254 nm, 30 °C) t_R (3*S*,4*S*): 18.0 min, t_R (3*R*,4*R*): 22.7 min, 80.96:19.04 er; **^{1}H NMR** (400 MHz, $CDCl_3$) δ_H: 1.21 (d, *J* 7.1 Hz, 3H, CH_3), 1.23 (d, *J* 7.1 Hz, 3H, CH_3), 2.66 (hept, *J* 6.8 Hz, 1H, C*H*CH_3), 4.53 (td, *J* 5.7, 1.5 Hz, 1H, C*H*OH), 5.54 (ddd, *J* 7.0, 5.6, 1.1 Hz, 1H, C*H*OC(O)*i*-Pr), 6.26 (ddd, *J* 16.0, 6.5, 5.2 Hz, 2H, PhCH=C*H*), 6.75 (dt, *J* 16.0, 1.5 Hz, 2H, PhC*H*=CH), 7.32 – 7.53 (m, 10H, ArH).

(1*E*,3*R*,4*R*,5*E*)-1,6-diphenylhexa-1,5-diene-3,4-diyl bis(2-methylpropanoate) (7f)



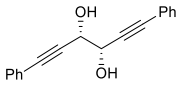
(422 mg, 52%) as a colourless solid with spectroscopic data in accordance with the literature.[3] **mp** 78−80 °C {Lit.[3] 84−85 °C}; $[\alpha]_D^{20}$ −4.57 (*c* 0.35 in $CHCl_3$) {Lit.[3] $[\alpha]_D^{20}$ (*ent*, 79:21 er) +10.8 (*c* 1.0 in $CHCl_3$)}; following hydrolysis to (1*E*,5*E*)-1,6-diphenylhexa-1,5-diene-3,4-diol: **Chiral HPLC analysis** Chiralpak ID (85:15 Hexane:IPA, flow rate 1 mL min^{-1}, 254 nm, 30 °C) t_R (1*S*,2*S*): 13.0 min, t_R (1*R*,2*R*): 14.7 min, 35.00:65.00 er; **^{1}H NMR** (400 MHz, $CDCl_3$) δ_H: 1.17 (d, *J* 7.1 Hz, 12H, C*H*$_3$), 2.56 – 2.71 (m, *J* 7.0 Hz, 2H, C*H*CH_3), 5.62 – 5.75 (m, 2H, C*H*OC(O)*i*-Pr), 6.08 – 6.23 (m, 2H, PhCH=C*H*), 6.72 (d, *J* 15.9 Hz, 2H, PhC*H*=CH), 7.23 – 7.43 (m, 10H, ArH).

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

According to the **General Procedure**, (±)-1,6-diphenylhexa-1,5-diyne-3,4-diol (525 mg, 2.0 mmol) and (*i*-PrCO)$_2$O (580 µL, 3.5 mmol, 1.75 equiv.), in $CHCl_3$ (10 mL total volume), and *i*-Pr$_2$NEt (610

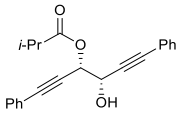
µL, 3.5 mmol, 1.75 equiv.) in $CHCl_3$ (10 mL total volume) gave crude products that were purified by by column chromatography (80:20 to 60:40 Hexane:EtOAc) to give:

(3*S*,4*S*)-1,6-diphenylhexa-1,5-diyne-3,4-diol (5g)



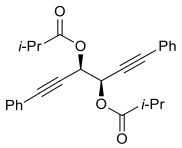
(10 mg, 2%) as a colourless solid with spectroscopic data in accordance with the literature.[3] **mp** 74−77 °C {Lit.[3] 78−79 °C}; $[\alpha]_D^{20}$ −101 (*c* 0.1 in $CHCl_3$) {Lit.[3] $[\alpha]_D^{20}$ (*ent*, >99:1 er) +97.2 (*c* 0.25 in $CHCl_3$)}; **Chiral HPLC analysis** Chiralcel OJ-H (80:20 Hexane:IPA, flow rate 1 mL min^{-1}, 211 nm, 30 °C) t_R (3*S*,4*S*): 13.6 min, t_R (3*R*,4*R*): 19.1 min, 77.37:22.63 er; **^{1}H NMR** (400 MHz, $CDCl_3$) δ_H: 4.77 (s, 2H, C*H*OH), 7.31 – 7.40 (m, 6H, ArH), 7.46 – 7.53 (m, 4H, ArH).

(3*S*,4*S*)-4-hydroxy-1,6-diphenylhexa-1,5-diyn-3-yl isobutyrate (6g)



(99 mg, 15%) as a colourless solid with spectroscopic data in accordance with the literature.[3] **mp** 71−72 °C {Lit.[3] 71 °C}; $[\alpha]_D^{20}$ +12.8 (*c* 1.0 in $CHCl_3$) {Lit.[3] $[\alpha]_D^{20}$ (*ent*, 85:15 er) −12.2 (*c* 1.0 in $CHCl_3$)}; following hydrolysis to 1,6-diphenylhexa-1,5-diyne-3,4-diol: **Chiral HPLC analysis** Chiralcel OJ-H (80:20 Hexane:IPA, flow rate 1 mL min^{-1}, 211 nm, 30 °C) t_R (3*S*,4*S*): 12.6 min, t_R (3*R*,4*R*): 18.1 min, 88.34:11.66 er; **^{1}H NMR** (500 MHz, $CDCl_3$) δ_H: 1.26 (d, *J* 7.0 Hz, 3H, CH_3), 1.27 (d, *J* 7.0 Hz, 3H, CH_3), 2.72 (hept, *J* 7.0 Hz, 1H, C*H*CH_3), 4.87 (d, *J* 5.7 Hz, 1H, C*H*OH), 5.86 (d, *J* 5.7 Hz, 1H, C*H*OC(O)*i*-Pr), 7.30 – 7.40 (m, 6H, ArH), 7.43 – 7.55 (m, 4H, ArH).

(3*R*,4*R*)-1,6-diphenylhexa-1,5-diyne-3,4-diyl bis(2-methylpropanoate) (7g)

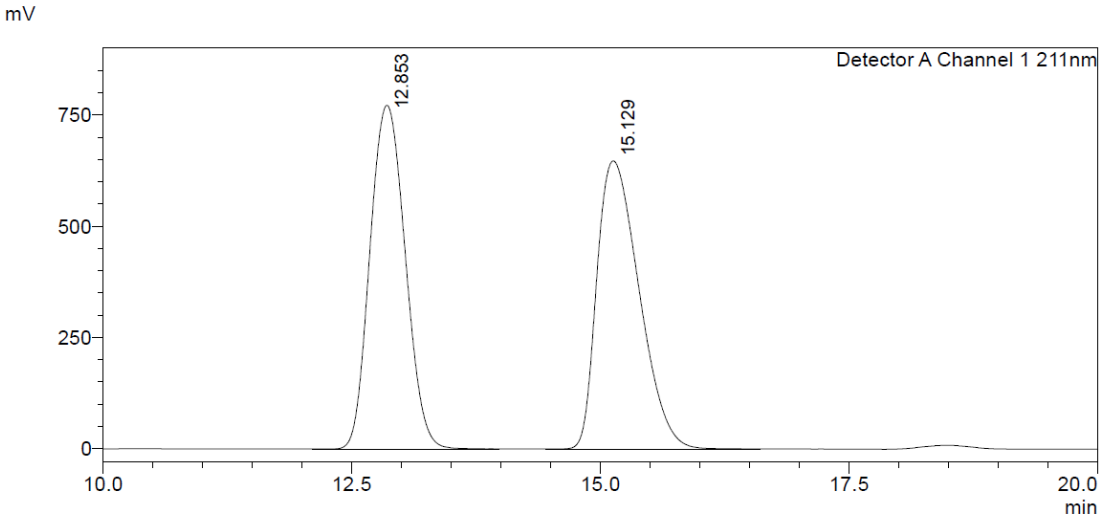


(569 mg, 70%) as a colourless oil with spectroscopic data in accordance with the literature.[3] $[\alpha]_D^{20}$ −10.4 (*c* 1.0 in $CHCl_3$) {Lit.[3] $[\alpha]_D^{20}$ (*ent*, 83:17 er) +39.8 (*c* 0.5 in $CHCl_3$)}; following hydrolysis to 1,6-diphenylhexa-1,5-diyne-3,4-diol: **Chiral HPLC analysis** Chiralcel OJ-H (80:20 Hexane:IPA, flow rate 1 mL min^{-1}, 211 nm, 30 °C) t_R (3*S*,4*S*): 12.6 min, t_R (3*R*,4*R*): 18.1 min, 41.64:58.36 er; **^{1}H NMR** (500 MHz, $CDCl_3$) δ_H: 1.24 (d, *J* 7.0 Hz, 6H, CH_3), 1.25 (d, *J* 7.0 Hz, 6H, CH_3), 2.68 (hept, *J* 7.0 Hz, 2H, C*H*CH_3), 6.00 (s, 2H, C*H*OC(O)*i*-Pr), 7.30 – 7.40 (m, 6H, ArH), 7.43 – 7.50 (m, 4H, ArH).

Procedure for the Kinetic Resolution of (±)-5a in flow-mode conditions (to lower conversion)

A packed bed reactor consisting of a vertically-mounted Omnifit glass chromatography column [borosilicate glass; length = 100 mm (70 mm adjustable bed height); internal diameter = 10 mm; maximum bed volume 5.6 mL], with a glass cooling jacket was loaded with PS-HyperBTM resin **8** (600 mg; f = 0.89 mmol g^{-1}). The resin was allowed to swell to its maximum volume by pumping $CHCl_3$ at 1 mL min^{-1} for 30 min at r.t. using a Gilson 305 HPLC pump. Two syringes were used to inject reagents using a Legato 200 series syringe pump by World Precision Instruments. The first syringe was filled with a solution of (±)-1,2-diphenylethane-1,2-diol **5a** (2 mmol, 1.0 equiv.) and (*i*-PrCO)$_2$O (2 mmol, 1.0 equiv.) in $CHCl_3$ (10 mL total volume) and the second syringe with *i*-Pr$_2$NEt (2 mmol, 1.0 equiv.) 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}. After complete addition of the reagents from the syringes, a Gilson 305 HPLC pump was connected, and $CHCl_3$ was pumped at 100 μL min^{-1} for 30 min to ensure elution of the products. A solution of 10% MeOH in $CHCl_3$ was then pumped at 200 μL min^{-1} for 30 min to wash the column and avoid cross contamination. The column was then prepared for the next KR by pumping $CHCl_3$ at 200 μL min^{-1} for 30 min. The mixture was diluted with CH_2Cl_2 and washed sequentially with HCl (1 M), saturated $NaHCO_3$ and brine. The organic layer was dried (Na_2SO_4), filtered and concentrated to give the crude products. The corresponding diol and monoester were then isolated by flash chromatography (60:40 Hexane:EtOAc).

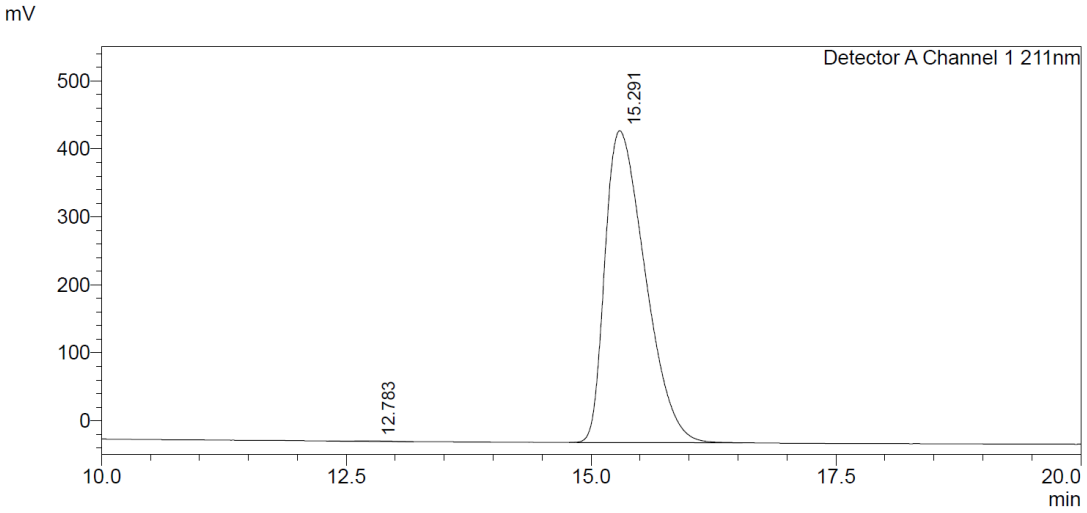
(1*S*,2*S*)-1,2-Diphenylethane-1,2-diol (5a): Chiral HPLC analysis Chiralpak ID (90:10 Hexane:IPA, flow rate 1.0 mL min^{-1}, 211 nm, 30 °C) t_R(1*R*,2*R*): 12.8 min, t_R(1*S*,2*S*): 15.3 min, 0.11:99.89 er.



<Peak Table>

Detector A Channel 1 211nm

Peak#	Ret. Time	Area%
1	12.853	49.934
2	15.129	50.066
Total		100.000



<Peak Table>

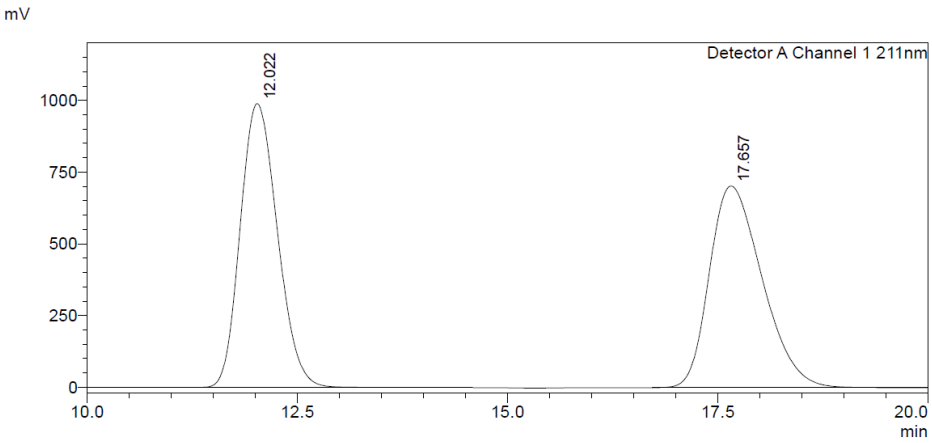
Detector A Channel 1 211nm

Peak#	Ret. Time	Area%
1	12.783	0.106
2	15.291	99.894
Total		100.000

Procedure for the Kinetic Resolution of (±)-6a in flow-mode conditions

A packed bed reactor consisting of a vertically-mounted Omnifit glass chromatography column [borosilicate glass; length = 100 mm (70 mm adjustable bed height); internal diameter = 10 mm; maximum bed volume 5.6 mL], with a glass cooling jacket was loaded with PS-HyperBTM resin **8** (600 mg; f = 0.89 mmol g^{-1}). The resin was allowed to swell to its maximum volume by pumping $CHCl_3$ at 1 mL min^{-1} for 30 min at r.t. using a Gilson 305 HPLC pump. Two syringes were used to inject reagents using a Legato 200 series syringe pump by World Precision Instruments. The first syringe was filled with a solution of 2-hydroxy-1,2-diphenylethyl isobutyrate **6a** (0.3 mmol, 1.0 equiv.) and $(i\text{-PrCO})_2O$ (0.26 mmol, 0.85 equiv.) in $CHCl_3$ (1.5 mL total volume) and the second syringe with $i\text{-Pr}_2NEt$ (0.26 mmol, 0.85 equiv.) in $CHCl_3$ (1.5 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}. After complete addition of the reagents from the syringes, a Gilson 305 HPLC pump was connected, and $CHCl_3$ was pumped at 100 μL min^{-1} for 30 min to ensure elution of the products. A solution of 10% MeOH in $CHCl_3$ was then pumped at 200 μL min^{-1} for 30 min to wash the column and avoid cross contamination. The column was then prepared for the next KR by pumping $CHCl_3$ at 200 μL min^{-1} for 30 min. The mixture was diluted with CH_2Cl_2 and washed sequentially with HCl (1 M), saturated $NaHCO_3$ and brine. The organic layer was dried (Na_2SO_4), filtered and concentrated to give the crude products. The corresponding monoester and diester were then isolated by flash chromatography (80:20 Hexane:EtOAc).

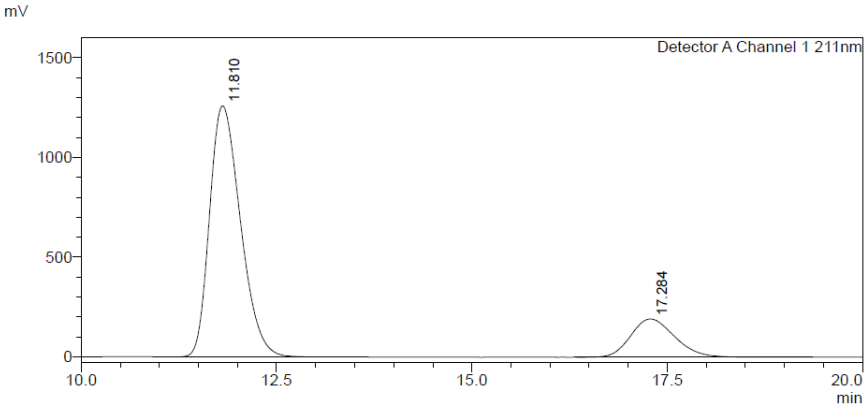
(1*S*,2*S*)-2-Hydroxy-1,2-diphenylethyl isobutyrate (6a): **Chiral HPLC analysis** Chiralcel OD-H (95:5 Hexane:IPA, flow rate 1 mL min^{-1}, 211 nm, 30 °C) t_R (1*S*,2*S*): 11.9 min, t_R (1*R*,2*R*): 17.5 min, 82.19:17.82 er.



<Peak Table>

Detector A Channel 1 211nm

Peak#	Ret. Time	Area%
1	12.022	49.984
2	17.657	50.016
Total		100.000

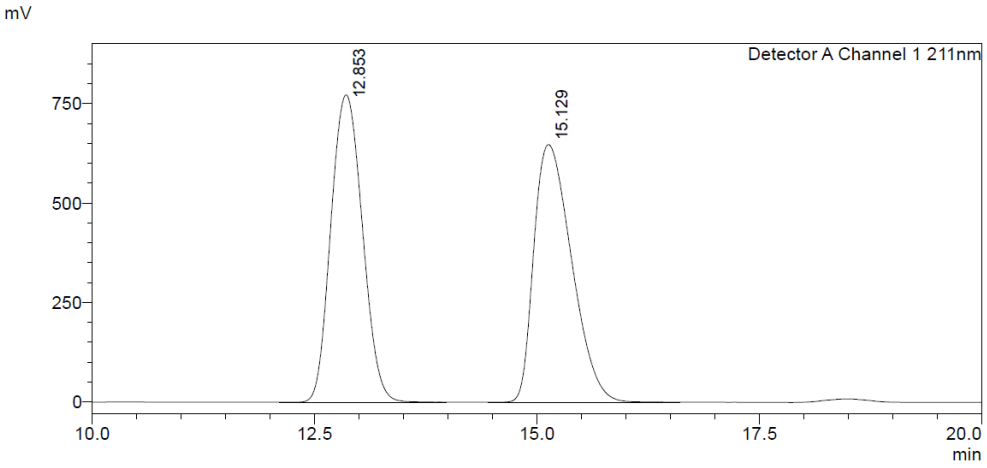


<Peak Table>

Detector A Channel 1 211nm

Peak#	Ret. Time	Area%
1	11.810	82.186
2	17.284	17.814
Total		100.000

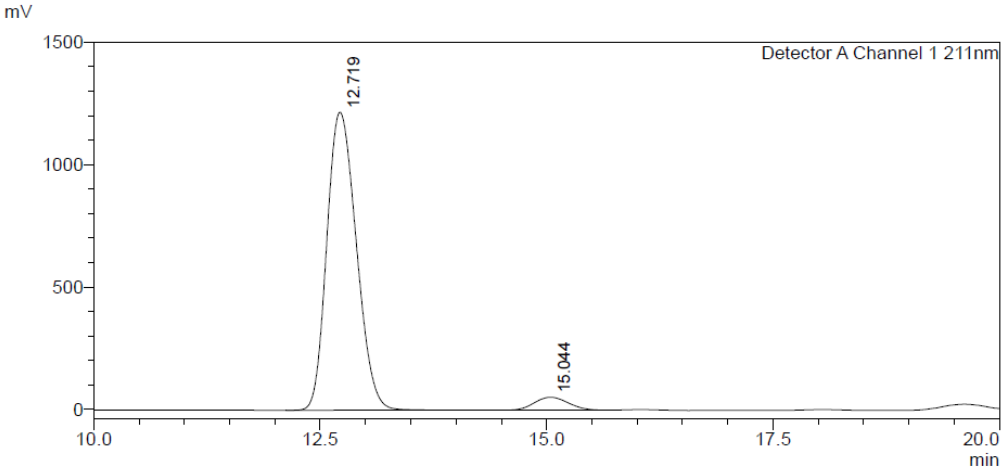
(1*R*,2*R*)-1,2-Diphenylethane-1,2-diyl bis(2-methylpropanoate) (7a): **Chiral HPLC analysis** Chiralpak ID (90:10 Hexane:IPA, flow rate 1.0 mL min^{-1}, 211 nm, 30 °C) t_R(1*R*,2*R*): 12.7 min, t_R(1*S*,2*S*): 15.4 min, 95.20:4.80 er.



<Peak Table>

Detector A Channel 1 211nm

Peak#	Ret. Time	Area%
1	12.853	49.934
2	15.129	50.066
Total		100.000



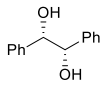
<Peak Table>

Detector A Channel 1 211nm

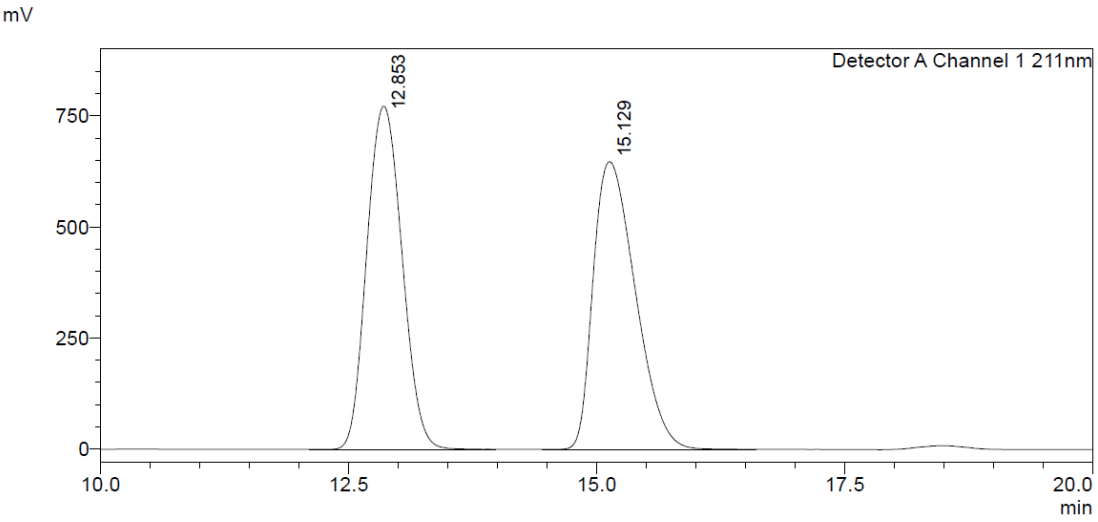
Peak#	Ret. Time	Area%
1	12.719	95.196
2	15.044	4.804
Total		100.000

HPLC chromatograms for the Sequential Acylative Kinetic Resolution of (±)-*syn*-1,2-diols 5

(1*S*,2*S*)-1,2-Diphenylethane-1,2-diol (5a)



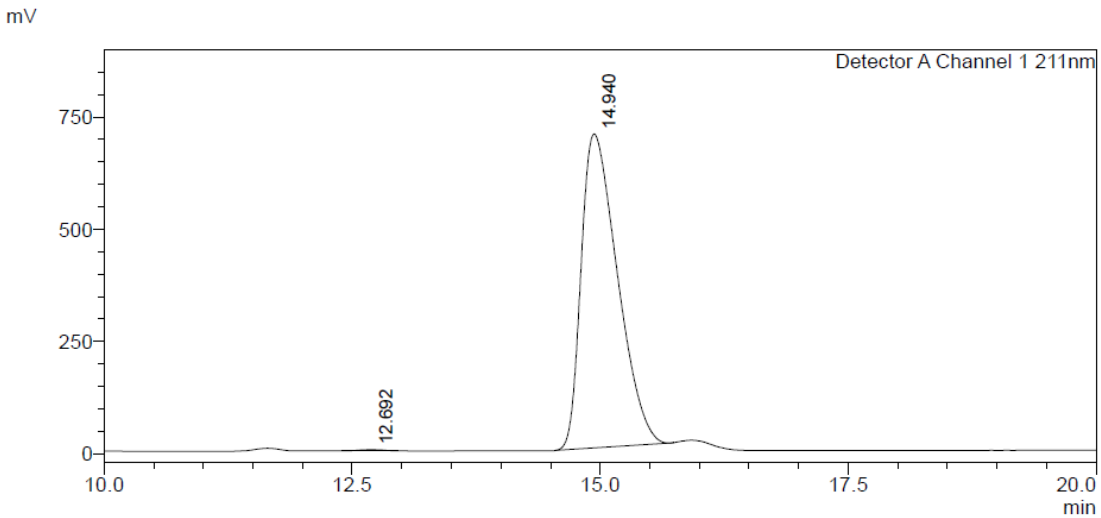
Chiral HPLC analysis Chiralpak ID (90:10 Hexane:IPA, flow rate 1.0 mL min^{-1}, 211 nm, 30 °C) t_R(1*R*,2*R*): 11.6 min, t_R(1*S*,2*S*): 14.9 min, 0.27:99.73 er.



<Peak Table>

Detector A Channel 1 211nm

Peak#	Ret. Time	Area%
1	12.853	49.934
2	15.129	50.066
Total		100.000

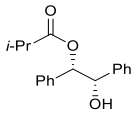


<Peak Table>

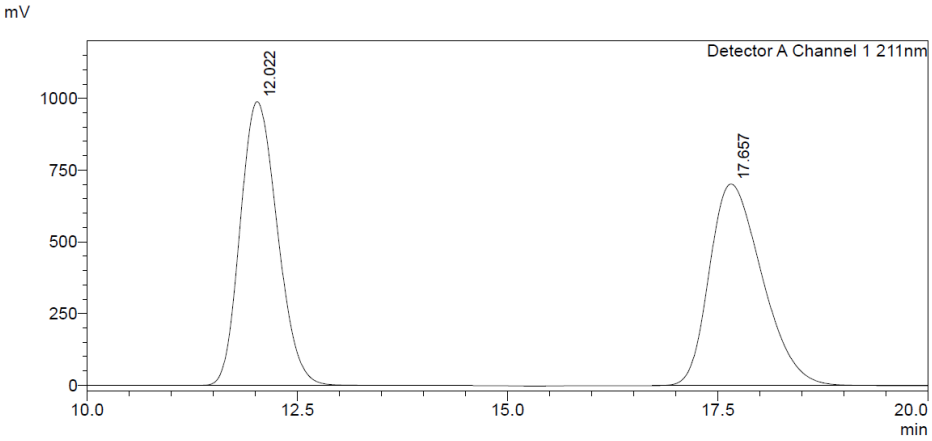
Detector A Channel 1 211nm

Peak#	Ret. Time	Area%
1	12.692	0.275
2	14.940	99.725
Total		100.000

(1*S*,2*S*)-2-Hydroxy-1,2-diphenylethyl isobutyrate (6a)



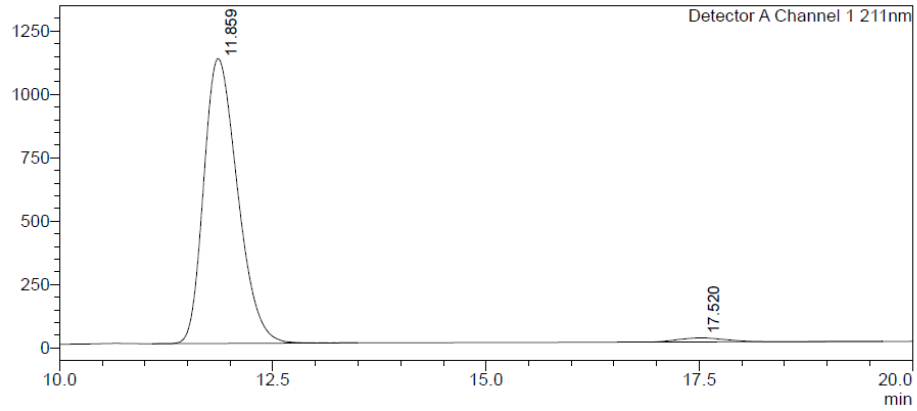
Chiral HPLC analysis Chiralcel OD-H (95:5 Hexane:IPA, flow rate 1 mL min^{-1}, 211 nm, 30 °C) t_R (1*S*,2*S*): 11.9 min, t_R (1*R*,2*R*): 17.5 min, 97.90:2.10 er.



<Peak Table>

Detector A Channel 1 211nm

Peak#	Ret. Time	Area%
1	12.022	49.984
2	17.657	50.016
Total		100.000

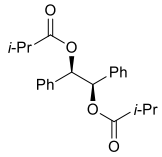


<Peak Table>

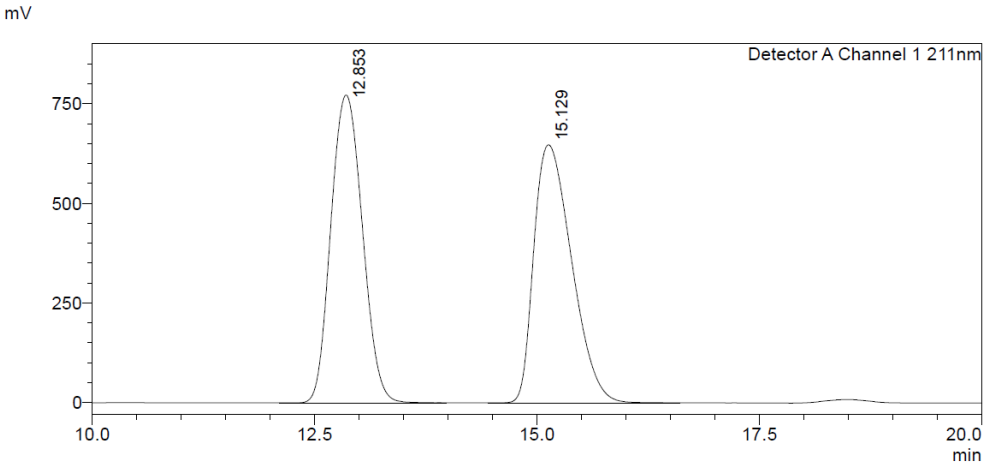
Detector A Channel 1 211nm

Peak#	Ret. Time	Area%
1	11.859	97.904
2	17.520	2.096
Total		100.000

(1*R*,2*R*)-1,2-Diphenylethane-1,2-diol (7a)



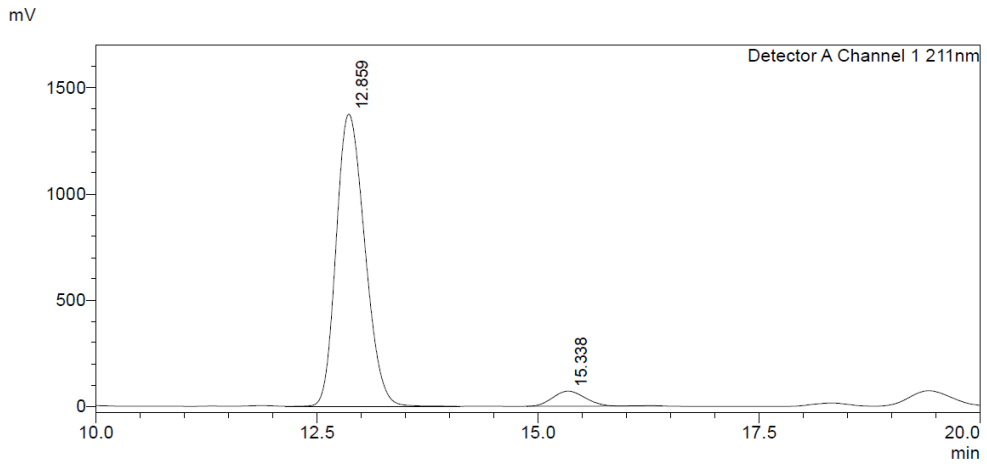
1,2-Diphenylethane-1,2-diyl bis(2-methylpropanoate) could not be separated by HPLC so it was hydrolysed to give 1,2-diphenylethane-1,2-diol; **Chiral HPLC analysis** Chiralpak ID (90:10 Hexane:IPA, flow rate 1.0 mL min^{-1}, 211 nm, 30 °C) t_R(1*R*,2*R*): 12.7 min, t_R(1*S*,2*S*): 15.4 min, 94.45:5.55 er.



<Peak Table>

Detector A Channel 1 211nm

Peak#	Ret. Time	Area%
1	12.853	49.934
2	15.129	50.066
Total		100.000

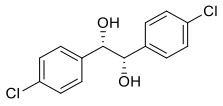


<Peak Table>

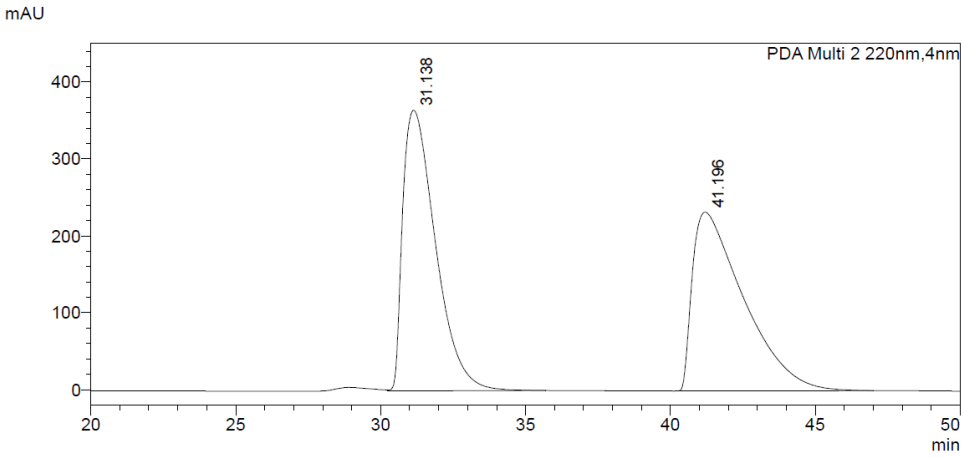
Detector A Channel 1 211nm

Peak#	Ret. Time	Area%
1	12.859	94.449
2	15.338	5.551
Total		100.000

(1*S*,2*S*)-1,2-bis(4-chlorophenyl)ethane-1,2-diol (5b)



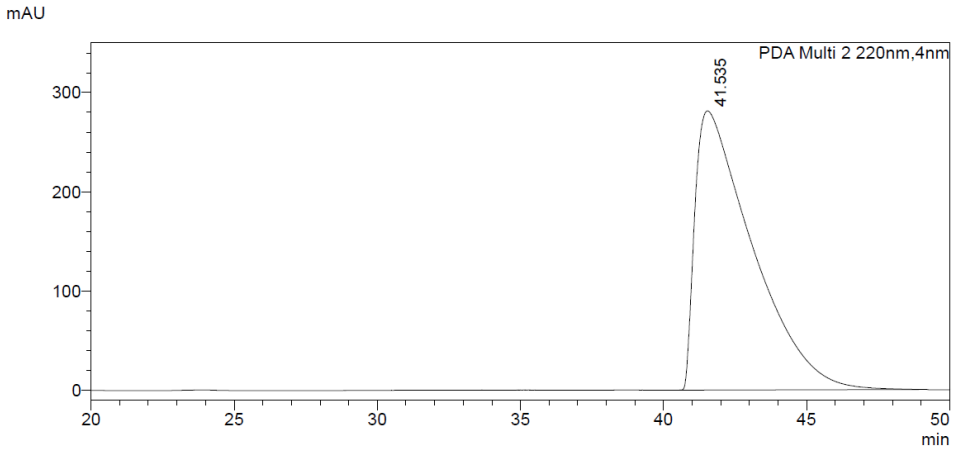
Chiral HPLC analysis Chiralpak AS-H (98:2 Hexane:IPA, flow rate 1 mL min^{-1}, 220 nm, 30 °C) t_R (1*S*,2*S*): 41.5 min, 0:100 er.



<Peak Table>

PDA Ch1 211nm

Peak#	Ret. Time	Area%
1	31.137	50.572
2	41.198	49.428
Total		100.000

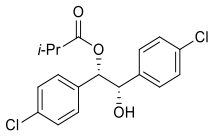


<Peak Table>

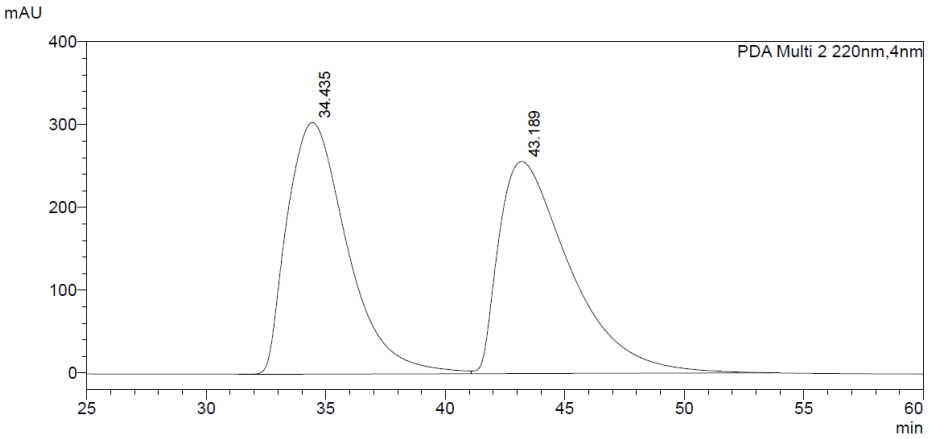
PDA Ch2 220nm

Peak#	Ret. Time	Area%
1	41.535	100.000
Total		100.000

(1*S*,2*S*)-1,2-bis(4-chlorophenyl)-2-hydroxyethyl isobutyrate (6b)



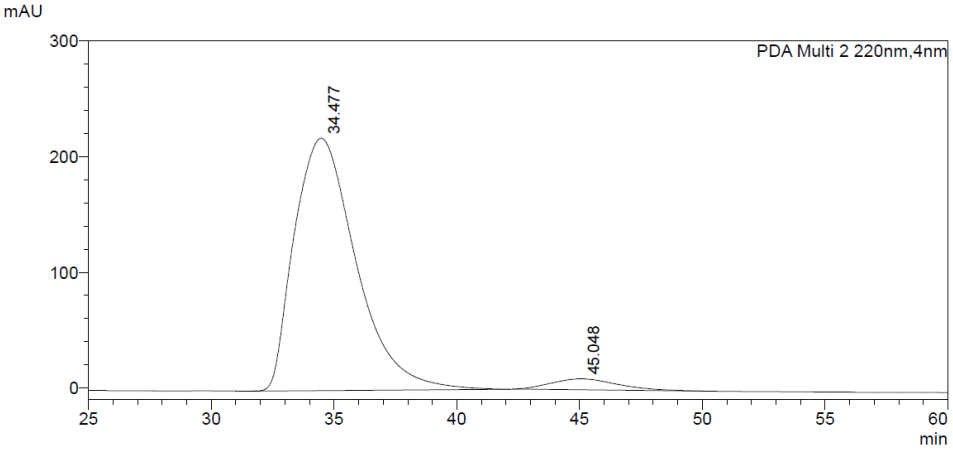
Chiral HPLC analysis Chiralpak AS-H (99.5:0.5 Hexane:IPA, flow rate 0.7 mL min^{-1}, 220 nm, 30 °C) t_R (1*S*,2*S*): 35.9 min, t_R (1*R*,2*R*): 46.7 min, 95.53:4.47 er.



<Peak Table>

PDA Ch2 220nm

Peak#	Ret. Time	Area%
1	34.435	50.018
2	43.189	49.982
Total		100.000

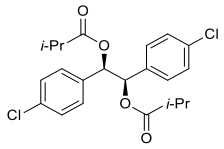


<Peak Table>

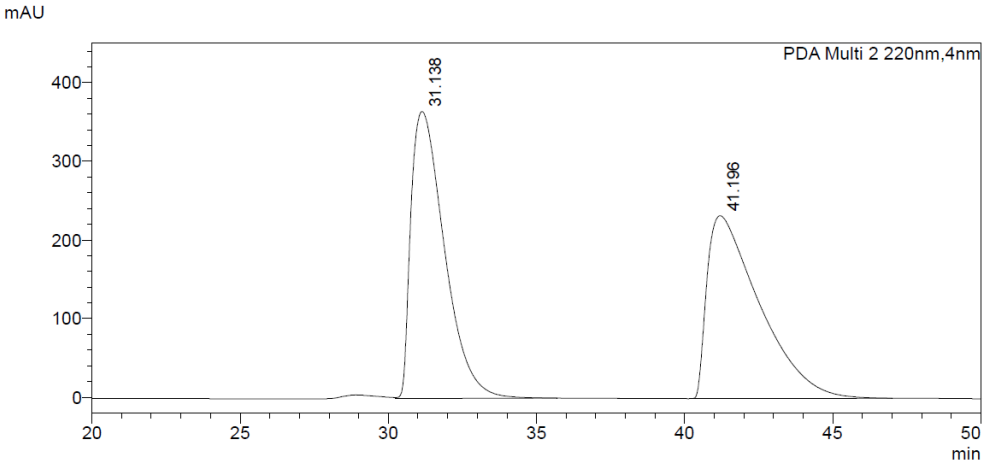
PDA Ch2 220nm

Peak#	Ret. Time	Area%
1	34.477	95.529
2	45.048	4.471
Total		100.000

(1*R*,2*R*)-1,2-bis(4-chlorophenyl)ethane-1,2-diyl bis(2-methylpropanoate) (7b)



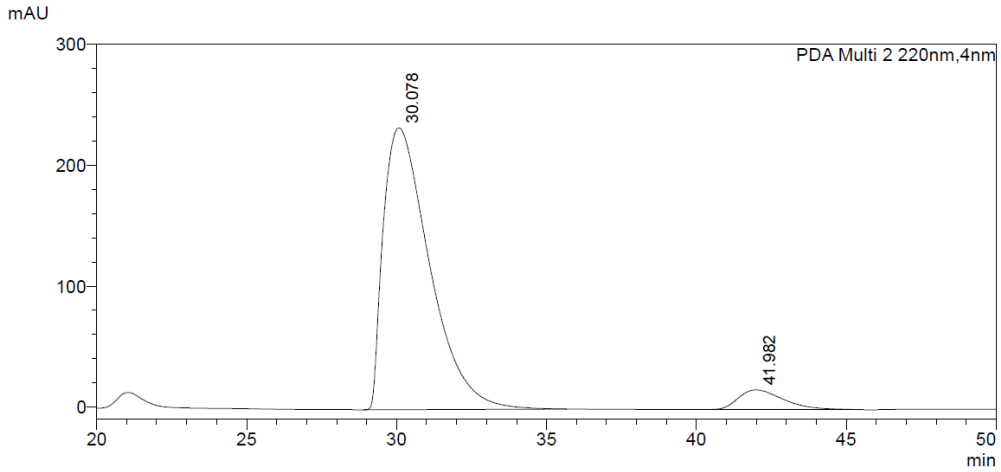
1,2-bis(4-chlorophenyl)ethane-1,2-diyl bis(2-methylpropanoate) could not be separated by HPLC so it was hydrolysed to give 1,2-bis(4-chlorophenyl)ethane-1,2-diol; **Chiral HPLC analysis** Chiralpak AS-H (98:2 Hexane:IPA, flow rate 1 mL min^{-1}, 220 nm, 30 °C) t_R (1*R*,2*R*): 31.0 min, t_R (1*S*,2*S*): 43.8 min, 93.62:6.38 er;



<Peak Table>

PDA Ch1 211nm

Peak#	Ret. Time	Area%
1	31.137	50.572
2	41.198	49.428
Total		100.000

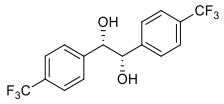


<Peak Table>

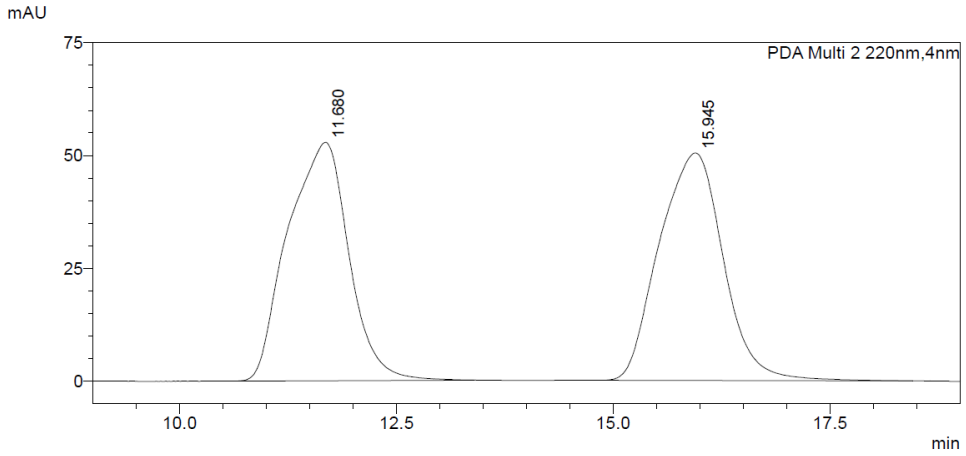
PDA Ch2 220nm

Peak#	Ret. Time	Area%
1	30.078	93.621
2	41.982	6.379
Total		100.000

(1*S*,2*S*)-1,2-bis(4-(trifluoromethyl)phenyl)ethane-1,2-diol (5c)



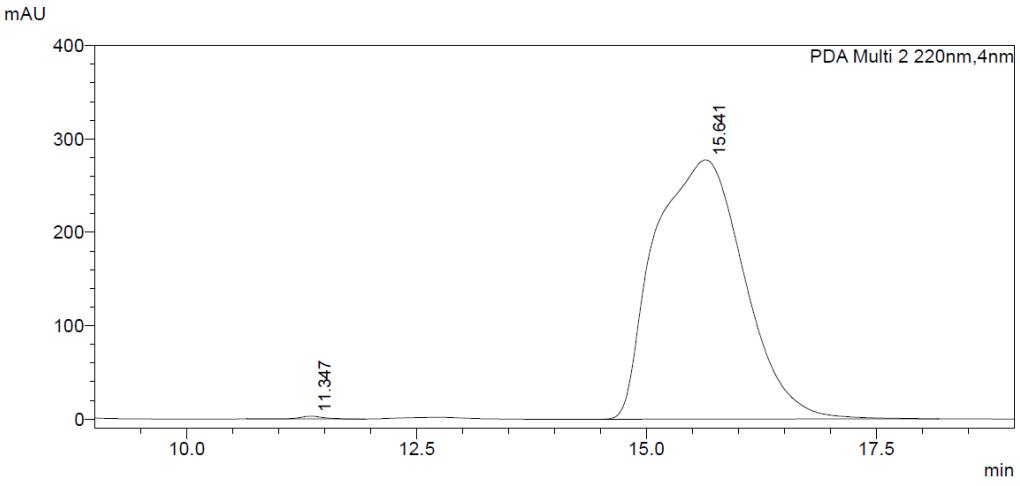
Chiral HPLC analysis Chiralpak AS-H (97:3 Hexane:IPA, flow rate 1 mL min^{-1}, 220 nm, 30 °C) t_R (1*R*,2*R*): 11.3 min, t_R (1*S*,2*S*): 15.6 min, 0.27:99.73 er.



<Peak Table>

PDA Ch2 220nm

Peak#	Ret. Time	Area%
1	11.680	49.936
2	15.945	50.064
Total		100.000

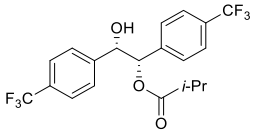


<Peak Table>

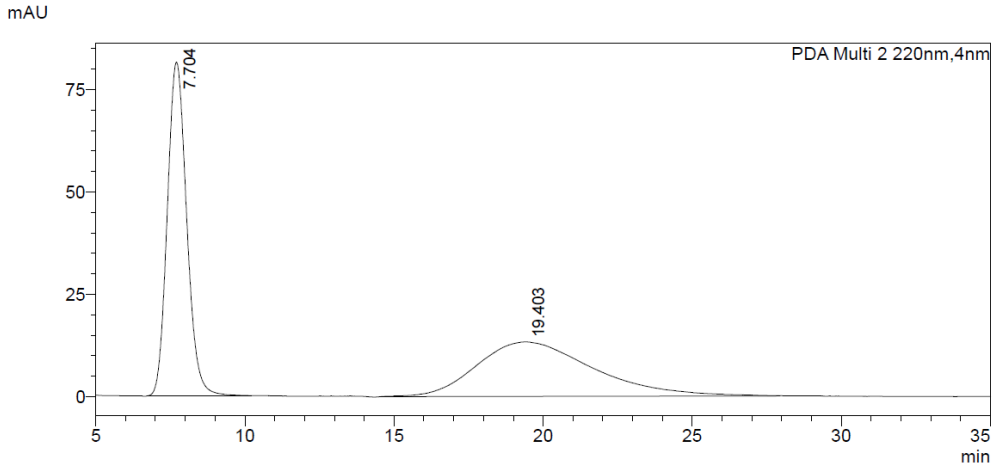
PDA Ch2 220nm

Peak#	Ret. Time	Area%
1	11.347	0.274
2	15.641	99.726
Total		100.000

(1*S*,2*S*)-2-hydroxy-1,2-bis(4-(trifluoromethyl)phenyl)ethyl isobutyrate (6c)



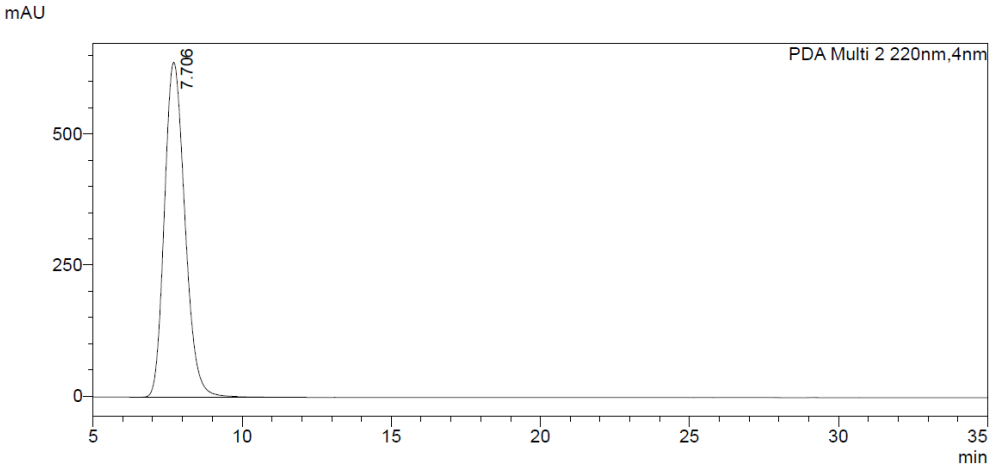
Chiral HPLC analysis Chiralcel OJ-H (95:5 Hexane:IPA, flow rate 1 mL min^{-1}, 220 nm, 30 °C) t_R (1*S*,2*S*): 7.7 min, 100:0 er.



<Peak Table>

PDA Ch2 220nm

Peak#	Ret. Time	Area%
1	7.704	50.553
2	19.403	49.447
Total		100.000

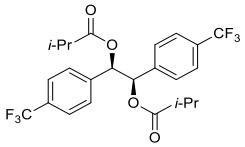


<Peak Table>

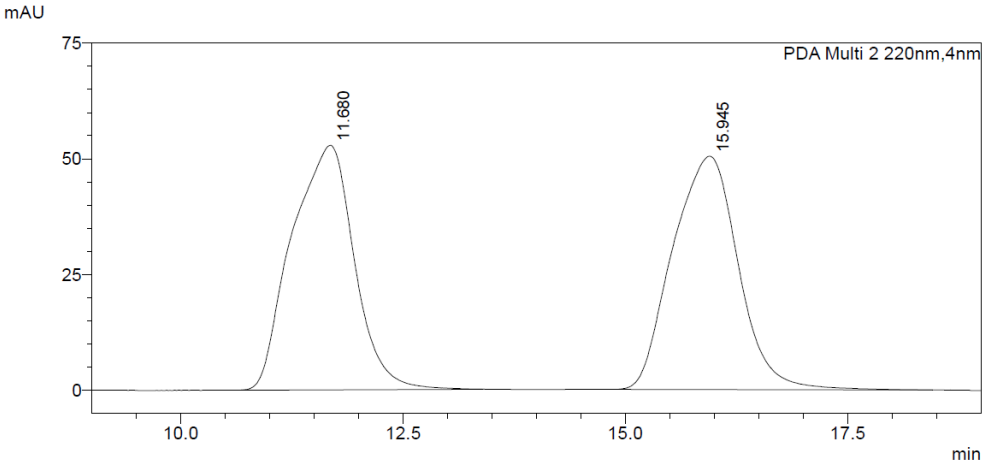
PDA Ch2 220nm

Peak#	Ret. Time	Area%
1	7.706	100.000
Total		100.000

(1*R*,2*R*)-1,2-bis(4-(trifluoromethyl)phenyl)ethane-1,2-diyl bis(2-methylpropanoate) (7c)



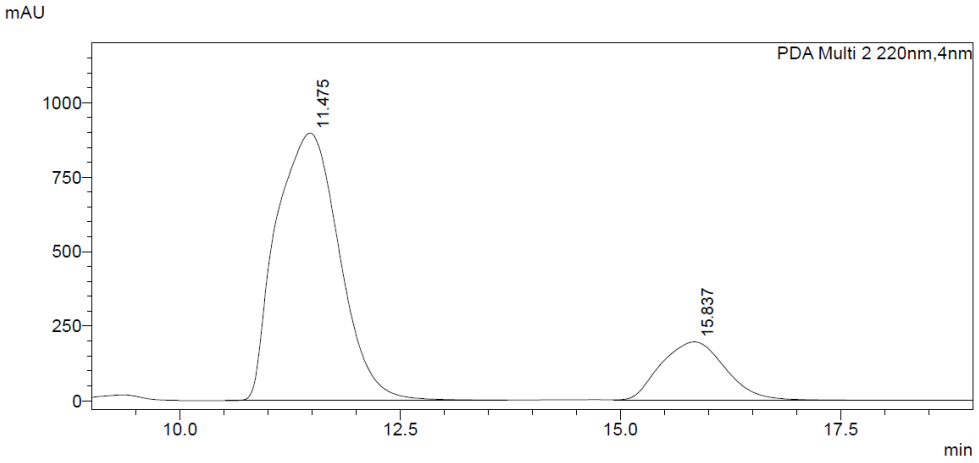
1,2-bis(4-(trifluoromethyl)phenyl)ethane-1,2-diyl bis(2-methylpropanoate) could not be separated by HPLC so it was hydrolysed to give 1,2-bis(4-(trifluoromethyl)phenyl)ethane-1,2-diol; **Chiral HPLC analysis** Chiralpak AS-H (97:3 Hexane:IPA, flow rate 1 mL min^{-1}, 220 nm, 30 °C) t_R (1*R*,2*R*): 11.5 min, t_R (1*S*,2*S*): 15.8 min, 81.67:18.33 er.



