Supplementary Material (ESI) for Organic & Biomolecular Chemistry

Supporting Information

Chiral Sulfoxides as Activators of Allyl Trichlorosilanes in the Stereoselective Allylation of Aldehydes.

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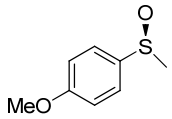
General Information.

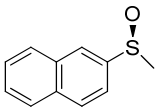
All reactions were performed in oven-dried (140°C) or flame-dried glassware under an atmosphere of dry nitrogen. All the solvents for the reactions were of reagent grade and were dried and distilled immediately before use (dichloromethane from calcium hydride, diethyl ether from lithium aluminium hydride). Column chromatographic purification of products was carried out using silica gel 60 (70-230 mesh, Merck). The reagents (Aldrich and Fluka) were used without further purification. The NMR spectra were recorded on a Bruker DRX 400 (400 MHz, 1H; 100 MHz, 13C). Spectra were referenced to residual chloroform (7.26 ppm, 1H, 77.23 ppm, 13C). Chemical shifts are reported in ppm, multiplicities are indicated by s (singlet), d (doublet), t (triplet), q (quartet), quint (quintept), m (multiplet) and br (broad). Coupling constants, J, are reported in Hz. Yields are given for isolated products showing one spot on a TLC plate and no impurities detectable in the NMR spectrum. Mass spectrometry analysis was carried out using an electrospray spectrometer Waters 4 micro quadrupole. HPLC analyses were performed with Waters Associates equipment (Waters 2487 Dual l absorbance Detector) and using a CHIRALPAK AD, CHIRALCEL OD, CHIRALCEL OB, CHIRALCEL AS, CHIRALCEL OD-H, CHIRALCEL AS-H, CHIRALCEL AD-H column with hexane/isopropyl alcohol mixtures and flow rates as indicated. Chiral GC (Supelco β-DEX 120 analyses were performed with FOCUS GC/FID Thermo Scientific. The HPLC and GC methods were calibrated with the corresponding racemic mixtures. Optical rotations were measured with a JASCO DIP-1000 polarimeter. Elemental analyses were performed with FLASHEA 1112 series-Thermo Scientific for CHNS-O. The absolute configuration of **9c**, **9m**, **11** ,**17b**, **18b** was established by Mosher methodology, while for the others it was determined comparing the optical rotation with the one reported in literature.

General Procedure for the catalytic asymmetric sulfoxidation.

In a flame-dried, 2-necked, round-bottom flask, a solution of (R,R)-diethyl tartrate (683 μL, 7.93 mmol), titanium tetraisopropoxide (257 μL, 0.99 mmol) and sulfide (3,0 mmol, 1.0 equiv) in dry CH_2Cl_2 (11 mL) under a nitrogen atmosphere was stirred at room temperature for 5 min. Then the temperature was cooled to 20 °C and after 20 min a solution of cumyl hydroperoxide (1.2 mL, 6 mmol in 11 ml of dry CH_2Cl_2) slowly added. At the end of the reaction, the mixture was quenched with a solution 10% of Na_2SO_3 (20 mL), extracted with 20x3 mL of CH_2Cl_2 and dried over anhydrous Na_2SO_4. Then the resulting gel was recovered with CH_2Cl_2 (50 mL) and filtered on a short pad of Celite. After removing the solvent under reduced pressure, the crude oil was purified by silica gel flash chromatography (eluent starting from Ep/ethyl acetate to a ethyl acetate) to afford the pure sulfoxide.



(*R*)-1-methoxy-4-(methylsulfinyl)benzene.[1] NMR data were in agreement with those reported in literature. e.e. 96 %. ^{1}H NMR δ 2.71 (s, 3H), 3.85 (s, 3H), 7.02 (d, 2H, J = 8.85 Hz), 7.60 (d, 2H, J = 8.85 Hz). ^{13}C NMR δ 43.6, 55.2, 114.5, 125.1, 136.2, 161.5. ESI-MS *m/z* 171 $[MH]^+$. $[\alpha]_D$ - 102 (*c* 1.0, $CHCl_3$). The enantiomeric excess was determined by chiral HPLC (Chiralcel OB hexane/2-propanol 80:20, 0,8 mL/min, 254 nm, t_S = 16.35 min. for minor enantiomer, t_R = 30.44 min. for major enantiomer.). Anal. calcd. for $C_8H_{10}O_2S$: C, 56.44; H, 5.92; S, 18.84 found: C, 56.69; H, 5.63; S, 18.66.

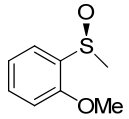


(*R*)-2-(methylsulfinyl)naphthalene.[2] ^{1}H NMR ($CDCl_3$) δ 2.73 *(s,* 3H), 7.53 (m, 2H), 8.21 (m, 5H). ^{13}C NMR δ 43.3, 118.9, 123.5, 126.9, 127.3, 127.6, 128.0, 129.1, 133.4, 133.9, 142.2. NMR data were in agreement with those reported in literature. ESI-MS *m/z* 191 $[MH]^+$. e.e. 98 %. The enantiomeric excess was determined by chiral HPLC (Chiralcel OB hexane/2- propanol 80:20, 0,8

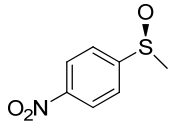
[1] A. B. E. Minidis, J-E. Backvall, *Chem. Eur. J.* **2001**, *7*, 297-302.

[2] J-M Brunel, P. Diter, M. Duetsch, H. B. Kagan, *J. Org. Chem*. **1995**, *60*, 8086-8088.

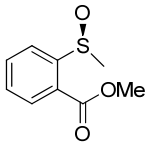
mL/min, 254 nm, t_S = 16.63 min. for minor enantiomer, t_R = 20.84 min. for major enantiomer.). Anal. calcd. for $C_{11}H_{10}OS$: C, 69.44; H, 5.30; S, 16.85 found: C, 69.69; H, 5.54; S, 16.62.



(*R*)-1-methoxy-2-(methylsulfinyl)benzene.[3] ^{1}H NMR ($CDC1_3$) δ 2.72 *(s,* 3H), 3.86 *(s,* 3H), 6.85 (d, *J* = 9 Hz, 2H), 7.88 (d, *J* = 9 Hz, 2H). ESI-MS *m/z* 171 $[MH]^+$. NMR data were in agreement with those reported in literature. e.e. 93 %. The enantiomeric excess was determined by chiral HPLC (Chiralcel OB hexane/2- propanol 80:20, 0,8 mL/min, 254 nm, t_S = 12.47 min. for minor enantiomer, t_R = 20.08 min. for major enantiomer.). Anal. calcd. for $C_8H_{10}O_2S$: C, 56.44; H, 5.92; S, 18.84 found: C, 56.66; H, 6.01; S, 18.44.



(*R*)-1-(methylsulfinyl)-4-nitrobenzene.[4] ^{1}H NMR ($CDCl_3$) δ 2.82 (s, 3H), 7.87 (d, 2H, *J* = 9.00 Hz), 8.40 (d, 2H, *J* = 9.00 Hz). ^{13}C NMR δ 43.7, 124.3, 124.6, 149.4, 153.2. ESI-MS *m/z* 186 $[MH]^+$. e.e. 94 %. NMR data were in agreement with those reported in literature. e.e. 94 %. The enantiomeric excess was determined by chiral HPLC (Chiralcel OB hexane/2- propanol 80:20, 0,8 mL/min, 254 nm, t_S = 20.70 min. for minor enantiomer, t_R = 24.41 min. for major enantiomer.). Anal. calcd. for $C_7H_7O_3NS$: C, 45.40; H, 3.81; N, 7.56; S, 17.31 found: C, 45.68; H, 3.99; N, 7.83; S, 17.40.



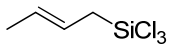
[3] M. A. M. Capozzi, C. Cardellicchio, F. Naso, P. Tortorella, *J. Org. Chem*. **2000**, 65, 2843-2846.

[4] P. J. Kropp, G. W. Breton, J. D. Fields, J. C. Tung, B. R. Loomis, *J. Am. Chem. Soc.* **2000**, 122, 4280-4285.

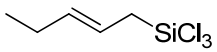
(*R*)-methyl 2-(methylsulfinyl)benzoate.[5] ^{1}H NMR δ ($CDCl_3$) 2.85 (s, 3H), 3.96 (s, 3H), 7.57 (t, 1H, *J*=7.6 Hz), 7.83 (t, 1H, *J*=6.6 Hz), 8.09 (d, 1H, *J*=7.6 Hz), 8.32 (d, 1H, *J*=7.9 Hz). ESI-MS *m/z* 198 $[MH]^+$. NMR data were in agreement with those reported in literature. e.e. 69 % (99% after crystallization in Hex/Et_2O 1:1 at +4 °C. The enantiomeric excess was determined by chiral HPLC (Chiralcel OB hexane/2- propanol 80:20, 0,8 mL/min, 254 nm, t_S = 15.38 min. for minor enantiomer, t_R = 27.26 min. for major enantiomer.). Anal. calcd. for $C_9H_{10}O_3S$: C, 54.53; H, 5.08; S, 16.17 found: C, 54.93; H, 5.40; S, 16.33.

General Procedure for the preparation of (*E*)-RCH=CHCH$_2$SiCl$_3$ (R = CH$_3$; CH$_3$CH$_2$; Ph).

In a flame-dried, 2-necked, round-bottom flask, a mixture of (*E*)-RCH=CHCH$_2$X (X = Cl; Br) (33.2 mmol), trichlorosilane (4.8 ml, 47.6 mmol) and dry ether (20 ml) was added drop wise for 20 min. to a suspension of CuCl (1.1 mmol) and triethylamine (5.05 ml, 36.3 mmol) in dry ether (25 ml) at room temperature. the reaction mixture was stirred for 20 min. at same temperature, filtered, and distilled under nitrogen (R = CH_3) to afford (*E*)-RCH=CHCH$_2$SiCl$_3$. However, more conveniently, for R = CH_3, CH_3CH_2, Ph the reaction mixture was filtered through a short pad of celite, concentrated *in vacuo* and used without further purification.



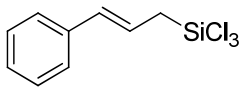
(*E*)-Crotyltrichlorosilane.[6a] ^{1}H NMR ($CDCl_3$, 400 MHz) δ 1.65 (d, 3H, *J* = 8.0 Hz), 2.18 (d, *J* = 6.5 Hz, 2H), 5.27-5.35 (m, 1H), 5.50-5.57 (m, 1H). NMR data were in agreement with those reported in literature; bp 137-140°C (lit. 142-143°C).[6b]This product was a 97:3 (*E/Z*)-mixture as shown by ^{1}H-NMR. ESI-MS *m/z* 188 $[MH]^+$.



[5] S. Okaki, H. Yang, T. Mtsui, Y. Goto, Y. Watanabe, *Tetrahedron Asymmetry* **1999**, 10, 183-192.

[6] a) A. V. Malkov, L. Dufkovà, L. Farrugia, P. Kocovsky, *Ang. Chem*, **2003**, 42 (31), 3674-3677.b) K. Iseki, Y. Kuroki, M. Takahashi, S. Kishimoto, Y. Kobayashi, *Tetrahedron*, **1997**, *53*, 3513-3526.

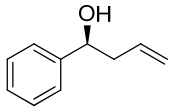
(*E*)-2-Pentenyltrichlorosilane.[7] ^{1}H NMR ($CDCl_3$, 400 MHz) δ 0.99 (t, 3H, *J* = 7.4 Hz), 2.04-2.08 (m, 2H), 2.26 (d, 2H, *J* = 7.5 Hz), 5.29-5.40 (m, 1H), 5.59-5.68 (m, 1H). ^{13}C NMR δ 12.7, 25.0, 28.3, 116.3, 136.7. NMR data were in agreement with those reported in literature. This product was a 95:5 (*E/Z*)-mixture as shown by ^{1}H-NMR. ESI-MS *m/z* 202 $[MH]^+$.



(*E*)-Cinnamyltrichlorosilane. ^{1}H NMR ($CDCl_3$, 400 MHz) δ 2.39 (d, 2H, *J* = 7.9 Hz), 6.00-6.07 (m, 1H), 6.40 (d, 1H, *J* = 15.8 Hz), 7.12-7.29 (m, 5H). ^{13}C NMR δ 28.9, 117.6, 125.1, 126.6, 127.6, 133.4, 135.8. This product was a 95:5 (*E/Z*)-mixture as shown by ^{1}H-NMR. ESI-MS *m/z* 265 $[MH]^+$.

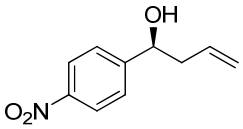
General Procedure for the Allylation of Aldehydes.

In a flame-dried, 2-necked, round-bottom flask, allyltrichlorosilane (46 μL, 0.325 mmol) was added to a solution of sulfoxide (0.75 mmol), diisopropylethylamine (40 μL, 0.25 mmol) and tetrabutylammonium iodide (0.30 mmol) in dry dichloromethane (1.5 mL) under nitrogen at -78 °C. After 5 min. of stirring at that temperature, aldehyde was added (0.25 mmol). At the end of the reaction, the mixture was quenched with saturated aqueous $NaHCO_3$ (1.5 mL), extracted with 20x3 mL of CH_2Cl_2 and dried over anhydrous Na_2SO_4. After removing the solvent under reduced pressure, the crude oil was purified by silica gel flash chromatography.

