Electronic Supplementary Information

Hydroalkoxylation in Brønsted Acidic Ionic Liquids and Its Application to the Synthesis of (±)–Centrolobin

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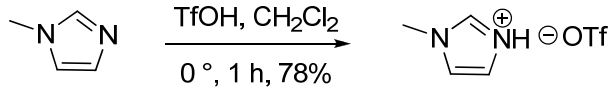
General methods: Reagents were purchased from Aldrich chemical (www.sigma-aldrich) or Alfa Aesar (www.alfa.com), and used without further purification. Reactions were performed in flame-dried glassware under positive Ar pressure with magnetic stirring. TLC was performed on 0.25 mm E. Merck silica gel 60 F_{254} plates and visualized under UV light (254 nm) or by staining with cerium ammonium molybdenate (CAM). Flash chromatography was performed on E. Merck 230−400 mesh silica gel 60. NMR spectra were recorded on Varian Unity 400 instruments at 24 °C. Chemical shifts are expressed in ppm relative to TMS (1H, 0 ppm), $CDCl_3$ (^{1}H, 7.26 ppm; ^{13}C, 77.2 ppm), DMSO-d_6 (^{1}H, 2.5 ppm; ^{13}C, 39.5 ppm), Benzene-d_6 (^{1}H, 7.16 ppm; ^{13}C, 128.1 ppm) or Acetone-d_6 (^{1}H, 2.05 ppm; ^{13}C, 206.3 ppm, 29.8 ppm); coupling constants are expressed in Hz. All ^{19}F NMR chemical shifts were

referenced to external CF_3CO_2H (0.0 ppm). Mass spectra were obtained with VG trio-2 GC-MS instrument. High resolution mass spectra were obtained with JEOL JMS-AX 505WA instrument. Alkenyl alcohol substrates **8a** and **8c–g** were purchased, and **8b, 8h–n** were synthesized according to the literature procedure.

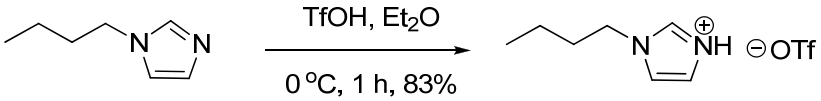
Synthesis of Brønsted acidic ionic liquids



1

1-Methylimidazolium triflate (1)

1-Methylimidazolium triflate (**1**) was synthesized according to the literature procedure.[1] In an oven-dried 5 mL roundbottom flask, triflic acid (188 mg, 1.26 mmol) was dissolved in anhydrous CH_2Cl_2 (0.4 mL) at 0 °C. 1-Methylimidazole (103 mg, 1.26 mmol) in anhydrous CH_2Cl_2 (0.4 mL) was then added dropwise and the mixture was stirred for 1 h. The solid formed was filtered off, washed with Et_2O to remove non-ionic residues, and dried *in vacuo* to afford analytically pure **1** (226 mg, 78%) as a white solid. **Mp**: 109 °C (Lit.[2] 92 °C). **^{1}H NMR** (400 MHz, DMSO-d_6): δ 14.13 (br s, 1H), 9.03 (s, 1H), 7.69 (s, 1H), 7.66 (s, 1H), 3.86 (s, 3H). **^{13}C NMR** (100 MHz, DMSO-d_6): δ 135.8, 123.1, 120.7 (q, J_{CF} = 320.4 Hz), 119.7, 35.4. **LRMS (FAB)** *m/z* (rel int): (pos) 83 ($[C_4H_7N_2]^+$, 100). **HRMS** *m/z* calcd for $C_4H_7N_2$ 83.0609, found 83.0608.

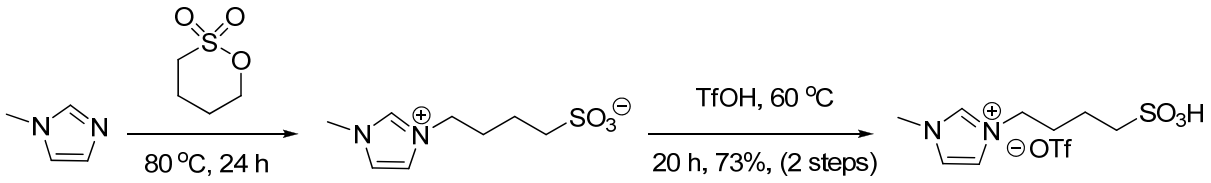


2

1-Butylimidazolium triflate (2)

1-Butylimidazolium triflate (**2**) was synthesized according to the literature procedure.[1,3] In an oven-dried 5 mL roundbottom flask, 1-butylimidazole (148 mg, 1.19 mmol) was dissolved in anhydrous CH_2Cl_2 (0.5 mL) at 0 °C. Triflic acid (170 mg, 1.13 mmol) was then added dropwise. After the mixture was stirred for 1 h, it was evaporated. The crude residue was washed repeatedly with Et_2O to remove non-ionic residues, and further dried *in vacuo* to afford analytically pure **2** (271 mg, 83%) as a brown oil. **^{1}H NMR** (400 MHz, DMSO-d_6): δ 14.20 (br s, 1H), 9.12 (s, 1H), 7.79 (s, 1H), 7.70 (s, 1H), 4.18 (t, J = 7.2 Hz, 2H), 1.79 (quintet, J = 7.2 Hz, 2H), 1.25 (sextet, J = 7.2 Hz, 2H), 0.90 (t, J = 7.2 Hz, 3H). **^{13}C NMR**

(100 MHz, DMSO-d_6): δ 135.2, 122.0, 120.7 (q, J_{CF} = 320.4 Hz), 119.9, 48.3, 31.4, 18.8, 13.3. **^{19}F NMR** (376 MHz, DMSO-d_6): δ −2.66 (s). **LRMS (FAB)** *m/z* (rel int): (pos) 125 ($[C_7H_{13}N_2]^+$, 100). **HRMS** *m/z* calcd for $C_7H_{13}N_2$ 125.1079, found 125.1082.

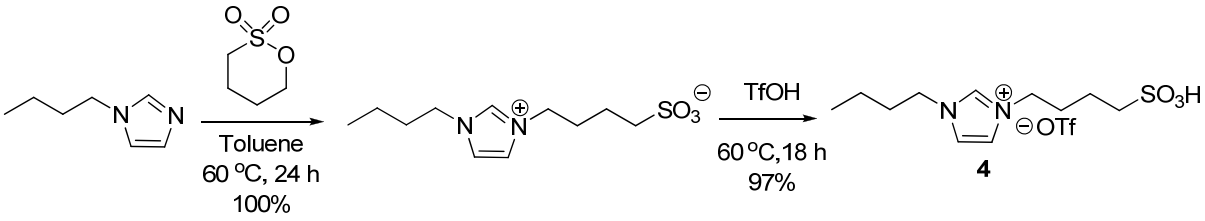


3

1-(Butyl-4-sulfonic)-3-methylimidazolium triflate (3)

1-(Butyl-4-sulfonic)-3-methylimidazolium triflate (**3**) was synthesized according to the literature procedure.[4,5] In a 5 mL vial, 1-methylimidazole (103 mg, 1.26 mmol) and 1,4-butanesultone (171 mg, 1.26 mmol) were stirred at 80 °C for 24 h. The mixture was cooled to rt. The precipitate was washed with Et_2O to remove non-ionic residues, and dried *in vacuo*. The white solid was used in the next step without further purification. **Mp**: 229 °C. **^{1}H NMR** (400 MHz, D_2O): δ 7.51 (s, 1H), 7.45 (s, 1H), 4.27 (t, *J* = 7.2 Hz, 2H), 3.91 (s, 3H), 2.98–2.95 (m, 2H), 2.08–2.01 (m, 2H), 1.80–1.72 (m, 2H). **^{1}H NMR** (400 MHz, DMSO-d_6): δ 9.12 (s, 1H), 7.77 (s, 1H), 7.70 (s, 1H), 4.18 (t, *J* = 6.8 Hz, 2H), 3.85 (s, 3H), 2.44 (m, 2H), 1.87 (quintet, *J* = 7.6 Hz, 2H), 1.53 (m, 2H). **^{13}C NMR** (100 MHz, DMSO-d_6): δ 136.5, 123.6, 122.3, 50.4, 48.5, 35.7, 28.6, 21.7. **LRMS (FAB)** *m/z* (rel int): (pos) 219 ($[M + H]^+$, 100). **HRMS** *m/z* calcd for $C_8H_{15}N_2O_3S$ 219.0803, found 219.0804.

Triflic acid (188 mg, 1.26 mmol) was added to 1-(butyl-4-sulfonate)-3-methylimidazolium zwitterions in a 5 mL vial at rt. The reaction mixture was stirred at 60 °C for 20 h. After cooled to rt, Et_2O (2 mL) was added to the reaction mixture. The ionic liquid-Et_2O mixture was stirred for a couple of minutes and stopped. Then, the ionic liquid-Et_2O mixture clearly showed two separate layers. The upper Et_2O layer was carefully removed by decantation. This procedure was repeated five times, and the residue was dried *in vacuo* to afford **3** as a colorless oil (336 mg, 73%, 2 steps). **^{1}H NMR** (400 MHz, D_2O): δ 8.74 (s, 1H), 7.50 (s, 1H), 7.44 (s, 1H), 4.25 (t, *J* = 7.2 Hz, 2H), 3.89 (s, 3H), 2.95 (m, 2H), 2.03 (m, 2H), 1.74 (m, 2H). **^{13}C NMR** (100 MHz, D_2O): δ 136.1, 123.8, 122.3, 119.8 (q, J_{CF} = 315.8 Hz), 50.2, 49.1, 35.8, 28.2, 21.1. **^{19}F NMR** (376 MHz, DMSO-d_6): δ −2.52 (s). **LRMS (FAB)** *m/z* (rel int): (pos) 219 ($[C_8H_{15}N_2O_3S]^+$, 100). **HRMS** *m/z* calcd for $C_8H_{15}N_2O_3S$ 219.0803, found 219.0807.

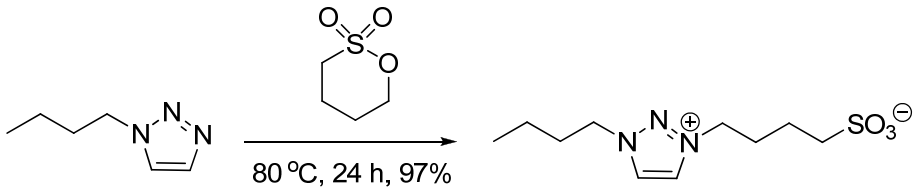


1-Butyl-3-(butyl-4-sulfonyl)imidazolium triflate (4)

1-Butyl-3-(butyl-4-sulfonyl)imidazolium triflate **(4)** was synthesized according to the literature procedure.[6] In a 5 mL vial, 1-butylimidazole (9.48 mg, 76.3 μmol) and 1,4-butanesultone (10.4 mg, 76.3 μmol) were dissolved in toluene (31.0 μL), and stirred at 60 °C for 24 h. The mixture was cooled to rt. The precipitate was washed with ether and toluene to remove non-ionic residues, and dried *in vacuo*. 1-Butyl-3-(butyl-4-sulfonate)imidazolium zwitterions was obtained as a white solid (20 mg, 100%). **Mp**: 160 °C (Lit.[7] 158 °C) **^{1}H NMR** (400 MHz, D_2O): δ 7.54–7.52 (m, 2H), 4.27 (t, *J* = 7.2 Hz, 2H), 4.22 (t, *J* = 7.2 Hz, 2H), 2.98–2.94 (m, 2H), 2.09–2.01 (m, 2H), 1.87 (quintet, *J* = 7.2 Hz, 2H), 1.78–1.72 (m, 2H), 1.33 (sextet, *J* = 7.6 Hz, 2H), 0.94 (t, *J* = 7.2 Hz, 3H). **^{1}H NMR** (400 MHz, D_2O): δ 8.84 (s, 1H), 7.55–7.53 (m, 2H), 4.28 (t, *J* = 7.2 Hz, 2H), 4.23 (t, *J* =7.2 Hz, 2H), 2.97 (m, 2H), 2.06 (m, 2H), 1.88 (quintet, *J* = 7.6 Hz, 2H), 1.77 (m, 2H), 1.34 (sextet, *J* = 7.6 Hz, 2H), 0.94 (t, *J* = 7.6 Hz, 3H). **^{1}H NMR** (400 MHz, DMSO-d_6): δ 9.21 (s, 1H), 7.80 (s, 1H), 7.79 (s, 1H), 4.17 (q, *J* = 7.2 Hz, 4H), 2.44 (m, 2H), 1.87 (m, 2H), 1.78 (quintet, *J* = 7.2 Hz, 2H), 1.56–1.49 (m, 2H), 1.26 (sextet, *J* = 7.2 Hz, 2H), 0.90 (t, *J* = 7.2 Hz, 3H). **^{13}C NMR** (100 MHz, D_2O): δ 135.4, 122.7, 122.4, 50.2, 49.5, 49.1, 31.3, 28.2, 21.1, 18.9, 12.8. **LRMS (FAB)** *m/z* (rel int): (pos) 261 ($[M + H]^+$, 100). **HRMS** *m/z* calcd for $C_{11}H_{21}N_2O_3S$ 261.1273, found 261.1273.

In a 5 mL vial, triflic acid (550 mg, 3.66 mmol) was added to 1-butyl-3-(butyl-4-sulfonate)imidazolium zwitterions (954 mg, 3.66 mmol) at rt. The reaction mixture was stirred at 60 °C for 18 h. After cooled to rt, Et_2O (2 mL) was added to the reaction mixture. The ionic liquid-Et_2O mixture was stirred for a couple of minutes and stopped. Then, the ionic liquid-Et_2O mixture clearly showed two separate layers. The upper Et_2O layer was carefully removed by decantation. This procedure was repeated five times to remove non-ionic residues. The residue was dried *in vacuo* to afford **4** as a colorless oil (1.45 g, 97%). **^{1}H NMR** (400 MHz, D_2O): δ 8.78 (s, 1H), 7.50–7.48 (m, 2H), 4.23 (t, *J* = 7.2 Hz, 2H), 4.18 (t, *J*

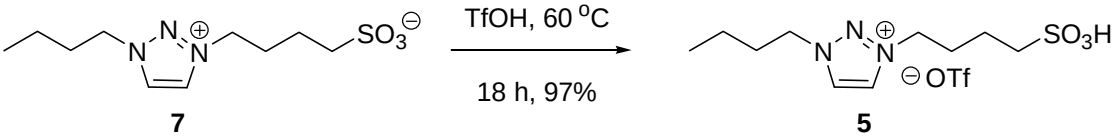
= 7.2 Hz, 2H), 2.92 (m, 2H), 2.00 (m, 2H), 1.83 (quintet, *J* = 7.2 Hz, 2H), 1.72 (m, 2H), 1.29 (sextet, *J* = 7.2 Hz, 2H), 0.89 (t, *J* = 7.2 Hz, 3H). **^{1}H NMR** (400 MHz, DMSO–d_6): δ 9.21 (s, 1H), 7.80 (s, 1H), 7.79 (s, 1H), 4.17 (q, *J* = 7.2 Hz, 4H), 2.48 (m, 2H), 1.89 (m, 2H), 1.77 (quintet, *J* = 7.2 Hz, 2H), 1.54 (m, 2H), 1.26 (sextet, *J* = 7.2 Hz, 2H), 0.90 (t, *J* = 7.2 Hz, 3H). **^{13}C NMR** (100 MHz, D_2O): δ 135.3, 122.6, 122.4, 119.8 (q, J_{CF} = 315.6 Hz), 50.2, 49.5, 49.1, 31.3, 28.2, 21.1, 18.9, 12.7. **LRMS (FAB)** *m/z* (rel int): (pos) 261 ($[C_{11}H_{21}N_2O_3S]^+$, 100). **HRMS** *m/z* calcd for $C_{11}H_{21}N_2O_3S$ 261.1273, found 261.1275. Anal. Calcd for $C_{12}H_{21}F_3N_2O_6S_2$: C, 35.12; H, 5.16; N, 6.83; S, 15.63. Found: C, 35.19; H, 5.12; N, 6.85; S, 15.62.



6 **7**

1-Butyl-3-(butyl-4-sulfonate)triazolium zwitterion (7)

In a 5 mL vial, 1-butyltriazole (151 mg, 1.21 mmol) and 1,4-butanesultone (181 mg, 1.21 mmol) were stirred at 80 °C for 24 h. The mixture was cooled to rt. The precipitate was washed with Et_2O to remove non-ionic residues, and dried *in vacuo*. 1-Butyl-3-(butyl-4-sulfonate)triazolium zwitterion (**7**) was obtained as a white solid (305 mg, 97%). **^{1}H NMR** (400 MHz, D_2O): δ 8.61 (s, 1H), 8.59 (s, 1H), 4.73 (t, *J* = 7.2 Hz, 2H), 4.68 (t, *J* = 7.2 Hz, 2H), 2.99 (m, 2H), 2.21 (m, 2H), 2.03 (quintet, *J* = 7.6 Hz, 2H), 1.82 (m, 2H), 1.38 (sextet, *J* = 7.6 Hz, 2H), 0.97 (t, *J* = 7.6 Hz, 3H). **^{1}H NMR** (400 MHz, DMSO-d_6): δ 8.94 (s, 1H), 8.93 (s, 1H), 4.64 (q, *J* = 7.2 Hz, 4H), 2.45 (m, 2H), 2.02 (m, 2H), 1.90 (quintet, *J* = 7.2 Hz, 2H), 1.57 (m, 2H), 1.30 (sextet, *J* = 7.2 Hz, 2H), 0.91 (t, *J* = 7.2 Hz, 3H). **^{13}C NMR** (100 MHz, D_2O): δ 133.1, 133.0, 56.3, 55.9, 52.7, 33.3, 30.1, 23.7, 21.5, 15.3. **LRMS (FAB)** *m/z* (rel int): (pos) 262 ($[M + H]^+$, 100). **HRMS** *m/z* calcd for $C_{10}H_{20}N_3O_3S$ 262.1225, found 262.1229. Anal. Calcd for $C_{10}H_{19}N_3O_3S$: C, 45.96; H, 7.33; N, 16.08; S, 12.27. Found: C, 45.98; H, 7.23; N, 16.01; S, 12.23.

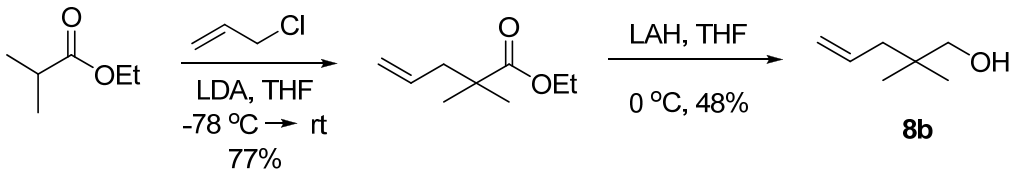


7 **5**

1-Butyl-3-(butyl-4-sulfonyl)triazolium triflate (5)

In a 5 mL vial, triflic acid (121 mg, 804 μmol) was added to 1-butyl-3-(butyl-4-sulfonate)triazolium zwitterion (**7**) (210 mg, 804 μmol) at rt. The reaction mixture was stirred at 60 °C for 18 h. After cooled to rt, Et_2O (2 mL) was added to the reaction mixture. The ionic liquid-Et_2O mixture was stirred for a couple of minutes and stopped. The upper Et_2O layer was carefully removed by decantation. This procedure was repeated five times to remove non-ionic residues. The residue was dried *in vacuo* to afford **5** as a colorless oil (322 mg, 97%). **^{1}H NMR** (400 MHz, D_2O): δ 8.51 (s, 1H), 8.50 (s, 1H), 4.65 (t, *J* = 7.2 Hz, 2H), 4.60 (t, *J* = 7.2 Hz, 2H), 2.91 (m, 2H), 2.13 (m, 2H), 1.95 (quintet, *J* = 7.6 Hz, 2H), 1.74 (m, 2H), 1.31 (sextet, *J* = 7.6 Hz, 2H), 0.89 (t, *J* = 7.6 Hz, 3H). **^{1}H NMR** (400 MHz, DMSO-d_6): δ 8.94 (s, 1H), 8.93 (s, 1H), 4.64 (q, *J* = 7.2 Hz, 4H), 2.48 (m, 2H), 2.02 (m, 2H), 1.90 (quintet, *J* = 7.2 Hz, 2H), 1.58 (m, 2H), 1.30 (sextet, *J* = 7.2 Hz, 2H), 0.91 (t, *J* = 7.2 Hz, 3H). **^{13}C NMR** (100 MHz, D_2O): δ 130.4 (2C), 119.8 (q, J_{CF} = 315.6 Hz), 53.7, 53.2, 50.1, 30.6, 27.5, 21.1, 18.9, 12.6. **LRMS (FAB)** *m/z* (rel int): (pos) 262 ($[C_{10}H_{20}N_3O_3S]^+$, 100). **HRMS** *m/z* calcd for $C_{10}H_{20}N_3O_3S$ 262.1225, found 262.1223. Anal. Calcd for $C_{11}H_{20}F_3N_3O_6S_2$: C, 32.11; H, 4.90; N, 10.21; S, 15.59. Found: C, 32.05; H, 4.96; N, 10.24; S, 15.59.

Syntheses of Alkenyl Alcohol Substrates

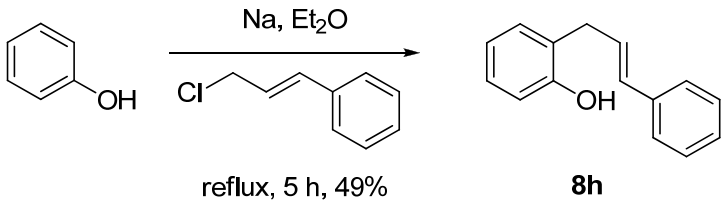


2,2-Dimethylpent-4-en-1-ol (8b)

8b was synthesized according to the literature procedure.[8] A 2.5 M solution of *n*-BuLi in hexane (9.6 mL, 24 mmol) was added dropwise to a solution of diisopropylammine (2.43 g, 24 mmol) in anhydrous THF (40 mL) at –50 °C under Ar atmosphere and stirred 30 min at –50 °C. After stirring an additional 30 min at –5 °C, the mixture was cooled to –50 °C. Ethyl isobutyrate (2.32 g, 20 mmol) was then added dropwise and stirred for 30 min at –50 °C. Finally, allyl chloride (1.84 g, 24 mmol) was added dropwise at –50 °C and the mixture was slowly warmed to rt. After stirring for 18 h at rt, the reaction mixture was cooled to 0 °C, and quenched by the addition of satd aq NH_4Cl (250 mL), then extracted with Et_2O (3 × 250 mL). The combined organic extracts were dried over $MgSO_4$, filtered, and concentrated by rotary

evaporation. Purification by silica flash chromatography (15:1→10:1 hexane/EtOAc) yielded ethyl 2,2-dimethylpent-4-enoate (2.40 g, 77%) as a colorless liquid. **TLC**: R_f 0.32 (15:1 hexane/EtOAc). **^{1}H NMR** (400MHz, $CDCl_3$): δ 5.73 (m, 1H), 5.06 (m, 1H), 5.03 (m, 1H), 4.12 (q, J = 7.2 Hz, 2H), 2.28 (dt, J = 7.2, 1.2 Hz, 2H), 1.25 (t, J = 7.2 Hz, 3H), 1.17 (s, 6H). **^{13}C NMR** (100 MHz, $CDCl_3$): δ 177.7, 134.5, 118.0, 60.5, 45.0, 42.4, 25.0, 14.5. **LRMS (ESI)** *m/z* (rel int) (pos) 83 ($[M - COOC_2H_5]^+$, 100). **HRMS** *m/z* calcd for $C_9H_{16}O_2$ 156.1150; found 156.1148. **IR** (KBr film): 3080, 2980, 2935, 1731, 1149 cm^{-1}.

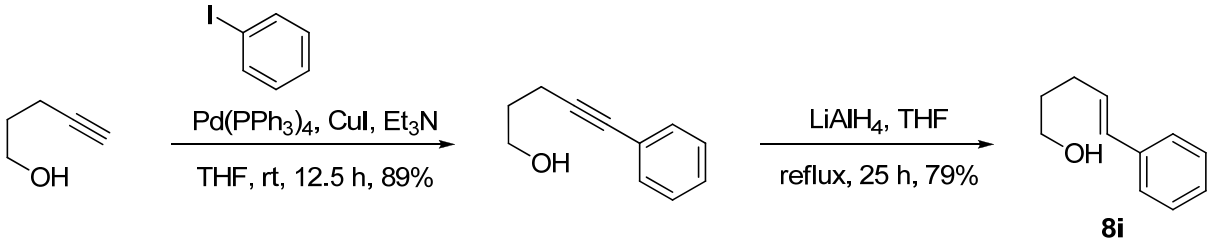
Ethyl 2,2-dimethylpent-4-enoate (1.79 g, 11.5 mmol) in anhydrous THF (21 mL) was added dropwise with stirring to a suspension of $LiAlH_4$ (873 mg, 23 mmol) in anhydrous THF (4 mL) at 0 °C. After stirring for 30 min at 0 °C, the reaction mixture was diluted with Et_2O (10 mL), quenched by the sequential addition of water (0.8 mL), a 15% aq NaOH (0.8 mL), and additional water (2.4 mL). The resulting white precipitate was filtered off and washed with Et_2O. The filtrate was dried over $MgSO_4$, filtered, and concentrated in vacuo. Purification by silica flash chromatography (4:1 hexane/EtOAc) yielded **8b** (0.63 g, 48%) as a yellow liquid. **TLC**: R_f 0.33 (5:1 hexane/EtOAc). **^{1}H NMR** (400MHz, $CDCl_3$): δ 5.85 (m, 1H), 5.07 (m, 1H), 5.03 (m, 1H), 3.33 (d, J = 4.0 Hz, 2H), 2.03 (dt, J = 8.0, 1.2 Hz, 2H), 1.37 (t, J = 4.0 Hz, 1H), 0.89 (s, 6H). **^{13}C NMR** (100MHz, $CDCl_3$): δ 135.5, 117.3, 72.0, 43.6, 35.7, 24.0. **LRMS (CI)** *m/z* (rel int): (pos) 113 ($[M - H]^+$, 30). **HRMS** *m/z* calcd for $C_7H_{14}O$ 114.1045, found 114.1053. **IR** (KBr film): 3353, 3078, 2960, 1641, 1043, 997, 913 cm^{-1}.



2-Cinnamylphenol (8h)

8h was synthesized according to the literature procedure.[9] In an oven-dried 50 mL roundbottom flask, phenol (1.07 g, 11.4 mmol) was dissolved in anhydrous Et_2O (11 mL). Na (0.455 g) was added and the reaction was stirred at rt for 30 min. Cinnamyl chloride (1.83 g, 11.4 mmol) was then added dropwise and the mixture was heated at reflux with stirring for 5 h. The mixture was cooled to rt and diluted with 0.1 N aq HCl (70 mL), then extracted with Et_2O (3 × 100 mL). The combined organic extracts were dried over $MgSO_4$, filtered, and concentrated by rotary evaporation. Purification by silica flash chromatography (5:1

hexane/EtOAc) yielded **8h** (1.17 g, 49%) as a pale yellow solid. **TLC**: R_f 0.29 (3:1 hexane/EtOAc (× 2)). **Mp**: 43 °C. **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.37−7.27 (m, 4H), 7.23−7.13 (m, 3H), 6.91 (td, *J* = 7.2, 1.2 Hz, 1H), 6.82 (dd, *J* = 8.0, 1.2 Hz, 1H), 6.51 (d, *J* = 15.6 Hz, 1H), 6.39 (dt, *J* = 15.6, 6.4 Hz, 1H), 4.88 (s, 1H), 3.57 (d, *J* = 6.4 Hz, 2H). **^{13}C NMR** (100 MHz, $CDCl_3$): δ 154.2, 137.3, 131.7, 130.7, 128.7, 128.2, 128.1, 127.5, 126.4, 125.9, 121.2, 115.9, 34.2. **LRMS (EI)** *m/z* (rel int): (pos) 211 ($[M + H]^+$, 18), 210 ($[M]^+$, 100), 209 ($[M - H]^+$, 30), 193 ($[M - OH]^+$, 15). **HRMS** *m/z* calcd for $C_{15}H_{14}O$ 210.1045, found 210.1039. **IR** (KBr film): 3425, 3029, 1594, 1455, 1098, 968, 755, 737, 693 cm^{-1}



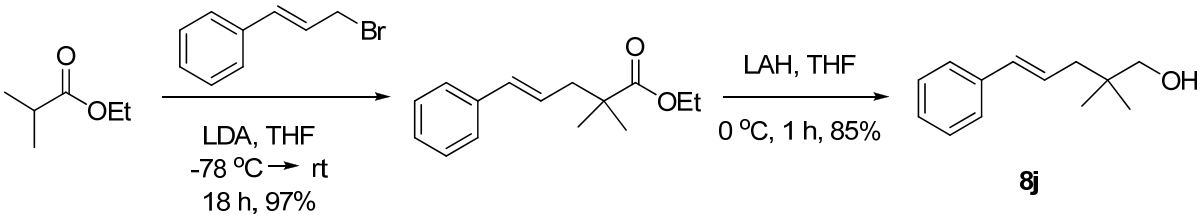
8i

(*E*)-5-Phenylpent-4-en-1-ol (8i)

8i was synthesized according to the literature procedure.[10] In an oven-dried 25 mL rooundbottom flask, $Pd(PPh_3)_4$ (129 mg, 112 μmol) was dissolved in anhydrous THF (5.6 mL). 4-Pentyn-1-ol (940 mg, 11.2 mmol), iodobenzene (4.56 g, 22.3 mmol), triethylamine (22.6 g, 223 mmol) and CuI (42.5 mg, 223 μmol) were added sequentially and stirred at rt for 12.5 h. The mixture was filtered over a celite and concentrated by rotary evaporation. Purification by silica flash chromatography (5:1 → 3:1 hexane/EtOAc) yielded 5-phenylpent-4-yn-1-ol (1.59 g, 89%) as a colorless liquid. **TLC**: R_f 0.19 (3:1 hexane/EtOAc). **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.40−7.38 (m, 2H), 7.29−7.27 (m, 3H), 3.83 (q, *J* = 5.6 Hz, 2H), 2.55 (t, *J* = 6.8 Hz, 2H), 1.87 (quintet, *J* = 6.8 Hz, 2H), 1.46 (t, *J* = 5.6 Hz, 1H). **^{13}C NMR** (100 MHz, $CDCl_3$): δ 131.7, 128.4, 127.9, 123.9, 89.5, 81.3, 62.0, 31.6, 16.2. **LRMS (ESI)** *m/z* (rel int): (pos) 161 ($[M + H]^+$, 27), 142 ($[M - OH_2]^+$, 14). **HRMS** *m/z* calcd for $C_{11}H_{12}O$ 160.0888, found 160.0887. **IR** (KBr film): 3301, 2947, 1599, 1442, 1061, 756, 692 cm^{-1}.