<Peak Table>

PDA Ch2 220nm

Peak#	Ret. Time	Area%
1	11.680	49.936
2	15.945	50.064
Total		100.000

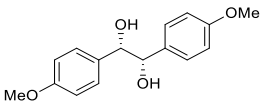


<Peak Table>

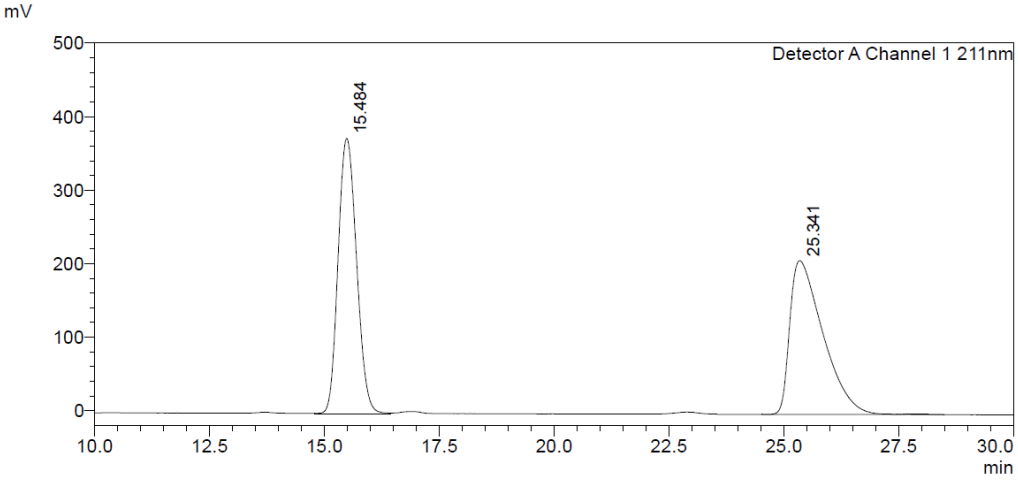
PDA Ch2 220nm

Peak#	Ret. Time	Area%
1	11.475	81.670
2	15.837	18.330
Total		100.000

(1S,2S)-1,2-bis(4-methoxyphenyl)ethane-1,2-diol (5d)



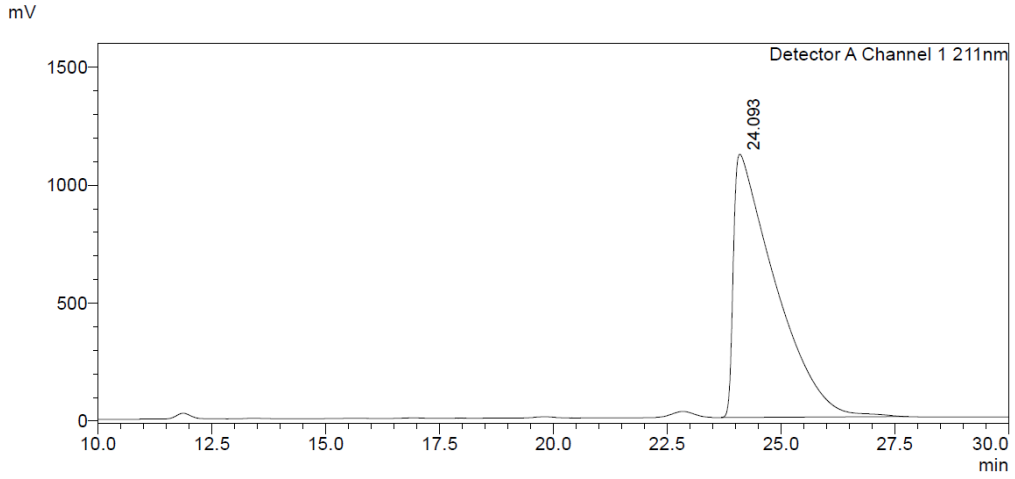
Chiral HPLC analysis Chiralpak ID (80:20 Hexane:IPA, flow rate 1 mL min^{-1}, 211 nm, 30 °C) t_R (1*S*,2*S*): 24.09 min, 0:100 er.



<Peak Table>

Detector A Channel 1 211nm

Peak#	Ret. Time	Area%
1	15.484	49.915
2	25.341	50.085
Total		100.000

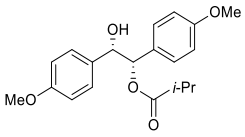


<Peak Table>

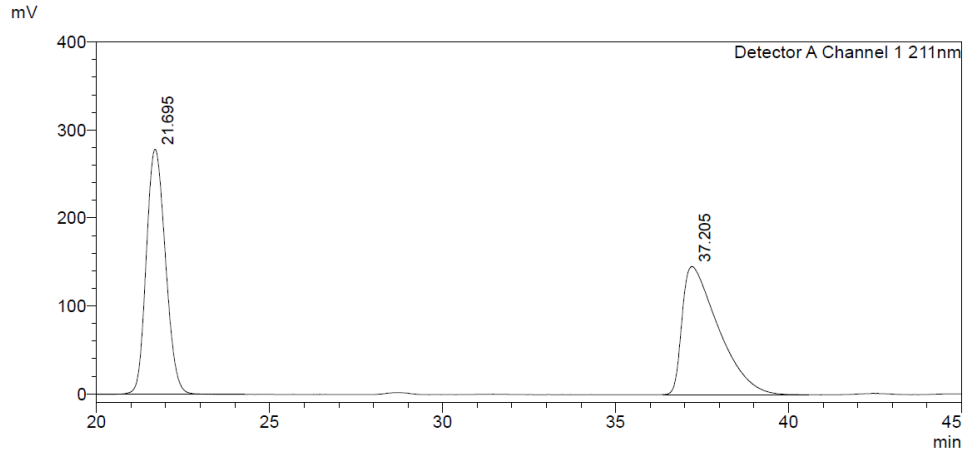
Detector A Channel 1 211nm

Peak#	Ret. Time	Area%
1	24.093	100.000
Total		100.000

(1*S*,2*S*)-2-hydroxy-1,2-bis(4-methoxyphenyl)ethyl isobutyrate (6d)



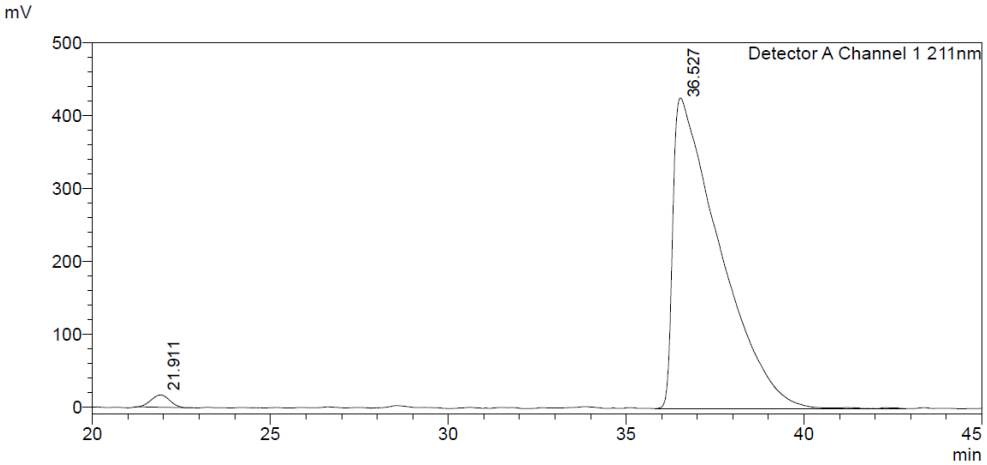
2-hydroxy-1,2-bis(4-methoxyphenyl)ethyl isobutyrate could not be separated by HPLC so it was hydrolysed to give 1,2-bis(4-methoxyphenyl)ethane-1,2-diol; **Chiral HPLC analysis** Chiralpak ID (85:15 Hexane:IPA, flow rate 1 mL min^{-1}, 211 nm, 30 °C) t_R (1*R*,2*R*): 21.91 min, t_R (1*S*,2*S*): 36.53 min, 1.56:98.44 er.



<Peak Table>

Detector A Channel 1 211nm

Peak#	Ret. Time	Area%
1	21.695	50.173
2	37.205	49.827
Total		100.000

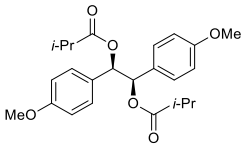


<Peak Table>

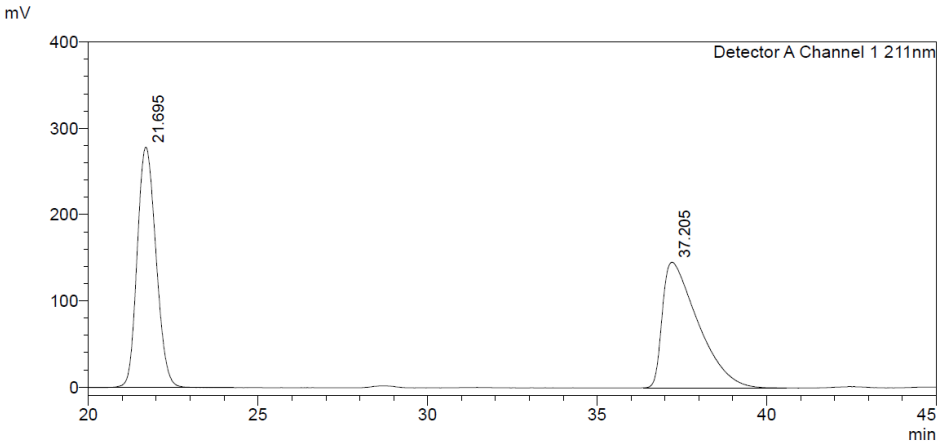
Detector A Channel 1 211nm

Peak#	Ret. Time	Area%
1	21.911	1.557
2	36.527	98.443
Total		100.000

(1*R*,2*R*)-1,2-bis(4-methoxyphenyl)ethane-1,2-diyl bis(2-methylpropanoate) (7d)



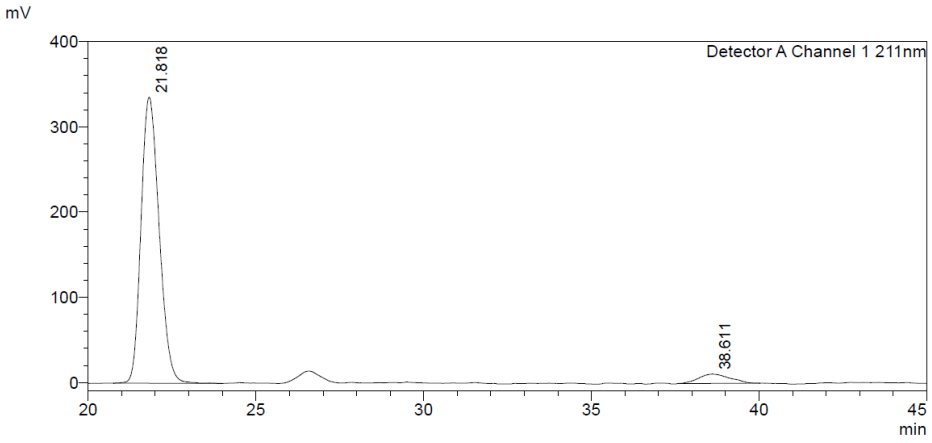
1,2-bis(4-methoxyphenyl)ethane-1,2-diyl bis(2-methylpropanoate) could not be separated by HPLC so it was hydrolysed to give 1,2-bis(4-methoxyphenyl)ethane-1,2-diol; **Chiral HPLC analysis** Chiralpak ID (85:15 Hexane:IPA, flow rate 1 mL min^{-1}, 211 nm, 30 °C) t_R (1*R*,2*R*): 21.81 min, t_R (1*S*,2*S*): 38.61 min, 94.66:5.34 er.



<Peak Table>

Detector A Channel 1 211nm

Peak#	Ret. Time	Area%
1	21.695	50.173
2	37.205	49.827
Total		100.000

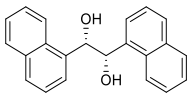


<Peak Table>

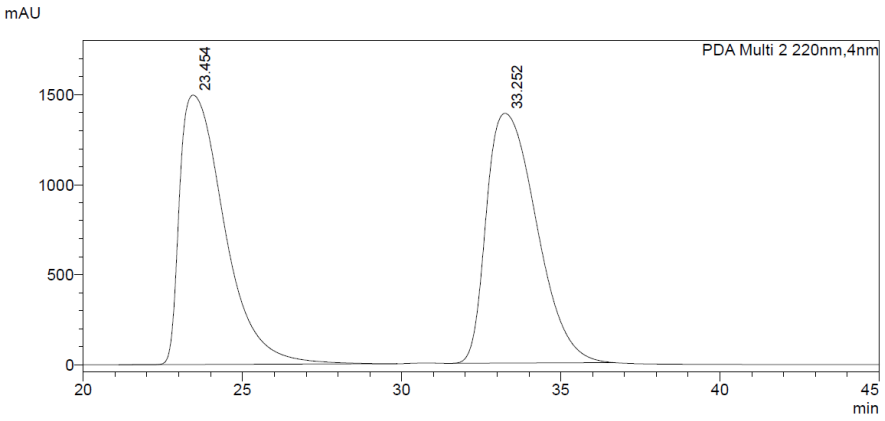
Detector A Channel 1 211nm

Peak#	Ret. Time	Area%
1	21.818	94.655
2	38.611	5.345
Total		100.000

(1*S*,2*S*)-1,2-di(naphthalen-1-yl)ethane-1,2-diol (5e)



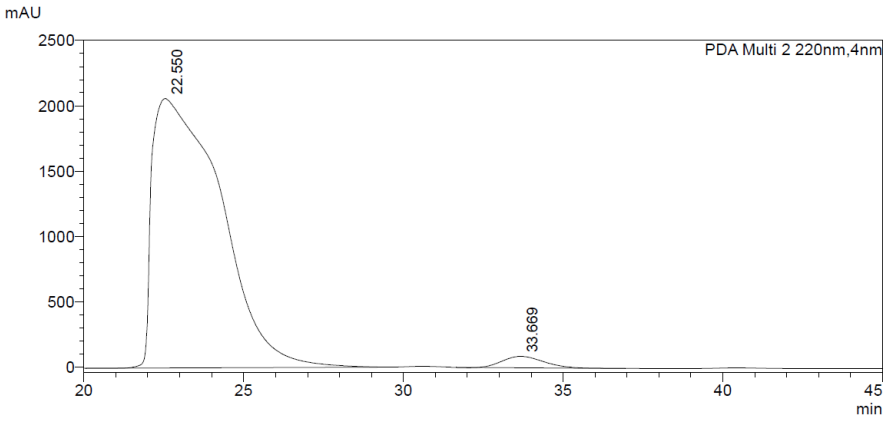
Chiral HPLC analysis Chiralpak OJ-H (80:20 Hexane:IPA, flow rate 1 mL min^{-1}, 220 nm, 30 °C) t_R (1*S*,2*S*): 22.6 min, , t_R (1*R*,2*R*): 33.7 min, 97.49:2.51 er.



<Peak Table>

PDA Ch2 220nm

Peak#	Ret. Time	Area%
1	23.454	49.112
2	33.252	50.888
Total		100.000

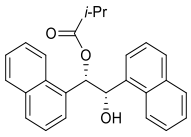


<Peak Table>

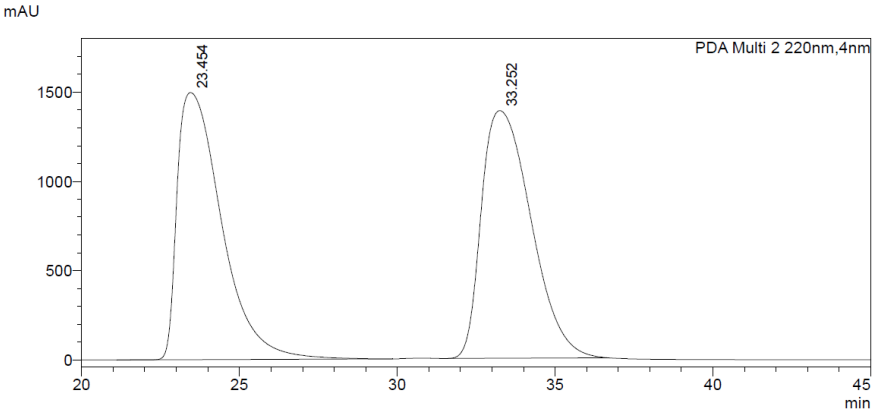
PDA Ch2 220nm

Peak#	Ret. Time	Area%
1	22.550	97.485
2	33.669	2.515
Total		100.000

(1*S*,2*S*)-2-hydroxy-1,2-di(naphthalen-1-yl)ethyl isobutyrate (6e)



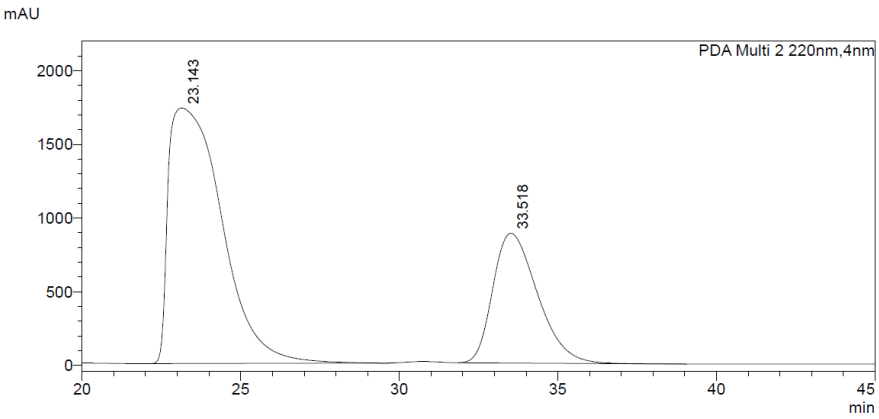
2-hydroxy-1,2-di(naphthalen-1-yl)ethyl isobutyrate could not be separated by HPLC so it was hydrolysed to give 1,2-di(naphthalen-1-yl)ethane-1,2-diol; **Chiral HPLC analysis** Chiralpak OJ-H (80:20 Hexane:IPA, flow rate 1 mL min^{-1}, 220 nm, 30 °C) t_R (1*S*,2*S*): 23.1 min, t_R (1*R*,2*R*): 33.5 min, 70.30:29.70 er.



<Peak Table>

PDA Ch2 220nm

Peak#	Ret. Time	Area%
1	23.454	49.112
2	33.252	50.888
Total		100.000

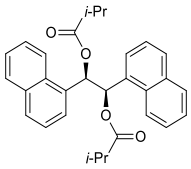


<Peak Table>

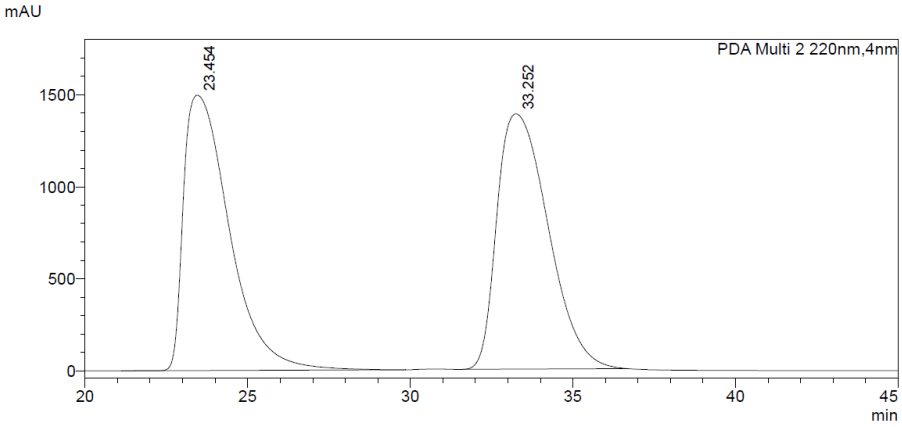
PDA Ch2 220nm

Peak#	Ret. Time	Area%
1	23.143	70.297
2	33.518	29.703
Total		100.000

(1*R*,2*R*)-1,2-di(naphthalen-1-yl)ethane-1,2-diyl bis(2-methylpropanoate) (7e)



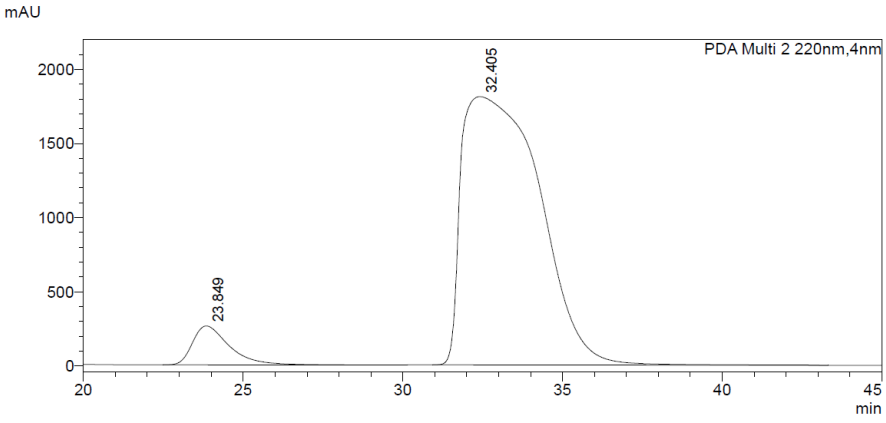
1,2-di(naphthalen-1-yl)ethane-1,2-diyl bis(2-methylpropanoate) could not be separated by HPLC so it was hydrolysed to give 1,2-di(naphthalen-1-yl)ethane-1,2-diol; **Chiral HPLC analysis** Chiralpak OJ-H (80:20 Hexane:IPA, flow rate 1 mL min^{-1}, 220 nm, 30 °C) t_R (1*S*,2*S*): 23.9 min, t_R (1*R*,2*R*): 32.4 min, 6.41:93.59 er.



<Peak Table>

PDA Ch2 220nm

Peak#	Ret. Time	Area%
1	23.454	49.112
2	33.252	50.888
Total		100.000

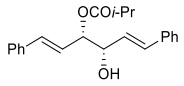


<Peak Table>

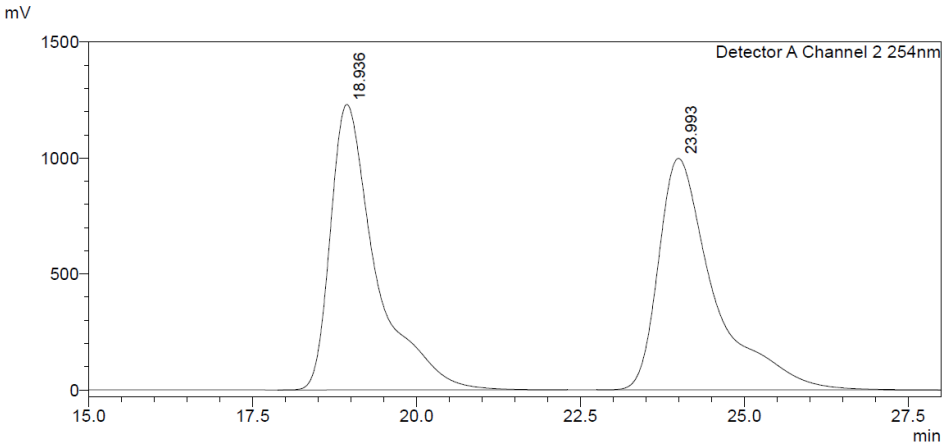
PDA Ch2 220nm

Peak#	Ret. Time	Area%
1	23.849	6.411
2	32.405	93.589
Total		100.000

(1*E*,3*S*,4*S*,5*E*)-4-hydroxy-1,6-diphenylhexa-1,5-dien-3-yl isobutyrate (6f)



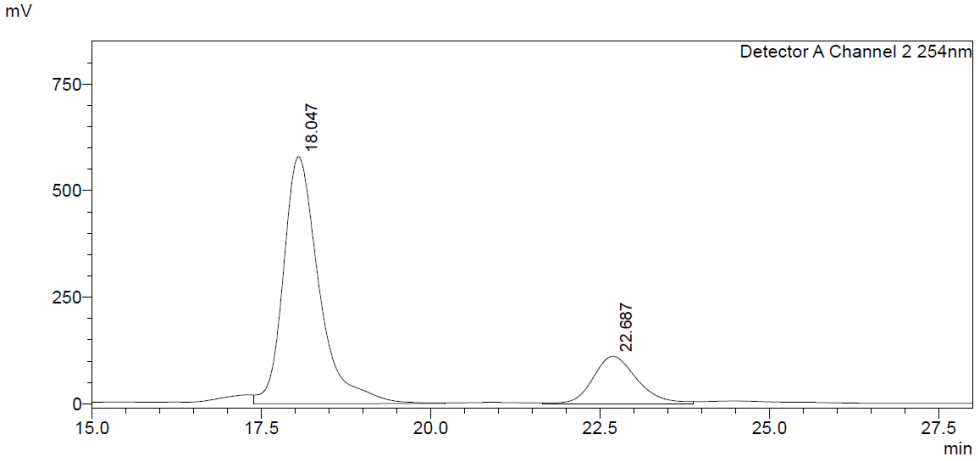
Chiral HPLC analysis Chiralpak IC (95:5 Hexane:IPA, flow rate 1 mL min^{-1}, 254 nm, 30 °C) t_R (3*S*,4*S*): 18.0 min, t_R (3*R*,4*R*): 22.7 min, 80.96:19.04 er.



<Peak Table>

Detector A Channel 2 254nm

Peak#	Ret. Time	Area%
1	18.936	50.198
2	23.993	49.802
Total		100.000

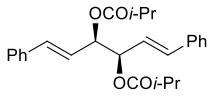


<Peak Table>

Detector A Channel 2 254nm

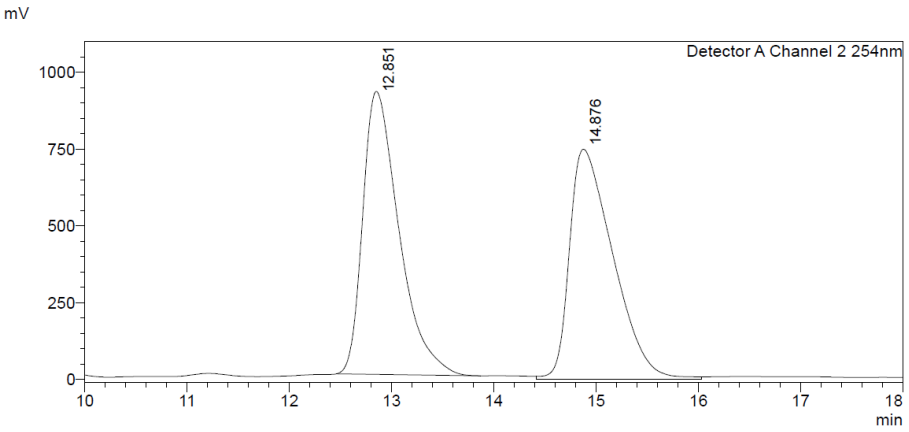
Peak#	Ret. Time	Area%
1	18.047	80.962
2	22.687	19.038
Total		100.000

(1*E*,3*R*,4*R*,5*E*)-1,6-diphenylhexa-1,5-diene-3,4-diyl bis(2-methylpropanoate) (7f)



(1*E*,5*E*)-1,6-diphenylhexa-1,5-diene-3,4-diyl bis(2-methylpropanoate) could not be separated by HPLC so it was hydrolysed to give (1*E*,5*E*)-1,6-diphenylhexa-1,5-diene-3,4-diol; **Chiral HPLC analysis** Chiralpak ID (85:15 Hexane:IPA, flow rate 1 mL min^{-1}, 254 nm, 30 °C) t_R (1*S*,2*S*): 13.0 min, t_R (1*R*,2*R*): 14.7 min, 35.00:65.00 er.

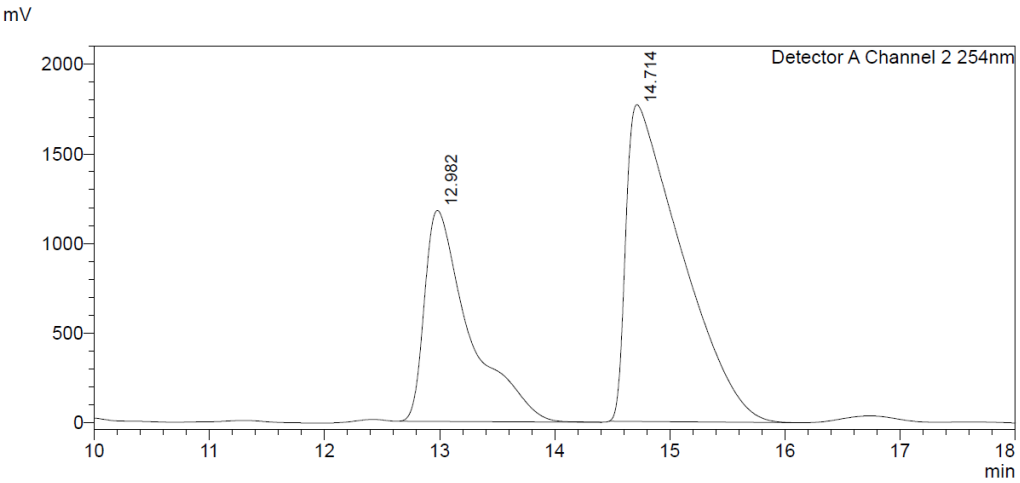
<Chromatogram>



<Peak Table>

Detector A Channel 2 254nm

Peak#	Ret. Time	Area%
1	12.851	50.699
2	14.876	49.301
Total		100.000

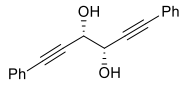


<Peak Table>

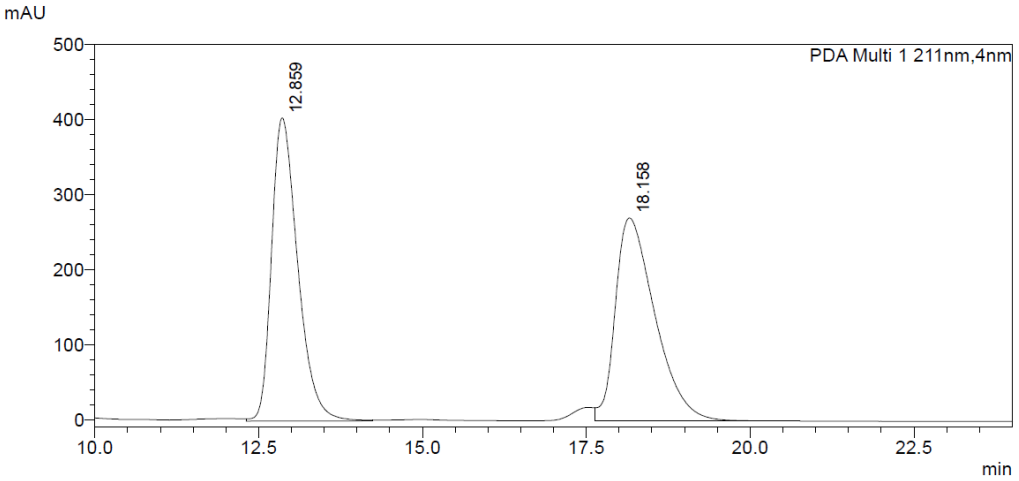
Detector A Channel 2 254nm

Peak#	Ret. Time	Area%
1	12.982	35.003
2	14.714	64.997
Total		100.000

(3*S*,4*S*)-1,6-diphenylhexa-1,5-diyne-3,4-diol (5g)



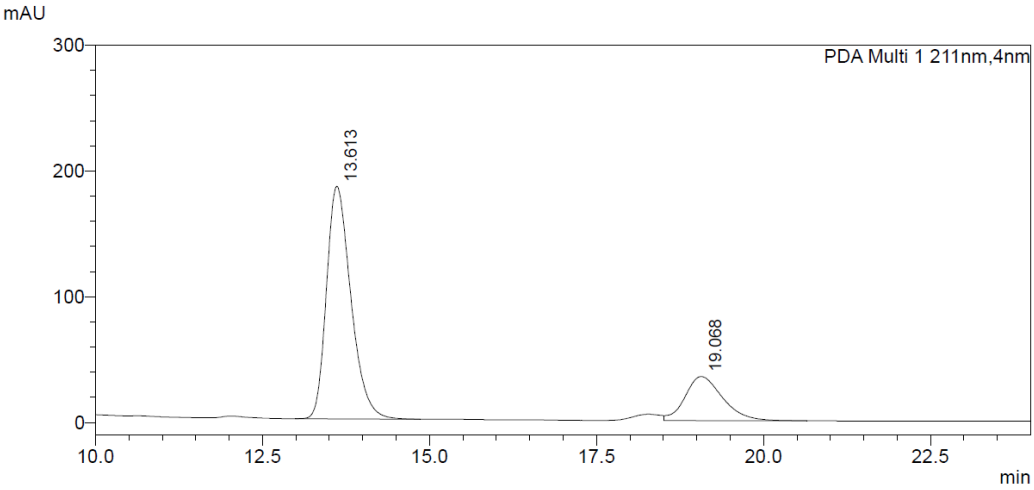
Chiral HPLC analysis Chiralcel OJ-H (80:20 Hexane:IPA, flow rate 1 mL min^{-1}, 211 nm, 30 °C) t_R (3*S*,4*S*): 13.6 min, t_R (3*R*,4*R*): 19.1 min, 77.37:22.63 er.



<Peak Table>

PDA Ch1 211nm

Peak#	Ret. Time	Area%
1	12.859	49.746
2	18.158	50.254
Total		100.000

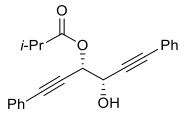


<Peak Table>

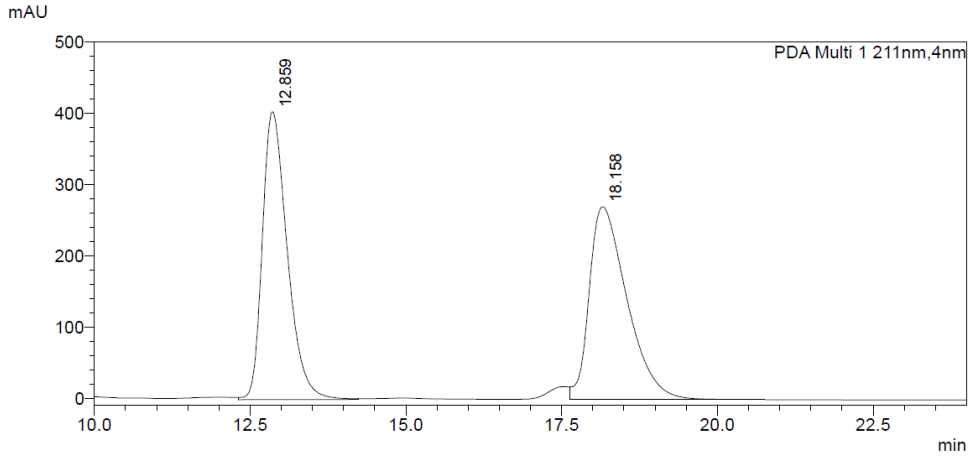
PDA Ch1 211nm

Peak#	Ret. Time	Area%
1	13.613	77.366
2	19.068	22.634
Total		100.000

(3*S*,4*S*)-4-hydroxy-1,6-diphenylhexa-1,5-diyn-3-yl isobutyrate (6g)



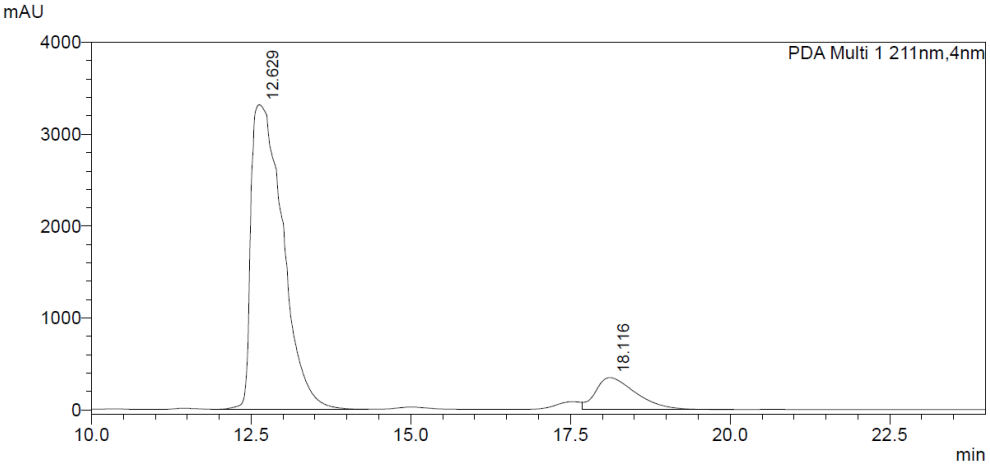
4-hydroxy-1,6-diphenylhexa-1,5-diyn-3-yl isobutyrate could not be separated by HPLC so it was hydrolysed to give 1,6-diphenylhexa-1,5-diyne-3,4-diol; **Chiral HPLC analysis** Chiralcel OJ-H (80:20 Hexane:IPA, flow rate 1 mL min^{-1}, 211 nm, 30 °C) t_R (3*S*,4*S*): 12.6 min, t_R (3*R*,4*R*): 18.1 min, 88.34:11.66 er.



<Peak Table>

PDA Ch1 211nm

Peak#	Ret. Time	Area%
1	12.859	49.746
2	18.158	50.254
Total		100.000

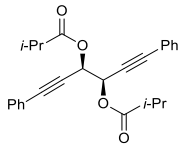


<Peak Table>

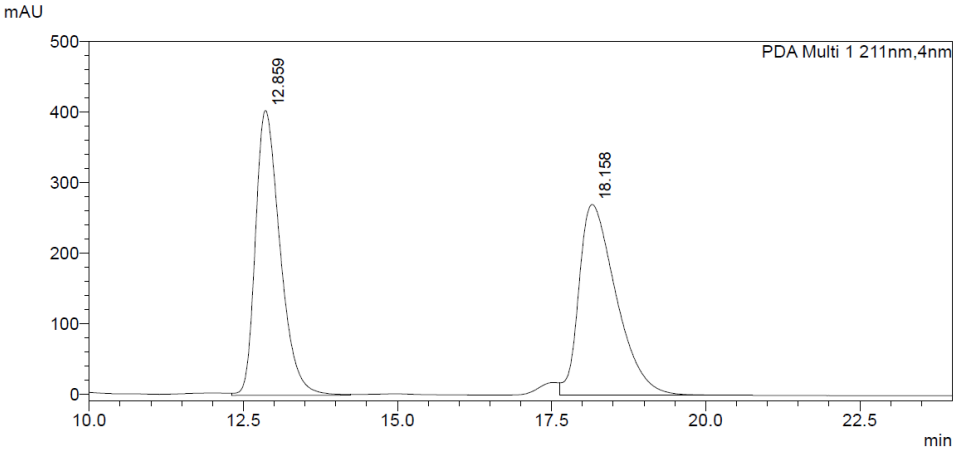
PDA Ch1 211nm

Peak#	Ret. Time	Area%
1	12.629	88.343
2	18.116	11.657
Total		100.000

(3*R*,4*R*)-1,6-diphenylhexa-1,5-diyne-3,4-diyl bis(2-methylpropanoate) (7g)



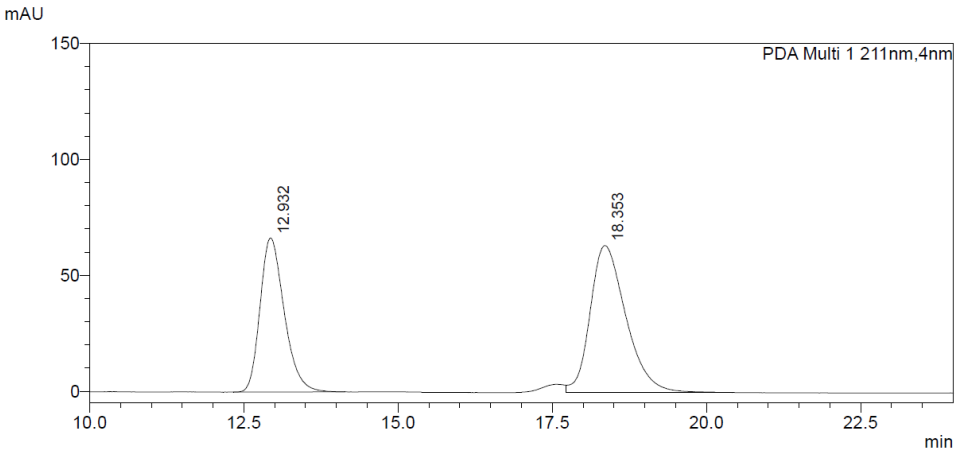
1,6-diphenylhexa-1,5-diyne-3,4-diyl bis(2-methylpropanoate) could not be separated by HPLC so it was hydrolysed to give 1,6-diphenylhexa-1,5-diyne-3,4-diol; **Chiral HPLC analysis** Chiralcel OJ-H (80:20 Hexane:IPA, flow rate 1 mL min^{-1}, 211 nm, 30 °C) t_R (3*S*,4*S*): 12.6 min, t_R (3*R*,4*R*): 18.1 min, 41.64:58.36 er.



<Peak Table>

PDA Ch1 211nm

Peak#	Ret. Time	Area%
1	12.859	49.746
2	18.158	50.254
Total		100.000



<Peak Table>

PDA Ch1 211nm

Peak#	Ret. Time	Area%
1	12.932	41.637
2	18.353	58.363
Total		100.000

General procedure for the Sequential Acylative Kinetic Resolution (±)-*anti*-1,3-diols 2 in flow-mode conditions

A packed bed reactor consisting of a vertically-mounted Omnifit glass chromatography column [borosilicate glass; length = 100 mm (70 mm adjustable bed height); internal diameter = 10 mm; maximum bed volume 5.6 mL], with a glass cooling jacket was loaded with PS-HyperBTM resin **8** (600 mg; f = 0.89 mmol g^{-1}). The resin was allowed to swell to its maximum volume by pumping 1:1 THF:$CHCl_3$ mixture at 1 mL min^{-1} for 30 min at r.t. using a Gilson 305 HPLC pump. Two syringes were used to inject reagents using a Legato 200 series syringe pump by World Precision Instruments. The first syringe was filled with a solution of the appropriate diol (±)-**2** (0.75 mmol, 1.0 equiv.) and $(EtCO)_2O$ (0.79 mmol, 1.05 equiv.) in 1:1 THF:$CHCl_3$ mixture (7.5 mL total volume) and the second syringe with *i*-Pr_2NEt (0.79 mmol, 1.05 equiv.) in 1:1 THF:$CHCl_3$ mixture (7.5 mL total volume). Both solutions were injected at 100 μL min^{-1}, mixed in a T-type mixing chamber, and passed through the reactor at a combined flow rate of 200 μL min^{-1}. After complete addition of the reagents from the syringes, a Gilson 305 HPLC pump was connected, and $CHCl_3$ was pumped at 200 μL min^{-1} for 30 min to ensure elution of the products. A solution of 10% MeOH in 1:1 THF:$CHCl_3$ mixture was then pumped at 200 μL min^{-1} for 30 min to wash the column and avoid cross contamination. The column was then prepared for the next KR by pumping 1:1 THF:$CHCl_3$ mixture at 200 μL min^{-1} for 30 min. The mixture was concentrated to remove THF and later diluted with CH_2Cl_2. So, it was washed sequentially with HCl (1 M), saturated $NaHCO_3$ and brine. The organic layer was dried (Na_2SO_4), filtered and concentrated to give the crude products which were purified by column chromatography.

Hydrolysis of diesters and monoesters

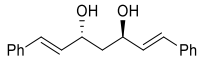
In some instances it was difficult to find conditions to separate the enantiomers of diesters or monoesters by HPLC using a chiral support, and therefore these products were hydrolysed to the diol prior to HPLC analysis: $LiOH•H_2O$ (3 equiv.) was added to a solution of the diester or monoester (1 equiv.) in MeOH (0.3 M) and allowed to stir at 50 °C until completion, based on TLC analysis. The mixture was diluted with EtOAc and washed sequentially with HCl (1 M), saturated $NaHCO_3$ and brine. The organic layer was dried (Na_2SO_4), filtered and concentrated to give the diol product.

Kinetic Resolution of (±)-(1*E*,6*E*)-1,7-diphenylhepta-1,6-diene-3,5-diol ((±)-2a)

According to the **General Procedure**, (±)-(1*E*,6*E*)-1,7-diphenylhepta-1,6-diene-3,5-diol (210 mg, 0.75 mmol) and propionic anhydride (101 μL, 0.787 mmol, 1.05 equiv.), in 1:1 $CHCl_3$:THF mixture (7.5 mL total volume), and *i*-Pr_2NEt (138 μL, 0.787 mmol, 1.05 equiv.) in 1:1 $CHCl_3$:THF mixture

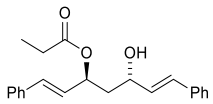
(7.5 mL total volume) gave crude products that were purified by column chromatography (80:20 to 60:40 Hexane:EtOAc) to give:

(1*E*,3*R*,5*R*,6*E*)-1,7-diphenylhepta-1,6-diene-3,5-diol (2a)



(63 mg, 30%) as a colourless solid with spectroscopic data in accordance with the literature.[6] **mp** 140−141 °C {Lit.[6] 149 °C}; $[\alpha]_D^{20}$ +25.0 (*c* 1.0 in $CHCl_3$) {Lit.[6] $[\alpha]_D^{25}$ (>99:1 er) +16.2 (*c* 0.50, $CHCl_3$)}; **Chiral HPLC analysis** Chiralpak IC (90:10 Hexane:IPA, flow rate 1.0 mL min^{-1}, 211 nm, 30 °C) t_R(3*S*,5*S*): 12.3 min, t_R(3*R*,5*R*): 14.0 min, 3.19:96.81 er; **^{1}H NMR** (500 MHz, $CDCl_3$) δ_H: 2.01 (t, *J* 5.4 Hz, 2H, C(2)H_2), 2.57 (d, *J* 3.9 Hz, 2H, OH), 4.72 (s, 2H, C*H*OH), 6.33 (dd, *J* 15.7, 6.2 Hz, 2H, PhCH=C*H*), 6.68 (d, *J* 15.7 Hz, 2H, PhC*H*=CH), 7.23 – 7.27 (m, 2H, ArH), 7.35 (t, *J* 7.3 Hz, 4H, ArH), 7.42 (d, *J* 7.5 Hz, 4H, ArH).

(1*E*,3S,5S,6*E*)-5-hydroxy-1,7-diphenylhepta-1,6-dien-3-yl propionate (3a)

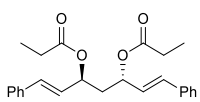


As an inseparable mixture of combined *anti* and *syn* diastereomers (80:20 $dr_{anti:syn}$) (45 mg, 18%) as a colourless oil with spectroscopic data in accordance with the literature.[6,12] $[\alpha]_D^{20}$ −21.5 (*c* 1.0 in $CHCl_3$).

Data for major diastereomer anti-**3a** in accordance with the literature:[6] following hydrolysis to (1*E*,6*E*)-1,7-diphenylhepta-1,6-diene-3,5-diol and the *anti*-diastereomer was purified through crystallization from hot Toluene:Hexane 1:1: **Chiral HPLC analysis** Chiralpak IC (90:10 Hexane:IPA, flow rate 1.0 mL min^{-1}, 211 nm, 30 °C) t_R(3R,5*R*): 12.2 min, t_R(3*R*,5*R*): 14.0 min, 64.99:35.01 er; **^{1}H NMR** (400 MHz, $CDCl_3$) δ_H: 1.11 – 1.24 (m, 3H, CH_3), 1.87 – 2.12 (m, 2H, C(2)H_2), 2.43 (q, *J* 7.5 Hz, 2H, C$H_2$$CH_3$), 2.62 (d, *J* 3.8 Hz, 1H, OH), 4.28 – 4.43 (m, 1H, C*H*OH), 5.71 – 5.82 (m, 1H, C*H*OC(O)Et), 6.15 – 6.34 (m, 2H, PhCH=C*H*), 6.66 (ddd, *J* 16.0, 6.0, 1.1 Hz, 2H, PhC*H*=CH), 7.25 – 7.31 (m, 2H, ArH), 7.32 – 7.38 (m, 4H, ArH), 7.38 – 7.44 (m, 4H, ArH).

Data for minor diastereomer syn-**3a** in accordance with the literature:[12] **^{1}H NMR** (400 MHz, $CDCl_3$) (*selected*) δ_H: 4.41 – 4.51 (m, 1H, C*H*OH), 5.59 – 5.73 (m, 1H, C*H*OC(O)Et).

(1*E*,3*S*,5*S*,6*E*)-1,7-diphenylhepta-1,6-diene-3,5-diyl dipropionate (4a)



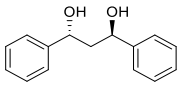
(99 mg, 34%) as a colourless solid with spectroscopic data in accordance with the literature.[6] **mp** 51−52 °C {Lit.[6] 56 °C}; $[\alpha]_D^{20}$ −5.0 (*c* 0.3 in $CHCl_3$) {Lit.[6] $[\alpha]_D^{25}$ (98:2 er) −44.1 (*c* 1.00, $CHCl_3$)};

Chiral HPLC analysis Chiralcel OJ-H (85:15 Hexane:IPA, flow rate 1.5 mL min^{-1}, 211 nm, 30 °C) t_R(3*R*,5*R*): 9.5 min, t_R(3*S*,5*S*): 13.4 min, 4.85:95.15 er.; **^{1}H NMR** (500 MHz, $CDCl_3$) δ_H: 1.16 (t, *J* 7.6 Hz, 6H, CH_3), 2.16 (dd, *J* 7.3, 6.6 Hz, 2H, CH_2), 2.37 (qd, *J* 7.5, 4.5 Hz, 4H, C*H*$_2$CH$_3$), 5.59 (td, *J* 7.4, 0.9 Hz, 2H, C*H*OC(O)Et), 6.18 (dd, *J* 15.9, 7.3 Hz, 2H, PhCH=C*H*), 6.66 (d, *J* 15.9 Hz 2H, PhC*H*=CH), 7.25 – 7.30 (m, 2H, ArH), 7.31 – 7.37 (m, 4H, ArH), 7.37 – 7.42 (m, 4H, ArH).

Kinetic Resolution of (±)-1,3-diphenylpropane-1,3-diol ((±)-2b)

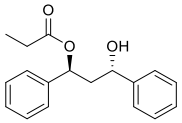
According to the **General Procedure**, (±)-1,3-diphenylpropane-1,3-diol (171 mg, 0.75 mmol) and propionic anhydride (101 µL, 0.787 mmol, 1.05 equiv.), in 1:1 $CHCl_3$:THF mixture (7.5 mL total volume), and *i*-Pr$_2$NEt (138 µL, 0.787 mmol, 1.05 equiv.) in 1:1 $CHCl_3$:THF mixture (7.5 mL total volume) gave crude products that were purified by column chromatography (80:20 to 60:40 Hexane:EtOAc) to give:

(1*R*,3*R*)-1,3-diphenylpropane-1,3-diol (2b)



(60 mg, 35%) as a colourless solid with spectroscopic data in accordance with the literature.[6] **mp** 141−143 °C {Lit.[6] 153°C}; $[\alpha]_D^{20}$ +75.0 (*c* 0.5 in $CHCl_3$) {Lit.[6] $[\alpha]_D^{25}$ (99:1 er) +115.9 (*c* 1.0, $CHCl_3$)}; **Chiral HPLC analysis** Chiralpak ID (90:10 Hexane:IPA, flow rate 1.0 mL min^{-1}, 211 nm, 30 °C) t_R(3*R*,5*R*): 11.4 min, 100:0 er.; **^{1}H NMR** (500 MHz, $CDCl_3$) δ_H: 2.21 (dd, *J* 6.2, 5.4 Hz, 2H, C(2)H$_2$), 5.01 (dd, *J* 6.3, 5.4 Hz, 2H, C*H*OH), 7.29 – 7.33 (m, 2H, ArH), 7.36 – 7.41 (m, 8H, ArH).

(1*S*,3*S*)-3-hydroxy-1,3-diphenylpropyl propionate (3b)



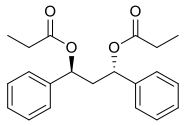
As an inseparable mixture of combined *anti* and *syn* diastereomers (90:10 dr$_{anti:syn}$) (24 mg, 10%) as a colourless oil. $[\alpha]_D^{20}$ −21.8 (*c* 0.45 in $CHCl_3$); following hydrolysis to 1,3-diphenylpropane-1,3-diol **Chiral HPLC analysis** Chiralpak ID (90:10 Hexane:IPA, flow rate 1.0 mL min^{-1}, 211 nm, 30 °C) t_R(3*R*,5*R*): 11.4 min, t_R(3*S*,5*S*): 14.6 min, 33.73:66.27 er; **IR** (neat) ν_{max} cm^{-1}: 3447 (OH), 1754 (C=O), 1495, 1456, 1350, 1277, 1188, 1063, 1022; **HRMS** (ESI$^+$) $C_{18}H_{20}O_3Na$ $[M+Na]^+$ found 307.1299, requires 307.1305 (−1.9 ppm).

