

# Pd-Catalyzed Desymmetric Intramolecular *O*-Arylation Reaction: Enantioselective Synthesis of (3,4-Dihydro-2H-chromen-3-yl)methanols

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## Supporting Information

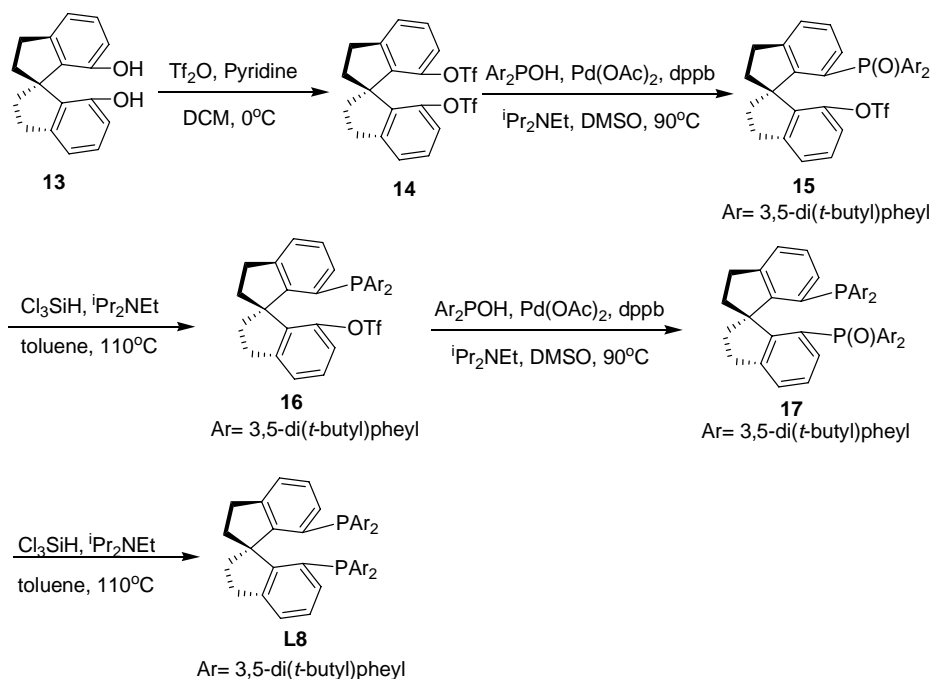
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## 1. General Remarks

$^1\text{H}$  NMR and  $^{13}\text{C}$  NMR spectra were recorded on a Bruker AV-400 or 500 MHz spectrometer. Chemical shifts ( $\delta$ ) are given in relative to tetramethylsilane ( $\delta$  0.00 ppm) in  $\text{CDCl}_3$ . Coupling constants,  $J$ , were reported in hertz unit (Hz). High resolution mass spectra (HRMS) were obtained on a Q-STAR Elite ESI-LC-MS/MS Spectrometer. Chemical names were generated using Cambridge Soft. ChemDraw Ultra 10.0. Optical rotations were measured on a WZZ-2S polarimeter. Enantiomeric ratios were determined by chiral HPLC using a chiralpak AD-H (Amylose tris (3,5-dimethylphenylcarbamate) coated on  $5\mu\text{m}$  silica-gel) or OD-H column (Cellulose tris (3,5-dimethylphenylcarbamate) coated on  $5\mu\text{m}$  silica-gel) with hexane and *i*-PrOH as solvents. Commercially obtained reagents were used without further purification.

## 2. Synthesis of Ligand L8:



Compound **L8** was prepared according to literature reported method<sup>[1]</sup>. To a solution of (*R*)-2,2',3,3'-tetrahydro-1,1'-spirobi[indene]-7,7'-diol **13** (3.0 g, 11.9 mmol) in  $\text{CH}_2\text{Cl}_2$  (70 mL) was added pyridine (3.8 mL, 47.6 mmol), and followed by dropwise addition of triflic anhydride (4.5 mL, 23.8 mmol) at  $0^\circ\text{C}$ . The mixture was stirred at room temperature over night. After removal of the solvent, the residue was diluted with EtOAc (50 mL) and then washed with 5 % aqueous HCl, saturated  $\text{NaHCO}_3$ , and brine. The organic layer was dried over anhydrous sodium sulfate, concentrated under reduced pressure. The residue was loaded on silica gel column and purified by  $\text{CH}_2\text{Cl}_2$  to afford the pure product **14** (5.8 g, 95%) as a white solid.  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.18-7.24 (m, 4H), 7.06 (d,  $J = 7.5$  Hz, 2H), 3.03 (m, 4H), 2.22-2.33 (m, 4H).  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ )  $\delta$  147.7, 145.9, 138.2, 129.4, 124.4, 119.3, 118.4, 116.7, 59.3, 38.6, 31.1.

EI-MS  $m/z$  516, ( $M^+$ ), 383, 367, 250, 233, 221, 202, 189, 165, 152, 107, 69.  $[\alpha]_D^{25} +146.1$  ( $c$  1.0,  $CHCl_3$ ).

A mixture of compound **14** (4.0 g, 7.8 mmol), bis(3,5-di-tert-butylphenyl)phosphine oxide (4.0 g, 9.4 mmol), palladium acetate (87.8 mg, 0.4 mmol) and 1,4-bis(diphenylphosphino)butane (dppb, 170 mg, 0.4 mmol) and diisopropylethylamine (4.0 g, 31 mmol) in DMSO (40 mL) was heated with stirring at 90 °C for 6 hours. After cooling to room temperature, the reaction mixture was diluted with EtOAc, washed with 5 % aqueous HCl and saturated  $NaHCO_3$ . The organic phase was dried over anhydrous  $Na_2SO_4$  and concentrated in vacuum. The residue was loaded on a silica gel column and purified with petroleum ether/EtOAc (3:1) to afford compound **15** (5.7 g, 92%) as a white solid.  $^1H$  NMR (500 MHz,  $CDCl_3$ )  $\delta$  7.55 (d,  $J$  = 0.5 Hz, 1H), 7.41 (m, 1H), 7.25 (d,  $J$  = 2.0 Hz, 1H), 7.18-7.22 (m, 3H), 7.07-7.14 (m, 2H), 7.04 (dd,  $J$  = 7.5, 2.0 Hz, 2H), 6.56 (d,  $J$  = 8.0 Hz, 1H), 3.22-3.29 (m, 1H), 3.02-3.12 (m, 4H), 2.25-2.38 (m, 3H), 1.26 (s, 18H), 1.70 (s, 18H).  $^{13}C$  NMR (125 MHz,  $CDCl_3$ )  $\delta$  152.8, 150.2, 150.1, 149.1, 145.9, 145.1, 141.9, 136.1, 135.2, 133.3, 131.4, 130.6, 128.4, 128.0, 127.8, 127.6, 126.0, 125.9, 125.8, 125.3, 124.7, 123.6, 119.1, 117.5, 116.6, 61.9, 39.7, 39.5, 34.9, 34.8, 31.7, 31.2, 31.2, 30.7. EI-MS  $m/z$  792 ( $M^+$ ), 777, 659, 644, 628, 576, 443, 4258, 322, 233, 149, 107, 57.  $[\alpha]_D^{25} +160.5$  ( $c$  1.0,  $CHCl_3$ ).

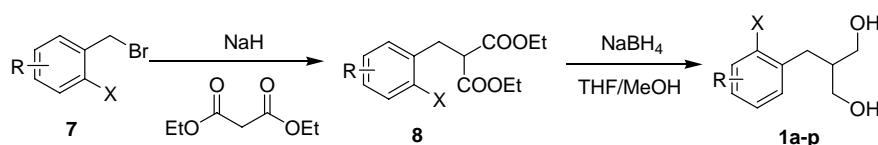
To a mixture of compound **15** (5.0 g, 6.3 mmol) and diisopropylethylamine (31.0 g, 240 mmol) in toluene (60 mL) was added  $Cl_3SiH$  (9.7 mL, 94.5 mmol) at 0 °C. The reaction mixture was stirred at 110 °C for overnight. After cooling to room temperature, the mixture was diluted with  $Et_2O$  and quenched with small amount of saturated  $NaHCO_3$ . The resulting suspension was filtered through Celite and the solid was washed with  $Et_2O$ . The combined organic layer was dried over  $Na_2SO_4$  and concentrated under reduced pressure. The residue was loaded on silica gel column and purified through chromatography with petroleum ether/EtOAc (50/1 to 20/1) as eluent to give compound **16** (4.2 g, 86%) as a white solid.  $^1H$  NMR (500 MHz,  $CDCl_3$ )  $\delta$  7.13-7.21 (m, 5H), 7.07 (t,  $J$  = 7.5 Hz, 1H), 6.91 (d,  $J$  = 7.5 Hz, 2H), 6.80 (d,  $J$  = 7.5 Hz, 2H), 6.73 (d,  $J$  = 7.5 Hz, 2H), 2.87-2.97 (m, 3H), 2.72 (m, 1H), 2.26 (m, 2H), 1.97 (m, 2H), 1.11 (s, 18H), 1.09 (s, 18H).  $^{13}C$  NMR (125 MHz,  $CDCl_3$ )  $\delta$  151.8, 150.1, 150.0, 148.1, 146.3, 143.3, 141.1, 137.0, 136.1, 133.6, 133.4, 128.8, 128.4, 128.2, 127.7, 127.6, 126.9, 125.1, 123.9, 122.0, 121.9, 119.3, 118.2, 116.7, 61.7, 61.6, 39.0, 38.9, 38.8, 34.7, 31.3, 31.2, 30.6. EI-MS  $m/z$  776 ( $M^+$ ), 719, 627, 601, 298, 263, 233, 147, 57.  $[\alpha]_D^{25} +116.5$  ( $c$  1.0,  $CHCl_3$ ).

The mixture of compound **16** (3.0 g, 3.9 mmol), bis(3,5-di-tert-butylphenyl)phosphine oxide (2.5 g, 5.8 mmol), palladium acetate (43 mg, 0.2 mmol), 1,4-bis(diphenylphosphino)butane (dppb, 82 mg, 0.2 mmol) and diisopropylethylamine (2.0 g, 15.6 mmol) in DMSO (40 mL) was heated with stirring at 90 °C for 6 hours. After cooling to room temperature, the reaction mixture was diluted with EtOAc, washed with 5 % aqueous HCl and saturated  $NaHCO_3$ . The organic phase was dried over anhydrous  $Na_2SO_4$  and concentrated under reduced pressure. The residue was loaded on a silica gel column and purified with petroleum ether(20/1 to 5/1) to give compound **17** (3.2 g, 83%) as a white solid.  $^1H$  NMR (500 MHz,  $CDCl_3$ )  $\delta$  7.66 (d,  $J$  = 8.0 Hz, 2H), 7.60 (dd,  $J$  =

11.5, 1.5 Hz, 2H), 7.44 (s, 1H), 7.31-7.36 (m, 3H), 7.24 (d,  $J = 6.0$ Hz, 2H), 7.09-7.16 (m, 3H), 6.55-7.07 (m, 3H), 6.56 (d,  $J = 6.0$  Hz, 2H), 3.01-3.10 (m, 2H), 2.66-2.72 (m, 2H), 2.36 (dd,  $J = 16.0, 9.5$  Hz, 1H), 2.03 (dd,  $J = 12.0, 7.5$  Hz, 1H), 1.86 (dd,  $J = 13.0, 9.0$  Hz, 1H), 1.55 (m, 1H), 1.28 (s, 18H), 1.25 (s, 18H), 1.00 (s, 18H), 0.92 (s, 18H).  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ )  $\delta$  157.8, 153.3, 150.2, 150.0, 149.3, 149.0, 146.2, 140.7, 135.8, 135.0, 134.9, 134.1, 132.7, 132.2, 132.1, 131.5, 129.6, 128.3, 127.3, 126.7, 126.5, 125.4, 125.2, 125.1, 124.5, 124.4, 122.3, 120.3, 64.8, 39.8, 38.6, 35.0, 34.8, 34.4, 31.2, 31.1, 30.4. ESI-MS  $m/z$  1053.4 ( $\text{M} + \text{H}^+$ ). HRMS calcd for  $\text{C}_{73}\text{H}_{99}\text{OP}_2^+$  ( $\text{M} + \text{H}^+$ ) 1053.7166, found 1053.7146.  $[\alpha]_{\text{D}}^{25} +165.5$  ( $c$  1.0,  $\text{CHCl}_3$ ).

To a mixture of compound **17** (3.1 g, 2.9 mmol) and diisopropylethylamine (15.0 g, 116 mmol) in toluene (60 mL) was added  $\text{Cl}_3\text{SiH}$  (4.5 mL, 43.4 mmol) at 0 °C. The reaction mixture was stirred at 110 °C for overnight. After cooling to room temperature, the mixture was diluted with  $\text{Et}_2\text{O}$  and quenched with small amount of saturated  $\text{NaHCO}_3$ . The resulting suspension was filtered through Celite and the solid was washed with  $\text{Et}_2\text{O}$ . The combined organic layer was dried over  $\text{Na}_2\text{SO}_4$  and concentrated under reduced pressure. The crude product was purified by silica gel column chromatography with petroleum ether/DCM (50/1 to 5/1) as eluent to give compound **L8** (2.6 g, 87%) as a white solid.  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.25 (s, 2H), 7.07-7.18 (m, 10H), 6.96 (s, 2H), 6.84 (s, 2H), 2.73 (m, 2H), 2.51 (m, 2H), 1.74 (m, 2H), 1.66 (m, 2H), 1.63 (s, 36 H), 1.87 (s, 36H);  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ )  $\delta$  152.9, 150.0, 148.8, 143.2, 138.5, 136.6, 136.6, 132.8, 129.8, 127.0, 126.1, 125.1, 122.6, 120.5, 64.7, 38.6, 34.8, 34.6, 31.4, 31.1, 30.3; ESI-MS  $m/z$  1037.4 ( $\text{M} + \text{H}^+$ ). HRMS calcd for  $\text{C}_{73}\text{H}_{99}\text{P}_2^+$  ( $\text{M} + \text{H}^+$ ) 1037.7217, found 1037.7205.  $[\alpha]_{\text{D}}^{25} +103.4$  ( $c$  1.0,  $\text{CHCl}_3$ ).

### 3. Synthesis of Substrates:



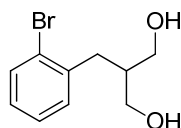
#### 2-(2-iodobenzyl)propane-1,3-diol (**1a**):

Typical procedure: Compound **1a** was prepared according to similar route of literature reported methods<sup>[2]</sup>: Ethyl malonate (6.2 mL, 40 mmol) was slowly added to the suspension of sodium hydride (1.6 g, 40 mmol) in THF at 0 °C. The reaction mixture was stirred at this temperature for 1.0 hours, then compound **7** (6.0 g, 20 mmol) in THF (10 mL) was added. The mixture was stirred at room temperature for another 3 hours and quenched by addition of  $\text{H}_2\text{O}$  (20 mL). The aqueous phase was extracted with ethyl acetate (50.0 mL  $\times$  3). The combined organic phase was concentrated under reduced pressure, and the residue was loaded on silica gel column and purified by flash chromatography (ethyl acetate/petroleum ether = 1/50 to 1/15) to afford the pure product **8** (7.0 g, 93%) as a colorless oil.

To a solution of compound **8** (7.0 g, 18.6 mmol) in THF (60 mL), was added  $\text{NaBH}_4$  (3.5 g, 93

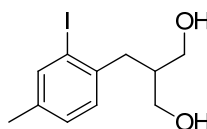
mmol) and the mixture was stirred at 0 °C for 15 minute. Then MeOH (12 mL) was added, and the mixture was stirred at room temperature for 4 hours. The reaction mixture was quenched by addition of H<sub>2</sub>O (20 mL). The organic solvents were removed under reduced pressure and the aqueous phase was extracted with ethyl acetate (50.0 mL × 3). The combined organic phase was concentrated under reduced pressure. The residue was loaded on silica gel column and purified by flash chromatography (ethyl acetate/petroleum ether = 1/3 to 1/1) to afford the pure product **1a** (4.9 g, 90%) as a white solid. <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) δ 7.74 (d, *J* = 8.0 Hz, 1H), 7.14-7.20 (m, 2H), 6.81 (m, 1H), 3.74 (dd, *J* = 11.0, 5.5 Hz, 2H), 3.62 (dd, *J* = 11.0, 7.0 Hz, 2H), 3.35 (br s, 2H), 2.65 (d, *J* = 7.0 Hz, 2H), 2.03 (m, 1H); <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>) δ 142.6, 139.6, 130.4, 128.2, 128.0, 100.9, 64.5, 42.3, 38.5; ESI-MS *m/z* 314.7 (M + Na)<sup>+</sup>; HRMS calcd for C<sub>10</sub>H<sub>13</sub>INaNO<sub>3</sub><sup>+</sup> (M + Na)<sup>+</sup> 314.9852, found 314.9852.

**2-(2-bromobenzyl)propane-1,3-diol (1a')**:



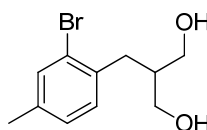
87% yield, white solid. <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) δ 7.46 (d, *J* = 8.0 Hz, 1H), 7.14-7.18 (m, 2H), 7.01 (m, 1H), 3.77 (dd, *J* = 10.0, 5.5 Hz, 2H), 3.65 (dd, *J* = 11.0, 7.0 Hz, 2H), 2.70 (d, *J* = 7.0 Hz, 2H), 2.07 (m, 1H), 1.87 (br s, 2H); <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>) δ 139.4, 132.9, 131.3, 127.9, 127.3, 124.7, 64.4, 42.3, 34.2; ESI-MS *m/z* 266.7 (M + Na)<sup>+</sup>; HRMS calcd for C<sub>10</sub>H<sub>13</sub>BrNaO<sub>2</sub><sup>+</sup> (M + Na)<sup>+</sup> 266.9991, found 266.9990.

**2-(2-iodo-4-methylbenzyl)propane-1,3-diol (1b)**:



89% yield, colorless oil. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.66 (s, 1H), 7.06-7.12 (m, 2H), 3.84 (dd, *J* = 10.4, 2.8 Hz, 2H), 3.71 (dd, *J* = 10.4, 6.8 Hz, 2H), 2.71 (d, *J* = 7.6 Hz, 2H), 2.27 (s, 3H), 2.10 (br s, 3H); <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>) δ 139.9, 139.4, 137.9, 130.0, 129.1, 100.8, 64.7, 42.4, 38.0, 20.2; ESI-MS *m/z* 329.6 (M + Na)<sup>+</sup>; HRMS calcd for C<sub>11</sub>H<sub>15</sub>INaO<sub>2</sub><sup>+</sup> (M + Na)<sup>+</sup> 329.0009, found 329.0013.

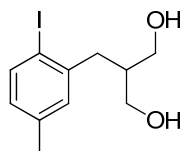
**2-(2-bromo-4-methylbenzyl)propane-1,3-diol (1b')**:



83% yield, colorless oil. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.37 (s, 1H), 7.11 (d, *J* = 8.0 Hz, 1H), 7.03 (d, *J* = 8.0 Hz, 1H), 3.83 (dd, *J* = 10.4, 2.4 Hz, 2H), 3.70 (dd, *J* = 10.4, 6.4 Hz, 2H), 2.72 (d, *J* =

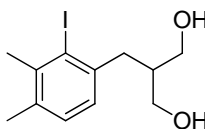
7.2 Hz, 2H), 2.30 (s, 3H), 2.10 (m, 1H);  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ )  $\delta$  137.9, 136.1, 133.3, 131.0, 128.2, 124.4, 64.8, 42.4, 33.7, 20.5; ESI-MS  $m/z$  259.1 ( $\text{M} + \text{H}$ ) $^+$ ; HRMS calcd for  $\text{C}_{11}\text{H}_{16}\text{BrO}_2^+$  ( $\text{M} + \text{Na}$ ) $^+$  259.03828, found 259.0330.

**2-(2-iodo-5-methylbenzyl)propane-1,3-diol (1c):**



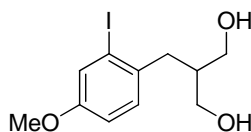
82% yield, colorless oil.  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.66 (d,  $J = 8.0$  Hz, 1H), 7.03 (d,  $J = 1.5$  Hz, 1H), 6.71 (dd,  $J = 8.0, 1.5$  Hz, 1H), 3.82 (dd,  $J = 11.0, 3.5$  Hz, 2H), 3.69 (dd,  $J = 11.0, 6.5$  Hz, 2H), 2.88 (br s, 2H), 2.67 (d,  $J = 7.5$  Hz, 2H), 2.26 (s, 3H), 2.10 (m, 1H);  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ )  $\delta$  142.3, 139.3, 138.2, 131.3, 129.1, 96.8, 64.9, 42.4, 38.4, 20.8; ESI-MS  $m/z$  329.6 ( $\text{M} + \text{Na}$ ) $^+$ ; HRMS calcd for  $\text{C}_{11}\text{H}_{15}\text{I}^+$  ( $\text{M} + \text{Na}$ ) $^+$  329.0009, found 329.0013.

**2-(2-iodo-3,4-dimethylbenzyl)propane-1,3-diol (1d):**



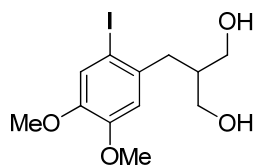
64% yield, white solid.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.01 (d,  $J = 8.0$  Hz, 1H), 6.95 (d,  $J = 8.0$  Hz, 1H), 3.86 (dd,  $J = 10.8, 3.2$  Hz, 2H), 3.72 (dd,  $J = 10.8, 6.8$  Hz, 2H), 2.79 (d,  $J = 7.6$  Hz, 2H), 2.47 (s, 3H), 2.34 (s, 3H), 2.25 (br s, 2H), 2.14 (m, 1H);  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ )  $\delta$  140.9, 140.4, 135.2, 129.4, 127.6, 109.8, 65.1, 42.2, 40.0, 26.6, 21.8; ESI-MS  $m/z$  343.2 ( $\text{M} + \text{Na}$ ) $^+$ ; HRMS calcd for  $\text{C}_{12}\text{H}_{17}\text{I}^+$  ( $\text{M} + \text{Na}$ ) $^+$  343.0165, found 343.0167.

**2-(2-iodo-4-methoxybenzyl)propane-1,3-diol (1e):**



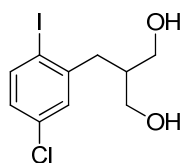
88% yield, white solid.  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.36 (s, 1H), 7.11 (d,  $J = 8.0$  Hz, 1H), 6.83 (d,  $J = 8.0$  Hz, 1H), 3.85 (dd,  $J = 11.0, 3.0$  Hz, 2H), 3.77 (s, 3H), 3.70 (dd,  $J = 11.0, 6.5$  Hz, 2H), 2.70 (d,  $J = 7.5$  Hz, 2H), 2.10 (br s, 2H), 2.07 (m, 1H);  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ )  $\delta$  158.2, 134.6, 130.4, 124.6, 114.4, 100.6, 65.3, 55.5, 42.7, 37.5; ESI-MS  $m/z$  344.9 ( $\text{M} + \text{Na}$ ) $^+$ ; HRMS calcd for  $\text{C}_{11}\text{H}_{15}\text{I}^+$  ( $\text{M} + \text{Na}$ ) $^+$  344.9958, found 344.9961.

**2-(2-iodo-4,5-dimethoxybenzyl)propane-1,3-diol (1f):**



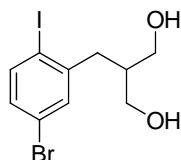
72% yield, white solid.  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.19 (s, 1H), 6.76 (s, 1H), 3.83 (dd,  $J = 11.0$ , 2.5 Hz, 2H), 3.83 (s, 3H), 3.82 (s, 3H), 3.70 (dd,  $J = 11.0$ , 6.5 Hz, 2H), 2.67 (d,  $J = 7.5$  Hz, 2H), 2.43 (br s, 2H), 2.07 (m, 1H);  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ )  $\delta$  149.3, 148.1, 135.1, 121.8, 113.2, 88.5, 65.0, 56.2, 56.0, 42.8, 38.2; ESI-MS  $m/z$  375.4 ( $\text{M} + \text{Na}$ ) $^+$ ; HRMS calcd for  $\text{C}_{12}\text{H}_{17}\text{I}\text{NaO}_4^+$  ( $\text{M} + \text{Na}$ ) $^+$  375.0064, found 375.0064.

**2-(5-chloro-2-iodobenzyl)propane-1,3-diol (1g):**



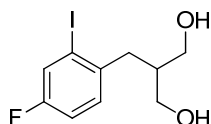
79% yield, white solid.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.71 (d,  $J = 8.4$  Hz, 1H), 7.23 (d,  $J = 2.0$  Hz, 1H), 6.90 (dd,  $J = 8.4$ , 2.0 Hz, 1H), 3.85 (dd,  $J = 10.8$ , 2.8 Hz, 2H), 3.70 (dd,  $J = 10.8$ , 6.0 Hz, 2H), 2.73 (d,  $J = 7.2$  Hz, 2H), 2.61 (br s, 2H), 2.06 (m, 1H);  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ )  $\delta$  144.6, 140.5, 134.5, 130.3, 128.2, 97.9, 64.4, 42.1, 38.4; ESI-MS  $m/z$  327.0 ( $\text{M} + \text{H}$ ) $^+$ ; HRMS calcd for  $\text{C}_{10}\text{H}_{13}\text{ClIO}_2^+$  ( $\text{M} + \text{H}$ ) $^+$  326.9643, found 326.9646.

**2-(5-bromo-2-iodobenzyl)propane-1,3-diol (1h):**



82% yield, white solid.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.65 (d,  $J = 8.0$  Hz, 1H), 7.39 (s, 1H), 7.04 (d,  $J = 8.0$  Hz, 1H), 3.85 (dd,  $J = 10.8$ , 3.2 Hz, 2H), 3.70 (dd,  $J = 10.8$ , 6.8 Hz, 2H), 2.73 (d,  $J = 6.8$  Hz, 2H), 2.33 (br s, 2H), 2.08 (m, 1H);  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ )  $\delta$  145.2, 140.9, 133.2, 131.2, 122.6, 98.7, 64.6, 42.6, 38.6; ESI-MS  $m/z$  370.9 ( $\text{M} + \text{H}$ ) $^+$ ; HRMS calcd for  $\text{C}_{10}\text{H}_{13}\text{BrIO}_2^+$  ( $\text{M} + \text{H}$ ) $^+$  370.9138, found 370.9142.

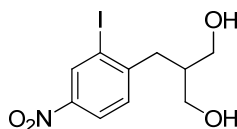
**2-(4-fluoro-2-iodobenzyl)propane-1,3-diol (1i):**



79% yield, yellow solid.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.54 (dd,  $J = 8.0$ , 2.0 Hz, 1H), 7.17-7.20 (m, 1H), 6.97-7.02 (m, 1H), 3.83 (dd,  $J = 10.8$ , 3.2 Hz, 2H), 3.67 (dd,  $J = 10.8$ , 6.4 Hz, 2H), 2.72 (d,  $J = 7.6$  Hz, 2H), 2.68 (br s, 2H), 2.05 (m, 1H);  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ )  $\delta$  160.5, 138.5, 130.8, 126.3, 115.2, 99.6, 64.8, 42.3, 37.5; ESI-MS  $m/z$  311.6 ( $\text{M} + \text{H}$ ) $^+$ ; HRMS calcd for  $\text{C}_{10}\text{H}_{13}\text{FO}_2^+$  ( $\text{M} + \text{H}$ ) $^+$  311.6064, found 311.6064.

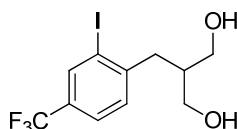
$\text{F I O}_2^+ (\text{M} + \text{H})^+$  310.9939, found 310.9941.

**2-(2-iodo-4-nitrobenzyl)propane-1,3-diol (1j):**



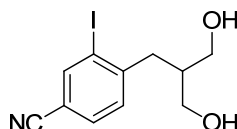
86% yield, yellow solid.  $^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.65 (d,  $J = 2.0$  Hz, 1H), 8.12 (dd,  $J = 8.4$ , 2.0 Hz, 1H), 7.42 (d,  $J = 8.4$  Hz, 1H), 3.84 (dd,  $J = 10.8$ , 3.2 Hz, 2H), 3.67 (dd,  $J = 10.8$ , 6.0 Hz, 2H), 2.90 (d,  $J = 7.6$  Hz, 2H), 2.35 (br s, 2H), 2.10 (m, 1H);  $^{13}\text{C NMR}$  (125 MHz,  $\text{CDCl}_3$ )  $\delta$  150.6, 146.5, 134.4, 130.5, 123.0, 99.7, 64.4, 41.9, 38.5; ESI-MS  $m/z$  360.2 ( $\text{M} + \text{Na}$ ) $^+$ ; HRMS calcd for  $\text{C}_{10}\text{H}_{12}\text{I N Na O}_4^+$  ( $\text{M} + \text{Na}$ ) $^+$  359.9703, found 359.9705.

**2-(2-iodo-4-(trifluoromethyl)benzyl)propane-1,3-diol (1k):**



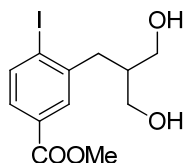
64% yield, colorless oil.  $^1\text{H NMR}$  (500 MHz,  $\text{CDCl}_3$ )  $\delta$  8.06 (s, 1H), 7.52 (d,  $J = 8.0$  Hz, 1H), 7.36 (d,  $J = 8.0$  Hz, 1H), 3.86 (dd,  $J = 11.0$ , 2.0 Hz, 2H), 3.70 (dd,  $J = 11.0$ , 6.0 Hz, 2H), 2.83 (d,  $J = 7.5$  Hz, 2H), 2.58 (br s, 2H), 2.10 (m, 1H);  $^{13}\text{C NMR}$  (125 MHz,  $\text{CDCl}_3$ )  $\delta$  147.0, 136.4, 130.4, 130.2, 125.0, 122.8, 100.3, 64.7, 42.0, 38.5; ESI-MS  $m/z$  382.9 ( $\text{M} + \text{Na}$ ) $^+$ ; HRMS calcd for  $\text{C}_{11}\text{H}_{12}\text{F}_3\text{I Na O}_2^+$  ( $\text{M} + \text{Na}$ ) $^+$  382.9726, found 382.9730.

**4-(3-hydroxy-2-(hydroxymethyl)propyl)-3-iodobenzonitrile (1l):**



92% yield, white solid.  $^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.09 (s, 1H), 7.56 (d,  $J = 8.0$  Hz, 1H), 7.37 (d,  $J = 8.0$  Hz, 1H), 3.86 (dd,  $J = 10.8$ , 3.2 Hz, 2H), 3.67 (dd,  $J = 10.8$ , 6.0 Hz, 2H), 2.86 (d,  $J = 7.2$  Hz, 2H), 2.15 (br s, 2H), 2.08 (m, 1H);  $^{13}\text{C NMR}$  (125 MHz,  $\text{CDCl}_3$ )  $\delta$  148.8, 142.5, 131.5, 130.7, 117.0, 111.6, 100.4, 64.1, 41.7, 38.7; ESI-MS  $m/z$  340.9 ( $\text{M} + \text{Na}$ ) $^+$ ; HRMS calcd for  $\text{C}_{11}\text{H}_{12}\text{I N Na O}_2^+$  ( $\text{M} + \text{Na}$ ) $^+$  339.9807, found 339.9805.

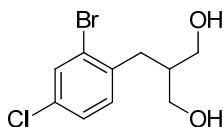
**methyl 3-(3-hydroxy-2-(hydroxymethyl)propyl)-4-iodobenzoate (1m):**



67% yield, colorless oil.  $^1\text{H NMR}$  (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.87 (d,  $J = 8.0$  Hz, 1H), 7.83 (s, 1H), 7.48 (d,  $J = 8.0$  Hz, 1H), 3.87 (s, 3H), 3.79 (dd,  $J = 11.0$ , 3.0 Hz, 2H), 3.66 (dd,  $J = 11.0$ , 6.0 Hz, 2H),

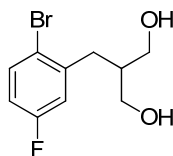
3.14 (br s, 2H), 2.77 (d,  $J = 7.5$  Hz, 2H), 2.08 (m, 1H);  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ )  $\delta$  166.8, 143.4, 139.9, 130.8, 130.1, 128.5, 107.2, 64.2, 52.3, 42.2, 38.4; ESI-MS  $m/z$  373.0 ( $\text{M} + \text{Na}$ ) $^+$ ; HRMS calcd for  $\text{C}_{12}\text{H}_{15}\text{INaO}_4^+$  ( $\text{M} + \text{Na}$ ) $^+$  372.9907, found 372.9906.

**2-(2-bromo-4-chlorobenzyl)propane-1,3-diol (1n):**



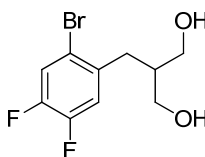
88% yield, white solid.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.55 (s, 1H), 7.17-7.23 (m, 2H), 3.82 (dd,  $J = 10.4, 3.4$  Hz, 2H), 3.68 (dd,  $J = 10.4, 7.2$  Hz, 2H), 2.74 (d,  $J = 7.2$  Hz, 2H), 2.42 (br s, 2H), 2.08 (m, 1H);  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ )  $\delta$  138.0, 132.7, 132.5, 132.0, 127.6, 124.9, 64.6, 42.1, 33.6; ESI-MS  $m/z$  279.0 ( $\text{M} + \text{H}$ ) $^+$ ; HRMS calcd for  $\text{C}_{10}\text{H}_{13}\text{BrClO}_2^+$  ( $\text{M} + \text{H}$ ) $^+$  278.9782, found 278.9784.

**2-(2-bromo-5-fluorobenzyl)propane-1,3-diol (1o):**



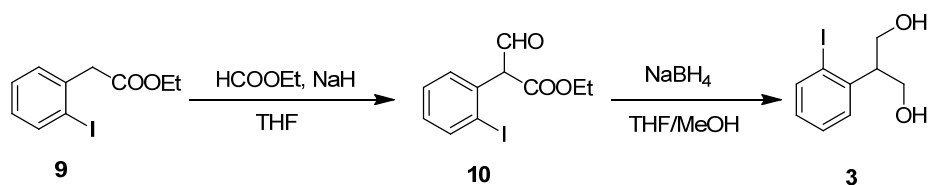
74% yield, colorless oil.  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.45-7.48 (m, 1H), 6.98 (d,  $J = 9.0$  Hz, 1H), 6.78-6.82 (m, 1H), 3.80 (dd,  $J = 11.0, 3.5$  Hz, 2H), 3.66 (dd,  $J = 11.0, 7.5$  Hz, 2H), 3.16 (br s, 2H), 2.73 (d,  $J = 7.0$  Hz, 2H), 2.09 (m, 1H);  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ )  $\delta$  161.8, 141.5, 134.0, 118.8, 118.1, 115.2, 64.4, 42.0, 34.3; ESI-MS  $m/z$  285.2 ( $\text{M} + \text{Na}$ ) $^+$ ; HRMS calcd for  $\text{C}_{10}\text{H}_{12}\text{BrFNaO}_2^+$  ( $\text{M} + \text{Na}$ ) $^+$  284.9897, found 284.9898.

**2-(2-bromo-4,5-difluorobenzyl)propane-1,3-diol (1p):**



83% yield, colorless oil.  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.37 (t,  $J = 8.5$  Hz, 1H), 7.09-7.13 (m, 1H), 3.82 (dd,  $J = 10.5, 2.5$  Hz, 2H), 3.66 (dd,  $J = 10.5, 6.0$  Hz, 2H), 2.73 (d,  $J = 7.5$  Hz, 2H), 2.41 (br s, 2H), 2.09 (m, 1H);  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ )  $\delta$  150.0, 148.0, 136.3, 121.5, 119.3, 117.7, 64.5, 42.0, 33.6; ESI-MS  $m/z$  281.0 ( $\text{M} + \text{H}$ ) $^+$ ; HRMS calcd for  $\text{C}_{10}\text{H}_{12}\text{BrF}_2\text{O}_2^+$  ( $\text{M} + \text{Na}$ ) $^+$  280.9979, found 280.9983.

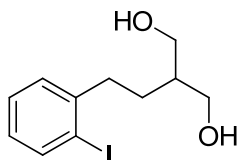
**2-(2-iodophenyl)propane-1,3-diol (3):**



Compound **3** was prepared according to similar route of literature reported methods<sup>[3]</sup>. Compound **9** (4.0 g, 14 mmol) in THF (15 mL) was slowly added into a suspension of sodium hydride (1.8 g, 45 mmol) in THF at 0 °C. The reaction mixture was stirred at this temperature for 1.0 hours and ethyl methanoate (2.5 mL, 30 mmol) was added at room temperature. The mixture was stirred for another 3 hours and quenched by addition of H<sub>2</sub>O (20 mL). The aqueous phase was extracted with ethyl acetate (15.0 mL × 3). The combined organic phase was concentrated under reduced pressure then the residue was loaded on silica gel column and purified by flash chromatography (ethyl acetate/petroleum ether = 1/50 to 1/20) to afford the pure product **10** (3.4 g, 76%) as a yellow solid.

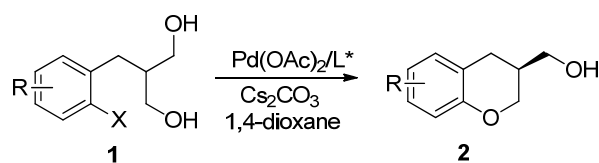
NaBH<sub>4</sub> (1.5 g, 39 mmol) was slowly added into a solution of compound **10** (2.2 g, 6.9 mmol) in THF (30 ml), and the mixture was stirred at 0 °C for 15 minute. Then MeOH (6 mL) was added and the mixture was stirred at room temperature for 4 hours. The reaction mixture was quenched H<sub>2</sub>O (20 mL) and concentrated under reduced pressure to remove THF and MeOH. The aqueous phase was extracted with ethyl acetate (50.0 mL × 3). The combined organic phase was concentrated under reduced pressure. The residue was loaded on silica gel column and purified by flash chromatography (ethyl acetate/petroleum ether = 1/3 to 1/1) to afford compound **3** (1.8 g, 92%) as a white solid. <sup>1</sup>H NMR (500 MHz, DMSO) δ 7.83 (dd, *J* = 8.0, 2.0 Hz, 1H), 7.32 (t, *J* = 8.0 Hz, 1H), 7.27 (dd, *J* = 8.0, 2.0 Hz, 1H), 6.95 (t, *J* = 8.0 Hz, 1H), 4.62 (t, *J* = 5.5 Hz, 2H), 3.63 (m, 4H), 3.18 (m, 1H); <sup>13</sup>C NMR (125 MHz, DMSO) δ 143.6, 139.0, 128.5, 128.2, 128.0, 103.0, 61.5, 53.6; ESI-MS *m/z* 300.96 (M + Na)<sup>+</sup>; HRMS calcd for C<sub>9</sub>H<sub>11</sub>INaO<sub>2</sub><sup>+</sup> (M + Na)<sup>+</sup> 300.9696, found 300.9697.

#### 2-(2-iodophenethyl)propane-1,3-diol (**5**):



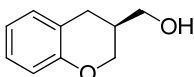
This compound was prepared similarly to **1a** (78%) as a white solid. <sup>1</sup>H NMR (400 MHz, CHCl<sub>3</sub>) δ 7.70 (d, *J* = 7.6 Hz, 1H), 7.16 (t, *J* = 7.6 Hz, 1H), 7.11 (d, *J* = 7.6 Hz, 1H), 6.77 (t, *J* = 8.0 Hz, 1H), 3.79 (dd, *J* = 10.4, 6.4 Hz, 2H), 3.66 (t, *J* = 10.0 Hz, 2H), 2.67 (t, *J* = 8.4 Hz, 2H), 2.46 (br s, 2H), 1.77 (m, 1H), 1.47 (m, 2H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ 144.8, 139.6, 129.3, 128.5, 127.8, 100.4, 65.7, 42.2, 38.6, 28.7; ESI-MS *m/z* 329.1 (M + Na)<sup>+</sup>. HRMS calcd for C<sub>11</sub>H<sub>15</sub>INaO<sub>2</sub><sup>+</sup> (M + Na)<sup>+</sup> 329.0009, found 329.0007.

#### 4. General Procedure for Pd-Catalyzed Coupling Reactions:



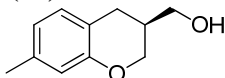
The reaction mixture of **1** (0.2 mmol), ligand (0.006 mmol), Pd(OAc)<sub>2</sub> (0.006 mmol) and Cs<sub>2</sub>CO<sub>3</sub> (0.4 mmol) in 1,4-dioxane (1.0 mL) were stirred at appointed temperature for 15 hours. After the reaction being completed, H<sub>2</sub>O (5.0 mL) and ethyl acetate (5.0 mL) were added into the mixture. The organic phase was separated and the aqueous phase was extracted with ethyl acetate (5.0 mL × 4). The solvent was removed under reduced pressure. The residue was loaded on silica column and purified by flash chromatography (ethyl acetate/ dichloromethane /petroleum ether = 1/3/10 to 1/1/3.5) to afford coupling products **2**.

##### (S)-chroman-3-ylmethanol (**2a**):



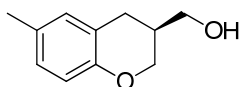
65% yield, yellow oil. <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) δ 7.10 (t, *J* = 7.5 Hz, 1H), 7.05 (d, *J* = 7.5 Hz, 1H), 6.86 (t, *J* = 7.5 Hz, 1H), 6.83 (d, *J* = 7.5 Hz, 1H), 4.30 (dd, *J* = 10.5, 2.0 Hz, 1H), 4.00 (dd, *J* = 10.5, 7.5 Hz, 1H), 3.69 (dd, *J* = 10.5, 6.0 Hz, 1H), 3.65 (dd, *J* = 10.5, 7.5 Hz, 1H), 2.87 (dd, *J* = 16.5, 6.0 Hz, 1H), 2.58 (dd, *J* = 16.5, 7.5 Hz, 1H), 2.27 (m, 1H); <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>) δ 154.5, 130.0, 127.3, 121.0, 120.5, 116.6, 67.6, 63.3, 34.8, 27.4; ESI-MS *m/z* 165.1 (M + H)<sup>+</sup>; HRMS calcd for C<sub>10</sub>H<sub>13</sub>O<sub>2</sub><sup>+</sup> (M + H)<sup>+</sup> 165.0910, found 165.0910; HPLC Chiralpak OD-H (hexane/*i*-PrOH = 97:3, 1.0 mL/min) τ<sub>minor</sub> = 23.8 min, τ<sub>major</sub> = 25.8 min. [α]<sub>D</sub><sup>25</sup> -19.3 (*c* 0.6, MeOH, 90:10 *er*), [α]<sub>D</sub><sup>25</sup> -12.7 (*c* 0.5, CHCl<sub>3</sub>, 90:10 *er*). [α]<sub>D</sub><sup>25</sup> -19.0 (*c* 0.3, CHCl<sub>3</sub>), 91%*ee*, lit.<sup>[4]</sup>

##### (S)-(7-methylchroman-3-yl)methanol (**2b**):



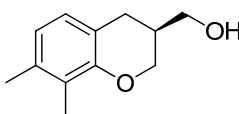
61% yield, colorless crystal. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 6.93 (d, *J* = 7.6 Hz, 1H), 6.67 (d, *J* = 7.6 Hz, 1H), 6.63 (s, 1H), 4.27 (dd, *J* = 10.4, 2.0 Hz, 1H), 4.00 (dd, *J* = 10.4, 8.0 Hz, 1H), 3.71 (dd, *J* = 10.4, 6.0 Hz, 1H), 3.63 (dd, *J* = 10.4, 8.0 Hz, 1H), 2.86 (dd, *J* = 16.0, 6.0 Hz, 1H), 2.55 (dd, *J* = 16.0, 8.0 Hz, 1H), 2.27 (m, 1H); <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>) δ 154.4, 137.2, 129.7, 121.4, 117.8, 116.9, 67.6, 63.5, 35.0, 27.0, 21.0; ESI-MS *m/z* 179.2 (M + H)<sup>+</sup>; HRMS calcd for C<sub>11</sub>H<sub>15</sub>O<sub>2</sub><sup>+</sup> (M + H)<sup>+</sup> 179.1067, found 179.1209; HPLC Chiralpak AD-H (hexane/*i*-PrOH = 95:5, 1.0 mL/min) τ<sub>minor</sub> = 11.4 min, τ<sub>major</sub> = 13.0 min. [α]<sub>D</sub><sup>25</sup> -27.8 (*c* 1.1, MeOH, 93:7 *er*).

##### (S)-(6-methylchroman-3-yl)methanol (**2c**):



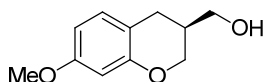
56% yield, yellow oil.  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  6.90 (d,  $J = 8.0$  Hz, 1H), 6.86 (s, 1H), 6.70 (d,  $J = 8.0$  Hz, 1H), 4.27 (dd,  $J = 10.5, 2.0$  Hz, 1H), 4.00 (dd,  $J = 10.5, 7.5$  Hz, 1H), 3.71 (dd,  $J = 10.5, 6.0$  Hz, 1H), 3.65 (dd,  $J = 10.5, 7.5$  Hz, 1H), 2.84 (dd,  $J = 16.0, 6.0$  Hz, 1H), 2.55 (dd,  $J = 16.0, 7.5$  Hz, 1H), 2.25 (s, 3H), 2.25 (m, 1H);  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ )  $\delta$  152.4, 130.3, 129.7, 127.9, 120.6, 116.3, 67.6, 63.5, 34.9, 27.4, 20.4; ESI-MS  $m/z$  179.2 ( $\text{M} + \text{H}$ ) $^+$ ; HRMS calcd for  $\text{C}_{11}\text{H}_{15}\text{O}_2^+$  ( $\text{M} + \text{H}$ ) $^+$  179.1067, found 179.1066; HPLC Chiralpak AD-H (hexane/*i*-PrOH = 97:3, 1.0 mL/min)  $\tau_{\text{minor}} = 21.2$  min,  $\tau_{\text{major}} = 20.1$  min.  $[\alpha]_{\text{D}}^{25} -18.3$  ( $c$  0.7, MeOH, 90:10 *er*).