5-Phenylpent-4-yn-1-ol (690 mg, 4.31 mmol) was added dropwise with stirring to a suspension of $LiAlH_4$ (860 mg, 21.5 mmol) in anhydrous THF (8.6 mL) at 0 °C. The reaction mixture was stirred at 0 °C for 20 min, and then brought to a reflux. After stirring for 25 h, the reaction mixture was cooled to 0 °C, and quenched by the sequential addition of water (0.86 mL), a 15% aqueous solution of NaOH (0.86 mL), and additional water (2.6 mL). The

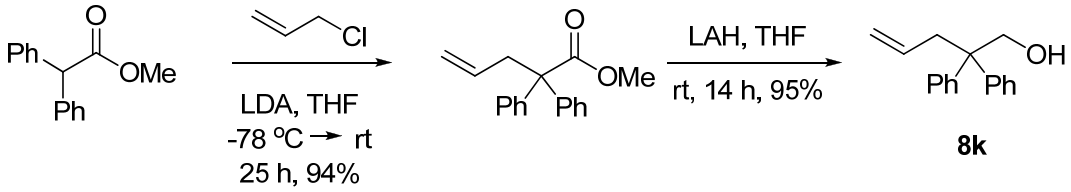
resulting white precipitate was filtered off and washed with ether. The filtrate was dried over $MgSO_4$, filtered, and concentrated *in vacuo*. Purification by silica flash chromatography (5:1 hexane/EtOAc) yielded **8i** (541 mg, 79%) as a colorless liquid. **TLC**: R_f 0.21 (3:1 hexane/EtOAc). **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.36−7.27 (m, 4H), 7.21 (m, 1H), 6.43 (d, *J* = 16.0 Hz, 1H), 6.23 (dt, *J* = 16.0, 6.8 Hz, 1H), 3.72 (q, *J* = 5.6 Hz, 2H), 2.32 (q, *J* = 6.4 Hz, 2H), 1.76 (quintet, *J* = 6.4 Hz, 2H), 1.27 (t, *J* = 5.6 Hz, 1H). **^{13}C NMR** (100 MHz, $CDCl_3$): δ 137.8, 130.6, 130.2, 128.7, 127.1, 126.1, 62.5, 32.4, 29.5. **LRMS (EI)** *m/z* (rel int): (pos) 163 ($[M + H]^+$, 10), 162 ($[M]^+$, 83), 144 ($[M - H_2O]^+$, 45), 129 ($[M - CH_2OH]^+$, 100). **HRMS** *m/z* calcd for $C_{11}H_{14}O$ 162.1045, found 162.1045. **IR** (KBr film): 3341, 3026, 2936, 1598, 1446, 1059, 966, 742, 693 cm^{-1}.



(*E*)-2,2-Dimethyl-5-phenylpent-4-en-1-ol (8j)

8j was synthesized according to the literature procedure.[8] A 2.5 M solution of *n*-BuLi in hexane (4.8 mL, 12 mmol) was added dropwise to a solution of diisopropylammine (1.21 g, 12 mmol) in anhydrous THF (20 mL) at −50 °C under Ar atmosphere and stirred 30 min at −5 °C. Ethyl isobutyrate (1.16 g, 10 mmol) was then added dropwise at −5 °C and stirred 30 min. Finally, cinnamyl bromide (2.36 g, 12 mmol) was added dropwise at −50 °C and the mixture was slowly warmed to rt. After stirring for 18 h, the reaction mixture was cooled to 0 °C, and quenched by the addition of satd aq NH_4Cl (100 mL) and water (20 mL), then extracted with Et_2O (3 × 100 mL). The combined organic extracts were dried over $MgSO_4$, filtered, and concentrated by rotary evaporation. Purification by silica flash chromatography (20:1 hexane/EtOAc) yielded (*E*)-ethyl 2,2-dimethyl-5-phenylpent-4-enoate (2.25 g, 97%) as a pale yellow oil. **TLC**: R_f 0.24 (20:1 hexane/EtOAc). **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.34−7.27 (m, 4H), 7.20 (m, 1H), 6.40 (d, *J* = 16.0 Hz, 1H), 6.15 (dt, *J* = 16.0, 7.6 Hz, 1H), 4.14 (q, *J* = 7.2 Hz, 2H), 2.43 (dd, *J* = 7.6, 1.2 Hz, 2H), 1.25 (t, *J* = 7.2 Hz, 3H), 1.22 (s, 6H). **^{13}C NMR** (100 MHz, $CDCl_3$): δ 177.7, 137.7, 133.2, 128.7, 127.3, 126.4, 126.3, 60.6, 44.1, 25.2, 14.5. **LRMS (ESI)** *m/z* (rel int): (pos) 233 ($[M + H]^+$, 8), 217 ($[M - COOCH_3]^+$, 100), 117 ($[PhCHCHCH_2]^+$, 41). **HRMS** *m/z* calcd for $C_{15}H_{20}O_2$ 232.1463, found 232.1469. **IR** (KBr film): 3028, 2977, 1723, 1599, 1471, 1189, 1134, 968, 744, 692 cm^{-1}.

(*E*)-Ethyl 2,2-dimethyl-5-phenylpent-4-enoate (1.16 g, 5 mmol) was added dropwise with stirring to a suspension of $LiAlH_4$ (0.38 g, 10 mmol) in anhydrous THF (3 mL) at 0 °C. After stirring for 1 h at 0 °C, the reaction mixture was quenched by the sequential addition of water (0.38 mL), a 15% aq NaOH (0.38 mL), and additional water (1.14 mL). The resulting white precipitate was filtered off and washed with ether. The filtrate was dried over $MgSO_4$, filtered, and concentrated *in vacuo*. Purification by silica flash chromatography (1:10→1:3 hexane/EtOAc) yielded **8j** (0.81 g, 85%) as a pale yellow oil **TLC**: R_f 0.24 (10:1 hexane/EtOAc). **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.37−7.28 (m, 4H), 7.20 (m, 1H), 6.41 (d, *J* = 16.0 Hz, 1H), 6.26 (dt, *J* = 16.0, 7.6 Hz, 1H), 3.38 (d, *J* = 5.6 Hz, 2H), 2.18 (dd, *J* = 7.6, 1.2 Hz, 2H), 1.37 (br t, *J* = 5.6 Hz, 1H), 0.95 (s, 6H). **^{13}C NMR** (100 MHz, $CDCl_3$): δ 137.7, 132.4, 128.5, 127.1, 127.0, 126.0, 71.8, 42.4, 36.2, 24.0. **LRMS (EI)** *m/z* (rel int): (pos) 190 ($[M]^+$, 40), 117 ($[PhCHCHCH_2]^+$, 100). **HRMS** *m/z* calcd for $C_{13}H_{18}O$ 190.1358, found 190.1364. **IR** (KBr film): 3369, 3027, 2958, 1599, 1471, 1040, 968, 758, 693 cm^{-1}.

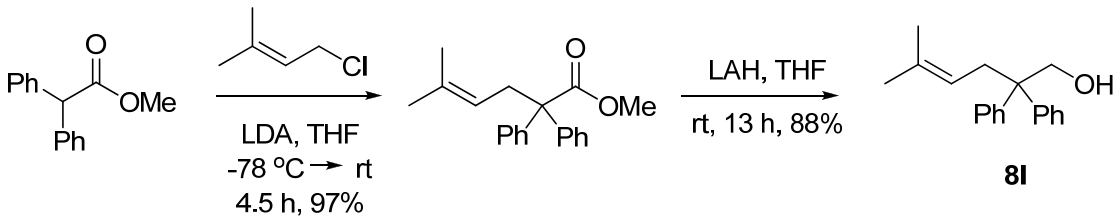


2,2-Diphenylpent-4-en-1-ol (8k)

8k was synthesized according to the literature procedure.[11] A 2.2 M solution of *n*-BuLi in hexane (1.51 mL, 3.32 mmol) was added dropwise to a solution of diisopropylammine (403 mg, 3.98 mmol) in anhydrous THF (2 mL) at −78 °C under Ar atmosphere and stirred 30 min at −78 °C. After stirring an additional 30 min at −5 °C, methyl 2,2-diphenylacetate (0.5 g, 2.21 mmol) in anhydrous THF (1.2 mL) was then added dropwise at −78 °C, stirred 10 min. Finally, allyl chloride (338 mg, 4.42 mmol) was added dropwise at −78 °C and the mixture was slowly warmed to rt. After stirring for 25 h at rt, the reaction mixture was cooled to 0 °C, and quenched by the addition of satd aq NH_4Cl (10 mL), then extracted with Et_2O (3 × 30 mL). The combined organic extracts were washed with brine, dried over $MgSO_4$, filtered, and concentrated by rotary evaporation. Purification by silica flash chromatography (10:1 hexane/EtOAc) yielded methyl 2,2-diphenylpent-4-enoate (555 mg, 94%) as a colorless liquid. **TLC**: R_f 0.37 (10:1 hexane/EtOAc). **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.32−7.22 (m, 10H), 5.60 (ddt, *J* = 18.0, 10.0, 6.8 Hz, 1H), 4.96−4.91 (m, 2H), 3.70 (s, 3H), 3.17 (dm, *J* =

6.8 Hz, 2H). **^{13}C NMR** (100 MHz, $CDCl_3$): δ 174.7, 142.7, 134.4, 129.1, 128.0, 127.0, 118.4, 60.5, 52.5, 43.0. **LRMS (ESI)** *m/z* (rel int): (pos) 267 ($[M + H]^+$, 42), 207 ($[M – COOCH_3]^+$, 65). **HRMS** *m/z* calcd for $C_{18}H_{18}O_2$ 266.1307, found 266.1318. **IR** (KBr film): 3062, 2951, 1732, 1641, 1599, 1446, 1225, 750, 700 cm^{-1}.

Methyl 2,2-diphenylpent-4-enoate (105 mg, 393 μmol) was added dropwise with stirring to a suspension of $LiAlH_4$ (20.9 mg, 550 μmol) in anhydrous THF (0.55 mL) at 0 °C. After stirring for 15 h at rt, the reaction mixture was cooled to 0 °C, diluted with Et_2O (5 mL), quenched by the sequential addition of water (21 μL), a 15% aq NaOH (21 μL), and additional water (63 μL). The resulting white precipitate was filtered off and washed with Et_2O. The filtrate was dried over $MgSO_4$, filtered, and concentrated *in vacuo*. Purification by silica flash chromatography (5:1 hexane/EtOAc) yielded **8k** (89.0 mg, 95%) as a white solid. **TLC**: R_f 0.31 (5:1 hexane/EtOAc). **Mp**: 44 °C (Lit.[12] 46−47 °C). **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.33−7.28 (m, 4H), 7.24−7.18 (m, 6H), 5.44 (ddt, *J* = 17.2, 10.0, 7.2 Hz, 1H), 5.09 (dm, *J* = 17.2 Hz, 1H), 5.00 (dm, *J* = 10.0 Hz, 1H), 4.16 (d, *J* = 6.8 Hz, 2H), 2.98 (dm, *J* = 7.2 Hz, 2H), 1.15 (t, *J* = 6.8 Hz, 1H). **^{13}C NMR** (100 MHz, $CDCl_3$): δ 145.5, 134.7, 128.5, 128.4, 126.6, 118.3, 68.1, 51.8, 41.2. **LRMS (CI)** *m/z* (rel int): (pos) 237 ($[M − H]^+$, 5), 221 ($[M − OH]^+$, 45), 197 ($[M – C_3H_5]^+$, 100). **IR** (KBr film): 3420, 3061, 2935, 1600, 1496, 1445, 1056, 758, 700 cm^{-1}.

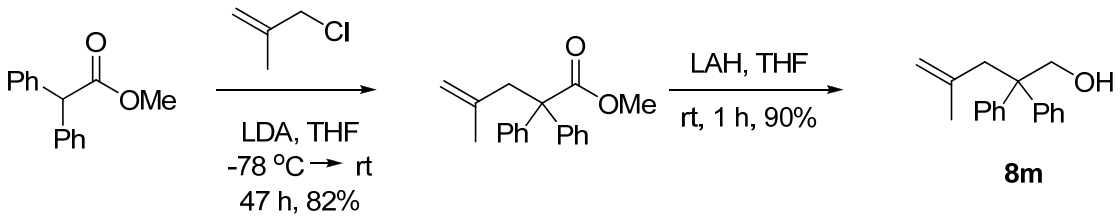


5-Methyl-2,2-diphenylhex-4-en-1-ol (8l)

8l was synthesized according to the literature procedure.[11] A 2.5 M solution of *n*-BuLi in hexane (7.96 mL, 19.9 mmol) was added dropwise to a solution of diisopropylammine (2.42 g, 23.9 mmol) in anhydrous THF (12 mL) at −78 °C under Ar atmosphere and stirred 30 min at −78 °C. After stirring an additional 30 min at −5 °C, methyl 2,2-diphenylacetate (3.0 g, 13.3 mmol) in anhydrous THF (7 mL) was then added dropwise at −78 °C, stirred 30 min. Finally, 1-chloro-3-methyl-2-butene (2.77 g, 26.5 mmol) was added dropwise at −78 °C and

the mixture was slowly warmed to rt. After stirring for 4.5 h at rt, the reaction mixture was cooled to 0 °C, and quenched by the addition of satd aq NH_4Cl (50 mL), then extracted with Et_2O (3 × 50 mL). The combined organic extracts were washed with brine, dried over $MgSO_4$, filtered, and concentrated by rotary evaporation. Purification by silica flash chromatography (20:1 hexane/EtOAc) yielded methyl 5-methyl-2,2-diphenylhex-4-enoate (3.79 g, 97%) as a yellow oil. **TLC**: R_f 0.64 (10:1 hexane/EtOAc). **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.30−7.21 (m, 10H), 5.01 (m, 1H), 3.69 (s, 3H), 3.08 (d, *J* = 6.8 Hz, 2H), 1.56 (s, 3H), 1.26 (s, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$): δ 175.1, 142.9, 135.1, 129.2, 127.9, 126.9, 119.7, 60.9, 52.5, 37.1, 26.1, 17.7. **LRMS (EI)** *m/z* (rel int): (pos) 294 ($[M]^+$, 3), 293 ($[M - H]^+$, 5), 235 ($[M - COOCH_3]^+$, 9). **HRMS** *m/z* calcd for $C_{20}H_{22}O_2$ 294.1620; found 294.1615. **IR** (KBr film): 3060, 3028, 2920, 1732, 1600, 1446, 1227, 761, 700 cm^{-1}.

Methyl 5-methyl-2,2-diphenylhex-4-enoate (3.79 g, 12.9 mmol) was added dropwise with stirring to a suspension of $LiAlH_4$ (684 mg, 18.0 mmol) in anhydrous THF (18 mL) at 0 °C. After stirring for 13 h at rt, the reaction mixture was cooled to 0 °C, diluted with Et_2O (20 mL), quenched by the sequential addition of water (0.7 mL), a 15% aq NaOH (0.7 mL), and additional water (2.1 mL). The resulting white precipitate was filtered off and washed with Et_2O. The filtrate was dried over $MgSO_4$, filtered, and concentrated *in vacuo*. Purification by silica flash chromatography (15:1 hexane/EtOAc) yielded **8l** (3.03 g, 88%) as a viscous colorless oil. **TLC**: R_f 0.27 (10:1 hexane/EtOAc). **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.31−7.27 (m, 4H), 7.23−7.18 (m, 6H), 4.85 (m, 1H), 4.12 (d, *J* = 6.8 Hz, 2H), 2.88 (d, *J* = 7.2 Hz, 2H), 1.60 (d, *J* = 1.2 Hz, 3H), 1.53 (d, *J* = 1.2 Hz, 3H), 1.16 (t, *J* = 6.8 Hz, 1H). **^{13}C NMR** (100 MHz, $CDCl_3$): δ 145.8, 134.8, 128.5, 128.3, 126.5, 120.0, 68.5, 52.5, 35.2, 26.2, 18.1. **LRMS (EI)** *m/z* (rel int): (pos) 266 ($[M]^+$, 2), 248 ($[M - H_2O]^+$, 1), 197 ($[Ph_2CCH_2OH]^+$, 100). **HRMS** *m/z* calcd for $C_{19}H_{22}O$ 266.1671; found 266.1667. **IR** (KBr film): 3429, 3087, 3027, 2969, 1600, 1444, 1379, 756, 700 cm^{-1}.

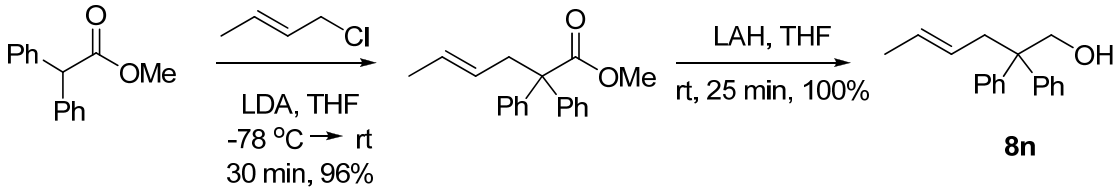


4-Methyl-2,2-diphenylpent-4-en-1-ol (8m)

8m was synthesized according to the literature procedure.[11] A 2.5 M solution of *n*-BuLi in

hexane (6.89 mL, 17.2 mmol) was added dropwise to a solution of diisopropylammine (1.74 g, 17.2 mmol) in anhydrous THF (12 mL) at −78 °C under Ar atmosphere and stirred 30 min at −78 °C. After stirring an additional 30 min at −5 °C, methyl 2,2-diphenylacetate (3.0 g, 13.3 mmol) in anhydrous THF (7 mL) was then added dropwise at −78 °C, stirred 10 min. Finally, allyl chloride (1.80 g, 19.9 mmol) was added dropwise at −78 °C and the mixture was slowly warmed to rt. After stirring for 47 h at rt, the reaction mixture was cooled to 0 °C, and quenched by the addition of satd aq NH_4Cl (50 mL), then extracted with Et_2O (3 × 50 mL). The combined organic extracts were washed with brine, dried over $MgSO_4$, filtered, and concentrated by rotary evaporation. Purification by silica flash chromatography (15:1 hexane/EtOAc) yielded methyl 4-methyl-2,2-diphenylpent-4-enoate (3.06 g, 82%) as a yellow oil. **TLC**: R_f 0.32 (10:1 hexane/EtOAc). **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.32−7.20 (m, 10H), 4.71 (m, 1H), 4.48 (m, 1H), 3.68 (s, 3H), 3.18 (d, J = 0.4 Hz, 2H), 1.36 (d, J = 0.4 Hz, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$): δ 174.5, 143.5, 142.3, 129.1, 128.0, 126.9, 115.5, 60.4, 52.3, 46.1, 24.5. **LRMS (EI)** *m/z* (rel int): (pos) 281 ($[M + H]^+$, 3), 280 ($[M]^+$, 12), 221 ($[M – CO_2CH_3]^+$, 63). **HRMS** *m/z* calcd for $C_{19}H_{20}O_2$ 280.1463; found 280.1469. **IR** (KBr film): 3060, 3028, 2950, 1724, 1642, 1599, 1443, 1264, 754, 698cm^{-1}.

Methyl 4-methyl-2,2-diphenylpent-4-enoate (2.70 g, 9.63 mmol) was added dropwise with stirring to a suspension of $LiAlH_4$ (512 mg, 13.5 mmol) in anhydrous THF (20 mL) at 0 °C. After stirring for 1 h at rt, the reaction mixture was cooled to 0 °C, diluted with Et_2O (20 mL), quenched by the sequential addition of water (0.7 mL), a 15% aq NaOH (0.7 mL), and additional water (2.1 mL). The resulting white precipitate was filtered off and washed with Et_2O. The filtrate was dried over $MgSO_4$, filtered, and concentrated *in vacuo*. Purification by silica flash chromatography (10:1 hexane/EtOAc) yielded **8m** (2.18 g, 90%) as a viscous colorless oil. **TLC**: R_f 0.22 (10:1 hexane/EtOAc). **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.31−7.26 (m, 4H), 7.23−7.18 (m, 6H), 4.84 (m, 1H), 4.66 (m, 1H), 4.25 (d, J = 6.8 Hz, 2H), 2.96 (s, 2H), 1.11 (dd, J = 1.2, 0.8 Hz, 3H), 1.09 (t, J = 6.8 Hz, 1H). **^{13}C NMR** (100 MHz, $CDCl_3$): δ 146.1, 142.9, 128.6, 128.4, 126.6, 115.9, 67.5, 51.7, 44.1, 24.6. **LRMS (CI)** *m/z* (rel int): (pos) 254 ($[M + H]^+$, 5), 253 ($[M]^+$, 3), 235 ($[M – OH]^+$, 41), 221 ($[M – CH_2OH]^+$, 18), 197 ($[Ph_2CCH_2OH]^+$, 100). **HRMS** *m/z* calcd for $C_{18}H_{20}O$ 252.1514; found 252.1516. **IR** (KBr film): 3443, 3060, 3028, 2947, 1642, 1600, 1445, 1025, 758, 700 cm^{-1}.



8n

(*E*)-2,2-Diphenylhex-4-en-1-ol (8n)

8n was synthesized according to the literature procedure.[11] A 2.5 M solution of *n*-BuLi in hexane (7.83 mL, 19.5 mmol) was added dropwise to a solution of diisopropylammine (1.74 g, 17.2 mmol) in anhydrous THF (13 mL) at –78 °C under Ar atmosphere and stirred 30 min at –78 °C. After stirring an additional 30 min at –5 °C, methyl 2,2-diphenylacetate (3.00 g, 13.3 mmol) in anhydrous THF (7 mL) was then added dropwise at –78 °C, stirred 10 min. Finally, crotyl bromide (3.16 g, 19.9 mmol) was added dropwise at –78 °C and the mixture was slowly warmed to rt. After stirring for 30 min at rt, the reaction mixture was cooled to 0 °C, and quenched by the addition of satd aq NH_4Cl (70 mL), then extracted with Et_2O (3 × 100 mL). The combined organic extracts were washed with brine, dried over $MgSO_4$, filtered, and concentrated by rotary evaporation. Purification by silica flash chromatography (10:1 hexane/EtOAc) yielded (*E*)-methyl 2,2-diphenylhex-4-enoate (3.56 g, 96%) as a colorless liquid. *E*:*Z* isomer = 5:1. **TLC**: R_f 0.66 (5:1 hexane/EtOAc). **^{1}H NMR** (*E* isomer, 400 MHz, $CDCl_3$): δ 7.32−7.22 (m, 10H), 5.32−5.18 (m, 2H), 3.69 (s, 3H), 3.08 (d, *J* = 5.6 Hz, 2H), 1.51 (br d, *J* = 6.0 Hz, 3H). **^{13}C NMR** (*E* isomer, 100 MHz, $CDCl_3$): δ 174.9, 142.9, 129.2, 128.0, 127.9, 126.9, 126.5, 60.8, 52.5, 41.8, 18.2. **LRMS (ESI)** *m/z* (rel int): (pos) 281 ($[M + H]^+$, 54), 280 ($[M]^+$, 28), 221 ($[M – COOCH_3]^+$, 100). **HRMS** *m/z* calcd for $C_{19}H_{20}O_2$ 280.1463, found 280.1467. **IR** (KBr film): 3059, 2950, 1732, 1599, 1446, 1222, 969, 751, 700 cm^{-1}.

(*E*)-Methyl 2,2-diphenylhex-4-enoate (3.27 g, 11.7 mmol) was added dropwise with stirring to a suspension of $LiAlH_4$ (619 mg, 16.3 mmol) in anhydrous THF (16 mL) at 0 °C. After stirring for 25 min at rt, the reaction mixture was cooled to 0 °C, diluted with Et_2O (24 mL), quenched by the sequential addition of water (0.62 mL), a 15% aq NaOH (0.62 mL), and additional water (1.86 mL). The resulting white precipitate was filtered off and washed with Et_2O. The filtrate was dried over $MgSO_4$, filtered, and concentrated *in vacuo*. Purification by silica flash chromatography (7:1 hexane/EtOAc) yielded **8n** (2.95 g, 100%) as a colorless oil. *E*:*Z* isomer = 6:1. **TLC**: R_f 0.30 (7:1 hexane/EtOAc). **^{1}H NMR** (*E* isomer, 400 MHz, $CDCl_3$): δ 7.32−7.16 (m, 10H), 5.51 (m, 1H), 5.07 (m, 1H), 4.14 (d, *J* = 6.8 Hz, 2H), 2.89 (dm, *J* = 6.4

Hz, 2H), 1.56 (dm, J = 5.6 Hz, 3H), 1.17 (t, J = 6.8 Hz, 1H). **^{13}C NMR** (*E* isomer, 100 MHz, $CDCl_3$): δ 145.7, 128.9, 128.4, 128.3, 126.9, 126.4, 68.1, 51.9, 39.9, 18.2. **LRMS (EI)** *m/z* (rel int): (pos) 252 ($[M]^+$, 2), 235 ($[M - OH]^+$, 5), 221 ($[M - CH_2OH]^+$, 23), 197 ($[Ph_2CCH_2OH]^+$, 100). **HRMS** *m/z* calcd for $C_{18}H_{20}O$ 252.1514, found 252.1515. **IR** (KBr film): 3428, 3027, 2934, 1599, 1445, 1058, 971, 754, 700 cm^{-1}.

Hydroalkoxylation in Brønsted acidic ionic liquids

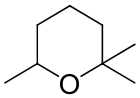
Representative NMR-Scale Reactions.

In an NMR tube, 2,2-diphenyl-4-penten-1-ol (**8k**) (12.2 mg, 51.0 μmol), mesitylene (6.3 mg, 51.0 μmol, internal standard), and the ionic liquid **5** (21.0 mg, 100 mol%) were dissolved in benzene-d_6 (0.75 mL). The tube was then heated at 80 °C for 24 h. The ensuing hydroalkoxylation/cyclization reaction was monitored by 1H NMR spectroscopy. The yield of **9k** was determined as 99% based on the internal standard mesitylene.

Typical Preparative-Scale Reactions.

In a 20 mL vial, 2,2-diphenyl-4-penten-1-ol (**8k**) (238 mg, 1.00 mmol) and the ionic liquid [BuTrBuSO$_3$H][OTf] (**5**) (412 mg, 100 mol%) were dissolved in benzene (5 mL). The reaction mixture was stirred at 80 °C for 43 h. Progress of the reaction was monitored by TLC. Upon the completion of the reaction, solvent was evaporated. The residue was purified by column chromatography (19:1 petroleum ether/Et_2O) to afford **9k** (220 mg, 93%) as a white solid.

All the products listed in Table 2 are known. Spectral data were identical to those previously reported.

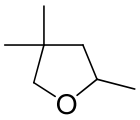


9a

2,2,6-Trimethyltetrahydropyran (9a)[13]

TLC: R_f 0.55 (5:1 hexane/EtOAc). **^{1}H NMR** (400 MHz, Benzene-d_6): δ 3.51 (m, 1H), 1.46−1.39 (m, 2H), 1.33−1.26 (m, 2H), 1.24 (s, 3H), 1.24−1.19 (m, 2H), 1.13 (d, J = 6.4 Hz, 3H), 1.08 (s, 3H). **^{1}H NMR** (400 MHz, $CDCl_3$): δ 3.67 (dqd, J = 11.2, 6.0, 2.4 Hz, 1H), 1.66−1.31 (m, 6H), 1.21 (s, 3H), 1.19 (s, 3H), 1.12 (d, J = 6.0 Hz, 3H). **^{13}C NMR** (100 MHz,

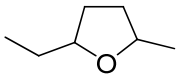
$CDCl_3$): δ 71.9, 66.6, 36.2, 33.6, 32.2, 23.0, 22.2, 20.3. **LRMS (ESI)** *m/z* (rel int): (pos) 128 ($[M]^+$, 29), 113 ($[M - CH_3]^+$, 66), 111 ($[M - OH]^+$, 43). **HRMS** *m/z* calcd for $C_8H_{16}O$ 128.1201, found 128.1203. **IR** (KBr film): 2934, 1457, 1091 cm^{-1}.



9b

2,4,4-Trimethyltetrahydrofuran (9b)[14]

TLC: R_f 0.62 (5:1 hexane/EtOAc). **^{1}H NMR** (400 MHz, Benzene-d_6): δ 4.00 (m, 1H), 3.48 (d, J = 8.0 Hz, 1H), 3.34 (d, J = 8.0 Hz, 1H), 1.48 (dd, J = 12.0, 6.4 Hz, 1H), 1.19 (d, J = 6.0 Hz, 3H), 1.07 (dd, J = 12.0, 8.8 Hz, 1H), 0.90 (s, 3H), 0.88 (s, 3H). **^{1}H NMR** (400 MHz, $CDCl_3$): δ 4.12 (m, 1H), 3.55 (d, J = 8.4 Hz, 1H), 3.43 (d, J = 8.4 Hz, 1H), 1.80 (dd, J = 12.0, 6.4 Hz, 1H), 1.30 (dd, J = 12.0, 8.8 Hz, 1H), 1.25 (d, J = 6.4 Hz, 3H), 1.10 (s, 3H), 1.08 (s, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$): δ 80.4, 75.5, 48.9, 40.2, 27.5, 26.9, 21.8. **LRMS (ESI)** *m/z* (rel int): (pos) 114 ($[M]^+$, 9), 113 ($[M - H]^+$, 100). **HRMS** *m/z* calcd for $C_7H_{14}O$ 114.1045, found 114.1042. **IR** (KBr film): 2928, 1461, 1073 cm^{-1}.