Data for major diastereomer anti-**3b**: **^{1}H NMR** (500 MHz, $CDCl_3$) δ_H: 1.19 (t, *J* 7.6 Hz, 3H, CH_3), 2.17 (ddd, *J* 14.5, 9.9, 3.4 Hz, 1H, C(2)H$_2$), 2.29 (ddd, *J* 14.6, 10.3, 3.4 Hz, 1H, C(2)H$_2$), 2.43 (qd, *J* 7.5, 4.4 Hz, 2H, C*H*$_2$CH$_3$), 2.77 (d, *J* 3.6 Hz, 1H, OH), 4.74 (dt, *J* 9.9, 3.5 Hz, 1H, C*H*OH), 6.11 (dd, *J* 10.3, 3.4 Hz, 1H, C*H*OC(O)Et), 7.29 – 7.34 (m, 2H, ArH), 7.34 – 7.42 (m, 8H, ArH); **^{13}C NMR**

(126 MHz, $CDCl_3$) δ_C: 9.2 (*C*H_3), 27.8 (*C*H_2CH$_3$), 46.6 (*C*(2)H_2), 70.4 (*C*HOH), 73.0 (*C*HOC(O)Et), 125.8 (2 × Ar*C*H), 126.4 (2 × Ar*C*H), 127.7 (Ar*C*H), 128.0 (Ar*C*H), 128.6 (4 × Ar*C*H), 140.4 (Ar*C*), 143.7 (Ar*C*), 174.0 (CHO*C*(O)Et).

Data for minor diastereomer syn-**3b** in accordance with the literature:[12] **^{1}H NMR** (500 MHz, $CDCl_3$) (*selected*) δ_H: 4.65 (dd, *J* 8.4, 4.8 Hz, 1H, C*H*OH), 5.90 (dd, *J* 7.8, 6.5 Hz, 1H, C*H*OC(O)Et).

(1*S*,3*S*)-1,3-diphenylpropane-1,3-diyl dipropionate (4b)

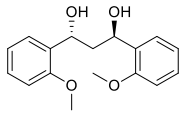


(105 mg, 39%) as a colourless oil with spectroscopic data in accordance with the literature.[6] $[\alpha]_D^{20}$ −89.4 (*c* 0.4 in $CHCl_3$) {Lit.[6] $[\alpha]_D^{25}$ (98:2 er) −48.9 (*c* 1.0, $CHCl_3$)}; following hydrolysis to 1,3-diphenylpropane-1,3-diol **Chiral HPLC analysis** Chiralpak ID (90:10 Hexane:IPA, flow rate 1.0 mL min^{-1}, 211 nm, 30 °C), t_R(3*R*,5*R*): 11.4 min, t_R(3*S*,5*S*): 14.5 min, 3.45:96.55 er; **^{1}H NMR** (500 MHz, $CDCl_3$) δ_H: 1.14 (t, *J* 7.5 Hz, 6H, CH_3), 2.24 – 2.46 (m, 6H, C*H*$_2$CH$_3$ and C(2)H_2), 5.90 (dd, *J* 7.7, 6.5 Hz, 2H, C*H*OC(O)Et), 7.29 – 7.39 (m, 10H, ArH).

Kinetic Resolution of (±)-1,3-bis(2-methoxyphenyl)propane-1,3-diol ((±)-2c)

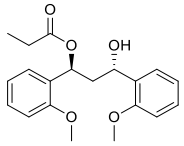
According to the **General Procedure**, (±)-1,3-bis(2-methoxyphenyl)propane-1,3-diol (216 mg, 0.75 mmol) and propionic anhydride (101 µL, 0.787 mmol, 1.05 equiv.), in 1:1 $CHCl_3$:THF mixture (7.5 mL total volume), and *i*-Pr$_2$NEt (138 µL, 0.787 mmol, 1.05 equiv.) in 1:1 $CHCl_3$:THF mixture (7.5 mL total volume) gave crude products that were purified by column chromatography (80:20 to 60:40 Hexane:EtOAc) to give:

(1*R*,3*R*)-1,3-bis(2-methoxyphenyl)propane-1,3-diol (2c)



(85 mg, 39%) as a colourless solid with spectroscopic data in accordance with the literature.[6] **mp** 106−107 °C {Lit.[6] 117 °C}; $[\alpha]_D^{20}$ +84.3 (*c* 1.0 in $CHCl_3$) {Lit.[6] $[\alpha]_D^{25}$ (>99:1 er) +121.8 (*c* 1.0, $CHCl_3$)}; **Chiral HPLC analysis** Chiralpak ID (80:20 Hexane:IPA, flow rate 1.0 mL min^{-1}, 211 nm, 30 °C) t_R(3*R*,5*R*): 16.9 min, t_R(3*S*,5*S*): 21.1 min, 97.50:2.50 er; **^{1}H NMR** (400 MHz, $CDCl_3$) δ_H: 2.31 (dd, *J* 6.3, 5.3 Hz, 2H, C(2)H_2), 3.83 (s, 6H, OCH_3), 5.23 (t, *J* 5.8 Hz, 2H, C*H*OH), 6.89 (dd, *J* 8.2, 1.1 Hz, 2H, ArH), 7.00 (td, *J* 7.5, 1.1 Hz, 2H, ArH), 7.23 – 7.30 (m, 2H, ArH), 7.41 – 7.48 (m, 2H, ArH).

(1*S*,3*S*)-3-hydroxy-1,3-bis(2-methoxyphenyl)propyl propionate (3c)

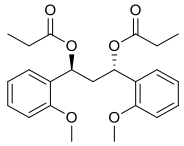


As an inseparable mixture of combined *anti* and *syn* diastereomers (94:6 $dr_{anti:syn}$) (56 mg, 22%) as a colourless oil. $[\alpha]_D^{20}$ −146.3 (*c* 0.6 in $CHCl_3$); following hydrolysis to 1,3-bis(2-methoxyphenyl)propane-1,3-diol **Chiral HPLC analysis** Chiralpak ID (80:20 Hexane:IPA, flow rate 1.0 mL min^{-1}, 211 nm, 30 °C) t_R(3*R*,5*R*): 16.7 min, t_R(3*S*,5*S*): 20.2 min, 14.77:85.23 er; **IR** (neat) ν_{max} cm^{-1}: 3450 (OH), 2940; 1732 (C=O), 1490, 1462; 1287; 1242; 1186; 1049; 1028; **HRMS** (ESI^+) $C_{20}H_{24}O_5Na$ $[M+Na]^+$ found 367.1502, requires 367.1516 (−3.8 ppm).

Data for major diastereomer anti-**3c**: **^{1}H NMR** (400 MHz, $CDCl_3$) δ_H: 1.20 (t, *J* 7.6 Hz, 3H, CH_3), 2.11 – 2.35 (m, 2H, C(2)H_2), 2.41 (qd, *J* 7.6, 0.9 Hz, 2H, C$H_2$$CH_3$), 3.20 (d, *J* 5.8 Hz, 1H, OH), 3.85 (s, 3H, OCH_3), 3.87 (s, 3H, OCH_3), 5.01 (dd, *J* 9.4, 3.5 Hz, 1H, C*H*OH), 6.50 (dd, *J* 9.9, 3.1 Hz, 1H, C*H*OC(O)Et), 6.88 (ddd, *J* 8.2, 4.4, 1.1 Hz, 2H, ArH), 6.92 − 7.01 (m, 2H, ArH), 7.20 – 7.28 (m, 2H, ArH), 7.35 (dd, *J* 7.6, 1.8 Hz, 1H, ArH), 7.38 – 7.44 (m, 1H, ArH); **^{13}C NMR** (126 MHz, $CDCl_3$) δ_C: 9.3 (*C*H_3), 27.9 (*C*$H_2$$CH_3$), 43.1 (*C*(2)$H_2$), 55.3 (O*C*$H_3$), 55.5 (O*C*$H_3$), 66.8 (*C*HOH), 68.1 (*C*HOC(O)Et), 110.3 (Ar*C*H), 110.6 (Ar*C*H), 120.5 (Ar*C*H), 120.8 (Ar*C*H), 126.2 (Ar*C*H), 126.8 (Ar*C*H), 128.2 (Ar*C*H), 128.7 (Ar*C*H), 129.4 (Ar*C*), 131.9 (Ar*C*), 156.0 (Ar*C*), 156.2 (Ar*C*), 174.3 (CHO*C*(O)Et).

Data for minor diastereomer syn-**3c** in accordance with the literature:[12] **^{1}H NMR** (400 MHz, $CDCl_3$) (*selected*) δ_H: 4.91 – 4.99 (m, 1H, C*H*OH), 6.16 – 6.30 (m, 1H, C*H*OC(O)Et).

(1*S*,3*S*)-1,3-bis(2-methoxyphenyl)propane-1,3-diyl dipropionate (4c)

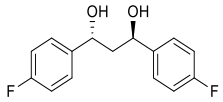


(75 mg, 25%) as a colorless oil with spectroscopic data in accordance with the literature.[6] $[\alpha]_D^{20}$ −48.0 (*c* 1.0 in $CHCl_3$) {Lit.[6] $[\alpha]_D^{25}$ (>99:1 er) −22.8 (*c* 1.0, $CHCl_3$)}; following hydrolysis to 1,3-bis(2-methoxyphenyl)propane-1,3-diol **Chiral HPLC analysis** Chiralpak ID (80:20 Hexane:IPA, flow rate 1.0 mL min^{-1}, 211 nm, 30 °C) t_R(3*R*,5*R*): 17.1 min, t_R(3*S*,5*S*): 20.4 min, 0.46:99.54 er; **^{1}H NMR** (400 MHz, $CDCl_3$) δ_H: 1.18 (t, *J* 7.6 Hz, 6H, CH_3), 2.27 (dd, *J* 7.3, 5.9 Hz, 2H, C(2)H_2), 2.39 (qd, *J* 7.5, 3.2 Hz, 4H, C$H_2$$CH_3$), 3.85 (s, 6H, OCH_3), 6.33 (dd, *J* 7.3, 5.9 Hz, 2H, C*H*OC(O)Et), 6.85 (dd, *J* 8.2, 1.1 Hz, 2H, ArH), 6.94 (td, *J* 7.5, 1.1 Hz, 2, ArH), 7.23 (ddd, *J* 8.2, 7.4, 1.8 Hz, 2H, ArH), 7.31 (dd, *J* 7.6, 1.8 Hz, 2H, ArH).

Kinetic Resolution of (±)-1,3-bis(4-fluorophenyl)propane-1,3-diol ((±)-2d)

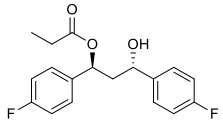
According to the **General Procedure**, (±)-1,3-bis(4-fluorophenyl)propane-1,3-diol (198 mg, 0.75 mmol) and propionic anhydride (101 µL, 0.787 mmol, 1.05 equiv.), in 1:1 $CHCl_3$:THF mixture (7.5 mL total volume), and *i*-Pr_2NEt (138 µL, 0.787 mmol, 1.05 equiv.) in 1:1 $CHCl_3$:THF mixture (7.5 mL total volume) gave crude products that were purified by column chromatography (80:20 to 60:40 Hexane:EtOAc) to give:

(1*R*,3*R*)-1,3-bis(4-fluorophenyl)propane-1,3-diol (2d)



(65 mg, 32%) as a colourless solid with spectroscopic data in accordance with the literature.[6] **mp** 115−117 °C {Lit.[6] 121.5 °C}; $[\alpha]_D^{20}$ +50.6 (*c* 1.0 in $CHCl_3$) {Lit.[6] $[\alpha]_D^{25}$ (>99:1 er) +73.5 (*c* 1.0, $CHCl_3$)}; **Chiral HPLC analysis** Chiralcel OJ-H (80:20 Hexane:IPA, flow rate 1.0 mL min^{-1}, 211 nm, 30 °C) t_R(3*R*,5*R*): 6.4 min, t_R(3*S*,5*S*): 7.3 min, 99.49:0.51 er; **^{1}H NMR** (400 MHz, $CDCl_3$) δ_H: 2.14 (dd, *J* 6.3, 5.3 Hz, 2H, C(2)H_2), 4.99 (t, *J* 5.8 Hz, 2H, C*H*OH), 7.02 – 7.11 (m, 4H, ArH), 7.31 – 7.39 (m, 4H, ArH); **^{19}F NMR** (377 MHz, $CDCl_3$) δ_F: −115.02.

(1*S*,3*S*)-1,3-bis(4-fluorophenyl)-3-hydroxypropyl propionate (3d)

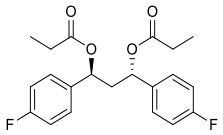


As an inseparable mixture of combined *anti* and *syn* diastereomers (92:8 $dr_{anti:syn}$) (34 mg, 14%) as a colourless oil. $[\alpha]_D^{20}$ −5.9 (*c* 0.45 in $CHCl_3$); following hydrolysis to 1,3-bis(4-fluorophenyl)propane-1,3-diol **Chiral HPLC analysis** Chiralcel OJ-H (80:20 Hexane:IPA, flow rate 1.0 mL min^{-1}, 211 nm, 30 °C) t_R(3*R*,5*R*): 6.4 min, t_R(3*S*,5*S*): 7.3 min, 40.40:59.60 er; **IR** (neat) ν_{max} cm^{-1}: 3447 (OH), 1732 (C=O), 1605; 1510, 1223; 1186; 1157; **HRMS** (ESI^+) $C_{18}H_{18}O_3F_2Na$ $[M+Na]^+$ found 343.1105, requires 343.1116 (−3.2 ppm).

Data for major diastereomer anti-**3d**: **^{1}H NMR** (400 MHz, $CDCl_3$) δ_H: 1.18 (t, *J* 7.6 Hz, 3H, CH_3), 2.11 (ddd, *J* 14.5, 9.8, 3.4 Hz, 1H, C(2)H_2), 2.24 (ddd, *J* 14.5, 10.2, 3.5 Hz, 1H, C(2)H_2), 2.42 (qd, *J* 7.5, 3.9 Hz, 2H, C$H_2$$CH_3$), 2.78 (bs, 1H, OH), 4.71 (dd, *J* 9.8, 3.5 Hz, 1H, C*H*OH), 6.06 (dd, *J* 10.3, 3.4 Hz, 1H, C*H*OC(O)Et), 6.99 – 7.11 (m, 4H, ArH), 7.31 – 7.42 (m, 4H, ArH); **^{13}C NMR** (126 MHz, $CDCl_3$) δ_C: 9.1 (*C*H_3), 27.8 (*C*$H_2$$CH_3$), 46.6 (*C*(2)$H_2$), 69.7 (*C*HOH), 72.3 (*C*HOC(O)Et), 115.3 (dd, $^2J_{C\text{-}F}$ 16.9 Hz, 2 × Ar*C*H), 115.5 (dd, $^2J_{C\text{-}F}$ 16.9 Hz, 2 × Ar*C*H), 127.4 (d, $^3J_{C\text{-}F}$ 8.1 Hz, 2 × Ar*C*H), 128.2 (d, $^3J_{C\text{-}F}$ 8.2 Hz, 2 × Ar*C*H), 136.1 (d, $^4J_{C\text{-}F}$ 3.1 Hz, Ar*C*), 139.3 (d, $^4J_{C\text{-}F}$ 3.0 Hz, Ar*C*), 161.3 (d, $^1J_{C\text{-}F}$ 246.2 Hz, Ar*C*), 163.4 (d, $^1J_{C\text{-}F}$ 246.2 Hz Ar*C*), 174.5 (CHO*C*(O)Et); **^{19}F NMR** (377 MHz, $CDCl_3$) δ_F: −113.90, −114.81.

Data for minor diastereomer syn-**3d** in accordance with the literature:[12] **^{1}H NMR** (400 MHz, $CDCl_3$) (*selected*) δ_H: 4.60 (dd, *J* 8.5, 4.8 Hz, 1H, C*H*OH), 5.87 (t, *J* 7.1 Hz, 1H, C*H*OC(O)Et); **^{19}F NMR** (377 MHz, $CDCl_3$) δ_F: −113.79, −114.46.

(1*S*,3*S*)-1,3-bis(4-fluorophenyl)propane-1,3-diyl dipropionate (4d)

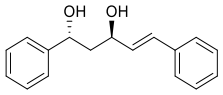


(113 mg, 40%) as a colourless oil with spectroscopic data in accordance with the literature.[6] $[\alpha]_D^{20}$ −66.1 (*c* 0.6 in $CHCl_3$) {Lit.[6] $[\alpha]_D^{25}$ (99:1 er) −68.4 (*c* 1.0, $CHCl_3$)}; following hydrolysis to 1,3-bis(4-fluorophenyl)propane-1,3-diol **Chiral HPLC analysis** Chiralcel OJ-H (80:20 Hexane:IPA, flow rate 1.0 mL min^{-1}, 211 nm, 30 °C) t_R(3*R*,5*R*): 6.5 min, t_R(3*S*,5*S*): 7.4 min, 6.72:93.28 er; **^{1}H NMR** (400 MHz, $CDCl_3$) δ_H: 1.13 (t, *J* 7.5 Hz, 6H, CH_3), 2.25 – 2.43 (m, 6H, CH_2CH$_3$ and C(2)H$_2$), 5.79 – 5.88 (m, 2H, C*H*OC(O)Et), 7.00 – 7.08 (m, 4H, ArH), 7.30 – 7.37 (m, 4H, ArH); **^{19}F NMR** (377 MHz, $CDCl_3$) δ_F: −113.71.

Kinetic Resolution of (±)-1,5-diphenylpent-4-ene-1,3-diol ((±)-2e)

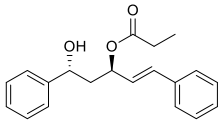
According to the **General Procedure**, (±)-1,5-diphenylpent-4-ene-1,3-diol (191 mg, 0.75 mmol) and propionic anhydride (101 µL, 0.787 mmol, 1.05 equiv.), in 1:1 $CHCl_3$:THF mixture (7.5 mL total volume), and *i*-Pr$_2$NEt (138 µL, 0.787 mmol, 1.05 equiv.) in 1:1 $CHCl_3$:THF mixture (7.5 mL total volume) gave crude products that were purified by column chromatography (80:20 to 60:40 Hexane:EtOAc) to give:

(1*R*,3*R*,*E*)-1,5-diphenylpent-4-ene-1,3-diol (2e)



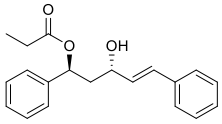
(60 mg, 32%) as a white solid with spectroscopic data in accordance with the literature.[6] **mp** 106−107 °C {Lit.[6] 106 °C}; $[\alpha]_D^{20}$ −13.5 (*c* 1.0 in $CHCl_3$) {Lit.[6] $[\alpha]_D^{25}$ (>99:1 er) −11.8 (*c* 1.0, $CHCl_3$)}; **Chiral HPLC analysis** Chiralcel OD-H (80:20 Hexane:IPA, flow rate 1.0 mL min^{-1}, 254 nm, 30 °C) t_R(3*S*,5*S*): 10.5 min, t_R(3*R*,5*R*): 17.5 min, 1.91:98.09 er; **^{1}H NMR** (400 MHz, $CDCl_3$) δ_H: 2.06 (ddd, *J* 14.5, 7.7, 3.3 Hz, 1H, C(2)H$_2$), 2.17 (ddd, *J* 14.6, 8.5, 3.5 Hz, 1H, C(2)H$_2$), 4.55 – 4.69 (m, 1H, C(3)*H*OH), 5.12 (dd, *J* 8.5, 3.4 Hz, 1H, C(1)*H*OH), 6.34 (dd, *J* 15.9, 6.1 Hz, 1H, PhCH=C*H*), 6.66 (dd, *J* 15.9, 1.4 Hz, 1H, PhC*H*=CH), 7.32 – 7.45 (m, 10H, ArH).

(3*R*,5*R*,*E*)-5-hydroxy-1,5-diphenylpent-1-en-3-yl propionate (3e′)



(21 mg, 9%) as a pale yellow oil. $[\alpha]_D^{20}$ −5.6 (*c* 0.25 in $CHCl_3$); following hydrolysis to 1,5-diphenylpent-4-ene-1,3-diol **Chiral HPLC analysis** Chiralcel OD-H (80:20 Hexane:IPA, flow rate 1.0 mL min^{-1}, 211 nm, 30 °C) t_R(3*S*,5*S*): 10.4 min, t_R(3*R*,5*R*): 18.1 min, 40.75:59.25 er; **IR** (neat) ν_{max} cm^{-1}: 3447 (OH), 3028, 1732 (C=O), 1494, 1450, 1273, 1186, 1080, 1066; **^{1}H NMR** (400 MHz, $CDCl_3$) δ_H: 1.21 (t, *J* 7.6 Hz, 3H, CH_3), 2.05 – 2.25 (m, 2H, C(2)H_2), 2.43 (q, *J* 7.5 Hz, 2H, C$H_2$$CH_3$), 4.66 – 4.82 (m, 1H, C*H*OH), 5.76 (qd, *J* 6.8, 1.1 Hz, 1H, C*H*OC(O)Et), 6.21 (dd, *J* 15.9, 7.0 Hz, 1H, PhCH=C*H*), 6.66 (d, *J* 15.9 Hz, 1H, PhC*H*=CH), 7.29 – 7.44 (m, 10H, ArH); **^{13}C NMR** (126 MHz, $CDCl_3$) δ_C: 9.2 (*C*H_3), 27.9 (*C*$H_2$$CH_3$), 44.6 (*C*(2)$H_2$), 70.3 (*C*HOH), 71.9 (*C*HOC(O)Et), 125.8 (2 × Ar*C*H), 126.6 (2 × Ar*C*H), 127.3 (Ar*C*H), 127.7 (Ar*C*H), 128.1 (PhCH=*C*H), 128.6 (4 × Ar*C*H), 132.4 (Ph*C*H=CH), 136.2 (Ar*C*), 143.7 (Ar*C*), 174.7 (CHO*C*(O)Et); **HRMS** (ESI^+) $C_{20}H_{22}O_3Na$ $[M+Na]^+$ found 333.1457, requires 333.1461 (−1.2 ppm).

(1*S*,3*S*,*E*)-3-hydroxy-1,5-diphenylpent-4-en-1-yl propionate (3e′′)

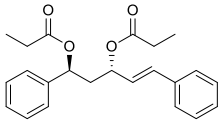


As an inseparable mixture of combined *anti* and *syn* diastereomers (89:11 $dr_{anti:syn}$) (14 mg, 6%) as a pale yellow oil. $[\alpha]_D^{20}$ +23.6 (*c* 0.25 in $CHCl_3$); following hydrolysis to 1,5-diphenylpent-4-ene-1,3-diol **Chiral HPLC analysis** Chiralcel OD-H (80:20 Hexane:IPA, flow rate 1.0 mL min^{-1}, 211 nm, 30 °C) t_R(3*S*,5*S*): 10.5 min, t_R(3*R*,5*R*): 18.4 min, 56.02:43.98 er; **IR** (neat) ν_{max} cm^{-1}: 3429 (OH), 1754 (C=O), 1494, 1450, 1275, 1186, 1080, 1068; **HRMS** (ESI^+) $C_{20}H_{22}O_3Na$ $[M+Na]^+$ found 333.1459, requires 333.1461 (−0.6 ppm).

Data for major diastereomer anti-**3e′′**: **^{1}H NMR** (500 MHz, $CDCl_3$) δ_H: 1.17 (t, *J* 7.5 Hz, 3H, CH_3), 2.05 (ddd, *J* 14.4, 9.5, 3.6 Hz, 1H, C(2)H_2), 2.43 (qd, *J* 7.6, 4.9 Hz, 2H, C$H_2$$CH_3$), 2.17 – 2.26 (m, 1H, OH), 4.30 − 4.39 (m, 1H, C*H*OH), 6.08 (dd, *J* 10.2, 3.6 Hz, 1H, C*H*OC(O)Et), 6.21 – 6.30 (m, 1H, PhCH=C*H*), 6.60 – 6.71 (m, 1H, PhC*H*=CH), 7.30 – 7.36 (m, 2H, ArH), 7.36 – 7.43 (m, 8H, ArH); **^{13}C NMR** (126 MHz, $CDCl_3$) δ_C: 9.1 (*C*H_3), 27.8 (*C*$H_2$$CH_3$), 44.7 (*C*(2)$H_2$), 68.9 (*C*HOH), 72.8 (*C*HOC(O)Et), 126.4 (2 × Ar*C*H), 126.5 (2 × Ar*C*H), 127.7 (Ar*C*H), 128.1 (Ar*C*H), 128.6 (4 × Ar*C*H), 130.4 (Ph*C*H=CH), 131.1 (PhCH=*C*H), 136.6 (Ar*C*), 140.4 (Ar*C*), 174.6 (CHO*C*(O)Et).

Data for minor diastereomer syn-**3e′′**: **^{1}H NMR** (500 MHz, $CDCl_3$) *(selected)* δ_H: 4.23 – 4.32 (m, 1H, C*H*OH), 6.00 (dd, *J* 7.7, 6.5 Hz, 1H, C*H*OC(O)Et), 6.56 (d, *J* 16.0 Hz, 1H, PhC*H*=CH); **^{13}C NMR** (126 MHz, $CDCl_3$) *(selected)* δ_C:70.1 (*C*HOH), 73.6 (*C*HOC(O)Et).

(1*S*,3*S*,*E*)-1,5-diphenylpent-4-ene-1,3-diyl dipropionate (4e)

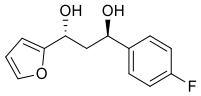


(100 mg, 36%) as a pale yellow oil with spectroscopic data in accordance with the literature.[6] $[\alpha]_D^{20}$ −53.2 (*c* 1.0 in $CHCl_3$) {Lit.[5] $[\alpha]_D^{25}$ (98:2 er) −53.12 (*c* 1.0, $CHCl_3$)}; following hydrolysis to 1,5-diphenylpent-4-ene-1,3-diol **Chiral HPLC analysis** Chiralcel OD-H (80:20 Hexane:IPA, flow rate 1.0 mL min^{-1}, 211 nm, 30 °C) t_R(3*S*,5*S*): 10.5 min, t_R(3*R*,5*R*): 18.0 min, 94.91:5.09 er; **^{1}H NMR** (400 MHz, $CDCl_3$) δ_H: 1.15 (dt, *J* 15.2, 7.5 Hz, 6H, CH_3), 2.17 – 2.44 (m, 6H, C*H*$_2$CH$_3$ and C(2)H$_2$), 5.58 (td, *J* 8.8, 4.7 Hz, 1H, C(1)*H*OC(O)Et), 5.90 (dd, *J* 9.7, 4.5 Hz, 1H, C(3)*H*OC(O)Et), 6.15 (dd, *J* 15.9, 7.3 Hz, 1H, PhCH=C*H*), 6.55 – 6.72 (m, 1H, PhC*H*=CH), 7.30 – 7.41 (m, 10H, ArH).

Kinetic Resolution of (±)-1-(4-fluorophenyl)-3-(furan-2-yl)propane-1,3-diol ((±)-2f)

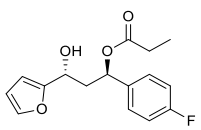
According to the **General Procedure**, (±)-1-(4-fluorophenyl)-3-(furan-2-yl)propane-1,3-diol (177 mg, 0.75 mmol) and propionic anhydride (101 µL, 0.787 mmol, 1.05 equiv.), in 1:1 $CHCl_3$:THF mixture (7.5 mL total volume), and *i*-Pr_2NEt (138 µL, 0.787 mmol, 1.05 equiv.) in 1:1 $CHCl_3$:THF mixture (7.5 mL total volume) gave crude products that were purified by column chromatography (80:20 to 60:40 Hexane:EtOAc) to give:

(1*R*,3*R*)-1-(4-fluorophenyl)-3-(furan-2-yl)propane-1,3-diol (2f)



(77 mg, 44%) as a pale yellow solid with spectroscopic data in accordance with the literature.[6] **mp** 141−144 °C {Lit.[6] 139 °C}; $[\alpha]_D^{20}$ +29.7 (*c* 0.5 in $CHCl_3$) {Lit.[6] $[\alpha]_D^{25}$ (97:3 er) +7.8 (*c* 0.5, $CHCl_3$)}; **Chiral HPLC analysis** Chiralcel ID (90:10 Hexane:IPA, flow rate 1.0 mL min^{-1}, 211 nm, 30 °C) t_R(3*R*,5*R*): 11.0 min, t_R(3*S*,5*S*): 12.8 min, 99.73:0.27 er.; **^{1}H NMR** (500 MHz, $CDCl_3$) δ_H: 2.00 – 2.16 (m, 2H, OH), 2.17 – 2.36 (m, 2H, C(2)H$_2$), 4.96 – 5.09 (m, 2H, C(1)*H*OH and C(3)*H*OH), 6.30 (dt, *J* 3.3, 0.8 Hz, 1H, ArH), 6.37 (dd, *J* 3.3, 1.8 Hz, 1H, ArH), 7.04 – 7.09 (m, 2H, ArH), 7.34 – 7.39 (m, 2H, ArH), 7.41 (dd, *J* 1.8, 0.9 Hz, 1H, ArH); **^{19}F NMR** (471 MHz, $CDCl_3$) δ_F: −115.04.

(1*R*,3*R*)-3-(4-fluorophenyl)-1-(furan-2-yl)-3-hydroxypropyl propionate (3f′)



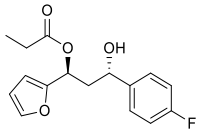
As an inseparable mixture of combined *anti* and *syn* diastereomers (82:18 $dr_{anti:syn}$) (9 mg, 4%) as a pale yellow oil. $[\alpha]_D^{20}$ +24.5 (*c* 0.1 in $CHCl_3$); following hydrolysis to 1-(4-fluorophenyl)-3-(furan-2-

yl)propane-1,3-diol **Chiral HPLC analysis** Chiralcel ID (90:10 Hexane:IPA, flow rate 1.0 mL min^{-1}, 211 nm, 30 °C) t_R(3*R*,5*R*): 11.1 min, t_R(3*S*,5*S*): 12.8 min, 72.97:27.03 er; **IR** (neat) ν_{max} cm^{-1}: 3439 (OH), 1732 (C=O), 1606, 1510, 1223, 1182, 1157, 1080, 1065, 1011; **HRMS** ($C_{16}H_{17}O_4FNa$ $[M+Na]^+$ found 315.0995, requires 315.1009 (−4.4 ppm).

Data for major diastereomer anti-**3f′**: **^{1}H NMR** (500 MHz, $CDCl_3$) δ_H: 1.16 (t, *J* 7.5 Hz, 3H, CH_3), 2.26 (ddd, *J* 14.4, 9.7, 3.6 Hz, 1H, C(2)H_2), 2.34 – 2.47 (m, 3H, C$H_2$$CH_3$ and C(2)H_2), 2.70 (d, *J* 4.9 Hz, 1H, OH), 4.75 (dt, *J* 9.2, 4.2 Hz, 1H, C(1)*H*OH), 6.04 (dd, *J* 10.3, 3.6 Hz, 1H, C(3)*H*OC(O)Et), 6.28 (dt, *J* 3.2, 0.8 Hz, 1H, ArH), 6.33 – 6.38 (m, 1H, ArH), 7.02 – 7.09 (m, 2H, ArH), 7.33 – 7.38 (m, 2H, ArH), 7.40 (dd, *J* 1.8, 0.8 Hz, 1H, ArH); **^{13}C NMR** (126 MHz, $CDCl_3$) δ_C: 9.1 (*C*H_3), 27.7 (*C*$H_2$$CH_3$), 42.6 (*C*(2)$H_2$), 64.0 (*C*(1)HOH), 72.0 (*C*(3)HOC(O)Et), 106.2 (Ar*C*H), 110.3 (Ar*C*H), 115.5 (d, $^2J_{C\text{-}F}$ 21.5 Hz, 2 ×Ar*C*H), 128.2 (d, $^3J_{C\text{-}F}$ 8.2 Hz 2 ×Ar*C*H), 136.2 (d, $^4J_{C\text{-}F}$ 3.2 Hz, Ar*C*), 142.2 (Ar*C*H), 155.6 (Ar*C*), 162.4 (d, $^1J_{C\text{-}F}$ 246.6 Hz, Ar*C*), 174.4 (CHO*C*(O)Et); **^{19}F NMR** (471 MHz, $CDCl_3$) δ_F: −113.95

Data for minor diastereomer syn-**3f′**: **^{1}H NMR** (500 MHz, $CDCl_3$) *(selected)* δ_H: 4.61 (dd, *J* 8.3, 5.4 Hz, 1H, C(1)*H*OH), 5.89 (t, *J* 7.2 Hz, 1H, C(3)*H*OC(O)Et); **^{13}C NMR** *(selected)* (126 MHz, $CDCl_3$) δ_C: 64.9 (*C*(1)HOH), 72.8 (*C*(3)HOC(O)Et); **^{19}F NMR** (471 MHz, $CDCl_3$) δ_F: −113.85.

(1*S*,3*S*)-1-(4-fluorophenyl)-3-(furan-2-yl)-3-hydroxypropyl propionate (3f′′)

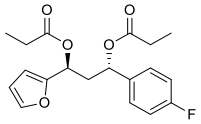


As an inseparable mixture of combined *anti* and *syn* diastereomers (71:29 $dr_{anti:syn}$) (14 mg, 6%) as a pale yellow oil. $[\alpha]_D^{20}$ −15.7 (*c* 0.07 in $CHCl_3$); following hydrolysis to 1-(4-fluorophenyl)-3-(furan-2-yl)propane-1,3-diol **Chiral HPLC analysis** Chiralcel ID (90:10 Hexane:IPA, flow rate 1.0 mL min^{-1}, 211 nm, 30 °C) t_R(3*R*,5*R*): 11.1 min, t_R(3*S*,5*S*): 12.8 min, 45.34:54.66 er; **IR** (neat) ν_{max} cm^{-1}: 3447 (OH), 2984, 1732 (C=O), 1605, 1510, 1221, 1182, 1157, 1067, 1013; **HRMS** (ESI^+) $C_{16}H_{17}O_4FNa$ $[M+Na]^+$ found 315.0997, requires 315.1009 (−3.8 ppm).

Data for major diastereomer anti-**3f′′**:**^{1}H NMR** (500 MHz, $CDCl_3$) δ_H: 1.18 (t, *J* 7.6 Hz, 3H, CH_3), 2.25 (ddd, *J* 14.4, 9.9, 3.8 Hz, 1H, C(2)H_2), 2.34 – 2.49 (m, 3H, C$H_2$$CH_3$ and C(2)H_2), 2.83 (d, *J* 3.7 Hz, 1H, OH), 4.69 (dt, *J* 9.8, 3.6 Hz, 1H, C(1)*H*OH), 6.15 (dd, *J* 10.1, 3.7 Hz, 1H, C(3)*H*OC(O)Et), 6.24 – 6.46 (m, 2H, ArH), 6.96 − 7.11 (m, 2H, ArH), 7.32 – 7.38 (m, 2H, ArH), 7.41 (dd, *J* 1.8, 0.9 Hz, 1H, ArH); **^{13}C NMR** (126 MHz, $CDCl_3$) δ_C: 9.1 (*C*H_3), 27.6 (*C*$H_2$$CH_3$), 42.5 (*C*(2)$H_2$), 65.9 (*C*(3)HOC(O)Et), 69.4 (*C*(1)HOH), 108.5 (Ar*C*H), 110.3 (Ar*C*H), 115.3 (d, $^2J_{C\text{-}F}$ 21.4 Hz, 2 × Ar*C*H), 127.4 (d, $^3J_{C\text{-}F}$ 8.1 Hz, 2 × Ar*C*H), 139.2 (d, $^4J_{C\text{-}F}$ 3.0 Hz, Ar*C*), 142.7 (Ar*C*H), 152.2 (Ar*C*), 161.2 (d, $^1J_{C\text{-}F}$ 245.6 Hz, Ar*C*), 174.7 (CHO*C*(O)Et); **^{19}F NMR** (471 MHz, $CDCl_3$) δ_F: −114.90.

Data for minor diastereomer syn-**3f′′**: **^{1}H NMR** (500 MHz, $CDCl_3$) *(selected)* δ_H: 4.64 (dd, *J* 8.6, 4.7 Hz, 1H, C(1)*H*OH), 6.04 (t, *J* 7.2 Hz, 1H, C(3)*H*OC(O)Et); **^{13}C NMR** (126 MHz, $CDCl_3$) *(selected)* δ_C: 45.0 (*C*(2)H_2), 66.5 (*C*(3)HOC(O)Et), 71.0 (*C*(1)HOH); **^{19}F NMR** (471 MHz, $CDCl_3$) δ_F: −114.65.

(1*S*,3*S*)-1-(4-fluorophenyl)-3-(furan-2-yl)propane-1,3-diyl dipropionate (4f)

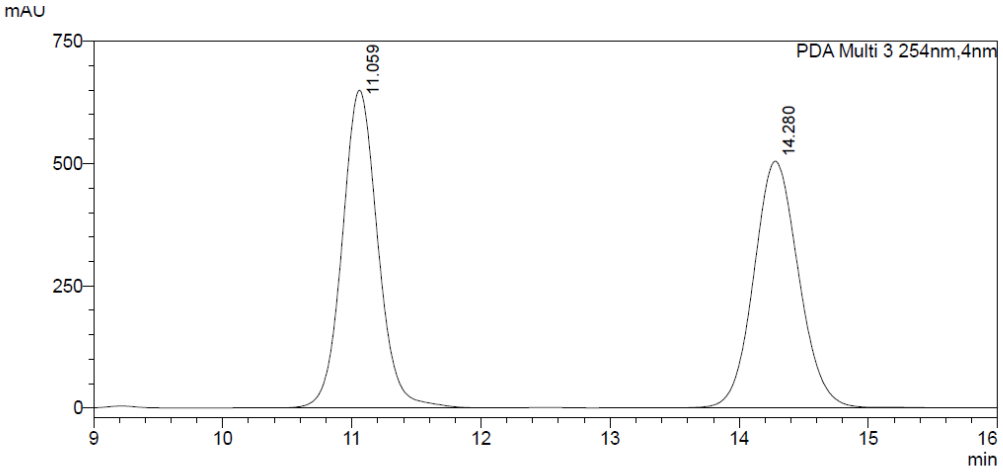


(82 mg, 32%) as a yellow oil with spectroscopic data in accordance with the literature.[6] $[\alpha]_D^{20}$ −88.8 (*c* 1.2 in $CHCl_3$) {Lit.[6] $[\alpha]_D^{25}$ (98:2 er) −135.3 (*c* 1.0, $CHCl_3$)}; following hydrolysis to 1,5-diphenylpent-4-ene-1,3-diol **Chiral HPLC analysis** Chiralcel ID (90:10 Hexane:IPA, flow rate 1.0 mL min^{-1}, 211 nm, 30 °C) t_R(3*R*,5*R*): 11.1 min, t_R(3*S*,5*S*): 12.8 min, 7.98:92.02 er; **^{1}H NMR** (500 MHz, $CDCl_3$) δ_H: 1.13 (q, *J* 7.5 Hz, 6H, CH_3), 2.27 – 2.42 (m, 4H, C*H*$_2$$CH_3$), 2.44 – 2.57 (m, 2H, C(2)H_2), 5.79 – 5.86 (m, 1H, C(1)*H*OC(O)Et), 5.93 – 6.03 (m, 1H, C(3)*H*OC(O)Et), 6.35 (d, *J* 1.4 Hz, 2H, ArH), 7.01 – 7.09 (m, 2H, ArH), 7.30 – 7.37 (m, 2H, ArH), 7.40 (t, *J* 1.3 Hz, 1H, ArH); **^{19}F NMR** (471 MHz, $CDCl_3$) δ_F: −113.79.

Procedure for the Kinetic Resolution of (±)-3a in flow-mode conditions

A packed bed reactor consisting of a vertically-mounted Omnifit glass chromatography column [borosilicate glass; length = 100 mm (70 mm adjustable bed height); internal diameter = 10 mm; maximum bed volume 5.6 mL], with a glass cooling jacket was loaded with PS-HyperBTM resin **8** (600 mg; f = 0.89 mmol g^{-1}). The resin was allowed to swell to its maximum volume by pumping 1:1 THF:$CHCl_3$ mixture at 1 mL min^{-1} for 30 min at r.t. using a Gilson 305 HPLC pump. Two syringes were used to inject reagents using a Legato 200 series syringe pump by World Precision Instruments. The first syringe was filled with a solution of (±)-(1*E*,6*E*)-5-hydroxy-1,7-diphenylhepta-1,6-dien-3-yl propionate **3a** (0.3 mmol, 1.0 equiv.) and $(EtCO)_2O$ (0.17 mmol, 0.55 equiv.) in 1:1 THF:$CHCl_3$ mixture (3.0 mL total volume) and the second syringe with *i*-Pr_2NEt (0.17 mmol, 0.55 equiv.) in 1:1 THF:$CHCl_3$ mixture (3.0 mL total volume). Both solutions were injected at 100 μL min^{-1}, mixed in a T-type mixing chamber, and passed through the reactor at a combined flow rate of 200 μL min^{-1}. After complete addition of the reagents from the syringes, a Gilson 305 HPLC pump was connected, and $CHCl_3$ was pumped at 200 μL min^{-1} for 30 min to ensure elution of the products. A solution of 10% MeOH in 1:1 THF:$CHCl_3$ mixture was then pumped at 200 μL min^{-1} for 30 min to wash the column and avoid cross contamination. The column was then prepared for the next KR by pumping 1:1 THF:$CHCl_3$ mixture at 200 μL min^{-1} for 30 min. The mixture was concentrated to remove THF and later diluted with CH_2Cl_2. So, it was washed sequentially with HCl (1 M), saturated $NaHCO_3$ and brine. The organic layer was dried (Na_2SO_4), filtered and concentrated to give the crude products. The corresponding monoester and diester were then isolated by flash chromatography (90:10 Hexane:EtOAc).

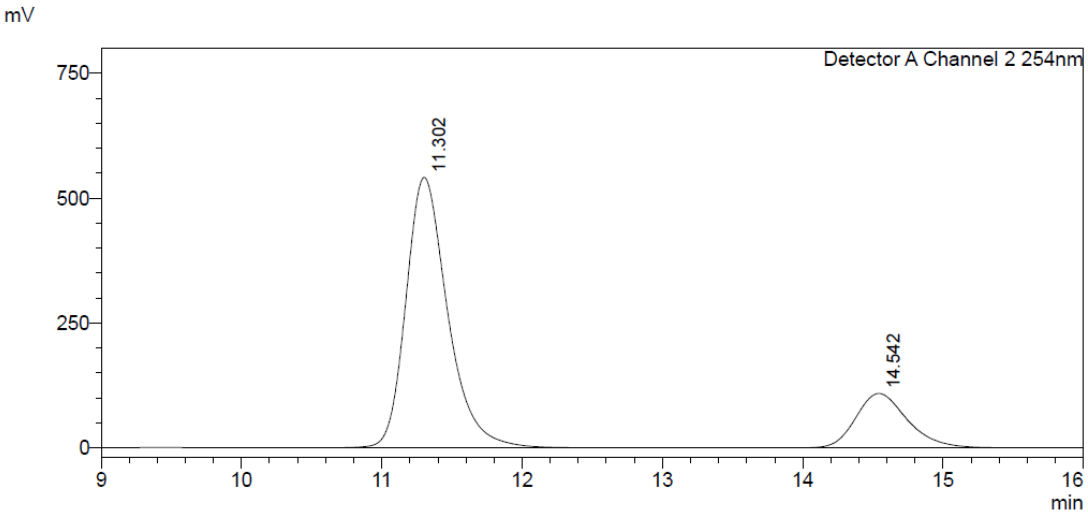
(1*E*,3*R*,5*R*,6*E*)-5-hydroxy-1,7-diphenylhepta-1,6-dien-3-yl propionate (3a): **Chiral HPLC analysis** Chiralpak AD-H (85:15 Hexane:IPA, flow rate 1.0 mL min^{-1}, 254 nm, 30 °C) t_R(3*R*,5*R*): 12.2 min, t_R(3*S*,5*S*): 14.0 min, 80.11:19.89 er.



<Peak Table>

PDA Ch3 254nm

Peak#	Ret. Time	Area%
1	11.059	50.448
2	14.280	49.552
Total		100.000

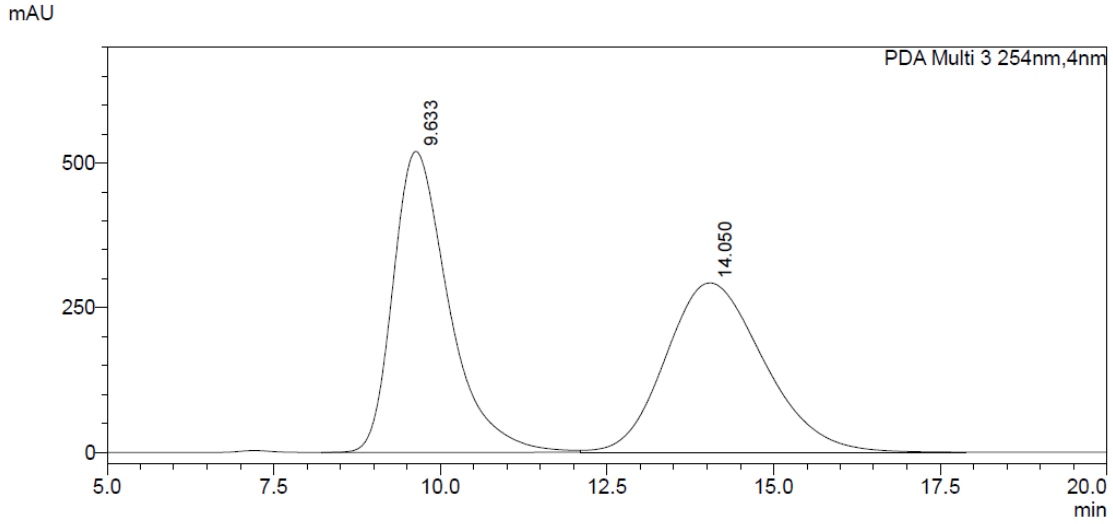


<Peak Table>

Detector A Channel 2 254nm

Peak#	Ret. Time	Area%
1	11.302	80.112
2	14.542	19.888
Total		100.000

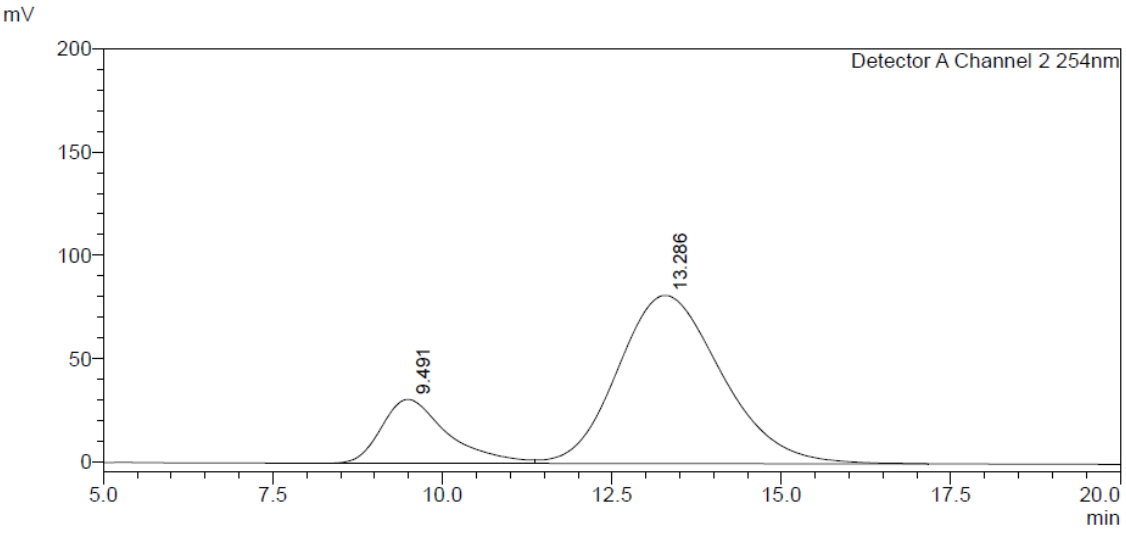
(1*E*,3*S*,5*S*,6*E*)-1,7-diphenylhepta-1,6-diene-3,5-diyl dipropionate (4a): **Chiral HPLC analysis** Chiralcel OJ-H (85:15 Hexane:IPA, flow rate 1.5 mL min^{-1}, 254 nm, 30 °C) t_R(3*R*,5*R*): 9.5 min, t_R(3*S*,5*S*): 13.3 min, 19.38:80.62 er.



<Peak Table>

PDA Ch3 254nm

Peak#	Ret. Time	Area%
1	9.633	50.208
2	14.050	49.792
Total		100.000



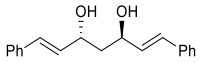
<Peak Table>

Detector A Channel 2 254nm

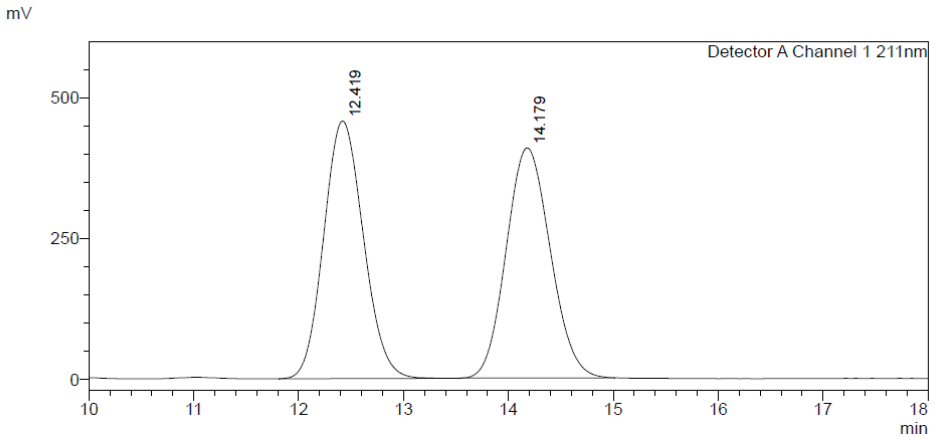
Peak#	Ret. Time	Area%
1	9.491	19.376
2	13.286	80.624
Total		100.000

HPLC chromatograms for the Sequential Acylative Kinetic Resolution of (±)-*anti*-1,3-diols 2

(1*E*,3*R*,5*R*,6*E*)-1,7-diphenylhepta-1,6-diene-3,5-diol (2a)



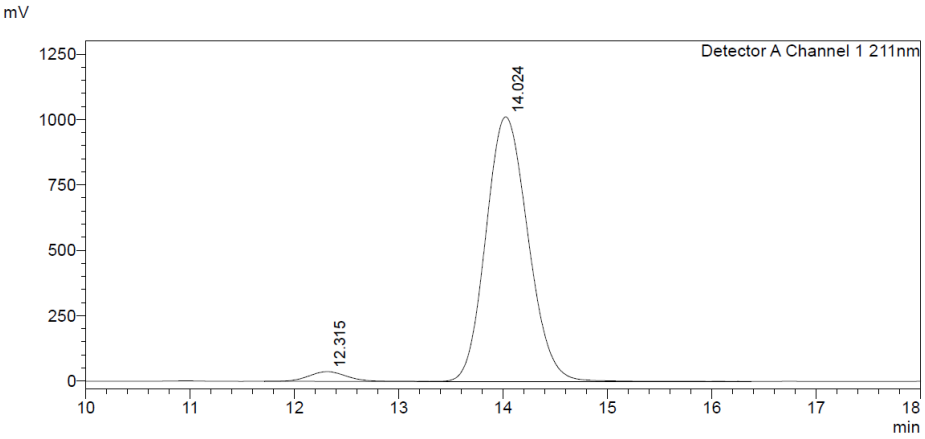
Chiral HPLC analysis Chiralpak IC (90:10 Hexane:IPA, flow rate 1.0 mL min^{-1}, 211 nm, 30 °C) t_R(3*S*,5*S*): 12.3 min, t_R(3*R*,5*R*): 14.0 min, 3.19:96.81 er.



<Peak Table>

Detector A Channel 1 211nm

Peak#	Ret. Time	Area%
1	12.419	50.139
2	14.179	49.861
Total		100.000

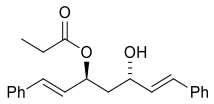


<Peak Table>

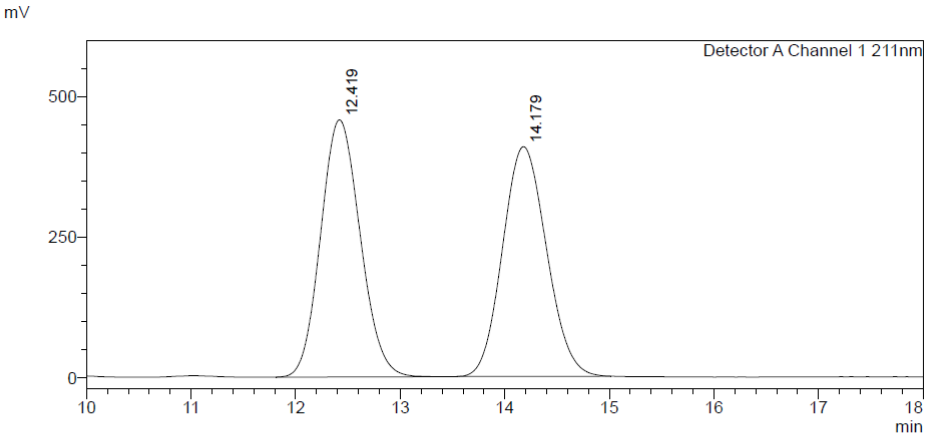
Detector A Channel 1 211nm

Peak#	Ret. Time	Area%
1	12.315	3.195
2	14.024	96.805
Total		100.000

(1*E*,3*S*,5*S*,6*E*)-5-hydroxy-1,7-diphenylhepta-1,6-dien-3-yl propionate (3a)



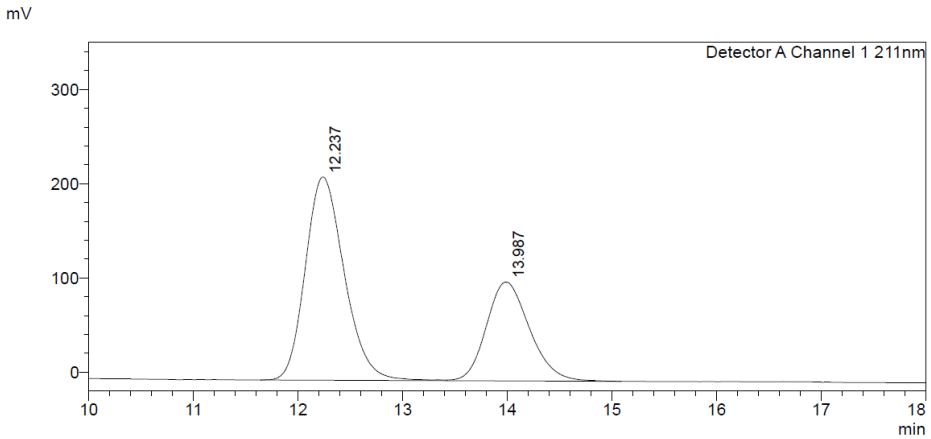
(1*E*,6*E*)-5-hydroxy-1,7-diphenylhepta-1,6-dien-3-yl propionate was hydrolysed to (1*E*,6*E*)-1,7-diphenylhepta-1,6-diene-3,5-diol and the *anti*-diastereomer was purified through crystallization form hot Toluene:Hexane 1:1. **Chiral HPLC analysis** Chiralpak IC (90:10 Hexane:IPA, flow rate 1.0 mL min^{-1}, 211 nm, 30 °C) t_R(3*S*,5*S*): 12.2 min, t_R(3*R*,5*R*): 14.0 min, 64.99:35.01 er.