**(S)-(7,8-dimethylchroman-3-yl)methanol (2d):**



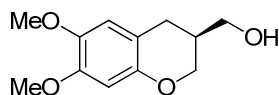
72% yield, white solid.  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  6.80 (d,  $J = 7.5$  Hz, 1H), 6.68 (d,  $J = 7.5$  Hz, 1H), 4.33 (dd,  $J = 10.5, 2.0$  Hz, 1H), 4.31 (dd,  $J = 10.5, 7.5$  Hz, 1H), 3.71 (dd,  $J = 10.5, 6.0$  Hz, 1H), 3.66 (dd,  $J = 10.5, 7.5$  Hz, 1H), 2.85 (dd,  $J = 16.5, 6.0$  Hz, 1H), 2.55 (dd,  $J = 16.5, 7.5$  Hz, 1H), 2.25 (s, 3H), 2.24 (m, 1H), 2.11 (s, 3H);  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ )  $\delta$  152.5, 135.5, 126.6, 124.1, 121.6, 117.8, 67.7, 63.6, 34.9, 27.5, 19.8, 11.7; ESI-MS  $m/z$  193.6 ( $\text{M} + \text{H}$ ) $^+$ ; HRMS calcd for  $\text{C}_{12}\text{H}_{17}\text{O}_2^+$  193.1223, found 193.1222; HPLC Chiralpak AD-H (hexane/*i*-PrOH = 95:5, 1.0 mL/min)  $\tau_{\text{minor}} = 8.4$  min,  $\tau_{\text{major}} = 9.4$  min.  $[\alpha]_{\text{D}}^{25} -5.3$  ( $c$  1.5, MeOH, 70:30 *er*).

**(S)-(7-methoxychroman-3-yl)methanol (2e):**



52% yield, yellow oil.  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  6.94 (d,  $J = 8.5$  Hz, 1H), 6.93 (dd,  $J = 8.5, 2.0$  Hz, 1H), 6.37 (d,  $J = 2.0$  Hz, 1H), 4.28 (dd,  $J = 10.5, 2.0$  Hz, 1H), 3.98 (dd,  $J = 10.5, 8.0$  Hz, 1H), 3.68 (s, 3H), 3.65 (dd,  $J = 10.5, 6.0$  Hz, 1H), 3.62 (dd,  $J = 10.5, 8.0$  Hz, 1H), 2.79 (dd,  $J = 16.0, 6.0$  Hz, 1H), 2.51 (dd,  $J = 16.0, 8.0$  Hz, 1H), 2.23 (m, 1H);  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ )  $\delta$  159.0, 155.2, 130.4, 113.4, 113.0, 107.4, 101.4, 67.7, 63.4, 55.3, 34.9, 26.7; ESI-MS  $m/z$  195.3 ( $\text{M} + \text{H}$ ) $^+$ ; HRMS calcd for  $\text{C}_{11}\text{H}_{15}\text{O}_3^+$  ( $\text{M} + \text{H}$ ) $^+$  195.1016, found 195.1016; HPLC Chiralpak AD-H (hexane/*i*-PrOH = 97:3, 1.0 mL/min)  $\tau_{\text{minor}} = 29.2$  min,  $\tau_{\text{major}} = 32.7$  min.  $[\alpha]_{\text{D}}^{25} -21.5$  ( $c$  1.5, MeOH, 91:9 *er*).

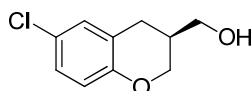
**(S)-(6,7-dimethoxychroman-3-yl)methanol (2f):**



35% yield, white solid.  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  6.52 (s, 1H), 6.37 (s, 1H), 4.23 (dd,  $J =$

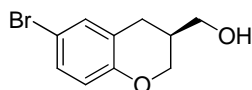
10.5, 2.0 Hz, 1H), 3.92 (dd,  $J = 10.5, 7.5$  Hz, 1H), 3.80 (s, 6H), 3.69 (dd,  $J = 10.5, 6.0$  Hz, 1H), 3.63 (dd,  $J = 10.5, 7.5$  Hz, 1H), 2.78 (dd,  $J = 16.5, 6.0$  Hz, 1H), 2.48 (dd,  $J = 16.5, 7.5$  Hz, 1H), 2.22 (m, 1H);  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ )  $\delta$  148.6, 148.5, 143.5, 113.0, 111.6, 101.0, 67.8, 63.6, 56.7, 56.1, 53.2, 27.1; ESI-MS  $m/z$  225.3 ( $\text{M} + \text{H}^+$ ); HRMS calcd for  $\text{C}_{12}\text{H}_{17}\text{O}_4^+$  ( $\text{M} + \text{H}^+$ )<sup>+</sup> 225.1123, found 225.1122; HPLC Chiralpak OD-H (hexane/*i*-PrOH = 90:10, 1.0 mL/min)  $\tau_{\text{minor}} = 20.9$  min,  $\tau_{\text{major}} = 18.6$  min.  $[\alpha]_{\text{D}}^{25} -26.7$  ( $c$  0.5, MeOH, 89% *ee*).

**(S)-(6-chlorochroman-3-yl)methanol (2g):**



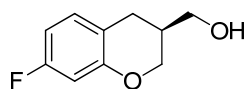
51% yield, white solid.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.02 (d,  $J = 8.8$  Hz, 1H), 7.02 (s, 1H), 6.73 (d,  $J = 8.8$  Hz, 1H), 4.27 (dd,  $J = 10.8, 2.0$  Hz, 1H), 3.98 (dd,  $J = 10.8, 8.0$  Hz, 1H), 3.71 (dd,  $J = 10.8, 6.0$  Hz, 1H), 3.64 (dd,  $J = 10.8, 8.0$  Hz, 1H), 2.85 (dd,  $J = 16.0, 6.0$  Hz, 1H), 2.58 (dd,  $J = 16.0, 8.0$  Hz, 1H), 2.25 (m, 1H);  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ )  $\delta$  153.2, 129.5, 127.3, 125.1, 122.6, 117.9, 67.7, 63.2, 34.4, 27.2; ESI-MS  $m/z$  199.1 ( $\text{M} + \text{H}^+$ ); HRMS calcd for  $\text{C}_{10}\text{H}_{12}\text{ClO}_2^+$  ( $\text{M} + \text{H}^+$ )<sup>+</sup> 199.0520, found 199.0520; HPLC Chiralpak OD-H (hexane/*i*-PrOH = 97:3, 1.0 mL/min)  $\tau_{\text{minor}} = 27.5$  min,  $\tau_{\text{major}} = 29.3$  min.  $[\alpha]_{\text{D}}^{25} -19.6$  ( $c$  0.5, MeOH, 90.5:9.5 *er*).

**(S)-(6-bromochroman-3-yl)methanol (2h):**



53% yield, colorless crystal.  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.16-7.17 (m, 2H), 6.67 (d,  $J = 9.0$  Hz, 1H), 4.27 (dd,  $J = 10.5, 2.0$  Hz, 1H), 3.98 (dd,  $J = 10.5, 7.5$  Hz, 1H), 3.67 (dd,  $J = 10.5, 6.0$  Hz, 1H), 3.63 (dd,  $J = 10.5, 7.5$  Hz, 1H), 2.83 (dd,  $J = 16.5, 6.0$  Hz, 1H), 2.55 (dd,  $J = 16.5, 7.5$  Hz, 1H), 2.23 (m, 1H);  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ )  $\delta$  153.7, 132.4, 130.1, 123.2, 118.3, 112.4, 67.6, 63.1, 34.4, 27.1; ESI-MS  $m/z$  243.0 ( $\text{M} + \text{H}^+$ ); HRMS calcd for  $\text{C}_{10}\text{H}_{12}\text{BrO}_2^+$  ( $\text{M} + \text{H}^+$ )<sup>+</sup> 243.0015, found 243.0011; HPLC Chiralpak AD-H (hexane/*i*-PrOH = 97:3, 1.0 mL/min)  $\tau_{\text{minor}} = 23.8$  min,  $\tau_{\text{major}} = 25.2$  min.  $[\alpha]_{\text{D}}^{25} -9.8$  ( $c$  0.75, MeOH, 87.5:12.5 *er*).

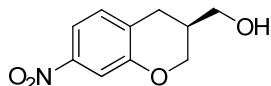
**(S)-(7-fluorochroman-3-yl)methanol (2i):**



63% yield, yellow oil.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  6.97 (t,  $J = 7.6$  Hz, 1H), 6.51-6.59 (m, 2H), 4.29 (dd,  $J = 10.4, 2.0$  Hz, 1H), 3.98 (dd,  $J = 10.4, 8.0$  Hz, 1H), 3.71 (dd,  $J = 10.4, 6.0$  Hz, 1H), 3.65 (dd,  $J = 10.4, 8.0$  Hz, 1H), 2.83 (dd,  $J = 16.0, 6.0$  Hz, 1H), 2.53 (dd,  $J = 16.0, 8.0$  Hz, 1H), 2.24 (m, 1H);  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ )  $\delta$  161.9, 155.3, 130.6, 116.6, 107.6, 103.7, 67.8, 63.3, 34.6, 26.8; ESI-MS  $m/z$  183.7 ( $\text{M} + \text{H}^+$ ); HRMS calcd for  $\text{C}_{10}\text{H}_{12}\text{FO}_2^+$  ( $\text{M} + \text{H}^+$ )<sup>+</sup> 183.0816, found

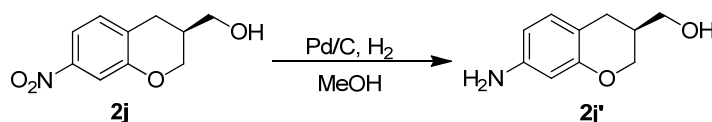
183.0817; HPLC Chiralpak OD-H (hexane/*i*-PrOH = 97:3, 1.0 mL/min)  $\tau_{\text{minor}} = 20.6$  min,  $\tau_{\text{major}} = 21.9$  min.  $[\alpha]_{\text{D}}^{25} -23.2$  (*c* 1.0, MeOH, 89:11 *er*).

**(S)-(7-nitrochroman-3-yl)methanol (2j):**



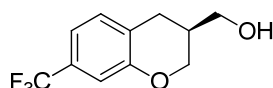
57% yield, yellow solid.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.70 (d,  $J = 8.4$  Hz, 1H), 7.66 (s, 1H), 7.18 (d,  $J = 8.4$  Hz, 1H), 4.36 (dd,  $J = 10.6, 2.0$  Hz, 1H), 4.01 (dd,  $J = 10.6, 8.0$  Hz, 1H), 3.73 (dd,  $J = 10.6, 6.0$  Hz, 1H), 3.68 (dd,  $J = 10.6, 8.0$  Hz, 1H), 2.95 (dd,  $J = 16.0, 6.0$  Hz, 1H), 2.70 (dd,  $J = 16.0, 8.0$  Hz, 1H), 2.31 (m, 1H);  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ )  $\delta$  154.9, 147.3, 130.4, 128.8, 115.1, 112.0, 68.0, 62.9, 34.0, 27.5 ; ESI-MS  $m/z$  210.1 ( $\text{M} + \text{H}$ ) $^+$ ; HRMS calcd for  $\text{C}_{10}\text{H}_{12}\text{NO}_4^+$  210.0762, found 210.0761.

**(S)-(7-aminochroman-3-yl)methanol (2j')**



Compound **2j'** was prepared according to similar route of literature reported methods<sup>[5]</sup>. In a round bottom flask was charged with compound **2j** (32 mg, 0.15mmol ), Pd/C (15 mg ) and MeOH (2.5 mL ). The mixture was stirred under the  $\text{H}_2$  at 30 °C for 5 hours. After carefully venting hydrogen, most of the reaction solvent was removed under reduced pressure. The residue was loaded on silica gel column and purified by flash chromatography (ethyl acetate/petroleum ether = 1/3 to 1/1) to afford the pure product **2j'** (21 mg, 79%) as a reddish brown solid.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  6.81 (d,  $J = 8.0$  Hz, 1H), 6.23 (d,  $J = 8.0$  Hz, 1H), 6.16 (s, 1H), 4.24 (dd,  $J = 10.6, 2.0$  Hz, 1H), 3.92 (dd,  $J = 10.6, 8.0$  Hz, 1H), 3.68 (dd,  $J = 10.6, 6.0$  Hz, 1H), 3.62 (dd,  $J = 10.6, 8.0$  Hz, 1H), 2.89 (br s, 2H), 2.75 (dd,  $J = 16.0, 6.0$  Hz, 1H), 2.46 (dd,  $J = 16.0, 8.0$  Hz, 1H), 2.23 (m, 1H); ESI-MS  $m/z$  180.0 ( $\text{M} + \text{H}$ ) $^+$ ; HRMS calcd for  $\text{C}_{10}\text{H}_{14}\text{NO}_2^+$  ( $\text{M} + \text{H}$ ) $^+$  180.1019, found 180.1017; HPLC Chiralpak AD-H (hexane/*i*-PrOH = 85:15, 1.0 mL/min)  $\tau_{\text{minor}} = 20.2$  min,  $\tau_{\text{major}} = 22.0$  min.  $[\alpha]_{\text{D}}^{25} -22.5$  (*c* 0.5, MeOH, 81.6:18.4 *er*).

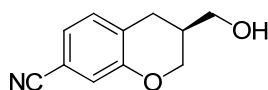
**(S)-(7-(trifluoromethyl)chroman-3-yl)methanol (2k):**



60% yield, colorless oil.  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.20 (d,  $J = 8.0$  Hz, 1H), 7.07 (d,  $J = 8.0$  Hz, 1H), 7.05 (s, 1H), 4.32 (dd,  $J = 10.5, 2.0$  Hz, 1H), 4.03 (dd,  $J = 10.5, 7.5$  Hz, 1H), 3.71 (dd,  $J = 10.5, 6.0$  Hz, 1H), 3.63 (dd,  $J = 10.5, 7.5$  Hz, 1H), 2.91 (dd,  $J = 16.5, 6.0$  Hz, 1H), 2.62 (dd,  $J = 16.5, 7.5$  Hz, 1H), 2.23 (m, 1H);  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ )  $\delta$  154.6, 130.5, 129.6, 125.0, 122.9, 116.8, 113.8, 67.8, 63.1, 34.3, 27.3; ESI-MS  $m/z$  233.3 ( $\text{M} + \text{H}$ ) $^+$ ; HRMS calcd for  $\text{C}_{11}\text{H}_{12}\text{F}_3\text{O}_2^+$  ( $\text{M} + \text{H}$ ) $^+$  233.0784, found 233.0783; HPLC Chiralpak AD-H (hexane/*i*-PrOH = 95:5, 1.0 mL/min)

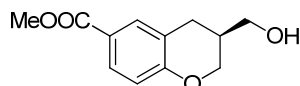
$\tau_{\text{minor}} = 9.5 \text{ min}$ ,  $\tau_{\text{major}} = 10.3 \text{ min}$ .  $[\alpha]_{\text{D}}^{25} -22.2$  (*c* 1.5, MeOH, 87:13 *er*).

**(S)-3-(hydroxymethyl)chroman-7-carbonitrile (2l):**



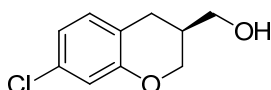
70% yield, white solid.  $^1\text{H NMR}$  (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.12 (m, 2H), 7.07 (s, 1H), 4.33 (dd, *J* = 10.5, 2.0 Hz, 1H), 4.07 (dd, *J* = 10.5, 8.0 Hz, 1H), 3.86 (s, 3H), 3.71 (dd, *J* = 10.5, 6.0 Hz, 1H), 3.63 (dd, *J* = 10.5, 8.0 Hz, 1H), 2.90 (dd, *J* = 16.0, 6.0 Hz, 1H), 2.62 (dd, *J* = 16.0, 8.0 Hz, 1H), 2.27 (m, 1H);  $^{13}\text{C NMR}$  (125 MHz,  $\text{CDCl}_3$ )  $\delta$  167.0, 158.7, 132.1, 129.1, 122.3, 120.8, 166.6, 68.1, 63.1, 51.8, 34.4, 27.3; ESI-MS *m/z* 190.2 (*M* + *H*) $^+$ ; HRMS calcd for  $\text{C}_{11}\text{H}_{12}\text{NO}_2^+$  (*M* + *H*) $^+$  190.0863, found 190.0864; HPLC Chiralpak AD-H (hexane/*i*-PrOH = 90:10, 1.0 mL/min)  $\tau_{\text{minor}} = 15.9 \text{ min}$ ,  $\tau_{\text{major}} = 16.5 \text{ min}$ .  $[\alpha]_{\text{D}}^{25} -36.7$  (*c* 0.9, MeOH, 89:10 *er*).

**(S)-methyl 3-(hydroxymethyl)chroman-6-carboxylate (2m):**



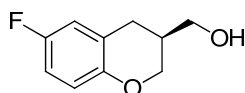
45% yield, yellow oil.  $^1\text{H NMR}$  (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.77 (s, 1H), 7.76 (d, *J* = 9.0 Hz, 1H), 6.81 (d, *J* = 9.0 Hz, 1H), 4.35 (dd, *J* = 10.5, 2.0 Hz, 1H), 4.03 (dd, *J* = 10.5, 7.5 Hz, 1H), 3.71 (dd, *J* = 10.5, 6.0 Hz, 1H), 3.63 (dd, *J* = 10.5, 7.5 Hz, 1H), 2.91 (dd, *J* = 16.5, 6.0 Hz, 1H), 2.62 (dd, *J* = 16.5, 7.5 Hz, 1H), 2.23 (m, 1H);  $^{13}\text{C NMR}$  (125 MHz,  $\text{CDCl}_3$ )  $\delta$  154.8, 130.9, 127.0, 123.8, 120.3, 118.7, 110.8, 67.9, 63.0, 34.1, 27.5; ESI-MS *m/z* 223.4 (*M* + *H*) $^+$ ; HRMS calcd for  $\text{C}_{12}\text{H}_{15}\text{O}_4^+$  (*M* + *H*) $^+$  223.0965, found 223.0961; HPLC Chiralpak AD-H (hexane/*i*-PrOH = 90:10, 1.0 mL/min)  $\tau_{\text{minor}} = 12.7 \text{ min}$ ,  $\tau_{\text{major}} = 13.5 \text{ min}$ .  $[\alpha]_{\text{D}}^{25} -13.0$  (*c* 0.75, MeOH, 73.6:26.4 *er*).

**(S)-(7-chlorochroman-3-yl)methanol (2n):**



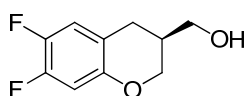
44% yield, white solid.  $^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ )  $\delta$  6.95 (d, *J* = 8.4 Hz, 1H), 6.82 (d, *J* = 8.4 Hz, 1H), 6.80 (s, 1H), 4.26 (dd, *J* = 10.8, 2.0 Hz, 1H), 3.98 (dd, *J* = 10.8, 8.4 Hz, 1H), 3.70 (dd, *J* = 10.8, 5.6 Hz, 1H), 3.65 (dd, *J* = 10.8, 8.4 Hz, 1H), 2.83 (dd, *J* = 16.0, 5.6 Hz, 1H), 2.55 (dd, *J* = 16.0, 8.4 Hz, 1H), 2.24 (m, 1H);  $^{13}\text{C NMR}$  (125 MHz,  $\text{CDCl}_3$ )  $\delta$  155.1, 132.2, 130.8, 120.6, 119.5, 116.7, 67.7, 63.2, 34.5, 26.9; ESI-MS *m/z* 268.1 (*M* + *H*) $^+$ ; ESI-MS *m/z* 199.1 (*M* + *H*) $^+$ ; HRMS calcd for  $\text{C}_{10}\text{H}_{12}\text{ClO}_2^+$  (*M* + *H*) $^+$  199.0520, found 199.0520; HPLC Chiralpak AD-H (hexane/*i*-PrOH = 95:5, 1.0 mL/min)  $\tau_{\text{minor}} = 12.6 \text{ min}$ ,  $\tau_{\text{major}} = 13.6 \text{ min}$ .  $[\alpha]_{\text{D}}^{25} -17.0$  (*c* 1.0, MeOH, 84:16 *er*).

**(S)-(6-fluorochroman-3-yl)methanol (2o):**



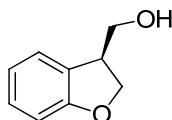
64% yield, yellow oil.  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  6.71 - 6.79 (m, 3H), 4.24 (dd,  $J = 11.0, 2.0$  Hz, 1H), 3.98 (dd,  $J = 11.0, 7.5$  Hz, 1H), 3.68 (dd,  $J = 11.0, 6.0$  Hz, 1H), 3.65 (dd,  $J = 11.0, 7.5$  Hz, 1H), 2.83 (dd,  $J = 16.5, 6.0$  Hz, 1H), 2.55 (dd,  $J = 16.5, 7.5$  Hz, 1H), 2.23 (m, 1H);  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ )  $\delta$  156.8, 150.5, 112.1, 117.4, 115.7, 113.9, 67.6, 63.1, 34.5, 27.4; ESI-MS  $m/z$  268.1 ( $\text{M} + \text{H}$ ) $^+$ ; ESI-MS  $m/z$  183.7 ( $\text{M} + \text{H}$ ) $^+$ ; HRMS calcd for  $\text{C}_{10}\text{H}_{12}\text{FO}_2$  $^+$  ( $\text{M} + \text{H}$ ) $^+$  183.0816, found 183.0817; HPLC Chiralpak AD-H (hexane/*i*-PrOH = 97:3, 1.0 mL/min)  $\tau_{\text{minor}} = 23.6$  min,  $\tau_{\text{major}} = 25.6$  min.  $[\alpha]_{\text{D}}^{25} -4.0$  (*c* 0.8, MeOH, 86:14 *er*).

**(S)-(6,7-difluorochroman-3-yl)methanol (2p):**



43% yield, yellow oil.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  6.81 (t,  $J = 9.6$  Hz, 1H), 6.60 (t,  $J = 9.6$  Hz, 1H), 4.24 (d,  $J = 10.6$  Hz, 1H), 3.96 (dd,  $J = 10.6, 8.0$  Hz, 1H), 3.68 (dd,  $J = 10.6, 5.6$  Hz, 1H), 3.63 (dd,  $J = 10.6, 8.0$  Hz, 1H), 2.80 (dd,  $J = 16.4, 5.6$  Hz, 1H), 2.52 (dd,  $J = 16.4, 8.0$  Hz, 1H), 2.22 (m, 1H);  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ )  $\delta$  150.3, 149.8, 148.9, 144.7, 117.1, 116.6, 67.6, 63.0, 34.3, 26.7; ESI-MS  $m/z$  201.6 ( $\text{M} + \text{H}$ ) $^+$ ; HRMS calcd for  $\text{C}_{10}\text{H}_{11}\text{F}_2\text{O}_2$  $^+$  ( $\text{M} + \text{H}$ ) $^+$  201.0722, found 201.0716; HPLC Chiralpak AD-H (hexane/*i*-PrOH = 97:3, 1.0 mL/min)  $\tau_{\text{minor}} = 21.6$  min,  $\tau_{\text{major}} = 22.7$  min.  $[\alpha]_{\text{D}}^{20} -20.3$  (*c* 0.5, MeOH, 88.6:11.4 *er*).

**(S)-(2,3-dihydrobenzofuran-3-yl)methanol (4):**

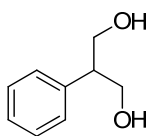


60% yield, yellow oil.  $^1\text{H}$  NMR (500 MHz,  $\text{CHCl}_3$ )  $\delta$  7.22 (d,  $J = 7.5$  Hz, 1H), 7.15 (t,  $J = 7.5$  Hz, 1H), 6.87 (t,  $J = 7.5$  Hz, 1H), 6.81 (d,  $J = 7.5$  Hz, 1H), 4.62 (t,  $J = 5.5$  Hz, 1H), 4.46 (t,  $J = 5.5$  Hz, 1H), 3.78 (m, 2H), 3.62 (m, 1H);  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ )  $\delta$  160.4, 128.8, 127.0, 124.6, 120.4, 109.7, 64.9, 44.5; ESI-MS  $m/z$  151.1 ( $\text{M} + \text{H}$ ) $^+$ ; HRMS calcd for  $\text{C}_9\text{H}_{11}\text{O}_2$  $^+$  ( $\text{M} + \text{H}$ ) $^+$  151.0754, found 151.0751. HPLC Chiralpak AD-H (hexane/*i*-PrOH = 97:3, 1.0 mL/min)  $\tau_{\text{minor}} = 19.0$  min,  $\tau_{\text{major}} = 17.6$  min.  $[\alpha]_{\text{D}}^{25} -9.9$  (*c* 0.75, MeOH, 74.7:25.3 *er*).  $[\alpha]_{\text{D}}^{24} -17.6$  (93%*ee*) lit.<sup>[6]</sup>

**5. Determination of Byproducts:**

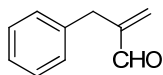
Two byproducts were identified in these reactions as dehalogenation and  $\beta$ -H elimination/dehydroxylation byproducts.

**2-phenylpropane-1, 3-diol (11):**



colorless oil.  $^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.11-7.23 (m, 5H), 3.74 (dd,  $J = 13.8, 2.8$  Hz, 3H), 3.61 (dd,  $J = 13.8, 7.6$  Hz, 1H), 2.56 (d,  $J = 7.6$  Hz, 1H), 2.00 (m, 1H); ESI-MS  $m/z$  153.1 ( $\text{M} + \text{H}$ ) $^+$ .

**2-benzylacrylaldehyde (12):**



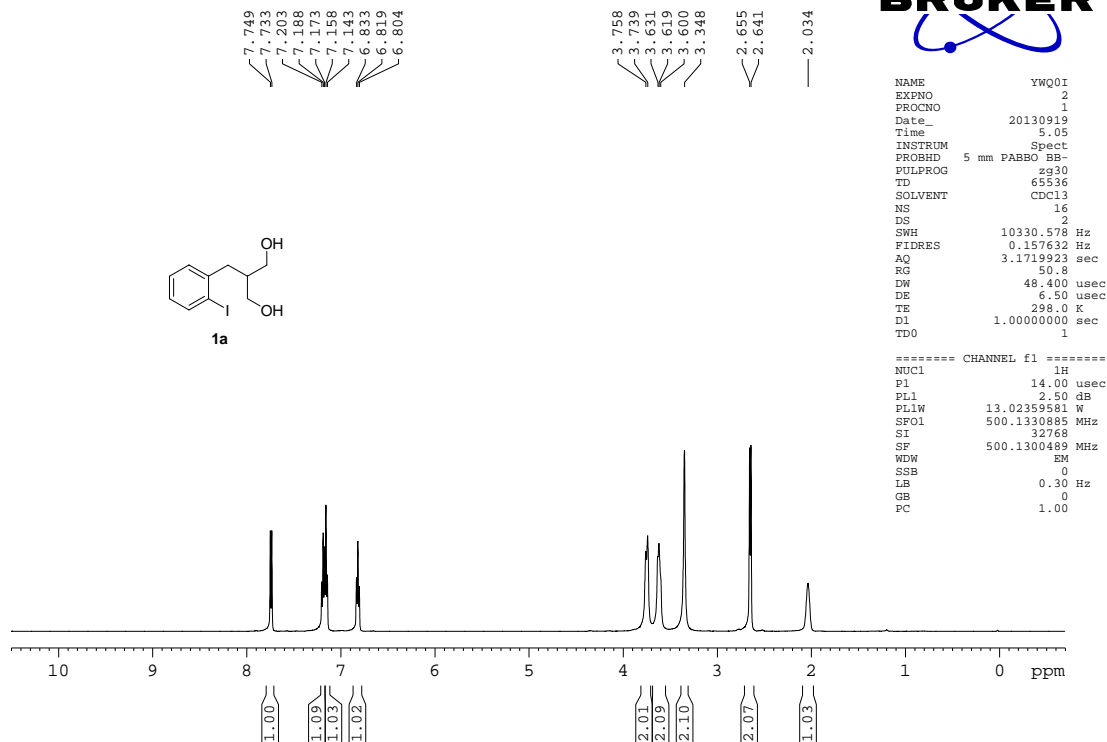
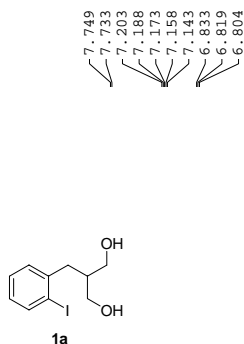
colorless oil.  $^1\text{H NMR}$  (400 MHz,  $\text{CD Cl}_3$ )  $\delta$  9.6 (s, 1H), 7.17-7.31 (m, 5H), 6.08 (d,  $J = 13.8$  Hz, 2H), 3.35 (s, 2H); ESI-MS  $m/z$  147.1 ( $\text{M} + \text{H}$ ) $^+$ .

**6. References:**

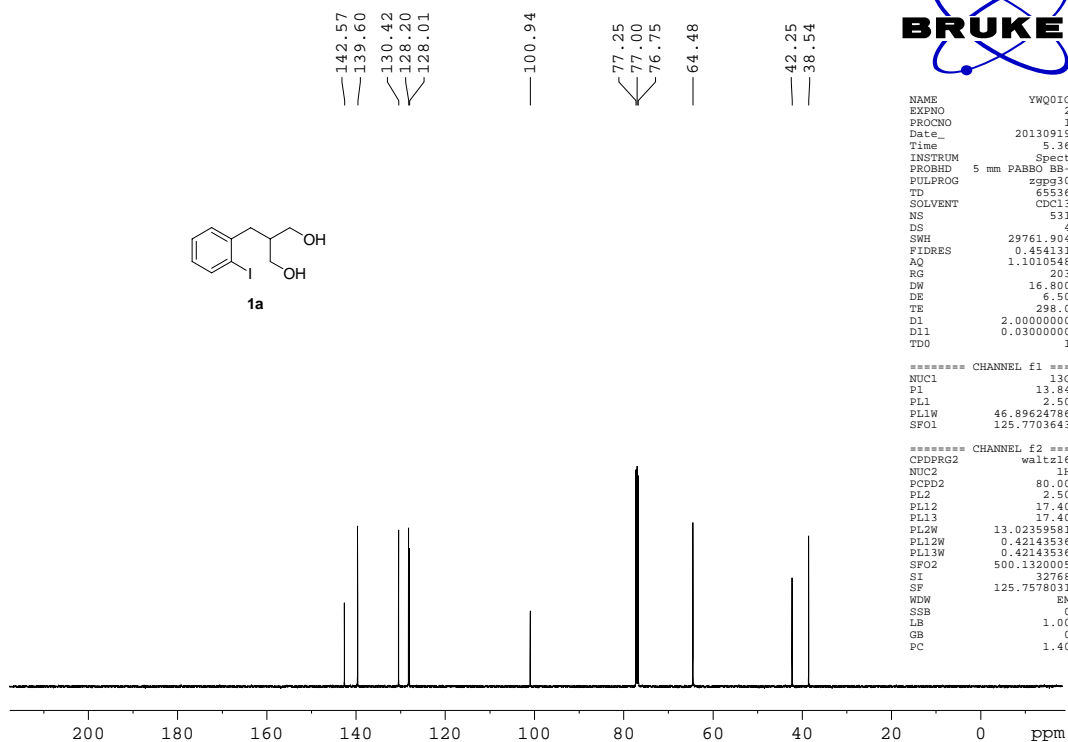
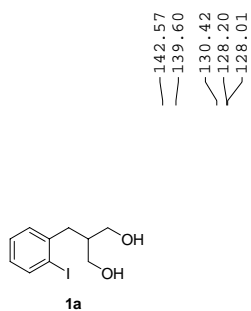
- [1] Jianhua Xie, Lixin Wang; Qilin Zhou, et al.; *J. Am. Chem. Soc.*, 2003, 125(15), 4404-4405.
- [2] Punit P. Seth, Alycia Miyaji, Richard H. Griffey, et al.; *J. Med. Chem.*, 2005, 48(23), 7099-7102.
- [3] Egle M. Beccalli, Concetta La Rosa, Alessandro Marchesini, et al.; *J. Org. Chem.*, 1984, 49(22), 4287-4290.
- [4] Elisabetta Brenna, Francesco G. Gatti, Alessandro Sacchetti, et al.; *J. Org. Chem.*, 2013, 78, 4811- 4822.
- [5] Fengwei Zhang, Jun Jin, Jiantai Ma, et al.; *Green Chem.*, 2011, 13, 1238-1243.
- [6] Rebecca N. Loy and Eric N. Jacobsen.; *J. Am. Chem. Soc.*, 2009, 131(8), 2786-2787.

# 7. <sup>1</sup>H NMR and <sup>13</sup>C NMR Spectra:

YWQ01H



YWQ01H



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NAME          YWQ01C
EXPNO         2
PROCNO        1
Date_         20130919
Time          5.36
INSTRUM       Spect
PROBHD        5 mm PABBO BB-
PULPROG       zgpg30
TD            65536
SOLVENT       CDCl3
NS            531
DS            4
SWH           29761.904 Hz
FIDRES        0.454131 Hz
AQ            1.1010548 sec
RG            203
DW            16.800 usec
DE            6.50 usec
TE            298.0 K
D1            2.0000000 sec
D11           0.0300000 sec
TD0           1
    
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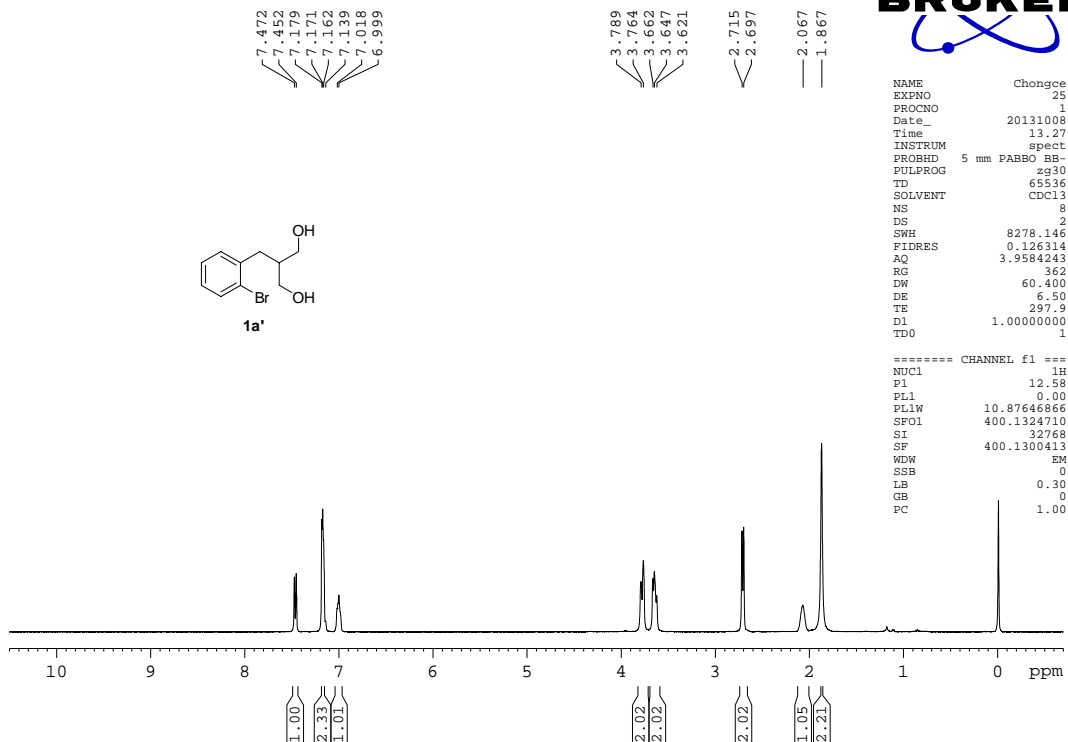
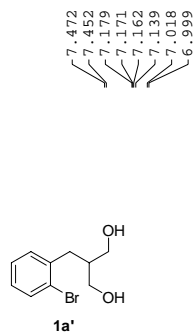
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***** CHANNEL f1 *****
NUC1          13C
P1            13.84 usec
PL1           2.50 dB
PL1W          46.89624786 W
SFO1          125.7703643 MHz
    
```

```

***** CHANNEL F2 *****
CPDPRG2       waltz16
NUC2          1H
PCPD2         80.00 usec
PL2           2.50 dB
PL12          17.40 dB
PL13          17.40 dB
PL2W          13.02359581 W
PL12W         0.42143536 W
PL13W         0.42143536 W
SFO2          500.1320005 MHz
SI            32768
SF            125.7576831 MHz
WDW           EM
SSB           0
LB            1.00 Hz
GB            0
PC            1.40
    
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Y0021



```

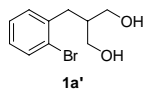
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PROCNO        1
Date_         20131008
Time          13.27
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PROBHD        5 mm PABBO BB-
PULPROG       zg30
TD            65536
SOLVENT       CDCl3
NS            8
DS            2
SWH           8278.146 Hz
FIDRES        0.126314 Hz
AQ            3.9584243 sec
RG            362
DW            60.400 usec
DE            6.50 usec
TE            297.9 Hz
D1            1.0000000 sec
TD0           1
    
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```

***** CHANNEL f1 *****
NUC1          1H
P1            12.58 usec
PL1           0.00 dB
PL1W          10.87646866 W
SFO1          400.1324710 MHz
SI            32768
SF            400.1300413 MHz
WDW           EM
SSB           0
LB            0.30 Hz
GB            0
PC            1.00
    
```

YWQ0020

139.35  
132.88  
131.30  
127.87  
127.33  
124.70  
  
77.26  
77.00  
76.75  
64.44  
  
42.26  
34.19



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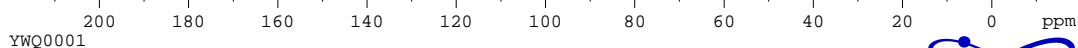
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PROCNO        1
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Time          23.26
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PULPROG       zgpg30
TD            32768
SOLVENT       CDCl3
NS            1024
DS            4
SWH           29761.904 Hz
FIDRES       0.908261 Hz
AQ           0.5505524 sec
RG           203
DW           16.800 usec
DE           6.50 usec
TE           300.7 K
D1           2.0000000 sec
D11          0.0300000 sec
TD0          1
  
```

```

===== CHANNEL f1 =====
NUC1          13C
P1           11.66 usec
PL1          0.00 dB
PL1W         83.39463043 W
SFO1         125.7703643 MHz
  
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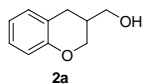
===== CHANNEL F2 =====
CPDPRG2      waltz16
NUC2          1H
PCPD2        80.00 usec
PL2          2.50 dB
PL12         17.40 dB
PL13         17.40 dB
PL2W         13.02359581 W
PL12W        0.42143536 W
PL13W        0.42143536 W
SFO2         500.1320005 MHz
SI           32768
SF           125.7578005 MHz
WDW          EM
SSB          0
LB           1.00 Hz
GB           0
PC           1.40
  
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YWQ0001



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7.095  
7.079  
7.059  
7.044  
6.872  
6.857  
6.843  
6.822  
6.805  
4.306  
4.303  
4.301  
4.285  
4.282  
4.280  
4.020  
4.005  
3.999  
3.984  
3.715  
3.703  
3.693  
3.681  
3.658  
3.643  
3.637  
3.621  
2.897  
2.885  
2.864  
2.853  
2.609  
2.593  
2.576  
2.561  
2.286  
2.271  
2.259  
2.244  
2.155



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NAME          YWQ0001
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PROCNO        1
Date_         20130730
Time          16.55
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PULPROG       zg30
TD            65536
SOLVENT       CDCl3
NS            8
DS            0
SWH           10330.578 Hz
FIDRES       0.157632 Hz
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RG           50.8
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DE           6.50 usec
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TD0          1
  
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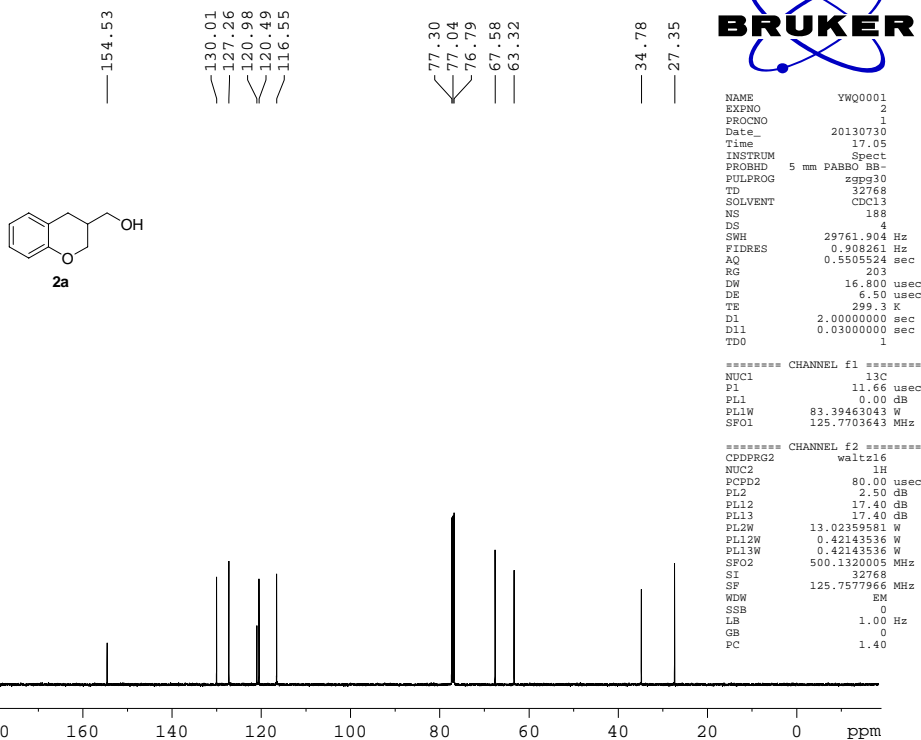
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P1           14.00 usec
PL1          2.50 dB
PL1W         13.02359581 W
SFO1         500.1330885 MHz
SI           32768
SF           500.1300227 MHz
WDW          EM
SSB          0
LB           0.30 Hz
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PC           1.00
  
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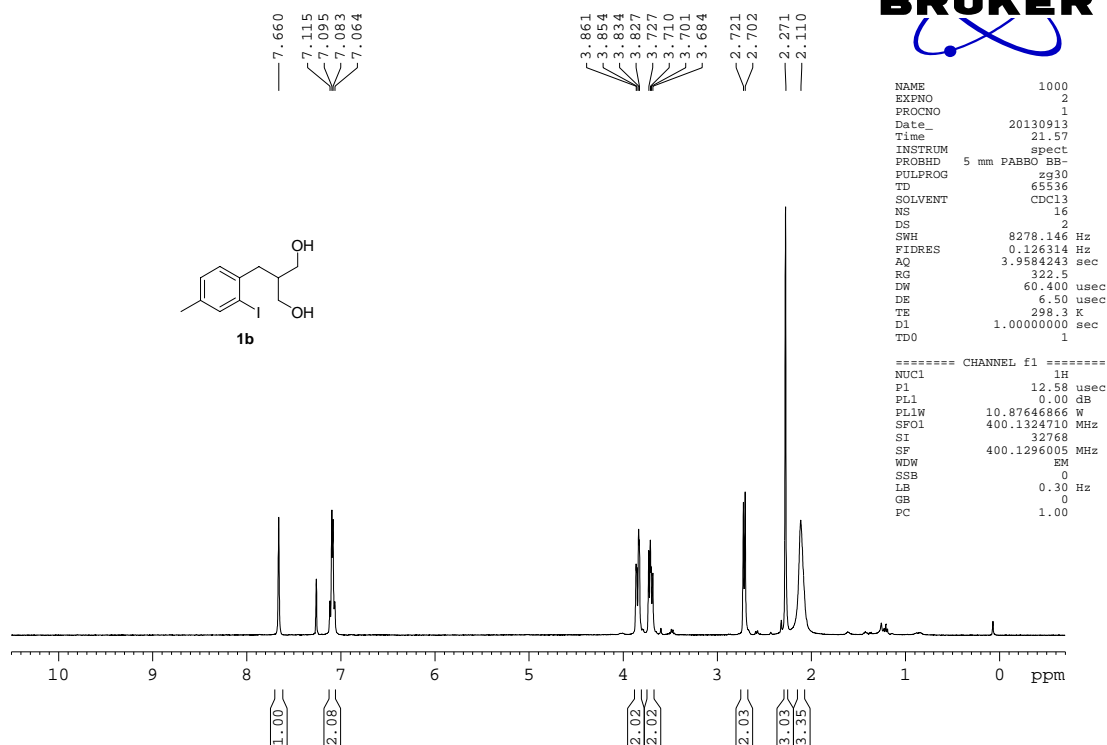


1.07  
1.00  
1.06  
0.97  
  
1.05  
1.03  
1.10  
1.04  
  
1.05  
1.05  
1.07

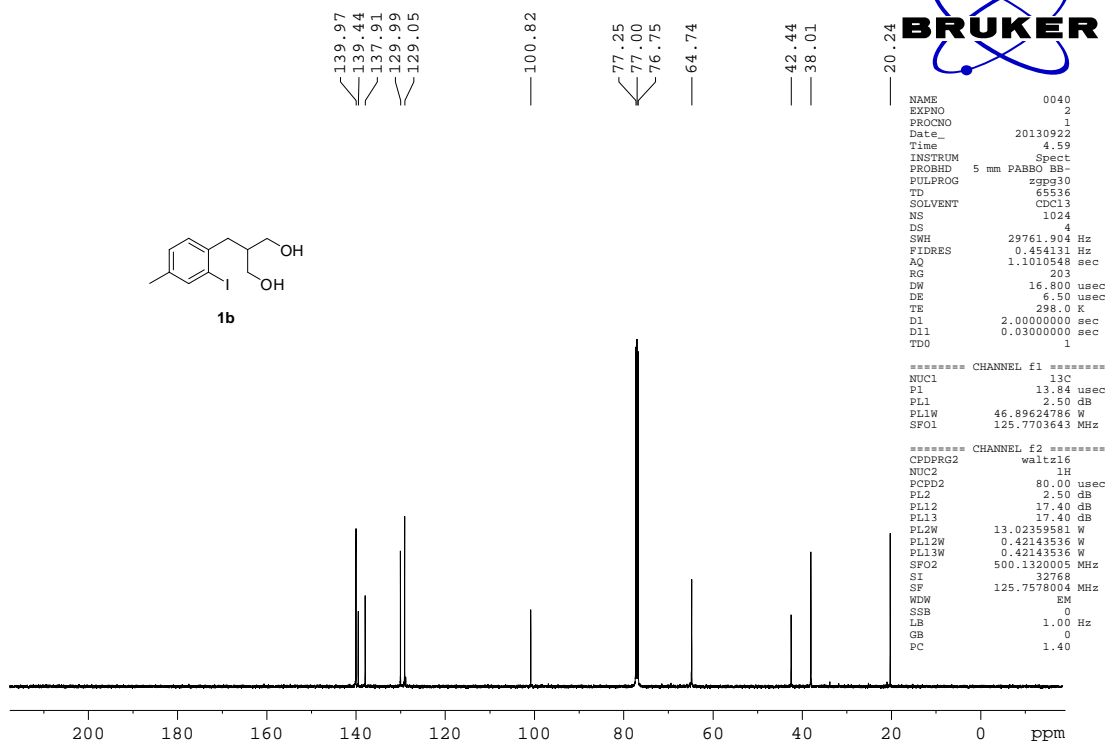
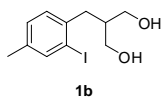
YWQ0001



