Asymmetric Methallylation of Ketones Catalyzed by a Highly Active Organocatalyst 3,3'-F_2-BINOL

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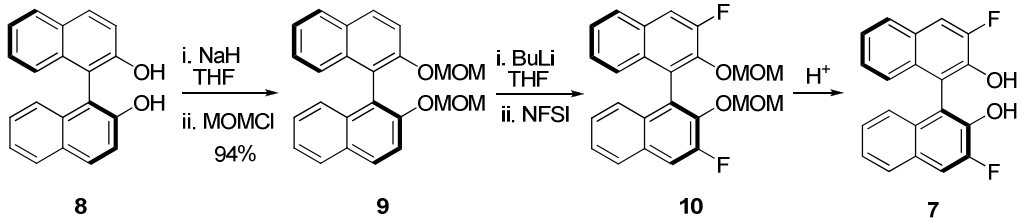
General Methods.

All reactions were carried out under an atmosphere of argon or nitrogen in dry glassware with magnetic stirring. Commercially available *n*-BuLi, MOMCl, THF, NFSI (N-fluorobenzenesulfonimide), anhydrous *t*-BuOH and *t*-AmOH were received and used without any further purification. Ketones **11j**,[1] **11k**[1], **11p**[2] were synthesized according to literature procedure. Column chromatography was performed on silica gel 60 (230-400 mesh). Thin layer chromatography was performed on 0.25 mm silica gel 60-F plates. Visualization was accomplished with UV light, $KMnO_4$, aqueous ceric ammonium molybdate, or bromocresol green dips followed by heating. The stereochemistry of methallylation products were assigned by comparison with optical rotation of known compounds such as **12*o***.

^{1}H NMR and spectra were recorded on a 400 MHz spectrometer at ambient temperature. Data are reported as follows: chemical shift in parts per million (δ, ppm) from an internal standard [tetramethylsilane (TMS) or deuterated chloroform ($CDCl_3$)], multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, br = broad), integration, and coupling constant (Hz). ^{13}C NMR and spectra were recorded on a 400 MHz spectrometer at ambient temperature. Chemical shifts are reported in ppm from $CDCl_3$ taken as 77.0 ppm. Melting points are uncorrected. Optical rotations were measured on an Autopd® III automatic polarimeter.

Synthesis of (*S*)-3,3'-difluoro-1,1'-binaphthyl-2,2'-diol 7[3]



8 **9** **10** **7**

A dry and clean flask was charged with 60% NaH (10.48 g, 261.94 mmol, 2.5 equiv) under nitrogen followed by addition of 100 mL of THF. After the resulting suspension was cooled to 0 ^{o}C, a solution of (*S*)-1,1'-binaphthyl-2,2'-diol **8** (30 g, 104.78 mmol) in 200 mL of THF was added while maintaining the internal temperature below 5 ^{o}C. After 1 h, MOMCl (20.25 g, 251.46 mmol, 2.4 equiv) was added while maintaining the internal temperature below 10 ^{o}C. After the solution was stirred for 1h at 0-5 ^{o}C, the reaction was quenched with water and extracted with EtOAc. The combined organic layers were washed with brine, dried over anhydrous Na_2SO_4. Removal of the solvent gave the product as a pale yellow solid, which was slurried in a mixture solvent (5% EtOAc in Hexane) to give 37 g of the product **9** as an off-white solid with 94% yield.

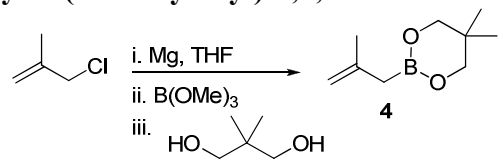
To a dry 500-mL 3-neck flask was charged **9** (10 g, 26.71 mmol) under nitrogen followed by addition of 100 mL of THF via a syringe. After the resulting solution was cooled to -15 ^{o}C, a solution of 2.5 M n-BuLi (25.64 ml, 64.10 mmol, 2.4 equiv) in hexane was added while maintaining the internal temperature below -10 ^{o}C. After complete addition, the mixture was kept at 0 ^{o}C for 1h to give a thick suspension. The suspension was cooled to -70 ^{o}C. NFSI (21.03 g, 64.68 mmol, 2.42 equiv) in 100 mL of THF was added while maintaining the internal temperature < -69 ^{o}C. After complete addition, the resulting mixture was kept < -69 ^{o}C for 30 min. Then the mixture was allowed to warm up to room temperature. Water was added to quench the reaction followed by addition of EtOAc. The combined organic layers were washed with aq. $NaHCO_3$, water and brine successively, and dried over anhydrous Na_2SO_4. Removal of the solvent under vacuum gave the crude product, which was purified by silica gel chromatography to give the product **10** as a liquid with 91% yield.

To a flask was charged **10** (6.0 g) and Amberlyst® ion-exchange resin 15 (6.0 g). After addition of 30 mL of THF and 30 mL of MeOH, the resulting mixture was heated at reflux for 3 h. After the mixture was cooled to room temperature, the solid was removed

by filtration. The crude product on removal of the solvent was purified on silica gel to give the pure product **7** as a white solid with 93% yield and > 99% ee.

$[\alpha]^{22}_{D}$ +22.9 [*c* 0.66, $CHCl_3$]; Melting point: 177-179 ^{o}C; ^{1}H NMR (600.02 MHz, DMSO-d_6), δ 9.57 (s, 2H), 7.89(d, *J* = 8.4 Hz, 2H), 7.85 (d, *J* = 12 Hz, 2H), 7.33 (t, *J* = 7.8 Hz, 2H), 7.20 (t, *J* = 7.8 Hz, 2H), 6.91 (d, J = 8.4 Hz, 2H); ^{13}C-NMR (150.92 MHz, DMSO-d_6) δ, 152.1, 143.1, 130.6, 127.6, 127.4, 125.5, 124.2, 123.8, 118.0, 111.7; ^{19}F NMR ($CDCl_3$), δ -135.1 (m, 1F). HRMS (ES pos.): m/z calcd for $C_{20}H_{13}F_2O_2^+$ (M + H^+): 323.0878, found: 323.0871; Chiral HPLC conditions: Chiralpak OD-H, 4.6 x 250 mm; 75:25 heptane/2-propanol, 1.2 mL/min, 220 nm, (*R*)-**7**, *t* = 5.89 min, (*S*)-**7**, *t* = 8.59 min.

Synthesis of 5,5-Dimethyl-2-(2-methylallyl)-1,3,2-dioxaborinane 4



To a dry flask was charged Mg turnings (12.0 g, 497 mmol) and THF (350 mL) under argon followed by addition of 1.2 M DIBAL-H in heptane (8.28 mL, 0.03 equiv). The solution was left for 1 h and gradually became grey. 3-chloro-2-methylprop-1-ene (30 g, 331 mmol) was added to the flask while maintaining the temperature between 20-25 ^{o}C. After the complete addition, the mixture was stirred for 2 h at that temperature. The solution was titrated with bi-pyridine as indicator.[4] The concentration is about 0.62 M.

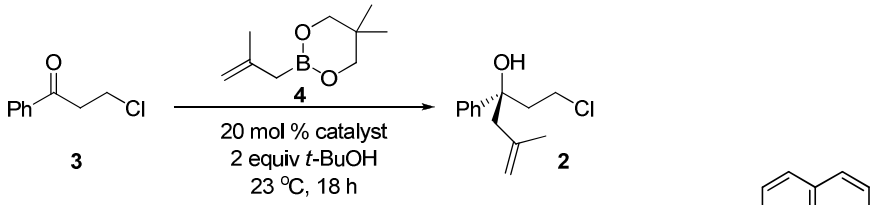
To a dry flask was charged the Grignard reagent (248 mmol, 400 mL) prepared above and MTBE (200 mL) under nitrogen. After the mixture was cooled to -60 ^{o}C (dry-ice bath), $B(OMe)_3$ (25.8 g, 248 mmol, 1 equiv) was added while maintaining the internal temperature below -60 ^{o}C. The resulting solution was stirred at -50 to -60 ^{o}C for 30 min, and then slowly warm to 0 ^{o}C over 30 min. AcCl (17.5 g, 223.2 mmol, 0.9 equiv) was charged while controlling the temperature below 5 ^{o}C. After that, the reaction mixture was allowed to warm to room temperature and then 3,3-dimethyl-propane-1,3-diol (23.0 g, 223 mmol, 0.9 equiv) was charged into the flask in one portion. The resulting reaction mixture was stirred at 23 ^{o}C overnight. Dioxane (32.78 g, 372 mmol, 1.5 equiv) was added to the mixture. After 2 h, the solid was filtered off through a Celite pad, which was rinsed with MTBE (200 mL). The residue on removal of the solvent was diluted with hexane (100 mL). The solid was removed by filtration. The filtrate was concentrated and

purified by passing a short silica gel column with 10% MTBE in hexanes as elute or by distillation under vacuum to give the boronate **4**.

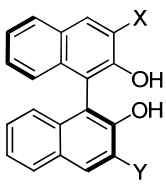
^{1}H NMR (400 MHz, $CDCl_3$): δ 4.66 (s, 1H), 4.62 (s, 1H), 3.61(s, 4H), 1.77 (s, 3H), 1.67 (s, 2H), 0.96 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 144.1, 109.4, 72.2, 31.6, 24.5, 21.8. HRMS (ES pos.): m/z calcd for $C_9H_{18}BO_2^+$ (M + H^+): 169.1400, found: 169.1371.

Screen results of various substituted BINOLs.

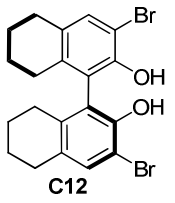
To a dry and clean flask was charged ketone **3** (2 mmol) and substituted BINOL **C1-C16** (0.20 equiv) under N_2 followed by addition of *t*-BuOH (0.30 g, 4.0 mmol, 2 equiv) and **4** (3 mmol, 1.5 equiv) via syringe. Then the mixture was stirred at 23 ^{o}C overnight. The er was determined on chiral HPLC. Chiral HPLC conditions: Chiralcel OJ-RH, 4.6 x 150 mm, 5 μm, mobile phase A (acetonitrile), mobile phase B (0.1% HCO_2H in water, adjusted with NH_4OH to pH = 4.0), 52:48 A/B, λ = 220 nm, flow rate: 1.3 mL min^{-1}. The samples for HPLC were diluted with MeOH.



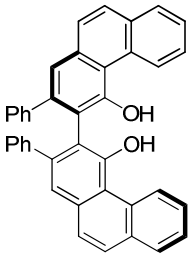
entry	diol	er
1	**8**	51:49
2	**C1**	68:32
3	**C2**	80:20
4	**C3**	60:40
5	**C4**	60:40
6*	**C5**	80:20
7*	**C6**	80:20
8	**C7**	50:50
9	**C8**	50:50
10	**C9**	50:50
11	**C10**	61:39
12	**C11**	50:50
13	**C12**	54:46
14	**C13**	56:44
15	**C14**	54:46
16	**C15**	50:50
17	**C16**	50:50



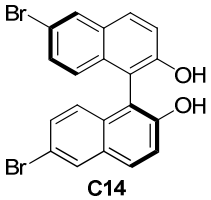
8 X = Y = H
C1 X = Br, Y = H
C2 X = Y = I
C3 X = Y = Me
C4 X = Y = 2-(2-hydroxypropanyl)
C5 X = Y = Ph
C6 X = Y = 3',4',5'-trifluorophenyl
C7 X = Y = $SiPh_3$
C8 X = Y = 2,4,6-trisopropylphenyl
C9 X = Y = 9-anthracenyl
C10 X = SO_2CF_3, Y = H
C11 X = CF_3, Y = H



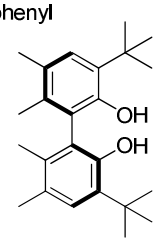
C12



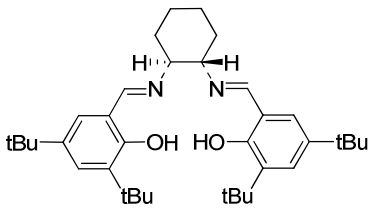
C13



C14



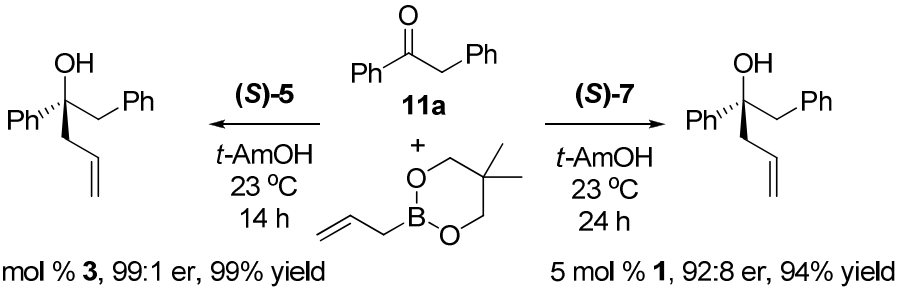
C15



C16

* 3.0 equiv of **4** and 2.0 eqiv of *t*-BuOH were used.

Allylation of 11a catalyzed by 3,3'-Br_2-BINOL 5 and 3,3'-F_2-BINOL 7.



General Procedure for the Asymmetric Methallylation of Ketones with 3,3'-F_2-BINOL 7 as the organocatalyst

To a dry and clean flask was charged ketone (2 mmol) and **7** (12.9 mg, 0.04 mmol, 0.05 equiv) under N_2 followed by addition of *t*-AmOH (0.29 g, 4.0 mmol, 2 equiv) and **4** (3 mmol, 1.5 equiv) via syringe. Then the mixture was stirred at the temperature as indicated in the table 2. The mixture was cooled to room temperature if the reaction was run at 40 ^oC. The reaction mixture was directly purified by flash chromatography (0-5% EtOAc in hexanes) on silica gel to yield the product.

(*R*)-4-Methyl-1,2-diphenylpent-4-en-2-ol 12a

The general procedure was applied to 1,2-diphenylethanone (0.39 g, 2.0 mmol) in the presence of 2 mol % **7**. The crude product was purified by column chromatography on silica gel (hexanes: EtOAc = 5:95) to give the product as an oil (0.49 g, 98 yield, 93:7 er). ^1H NMR (400 MHz, $CDCl_3$): δ 7.35-7.14 (m, 8H), 6.97-6.95 (m, 2H), 4.82 (s, 1H), 4.67 (s, 1H), 3.16 (d, *J* = 13.4 Hz, 1H), 3.06 (d, *J* = 13.4 Hz, 1H), 2.76 (d, *J* = 13.6 Hz, 1H), 2.57 (d, *J* = 13.6 Hz, 1H), 2.26 (s, 1H), 1.37 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 145.8, 142.3, 136.6, 130.8, 127.8, 126.5, 125.7, 115.7, 75.6, 50.1, 49.9, 24.3. HRMS (ES pos.): m/z calcd for $C_{18}H_{19}^+$ ($M + H^+ - H_2O$): 235.1481, found: 235.1476. Optical Rotation: $[\alpha]_D^{22}$ +21.2 (c = 1.13, $CHCl_3$) for an enantiomerically enriched sample of 93:7 er.

Chiral HPLC conditions: Chiralcel OJ-RH, 4.6 x 150 mm, 5 μm, mobile phase A (acetonitrile), mobile phase B (0.1% HCO_2H in water, adjusted with NH_4OH to pH = 4.0), 52:48 A/B, λ = 220 nm, flow rate: 1.3 mL min^{-1}. The samples for HPLC were diluted with MeOH. (*R*)-**12a**, t_{major} = 8.57 min; (*S*)-**12a**, t_{minor} = 8.06 min.

(*R*)-1-(3-Methoxyphenyl)-4-methyl-2-phenylpent-4-en-2-ol 12b

The general procedure was applied to 2-(3-methoxyphenyl)-1-phenylethanone (113 mg, 0.5 mmol). The crude product was purified by column chromatography on silica gel (hexanes: EtOAc = 5:95) to give the product (134 mg, 95% yield, 96:4 er). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.37-7.26 (m, 4H), 7.23-7.17 (m, 1H), 7.09 (t, 1H, *J* = 7.9 Hz), 6.73-6.68 (m, 1H), 6.59 (d, 1H, *J* = 7.4 Hz), 6.43 (bs, 1H), 4.82 (bs, 1H), 4.68 (s, 1H), 3.63 (s, 3H), 3.15 (d, 1H, *J* = 13.4 Hz), 3.03 (d, 1H, *J* = 13.4 Hz), 2.76 (d, 1H, *J* = 13.6 Hz), 2.58 (d, 1H, *J* = 13.6 Hz), 2.27 (s, 1H), 1.39 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 159.1, 145.8, 142.3, 138.0, 128.7, 127.8, 126.4, 125.7, 123.2, 115.9, 115.7, 112.5, 75.6, 55.0, 50.1, 50.0, 24.3. HRMS (ES pos.): m/z calcd for $C_{19}H_{26}NO_2$ (M + NH_4^+): 300.1958, found: 300.1954. Optical Rotation: $[\alpha]_D^{22}$ +21.1 (c = 0.77, $CHCl_3$) for an enantiomerically enriched sample of 96:4 er.

Chiral HPLC conditions: Chiralcel OJ-RH, 4.6 x 150 mm, 5 μm; mobile phase A (acetonitrile), mobile phase B (0.1% HCO_2H in water, adjusted with NH_4OH to pH = 4.0), 52:48 A/B, λ = 220 nm, flow rate: 1.3 mL min^{-1}. The samples for HPLC were diluted with MeOH. (*R*)-**12b**, t_{major} = 7.15 min; (*S*)-**12b**, t_{minor} = 6.64 min

(*R*)-2-(2-Methoxyphenyl)-4-methyl-1-phenylpent-4-en-2-ol 12c

The general procedure was applied to 1-(2-methoxyphenyl)-2-phenylethanone (250 mg, 1.1 mmol). The crude product was purified by column chromatography on silica gel (hexanes: EtOAc = 5:95) to give the product (302 mg, 97% yield, 95:5 er). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.24-7.17 (m, 2H), 7.16-7.10 (m, 3H), 6.99-6.94 (m, 2H), 6.88-6.79 (m, 2H), 4.76 (bs, 1H), 4.67 (bs, 1H), 3.88 (s, 3H), 3.45 (d, 1H, *J* = 13.3 Hz), 3.20 (d, 1H, *J* = 13.5 Hz), 3.09 (d, 1H, *J* = 13.3 Hz), 2.86 (s, 1H), 2.46 (d, 1H, *J* = 13.5 Hz), 1.38(s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 142.9, 142.8, 134.8, 130.8, 129.3, 128.6, 126.6, 125.3, 125.1, 125.0, 123.6, 115.8, 74.7, 50.5, 30.2, 24.0. HRMS (ES pos.): m/z calcd for $C_{19}H_{21}O^+$ (M + H^+ -H_2O): 265.1287, found: 265.1581. Optical Rotation: $[\alpha]_D^{22}$ +27.1 (c = 0.90, $CHCl_3$) for an enantiomerically enriched sample of 92.5:7.5 er.

Chiral HPLC conditions: Chiralcel OJ-RH, 4.6 x 150 mm, 5 μm; mobile phase A (acetonitrile), mobile phase B (0.1% HCO_2H in water, adjusted with NH_4OH to pH =

4.0), 48:52 A/B, λ = 220 nm, flow rate: 1.3 mL min^{-1}. The samples for HPLC were diluted with MeOH. (*R*)-**12c**, t_{major} = 10.58 min; (*S*)-**12c**, t_{minor} = 11.28 min.

(*R*)-2-(4-Fluorophenyl)-4-methyl-1-phenylpent-4-en-2-ol 12d

The general procedure was applied to 1-(4-fluorophenyl)-2-phenylethanone (107.1 mg, 0.5 mmol) in the presence of 5 mol % of **7** (8.06 mg, 0.03 mmol). The crude product was purified by column chromatography on silica gel (hexanes: EtOAc = 5:95) to give the product (136 mg, 97% yield, 96:4 er). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.31-7.24 (m, 2H), 7.19-7.14 (m, 3H), 6.99-6.91 (m, 4H), 4.84 (bs, 1H), 4.67 (bs, 1H), 3.12 (d, 1H, *J* = 13.4 Hz), 3.04 (d, 1H, *J* = 13.4 Hz), 2.74 (d, 1H, *J* = 13.6 Hz), 2.57 (d, 1H, *J* = 13.6 Hz), 2.27 (s, 1H), 1.39 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 162.8, 160.0, 142.1, 141.5, 136.3, 130.7, 127.9, 127.4, 127.3, 126.6, 116.0, 114.6, 114.4, 75.3, 50.2, 50.1, 24.3. HRMS (ES pos.): m/z calcd for $C_{18}H_{23}NFO$ ($M + NH_4^+$): 288.1758, found: 288.1749. Optical Rotation: $[\alpha]_D^{22}$ +22.3 (c = 0.785, $CHCl_3$) for an enantiomerically enriched sample of 96:4 er.

Chiral HPLC conditions: Chiralcel OJ-RH, 4.6 x 150 mm, 5 μm; mobile phase A (acetonitrile), mobile phase B (0.1% HCO_2H in water, adjusted with NH_4OH to pH = 4.0), 60:40 A/B, λ = 220 nm, flow rate: 1.3 mL min^{-1}. The samples for HPLC were diluted with MeOH. (*R*)-**12d**, t_{major} = 4.75 min; (*S*)-**12d**, t_{minor} = 4.41 min.

(*R*)-2-(4-Chlorophenyl)-4-methyl-1-phenylpent-4-en-2-ol 12e

The general procedure was applied to 1-(4-chlorophenyl)-2-phenylethanone (115.3 mg, 0.5 mmol) in the presence of 5 mol % of **7** (8.06 mg, 0.03 mmol). The crude product was purified by column chromatography on silica gel (hexanes: EtOAc = 5:95) to give the product **12e** (139 mg, 97% yield, 96:4 er). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.28-7.21 (m, 4H), 7.19-7.14 (m, 3H), 6.97-6.91 (m, 2H), 4.83 (bs, 1H), 4.66 (bs, 1H), 3.12 (d, 1H, *J* = 13.4 Hz), 3.03 (d, 1H, *J* = 13.4 Hz), 2.73 (d, 1H, *J* = 13.6 Hz), 2.56 (d, 1H, *J* = 13.6 Hz), 2.26 (s, 1H), 1.39 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 144.5, 141.9, 136.2, 132.3, 130.7, 128.0, 127.3, 126.6, 116.1, 75.4, 50.1, 50.0, 24.4. HRMS (ES pos.): m/z calcd for $C_{18}H_{23}NClO$ ($M + NH_4^+$): 304.1463, found: 304.1451. Optical Rotation: $[\alpha]_D^{22}$ +29.5 (c = 0.59, $CHCl_3$) for an enantiomerically enriched sample of 96:4 er.

Chiral HPLC conditions: Chiralcel OJ-RH, 4.6 x 150 mm, 5 μm; mobile phase A (acetonitrile), mobile phase B (0.1% HCO_2H in water, adjusted with NH_4OH to pH = 4.0), 60:40 A/B, λ = 220 nm, flow rate: 1.3 mL min^{-1}. The samples for HPLC were diluted with MeOH. (*R*)-**12e**, t_{major} = 6.03 min; (*S*)-**12e**, t_{minor} = 5.75 min.

(*R*)-2-(4-Bromophenyl)-4-methyl-1-phenylpent-4-en-2-ol 12f

The general procedure was applied to 1-(4-bromophenyl)-2-phenylethanone (137.6 mg, 0.5 mmol) in the presence of 5 mol % of **7** (8.06 mg, 0.03 mmol). The crude product was purified by column chromatography on silica gel (hexanes: EtOAc = 5:95) to give the product (162 mg, 98% yield, 97:3 er). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.42-7.37(m, 2H), 7.23-7.15 (m, 5H), 6.97-6.91 (m, 2H), 4.84 (bs, 1H), 4.67 (s, 1H), 3.12 (d, 1H, *J* = 13.4 Hz), 3.03 (d, 1H, *J* = 13.4 Hz), 2.73 (d, 1H, *J* = 13.7 Hz), 2.56 (d, 1H, *J* = 13.7 Hz), 2.26 (s, 1H), 1.40 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 145.0, 141.9, 136.1, 130.9, 130.7, 128.0, 127.6, 126.6, 120.4, 116.1, 75.4, 50.1, 50.0, 24.4. HRMS (ES pos.): m/z calcd for $C_{18}H_{23}NBrO$ (M + NH_4^+): 348.0958, found: 348.0940. Optical Rotation: $[\alpha]_D^{22}$ +40 (c = 0.80, $CHCl_3$) for an enantiomerically enriched sample of 97:3 er.

Chiral HPLC conditions: Chiralpak OD-H, 4.6 x 250 mm; 98:2 heptane/*iso*-propanol, 1.0 mL/min; 220 nm; (*R*)-**12f**, *t* = 16.47 min; (*S*)-**12f**, *t* = 19.54 min.

(*R*)-2-(4-Methoxyphenyl)-4-methyl-1-(naphthalen-1-yl)pent-4-en-2-ol 12g

The general procedure was applied to 1-(4-methoxyphenyl)-2-(naphthalen-1-yl)ethanone (138 mg, 0.5 mmol) in the presence of 5 mol % of **7** (8.06 mg, 0.03 mmol). The crude product was purified by column chromatography on silica gel (hexanes: EtOAc = 5:95) to give the product (159.6 mg, 96% yield, 93:7 er). ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.14-8.06 (m, 1H), 7.84-7.77 (m, 1H), 7.70 (d, 1H, *J* = 8.1 Hz), 7.48-7.38 (m, 2H), 7.34-7.27 (m, 3H), 7.11 (d, 1H, *J* = 7.0 Hz), 6.84-6.78 (m, 2H), 4.81 (s, 1H), 4.63 (s, 1H), 3.78 (s, 3H), 3.66 (d, 1H, *J* = 14.0 Hz), 3.46 (d, 1H, *J* = 14.0 Hz), 2.83 (d, 1H, *J* = 13.6 Hz), 2.60 (d, 1H, *J* = 13.6 Hz), 2.23 (s, 1H), 1.37 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 158.2, 142.5, 138.5, 133.8, 133.5, 133.2, 129.4, 128.5, 127.2, 126.8, 125.6, 125.3, 125.0, 115.8, 113.2, 76.0, 55.2, 49.8, 45.9, 24.4. HRMS (ES pos.): m/z calcd for $C_{23}H_{28}NO_2$ (M + NH_4^+): 350.2115, found: 350.2093. Optical Rotation: $[\alpha]_D^{22}$ +37.4 (c = 0.96, $CHCl_3$) for an enantiomerically enriched sample of 93:7 er.

Chiral HPLC conditions: Chiralcel OJ-RH, 4.6 x 150 mm, 5 μm; mobile phase A (acetonitrile), mobile phase B (0.1% HCO_2H in water, adjusted with NH_4OH to pH = 4.0), 52:48 A/B, λ = 220 nm, flow rate: 1.3 mL min^{-1}. The samples for HPLC were diluted with MeOH. (*R*)-**12g**, t_{major} = 26.23 min; (*S*)-**12g**, t_{minor} = 27.77 min.

(*R*)-2-(4-Methoxyphenyl)-4-methyl-1-(naphthalen-2-yl)pent-4-en-2-ol 12h

The general procedure was applied to 1-(4-methoxyphenyl)-2-(naphthalen-2-yl)ethanone (138 mg, 0.5 mmol) in the presence of 5 mol % of **7** (16.73 mg, 0.05 mmol). The crude product was purified by column chromatography on silica gel (hexanes: EtOAc = 5:95) to give the product **12h** (164 mg, 99% yield, 98:2 er). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.79-7.68 (m, 2H), 7.61 (d, 1H, *J* = 8.4 Hz), 7.49 (s, 1H), 7.45-7.37 (m, 2H), 7.28-7.22 (m, 2H), 7.06-7.01 (m, 1H), 6.84-6.78 (m, 2H), 4.83(s, 1H), 4.67 (s, 1H), 3.79 (s, 3H), 3.30 (d, 1H, *J* = 13.4 Hz), 3.19 (d, 1H, *J* = 13.Hz), 2.77 (d, 1H, *J* = 13.6 Hz), 2.59 (d, 1H, *J* = 13.6 Hz), 2.29 (s, 1H), 1.40(s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 158.2, 142.5, 137.9, 134.4, 133.2, 132.2, 129.4, 129.3, 127.7, 127.5, 127.1, 126.8, 125.7, 125.4, 115.7, 113.2, 75.5, 55.2, 50.2, 50.1, 24.4. HRMS (ES pos.): m/z calcd for $C_{23}H_{23}O$ (M + H^+-H_2O): 315.1743, found: 315.1730. Optical Rotation: $[\alpha]_D^{22}$ +80.4 (c = 0.83, $CHCl_3$) for an enantiomerically enriched sample of 98:2 er.

Chiral HPLC conditions: Chiralcel OJ-RH, 4.6 x 150 mm, 5 μm; mobile phase A (acetonitrile), mobile phase B (0.1% HCO_2H in water, adjusted with NH_4OH to pH = 4.0), 60:40 A/B, λ = 220 nm, flow rate: 1.3 mL min^{-1}. The samples for HPLC were diluted with MeOH. (*R*)-**12h**, t_{major} = 10.31 min; (*S*)-**12h**, t_{minor} = 11.33 min.

(*R*)-1-Chloro-3-(4-fluorophenyl)-5-methylhex-5-en-3-ol 12i

The general procedure was applied to 3-chloro-1-(4-fluorophenyl)propan-1-one (96 mg, 0.5 mmol) in the presence of 5 mol % of **7** (8.06 mg, 0.03 mmol). The crude product was purified by column chromatography on silica gel (hexanes: EtOAc = 5:95) to give the product (115 mg, 95% yield, 85:15 er). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.39-7.32 (m, 2H), 7.06-7.00 (m, 2H), 4.94 (bs, 1H), 4.77 (bs, 1H), 3.60-3.52 (m, 1H), 3.18-3.10 (m, 1H), 2.64 (d, 1H, *J* = 13.3 Hz), 2.54 (d, 1H, *J* = 13.3 Hz), 2.52 (s, 1H), 2.38-2.24 (m, 2H), 1.32 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 141.5, 140.4, 126.8, 116.8, 115.2, 115.0, 74.2, 51.5, 46.4, 40.0, 24.3. HRMS (ES pos.): m/z calcd for $C_{13}H_{15}ClF^+$ (M + H^+ -H_2O):

225.0841, found: 225.0837. Optical Rotation: $[\alpha]_D^{22}$ -56.3 (c = 0.70, $CHCl_3$) for an enantiomerically enriched sample of 85:15 er.

Chiral HPLC conditions: Chiralcel OJ-RH, 4.6 x 150 mm, 5 μm; mobile phase A (acetonitrile), mobile phase B (0.1% HCO_2H in water, adjusted with NH_4OH to pH = 4.0), 52:48 A/B, λ = 220 nm, flow rate: 1.3 mL min^{-1}. The samples for HPLC were diluted with MeOH. (*R*)-**12i**, t_{major} = 5.32 min; (*S*)-**12i**, t_{minor} = 5.63 min.