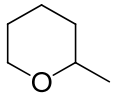
9c

2-Ethyl-5-methyltetrahydrofuran (9c)[15]

1.8:1 mixture of *trans:cis* determined by ^{1}H NMR analysis and *trans/cis* relationship established by analogy to 2-isopropyl-5-methyltetrahydrofuran.

TLC: R_f 0.63 (5:1 hexane/EtOAc). **^{1}H NMR** (400 MHz, Benzene-d_6): δ 3.97 (m, 1H, *trans*), 3.81 (m, 1H, *trans*), 3.81 (m, 1H, *cis*), 3.62 (m, 1H, *cis*), 1.76−1.19 (m, 6H, *trans/cis*), 1.18 (d, J = 6.0 Hz, 3H, *cis*), 1.14 (d, J = 6.0 Hz, 3H, *trans*), 0.93 (t, J = 7.6 Hz, 3H, *cis*), 0.91 (t, J = 7.6 Hz, 3H, *trans*). **^{1}H NMR** (400 MHz, $CDCl_3$): δ 4.10 (m, 1H, *trans*), 3.92 (m, 1H, *trans*), 3.92 (m, 1H, *cis*), 3.74 (m, 1H, *cis*), 2.06−1.99 (m, 2H, *trans*), 1.96−1.93 (m, 2H, *cis*), 1.61−1.39 (m, 4H, *trans/cis*), 1.24 (d, J = 6.0 Hz, 3H, *cis*), 1.21 (d, J = 6.0 Hz, 3H, *trans*), 0.93 (t, J = 7.2 Hz, 3H, *cis*), 0.91 (t, J = 7.2 Hz, 3H, *trans*). **^{13}C NMR** (100 MHz, $CDCl_3$): δ 81.1 (*cis*), 80.3 (*trans*), 75.4 (*cis*), 74.7 (*trans*), 34.2 (*trans*), 33.1 (*cis*), 32.1 (*trans*), 31.0 (*cis*), 29.1 (*trans/cis*), 21.6 (*trans/cis*), 10.5 (*trans/cis*). **LRMS (CI)** *m/z* (rel int): (pos) 115 ([M +

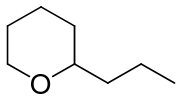
H]$^+$, 48), 97 ([M − OH]$^+$, 100). **HRMS** *m/z* calcd for $C_7H_{15}O$ 115.1123, found 115.1119. **IR** (KBr film): 2925, 1461, 1121 cm^{-1}.



9d

2-Methyltetrahydropyran (9d)[13]

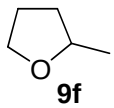
TLC: R_f 0.54 (5:1 hexane/EtOAc). **^{1}H NMR** (400 MHz, Benzene-d_6): δ 3.87 (m, 1H), 3.58 (m, 1H), 3.19 (m, 1H), 1.60−1.10 (m, 6H), 1.14 (d, J = 6.0 Hz, 3H). **^{1}H NMR** (400 MHz, $CDCl_3$): δ 3.96 (dm, J = 15.2 Hz, 1H), 3.47−3.37 (m, 2H), 1.80 (m, 1H), 1.60−1.45 (m, 3H), 1.30−1.21 (m, 2H), 1.15 (d, J = 6.4 Hz, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$): δ 74.1, 68.7, 33.9, 26.1, 23.8, 22.4. **LRMS (ESI)** *m/z* (rel int): (pos) 123 ([M + Na]$^+$, 12), 101 ([M + H]$^+$, 7), 100 ([M]$^+$, 9), 85 ([M – CH_3]$^+$, 100), 83 ([M – OH]$^+$, 33). **HRMS** *m/z* calcd for $C_6H_{12}O$ 100.0888, found 100.0893. **IR** (KBr film): 2928, 1460, 1125 cm^{-1}.



9e

2-Propyltetrahydropyran (9e)[16]

TLC: R_f 0.63 (5:1 hexane/EtOAc). **^{1}H NMR** (400 MHz, Benzene-d_6): δ 3.90 (m, 1H), 3.71 (m, 1H), 3.22 (m, 1H), 3.10 (m, 1H), 1.61−1.14 (m, 9H), 0.89 (t, J = 7.2 Hz, 3H). **^{1}H NMR** (400 MHz, $CDCl_3$): δ 3.97 (dm, J = 11.6 Hz, 1H), 3.41 (td, J = 11.6, 2.4 Hz, 1H), 3.24 (m, 1H), 1.81 (m, 1H), 1.61−1.20 (m, 9H), 0.91 (t, J = 7.2 Hz, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$): δ 77.8, 68.7, 39.0, 32.2, 26.5, 23.8, 18.9, 14.4. **LRMS (CI)** *m/z* (rel int): (pos) 129 ([M + H]$^+$, 23), 128 ([M]$^+$, 21), 111 ([M − OH]$^+$, 100). **HRMS** *m/z* calcd for $C_8H_{17}O$ 129.1279, found 129.1283. **IR** (KBr film): 2934, 1461, 1091 cm^{-1}.

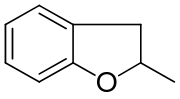


9f

2-Methyltetrahydrofuran (9f)[13]

TLC: R_f 0.64 (5:1 hexane/EtOAc). **^{1}H NMR** (400 MHz, Benzene-d_6): δ 3.77 (m, 2H), 3.54 (m, 1H), 1.64−1.48 (m, 4H), 1.15 (d, J = 6.0 Hz, 3H). **^{1}H NMR** (400 MHz, $CDCl_3$): δ 3.97−3.87 (m, 2H), 3.71 (td, J = 8.0, 6.4 Hz, 1H), 2.05−1.83 (m, 3H), 1.43 (m, 1H), 1.23 (d, J

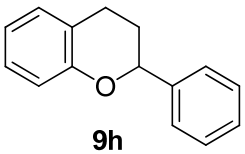
= 6.0 Hz, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$): δ 75.4, 67.9, 33.3, 26.1, 21.2. **LRMS (ESI)** *m/z* (rel int): (pos) 109 ($[M + Na]^+$, 7), 87 ($[M + H]^+$, 3), 86 ($[M]^+$, 3), 85 ($[M – H]^+$, 100), 69 ($[M – OH]^+$, 5). **HRMS** *m/z* calcd for $C_5H_{10}O$ 86.0732, found 86.0733. **IR** (KBr film): 2921, 1458, 1099 cm^{-1}.



9g

2,3-Dihydro-2-methylbenzofuran (9g)[13]

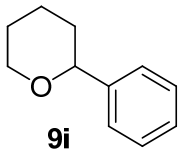
TLC: R_f 0.66 (5:1 hexane/EtOAc). **^{1}H NMR** (400 MHz, Benzene-d_6): δ 7.01−6.94 (m, 2H), 6.83 (d, *J* = 8.0 Hz, 1H), 6.76 (t, *J* = 8.0 Hz, 1H), 4.50 (m, 1H), 2.75 (dd, *J* = 15.2, 8.8 Hz, 1H), 2.31 (dd, *J* = 15.2, 7.6 Hz, 1H), 1.09 (d, *J* = 6.0 Hz, 3H). **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.15 (d, *J* = 7.6 Hz, 1H), 7.10 (t, *J* = 7.6 Hz, 1H), 6.82 (t, *J* = 7.2 Hz, 1H), 6.75 (d, *J* = 7.6 Hz, 1H), 4.92 (m, 1H), 3.31 (dd, *J* = 15.2, 8.8 Hz, 1H), 2.82 (dd, *J* = 15.2, 7.6 Hz, 1H), 1.47 (d, *J* = 6.4 Hz, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$): δ 159.7, 128.1, 127.2, 125.1, 120.3, 109.5, 79.7, 37.3, 21.9. **LRMS (EI)** *m/z* (rel int): (pos) 135 ($[M + H]^+$, 15), 134 ($[M]^+$, 100), 133 ($[M − H]^+$, 45), 119 ($[M − CH_3]^+$, 47). **HRMS** *m/z* calcd for $C_9H_{10}O$ 134.0732, found 134.0730. **IR** (KBr film): 3052, 2975, 1598, 1481, 1464, 1233, 1036, 750 cm^{-1}.



9h

2-Phenylchromane (9h)[17]

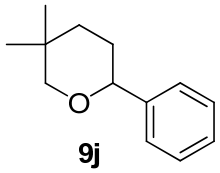
TLC: R_f 0.62 (5:1 hexane/EtOAc). **Mp**: 38 °C (Lit.[18] 40−41 °C). **^{1}H NMR** (400 MHz, Benzene-d_6): δ 7.28−7.25 (m, 2H), 7.18−7.16 (m, 2H) 7.11−7.01 (m, 3H), 6.91 (d, *J* = 7.6 Hz, 1H), 6.82 (t, *J* = 7.6 Hz, 1H), 4.74 (dd, *J* = 8.0, 4.4 Hz, 1H), 2.53 (m, 1H), 2.35 (dt, *J* = 16.4, 4.4 Hz, 1H), 1.76−1.70 (m, 2H). **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.46−7.36 (m, 4H), 7.32 (m, 1H), 7.15−7.08 (m, 2H), 6.91 (d, *J* = 8.4 Hz, 1H), 6.88 (td, *J* = 7.6, 1.2 Hz, 1H), 5.07 (dd, *J* = 10.0, 2.4 Hz, 1H), 3.01 (ddd, *J* = 16.4, 11.2, 5.2 Hz, 1H), 2.80 (ddd, *J* = 16.4, 5.2, 3.6 Hz, 1H), 2.21 (m, 1H), 2.09 (m, 1H). **^{13}C NMR** (100 MHz, $CDCl_3$): δ 155.3, 142.0, 129.7, 128.7, 128.0, 127.5, 126.2, 122.0, 120.5, 117.1, 78.0, 30.2, 25.3. **LRMS (FAB)** *m/z* (rel int): (pos) 210 ($[M]^+$, 20), 209 ($[M − H]^+$, 10). **HRMS** *m/z* calcd for $C_{15}H_{14}O$ 210.1045, found 210.1040.



9i

2-Phenyltetrahydropyran (9i)[19]

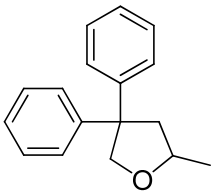
TLC: R_f 0.74 (3:1 hexane/EtOAc). **^{1}H NMR** (400 MHz, Benzene-d_6): δ 7.40 (d, J = 6.0 Hz, 2H), 7.21 (d, J = 6.0 Hz, 2H), 7.10 (m, 1H), 4.16 (d, J = 10.8 Hz, 1H), 4.00 (dm, J = 10.8 Hz, 1H), 3.32 (m, 1H), 1.63−1.13 (m, 6H). **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.30−7.23 (m, 5H), 4.32 (dd, J = 10.4, 2.4 Hz, 1H), 4.14 (m, 1H), 3.62 (td, J = 10.4, 2.4 Hz, 1H), 1.95 (m, 1H), 1.83 (m, 1H), 1.72−1.56 (m, 4H). **^{13}C NMR** (100 MHz, $CDCl_3$): δ 143.5, 128.5, 127.4, 126.0, 80.3, 69.2, 34.2, 26.1, 24.2. **LRMS (CI)** *m/z* (rel int):(pos) 163 ($[M + H]^+$, 18), 162 ($[M]^+$, 28), 161 ($[M - H]^+$, 100), 145 ($[M - OH]^+$, 20). **HRMS** *m/z* calcd for $C_{11}H_{13}O$ 161.0966, found 161.0961. **IR** (KBr film): 3032, 2938, 1605, 1494, 1453, 1090, 751, 699 cm^{-1}.



9j

5,5-Dimethyl-2-phenyltetrahydropyran (9j)[8]

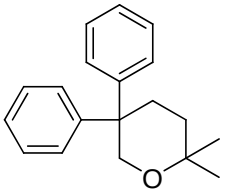
TLC: R_f 0.64 (5:1 hexane/EtOAc). **^{1}H NMR** (400 MHz, Benzene-d_6): δ 7.42 (d, J = 6.4 Hz, 2H), 7.21 (t, J = 6.4 Hz, 2H), 7.11 (t, J = 6.4 Hz, 1H), 4.08 (dd, J = 11.2, 2.8 Hz, 1H), 3.56 (dd, J = 11.2, 2.8 Hz, 1H), 3.13 (dd, J = 11.2, 0.8 Hz, 1H), 1.66 (m, 1H), 1.49 (m, 1H), 1.32 (m, 1H), 1.23 (dt, J = 13.2, 4.4 Hz, 1H), 1.04 (s, 3H), 0.65 (s, 3H). **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.39−7.24 (m, 5H), 4.24 (dd, J = 11.2, 2.8 Hz, 1H), 3.62 (dd, J = 11.2, 2.8 Hz, 1H), 3.34 (d, J = 11.2 Hz, 1H), 1.85−1.49 (m, 4H), 1.13 (s, 3H), 0.87 (s, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$): δ 143.4, 128.5, 127.4, 126.0, 80.4, 78.9, 37.5, 30.6, 30.0, 27.4, 23.7. **LRMS (FAB)** *m/z* (rel int): (pos) 191 ($[M + H]^+$, 22), 190 ($[M]^+$, 25), 189 ($[M - H]^+$, 80). **HRMS** *m/z* calcd for $C_{13}H_{17}O$ 189.1279, found 189.1281. **IR** (KBr film): 3032, 2949, 1606, 1495, 1453, 1093, 751, 699 cm^{-1}.



9k

2-Methyl-4,4-diphenyltetrahydrofuran (9k)[13]

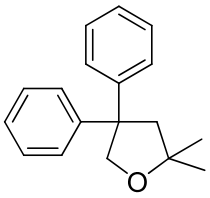
TLC: R_f 0.57 (5:1 hexane/EtOAc). **Mp**: 45 °C (Lit.[12] 42−44 °C). **^{1}H NMR** (400 MHz, Benzene-d_6): δ 7.26−7.23 (m, 2H), 7.12−6.99 (m, 8H), 4.49 (dd, J = 8.8, 0.8 Hz, 1H), 4.10 (d, J = 8.8 Hz, 1H), 4.05 (m, 1H), 2.26 (ddd, J = 12.0, 6.0, 0.8 Hz, 1H), 1.97 (dd, J = 12.0, 9.2 Hz, 1H), 1.15 (d, J = 6.0 Hz, 3H). **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.32−7.16 (m, 10H), 4.58 (dd, J = 8.8, 0.8 Hz, 1H), 4.17 (d, J = 8.8 Hz, 1H), 4.17 (m, 1H), 2.63 (ddd, J = 12.0, 6.0, 0.8 Hz, 1H), 2.27 (dd, J = 12.0, 9.6 Hz, 1H), 1.30 (d, J = 6.0 Hz, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$): δ 146.6, 146.5, 128.6, 128.5, 127.4 (2C), 126.6, 126.4, 77.1, 75.0, 56.6, 46.8, 21.6. **LRMS (FAB)** *m/z* (rel int): (pos) 239 ($[M + H]^+$, 15), 238 ($[M]^+$, 20), 237 ($[M - H]^+$, 55). **HRMS** *m/z* calcd for $C_{17}H_{17}O$ 237.1279, found 237.1277.



9l

2,2-Dimethyl-5,5-diphenyltetrahydropyran (9l)[8]

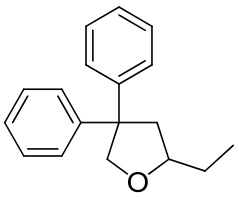
TLC: R_f 0.47 (10:1 hexane/EtOAc). **Mp**: 85 °C (Lit.[12] 85−86 °C). **^{1}H NMR** (400 MHz, Benzene-d_6): δ 7.32−7.30 (m, 4H), 7.14−7.09 (m, 4H) 7.05−6.99 (m, 2H), 4.04 (s, 2H), 2.21−2.18 (m, 2H), 1.26−1.23 (m, 2H), 1.11 (s, 6H). **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.33−7.25 (m, 8H), 7.19−7.15 (m, 2H), 4.05 (s, 2H), 2.44−2.41 (m, 2H), 1.42−1.38 (m, 2H), 1.23 (s, 6H). **^{13}C NMR** (100 MHz, $CDCl_3$): δ 146.6, 128.3, 128.1, 126.1, 71.4, 69.2, 46.1, 32.8, 31.0, 26.5. **LRMS (EI)** *m/z* (rel int): (pos) 266 ($[M]^+$, 10), 236 ($[M - 2CH_3]^+$, 18), 180 ($[Ph_2CCH_2]^+$, 100). **HRMS** *m/z* calcd for $C_{19}H_{22}O$ 266.1671, found 266.1676.



9m

2,2-Dimethyl-4,4-diphenyltetrahydrofuran (9m)[20]

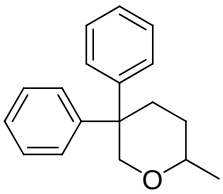
TLC: R_f 0.24 (10:1 hexane/EtOAc). **Mp**: 64 °C. **^{1}H NMR** (400 MHz, Benzene-d_6): δ 7.20−7.17 (m, 4H), 7.11−7.06 (m, 4H) 7.01−6.97 (m, 2H), 4.35 (s, 2H), 2.35 (s, 2H), 1.12 (s, 6H). **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.29−7.25 (m, 8H), 7.19−7.14 (m, 2H), 4.43 (s, 2H), 2.60 (s, 2H), 1.18 (s, 6H). **^{13}C NMR** (100 MHz, $CDCl_3$): δ 147.0, 128.5, 127.4, 126.3, 81.6, 75.6, 56.9, 51.6, 29.4. **LRMS (EI)** *m/z* (rel int): (pos) 253 ($[M + H]^+$, 10), 252 ($[M]^+$, 40), 222 ($[M - 2CH_3]^+$, 93), 193 ($[M - C_3H_7O]^+$, 25). **HRMS** *m/z* calcd for $C_{18}H_{20}O$ 252.1514, found 252.1515.



9n

2-Ethyl-4,4-diphenyltetrahydrofuran (9n)[8]

TLC: R_f 0.51 (10:1 hexane/EtOAc). **^{1}H NMR** (400 MHz, Benzene-d_6): δ 7.29−7.27 (m, 2H), 7.13−6.99 (m, 8H), 4.53 (dd, *J* = 8.4, 0.8 Hz, 1H), 4.06 (d, *J* = 8.4 Hz, 1H), 3.89 (m, 1H), 2.30 (ddd, *J* = 12.0, 5.6, 1.2 Hz, 1H), 2.04 (dd, *J* = 12.0, 9.6 Hz, 1H), 1.59 (m, 1H), 1.38 (m, 1H), 0.86 (t, *J* = 7.2 Hz, 3H). **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.32−7.26 (m, 6H), 7.23−7.16 (m, 4H), 4.61 (dd, *J* = 8.8, 0.8 Hz, 1H), 4.12 (d, *J* = 8.8 Hz, 1H), 3.96 (m, 1H), 2.61 (ddd, *J* = 12.0, 6.0, 1.2 Hz, 1H), 2.18 (dd, *J* = 12.0, 10.0 Hz, 1H), 1.67 (m, 1H), 1.56 (m, 1H), 0.93 (t, *J* = 7.6 Hz, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$): δ 146.6, 146.3, 128.6, 128.5, 127.4, 127.3, 126.6, 126.4, 80.3, 76.9, 56.2, 44.6, 29.1, 10.5. **LRMS (EI)** *m/z* (rel int): (pos) 252 ($[M]^+$, 15), 223 ($[M - C_2H_5]^+$, 13), 193 ($[M - C_3H_7O]^+$, 100), 180 ($[Ph_2CCH_2]^+$, 75). **HRMS** *m/z* calcd for $C_{18}H_{20}O$ 252.1514, found 252.1515. **IR** (KBr film): 3059, 2966, 1599, 1460, 1447, 1079, 750, 693 cm^{-1}.



9n'

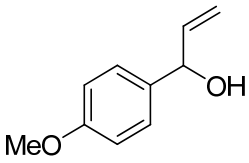
2-Methyl-5,5-diphenyltetrahydropyran (9n')[8]

TLC: R_f 0.54 (10:1 hexane/EtOAc). **^{1}H NMR** (400 MHz, Benzene-d_6): δ 7.51−7.48 (m, 2H), 7.40−7.37 (m, 4H), 7.20−7.14 (m, 4H), 4.66 (dd, *J* = 12.0, 3.2 Hz, 1H), 3.45 (d, *J* = 12.0 Hz, 1H), 3.28 (m, 1H), 2.24−2.15 (m, 2H), 1.38 (m, 1H), 1.20 (m, 1H), 1.09 (d, *J* = 6.4 Hz, 3H).**^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.44−7.41 (m, 2H), 7.31−7.23 (m, 4H), 7.19−7.14 (m, 4H), 4.63 (dd, *J* = 12.0, 2.8 Hz, 1H), 3.56 (d, *J* = 12.0 Hz, 1H), 3.56 (m, 1H), 2.50−2.37 (m, 2H), 1.55 (m, 1H), 1.24 (m, 1H), 1.17 (d, *J* = 6.0 Hz, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$): δ 147.0, 146.1, 129.1, 128.5, 128.2, 127.2, 126.4, 125.8, 75.3, 74.2, 46.0, 35.0, 30.0, 21.9. **LRMS (EI)** *m/z* (rel int): (pos) 252 ($[M]^+$, 15), 180 ($[Ph_2CCH_2]^+$, 100). **HRMS** *m/z* calcd for $C_{18}H_{20}O$ 252.1514, found 252.1519. **IR** (KBr film): 3059, 2935, 1600, 1495, 1446, 1095, 754, 700 cm^{-1}.

Reuse of [bmTr][PF_6] ionic liquid

In a 20 mL vial, 2,2-diphenyl-4-penten-1-ol (**8k**) (238 mg, 1.00 mmol) and ionic liquid **5** (412 mg, 1.00 mmol) were dissolved in benzene (5 mL). The reaction mixture was stirred at 80 °C, and the reaction progress was monitored by TLC. Upon completion of the reaction, Et_2O (5 mL) was added to the reaction mixture. The ionic liquid-Et_2O mixture was stirred for a couple of minutes and stopped. Then, the ionic liquid-Et_2O mixture clearly showed two separate layers although <0.5 mL of ionic liquid was used. The upper Et_2O layer was carefully removed by decantation. This procedure was repeated five times. The combined Et_2O fractions were evaporated. Purification by silica flash chromatography (petroleum ether/Et_2O = 19:1) afforded **9k** (226 mg, 95%) as a white solid. The recovered ionic liquid (406 mg) was dried *in vacuo* and reused for the next cycle.

Synthesis of (±)–Centrolobin

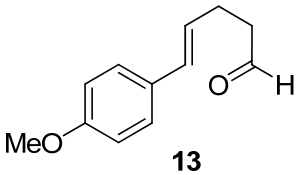


11

1-(4-Methoxyphenyl)prop-2-en-1-ol (11)

In a flame-dried 100 mL flask, *p*-anisaldehyde (3.57 mL, 29.4 mmol) was dissolved in anhydrous THF (24 mL) at 0 ^{o}C under argon atmosphere. Vinylmagnesium bromide (1.0 M in THF, 35.3 mL, 35.3 mmol) was then added via syringe dropwise. After the mixture was stirred for 1.5 h, it was quenched with sat. NH_4Cl (20 mL), and extracted with diethylether (3 × 70 mL). The combined organic layers were dried over anhydrous $MgSO_4$, filtered, and evaporated. The residue was purified by column chromatography (4:1 hexane/EtOAc) to give compound **11** (4.26 g, 88%) as a yellow oil.

TLC: R_f 0.26 (4:1 hexane/EtOAc). **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.30 (d, *J* = 8.8 Hz, 2H), 6.89 (d, *J* = 8.8 Hz, 2H), 6.09–6.01 (m, 1H), 5.34 (dt, *J* = 17.2, 1.2 Hz, 1H), 5.19 (dt, *J* = 10.0, 1.2 Hz, 1H), 5.17 (m, 1H), 3.80 (s, 3H), 1.82 (d, *J* = 4.0 Hz, 1H). **^{13}C NMR** (100 MHz, $CDCl_3$): δ 159.5, 140.6, 135.1, 127.9, 115.0, 114.2, 75.1, 55.5. **LRMS (FAB)** *m/z* (rel int): (pos) 164 ($[M]^+$, 25), 147 ($[M - OH]^+$, 100), 107 ($[PhOCH_3]^+$, 13). **HRMS** *m/z* calcd for $C_{10}H_{12}O_2$ 164.0837, found 164.0831. **IR** (KBr film): 3401, 3078, 2838, 1611, 1586, 1463, 1248, 831 cm^{-1}.



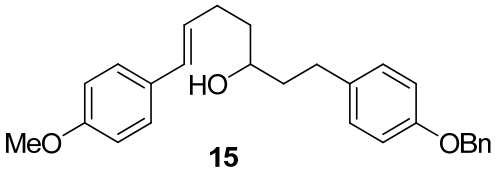
13

5-(4-Methoxyphenyl)pent-4-enal (13)

Under argon atmosphere, $Hg(OAc)_2$ (776 mg, 2.44 mmol), NaOAc (239 mg, 2.44 mmol), and allylic alcohol **11** were dissolved in *n*-butyl vinyl ether (30 mL). The reaction mixture was stirred at reflux for 17 h. The mixture was then cooled to rt. $NaSH{\cdot}nH_2O$ solution (228 mg in 100 ml H_2O) was added and extracted with EtOAc (3 × 100 mL). The combined organic layers were dried over anhydrous $MgSO_4$, filtered, and evaporated. Column chromatography (6:1 hexane/EtOAc) afforded compound **13** (3.82 g, 83%) as a yellow liquid.

TLC: R_f 0.24 (6:1 hexane/EtOAc). **^{1}H NMR** (400 MHz, $CDCl_3$): δ 9.82 (t, *J* = 1.4 Hz, 1H), 7.26 (d, *J* = 8.4 Hz, 2H), 6.84 (d, *J* = 8.4 Hz, 2H), 6.38 (d, *J* = 16.0 Hz, 1H), 6.06 (dt, *J* = 16.0,

6.8 Hz, 1H), 3.8 (s, 3H), 2.64–2.60 (m, 2H), 2.56–2.50 (m, 2H). **^{13}C NMR** (100 MHz, $CDCl_3$): δ 202.1, 159.2, 130.7, 130.2, 127.4, 126.1, 114.2, 55.5, 43.7, 25.7. **LRMS (EI)** *m/z* (rel int): (pos) 190 ($[M]^+$, 55), 147 ($[M - CH_2CHO]^+$, 48). **HRMS** *m/z* calcd for $C_{12}H_{14}O_2$ 190.0994, found 190.0995. **IR** (KBr film): 3031, 3004, 2908, 2836, 2724, 1722, 1607, 1463, 1247, 840 cm^{-1}.

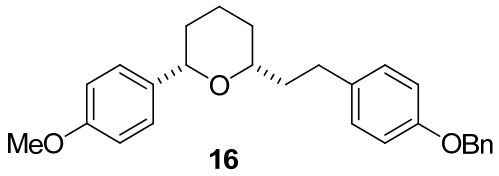


15

(±)-1-(4-(Benzyloxy)phenyl)-7-(4-methoxyphenyl)hept-6-en-3-ol (15)

Mg (230 mg, 9.46 mmol) in a flame-dried flask was heated with a torch, and cooled. This activation procedure was repeated three times. 1-(2-Bromoethyl)-4-(phenylmethoxy)benzene (**14)** (2.30 g, 7.88 mmol) in THF (10 mL) was then added. The resulting reaction mixture was heated at 70 °C for 2 h. **13** (1.00 g, 5.26 mmol) in THF (10 mL) was added dropwise at −78 °C and stirred for 1 h. The reaction mixture was stirred for an additional hour at rt. After the completion of the reaction, sat NH_4Cl (100 mL) was added and extracted with EtOAc (3 × 100 mL). The combined organic layers were dried over anhydrous Na_2SO_4, filtered, and evaporated. Column chromatography (4:1 hexane/EtOAc) afforded compound **15** (1.52 g, 72%) as a white solid.

TLC: R_f 0.38 (2:1 hexane/EtOAc). **Mp**: 82–84 °C. **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.44–7.32 (m, 5H), 7.26 (d, *J* = 8.8 Hz, 2H), 7.12 (d, *J* = 8.8 Hz, 2H), 6.90 (d, *J* = 8.8 Hz, 2H), 6.83 (d, *J* = 8.8 Hz, 2H), 6.35 (d, *J* = 16.0 Hz, 1H), 6.07 (dt, *J* = 16, 6.8 Hz, 1H), 5.03 (s, 2H), 3.79 (s, 3H), 3.69 (m, 1H), 2.73 (m, 1H), 2.63 (m, 1H), 2.35–2.26 (m, 2H), 1.80–1.73 (m, 2H), 1.68–1.61 (m, 2H), 1.39 (d, *J* = 5.2 Hz, 1H). **^{13}C NMR** (100 MHz, $CDCl_3$): δ 159.0, 157.3, 137.4, 134.6, 130.7, 129.9, 129.5, 128.8, 128.3, 128.1, 127.7, 127.3, 115.1, 114.2, 71.1, 70.3, 55.5, 39.5, 37.4, 31.4, 29.5. **LRMS (FAB)** *m/z* (rel int): (pos) 402 ($[M]^+$, 40), 197 ($[PhCH_2OC_6H_4CH_2]$)$^+$, 20), 107 ($[PhOCH_3]^+$, 31), 91 ($[PhCH_2]^+$, 89). **HRMS** *m/z* calcd for $C_{27}H_{30}O_3$ 402.2195, found 402.2201. Anal. Calcd for $C_{27}H_{30}O_3$: C, 80.56; H, 7.51. Found: C, 80.57; H, 7.52.



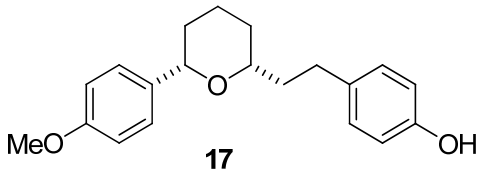
16

(±)-2-(4-(Benzyloxy)phenethyl)-tetrahydro-6-(4-methoxyphenyl)-2*H*-pyran (16)

In a 20 mL vial, alkenyl alcohol **15** (201 mg, 0.5 mmol) and ionic liquid **4** (210 mg, 0.5 mmol) were dissolved in anhydrous benzene (2.5 ml, 0.2 M). The biphasic solution was stirred at rt for 110 h. The reaction was concentrated by rotary evaporation. Purification with silica flash chromatography (10:1 hexane/EtOAc) yielded the **16** (154 mg, 77%) as a white solid.