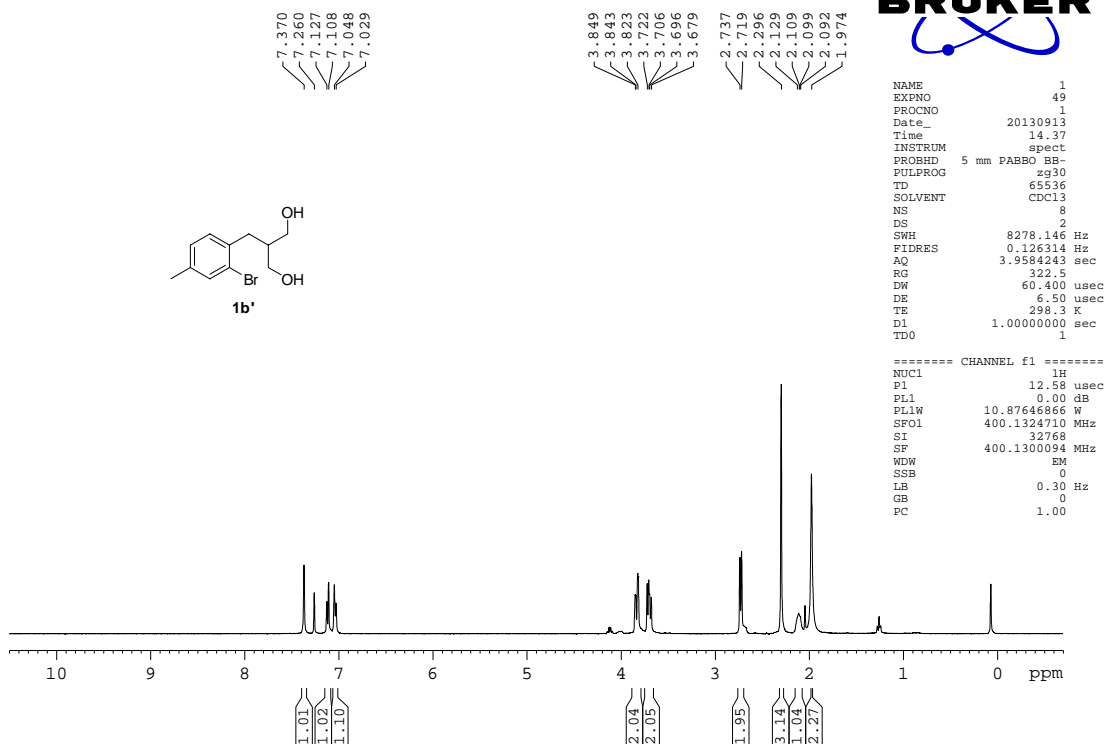
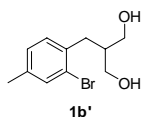
YWQ-0040



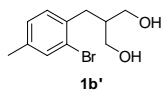
0040



LY-113554



YWQ0042C



137.88  
136.11  
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130.96  
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124.42

77.25  
77.00  
76.74

64.82  
42.36  
33.69

20.50



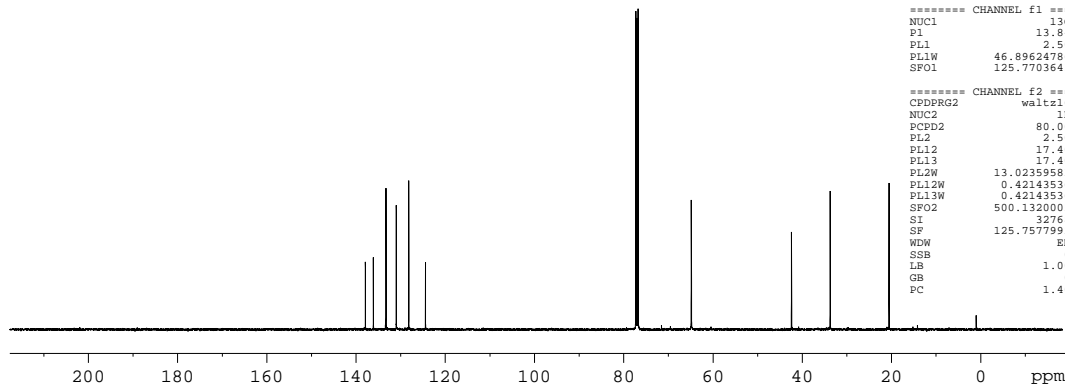
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EXPNO 1
PROCNO 1
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Time 10.53
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PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010548 sec
RG 203
DW 16.800 usec
DE 6.50 usec
TE 298.1 K
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D11 0.0300000 sec
TD0 1
  
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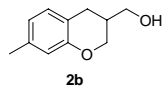
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***** CHANNEL f1 *****
NUC1 13C
P1 13.84 usec
PL1 2.50 dB
PL1W 46.8924786 W
SFO1 125.7703643 MHz

***** CHANNEL F2 *****
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 2.50 dB
PL12 17.40 dB
PL13 17.40 dB
PL2W 13.02359581 W
PL12W 0.42143536 W
PL13W 0.42143536 W
SFO2 500.1320005 MHz
SI 32768
SF 125.7577992 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
  
```



0040-P



7.258  
6.941  
6.922  
6.684  
6.665  
6.633

4.289  
4.263  
4.005  
3.985  
3.960  
3.728  
3.713  
3.702  
3.687  
3.671  
3.651  
3.625  
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2.271  
1.649

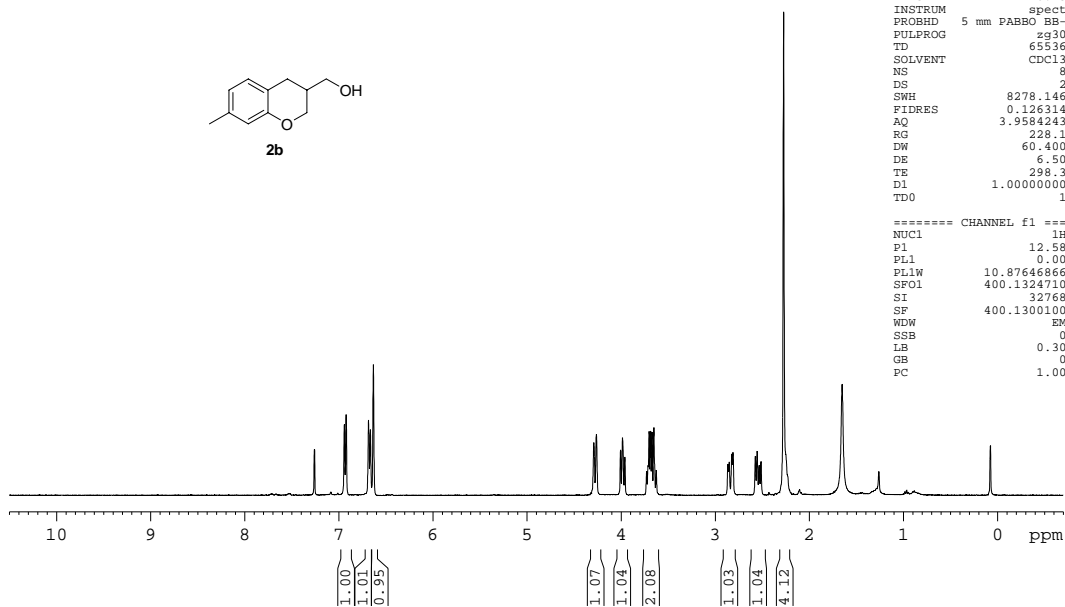


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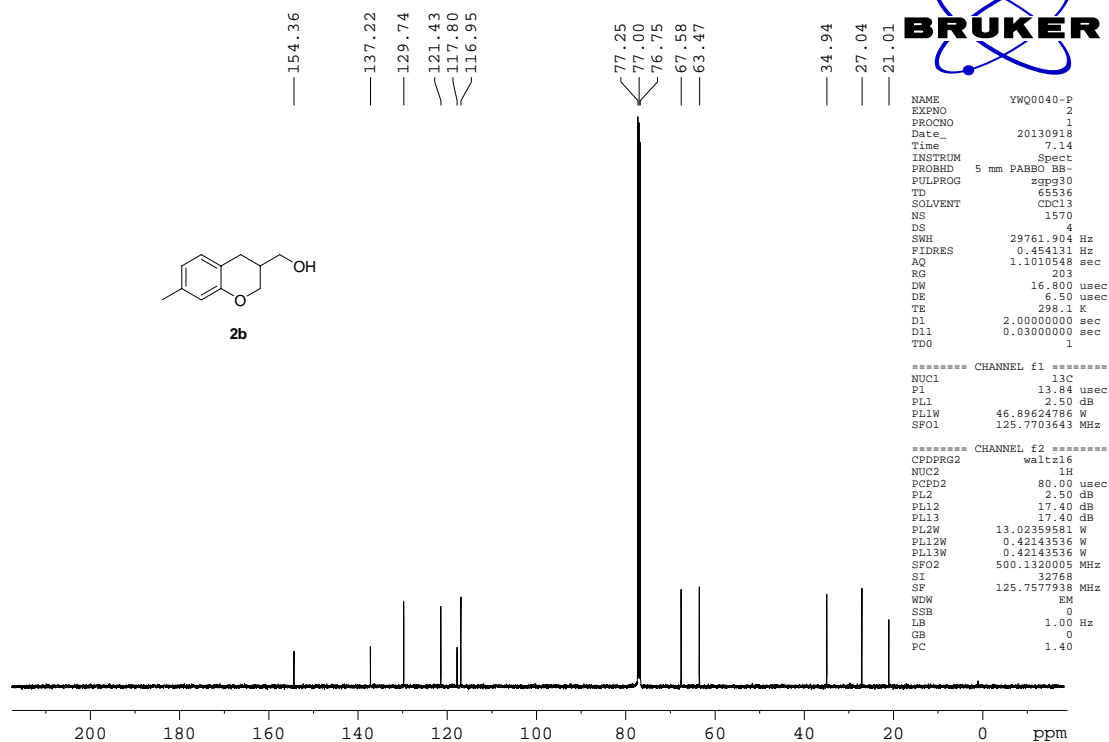
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PROCNO 1
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DS 2
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FIDRES 0.126314 Hz
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TD0 1
  
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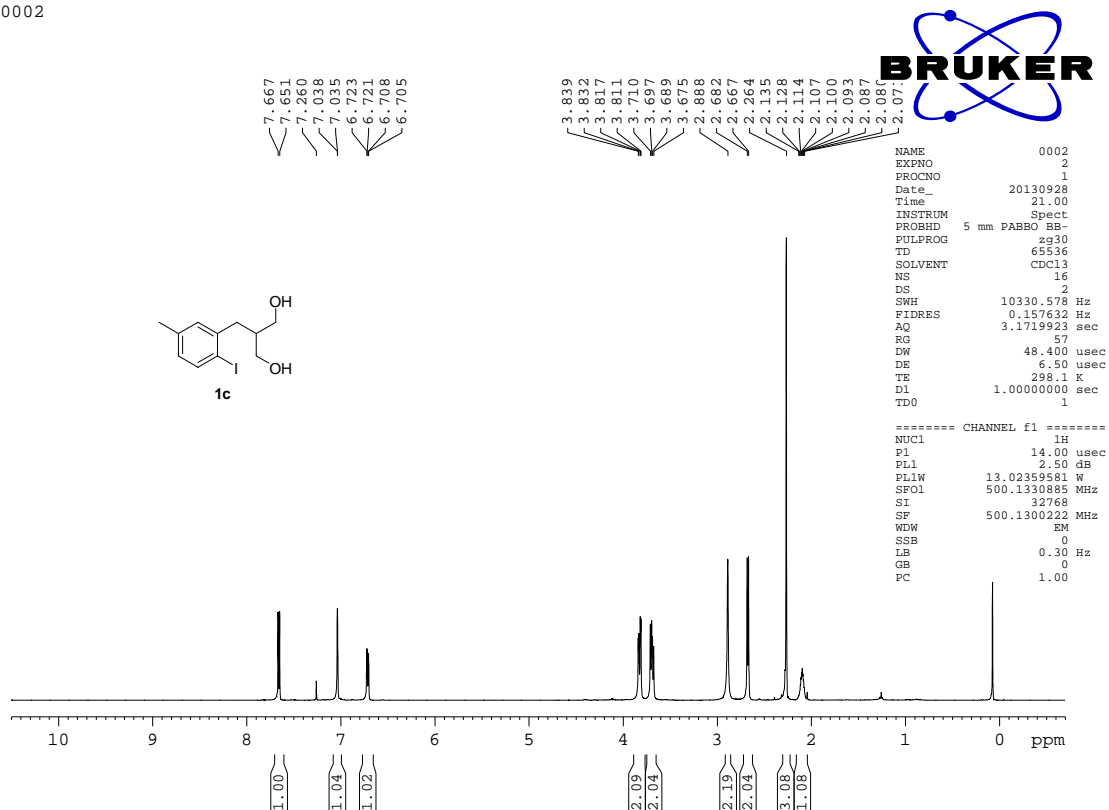
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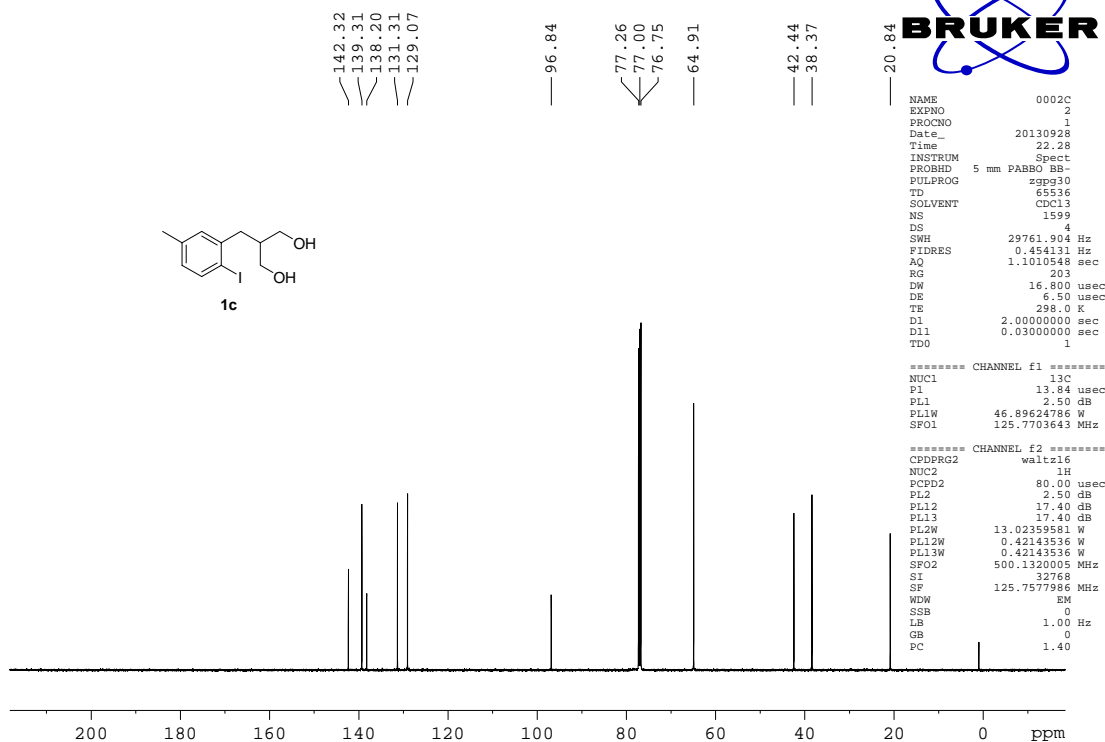
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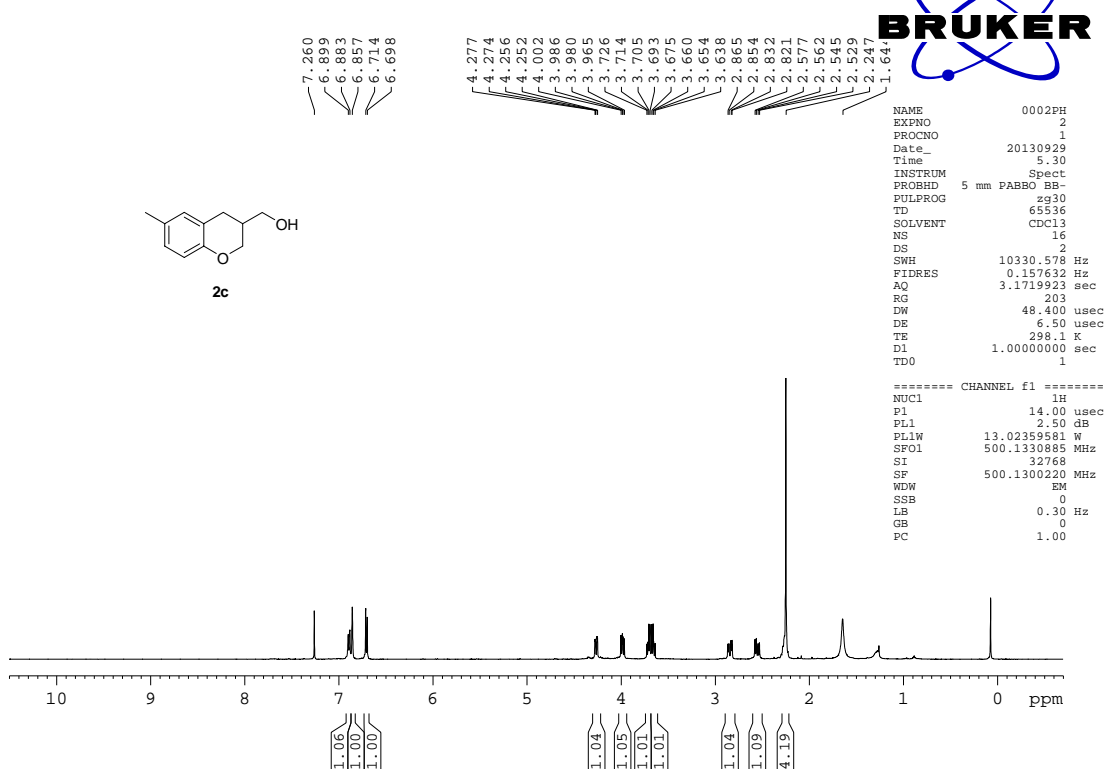
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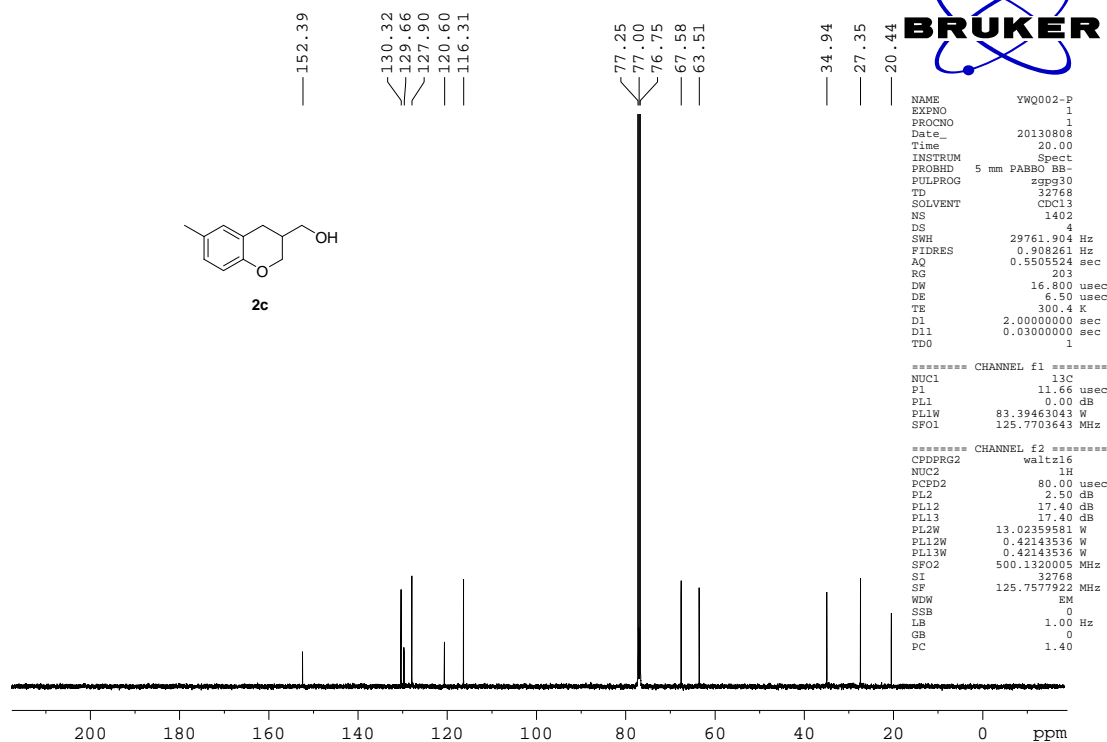
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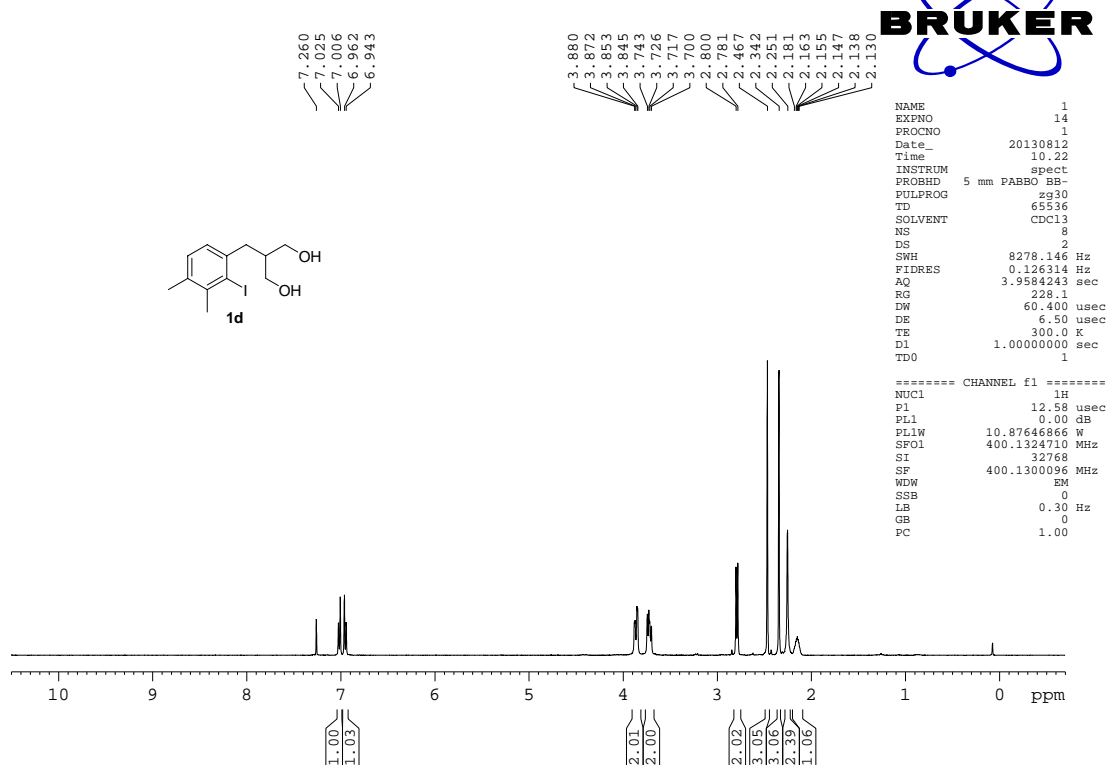
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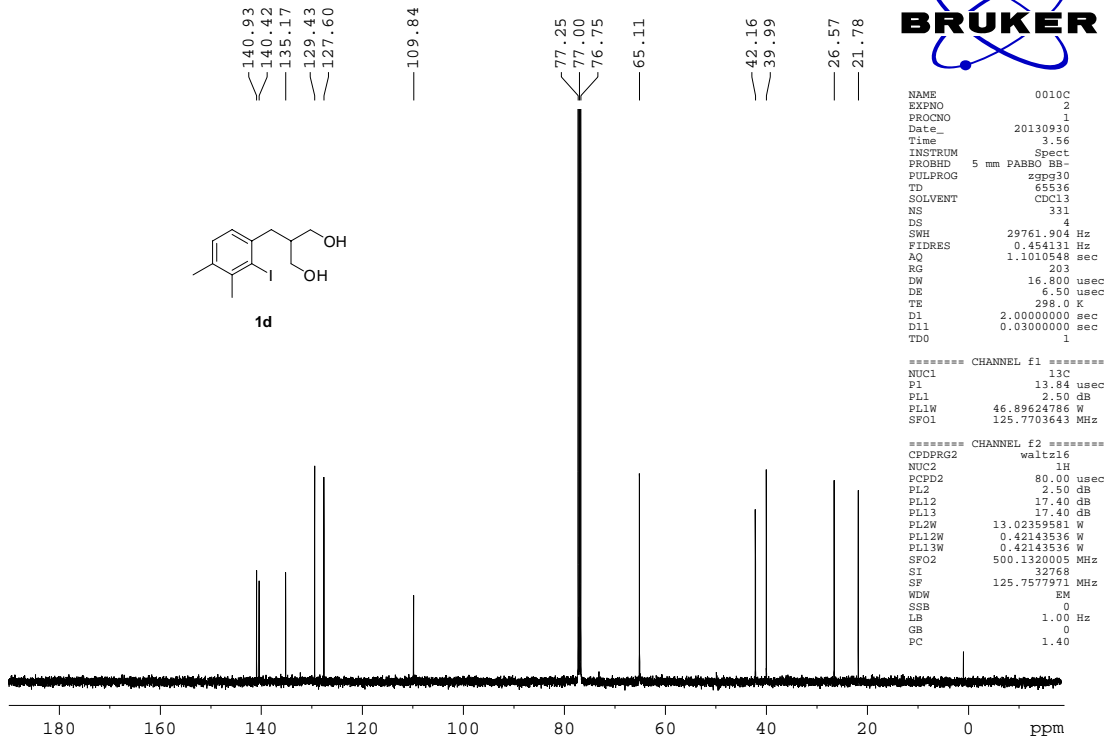
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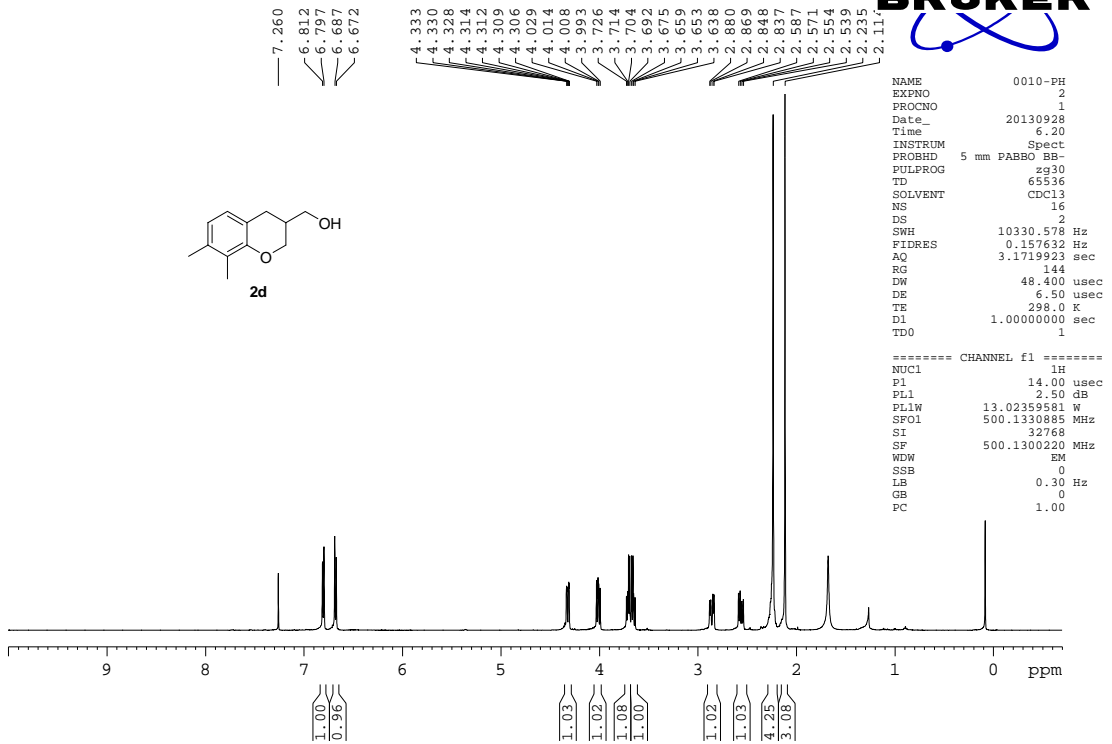
0010



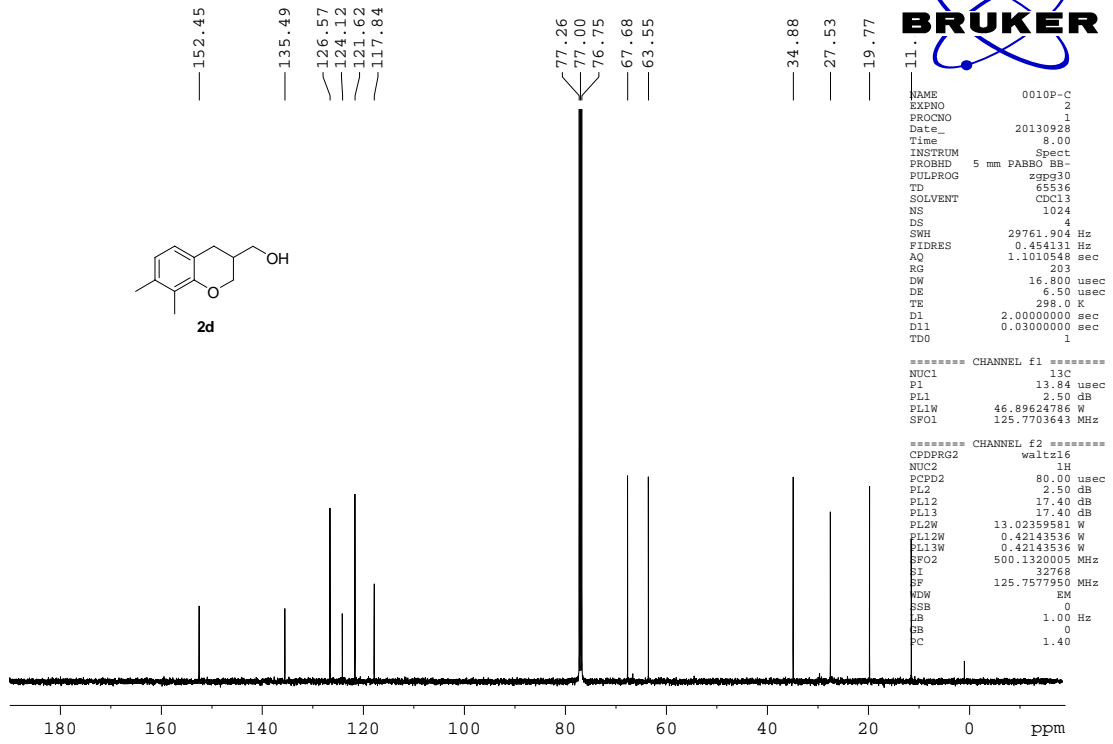
0010C



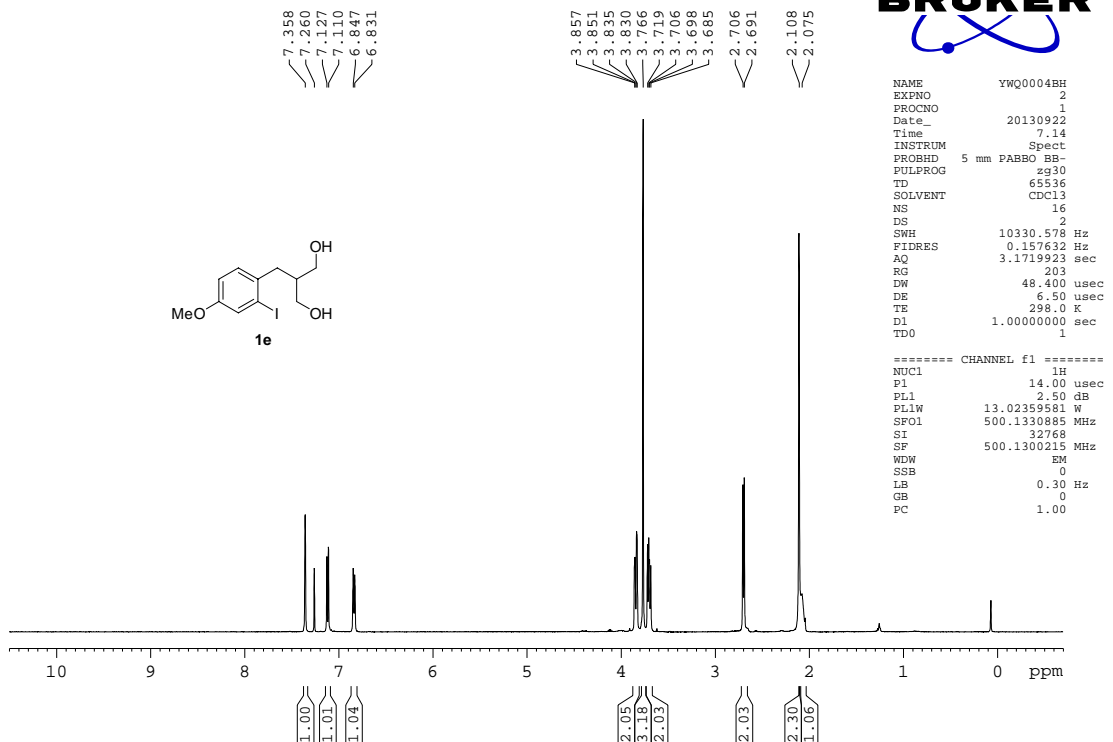
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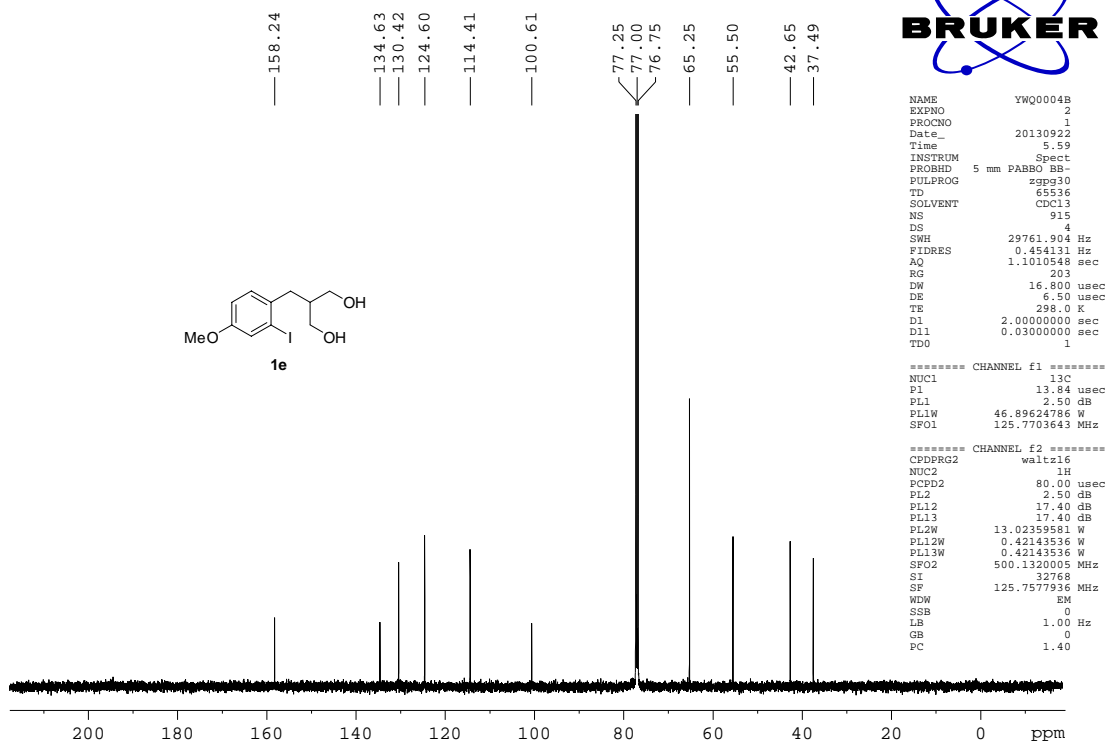
0010P-C



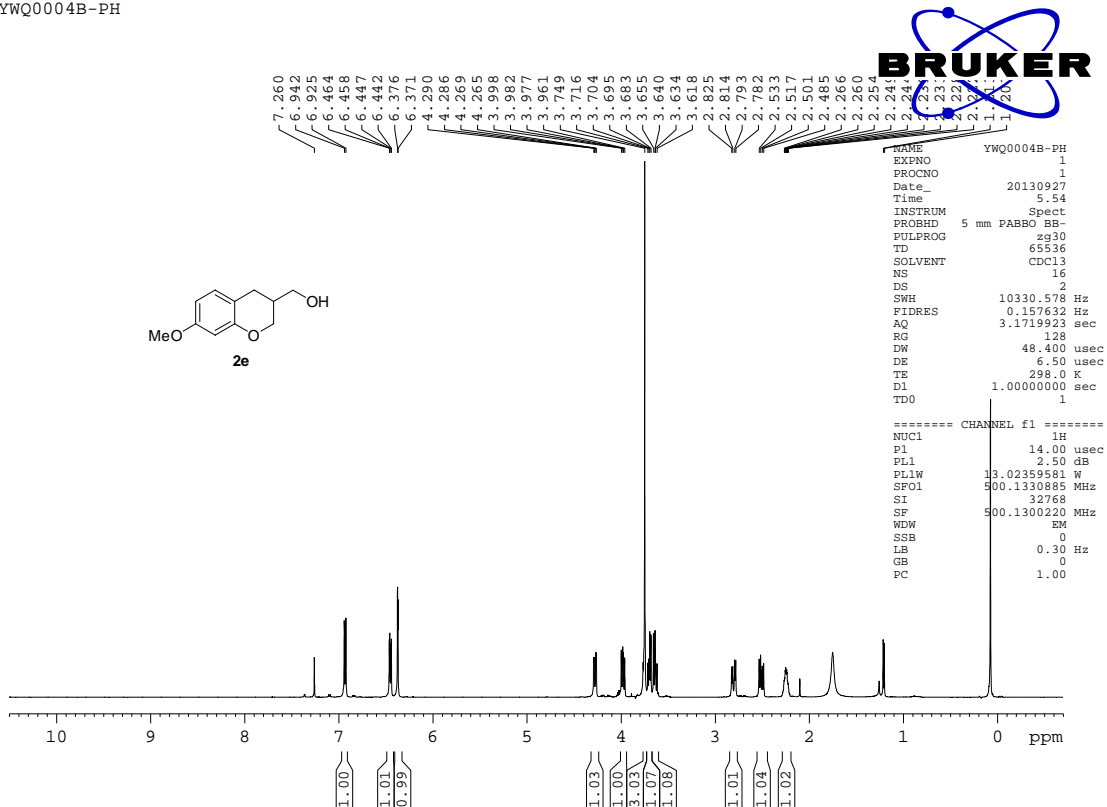
YWQ0004BH



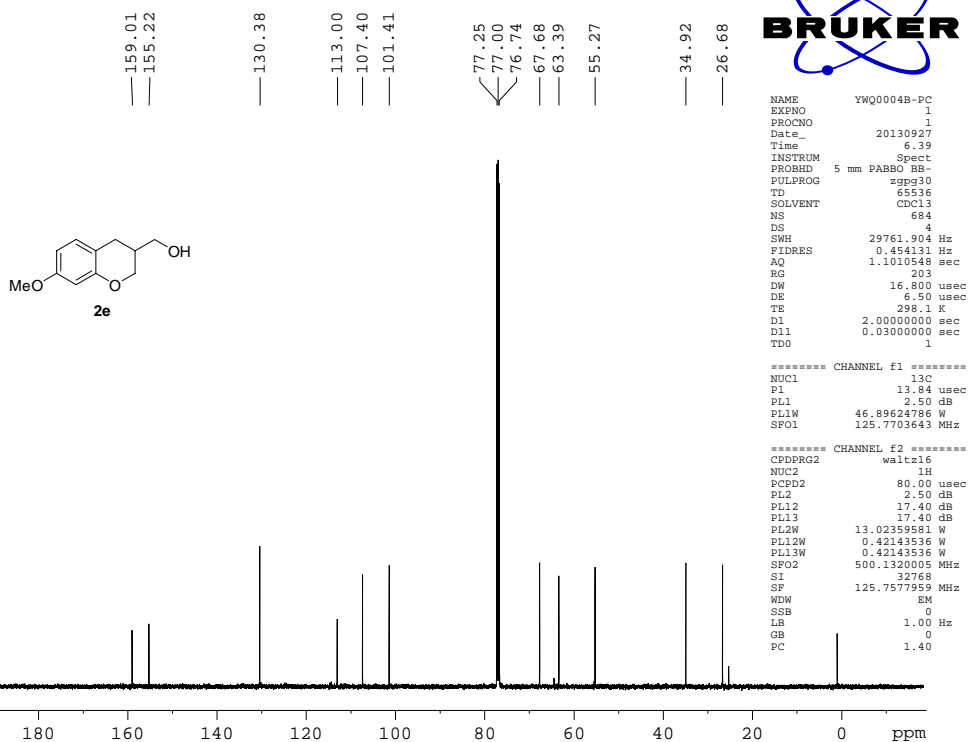
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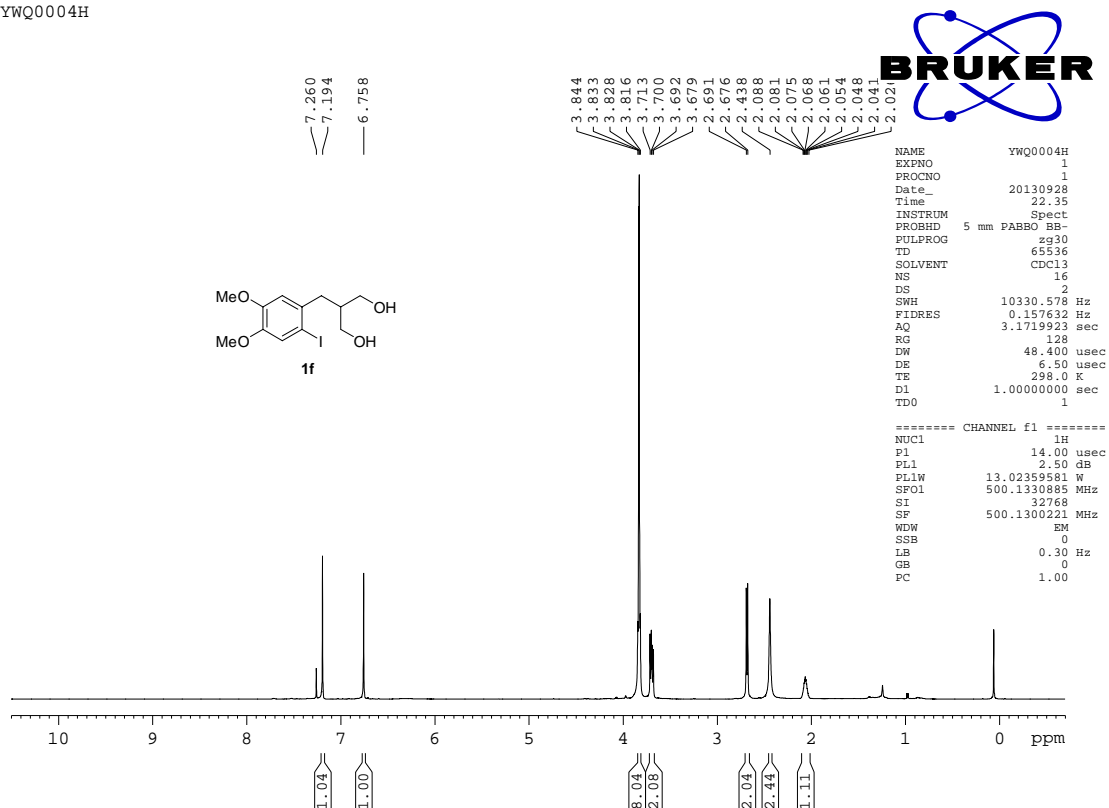
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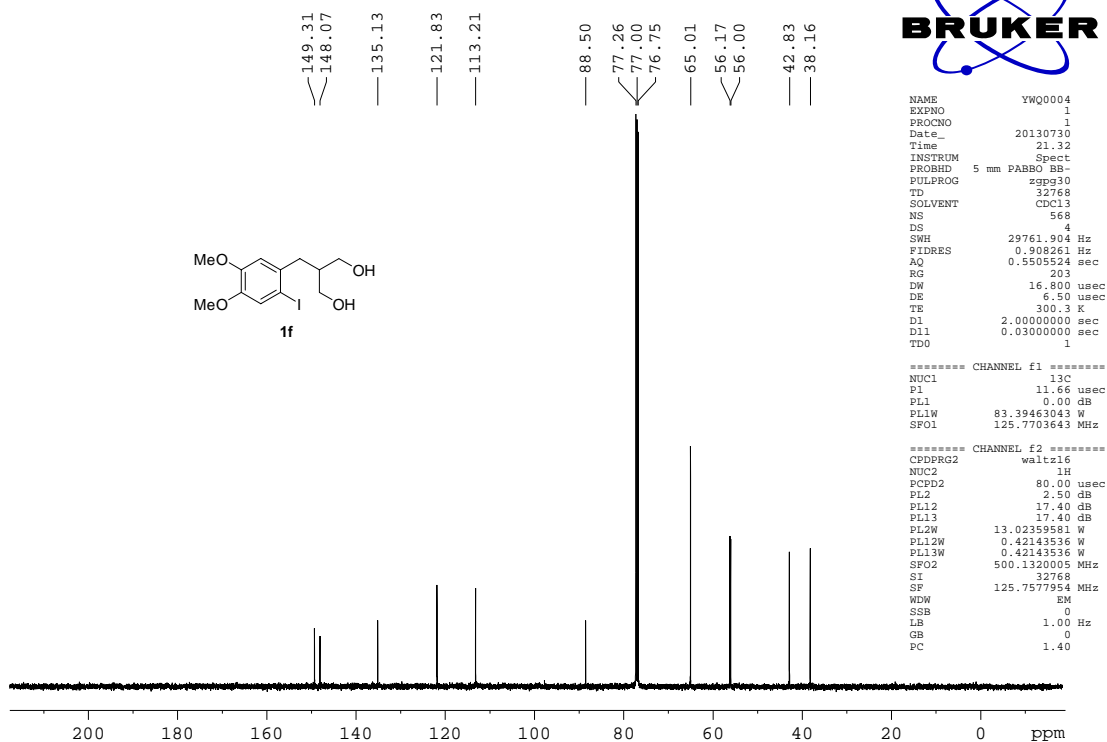
YWQ0004B-PC



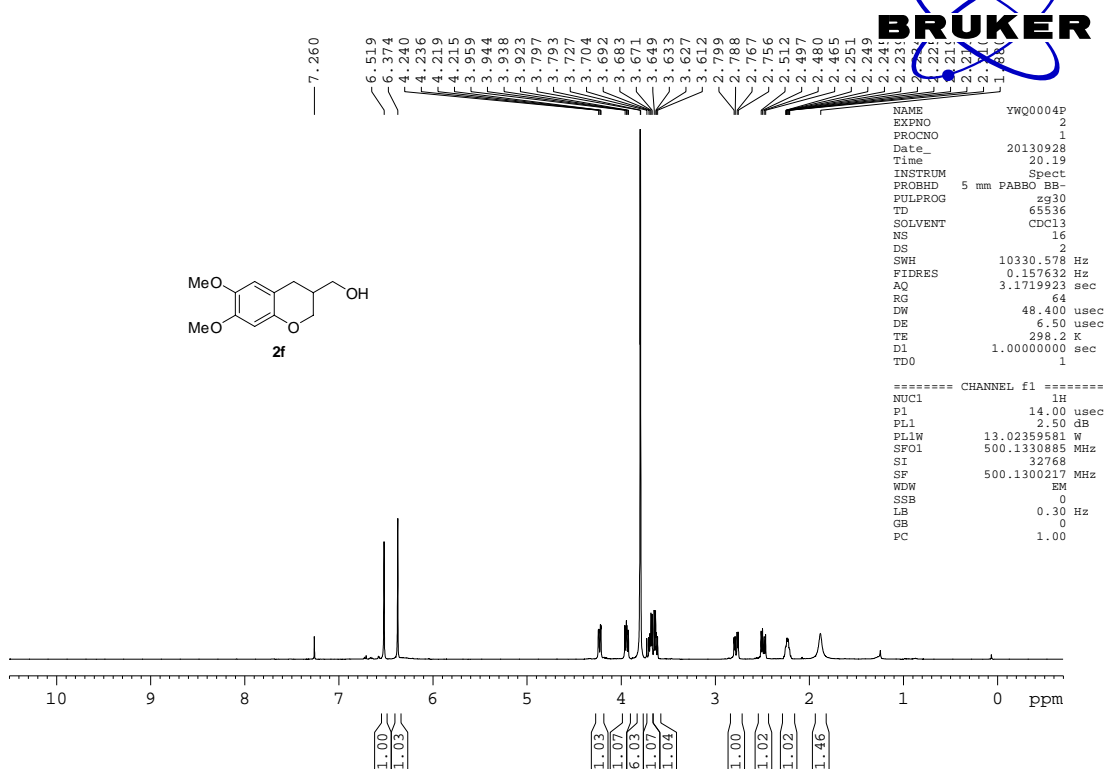
YWQ0004H



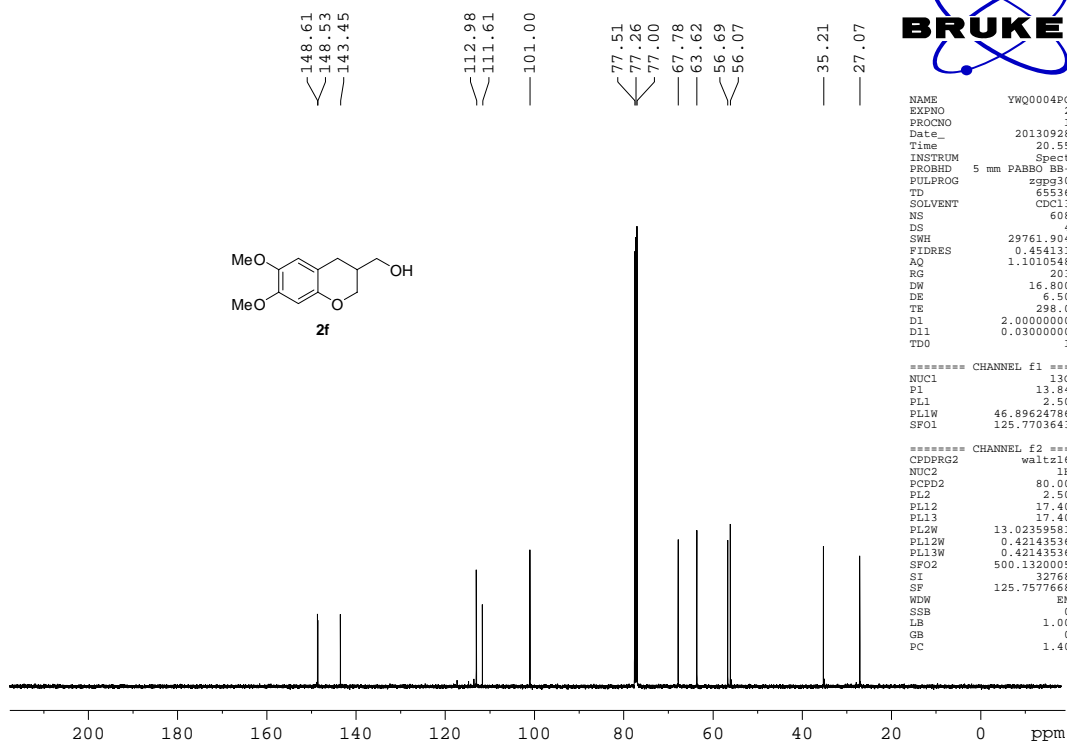
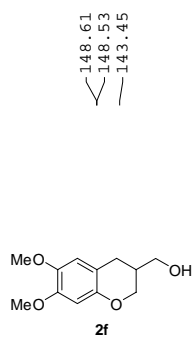
YWQ0004



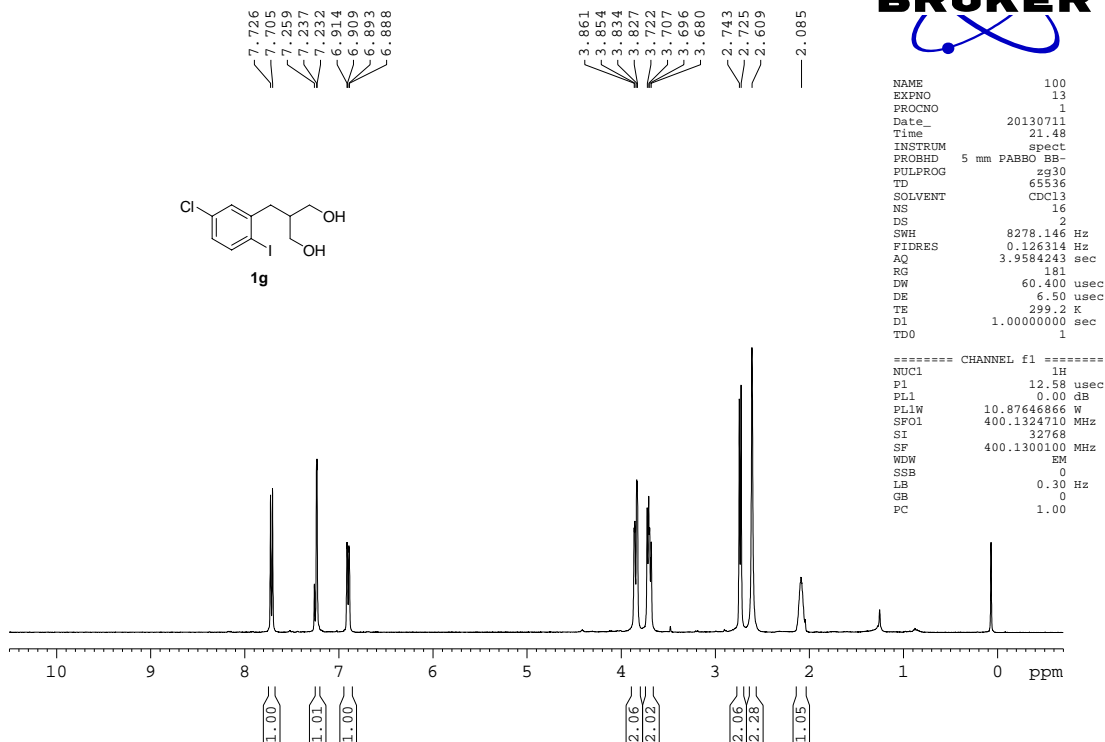
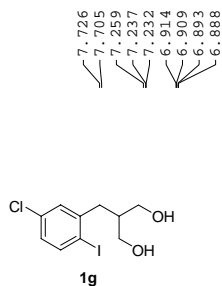
YWQ0004P



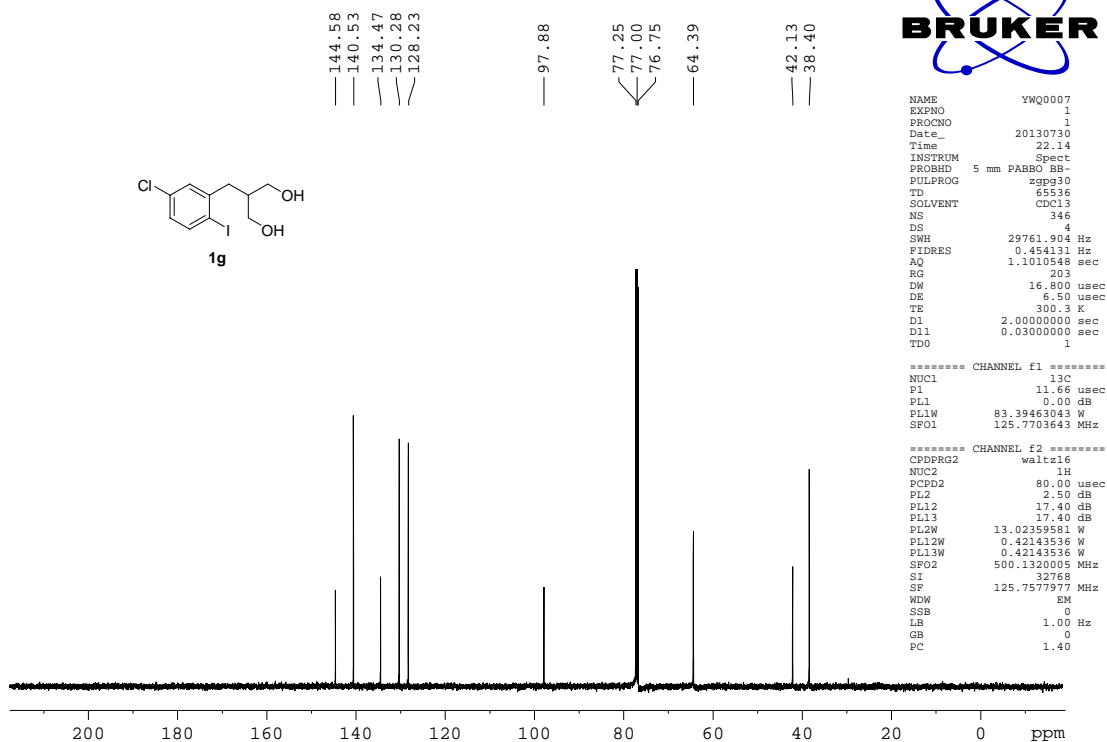
YWQ0004PC



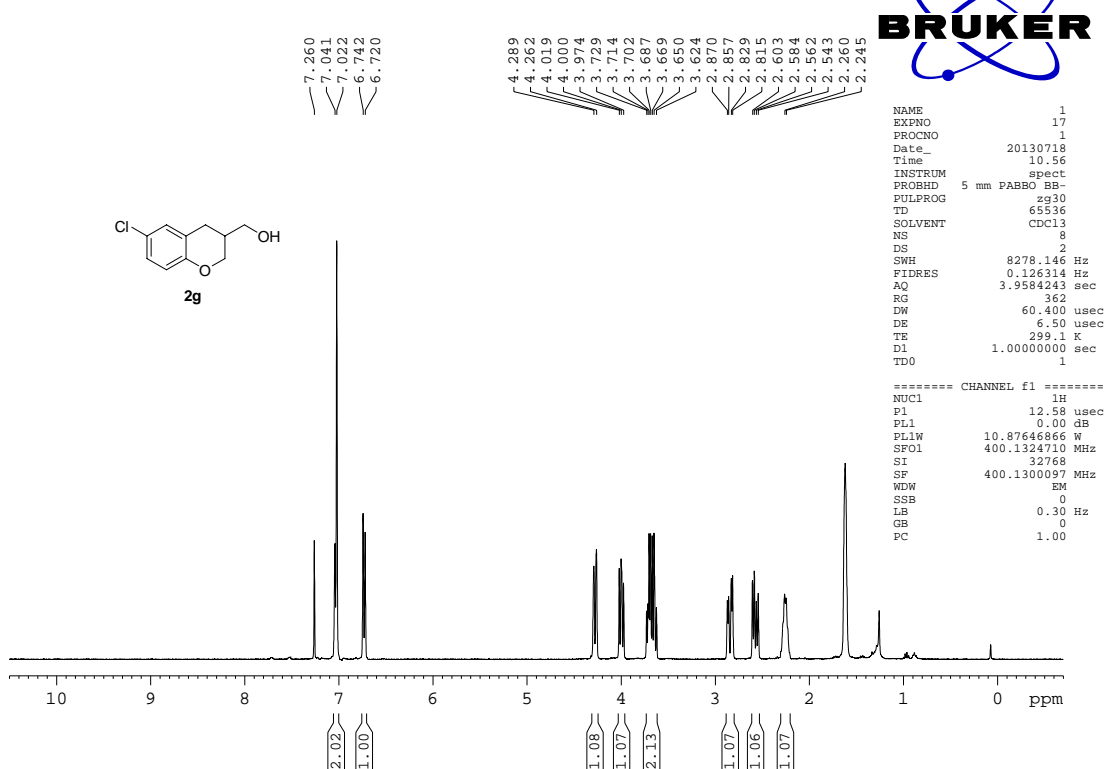
ywq0007



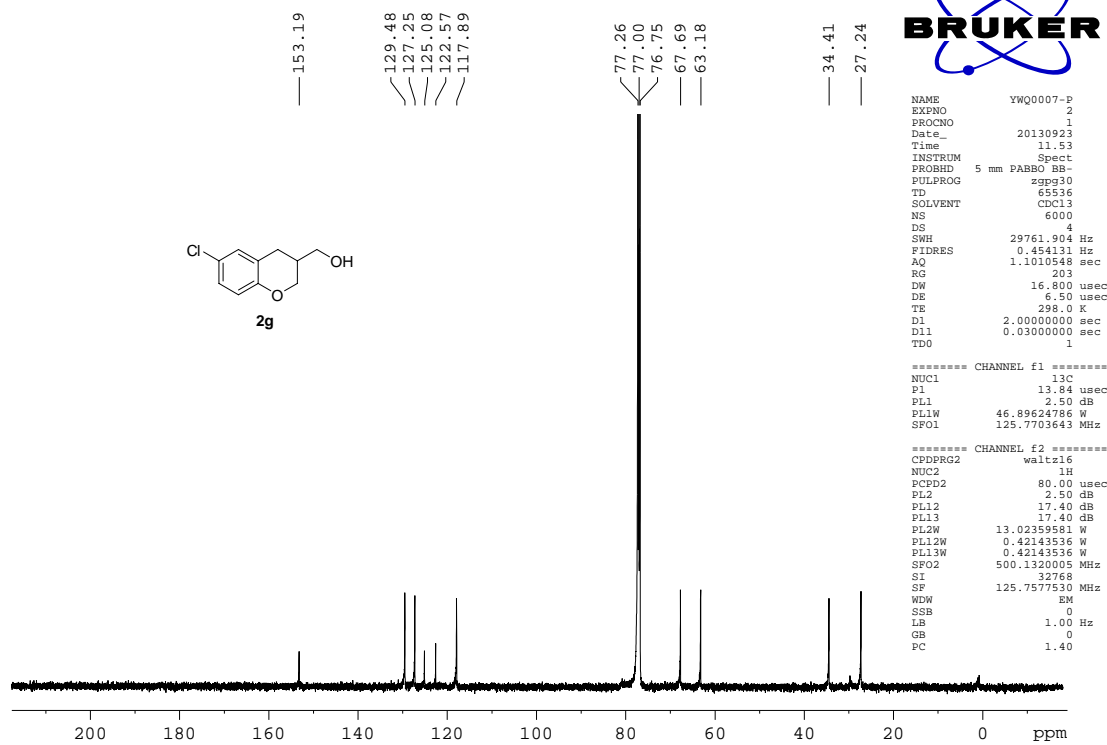
YWQ0007



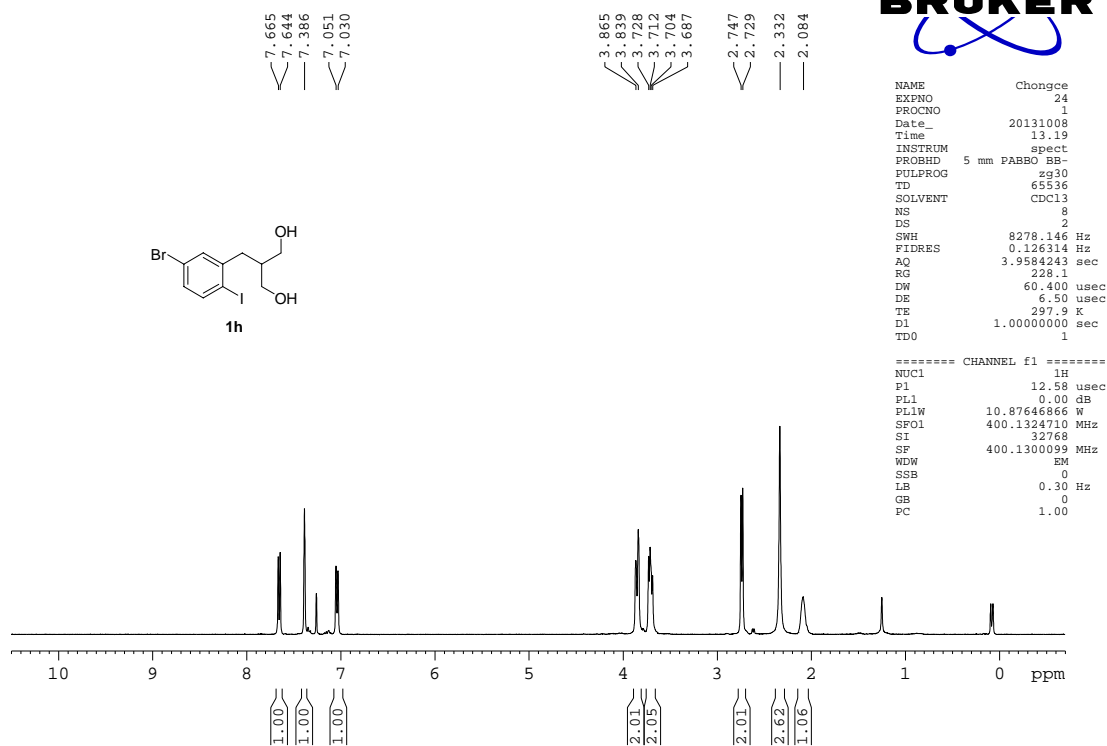
YWQ-007



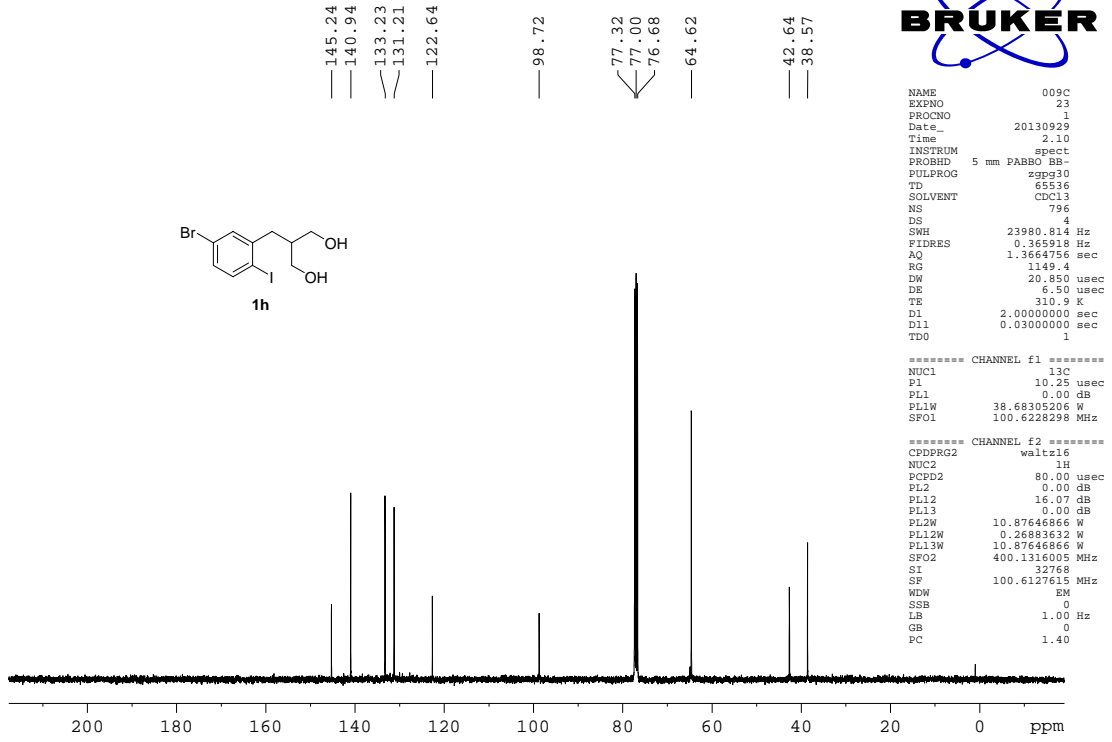
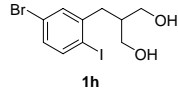
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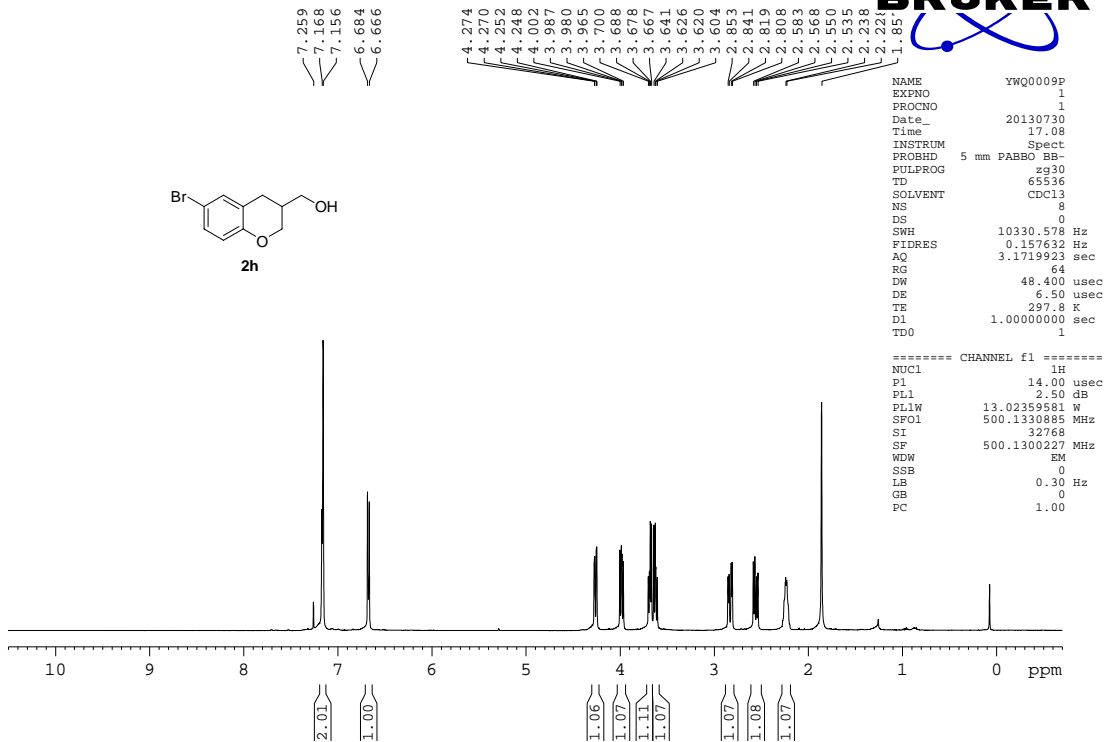
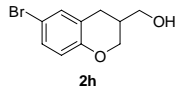
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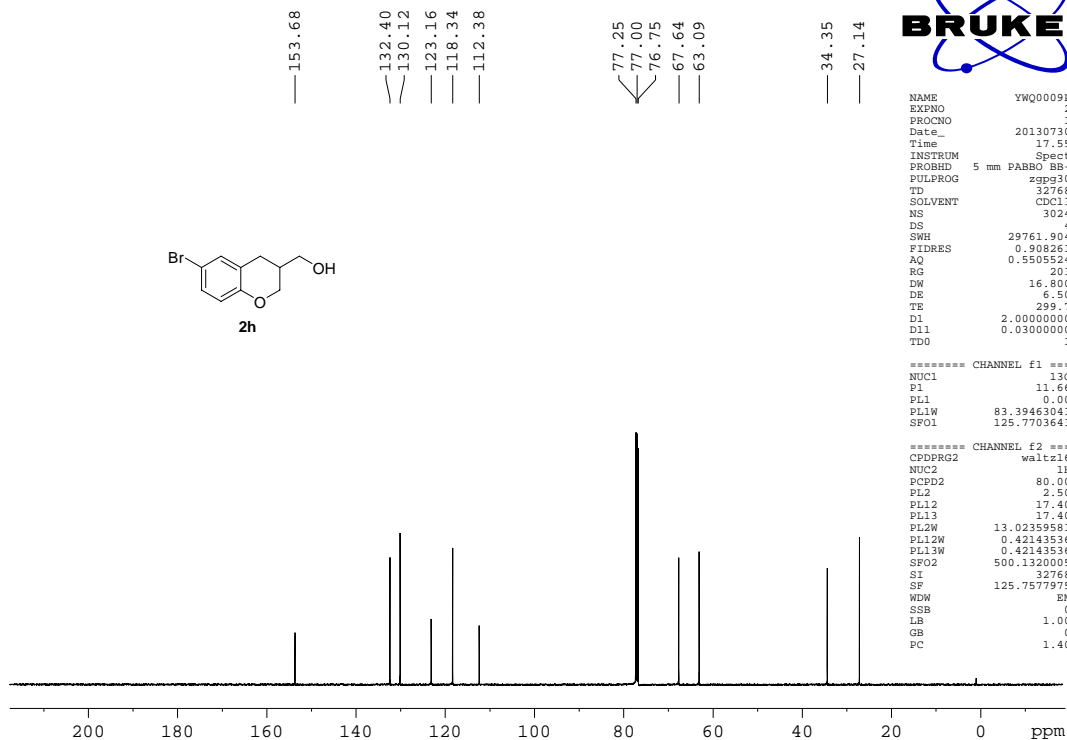
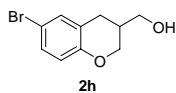
009C



YWQ0009P



YWQ0009P