<Peak Table>

Detector A Channel 1 211nm

Peak#	Ret. Time	Area%
1	12.419	50.139
2	14.179	49.861
Total		100.000

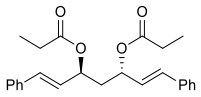


<Peak Table>

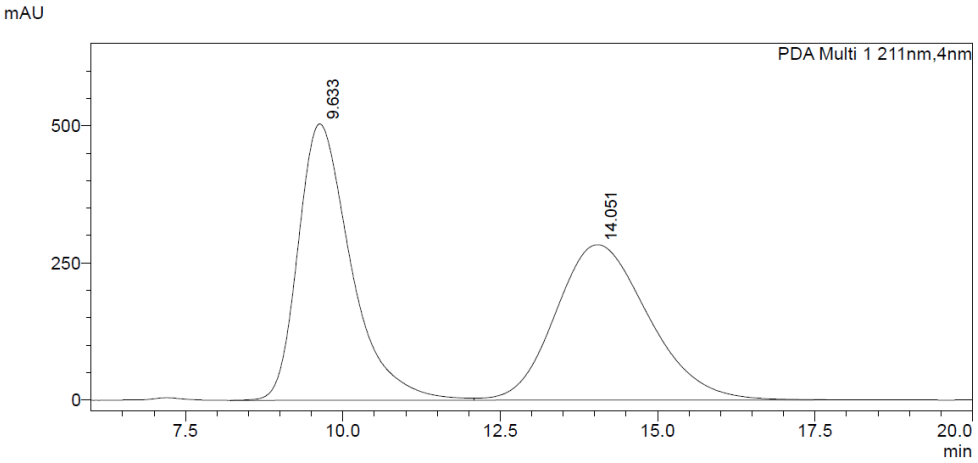
Detector A Channel 1 211nm

Peak#	Ret. Time	Area%
1	12.237	64.989
2	13.987	35.011
Total		100.000

(1*E*,3*S*,5*S*,6*E*)-1,7-diphenylhepta-1,6-diene-3,5-diyl dipropionate (4a)



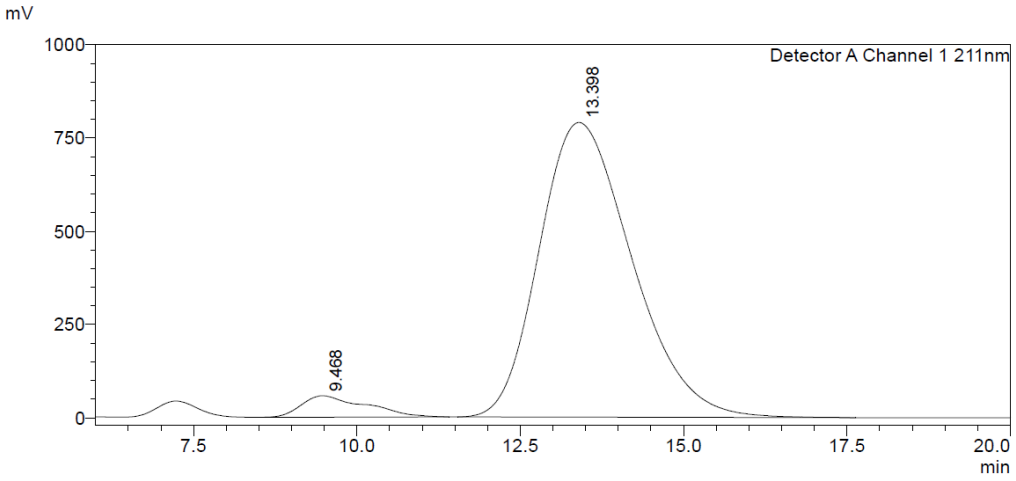
Chiral HPLC analysis Chiralcel OJ-H (85:15 Hexane:IPA, flow rate 1.5 mL min^{-1}, 211 nm, 30 °C) t_R(3*R*,5*R*): 9.5 min, t_R(3*S*,5*S*): 13.4 min, 4.85:95.15 er.



<Peak Table>

PDA Ch1 211nm

Peak#	Ret. Time	Area%
1	9.633	50.281
2	14.051	49.719
Total		100.000

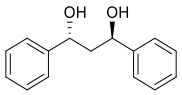


<Peak Table>

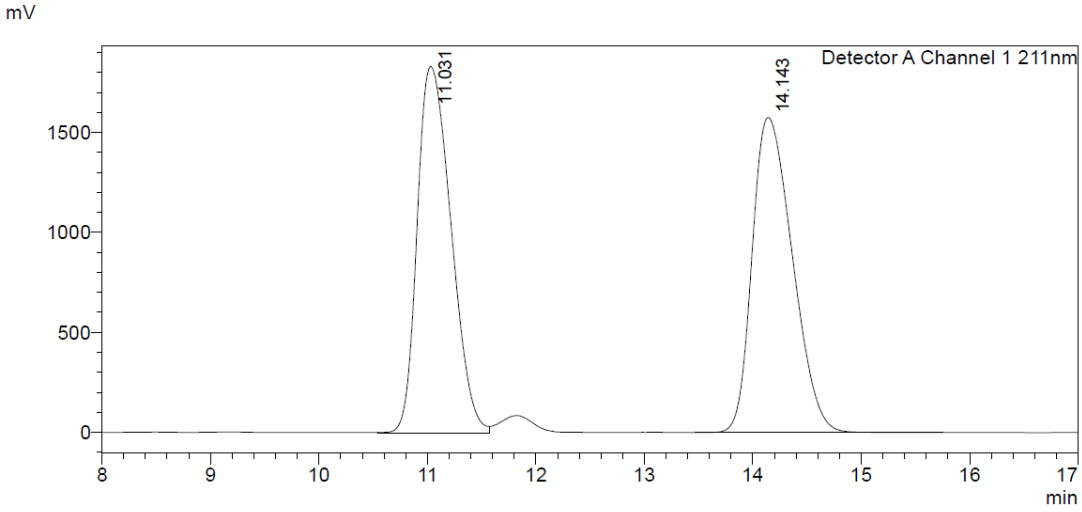
Detector A Channel 1 211nm

Peak#	Ret. Time	Area%
1	9.468	4.849
2	13.398	95.151
Total		100.000

(1*R*,3*R*)-1,3-diphenylpropane-1,3-diol (2b)



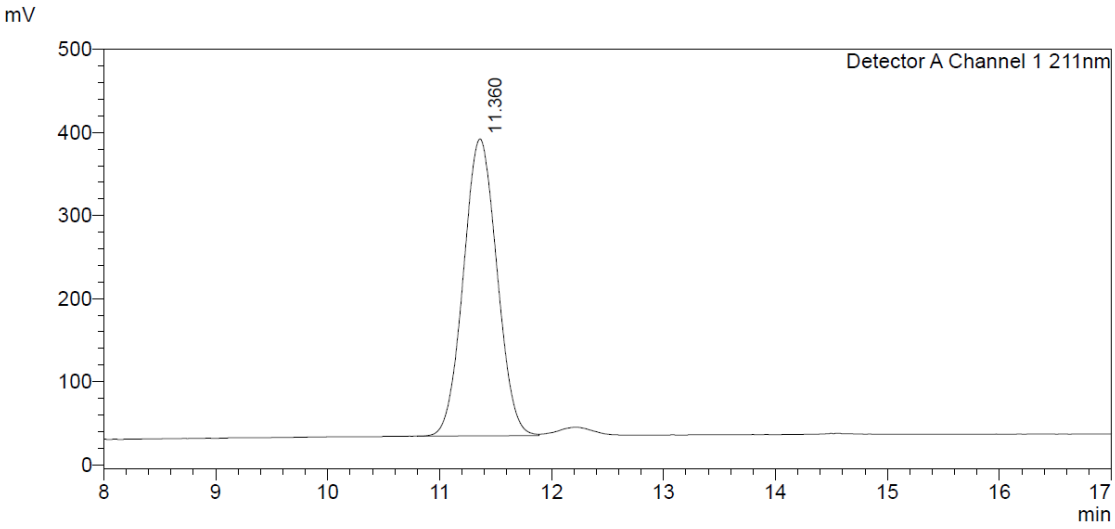
Chiral HPLC analysis Chiralpak ID (90:10 Hexane:IPA, flow rate 1.0 mL min^{-1}, 211 nm, 30 °C) t_R(3*R*,5*R*): 11.4 min, 100:0 er. The additional peak at $t_R \approx 12.2$ min could be identified as the meso diastereomer.



<Peak Table>

Detector A Channel 1 211nm

Peak#	Ret. Time	Area%
1	11.031	49.489
2	14.143	50.511
Total		100.000

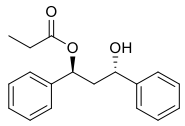


<Peak Table>

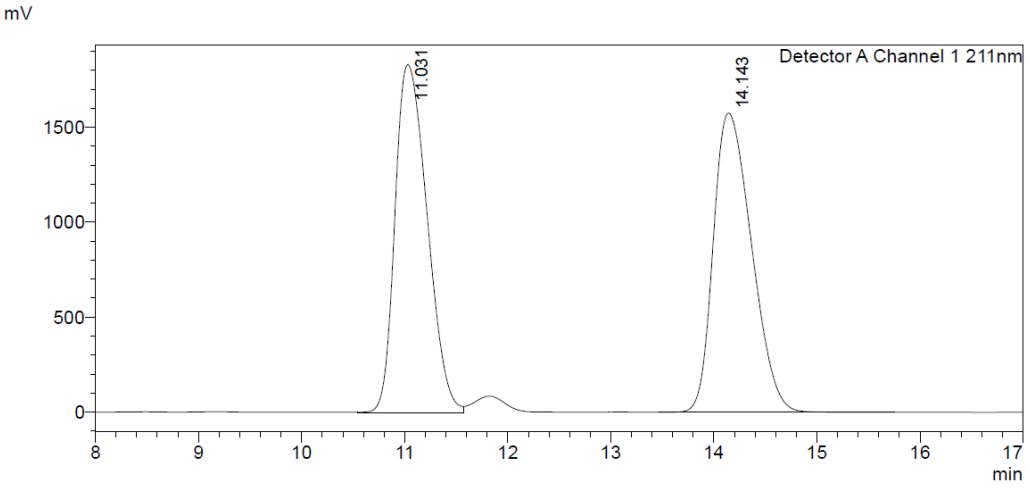
Detector A Channel 1 211nm

Peak#	Ret. Time	Area%
1	11.360	100.000
Total		100.000

(1*S*,3*S*)-3-hydroxy-1,3-diphenylpropyl propionate (3b)



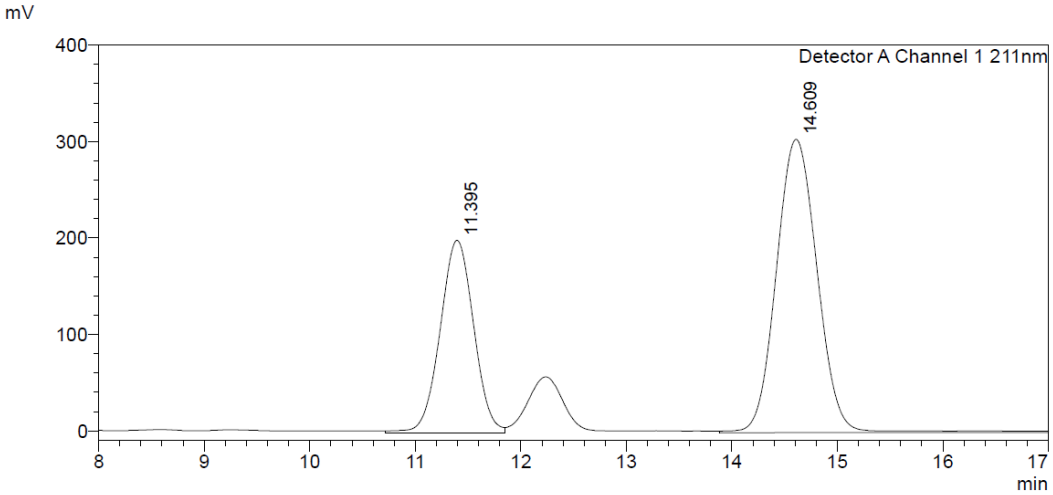
3-hydroxy-1,3-diphenylpropyl propionate was hydrolysed to give 1,3-diphenylpropane-1,3-diol; **Chiral HPLC analysis** Chiralpak ID (90:10 Hexane:IPA, flow rate 1.0 mL min^{-1}, 211 nm, 30 °C) t_R(3*R*,5*R*): 11.4 min t_R(3*S*,5*S*): 14.6 min, 33.73:66.27 er. The additional peak at $t_R \approx$ 12.2 min could be identified as the meso diastereomer.



<Peak Table>

Detector A Channel 1 211nm

Peak#	Ret. Time	Area%
1	11.031	49.489
2	14.143	50.511
Total		100.000

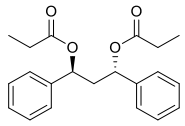


<Peak Table>

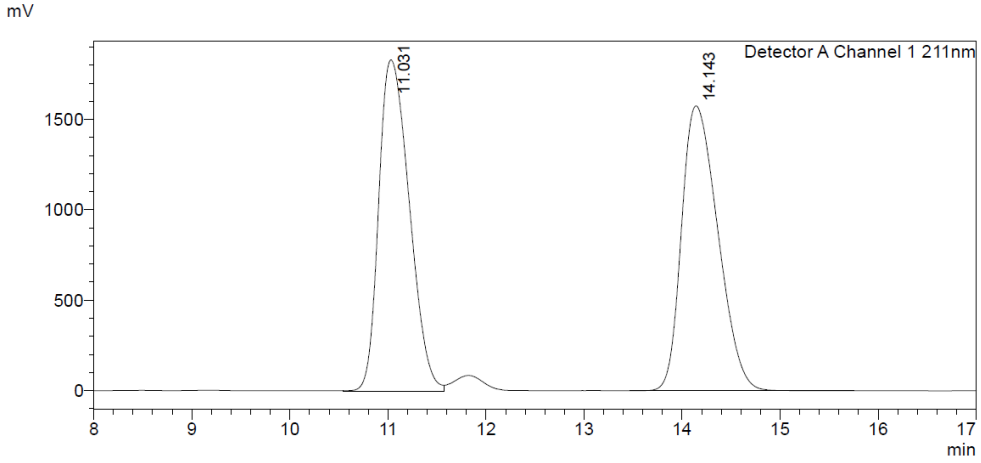
Detector A Channel 1 211nm

Peak#	Ret. Time	Area%
1	11.395	33.733
2	14.609	66.267
Total		100.000

(1*S*,3*S*)-1,3-diphenylpropane-1,3-diyl dipropionate (4b)



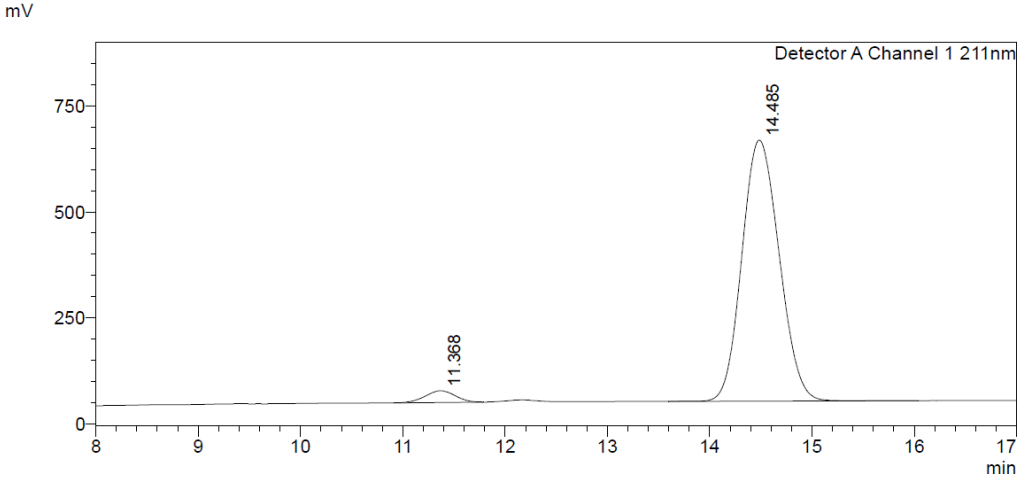
1,3-diphenylpropane-1,3-diyl dipropionate was hydrolysed to give 1,3-diphenylpropane-1,3-diol; **Chiral HPLC analysis** Chiralpak ID (90:10 Hexane:IPA, flow rate 1.0 mL min^{-1}, 211 nm, 30 °C) t_R(3*R*,5*R*): 11.4 min, t_R(3*S*,5*S*): 14.5 min, 3.45:96.55 er. The additional peak at $t_R \approx 12.2$ min could be identified as the meso diastereomer.



<Peak Table>

Detector A Channel 1 211nm

Peak#	Ret. Time	Area%
1	11.031	49.489
2	14.143	50.511
Total		100.000

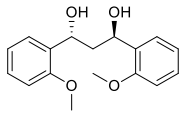


<Peak Table>

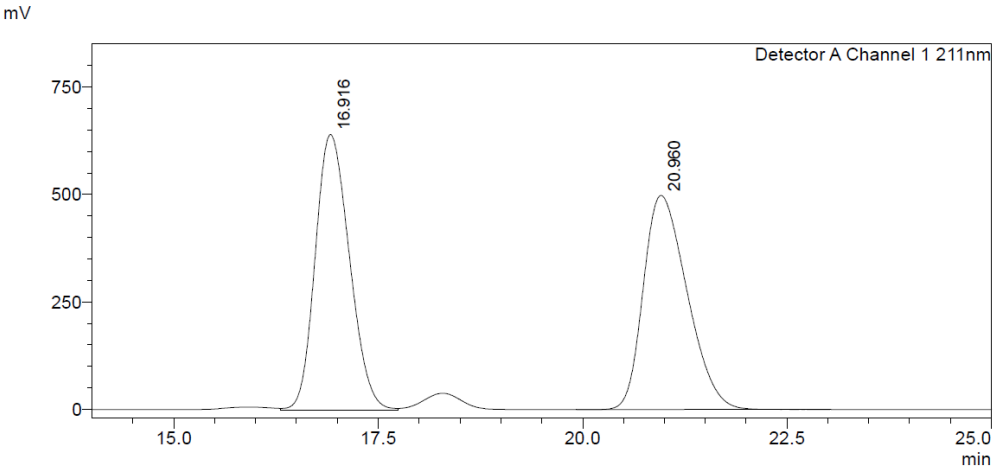
Detector A Channel 1 211nm

Peak#	Ret. Time	Area%
1	11.368	3.453
2	14.485	96.547
Total		100.000

(1*R*,3*R*)-1,3-bis(2-methoxyphenyl)propane-1,3-diol (2c)



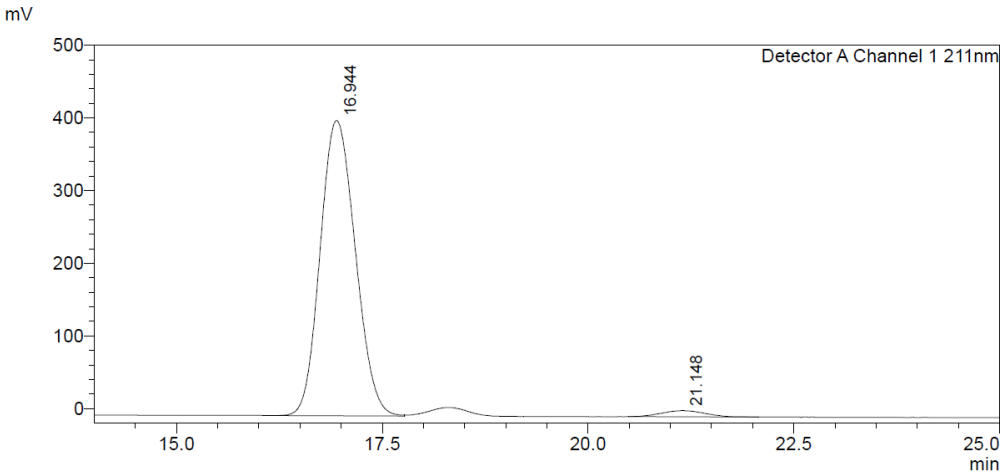
Chiral HPLC analysis Chiralpak ID (80:20 Hexane:IPA, flow rate 1.0 mL min^{-1}, 211 nm, 30 °C) t_R(3*R*,5*R*): 16.9 min, t_R(3*S*,5*S*): 21.1 min, 97.50:2.50 er. The additional peak at $t_R \approx 18.25$ min could be identified as the meso diastereomer.



<Peak Table>

Detector A Channel 1 211nm

Peak#	Ret. Time	Area%
1	16.916	50.110
2	20.960	49.890
Total		100.000

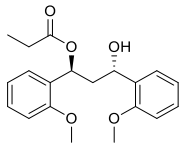


<Peak Table>

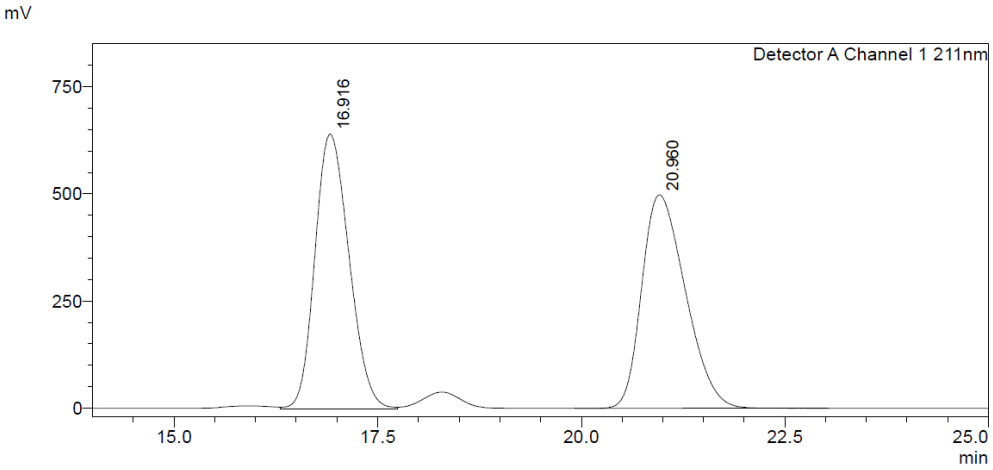
Detector A Channel 1 211nm

Peak#	Ret. Time	Area%
1	16.944	97.496
2	21.148	2.504
Total		100.000

(1*S*,3*S*)-3-hydroxy-1,3-bis(2-methoxyphenyl)propyl propionate (3c)



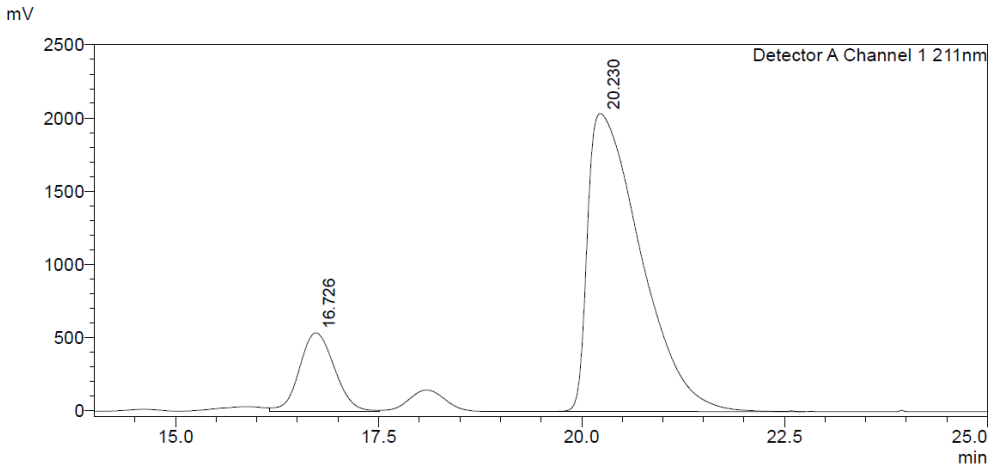
3-hydroxy-1,3-bis(2-methoxyphenyl)propyl propionate was hydrolysed to give 1,3-diphenylpropane-1,3-diol; **Chiral HPLC analysis** Chiralpak ID (80:20 Hexane:IPA, flow rate 1.0 mL min^{-1}, 211 nm, 30 °C) t_R(3*R*,5*R*): 16.7 min, t_R(3*S*,5*S*): 20.2 min, 14.77:85.23 er. The additional peak at $t_R \approx 18.25$ min could be identified as the meso diastereomer.



<Peak Table>

Detector A Channel 1 211nm

Peak#	Ret. Time	Area%
1	16.916	50.110
2	20.960	49.890
Total		100.000

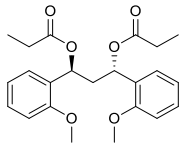


<Peak Table>

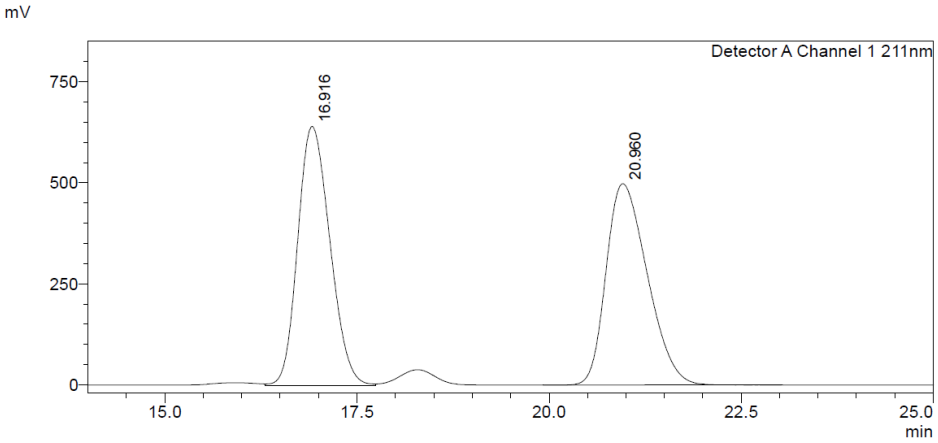
Detector A Channel 1 211nm

Peak#	Ret. Time	Area%
1	16.726	14.774
2	20.230	85.226
Total		100.000

(1*S*,3*S*)-1,3-bis(2-methoxyphenyl)propane-1,3-diyl dipropionate (4c)



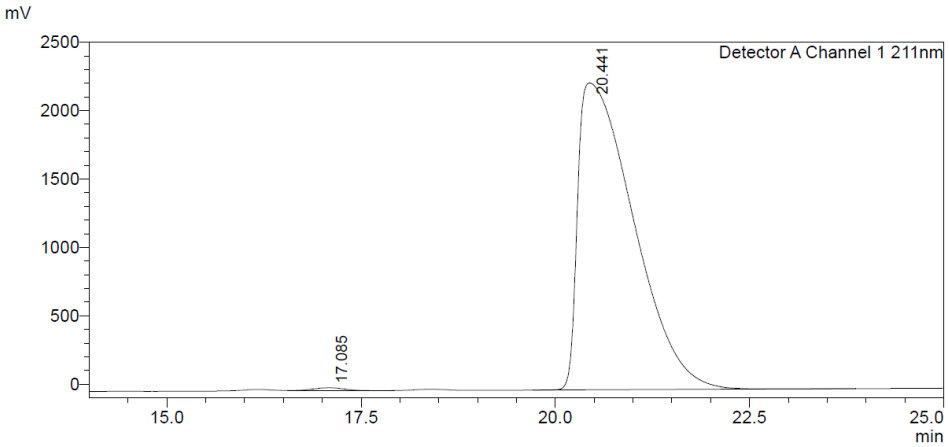
1,3-bis(2-methoxyphenyl)propane-1,3-diyl dipropionate was hydrolysed to give 1,3-bis(2-methoxyphenyl)propane-1,3-diol; **Chiral HPLC analysis** Chiralpak ID (80:20 Hexane:IPA, flow rate 1.0 mL min^{-1}, 211 nm, 30 °C) t_R(3*R*,5*R*): 17.1 min, t_R(3*S*,5*S*): 20.4 min, 0.46:99.54 er.



<Peak Table>

Detector A Channel 1 211nm

Peak#	Ret. Time	Area%
1	16.916	50.110
2	20.960	49.890
Total		100.000

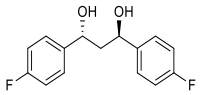


<Peak Table>

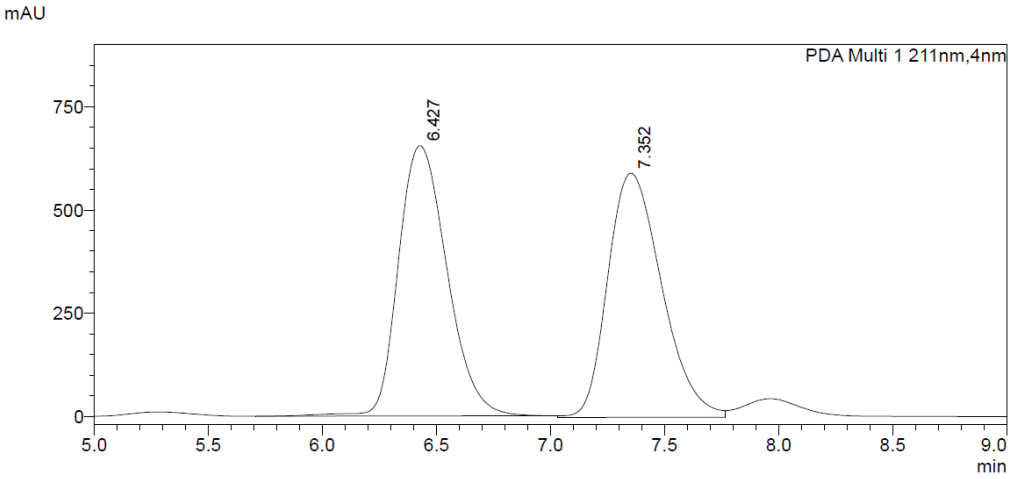
Detector A Channel 1 211nm

Peak#	Ret. Time	Area%
1	17.085	0.460
2	20.441	99.540
Total		100.000

(1*R*,3*R*)-1,3-bis(4-fluorophenyl)propane-1,3-diol (2d)



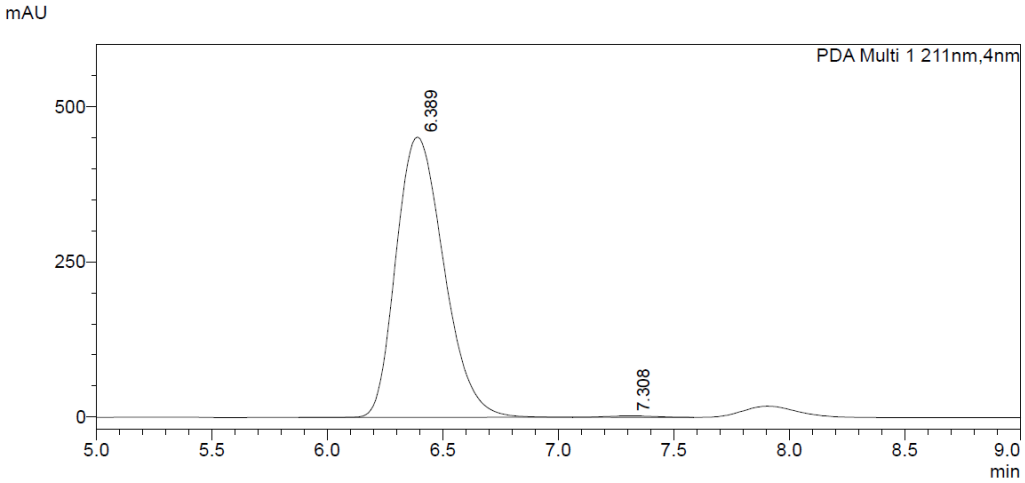
Chiral HPLC analysis Chiralcel OJ-H (80:20 Hexane:IPA, flow rate 1.0 mL min^{-1}, 211 nm, 30 °C) t_R(3*R*,5*R*): 6.4 min, t_R(3*S*,5*S*): 7.3 min, 99.49:0.51 er. The additional peak at $t_R \approx 7.9$ min could be identified as the meso diastereomer.



<Peak Table>

PDA Ch1 211nm

Peak#	Ret. Time	Area%
1	6.427	49.951
2	7.352	50.049
Total		100.000

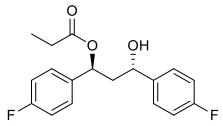


<Peak Table>

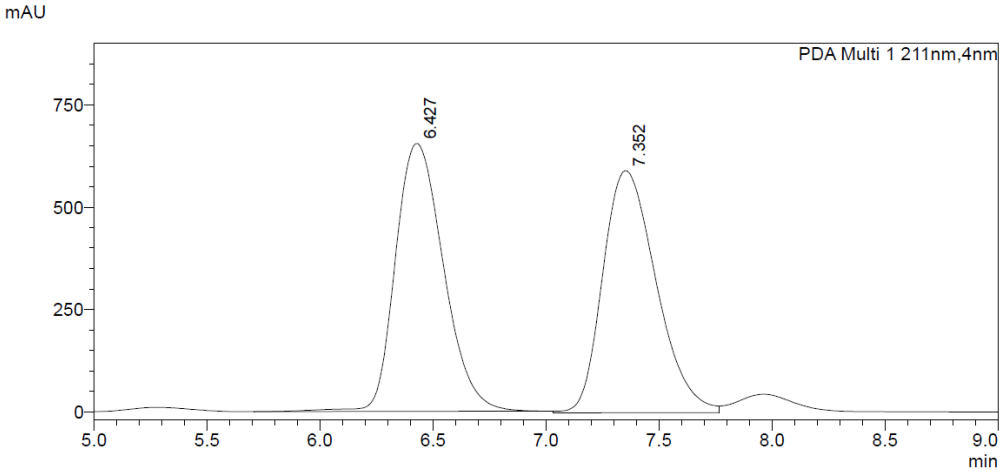
PDA Ch1 211nm

Peak#	Ret. Time	Area%
1	6.389	99.492
2	7.308	0.508
Total		100.000

(1*S*,3*S*)-1,3-bis(4-fluorophenyl)-3-hydroxypropyl propionate (3d)



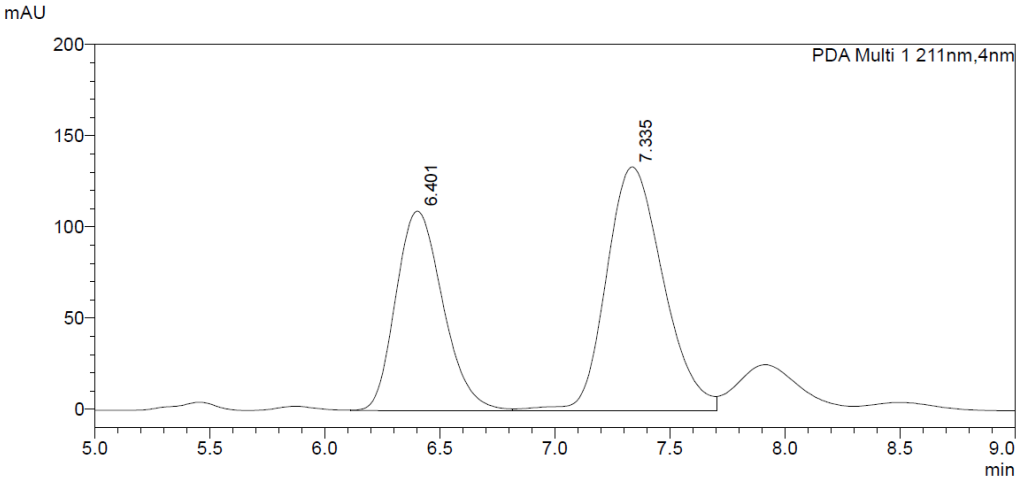
1,3-bis(4-fluorophenyl)-3-hydroxypropyl propionate was hydrolysed to give 1,3-bis(4-fluorophenyl)propane-1,3-diol; **Chiral HPLC analysis** Chiralcel OJ-H (80:20 Hexane:IPA, flow rate 1.0 mL min^{-1}, 211 nm, 30 °C) t_R(3*R*,5*R*): 6.4 min, t_R(3*S*,5*S*): 7.3 min, 40.40:59.60 er. The additional peak at $t_R \approx 7.9$ min could be identified as the meso diastereomer.



<Peak Table>

PDA Ch1 211nm

Peak#	Ret. Time	Area%
1	6.427	49.951
2	7.352	50.049
Total		100.000

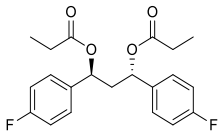


<Peak Table>

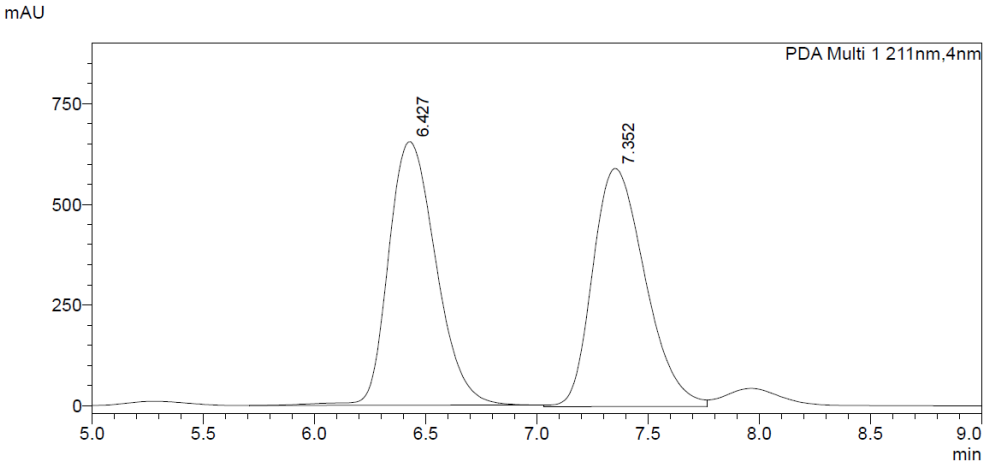
PDA Ch1 211nm

Peak#	Ret. Time	Area%
1	6.401	40.397
2	7.335	59.603
Total		100.000

(1*S*,3*S*)-1,3-bis(4-fluorophenyl)propane-1,3-diyl dipropionate (4d)



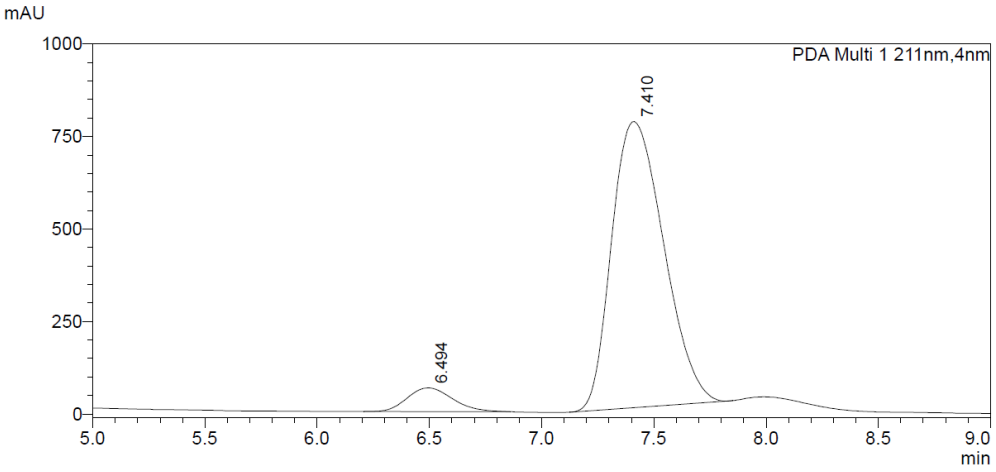
1,3-bis(4-fluorophenyl)propane-1,3-diyl dipropionate was hydrolysed to give 1,3-bis(4-fluorophenyl)propane-1,3-diol; **Chiral HPLC analysis** Chiralcel OJ-H (80:20 Hexane:IPA, flow rate 1.0 mL min^{-1}, 211 nm, 30 °C) t_R(3*R*,5*R*): 6.5 min, t_R(3*S*,5*S*): 7.4 min, 6.72:93.28 er. The additional peak at $t_R \approx 7.9$ min could be identified as the meso diastereomer.



<Peak Table>

PDA Ch1 211nm

Peak#	Ret. Time	Area%
1	6.427	49.951
2	7.352	50.049
Total		100.000

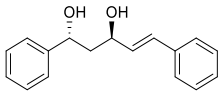


<Peak Table>

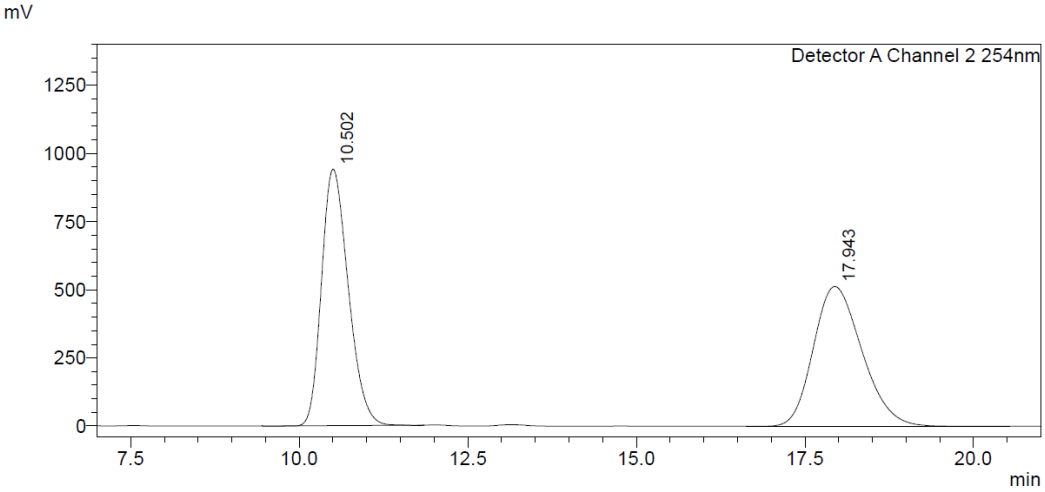
PDA Ch1 211nm

Peak#	Ret. Time	Area%
1	6.494	6.719
2	7.410	93.281
Total		100.000

(1*R*,3*R*,*E*)-1,5-diphenylpent-4-ene-1,3-diol (2e)



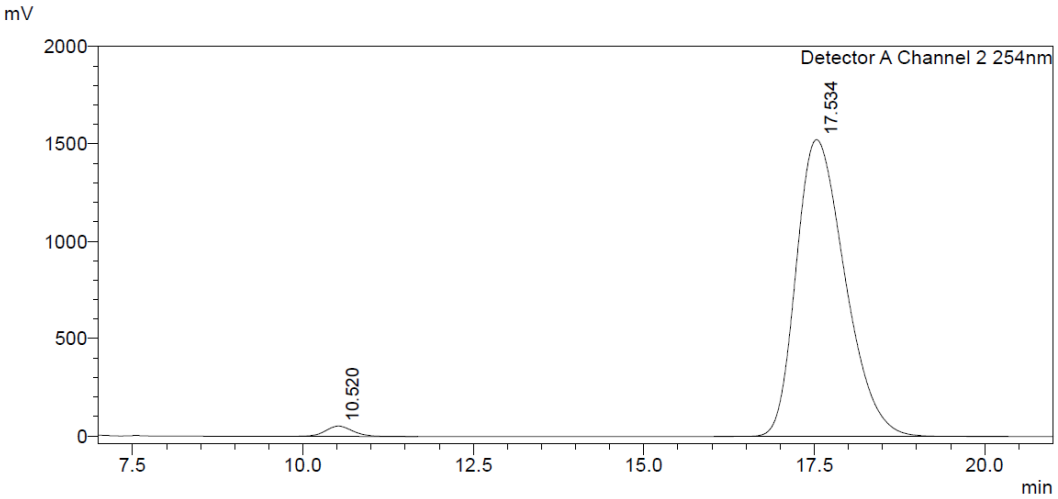
Chiral HPLC analysis Chiralcel OD-H (80:20 Hexane:IPA, flow rate 1.0 mL min^{-1}, 254 nm, 30 °C) t_R(3*S*,5*S*): 10.5 min, t_R(3*R*,5*R*): 17.5 min, 1.91:98.09 er.



<Peak Table>

Detector A Channel 2 254nm

Peak#	Ret. Time	Area%
1	10.502	50.290
2	17.943	49.710
Total		100.000

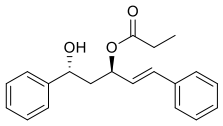


<Peak Table>

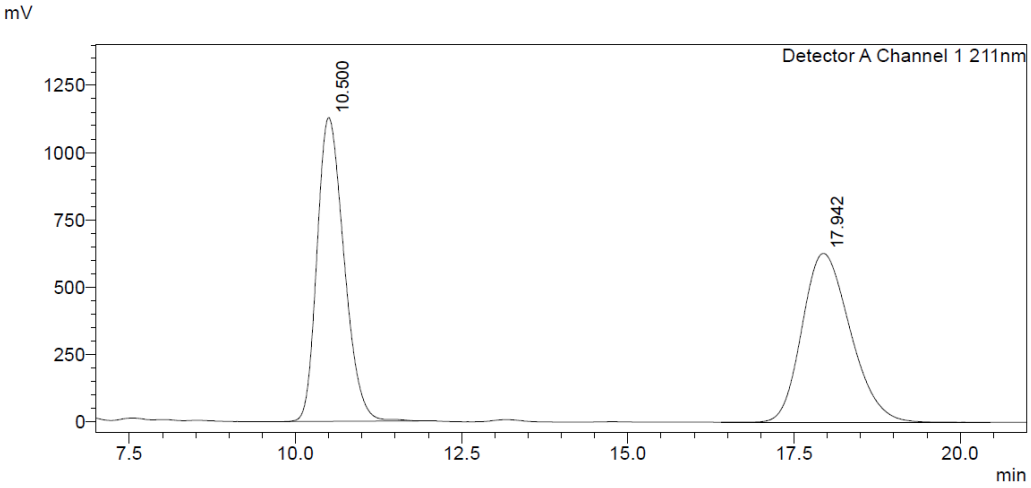
Detector A Channel 2 254nm

Peak#	Ret. Time	Area%
1	10.520	1.905
2	17.534	98.095
Total		100.000

(3*R*,5*R*,*E*)-5-hydroxy-1,5-diphenylpent-1-en-3-yl propionate (3e′)



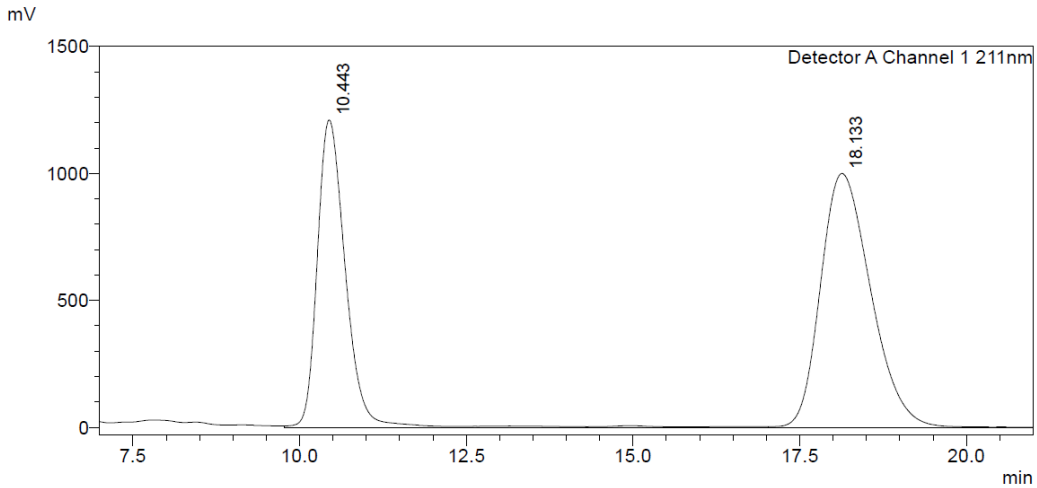
5-hydroxy-1,5-diphenylpent-1-en-3-yl propionate was hydrolysed to give 1,5-diphenylpent-4-ene-1,3-diol; **Chiral HPLC analysis** Chiralcel OD-H (80:20 Hexane:IPA, flow rate 1.0 mL min^{-1}, 211 nm, 30 °C) t_R(3*S*,5*S*): 10.4 min, t_R(3*R*,5*R*): 18.1 min, 40.75:59.25 er.



<Peak Table>

Detector A Channel 1 211nm

Peak#	Ret. Time	Area%
1	10.500	50.099
2	17.942	49.901
Total		100.000

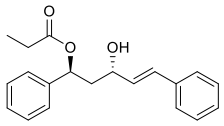


<Peak Table>

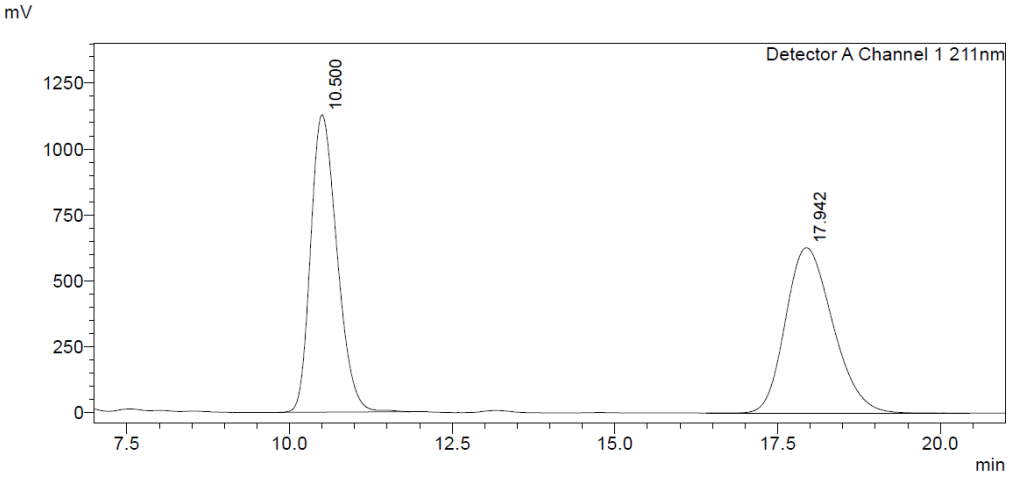
Detector A Channel 1 211nm

Peak#	Ret. Time	Area%
1	10.443	40.746
2	18.133	59.254
Total		100.000

(1*S*,3*S*,*E*)-3-hydroxy-1,5-diphenylpent-4-en-1-yl propionate (3e′′)



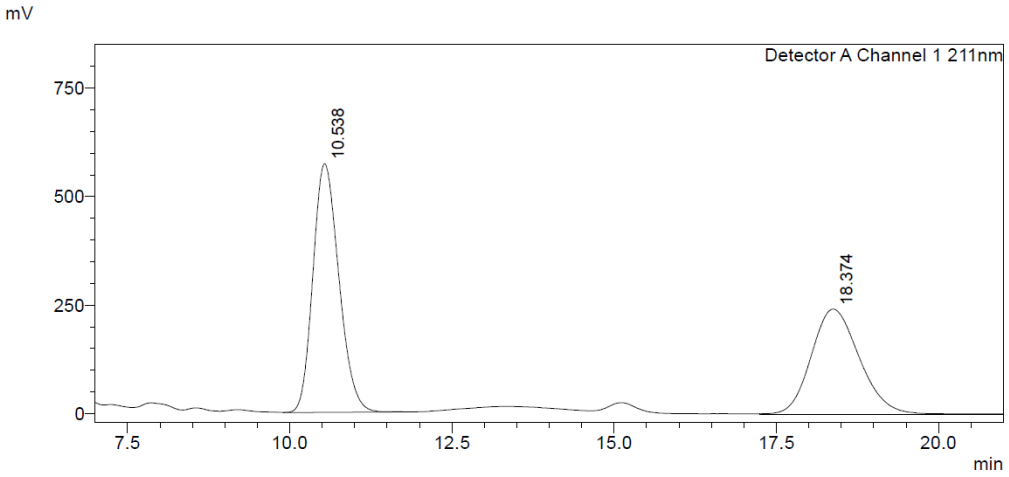
3-hydroxy-1,5-diphenylpent-4-en-1-yl propionate was hydrolysed to give 1,5-diphenylpent-4-ene-1,3-diol; **Chiral HPLC analysis** Chiralcel OD-H (80:20 Hexane:IPA, flow rate 1.0 mL min^{-1}, 211 nm, 30 °C) t_R(3*S*,5*S*): 10.5 min, t_R(3*R*,5*R*): 18.4 min, 56.02:43.98 er.