In a 20 mL vial, **15** (201 mg, 0.5 mmol) and ionic liquid **5** (210 mg, 0.5 mmol) were dissolved in anhydrous benzene (2.5 ml, 0.2 M). The biphasic solution was stirred at rt for 38 h. The reaction was concentrated by rotary evaporation. Purification with silica flash chromatography (10:1 hexane/EtOAc) yielded the **16** (165 mg, 82%) as a white solid.

TLC: R_f 0.53 (10:1 hexane/EtOAc). **Mp**: 66–67 °C. **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.44–7.30 (m, 7H), 7.11 (d, *J* = 8.8 Hz, 2H), 6.89 (d, *J* = 8.8 Hz, 2H), 6.88 (d, *J* = 8.8 Hz, 2H), 5.04 (s, 2H), 4.29 (dd, *J* = 11.2, 2.0 Hz, 1H), 3.80 (s, 3H) 3.43 (m, 1H), 2.78–2.63 (m, 2H), 2.04–1.86 (m, 2H), 1.83 (m, 1H), 1.73 (m, 1H), 1.65–1.59 (m, 2H), 1.50 (m, 1H), 1.32 (dd, *J* = 7.2, 4.0 Hz, 1H). **^{13}C NMR** (100 MHz, $CDCl_3$): δ 158.9, 157.1, 137.5, 136.1, 135.1, 129.6, 128.8, 128.1, 127.7, 127.3, 114.9, 113.8, 79.3, 77.4, 70.3, 55.5, 38.5, 33.6, 31.5, 31.0, 24.3. **LRMS (FAB)** *m/z* (rel int): (pos) 402 ($[M]^+$, 57), 197 ($[PhCH_2OC_6H_4CH_2]^+$, 32), 91 ($[PhCH_2]^+$, 100). **HRMS** *m/z* calcd for $C_{27}H_{30}O_3$ 402.2195, found 402.2189. Anal. Calcd for $C_{27}H_{30}O_3$: C, 80.56; H, 7.51. Found: C, 80.50; H, 7.64.



17

(±)-4-(2-(Tetrahydro-6-(4-methyoxyphenyl)-2*H*-pyran-2-yl)ethyl)phenol (17)

In a 15 mL roundbottom flask, **16** (100 mg, 0.248 mmol) was dissolved in anhydrous EtOH (4 mL). Solid 5% Pd/C (20 mg) was added and the flask was flushed with H_2 gas, then stirred under a balloon of H_2 at rt for 3 h. The mixture was filtered over a celite and concentrated by

rotary evaporation. Purification by silica flash chromatography (4: 1 hexane/EtOAc) yielded **17** (76.3 mg, 98%) as a white solid.

TLC: R_f 0.29 (4:1 hexane/EtOAc). **Mp**: 69–70 °C (Lit.[21] 82–85 °C). **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.31 (d, *J* = 8.4 Hz, 2H), 7.05 (d, *J* = 8.4 Hz, 2H), 6.88 (d, *J* = 8.4 Hz, 2H), 6.72 (d, *J* = 8.4 Hz, 2H), 4.67 (s, 1H), 4.29 (dd, *J* = 11.2, 2.0 Hz, 1H), 3.80 (s, 3H), 3.43 (m, 1H), 2.76–2.61 (m, 2H), 1.95–1.80 (m, 3H), 1.76–1.58 (m. 3H), 1.50 (m, 1H), 1.38–1.30 (m, 1H). **^{13}C NMR** (100 MHz, $CDCl_3$): δ 158.9, 153.7, 136.1, 134.9, 129.8, 127.3, 115.3, 113.8, 79.3, 77.4, 55.5, 38.5, 33.5, 31.5, 31.0, 24.3. **LRMS (EI)** *m/z* (rel int): (pos) 312 ($[M]^+$, 100), 294 ($[M - H_2O]^+$, 6), 107 ($[PhOCH_3]^+$, 83), 91 ($[PhCH_2]^+$, 6). **HRMS** *m/z* calcd for $C_{20}H_{24}O_3$ 312.1725, found 312.1726.

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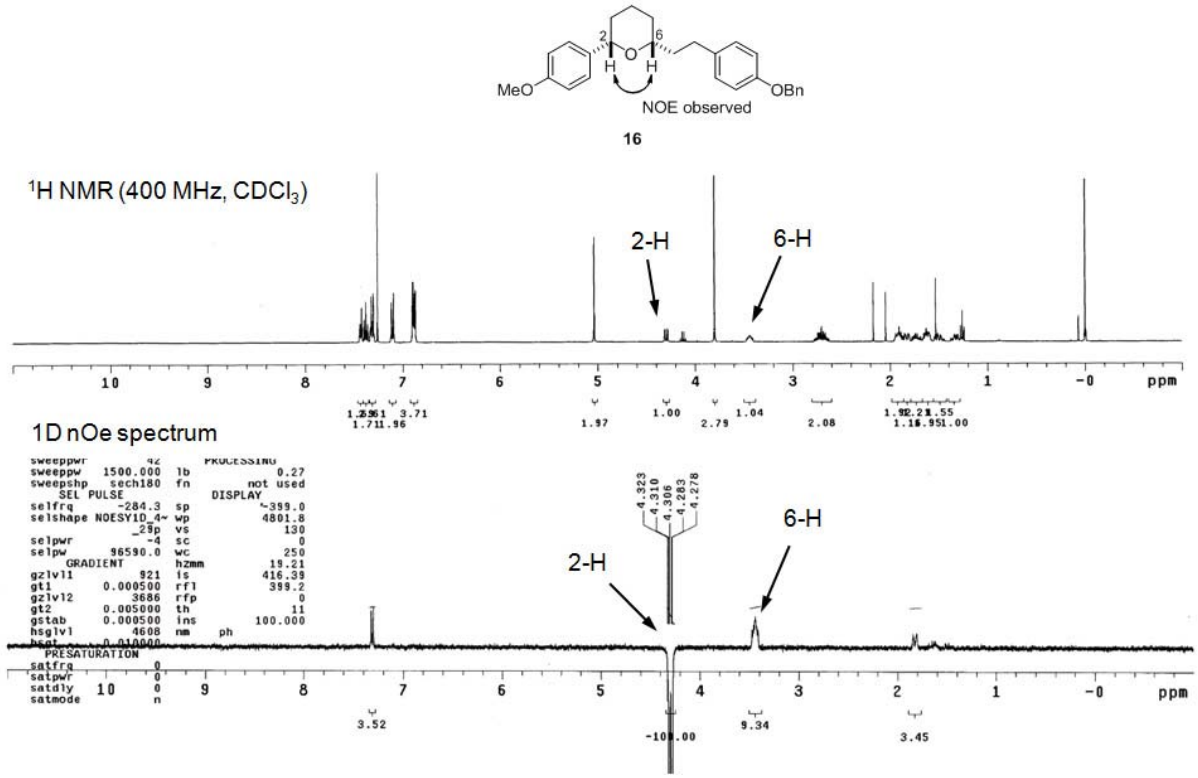
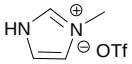
nOe spectrum of 16



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



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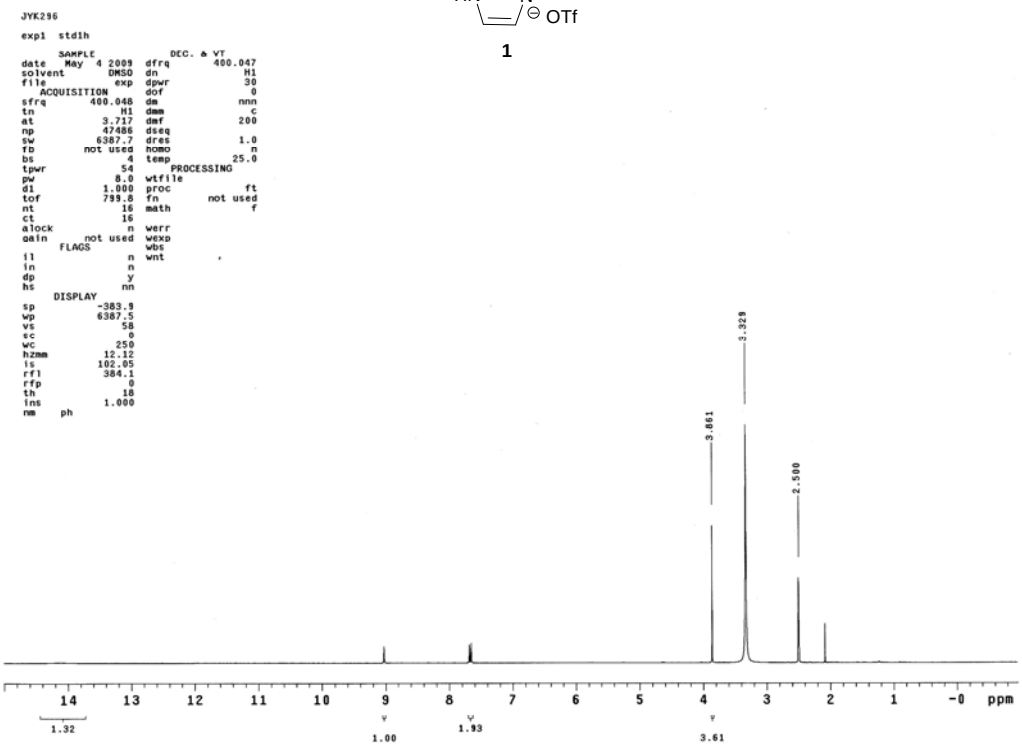

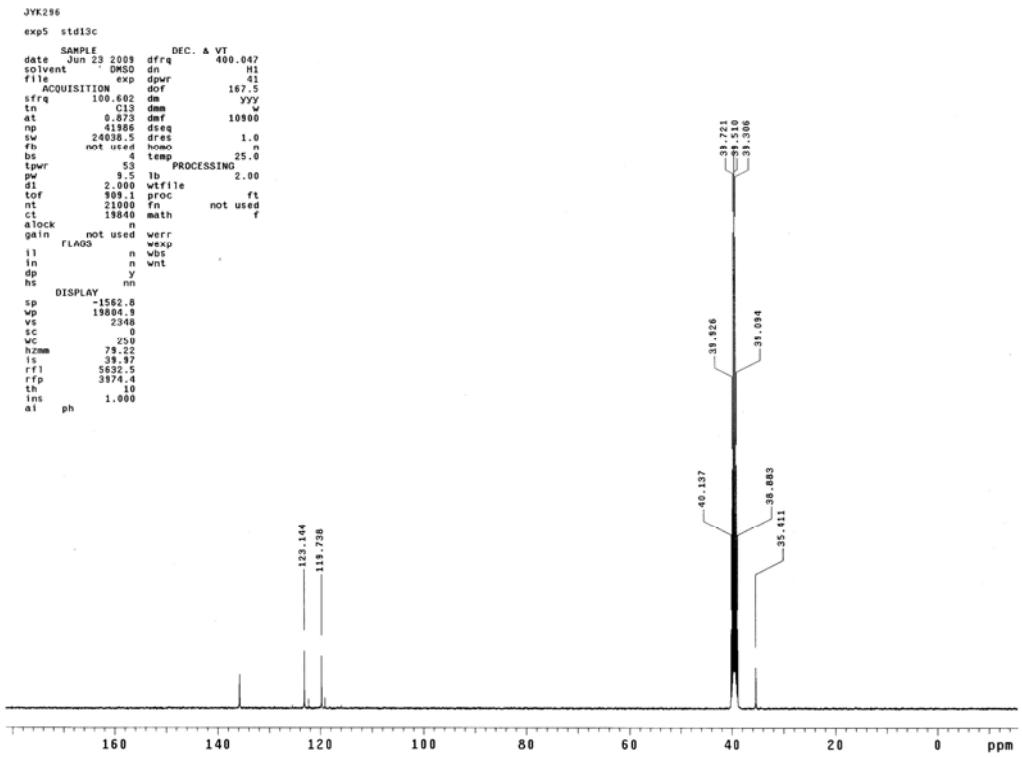

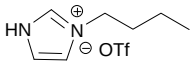
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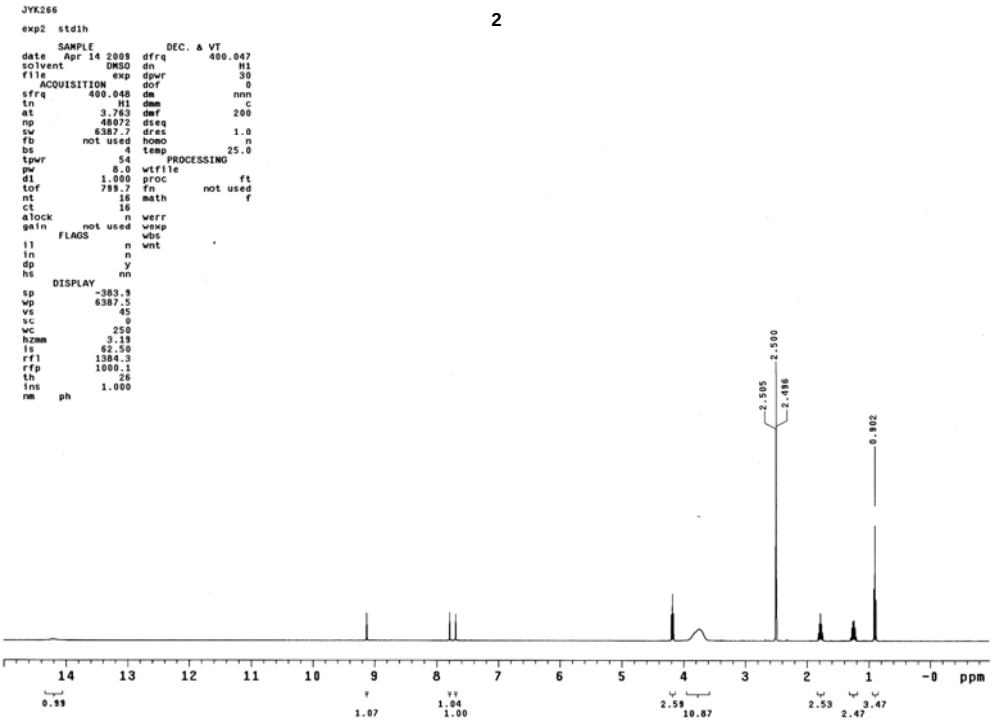
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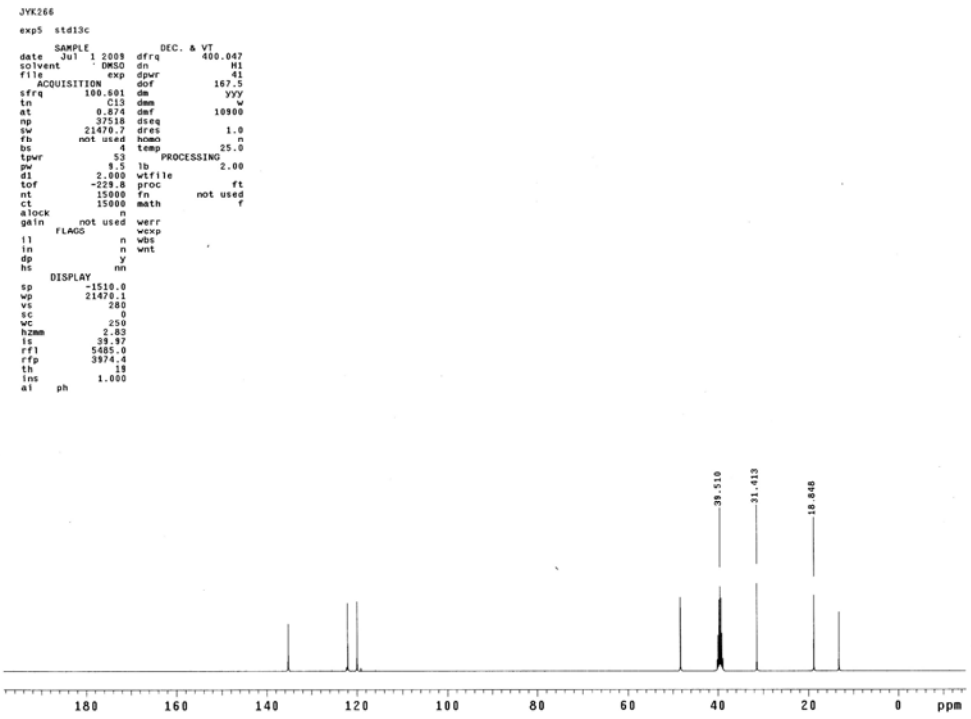
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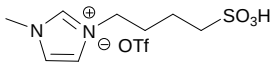
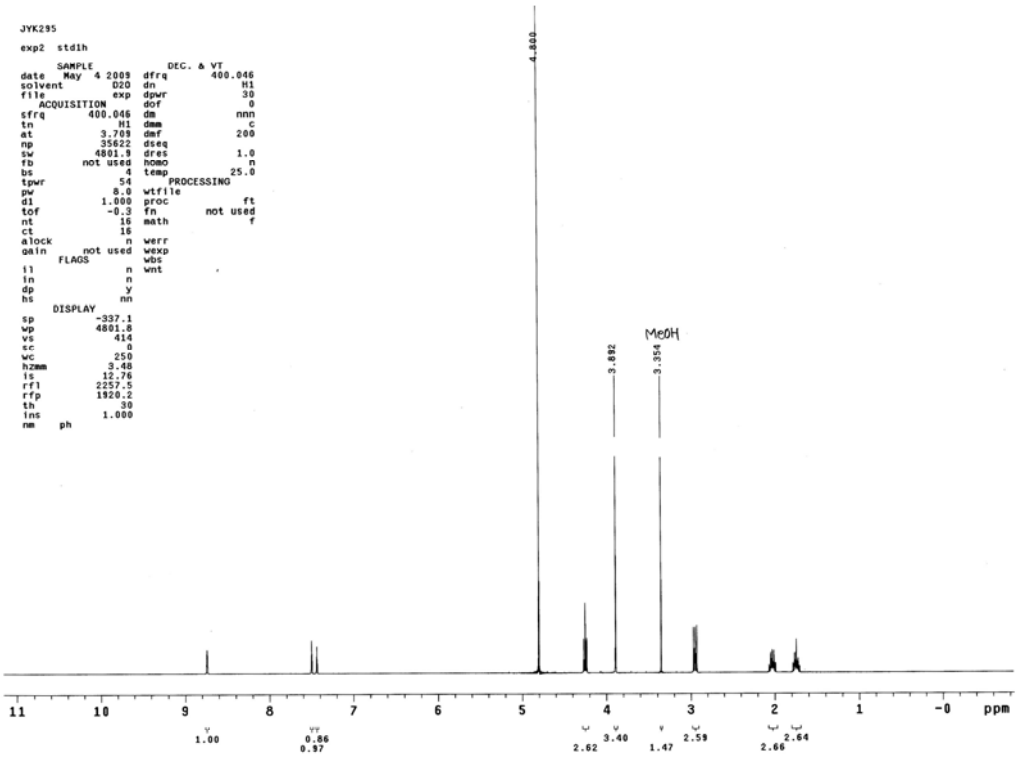
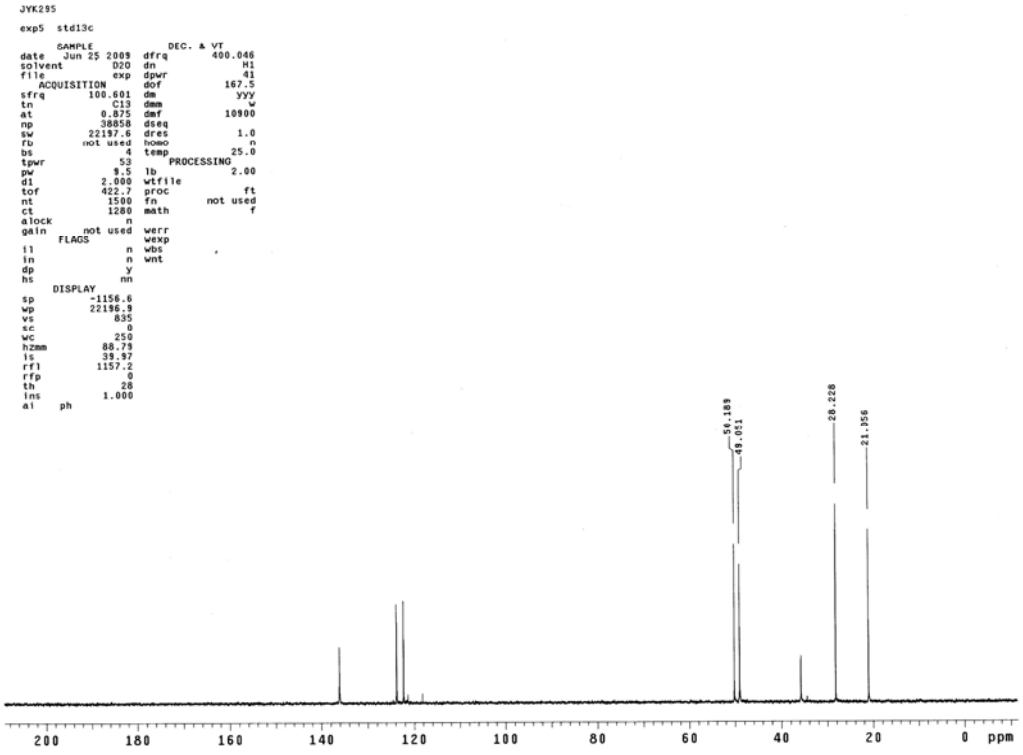
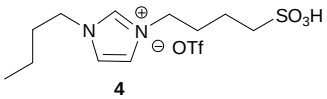
80
60
40
20
0
ppm


N
N
SO3H
OTf

3

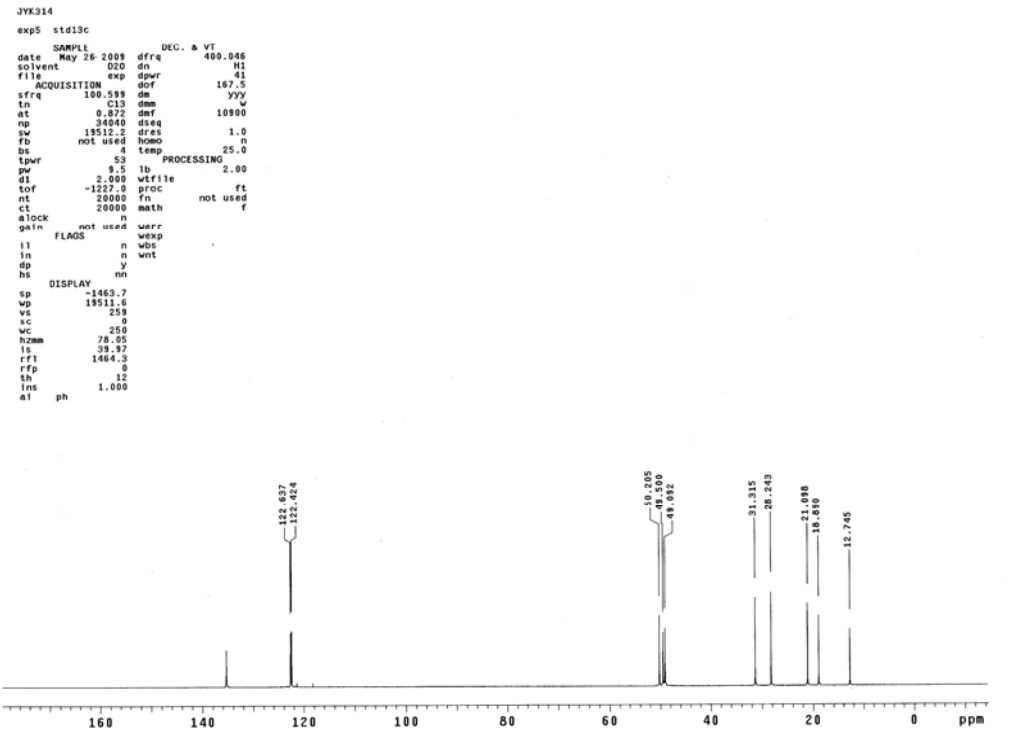

JYK295
exp2 std1h
SAMPLE
date May 4 2009
solvent D2O
file exp
ACQUISITION
sfrq 400.046
tn H1
at 3.709
np 35622
sw 4801.9
fb not used
bs 4
tpwr 54
pw 8.0
d1 1.000
tof -0.3
nt 16
ct 16
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -337.1
wp 4801.8
vs 414
sc 0
wc 250
hzmm 3.48
is 12.76
rfl 2257.5
rfp 1920.2
th 30
ins 1.000
nm ph
DEC. & VT
dfrq 400.046
dn H1
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
dseq
dres 1.0
homo n
temp 25.0
PROCESSING
wtfile
proc ft
fn not used
math f
werr
wexp
wbs
wnt
4.808
3.892
3.354
MeOH
11
10
9
8
7
6
5
4
3
2
1
-0
ppm
1.00
0.86
0.97
2.62
3.40
1.47
2.59
2.66
2.64

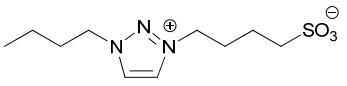

JYK295
exp5 std13c
SAMPLE
date Jun 25 2009
solvent D2O
file exp
ACQUISITION
sfrq 100.601
tn C13
at 0.875
np 38858
sw 22197.6
fb not used
bs 4
tpwr 53
pw 9.5
d1 2.000
tof 422.7
nt 1500
ct 1280
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -1156.6
wp 22196.9
vs 835
sc 0
wc 250
hzmm 88.79
is 39.97
rfl 1157.2
rfp 0
th 28
ins 1.000
ai ph
DEC. & VT
dfrq 400.046
dn H1
dpwr 41
dof 167.5
dm yyy
dmm w
dmf 10900
dseq
dres 1.0
homo n
temp 25.0
PROCESSING
lb 2.00
wtfile
proc ft
fn not used
math f
werr
wexp
wbs
wnt
56.189
49.051
28.228
21.956
200
180
160
140
120
100
80
60
40
20
0
ppm


N
N
SO3H
OTf

4

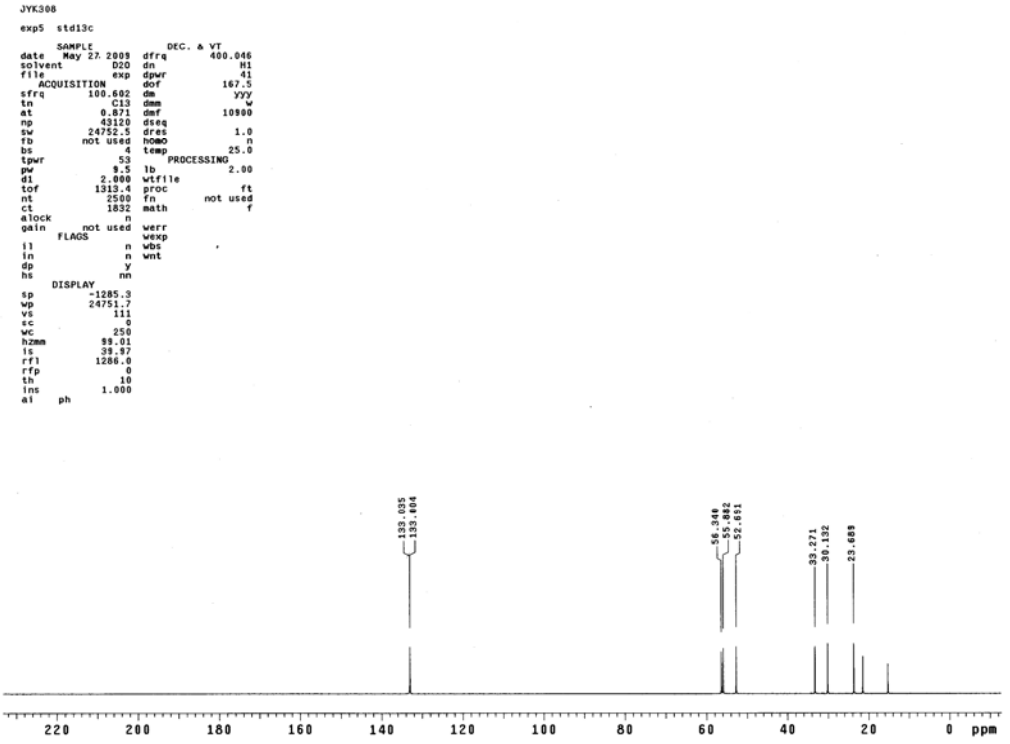
JYK314
exp2 std1h
SAMPLE
date May 22 2009
solvent D2O
file exp
ACQUISITION
sfrq 400.046
tn H1
at 3.665
np 35194
sw 4801.9
fb not used
bs 4
tpwr 54
pw 8.0
d1 1.000
tof -0.3
nt 16
ct 16
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -343.3
wp 4801.8
vs 50
sc 0
wc 250
hzmm 6.69
is 61.82
rfl 2263.6
rfp 1920.2
th 17
ins 1.000
nm ph
DEC. & VT
dfrq 400.046
dn H1
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
dseq
dres 1.0
homo n
temp 25.0
PROCESSING
wtfile
proc ft
fn not used
math f
werr
wexp
wbs
wnt
4.000
0.891
11
10
9
8
7
6
5
4
3
2
1
-0
ppm
1.00
1.92
2.24
2.23
2.15
2.36
2.262.20
2.21
3.08

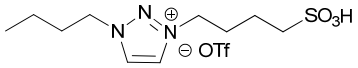

JYK314
exp5 std13c
SAMPLE
date May 26 2009
solvent D2O
file exp
ACQUISITION
sfrq 100.599
tn C13
at 0.872
np 34040
sw 19512.2
fb not used
bs 4
tpwr 53
pw 9.5
d1 2.000
tof -1227.0
nt 20000
ct 20000
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -1463.7
wp 19511.6
vs 259
sc 0
wc 250
hzmm 78.05
is 39.97
rfl 1464.3
rfp 0
th 12
ins 1.000
al ph
DEC. & VT
dfrq 400.046
dn H1
dpwr 41
dof 167.5
dm yyy
dmm w
dmf 10900
dseq
dres 1.0
homo n
temp 25.0
PROCESSING
lb 2.00
wtfile
proc ft
fn not used
math f
werr
wexp
wbs
wnt
122.637
122.424
50.205
49.580
49.082
31.315
28.243
21.098
18.880
12.745
160
140
120
100
80
60
40
20
0
ppm