```

NAME      YWQ0009P
EXPNO    2
PROCNO   1
Date_    20130730
Time     17.55
INSTRUM  Spect
PROBHD   5 mm PABBO BB-
PULPROG  zgpg30
TD       32768
SOLVENT  CDCl3
NS       3024
DS       4
SWH      29761.904 Hz
FIDRES   0.908261 Hz
AQ       0.5505524 sec
RG       203
DW       16.800 usec
DE       6.50 usec
TE       299.7 K
D1       2.0000000 sec
D11      0.0300000 sec
TD0      1
  
```

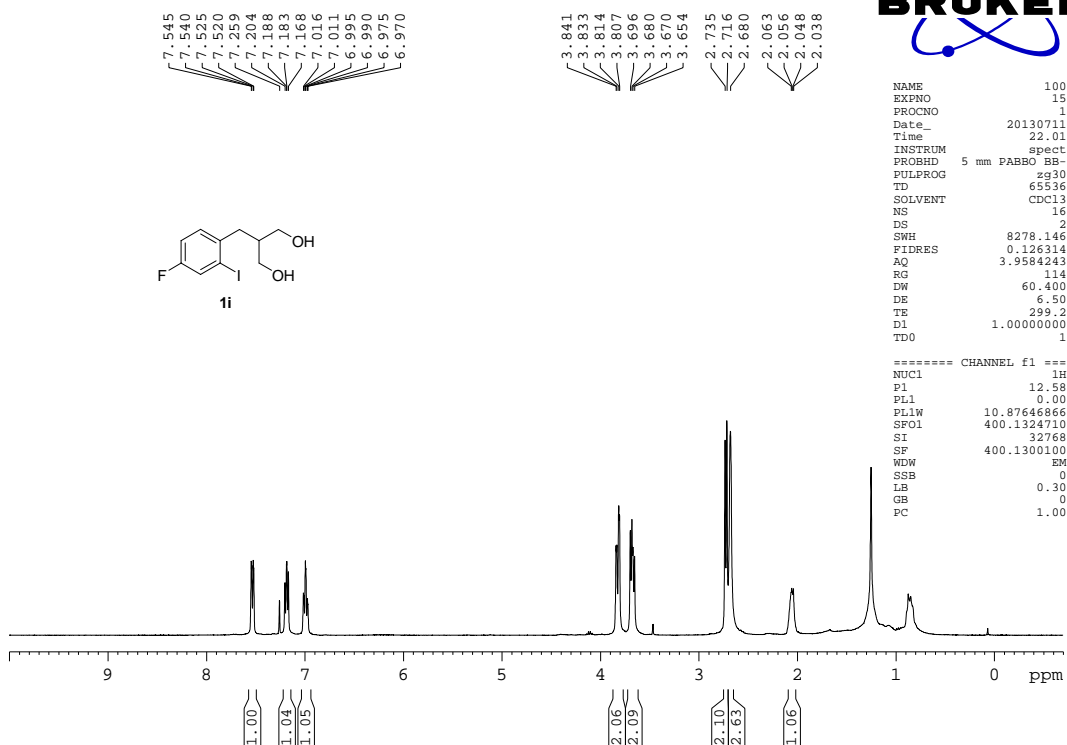
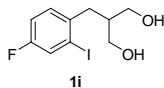
```

***** CHANNEL f1 *****
NUC1     13C
P1       11.66 usec
PL1     0.00 dB
PL1W    83.39463043 W
SFO1    125.7703643 MHz
  
```

```

***** CHANNEL F2 *****
CPDPRG2  waltz16
NUC2     1H
PCPD2    80.00 usec
PL2     2.50 dB
PL12    17.40 dB
PL13    17.40 dB
PL2W    13.02359581 W
PL12W   0.42143536 W
PL13W   0.42143536 W
SFO2    500.1320005 MHz
SI       32768
SF      125.7577975 MHz
WDW      EM
SSB      0
LB       1.00 Hz
GB       0
PC       1.40
  
```

ywq0006



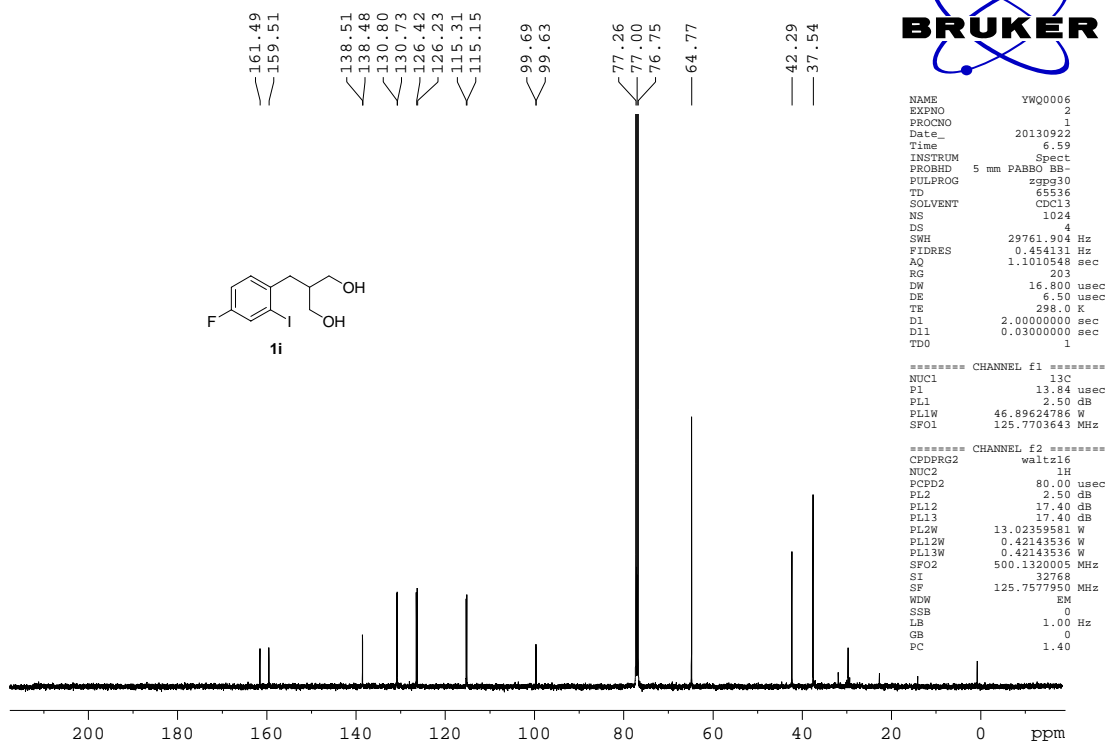
```

NAME      100
EXPNO    15
PROCNO   1
Date_    20130711
Time     22.01
INSTRUM  Spect
PROBHD   5 mm PABBO BB-
PULPROG  zg30
TD       65536
SOLVENT  CDCl3
NS       16
DS       2
SWH      8278.146 Hz
FIDRES   0.126314 Hz
AQ       3.9584243 sec
RG       114
DW       60.400 usec
DE       6.50 usec
TE       299.2 K
D1       1.0000000 sec
TD0      1
  
```

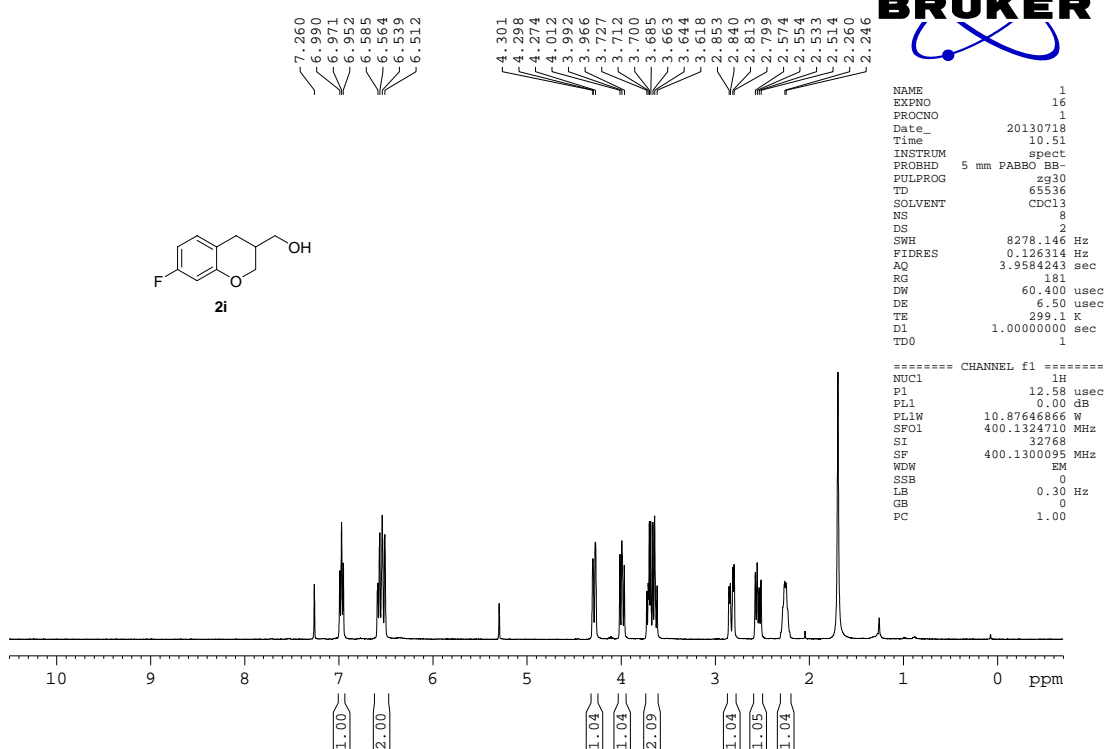
```