(*R*)-4-Methyl-1-phenyl-2-(thiophen-3-yl)pent-4-en-2-ol 12j

The general procedure was applied to 2-phenyl-1-(thiophen-3-yl)ethanone (0.21 g, 1.04 mmol) in the presence of 5 mol % of **7** (16.73 mg, 0.05 mmol). The crude product was purified by column chromatography on silica gel (hexanes: EtOAc = 5:95) to give the product (0.26 g, 98% yield, 96:4 er). ^{1}H NMR (400 MHz, $CDCl_3$), δ 7.27-7.25 (m, 1H), 7.20-7.16 (m, 3H), 6.97-6.93 (m, 4H), 4.86 (m, 1H), 4.70 (m, 1H), 3.02 (d, *J* = 13.32 Hz, 1H), 3.03 (d, *J* = 13.36 Hz, 1H), 2.67 (d, *J* = 13.44 Hz, 1H), 2.55 (d, *J* = 13.44 Hz, 1H), 2.33 (s, 1H), 1.44 (s, 3H). ^{13}C-NMR (100 MHz, $CDCl_3$) δ 148.1, 142.3, 136.5, 130.6, 127.8, 126.5, 126.0, 125.4, 120.6, 115.7, 75.0, 50.2, 49.6, 24.1. HRMS (ES pos.): m/z calcd for $C_{16}H_{22}NOS$ ($M + NH_4^+$): 276.1417, found: 276.1416. Optical Rotation: $[\alpha]_D^{22}$ +1.39 (c = 0.79, $CHCl_3$) for an enantiomerically enriched sample of 96:4 er.

Chiral HPLC conditions: Chiralcel OJ-RH, 4.6 x 150 mm, 5 μm; mobile phase A (acetonitrile), mobile phase B (0.1% HCO_2H in water, adjusted with NH_4OH to pH = 4.0), 40:60 A/B, λ = 220 nm, flow rate: 1.3 mL min^{-1}. The samples for HPLC were diluted with MeOH. (*R*)-**12j**, t_{major} = 23.36 min; (*S*)-**12j**, t_{minor} = 24.38 min.

(R)-4-Methyl-1-phenyl-2-(thiophen-2-yl)pent-4-en-2-ol 12k

The general procedure was applied to 2-phenyl-1-(thiophen-2-yl)ethanone (0.21 g, 1.04 mmol) in the presence of 5 mol % of **7** (16.73 mg, 0.05 mmol). The crude product was purified by column chromatography on silica gel (hexanes: EtOAc = 5:95) to give the product (0.26 g, 97% yield, 97:3 er). ^{1}H NMR (400 MHz, $CDCl_3$), δ 7.26-7.18 (m, 3H), 7.15-7.13 (m, 1H), 7.06-7.03 (m, 2H), 6.92-6.90 (m, 1H), 6.76-6.74 (m, 1H), 4.91 (m, 1h), 4.73 (m, 1H), 3.18 (d, *J* = 13.4 Hz, 1H), 3.10 (d, *J* = 13.4 Hz, 1H), 2.70 (d, *J* = 13.5 Hz, 1H), 2.61 (dd, *J* = 0.64, 13.5 Hz, 1H), 2.58 (s, 1H), 1.50 (s, 3H). ^{13}C-NMR (100 MHz, $CDCl_3$) δ 151.8, 142.1, 136.2, 130.7, 127.9, 126.7, 126.7, 123.7, 123.1, 116.1,

75.1, 51.1, 50.7, 24.2. HRMS (ES pos.): m/z calcd for $C_{16}H_{22}NOS$ (M + NH_4^+): 276.1417, found: 276.1419. Optical Rotation: $[\alpha]_D^{22}$ +13.5 (c = 0.89, $CHCl_3$) for an enantiomerically enriched sample of 96:4 er.

Chiral HPLC conditions: Chiralcel OJ-RH, 4.6 x 150 mm, 5 μm; mobile phase A (acetonitrile), mobile phase B (0.1% HCO_2H in water, adjusted with NH_4OH to pH = 4.0), 40:60 A/B, λ = 220 nm, flow rate: 1.3 mL min^{-1}. The samples for HPLC were diluted with MeOH. (*R*)-**12k**, t_{major} = 24.43 min; (*S*)-**12k**, t_{minor} = 26.30 min.

(*S*)-5-Methyl-1,3-diphenylhex-5-en-3-ol 12l

The general procedure was applied to 1,3-diphenylpropan-1-one (125 mg, 0.59 mmol) in the presence of 5 mol % of **7** (9.58 mg, 0.03 mmol). The crude product was purified by column chromatography on silica gel (hexanes: EtOAc = 5:95) to give the product (154 mg, 97% yield, 91:9 er). ^{1}H NMR (400 MHz, $CDCl_3$), δ 7.46-7.43 (m, 2H), 7.37-7.33 (m, 2H), 7.26-721 (m, 3H), 7.15-7.08 (m, 3H), 4.90 (m, 1H), 4.77 (bs, 1H), 2.72-2.56 (m, 3H), 2.42 (s, 1H), 2.30-2.06 (m, 3H), 1.30 (s, 3H). ^{13}C-NMR (100 MHz, $CDCl_3$) δ 145.9, 142.6, 142.4, 128.4, 128.3, 128.2, 126.5, 125.7, 125.4, 116.0, 75.0, 51.5, 45.5, 30.0, 24.3. HRMS (ES pos.): m/z calcd for $C_{19}H_{21}^+$ (M + H^+ -H_2O): 249.1638, found: 249.1639. Optical Rotation: $[\alpha]_D^{22}$ -75.6 (c = 0.88, $CHCl_3$) for an enantiomerically enriched sample of 91:9 er.

Chiral HPLC conditions: Chiralcel OJ-RH, 4.6 x 150 mm, 5 μm; mobile phase A (acetonitrile), mobile phase B (0.1% HCO_2H in water, adjusted with NH_4OH to pH = 4.0), 60:40 A/B, λ = 220 nm, flow rate: 1.3 mL min^{-1}. The samples for HPLC were diluted with MeOH. (*R*)-**12l**, t_{minor} = 8.17 min; (*S*)-**12l**, t_{major} = 7.36 min.

(*S*)-2-Methyl-4,7-diphenylhept-1-en-4-ol 12m

The general procedure was applied to 1,4-diphenylbutan-1-one (125 mg, 0.56 mmol) in the presence of 5 mol % of **7** (8.98 mg, 0.03 mmol). The crude product was purified by column chromatography on silica gel (hexanes: EtOAc = 5:95) to give the product (150 mg, 96% yield, 90:10 er). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.23-7.07 (m, 10 H), 4.86 (s, 1H), 4.72 (s, 1H), 2.63-2.49 (m, 4H), 2.33 (s, 1H), 1.91-1.80 (m, 3H), 1.38-1.29 (m, 1H), 1.27 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 146.1, 142.5, 142.4, 128.4, 128.2, 128.0, 126.4, 125.7, 125.3, 115.9, 75.0, 51.3, 43.1, 36.1, 25.3, 24.3. HRMS (ES pos.): m/z calcd

for $C_{20}H_{28}NO^+$ ($M + NH_4^+$): 298.2165, found: 298.2156. Optical Rotation: $[\alpha]_D^{22}$ -44.8 (c = 0.65, $CHCl_3$) for an enantiomerically enriched sample of 90:10 er.

Chiral HPLC conditions: Chiralcel OJ-RH, 4.6 x 150 mm, 5 μm; mobile phase A (acetonitrile), mobile phase B (0.1% HCO_2H in water, adjusted with NH_4OH to pH = 4.0), 60:40 A/B, λ = 220 nm, flow rate: 1.3 mL min^{-1}. The samples for HPLC were diluted with MeOH. (*R*)-**12m**, t_{minor} = 8.34 min; (*S*)-**12m**, t_{major} = 6.83 min.

(*R*)-1-(Benzyloxy)-4-methyl-2-phenylpent-4-en-2-ol 12n

The general procedure was applied to 2-(benzyloxy)-1-phenylethanone (0.23 g, 1.02 mmol) in the presence of 5 mol % of **7** (6.55 mg, 0.02 mmol). The crude product was purified by column chromatography on silica gel (hexanes: EtOAc = 5:95) to give the product **12n** (269 mg, 93% yield, 85:15 er). ^{1}H NMR (400 MHz, $CDCl_3$), δ 7.44-7.42 (m, 2H), 7.34-7.22 (m, 8H), 4.78 (m, 1H), 4.63 (m, 1H), 4.54 (bs, 2H), 3.65 (d, *J* = 9.4 Hz, 1H), 3.62 (d, *J* = 9.3 Hz, 1H), 2.88 (s, 1H), 2.68 (d, *J* = 13.68 Hz), 2.57 (d, *J* = 13.68 Hz, 1H), 1.46 (s, 3H). ^{13}C-NMR (100 MHz, $CDCl_3$) δ 144.2, 141.9, 138.0, 128.4, 128.0, 127.8, 127.7, 126.8, 125.5, 115.2, 77.3, 75.5, 73.4, 47.1, 24.3. HRMS (ES pos.): m/z calcd for $C_{19}H_{26}NO_2$ ($M + NH_4^+$): 300.1958, found: 300.1959. Optical Rotation: $[\alpha]_D^{22}$ -24.3 (c = 1.02, $CHCl_3$) for an enantiomerically enriched sample of 84:16 er.

Chiral HPLC conditions: Chiralcel OJ-3, 4.6 x 150 mm, 5 μm; mobile phase A (heptane), mobile phase B (IPA), 92:8 A/B, λ = 220 nm, flow rate: 1.0 mL min^{-1}. The samples for HPLC were diluted with MeOH. (*R*)-**12n**, t_{major} = 6.63 min; (*S*)-**12n**, t_{minor} = 9.53 min.

(*S*)-4-Methyl-2-phenylpent-4-en-2-ol 12o[5]

The general procedure was applied to acetophenone (60 mg, 0.50 mmol) in the presence of 5 mol % of **7** (0.025 mmol). The crude product was purified by column chromatography on silica gel (hexanes: EtOAc = 5:95) to give the product **12n** (85 mg, 96% yield, 78:22 er). Optical Rotation: $[\alpha]_D^{22}$ -43.2 (c = 3.49, $CHCl_3$) for an enantiomerically enriched sample of 78:22 er.

Chiral HPLC conditions: Chiralcel OJ-RH, 4.6 x 150 mm, 5 μm, mobile phase A (acetonitrile), mobile phase B (0.1% HCO_2H in water, adjusted with NH_4OH to pH =

4.0), 52:48 A/B, λ = 220 nm, flow rate: 1.3 mL min^{-1}. The samples for HPLC were diluted with MeOH. (*R*)-**12o**, t_{minor} = 3.75 min; (*S*)-**12o**, t_{major} = 4.89 min.

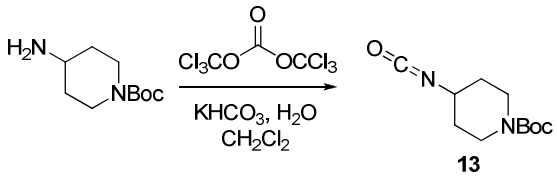
(*R*)-tert-Butyl 3-hydroxy-5-methyl-3-phenylhex-5-enoate 12p

The general procedure was applied to tert-butyl 3-oxo-3-phenylpropanoate (0.23 g, 0.98 mmol) in the presence of 5 mol % of **1** (15.82 mg, 0.05 mmol). The crude product was purified by column chromatography on silica gel (hexanes: EtOAc = 5:95) to give the product **12p** (275 mg, 95% yield, 78:22 er). ^{1}H NMR (400 MHz, $CDCl_3$), δ 7.44-7.42 (m, 2H), 7.32-7.29 (m, 2H), 7.26-7.21 (m, 1H), 4.82 (m, 1H), 4.63 (m, 1H), 4.42 (bs, 1H), 2.89 (d, *J* = 11.61 Hz, 1H), 2.77 (d, *J* = 15.52 Hz, 1H), 2.50 (d, *J* = 13.56 Hz, 1H), 2.45 (d, *J* = 13.52 Hz, 1H), 1.62 (s, 3H), 1.23 (s, 9H). ^{13}C-NMR (100 MHz, $CDCl_3$) δ 172.3, 146.0, 142.0, 127.9, 126.7, 125.3, 115.3, 81.7, 75.3, 51.4, 45.8, 27.8, 24.3. HRMS (ES pos.): m/z calcd for $C_{34}H_{48}NaO_6$ (2M + Na^+): 575.3343, found: 575.3352. Optical Rotation: $[\alpha]_D^{22}$ +11.8 (c = 0.89, $CHCl_3$) for an enantiomerically enriched sample of 80:20 er.

Chiral HPLC conditions: Chiralcel OJ-RH, 4.6 x 150 mm, 5 μm; mobile phase A (acetonitrile), mobile phase B (0.1% HCO_2H in water, adjusted with NH_4OH to pH = 4.0), 48:52 A/B, λ = 220 nm, flow rate: 1.3 mL min^{-1}. The samples for HPLC were diluted with MeOH. (*R*)-**12p**, t_{major} = 7.31 min; (*S*)-**12p**, t_{minor} = 9.10 min.

Synthesis of enantioenriched (*R*)-tert-butyl 4-(6-(2-methylallyl)-2-oxo-6-phenyl-1,3-oxazinan-3-yl)piperidine-1-carboxylate 1

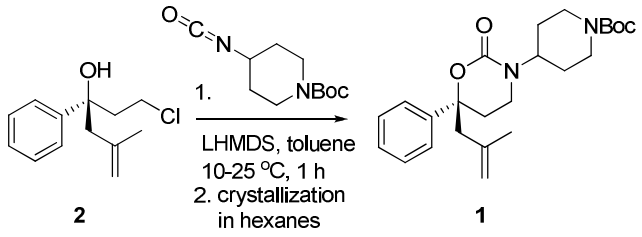
*Synthesis of tert-butyl 4-isocyanatopiperidine-1-carboxylate **13***



13

To a flask was charged $KHCO_3$ (5.75 g; 57.42 mmol) and H_2O (50.00 ml; 10.000 V) followed by addition of CH_2Cl_2 (50.00 ml; 10.000 V) and 4-amino-1-Boc-piperidine (5.00 g; 24.97 mmol). The resulting mixture was cooled to 0 ^oC. Triphosgene (2.56 g; 8.44 mmol) was charged in one portion. After the mixture was stirred for 1h, the organic layer was separated. The aqueous layer was extracted with CH_2Cl_2 (2 x 30 mL). The combined organic layers were washed successively with water and brine, and dried with

anhydrous Na_2SO_4. After removal of the solvent, the residue was dissolved in hexanes. After the mixture was stirred for 1h, the solid (small amount of urea) was filtered off. The filtrate was concentrated to give the tert-butyl 4-isocyanatopiperidine-1-carboxylate **13** as a liquid, which was used for next step directly.



*Synthesis of (R)-tert-butyl 4-(6-(2-methylallyl)-2-oxo-6-phenyl-1,3-oxazinan-3-yl)piperidine-1-carboxylate **1***

To a dry flask was charged (*R*)-1-chloro-5-methyl-3-phenylhex-5-en-3-ol **2** (3.20 g, 13.67 mmol) with 87:13 er, tert-butyl 4-isocyanatopiperidine-1-carboxylate **13** (13.67 mmol) and anhydrous toluene (35 mL). After the mixture was cooled to ~10 °C, LiHMDS (1M in toluene, 14.36 mL) was added while maintaining the internal temperature below 25 °C. After 1h, the mixture was extracted with EtOAc (2 X 100 mL), washed successively with aq. NH_4Cl and water, and dried with anhydrous Na_2SO_4. After removal of the solvent, the residue was stirred in 15 mL of hexanes at rt. After overnight, the white solid was collected and washed with 10 mL of 5:1 hexanes/EtOAc to give the product **1** with 99.4:0.6 er (3.5 g, 62% yield).

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.37-7.27 (m, 5H), 4.87 (s, 1H), 4.68 (s, 1H),4.27-4.07 (m, 3H), 3.08-3.04 (m, 1H), 2.75-2.53 (m, 5H), 2.38-2.35 (m, 1H), 2.6-2.21 (m, 1H), 1.66 (bs, 4H), 1.42 (s, 9H), 1.30-1.24 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 154.5, 152.9, 141.9, 140.5, 128.6, 127.6, 124.8, 116.3, 83.1, 79.6, 53.7, 50.8, 43.1, 36.6, 30.4, 28.4, 28.3, 24.3. HRMS (ES pos.): m/z calcd for $C_{24}H_{35}N_2O_4^+$ ($M + H^+ - H_2O$): 415.2591, found: 415.2598.

Chiral HPLC conditions: Chiralpak AD-H, 4.6 x 250 mm; 85:15 pentane/*iso*-propanol, 1.0 mL/min; 220 nm; (S)-**1**, t_{minor} = 5.47 min; (*R*)-**1**, t_{major} = 6.01 min.

(*R*)-4-Methylene-1,2-diphenylhexan-2-ol 16

The general procedure was applied to 1,2-diphenylethanone (0.20, 1.02 mmol) in the presence of 5 mol % of **7** (16.42 mg, 0.05 mmol) and boronate **14** (1.53 mmol, 1.5 equiv).

The crude product was purified by column chromatography on silica gel (hexanes: EtOAc = 5:95) to give the product (251 mg, 93% yield, 95:5 er). ^{1}H NMR (400 MHz, $CDCl_3$), δ 7.34-7.24 (m, 4H), 7.21-7.14 (m, 4H), 6.97-6.95 (m, 2H), 4.84 (m, 1H), 4.70 (s, 1H), 3.17 (d, *J* = 13.4 Hz, 1H), 3.06 (d, *J* = 13.7 Hz, 1H), 2.79 (d, *J* = 13.7 Hz, 1H), 2.53 (d, *J* = 13.7 Hz, 1H), 2.30 (s, 1H), 1.60 (q, *J* = 7.2 Hz, 2H), 0.83 (t, *J* = 7.4 Hz, 3H). ^{13}C-NMR (100 MHz, $CDCl_3$) δ 147.8, 146.0, 136.7, 130.8, 127.8, 126.4, 126.4, 125.6, 113.3, 75.5, 49.9, 48.5, 29.9, 12.3. HRMS (ES pos.): m/z calcd for $C_{19}H_{26}NO$ ($M + NH_4^+$): 284.2009, found: 284.2003. Optical Rotation: $[\alpha]_D^{22}$ +15.6 (c = 0.90, $CHCl_3$) for an enantiomerically enriched sample of 95:5 er.

Chiral HPLC conditions: Chiralpak OD-H, 4.6 x 250 mm; 98:2 heptane/*iso*-propanol, 1.0 mL/min; 220 nm; (*R*)-**16**, t_{major} = 7.71 min; (*S*)-**16**, t_{minor} = 11.45 min.

(*R*)-4-Methylene-1,2-diphenyloctan-2-ol 17

The general procedure was applied to 1,2-diphenylethanone in the presence of 5 mol % of **7** (16.73 mg, 0.05 mmol) and boronate **15** (1.53 mmol, 1.5 equiv). The crude product was purified by column chromatography on silica gel (hexanes: EtOAc = 5:95) to give the product **17** (294 mg, 95% yield, 93:7 er). ^{1}H NMR (400 MHz, $CDCl_3$), δ 7.34-7.25 (m, 4H), 7.21-7.14 (m, 4H), 6.98-6.96 (m, 2H), 4.82 (s, 1H), 4.70 (s, 1H), 3.17 (d, *J* = 13.4 Hz, 1H), 3.06 (d, *J* = 13.3 Hz, 1H), 2.79 (d, *J* = 13.6 Hz, 1H), 2.53 (d, *J* = 13.7 Hz, 1H), 2.32 (s, 1H), 1.51-1.59 (m, 1H), 1.53-1.46 (m, 1H), 1.23-1.07 (m, 4H), 0.77 (t, *J* = 7.2 Hz, 3H). ^{13}C-NMR (100 MHz, $CDCl_3$) δ 146.4, 146.0, 136.7, 130.8, 127.8, 126.4, 126.4, 125.6, 114.5, 75.5, 49.9, 48.1, 36.7, 29.9, 22.3, 13.9. HRMS (ES pos.): m/z calcd for $C_{21}H_{30}NO$ ($M + NH_4^+$): 312.2322, found: 312.2314. Optical Rotation: $[\alpha]_D^{22}$ +17.1 (c = 0.55, $CHCl_3$) for an enantiomerically enriched sample of 93:7 er.

Chiral HPLC conditions: Chiralcel OJ-RH, 4.6 x 150 mm, 5 μm; mobile phase A (acetonitrile), mobile phase B (0.1% HCO_2H in water, adjusted with NH_4OH to pH = 4.0), 60:40 A/B, λ = 220 nm, flow rate: 1.3 mL min^{-1}. The samples for HPLC were diluted with MeOH. (*R*)-**17**, t_{major} = 7.09 min; (*S*)-**17**, t_{major} = 8.24 min.

References:

1. X. Wu, T-A. Chen, L. Zhu, R. D. Rieke, *Tetrahedron Lett.* **1994**, *35*, 3673.

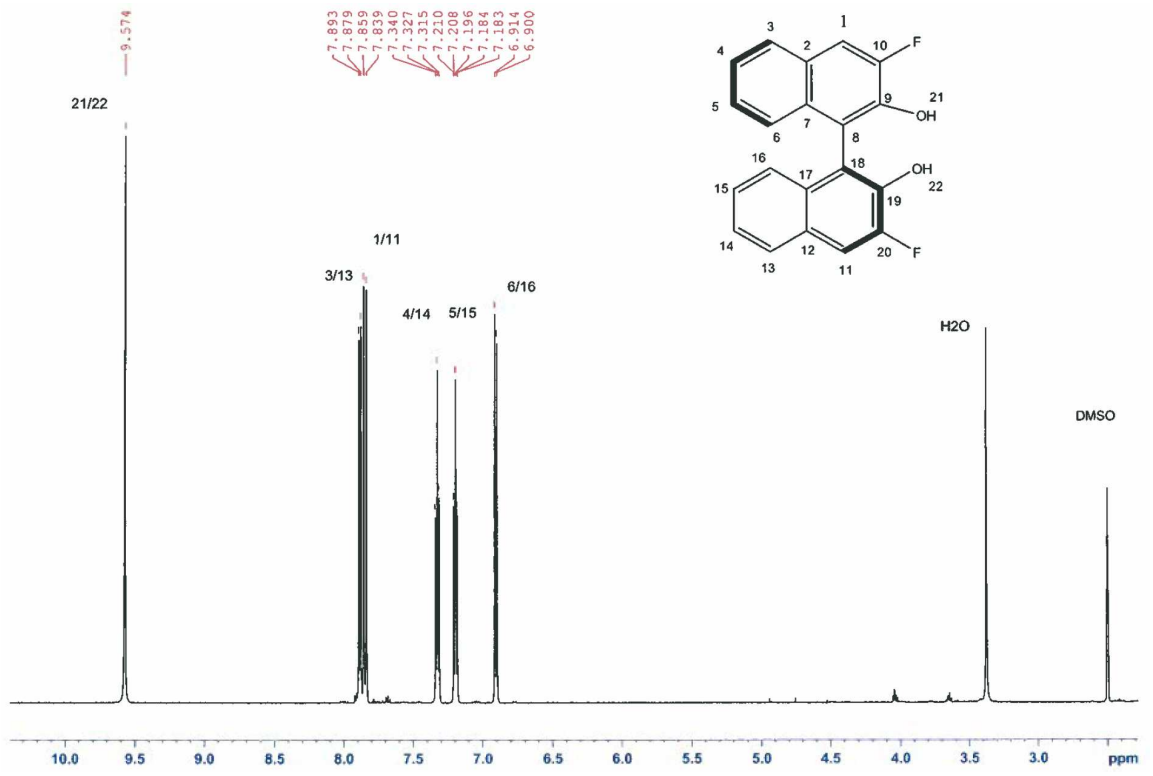
2. J. A. Turner, W. S. Jacks, *J. Org. Chem.* **1989**, *54*, 4229.

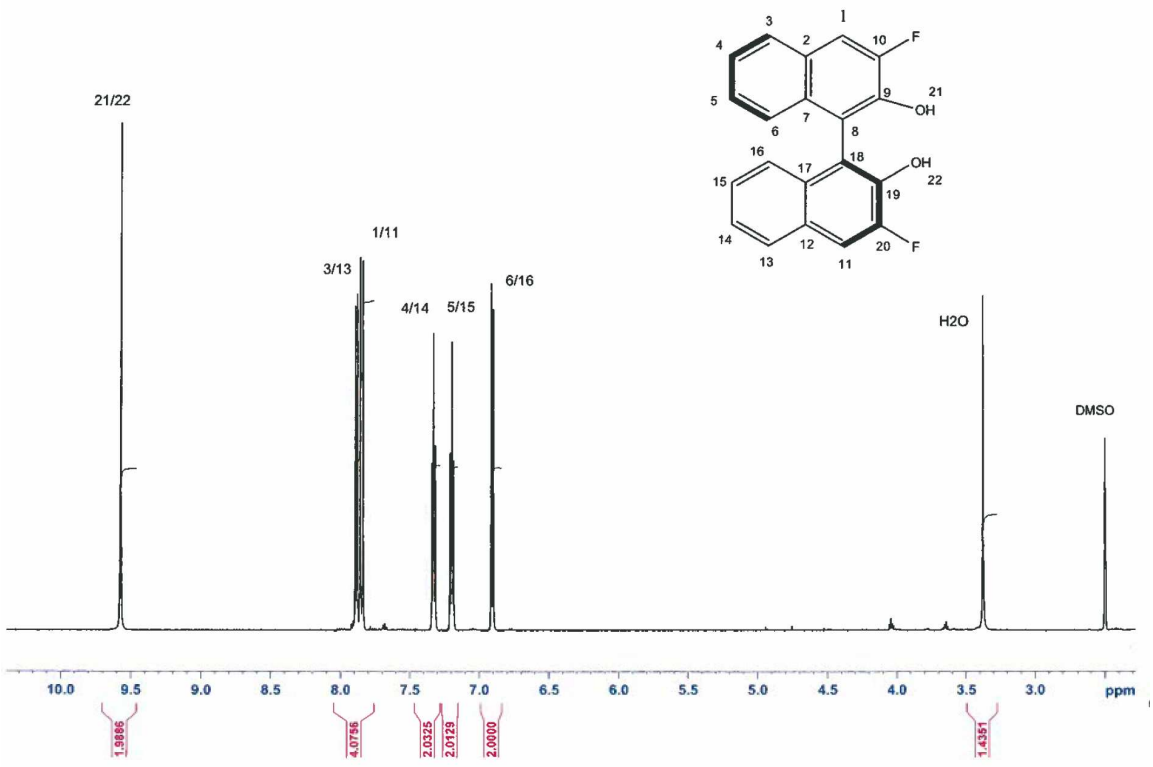
3. No racemization was observed in 1:1 THF:3M HCl after 16 h at 60 ^{o}C. However, the er changed from >99:1 to 90.5:9.5 in 2M NaOH after 16 h at 100 ^{o}C.

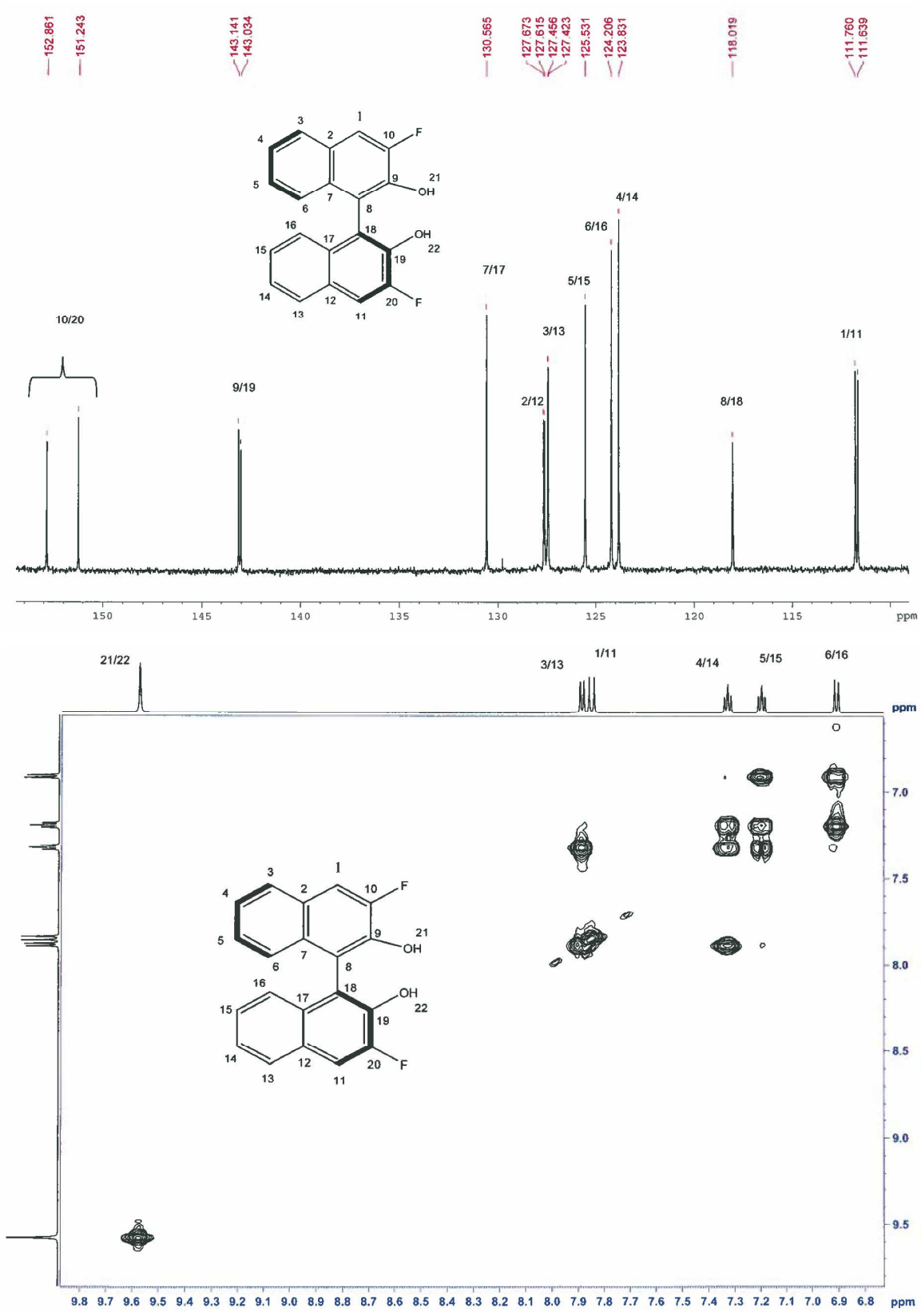
4. T. R. Hoye, A. W. Aspaas, B. M. Eklov, T. D. Ryba. *Org. Lett.* **2005**, *7*, 2205.