<Peak Table>

Detector A Channel 1 211nm

Peak#	Ret. Time	Area%
1	10.500	50.099
2	17.942	49.901
Total		100.000

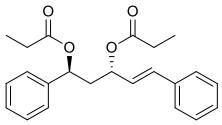


<Peak Table>

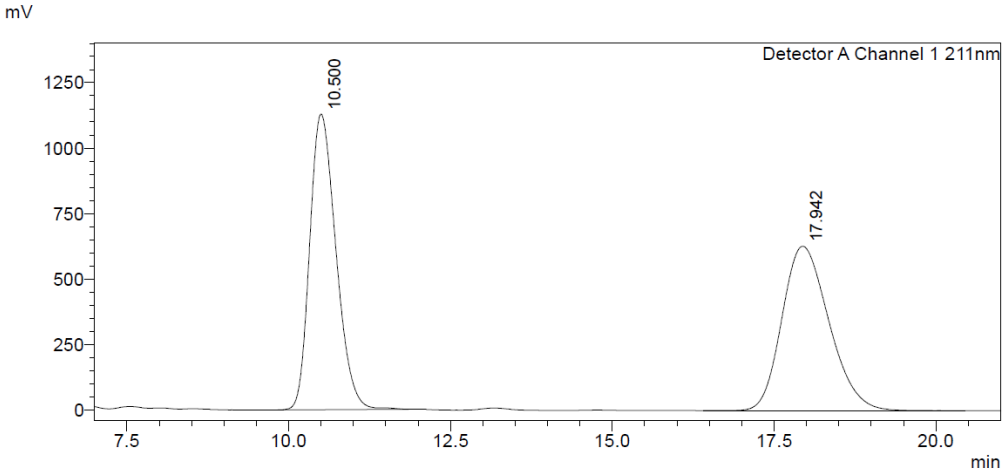
Detector A Channel 1 211nm

Peak#	Ret. Time	Area%
1	10.538	56.020
2	18.374	43.980
Total		100.000

(1*S*,3*S*,*E*)-1,5-diphenylpent-4-ene-1,3-diyl dipropionate (4e)



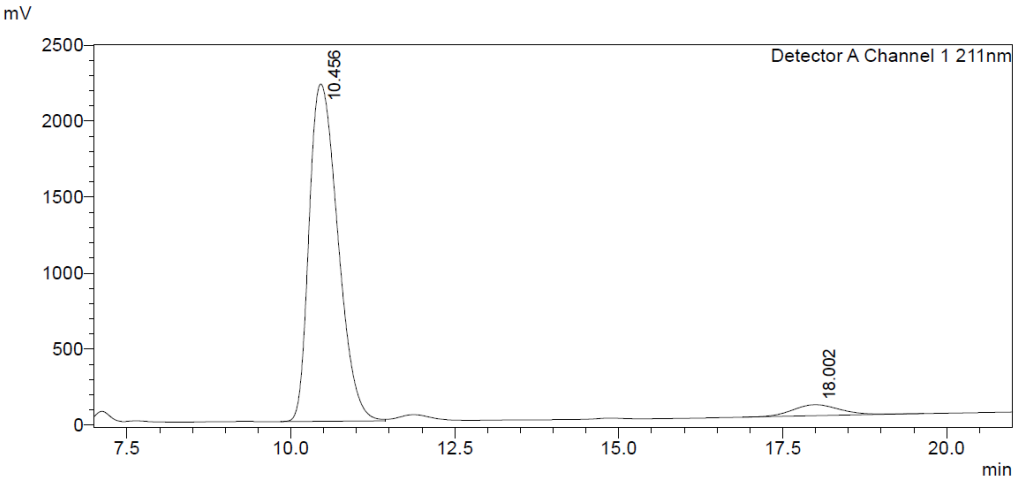
1,5-diphenylpent-4-ene-1,3-diyl dipropionate was hydrolysed to give 1,5-diphenylpent-4-ene-1,3-diol; **Chiral HPLC analysis** Chiralcel OD-H (80:20 Hexane:IPA, flow rate 1.0 mL min^{-1}, 211 nm, 30 °C) t_R(3*S*,5*S*): 10.5 min, t_R(3*R*,5*R*): 18.0 min, 94.91:5.09 er.



<Peak Table>

Detector A Channel 1 211nm

Peak#	Ret. Time	Area%
1	10.500	50.099
2	17.942	49.901
Total		100.000

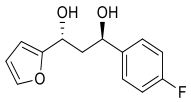


<Peak Table>

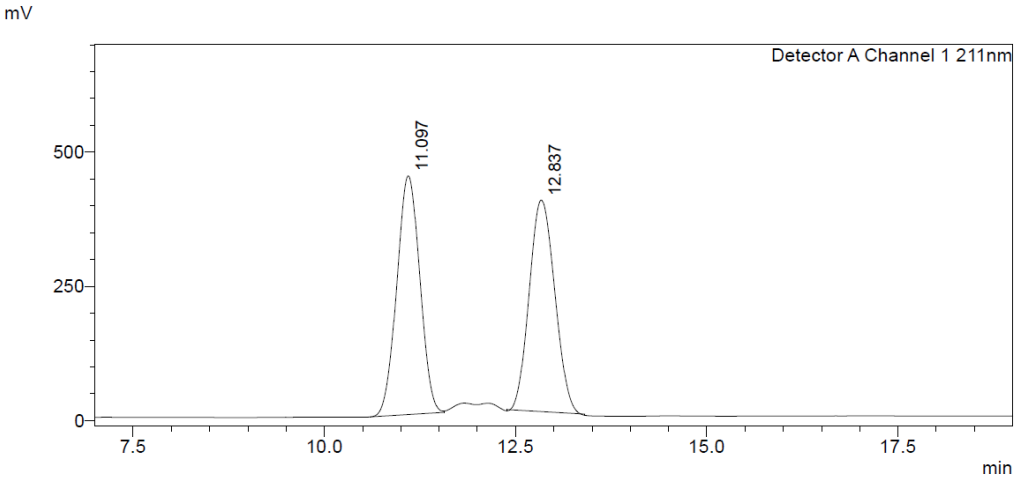
Detector A Channel 1 211nm

Peak#	Ret. Time	Area%
1	10.456	94.911
2	18.002	5.089
Total		100.000

(1*R*,3*R*)-1-(4-fluorophenyl)-3-(furan-2-yl)propane-1,3-diol (2f)



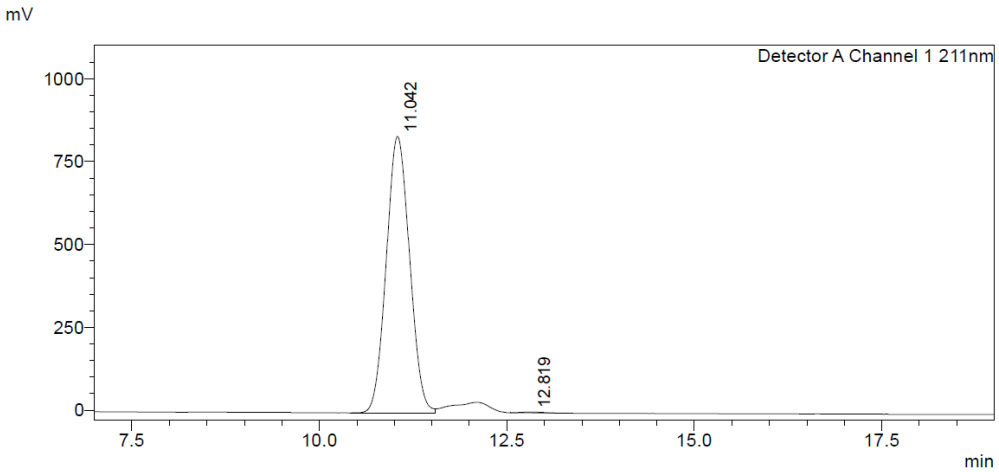
Chiral HPLC analysis Chiralcel ID (90:10 Hexane:IPA, flow rate 1.0 mL min^{-1}, 211 nm, 30 °C) t_R(3*R*,5*R*): 11.0 min, t_R(3*S*,5*S*): 12.8 min, 99.73:0.27 er. The additional peaks at $t_R \approx 12.0$ min could be identified as the diastereomer.



<Peak Table>

Detector A Channel 1 211nm

Peak#	Ret. Time	Area%
1	11.097	50.511
2	12.837	49.489
Total		100.000

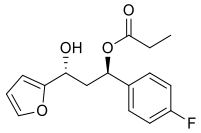


<Peak Table>

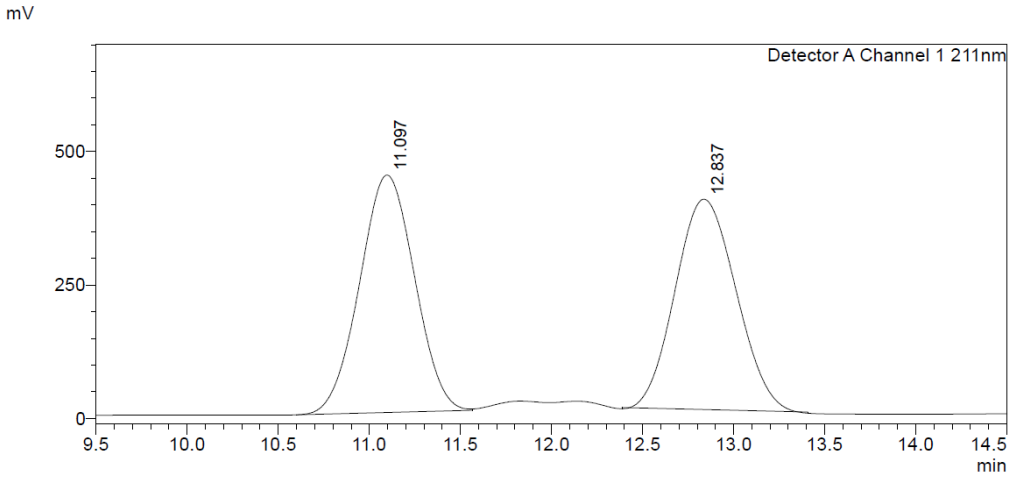
Detector A Channel 1 211nm

Peak#	Ret. Time	Area%
1	11.042	99.734
2	12.819	0.266
Total		100.000

(1*R*,3*R*)-3-(4-fluorophenyl)-1-(furan-2-yl)-3-hydroxypropyl propionate (3f′)



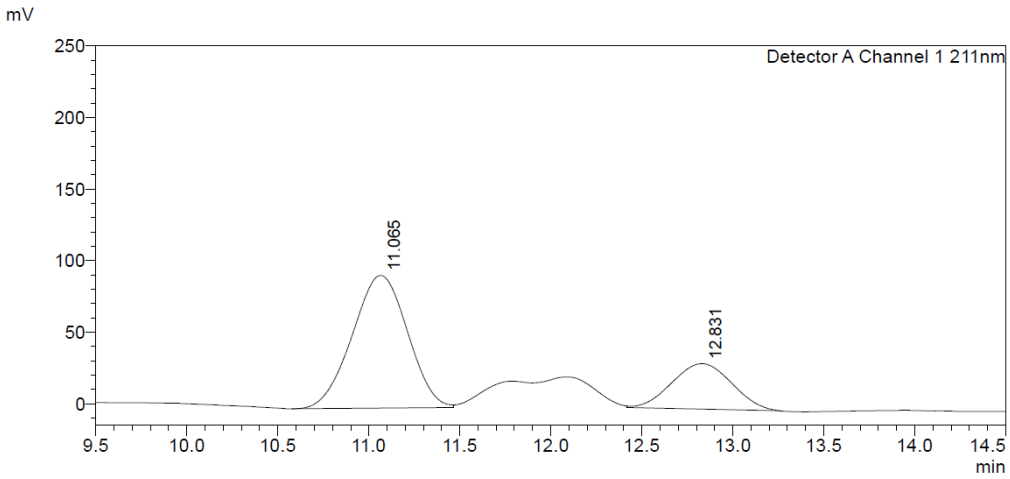
3-(4-fluorophenyl)-1-(furan-2-yl)-3-hydroxypropyl propionate was hydrolysed to give 1-(4-fluorophenyl)-3-(furan-2-yl)propane-1,3-diol; **Chiral HPLC analysis** Chiralcel ID (90:10 Hexane:IPA, flow rate 1.0 mL min^{-1}, 211 nm, 30 °C) t_R(3*R*,5*R*): 11.1 min, t_R(3*S*,5*S*): 12.8 min, 72.97:27.03 er. The additional peaks at $t_R \approx 12.0$ min could be identified as the diastereomer.



<Peak Table>

Detector A Channel 1 211nm

Peak#	Ret. Time	Area%
1	11.097	50.511
2	12.837	49.489
Total		100.000

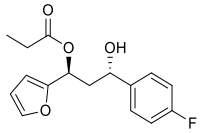


<Peak Table>

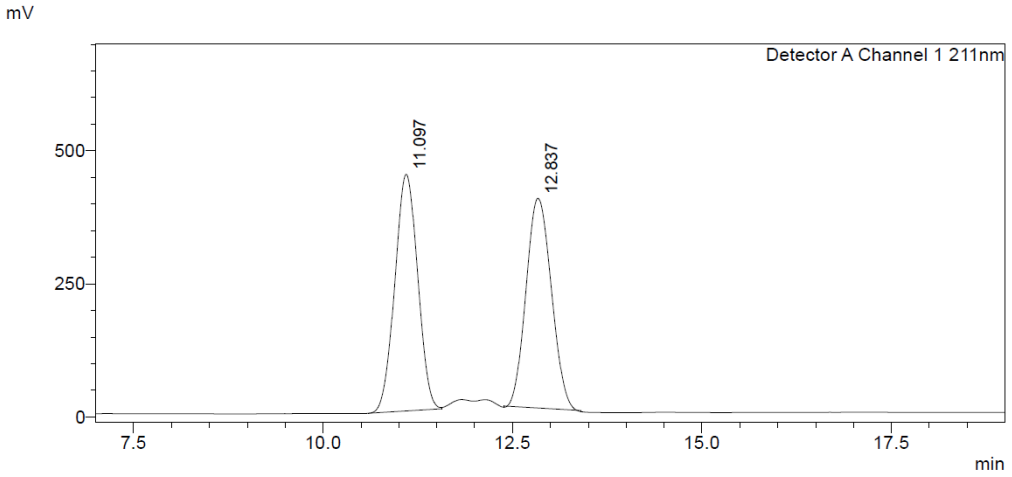
Detector A Channel 1 211nm

Peak#	Ret. Time	Area%
1	11.065	72.974
2	12.831	27.026
Total		100.000

(1*S*,3*S*)-1-(4-fluorophenyl)-3-(furan-2-yl)-3-hydroxypropyl propionate (3f′′)



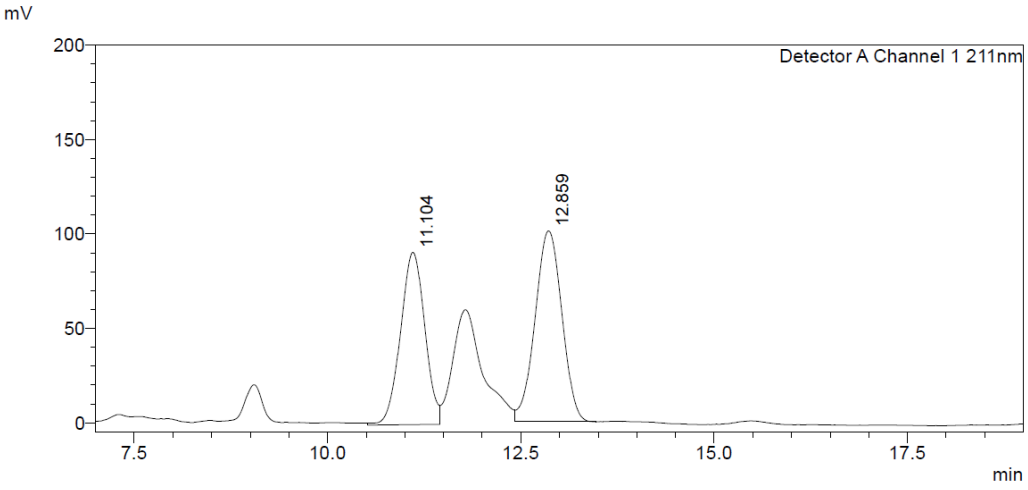
1-(4-fluorophenyl)-3-(furan-2-yl)-3-hydroxypropyl propionate was hydrolysed to give 1-(4-fluorophenyl)-3-(furan-2-yl)propane-1,3-diol; **Chiral HPLC analysis** Chiralcel ID (90:10 Hexane:IPA, flow rate 1.0 mL min^{-1}, 211 nm, 30 °C) t_R(3*R*,5*R*): 11.1 min, t_R(3*S*,5*S*): 12.8 min, 45.34:54.66 er. The additional peaks at $t_R \approx 12.0$ min could be identified as the diastereomer.



<Peak Table>

Detector A Channel 1 211nm

Peak#	Ret. Time	Area%
1	11.097	50.511
2	12.837	49.489
Total		100.000

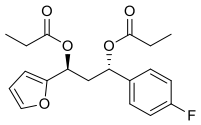


<Peak Table>

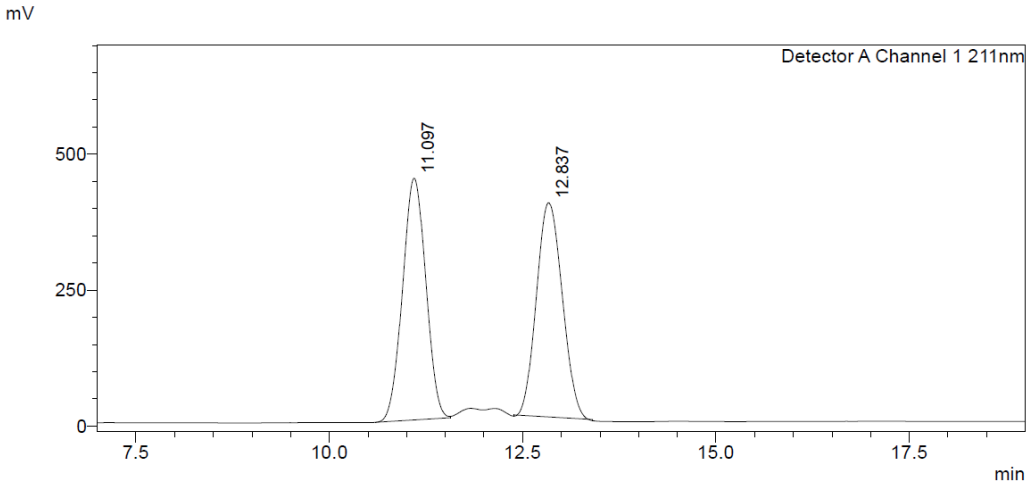
Detector A Channel 1 211nm

Peak#	Ret. Time	Area%
1	11.104	45.344
2	12.859	54.656
Total		100.000

(1*S*,3*S*)-1-(4-fluorophenyl)-3-(furan-2-yl)propane-1,3-diyl dipropionate (4f)



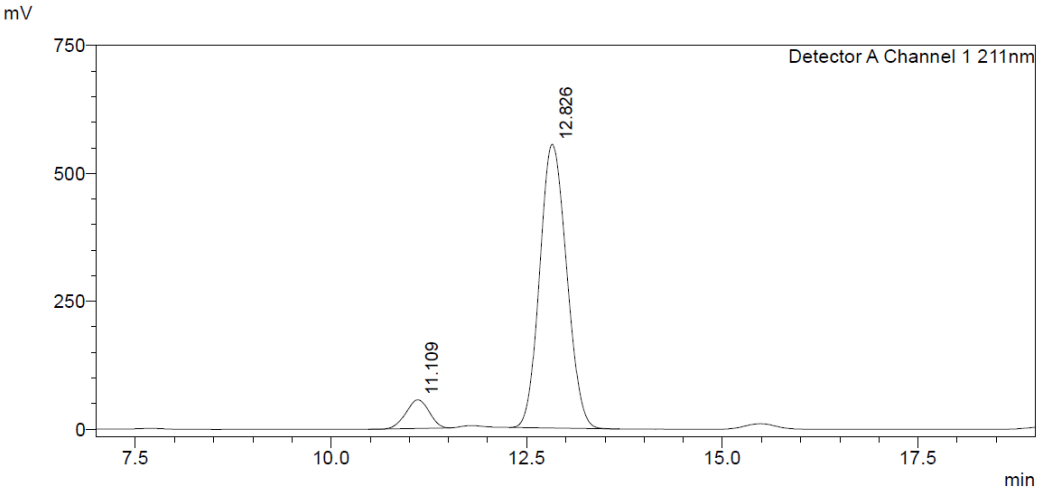
1-(4-fluorophenyl)-3-(furan-2-yl)propane-1,3-diyl dipropionate was hydrolysed to give 1-(4-fluorophenyl)-3-(furan-2-yl)propane-1,3-diol; **Chiral HPLC analysis** Chiralcel ID (90:10 Hexane:IPA, flow rate 1.0 mL min^{-1}, 211 nm, 30 °C) t_R(3*R*,5*R*): 11.1 min, t_R(3*S*,5*S*): 12.8 min, 7.98:92.02 er.



<Peak Table>

Detector A Channel 1 211nm

Peak#	Ret. Time	Area%
1	11.097	50.511
2	12.837	49.489
Total		100.000



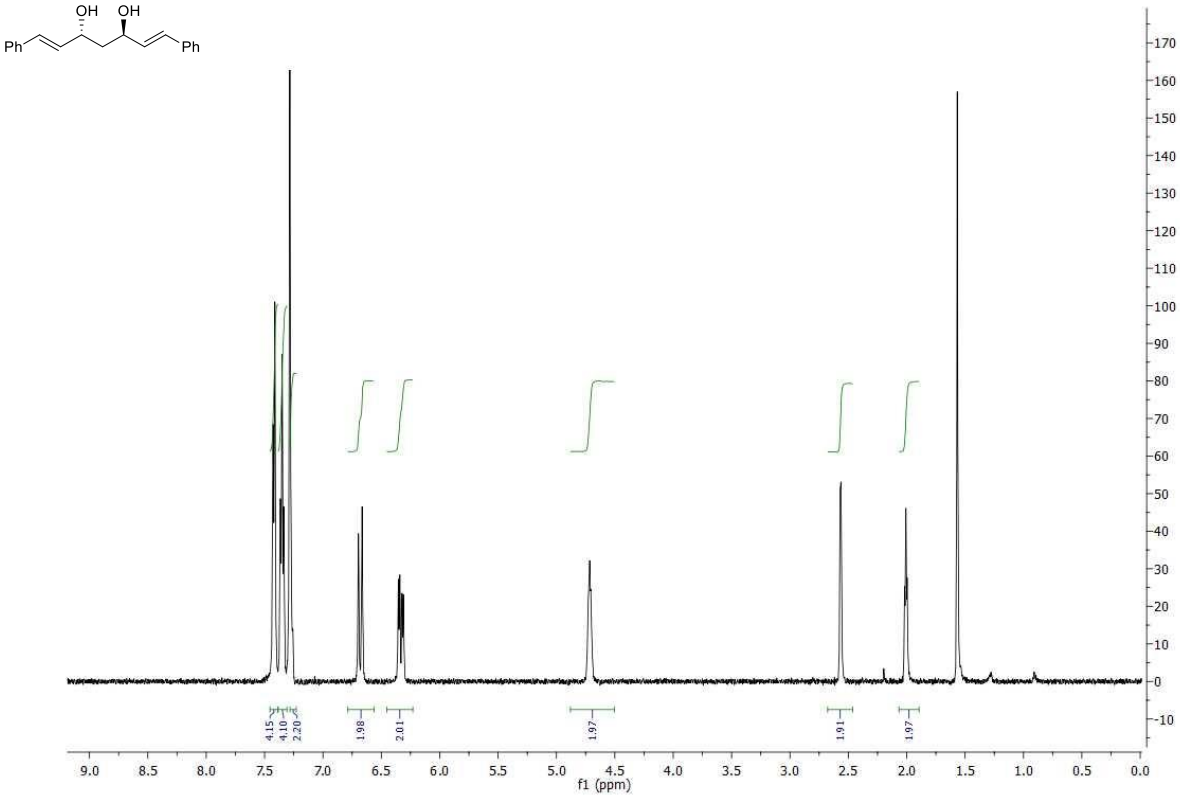
<Peak Table>

Detector A Channel 1 211nm

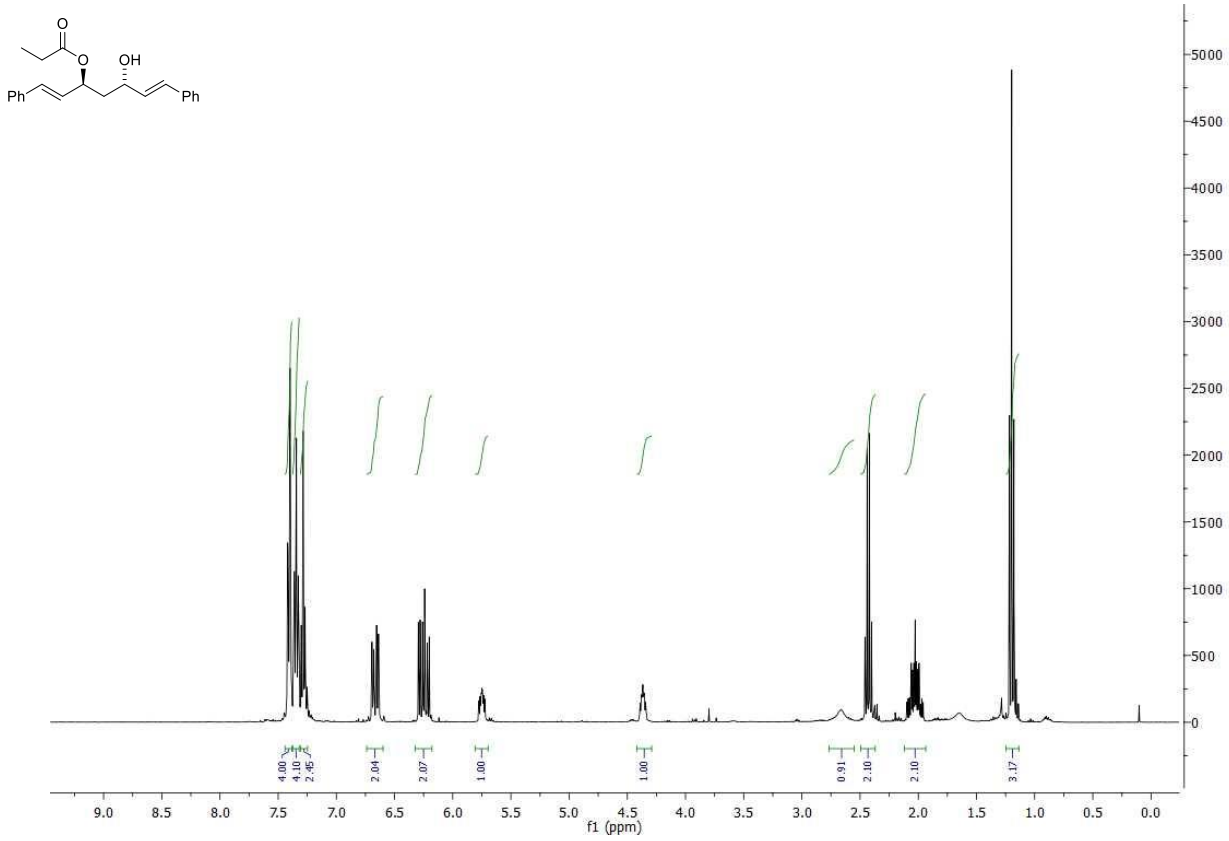
Peak#	Ret. Time	Area%
1	11.109	7.976
2	12.826	92.024
Total		100.000

NMR Spectra

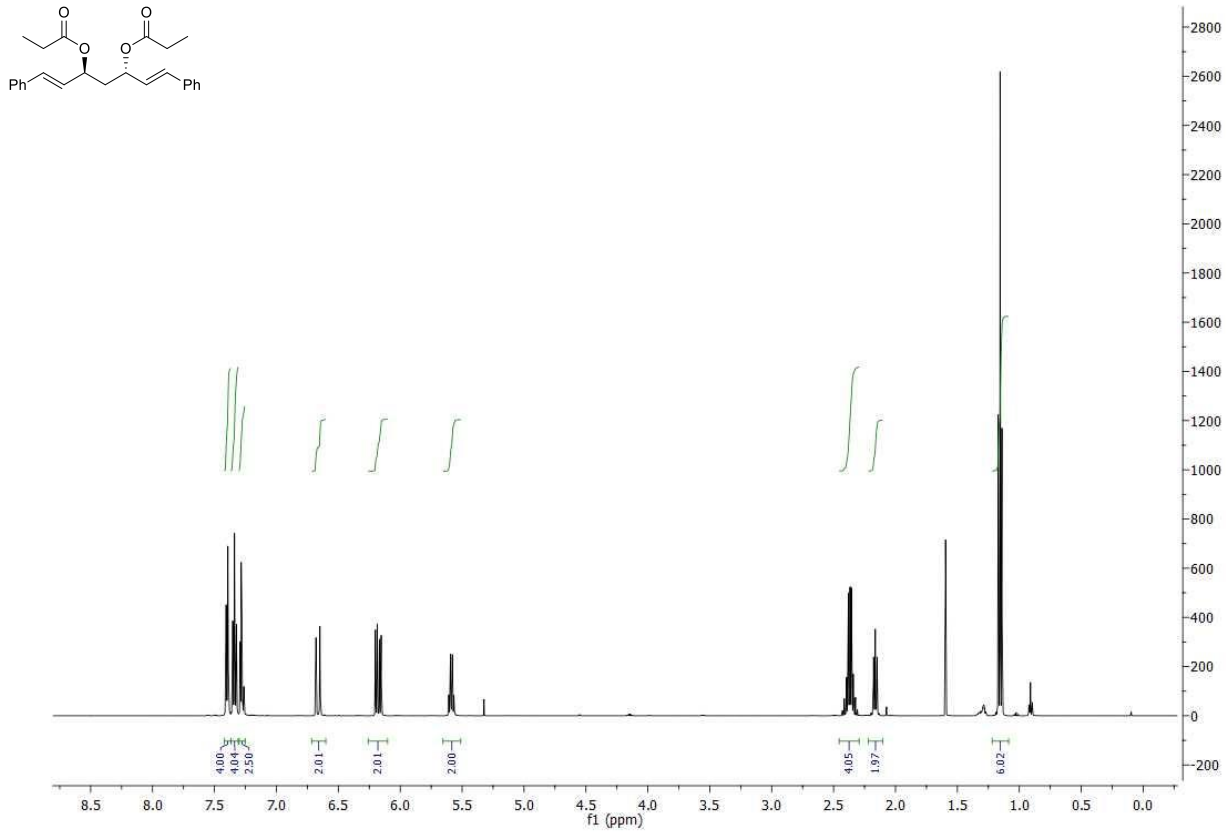
(1*E*,3*R*,5*R*,6*E*)-1,7-diphenylhepta-1,6-diene-3,5-diol (2a)



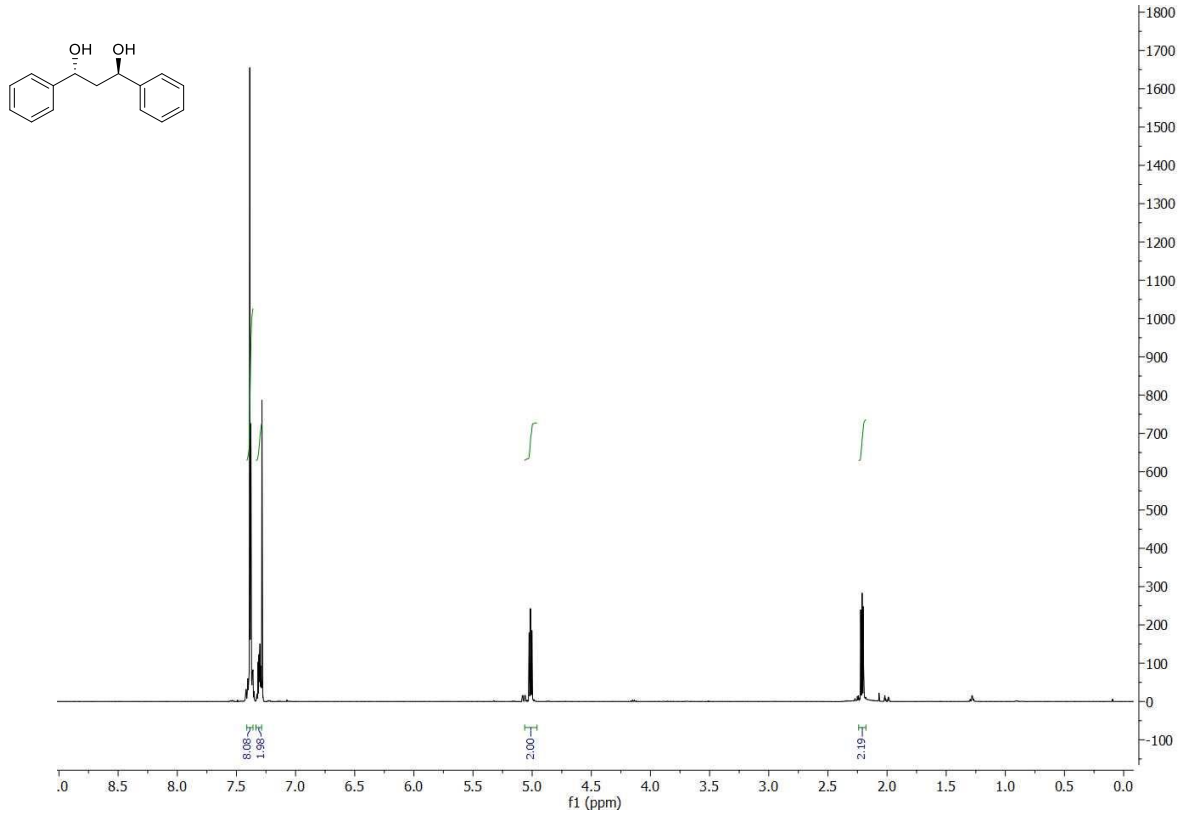
(1*E*,3S,5S,6*E*)-5-hydroxy-1,7-diphenylhepta-1,6-dien-3-yl propionate (3a)



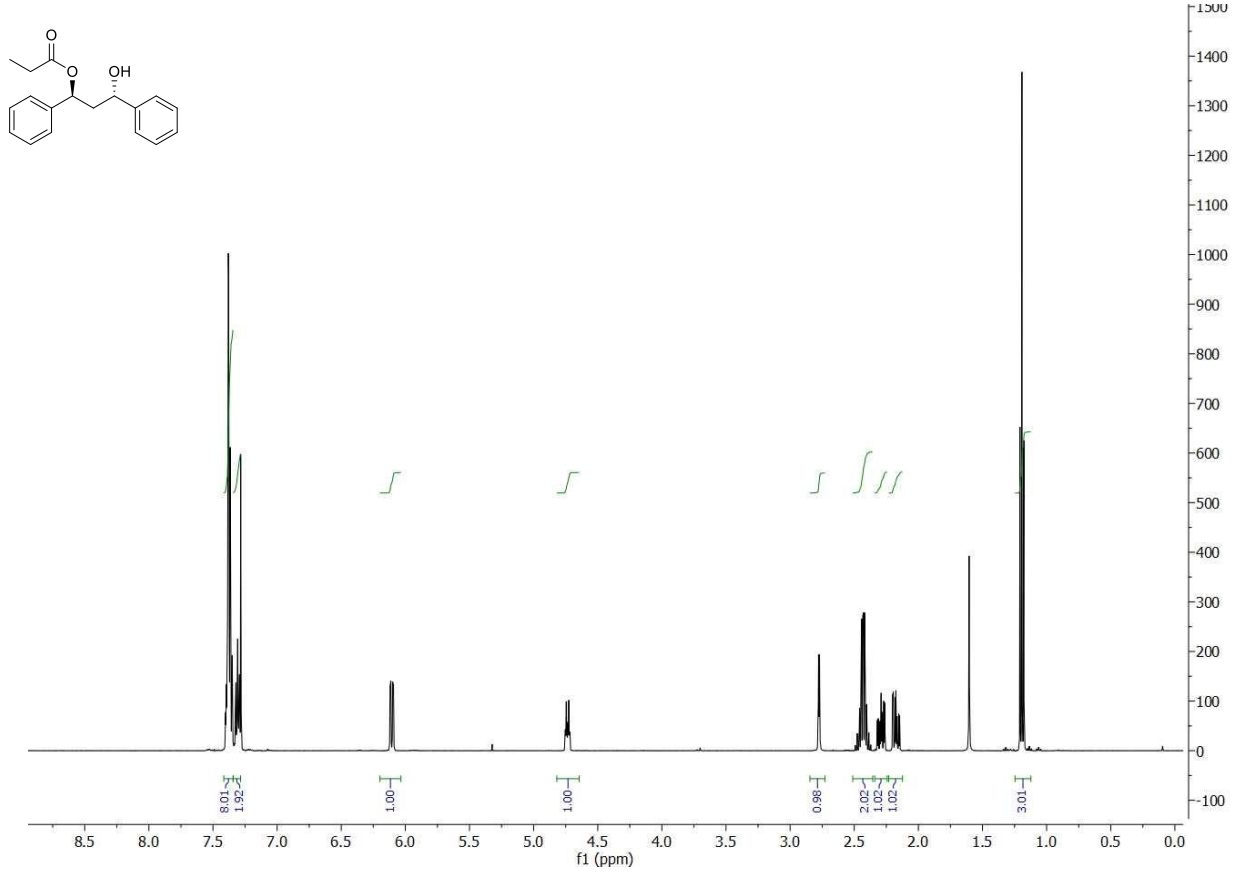
(1*E*,3*S*,5*S*,6*E*)-1,7-diphenylhepta-1,6-diene-3,5-diyl dipropionate (4a)

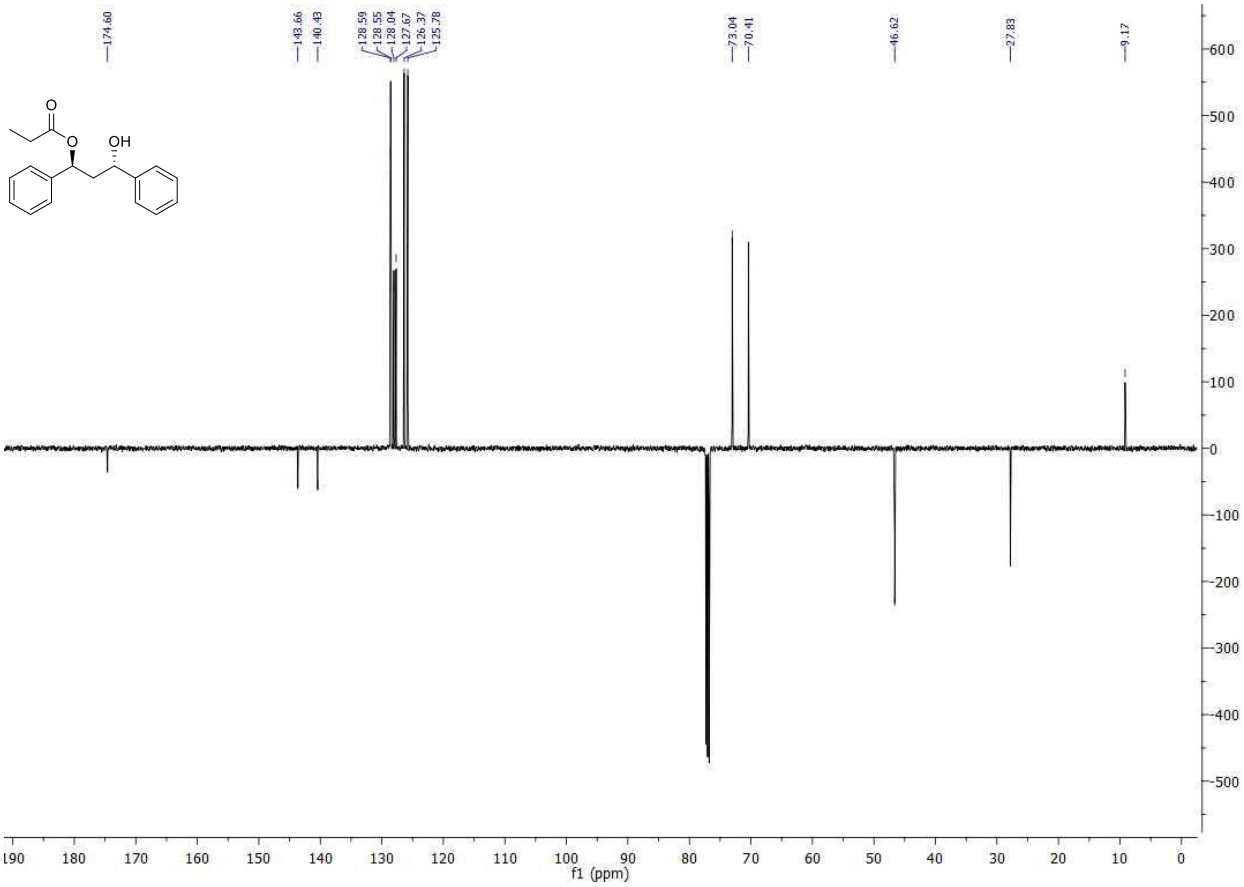


(1*R*,3*R*)-1,3-diphenylpropane-1,3-diol (2b)

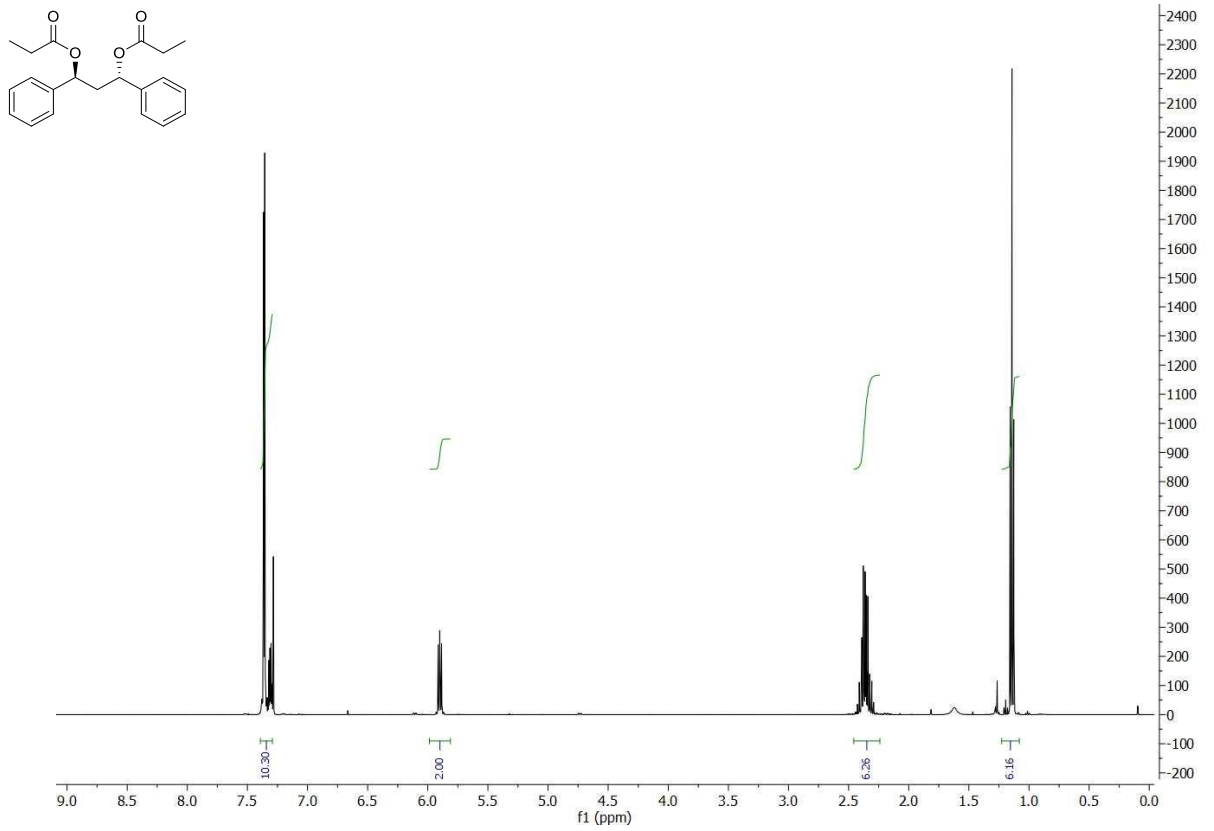


(1*S*,3*S*)-3-hydroxy-1,3-diphenylpropyl propionate (3b)

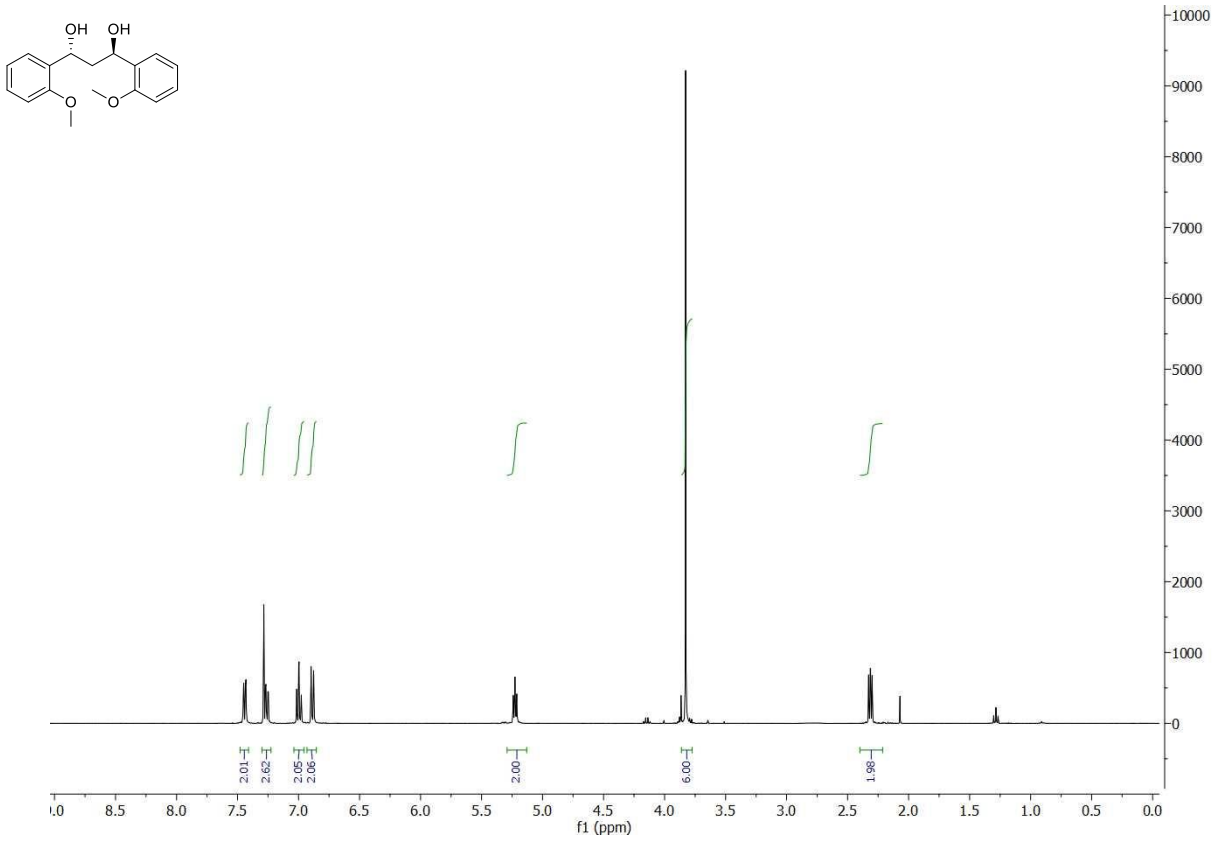




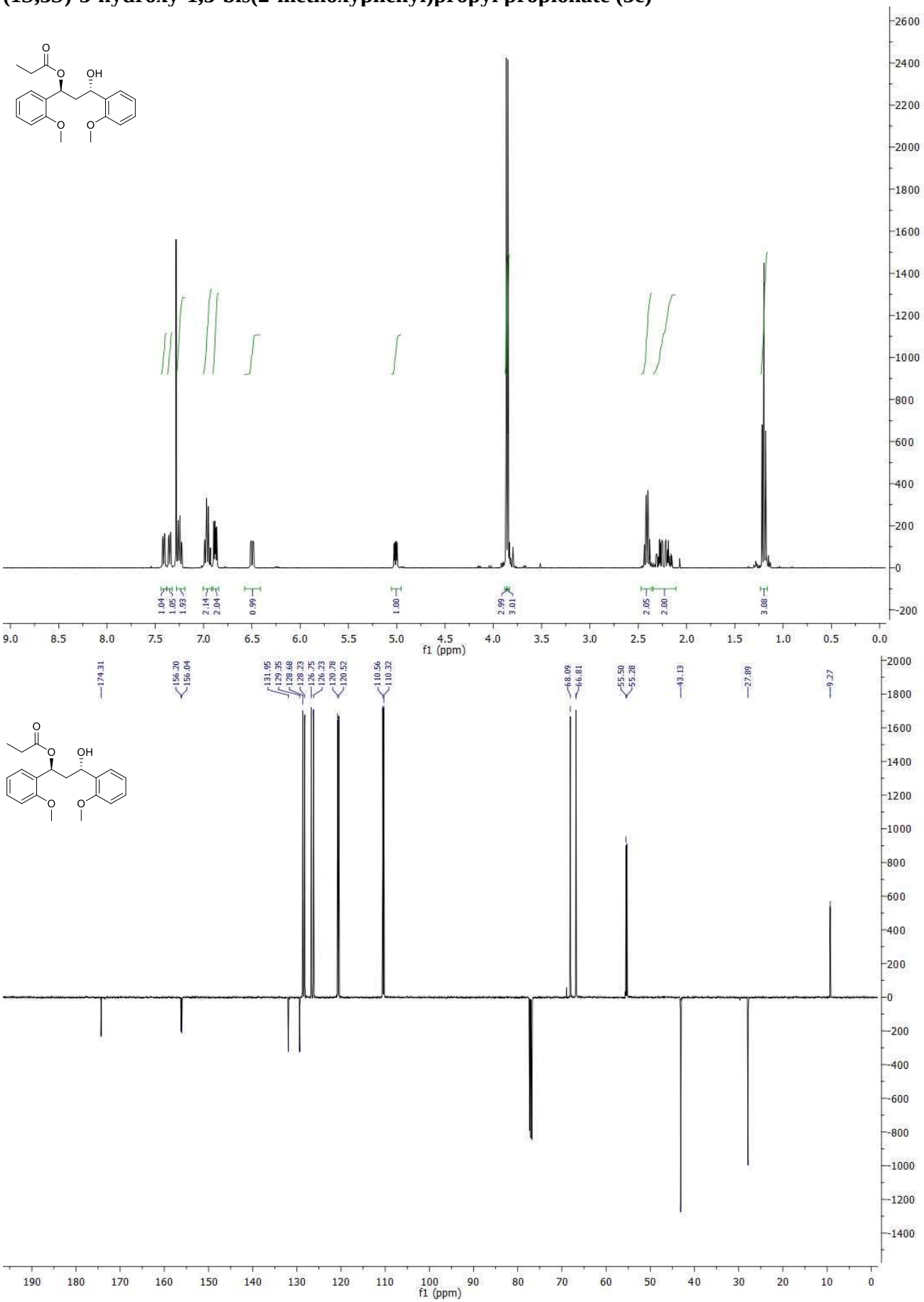
(1*S*,3*S*)-1,3-diphenylpropane-1,3-diyl dipropionate (4b)



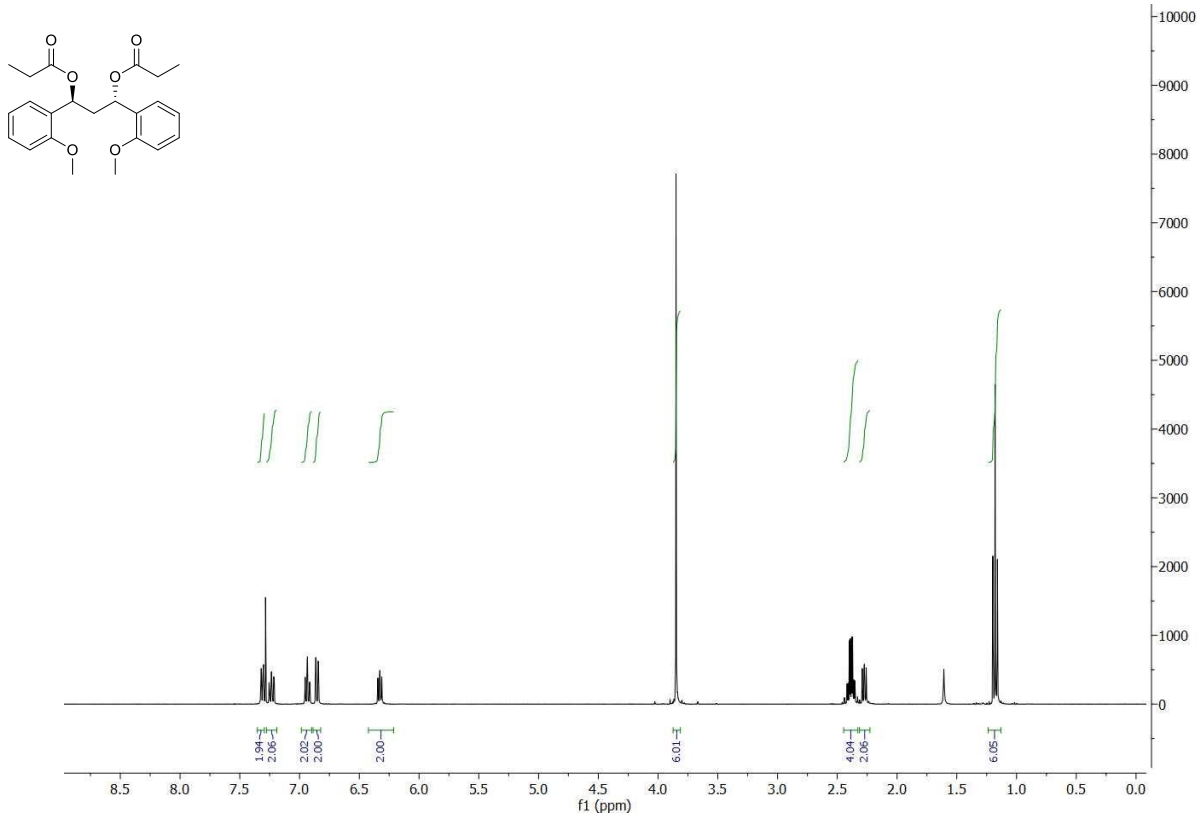
(1*R*,3*R*)-1,3-bis(2-methoxyphenyl)propane-1,3-diol (2c)