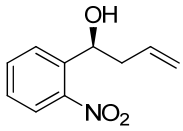


(S)-(-)-1-Phenyl-but-3-en-1-ol.[6] This product was purified with petroleum ether to petroleum ether: Et_2O 70:30 mixture. ^{1}HNMR ($CDCl_3$, 400 MHz) δ 1.77 (s, 1H), 2.27-2.41 (m, 2H), 4.26-4.31 (m, 1H), 5.07-5.18 (m, 2H), 5.73-5.84 (m,1H), 6.17 (dd, *J* = 16.0, 6.4 Hz, 1H), 6.53 (d, *J* = 16.0 Hz, 1H), 7.11-7.36 (m, 5H); ^{13}C NMR: δ 144.22, 134.74, 128.39, 127.47, 126.08, 117.85, 73.56, 43.72. NMR data were in agreement with those reported in literature. ESI-MS *m/z* 149 $[MH]^+$. $[\alpha]_D$ - 40 (*c* 1.0, $CHCl_3$). e.e. 60 %. The enantiomeric excess was determined by chiral HPLC (Chiralcel OB hexane/2- propanol 95:5, 0.6 mL/min, 254 nm, t_S = 10.90 min. for major enantiomer, t_R = 12.69 min. for minor enantiomer.).

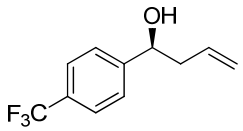
[7] D. A. Evans, Y. Aye, J. Wu, *Org. Lett.* , **2006**, 8 (10), 2071-2073.



(S)-(-)-1-(4-Nitrophenyl)-but-3-en-1-ol.[6] This product was purified with petroleum ether to petroleum ether: Et_2O 60:40 mixture as eluant. ^1H NMR ($CDCl_3$, 400 MHz) δ 8.14 (d, 2 H, J = 8.5 Hz), 7.52 (d, 2 H, J = 8.5 Hz), 5.74-5.82 (m, 1 H), 5.11-5.15 (m, 2 H), 4.87 (t, 1 H, J = 6.0 Hz), 3.17 (bs, 1H, alcohol), 2.45-2.56 (m, 2 H). ^{13}C NMR: δ 151.52, 147.067, 133.36, 126.65, 123.46, 118.96, 72.35, 43.61. NMR data were in agreement with those reported in the literature. ESI-MS *m/z* 194 $[MH]^+$. $[\alpha]_D$ - 50 (*c* 1.0, $CHCl_3$). e.e. 82 %. The enantiomeric excess was determined by chiral HPLC (Chiralcel OD, hexane/2- propanol 99:1, 0.5 mL/min, 210 nm, t_R = 108.93 min for minor enantiomer, t_S = 113.22 min for major enantiomer).



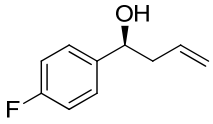
(S)-(-)-1-(2-Nitrophenyl)-but-3-en-1-ol.[8] This product was purified with petroleum ether to petroleum ether: Et_2O 60:40 mixture as eluant. ^1H NMR ($CDCl_3$, 300 MHz) δ 7.78 (1H, dd, J = 8.0, 1.0 Hz), 7.68 (1H, dd, J = 8.0, 1.0 Hz), 7.51 (1H, dt, J = 7.5, 1.0 Hz), 7.28 (1H, dt, J = 8.0, 1.5 Hz), 5.82 - 5.67 (1H, m), 5.20 - 5.01 (2H, m), 2.60 - 2.46 (1H, m), 2.43 (1H, br s), 2.33 - 2.21 (1H, m); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 147.59, 139.21, 133.93, 133.43, 128.07, 128.02, 124.33, 118.99, 68.27, 42.78; NMR data were in agreement with those reported in the literature. ESI-MS *m/z* 194 $[MH]^+$. $[\alpha]_D$ - 31 (*c* 1.0, $CHCl_3$). e.e. 72 %. The enantiomeric excess was determined by chiral HPLC (Chiralcel OD, hexane/2- propanol 99:1, 0.5 mL/min, 210 nm, t_R = 72.50 min for minor enantiomer, t_S = 77.65 min for major enantiomer).



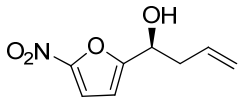
(S)-(-)-1-[(4-Trifluoromethyl)-phenyl]-but-3-en-1-ol.[6] This product was purified with petroleum ether to petroleum ether: Et_2O 60:40 mixture as eluant. ^1H-NMR ($CDCl_3$, 400 MHz) δ 2.00 (s, 1H), 2.35-2.51 (m, 2H), 4.71-4.75 (m, 1H), 5.08-5.13 (m, 2H), 5.67-5.77 (m, 1H), 7.39-7.41 (m, 2H), 7.52-7.54 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 144.0, 134.5, 129.8, 127.4, 125.4, 124.2, 117.0,

[8] A. N. Thadani, R. A. Batey *Org. Lett.,* **2006**, *4*, 3827-3830.

76.8, 44.8. NMR data were in agreement with those reported in the literature. ESI-MS *m/z* 217 $[MH]^+$. $[\alpha]_D$ - 23 (*c* 1.0, $CHCl_3$). e.e. 66 %. The enantiomeric excess was determined by chiral GC (Supelco β-DEX 120 column, oven: 100°C for 5 min, then 1°C/min to 200°C, 10 min at that temperature, t_R = 59.32 min for minor enantiomer, t_S = 59. 94 min for major enantiomer). Anal. calcd. for $C_{11}H_{11}F_3O$: C 61.11; H 5.13; found: C 60.97; H 5.24.



(*S*)-(-)-1-(4-Fluorophenyl)-but-3-en-1-ol.[6] This product was purified with petroleum ether to petroleum ether: Et_2O 60:40 mixture as eluant. ^{1}H-NMR ($CDCl_3$, 400 MHz) δ 2.10 (s, 1H), 2.44-2.57 (m, 2H), 4.72-7.76 (m, 1H), 5.16-5.21 (m, 2H), 5.76-5.86 (m, 1H), 7.02-7.08 (m, 2H), 7.32-7.37 (m, 2H). ^{13}C NMR (50 MHz, $CDCl_3$) δ 164.6, 139.6, 134.1, 127.5, 118.6, 115.4, 72.6, 43.9. NMR data were in agreement with those reported in the literature. ESI-MS *m/z* 167 $[MH]^+$. $[\alpha]_D$ - 22 (*c* 1.0, $CHCl_3$). e.e. 62 %. The enantiomeric excess was determined by chiral GC (Supelco β-DEX 120 column, oven: 100°C for 5 min, then 1°C/min to 200°C, 10 min at that temperature, t_R = 53.88 min for minor enantiomer, t_S = 54.31 min for major enantiomer).

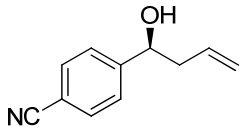


(S)-(-)-1-[(5-Nitro)-2-Furyl]-but-3-en-1-ol[9]**.** This product was purified with petroleum ether to petroleum ether: Et_2O 60:40 mixture as eluant. ^{1}H NMR ($CDCl_3$, 400 MHz) δ 2.56 (br s, 1H), 2.58–2.74 (m, 2H), 4.83 (t, 1H, J = 5.5 Hz), 5.19–5.24 (m, 2H), 5.75–5.81 (m, 1H), 6.52 (d, 1H, J = 3.6 Hz), 7.27 (d, 1H, J = 3.6 Hz). ^{13}C NMR ($CDCl_3$, 100 MHz) δ 39.9, 66.7, 109.4, 112.4, 119.9, 132.0, 150.6, 159.7. ESI-MS m/z 184 [MH]+. .NMR data were in agreement with those reported in the literature. ESI-MS *m/z* 184 $[MH]^+$. $[\alpha]_D$ - 75 (*c* 1.0, $CHCl_3$). e.e. 90 %. The enantiomeric excess was determined by chiral HPLC (Chiralcel AD, hexane/2- propanol 95:5, 0.8 mL/min, 254 nm, t_S = 18.04 min for major enantiomer, t_R = 20.97 min for minor enantiomer). Anal. calcd. for $C_8H_9NO_4$: C 52.46; H 4.95; N 7.65 found: C 52.27; H 4.76; N 7.96.

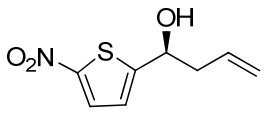
(R)-(+)-1-[(5-Nitro)-2-Furyl]-but-3-en-1-ol.[9] This product was purified with petroleum ether to petroleum ether: Et_2O 60:40 mixture as eluant. ^{1}H NMR ($CDCl_3$, 400 MHz) d 2.56 (br s, 1H),

[9] A. Massa, M. R. Acocella, V. De Sio, R. Villano, A. Scettri, *Tetrahedron Asymmetry* **2009**, 30, 202-204.

2.58–2.74 (m, 2H), 4.83 (t, 1H, J = 5.5 Hz), 5.19–5.24 (m, 2H), 5.75–5.81 (m, 1H), 6.52 (d, 1H, J = 3.6 Hz), 7.27 (d, 1H, J = 3.6 Hz). ^{13}C NMR ($CDCl_3$, 100 MHz) δ 39.9, 66.7, 109.4, 112.4, 119.9, 132.0, 150.6, 159.7. NMR data were in agreement with those reported in the literature. ESI-MS *m/z* 184 $[MH]^+$. $[\alpha]_D$ + 75 (*c* 1.0, $CHCl_3$). e.e. 86 %. The enantiomeric excess was determined by chiral HPLC (Chiralcel AD, hexane/2- propanol 95:5, 0.8 mL/min, 254 nm, t_S = 18.08 min for minor enantiomer, t_R = 20.99 min for major enantiomer). Anal. calcd. for $C_8H_9NO_4$: C 52.46; H 4.95; N 7.65 found: C 52.15; H 4.66; N 7.74.



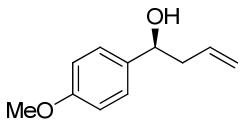
(*S*)-(-)-1-(4-Cyanophenyl)-but-3-en-1-ol.[8] This product was purified with petroleum ether to petroleum ether: Et_2O 60:40 mixture as eluant. ESI-MS *m/z* 174 $[MH]^+$. $[\alpha]_D$ - 37 (*c* 1.0, $CHCl_3$). e.e. 72 %. ^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.58 (2H, d, *J* = 8.5 Hz), 7.43 (2H, d, *J* = 8.0 Hz), 5.79 - 5.68 (1H, m), 5.14 - 5.08 (2H, m), 4.76 (1H, dd, *J* = 7.5, 5.0 Hz), 2.73 (1H, br s), 2.52 - 2.36 (2H, m); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 149.22, 133.27, 132.00, 126.39, 119.01, 118.72, 110.72, 72.24, 43.57. NMR data were in agreement with those reported in the literature. The enantiomeric excess was determined by chiral HPLC (Chiralcel AD, hexane/2- propanol 97:3, 0.8 mL/min, 220 nm, t_R = 38.24 min for minor enantiomer, t_S = 40.65 min for major enantiomer).



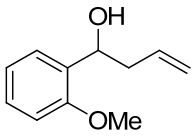
(*S*)-(-)-1-[(5-Nitro)-2-Thiophenyl]-but-3-ene-1-ol.[9] This product was purified with petroleum ether to petroleum ether: Et_2O 60:40 mixture as eluant. ^{1}H NMR ($CDCl_3$, 400 MHz) δ 2.51–2.68 (m, 2H), 2.82 (br s, 1H), 4.97–5.02(dd, 1H, J = 5.1, 7.3 Hz), 5.19–5.30 (m, 2H), 5.72–5.89 (m, 1H),6.89–6.91 (dd, 1H, J = 0.9, 4.2 Hz), 7.80 (d, 1H, J = 4.2). ^{13}C NMR ($CDCl_3$, 100 MHz) δ 43.1, 68.8, 119.9, 122.0, 128.3, 131.8, 145.9, 156.8.NMR data were in agreement with those reported in the literature. ESI-MS *m/z* 200 $[MH]^+$. $[\alpha]_D$ - 6 (*c* 3.0, $CHCl_3$). e.e. 85 %. The enantiomeric excess was determined by chiral HPLC (Chiralcel AD, hexane/2- propanol 95:5, 0.8 mL/min, 254 nm, t_S = 18.38 min for minor enantiomer, t_R = 20.29 min for major enantiomer). Anal. calcd. for $C_8H_9NO_3S$: C 48.23; H 4.55; N 7.03; S 16.09 found: C 48.52; H 4.64; N 7.25; S 16.29.

(*R*)-(+)-1-[(5-Nitro)-2-Thiophenyl]-but-3-ene-1-ol.[9] This product was purified with petroleum ether to petroleum ether: Et_2O 60:40 mixture as eluant. ^{1}H NMR ($CDCl_3$, 400 MHz) δ 2.51–2.68

(m, 2H), 2.82 (br s, 1H), 4.97–5.02 (dd, 1H, J = 5.1, 7.3 Hz), 5.19–5.30 (m, 2H), 5.72–5.89 (m, 1H), 6.89–6.91 (dd, 1H, J = 0.9, 4.2 Hz), 7.80 (d, 1H, J = 4.2). ^{13}C NMR ($CDCl_3$, 100 MHz) δ 43.1, 68.8, 119.9, 122.0, 128.3, 131.8, 145.9, 156.8.NMR data were in agreement with those reported in the literature. ESI-MS *m/z* 200 $[MH]^+$. $[\alpha]_D$ + 6 (*c* 3.0, $CHCl_3$). e.e. 84 %. The enantiomeric excess was determined by chiral HPLC (Chiralcel AD, hexane/2- propanol 95:5, 0.8 mL/min, 254 nm, t_S = 18.30 min for major enantiomer, t_R = 20.24 min for minor enantiomer). Anal. calcd. for $C_8H_9NO_3S$: C 48.23; H 4.55; N 7.03; S 16.09 found: C 48.44; H 4.25; N 7.34; S 16.38.