5. J. G. Kim, E. H. Camp, P. J. Walsh, *Org. Lett*. **2006**, *8*, 4413-4416.

(*S*)-3,3'-Difluoro-1,1'-binaphthyl-2,2'-diol 7



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143.141
143.034
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127.673
127.615
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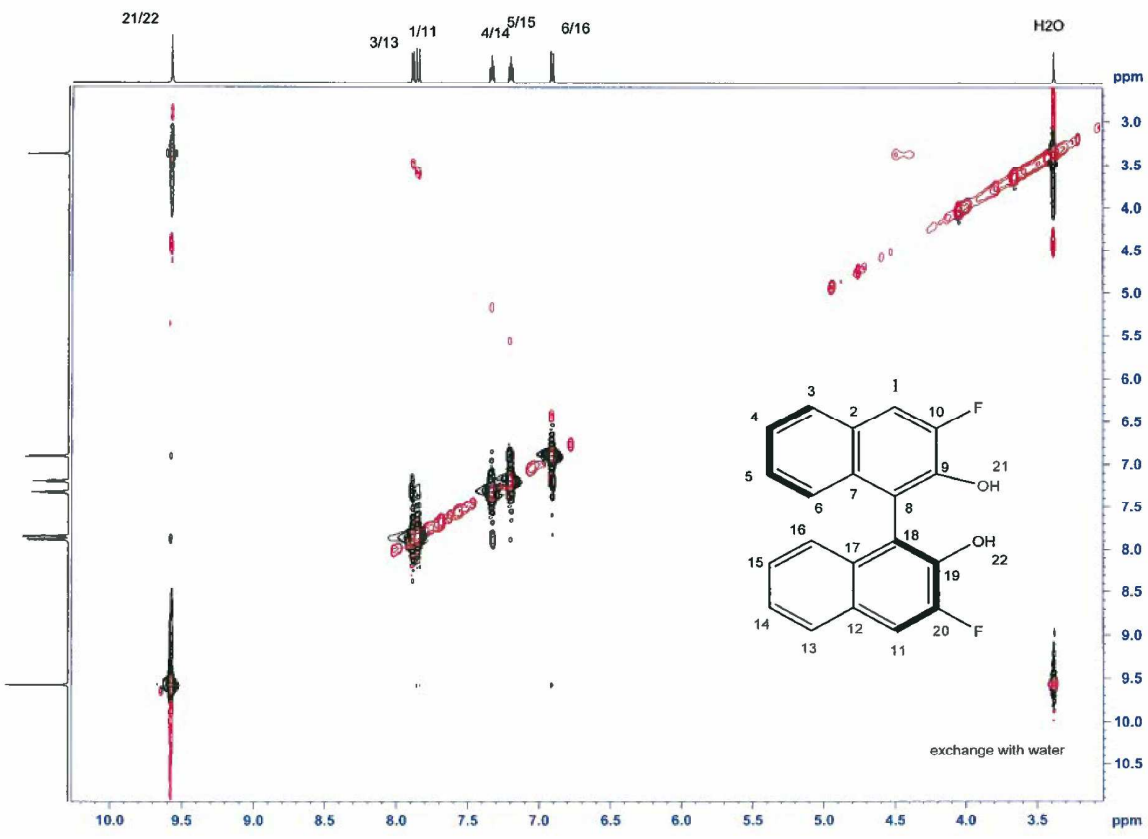
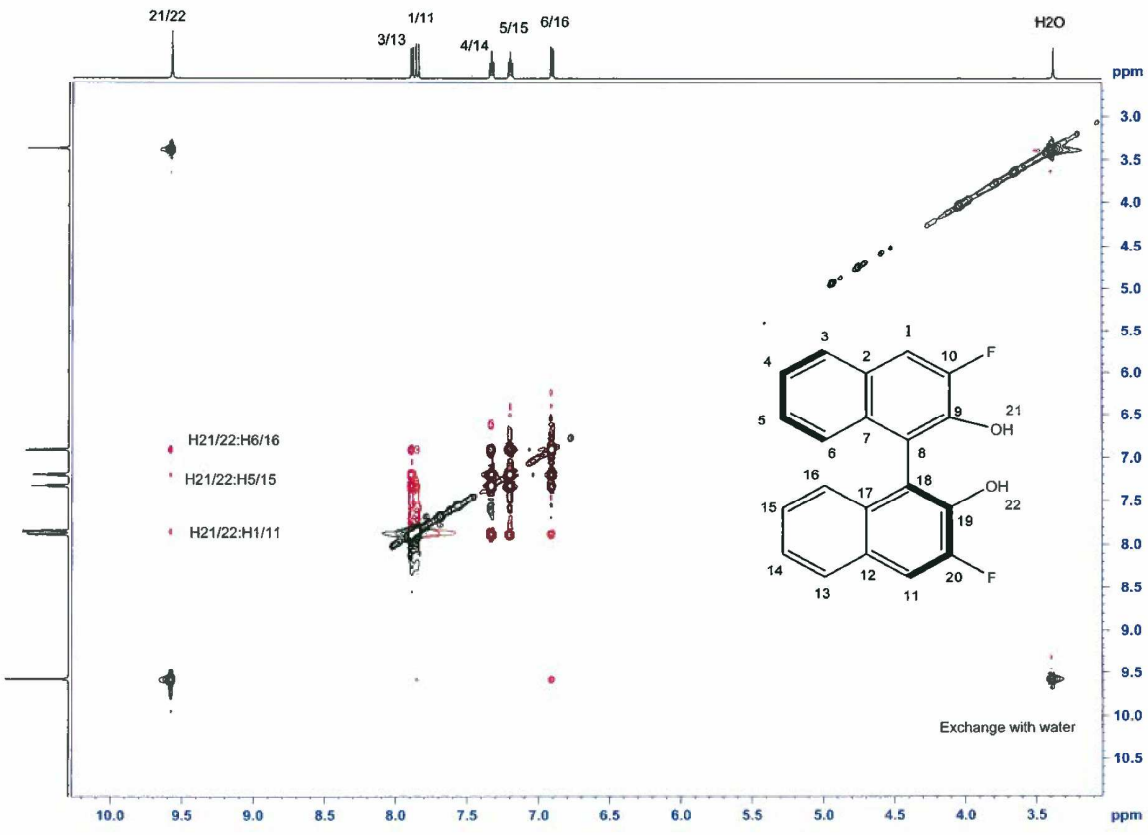
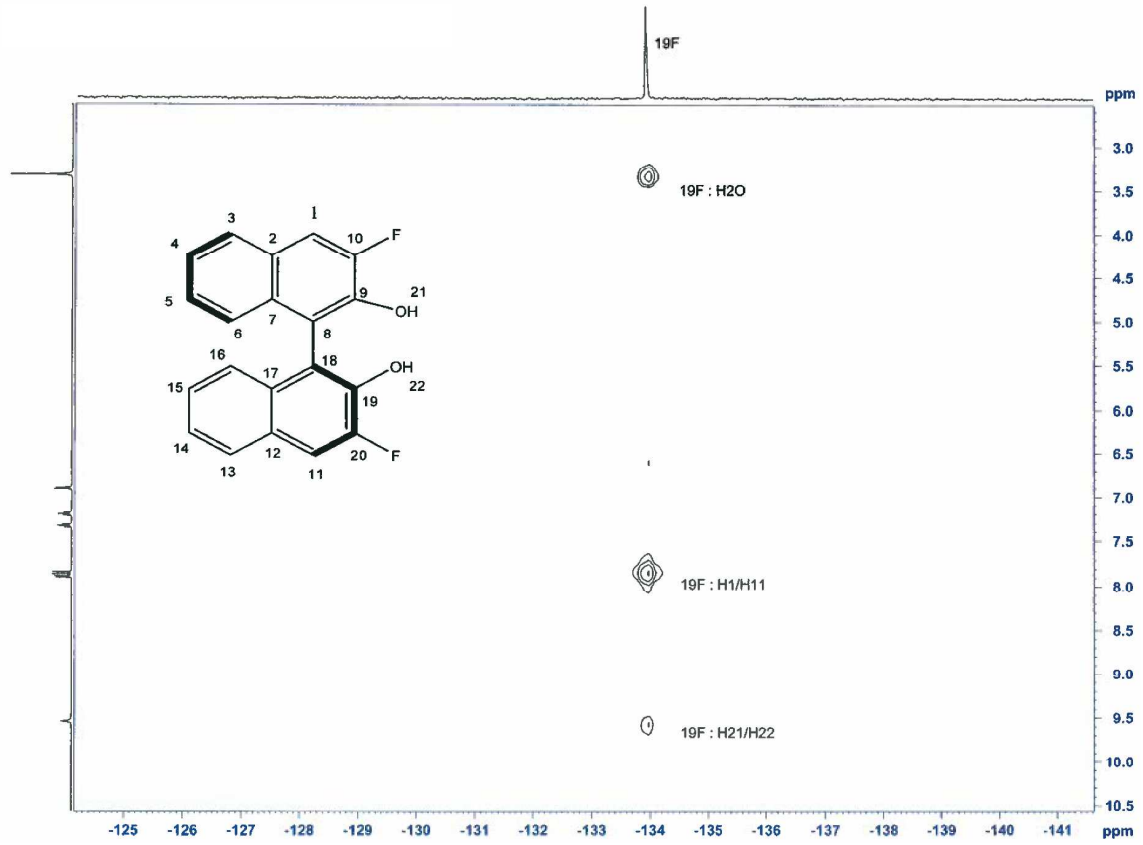
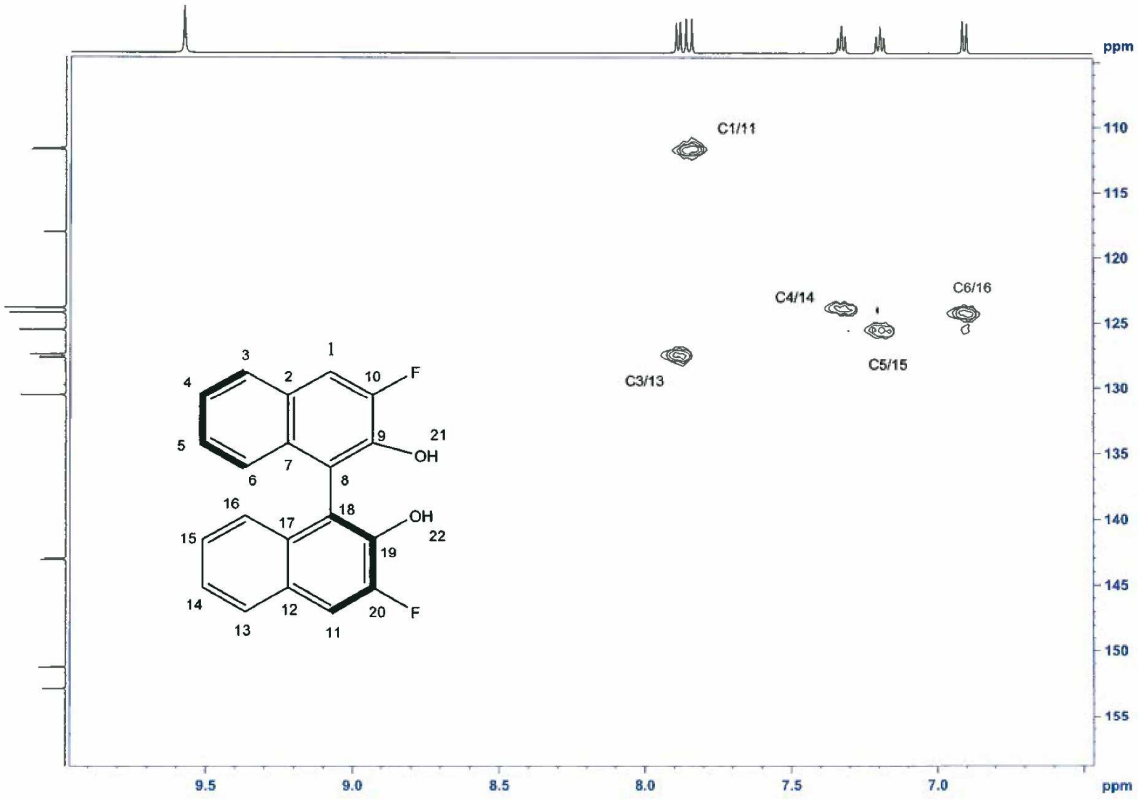
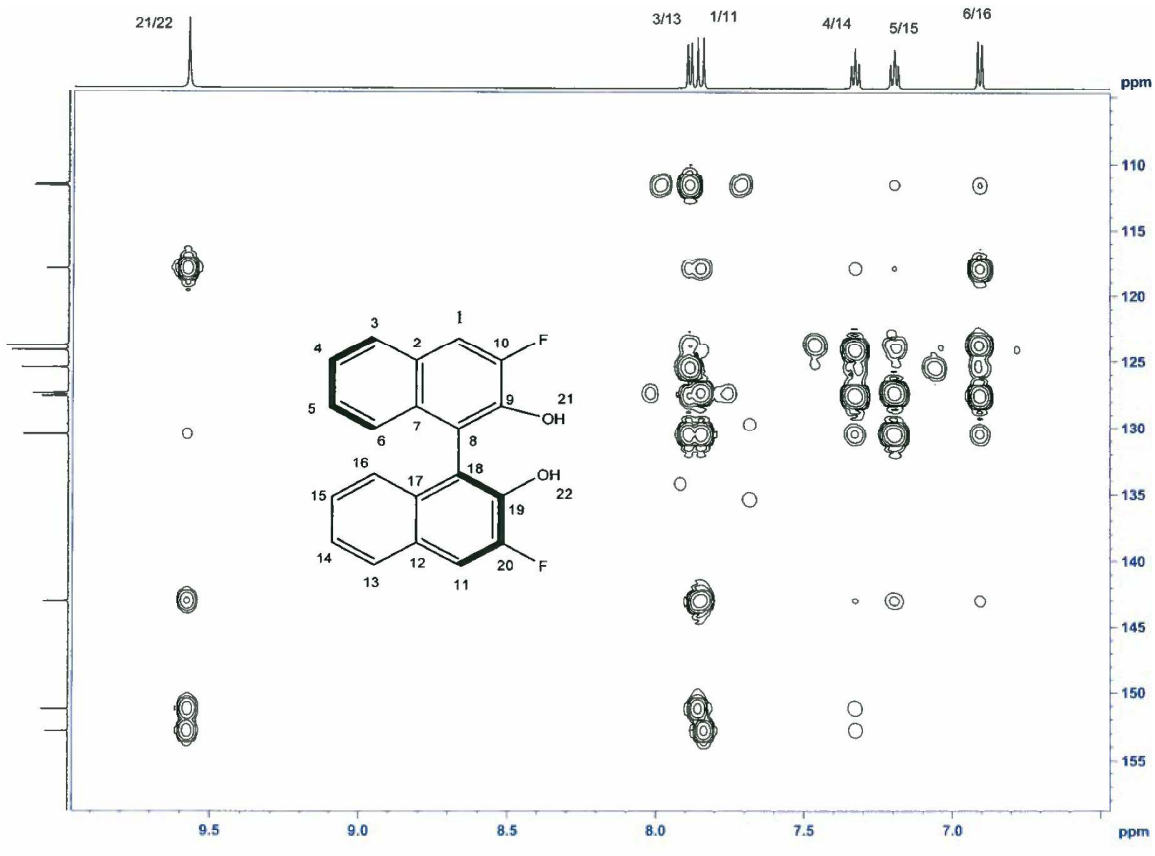
127.423
125.531
124.206
123.831
118.019
111.760
111.639
10/20
9/19
7/17
2/12
3/13
5/15
6/16
4/14
8/18
1/11
150
145
140
135
130
125
120
115
ppm
21/22
3/13
1/11
4/14
5/15
6/16
ppm
7.0
7.5
8.0
8.5
9.0
9.5
9.8 9.7 9.6 9.5 9.4 9.3 9.2 9.1 9.0 8.9 8.8 8.7 8.6 8.5 8.4 8.3 8.2 8.1 8.0 7.9 7.8 7.7 7.6 7.5 7.4 7.3 7.2 7.1 7.0 6.9 6.8
ppm


21/22
3/13
1/11
4/14
5/15
6/16
H2O
ppm
exchange with water


21/22
3/13
1/11
4/14
5/15
6/16
H2O
ppm
H21/22:H6/16
H21/22:H5/15
H21/22:H1/11
Exchange with water

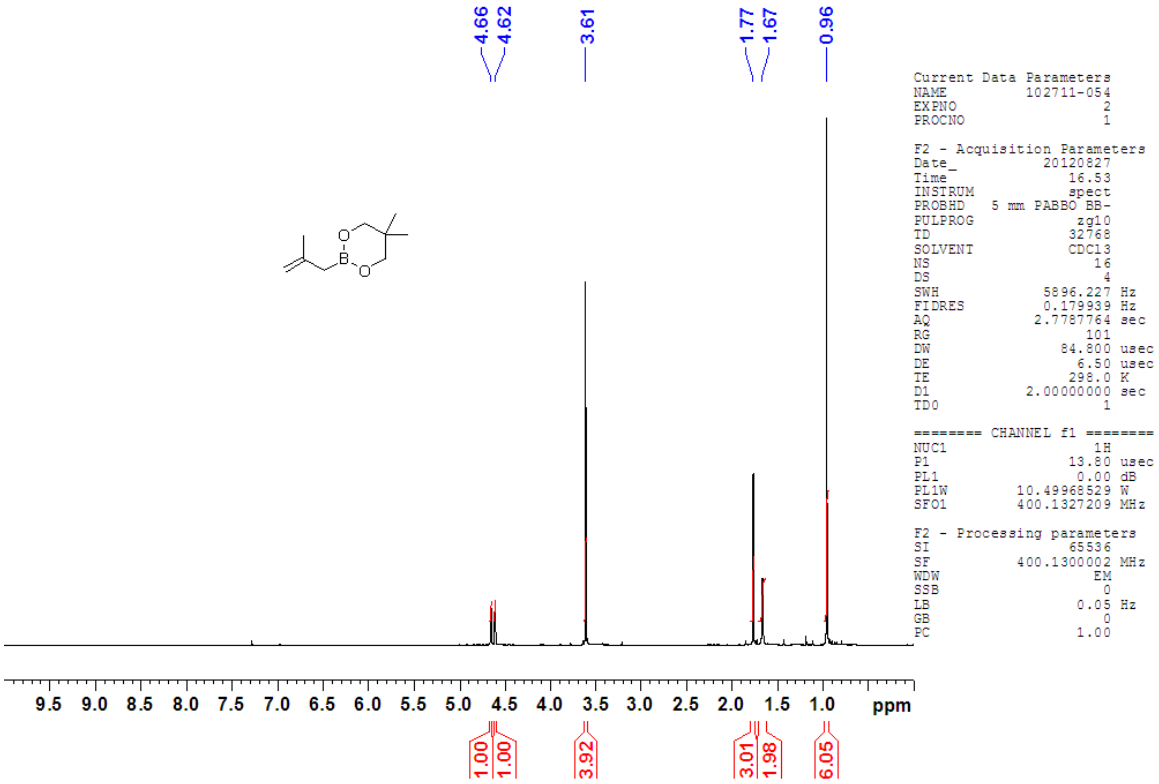

19F
ppm
19F : H2O
19F : H1/H11
19F : H21/H22
-125 -126 -127 -128 -129 -130 -131 -132 -133 -134 -135 -136 -137 -138 -139 -140 -141 ppm


ppm
C1/11
C4/14
C6/16
C5/15
C3/13
9.5 9.0 8.5 8.0 7.5 7.0 ppm


21/22
3/13
1/11
4/14
5/15
6/16
ppm
110
115
120
125
130
135
140
145
150
155
9.5
9.0
8.5
8.0
7.5
7.0
ppm
1
2
3
4
5
6
7
8
9
10
F
21
OH
16
17
18
OH
22
15
19
14
12
20
F
13
11

5,5-Dimethyl-2-(2-methylallyl)-1,3,2-dioxaborinane 4

102711-054-H

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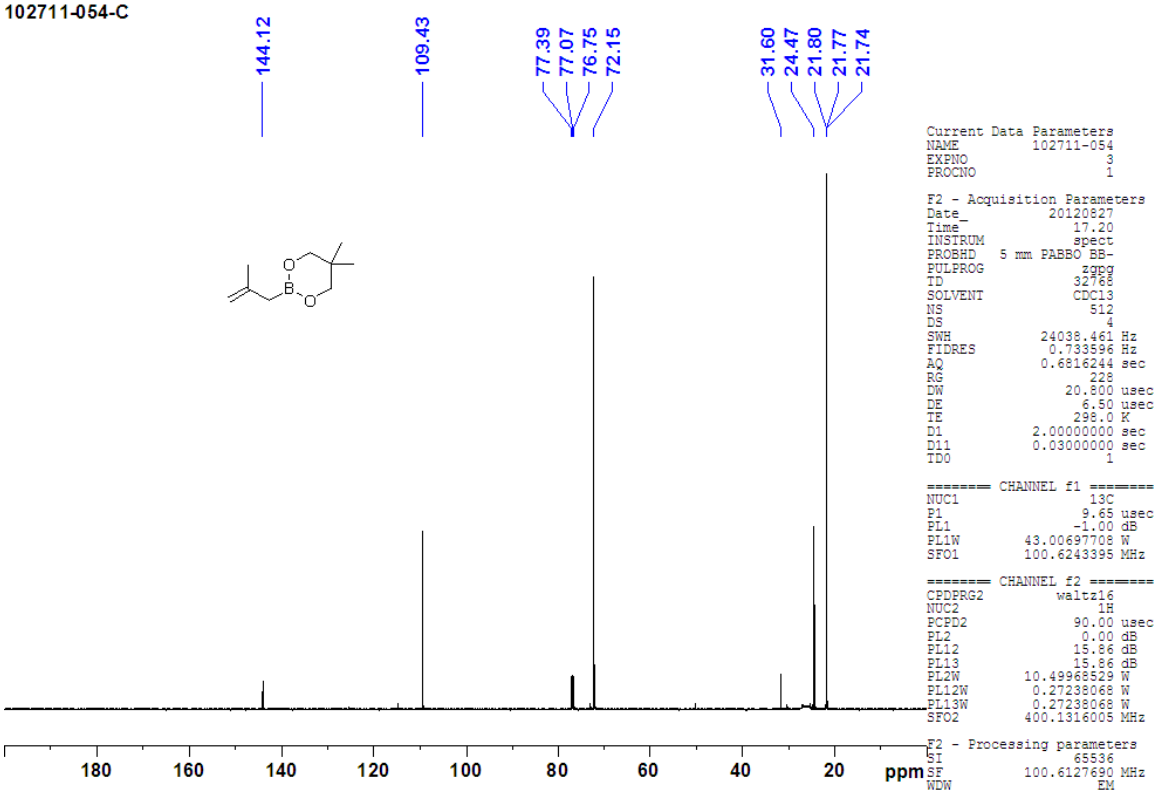
Current Data Parameters
NAME          102711-054
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20120827
Time               16.53
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zg10
TD                 32768
SOLVENT            CDCl3
NS                    16
DS                     4
SWH             5896.227 Hz
FIDRES          0.179939 Hz
AQ              2.7787764 sec
RG                   101
DW                84.800 usec
DE                 6.50 usec
TE                298.0 K
D1             2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 13.80 usec
PL1                0.00 dB
PL1W         10.49968529 W
SFO1         400.1327209 MHz

F2 - Processing parameters
SI                 65536
SF           400.1300002 MHz
WDW                   EM
SSB                    0
LB                  0.05 Hz
GB                     0
PC                  1.00
```

102711-054-C




```
Current Data Parameters
NAME          102711-054
EXPNO                  3
PROCNO                 1

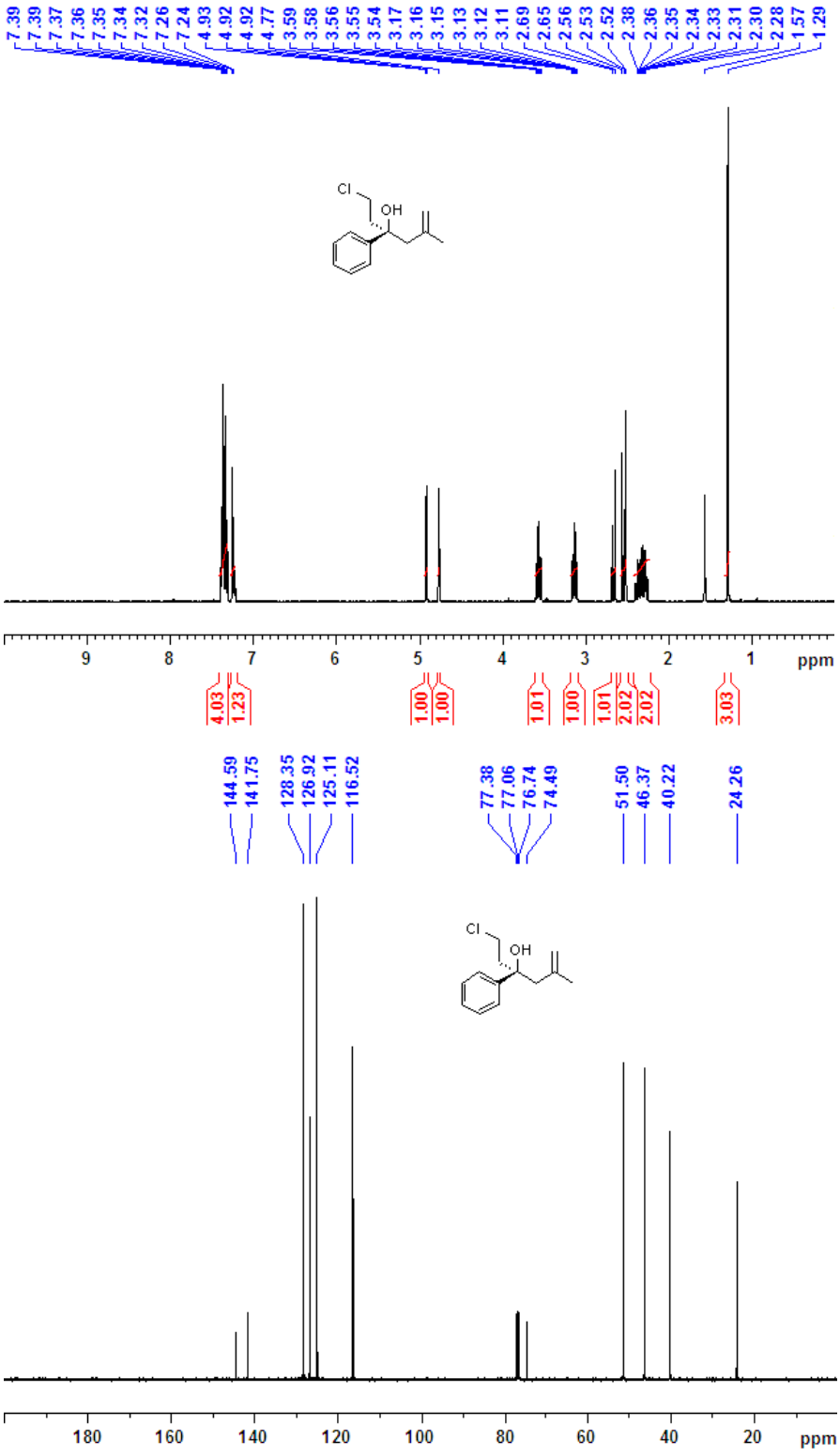
F2 - Acquisition Parameters
Date_           20120827
Time               17.20
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zgpg
TD                 32768
SOLVENT            CDCl3
NS                   512
DS                     4
SWH            24038.461 Hz
FIDRES          0.733596 Hz
AQ              0.6816244 sec
RG                   228
DW                20.800 usec
DE                 6.50 usec
TE                298.0 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                 9.65 usec
PL1               -1.00 dB
PL1W         43.00697708 W
SFO1         100.6243395 MHz

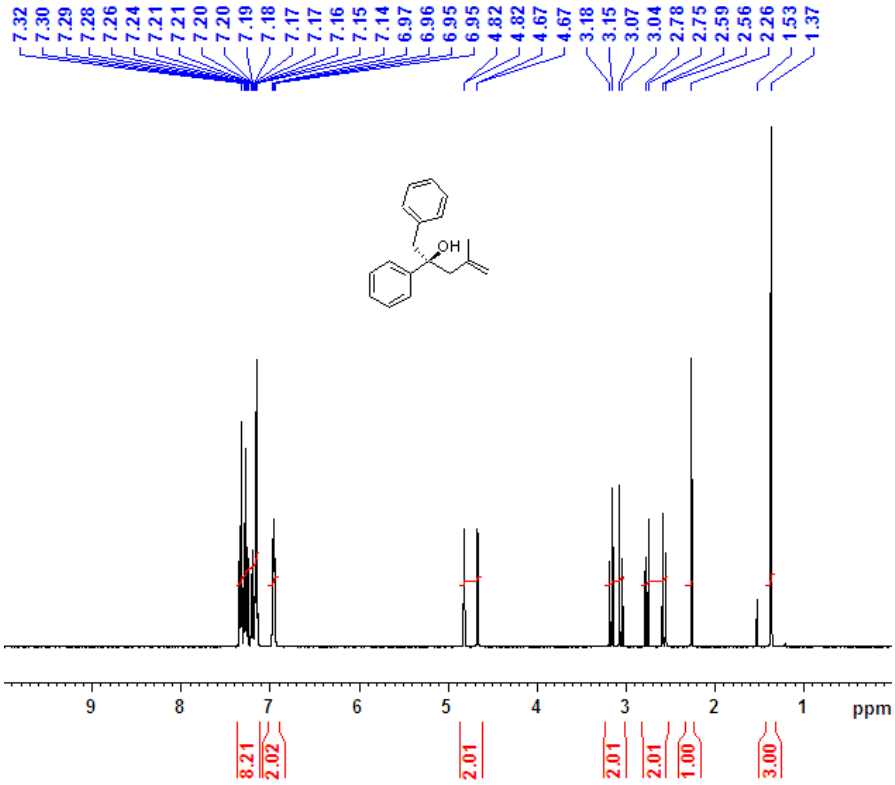
======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                  1H
PCPD2              90.00 usec
PL2                0.00 dB
PL12              15.86 dB
PL13              15.86 dB
PL2W         10.49968529 W
PL12W         0.27238068 W
PL13W         0.27238068 W
SFO2         400.1316005 MHz