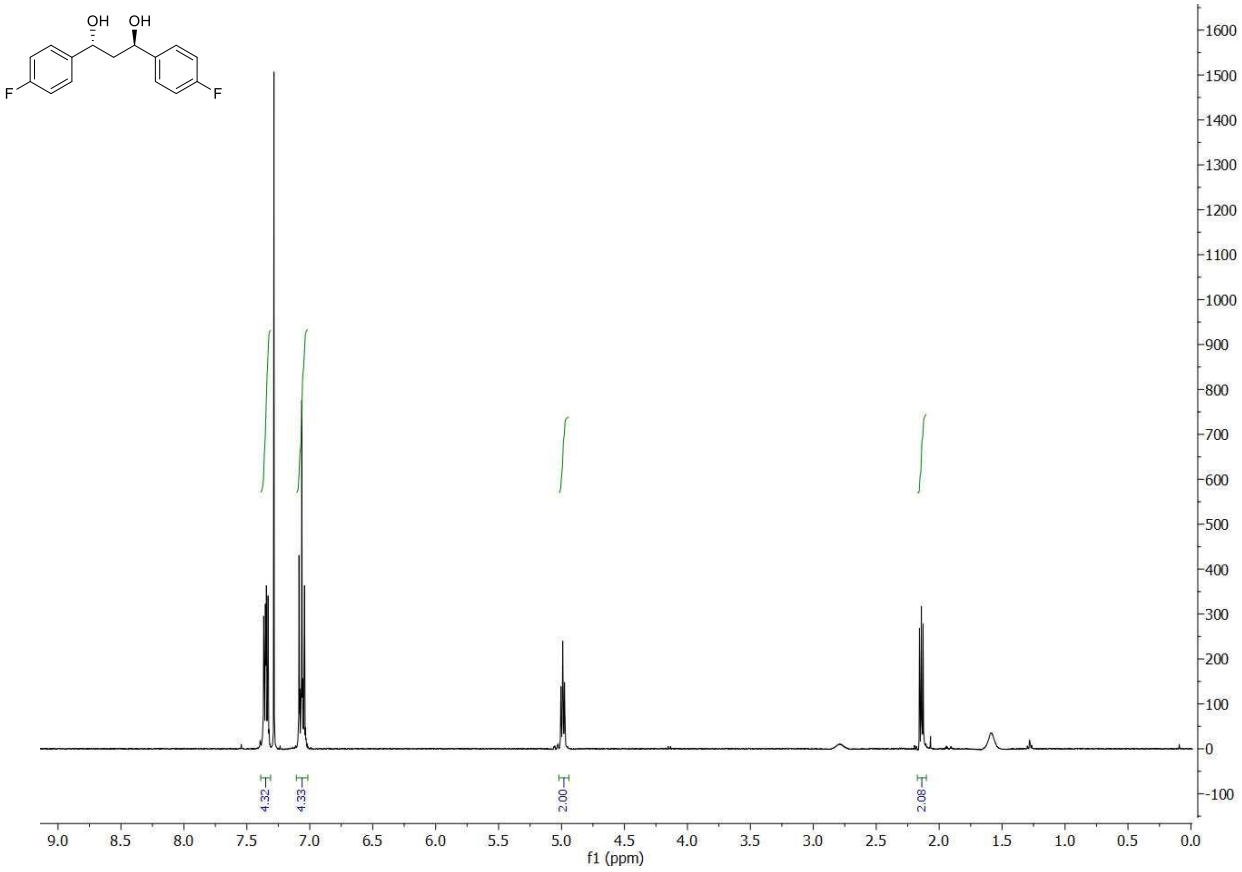
(1*S*,3*S*)-3-hydroxy-1,3-bis(2-methoxyphenyl)propyl propionate (3c)

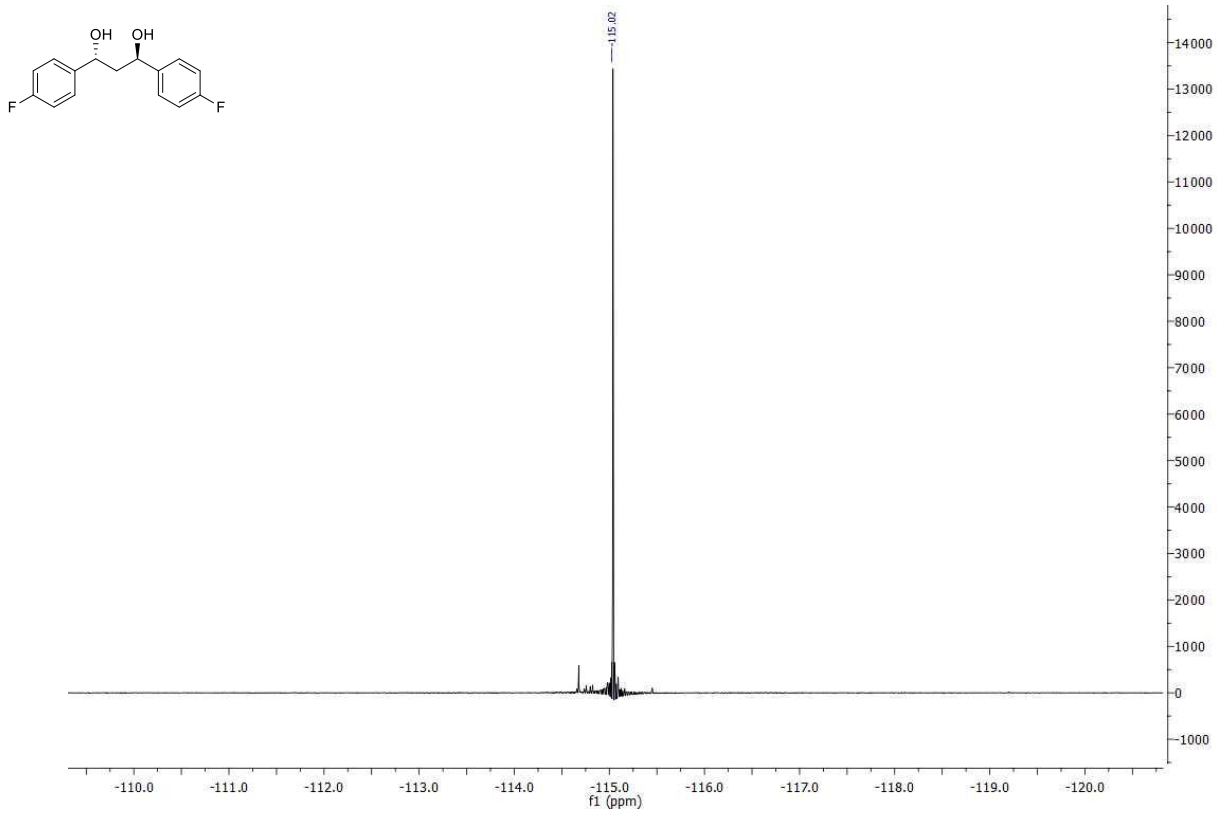


(1*S*,3*S*)-1,3-bis(2-methoxyphenyl)propane-1,3-diyl dipropionate (4c)

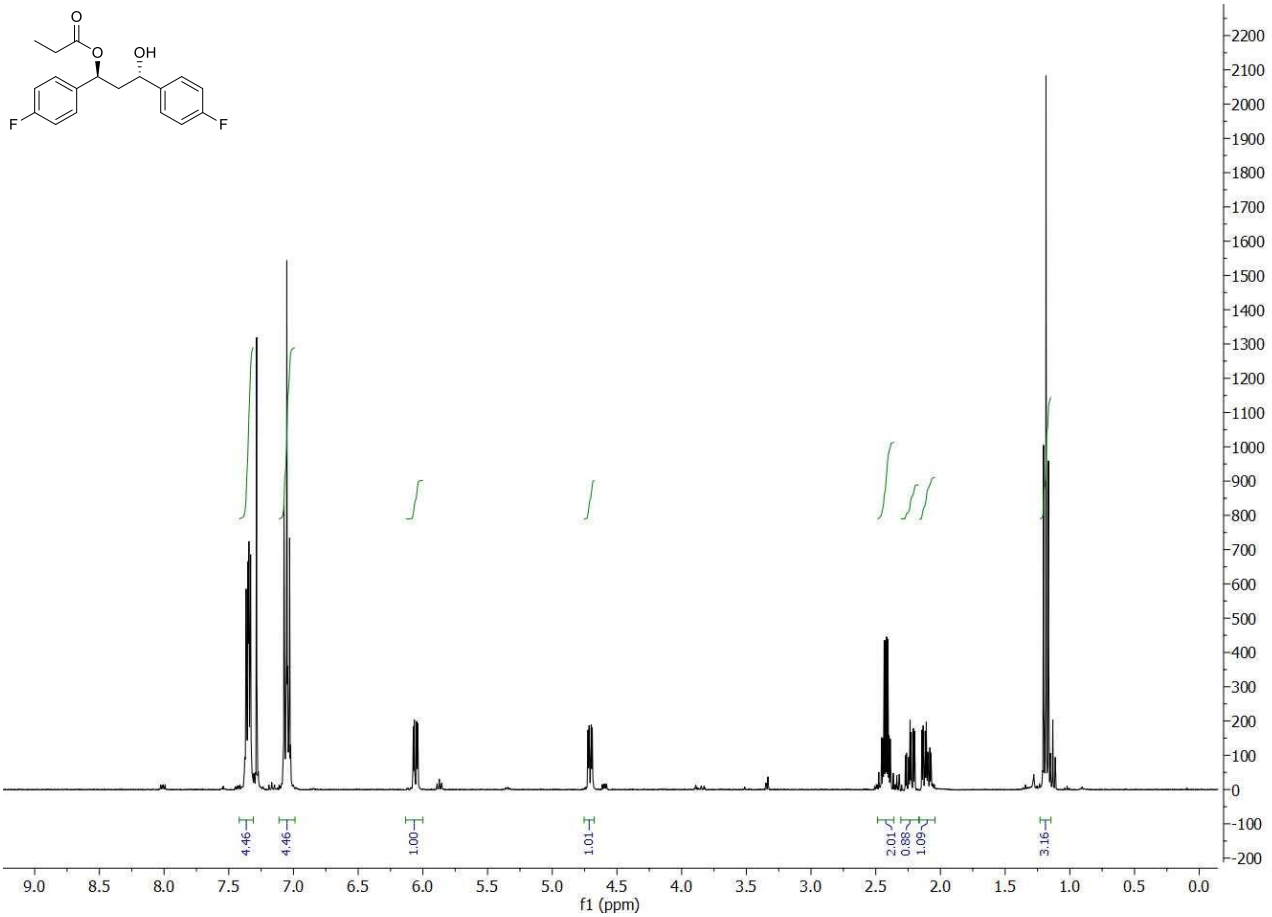


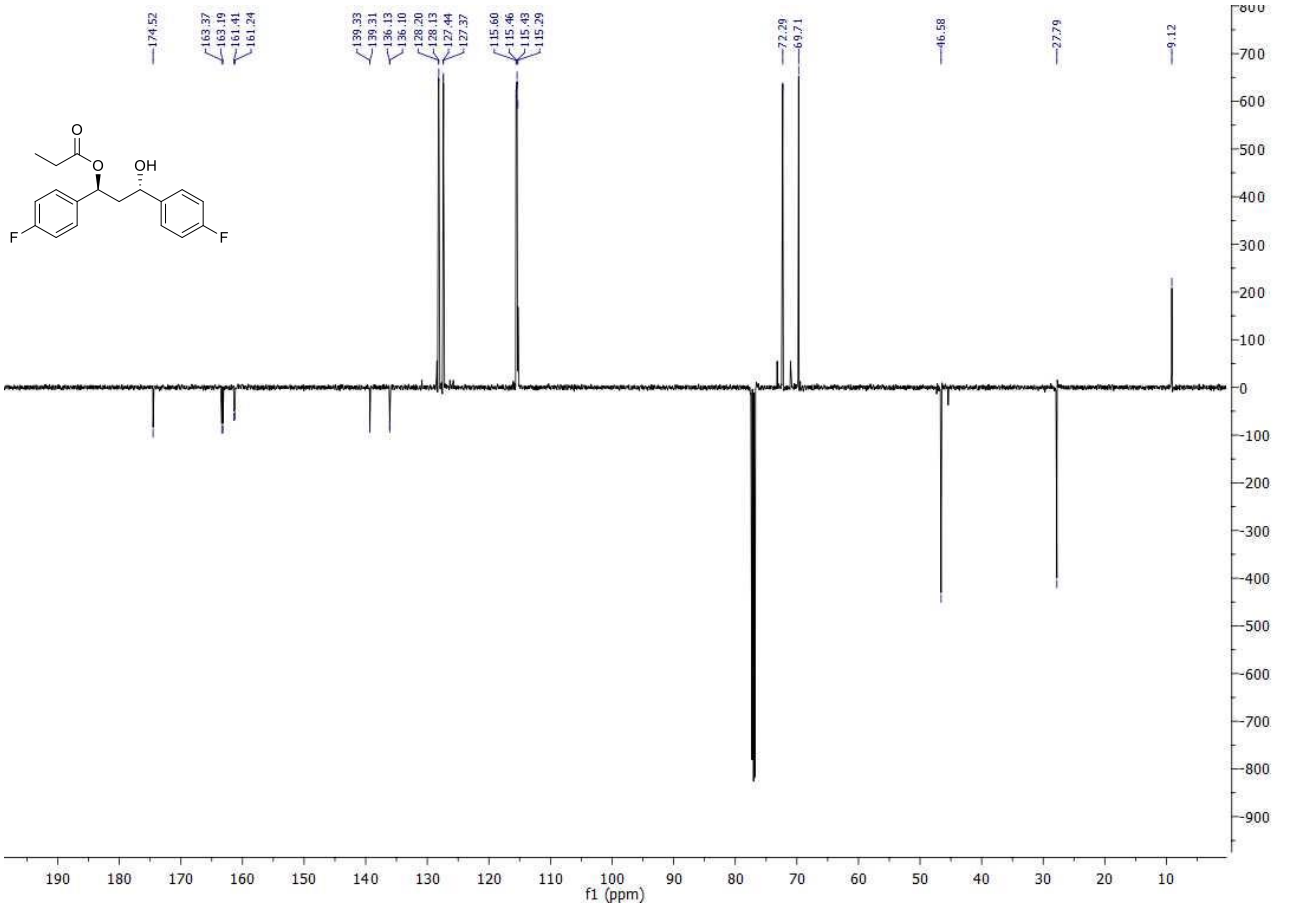
(1*R*,3*R*)-1,3-bis(4-fluorophenyl)propane-1,3-diol (2d)

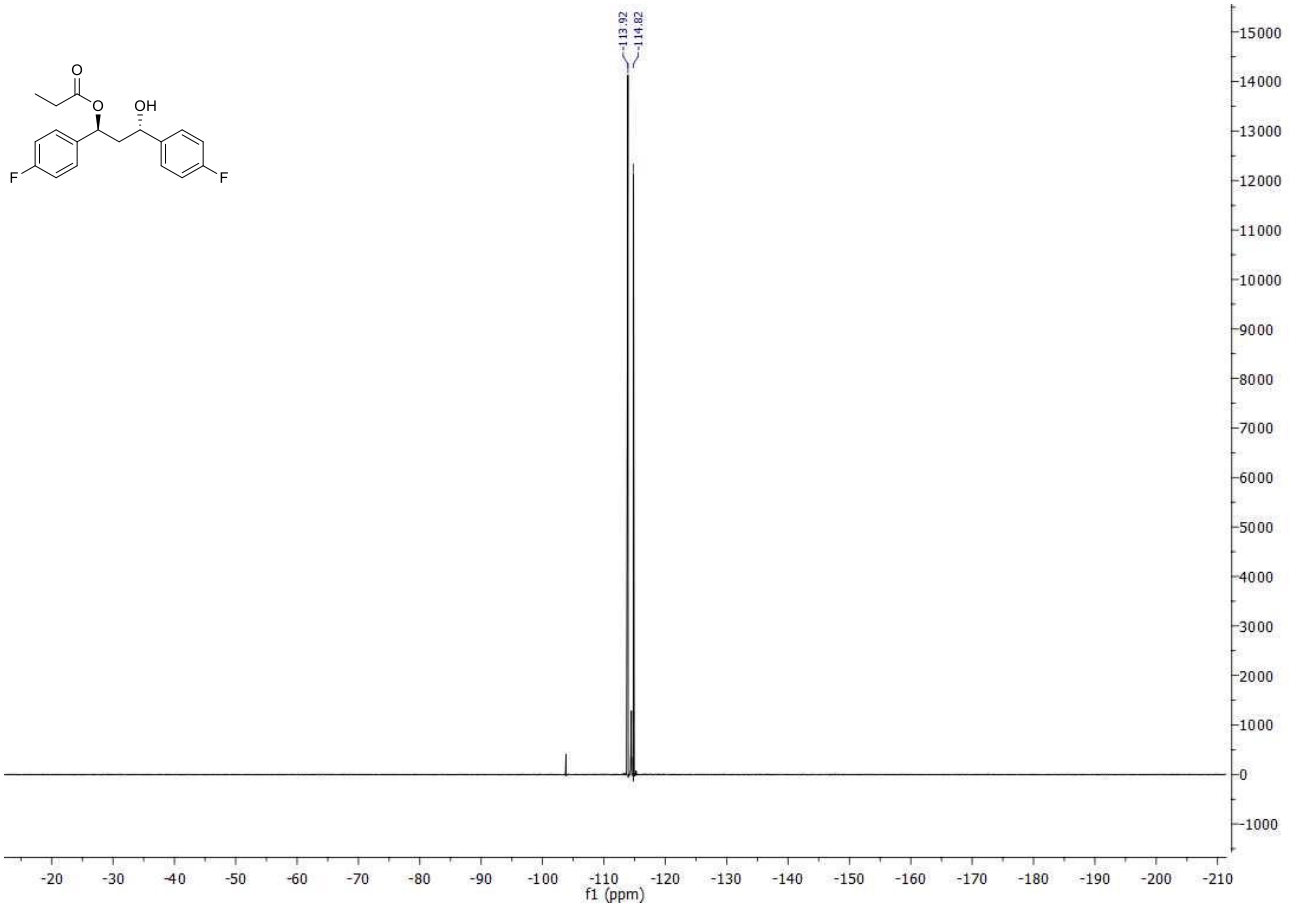




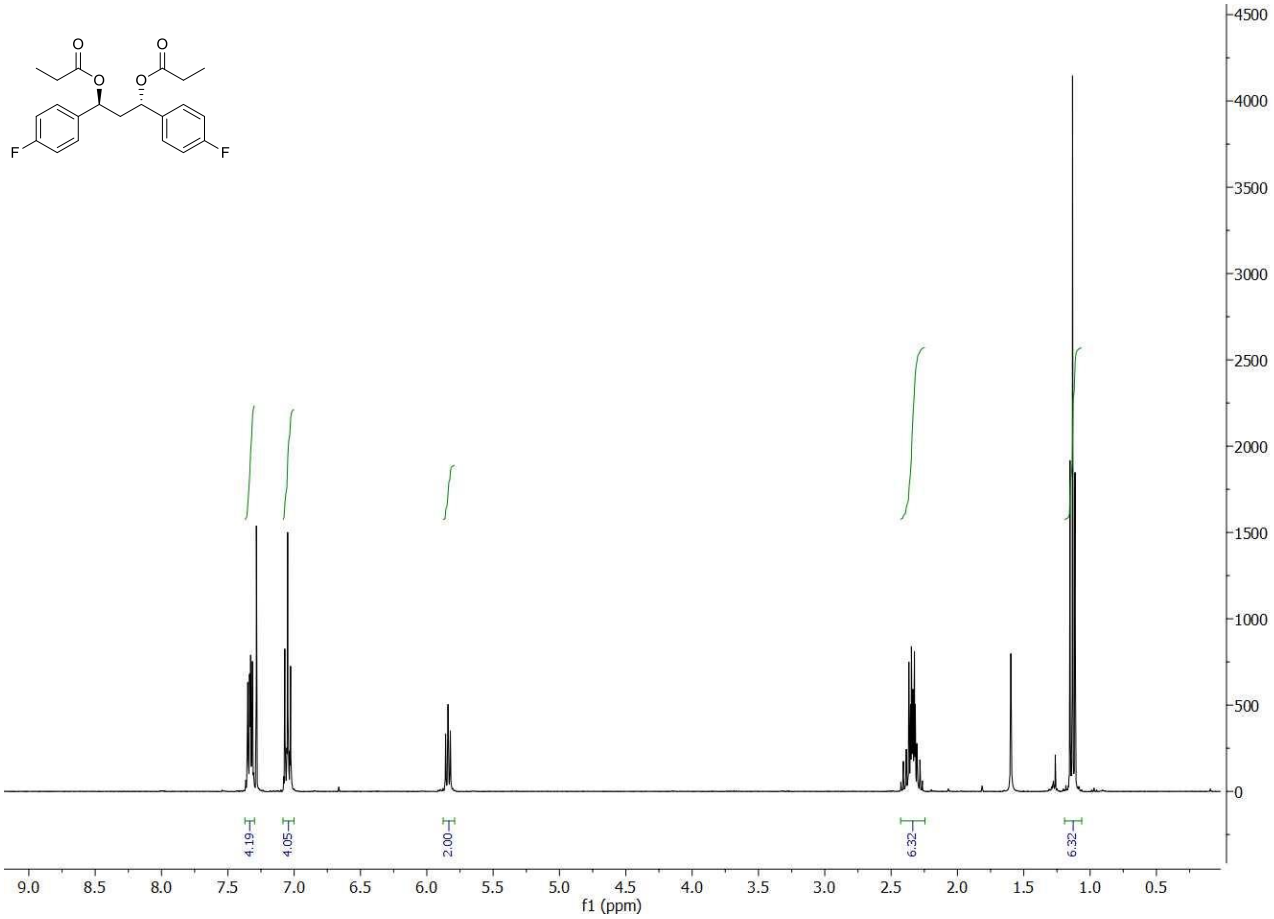
(1*S*,3*S*)-1,3-bis(4-fluorophenyl)-3-hydroxypropyl propionate (3d)

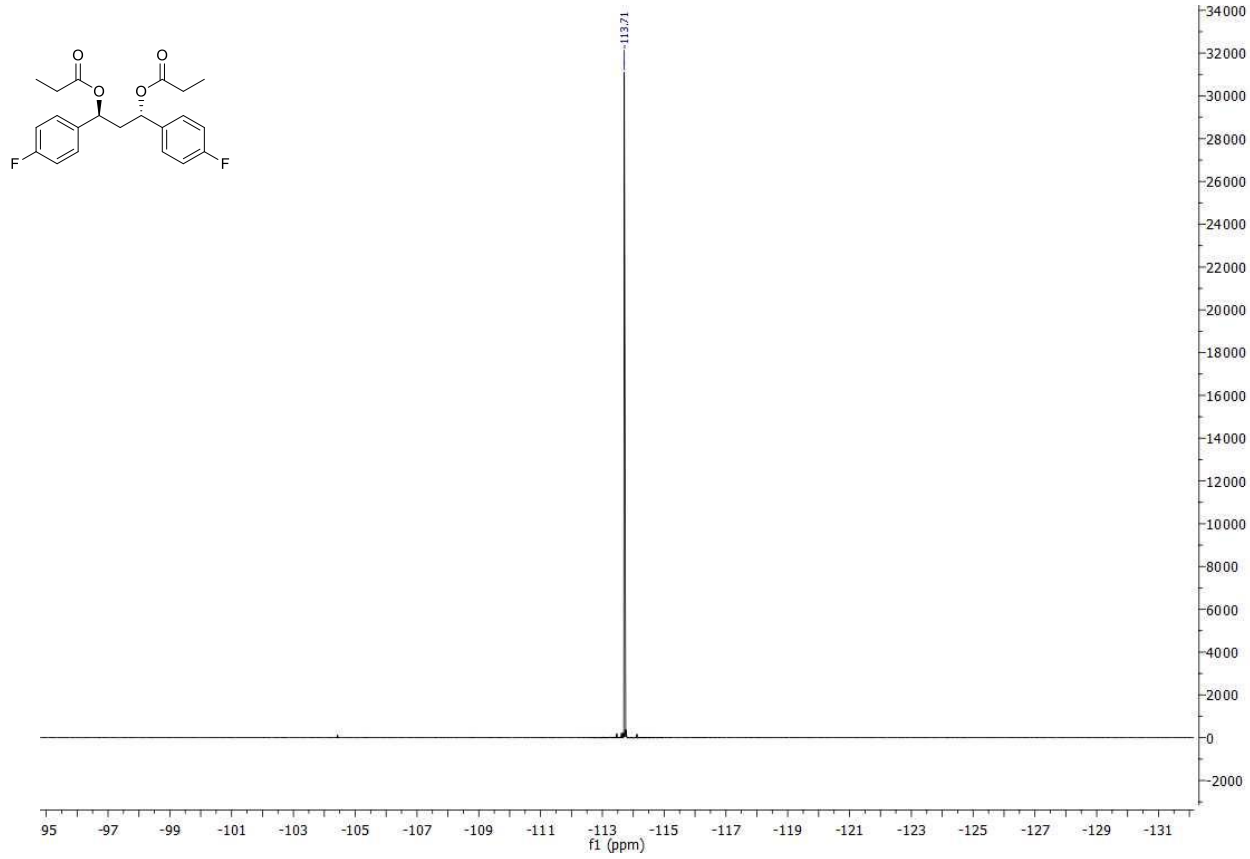



174.52
163.37
163.19
161.41
161.24
139.33
139.31
136.13
136.10
128.20
128.13
127.44
127.37
115.60
115.46
115.48
115.29
72.29
69.71
46.58
27.79
9.12
O
O
OH
F
F
f1 (ppm)

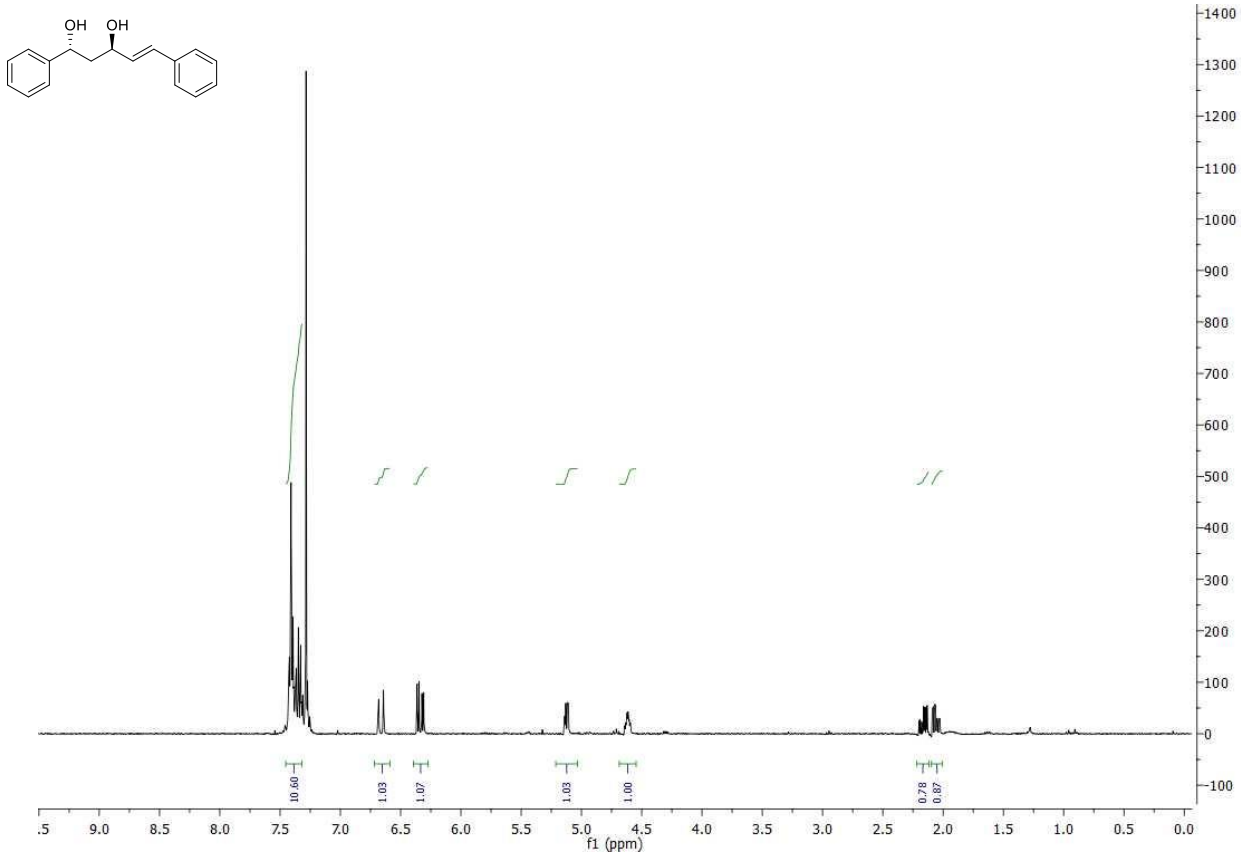

-113.92
-114.82
O
O
OH
F
F
f1 (ppm)

(1*S*,3*S*)-1,3-bis(4-fluorophenyl)propane-1,3-diyl dipropionate (4d)

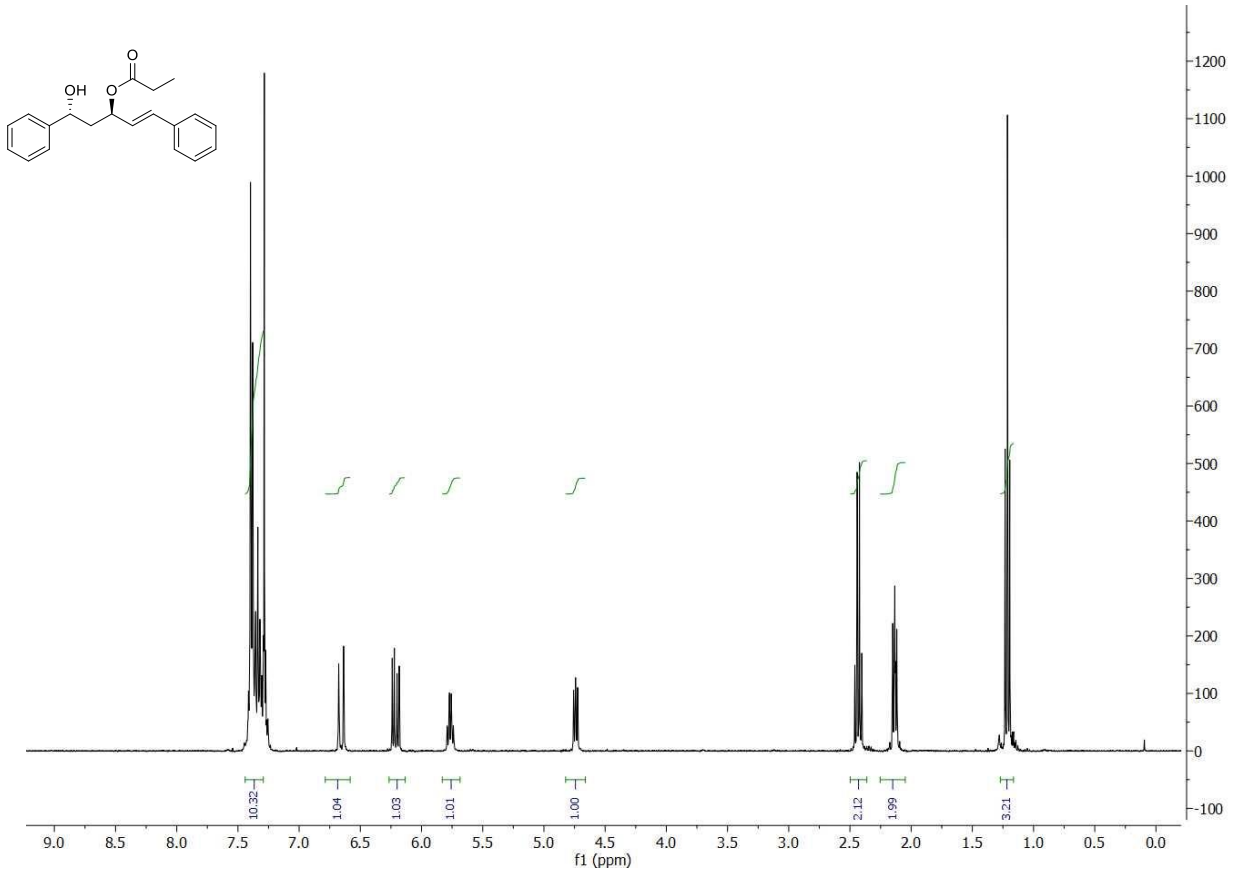


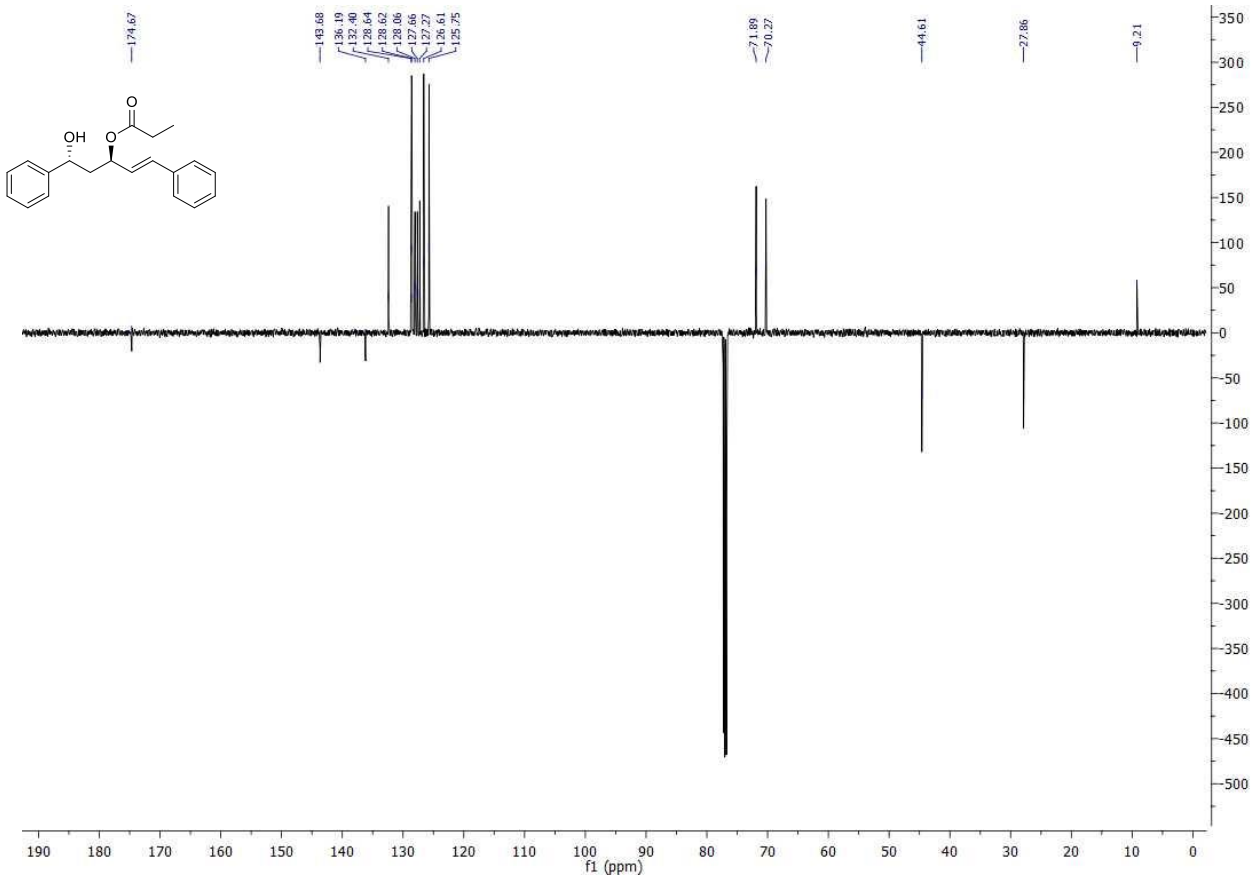


(1*R*,3*R*,*E*)-1,5-diphenylpent-4-ene-1,3-diol (2e)

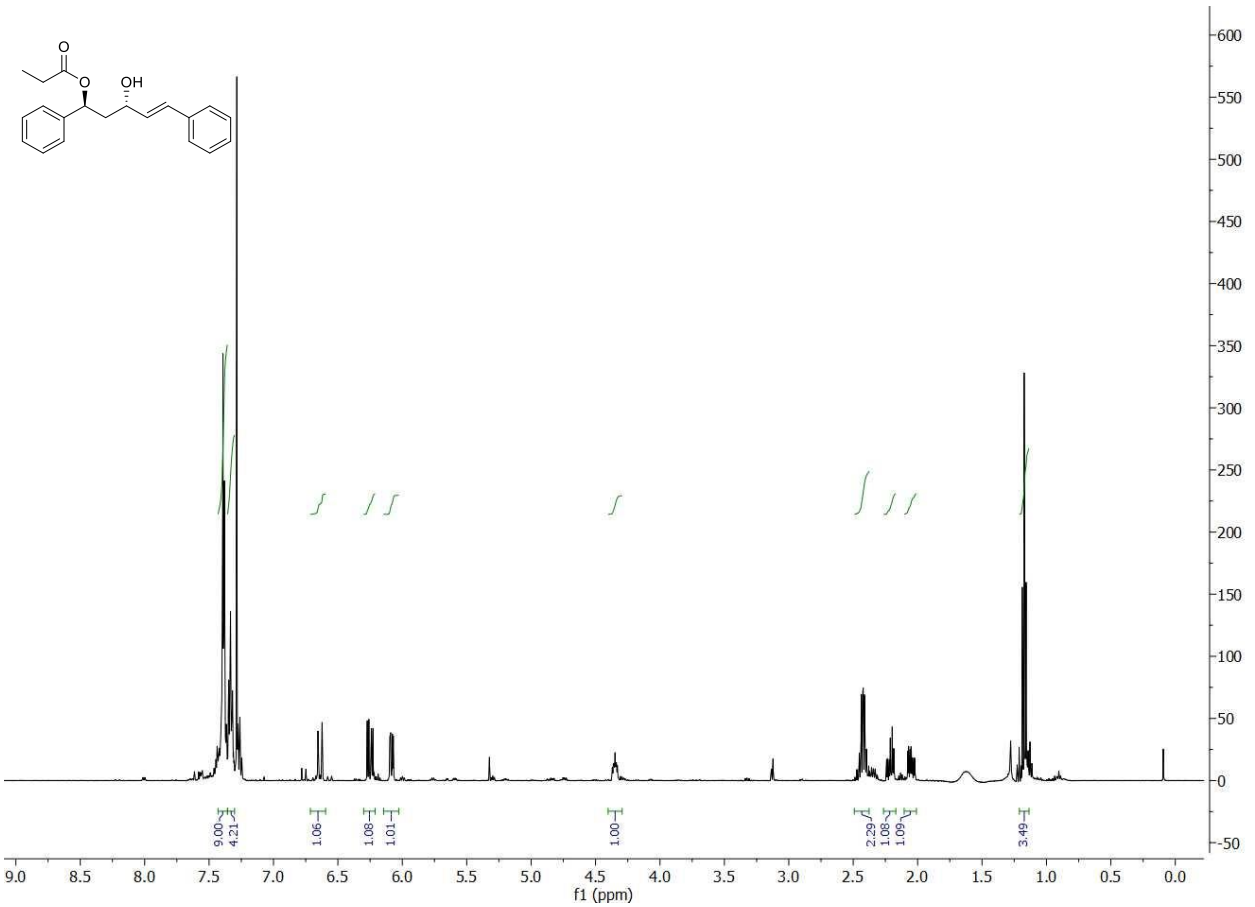


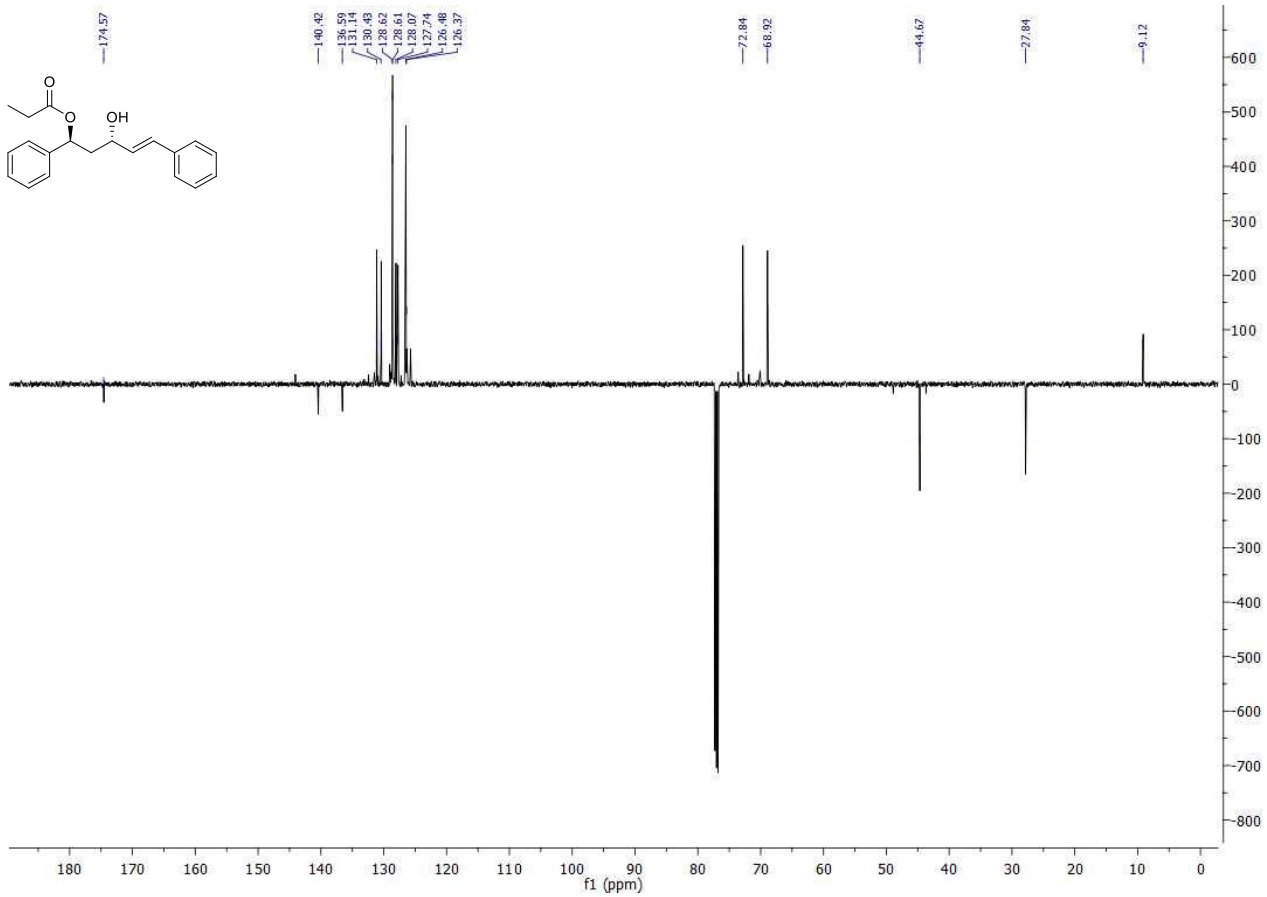
(3*R*,5*R*,*E*)-5-hydroxy-1,5-diphenylpent-1-en-3-yl propionate (3e')



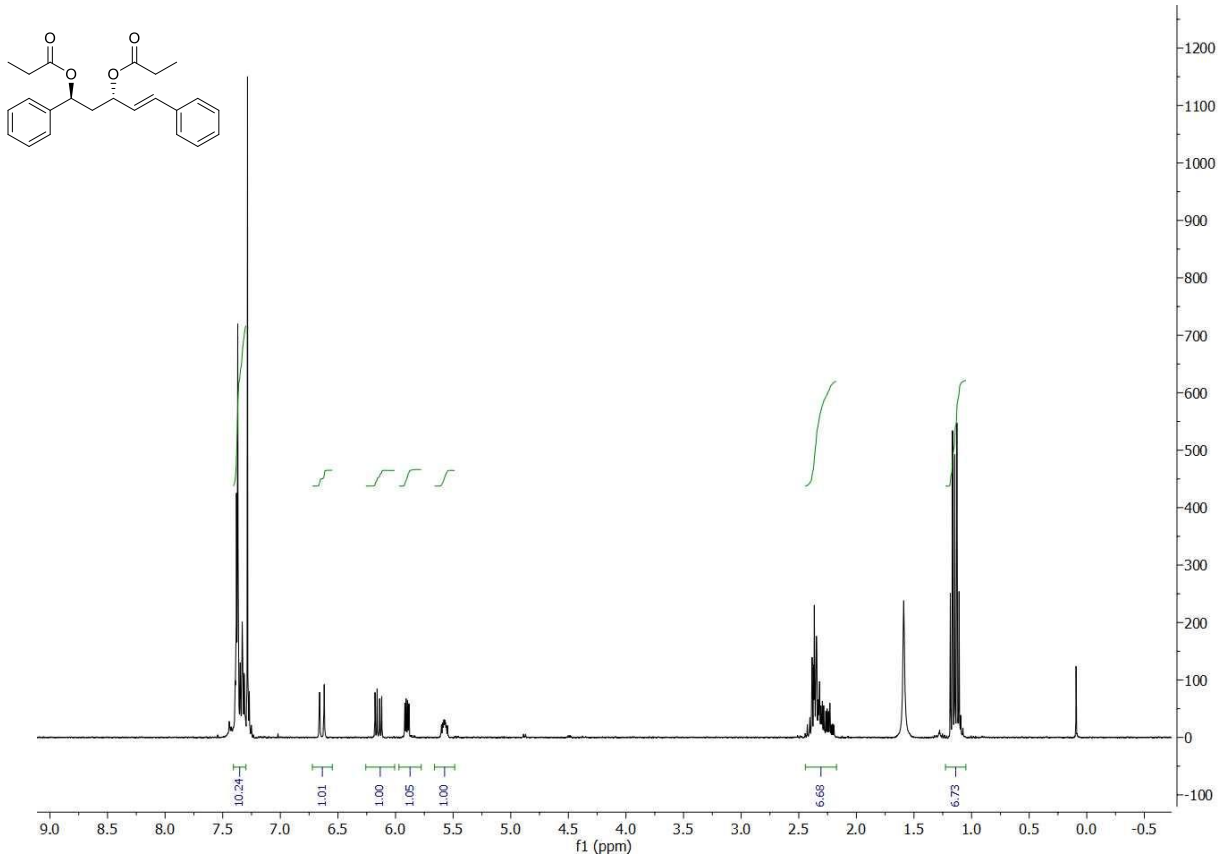


(1*S*,3*S*,*E*)-3-hydroxy-1,5-diphenylpent-4-en-1-yl propionate (3e'')

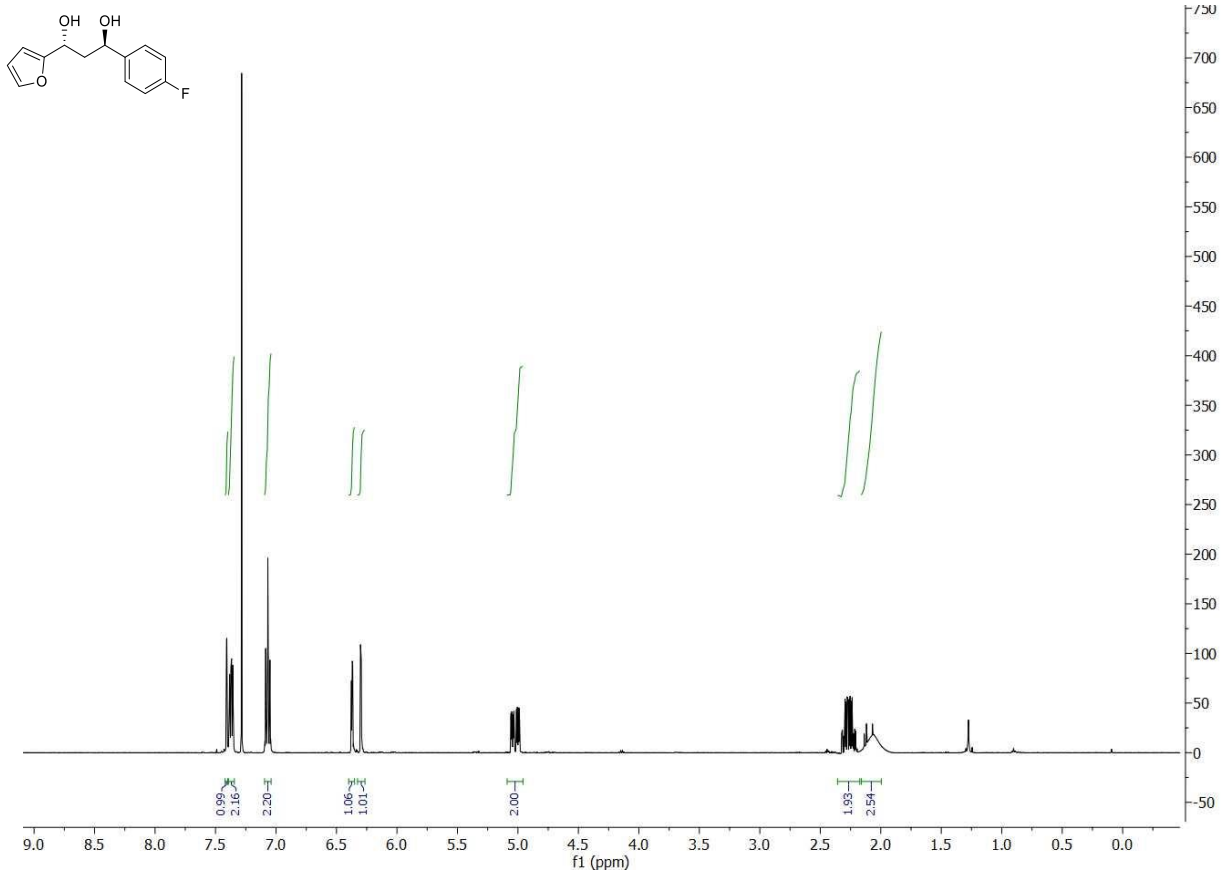


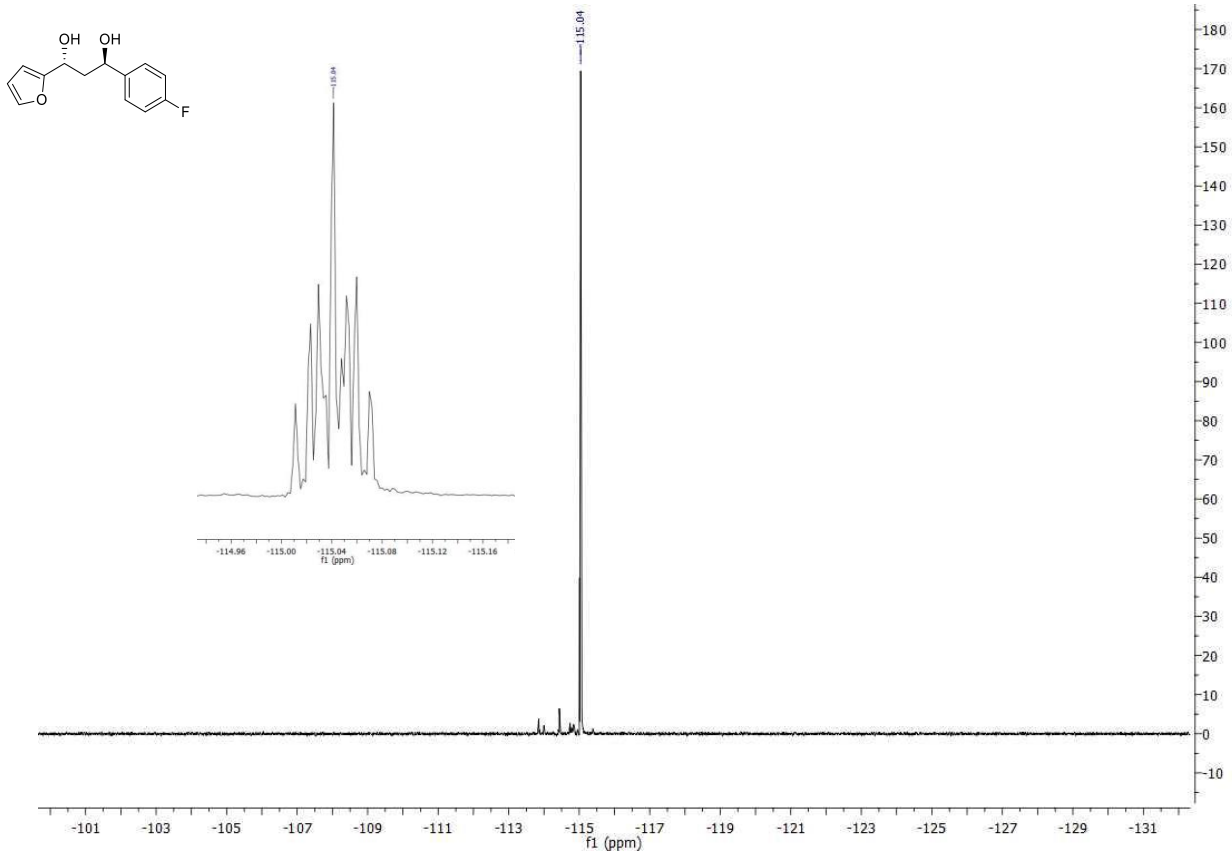


(1*S*,3*S*,*E*)-1,5-diphenylpent-4-ene-1,3-diyl dipropionate (4e)