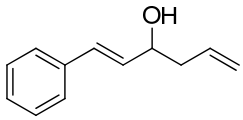


(*S*)-(-)-1-(4-Methoxyphenyl)-but-3-en-1-ol.[6] This product was purified with petroleum ether to petroleum ether: Et_2O 70:30 mixture as eluant. ^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.27 (2H, d, *J* = 8.5 Hz), 6.87(2H, d, *J* = 9.0 Hz), 5.82 - 5.73 (1H, m), 5.18 - 5.09 (2H, m), 4.67 (1H, t, *J* = 6.5 Hz), 3.79 (3H, s),2.48 (2H, t, *J* = 7.0 Hz), 2.04 (1H, br s); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 158.96, 136.00, 134.58, 127.04, 118.21, 113.74, 72.93, 55.24, 43.73. NMR data were in agreement with those reported in the literature. ESI-MS *m/z* 179 $[MH]^+$. $[\alpha]_D$ - 34 (*c* 1.0, $CHCl_3$). e.e. 53 %. The enantiomeric excess was determined by chiral HPLC (Chiralcel OD, hexane/2- propanol 98:2, 0.8 mL/min, 254 nm, t_R = 22.98 min for minor enantiomer, t_S = 27.22 min for major enantiomer).

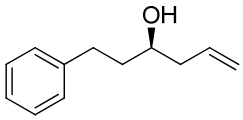


(S)-(-)-1-(2-Methoxyphenyl)-but-3-en-1-ol.[10] This product was purified with petroleum ether to petroleum ether: Et_2O 60:40 mixture as eluant. ^{1}H NMR ($CDCl_3$, 400 MHz) δ 2.39-2.55 (m, 3H), 3.77 (s, 3H), 4.87-4.90 (m, 1H), 5.01-5.09 (m, 2H), 5.72-5.82 (m, 1H), 6.80 (d, *J* = 8.4 Hz, 1H), 6.86-6.90 (t, *J* = 7.6 Hz, 1H), 7.15-7.19 (m, 1H), 7.25 (d, *J* = 8.4 Hz, 1H). ^{13}C NMR (100Hz, $CDCl_3$): δ 41.8, 55.1, 69.2, 110.2, 117.2, 120.5, 126.6, 128.1, 131.8, 135.2, 156.1. NMR data were in agreement with those reported in the literature. ESI-MS *m/z* 179 $[MH]^+$. $[\alpha]_D$ - 30 (*c* 1.0, $CHCl_3$). e.e. 34 %. The enantiomeric excess was determined by chiral HPLC (Chiralcel OD, hexane/2-propanol 99:1, 0.8 mL/min, 220 nm, t_S = 28.32 min for major enantiomer, t_R = 32.45 min for minor enantiomer).

[10] A. V. Malkov, M. Bell, F. Castelluzzo, P. Kocovsky *Org. Lett.* **2005,**Vol**.** 7**,** No. 15, 3219-3222.



1-Phenyl-hexa-1,5-dien-3-ol.[6] This product was purified with petroleum ether to petroleum ether: Et_2O 70:30 mixture. ^1H NMR (300 MHz, $CDCl_3$): δ 7.21-7.40 (m, 5H), 6.60 (d, J = 15.9 Hz, 1H), 6.25 (dd, J =15.9, 6.3 Hz, 1H), 5.79-5.93 (m, 1H), 5.15-5.21 (m, 2H), 4.35-4.37 (m, 1H), 2.33-2.48 (m,2H), 1.80 (br, 1H) ^{13}C NMR (75.4 MHz, $CDCl_3$): δ 136.6, 134.0, 131.5, 130.3, 128.5, 127.6, 126.4, 118.4, 71.6, 42.0. NMR data were in agreement with those reported in the literature. ESI-MS *m/z* 175 $[MH]^+$. e.e. 54 %. The enantiomeric excess was determined by chiral HPLC (Chiralcel OB hexane/2- propanol 95:5, 0,6 mL/min, 254 nm, t_R = 15.09 min for minor enantiomer, t_S = 16.44 min. for major enantiomer).

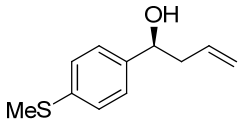


(R)-(+)-1-Phenyl-5-hexen-3-ol.[11] This product was purified with petroleum ether to petroleum ether: Et_2O 70:30 mixture as eluant. ^1H NMR (300 MHz, $CDCl_3$): δ 7.31-7.16 (m, 5H), 5.89-5.75 (m, 1H), 5.17-5.12 (m, 2H), 3.68 (m, 1H), 2.74 (m, 2H), 2.36-2.14 (m, 2H), 1.81-1.76 (m, 2H) ^{13}C NMR (75.4 MHz, CDCl3): δ 142.0, 134.6, 128.4, 125.8, 118.2, 70.0, 42.0, 38.4, 32.0. NMR data were in agreement with those reported in the literature. ESI-MS *m/z* 177 $[MH]^+$. $[\alpha]_D$ + 30 (*c* 1.0, $CHCl_3$). e.e. 74 %. The enantiomeric excess was determined by chiral HPLC (Chiralcel OD, hexane/2- propanol 99:1, 1.0 mL/min, 220 nm, t_S = 16.93 min for minor enantiomer, t_R = 29.37 min for major enantiomer).

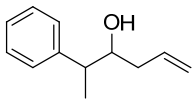
(S)-(-)-1-Phenyl-5-hexen-3-ol.[11] This product was purified with petroleum ether to petroleum ether: Et_2O 70:30 mixture as eluant. ^1H NMR (300 MHz, $CDCl_3$): δ 7.31-7.16 (m, 5H), 5.89-5.75 (m, 1H), 5.17-5.12 (m, 2H), 3.68 (m, 1H), 2.74 (m, 2H), 2.36-2.14 (m, 2H), 1.81-1.76 (m, 2H) ^{13}C NMR (75.4 MHz, $CDCl_3$): δ 142.0, 134.6, 128.4, 125.8, 118.2, 70.0, 42.0, 38.4, 32.0. NMR data in agreement with those reported in the literature. ESI-MS *m/z* 177 $[MH]^+$. $[\alpha]_D$ - 31 (*c* 1.0, $CHCl_3$). e.e. 76 %. The enantiomeric excess was determined by chiral HPLC (Chiralcel OD, hexane/2-

[11] Q. Yao, M. Sheets, *J. Org. Chem.* **2006**, *71*, 5384-5387.

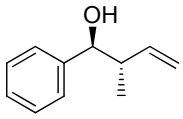
propanol 99:1, 1.0 mL/min, 220 nm, t_S = 16.99 min for major enantiomer, t_R = 29.44 min for minor enantiomer).



(*S*)-(-)-1-(4-Methylthiophenyl)-but-3-en-1-ol.[12] This product was purified with petroleum ether to petroleum ether: Et_2O 70:30 mixture as eluant. ^1H NMR (200 MHz, $CDCl_3$): δ 7.35-7.21 (4H, m,), 5.85-5.71 (1H, m,), 5.19-5.10 (2H, m), 4.70 (1H, dd, J = 3.40 Hz, J = 8.10 Hz), 2.56-2.44 (2H, m), 2.47 (3H, s) 2.20 (1H, s). ^{13}C NMR (50MHz, $CDCl_3$): δ 140.7, 137.3, 134.2, 126.5, 126.3, 118.2, 72.8, 43.5, 15.8. NMR data were in agreement with those reported in the literature. ESI-MS *m/z* 195 $[MH]^+$. $[\alpha]_D$ - 21 (*c* 1.0, $CHCl_3$). e.e. 65 %. The enantiomeric excess was determined by chiral HPLC (Chiralcel AS-H, hexane/2- propanol 95:5, 0.6 mL/min, 254 nm, t_R = 17.68 min for minor enantiomer, t_S = 22.75 min for major enantiomer).



2-phenylhex-5-en-3-ol.[13] This product was purified with petroleum ether to petroleum ether: Et_2O 95:5 mixture as eluant and obtained as a 65/35 diastereoisomeric mixture. ^1H NMR ($CDC1_3$) major isomer δ 1.32 (3 H, d, *J* = 7 Hz), 1.78 (1 H, br s), 1.96-2.24 (2 H, m), 2.68-2.84 (1 H, m), 3.62-3.77 (1 H, m), 5.02-5.16 (2 H, m), 5.70-5.97 (1 H, m), 7.14-7.36 (5 H, m), minor isomer δ 1.28 (3 H, d, *J* = 7 Hz). ^{13}C NMR ($CDCl_3$) major isomer δ 16.3, 39.4, 45.3, 74.9, 117.9, 126.7, 132.7, 128.4 (2 C), 135.0, 143.2, 144.3, minor isomer δ 17.6, 38.9, 117.5, 126.5, 128.1 (2C). NMR data were in agreement with those reported in literature. ESI-MS *m/z* 177 $[MH]^+$. Anal. calcd. for $C_{12}H_{16}O$: C 81.77; H 9.15; found: C 81.65; H 9.26.

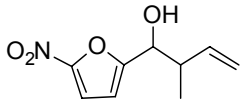


(1*S*,2*S*)-(-)-2-Methyl-1-Phenyl-but-3-en-1-ol (-)-*anti*.[14] This product was purified with petroleum ether to petroleum ether: Et_2O 70:30 mixture as eluant. ^1H NMR ($CDCl_3$, 400 MHz) δ 7.37 - 7.24

[12] M. Bandini, P. G. Cozzi, A. Umani-Ronchi *Tetrahedron* **2001**, 57, 835-843
[13] C. H. Heathcock, S. Kiyooka, T. A. Blumenkopf, *J. Org. Chem*. **1984**, *49*, 4214-4223.

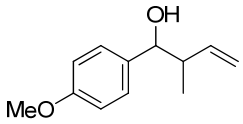
[14] J. F. Traverse, Y. Zhao, A. H. Hoveyda, M. L. Snapper, *Org. Lett.* **2005**, *7*, 3151-3154.

(5H, m), 5.81 (1H, ddd, *J* = 17.0, 10.5, 8.0 Hz), 5.23 - 5.16 (2H, m), 4.34 (1H, d, *J* = 8.0 Hz), 2.48 (1H, sextet, *J* = 7.0 Hz), 2.29 (1H, br s), 0.87 (3H, d, *J* = 7.0 Hz); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 142.4, 140.6, 128.1, 127.5, 126.8, 116.7, 77.7, 46.2, 16.4. NMR data were in agreement with those reported in literature. ESI-MS *m/z* 163 $[MH]^+$. $[\alpha]_D$ - 40 (*c* 1.0, $CHCl_3$). e.e. 68 %. The enantiomeric excess was determined by chiral HPLC (Chiralcel AS-H hexane/2- propanol 95:5, 0.5 mL/min, 220 nm, *t* = 9.96 min. for minor enantiomer, *t* = 11.20 min. for major enantiomer.).



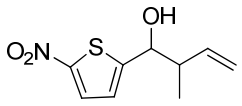
(1*S*,2*S*)-(-)-1-(5-Nitrofuryl)-2-methylbut-3-en-1-ol (-)-*anti*. This product was purified with petroleum ether to petroleum ether: Et_2O 80:20 mixture as eluant. ^{1}H NMR ($CDCl_3$, 400 MHz) δ 1.05 (d, 3H, *J* = 6.8 Hz), 2.68-2.77 (m, 1H), 4.53 (d, 1H, *J* = 6.5 Hz), 5.15-5.21 (m, 2H), 5.67-5.78 (m, 1H), 6.50 (d, 1H, *J* = 3.7 Hz), 7.25 (d, 1H, *J* = 3.7 Hz). ^{13}C NMR (100 MHz, $CDCl_3$) δ 16.0, 43.4, 71.3, 110.2, 112.3, 118.2, 138.0, 151.1, 159.4. ESI-MS *m/z* 198 $[MH]^+$. $[\alpha]_D$ - 102 (*c* 1.0, $CHCl_3$). e.e. 87 %. The enantiomeric excess was determined by chiral HPLC (Chiralcel AD hexane/2- propanol 99:1, 0.6 mL/min, 254 nm, *t* = 61.28 min. for minor enantiomer, *t* = 64.42 min. for major enantiomer.). Anal. calcd. for $C_9H_{11}NO_4$: C 54.82; H 5.62; N 7.10 found: C 54.54; H 5.91; N 7.21.

(1*R*,2*R*)-(+)-1-(5-Nitrofuryl)-2-methylbut-3-en-1-ol (+)-*anti*. This product was purified with petroleum ether to petroleum ether: Et_2O 80:20 mixture as eluant. ^{1}H NMR ($CDCl_3$, 400 MHz) δ 1.05 (d, 3H, *J* = 6.8 Hz), 2.68-2.77 (m, 1H), 4.53 (d, 1H, *J* = 6.5 Hz), 5.15-5.21 (m, 2H), 5.67-5.78 (m, 1H), 6.50 (d, 1H, *J* = 3.7 Hz), 7.25 (d, 1H, *J* = 3.7 Hz). ^{13}C NMR ($CDCl_3$, 100 MHz) δ 16.0, 43.4, 71.3, 110.2, 112.3, 118.2, 138.0, 151.1, 159.4. NMR data were in agreement with those reported for (1*S*,2*S*)-(-)-1-(5-Nitrofuryl)-2-methylbut-3-en-1-ol. ESI-MS *m/z* 198 $[MH]^+$. $[\alpha]_D$ + 102 (*c* 1.0, $CHCl_3$). e.e. 87 %. The enantiomeric excess was determined by chiral HPLC (Chiralcel AD hexane/2- propanol 99:1, 0.6 mL/min, 254 nm, *t* = 61.19 min. for major enantiomer, *t* = 64.50 min. for minor enantiomer.). Anal. calcd. for $C_9H_{11}NO_4$: C 54.82; H 5.62; N 7.10 found: C 54.61; H 5.91; N 7.00.