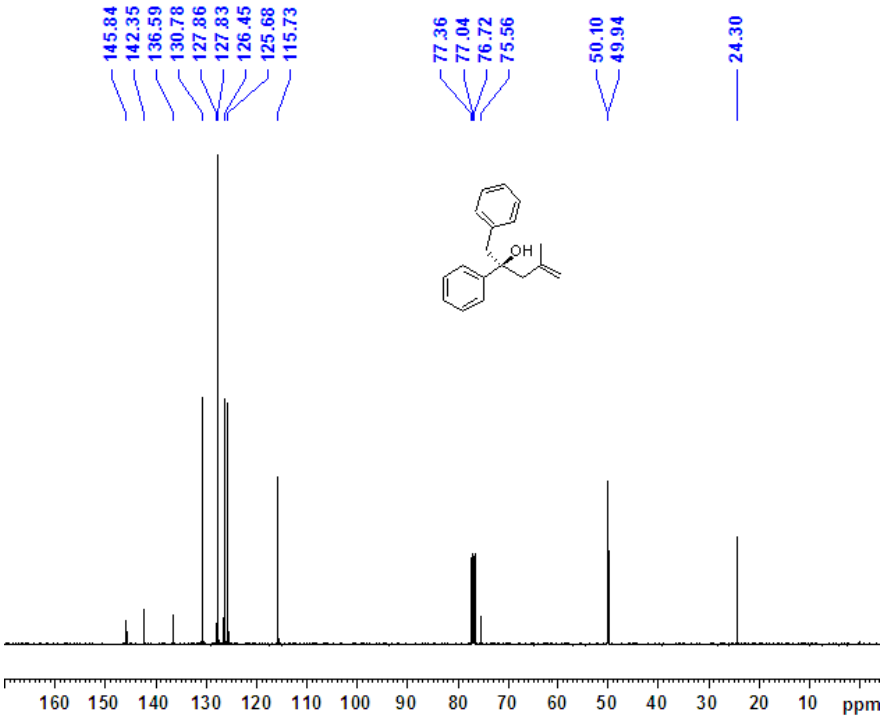
F2 - Processing parameters
SI                 65536
SF           100.6127690 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```

(*R*)-1-Chloro-5-methyl-3-phenylhex-5-en-3-ol 2

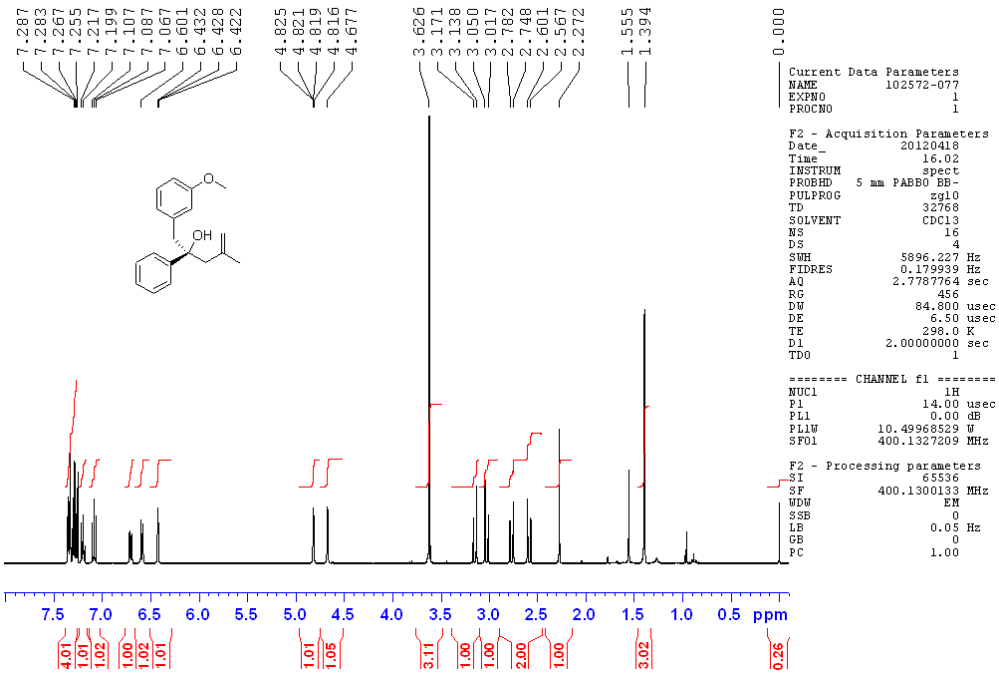


(*R*)-4-Methyl-1,2-diphenylpent-4-en-2-ol 12a





(*R*)-1-(3-Methoxyphenyl)-4-methyl-2-phenylpent-4-en-2-ol 12b

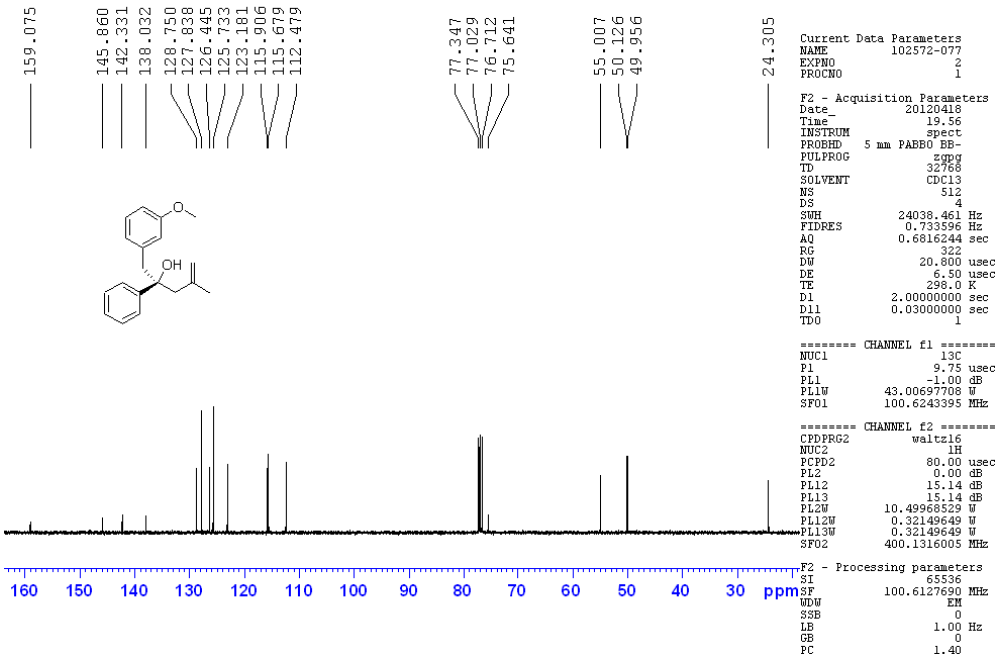



```
Current Data Parameters
NAME          102572-077
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20120418
Time               16.02
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zg10
TD                 32768
SOLVENT            CDCl3
NS                    16
DS                     4
SWH             5896.227 Hz
FIDRES          0.179939 Hz
AQ              2.7787764 sec
RG                   456
DW                84.800 usec
DE                 6.50 usec
TE                298.0 K
D1             2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                14.00 usec
PL1                0.00 dB
PL1W         10.49968529 W
SF01         400.1327209 MHz

F2 - Processing parameters
SI                 65536
SF          400.1300133 MHz
WDW                   EM
SSB                    0
LB                  0.05 Hz
GB                     0
PC                  1.00
```




```
Current Data Parameters
NAME          102572-077
EXPNO                  2
PROCNO                 1

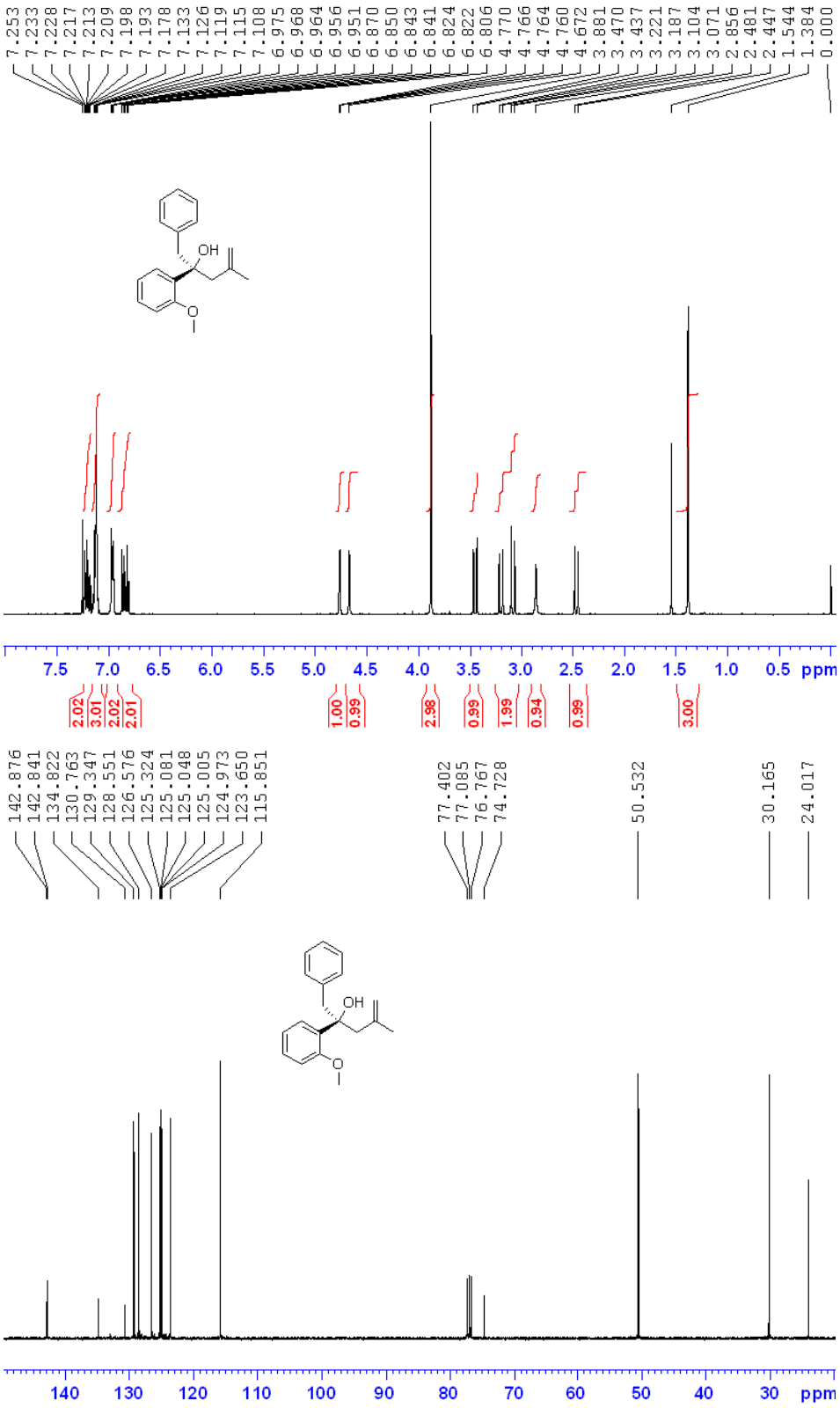
F2 - Acquisition Parameters
Date_           20120418
Time               19.56
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zgpg
TD                 32768
SOLVENT            CDCl3
NS                   512
DS                     4
SWH            24038.461 Hz
FIDRES          0.733596 Hz
AQ              0.6816244 sec
RG                   322
DW                20.800 usec
DE                 6.50 usec
TE                298.0 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                 9.75 usec
PL1               -1.00 dB
PL1W         43.00697708 W
SF01        100.6243395 MHz

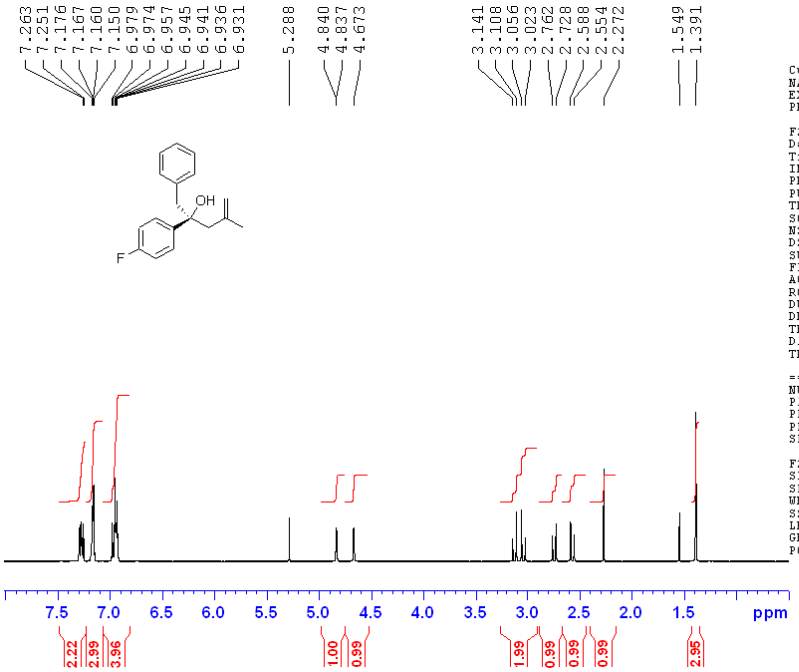
======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2             80.00 usec
PL2                0.00 dB
PL12              15.14 dB
PL13              15.14 dB
PL2W         10.49968529 W
PL12W         0.32149649 W
PL13W         0.32149649 W
SF02        400.1316005 MHz

F2 - Processing parameters
SI                 65536
SF          100.6127690 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```

(*R*)-2-(2-Methoxyphenyl)-4-methyl-1-phenylpent-4-en-2-ol 12c



(*R*)-2-(4-Fluorophenyl)-4-methyl-1-phenylpent-4-en-2-ol 12d

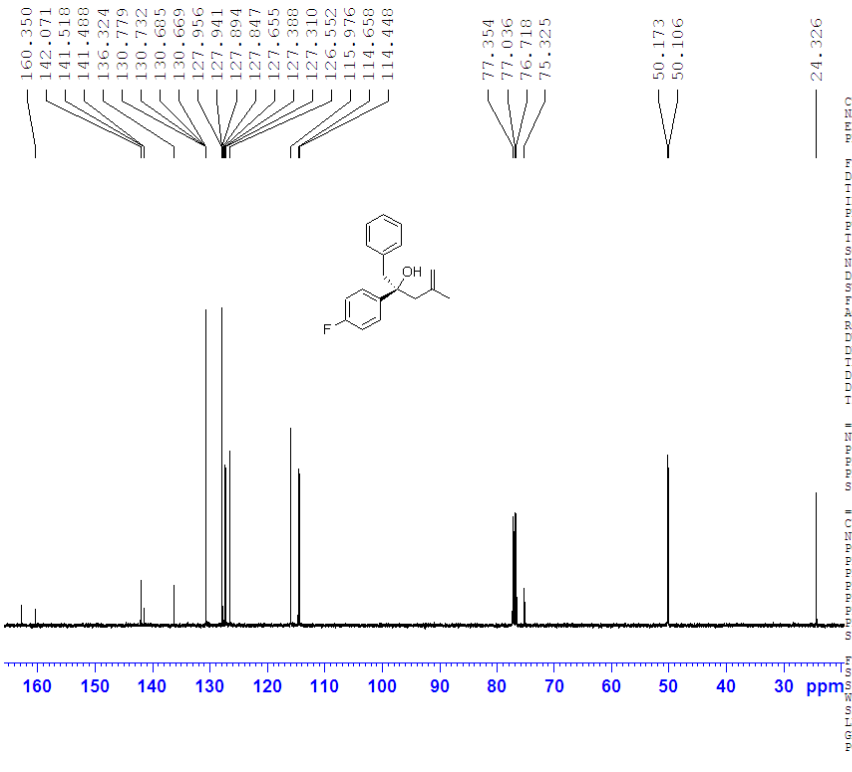



```
Current Data Parameters
NAME          102572-085
EXPNO                  3
PROCNO                 1

F2 - Acquisition Parameters
Date_           20120423
Time               12.57
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zg10
TD                 32768
SOLVENT            CDCl3
NS                    16
DS                     4
SWH             5896.227 Hz
FIDRES          0.179939 Hz
AQ              2.7787764 sec
RG                   406
DW                84.800 usec
DE                 6.50 usec
TE                298.0 K
D1             2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                14.00 usec
PL1                0.00 dB
PL1W         10.49968529 W
SFO1        400.1327209 MHz

F2 - Processing parameters
SI                 65536
SF          400.1300153 MHz
WDW                   EM
SSB                    0
LB                  0.05 Hz
GB                     0
PC                  1.00
```




```
Current Data Parameters
NAME          102572-085
EXPNO                  3
PROCNO                 1

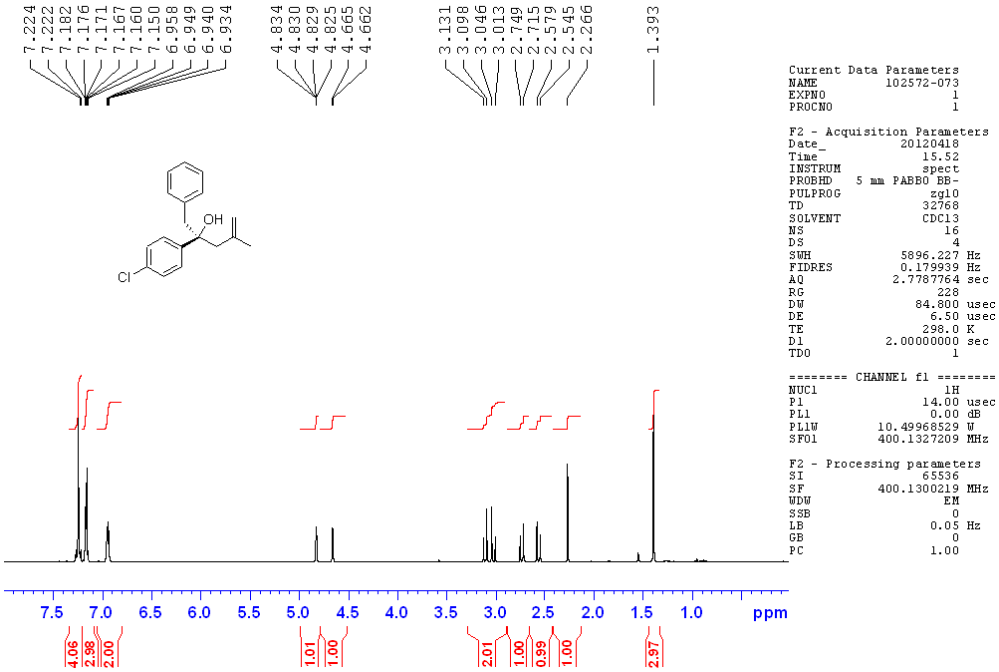
F2 - Acquisition Parameters
Date_           20120817
Time               15.56
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zgpg
TD                 32768
SOLVENT            CDCl3
NS                   512
DS                     4
SWH            24038.461 Hz
FIDRES          0.733596 Hz
AQ              0.6816244 sec
RG                   228
DW                20.800 usec
DE                 6.50 usec
TE                298.0 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                    1

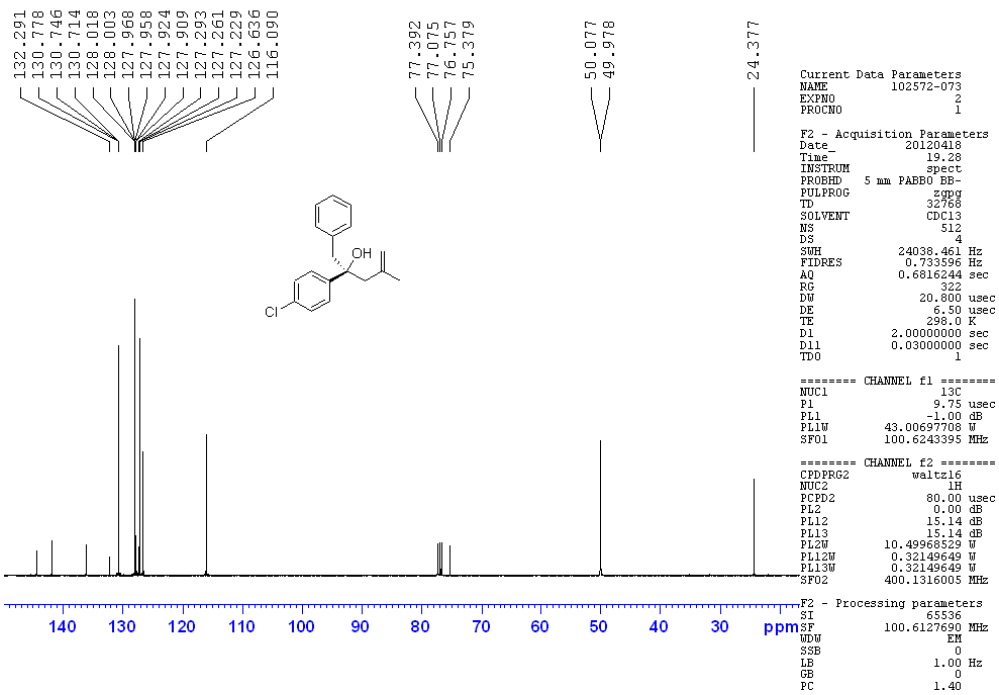
======== CHANNEL f1 ========
NUC1                 13C
P1                 9.65 usec
PL1               -1.00 dB
PL1W        43.00697708 W
SFO1       100.6243395 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2             90.00 usec
PL2                0.00 dB
PL12              15.86 dB
PL13              15.86 dB
PL2W        10.49968529 W
PL12W        0.27238068 W
PL13W        0.27238068 W
SFO2       400.1316005 MHz

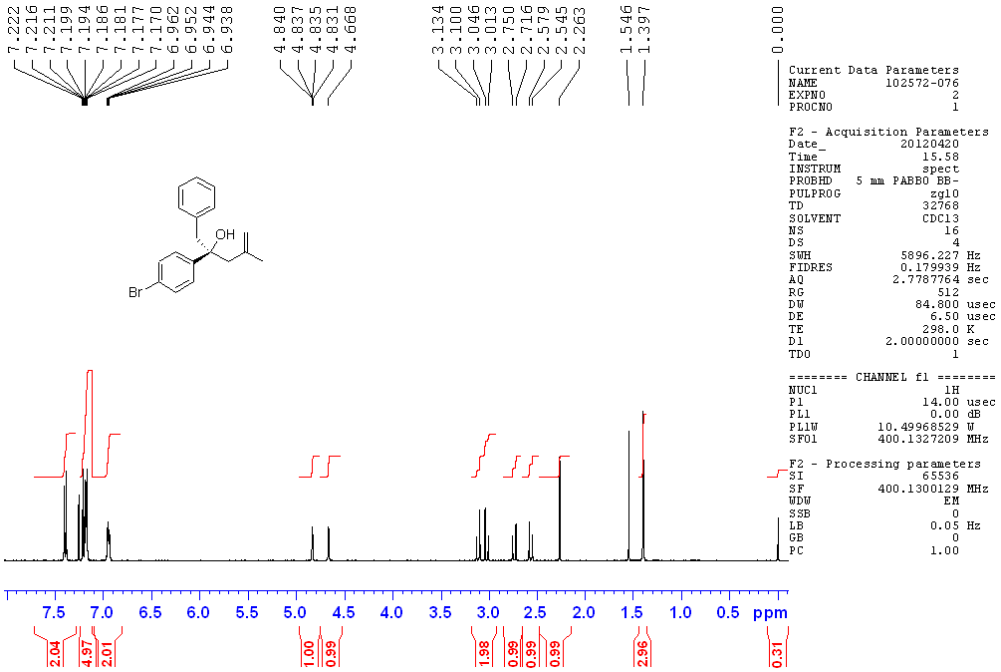
F2 - Processing parameters
SI                 65536
SF         100.6127690 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```

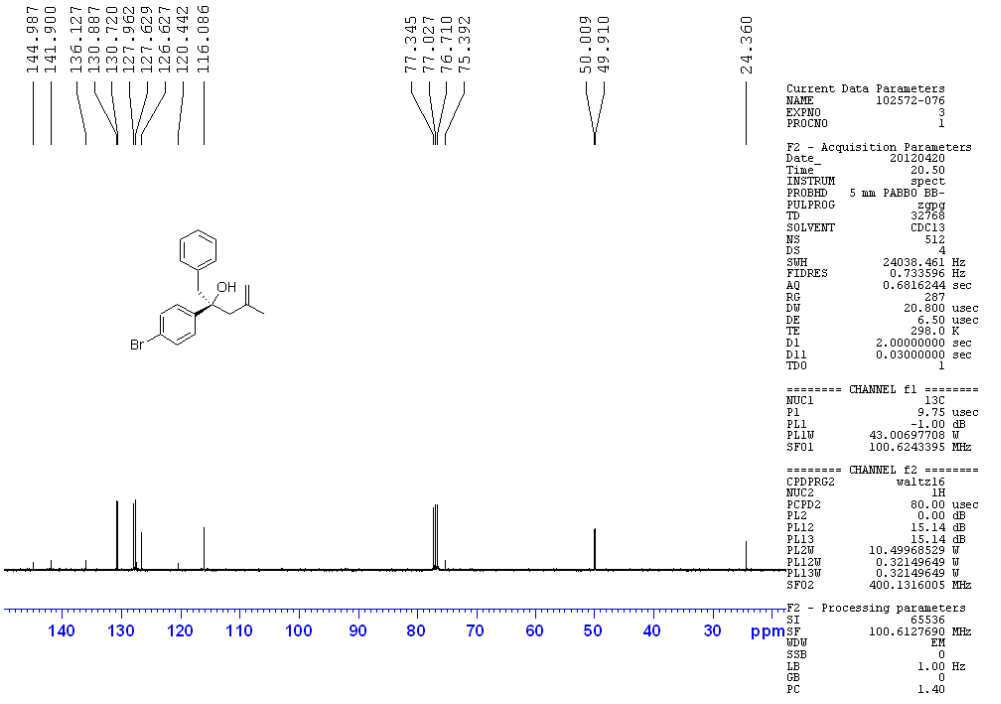
(*R*)-2-(4-Chlorophenyl)-4-methyl-1-phenylpent-4-en-2-ol 12e