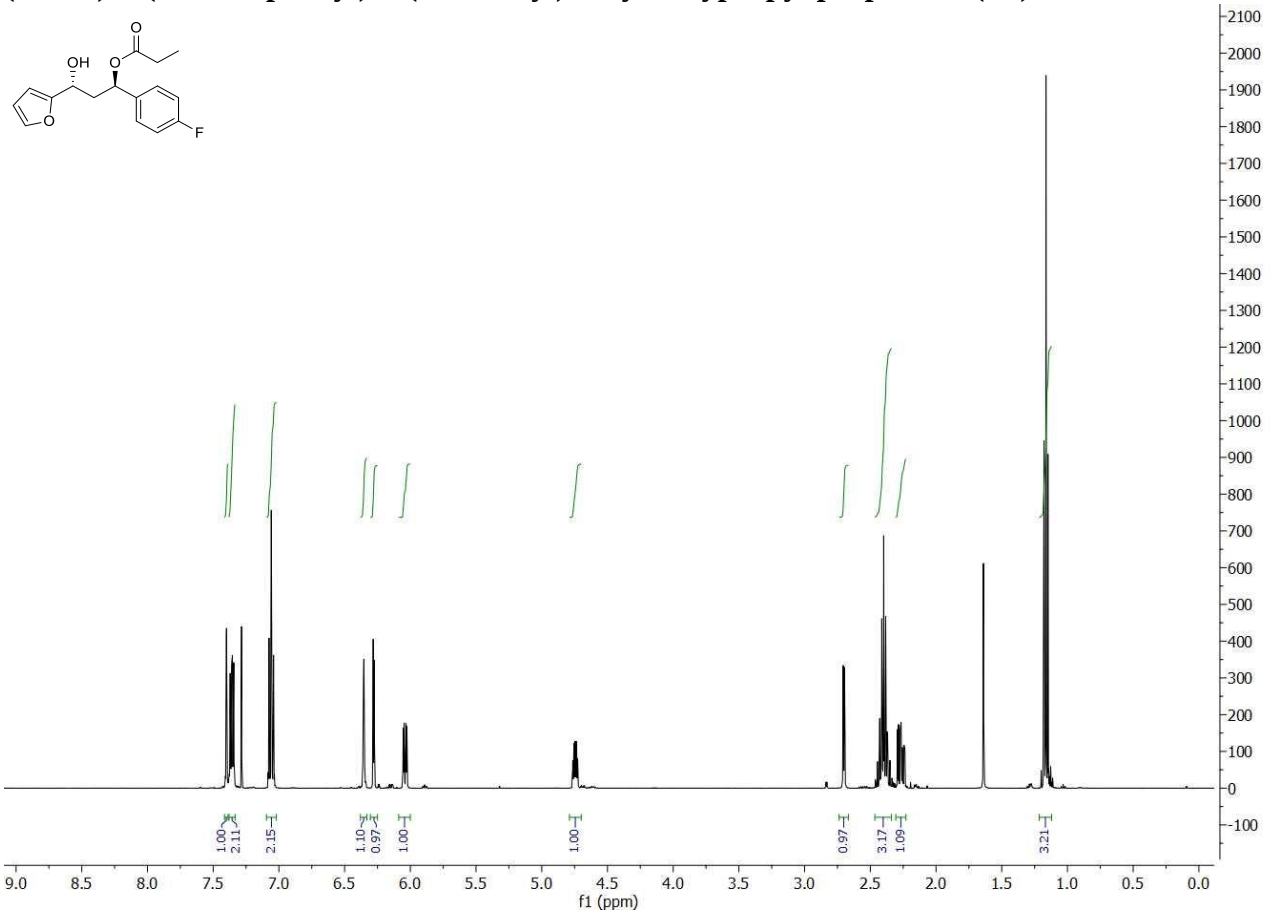


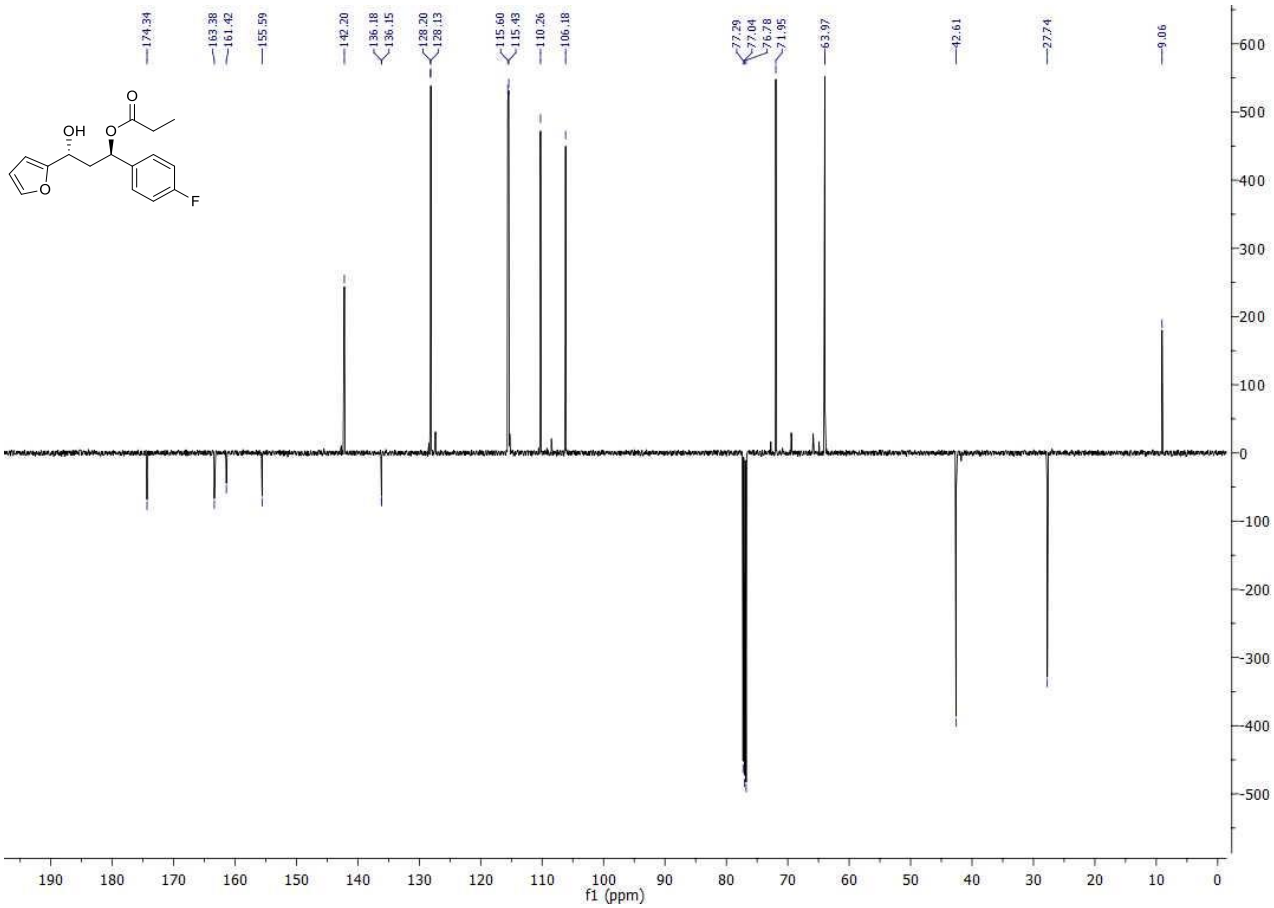
(1*R*,3*R*)-1-(4-fluorophenyl)-3-(furan-2-yl)propane-1,3-diol (2f)

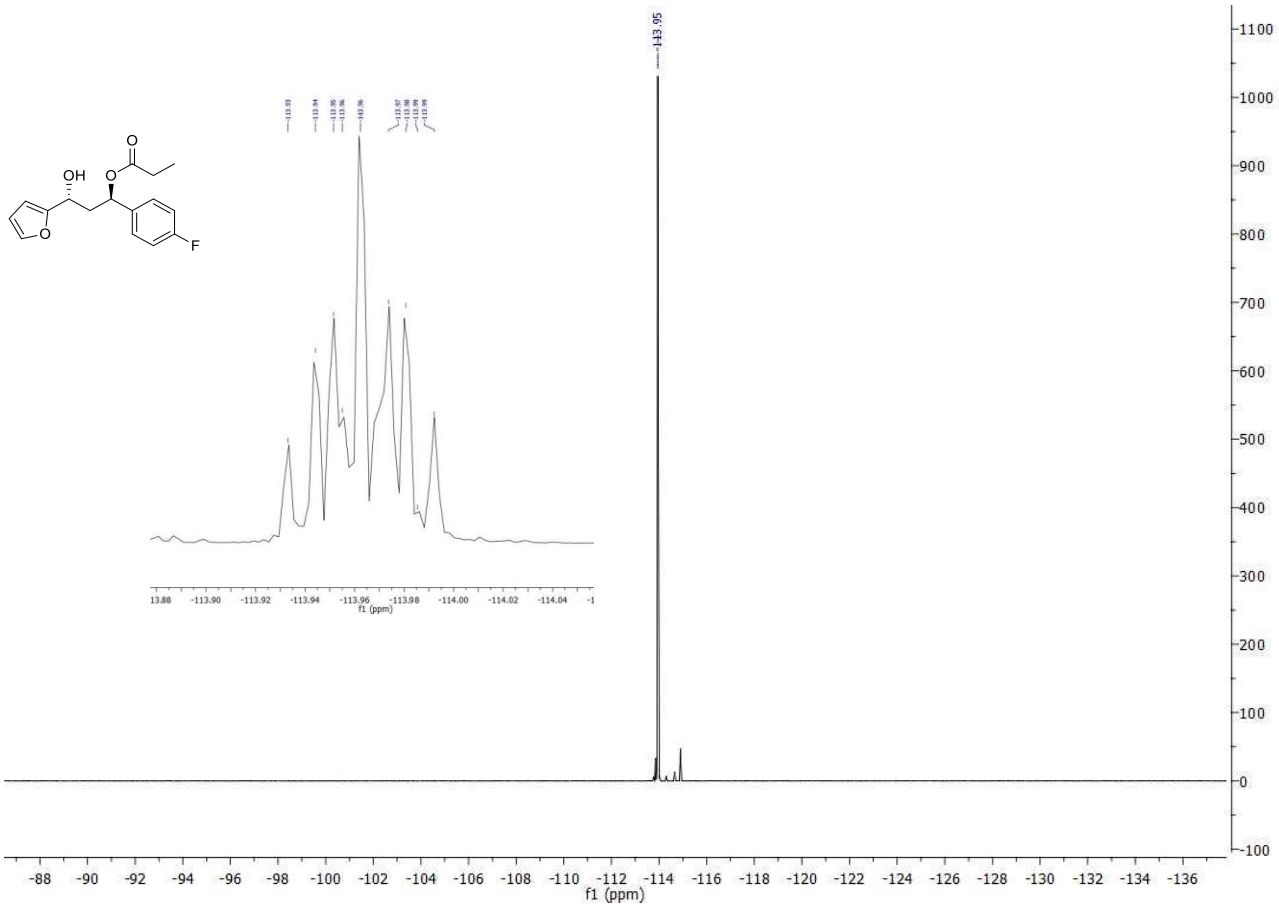




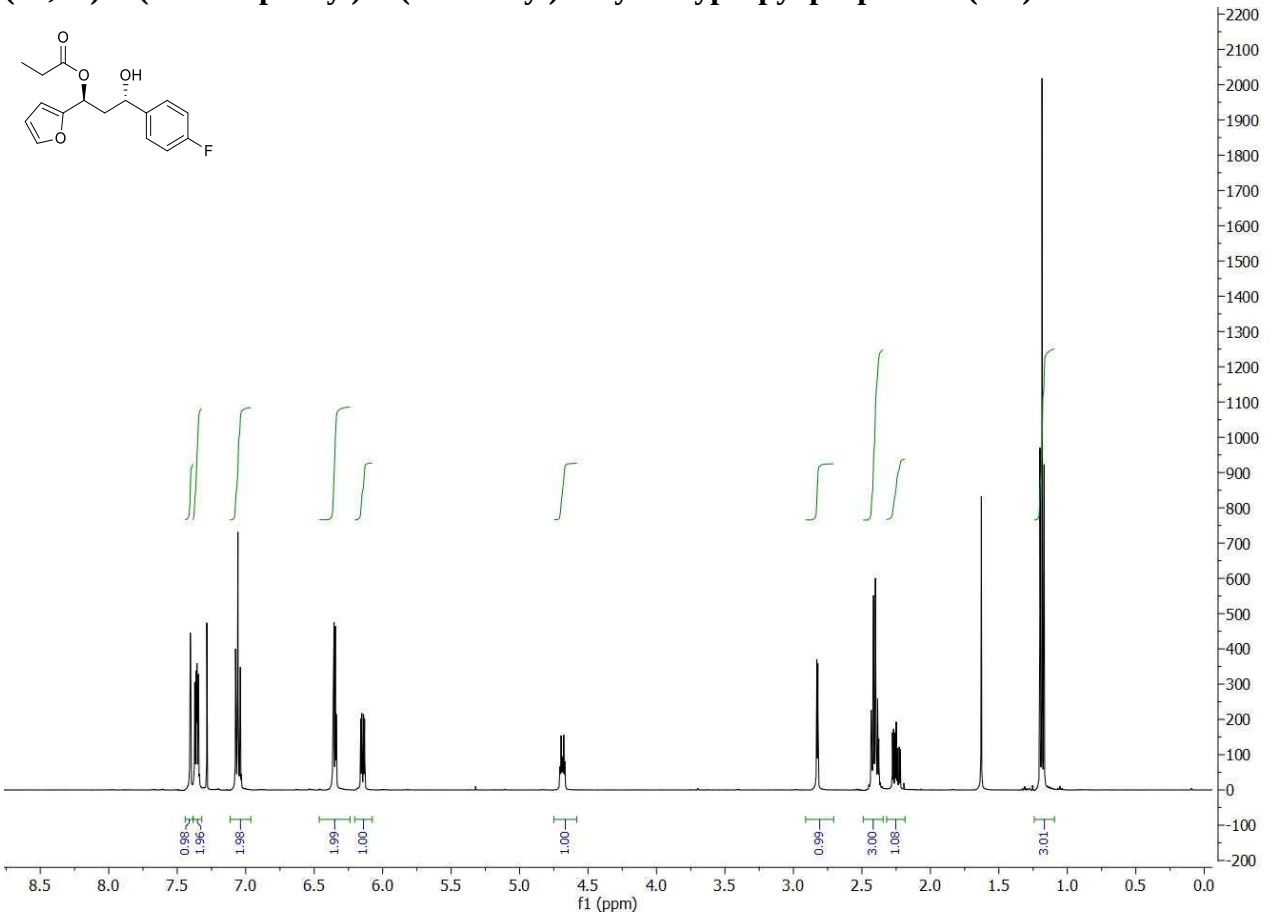
(1*R*,3*R*)-3-(4-fluorophenyl)-1-(furan-2-yl)-3-hydroxypropyl propionate (3f′)

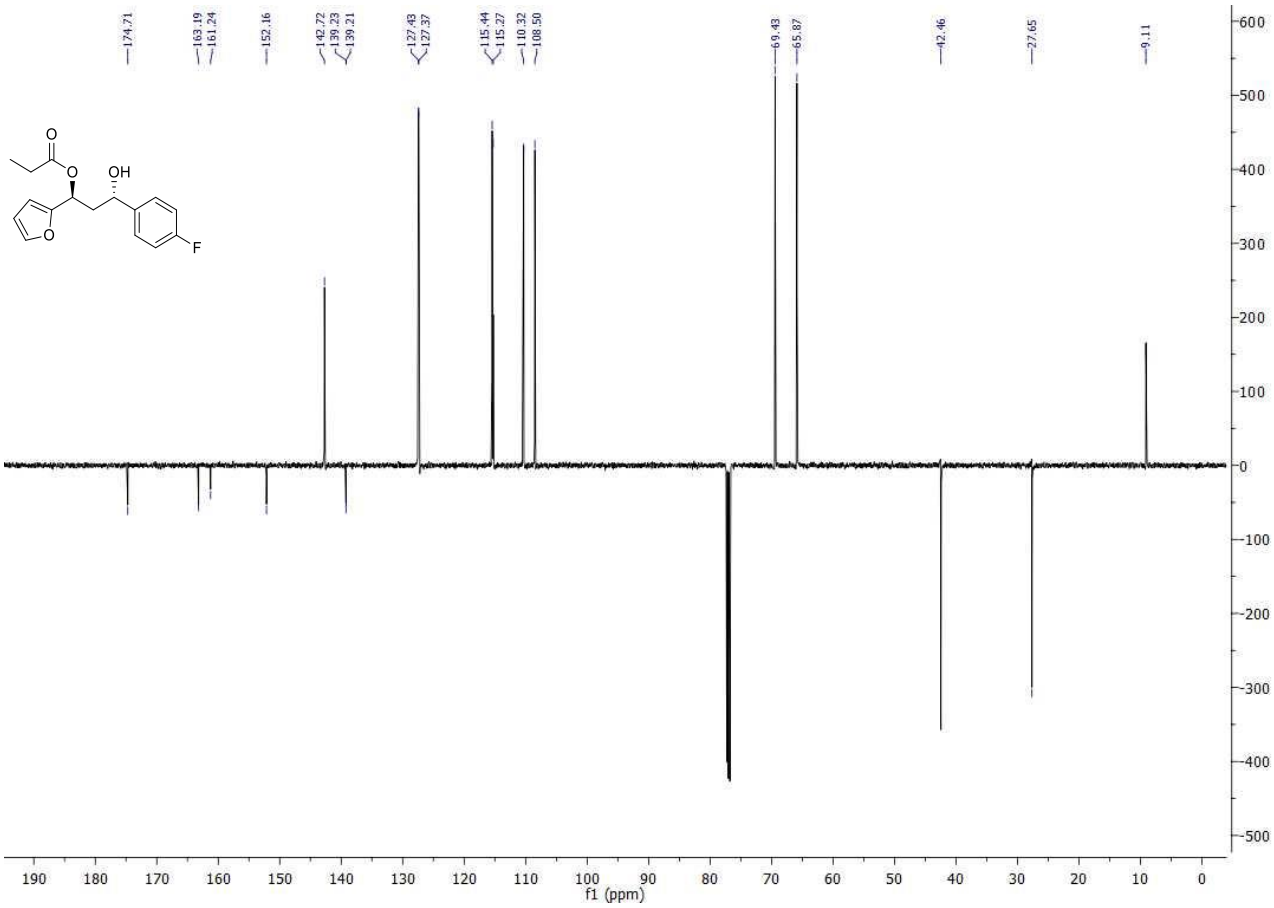


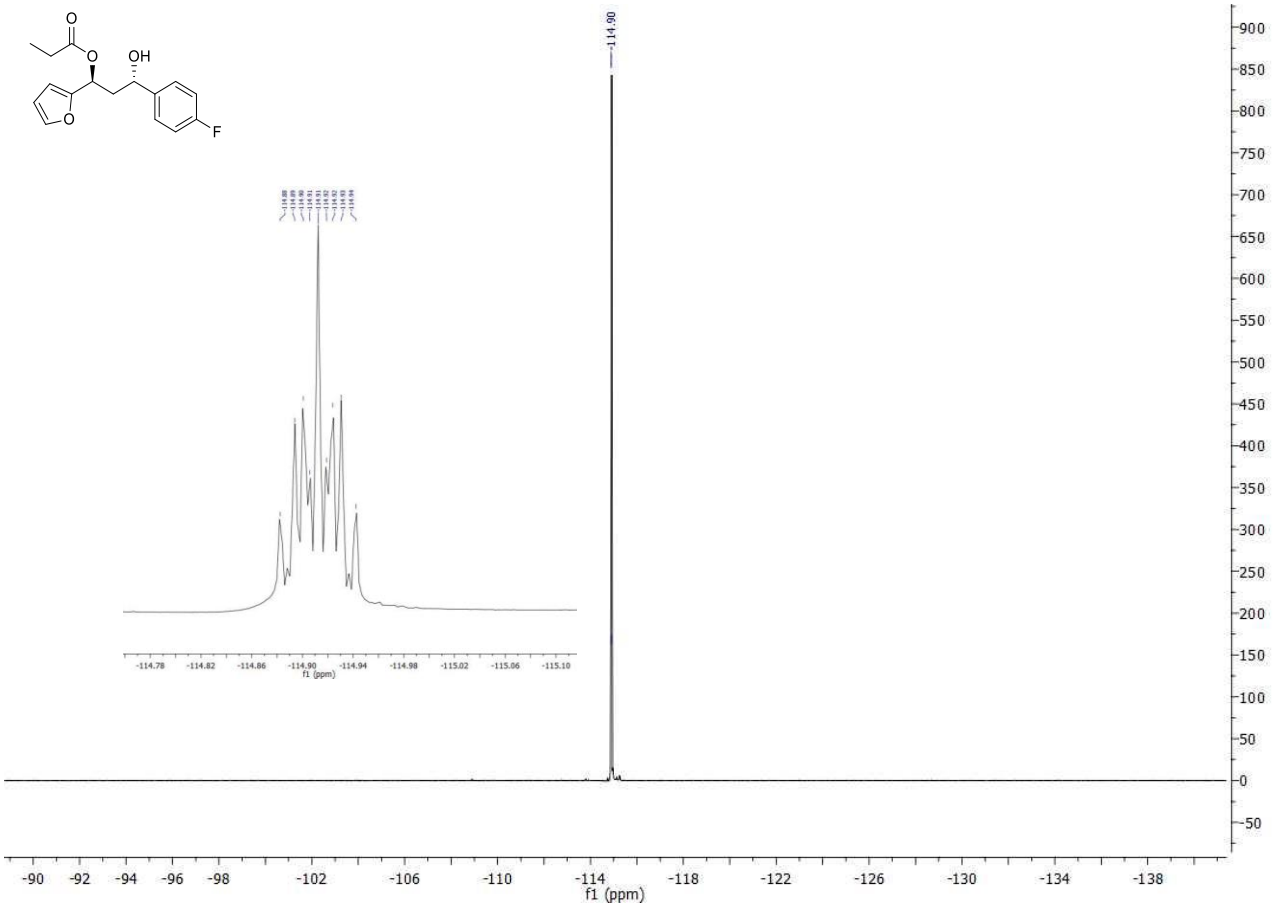




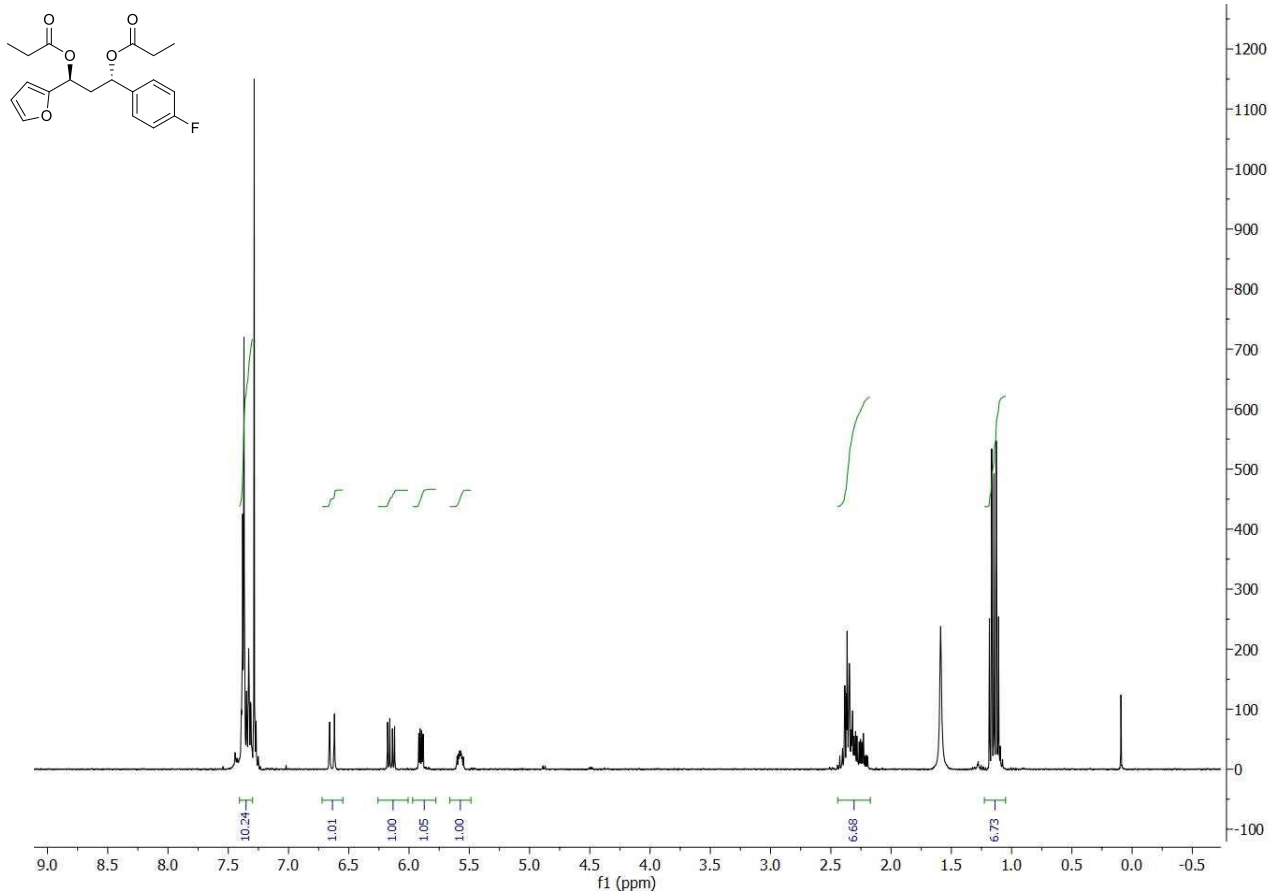
(1*S*,3*S*)-1-(4-fluorophenyl)-3-(furan-2-yl)-3-hydroxypropyl propionate (3f′′)

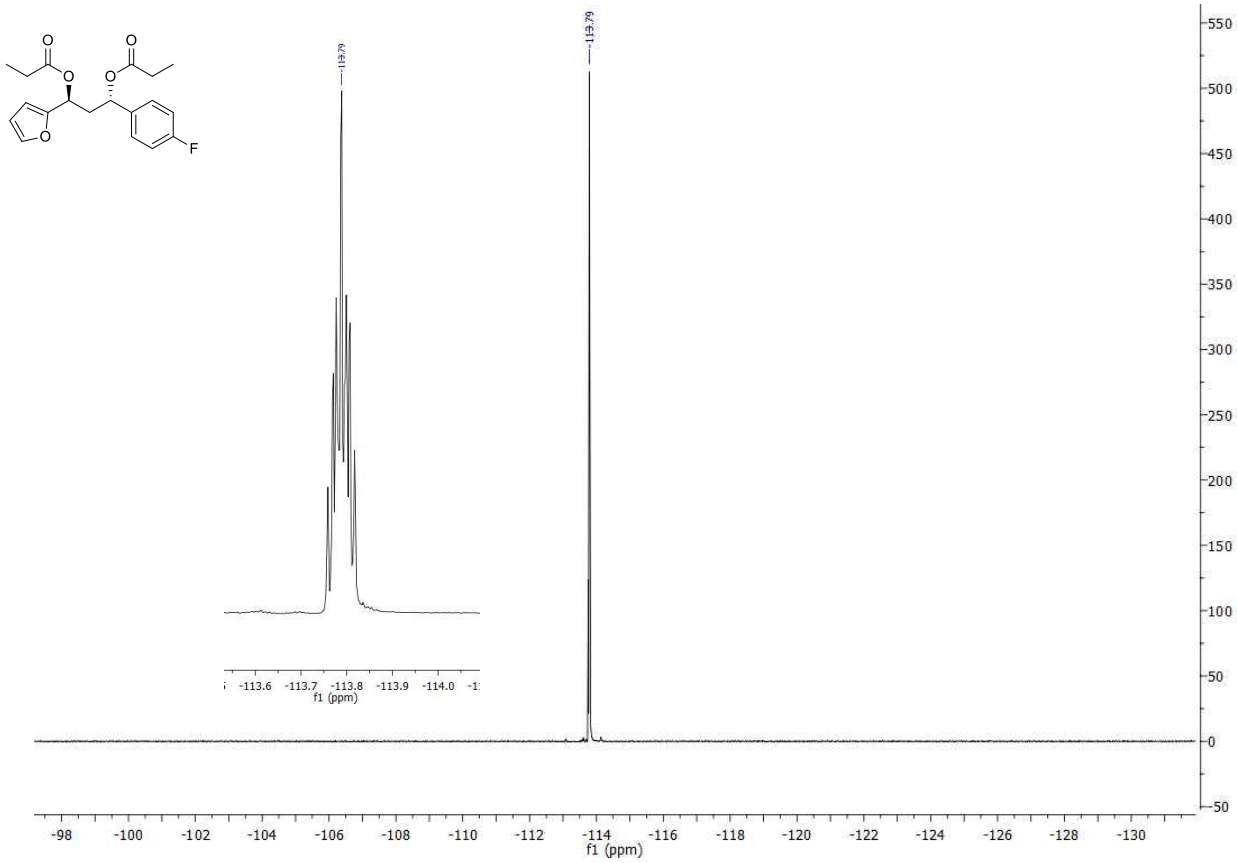



174.71
163.19
161.24
152.16
142.72
139.23
139.21
127.43
127.37
115.44
115.27
110.32
108.50
69.43
65.87
42.46
27.65
9.11
O
O
OH
F
f1 (ppm)

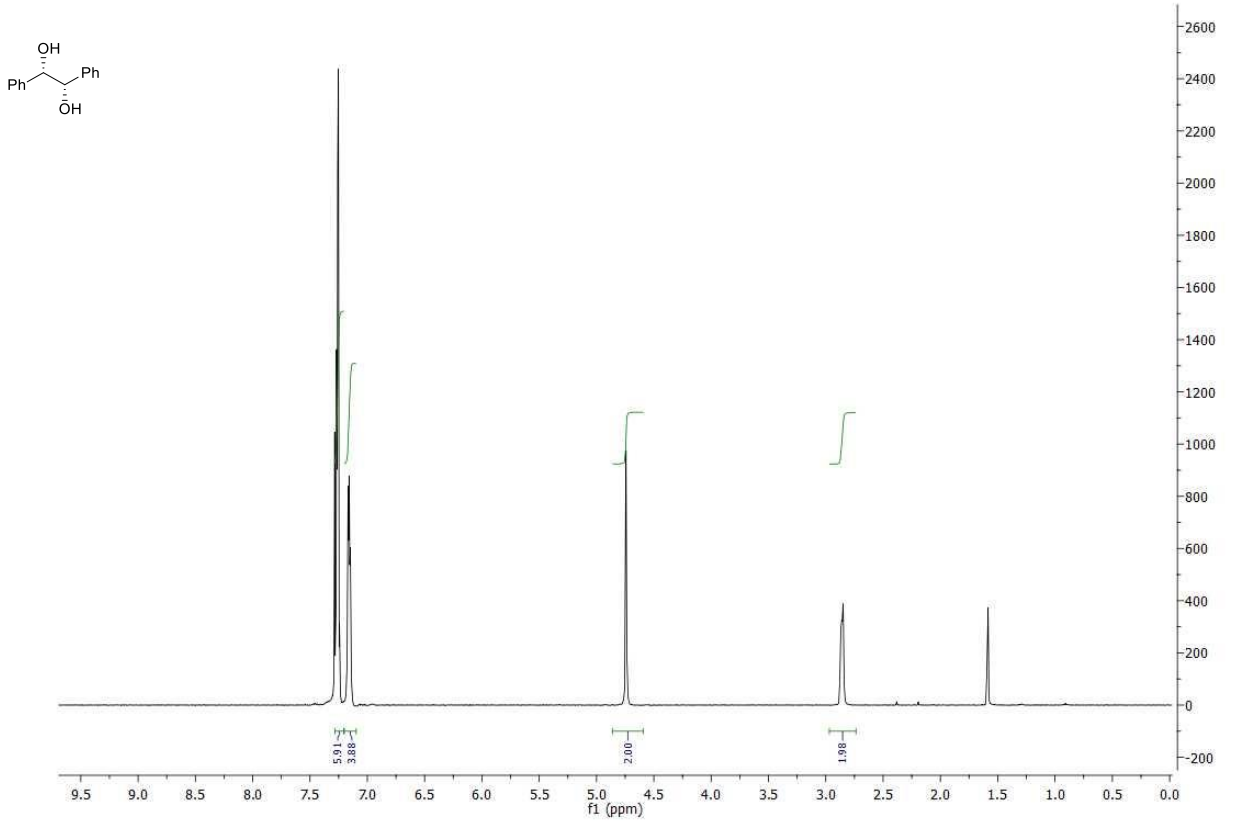

-114.90
O
O
OH
F
f1 (ppm)

(1*S*,3*S*)-1-(4-fluorophenyl)-3-(furan-2-yl)propane-1,3-diyl dipropionate (4f)

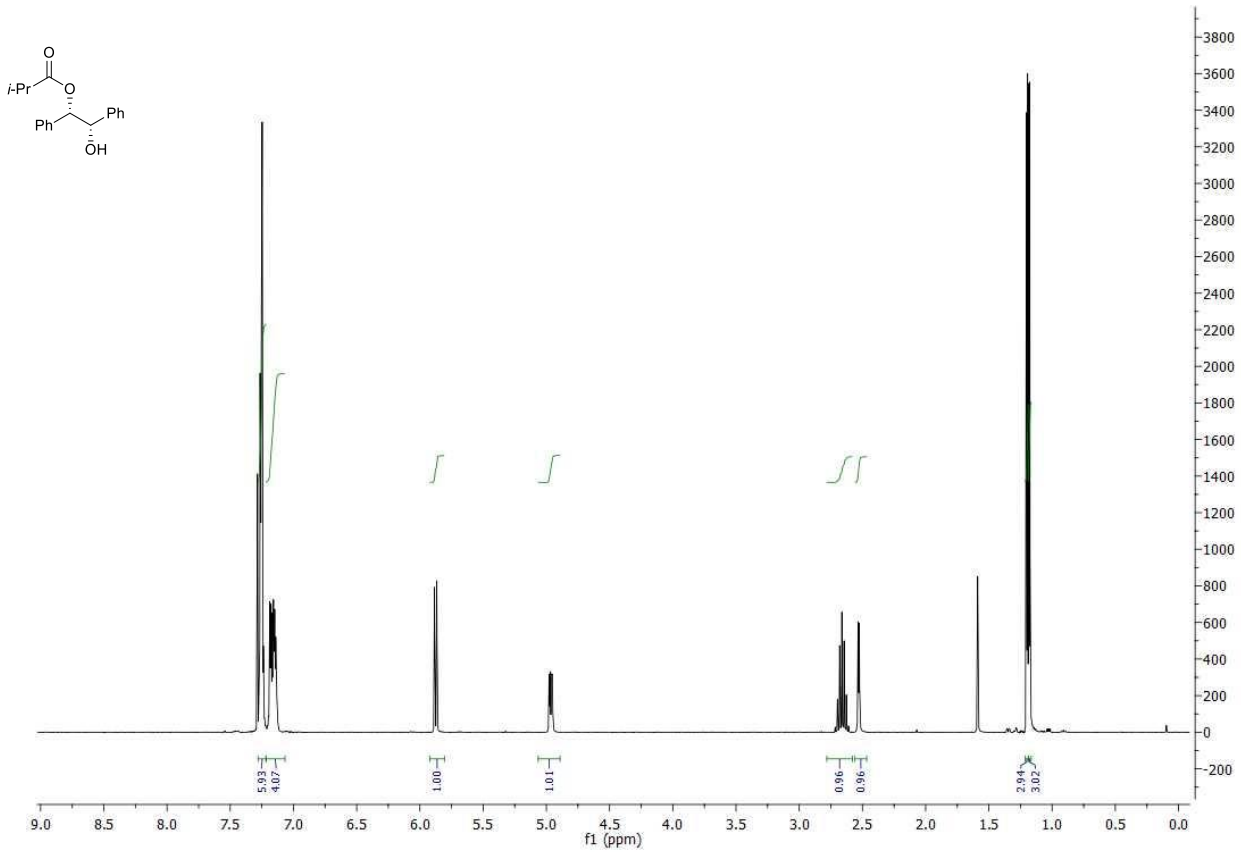




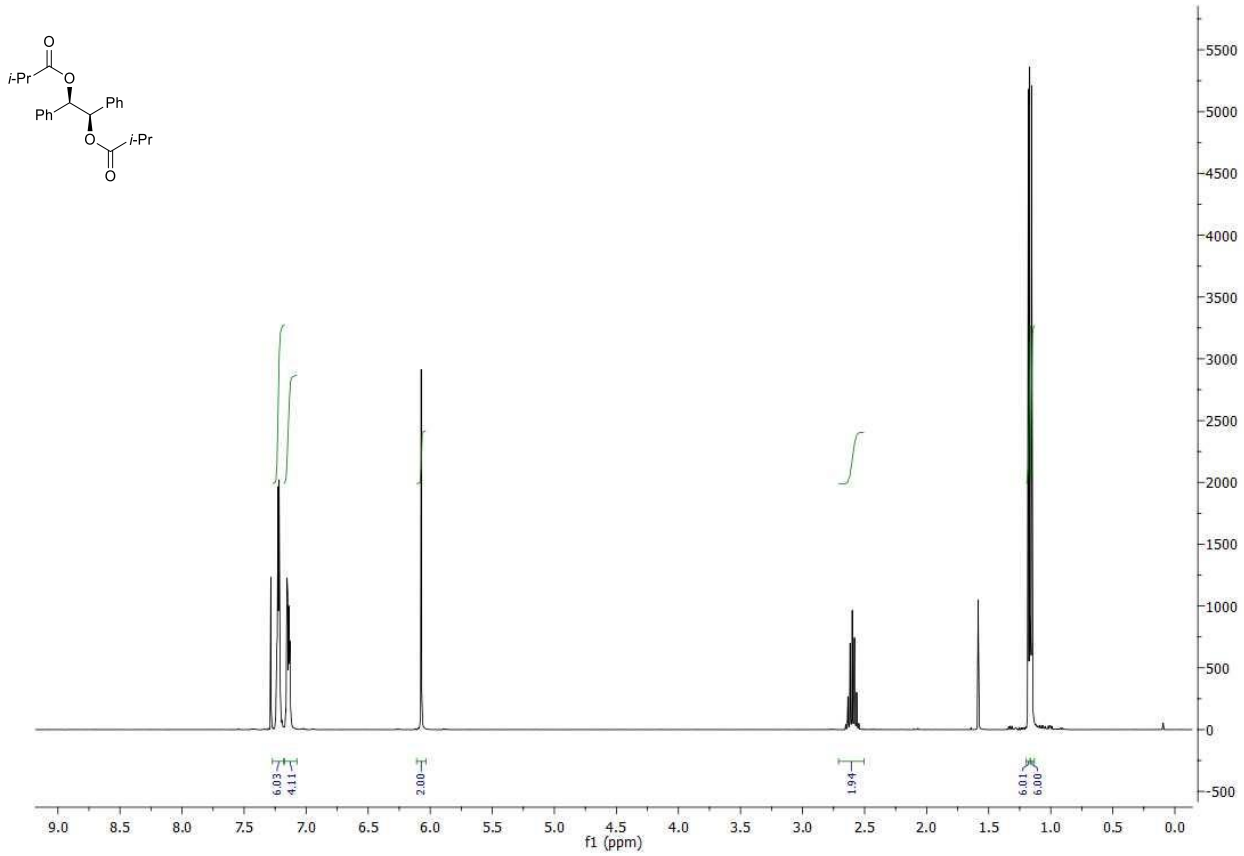
(1*S*,2*S*)-1,2-Diphenylethane-1,2-diol (5a)



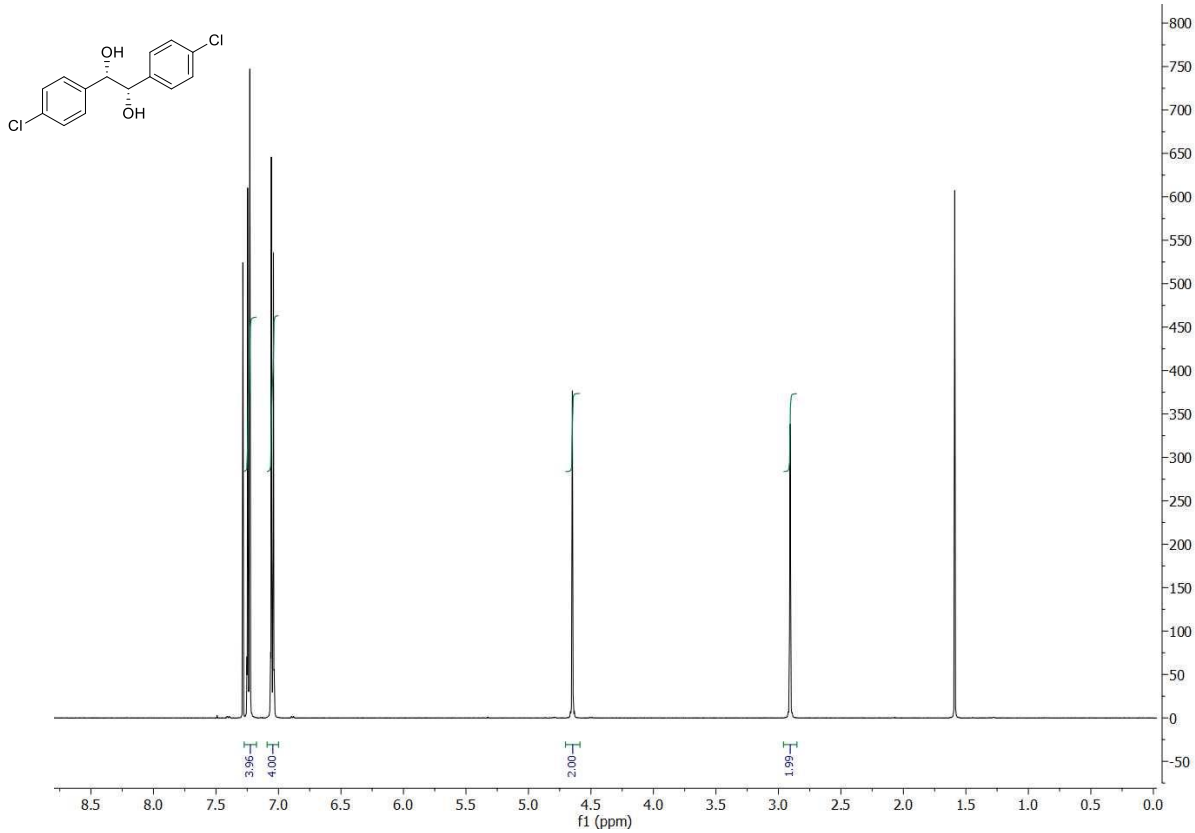
(1*S*,2*S*)-2-Hydroxy-1,2-diphenylethyl isobutyrate (6a)



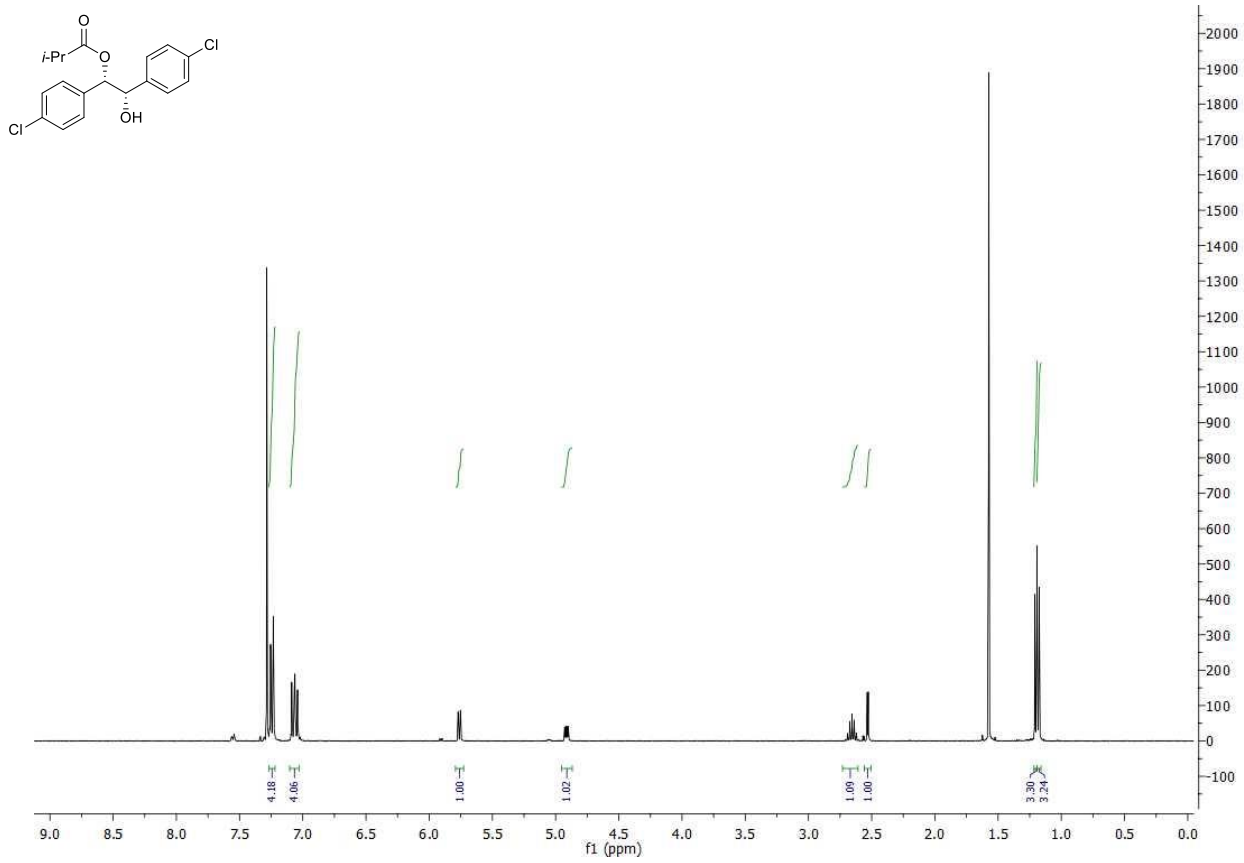
(1*R*,2*R*)-1,2-Diphenylethane-1,2-diyl bis(2-methylpropanoate) (7a)



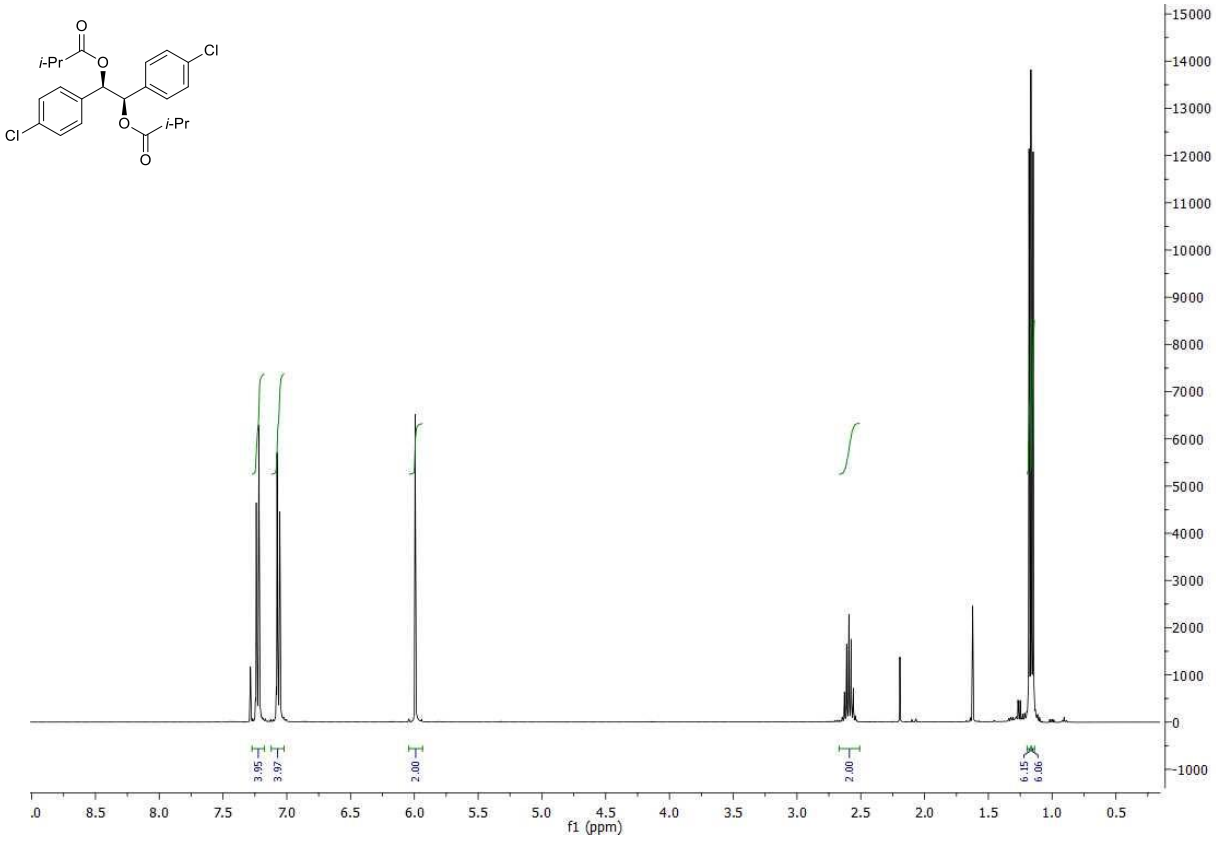
(1*S*,2*S*)-1,2-bis(4-chlorophenyl)ethane-1,2-diol (5b)



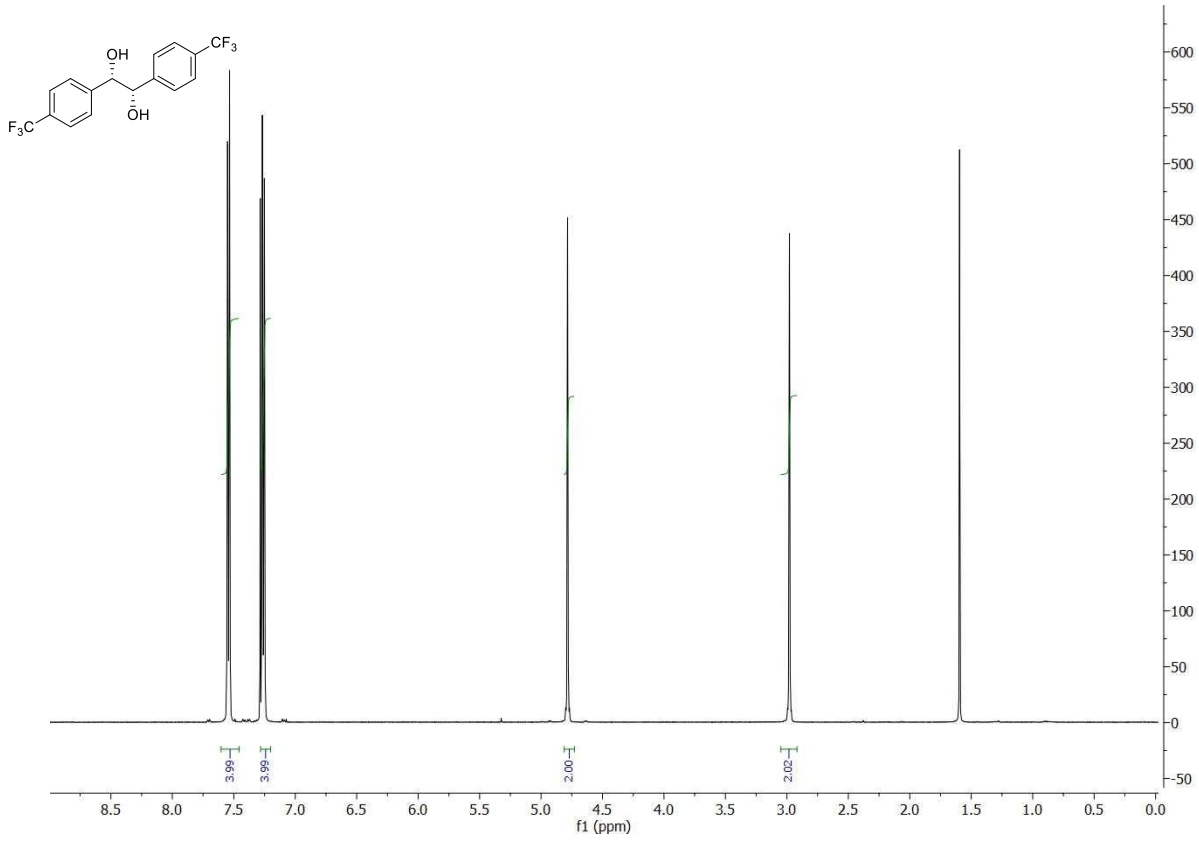
(1*S*,2*S*)-1,2-bis(4-chlorophenyl)-2-hydroxyethyl isobutyrate (6b)