(1*S*,2*S*)-(-)-1-(4-Methoxyphenyl)-2-methylbut-3-en-1-ol (-)-*anti*.[8] This product was purified with petroleum ether to petroleum ether: Et_2O 80:20 mixture as eluant. ^1H NMR ($CDCl_3$, 400 MHz) δ 7.25 (2H, d, *J* = 9.0 Hz), 6.88 (2H, d, *J* = 9.0 Hz), 5.81 (1H, ddd, *J* = 17.0, 10.5, 8.0 Hz), 5.24 - 5.15 (2H, m), 4.29 (1H, d, *J* = 8.0 Hz), 3.80 (3H, s), 2.45 (1H, sextet, *J* = 7.0 Hz), 2.29 (1H, br s), 0.83 (3H, d, *J* = 7.0 Hz); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 159.0, 140.9, 134.6, 127.9, 116.5, 113.6, 77.4, 55.2, 46.3, 16.5. NMR data were in agreement with those reported in literature. ESI-MS *m/z* 193 $[MH]^+$. $[\alpha]_D$ - 38 (*c* 1.0, $CHCl_3$). e.e. 70 %. The enantiomeric excess was determined by chiral HPLC (Chiralcel AS-H hexane/2- propanol 95:5, 0.6 mL/min, 220 nm, *t* = 12.90 min. for minor enantiomer, *t* = 17.15 min. for major enantiomer.). Anal. calcd. for $C_{12}H_{16}O_2$: C 74.97; H 8.39; found: C 74.85; H 8.50.

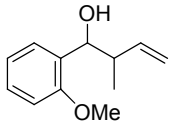
(1*R*,2*R*)-(+)-1-(4-Methoxyphenyl)-2-methylbut-3-en-1-ol (+)-*anti*.[8] This product was purified with petroleum ether to petroleum ether: Et_2O 80:20 mixture as eluant. ^1H NMR ($CDCl_3$, 400 MHz) δ 7.25 (2H, d, *J* = 9.0 Hz), 6.88 (2H, d, *J* = 9.0 Hz), 5.81 (1H, ddd, *J* = 17.0, 10.5, 8.0 Hz), 5.24 - 5.15 (2H, m), 4.29 (1H, d, *J* = 8.0 Hz), 3.80 (3H, s), 2.45 (1H, sextet, *J* = 7.0 Hz), 2.29 (1H, br s), 0.83 (3H, d, *J* = 7.0 Hz); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 159.0, 140.9, 134.6, 127.9, 116.5, 113.6, 77.4, 55.2, 46.3, 16.5. NMR data were in agreement with those reported in literature. ESI-MS *m/z* 193 $[MH]^+$. $[\alpha]_D$ + 43 (*c* 1.0, $CHCl_3$). e.e. 77 %. The enantiomeric excess was determined by chiral HPLC (Chiralcel AS-H hexane/2- propanol 95:5, 0.6 mL/min, 220 nm, *t* = 12.14 min. for major enantiomer, *t* = 15.92 min. for minor enantiomer.). Anal. calcd. for $C_{12}H_{16}O_2$: C 74.97; H 8.39; found: C 74.71; H 8.61.



(1*S*,2*S*)-(-)-1-(5-Nitrothiophenyl)-2-methylbut-3-ene-1-ol (-)-*anti*. This product was purified with petroleum ether to petroleum ether: Et_2O 80:20 mixture as eluant. ^1H NMR ($CDCl_3$, 400 MHz) δ 1.04 (d, 3H, *J* = 6.8 Hz), 2.47-2.54 (m, 1H), 4.68 (d, 1H, *J* = 6.5 Hz), 5.17-5.24 (m, 2H), 5.68-5.80 (m, 1H), 6.88 (d, 1H, *J* = 4.1 Hz), 7.78 (d, 1H, *J* = 4.1 Hz). ^{13}C NMR ($CDCl_3$, 100 MHz) δ 16.0, 45.5, 73.8, 118.7, 123.4, 128.3, 138.2, 146.6, 156.0. ESI-MS *m/z* 214 $[MH]^+$. $[\alpha]_D$ - 23 (*c* 3.0,

$CHCl_3$). e.e. 82 %. The enantiomeric excess was determined by chiral HPLC (Chiralcel OD hexane/2- propanol 95:5, 0.6 mL/min, 254 nm, *t* = 20.35 min. for major enantiomer, *t* = 23.18 min. for minor enantiomer.). Anal. calcd. for $C_9H_{11}NO_3S$: C 50.69; H 5.20; N 6.57; S 15.04 found: C 50.99; H 5.01; N 6.76; S 15.33.

(1*R*,2*R*)-(+)-1-(5-Nitrothiophenyl)-2-methylbut-3-ene-1-ol (+)-*anti*. This product was purified with petroleum ether to petroleum ether: Et_2O 80:20 mixture as eluant. ^1H NMR ($CDCl_3$, 400 MHz) δ 1.04 (d, 3H, *J* = 6.8 Hz), 2.47-2.54 (m, 1H), 4.68 (d, 1H, *J* = 6.5 Hz), 5.17-5.24 (m, 2H), 5.68-5.80 (m, 1H), 6.88 (d, 1H, *J* = 4.1 Hz), 7.78 (d, 1H, *J* = 4.1 Hz). ^{13}C NMR ($CDCl_3$, 100 MHz) δ 16.0, 45.5, 73.8, 118.7, 123.4, 128.3, 138.2, 146.6, 156.0. NMR data were in agreement with those reported for (1*S,*2*S*)-(-)-1-(5-Nitrothiophenyl)-2-methylbut-3-ene-1-ol. ESI-MS *m/z* 214 $[MH]^+$. $[\alpha]_D$ + 29 (*c* 3.0, $CHCl_3$).). e.e. 84 %. The enantiomeric excess was determined by chiral HPLC (Chiralcel OD hexane/2- propanol 95:5, 0.6 mL/min, 254 nm, *t* = 20.58 min. for minor enantiomer, *t* = 23.46 min. for major enantiomer.). Anal. calcd. for $C_9H_{11}NO_3S$: C 50.69; H 5.20; N 6.57; S 15.04 found: C 50.99; H 5.04; N 6.68; S 15.34.

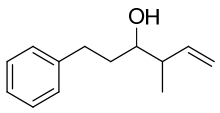


(1*S*,2*S*)-(-)-1-(2-Methoxyphenyl)-2-methylbut-3-en-1-ol (-)-*anti*.[15] This product was purified with petroleum ether to petroleum ether: Et_2O 80:20 mixture as eluant. ^1H NMR (300 MHz, $CDCl_3$) δ 0.91 (d, *J* = 6.9 Hz, 3H), 2.25-2.57 (br, 1H), 2.53-2.69 (m, 1H), 3.83 (s, 3H), 4.69 (d, *J* = 7.55Hz, 1H), 5.06-5.15 (m, 2H), 5.81-5.96 (m, 1H), 6.81-6.99 (m, 2H), 7.16-7.32 (m, 2H). ^{13}C NMR (75 MHz, $CDCl_3$) 16.9, 44.8, 55.4, 73.9, 110.6, 115.8, 120.8, 128.3, 128.4, 131.0, 141.2, 156.9. NMR data were in agreement with those reported in literature. ESI-MS *m/z* 193 $[MH]^+$. $[\alpha]_D$ - 21 (*c* 1.0, $CHCl_3$). e.e. 58 %. The enantiomeric excess was determined by chiral HPLC (Chiralcel OD hexane/2- propanol 95:5, 0.6 mL/min, 220 nm, *t* = 11.09 min. for major enantiomer, *t* = 12.01 min. for minor enantiomer.). Anal. calcd. for $C_{12}H_{16}O_2$: C 74.97; H 8.39; found: C 74.85; H 8.50.

(1*R*,2*R*)-(+)-1-(2-Methoxyphenyl)-2-methylbut-3-en-1-ol (+)-*anti*.[14] This product was purified with petroleum ether to petroleum ether: Et_2O 80:20 mixture as eluant. ^1H NMR (300 MHz, $CDCl_3$)

[15] Li Jiaming, Zha Zhenggen, Sun Lilin, Zhang Yan, Wang Zhiyong. *Chemistry lett.* **2006**, 35, *5*, 498-499.

δ 0.91 (d, *J* = 6.9 Hz, 3H), 2.25-2.57 (br, 1H), 2.53-2.69 (m, 1H), 3.83 (s, 3H), 4.69 (d, *J* = 7.55Hz, 1H), 5.06-5.15 (m, 2H), 5.81-5.96 (m, 1H), 6.81-6.99 (m, 2H), 7.16-7.32 (m, 2H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 16.9, 44.8, 55.4, 73.9, 110.6, 115.8, 120.8, 128.3, 128.4, 131.0, 141.2, 156.9. NMR data were in agreement with those reported in literature. ESI-MS *m/z* 193 $[MH]^+$. $[\alpha]_D$ + 29 (*c* 1.0, $CHCl_3$). e.e. 60 %. The enantiomeric excess was determined by chiral HPLC (Chiralcel OD hexane/2- propanol 95:5, 0.6 mL/min, 220 nm, *t* = 10.87 min. for minor enantiomer, *t* = 11.71 min. for major enantiomer.). Anal. calcd. for $C_{12}H_{16}O_2$: C 74.97; H 8.39; found: C 74.73; H 8.55.

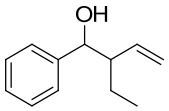


(3*R*,4*S*)-(+)-4-Methyl-1-phenyl-5-hexen-3-ol (+)-*anti*.[16] This product was purified with petroleum ether to petroleum ether: Et_2O 85:15 mixture as eluant. ^{1}H NMR (300 MHz, $CDCI_3$) δ 7.14-7.30 (m, 5 H), 5.73 (ddd, J = 18.0, 11.5, 8.5 Hz, I H), 5.09-5.16 (m, 2 H), 3.40 (m, 1 H), 2.84 (m, 1 H), 2.67 (m, 1 H), 2.22 (m, 1 H), 1.55-1.90 (m, 3 H), 1.02 (d, *J* = 6.8 Hz, 3 H). ^{13}C NMR δ 11.0, 22.6, 53.8, 75.8, 118.2, 126.1, 126.8, 127.4, 138.3, 141.7. NMR data were in agreement with those reported in literature. ESI-MS *m/z* 191 $[MH]^+$. $[\alpha]_D$ + 10 (*c* 2.0, $CHCl_3$). e.e. 64 %. The enantiomeric excess was determined by chiral HPLC (Chiralcel OD hexane/2- propanol 97:3, 1.0 mL/min, 254 nm, *t* = 12.00 min. for minor enantiomer, *t* = 18.15 min. for major enantiomer.).

(3*S*,4*R*)-(-)-4-Methyl-1-phenyl-5-hexen-3-ol (-)-*anti*.[16] This product was purified with petroleum ether to petroleum ether: Et_2O 85:15 mixture as eluant. ^{1}H NMR (300 MHz, $CDCI_3$) δ 7.14-7.30 (m, 5 H), 5.73 (ddd, J = 18.0, 11.5, 8.5 Hz, I H), 5.09-5.16 (m, 2 H), 3.40 (m, 1 H), 2.84 (m, 1 H), 2.67 (m, 1 H), 2.22 (m, 1 H), 1.55-1.90 (m, 3 H), 1.02 (d, *J* = 6.8 Hz, 3 H). ^{13}C NMR δ 11.0, 22.6, 53.8, 75.8, 118.2, 126.1, 126.8, 127.4, 138.3, 141.7. NMR data were in agreement with those reported for (3*R*,4*S*)-(+)-4-Methyl-1-phenyl-5-hexen-3-ol (+)-*anti*. ESI-MS *m/z* 191 $[MH]^+$. $[\alpha]_D$ - 10 (*c* 2.0, $CHCl_3$). e.e. 64 %. The enantiomeric excess was determined by chiral HPLC (Chiralcel OD

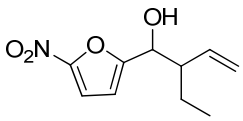
[16] W. R. Roush, K. Ando, D. B. Powers, A. D. Palkowitz, R. L. Halterman, *J. Am. Chem. Soc*, **1990**, *112 (17)*, 6339-6348.

hexane/2- propanol 97:3, 1,0 mL/min, 254 nm, *t* = 12.24 min. for major enantiomer, *t* = 18.65 min. for minor enantiomer.).



(1*S*,2*S*)-(-)-2-Ethyl-1-Phenyl-but-3-en-1-ol (-)-*anti*.[17] This product was purified with petroleum ether to petroleum ether: Et_2O 70:30 mixture as eluant. ^{1}H NMR δ ($CDCl_3$, 400 MHz) 0.81 (t, 3H, *J* = 7.4 Hz), 1.16-1.30 (m, 2H), 2.02 (brs, 1H), 2.15-2.24 (m, 1H), 4.41 (d, 1H, *J* = 7.9 Hz), 5.17-5.30 (m, 2H), 5.60-5.72 (m, 2H), 7.26-7.36 (m, 5H). ^{13}C NMR ($CDCl_3$, 100 MHz) δ 11.0, 22.6, 53.8, 75.8, 118.2, 126.1, 126.8, 127.4, 138.3, 141.7. NMR data were in agreement with those reported in literature. ESI-MS *m/z* 177 $[MH]^+$. $[\alpha]_D$ - 32 (*c* 1.0, $CHCl_3$). e.e. 72 %. The enantiomeric excess was determined by chiral HPLC (Chiralcel AS-H hexane/2- propanol 97:3, 0.5 mL/min, 220 nm, *t* = 11.67 min. for minor enantiomer, *t* = 13.74 min. for major enantiomer.). Anal. calcd. for $C_{12}H_{16}O$: C 81.77; H 9.15 found: C 81.48; H 9.46.

(1*R*,2*R*)-(+)-2-Ethyl-1-Phenyl-but-3-en-1-ol (+)-*anti*.[16] This product was purified with petroleum ether to petroleum ether: Et_2O 70:30 mixture as eluant. ^{1}H NMR ($CDCl_3$, 400 MHz) δ 0.81 (t, 3H, *J* = 7.4 Hz), 1.16-1.30 (m, 2H), 2.02 (brs, 1H), 2.15-2.24 (m, 1H), 4.41 (d, 1H, *J* = 7.9 Hz), 5.17-5.30 (m, 2H), 5.60-5.72 (m, 2H), 7.26-7.36 (m, 5H). ^{13}C NMR ($CDCl_3$, 100 MHz)δ 11.0, 22.6, 53.8, 75.8, 118.2, 126.1, 126.8, 127.4, 138.3, 141.7. NMR data were in agreement with those reported in literature. ESI-MS *m/z* 177 $[MH]^+$. $[\alpha]_D$ + 30 (*c* 1.0, $CHCl_3$). e.e. 72 %. The enantiomeric excess was determined by chiral HPLC (Chiralcel AS-H hexane/2- propanol 97:3, 0.5 mL/min, 220 nm, *t* = 11.94 min. for major enantiomer, *t* = 13.70 min. for minor enantiomer.). Anal. calcd. for $C_{12}H_{16}O$: C 81.77; H 9.15 found: C 81.22; H 9.64.