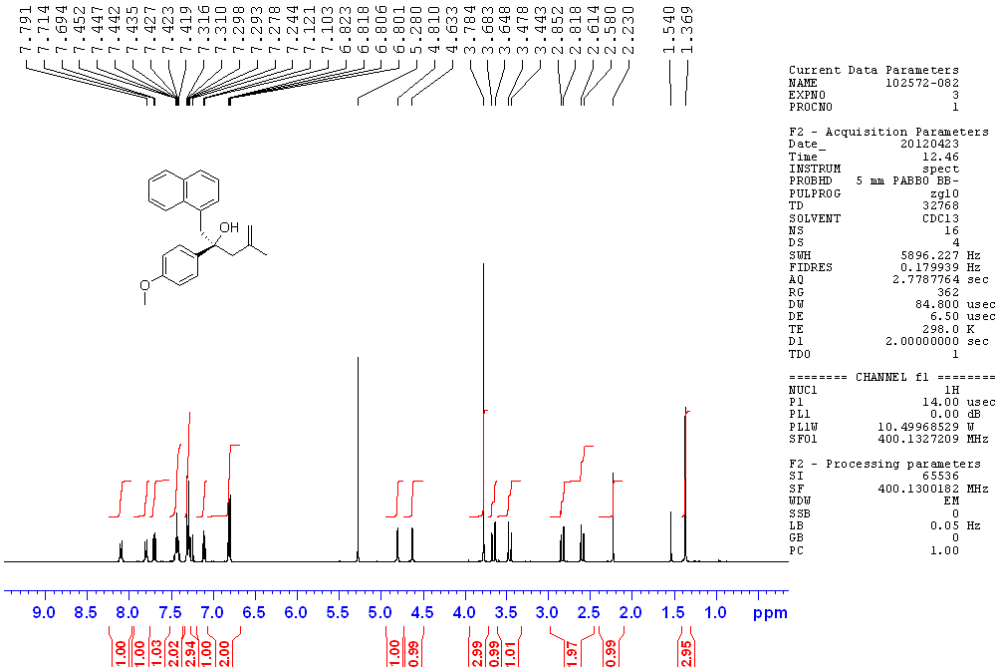


(*R*)-2-(4-Bromophenyl)-4-methyl-1-phenylpent-4-en-2-ol 12f





(*R*)-2-(4-Methoxyphenyl)-4-methyl-1-(naphthalen-1-yl)pent-4-en-2-ol 12g

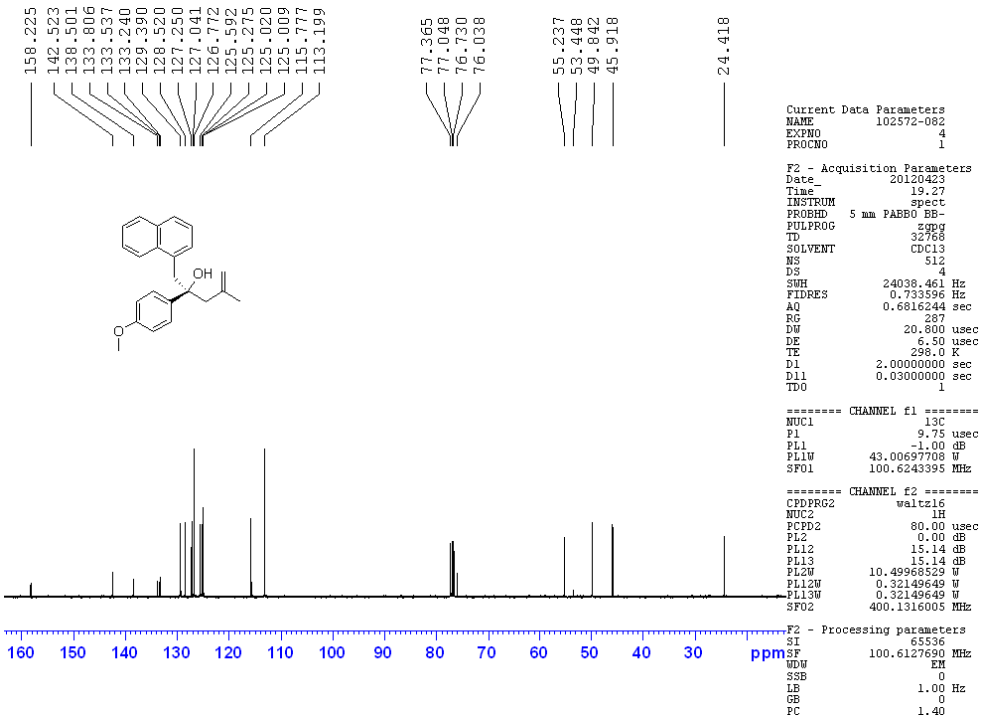



```
Current Data Parameters
NAME          102572-082
EXPNO                  3
PROCNO                 1

F2 - Acquisition Parameters
Date_           20120423
Time               12.46
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zg10
TD                 32768
SOLVENT            CDCl3
NS                    16
DS                     4
SWH             5896.227 Hz
FIDRES          0.179939 Hz
AQ             2.7787764 sec
RG                   362
DW                84.800 usec
DE                 6.50 usec
TE                298.0 K
D1            2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 14.00 usec
PL1                 0.00 dB
PL1W         10.49968529 W
SF01        400.1327209 MHz

F2 - Processing parameters
SI                 65536
SF          400.1300182 MHz
WDW                   EM
SSB                    0
LB                  0.05 Hz
GB                     0
PC                  1.00
```




```
Current Data Parameters
NAME          102572-082
EXPNO                  4
PROCNO                 1

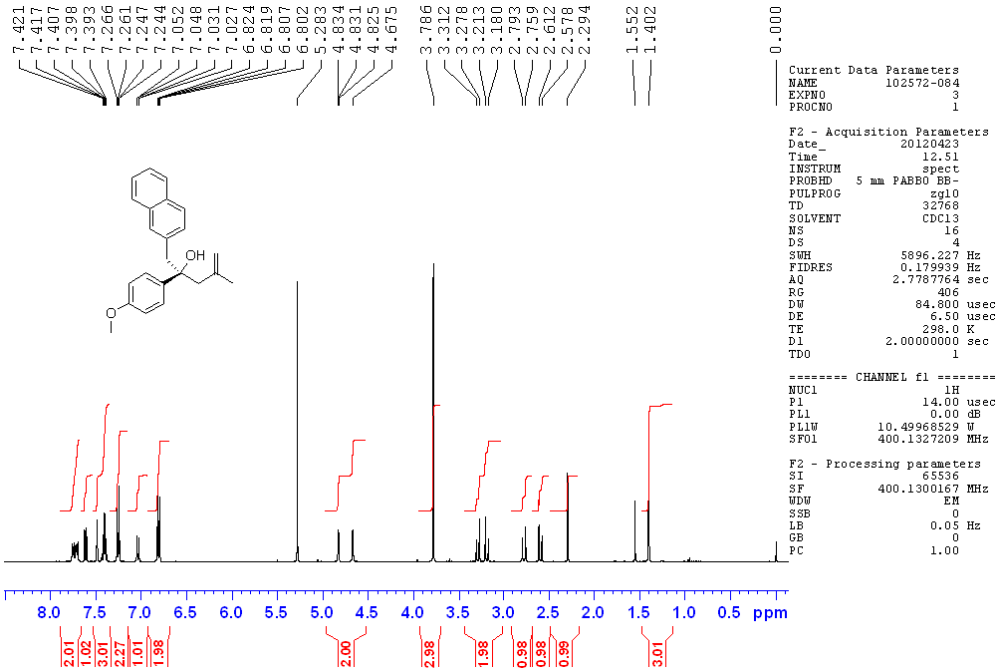
F2 - Acquisition Parameters
Date_           20120423
Time               19.27
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zgpg
TD                 32768
SOLVENT            CDCl3
NS                   512
DS                     4
SWH            24038.461 Hz
FIDRES          0.733596 Hz
AQ             0.6816244 sec
RG                   287
DW                20.800 usec
DE                 6.50 usec
TE                298.0 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                  9.75 usec
PL1                -1.00 dB
PL1W         43.00697708 W
SF01        100.6243395 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2              80.00 usec
PL2                 0.00 dB
PL12               15.14 dB
PL13               15.14 dB
PL2W         10.49968529 W
PL12W         0.32149649 W
PL13W         0.32149649 W
SF02        400.1316005 MHz

F2 - Processing parameters
SI                 65536
SF          100.6127690 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```

(*R*)-2-(4-Methoxyphenyl)-4-methyl-1-(naphthalen-2-yl)pent-4-en-2-ol 12h

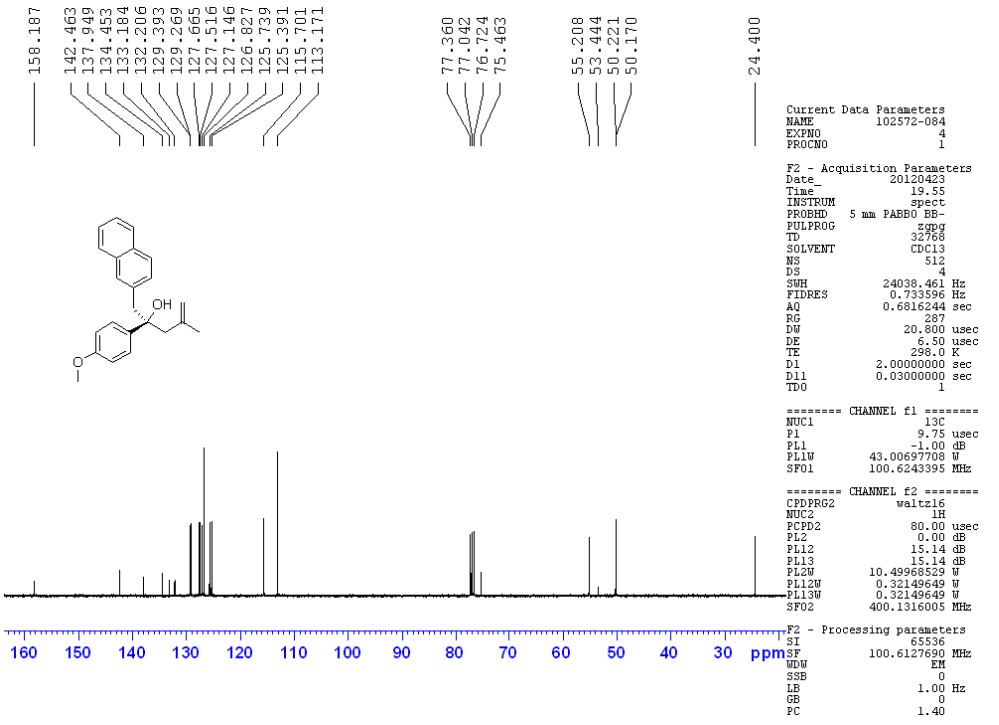



```
Current Data Parameters
NAME          102572-084
EXPNO                  3
PROCNO                 1

F2 - Acquisition Parameters
Date_           20120423
Time               12.51
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zg10
TD                 32768
SOLVENT            CDCl3
NS                    16
DS                     4
SWH             5896.227 Hz
FIDRES          0.179939 Hz
AQ              2.7787764 sec
RG                   406
DW                84.800 usec
DE                 6.50 usec
TE                298.0 K
D1             2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 14.00 usec
PL1                 0.00 dB
PL1W         10.49968529 W
SF01        400.1327209 MHz

F2 - Processing parameters
SI                 65536
SF          400.1300167 MHz
WDW                   EM
SSB                    0
LB                  0.05 Hz
GB                     0
PC                  1.00
```




```
Current Data Parameters
NAME          102572-084
EXPNO                  4
PROCNO                 1

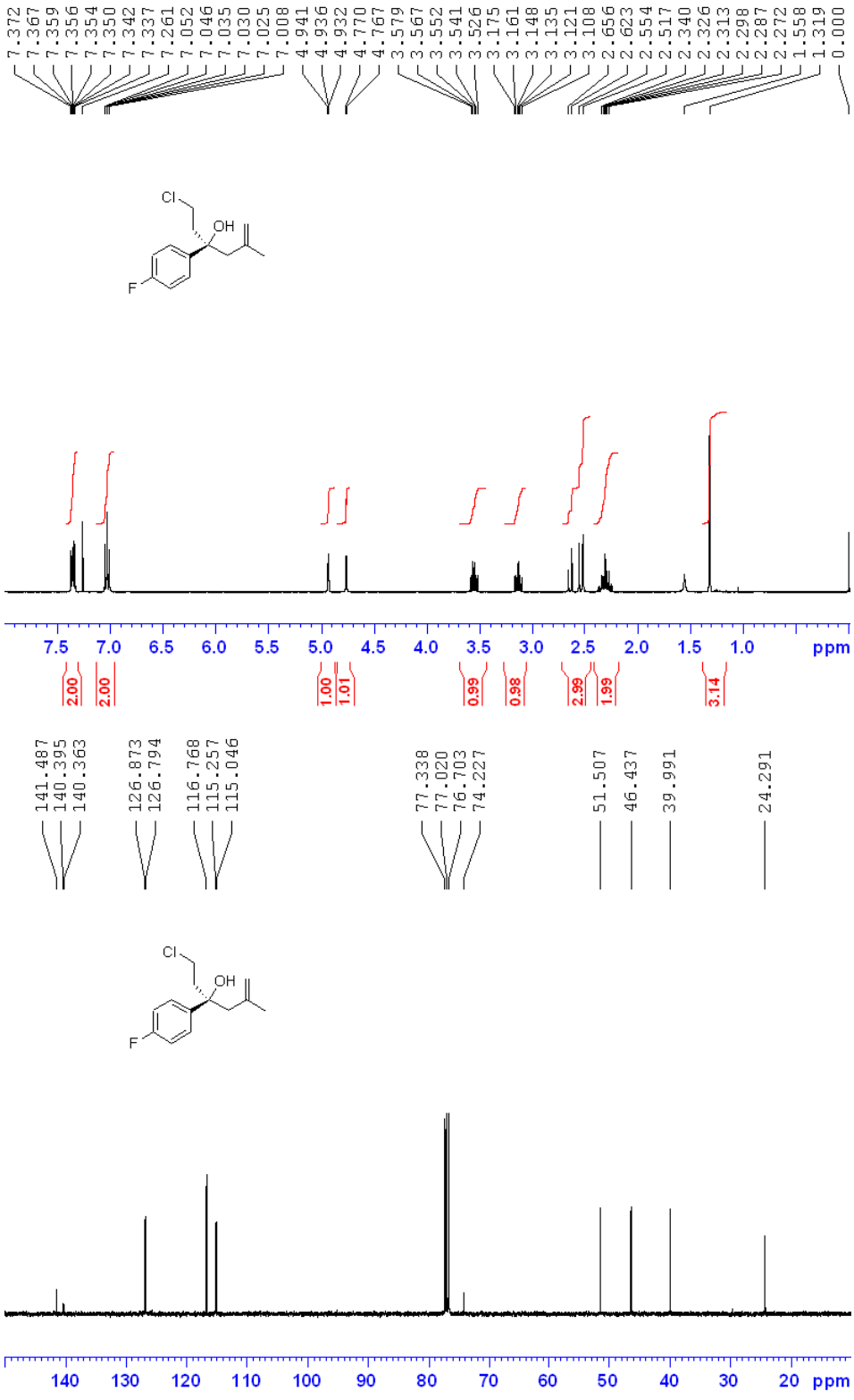
F2 - Acquisition Parameters
Date_           20120423
Time               19.55
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zgpg
TD                 32768
SOLVENT            CDCl3
NS                   512
DS                     4
SWH            24038.461 Hz
FIDRES          0.733596 Hz
AQ              0.6816244 sec
RG                   287
DW                20.800 usec
DE                 6.50 usec
TE                298.0 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                  9.75 usec
PL1                -1.00 dB
PL1W         43.00697708 W
SF01        100.6243395 MHz

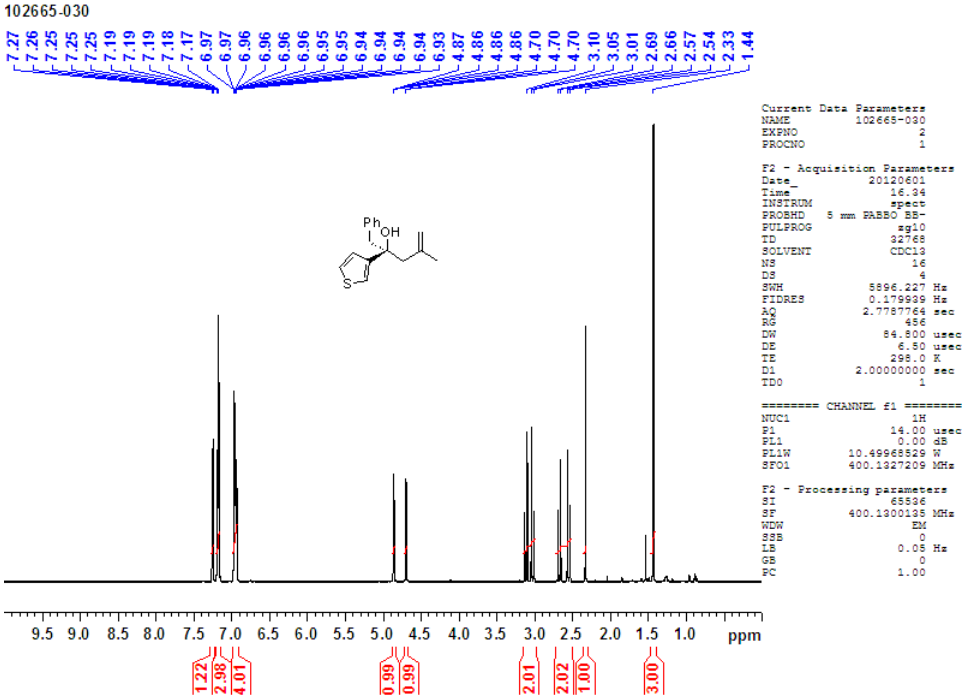
======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2              80.00 usec
PL2                 0.00 dB
PL12               15.14 dB
PL13               15.14 dB
PL2W         10.49968529 W
PL12W         0.32149649 W
PL13W         0.32149649 W
SF02        400.1316005 MHz

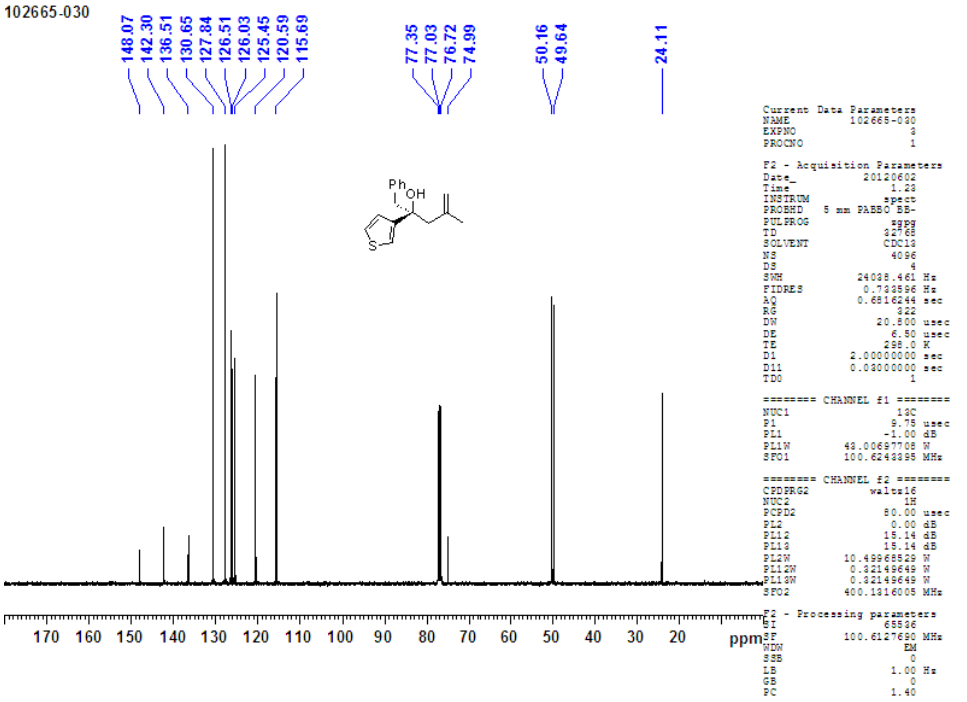
F2 - Processing parameters
SI                 65536
SF          100.6127690 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```

(*R*)-1-Chloro-3-(4-fluorophenyl)-5-methylhex-5-en-3-ol 12i

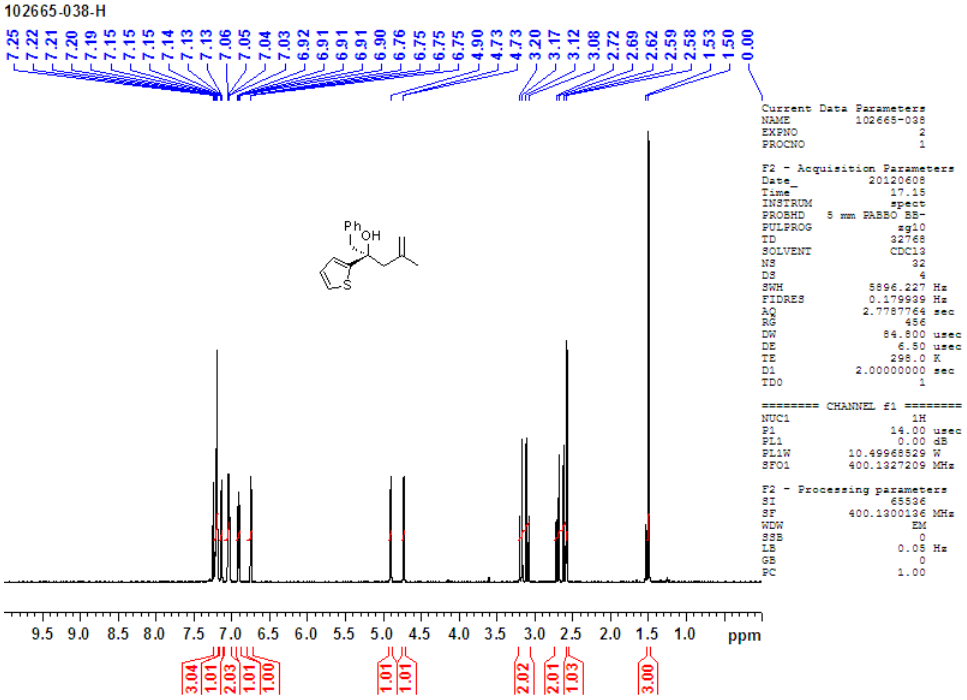


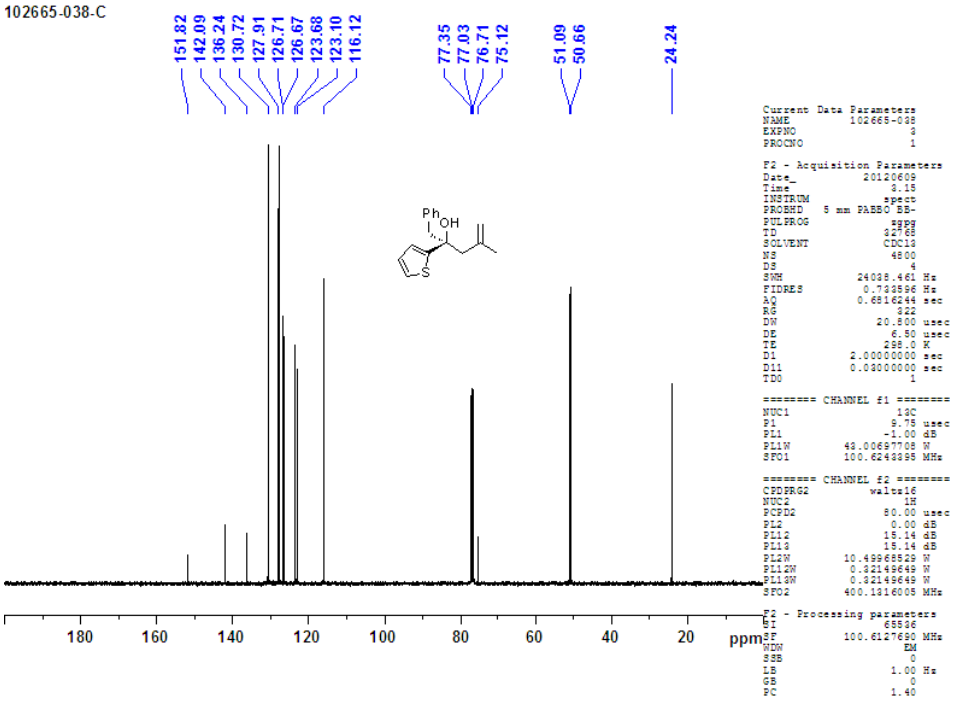
(*R*)-4-Methyl-1-phenyl-2-(thiophen-3-yl)pent-4-en-2-ol 12j