***** CHANNEL f1 *****
NUC1     1H
P1       12.58 usec
PL1     0.00 dB
PL1W    10.87646866 W
SFO1    400.1324710 MHz
SI       32768
SF      400.1300100 MHz
WDW      EM
SSB      0
LB       0.30 Hz
GB       0
PC       1.00
  
```

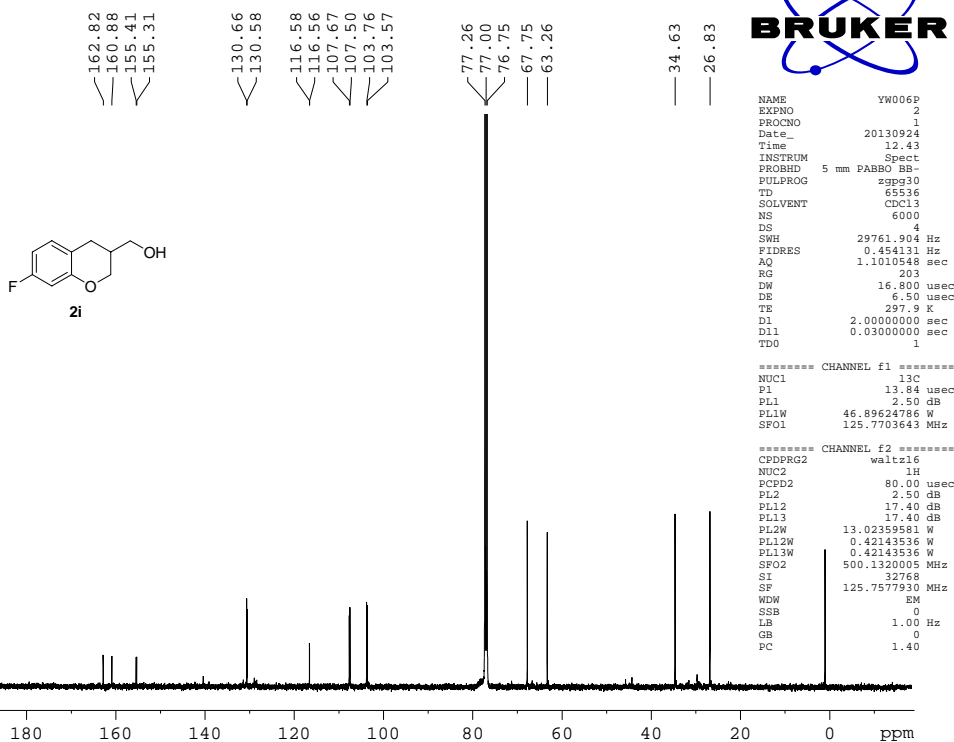
YWQ0006



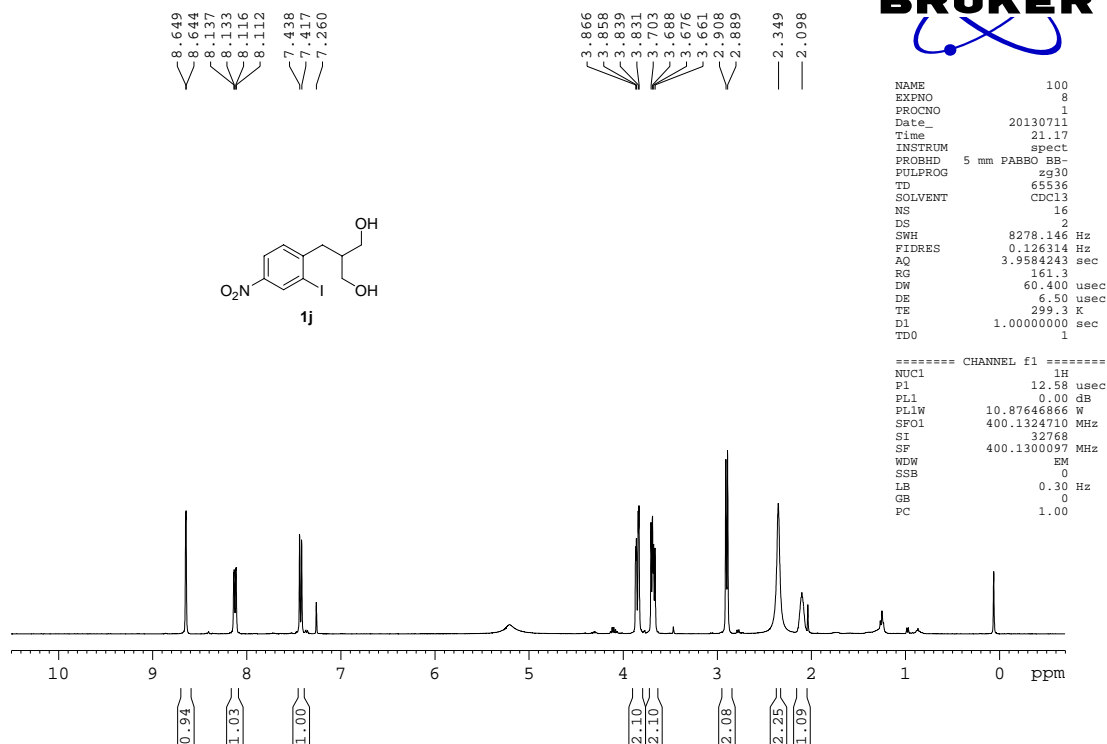
YWQ-006



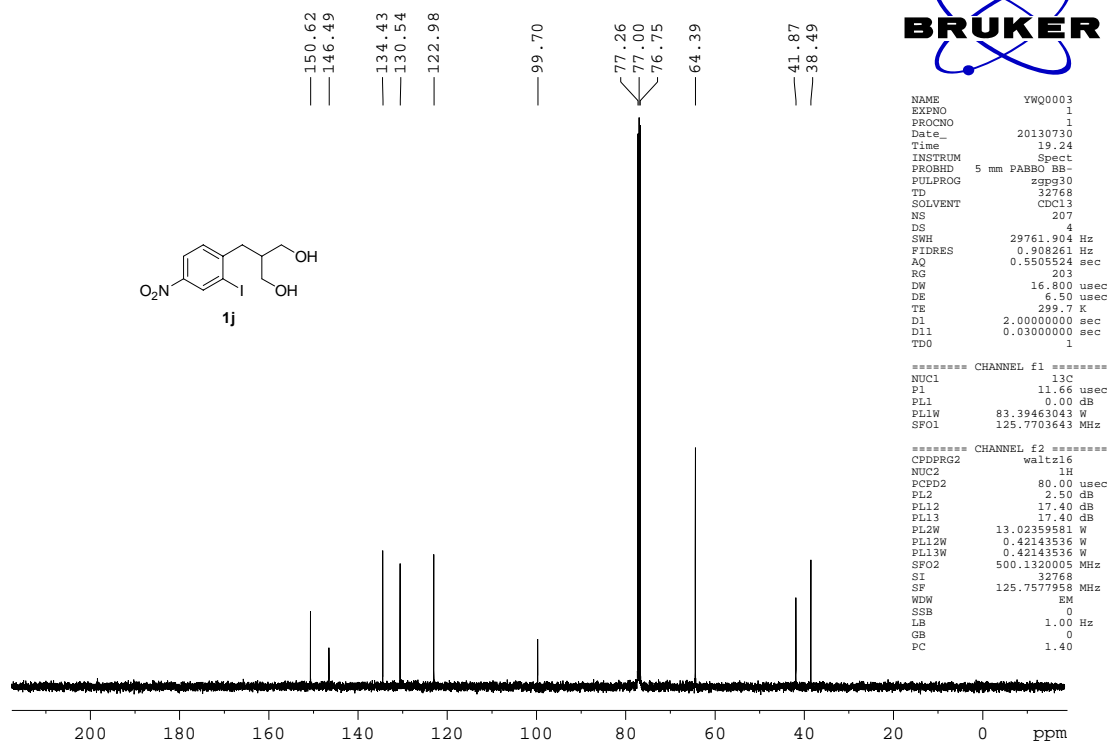
YW006P



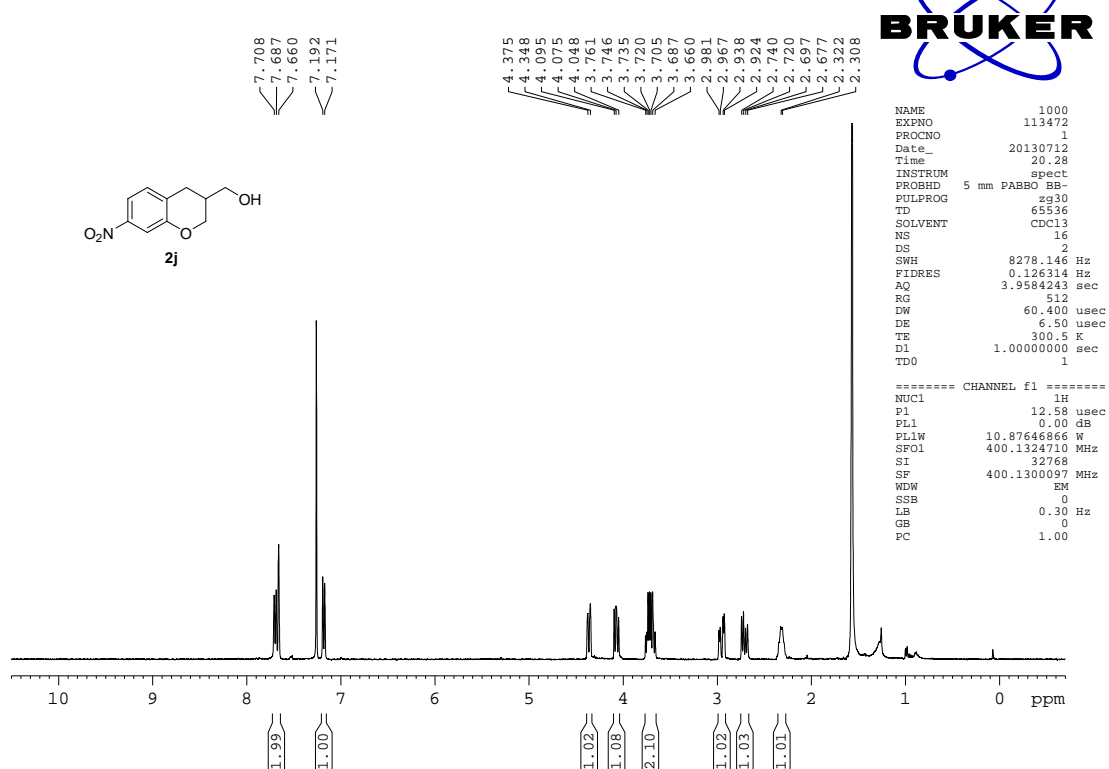
ywq0003



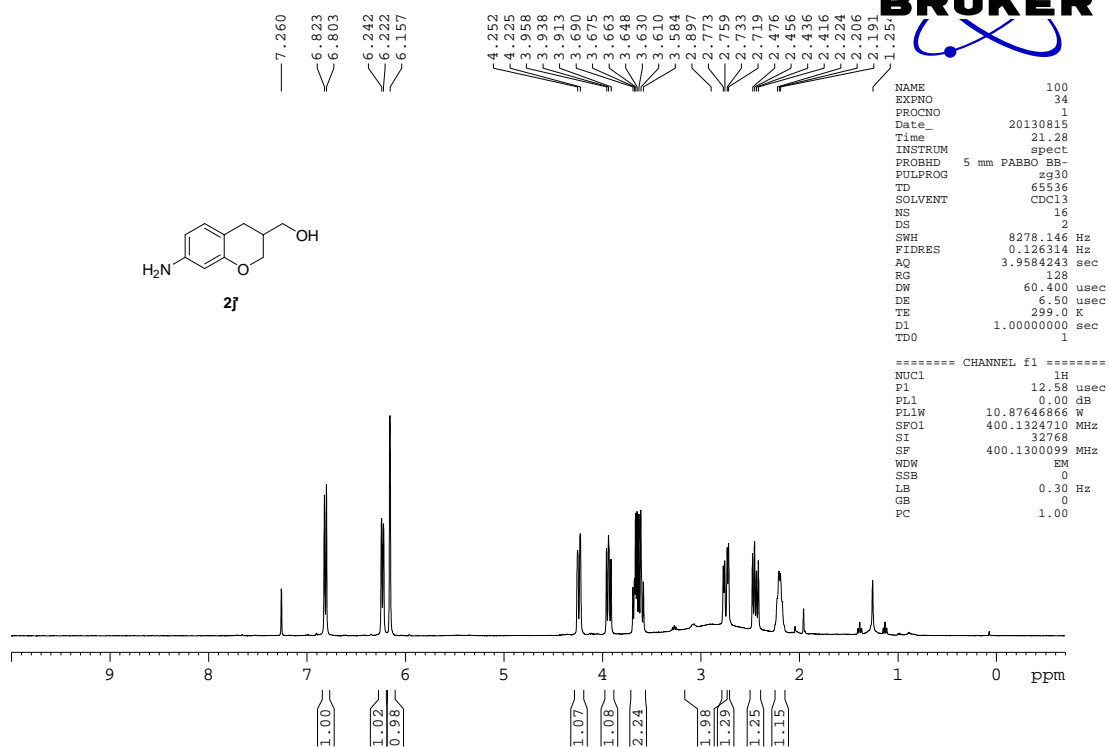
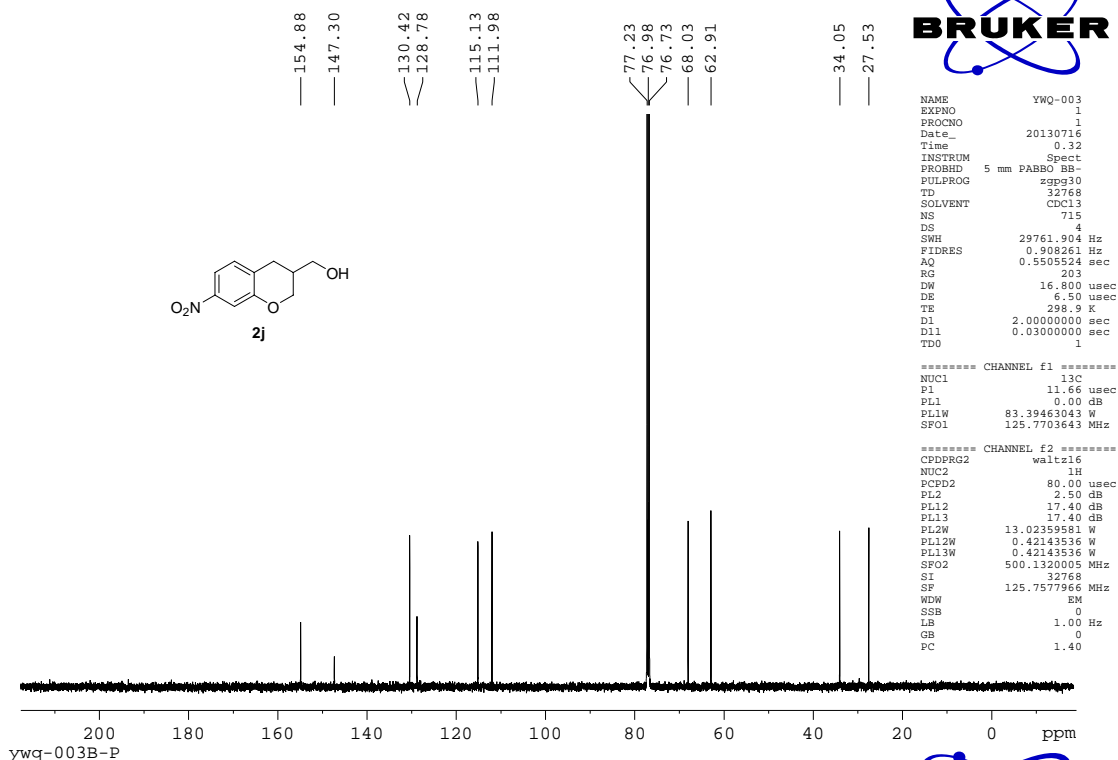
YWQ0003



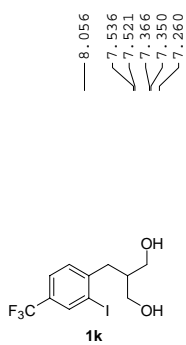
LY-113472



YWQ-003



0026H

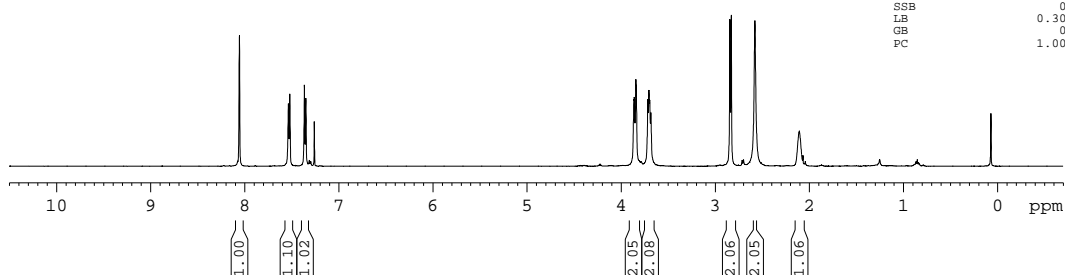


3.864  
3.860  
3.844  
3.838  
3.716  
3.704  
3.696  
3.683  
2.844  
2.829  
2.578  
2.104



```

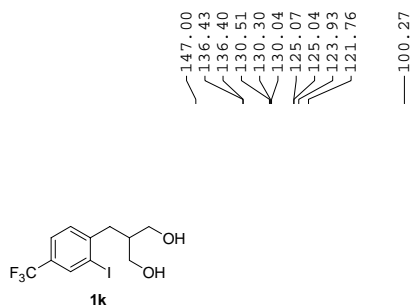
NAME          0026H
EXPNO         2
PROCNO        1
Date_         20130929
Time          5.37
INSTRUM       Spect
PROBHD        5 mm PABBO BB-
PULPROG       zg30
TD            65536
SOLVENT       CDCl3
NS            16
DS            2
SWH           10330.578 Hz
FIDRES        0.157632 Hz
AQ            3.1719923 sec
RG            161
DW            48.400 usec
DE            6.50 usec
TE            298.1 K
D1            1.00000000 sec
D11           1
TDO           1
  
```



```

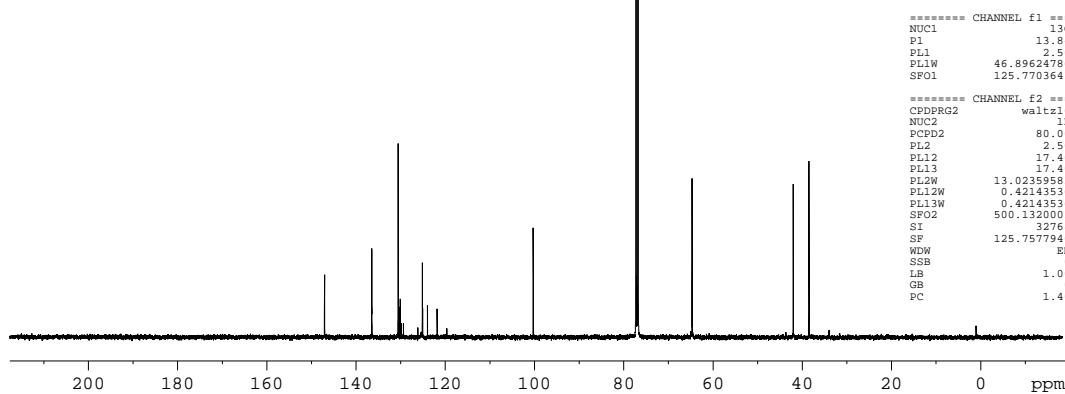
===== CHANNEL f1 =====
NUC1          1H
P1            14.00 usec
PL1           2.50 dB
PL1W          13.02359581 W
SF01          500.1330885 MHz
SI            32768
SF            500.1300221 MHz
WDW           EM
SSB           0
LB            0.30 Hz
GB            0
PC            1.00
  
```

0026CC



```

NAME          0026CC
EXPNO         2
PROCNO        1
Date_         20130929
Time          7.35
INSTRUM       Spect
PROBHD        5 mm PABBO BB-
PULPROG       zgpg30
TD            65536
SOLVENT       CDCl3
NS            2048
DS            4
SWH           29761.904 Hz
FIDRES        0.454131 Hz
AQ            1.1010548 sec
RG            203
DW            16.800 usec
DE            6.50 usec
TE            297.9 K
D1            2.00000000 sec
D11           0.03000000 sec
TDO           1
  
```

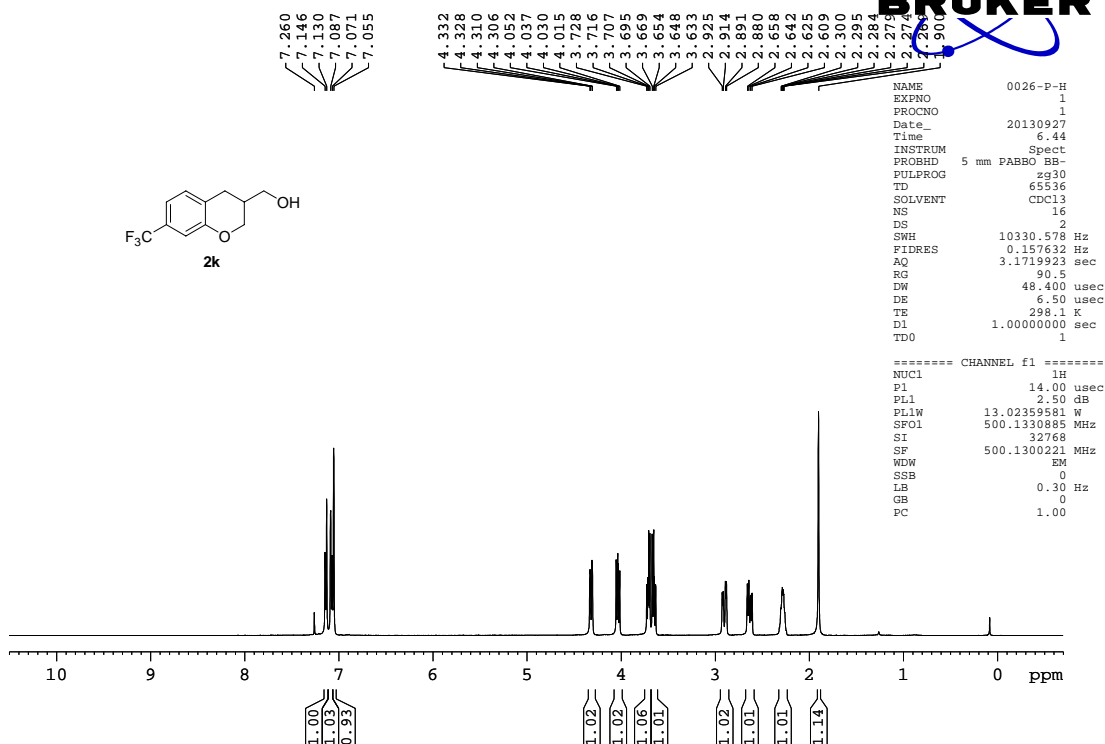
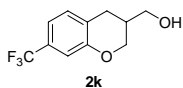


```

===== CHANNEL f1 =====
NUC1          13C
P1            13.84 usec
PL1           2.50 dB
PL1W          46.89624786 W
SF01          125.7703643 MHz

===== CHANNEL f2 =====
CPDPRG2       waltz16
NUC2          1H
PCPD2         80.00 usec
PL2           2.50 dB
PL12          17.40 dB
PL13          17.40 dB
PL2W          13.02359581 W
PL12W         0.42143536 W
PL13W         0.42143536 W
SF02          500.1320005 MHz
SI            32768
SF            125.7577940 MHz
WDW           EM
SSB           0
LB            1.00 Hz
GB            0
PC            1.40
  
```

0026-P-H



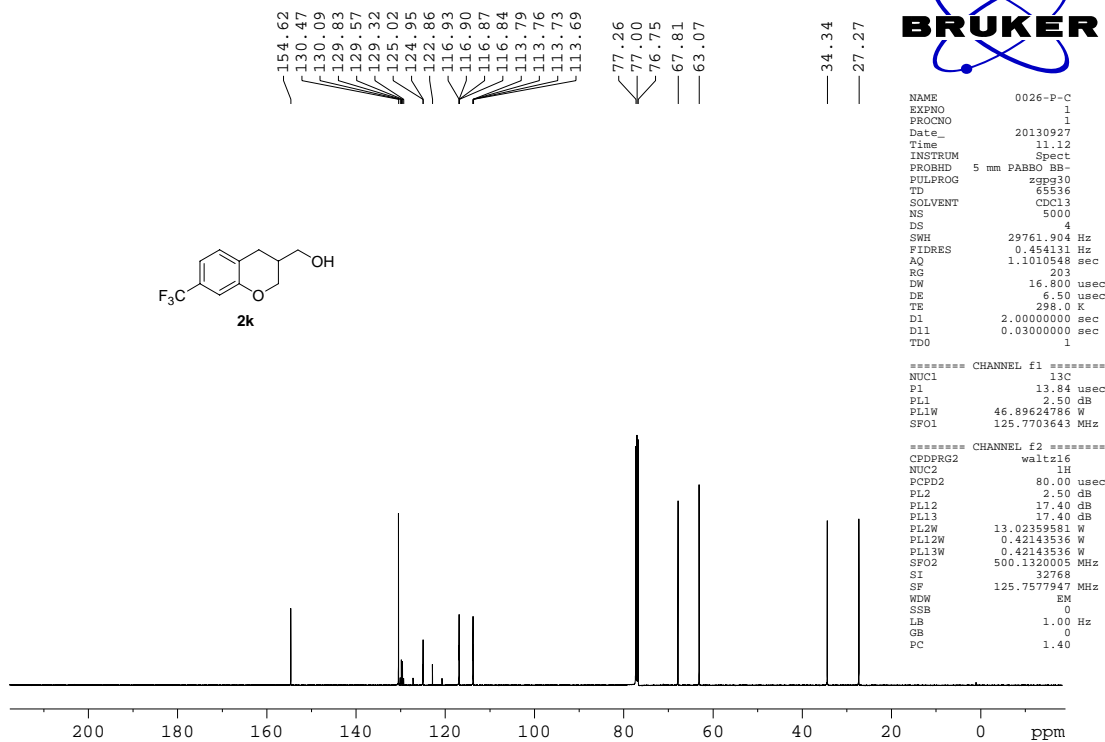
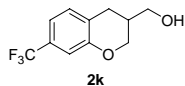
```

NAME          0026-P-H
EXPNO         1
PROCNO        1
Date_         20130927
Time          6.44
INSTRUM       Spect
PROBHD        5 mm PABBO BB-
PULPROG       zg30
TD            65536
SOLVENT       CDCl3
NS            16
DS            2
SWH           10330.578 Hz
FIDRES        0.157632 Hz
AQ            3.1719923 sec
RG            90.5
DW            48.400 usec
DE            6.50 usec
TE            298.1 K
D1            1.00000000 sec
TDO           1
  
```

```

===== CHANNEL f1 =====
NUC1          1H
P1            14.00 usec
PL1           2.50 dB
PL1W          13.02359581 W
SF01          500.1330885 MHz
SI            32768
SF            500.1300221 MHz
WDW           EM
SSB           0
LB            0.30 Hz
GB            0
PC            1.00
  
```

0026-P-C



```

NAME          0026-P-C
EXPNO         1
PROCNO        1
Date_         20130927
Time          11.12
INSTRUM       Spect
PROBHD        5 mm PABBO BB-
PULPROG       zgpg30
TD            65536
SOLVENT       CDCl3
NS            5000
DS            4
SWH           29761.904 Hz
FIDRES        0.454131 Hz
AQ            1.1010548 sec
RG            203
DW            16.800 usec
DE            6.50 usec
TE            298.0 K
D1            2.00000000 sec
D11           0.03000000 sec
TDO           1
  
```

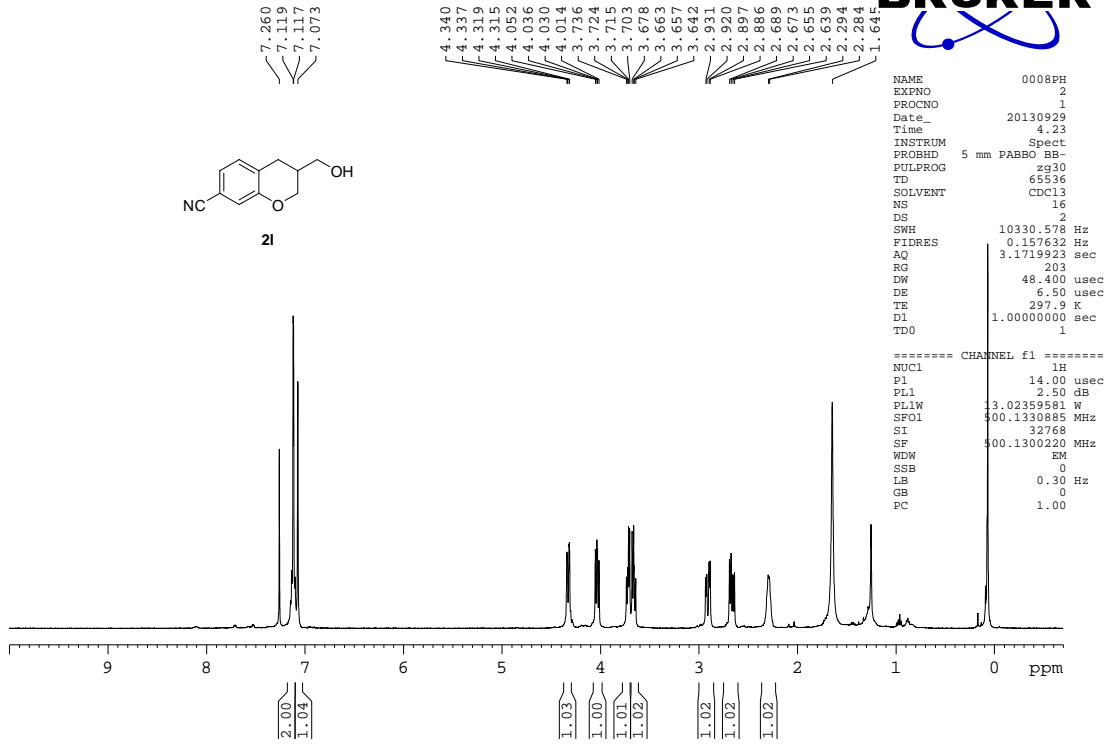
```

===== CHANNEL f1 =====
NUC1          13C
P1            13.84 usec
PL1           2.50 dB
PL1W          46.89624786 W
SF01          125.7703643 MHz

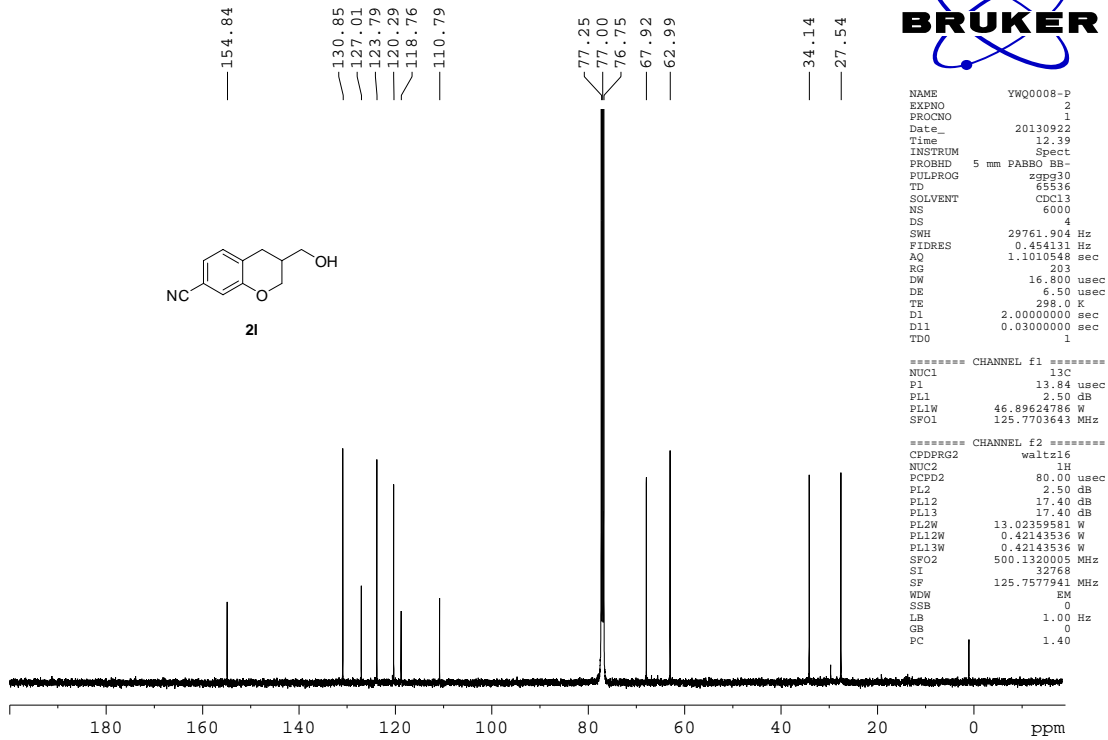
===== CHANNEL f2 =====
CPDPRG2       waltz16
NUC2          1H
PCPD2         80.00 usec
PL2           2.50 dB
PL12          17.40 dB
PL13          17.40 dB
PL1W          13.02359581 W
PL12W         0.42143536 W
PL13W         0.42143536 W
SF02          500.1320005 MHz
SI            32768
SF            125.7577947 MHz
WDW           EM
SSB           0
LB            1.00 Hz
GB            0
PC            1.40
  
```



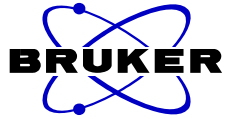
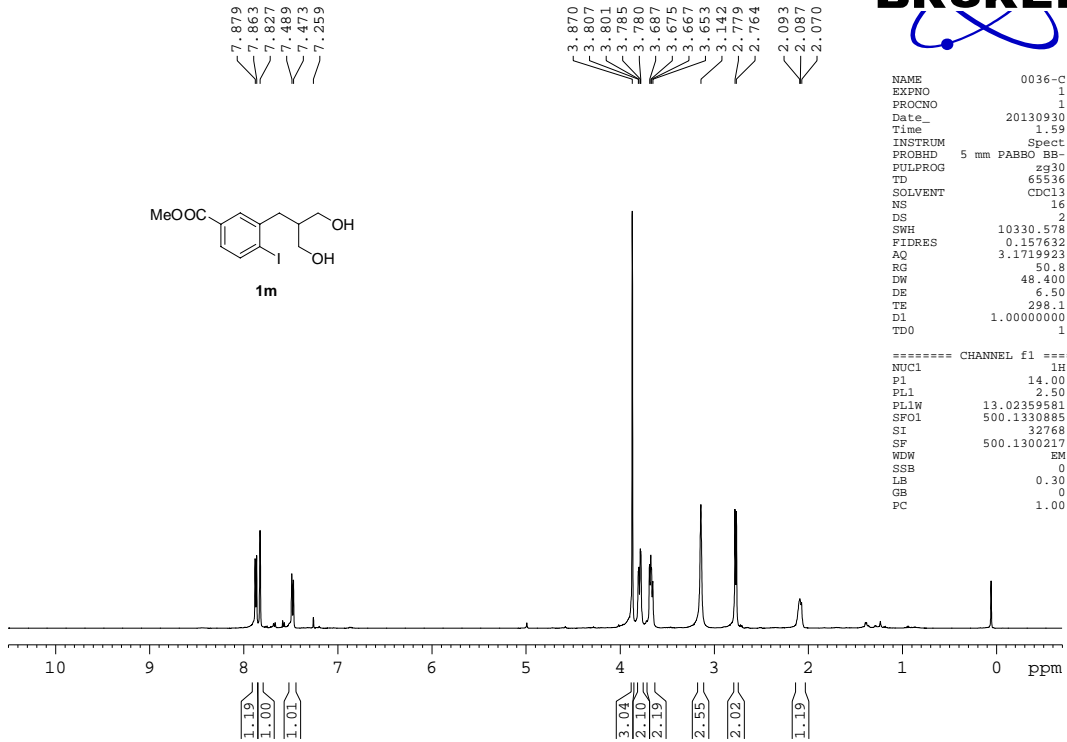
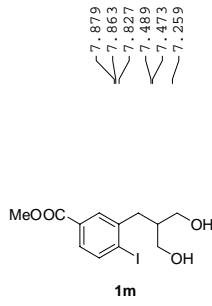
0008PH



YWQ0008-P



0036-C

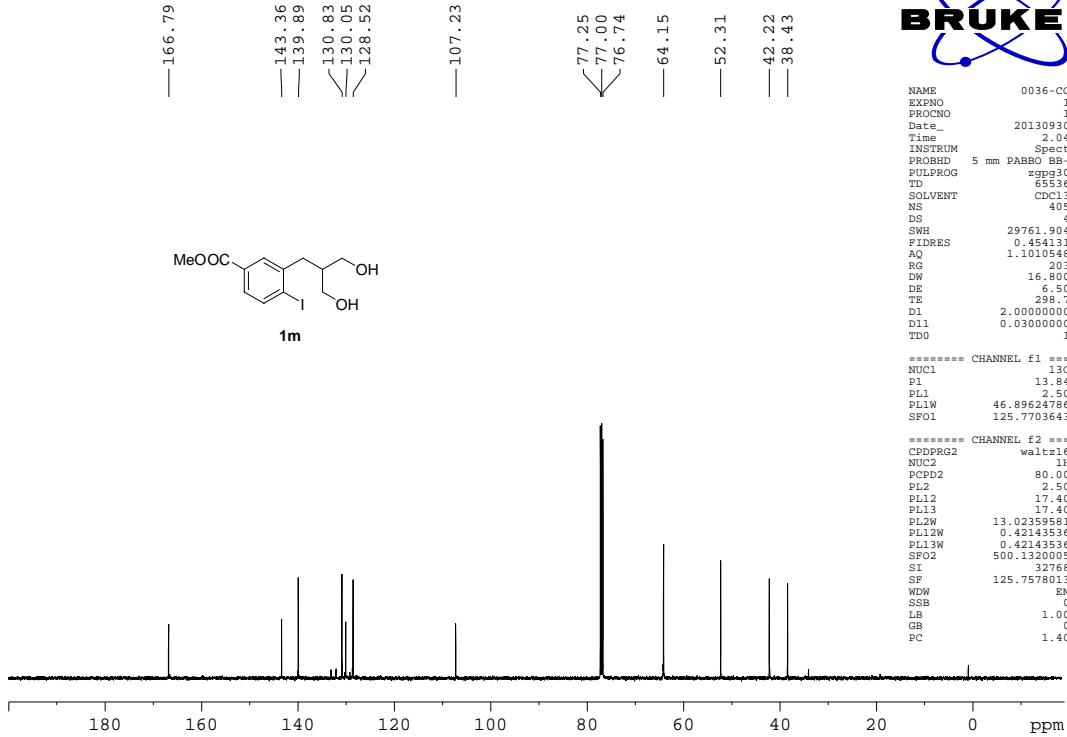
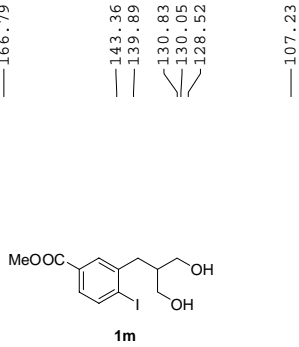


```

NAME          0036-C
EXPNO         1
PROCNO        1
Date_         20130930
Time          1.59
INSTRUM       Spect
PROBHD        5 mm PABBO BB-
PULPROG       zg30
TD            65536
SOLVENT       CDCl3
NS            16
DS            2
SWH           10330.578 Hz
FIDRES        0.157632 Hz
AQ            3.1719923 sec
RG            50.8
DW            48.400 usec
DE            6.50 usec
TE            298.1 K
D1            1.00000000 sec
TD0           1

===== CHANNEL f1 =====
NUC1          1H
P1            14.00 usec
PL1           2.50 dB
PL1W          13.02359581 W
SF01          500.1330885 MHz
SI            32768
SF            500.1300217 MHz
WDW           EM
SSB           0
LB            0.30 Hz
GB            0
PC            1.00
    
```

0036-CC



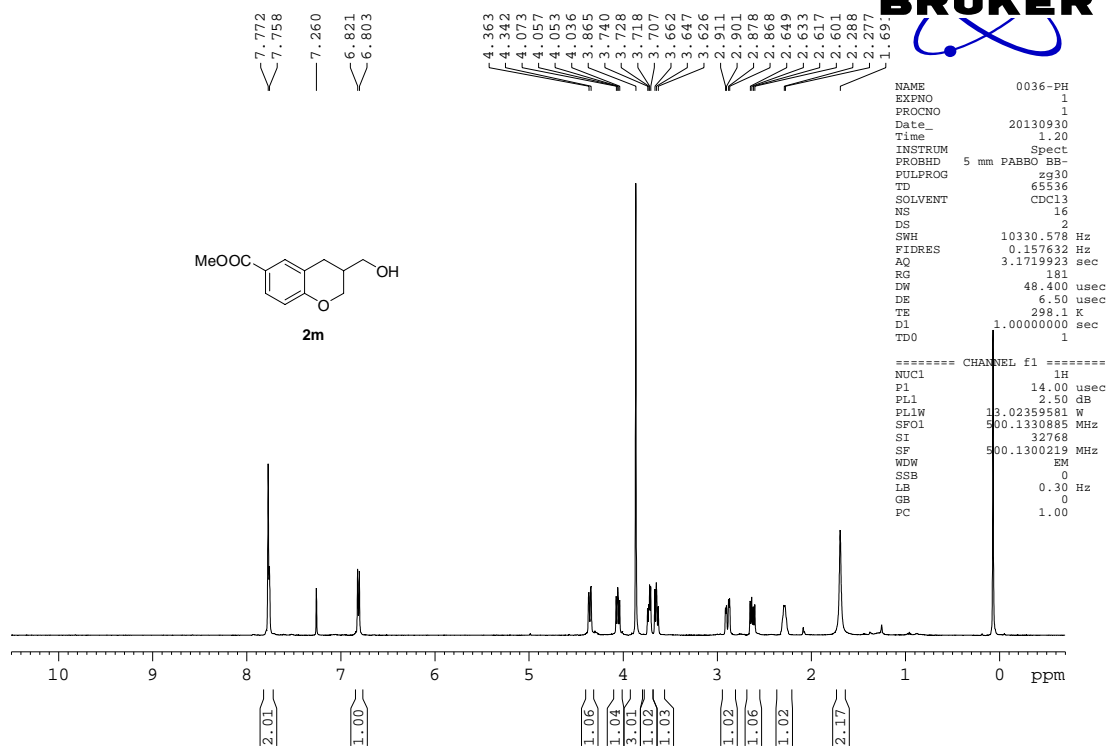
```

NAME          0036-CC
EXPNO         1
PROCNO        1
Date_         20130930
Time          2.04
INSTRUM       Spect
PROBHD        5 mm PABBO BB-
PULPROG       zgpg30
TD            65536
SOLVENT       CDCl3
NS            405
DS            4
SWH           29761.904 Hz
FIDRES        0.454131 Hz
AQ            1.1010548 sec
RG            203
DW            16.800 usec
DE            6.50 usec
TE            298.7 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0           1

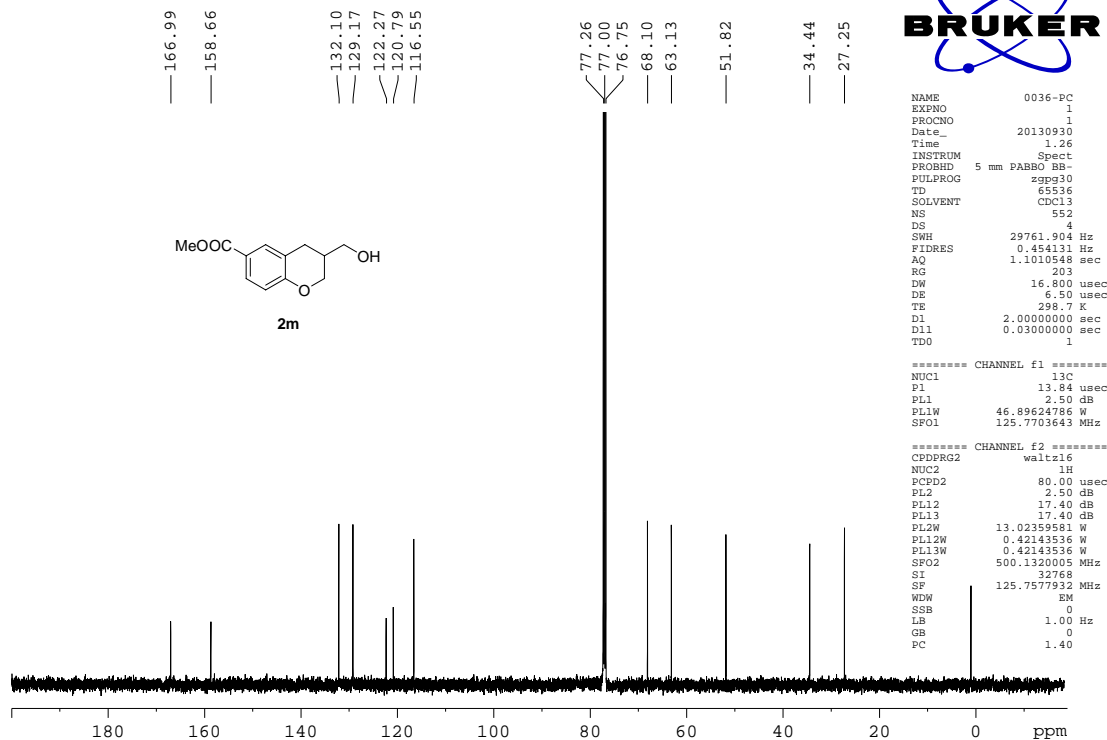
===== CHANNEL f1 =====
NUC1          13C
P1            13.84 usec
PL1           2.50 dB
PL1W          46.89624786 W
SF01          125.7703643 MHz

===== CHANNEL f2 =====
CPDPRG2       waltz16
NUC2          1H
PCPD2         80.00 usec
PL2           2.50 dB
PL12          17.40 dB
PL13          17.40 dB
PL1W          13.02359581 W
PL12W         0.42143536 W
PL13W         0.42143536 W
SF02          500.1320005 MHz
SI            32768
SF            125.7578013 MHz
WDW           EM
SSB           0
LB            1.00 Hz
GB            0
PC            1.40
    
```

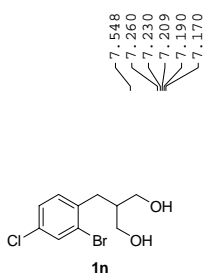
0036-PH



0036-PC



LY-113555



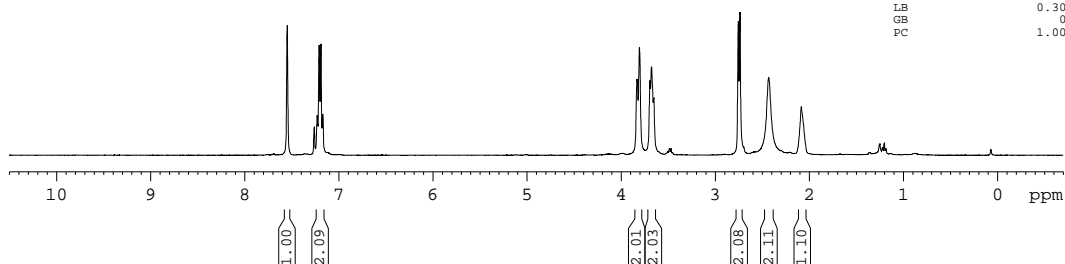
3.831  
3.805  
3.693  
3.677  
3.653

2.754  
2.736  
2.429

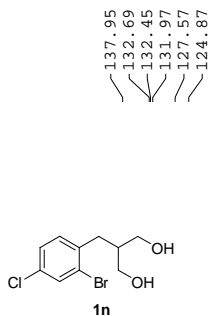
2.083

```

NAME          1000
EXPNO         113555
PROCNO        1
Date_         20130913
Time          21.51
INSTRUM       spect
PROBHD        5 mm PABBO BB-
PULPROG       zg30
TD            65536
SOLVENT       CDCl3
NS            16
DS            2
SWH           8278.146 Hz
FIDRES        0.126314 Hz
AQ            3.9584243 sec
RG            181
DW            60.400 usec
DE            6.50 usec
TE            298.3 K
D1            1.00000000 sec
D11           1
D10           1
    
```

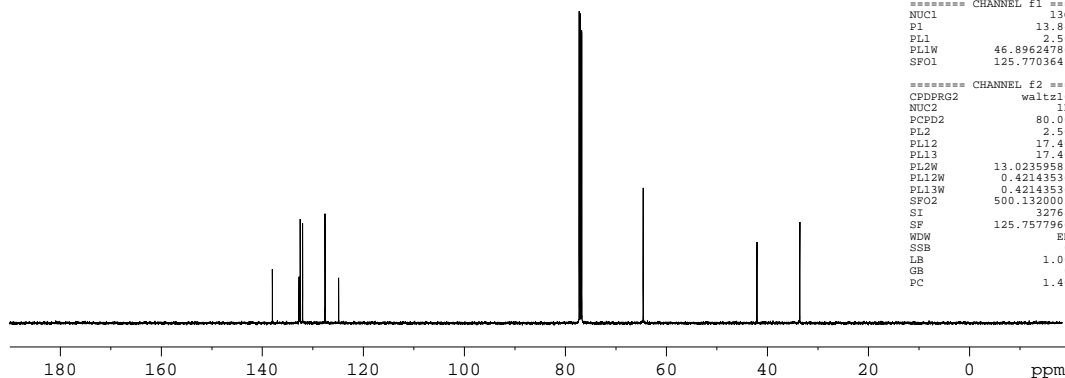


YWQ00041C



```

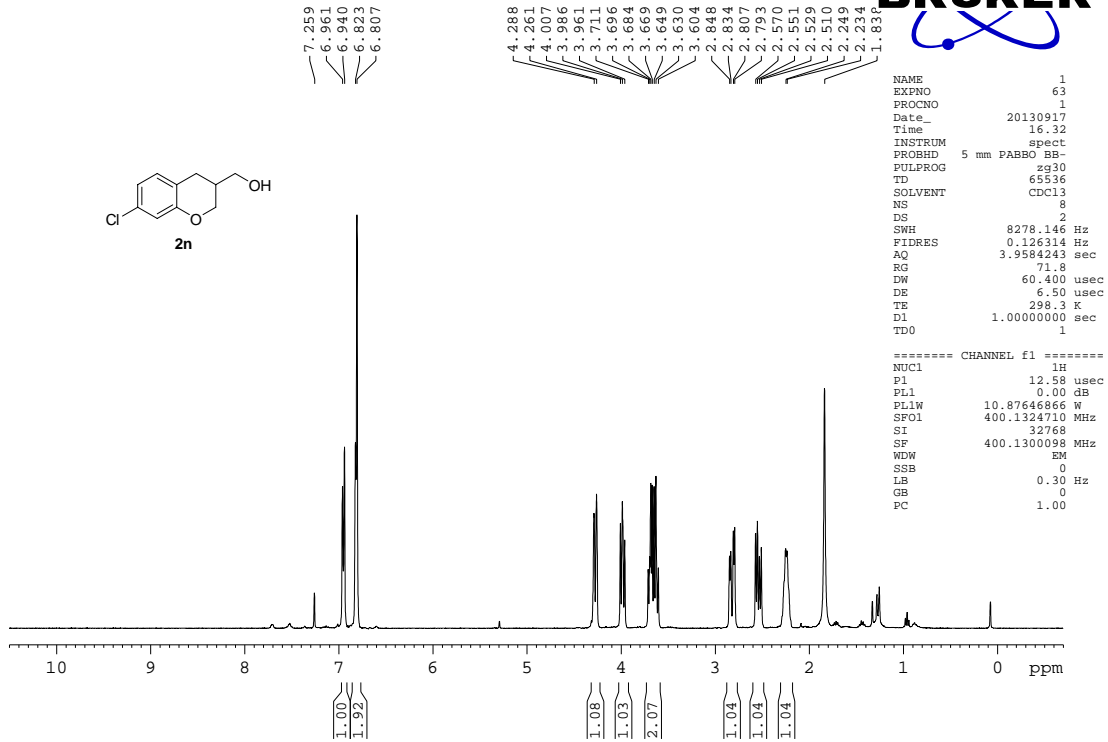
NAME          YWQ0041C
EXPNO         1
PROCNO        1
Date_         20130919
Time          9.16
INSTRUM       Spect
PROBHD        5 mm PABBO BB-
PULPROG       zgpg30
TD            65536
SOLVENT       CDCl3
NS            786
DS            4
SWH           29761.904 Hz
FIDRES        0.454131 Hz
AQ            1.1010548 sec
RG            203
DW            16.800 usec
DE            6.50 usec
TE            298.1 K
D1            2.00000000 sec
D11           0.03000000 sec
D10           1
    