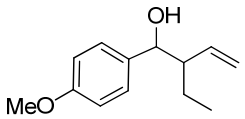


(1*S*,2*S*)-(-)-1-(5-Nitrofuryl)-2-ethylbut-3-en-1-ol (-)-*anti*. This product was purified with petroleum ether to petroleum ether: Et_2O 70:30 mixture as eluant. ^{1}H NMR ($CDCl_3$, 400 MHz) δ 0.88 (t, 3H, *J* = 7.4 Hz), 1.34-1.48 (m, 2H), 2.43-2.47 (m, 1H), 4.63 (d, 1H, *J* = 6.3 Hz), 5.13-5.26

[17] P. Jones, P. Knochel, *J. Org. Chem*. **1999**, *64*, 186-195.

(m, 2H), 5.56-5.68 (m, 1H), 6.51 (d, 1H, *J* = 3.7 Hz), 7.25 (d, 1H, *J* = 3.7 Hz). ^{13}C NMR ($CDCl_3$, 100 MHz)δ 10.5, 22.3, 50.3, 69.0, 109.3, 110.7, 118.0, 135.6, 149.9, 158.6. ESI-MS *m/z* 212 $[MH]^+$. $[\alpha]_D$ - 76 (*c* 1.0, $CHCl_3$). e.e. 86 %. The enantiomeric excess was determined by chiral HPLC (Chiralcel AD-H hexane/2- propanol 97:3, 0.6 mL/min, 254 nm, *t* = 29.48 min. for major enantiomer, *t* = 31.27 min. for minor enantiomer.). Anal. calcd. for $C_{10}H_{13}NO_4$: C 56.86; H 6.20; N 6.63 found: C 56.47; H 6.71; N 6.91.

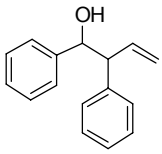
(1*R*,2*R*)-(+)-1-(5-Nitrofuryl)-2-ethylbut-3-en-1-ol (+)-*anti*. This product was purified with petroleum ether to petroleum ether: Et_2O 70:30 mixture as eluant. ^{1}H NMR ($CDCl_3$, 400 MHz) δ 0.88 (t, 3H, *J* = 7.4 Hz), 1.34-1.48 (m, 2H), 2.43-2.47 (m, 1H), 4.63 (d, 1H, *J* = 6.3 Hz), 5.13-5.26 (m, 2H), 5.56-5.68 (m, 1H), 6.51 (d, 1H, *J* = 3.7 Hz), 7.25 (d, 1H, *J* = 3.7 Hz). ^{13}C NMR ($CDCl_3$, 100 MHz) δ 10.5, 22.3, 50.3, 69.0, 109.3, 110.7, 118.0, 135.6, 149.9, 158.6. NMR data were in agreement with those reported for (1*S*,2*S*)-(-)-1-(5-Nitrofuryl)-2-ethylbut-3-en-1-ol. ESI-MS *m/z* 212 $[MH]^+$. $[\alpha]_D$ + 75 (*c* 1.0, $CHCl_3$). e.e. 85 %. The enantiomeric excess was determined by chiral HPLC (Chiralcel AD-H hexane/2- propanol 97:3, 0.6 mL/min, 254 nm, *t* = 28.84 min. for minor enantiomer, *t* = 30.60 min. for major enantiomer.). Anal. calcd. for $C_{10}H_{13}NO_4$: C 56.86; H 6.20; N 6.63 found: C 56.62; H 6.36; N 6.84.



(1*S*,2*S*)-(-)-1-(4-Methoxyphenyl)-2-ethylbut-3-en-1-ol (-)-*anti*.[18] This product was purified with petroleum ether to petroleum ether: Et_2O 70:30 mixture as eluant. ^{1}H NMR ($CDCl_3$, 400 MHz) δ 0.87 (t, 3H, *J* = 7.4 Hz), 1.17-1.37 (m, 2H), 2.20-2.36 (m, 1H), 3.90 (s, 3H), 4.43 (d, 1H, *J* = 8.1 Hz), 5.26-5.38 (m, 2H), 5.67-5.82 (m, 1H), 6.96 (d, 2H, *J* = 7.3 Hz), 7.34 (d, 2H, *J* = 7.3 Hz). ^{13}C NMR ($CDCl_3$, 100 MHz) δ 10.7, 22.4, 53.7, 54.2, 75.2, 112.6, 117.8, 127.1, 133.7, 138.4, 158.1. NMR data were in agreement with those reported in literature. ESI-MS *m/z* 207 $[MH]^+$. $[\alpha]_D$ - 47 (*c* 1.0, $CHCl_3$). e.e. 74 %. The enantiomeric excess was determined by chiral HPLC (Chiralcel AS-H hexane/2- propanol 95:5, 0.6 mL/min, 220 nm, *t* = 11.14 min. for minor enantiomer, *t* = 14.13 min. for major enantiomer.). Anal. calcd. for $C_{13}H_{18}O_2$: C 75.69; H 8.80 found: C 75.70; H 8.46.

[18] Y. Ahn, W. W. Doubleday, T. Cohen, *Syn. Comm.* **1995**, *25*, 33-41.

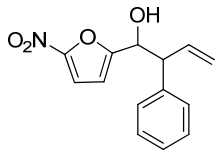
(1*R*,2*R*)-(+)-1-(4-Methoxyphenyl)-2-ethylbut-3-en-1-ol (+)-*anti.*[17] This product was purified with petroleum ether to petroleum ether: Et_2O 70:30 mixture as eluant. ^1H NMR ($CDCl_3$, 400 MHz) δ 0.87 (t, 3H, *J* = 7.4 Hz), 1.17-1.37 (m, 2H), 2.20-2.36 (m, 1H), 3.90 (s, 3H), 4.43 (d, 1H, *J* = 8.1 Hz), 5.26-5.38 (m, 2H), 5.67-5.82 (m, 1H), 6.96 (d, 2H, *J* = 7.3 Hz), 7.34 (d, 2H, *J* = 7.3 Hz). ^{13}C NMR ($CDCl_3$, 100 MHz) δ 10.7, 22.4, 53.7, 54.2, 75.2, 112.6, 117.8, 127.1, 133.7, 138.4, 158.1. NMR data were in agreement with those reported in literature. ESI-MS *m/z* 207 $[MH]^+$. $[\alpha]_D$ + 48 (*c* 1.0, $CHCl_3$). e.e. 80 %. The enantiomeric excess was determined by chiral HPLC (Chiralcel AS-H hexane/2- propanol 95:5, 0.6 mL/min, 220 nm, *t* = 11.22 min. for major enantiomer, *t* = 14.65 min. for minor enantiomer.). Anal. calcd. for $C_{13}H_{18}O_2$: C 75.69; H 8.80 found: C 75.38; H 8.97.



(1*S*,2*R*)-(-)-1,2-Diphenyl-but-3-en-1-ol (-)-*anti.*[19] This product was purified with petroleum ether to petroleum ether: Et_2O 70:30 mixture as eluant. ^1H NMR (300 MHz, $CDCl_3$) δ 7.26-7.04 (m, 10H), 6.26 (ddd, *J* = 16.8, 10.2 and 8.7 Hz, 1H), 5.29-5.21 (m, 2H), 4.85 (dd, *J* = 7.8 and 2.4 Hz, 1H), 3.55 (t, *J* = 8.4 Hz, 1H), 2.30 (d, *J* = 2.7 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 142.1, 140.9, 138.1, 128.6, 128.2, 127.7, 126.9, 118.7, 77.5, 59.5;NMR data were in agreement with those reported in literature. ESI-MS *m/z* 225 $[MH]^+$. $[\alpha]_D$ - 25 (*c* 3.0, $CHCl_3$). e.e. 68 %. The enantiomeric excess was determined by chiral HPLC (Chiralcel OD hexane/2- propanol 98:2, 0.5 mL/min, 254 nm, *t* = 42.70 min. for minor enantiomer, *t* = 46.82 min. for major enantiomer.).

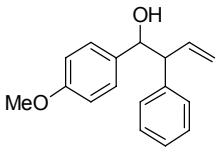
(1*R*,2*S*)-(+)-1,2-Diphenyl-but-3-en-1-ol (+)-*anti.*[18] This product was purified with petroleum ether to petroleum ether: Et_2O 70:30 mixture as eluant. ^1H NMR (300 MHz, $CDCl_3$) δ 7.26-7.04 (m, 10H), 6.26 (ddd, *J* = 16.8, 10.2 and 8.7 Hz, 1H), 5.29-5.21 (m, 2H), 4.85 (dd, *J* = 7.8 and 2.4 Hz, 1H), 3.55 (t, *J* = 8.4 Hz, 1H), 2.30 (d, *J* = 2.7 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 142.1, 140.9, 138.1, 128.6, 128.2, 127.7, 126.9, 118.7, 77.5, 59.5;NMR data were in agreement with those reported in literature. ESI-MS *m/z* 225 $[MH]^+$. $[\alpha]_D$ + 25 (*c* 3.0, $CHCl_3$). e.e. 70 %. The enantiomeric excess was determined by chiral HPLC (Chiralcel OD hexane/2- propanol 98:2, 0.5 mL/min, 254 nm, *t* = 42.60 min. for major enantiomer, *t* = 46.69 min. for minor enantiomer.).

[19] M. Bandini, P. Cozzi, A. Umani-Ronchi, *Ang. Chem.* **2000**, (39) 13, 2327-2330.



(1*S*,2*R*)-(-)-1-(5-Nitrofuryl)-2-phenylbut-3-en-1-ol (-)-*anti.* This product was purified with petroleum ether to petroleum ether: Et_2O 90:10 mixture as eluant. ^1H NMR ($CDCl_3$, 400 MHz) δ 2.65 (brs, 1H), 3.83 (t, 1H, *J* = 8.2 Hz, *J* = 7.1 Hz), 4.95 (t, 1H, *J* = 7.1 Hz, *J* = 3.48 Hz), 5.22-5.30 (m, 2H), 6.11-6.23 (m, 1H), 6.30 (d, 1H, *J* = 3.7 Hz), 7.13-7.29 (m, 6H). ^{13}C NMR ($CDCl_3$, 100 MHz) δ 54.6, 70.1, 109.7, 111.2, 117.4, 118.6, 126.3, 126.9, 127.8, 134.4, 138.1, 157.1. ESI-MS *m/z* 260 $[MH]^+$. $[\alpha]_D$ - 15 (*c* 1.0, $CHCl_3$). e.e. 97 %. The enantiomeric excess was determined by chiral HPLC (Chiralcel AS-H hexane/2- propanol 98:2, 0.5 mL/min, 220 nm, *t* = 92.80 min. for major enantiomer, *t* = 100.01 min. for minor enantiomer.). Anal. calcd. for $C_{14}H_{13}NO_4$: C 64.86; H 5.05; N 5.40 found: C 64.67; H 5.24; N 5.68.

(1*R*,2*S*)-(+)-1-(5-Nitrofuryl)-2-phenylbut-3-en-1-ol (+)-*anti.* This product was purified with petroleum ether to petroleum ether: Et_2O 90:10 mixture as eluant. NMR data were in agreement with those reported for (1*S*,2*R*)-(-)-1-(5-Nitrofuryl)-2-phenylbut-3-en-1-ol. ESI-MS *m/z* 260 $[MH]^+$. $[\alpha]_D$ + 14 (*c* 1.0, $CHCl_3$). e.e. 97 %. The enantiomeric excess was determined by chiral HPLC (Chiralcel AS-H hexane/2- propanol 98:2, 0.5 mL/min, 220 nm, *t* = 92.70 min. for minor enantiomer, *t* = 99.92 min. for major enantiomer.). Anal. calcd. for $C_{14}H_{13}NO_4$: C 64.86; H 5.05; N 5.40 found: C 64.64; H 5.17; N 5.61.

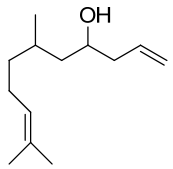


(1*S*,2*R*)-(-)-1-(4-Methoxyphenyl)-2-phenylbut-3-en-1-ol (-)-*anti.*[20] This product was purified with petroleum ether to petroleum ether: Et_2O 80:20 mixture as eluant. ^1H NMR (300 MHz, $CDCl_3$) δ 7.17-7.07 (m, 7H, Ar-H), 6.79-6.68 (m, 2H, Ar-H), 6.22 (ddd, *J* = 17.1, 10.2 and 8.4 Hz, 1H, CH), 5.14-4.98 (m, 3H, CH and CH_2), 3.72-3.65 (m, 4H, CH and CH_3), 2.59 (d, *J* = 6.0 Hz, 1H, OH); ^{13}C NMR (75 MHz, $CDCl_3$) δ 156.4, 141.7, 138.0, 130.2, 128.3, 128.1, 128.0, 126.3, 120.4, 117.6,

[20] S. Zhu, Y. Yang, L. Wang, B. Liu, Q. Zhou, *Org. Lett.,* **2005**, 7 (12), 2333-2335.

110.3, 73.9, 56.7, 55.2. NMR data were in agreement with those reported in literature. ESI-MS *m/z* 255 $[MH]^+$. $[\alpha]_D$ - 12 (*c* 2.0, $CHCl_3$). e.e. 66 %. The enantiomeric excess was determined by chiral HPLC (Chiralcel AD-H hexane/2- propanol 98:2, 0.9 mL/min, 220 nm, *t* = 41.79 min. for minor enantiomer, *t* = 47.69 min. for major enantiomer.).