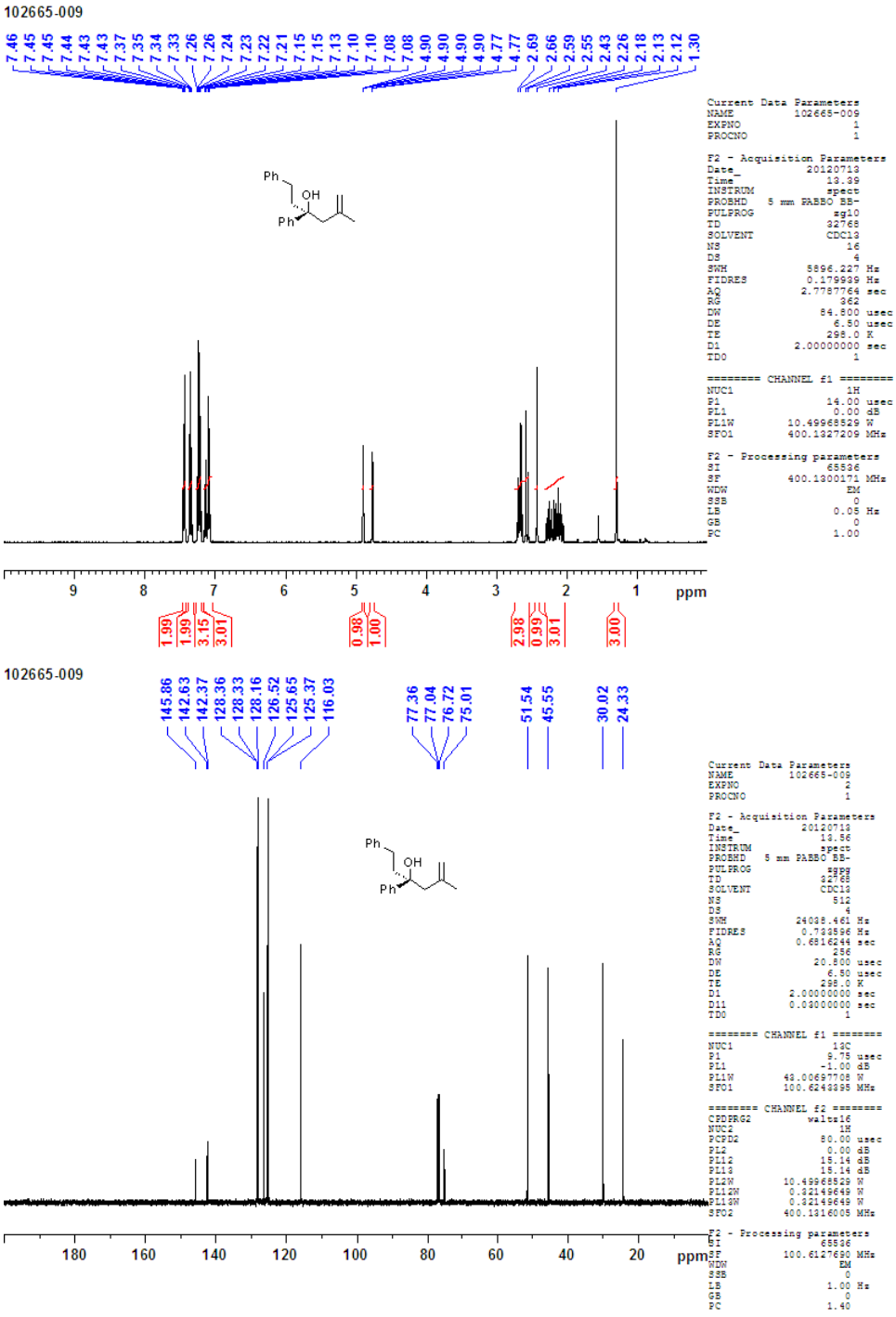


(*R*)-4-Methyl-1-phenyl-2-(thiophen-3-yl)pent-4-en-2-ol 12k

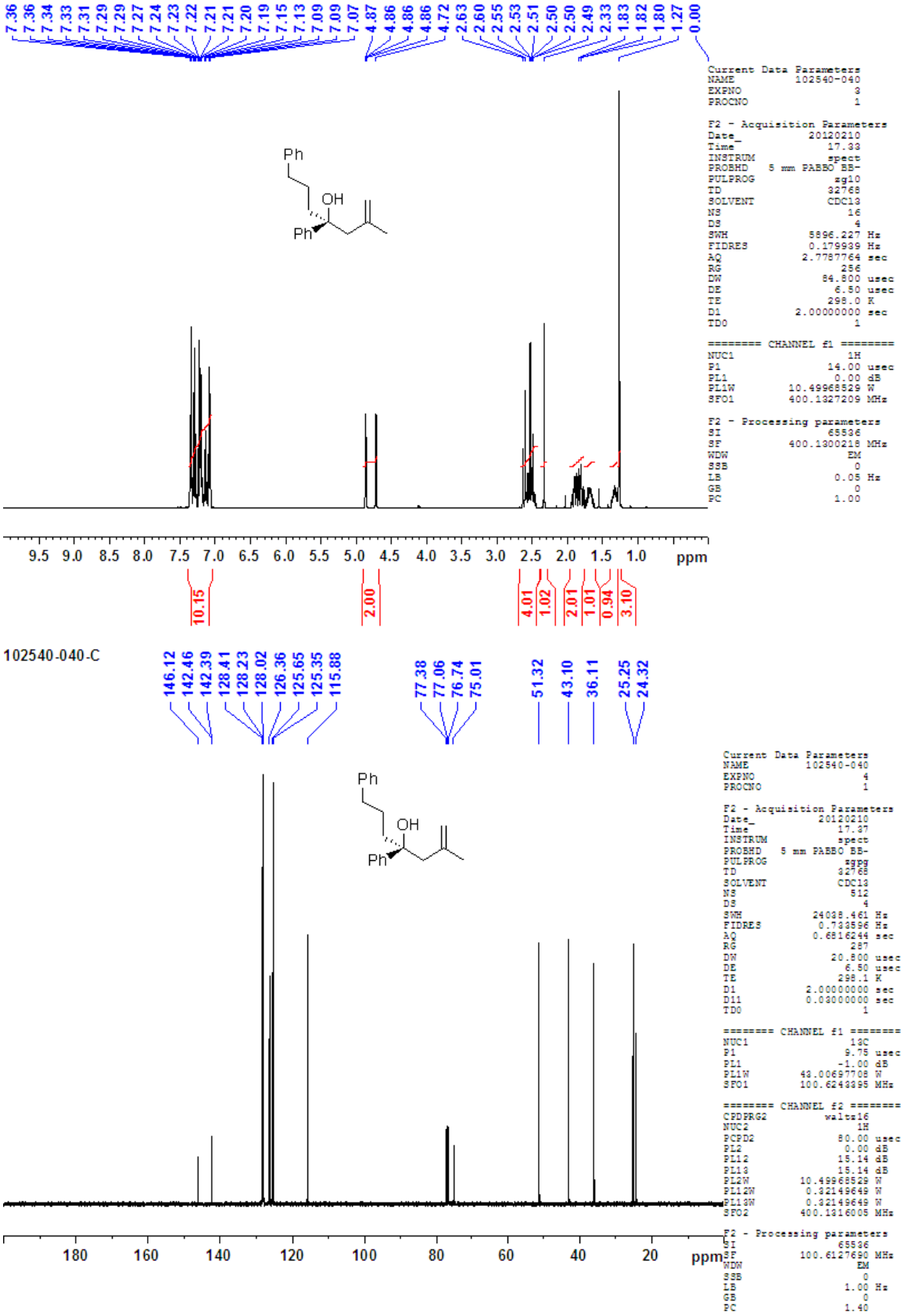




(*S*)-5-Methyl-1,3-diphenylhex-5-en-3-ol 12l



(*S*)-2-Methyl-4,7-diphenylhept-1-en-4-ol 12m




```
Current Data Parameters
NAME                  102540-040
EXPNO                          3
PROCNO                         1

F2 - Acquisition Parameters
Date_                   20120210
Time                       17.33
INSTRUM                    spect
PROBHD     5 mm PABBO BB-
PULPROG                    zg10
TD                        32768
SOLVENT                   CDCl3
NS                           16
DS                            4
SWH                    5896.227 Hz
FIDRES                 0.179939 Hz
AQ                     2.7787764 sec
RG                          256
DW                       84.800 usec
DE                        6.50 usec
TE                       298.0 K
D1                    2.00000000 sec
TD0                            1

======== CHANNEL f1 ========
NUC1                         1H
P1                        14.00 usec
PL1                        0.00 dB
PL1W                10.49968529 W
SFO1                400.1327209 MHz

F2 - Processing parameters
SI                         65536
SF                  400.1300218 MHz
WDW                           EM
SSB                            0
LB                         0.05 Hz
GB                             0
PC                          1.00
```

```
Current Data Parameters
NAME                  102540-040
EXPNO                          4
PROCNO                         1

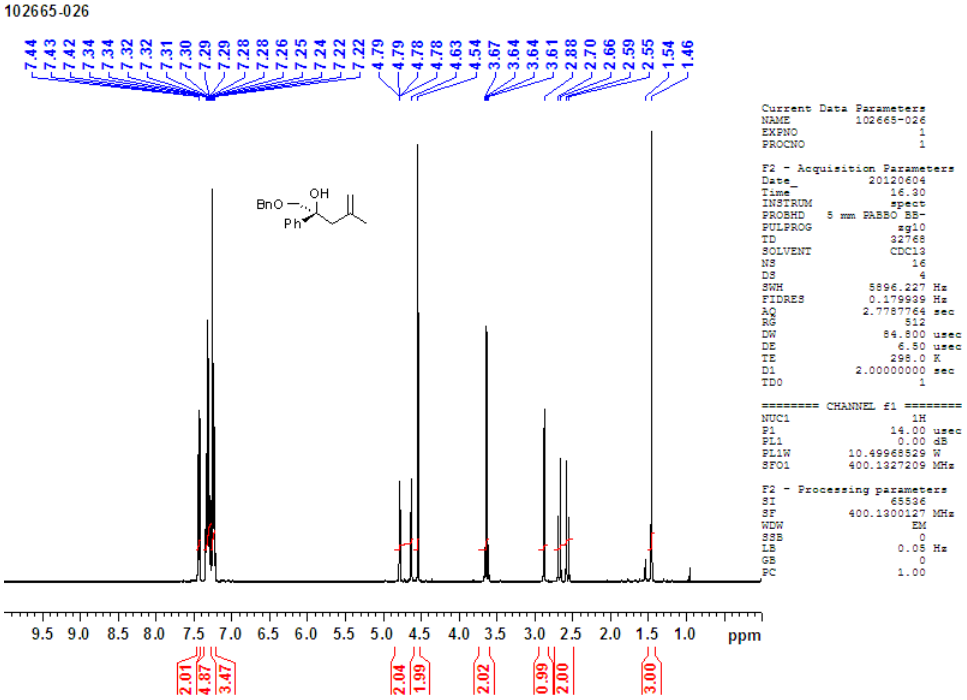
F2 - Acquisition Parameters
Date_                   20120210
Time                       17.37
INSTRUM                    spect
PROBHD     5 mm PABBO BB-
PULPROG                  zgpg
TD                        32768
SOLVENT                   CDCl3
NS                           512
DS                            4
SWH                   24038.461 Hz
FIDRES                 0.733596 Hz
AQ                     0.6816244 sec
RG                          287
DW                       20.800 usec
DE                        6.50 usec
TE                       298.1 K
D1                    2.00000000 sec
D11                   0.03000000 sec
TD0                            1

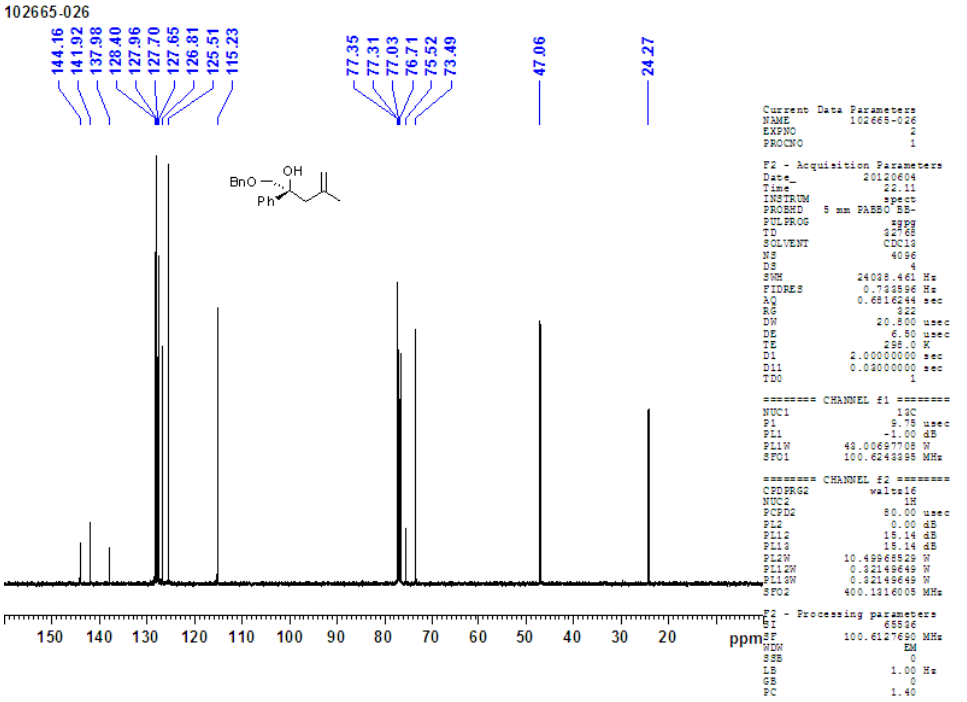
======== CHANNEL f1 ========
NUC1                         13C
P1                         9.75 usec
PL1                       -1.00 dB
PL1W                43.00697708 W
SFO1                100.6243395 MHz

======== CHANNEL f2 ========
CPDPRG2                  waltz16
NUC2                         1H
PCPD2                     80.00 usec
PL2                        0.00 dB
PL12                      15.14 dB
PL13                      15.14 dB
PL2W                10.49968529 W
PL12W                0.32149649 W
PL13W                0.32149649 W
SFO2                400.1316005 MHz

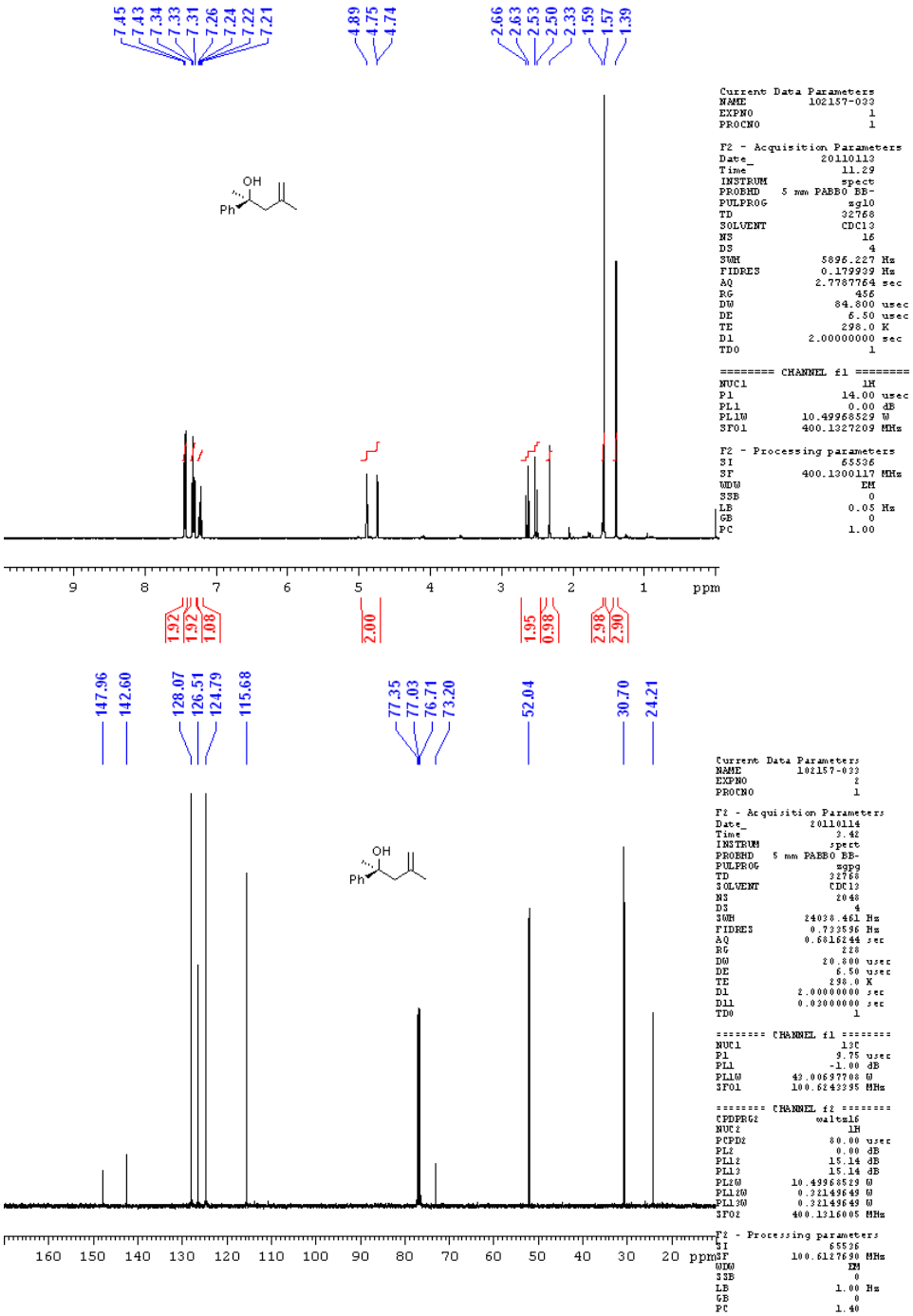
F2 - Processing parameters
SI                         65536
SF                  100.6127690 MHz
WDW                           EM
SSB                            0
LB                         1.00 Hz
GB                             0
PC                          1.40
```

(*R*)-1-(Benzyloxy)-4-methyl-2-phenylpent-4-en-2-ol 12n





(S)-4-Methyl-2-phenylpent-4-en-2-ol 12o



Current Data Parameters
NAME 102157-033
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20110113
Time 11.29
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg10
TD 32768
SOLVENT CDCl3
NS 16
DS 4
SWH 5896.227 Hz
FIDRES 0.179939 Hz
AQ 2.7787764 sec
RG 456
DW 84.800 usec
DE 6.50 usec
TE 298.0 K
D1 2.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 14.00 usec
PL1 0.00 dB
PL1W 10.49968529 W
SFO1 400.1327209 MHz

F2 - Processing parameters
SI 65536
SF 400.1300117 MHz
WDW EM
SSB 0
LB 0.05 Hz
GB 0
PC 1.00

Current Data Parameters
NAME 102157-033
EXPNO 2
PROCNO 1

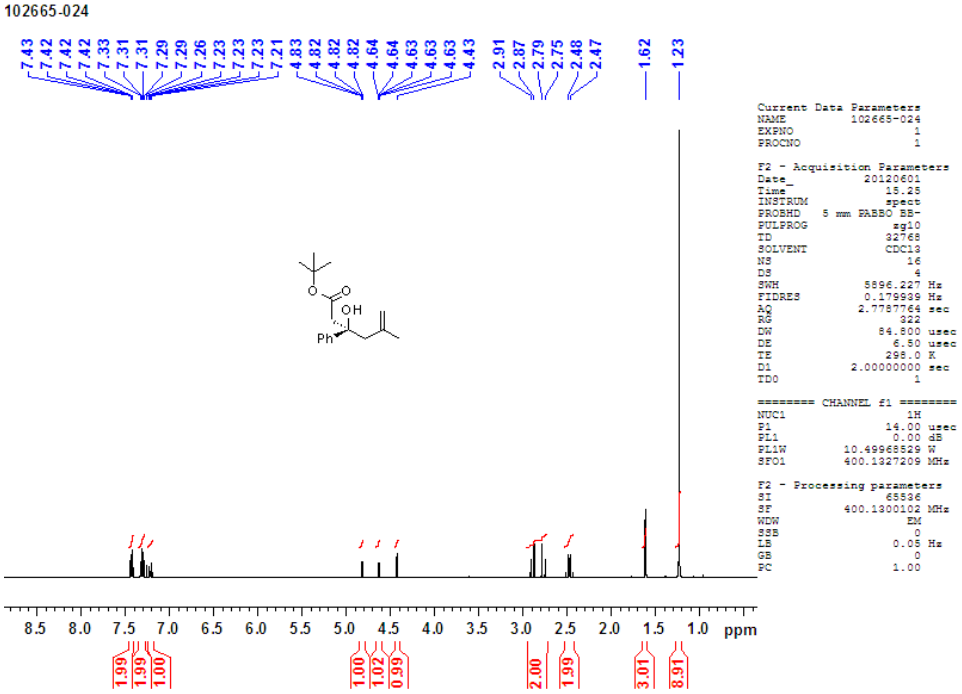
F2 - Acquisition Parameters
Date_ 20110114
Time 2.42
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg
TD 32768
SOLVENT CDCl3
NS 2048
DS 4
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 0.6816244 sec
RG 228
DW 20.800 usec
DE 6.50 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

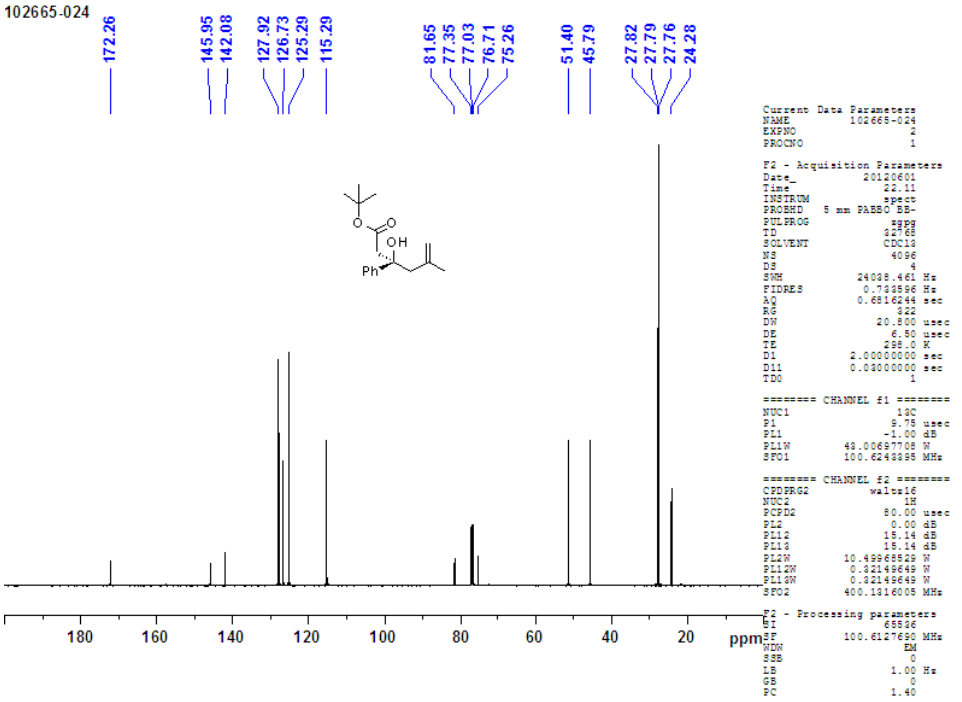
======== CHANNEL f1 ========
NUC1 13C
P1 9.75 usec
PL1 -1.00 dB
PL1W 42.00697708 W
SFO1 100.6243295 MHz

======== CHANNEL f2 ========
CPDPRG2 waltm16
NUC2 1H
PCPD2 80.00 usec
PL2 0.00 dB
PL12 15.14 dB
PL13 15.14 dB
PL2W 10.49968529 W
PL12W 0.3214964g W
PL13W 0.3214964g W
SFO2 400.1316005 MHz

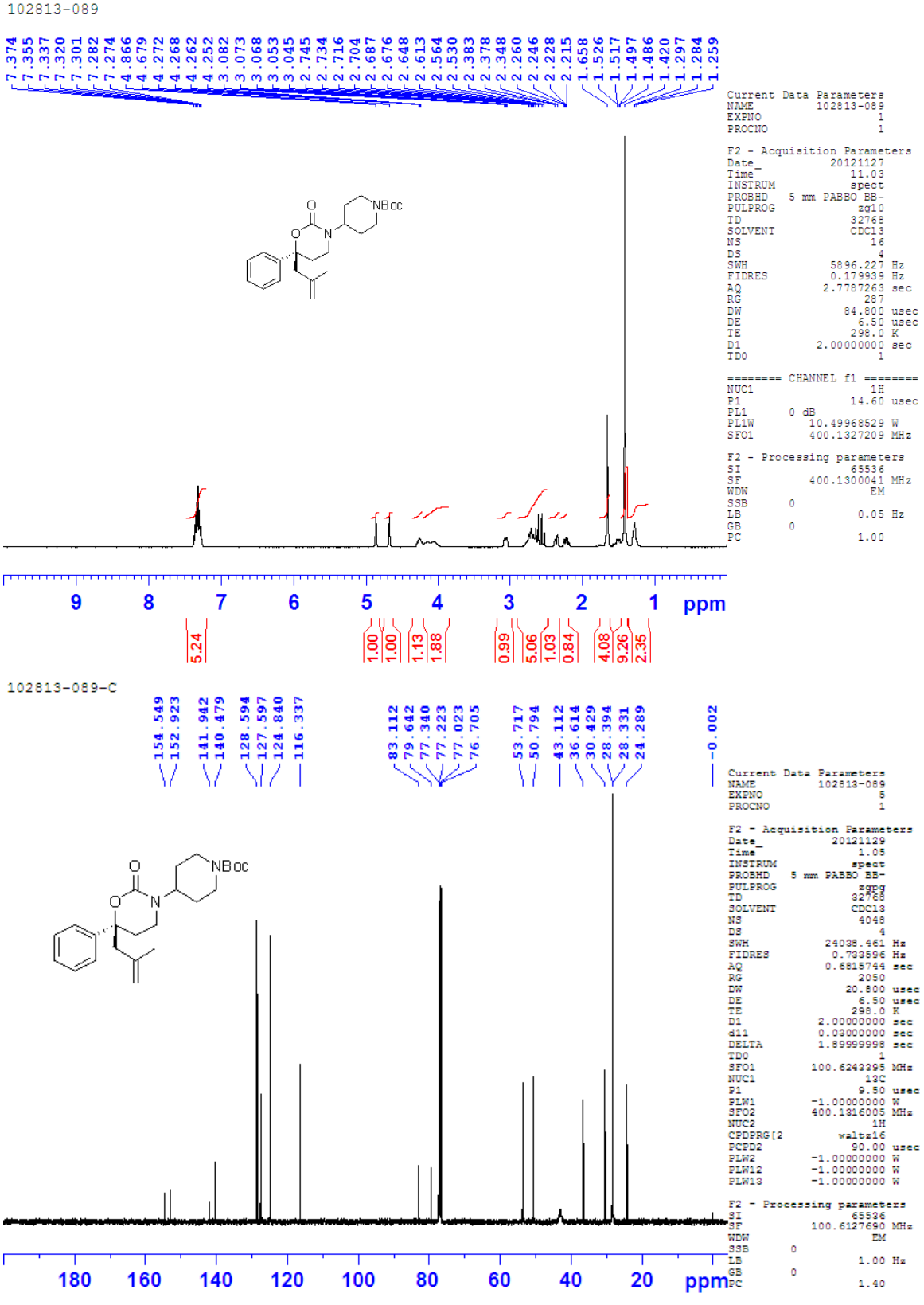
F2 - Processing parameters
SI 65536
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

(*R*)-tert-Butyl 3-hydroxy-5-methyl-3-phenylhex-5-enoate 12p



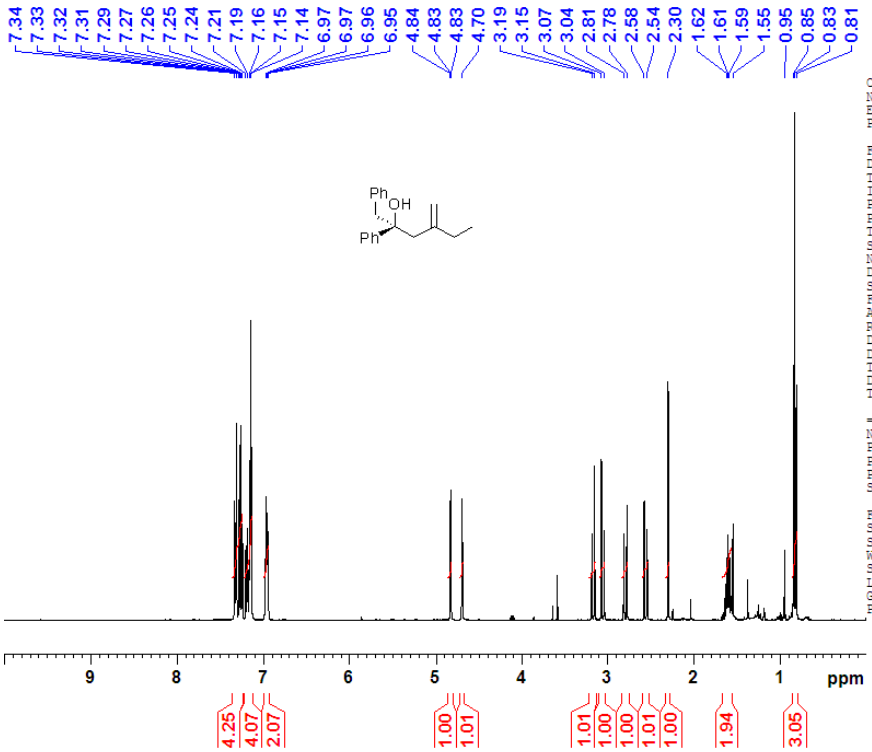


(*R*)-tert-butyl 4-(6-(2-methylallyl)-2-oxo-6-phenyl-1,3-oxazinan-3-yl)piperidine-1-carboxylate 1



(*R*)-4-Methylene-1,2-diphenylhexan-2-ol 16

102615-046-H



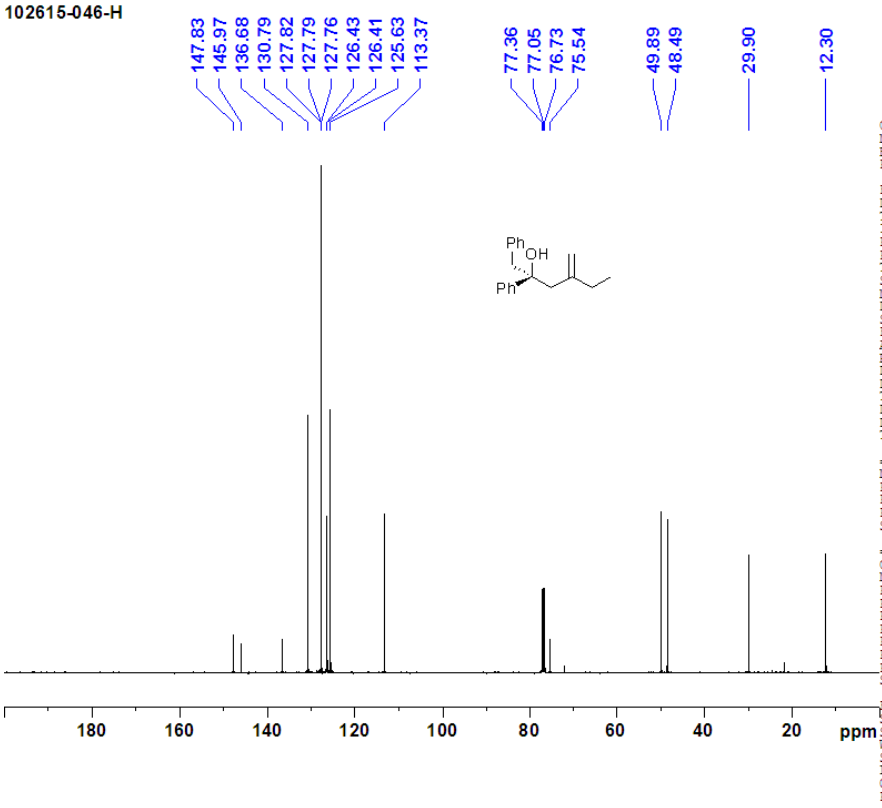

```
Current Data Parameters
NAME          102615-046
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_          20120824
Time             16.48
INSTRUM          spect
PROBHD   5 mm PABBO BB-
PULPROG          zg10
TD              32768
SOLVENT          CDCl3
NS                  16
DS                   4
SWH           5896.227 Hz
FIDRES        0.179939 Hz
AQ            2.7787764 sec
RG                 322
DW              84.800 usec
DE               6.50 usec
TE              298.0 K
D1          2.00000000 sec
TD0                  1

======== CHANNEL f1 ========
NUC1                1H
P1               13.80 usec
PL1               0.00 dB
PL1W       10.49968529 W
SFO1       400.1327209 MHz

F2 - Processing parameters
SI                65536
SF         400.1300168 MHz
WDW                  EM
SSB                   0
LB                 0.05 Hz
GB                    0
PC                 1.00
```