7

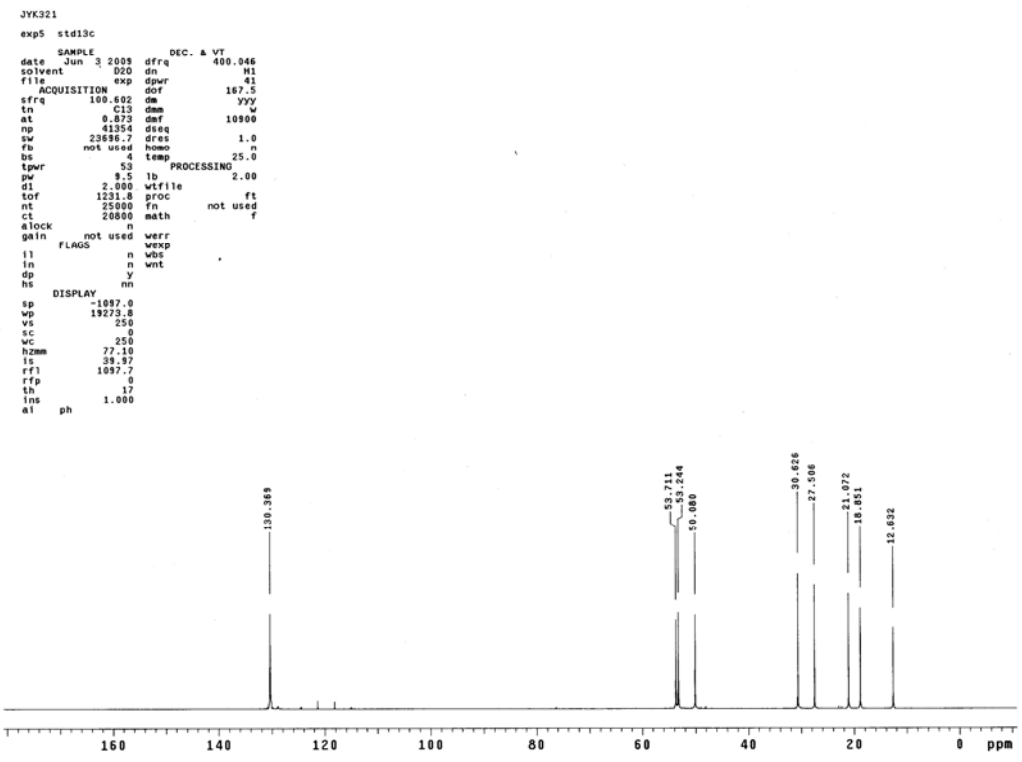
JYK308
exp1 std1h
SAMPLE
date May 25 2009
solvent D2O
file exp
ACQUISITION
sfrq 400.046
tn H1
at 3.757
np 36084
sw 4801.9
fb not used
bs 4
tpwr 54
pw 8.0
d1 1.000
tof -0.4
nt 16
ct 16
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -336.7
wp 4801.8
vs 30
sc 0
wc 250
hzmm 1.50
is 171.45
rfl 2257.1
rfp 1920.2
th 13
ins 1.000
nm ph
DEC. & VT
dfrq 400.046
dn H1
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
dseq
dres 1.0
homo n
temp 25.0
PROCESSING
wtfile
proc ft
fn not used
math f
werr
wexp
wbs
wnt
4.800
0.968
11 10 9 8 7 6 5 4 3 2 1 -0 ppm
0.78
1.00
1.01
1.00
1.03
1.01
1.03
0.98
1.35

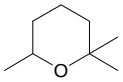

JYK308
exp5 std13c
SAMPLE
date May 27 2009
solvent D2O
file exp
ACQUISITION
sfrq 100.602
tn C13
at 0.871
np 43120
sw 24752.5
fb not used
bs 4
tpwr 53
pw 9.5
d1 2.000
tof 1313.4
nt 2500
ct 1832
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -1285.3
wp 24751.7
vs 111
sc 0
wc 250
hzmm 99.01
is 39.87
rfl 1286.0
rfp 0
th 10
ins 1.000
ai ph
DEC. & VT
dfrq 400.046
dn H1
dpwr 41
dof 167.5
dm yyy
dmm w
dmf 10900
dseq
dres 1.0
homo n
temp 25.0
PROCESSING
lb 2.00
wtfile
proc ft
fn not used
math f
werr
wexp
wbs
wnt
133.035
133.404
56.346
55.882
52.691
33.271
30.132
23.689
220 200 180 160 140 120 100 80 60 40 20 0 ppm


SO3H
N
N
⊕
⊖ OTf

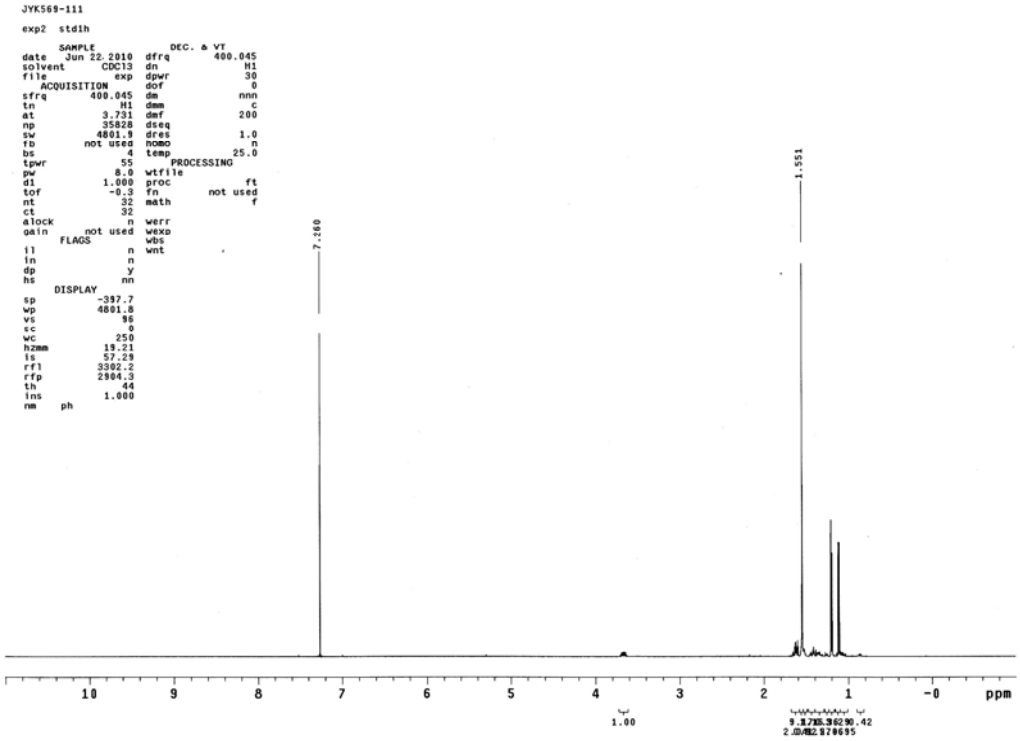
5

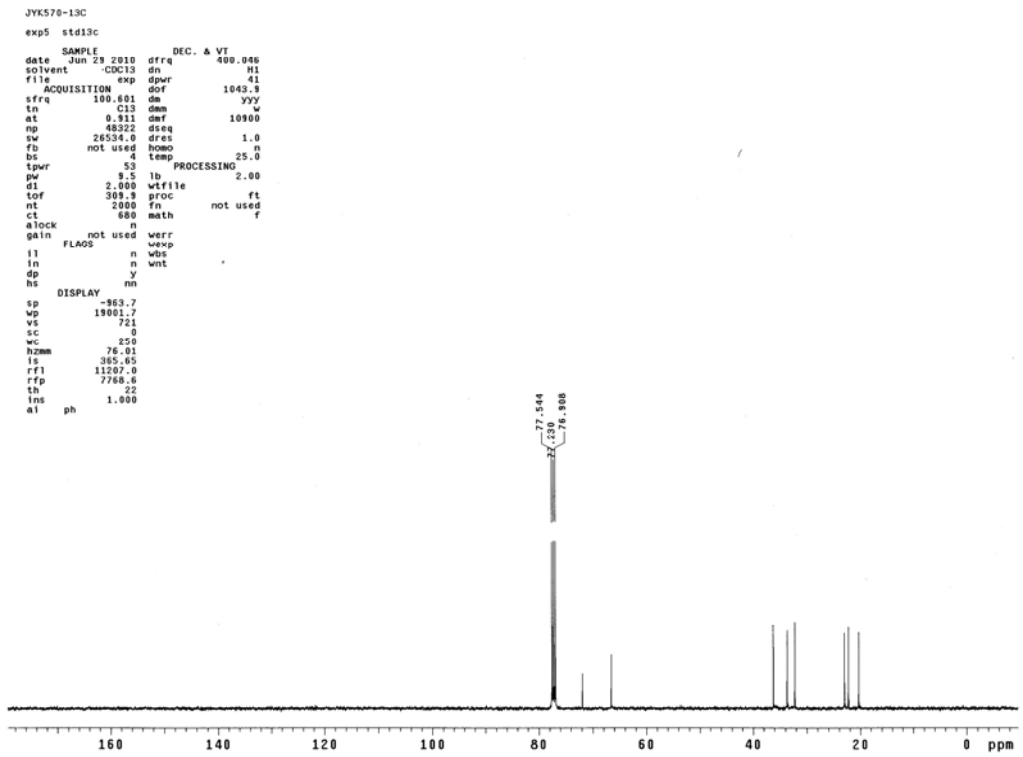
JYK321-080529
exp2 std1h
SAMPLE
date May 29 2008
solvent D2O
file exp
ACQUISITION
sfrq 400.046
tn H1
at 3.760
np 36114
sw 4801.9
fb not used
bs 4
tpwr 54
pw 8.0
d1 1.000
tof -0.4
nt 16
ct 16
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -347.1
wp 4801.8
vs 33
sc 0
wc 250
hzmm 7.25
is 125.00
rfl 2267.5
rfp 1920.2
th 8
ins 1.000
nm ph
DEC. & VT
dfrq 400.046
dn H1
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
dseq
dres 1.0
homo n
temp 25.0
PROCESSING
wtfile
proc ft
fn not used
math f
werr
wexp
wbs
wnt
4.800
4.646
4.599
0.906
0.887
11
10
9
8
7
6
5
4
3
2
1
-0
ppm
0.83
1.83
1.00
0.98
1.00
1.00
1.06
1.03
1.45

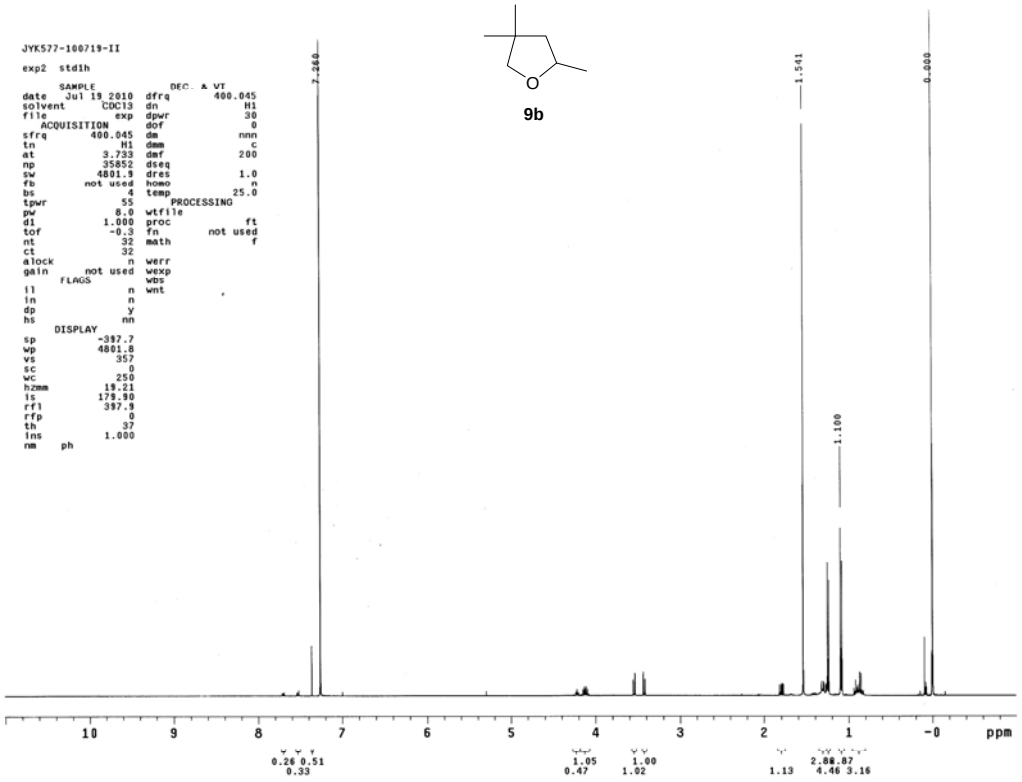

JYK321
exp5 std13c
SAMPLE
date Jun 3 2009
solvent D2O
file exp
ACQUISITION
sfrq 100.602
tn C13
at 0.873
np 41354
sw 23696.7
fb not used
bs 4
tpwr 53
pw 9.5
d1 2.000
tof 1231.8
nt 25000
ct 20800
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -1097.0
wp 19273.8
vs 250
sc 0
wc 250
hzmm 77.10
is 39.97
rfl 1097.7
rfp 0
th 17
ins 1.000
ai ph
DEC. & VT
dfrq 400.046
dn H1
dpwr 41
dof 167.5
dm yyy
dmm w
dmf 10900
dseq
dres 1.0
homo n
temp 25.0
PROCESSING
lb 2.00
wtfile
proc ft
fn not used
math f
werr
wexp
wbs
wnt
130.369
53.711
53.244
50.080
30.626
27.506
21.072
18.851
12.632
160
140
120
100
80
60
40
20
0
ppm



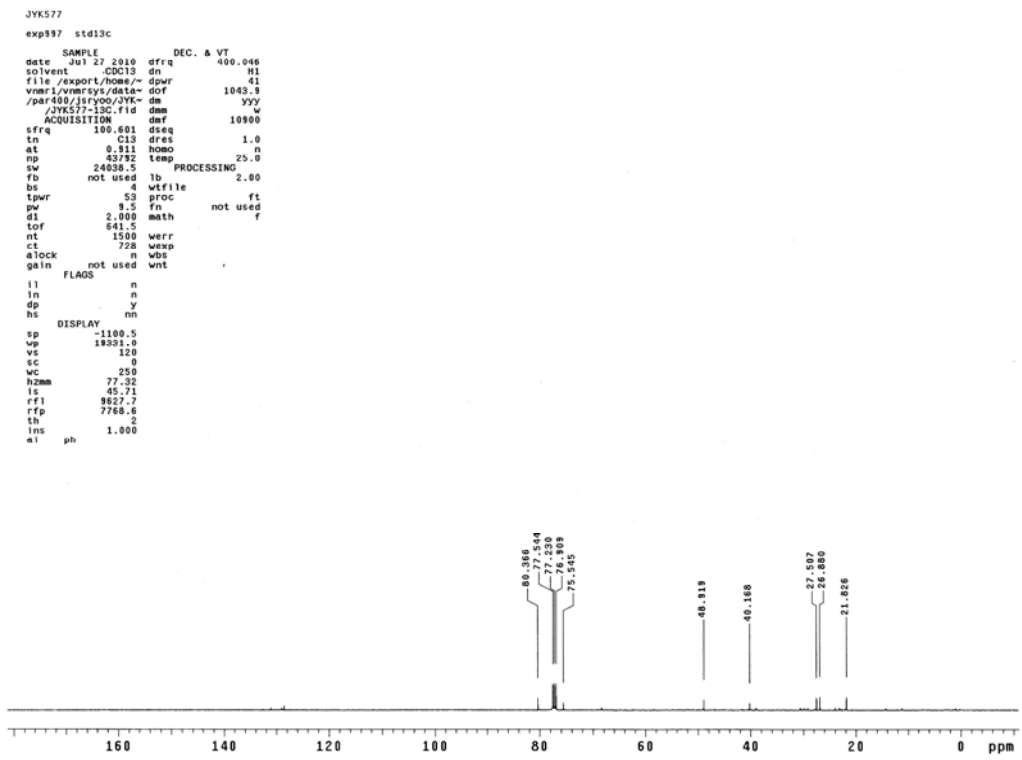
9a

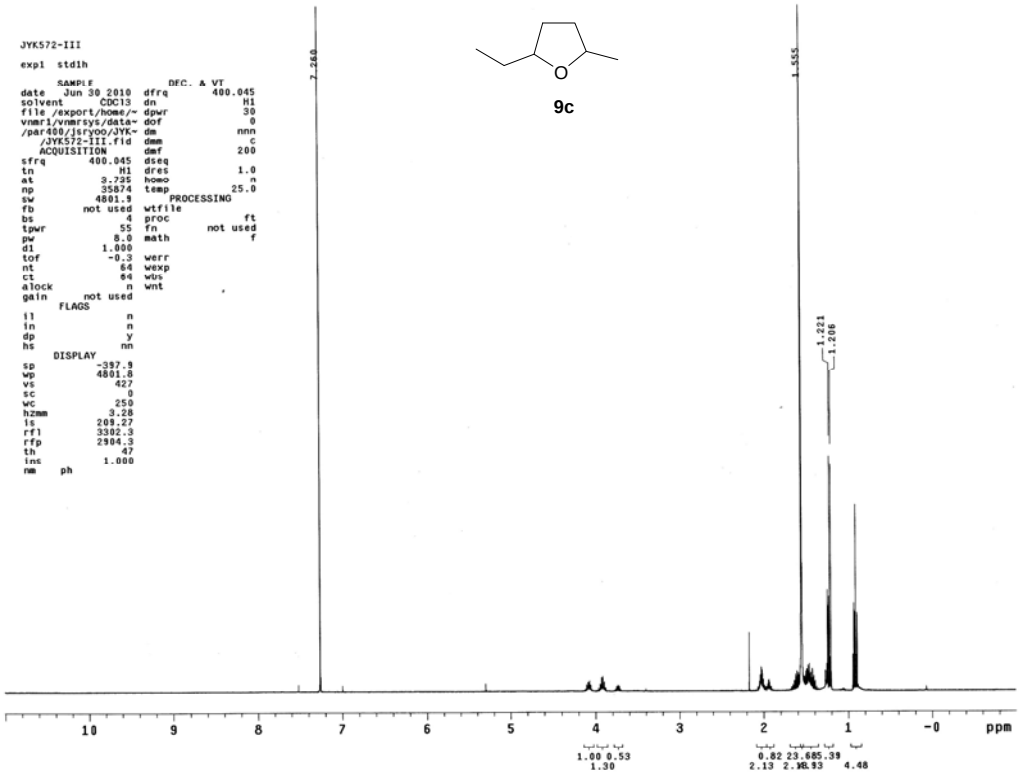

JYK569-111
exp2 std1h
SAMPLE
date Jun 22 2010
solvent CDCl3
file exp
ACQUISITION
sfrq 400.045
tn H1
at 3.731
np 35828
sw 4801.9
fb not used
bs 4
tpwr 55
pw 8.0
d1 1.000
tof -0.3
nt 32
ct 32
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -317.7
wp 4801.8
vs 96
sc 0
wc 250
hzmm 19.21
is 57.29
rf1 3302.2
rfp 2904.3
th 44
ins 1.000
nm ph
DEC. & VT
dfrq 400.045
dn H1
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
dseq
dres 1.0
homo n
temp 25.0
PROCESSING
wtfile
proc ft
fn not used
math f
werr
wexp
wbs
wnt
7.260
1.551
10 9 8 7 6 5 4 3 2 1 -0 ppm
1.00


JYK570-13C
exp5 std13c
SAMPLE
date Jun 25 2010
solvent CDCl3
file exp
ACQUISITION
sfrq 100.601
tn C13
at 0.911
np 48322
sw 26534.0
fb not used
bs 4
tpwr 53
pw 9.5
d1 2.000
tof 309.9
nt 2000
ct 680
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -963.7
wp 19001.7
vs 721
sc 0
wc 250
hzmm 76.01
is 365.65
rf1 11207.0
rfp 768.6
th 22
ins 1.000
al ph
DEC. & VT
dfrq 400.046
dn H1
dpwr 41
dof 1043.9
dm yyy
dmm w
dmf 10900
dseq
dres 1.0
homo n
temp 25.0
PROCESSING
lb 2.00
wtfile
proc ft
fn not used
math f
werr
wexp
wbs
wnt
77.544
77.330
76.908
160 140 120 100 80 60 40 20 0 ppm

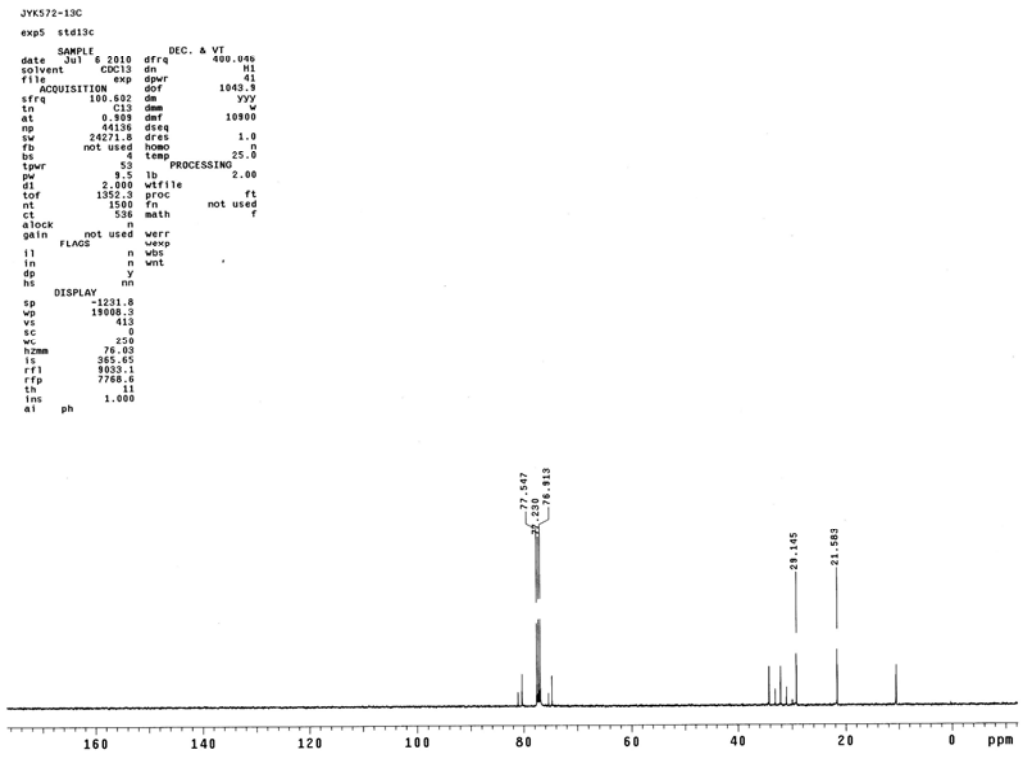

JYK577-100719-II
exp2 std1h
SAMPLE
date Jul 19 2010
solvent CDCl3
file exp
ACQUISITION
sfrq 400.045
tn H1
at 3.733
np 35852
sw 4801.8
fb not used
bs 4
tpwr 55
pw 8.0
d1 1.000
tof -0.3
nt 32
ct 32
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -387.7
wp 4801.8
vs 357
sc 0
wc 250
hzmm 19.21
is 179.90
rfl 387.9
rfp 0
th 37
ins 1.000
nm ph
DEC. & VT
dfrq 400.045
dn H1
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
dseq
dres 1.0
homo n
temp 25.0
PROCESSING
wtfile
proc ft
fn not used
math f
werr
wexp
wbs
wnt
7.260
1.541
1.100
0.000
10
9
8
7
6
5
4
3
2
1
-0
ppm
0.26 0.51
0.33
1.05
0.47
1.00
1.02
1.13
2.88 87
4.46 3.16

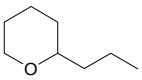
9b


JYK577
exp997 std13c
SAMPLE
date Jul 27 2010
solvent CDCl3
file /export/home/~
vnmr1/vnmrsys/data~
/par400/jsryoo/JYK~
/JYK577-13C.fid
ACQUISITION
sfrq 100.601
tn C13
at 0.911
np 43792
sw 24038.5
fb not used
bs 4
tpwr 53
pw 9.5
d1 2.000
tof 641.5
nt 1500
ct 728
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -1100.5
wp 19331.0
vs 120
sc 0
wc 250
hzmm 77.32
is 45.71
rfl 9627.7
rfp 7768.6
th 2
ins 1.000
ai ph
DEC. & VT
dfrq 400.046
dn H1
dpwr 41
dof 1043.8
dm yyy
dmm W
dmf 10900
dseq
dres 1.0
homo n
temp 25.9
PROCESSING
lb 2.00
wtfile
proc ft
fn not used
math f
werr
wexp
wbs
wnt
80.366
77.544
77.230
76.909
75.545
48.919
40.168
27.597
26.880
21.826
160
140
120
100
80
60
40
20
0
ppm


JYK572-III
exp1 std1h
SAMPLE
date Jun 30 2010
solvent CDCl3
file /export/home/~
vnmr1/vnmrsys/data~
/par400/jsryoo/JYK~
/JYK572-III.fid
ACQUISITION
sfrq 400.045
tn H1
at 3.735
np 35874
sw 4801.9
fb not used
bs 4
tpwr 55
pw 8.0
d1 1.000
tof -0.3
nt 64
ct 64
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -397.9
wp 4801.8
vs 427
sc 0
wc 250
hzmm 3.28
is 209.27
rf1 3302.3
rfp 2904.3
th 47
ins 1.000
nm ph
DEC. & VT
dfrq 400.045
dn H1
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
dseq
dres 1.0
homo n
temp 25.0
PROCESSING
wtfile
proc ft
fn not used
math f
werr
wexp
wbs
wnt
7.260
1.555
1.221
1.206
10 9 8 7 6 5 4 3 2 1 -0 ppm
1.00 0.53
1.30
0.82 23.685.39
2.13 2.93 4.48

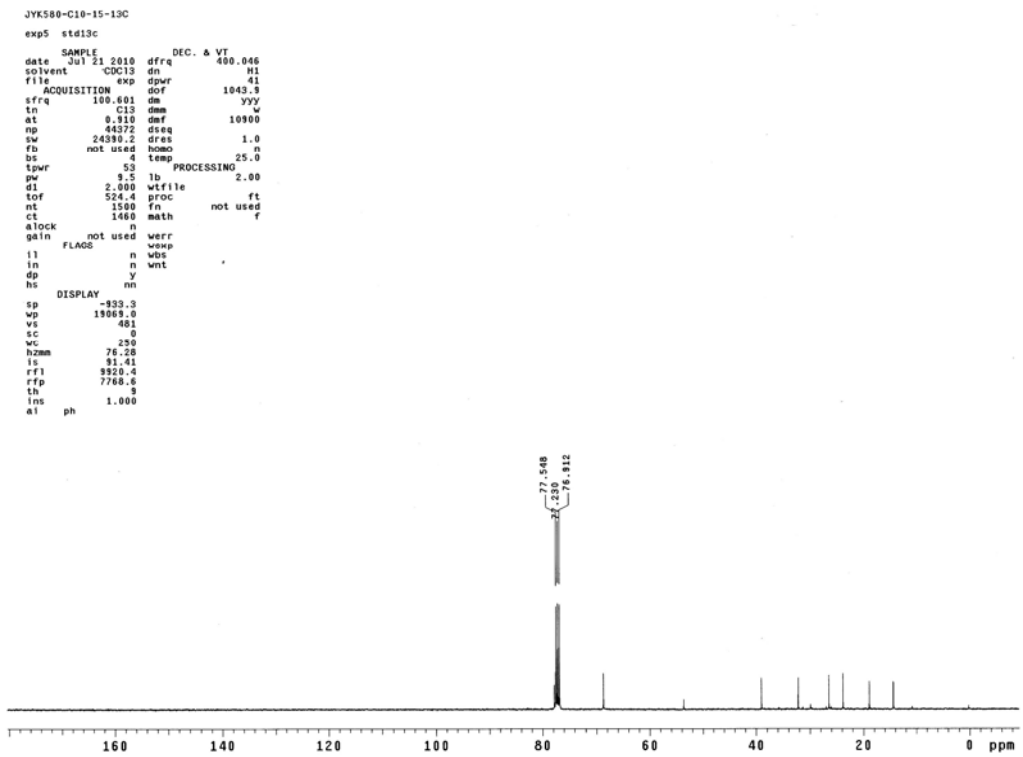
9c


JYK572-13C
exp5 std13c
SAMPLE
date Jul 6 2010
solvent CDCl3
file exp
ACQUISITION
sfrq 100.602
tn C13
at 0.909
np 44136
sw 24271.8
fb not used
bs 4
tpwr 53
pw 9.5
d1 2.000
tof 1352.3
nt 1500
ct 536
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -1231.8
wp 19008.3
vs 413
sc 0
wc 250
hzmm 76.03
is 365.65
rf1 9033.1
rfp 7768.6
th 11
ins 1.000
al ph
DEC. & VT
dfrq 400.046
dn H1
dpwr 41
dof 1043.9
dm yyy
dmm w
dmf 10900
dseq
dres 1.0
homo n
temp 25.0
PROCESSING
lb 2.00
wtfile
proc ft
fn not used
math f
werr
wexp
wbs
wnt
77.547
77.230
76.913
29.145
21.583
160 140 120 100 80 60 40 20 0 ppm