(1*R*,2*S*)-(+)-1-(4-Methoxyphenyl)-2-phenylbut-3-en-1-ol (+)-*anti.*[19] This product was purified with petroleum ether to petroleum ether: Et_2O 80:20 mixture as eluant. ^1H NMR (300 MHz, $CDCl_3$) δ 7.17-7.07 (m, 7H, Ar-H), 6.79-6.68 (m, 2H, Ar-H), 6.22 (ddd, *J* = 17.1, 10.2 and 8.4 Hz, 1H, CH), 5.14-4.98 (m, 3H, CH and CH_2), 3.72-3.65 (m, 4H, CH and CH_3), 2.59 (d, *J* = 6.0 Hz, 1H, OH); ^{13}C NMR (75 MHz, $CDCl_3$) δ 156.4, 141.7, 138.0, 130.2, 128.3, 128.1, 128.0, 126.3, 120.4, 117.6, 110.3, 73.9, 56.7, 55.2. NMR data were in agreement with those reported in literature. ESI-MS *m/z* 255 $[MH]^+$. $[\alpha]_D$ + 12 (*c* 2.0, $CHCl_3$). e.e. 66 %. The enantiomeric excess was determined by chiral HPLC (Chiralcel AD-H hexane/2- propanol 98:2, 0.9 mL/min, 220 nm, *t* = 41.25 min. for major enantiomer, *t* = 47.47 min. for minor enantiomer.).

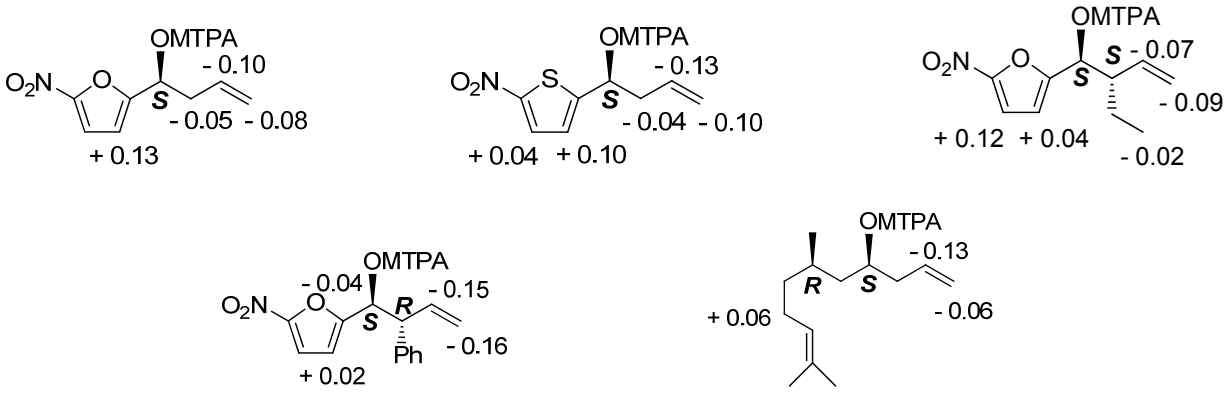


(4*R*,6*R*)/ (4*S*,6*R*)-10-dimethylundeca-1,9-dien-4-ol.[21] This product was purified with petroleum ether to petroleum ether: Et_2O 80:20 mixture as eluant. ^1H NMR (400 MHz, $CDCl_3$) δ (ppm): 5.79 (ddt, *J* = 16.5, 10.7, 7.4 Hz, 1H), 5.13 (bd, *J* = 16.5 Hz, 1H), 5.06 (bd, *J* = 10.7 Hz, 1H), 5.06 (bt, *J* = 6.5 Hz, 1H), 3.75-3.70 (m, 1H), 2.30-1.94(m, 4H), 1.66 (s, 3H), 1.58 (s, 3H), 1.50-1.17 (m, 5H), 0.88 (d, *J* = 6.2 Hz, 3H, ***R,R*-11**), 0.84 (d, *J* = 6.2 Hz, 3H, ***SR*-12**); ^{13}C NMR (100 MHz, $CDCl_3$; DEPT) δ (ppm): 134.9 (CH), 131.5 (C), 124.8 (CH), 118.1 (CH_2), 68.7 (CH), 44.3 (CH_2), 42.2 (CH_2), 37.9 (CH_2), 28.9 (CH), 25.7(CH_3), 25.4 (CH_2), 20.2 (CH_3), 17.7 (CH_3); NMR data were in agreement with those reported in literature. ESI-MS *m/z* 197 $[MH]^+$. Anal. calcd. for $C_{13}H_{24}O$: C 79.53; H 12.32; found: C 79.71; H 12.43.

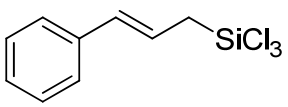
General Procedure for the Determination of the Absolute Configuration by Mosher's Esters.

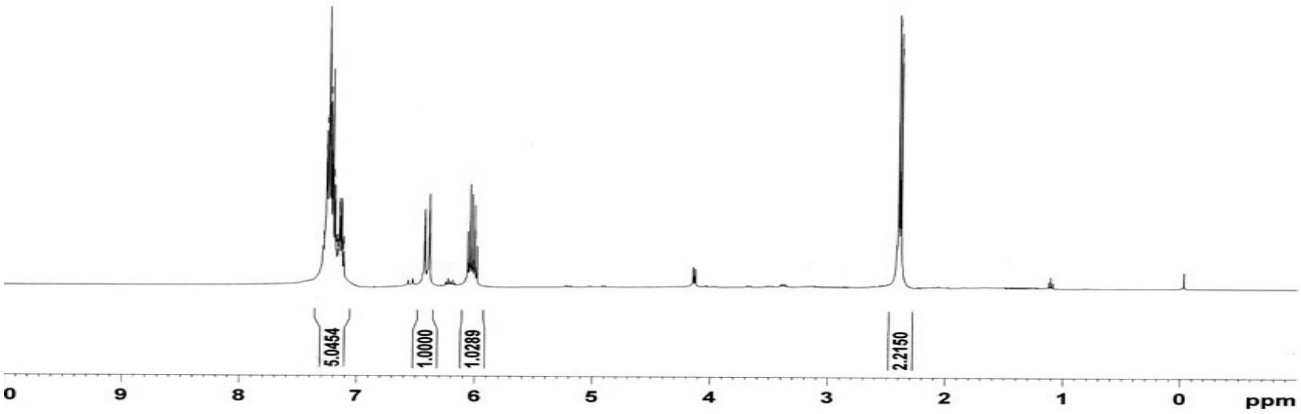
[21] R.E. Estevez, J. Justicia, B. Bazdi, N. Fuentes, M. Paradas, D. Choquesillo-Lazarate, J. M. Garcia-Ruiz, R. Robles, A Gansauer, J. M. Cuerva, J. E. Oltra, *Chem. Eur. J.*, **2009**, *15*, 2774-2791.

In a 5 ml flask, pyridine (67 µL, 0.83 mmol), 4-dimethilaminopyridine (0.4 mg, 0.0031 mmol) and (*R*) or (*S*)-α-methoxy-α-trifluoromethyl-phenylacetic acid chloride (29 µL, 0.155 mmol) were added to the homoallylic alcohol (0.031 mmol) at room temperature. After 30 min. of stirring at that temperature, the reaction was quenched with solution 1M of HCl (1 ml), extracted with 1x3 mL of CH_2Cl_2, dried over anhydrous Na_2SO_4 and filtered. After removing the solvent under reduced pressure, the crude was purified by silica gel flash chromatography (eluent Hex/Et_2O 9:1) to give the pure (*S*) or (*R*) Mosher' ester respectively.



The ^{1}H-NMR and ^{13}C-NMR spectra of new compounds.

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0
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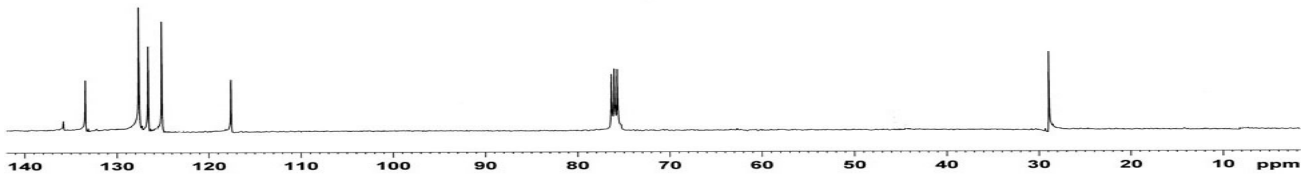
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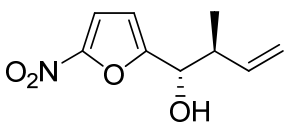
120
110
100
90
80
70
60
50
40
30
20
10
ppm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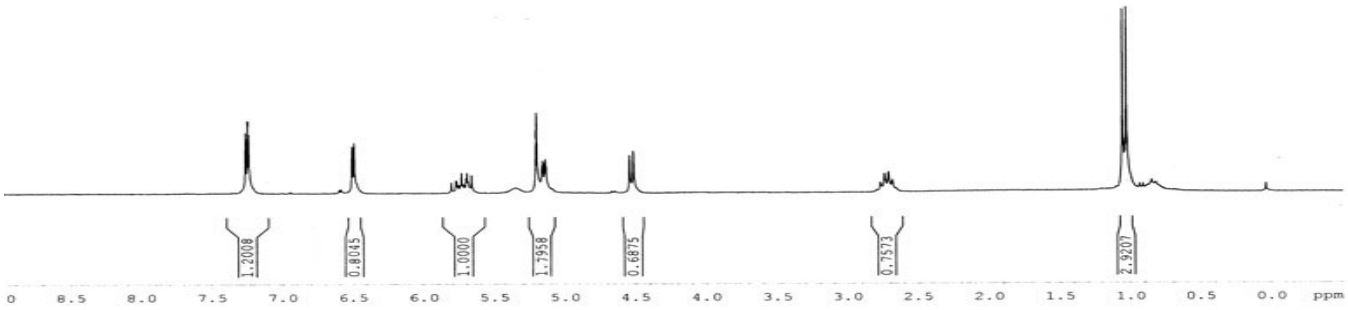
O
OH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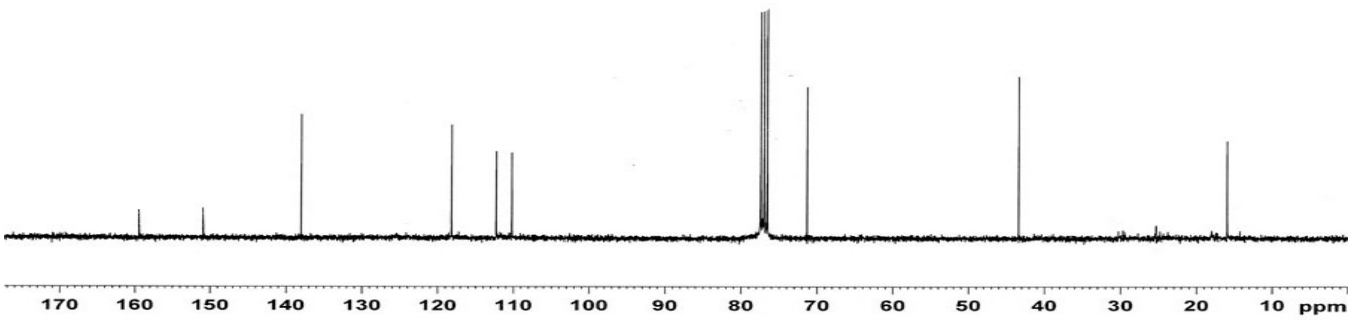
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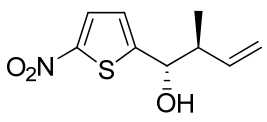
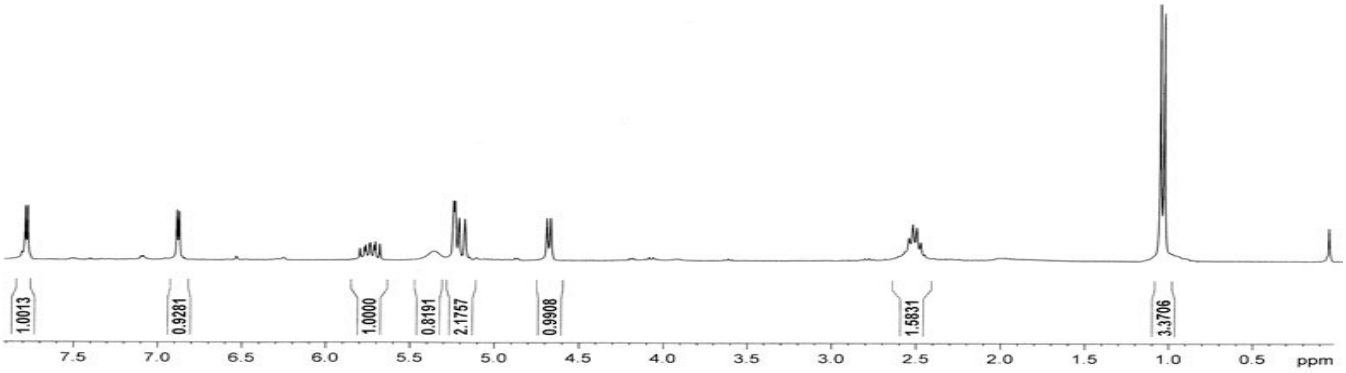
10
ppm