102615-046-H




```
Current Data Parameters
NAME          102615-046
EXPNO                  4
PROCNO                 1

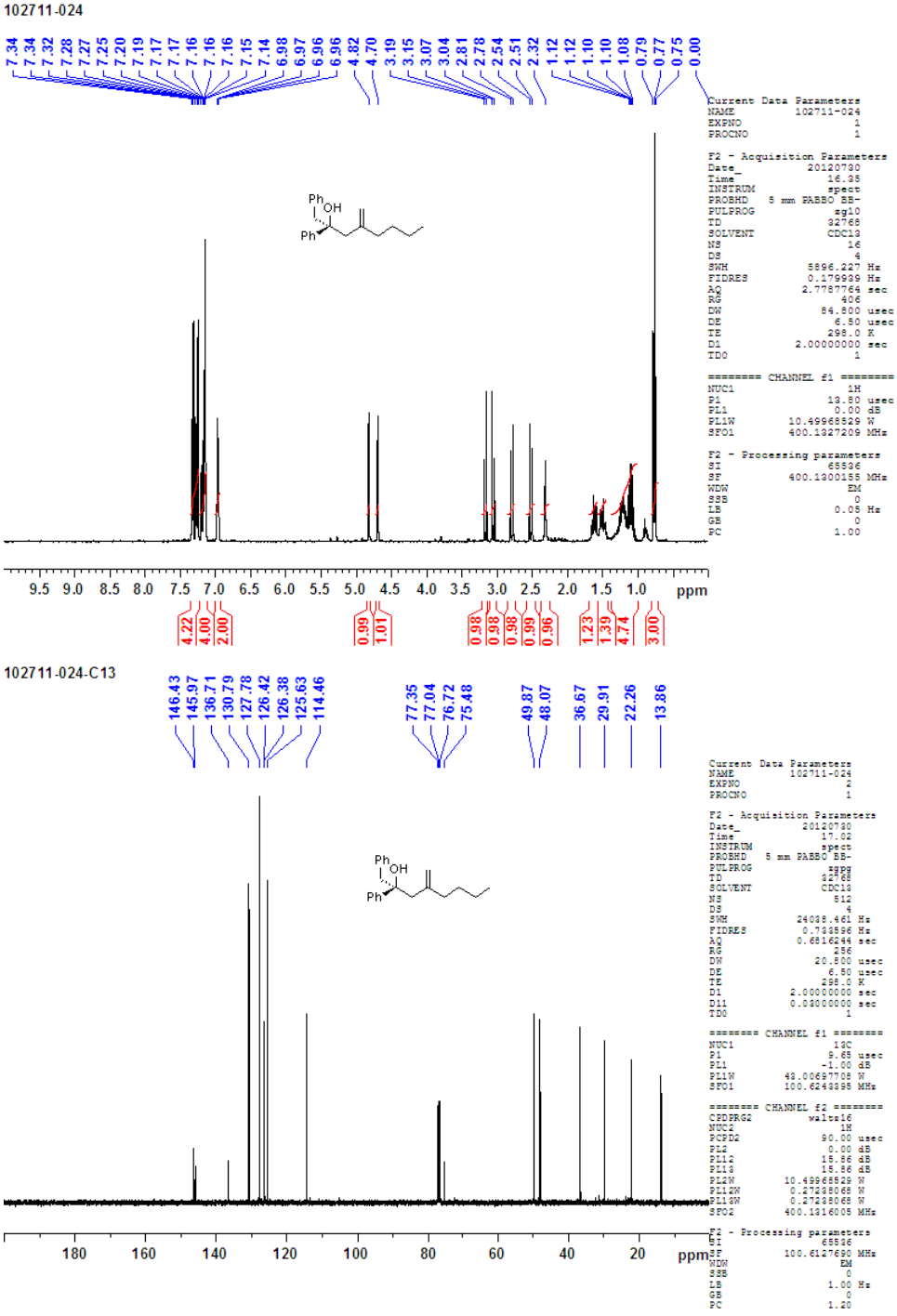
F2 - Acquisition Parameters
Date_          20120825
Time             23.43
INSTRUM          spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg
TD              32768
SOLVENT          CDCl3
NS                4096
DS                   4
SWH          24038.461 Hz
FIDRES        0.733596 Hz
AQ            0.6816244 sec
RG                 203
DW              20.800 usec
DE               6.50 usec
TE              298.0 K
D1          2.00000000 sec
D11         0.03000000 sec
TD0                  1

======== CHANNEL f1 ========
NUC1               13C
P1                9.65 usec
PL1              -1.00 dB
PL1W       43.00697708 W
SFO1       100.6243395 MHz

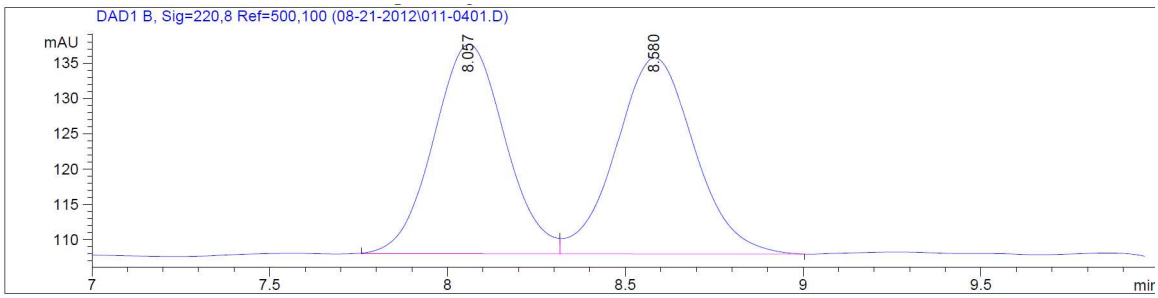
======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                1H
PCPD2           90.00 usec
PL2               0.00 dB
PL12             15.86 dB
PL13             15.86 dB
PL2W       10.49968529 W
PL12W       0.27238068 W
PL13W       0.27238068 W
SFO2       400.1316005 MHz

F2 - Processing parameters
SI                65536
SF         100.6127690 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```

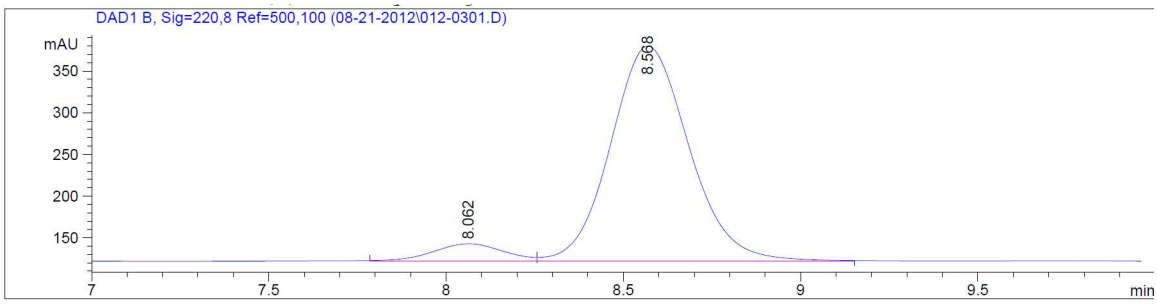
(*R*)-4-Methylene-1,2-diphenyloctan-2-ol 17



(*R*)-4-Methyl-1,2-diphenylpent-4-en-2-ol 12a

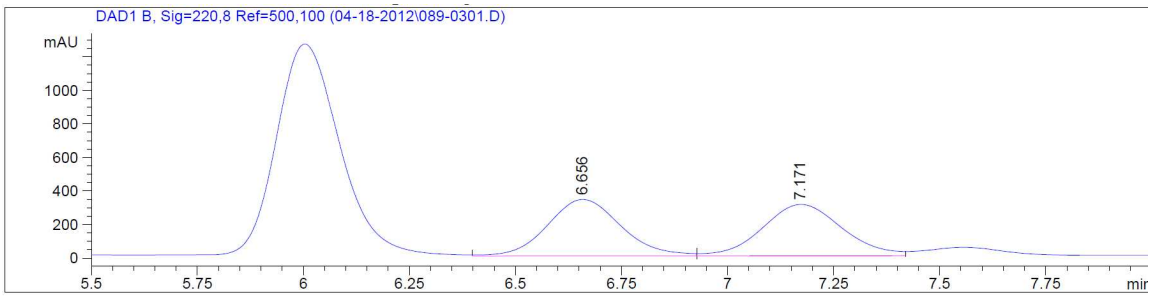


Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	8.057	BV	0.2172	414.81424	29.64688	49.2910
2	8.580	VB	0.2353	426.74731	27.76923	50.7090
Totals :				841.56155	57.41611	

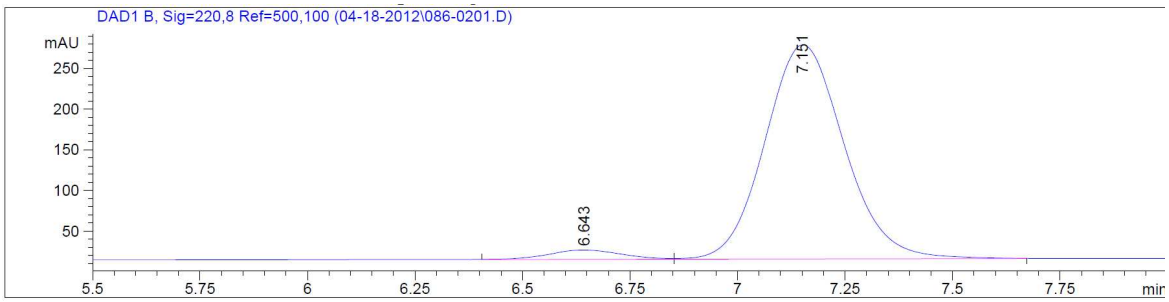


Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	8.062	BV	0.2102	276.80423	20.41046	6.5591
2	8.568	VB	0.2346	3943.35522	257.55811	93.4409
Totals :				4220.15945	277.96856	

(*R*)-1-(3-Methoxyphenyl)-4-methyl-2-phenylpent-4-en-2-ol 12b

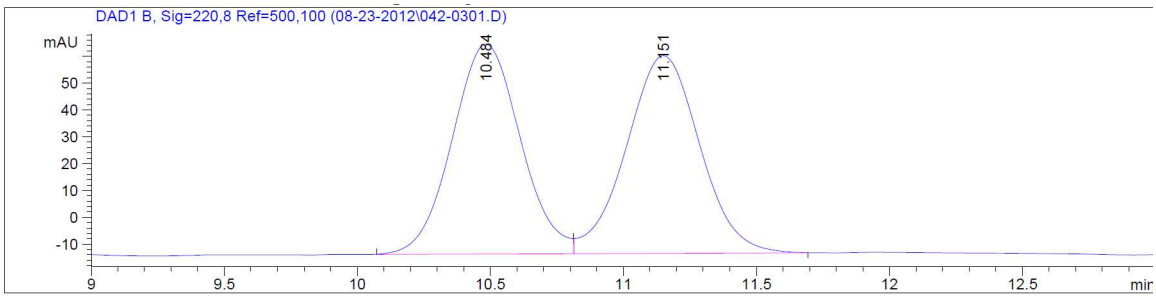


Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.656	VV	0.1869	4072.97974	337.28018	50.3854
2	7.171	VV	0.2027	4010.67310	306.36887	49.6146
Totals :				8083.65283	643.64905	

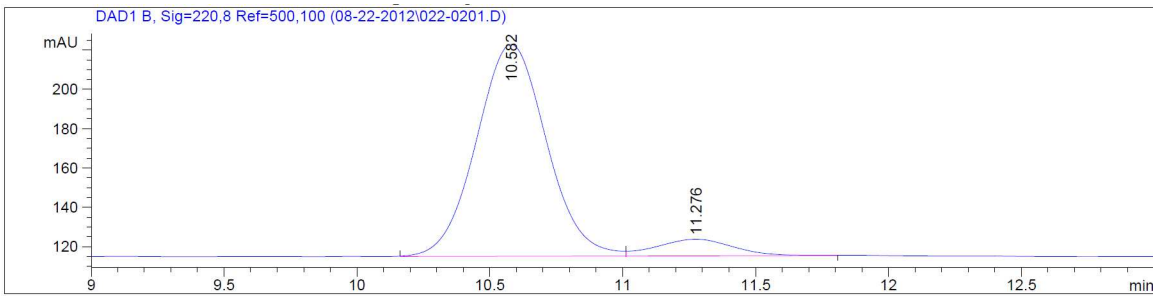


Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.643	BV	0.1809	135.64973	11.55721	3.8567
2	7.151	VB	0.1977	3381.61890	263.54947	96.1433
Totals :				3517.26863	275.10667	

(*R*)-2-(2-Methoxyphenyl)-4-methyl-1-phenylpent-4-en-2-ol 12c

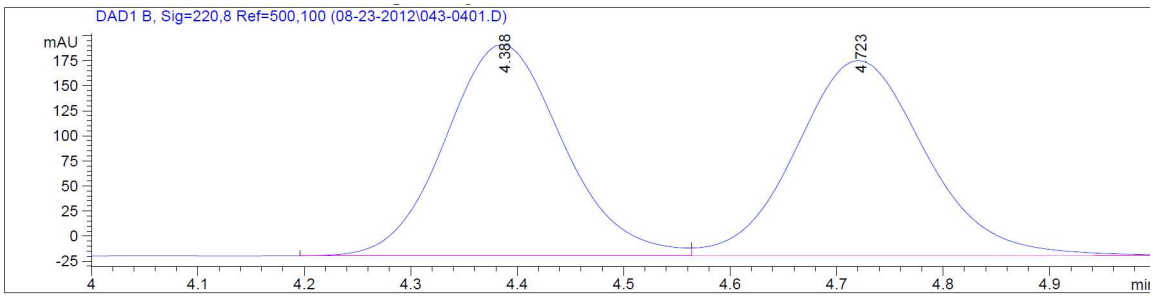


Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	10.484	BV	0.2760	1394.80823	78.24401	49.6480
2	11.151	VB	0.2960	1414.58472	73.68077	50.3520
Totals :				2809.39294	151.92478	

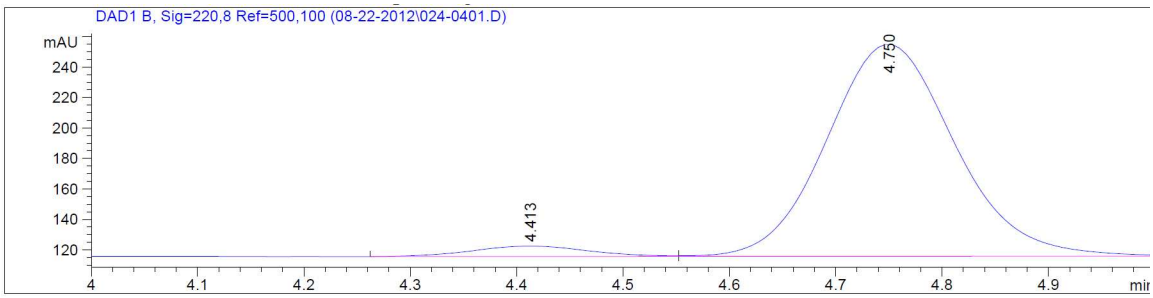


Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	10.582	BV	0.2833	1969.88599	107.75513	92.1333
2	11.276	VB	0.3043	168.19673	8.44598	7.8667
Totals :				2138.08272	116.20111	

(*R*)-2-(4-Fluorophenyl)-4-methyl-1-phenylpent-4-en-2-ol 12d

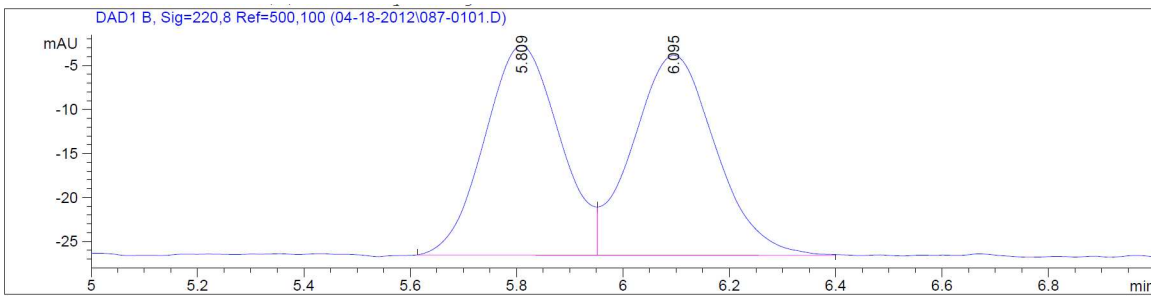


Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	4.388	BV	0.1201	1629.31494	211.03214	49.5229
2	4.723	VV	0.1294	1660.70715	194.92235	50.4771
Totals :				3290.02209	405.95448	

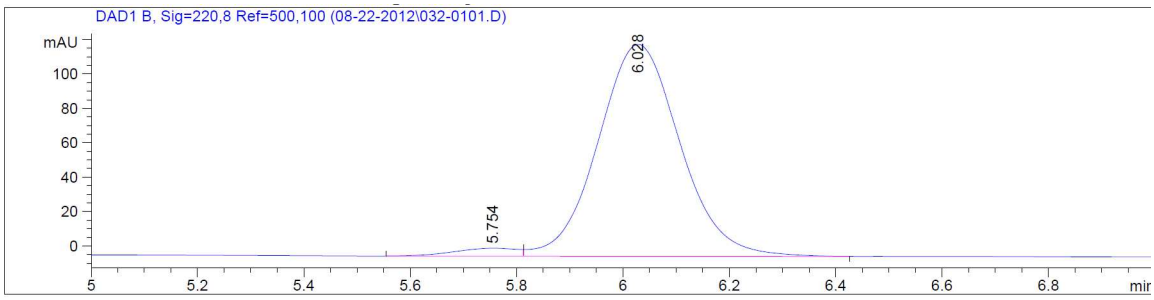


Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	4.413	BV	0.1170	50.69893	6.79719	4.1702
2	4.750	VB	0.1297	1165.04358	139.14168	95.8298
Totals :				1215.74251	145.93887	

(*R*)-2-(4-Chlorophenyl)-4-methyl-1-phenylpent-4-en-2-ol 12e

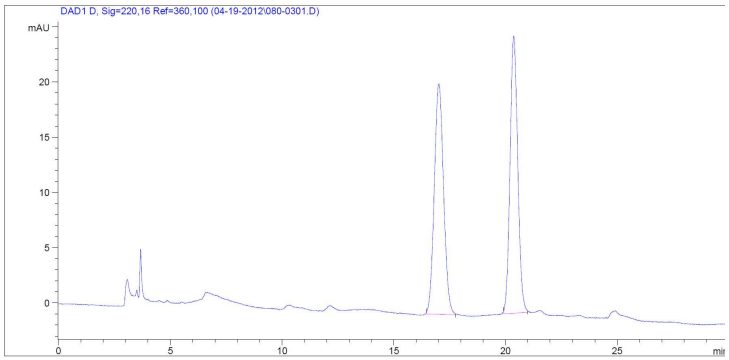


Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	5.809	BV	0.1544	233.62427	23.82161	48.6751
2	6.095	VB	0.1645	246.34253	22.72545	51.3249
Totals :				479.96680	46.54706	

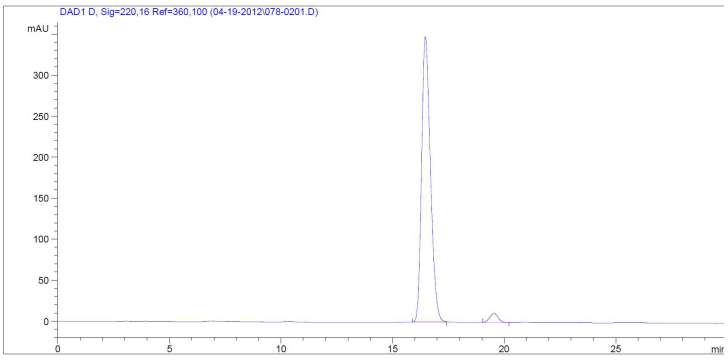


Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	5.754	BV	0.1259	37.21839	4.52992	2.7425
2	6.028	VB	0.1649	1319.85571	123.25403	97.2575
Totals :				1357.07410	127.78395	

(*R*)-2-(4-bromophenyl)-4-methyl-1-phenylpent-4-en-2-ol 12f

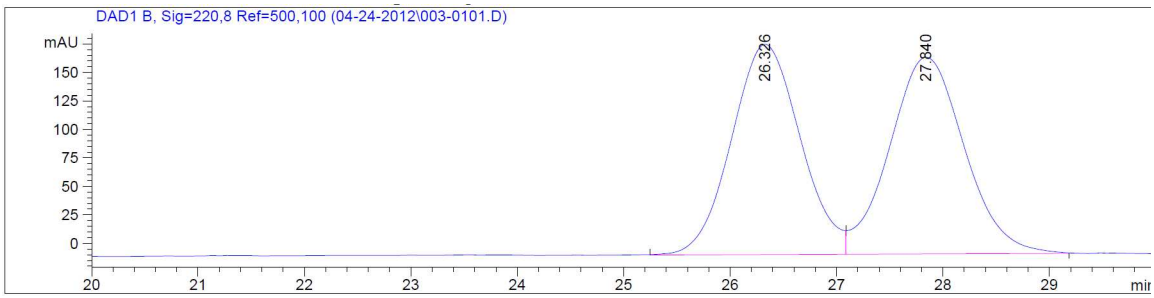


Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	17.019	BB	0.4378	603.79785	20.85466	50.2976
2	20.372	BB	0.3719	596.65350	25.04594	49.7024
Totals :				1200.45135	45.90060	

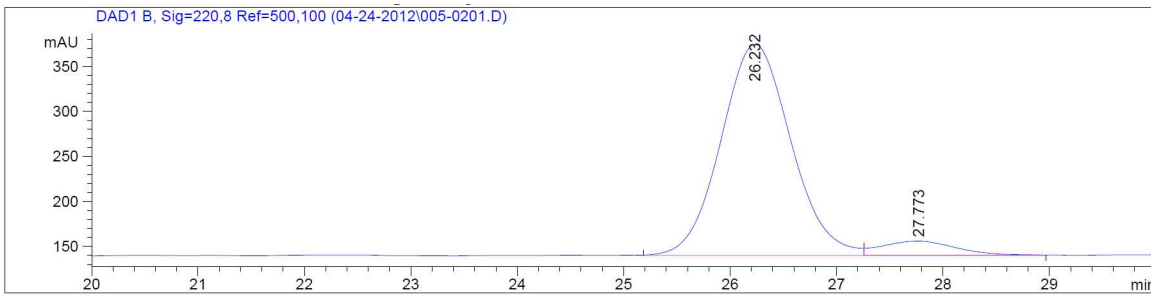


Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	16.472	BB	0.4268	9634.00586	348.17361	97.0468
2	19.544	BB	0.3992	293.17133	11.20531	2.9532
Totals :				9927.17719	359.37892	

(*R*)-2-(4-Methoxyphenyl)-4-methyl-1-(naphthalen-1-yl)pent-4-en-2-ol 12g

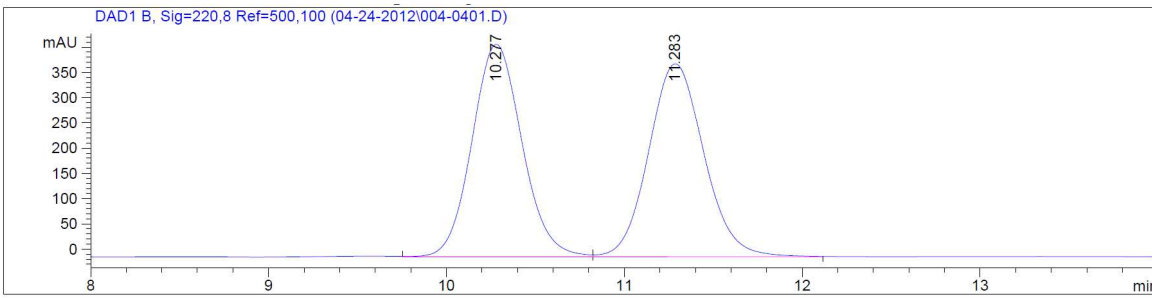


Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	26.326	VV	0.6579	8287.64160	184.54683	49.5631
2	27.840	VB	0.7081	8433.74512	172.57779	50.4369
Totals :				1.67214e4	357.12462	

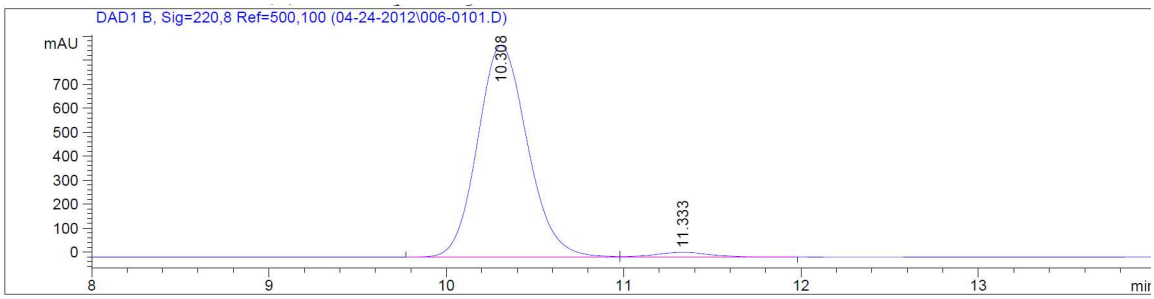


Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	26.232	BV	0.6838	1.07003e4	234.72682	93.2776
2	27.773	VV	0.5816	771.15674	15.85055	6.7224
Totals :				1.14715e4	250.57737	

(*R*)-2-(4-methoxyphenyl)-4-methyl-1-(naphthalen-2-yl)pent-4-en-2-ol 12h

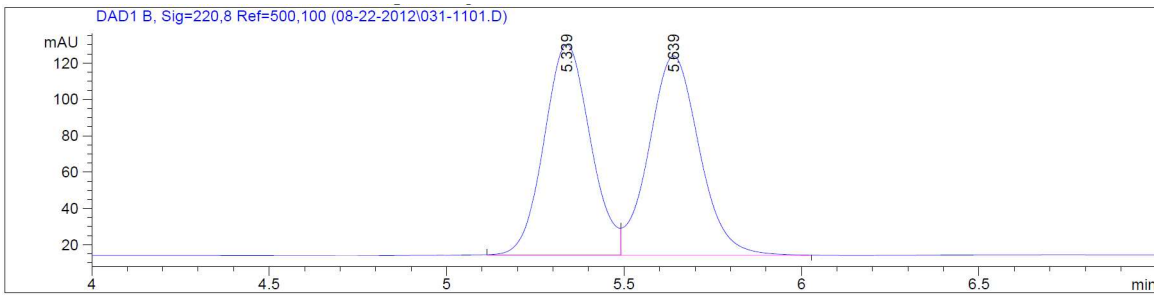


Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	10.277	VV	0.3044	8240.25391	420.97717	49.9634
2	11.283	VB	0.3338	8252.31152	382.33133	50.0366
Totals :				1.64926e4	803.30850	

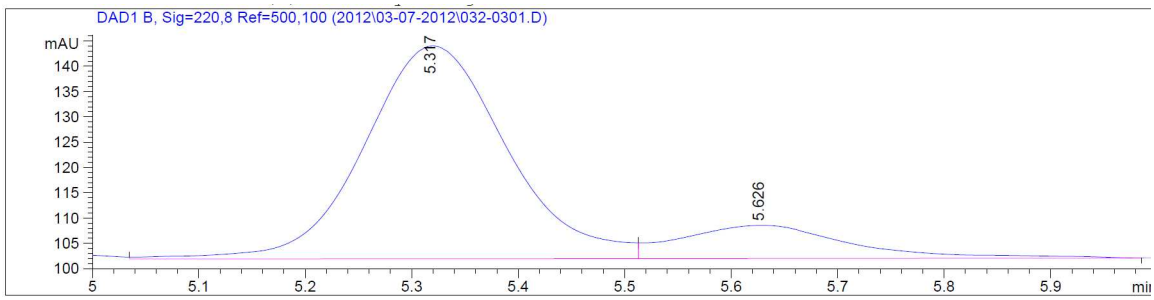


Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	10.308	BV	0.3036	1.72128e4	882.19946	97.6047
2	11.333	VB	0.3187	422.42014	19.50048	2.3953
Totals :				1.76352e4	901.69994	

(*R*)-1-Chloro-3-(4-fluorophenyl)-5-methylhex-5-en-3-ol 12i

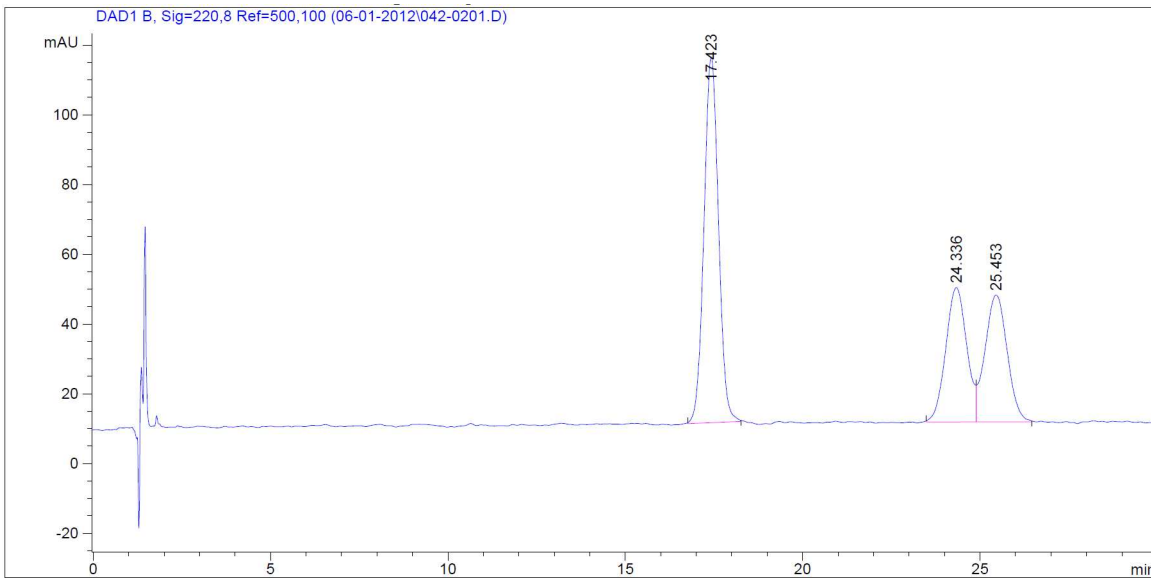


Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	5.339	BV	0.1416	1052.29944	116.29847	49.1793
2	5.639	VB	0.1534	1087.41907	109.94777	50.8207
Totals :				2139.71851	226.24624	

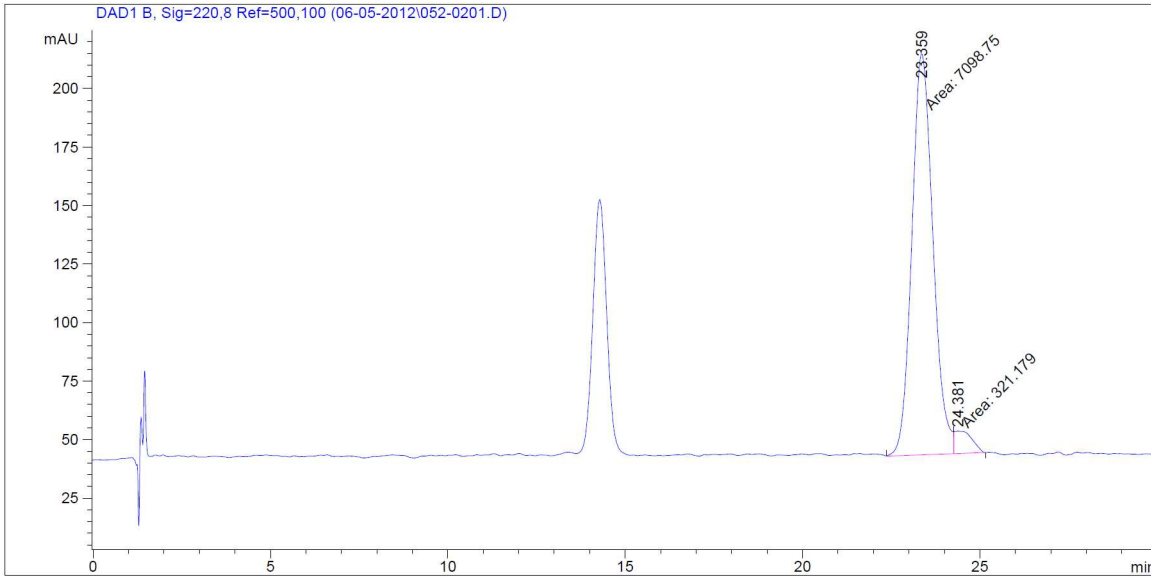


Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	5.317	VV	0.1427	392.72311	42.14299	84.5432
2	5.626	VV	0.1574	71.80025	6.57554	15.4568
Totals :				464.52337	48.71853	

(*R*)-4-methyl-1-phenyl-2-(thiophen-3-yl)pent-4-en-2-ol 12j

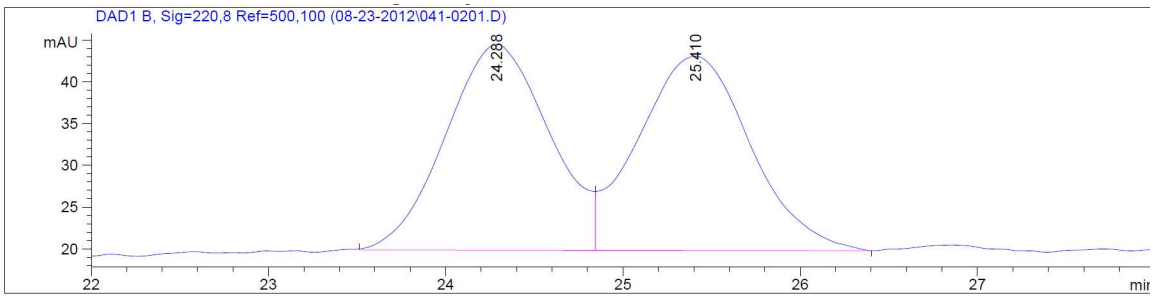


Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	17.423	BB	0.4430	2975.52954	104.86835	48.3919
2	24.336	BV	0.6237	1584.33020	38.53163	25.7664
3	25.453	VB	0.6620	1588.95581	36.35072	25.8417
Totals :				6148.81555	179.75069	

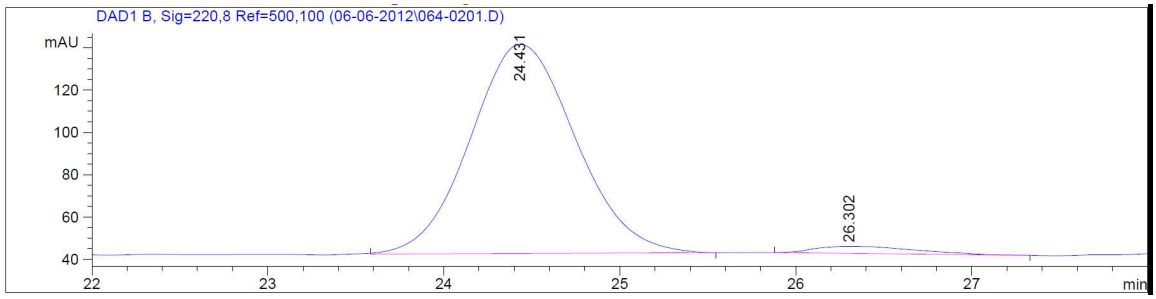


Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	23.359	MF	0.6905	7098.75293	171.33400	95.6714
2	24.381	FM	0.5581	321.17899	9.59143	4.3286
Totals :				7419.93192	180.92543	

(*R*)-4-Methyl-1-phenyl-2-(thiophen-3-yl)pent-4-en-2-ol 12k

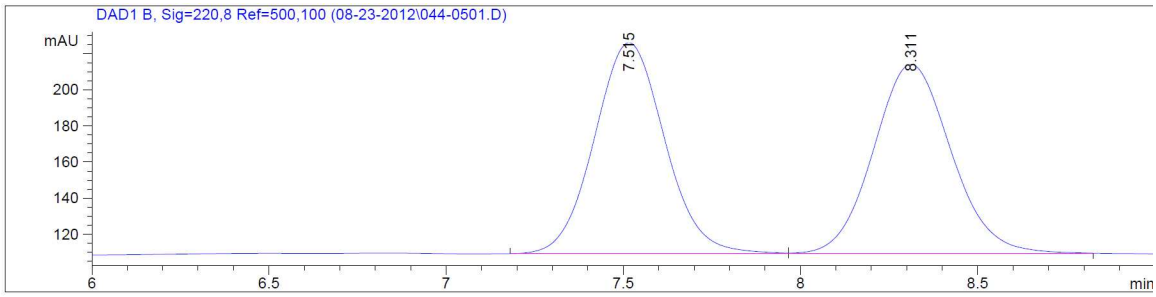


Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	24.288	BV	0.6258	1001.28320	24.65535	49.4156
2	25.410	VB	0.6621	1024.96594	23.25925	50.5844
Totals :				2026.24915	47.91460	

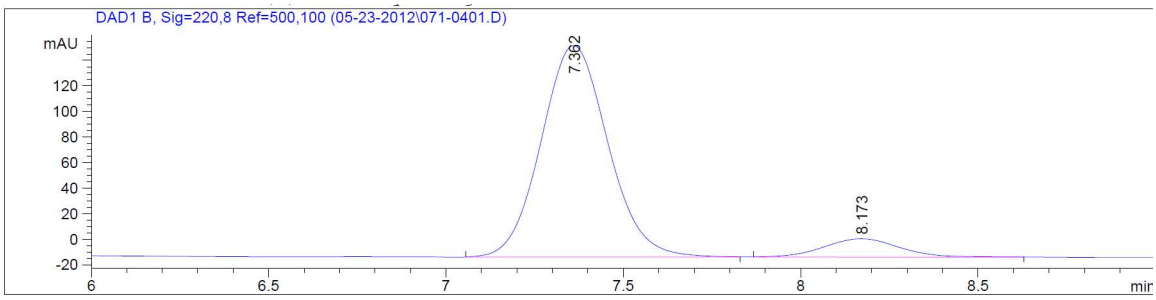


Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	24.431	BB	0.6510	4120.61377	99.12616	96.8472
2	26.302	BB	0.5441	134.14339	3.30112	3.1528
Totals :				4254.75716	102.42728	

(*S*)-5-Methyl-1,3-diphenylhex-5-en-3-ol 12l

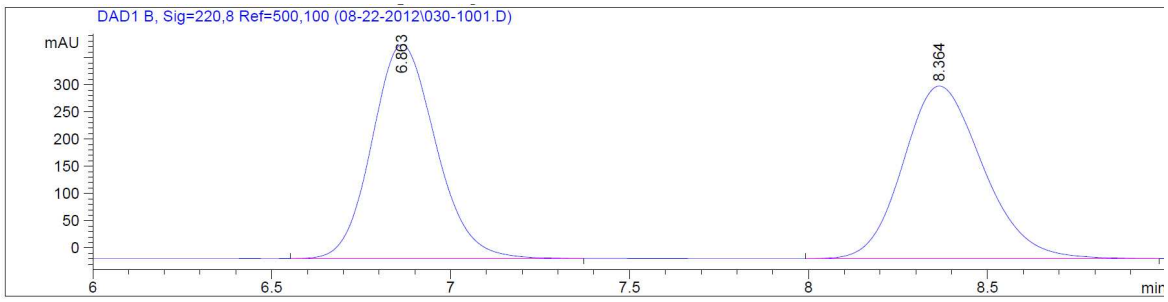


Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	7.515	BV	0.2152	1614.78674	116.84342	50.1815
2	8.311	VB	0.2364	1603.10864	104.83906	49.8185
Totals :				3217.89539	221.68248	

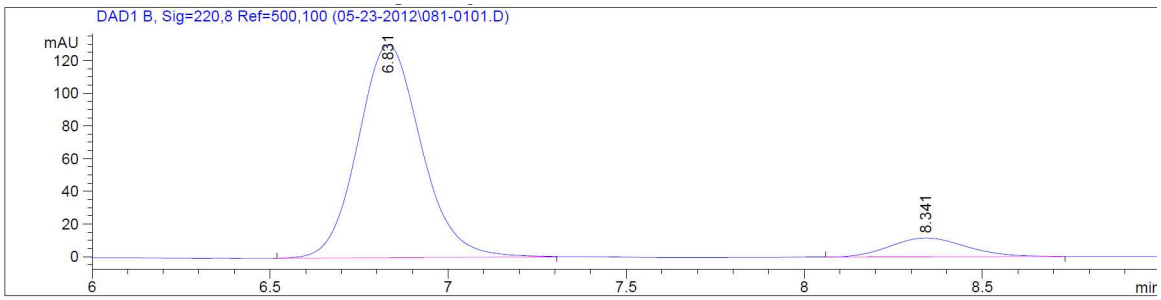


Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	7.362	BB	0.2027	2165.67236	165.50398	90.9961
2	8.173	BB	0.2322	214.28937	14.35128	9.0039
Totals :				2379.96173	179.85526	

(*S*)-2-methyl-4,7-diphenylhept-1-en-4-ol 12m

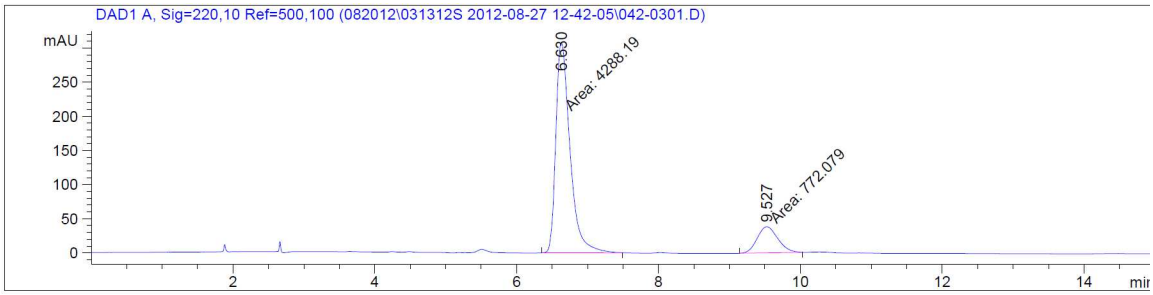


Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.863	BB	0.1928	4886.65625	393.63443	49.7315
2	8.364	BB	0.2395	4939.41992	317.44073	50.2685
Totals :				9826.07617	711.07516	

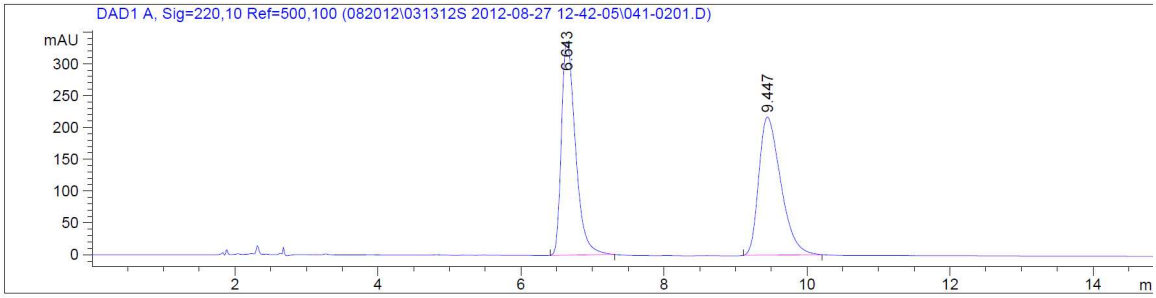


Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.831	BB	0.1899	1609.17676	130.46039	90.2623
2	8.341	BB	0.2329	173.60240	11.58259	9.7377
Totals :				1782.77916	142.04298	

(*R*)-1-(Benzyloxy)-4-methyl-2-phenylpent-4-en-2-ol 12n

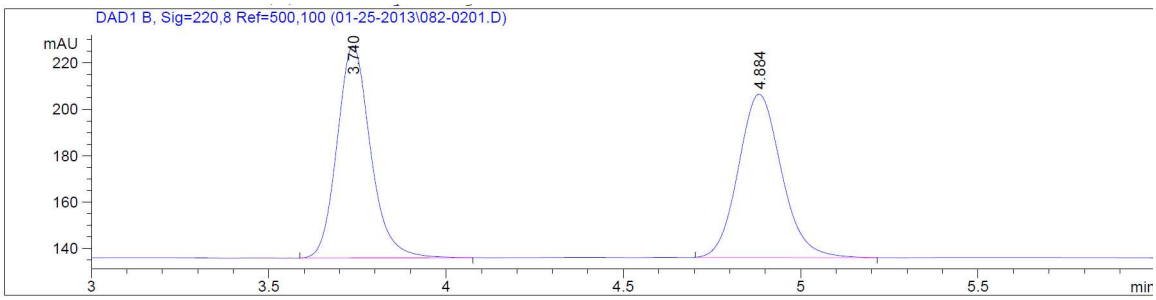


Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.630	MM	0.2315	4288.18701	308.76804	84.7423
2	9.527	MM	0.3362	772.07861	38.27243	15.2577
Totals :				5060.26562	347.04047	

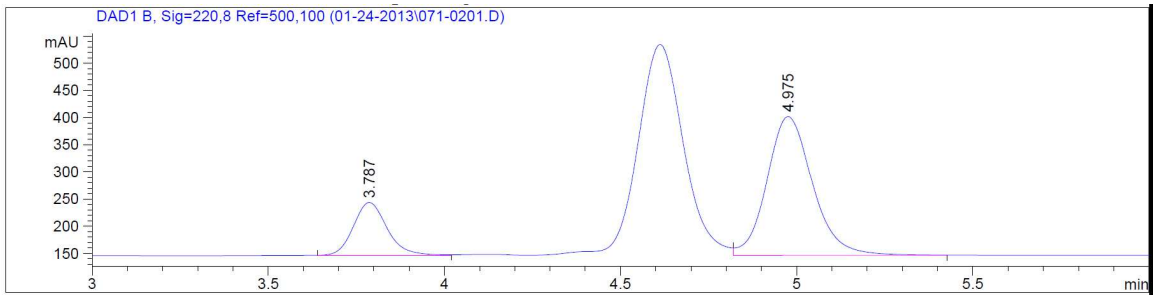


Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.643	MM	0.2272	4575.40479	335.64401	49.7491
2	9.447	MM	0.3554	4621.55811	216.74353	50.2509
Totals :				9196.96289	552.38754	

(*S*)-4-Methyl-2-phenylpent-4-en-2-ol 12o

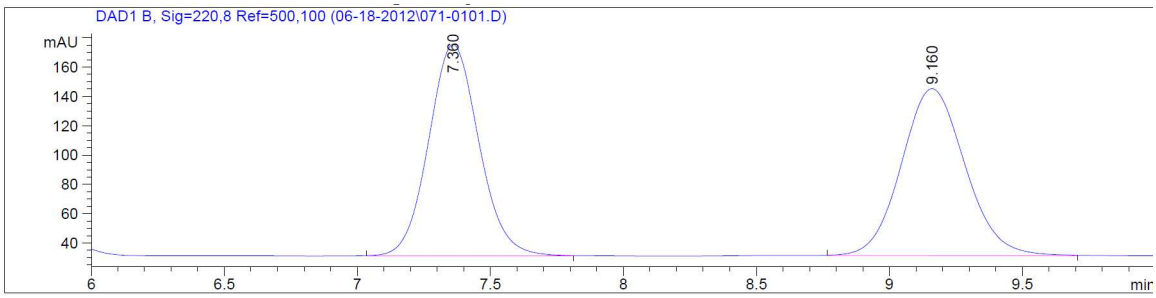


Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	3.740	BB	0.1004	600.65021	91.68155	50.2221