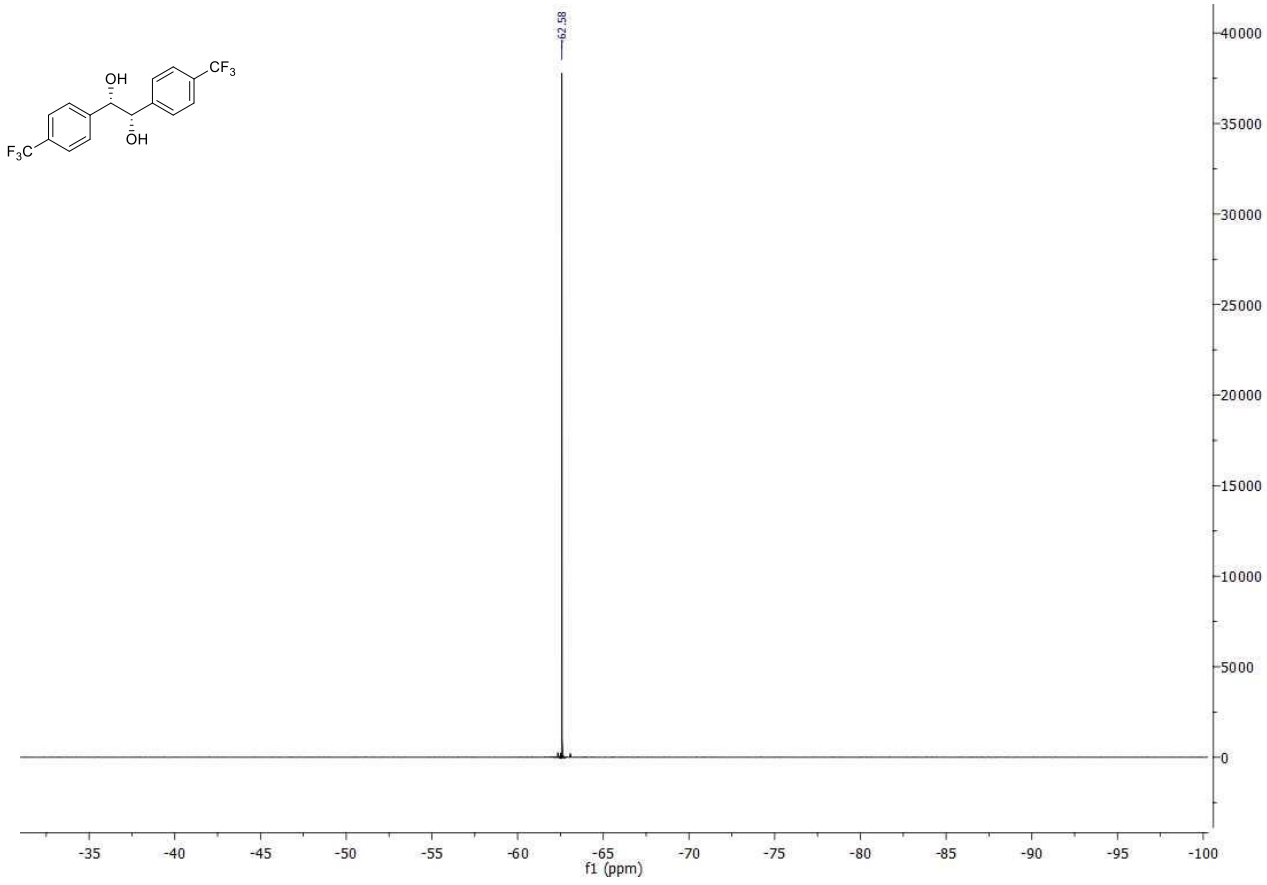


(1*R*,2*R*)-1,2-bis(4-chlorophenyl)ethane-1,2-diyl bis(2-methylpropanoate) (7b)

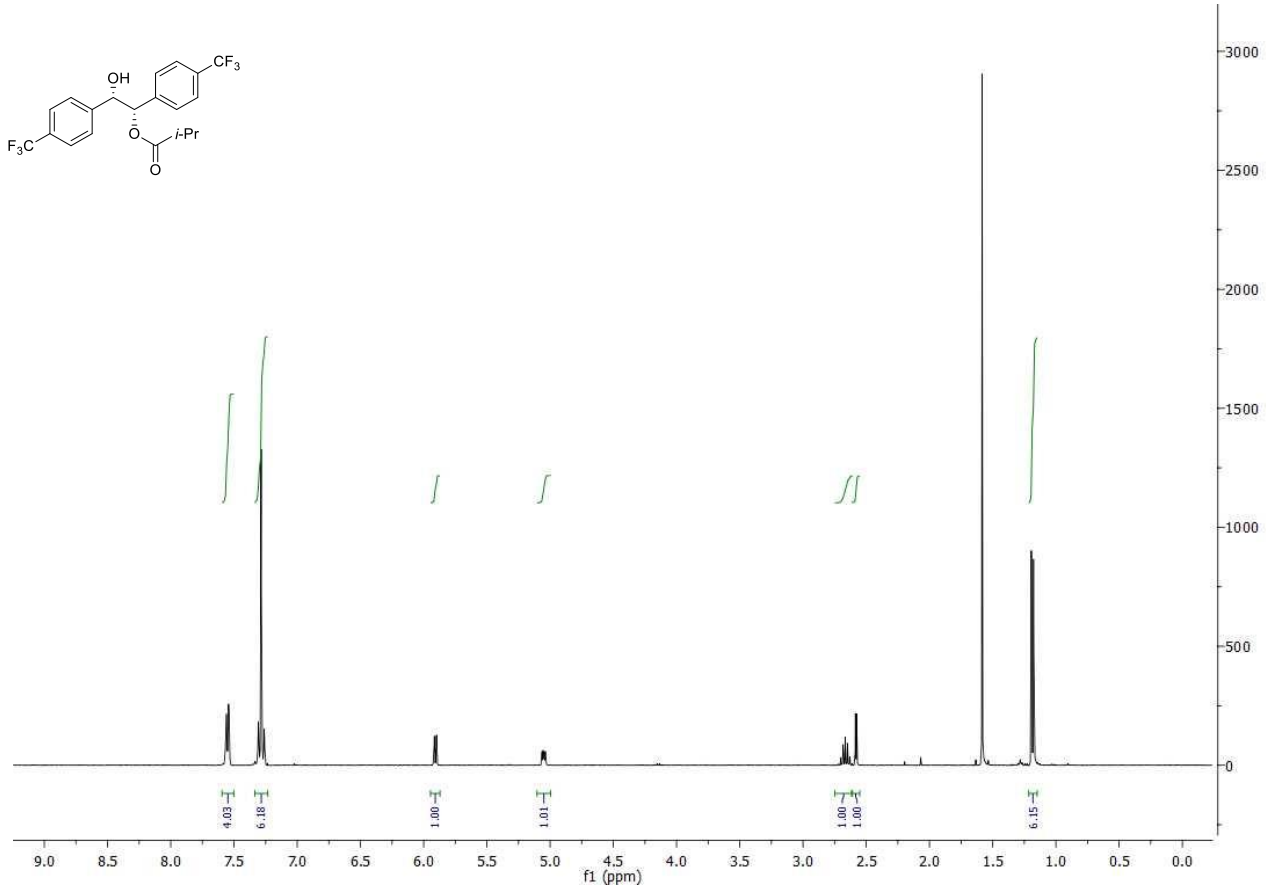


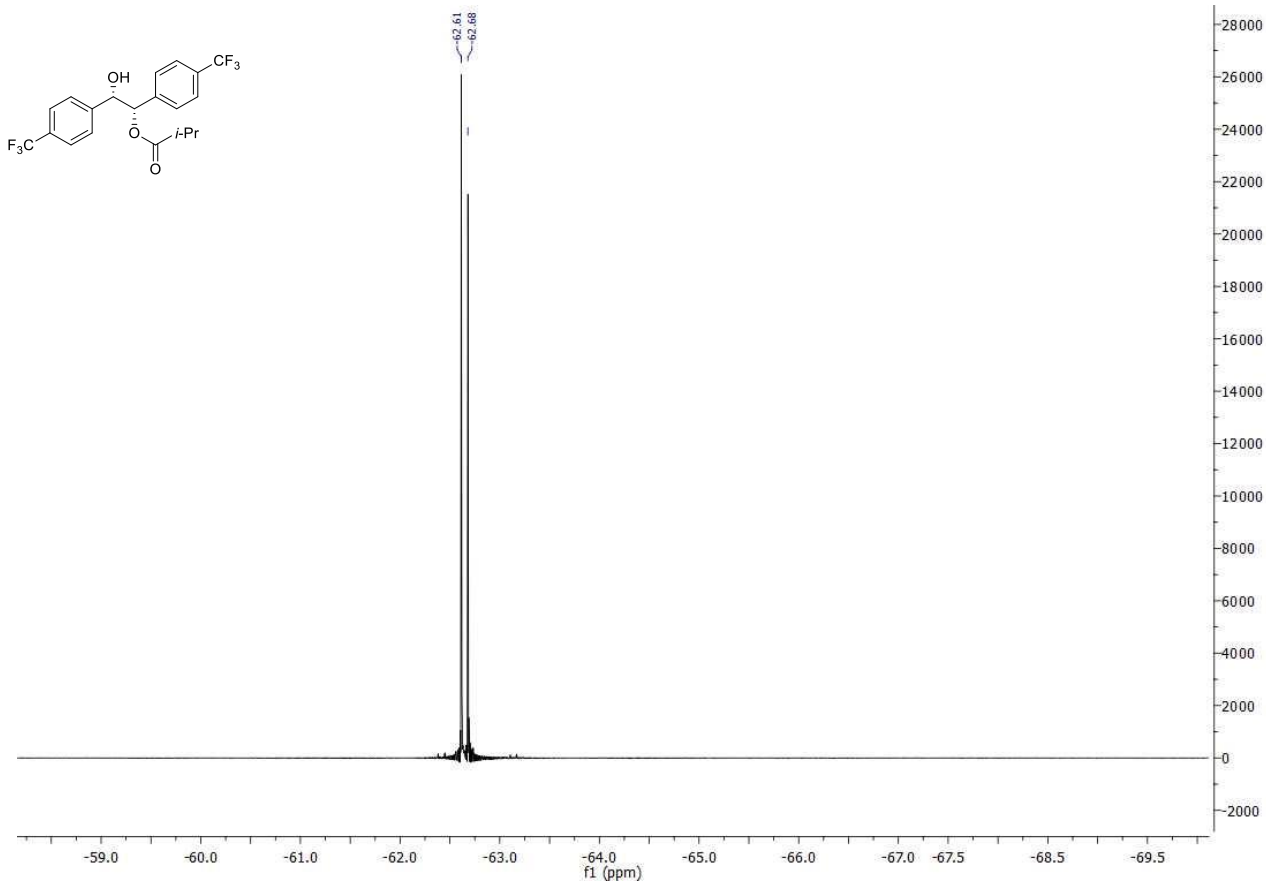
(1*S*,2*S*)-1,2-bis(4-(trifluoromethyl)phenyl)ethane-1,2-diol (5c)



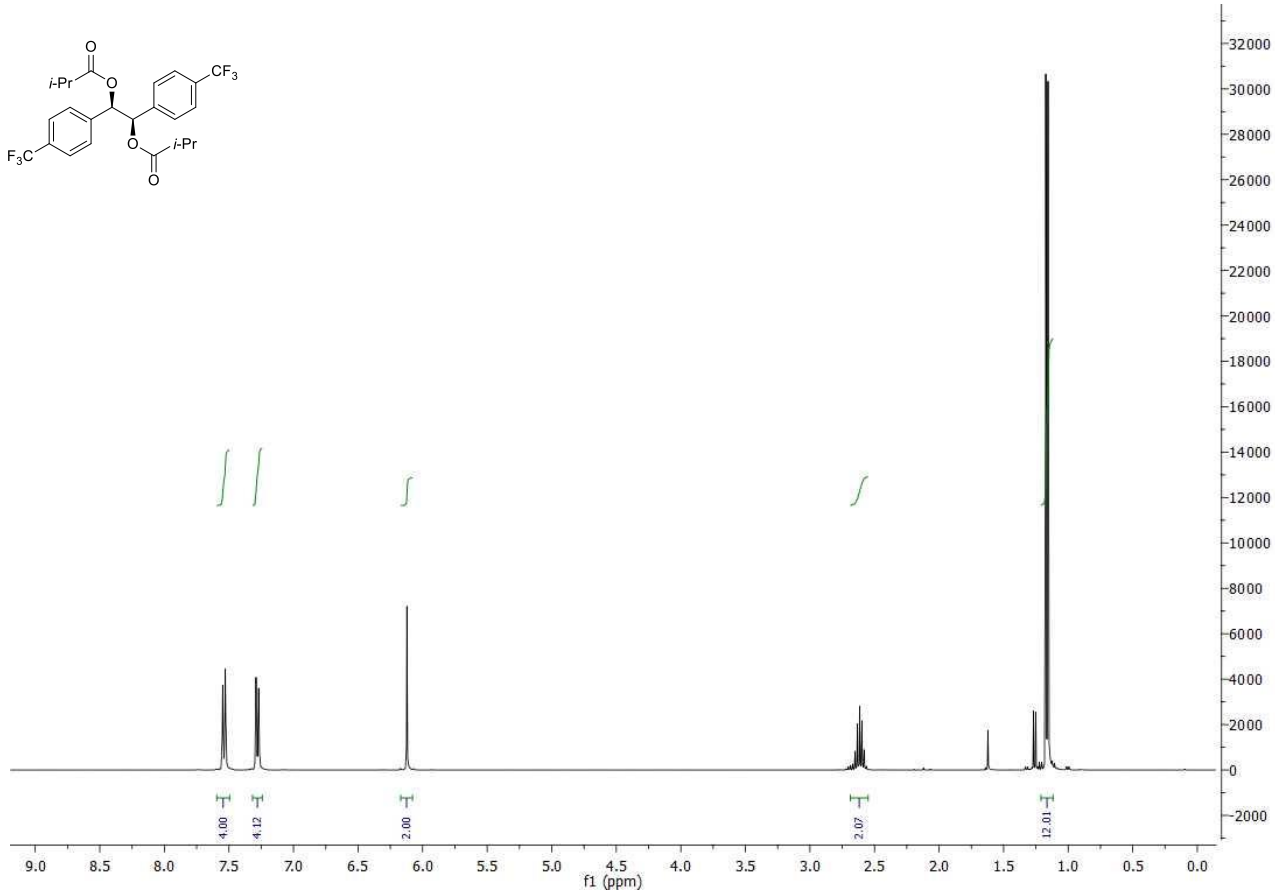


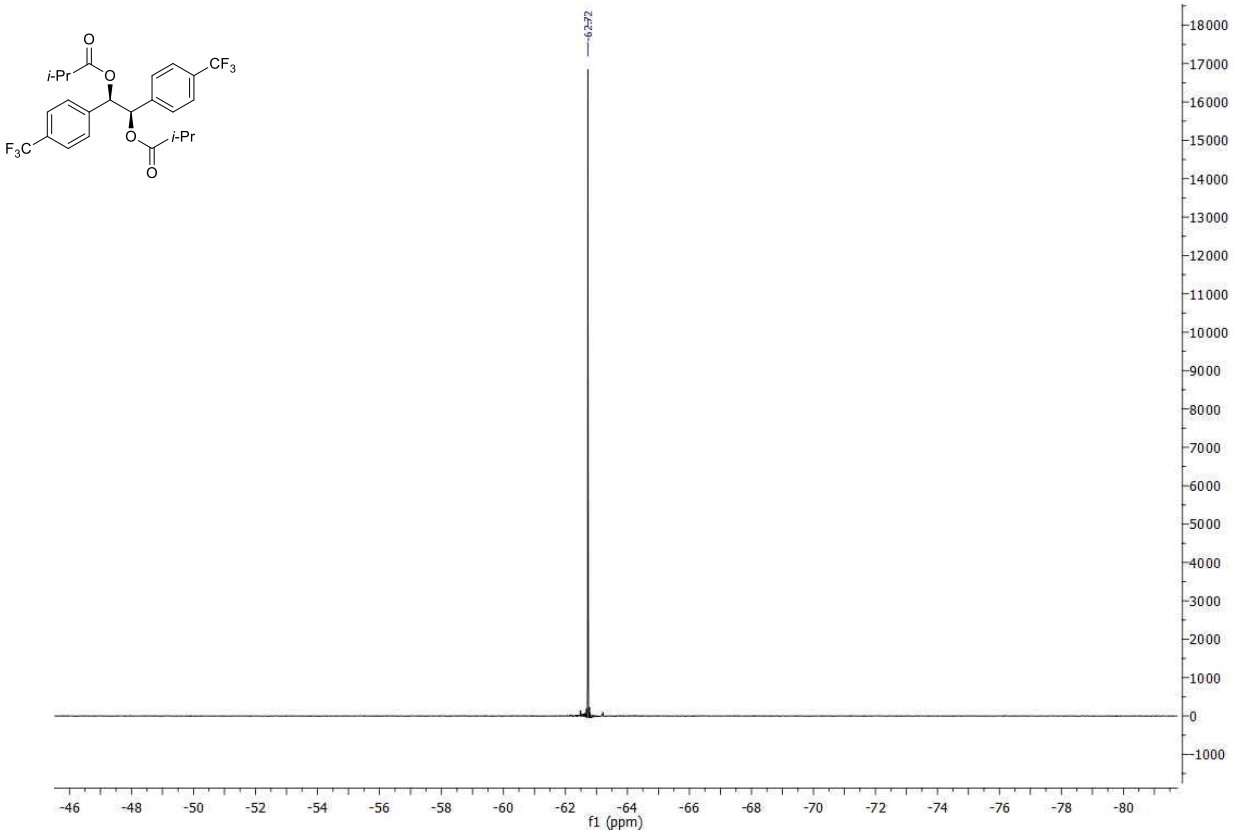
(1*S*,2*S*)-2-hydroxy-1,2-bis(4-(trifluoromethyl)phenyl)ethyl isobutyrate (6c)



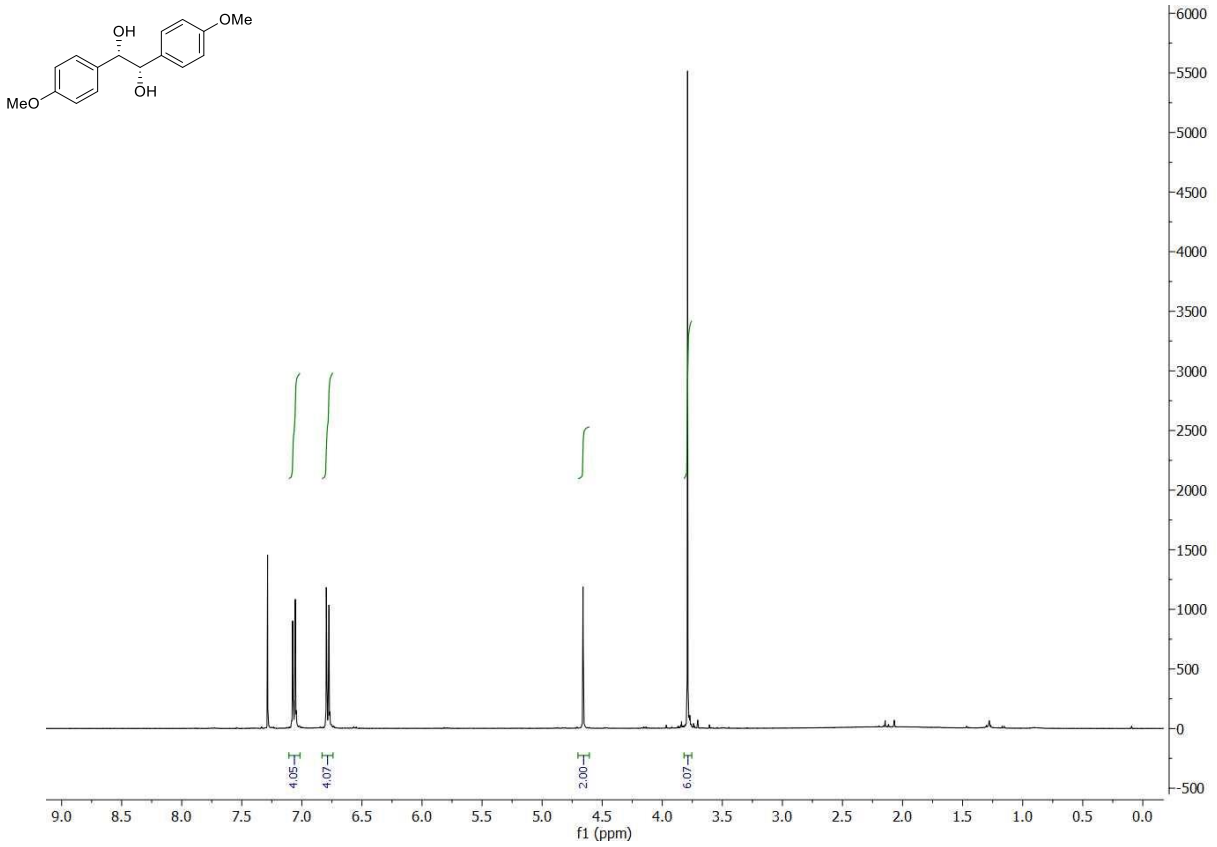


(1*R*,2*R*)-1,2-bis(4-(trifluoromethyl)phenyl)ethane-1,2-diyl bis(2-methylpropanoate) (7c)

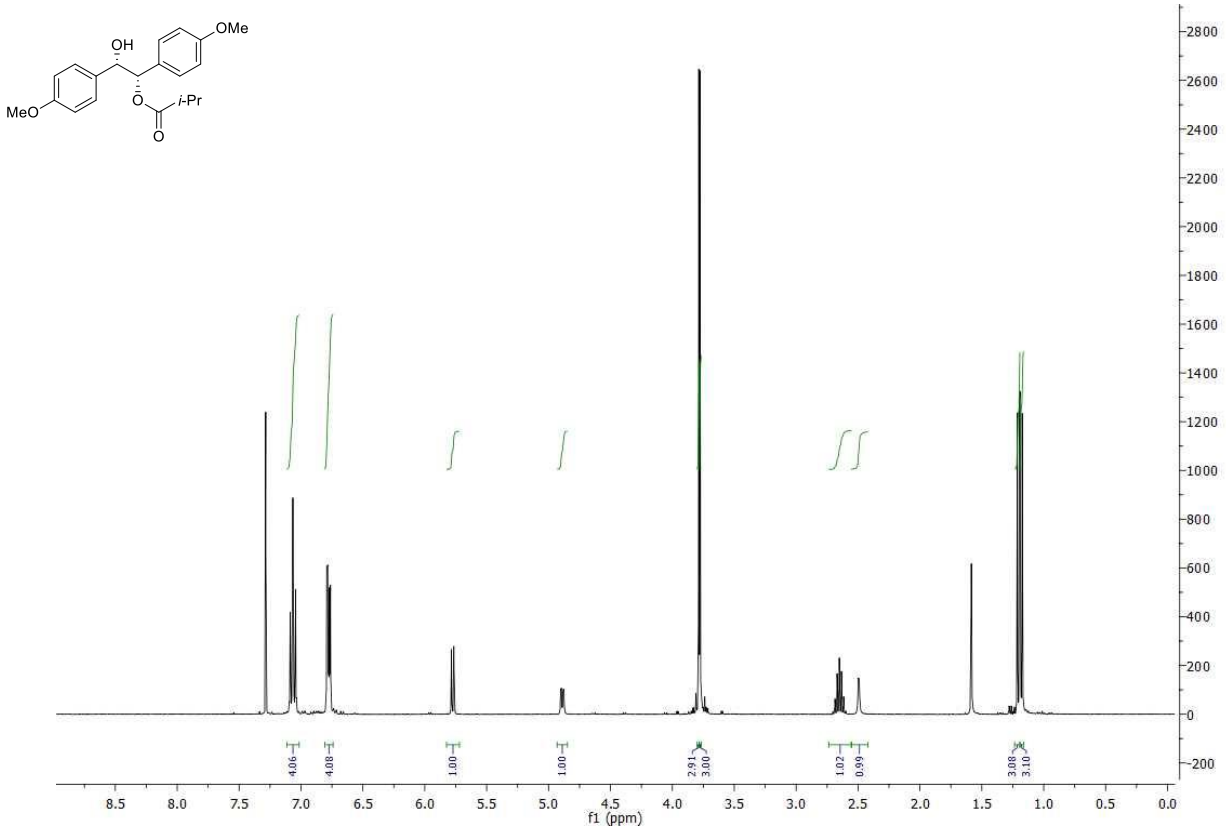




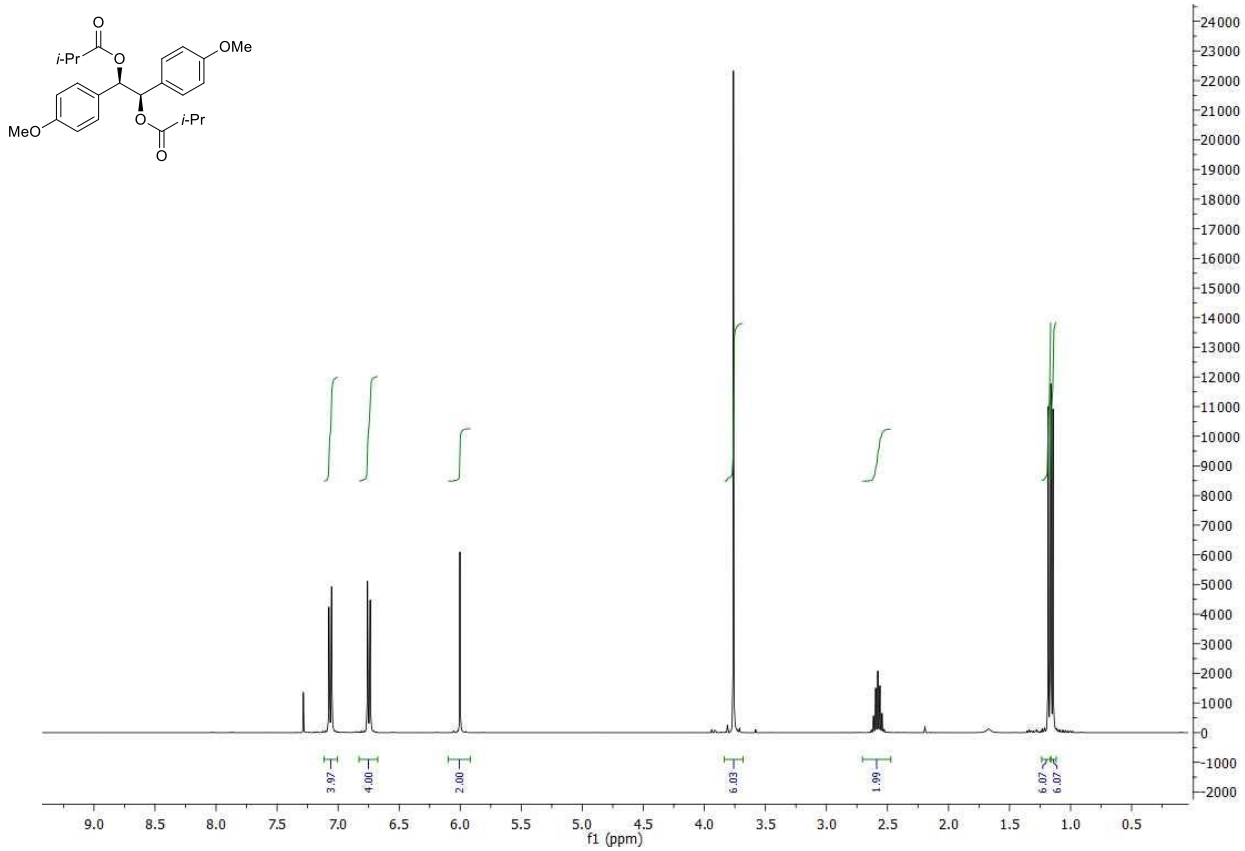
(1*S*,2*S*)-1,2-bis(4-methoxyphenyl)ethane-1,2-diol (5d)



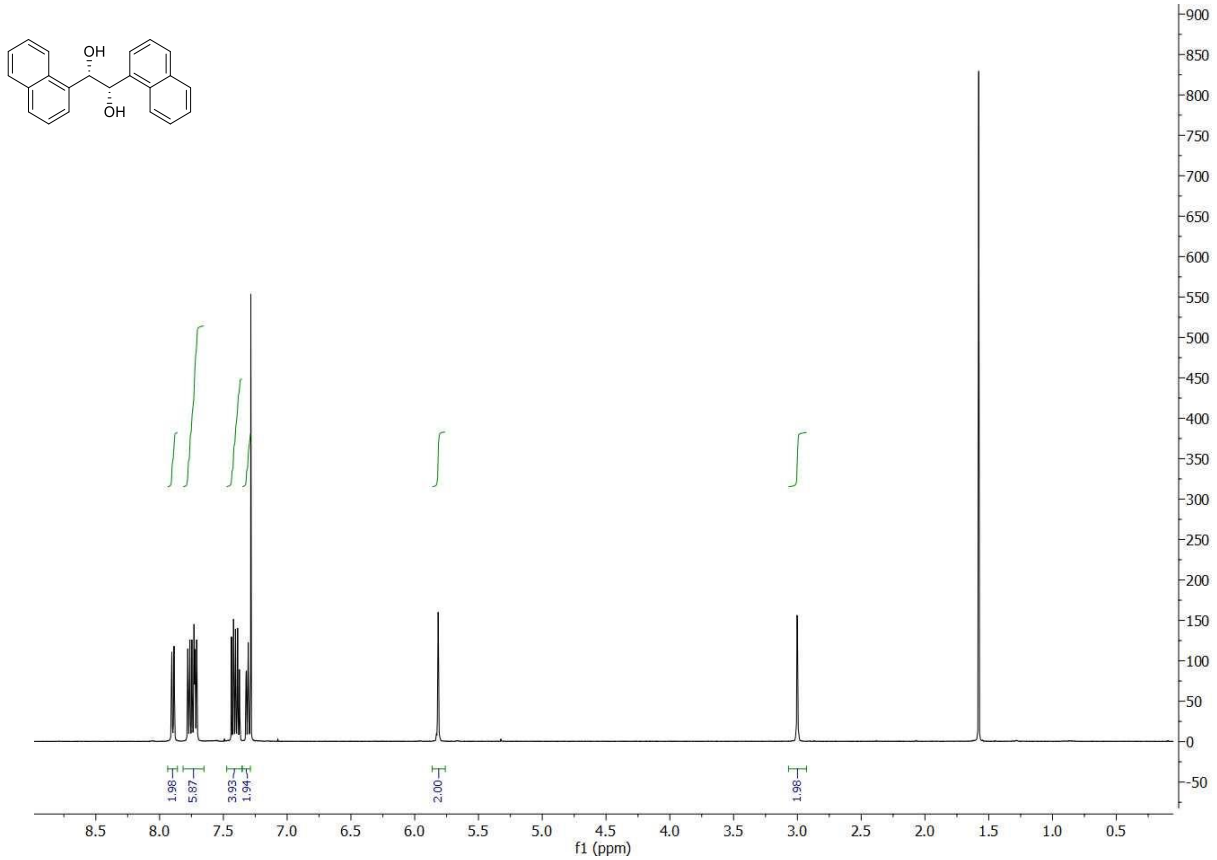
(1*S*,2*S*)-2-hydroxy-1,2-bis(4-methoxyphenyl)ethyl isobutyrate (6d)



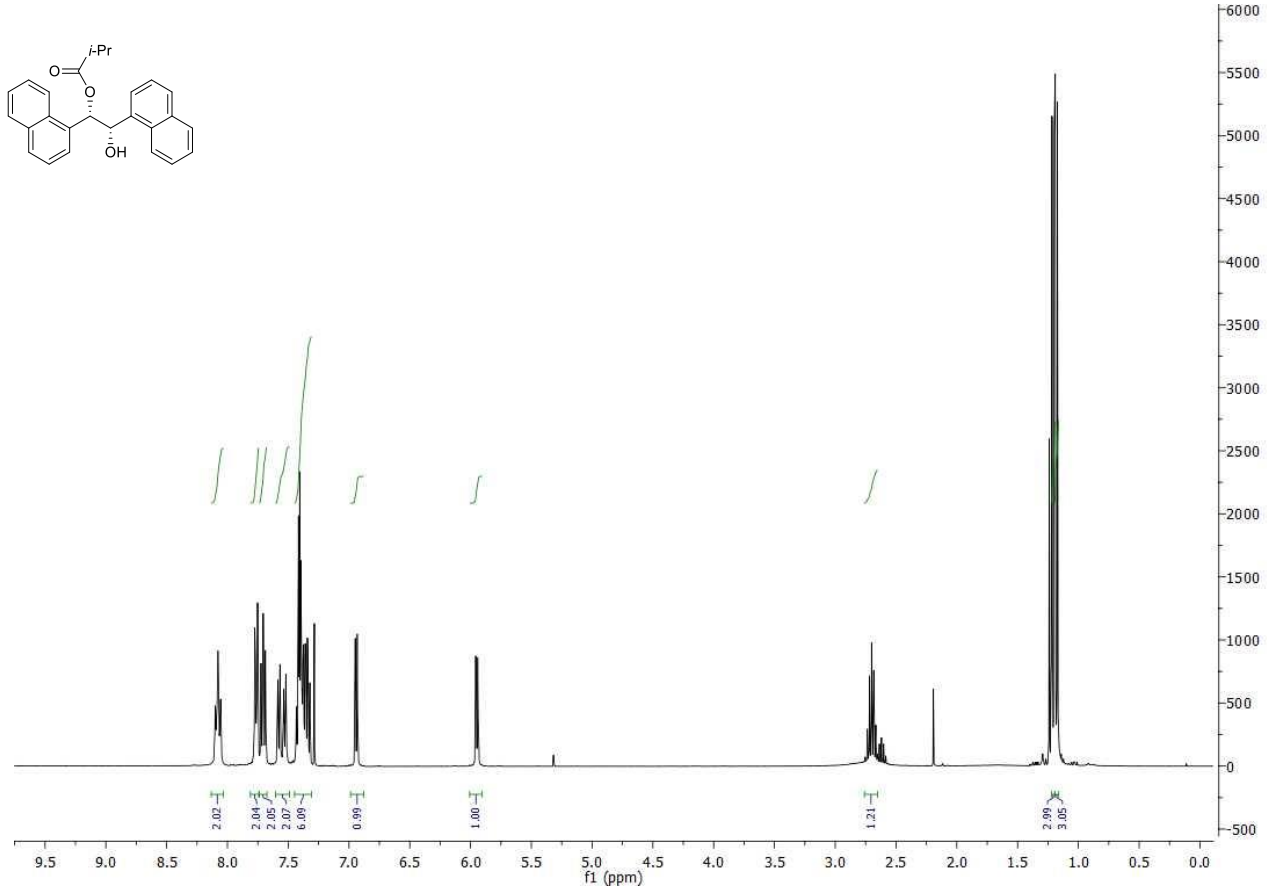
(1*R*,2*R*)-1,2-bis(4-methoxyphenyl)ethane-1,2-diyl bis(2-methylpropanoate) (7d)



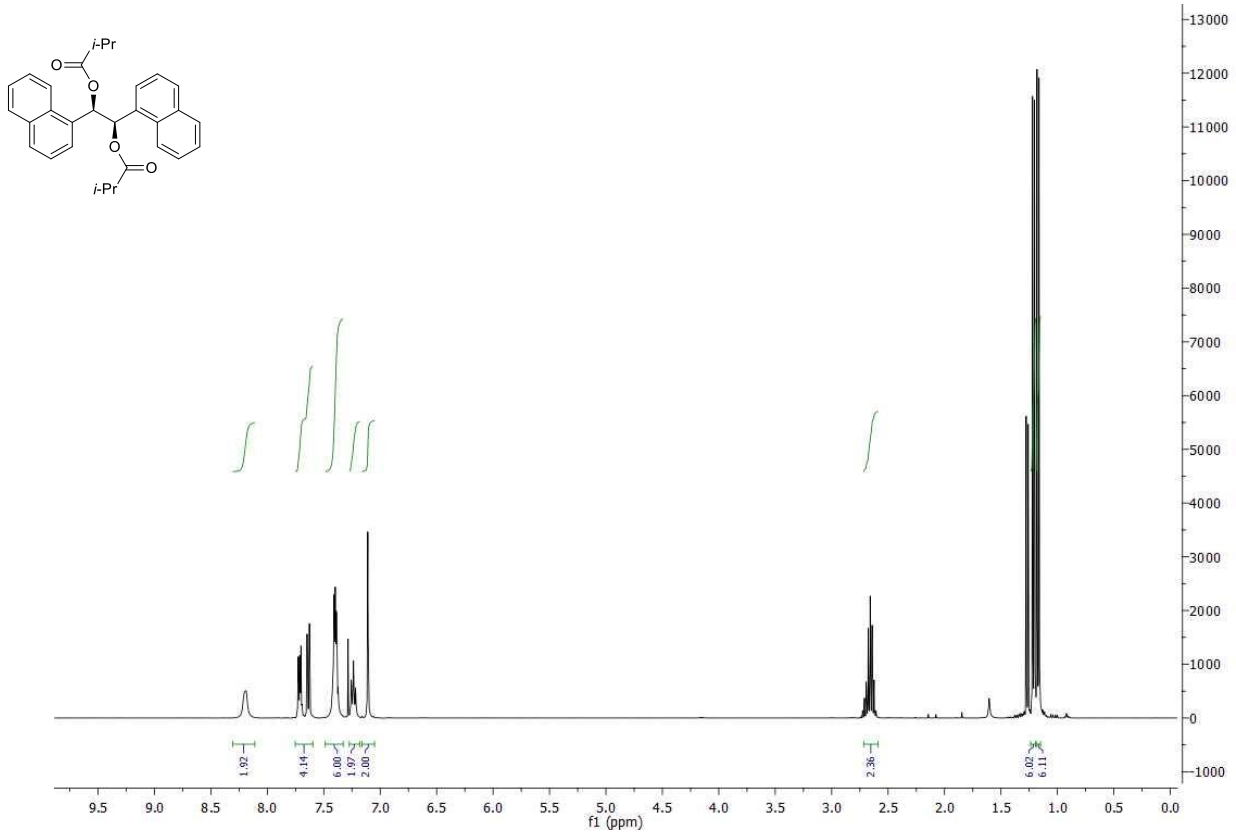
(1*S*,2*S*)-1,2-di(naphthalen-1-yl)ethane-1,2-diol (5e)



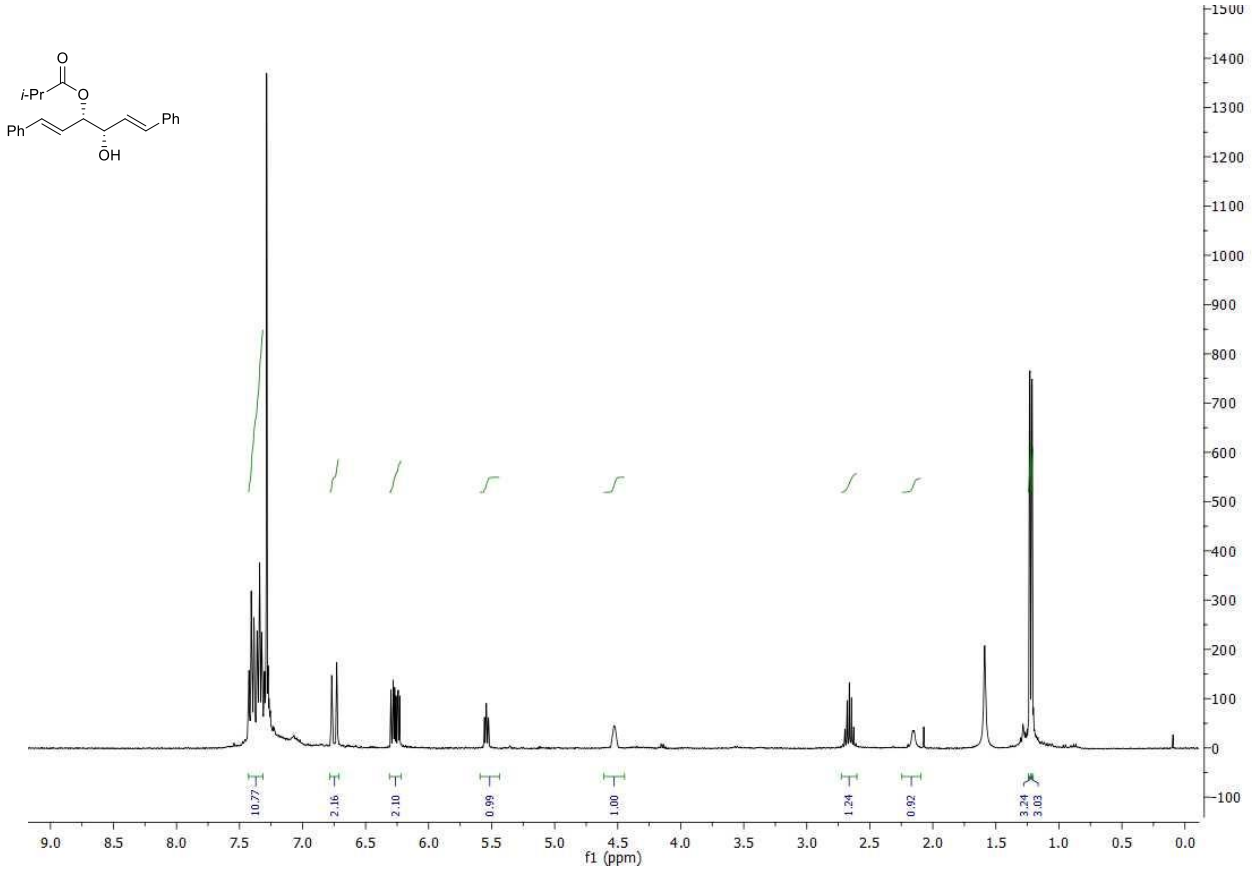
(1*S*,2*S*)-2-hydroxy-1,2-di(naphthalen-1-yl)ethyl isobutyrate (6e)



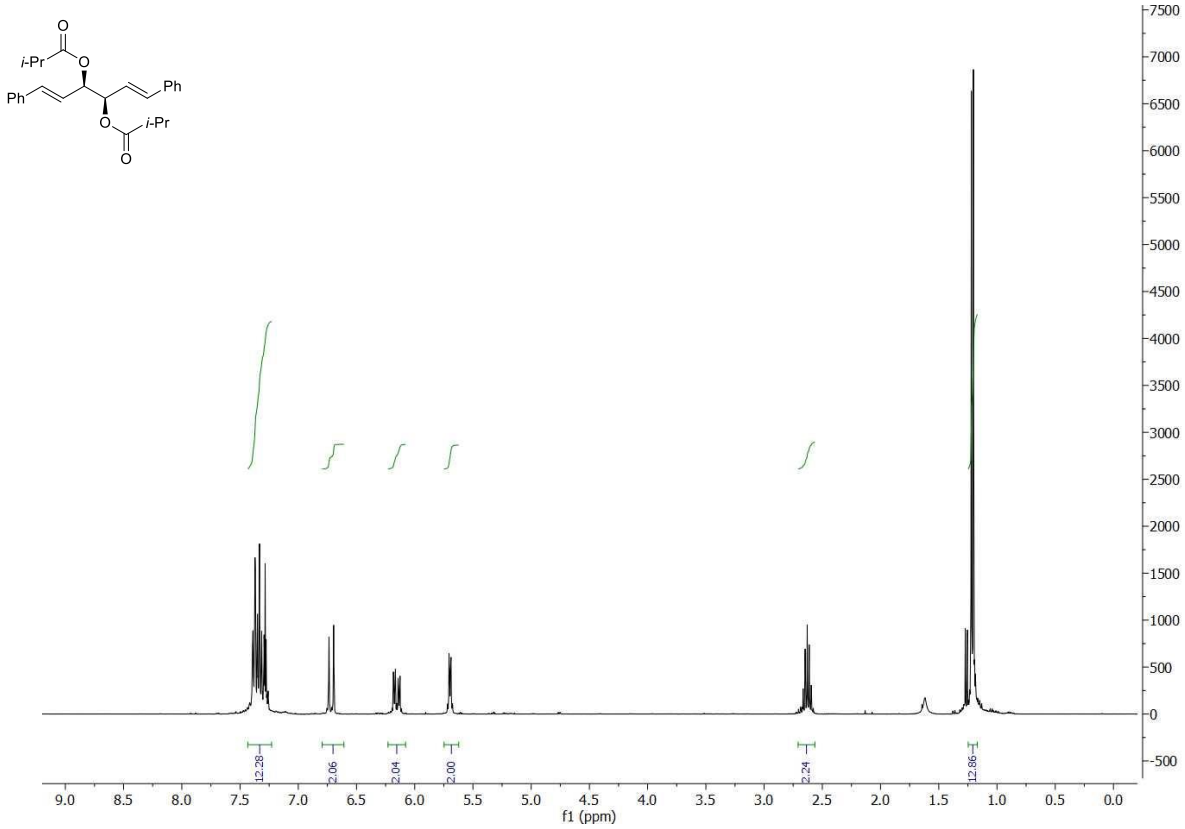
(1*R*,2*R*)-1,2-di(naphthalen-1-yl)ethane-1,2-diyl bis(2-methylpropanoate) (7e)



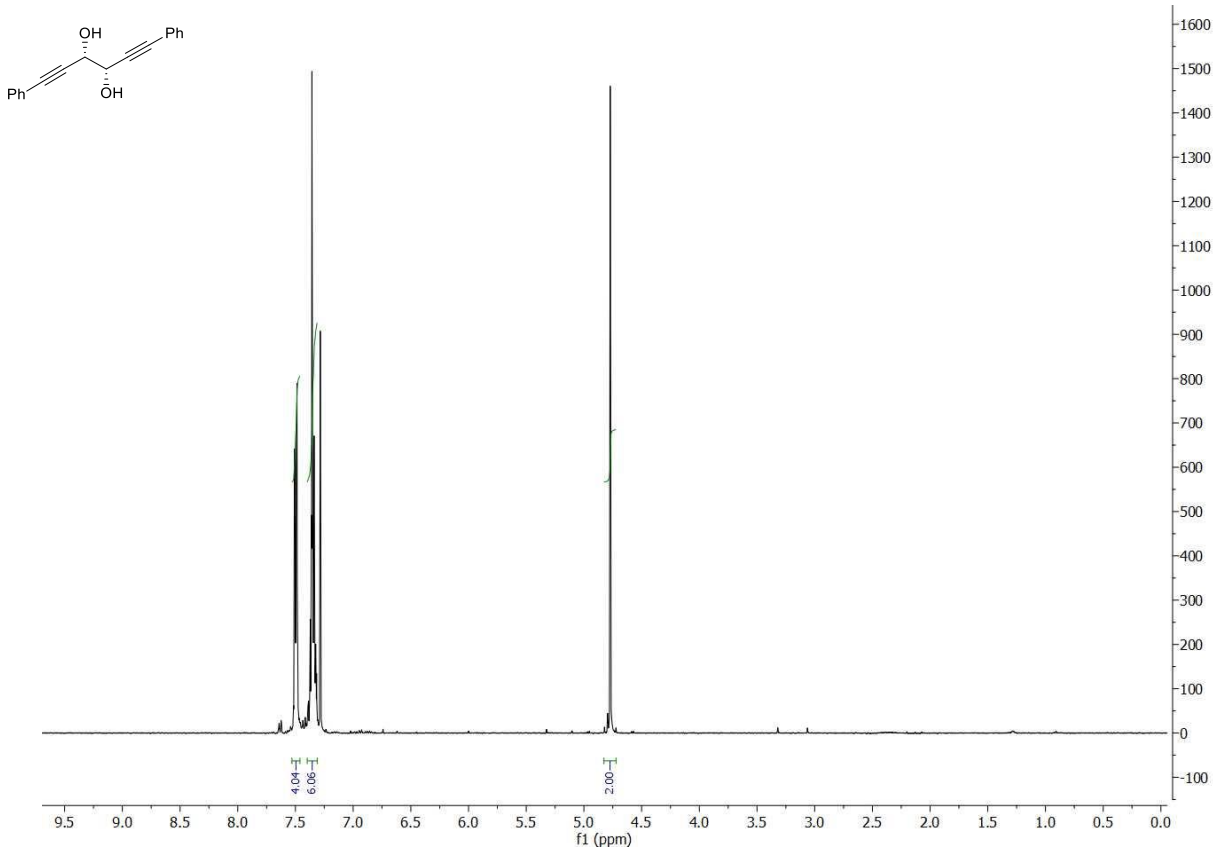
(1*E*,3*S*,4*S*,5*E*)-4-hydroxy-1,6-diphenylhexa-1,5-dien-3-yl isobutyrate (6f)



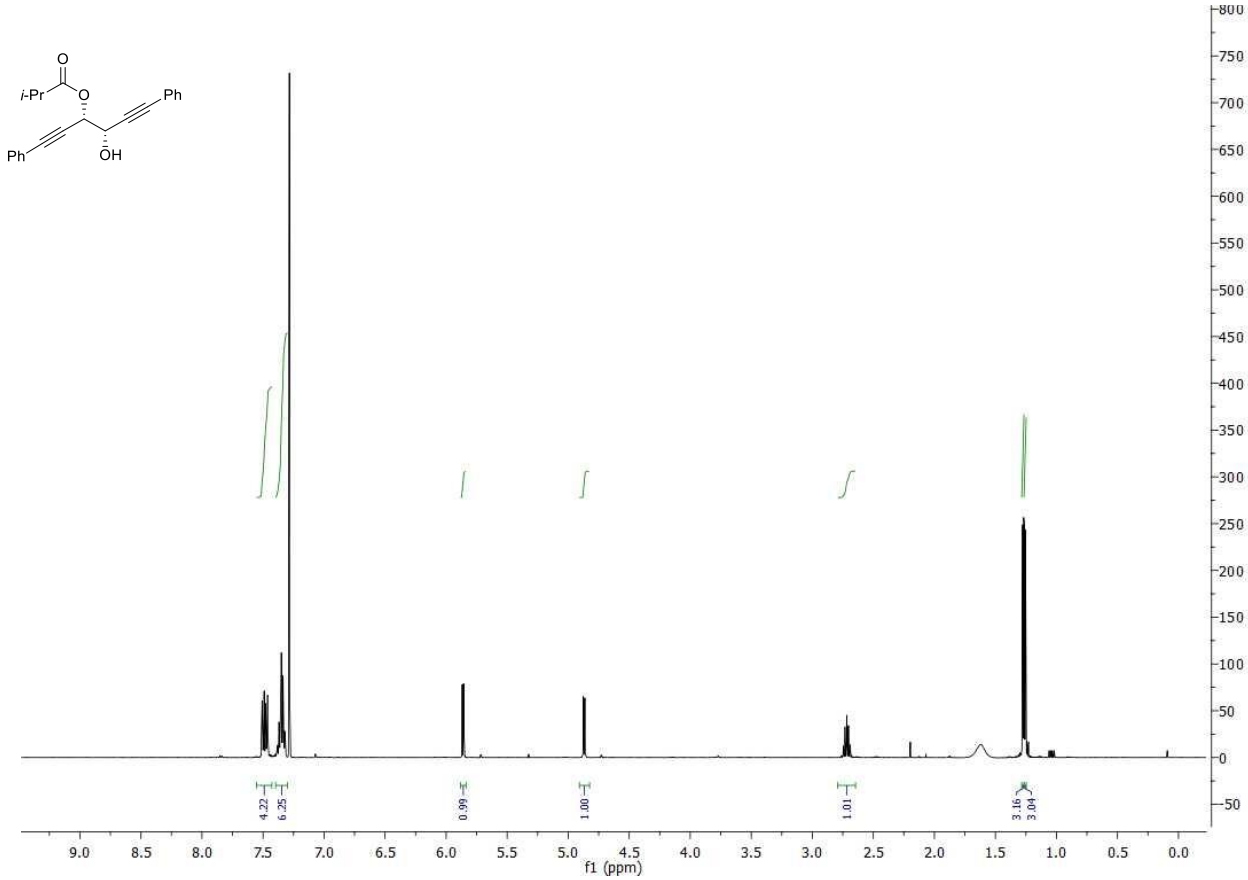
(1*E*,3*R*,4*R*,5*E*)-1,6-diphenylhexa-1,5-diene-3,4-diyl bis(2-methylpropanoate) (7f)



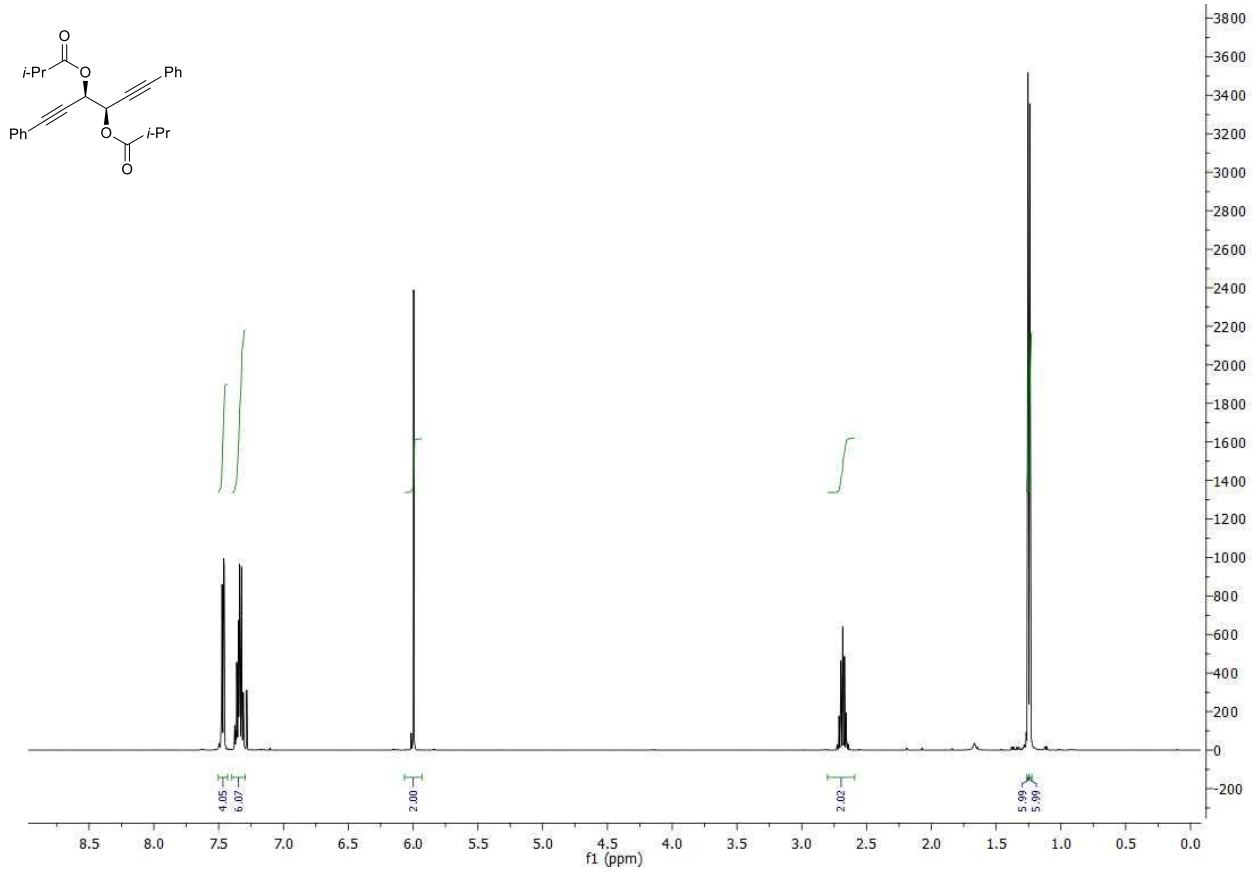
(3*S*,4*S*)-1,6-diphenylhexa-1,5-diyne-3,4-diol (5g)



(3*S*,4*S*)-4-hydroxy-1,6-diphenylhexa-1,5-diyn-3-yl isobutyrate (6g)



(3*R*,4*R*)-1,6-diphenylhexa-1,5-diyne-3,4-diyl bis(2-methylpropanoate) (7g)



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