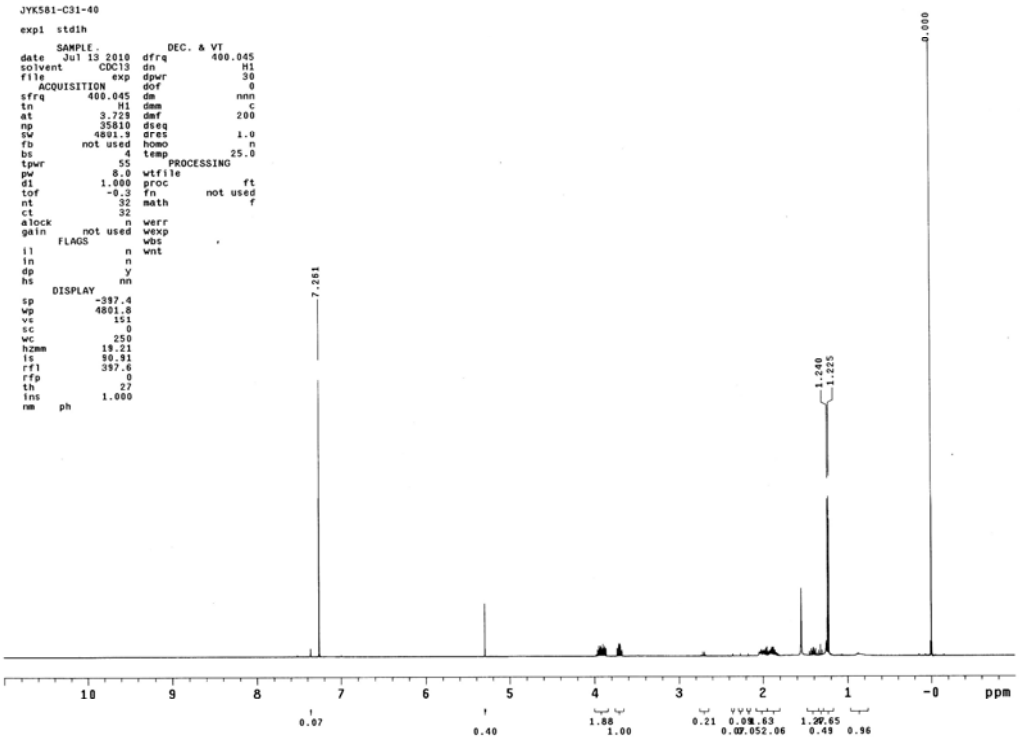


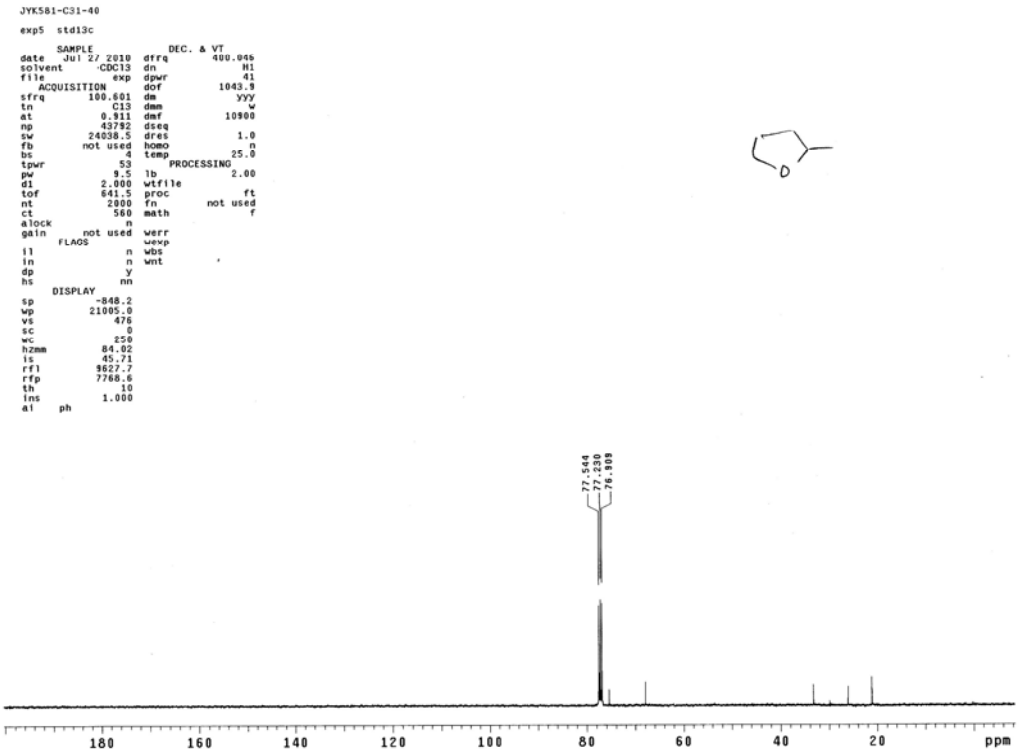
9e

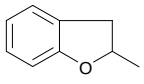
JYK580-C10-15
exp1 std1h
SAMPLE
date Jul 9 2010
solvent CDCl3
file exp
ACQUISITION
sfrq 400.045
tn H1
at 3.737
np 35892
sw 4801.8
fb not used
bs 4
tpwr 55
pw 8.0
d1 1.000
tof -0.3
nt 16
ct 16
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -397.7
wp 4801.8
vs 188
sc 0
wc 250
hzmm 0.18
is 4.88
rf1 397.9
rfp 0
th 23
ins 1.000
nm ph
DEC. & VT
dfrq 400.045
dn H1
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
dseq
dres 1.0
homo n
temp 25.0
PROCESSING
wtfile
proc ft
fn not used
math f
werr
wexp
wbs
wnt
7.260
1.541
0.905
0.000
10
9
8
7
6
5
4
3
2
1
-0
ppm
0.11
1.00
1.11
0.95
1.15
4.31
1.52
1.72
2.52
3.31


JYK580-C10-15-13C
exp5 std13c
SAMPLE
date Jul 21 2010
solvent CDCl3
file exp
ACQUISITION
sfrq 100.601
tn C13
at 0.910
np 44372
sw 24380.2
fb not used
bs 4
tpwr 53
pw 9.5
d1 2.000
tof 524.4
nt 1500
ct 1460
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -933.3
wp 19069.0
vs 481
sc 0
wc 250
hzmm 76.28
is 91.41
rf1 9920.4
rfp 7760.6
th 9
ins 1.000
al ph
DEC. & VT
dfrq 400.046
dn H1
dpwr 41
dof 1043.9
dm yyy
dmm w
dmf 10900
dseq
dres 1.0
homo n
temp 25.0
PROCESSING
lb 2.00
wtfile
proc ft
fn not used
math f
werr
wexp
wbs
wnt
77.548
77.230
76.912
160
140
120
100
80
60
40
20
0
ppm

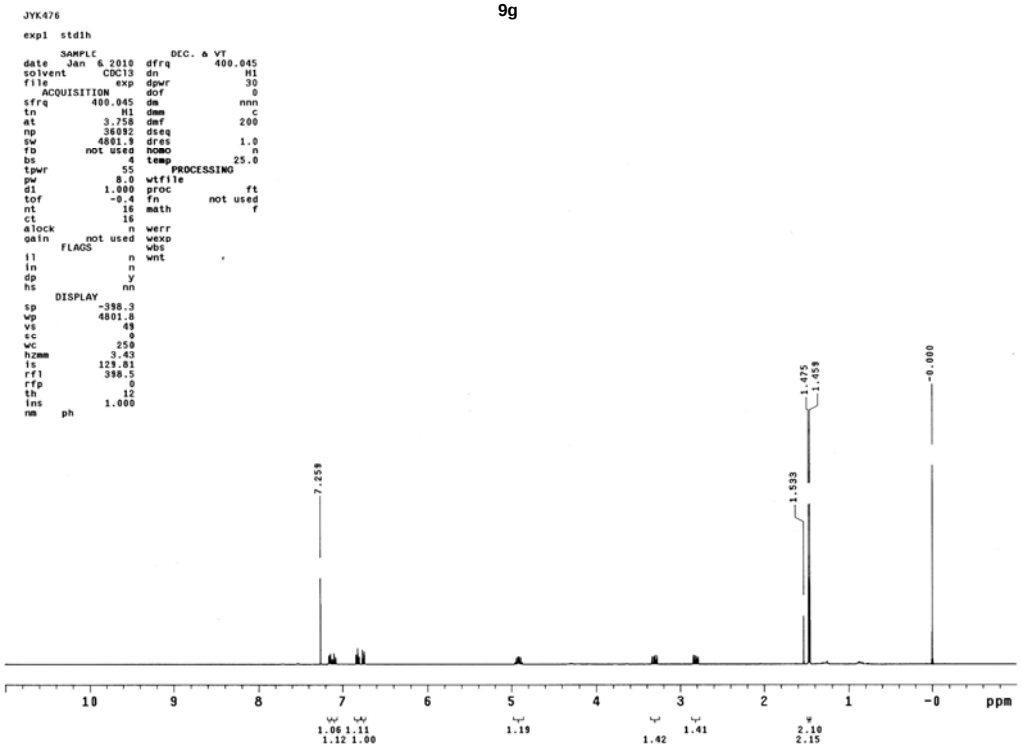
9f

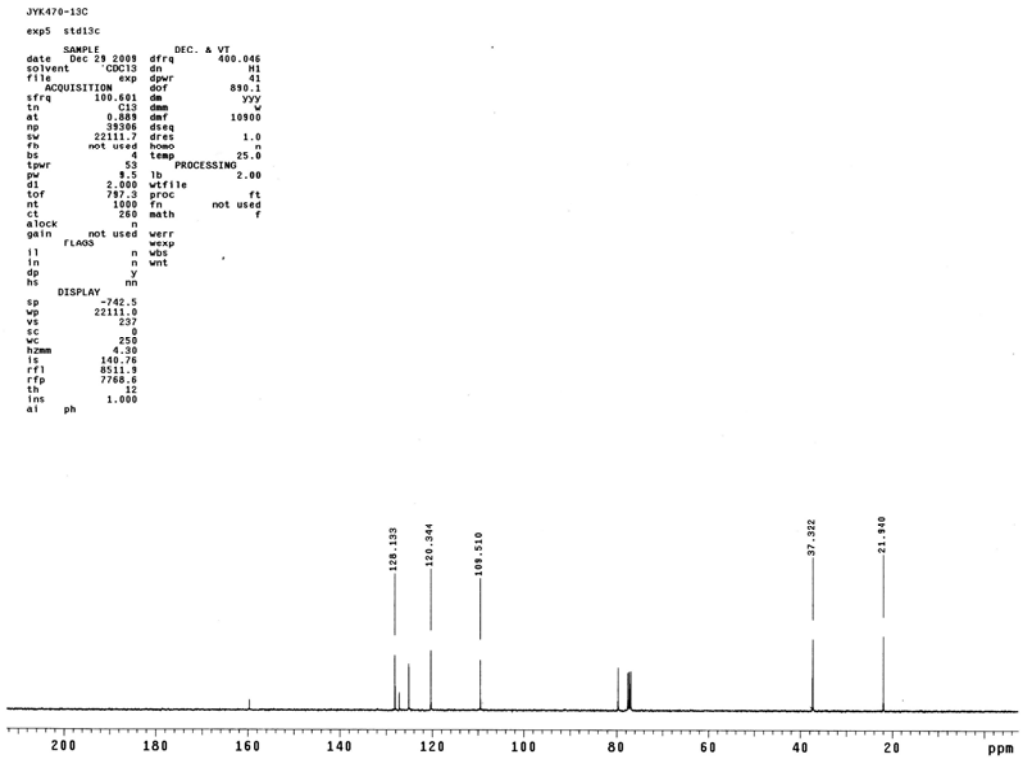

JYK581-C31-40
exp1 std1h
SAMPLE
date Jul 13 2010
solvent CDCl3
file exp
ACQUISITION
sfrq 400.045
tn H1
at 3.729
np 35810
sw 4801.9
fb not used
bs 4
tpwr 55
pw 8.0
d1 1.000
tof -0.3
nt 32
ct 32
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -397.4
wp 4801.8
vs 151
sc 0
wc 250
hzmm 19.21
is 90.91
rfl 397.6
rfp 0
th 27
ins 1.000
nm ph
DEC. & VT
dfrq 400.045
dn H1
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
dseq
dres 1.0
homo n
temp 25.0
PROCESSING
wtfile
proc ft
fn not used
math f
werr
wexp
wbs
wnt
7.261
1.240
1.225
0.000
10
9
8
7
6
5
4
3
2
1
-0
ppm
0.07
0.40
1.88
1.00
0.21
0.05
1.63
2.06
1.29
0.49
2.65
0.96

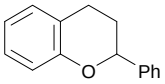

JYK581-C31-40
exp5 std13c
SAMPLE
date Jul 27 2010
solvent CDCl3
file exp
ACQUISITION
sfrq 100.601
tn C13
at 0.911
np 43782
sw 24038.5
fb not used
bs 4
tpwr 53
pw 9.5
d1 2.000
tof 641.5
nt 2000
ct 560
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -848.2
wp 21005.0
vs 476
sc 0
wc 250
hzmm 84.02
is 45.71
rfl 9627.7
rfp 7768.6
th 10
ins 1.000
al ph
DEC. & VT
dfrq 400.046
dn H1
dpwr 41
dof 1043.9
dm yyy
dmm w
dmf 10900
dseq
dres 1.0
homo n
temp 25.0
PROCESSING
lb 2.00
wtfile
proc ft
fn not used
math f
werr
wexp
wbs
wnt
77.544
77.230
76.909
180
160
140
120
100
80
60
40
20
ppm



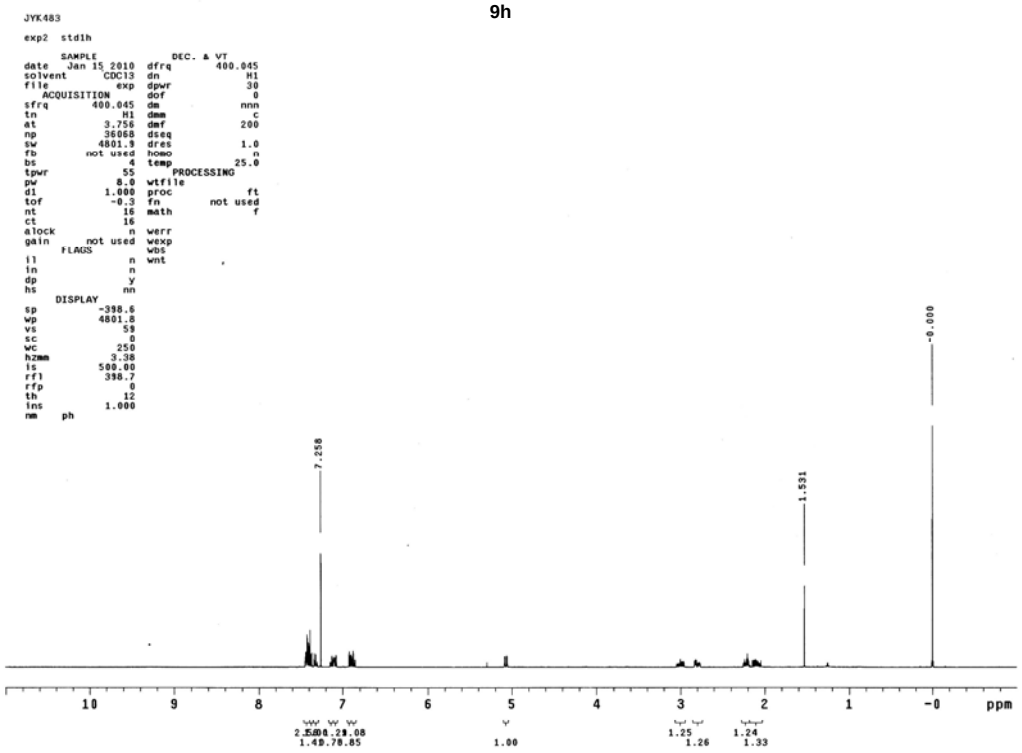
9g

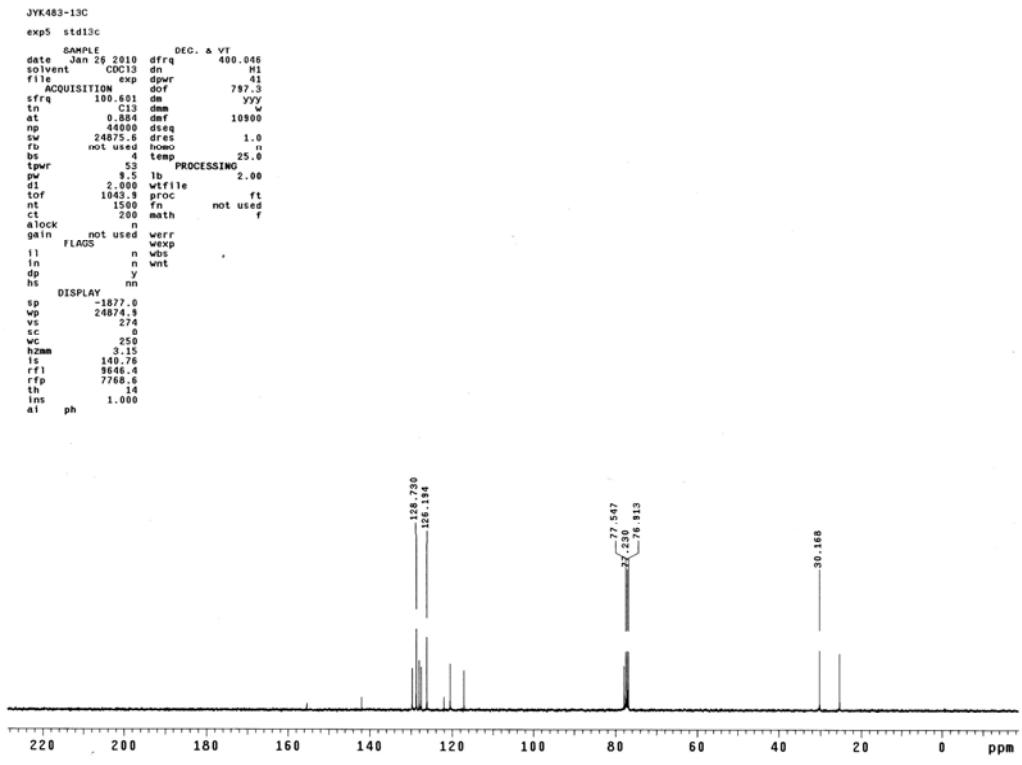

JYK476
exp1 std1h
SAMPLE
date Jan 6 2010
solvent CDCl3
file exp
ACQUISITION
sfrq 400.045
tn H1
at 3.758
np 36092
sw 4801.9
fb not used
bs 4
tpwr 55
pw 8.0
d1 1.000
tof -0.4
nt 16
ct 16
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -398.3
wp 4801.8
vs 49
sc 0
wc 250
hzmm 3.43
is 129.81
rfl 398.5
rfp 0
th 12
ins 1.000
nm ph
DEC. & VT
dfrq 400.045
dn H1
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
dseq
dres 1.0
homo n
temp 25.0
PROCESSING
wtfile
proc ft
fn not used
math f
werr
wexp
wbs
wnt
7.259
1.533
1.475
1.459
0.000
10
9
8
7
6
5
4
3
2
1
-0
ppm
1.06 1.11
1.12 1.00
1.19
1.42
1.41
2.10
2.15

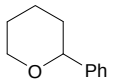

JYK470-13C
exp5 std13c
SAMPLE
date Dec 29 2009
solvent CDCl3
file exp
ACQUISITION
sfrq 100.601
tn C13
at 0.889
np 39306
sw 22111.7
fb not used
bs 4
tpwr 53
pw 9.5
d1 2.000
tof 797.3
nt 1000
ct 260
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -742.5
wp 22111.0
vs 237
sc 0
wc 250
hzmm 4.30
is 140.76
rfl 8511.9
rfp 7768.6
th 12
ins 1.000
ai ph
DEC. & VT
dfrq 400.046
dn H1
dpwr 41
dof 890.1
dm yyy
dmm w
dmf 10900
dseq
dres 1.0
homo n
temp 25.0
PROCESSING
lb 2.00
wtfile
proc ft
fn not used
math f
wbs
wnt
128.133
120.344
109.510
37.322
21.840
200
180
160
140
120
100
80
60
40
20
ppm


O
Ph

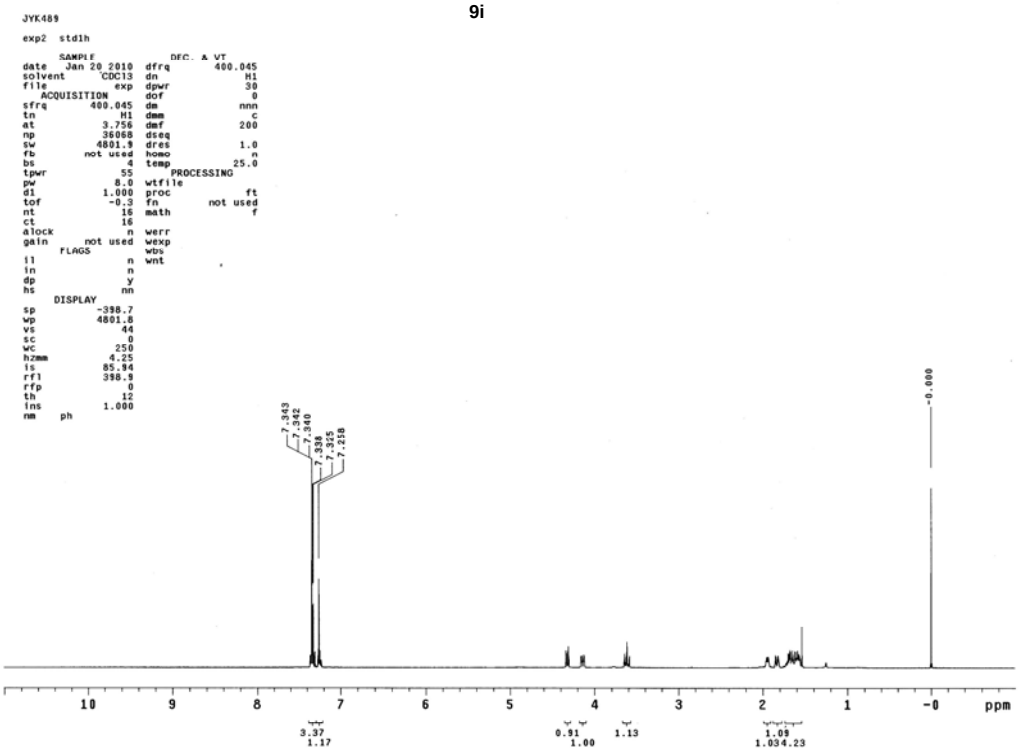
9h

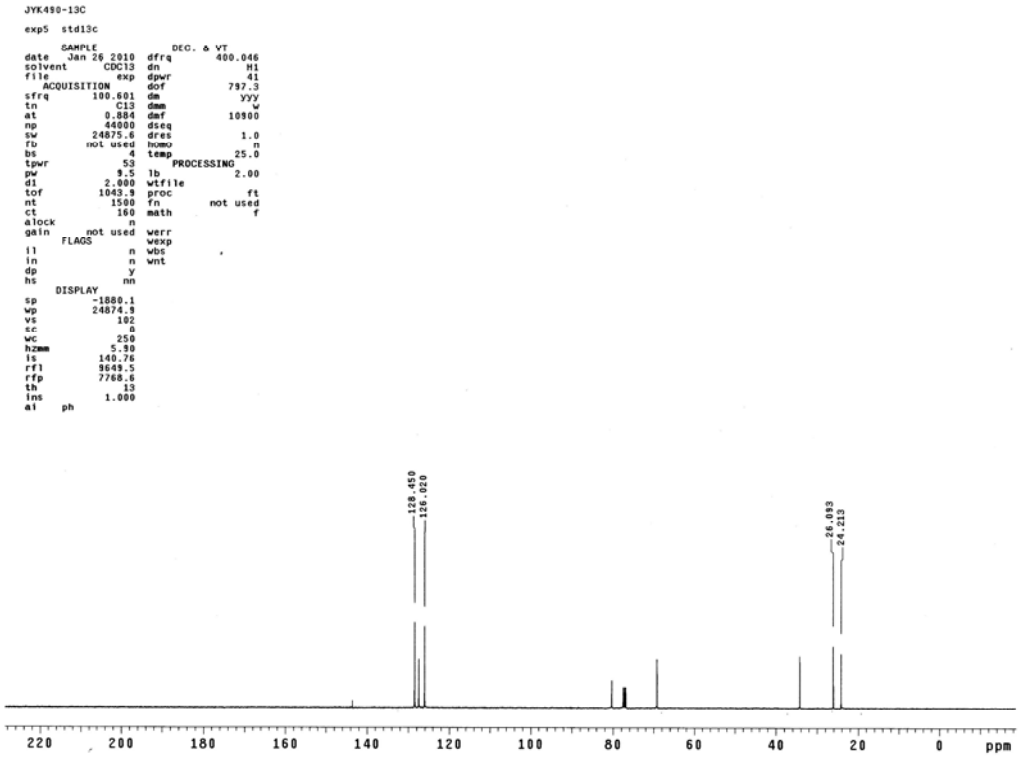

JYK483
exp2 std1h
SAMPLE
date Jan 15 2010
solvent CDCl3
file exp
ACQUISITION
sfrq 400.045
tn H1
at 3.756
np 36068
sw 4801.9
fb not used
bs 4
tpwr 55
pw 8.0
d1 1.000
tof -0.3
nt 16
ct 16
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -398.6
wp 4801.8
vs 59
sc 0
wc 250
hzmm 3.38
is 500.00
rfl 398.7
rfp 0
th 12
ins 1.000
nm ph
DEC. & VT
dfrq 400.045
dn H1
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
dseq
dres 1.0
homo n
temp 25.0
PROCESSING
wtfile
proc ft
fn not used
math f
werr
wexp
wbs
wnt
7.258
1.531
0.000
10
9
8
7
6
5
4
3
2
1
-0
ppm
2.50
0.21
0.08
1.49
0.78
0.85
1.00
1.25
1.26
1.24
1.33


JYK483-13C
exp5 std13c
SAMPLE
date Jan 26 2010
solvent CDCl3
file exp
ACQUISITION
sfrq 100.601
tn C13
at 0.884
np 44000
sw 24875.6
fb not used
bs 4
tpwr 53
pw 9.5
d1 2.000
tof 1043.9
nt 1500
ct 200
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -1877.0
wp 24874.9
vs 274
sc 0
wc 250
hzmm 3.15
is 140.76
rfl 9646.4
rfp 7768.6
th 14
ins 1.000
ai ph
DEC. & VT
dfrq 400.046
dn H1
dpwr 41
dof 797.3
dm yyy
dmm W
dmf 10900
dseq
dres 1.0
homo n
temp 25.0
PROCESSING
lb 2.00
wtfile
proc ft
fn not used
math f
werr
wexp
wbs
wnt
128.730
126.194
77.547
77.230
76.913
30.168
220
200
180
160
140
120
100
80
60
40
20
0
ppm


O
Ph

9i


JYK489
exp2 std1h
SAMPLE
date Jan 20 2010
solvent CDCl3
file exp
ACQUISITION
sfrq 400.045
tn H1
at 3.756
np 36068
sw 4801.9
fb not used
bs 4
tpwr 55
pw 8.0
d1 1.000
tof -0.3
nt 16
ct 16
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -398.7
wp 4801.8
vs 44
sc 0
wc 250
hzmm 4.25
is 85.94
rfl 398.9
rfp 0
th 12
ins 1.000
nm ph
DEC. & VT
dfrq 400.045
dn H1
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
dseq
dres 1.0
homo n
temp 25.0
PROCESSING
wtfile
proc ft
fn not used
math f
werr
wexp
wbs
wnt
7.343
7.342
7.340
7.338
7.325
7.218
-0.000
10
9
8
7
6
5
4
3
2
1
-0
ppm
3.37
1.17
0.91
1.00
1.13
1.09
1.034.23