2	4.884	BB	0.1305	595.33752	70.51340	49.7779
Totals :				1195.98773	162.19495	

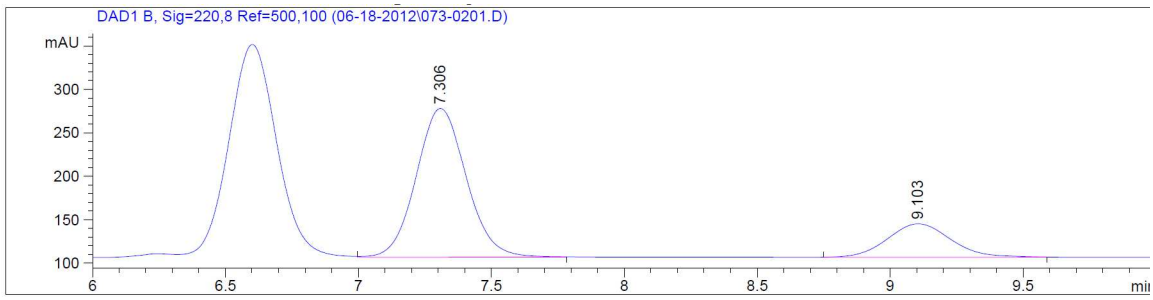


Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	3.787	BV	0.1038	651.06793	97.62309	22.0325
2	4.975	VB	0.1372	2303.97070	255.51031	77.9675
Totals :				2955.03864	353.13340	

(*R*)-tert-Butyl 3-hydroxy-5-methyl-3-phenylhex-5-enoate 12p

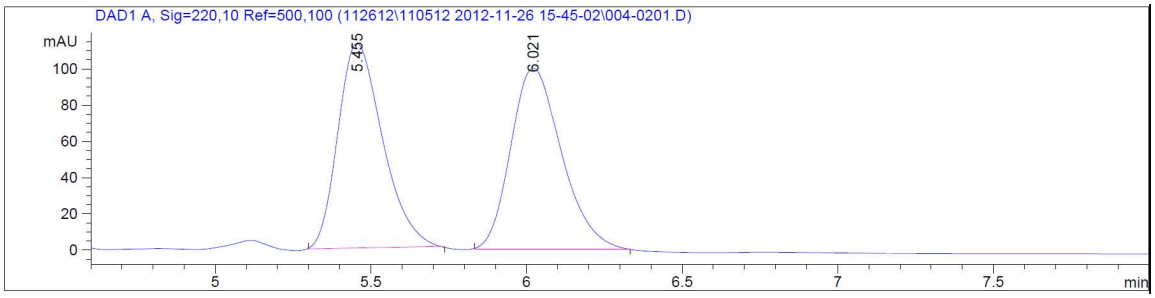


Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	7.360	BB	0.2033	1896.80212	144.35103	49.9853
2	9.160	BB	0.2586	1897.91858	113.80959	50.0147
Totals :				3794.72070	258.16062	

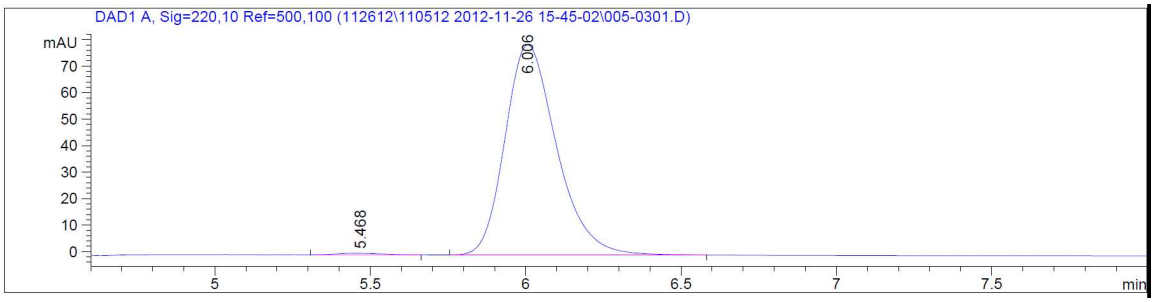


Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	7.306	VB	0.2027	2240.48779	171.14331	77.7966
2	9.103	BB	0.2558	639.44269	38.49627	22.2034
Totals :				2879.93048	209.63958	

(*R*)-tert-butyl 4-(6-(2-methylallyl)-2-oxo-6-phenyl-1,3-oxazinan-3-yl)piperidine-1-carboxylate 1

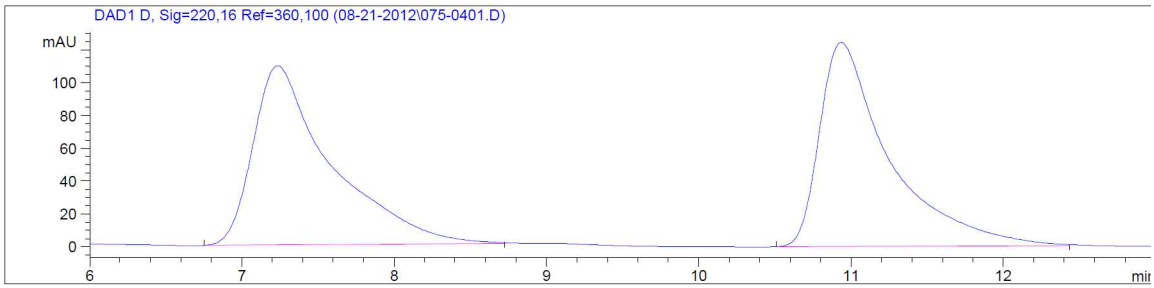


Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	5.455	MM	0.1660	1125.76025	112.99730	49.9209
2	6.021	MM	0.1884	1129.32776	99.87939	50.0791
Totals :				2255.08801	212.87669	

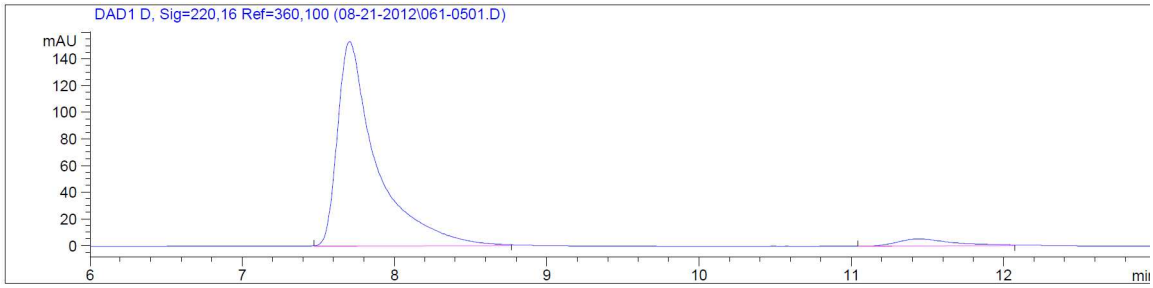


Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	5.468	MM	0.1582	5.86168	6.17572e-1	0.6366
2	6.006	MM	0.1923	914.86511	79.28283	99.3634
Totals :				920.72680	79.90040	

(*R*)-4-Methylene-1,2-diphenylhexan-2-ol 16

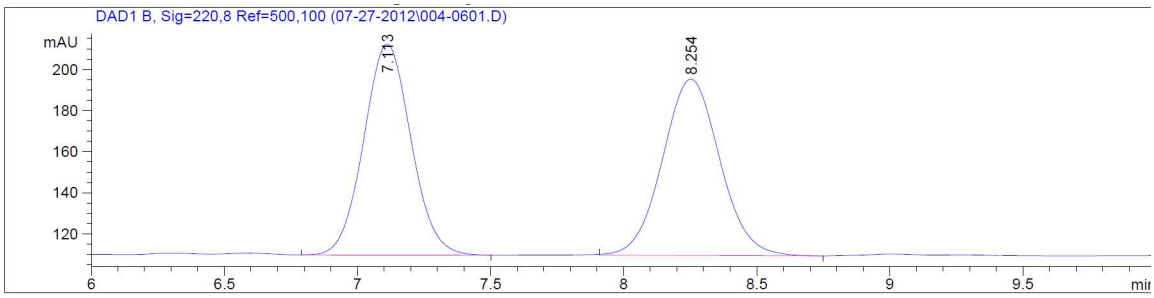


Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	7.233	BB	0.4989	3884.16479	109.21716	49.6043
2	10.934	BB	0.4541	3946.13037	124.38062	50.3957
Totals :				7830.29517	233.59777	

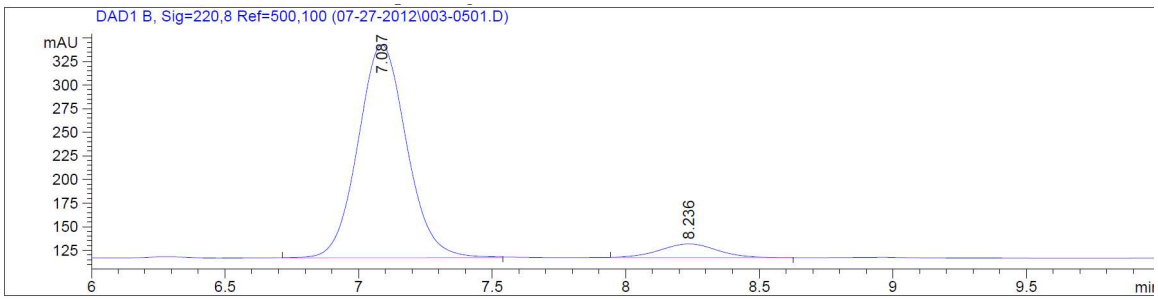


Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	7.709	BB	0.2512	2725.96973	153.45779	95.4457
2	11.447	BB	0.3722	130.07387	5.22889	4.5543
Totals :				2856.04359	158.68668	

(*R*)-4-Methylene-1,2-diphenyloctan-2-ol 17



Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	7.113	VB	0.1924	1268.09448	102.46413	49.6507
2	8.254	BB	0.2329	1285.93921	85.75674	50.3493
Totals :				2554.03369	188.22087	



Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	7.087	BB	0.1952	2845.49854	225.58667	93.0212
2	8.236	BB	0.2288	213.47958	14.41169	6.9788
Totals :				3058.97812	239.99836	

X-Ray Crystallograpgic Data for Compound 7

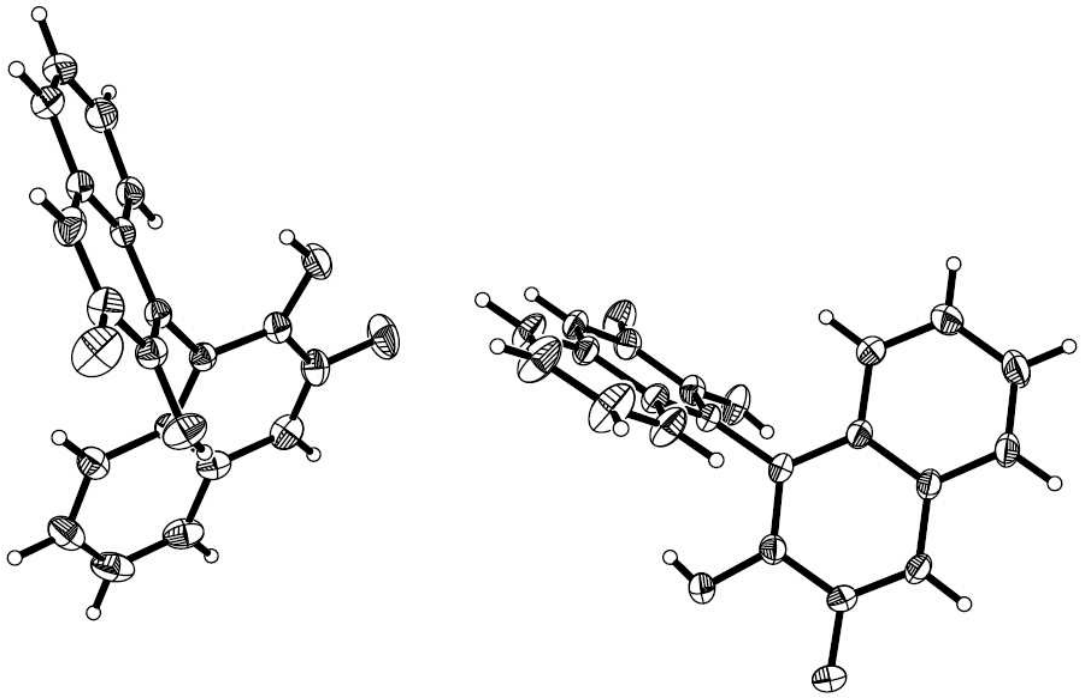


Figure 1. ORTEP (50% probability level) diagram of asymmetric unit of **7** expanded to show the full fragments.

Table 1. Crystal data and structure refinement for **7**.

Identification code	3,3'-F_2-BINOL	
Empirical formula	C20 H12 F2 O2	
Formula weight	322.30	
Temperature	173(2) K	
Wavelength	0.71073 Å	
Crystal system	Hexagonal	
Space group	P3(2)21	
Unit cell dimensions	a = 10.6787(7) Å	α= 90°.
	b = 10.6787(7) Å	β= 90°.
	c = 23.2221(15) Å	γ = 120°.
Volume	2293.3(3) $Å^3$	
Z	6	
Density (calculated)	1.400 Mg/m^3	
Absorption coefficient	0.106 mm^{-1}	
F(000)	996	
Crystal size	0.32 x 0.26 x 0.24 mm^3	
Theta range for data collection	2.20 to 28.80°.	
Index ranges	-14<=h<=14, -14<=k<=14, -30<=l<=31	
Reflections collected	24741	
Independent reflections	3825 [R(int) = 0.0253]	
Completeness to theta = 28.80°	96.9 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.9750 and 0.9668	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	3825 / 0 / 222	
Goodness-of-fit on F^2	1.038	
Final R indices [I>2sigma(I)]	R1 = 0.0347, wR2 = 0.1079	
R indices (all data)	R1 = 0.0391, wR2 = 0.1215	
Absolute structure parameter	0.8(6)	
Largest diff. peak and hole	0.267 and -0.226 $e.Å^{-3}$	

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for **7**. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
F(23)	4952(1)	8248(1)	1064(1)	39(1)
O(22)	6384(1)	6915(1)	856(1)	35(1)
C(21)	4813(1)	5611(1)	55(1)	22(1)
C(24)	3364(2)	7159(1)	286(1)	25(1)
C(29)	3614(1)	5377(2)	-293(1)	24(1)
C(210)	2889(2)	6169(2)	-180(1)	24(1)
C(22)	5251(1)	6593(1)	499(1)	25(1)
C(25)	1725(2)	5950(2)	-536(1)	35(1)
C(26)	1288(2)	4993(2)	-984(1)	48(1)
C(28)	3122(2)	4383(2)	-758(1)	35(1)
C(23)	4491(2)	7335(1)	611(1)	26(1)
C(27)	1988(2)	4196(2)	-1092(1)	48(1)
O(12)	8211(1)	60(1)	742(1)	34(1)
F(13)	9544(1)	2700(1)	338(1)	46(1)
C(18)	11614(1)	1667(2)	2365(1)	28(1)
C(11)	9864(1)	749(1)	1550(1)	21(1)
C(12)	9355(1)	1045(2)	1050(1)	25(1)
C(19)	11070(1)	1896(1)	1841(1)	23(1)
C(14)	11196(2)	3588(2)	1097(1)	31(1)
C(15)	12886(2)	4476(2)	1924(1)	35(1)
C(13)	10064(2)	2473(2)	835(1)	31(1)
C(110)	11731(1)	3332(1)	1620(1)	27(1)
C(17)	12718(2)	2805(2)	2656(1)	35(1)
C(16)	13362(2)	4214(2)	2434(1)	40(1)

Table 3. Bond lengths [Å] and angles [°] for **7**. Symmetry transformations used to generate equivalent atoms: #1 y,x,-z #2 x-y,-y,-z+1/3

F(23)-C(23)	1.3484(14)	C(22)-O(22)-H(22)	107.4(16)
O(22)-C(22)	1.3613(16)	C(22)-C(21)-C(29)	119.42(11)
O(22)-H(22)	0.76(2)	C(22)-C(21)-C(21)#1	119.19(11)
C(21)-C(22)	1.3759(17)	C(29)-C(21)-C(21)#1	121.39(11)
C(21)-C(29)	1.4263(17)	C(23)-C(24)-C(210)	119.47(11)
C(21)-C(21)#1	1.498(2)	C(23)-C(24)-H(24)	120.3
C(24)-C(23)	1.3520(19)	C(210)-C(24)-H(24)	120.3
C(24)-C(210)	1.4172(17)	C(28)-C(29)-C(21)	121.74(11)
C(24)-H(24)	0.9500	C(28)-C(29)-C(210)	118.41(11)
C(29)-C(28)	1.4192(18)	C(21)-C(29)-C(210)	119.84(11)
C(29)-C(210)	1.4275(17)	C(25)-C(210)-C(24)	121.83(12)
C(210)-C(25)	1.4110(17)	C(25)-C(210)-C(29)	119.20(12)
C(22)-C(23)	1.4137(18)	C(24)-C(210)-C(29)	118.97(11)
C(25)-C(26)	1.366(2)	O(22)-C(22)-C(21)	124.74(12)
C(25)-H(25)	0.9500	O(22)-C(22)-C(23)	115.66(11)
C(26)-C(27)	1.407(2)	C(21)-C(22)-C(23)	119.59(11)
C(26)-H(26)	0.9500	C(26)-C(25)-C(210)	120.85(13)
C(28)-C(27)	1.366(2)	C(26)-C(25)-H(25)	119.6
C(28)-H(28)	0.9500	C(210)-C(25)-H(25)	119.6
C(27)-H(27)	0.9500	C(25)-C(26)-C(27)	120.11(14)
O(12)-C(12)	1.3506(16)	C(25)-C(26)-H(26)	119.9
O(12)-H(12)	0.8400	C(27)-C(26)-H(26)	119.9
F(13)-C(13)	1.3536(15)	C(27)-C(28)-C(29)	120.67(13)
C(18)-C(17)	1.376(2)	C(27)-C(28)-H(28)	119.7
C(18)-C(19)	1.4200(17)	C(29)-C(28)-H(28)	119.7
C(18)-H(18)	0.9500	F(23)-C(23)-C(24)	120.96(11)
C(11)-C(12)	1.3844(17)	F(23)-C(23)-C(22)	116.37(11)
C(11)-C(19)	1.4279(17)	C(24)-C(23)-C(22)	122.67(11)
C(11)-C(11)#2	1.488(2)	C(28)-C(27)-C(26)	120.74(14)
C(12)-C(13)	1.4123(19)	C(28)-C(27)-H(27)	119.6
C(19)-C(110)	1.4247(18)	C(26)-C(27)-H(27)	119.6
C(14)-C(13)	1.345(2)	C(12)-O(12)-H(12)	109.5
C(14)-C(110)	1.4264(19)	C(17)-C(18)-C(19)	120.85(14)
C(14)-H(14)	0.9500	C(17)-C(18)-H(18)	119.6
C(15)-C(16)	1.370(2)	C(19)-C(18)-H(18)	119.6
C(15)-C(110)	1.4161(19)	C(12)-C(11)-C(19)	119.36(11)
C(15)-H(15)	0.9500	C(12)-C(11)-C(11)#2	119.29(12)
C(17)-C(16)	1.404(3)	C(19)-C(11)-C(11)#2	121.33(11)
C(17)-H(17)	0.9500	O(12)-C(12)-C(11)	124.90(12)
C(16)-H(16)	0.9500	O(12)-C(12)-C(13)	116.20(12)
		C(11)-C(12)-C(13)	118.90(12)
		C(18)-C(19)-C(110)	117.95(12)
		C(18)-C(19)-C(11)	121.88(12)
		C(110)-C(19)-C(11)	120.12(11)
		C(13)-C(14)-C(110)	119.00(12)
		C(13)-C(14)-H(14)	120.5
		C(110)-C(14)-H(14)	120.5
		C(16)-C(15)-C(110)	120.43(14)
		C(16)-C(15)-H(15)	119.8
		C(110)-C(15)-H(15)	119.8
		C(14)-C(13)-F(13)	119.65(12)
		C(14)-C(13)-C(12)	123.72(12)
		F(13)-C(13)-C(12)	116.62(12)
		C(15)-C(110)-C(19)	119.85(12)
		C(15)-C(110)-C(14)	121.29(13)
		C(19)-C(110)-C(14)	118.85(12)
		C(18)-C(17)-C(16)	120.63(14)
		C(18)-C(17)-H(17)	119.7
		C(16)-C(17)-H(17)	119.7
		C(15)-C(16)-C(17)	120.25(13)
		C(15)-C(16)-H(16)	119.9
		C(17)-C(16)-H(16)	119.9

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for **7**. The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2\ a^{*2}U^{11} + ...\ + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
F(23)	46(1)	38(1)	41(1)	-19(1)	-14(1)	28(1)
O(22)	35(1)	35(1)	44(1)	-13(1)	-17(1)	24(1)
C(21)	22(1)	23(1)	26(1)	2(1)	1(1)	15(1)
C(24)	29(1)	27(1)	28(1)	0(1)	2(1)	20(1)
C(29)	25(1)	28(1)	25(1)	-1(1)	-1(1)	17(1)
C(210)	26(1)	32(1)	23(1)	1(1)	0(1)	20(1)
C(22)	24(1)	23(1)	28(1)	0(1)	-4(1)	12(1)
C(25)	36(1)	56(1)	30(1)	-8(1)	-7(1)	36(1)
C(26)	48(1)	82(1)	36(1)	-21(1)	-20(1)	48(1)
C(28)	37(1)	45(1)	36(1)	-15(1)	-9(1)	29(1)
C(23)	29(1)	22(1)	27(1)	-4(1)	-2(1)	14(1)
C(27)	51(1)	70(1)	41(1)	-30(1)	-22(1)	43(1)
O(12)	33(1)	30(1)	35(1)	2(1)	-13(1)	13(1)
F(13)	47(1)	44(1)	43(1)	17(1)	-10(1)	20(1)
C(18)	22(1)	37(1)	26(1)	-3(1)	0(1)	15(1)
C(11)	20(1)	23(1)	21(1)	0(1)	1(1)	12(1)
C(12)	24(1)	28(1)	26(1)	0(1)	-2(1)	14(1)
C(19)	20(1)	27(1)	22(1)	-2(1)	4(1)	12(1)
C(14)	28(1)	25(1)	38(1)	7(1)	8(1)	12(1)
C(15)	29(1)	26(1)	42(1)	-6(1)	6(1)	8(1)
C(13)	32(1)	35(1)	29(1)	8(1)	0(1)	19(1)
C(110)	23(1)	25(1)	29(1)	-1(1)	7(1)	10(1)
C(17)	27(1)	49(1)	28(1)	-8(1)	-3(1)	18(1)
C(16)	27(1)	44(1)	39(1)	-17(1)	-2(1)	11(1)

Table 5. Hydrogen-bond geometry (Å , °).

D - H···A	D – H	H···A	D···A	D - H···A
O(1)-H(1A)···O(2)	0.84	2.19	2.942(2)	149
O(2)-H(2A)···O(1)	0.84	2.11	2.942(2)	171
O(14)-H(14A)···F(2)	0.95	2.49	3.232(3)	134