O2N
S
OH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4.0
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2.0
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1.0
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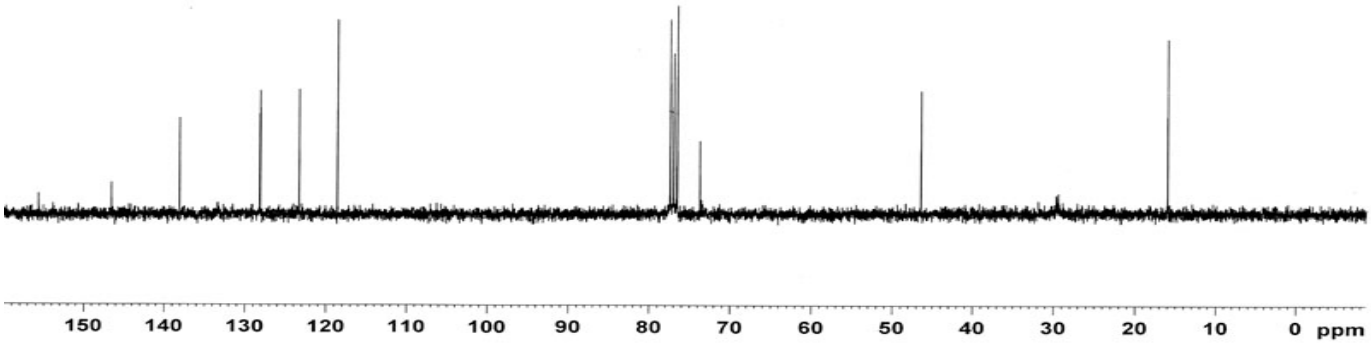
ppm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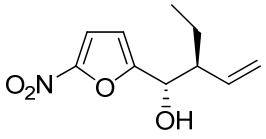
O2N
O
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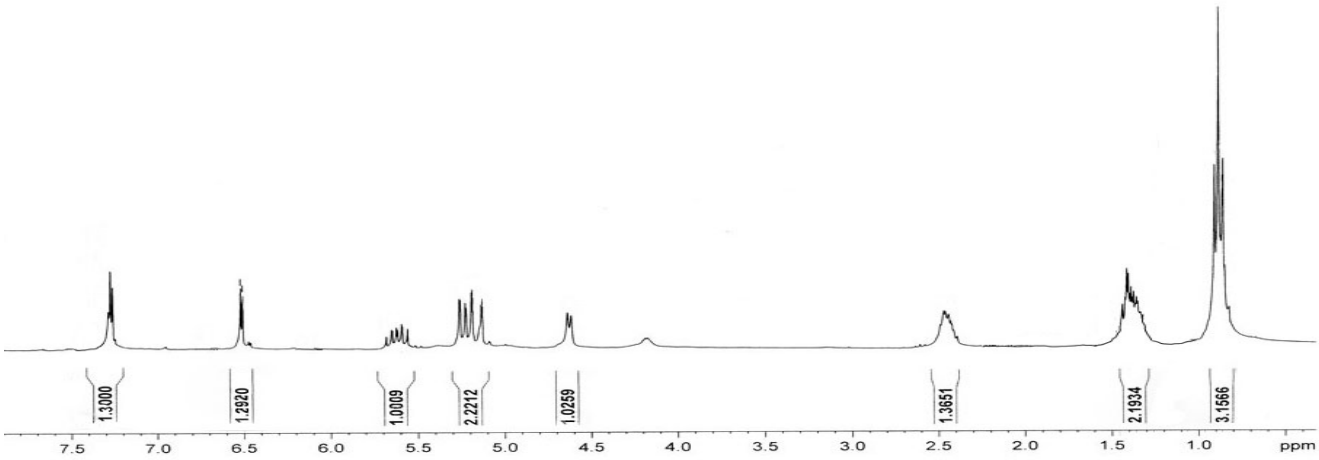
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5.0
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4.0
3.5
3.0
2.5
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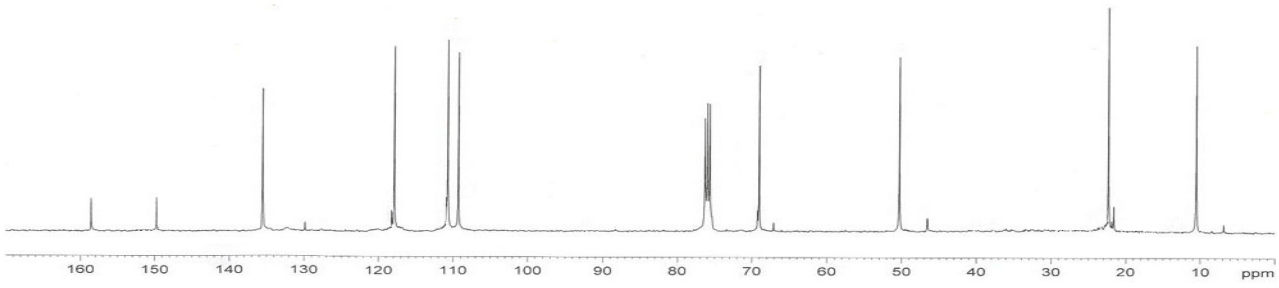
1.5
1.0
ppm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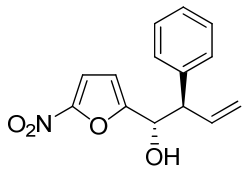
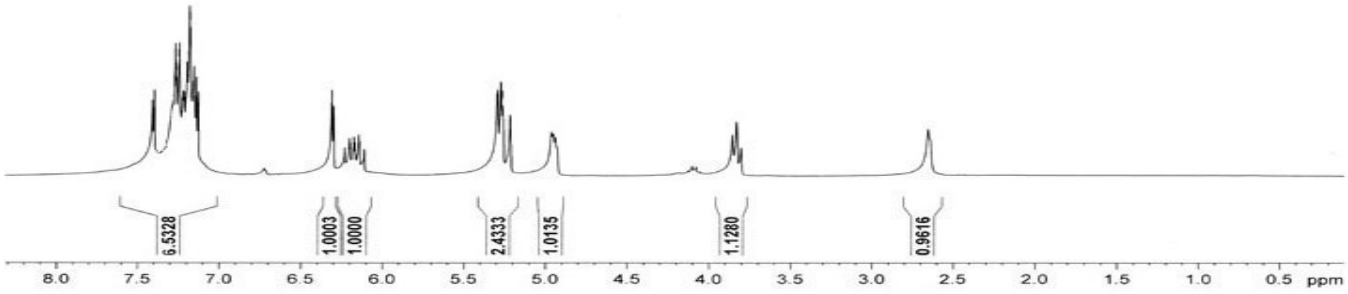
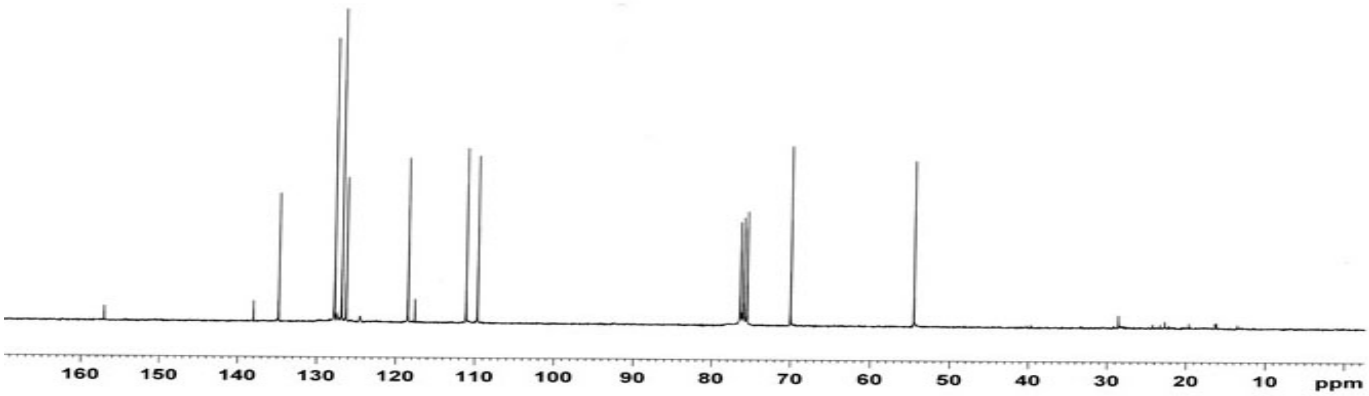
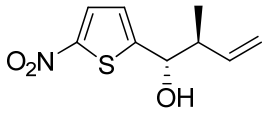
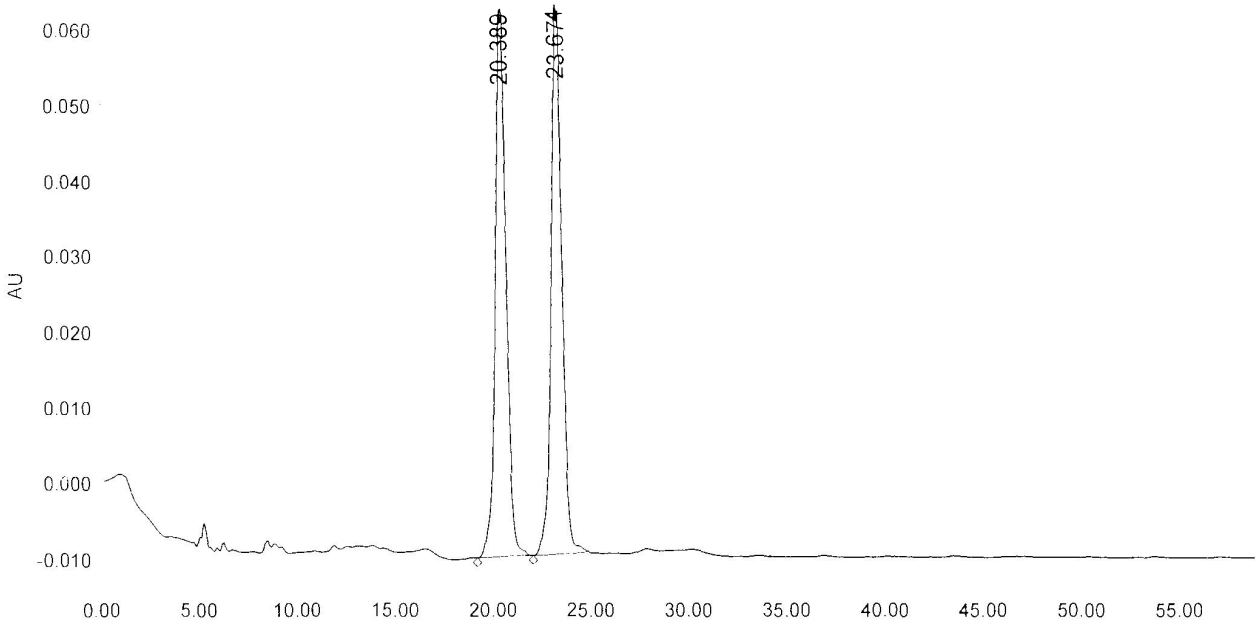


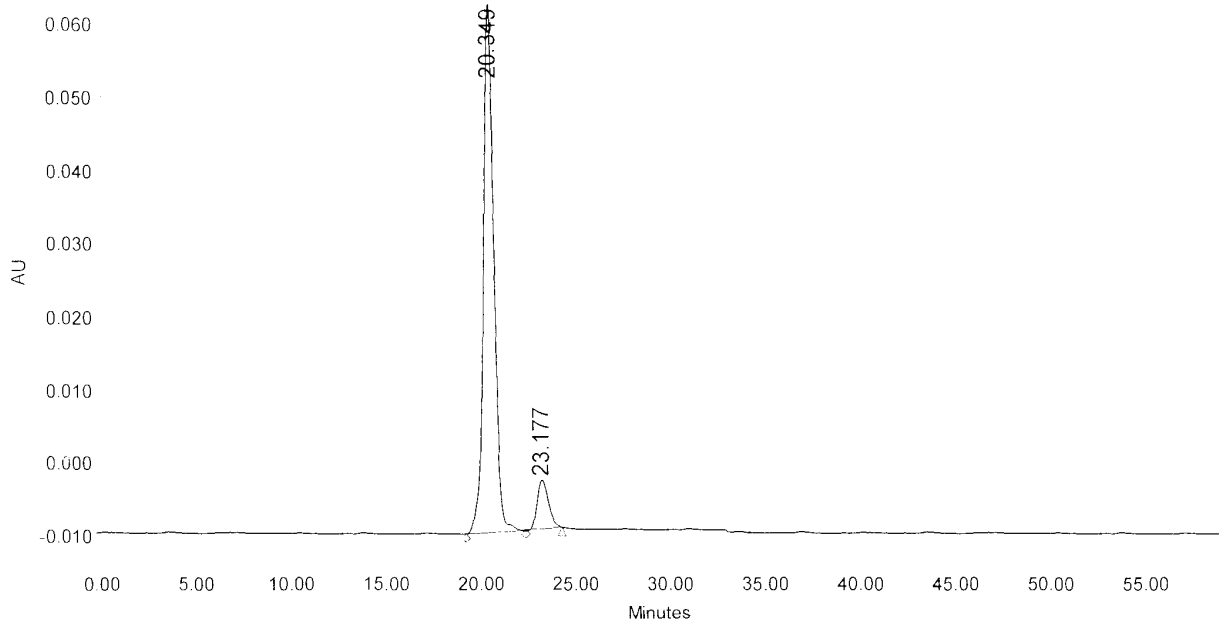




The HPLC of new compounds.