```



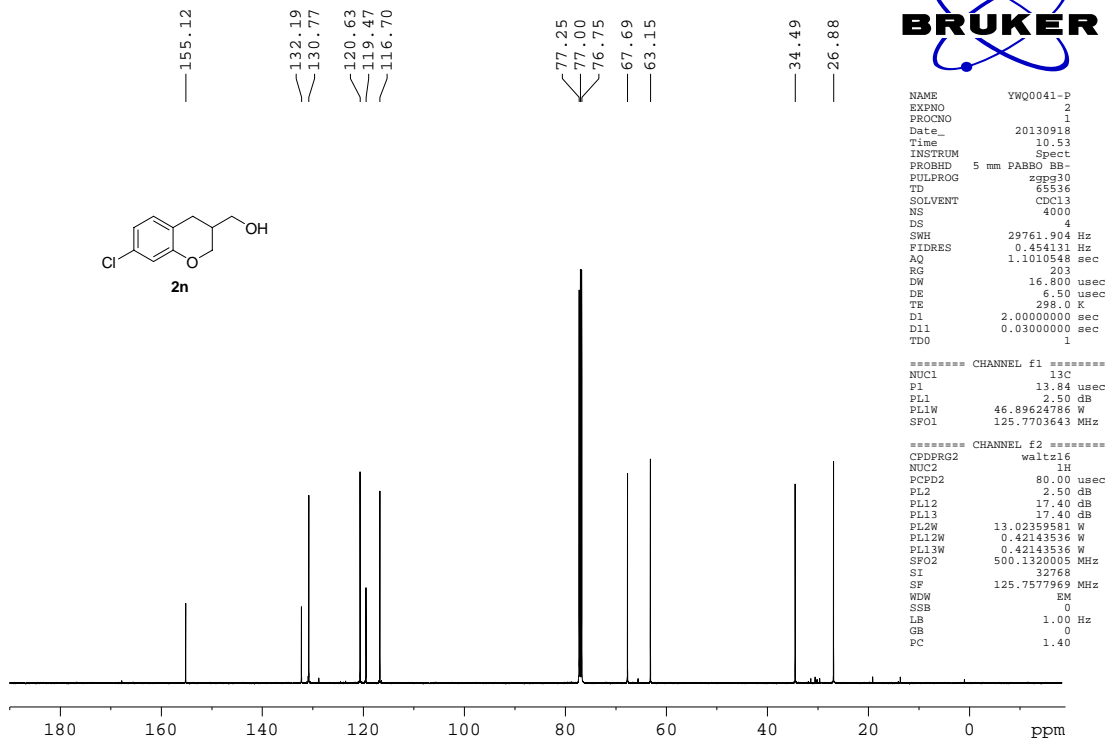
```

===== CHANNEL f1 =====
NUC1          13C
P1            13.84 usec
PL1           2.50 dB
PL1W          46.89624786 W
SF01          125.7703643 MHz

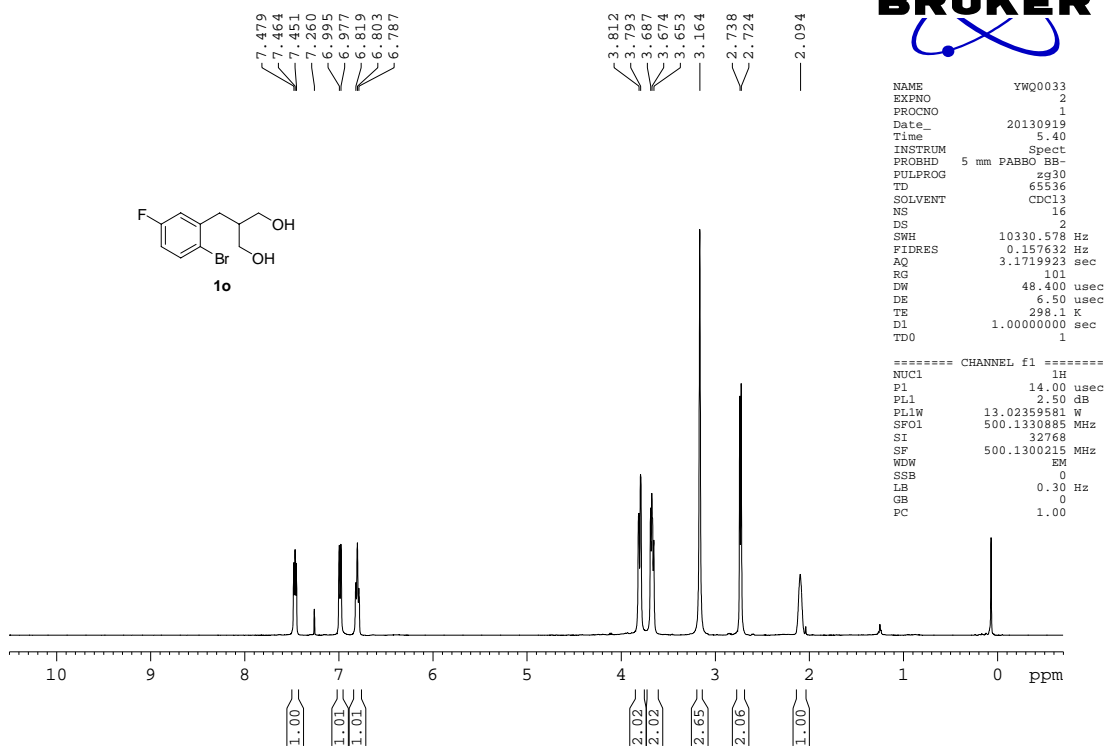
===== CHANNEL f2 =====
CPDPRG2       waltz16
NUC2          1H
PCPD2         80.00 usec
PL2           2.50 dB
PL12          17.40 dB
PL13          17.40 dB
PL1W          13.02359561 W
PL2W          0.42143536 W
PL12W         0.42143536 W
PL13W         0.42143536 W
SFO2          500.1320005 MHz
SI            32768
SF            125.7577966 MHz
WDW           EM
SSB           0
LB            1.00 Hz
GB            0
PC            1.40
    
```



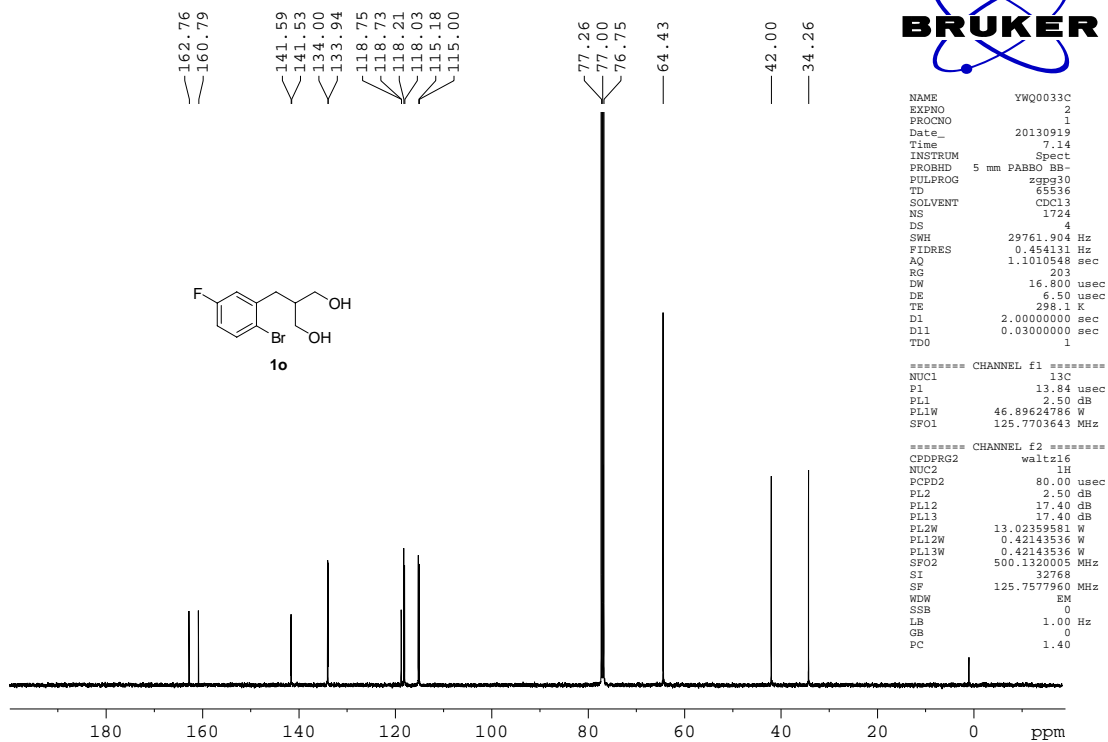
YWQ0041-P



YWQ0033H

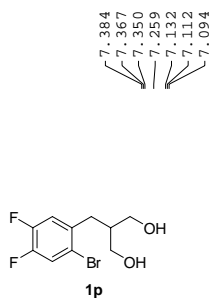


YWQ0033C





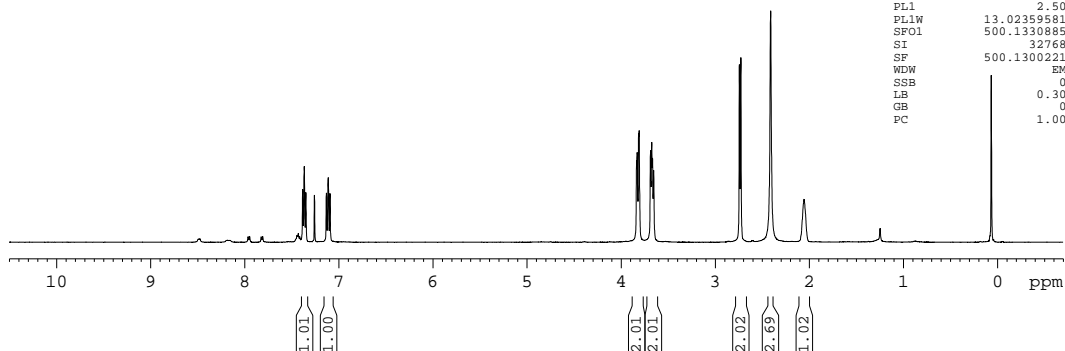
YWQ0039



3.834  
3.829  
3.813  
3.808  
3.687  
3.675  
3.666  
3.654  
2.742  
2.727  
2.410  
2.055

```

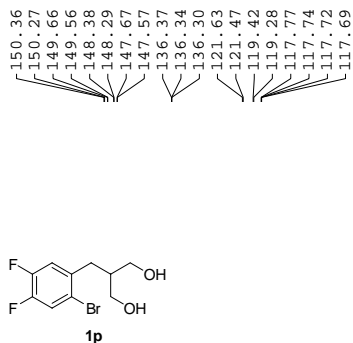
NAME          YWQ0039
EXPNO         1
PROCNO        1
Date_         20130916
Time          5.22
INSTRUM       Spect
PROBHD        5 mm PABBO BB-
PULPROG       zg30
TD            65536
SOLVENT       CDCl3
NS            16
DS            2
SWH           10330.578 Hz
FIDRES        0.157632 Hz
AQ            3.1719923 sec
RG            161
DW            48.400 usec
DE            6.50 usec
TE            298.3 K
D1            1.0000000 sec
D11           1
TDO           1
    
```



```

===== CHANNEL f1 =====
NUC1          1H
P1            14.00 usec
PL1           2.50 dB
PL1W          13.02359581 W
SF01          500.1330885 MHz
SI            32768
SF            500.1300221 MHz
WDW           EM
SSB           0
LB            0.30 Hz
GB            0
PC            1.00
    
```

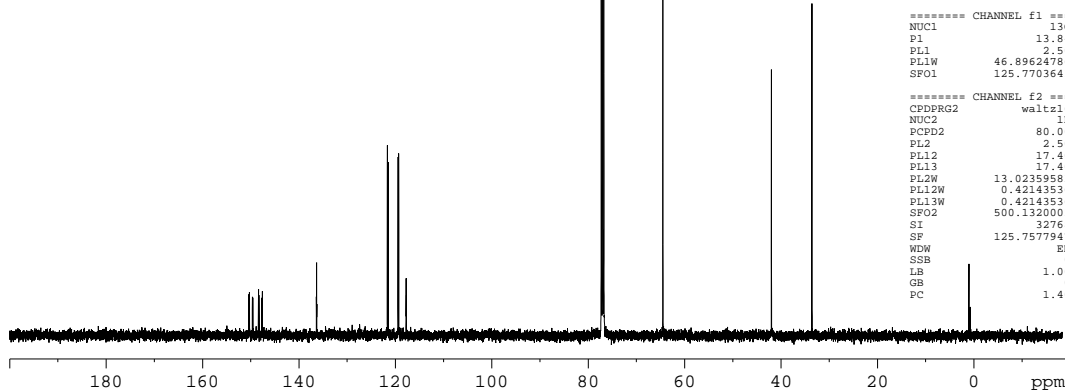
YWQ0039C



77.25  
77.00  
76.75  
64.49  
41.96  
33.56

```

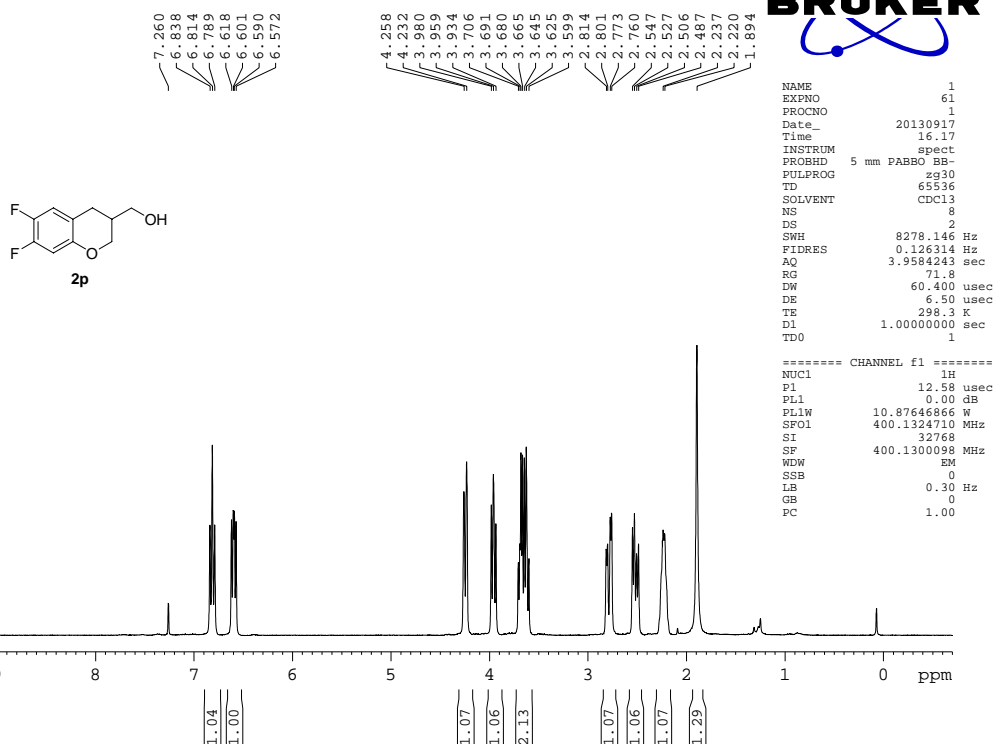
NAME          YWQ0039C
EXPNO         1
PROCNO        1
Date_         20130927
Time          5.45
INSTRUM       Spect
PROBHD        5 mm PABBO BB-
PULPROG       zgpg30
TD            65536
SOLVENT       CDCl3
NS            1043
DS            4
SWH           29761.904 Hz
FIDRES        0.454131 Hz
AQ            1.1010548 sec
RG            203
DW            16.800 usec
DE            6.50 usec
TE            298.0 K
D1            2.0000000 sec
D11           0.03000000 sec
TDO           1
    
```



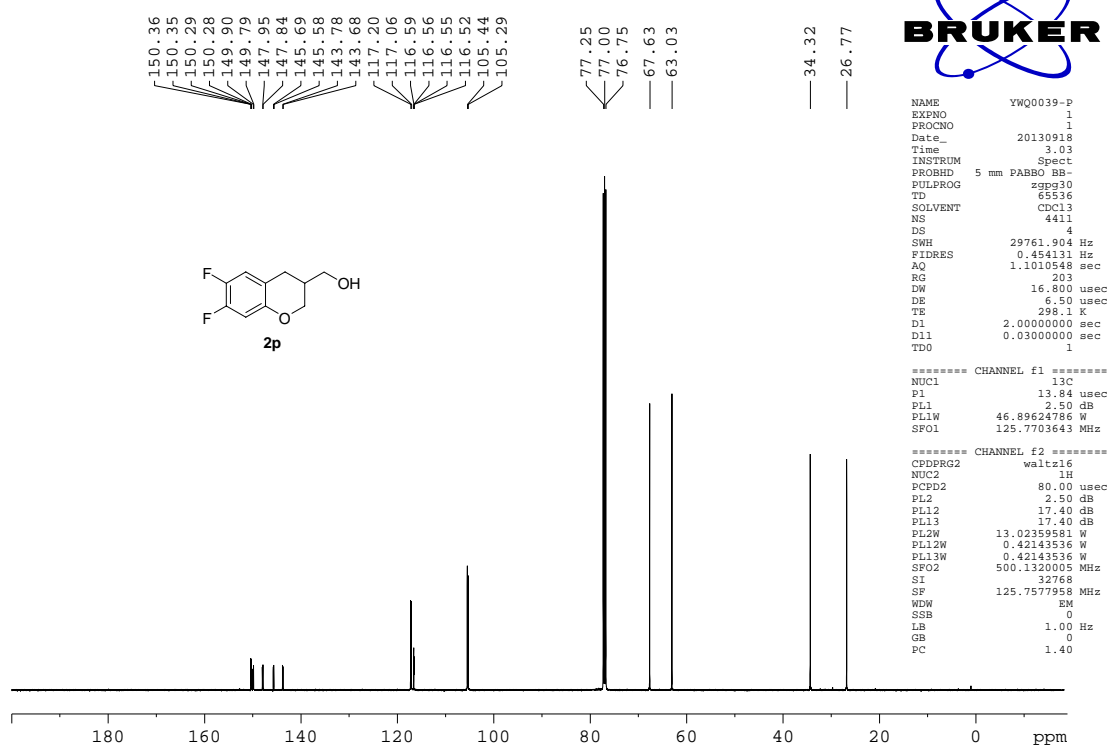
```

===== CHANNEL f1 =====
NUC1          13C
P1            13.84 usec
PL1           2.50 dB
PL1W          46.89624786 W
SF01          125.7703643 MHz

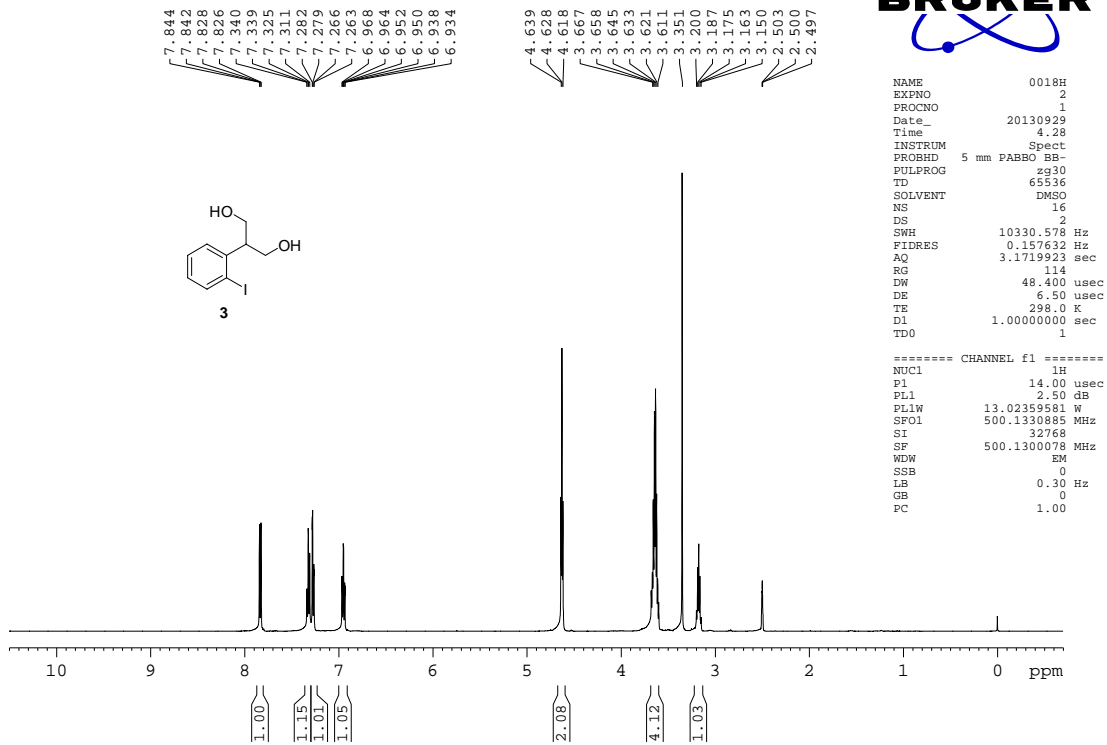
===== CHANNEL f2 =====
CPDPRG2       waltz16
NUC2          1H
PCPD2         80.00 usec
PL2           2.50 dB
PL12          17.40 dB
PL13          17.40 dB
PL1W          13.02359581 W
PL12W         0.42143536 W
PL13W         0.42143536 W
SF02          500.1320005 MHz
SI            32768
SF            125.7577947 MHz
WDW           EM
SSB           0
LB            1.00 Hz
GB            0
PC            1.40
    
```



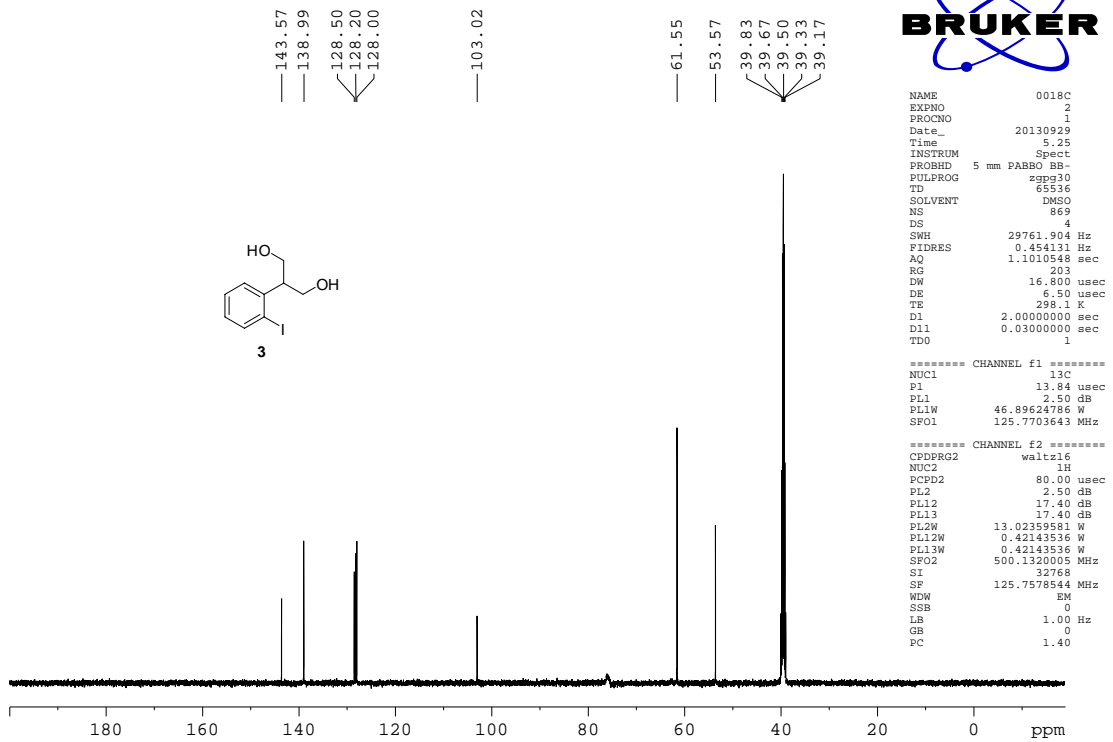
YWQ0039-P



0018H

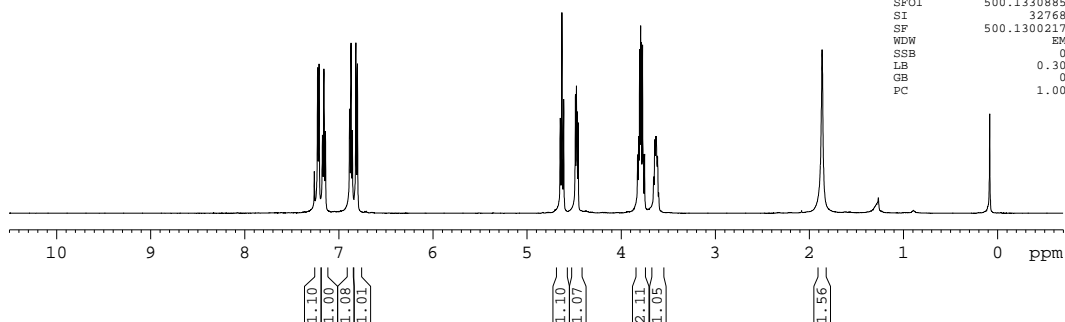
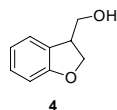


0018C



0018P

7.260  
7.225  
7.210  
7.174  
7.158  
7.143  
6.885  
6.871  
6.856  
6.818  
6.802  
4.646  
4.628  
4.610  
4.485  
4.475  
4.467  
4.457  
3.824  
3.813  
3.803  
3.791  
3.785  
3.772  
3.764  
3.750  
3.653  
3.642  
3.629  
3.624  
3.612  
3.600  
1.861



```

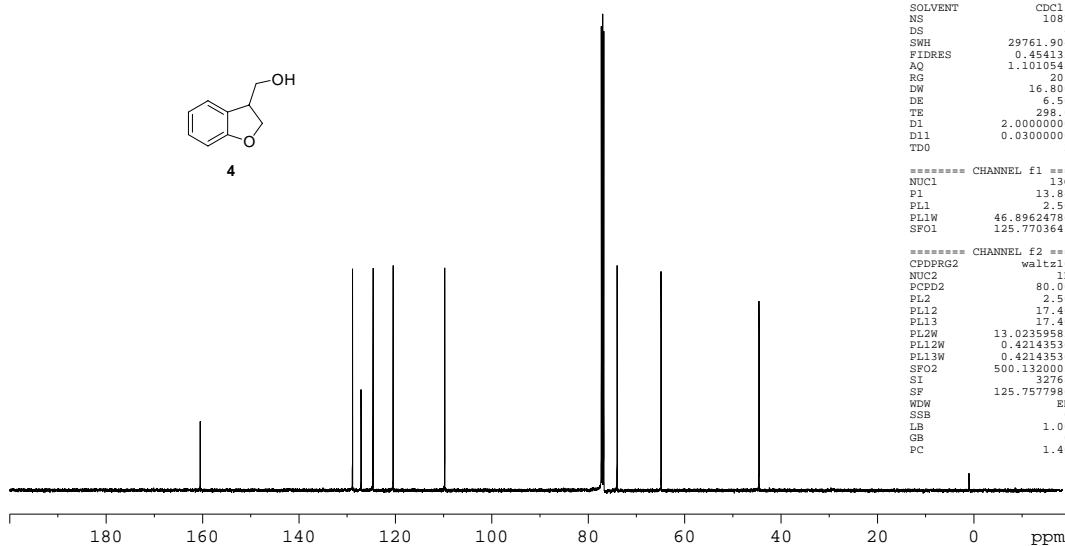
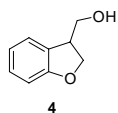
NAME      0018P
EXPNO    2
PROCNO   1
Date_    20130929
Time     3.17
INSTRUM  Spect
PROBHD   5 mm PABBO BB-
PULPROG  zg30
TD       65536
SOLVENT  CDCl3
NS       16
DS       2
SWH      10330.578 Hz
FIDRES   0.157632 Hz
AQ       3.1719923 sec
RG       101
DW       48.400 usec
DE       6.50 usec
TE       298.0 K
D1       1.00000000 sec
TDO      1
    
```

```

===== CHANNEL f1 =====
NUC1      1H
P1        14.00 usec
PL1       2.50 dB
PL1W      13.02359581 W
SFO1      500.1330885 MHz
SI        32768
SF        500.1300217 MHz
WDW       EM
SSB       0
LB        0.30 Hz
GB        0
PC        1.00
    
```

0018PC

160.44  
128.85  
127.07  
124.58  
120.42  
109.71  
77.25  
77.00  
76.75  
73.99  
64.86  
44.54



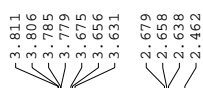
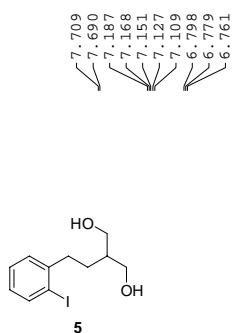
```

NAME      0018PC
EXPNO    2
PROCNO   1
Date_    20130929
Time     4.18
INSTRUM  Spect
PROBHD   5 mm PABBO BB-
PULPROG  zgpg30
TD       65536
SOLVENT  CDCl3
NS       1087
DS       4
SWH      29761.904 Hz
FIDRES   0.454131 Hz
AQ       1.1010548 sec
RG       203
DW       16.800 usec
DE       6.50 usec
TE       298.0 K
D1       2.00000000 sec
D11      0.03000000 sec
TDO      1
    
```

```

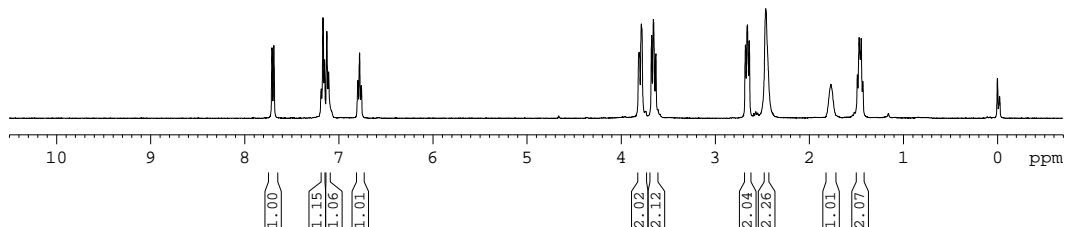
===== CHANNEL f1 =====
NUC1      13C
P1        13.84 usec
PL1       2.50 dB
PL1W      46.89624786 W
SFO1      125.7703643 MHz

===== CHANNEL f2 =====
CPDPRG2  waltz16
NUC2      1H
PCPD2    80.00 usec
PL2       2.50 dB
PL12     17.40 dB
PL13     17.40 dB
PL2W     13.02359581 W
PL12W    0.42143536 W
PL13W    0.42143536 W
SFO2     500.1320005 MHz
SI        32768
SF        125.7577986 MHz
WDW       EM
SSB       0
LB        1.00 Hz
GB        0
PC        1.40
    
```

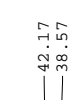
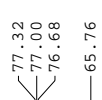
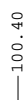
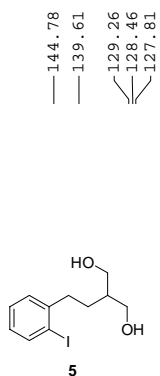


```

NAME      Chongce
EXPNO     20
PROCNO    1
Date_     20131008
Time      12.49
INSTRUM   spect
PROBHD    5 mm PABBO BB-
PULPROG   zg30
TD         65536
SOLVENT   CDCl3
NS         8
DS         2
SWH        8278.146 Hz
FIDRES     0.126314 Hz
AQ         3.9584243 sec
RG         181
DW         60.400 usec
DE         6.50 usec
TE         297.9 K
D1         1.00000000 sec
TD0        1
    
```



ywq-0012C



```

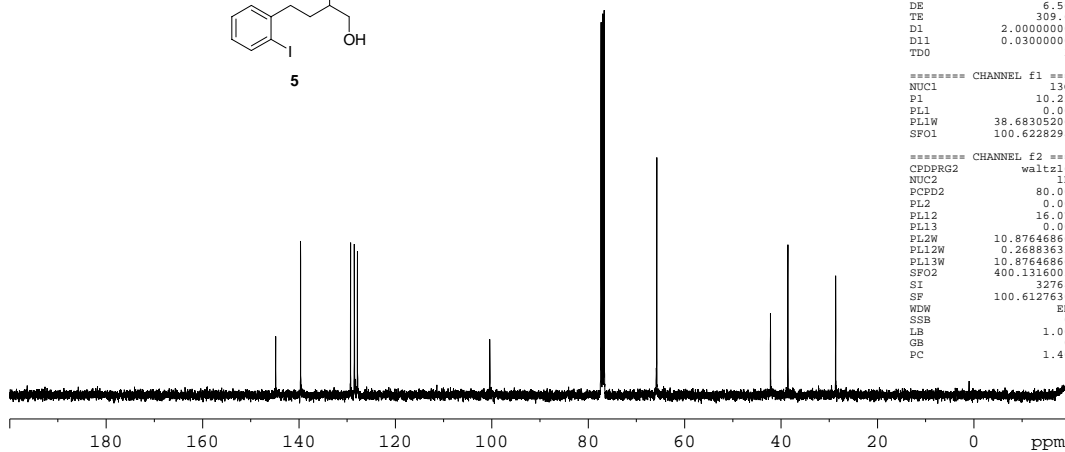
NAME      0012
EXPNO     67
PROCNO    1
Date_     20131001
Time      12.45
INSTRUM   spect
PROBHD    5 mm PABBO BB-
PULPROG   zgpg30
TD         65536
SOLVENT   CDCl3
NS         307
DS         4
SWH        23980.814 Hz
FIDRES     0.365918 Hz
AQ         1.3664756 sec
RG         1149.4
DW         20.850 usec
DE         6.50 usec
TE         309.6 K
D1         2.00000000 sec
D11        0.03000000 sec
TD0        1
    
```

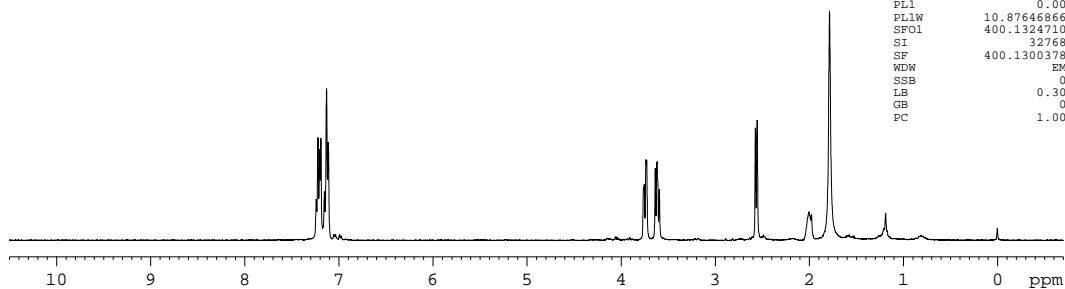
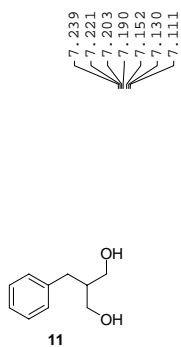
```

===== CHANNEL f1 =====
NUC1      13C
P1        10.25 usec
PL1       0.00 dB
PL1W      38.68305206 W
SF01      100.6228298 MHz
    
```

```

===== CHANNEL f2 =====
CPDPRG2   waltz16
NUC2      1H
PCPD2     80.00 usec
PL2       0.00 dB
PL12      16.07 dB
PL13      0.00 dB
PL2W      10.87646866 W
PL12W     0.26883632 W
PL13W     10.87646866 W
SFO2      400.1316005 MHz
SI         32768
SF         100.6127630 MHz
WDW        EM
SSB        0
LB         1.00 Hz
GB         0
PC         1.40
    
```





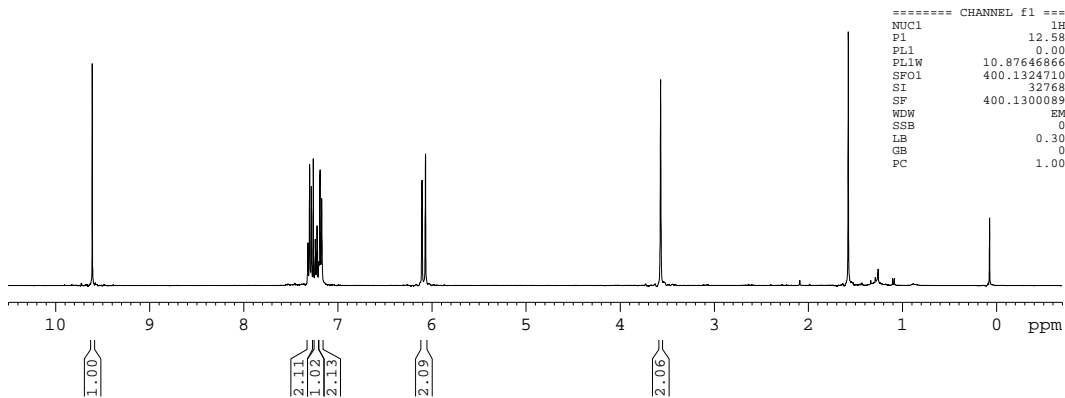
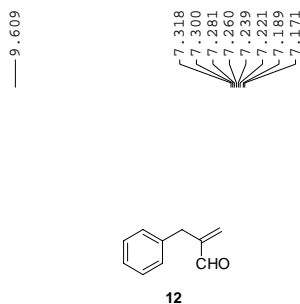
```

NAME          22
EXPNO         22
PROCNO        1
Date_         20130925
Time          11.40
INSTRUM       spect
PROBHD        5 mm PABBO BB-
PULPROG       zg30
TD            65536
SOLVENT       CDCl3
NS            8
DS            2
SWH           8278.146 Hz
FIDRES        0.126314 Hz
AQ            3.9584243 sec
RG            362
DW            60.400 usec
DE            6.50 usec
TE            298.7 K
D1            1.00000000 sec
TD0           1
    
```

```

===== CHANNEL f1 =====
NUC1          1H
P1            12.58 usec
PL1           0.00 dB
PL1W          10.87646866 W
SF01          400.1324710 MHz
SI            32768
SF            400.1300379 MHz
WDW           EM
SSB           0
LB            0.30 Hz
GB            0
PC            1.00
    
```

35732C-2



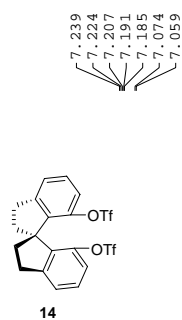
```

NAME          22
EXPNO         22
PROCNO        1
Date_         20130529
Time          23.32
INSTRUM       spect
PROBHD        5 mm PABBO BB-
PULPROG       zg30
TD            65536
SOLVENT       CDCl3
NS            16
DS            2
SWH           8278.146 Hz
FIDRES        0.126314 Hz
AQ            3.9584243 sec
RG            322.5
DW            60.400 usec
DE            6.50 usec
TE            299.0 K
D1            1.00000000 sec
TD0           1
    
```

```

===== CHANNEL f1 =====
NUC1          1H
P1            12.58 usec
PL1           0.00 dB
PL1W          10.87646866 W
SF01          400.1324710 MHz
SI            32768
SF            400.1300089 MHz
WDW           EM
SSB           0
LB            0.30 Hz
GB            0
PC            1.00
    
```

Y-Y-1

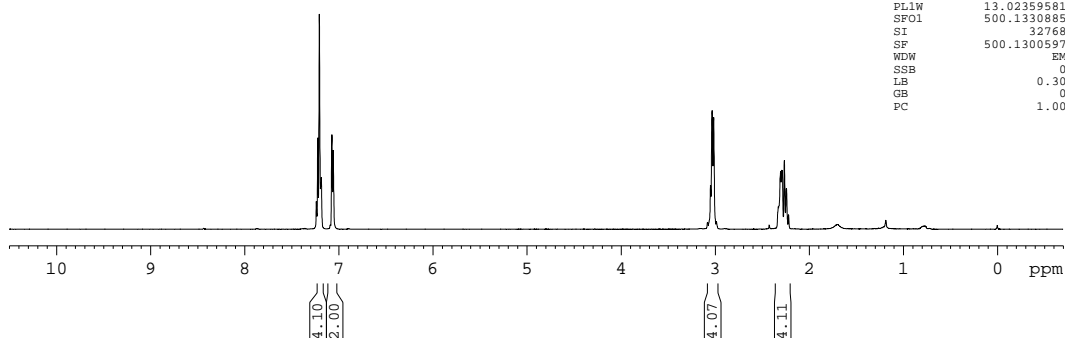


3.050  
3.033  
3.016  
2.327  
2.303  
2.288  
2.263  
2.242  
2.219



```

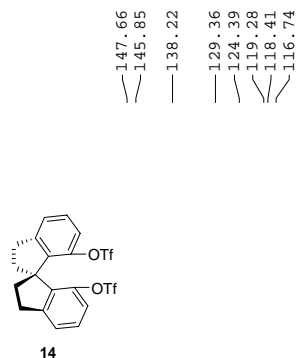
NAME          Y-Y-1
EXPNO         1
PROCNO        1
Date_         20131108
Time          6.28
INSTRUM       Spect
PROBHD        5 mm PABBO BB-
PULPROG       zg30
TD            65536
SOLVENT       CDCl3
NS            16
DS            2
SWH           10330.578 Hz
FIDRES        0.157632 Hz
AQ            3.1719923 sec
RG            203
DW            48.400 usec
DE            6.50 usec
TE            298.1 K
D1            1.00000000 sec
D11           1
TDO           1
    
```



```

===== CHANNEL f1 =====
NUC1          1H
P1            14.00 usec
PL1           2.50 dB
PL1W          13.02359581 W
SF01          500.1330885 MHz
SI            32768
SF            500.1300597 MHz
WDW           EM
SSB           0
LB            0.30 Hz
GB            0
PC            1.00
    
```

Y-Y-3CC



77.25  
77.00  
76.75

59.34

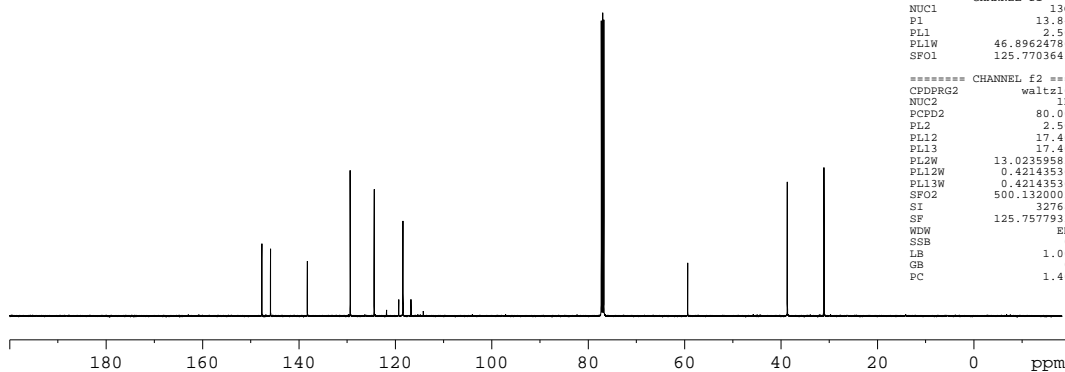
38.67

31.07



```

NAME          Y-Y-3CC
EXPNO         1
PROCNO        1
Date_         20131108
Time          8.31
INSTRUM       Spect
PROBHD        5 mm PABBO BB-
PULPROG       zgpg30
TD            65536
SOLVENT       CDCl3
NS            1362
DS            4
SWH           29761.904 Hz
FIDRES        0.454131 Hz
AQ            1.1010548 sec
RG            203
DW            16.800 usec
DE            6.50 usec
TE            298.0 K
D1            2.00000000 sec
D11           0.03000000 sec
TDO           1
    