JYK490-13C
exp5 std13c
SAMPLE
date Jan 26 2010
solvent CDCl3
file exp
ACQUISITION
sfrq 100.601
tn C13
at 0.884
np 44000
sw 24875.6
fb not used
bs 4
tpwr 53
pw 9.5
d1 2.000
tof 1043.9
nt 1500
ct 160
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -1880.1
wp 24874.9
vs 102
sc 0
wc 250
hzmm 5.90
is 140.76
rfl 9649.5
rfp 7768.6
th 13
ins 1.000
ai ph
DEC. & VT
dfrq 400.046
dn H1
dpwr 41
dof 797.3
dm yyy
dmm w
dmf 10800
dseq
dres 1.0
homo n
temp 25.0
PROCESSING
lb 2.00
wtfile
proc ft
fn not used
math f
werr
wexp
wbs
wnt
128.450
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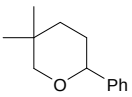
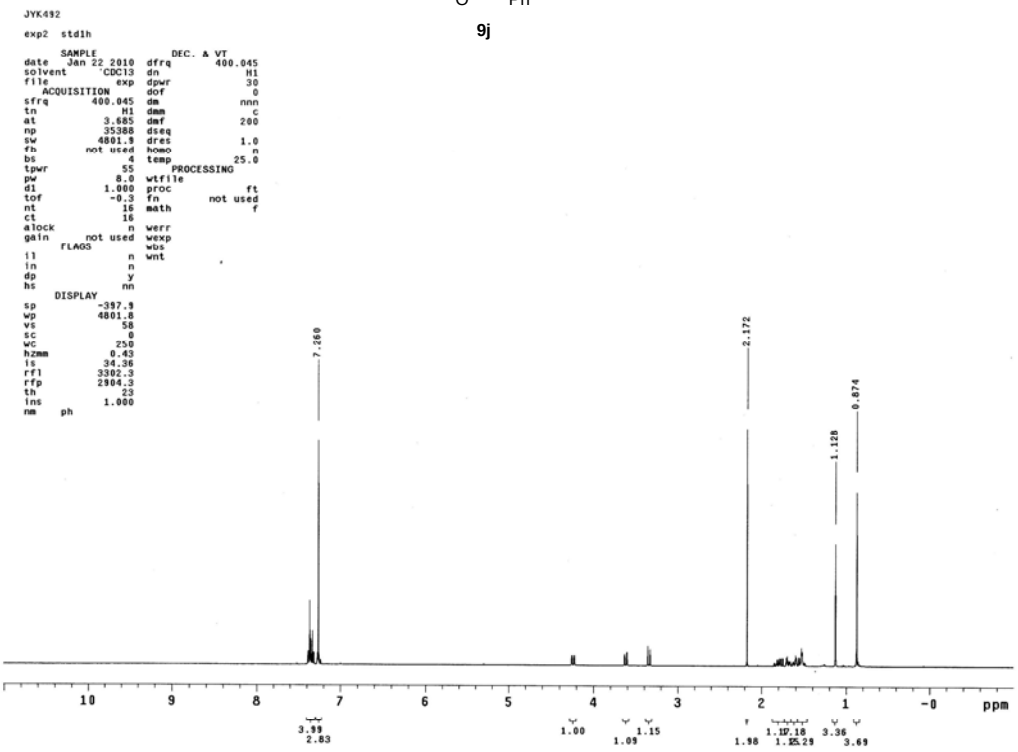
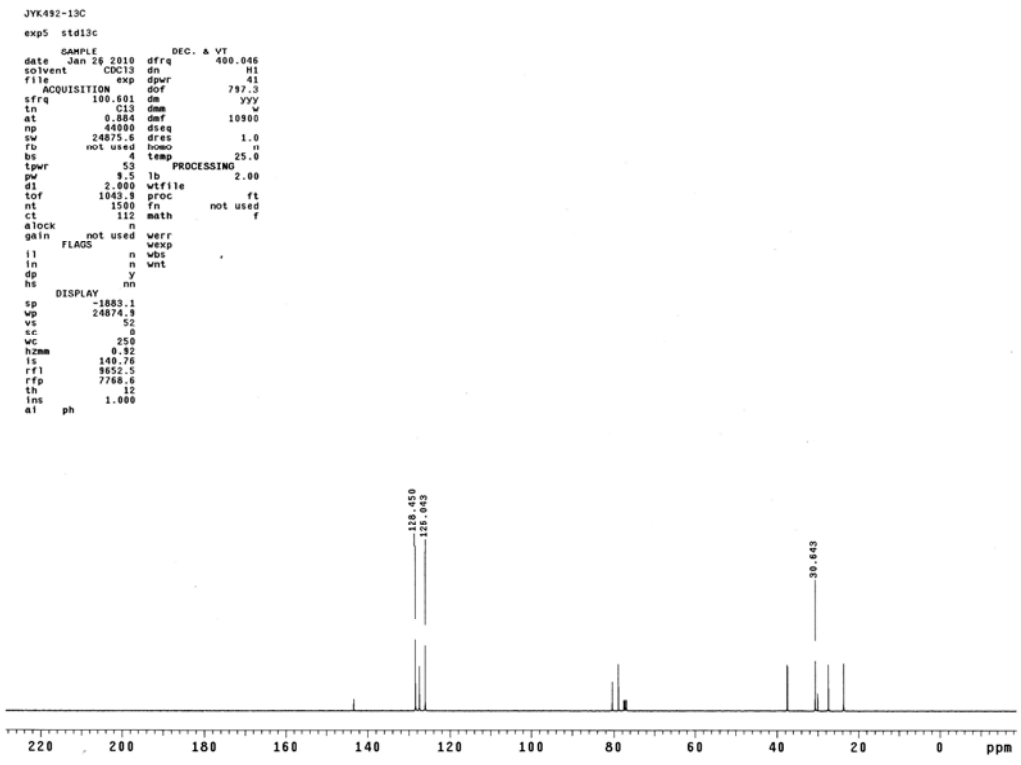
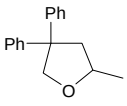
26.083
24.213
220
200
180
160
140
120
100
80
60
40
20
0
ppm


O
Ph

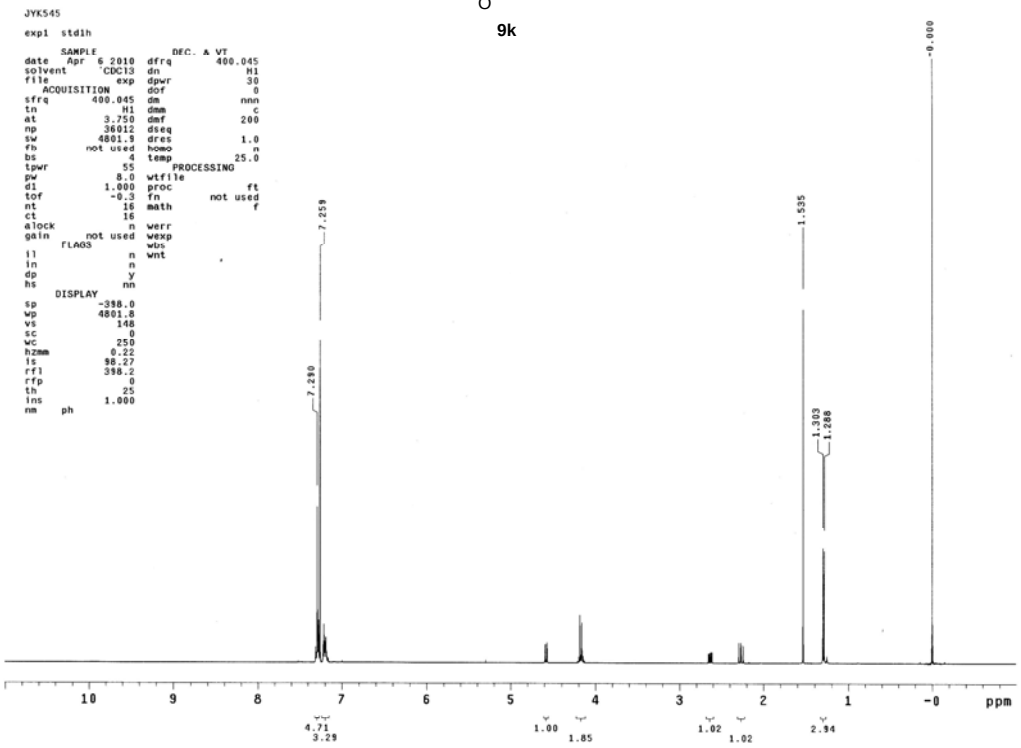
9j

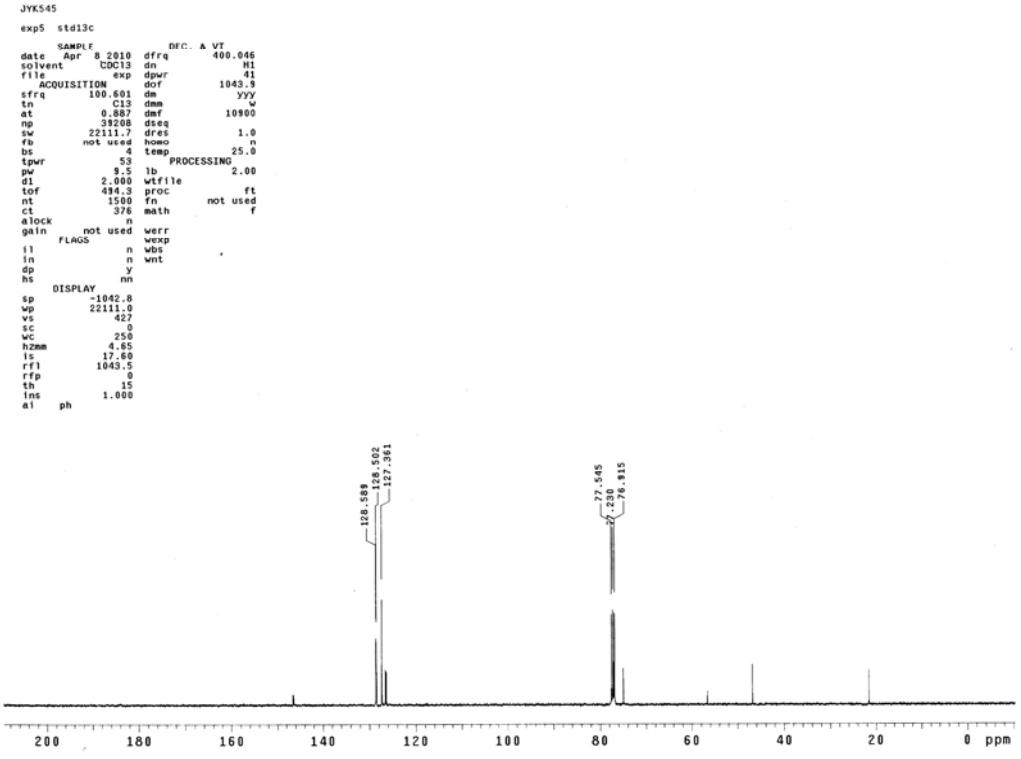

JYK492
exp2 std1h
SAMPLE
date Jan 22 2010
solvent CDCl3
file exp
ACQUISITION
sfrq 400.045
tn H1
at 3.685
np 35388
sw 4801.9
fb not used
bs 4
tpwr 55
pw 8.0
d1 1.000
tof -0.3
nt 16
ct 16
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -397.9
wp 4801.8
vs 58
sc 0
wc 250
hzmm 0.43
is 34.36
rfl 3302.3
rfp 2904.3
th 23
ins 1.000
nm ph
DEC. & VT
dfrq 400.045
dn H1
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
dseq
dres 1.0
homo n
temp 25.0
PROCESSING
wtfile
proc ft
fn not used
math f
werr
wexp
wbs
wnt
7.260
2.172
1.128
0.874
10
9
8
7
6
5
4
3
2
1
-0
ppm
3.99
2.83
1.00
1.15
1.09
1.98
3.36
3.69

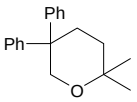

JYK492-13C
exp5 std13c
SAMPLE
date Jan 26 2010
solvent CDCl3
file exp
ACQUISITION
sfrq 100.601
tn C13
at 0.884
np 44000
sw 24875.6
fb not used
bs 4
tpwr 53
pw 9.5
d1 2.000
tof 1043.9
nt 1500
ct 112
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -1883.1
wp 24874.9
vs 52
sc 0
wc 250
hzmm 0.92
is 140.76
rfl 9652.5
rfp 7768.6
th 12
ins 1.000
al ph
DEC. & VT
dfrq 400.046
dn H1
dpwr 41
dof 797.3
dm yyy
dmm w
dmf 10900
dseq
dres 1.0
homo n
temp 25.0
PROCESSING
lb 2.00
wtfile
proc ft
fn not used
math f
werr
wexp
wbs
wnt
128.459
128.043
39.643
220
200
180
160
140
120
100
80
60
40
20
0
ppm


Ph
Ph
O

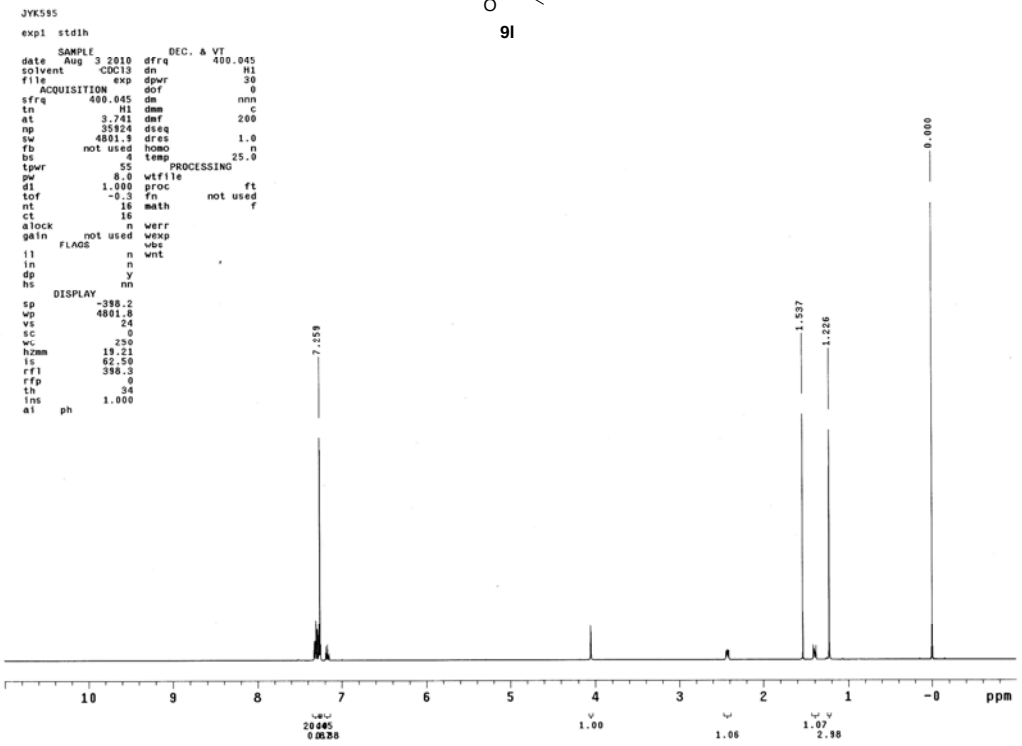
9k

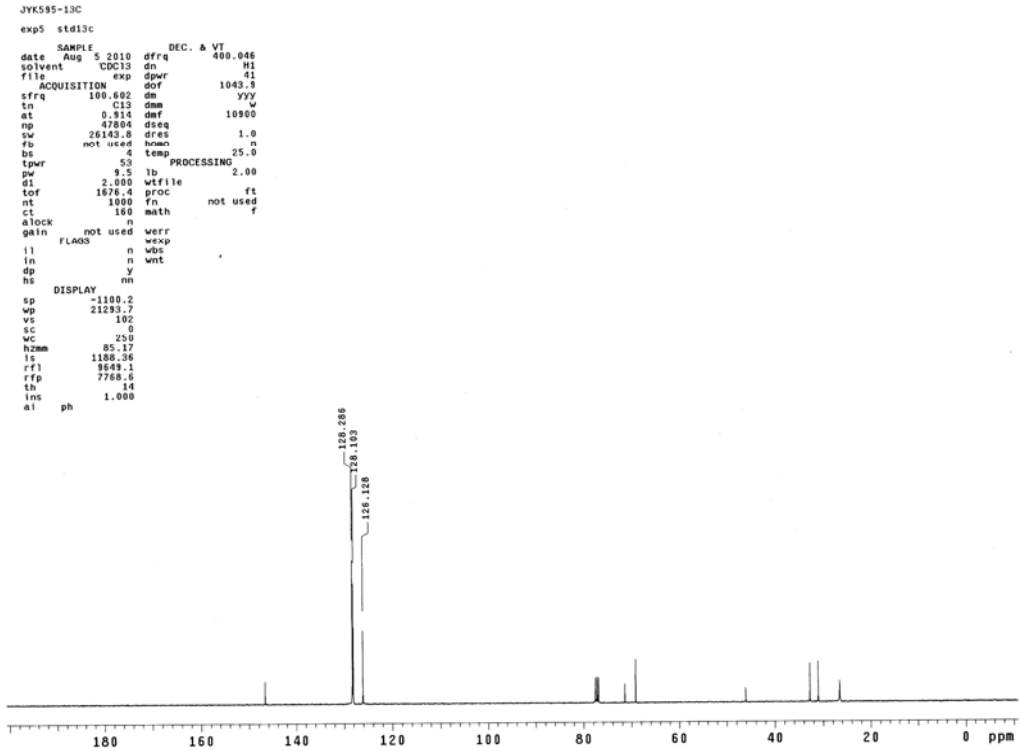

JYK545
exp1 std1h
SAMPLE
date Apr 6 2010
solvent CDCl3
file exp
ACQUISITION
sfrq 400.045
tn H1
at 3.750
np 36012
sw 4801.9
fb not used
bs 4
tpwr 55
pw 8.0
d1 1.000
tof -0.3
nt 16
ct 16
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -388.0
wp 4801.8
vs 148
sc 0
wc 250
hzmm 0.22
is 98.27
rfl 398.2
rfp 0
th 25
ins 1.000
nm ph
DEC. & VT
dfrq 400.045
dn H1
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
dseq
dres 1.0
homo n
temp 25.0
PROCESSING
wtfile
proc ft
fn not used
math f
werr
wexp
wbs
wnt
7.259
7.280
1.535
1.303
1.288
0.000
10
9
8
7
6
5
4
3
2
1
-0
ppm
4.71
3.29
1.00
1.85
1.02
1.02
2.94

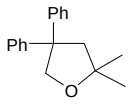

JYK545
exp5 std13c
SAMPLE
date Apr 8 2010
solvent CDCl3
file exp
ACQUISITION
sfrq 100.601
tn C13
at 0.887
np 39208
sw 22111.7
fb not used
bs 4
tpwr 53
pw 9.5
d1 2.000
tof 494.3
nt 1500
ct 376
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -1042.8
wp 22111.0
vs 427
sc 0
wc 250
hzmm 4.65
is 17.60
rfl 1043.5
rfp 0
th 15
ins 1.000
ai ph
DEC. & VT
dfrq 400.046
dn H1
dpwr 41
dof 1043.9
dm yyy
dmm W
dmf 10900
dseq
dres 1.0
homo n
temp 25.0
PROCESSING
lb 2.00
wtfile
proc ft
fn not used
math f
werr
wexp
wbs
wnt
128.589
128.502
127.361
77.545
77.230
76.915
200
180
160
140
120
100
80
60
40
20
0
ppm


Ph
Ph
O

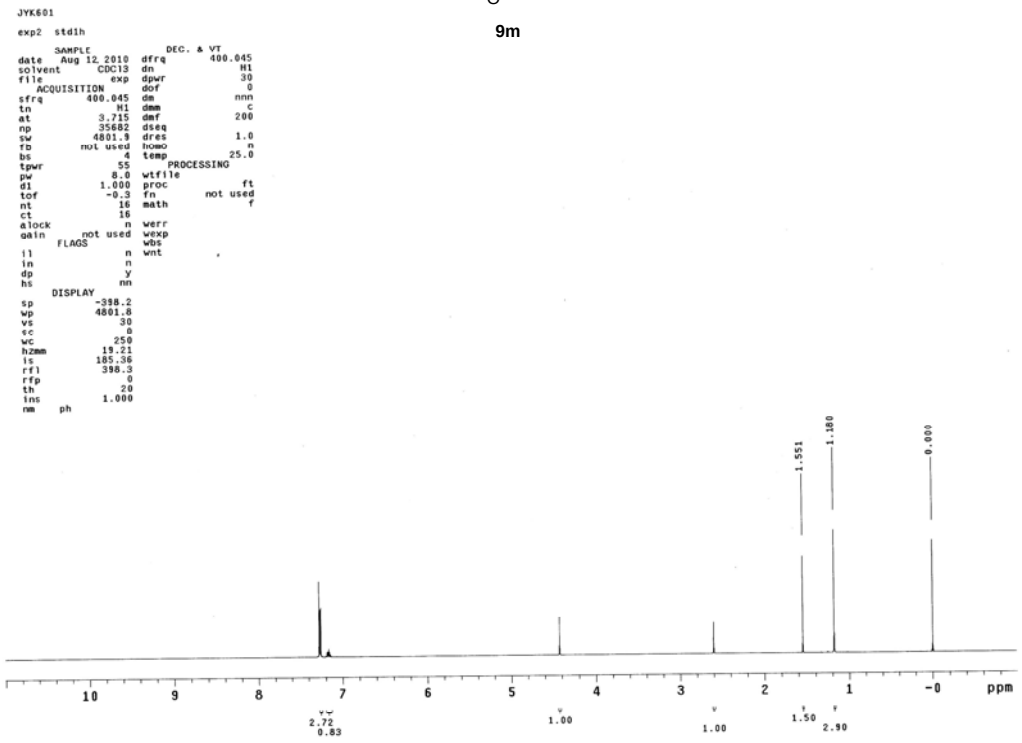
9l

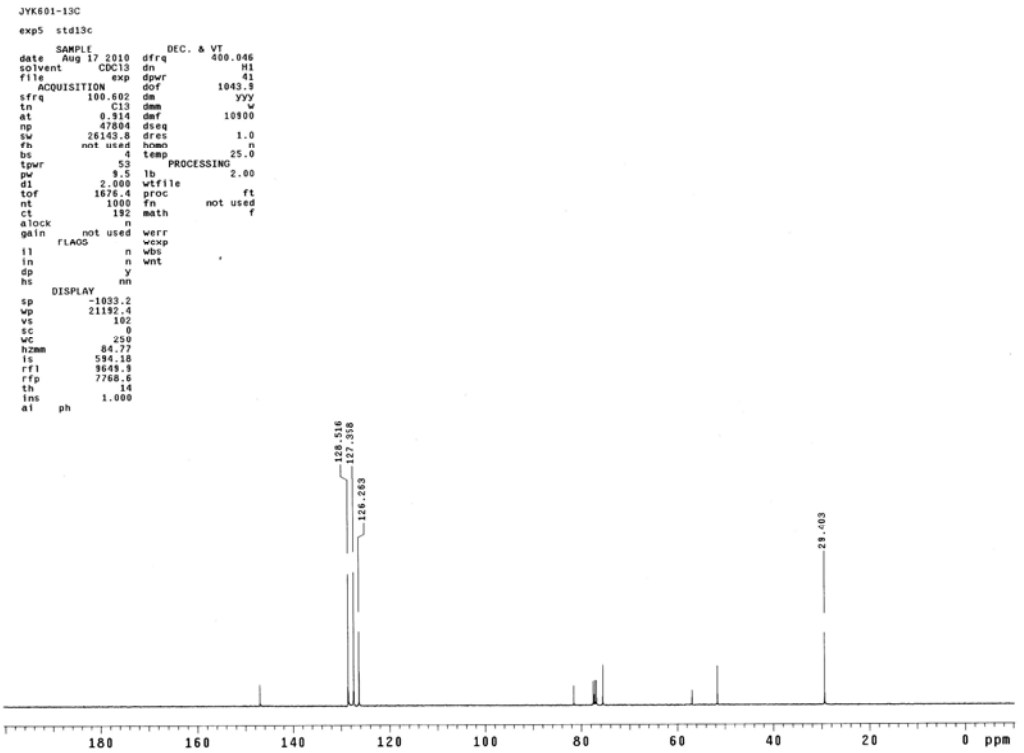

JYK595
exp1 std1h
SAMPLE
date Aug 3 2010
solvent CDCl3
file exp
ACQUISITION
sfrq 400.045
tn H1
at 3.741
np 35924
sw 4801.9
fb not used
bs 4
tpwr 55
pw 8.0
dl 1.000
tof -0.3
nt 16
ct 16
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -398.2
wp 4801.8
vs 24
sc 0
wc 250
hzmm 19.21
is 62.50
rfl 398.3
rfp 0
th 34
ins 1.000
ai ph
DEC. & VT
dfrq 400.045
dn H1
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
dseq
dres 1.0
homo n
temp 25.0
PROCESSING
wtfile
proc ft
fn not used
math f
werr
wexp
wbs
wnt
7.259
1.537
1.226
0.000
10
9
8
7
6
5
4
3
2
1
-0
ppm
1.00
1.06
1.07
2.98

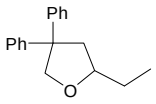

JYK595-13C
exp5 std13c
SAMPLE
date Aug 5 2010
solvent CDCl3
file exp
ACQUISITION
sfrq 100.602
tn C13
at 0.914
np 47804
sw 26143.8
fb not used
bs 4
tpwr 53
pw 9.5
dl 2.000
tof 1676.4
nt 1000
ct 160
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -1100.2
wp 21293.7
vs 102
sc 0
wc 250
hzmm 85.17
is 1188.36
rfl 9649.1
rfp 7768.6
th 14
ins 1.000
ai ph
DEC. & VT
dfrq 400.046
dn H1
dpwr 41
dof 1043.9
dm yyy
dmm w
dmf 10900
dseq
dres 1.0
homo n
temp 25.0
PROCESSING
lb 2.00
wtfile
proc ft
fn not used
math f
werr
wexp
wbs
wnt
128.286
128.193
126.128
180
160
140
120
100
80
60
40
20
0
ppm


Ph
Ph
O

9m

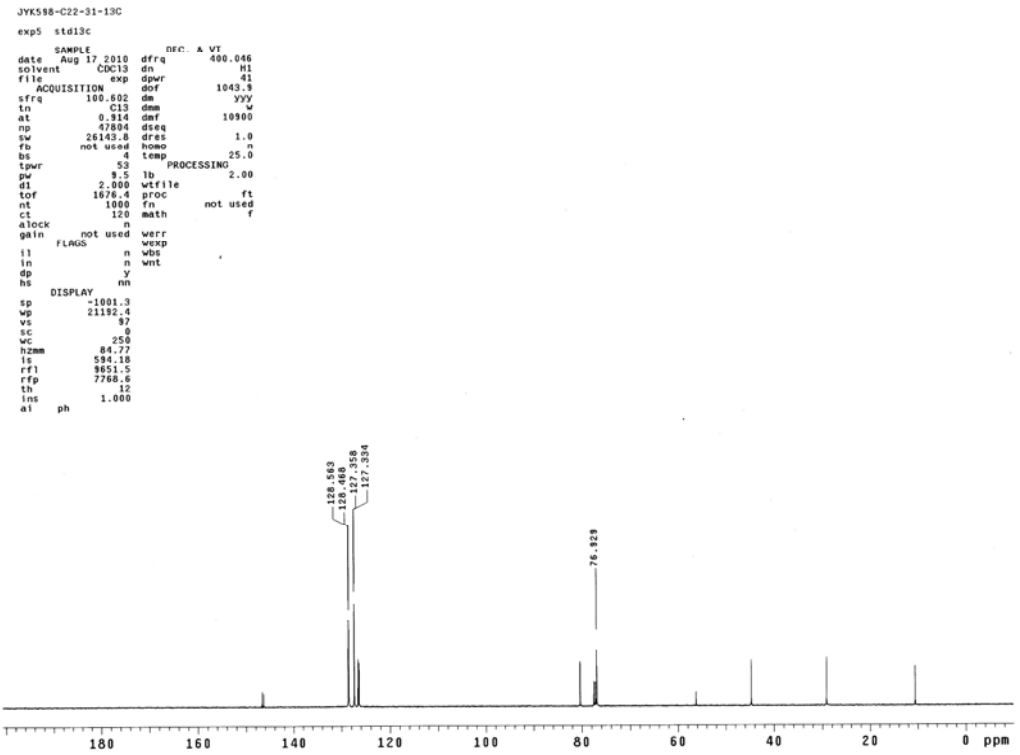

JYK601
exp2 std1h
SAMPLE
date Aug 12 2010
solvent CDCl3
file exp
ACQUISITION
sfrq 400.045
tn H1
at 3.715
np 35682
sw 4801.9
fb not used
bs 4
tpwr 55
pw 8.0
d1 1.000
tof -0.3
nt 16
ct 16
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -398.2
wp 4801.8
vs 30
sc 0
wc 250
hzmm 19.21
is 185.36
rfl 398.3
rfp 0
th 20
ins 1.000
nm ph
DEC. & VT
dfrq 400.045
dn H1
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
dseq
dres 1.0
homo n
temp 25.0
PROCESSING
wtfile
proc ft
fn not used
math f
werr
wexp
wbs
wnt
1.551
1.380
0.000
10
9
8
7
6
5
4
3
2
1
-0
ppm
2.72
0.83
1.00
1.00
1.50
2.90

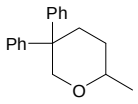

JYK601-13C
exp5 std13c
SAMPLE
date Aug 17 2010
solvent CDCl3
file exp
ACQUISITION
sfrq 100.602
tn C13
at 0.914
np 47804
sw 26143.8
fb not used
bs 4
tpwr 53
pw 9.5
d1 2.000
tof 1676.4
nt 1000
ct 192
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -1033.2
wp 21192.4
vs 102
sc 0
wc 250
hzmm 84.77
is 594.18
rfl 9649.9
rfp 7768.6
th 14
ins 1.000
al ph
DEC. & VT
dfrq 400.046
dn H1
dpwr 41
dof 1043.9
dm yyy
dmm w
dmf 10900
dseq
dres 1.0
homo n
temp 25.0
PROCESSING
lb 2.00
wtfile
proc ft
fn not used
math f
werr
wexp
wbs
wnt
128.516
127.358
126.263
28.403
180
160
140
120
100
80
60
40
20
0
ppm