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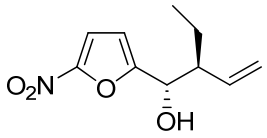

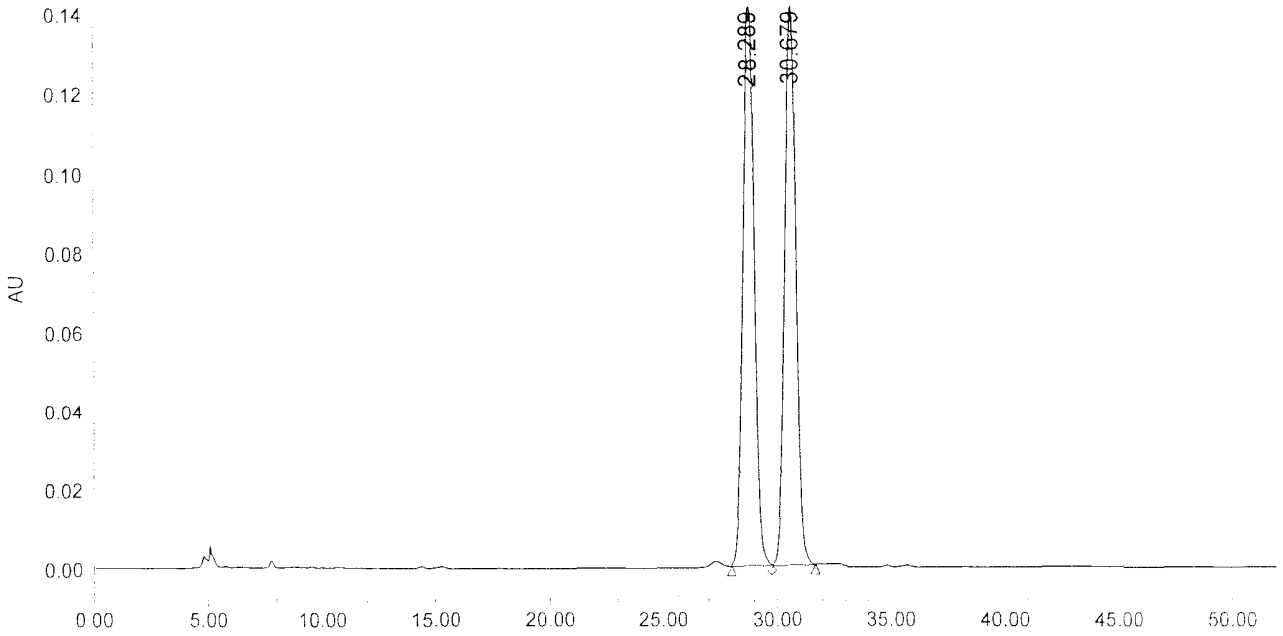

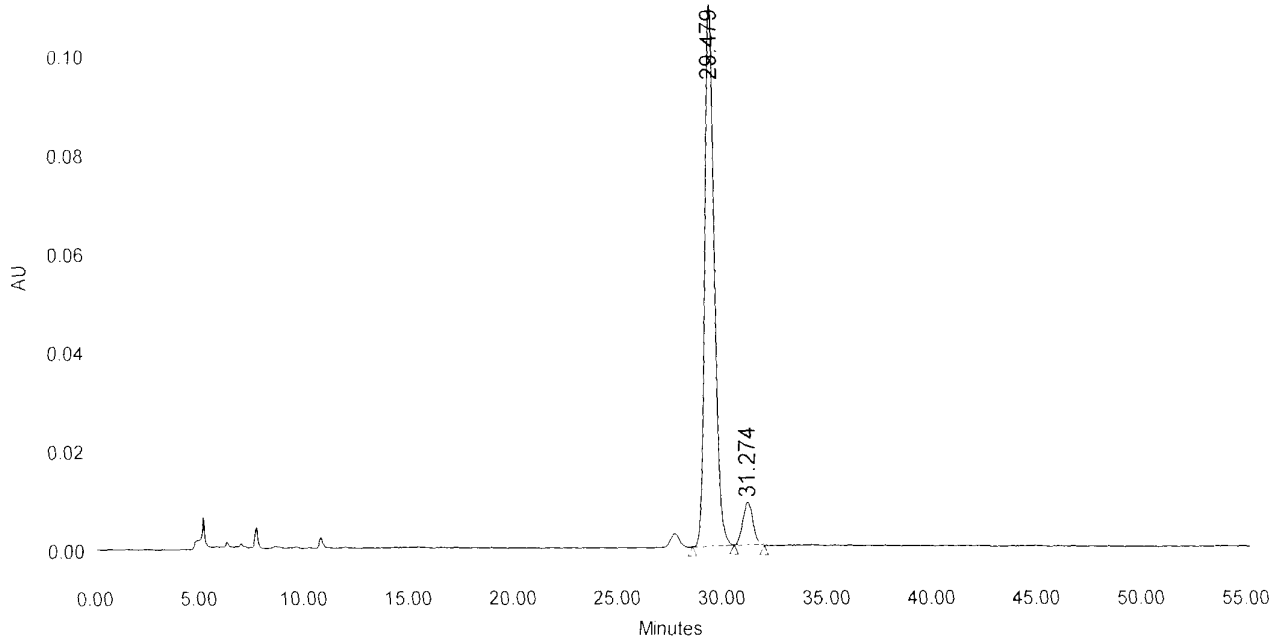

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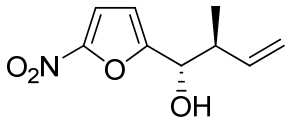

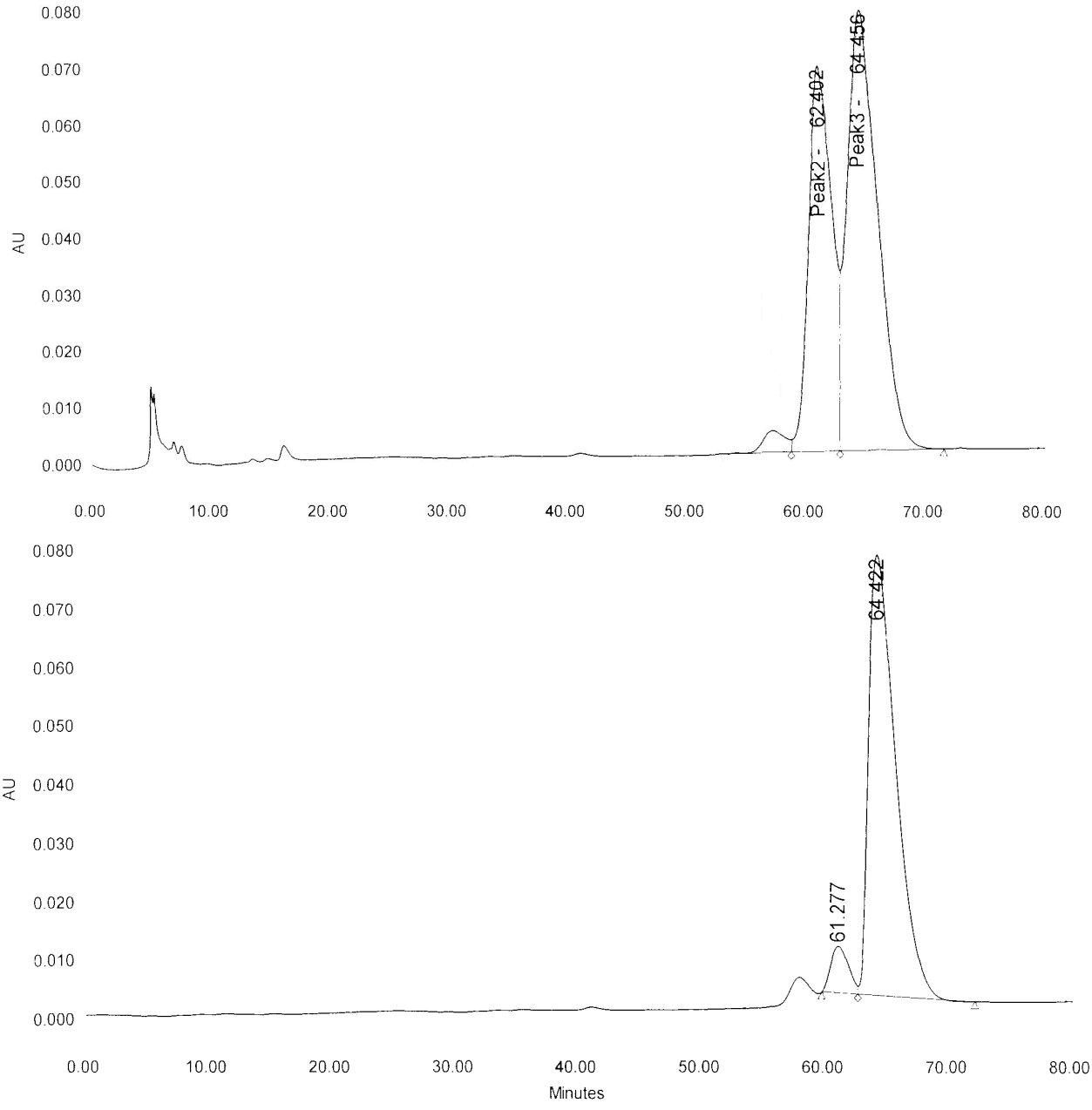

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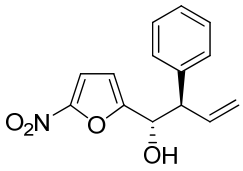

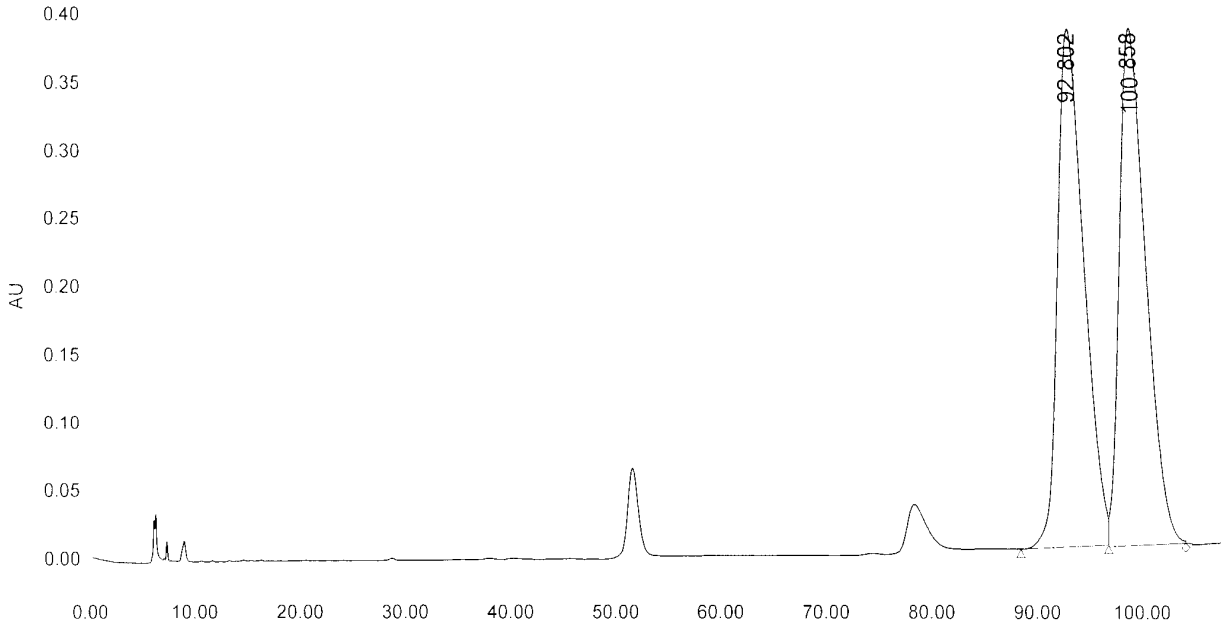

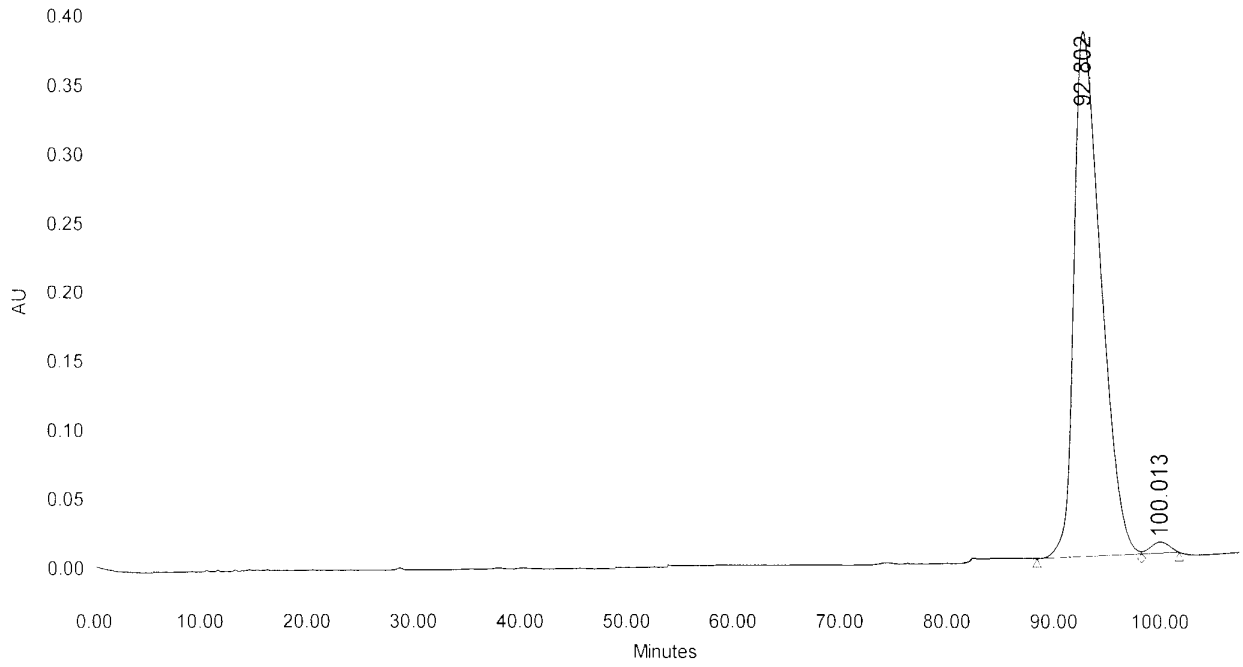

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