```

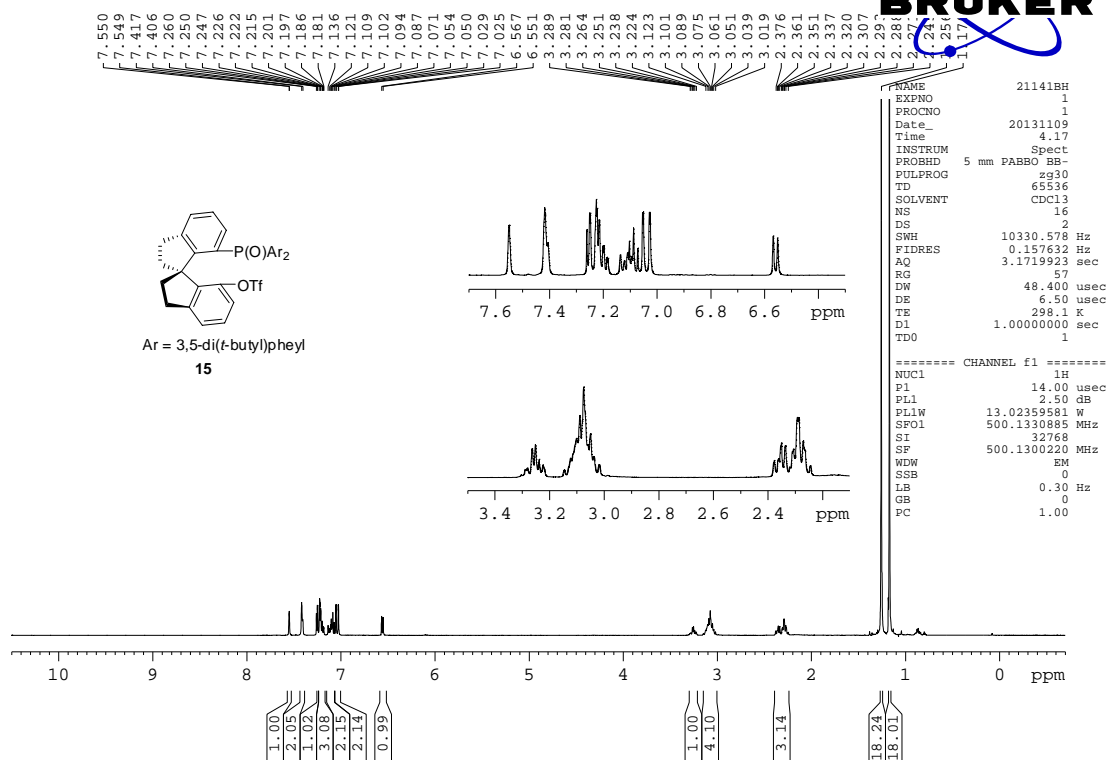


```

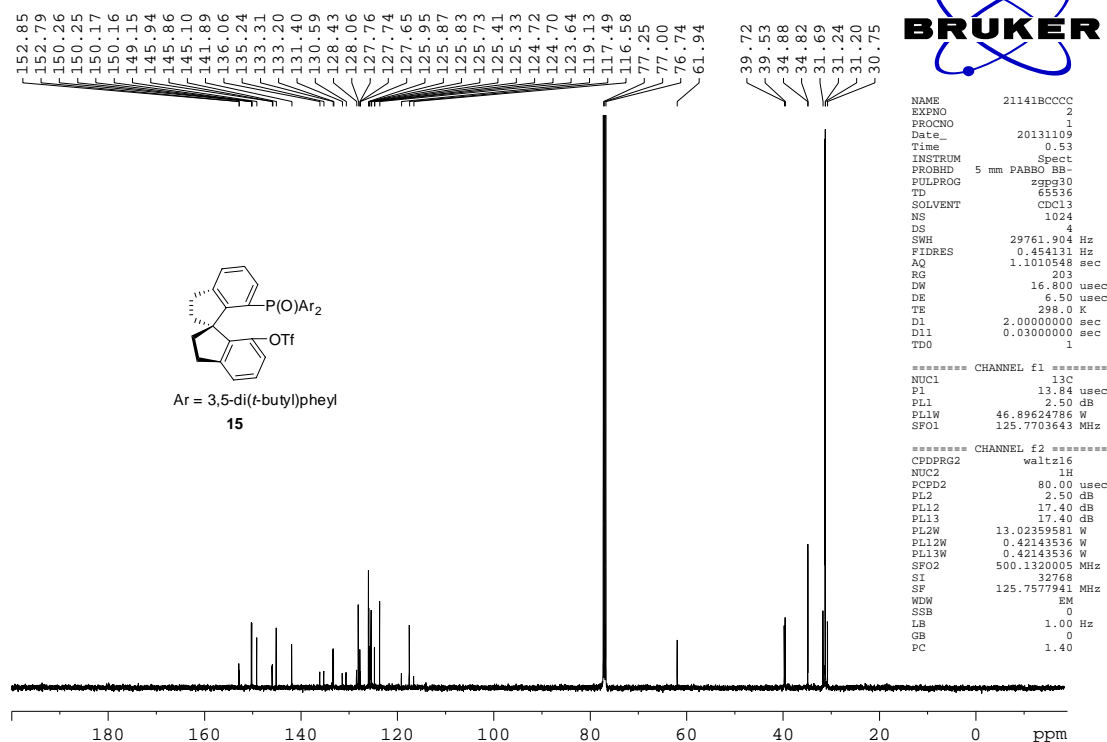
===== CHANNEL f1 =====
NUC1          13C
P1            13.84 usec
PL1           2.50 dB
PL1W          46.89624786 W
SF01          125.7703643 MHz

===== CHANNEL f2 =====
CPDPRG2       waltz16
NUC2          1H
PCPD2         80.00 usec
PL2           2.50 dB
PL12          17.40 dB
PL13          17.40 dB
PL14          17.40 dB
PL2W          13.02359581 W
PL12W         0.42143536 W
PL13W         0.42143536 W
PL14W         0.42143536 W
SF02          500.1320095 MHz
SI            32768
SF            125.7577931 MHz
WDW           EM
SSB           0
LB            1.00 Hz
GB            0
PC            1.40
    