Ph
Ph
O

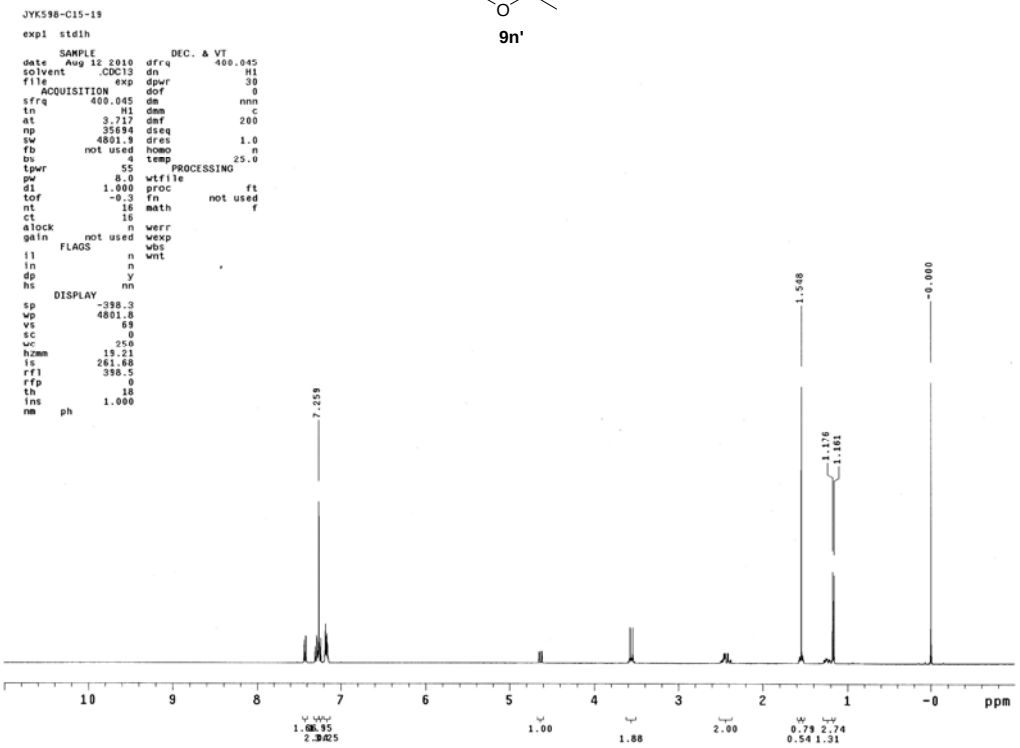
9n

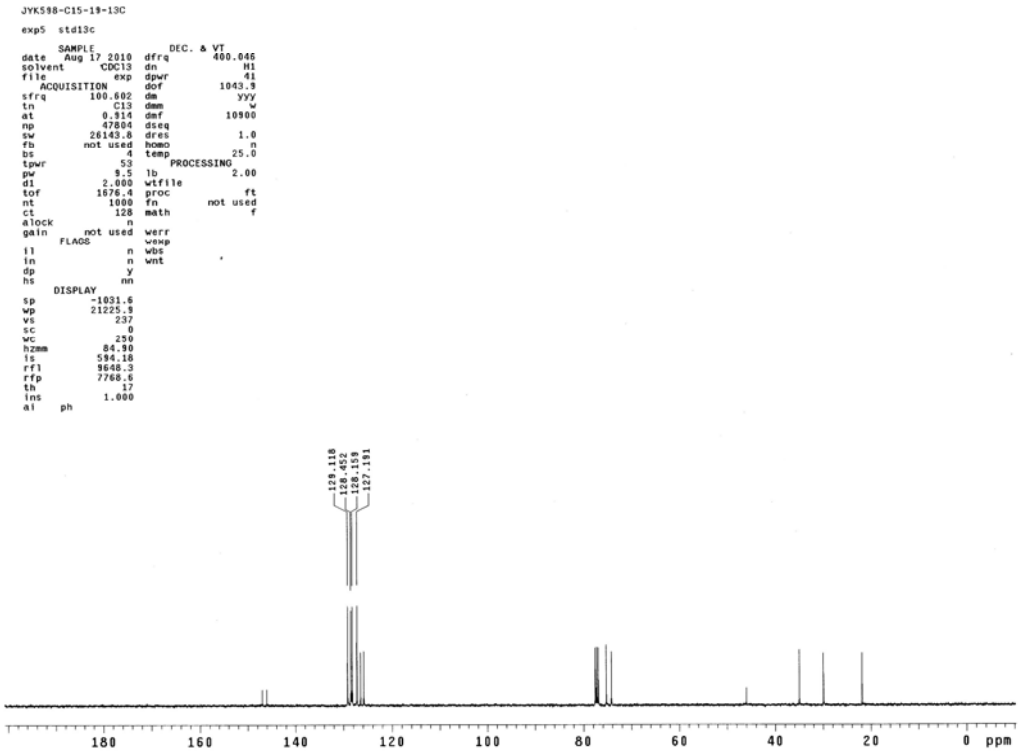
JYK598-C22-31
exp2 std1h
SAMPLE
date Aug 12 2010
solvent CDCl3
file exp
ACQUISITION
sfrq 400.045
tn H1
at 3.715
np 35682
sw 4801.9
fb not used
bs 4
tpwr 55
pw 8.0
d1 1.000
tof -0.3
nt 16
ct 16
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -398.6
wp 4801.8
vs 59
sc 0
wc 250
hzmm 19.21
is 65.42
rfl 398.7
rfp 0
th 19
ins 1.000
nm ph
DEC. & VT
dfrq 400.045
dn H1
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
dseq
dres 1.0
homo n
temp 25.0
PROCESSING
wtfile
proc ft
fn not used
math f
werr
wexp
wbs
wnt
7.258
1.553
0.934
-0.000
10
9
8
7
6
5
4
3
2
1
-0
ppm
4.62
3.26
0.98
0.91
0.95
1.00
1.01
0.50
1.04
0.75
2.73

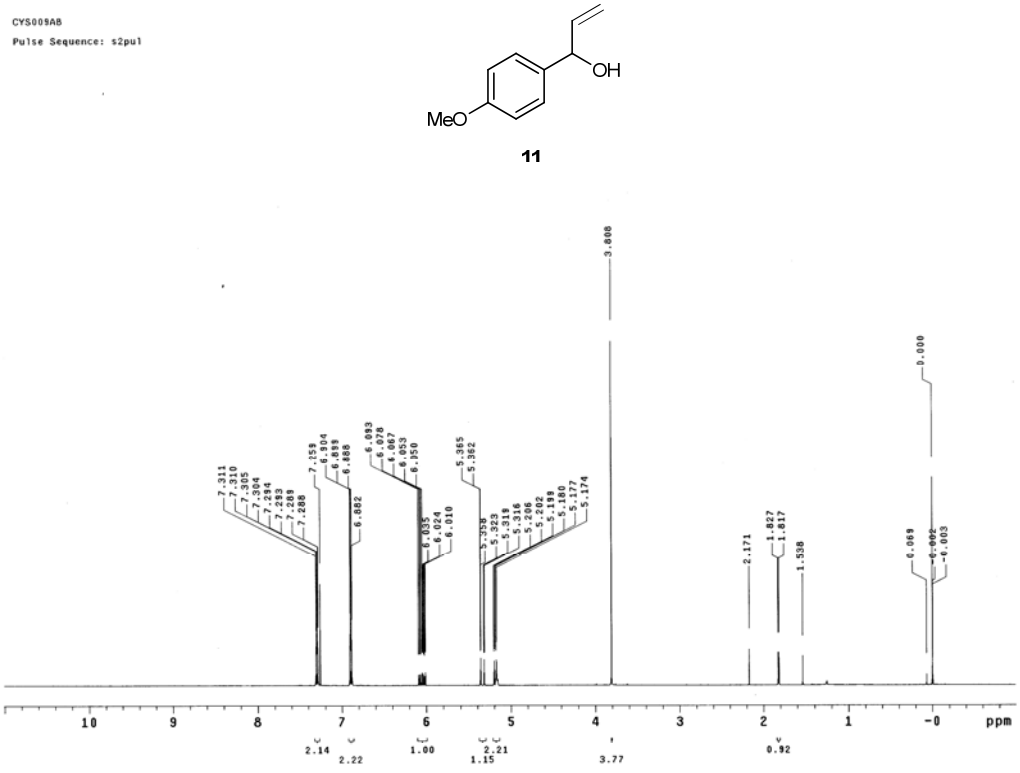

JYK598-C22-31-13C
exp5 std13c
SAMPLE
date Aug 17 2010
solvent CDCl3
file exp
ACQUISITION
sfrq 100.602
tn C13
at 0.914
np 47804
sw 26143.8
fb not used
bs 4
tpwr 53
pw 9.5
d1 2.000
tof 1676.4
nt 1000
ct 120
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -1001.3
wp 21192.4
vs 97
sc 0
wc 250
hzmm 84.77
is 594.18
rfl 9651.5
rfp 7768.6
th 12
ins 1.000
ai ph
DEC. & VT
dfrq 400.046
dn H1
dpwr 41
dof 1043.9
dm yyy
dmm w
dmf 10900
dseq
dres 1.0
homo n
temp 25.0
PROCESSING
lb 2.00
wtfile
proc ft
fn not used
math f
werr
wexp
wbs
wnt
126.563
128.048
127.358
127.394
76.929
180
160
140
120
100
80
60
40
20
0
ppm

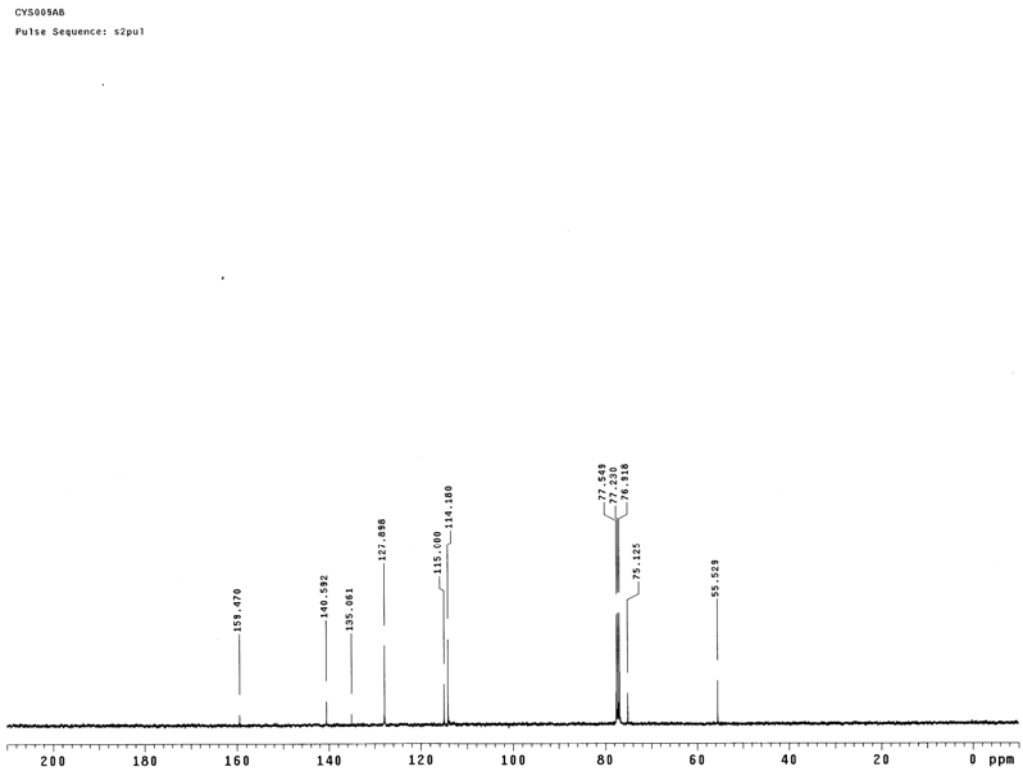

Ph
Ph
O

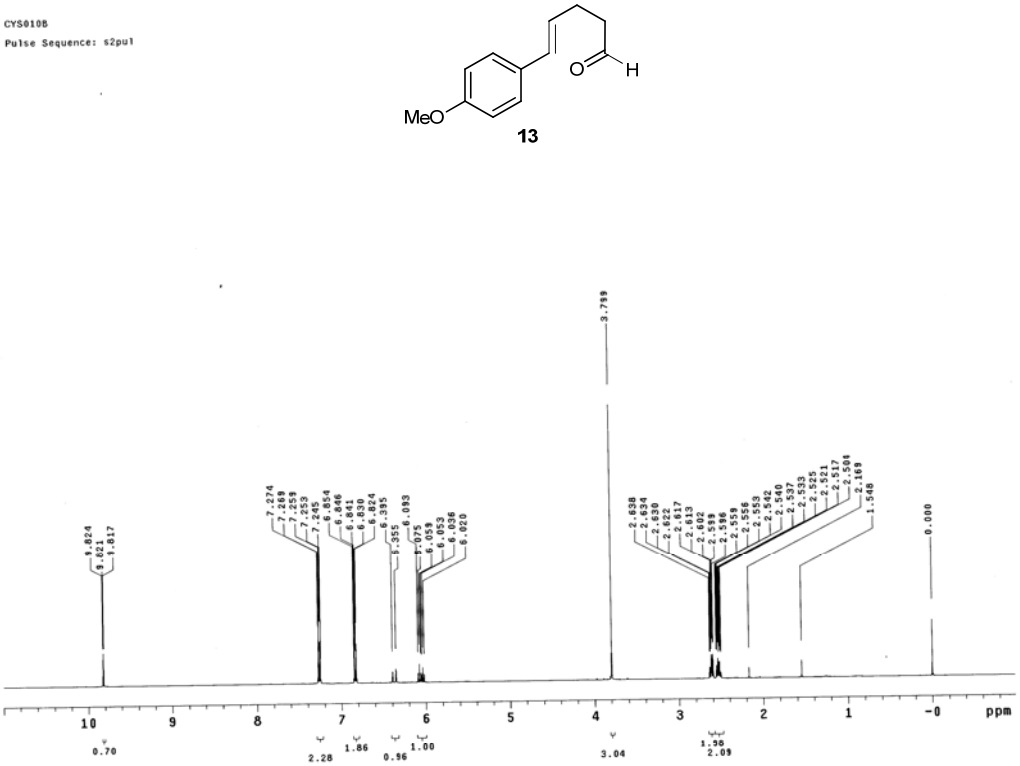
9n'

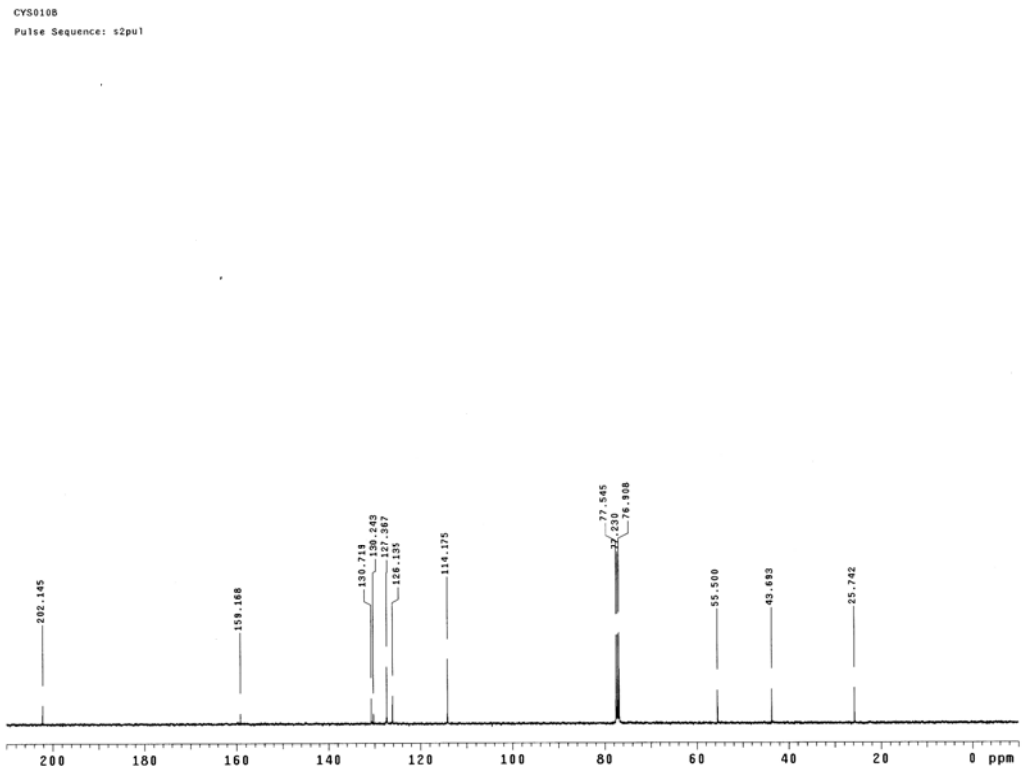

JYK598-C15-19
expl std1h
SAMPLE
date Aug 12 2010
solvent CDCl3
file exp
ACQUISITION
sfrq 400.045
tn H1
at 3.717
np 35694
sw 4801.9
fb not used
bs 4
tpwr 55
pw 8.0
dl 1.000
tof -0.3
nt 16
ct 16
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -398.3
wp 4801.8
vs 69
sc 0
wc 250
hzmm 19.21
is 261.68
rfl 398.5
rfp 0
th 18
ins 1.000
nm ph
DEC. & VT
dfrq 400.045
dn H1
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
dseq
dres 1.0
homo n
temp 25.0
PROCESSING
wtfile
proc ft
fn not used
math f
werr
wexp
wbs
wnt
7.259
1.548
1.176
1.161
0.000
10
9
8
7
6
5
4
3
2
1
-0
ppm
1.00
1.88
2.00
0.54
1.31

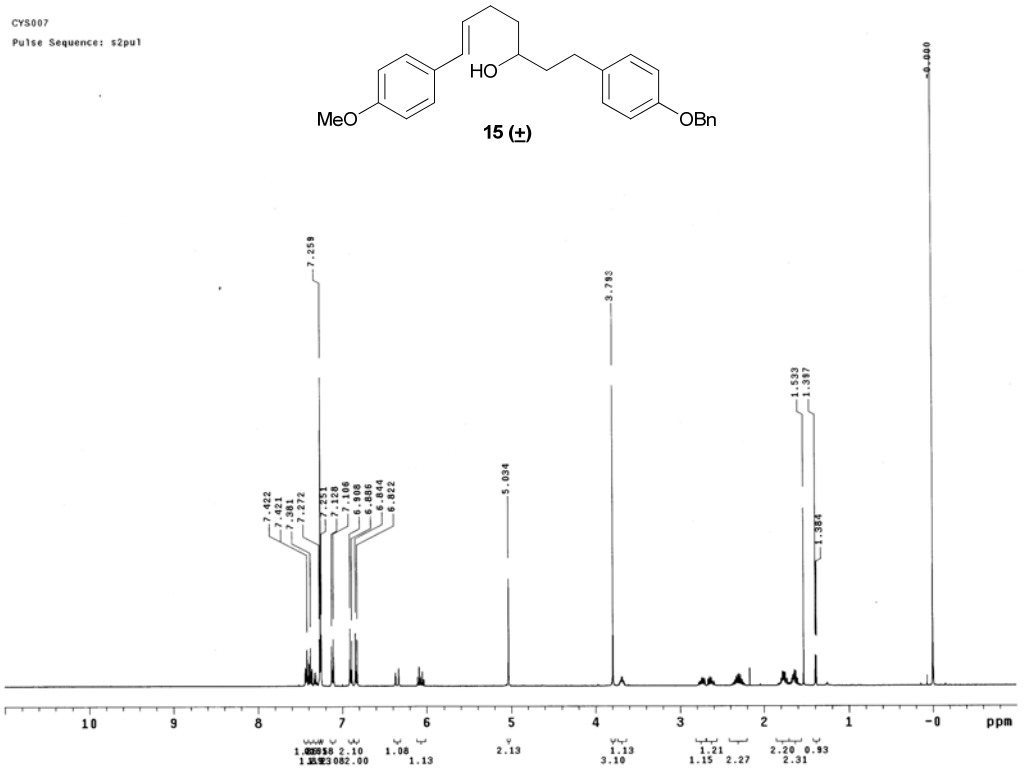

JYK598-C15-19-13C
exp5 std13c
SAMPLE
date Aug 17 2010
solvent CDCl3
file exp
ACQUISITION
sfrq 100.602
tn C13
at 0.914
np 47804
sw 26143.8
fb not used
bs 4
tpwr 53
pw 9.5
dl 2.000
tof 1676.4
nt 1000
ct 128
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -1031.6
wp 21225.9
vs 237
sc 0
wc 250
hzmm 84.90
is 594.18
rfl 9648.3
rfp 7768.6
th 17
ins 1.000
al ph
DEC. & VT
dfrq 400.046
dn H1
dpwr 41
dof 1043.9
dm yyy
dmm w
dmf 10900
dseq
dres 1.0
homo n
temp 25.0
PROCESSING
lb 2.00
wtfile
proc ft
fn not used
math f
werr
wexp
wbs
wnt
129.118
128.452
128.159
127.391
180
160
140
120
100
80
60
40
20
0
ppm

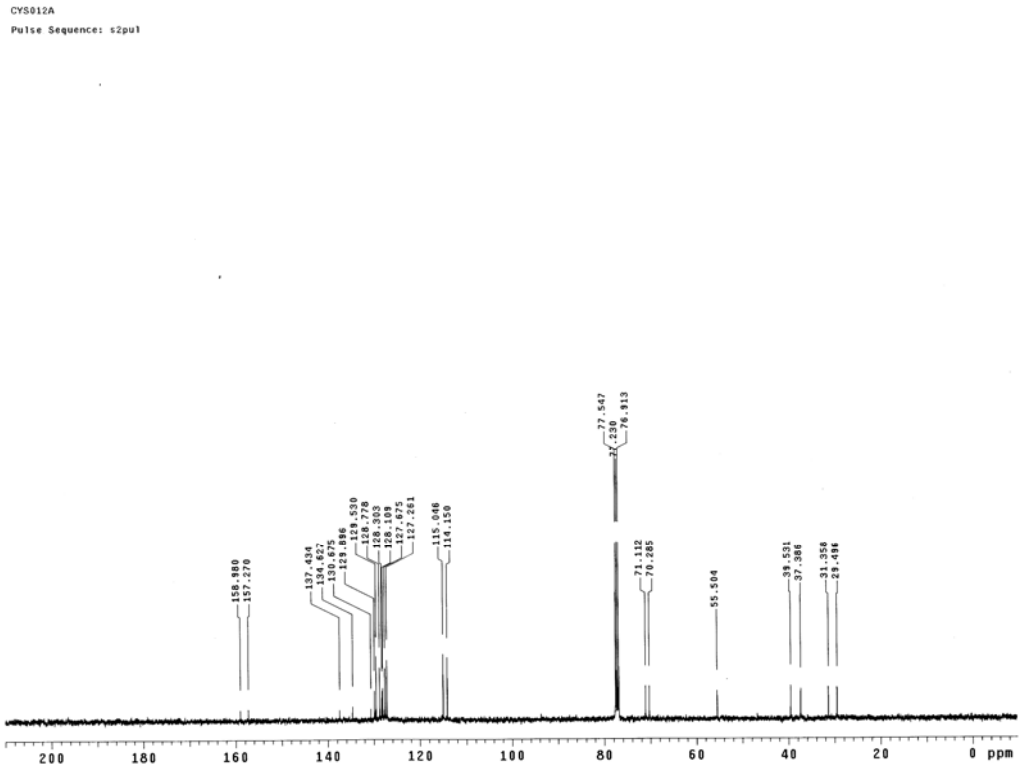

CYS009AB
Pulse Sequence: s2pul
OH
MeO
11
7.311
7.310
7.305
7.300
7.294
7.293
7.289
7.288
7.159
6.904
6.899
6.888
6.882
6.093
6.078
6.067
6.053
6.050
6.035
6.024
6.010
5.365
5.362
5.323
5.319
5.316
5.206
5.198
5.180
5.177
5.174
3.808
2.171
1.827
1.817
1.538
0.069
0.000
-0.003
10 9 8 7 6 5 4 3 2 1 -0 ppm
2.14
2.22
1.00
2.21
1.15
3.77
0.92

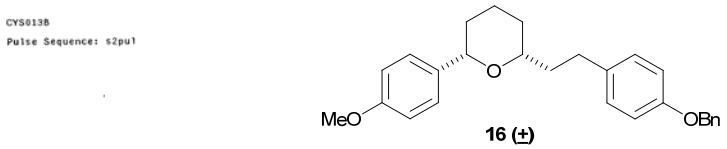

CYS009AB
Pulse Sequence: s2pul
159.470
140.592
135.061
127.898
115.000
114.180
77.549
77.230
76.910
75.125
55.529
200 180 160 140 120 100 80 60 40 20 0 ppm


CYS010B
Pulse Sequence: s2pul
MeO
O
H
13
ppm

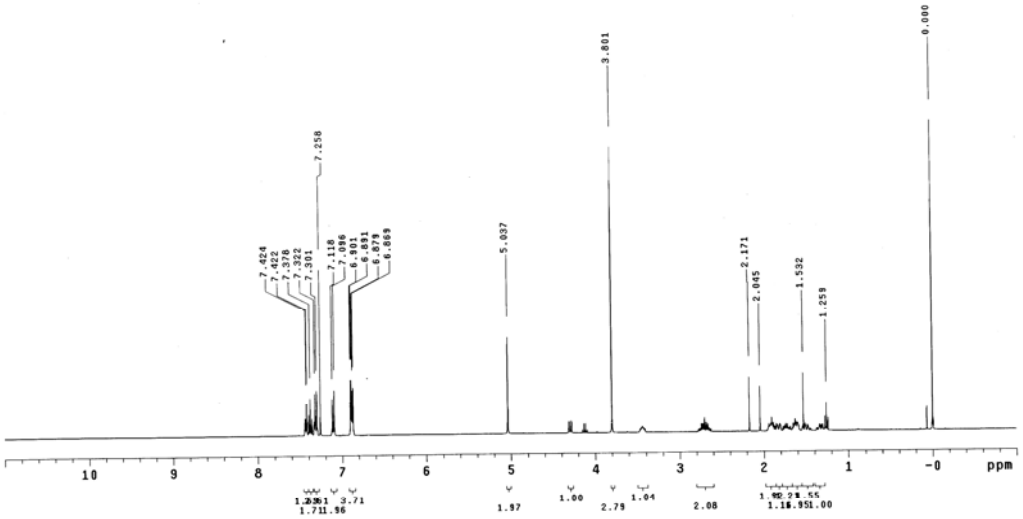

CYS010B
Pulse Sequence: s2pul
ppm

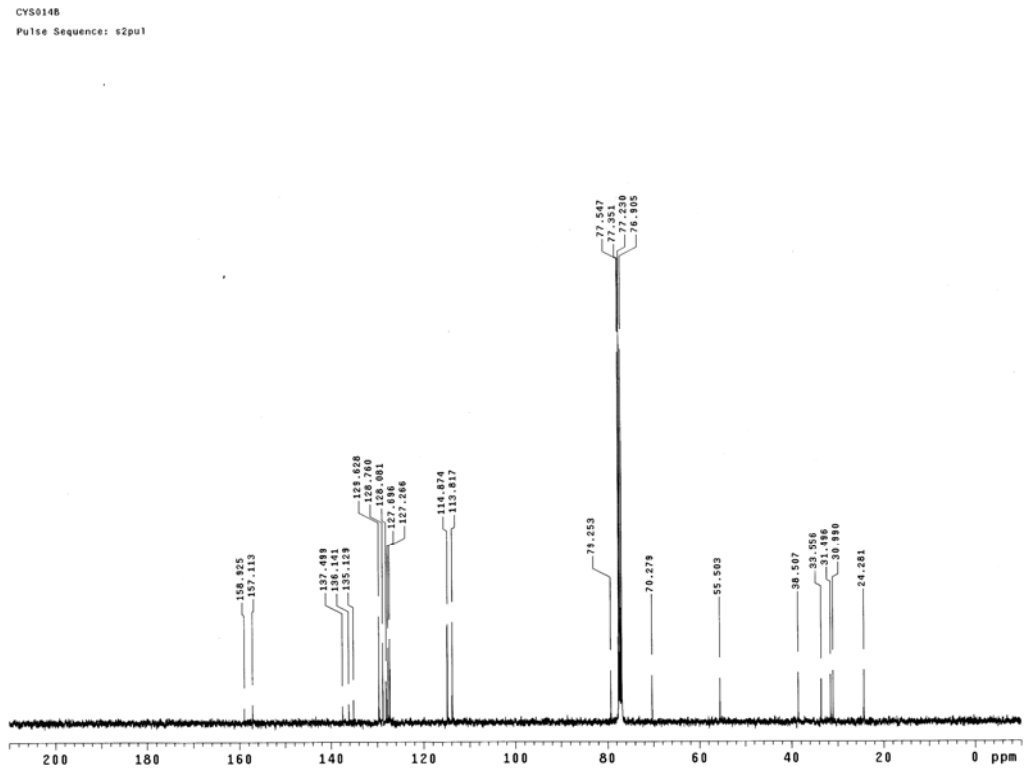

CYS007
Pulse Sequence: s2pul
HO
MeO
OBn
15 (±)
7.259
3.793
1.533
1.397
7.422
7.421
7.361
7.272
7.251
7.128
7.106
6.908
6.886
6.844
6.822
5.034
1.384
0.000
10
9
8
7
6
5
4
3
2
1
-0
ppm
2.10
1.08
1.13
2.13
1.13
3.10
1.21
1.15
2.27
2.20
0.93
2.31


CYS012A
Pulse Sequence: s2pul
77.547
77.230
76.913
158.980
157.270
137.434
134.627
130.675
129.896
129.530
128.778
128.303
128.109
127.675
127.281
115.046
114.159
71.112
70.285
55.504
39.531
37.386
31.356
29.496
200
180
160
140
120
100
80
60
40
20
0 ppm

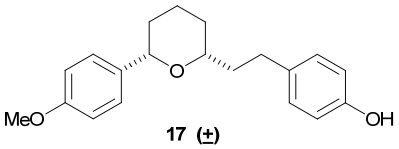

MeO
O
OBn

16 (±)

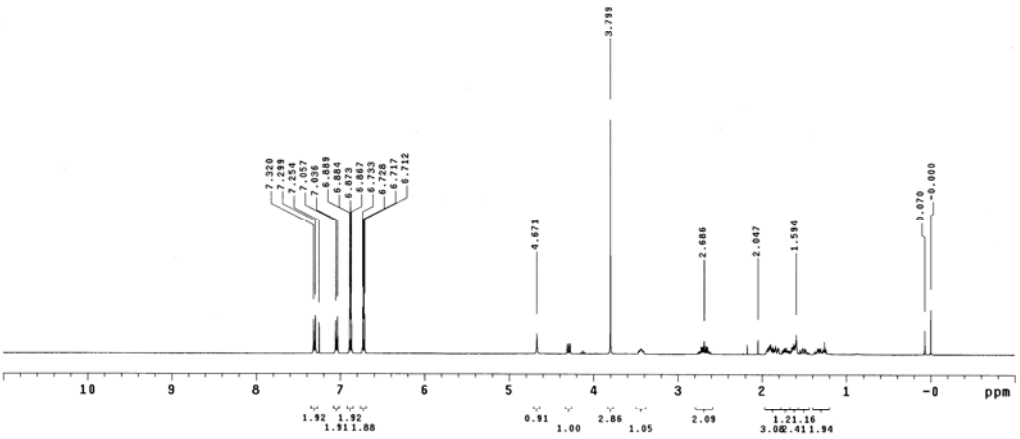

CYS013B
Pulse Sequence: s2pul
7.424
7.422
7.320
7.322
7.301
7.258
7.116
7.096
6.901
6.891
6.879
6.869
5.037
3.601
2.171
2.045
1.532
1.259
0.000
10
9
8
7
6
5
4
3
2
1
-0
ppm
1.97
1.00
2.79
1.04
2.08


CYS014B
Pulse Sequence: s2pul
158.325
157.113
137.499
136.141
135.129
128.628
128.760
126.681
127.696
127.266
114.874
113.817
77.547
77.351
77.230
76.905
73.253
70.279
55.503
38.507
33.556
31.496
30.990
24.281
200
180
160
140
120
100
80
60
40
20
0 ppm

CYS015B
Pulse Sequence: s2pul



17 (±)



CYS015B
Pulse Sequence: s2pul