```

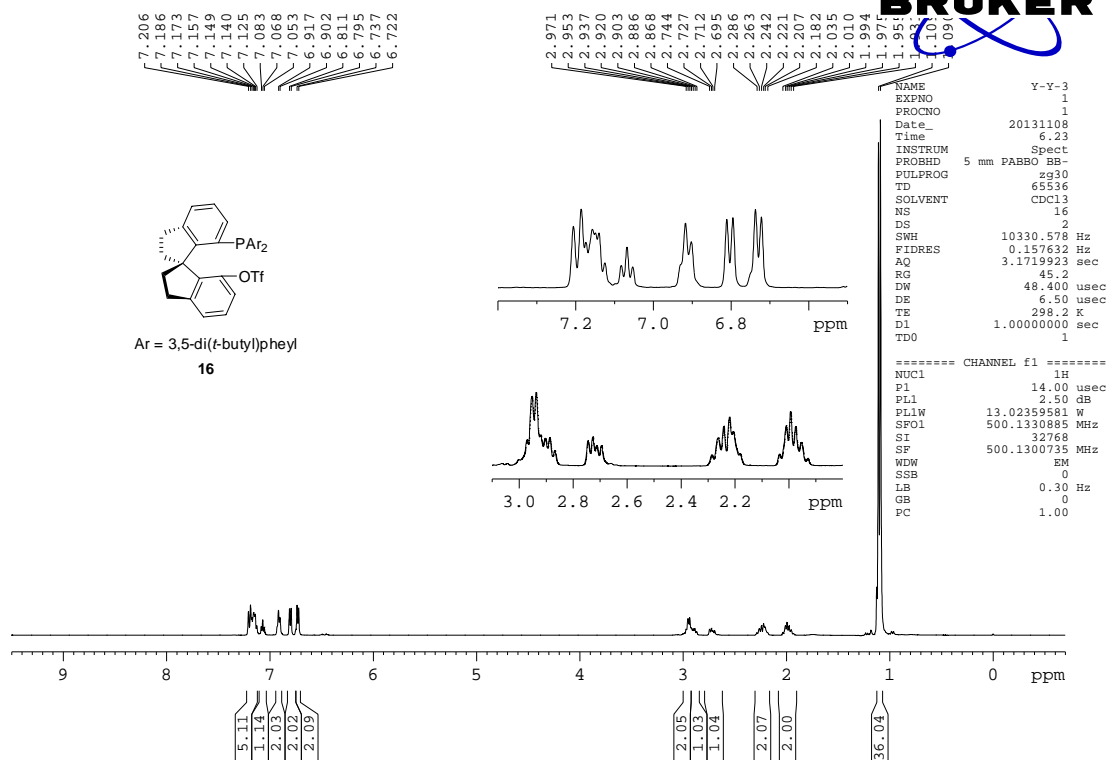
21141BH



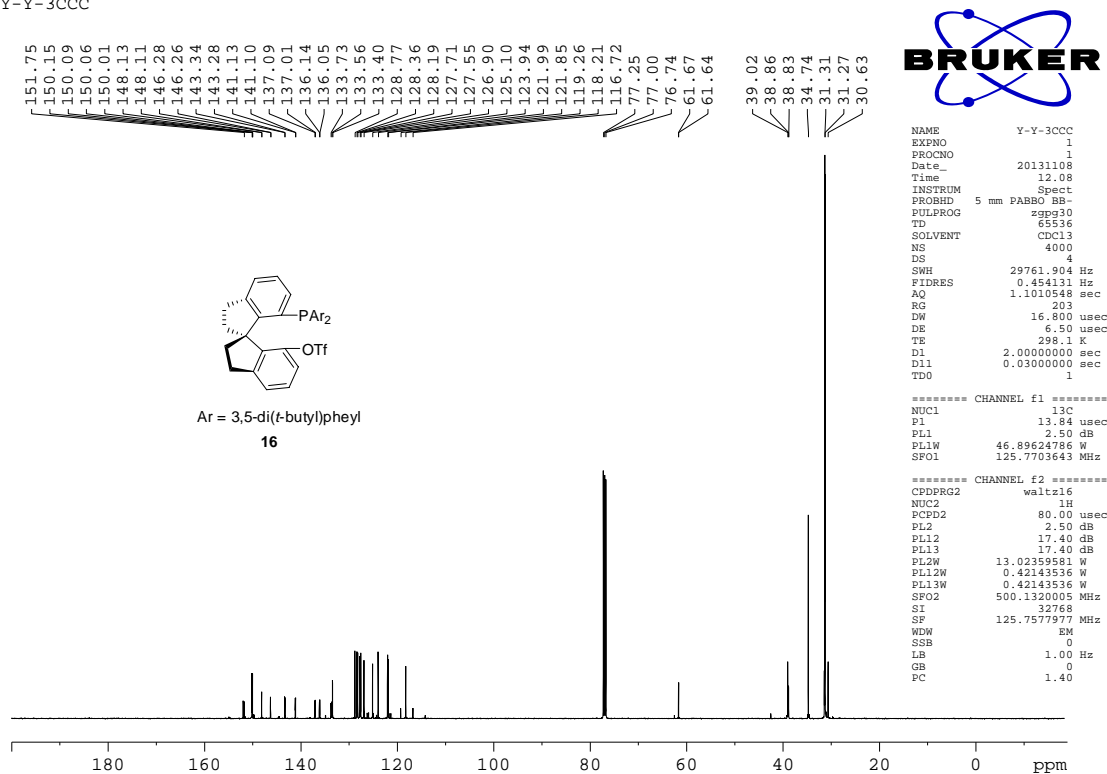
21141BCCCC



Y-Y-3

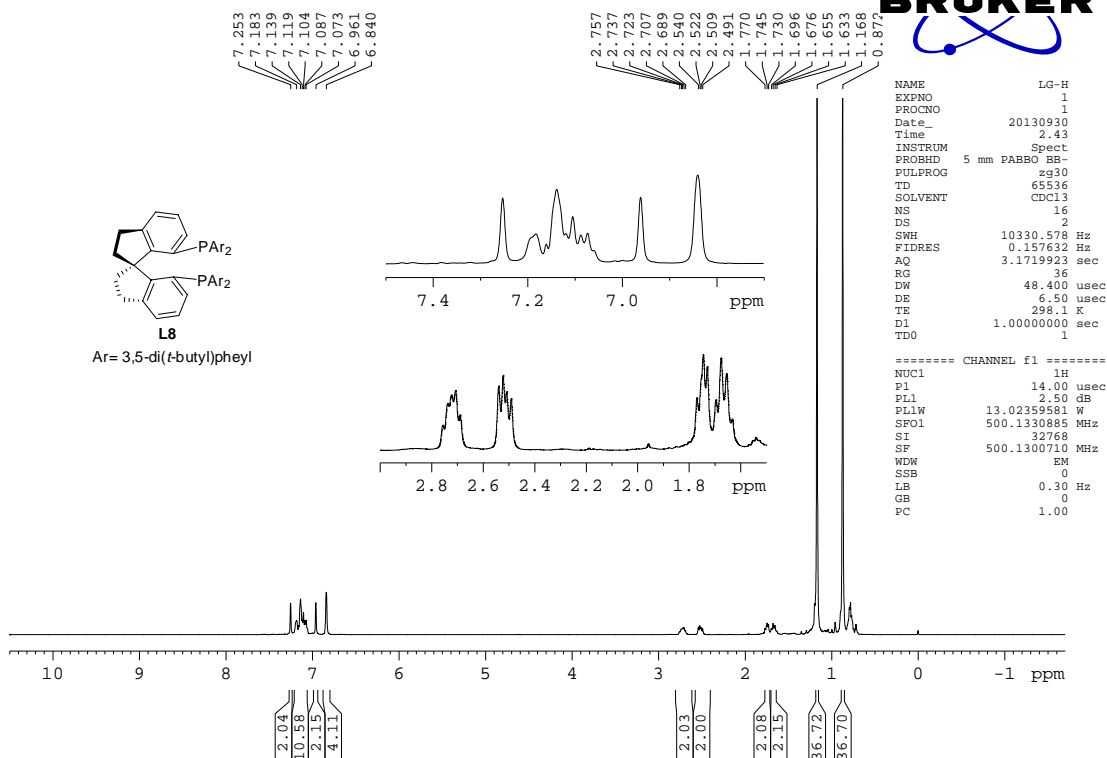


Y-Y-3CCC

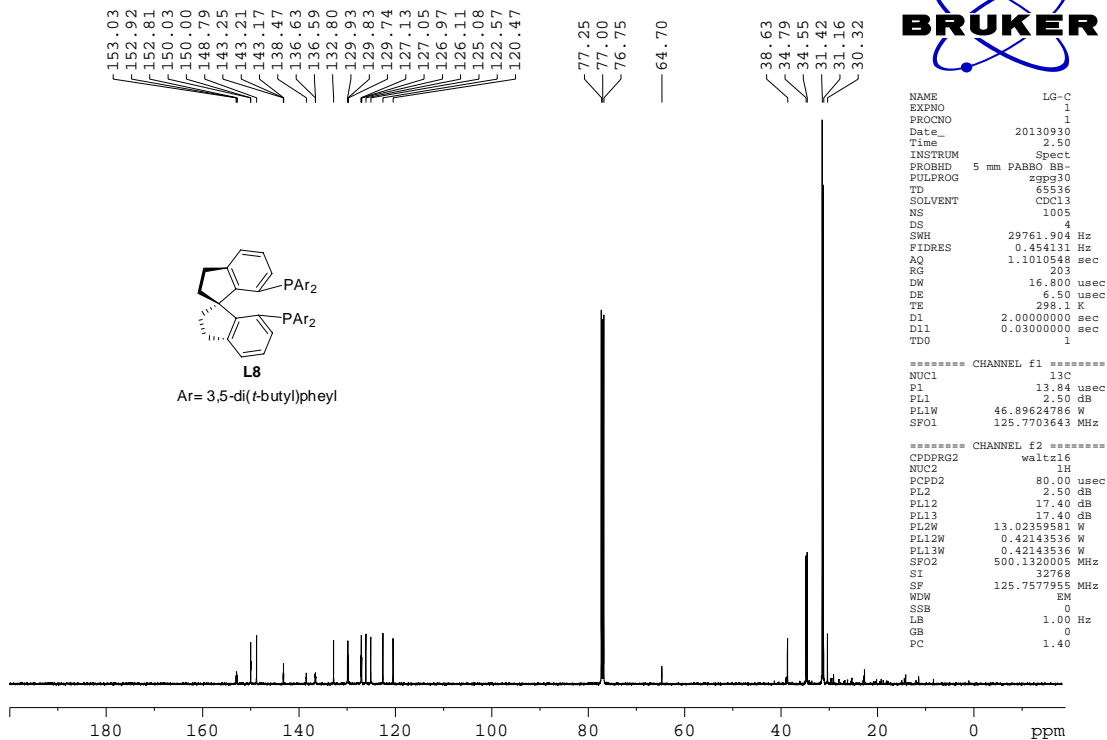




LG-H



LG-C

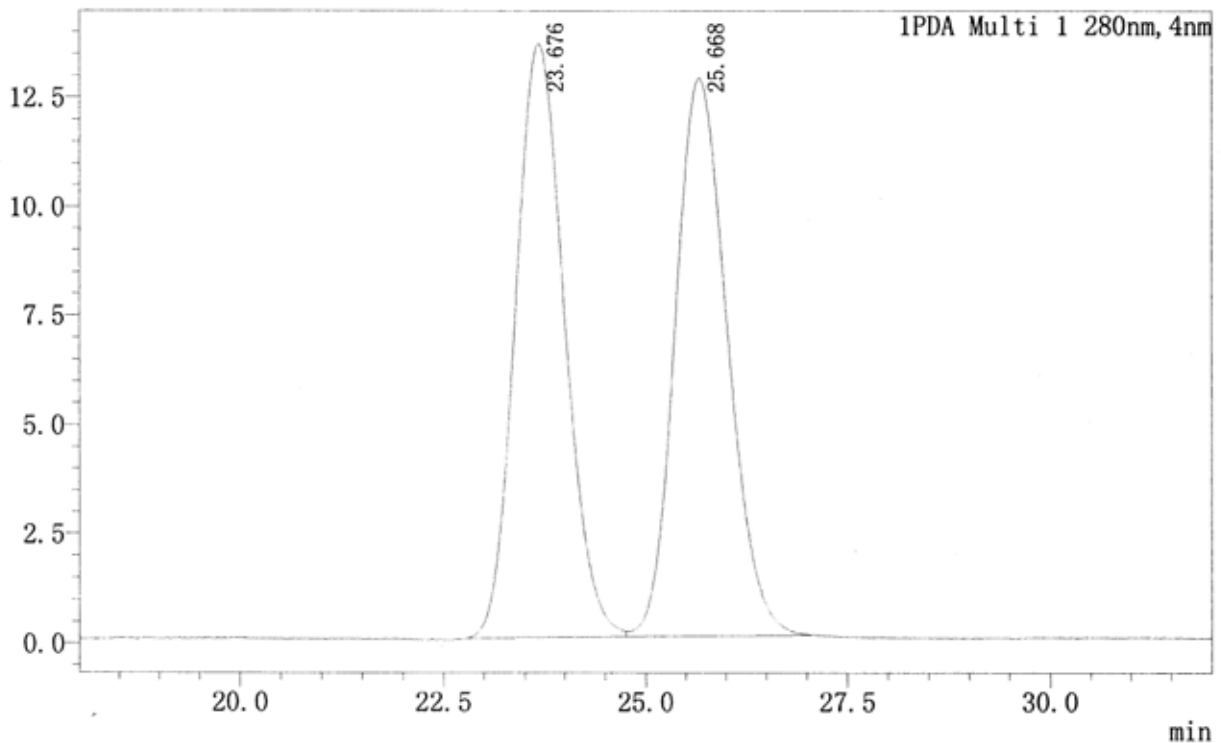


## 8. HPLC Spectra.

# Data Report

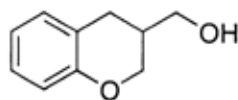
Sample Information  
Sample Name : 001P(RS),OD-H,3%  
Tray# : 1  
Vial# : 62  
Injection Volume : 7  
Data File : 001P(RS),OD-H,3%.lcd  
Method File : 3%,60min.lcm  
Report Format File : Templates.lsr  
Sample Type : Unknown  
Acquired by : System Administrator  
Date Acquired : 2013/8/2 12:24:30  
Date Processed : 2013/8/2 12:57:57

mAU



PDA Ch1 280nm

NO.	Ret. Time	Height (uAU)	Area(uAU*min)	Rel. Area %	Resolution(USP)
1	23.676	13593	573333	49.913	—
2	25.668	12758	575323	50.087	1.727

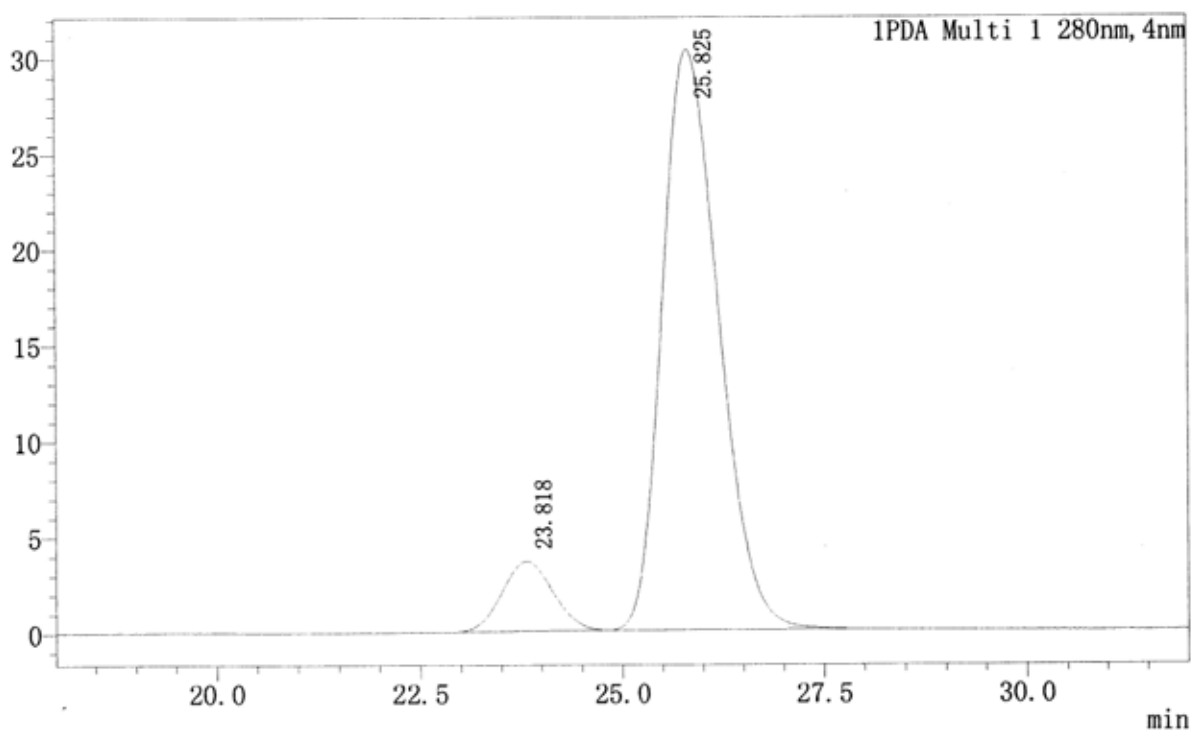


(rac)-2a

# Data Report

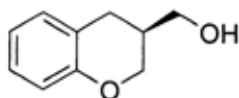
Sample Information  
Sample Name : 001-P,OD-H,3%  
Tray# : 1  
Vial# : 16  
Injection Volume : 10  
Data File : 001-P,OD-H,3%.lcd  
Method File : 3%,35MIN.lcm  
Report Format File : Templates.lsr  
Sample Type : Unknown  
Acquired by : System Administrator  
Date Acquired : 2013/8/7 15:23:33  
Date Processed : 2013/8/7 15:56:13

mAU



PDA Ch1 280nm

NO.	Ret. Time	Height (uAU)	Area (uAU*min)	Rel. Area %	Resolution (USP)
1	23.818	3599	157945	9.706	--
2	25.825	30245	1469363	90.294	1.632

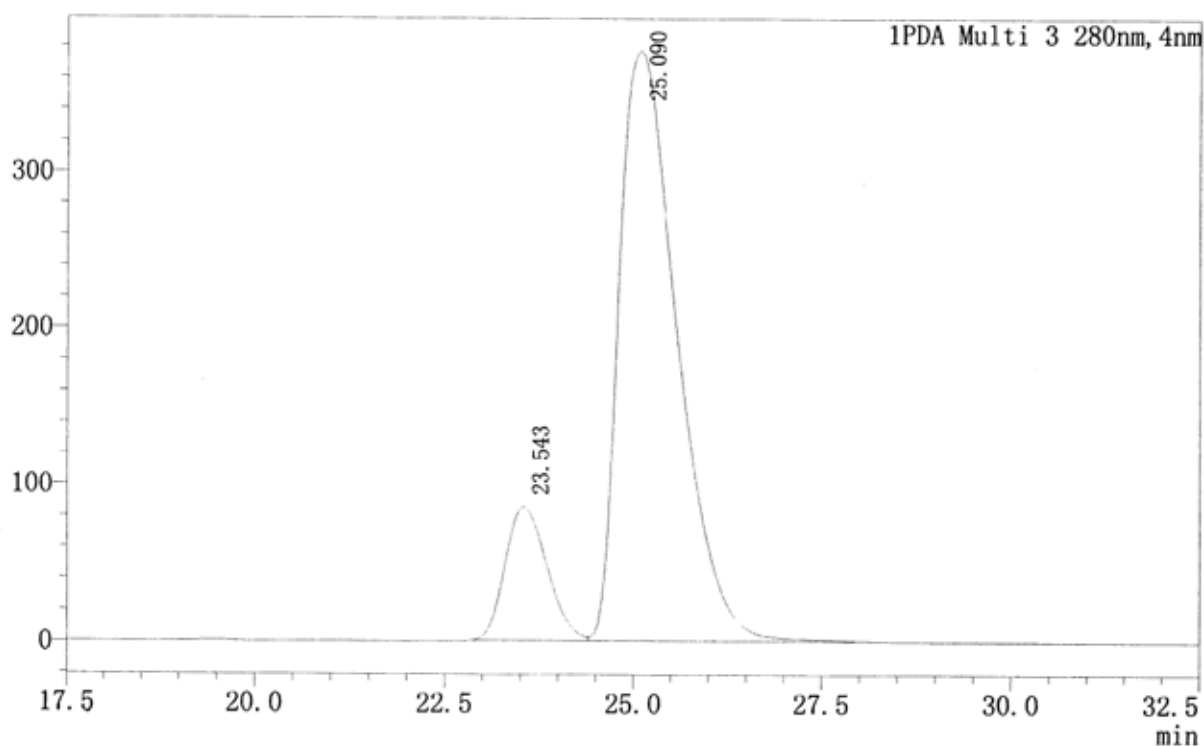


(S)-2a from 1a

# Data Report

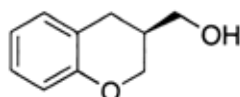
Sample Information  
Sample Name : 0020-P,3%,OD  
Tray# : 1  
Vial# : 3  
Injection Volume : 5  
Data File : 0020-P,3%,OD-H.lcd  
Method File : 3%,60MIN.lcm  
Report Format File : Templates.lsr  
Sample Type : Unknown  
Acquired by : System Administrator  
Date Acquired : 2013/9/29 0:01:48  
Date Processed : 2013/9/29 1:01:49

mAU



PDA Ch3 280nm

NO.	Ret. Time	Height (uAU)	Area (uAU*min)	Rel. Area %	Resolution(USP)
1	23.543	84989	3288879	14.020	--
2	25.090	376136	20169763	85.980	1.295

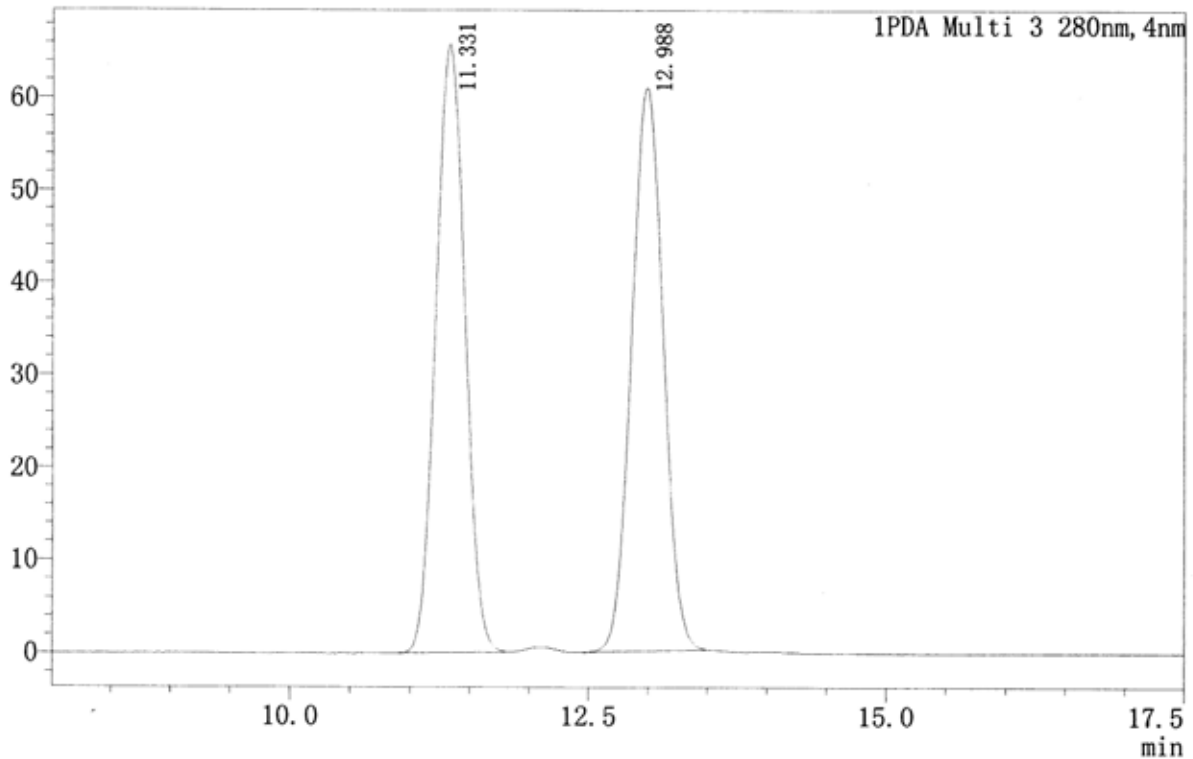


(S)-2a from 1a'

# Data Report

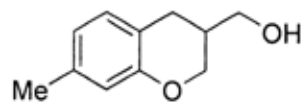
Sample Information  
Sample Name : 0040P(RS),AD-H,5%  
Tray# : 1  
Vial# : 81  
Injection Volume : 7  
Data File : 0040P(RS),AD-H,5%.lcd  
Method File : 5%,30MIN.lcm  
Report Format File : Templates.lsr  
Sample Type : Unknown  
Acquired by : System Administrator  
Date Acquired : 2013/9/16 11:50:43  
Date Processed : 2013/9/16 12:08:26

mAU



PDA Ch3 280nm

NO.	Ret. Time	Height (uAU)	Area (uAU*min)	Rel. Area %	Resolution (USP)
1	11.331	65768	1123683	49.911	—
2	12.988	60842	1127679	50.089	3.456

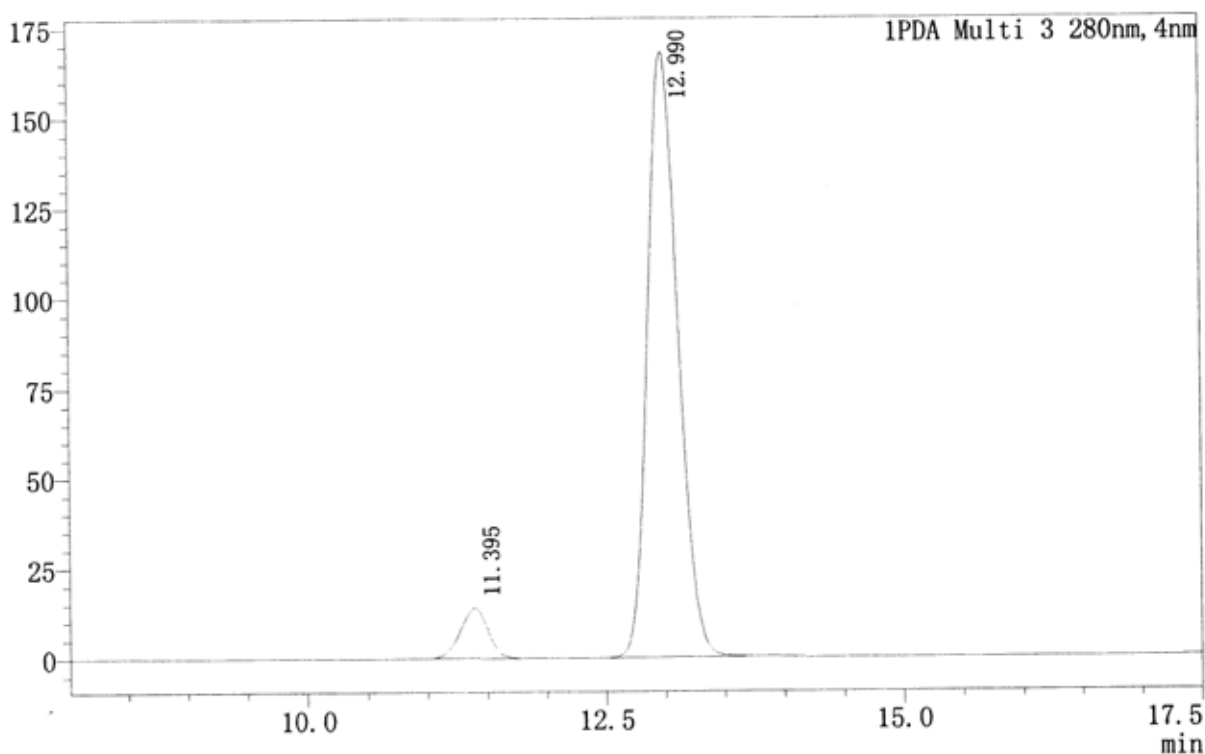


(rac)-2b

# Data Report

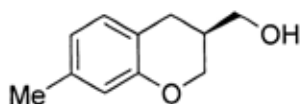
Sample Information  
Sample Name : 0040-P,AD-H,5%  
Tray# : 1  
Vial# : 2  
Injection Volume : 6  
Data File : 0040-P,AD-H,5%.lcd  
Method File : 5%,30MIN.lcm  
Report Format File : Templates.lsr  
Sample Type : Unknown  
Acquired by : System Administrator  
Date Acquired : 2013/9/16 12:30:22  
Date Processed : 2013/9/16 12:48:02

mAU



PDA Ch3 280nm

NO.	Ret. Time	Height (uAU)	Area (uAU*min)	Rel. Area %	Resolution(USP)
1	11.395	13877	223629	6.961	—
2	12.990	167951	2988827	93.039	3.488

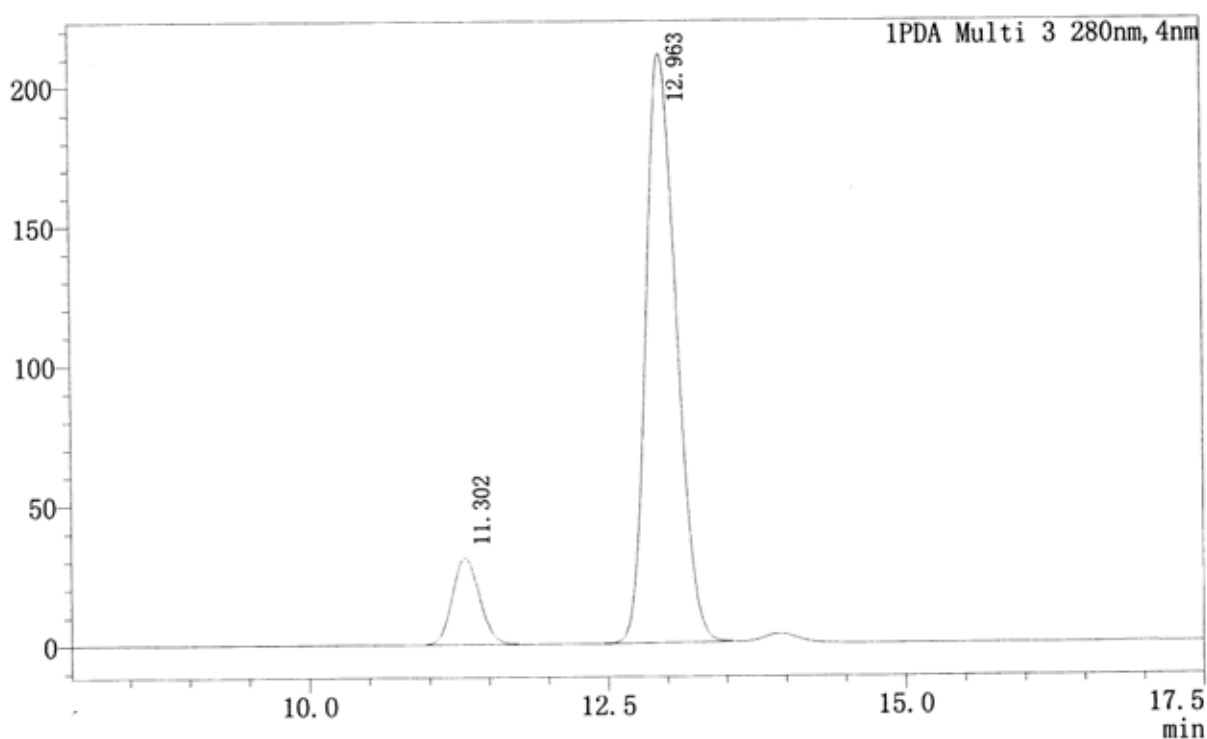


(S)-2b from 1b

# Data Report

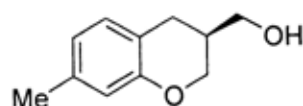
Sample Information  
Sample Name : 0042-P,AD-H,5%  
Tray# : 1  
Vial# : 1  
Injection Volume : 6  
Data File : 0042-P,AD-H,5%.lcd  
Method File : 5%,30MIN.lcm  
Report Format File : Templates.lsr  
Sample Type : Unknown  
Acquired by : System Administrator  
Date Acquired : 2013/9/16 15:05:50  
Date Processed : 2013/9/16 15:23:55

mAU



PDA Ch3 280nm

NO.	Ret. Time	Height (uAU)	Area (uAU*min)	Rel. Area %	Resolution (USP)
1	11.302	30997	492811	11.515	—
2	12.963	210965	3786754	88.485	3.647



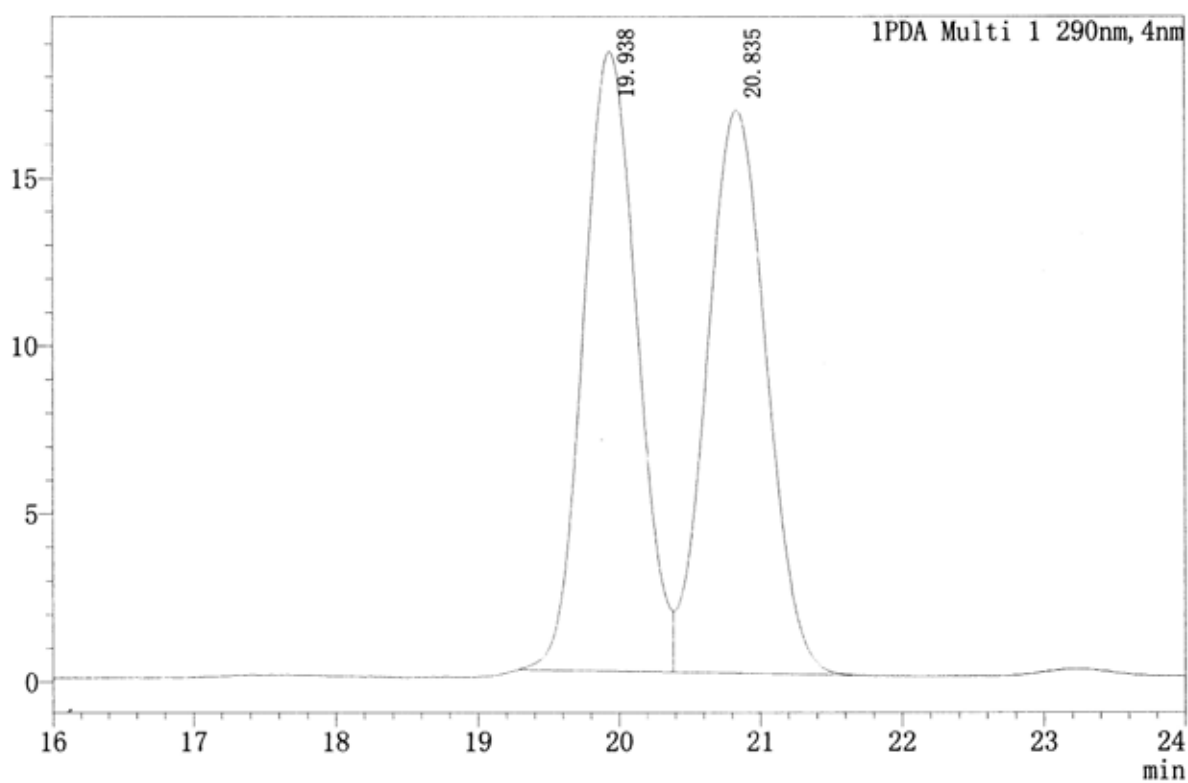
(S)-2b from 1b'

# Data Report

## Sample Information

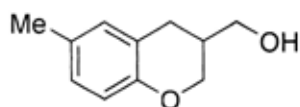
Sample Name : 002P(RS),AD-H,3%  
Tray# : 1  
Vial# : 93  
Injection Volume : 8  
Data File : 002P(RS),AD-H,3%,20'.lcl  
Method File : 5%,30MIN.lcm  
Report Format File : Templates.lsr  
Sample Type : Unknown  
Acquired by : System Administrator  
Date Acquired : 2013/8/8 13:16:50  
Date Processed : 2013/8/8 13:42:22

mAU



PDA Ch1 290nm

NO.	Ret. Time	Height (uAU)	Area (uAU*min)	Rel. Area %	Resolution(USP)
1	19.938	18434	488139	49.915	—
2	20.835	16742	489796	50.085	1.204

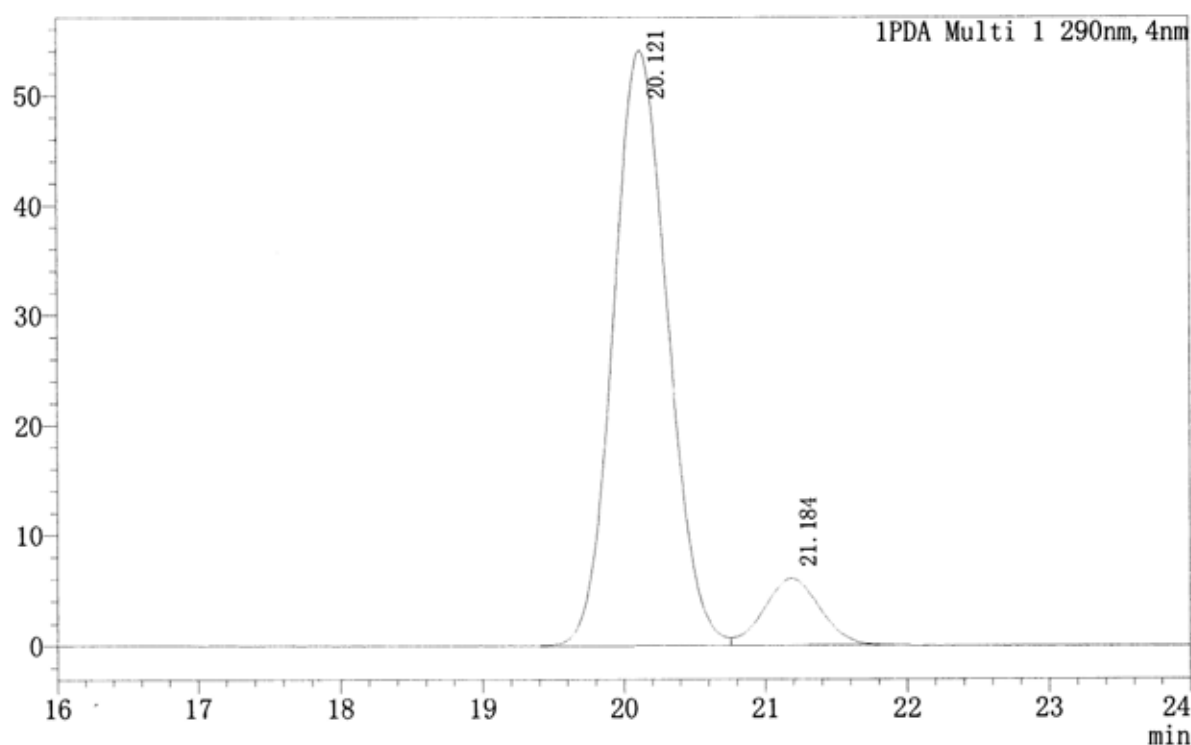


(rac)-2c

# Data Report

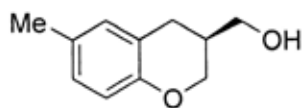
Sample Information  
Sample Name : 002-P,AD-H,3%  
Tray# : 1  
Vial# : 91  
Injection Volume : 5  
Data File : 002-P,AD-H,3%,15'.lcd  
Method File : 3%,35MIN.lcm  
Report Format File : Templates.lsr  
Sample Type : Unknown  
Acquired by : System Administrator  
Date Acquired : 2013/8/15 18:17:09  
Date Processed : 2013/8/15 18:42:13

mAU



PDA Ch1 290nm

NO.	Ret. Time	Height (uAU)	Area (uAU*min)	Rel. Area %	Resolution (USP)
1	20.121	54065	1444617	89.760	--
2	21.184	6008	164797	10.240	1.469

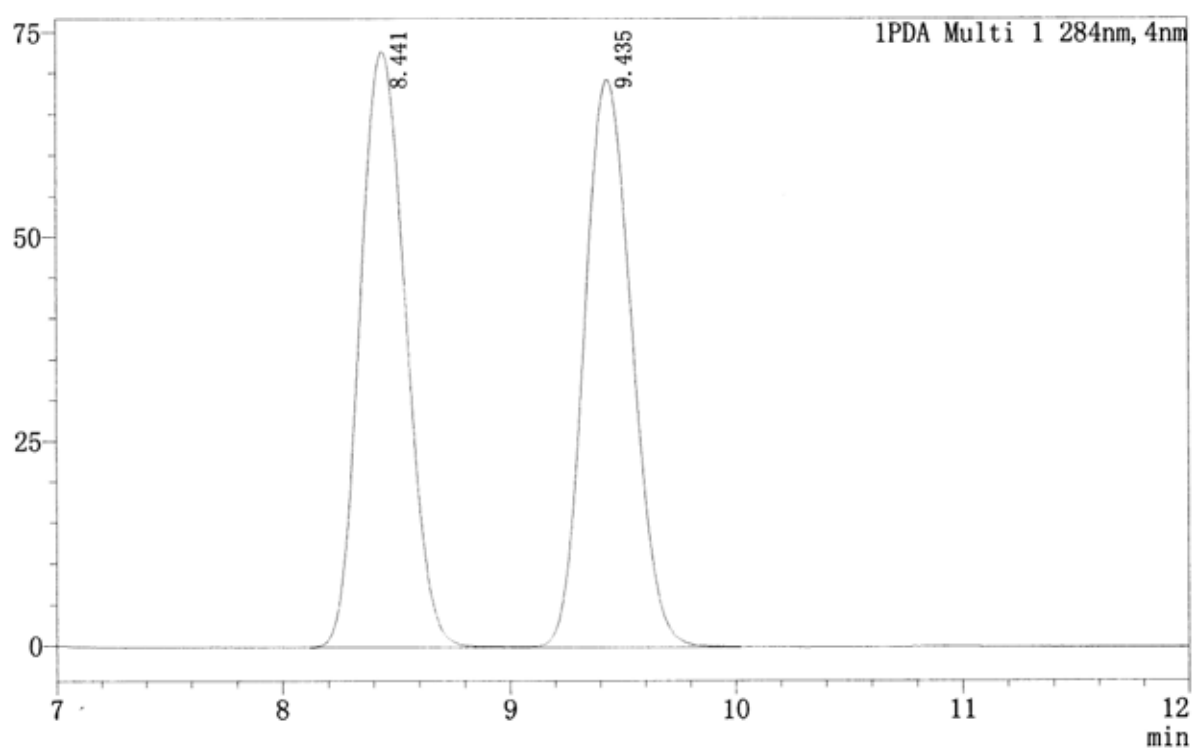


(S)-2c from 1c

# Data Report

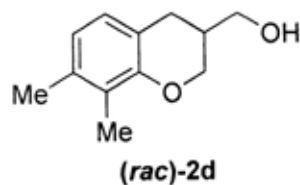
Sample Information  
Sample Name : 0010P(RS),AD-H,5%  
Tray# : 1  
Vial# : 95  
Injection Volume : 6  
Data File : 0010P(RS),AD-H5%.lcd  
Method File : 5%,30MIN.lcm  
Report Format File : Templates.lsr  
Sample Type : Unknown  
Acquired by : System Administrator  
Date Acquired : 2013/8/13 15:59:03  
Date Processed : 2013/8/13 16:13:57

mAU



PDA Ch1 284nm

NO.	Ret. Time	Height (uAU)	Area (uAU*min)	Rel. Area %	Resolution(USP)
1	8.441	72812	1013334	49.912	--
2	9.435	69314	1016888	50.088	2.572

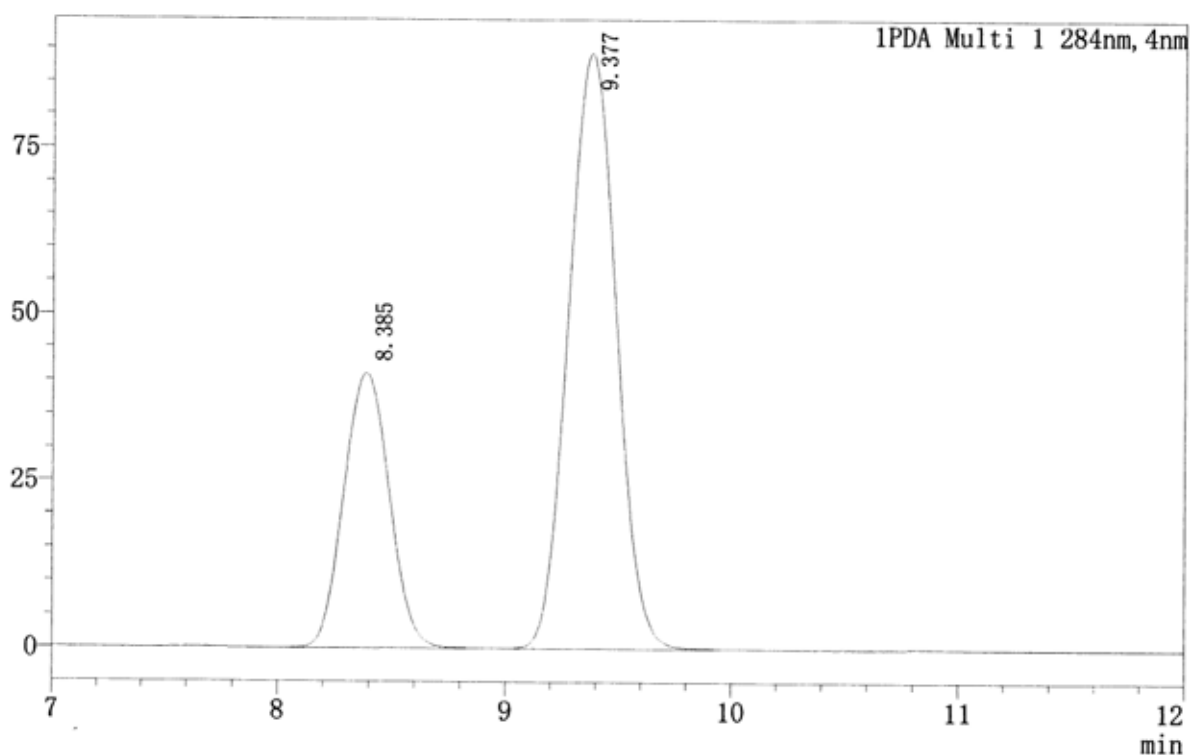


# Data Report

## Sample Information

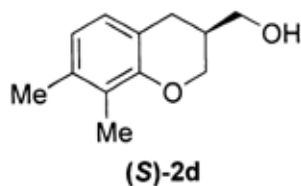
Sample Name : 0010-P,AD-H,5%  
Tray# : 1  
Vial# : 91  
Injection Volume : 6  
Data File : 0010-P,AD-H,5%.lcd  
Method File : 5%,30MIN.lcm  
Report Format File : Templates.lsr  
Sample Type : Unknown  
Acquired by : System Administrator  
Date Acquired : 2013/8/15 16:54:07  
Date Processed : 2013/8/15 17:06:46

mAU



PDA Ch1 284nm

NO.	Ret. Time	Height (uAU)	Area (uAU*min)	Rel. Area %	Resolution(USP)
1	8.385	41174	571482	30.294	--
2	9.377	89371	1314991	69.706	2.564

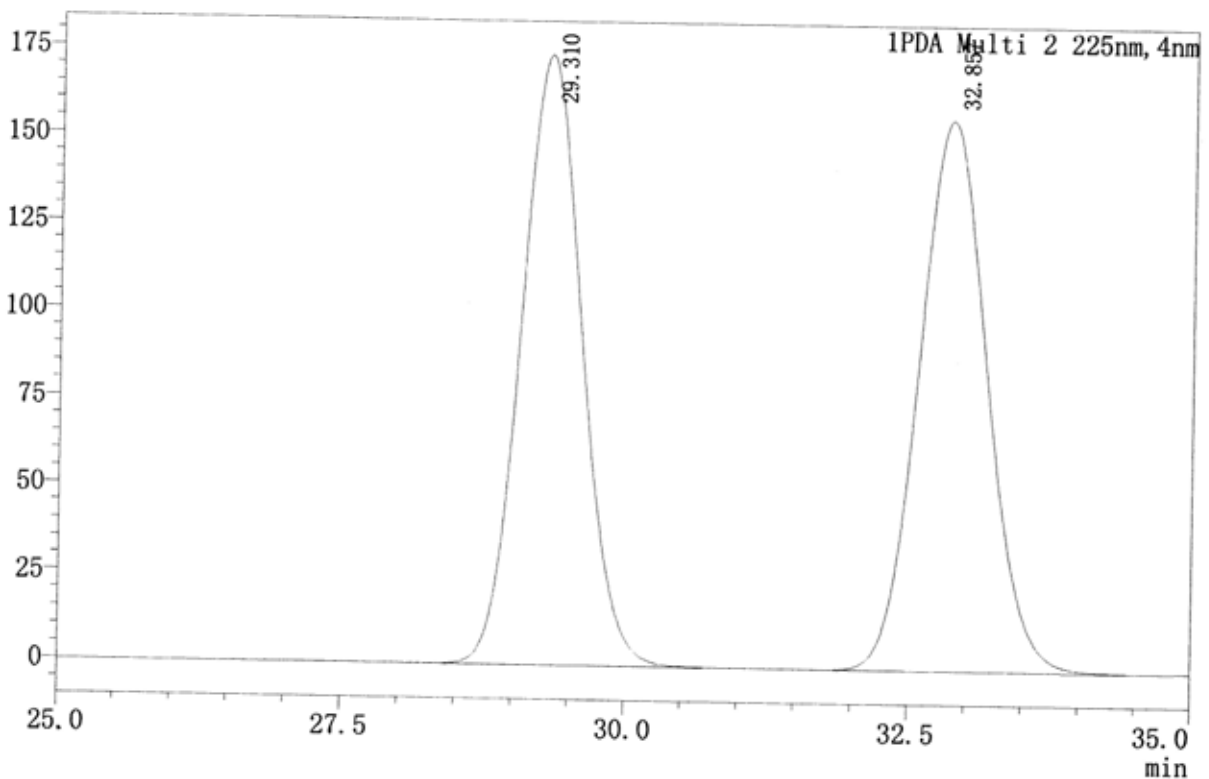


# Data Report

## Sample Information

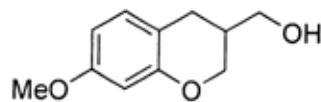
Sample Name : 004B-P(RS),AD-H,3%  
Tray# : 1  
Vial# : 82  
Injection Volume : 6  
Data File : 004B-P(RS),AD-H,3%,20.lcd  
Method File : 5%,30MIN.lcm  
Report Format File : Templates.lsr  
Sample Type : Unknown  
Acquired by : System Administrator  
Date Acquired : 2013/9/22 17:17:26  
Date Processed : 2013/9/22 17:53:20

mAU



PDA Ch2 225nm

NO.	Ret. Time	Height (uAU)	Area (uAU*min)	Rel. Area %	Resolution (USP)
1	29.310	173876	6497206	49.922	--
2	32.857	156941	6517445	50.078	3.403



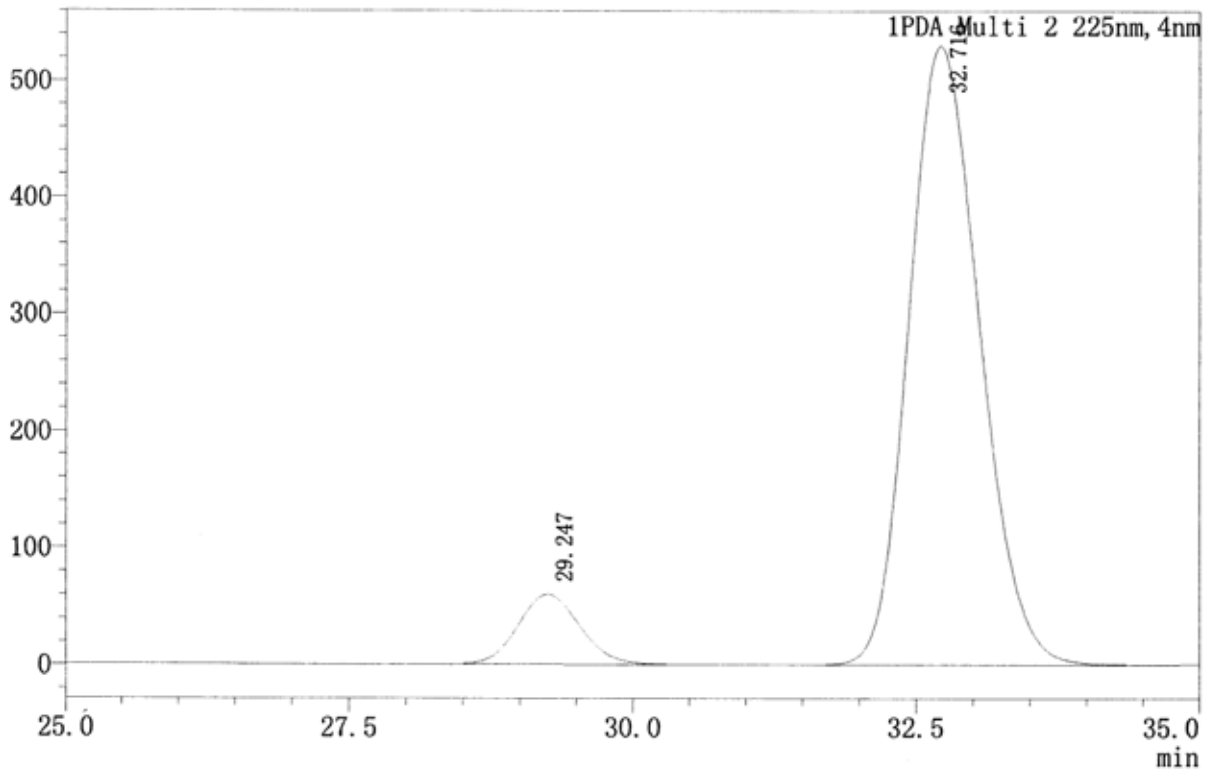
(rac)-2e

# Data Report

## Sample Information

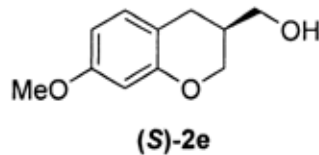
Sample Name : 004B-P,AD-H,3%  
Tray# : 1  
Vial# : 5  
Injection Volume : 7  
Data File : 004B-P,AD-H,3%,20.lcd  
Method File : 5%,30MIN.lcm  
Report Format File : Templates.lsr  
Sample Type : Unknown  
Acquired by : System Administrator  
Date Acquired : 2013/9/22 17:57:02  
Date Processed : 2013/9/22 18:34:37

mAU



PDA Ch2 225nm

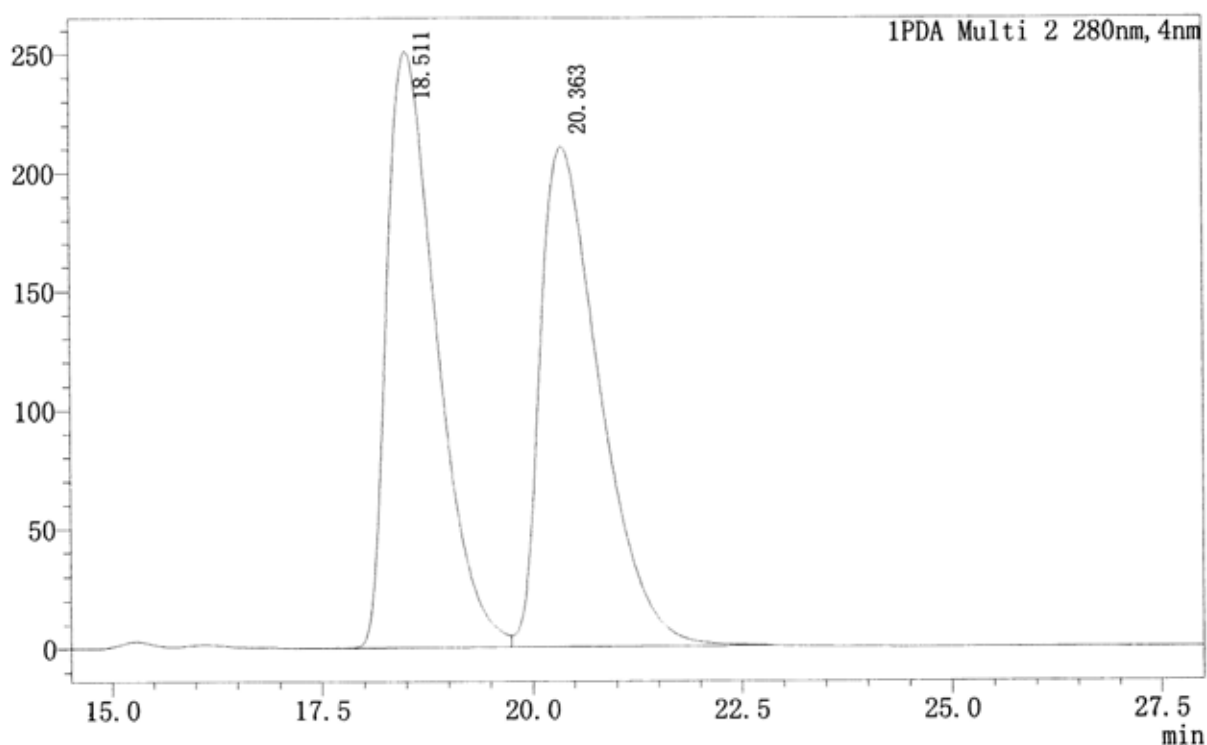
NO.	Ret. Time	Height (uAU)	Area (uAU*min)	Rel. Area %	Resolution(USP)
1	29.247	59879	2261499	8.992	—
2	32.716	530421	22887759	91.008	3.227



# Data Report

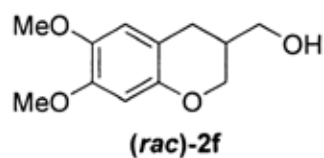
Sample Information  
Sample Name : 004-P(R/S),10%,OD  
Tray# : 1  
Vial# : 2  
Injection Volume : 5  
Data File : 004-P(RS),10%,OD-H.lcd  
Method File : 10%,60MIN.lcm  
Report Format File : Templates.lsr  
Sample Type : Unknown  
Acquired by : System Administrator  
Date Acquired : 2013/9/28 21:30:42  
Date Processed : 2013/9/28 22:30:44

mAU



PDA Ch2 280nm

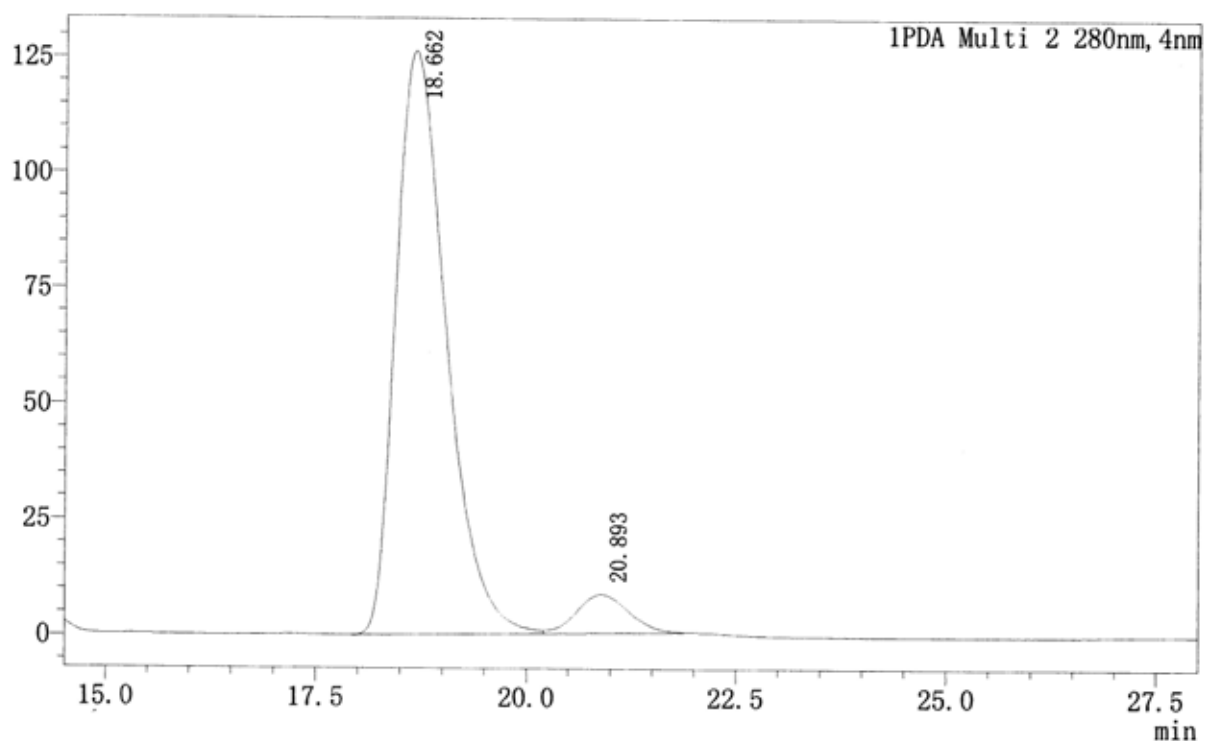
NO.	Ret. Time	Height (uAU)	Area (uAU*min)	Rel. Area %	Resolution(USP)
1	18.511	250909	10341512	49.725	--
2	20.363	210627	10455923	50.275	1.582



# Data Report

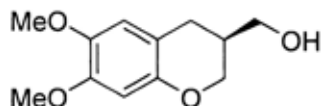
Sample Information  
Sample Name : 1916-02,10%,OD  
Tray# : 1  
Vial# : 1  
Injection Volume : 5  
Data File : 1916-02,10%,OD-H.lcd  
Method File : 10%,60MIN.lcm  
Report Format File : Templates.lsr  
Sample Type : Unknown  
Acquired by : System Administrator  
Date Acquired : 2013/9/28 22:42:03  
Date Processed : 2013/9/28 23:42:05

mAU



PDA Ch2 280nm

NO.	Ret. Time	Height (uAU)	Area (uAU*min)	Rel. Area %	Resolution (USP)
1	18.662	126177	5240438	93.598	—
2	20.893	8318	358418	6.402	2.018

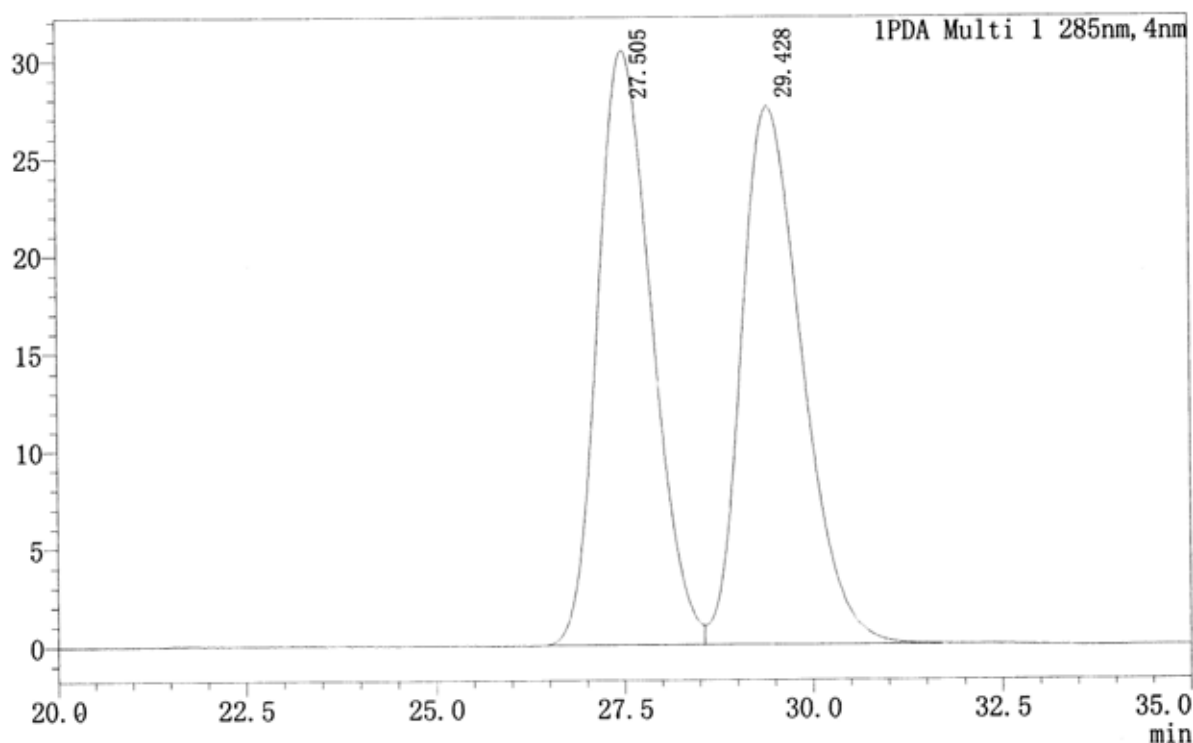


(S)-2f

# Data Report

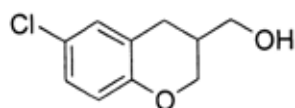
Sample Information  
Sample Name : 007P(RS),OD-H,3%  
Tray# : 1  
Vial# : 82  
Injection Volume : 6  
Data File : 007P(RS),OD-H,3%,15'.lcd  
Method File : 3%,35MIN.lcm  
Report Format File : Templates.lsr  
Sample Type : Unknown  
Acquired by : System Administrator  
Date Acquired : 2013/8/14 17:06:00  
Date Processed : 2013/8/14 17:53:20

mAU



PDA Ch1 285nm

NO.	Ret. Time	Height (uAU)	Area (uAU*min)	Rel. Area %	Resolution(USP)
1	27.505	30422	1519675	49.658	—
2	29.428	27544	1540624	50.342	1.380

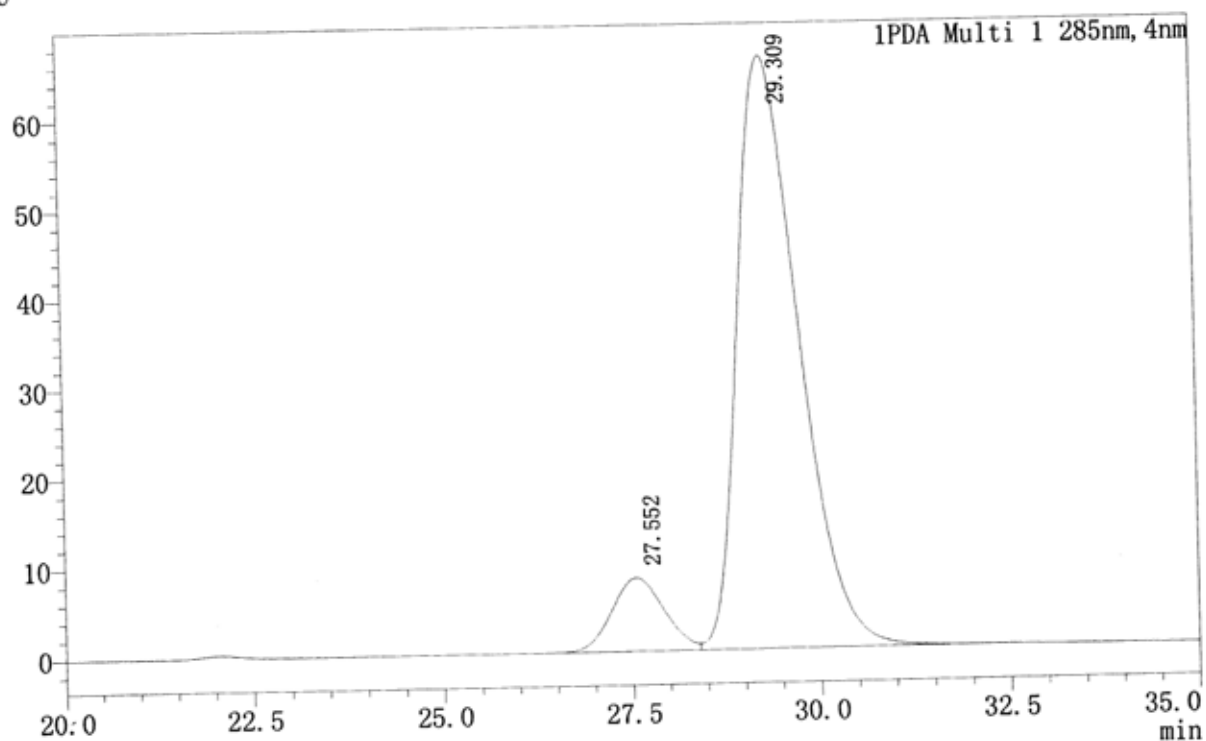


(rac)-2g

# Data Report

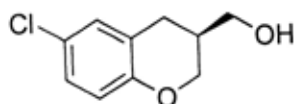
Sample Information  
Sample Name : 007-P,OD-H,3%  
Tray# : 1  
Vial# : 65  
Injection Volume : 6  
Data File : 007-P,OD-H,3%,15'.lcd  
Method File : 3%,35MIN.lcm  
Report Format File : Templates.lsr  
Sample Type : Unknown  
Acquired by : System Administrator  
Date Acquired : 2013/8/14 17:54:23  
Date Processed : 2013/8/14 18:34:31

mAU



PDA Ch1 285nm

NO.	Ret. Time	Height (uAU)	Area (uAU*min)	Rel. Area %	Resolution (USP)
1	27.552	8099	392013	9.537	--
2	29.309	66217	3718450	90.463	1.270

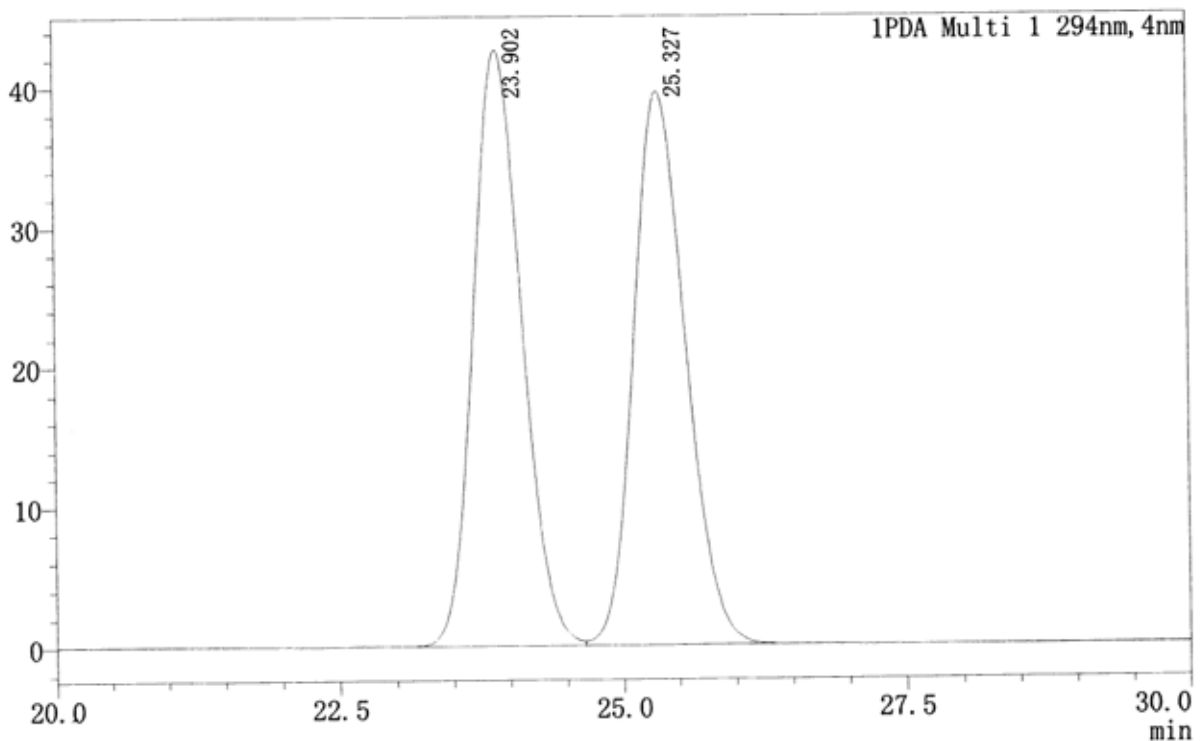


(S)-2g

# Data Report

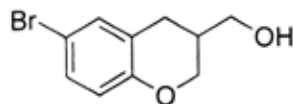
Sample Information  
Sample Name : 009P(RS),AD-H,3%  
Tray# : 1  
Vial# : 76  
Injection Volume : 6  
Data File : 009P(RS),AD-H,3%.lcd  
Method File : 3%,35MIN.lcm  
Report Format File : Templates.lsr  
Sample Type : Unknown  
Acquired by : System Administrator  
Date Acquired : 2013/8/16 13:32:57  
Date Processed : 2013/8/16 14:03:03

mAU



PDA Ch1 294nm

NO.	Ret. Time	Height (uAU)	Area (uAU*min)	Rel. Area %	Resolution (USP)
1	23.902	42621	1263898	49.988	—
2	25.327	39560	1264514	50.012	1.742

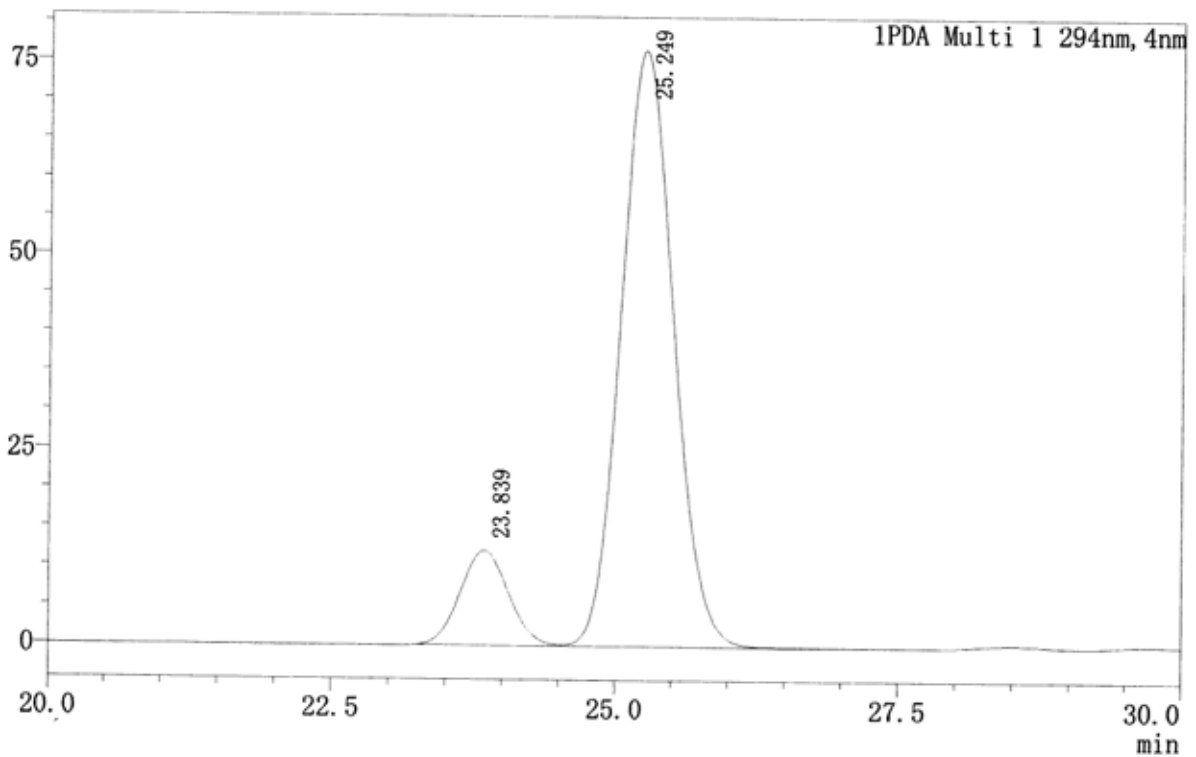


(rac)-2h

# Data Report

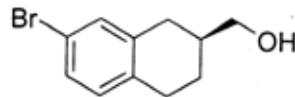
Sample Information  
Sample Name : 009-P,AD-H,3%  
Tray# : 1  
Vial# : 62  
Injection Volume : 6  
Data File : 009-P,AD-H,3%.lcd  
Method File : 3%,35MIN.lcm  
Report Format File : Templates.lsr  
Sample Type : Unknown  
Acquired by : System Administrator  
Date Acquired : 2013/8/16 12:58:48  
Date Processed : 2013/8/16 13:31:05

mAU



PDA Ch1 294nm

NO.	Ret. Time	Height (uAU)	Area (uAU*min)	Rel. Area %	Resolution (USP)
1	23.839	12051	357685	12.587	—
2	25.249	76642	2484065	87.413	1.706



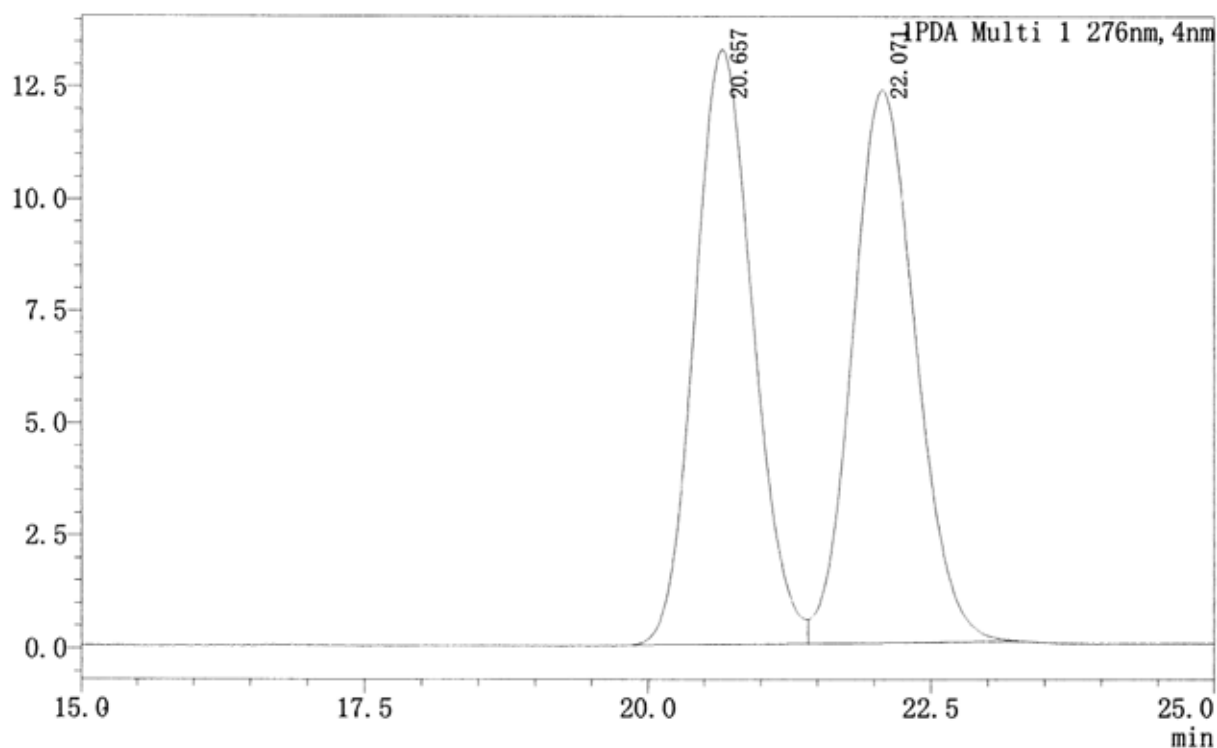
(S)-2h

# Data Report

## Sample Information

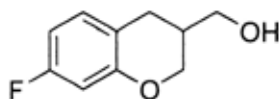
Sample Name : 006P(RS),OD-H,3%  
Tray# : 1  
Vial# : 63  
Injection Volume : 8  
Data File : 006P(RS),OD-H,3%,15'.lcd  
Method File : 3%,35MIN.lcm  
Report Format File : Templates.lsr  
Sample Type : Unknown  
Acquired by : System Administrator  
Date Acquired : 2013/8/14 16:10:14  
Date Processed : 2013/8/14 16:36:37

mAU



PDA Ch1 276nm

NO.	Ret. Time	Height (uAU)	Area (uAU*min)	Rel. Area %	Resolution (USP)
1	20.657	13235	474825	49.877	--
2	22.071	12310	477166	50.123	1.429

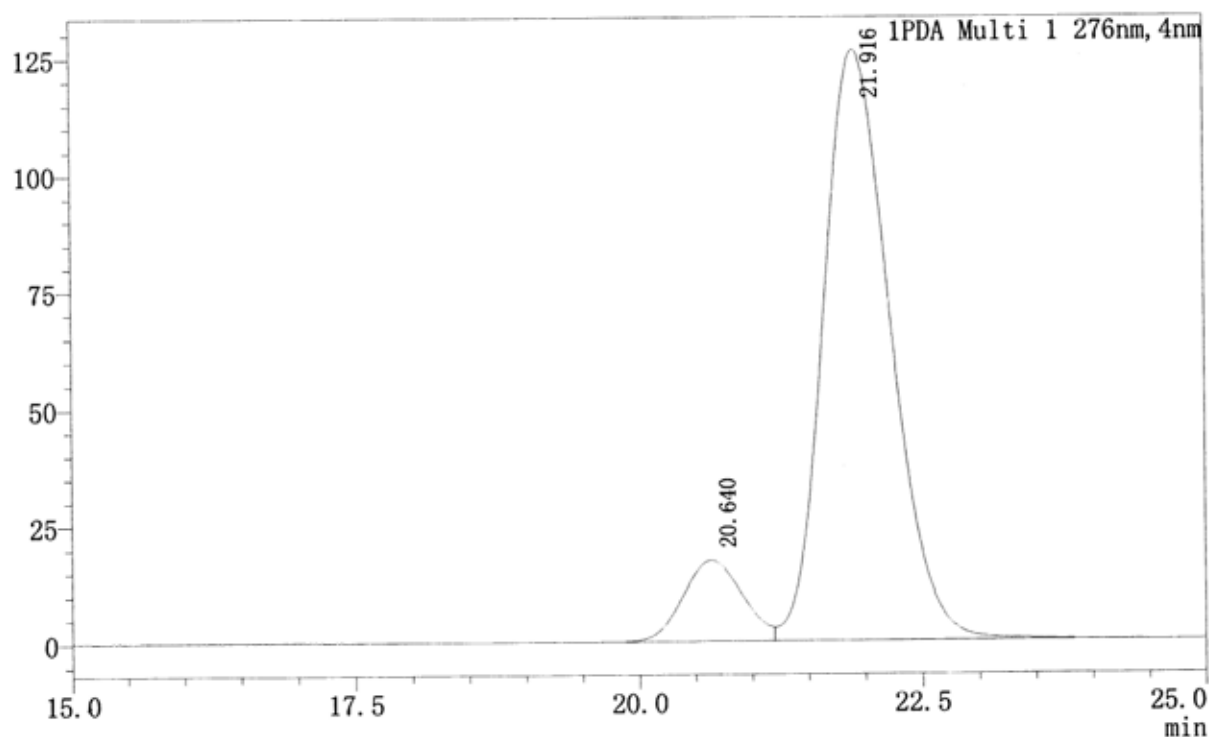


(rac)-2i

# Data Report

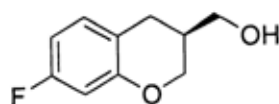
Sample Information  
Sample Name : 006-P,OD-H,3%  
Tray# : 1  
Vial# : 64  
Injection Volume : 6  
Data File : 006-P,OD-H,3%,15'.lcd  
Method File : 3%,35MIN.lcm  
Report Format File : Templates.lsr  
Sample Type : Unknown  
Acquired by : System Administrator  
Date Acquired : 2013/8/14 16:40:01  
Date Processed : 2013/8/14 17:05:07

mAU



PDA Ch1 276nm

NO.	Ret. Time	Height (uAU)	Area (uAU*min)	Rel. Area %	Resolution (USP)
1	20.640	17215	629704	10.909	—
2	21.916	126137	5142807	89.091	1.236



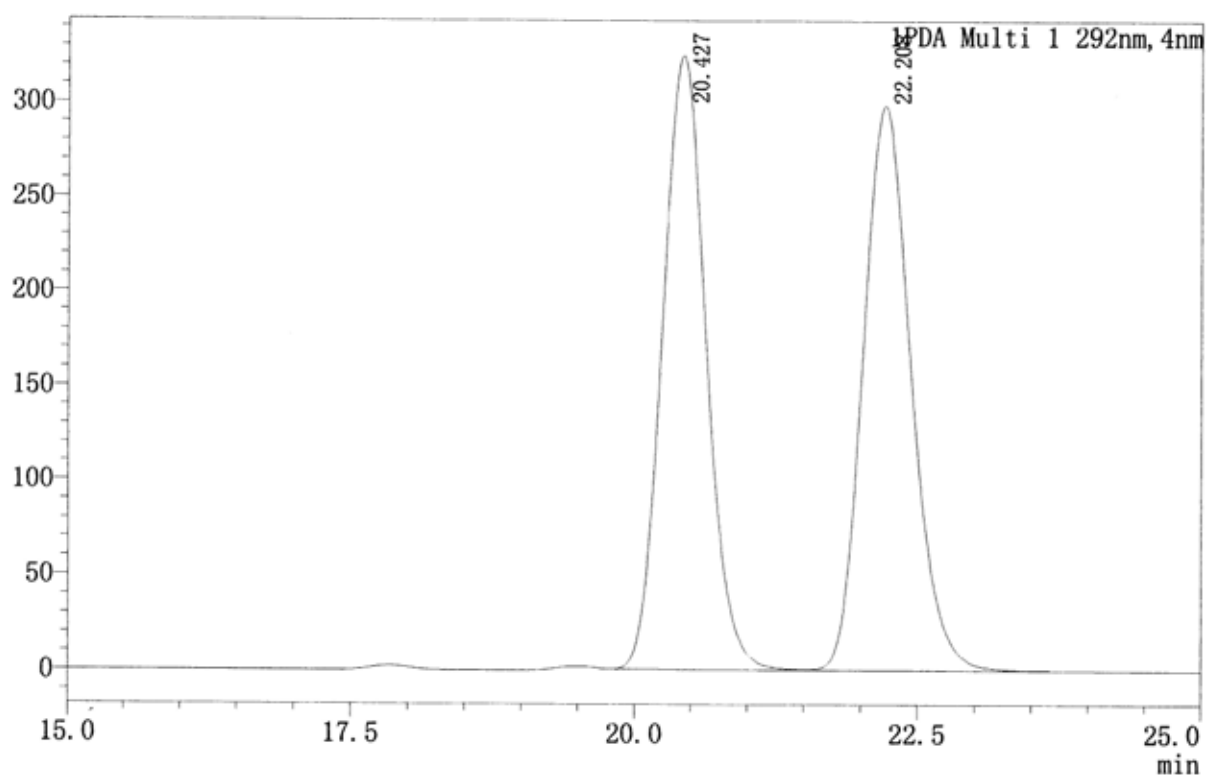
(S)-2i

# Data Report

## Sample Information

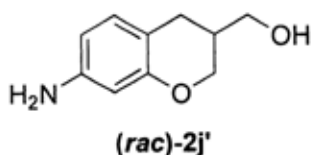
Sample Name : YWQ003B-P,AD-H,15%  
Tray# : 1  
Vial# : 46  
Injection Volume : 6  
Data File : YWQ003B-P(RS),AD-H,15%.lcd  
Method File : 15%,40MIN.lcm  
Report Format File : Templates.lsr  
Sample Type : Unknown  
Acquired by : System Administrator  
Date Acquired : 2013/8/15 23:19:59  
Date Processed : 2013/8/16 0:00:02

mAU



PDA Ch1 292nm

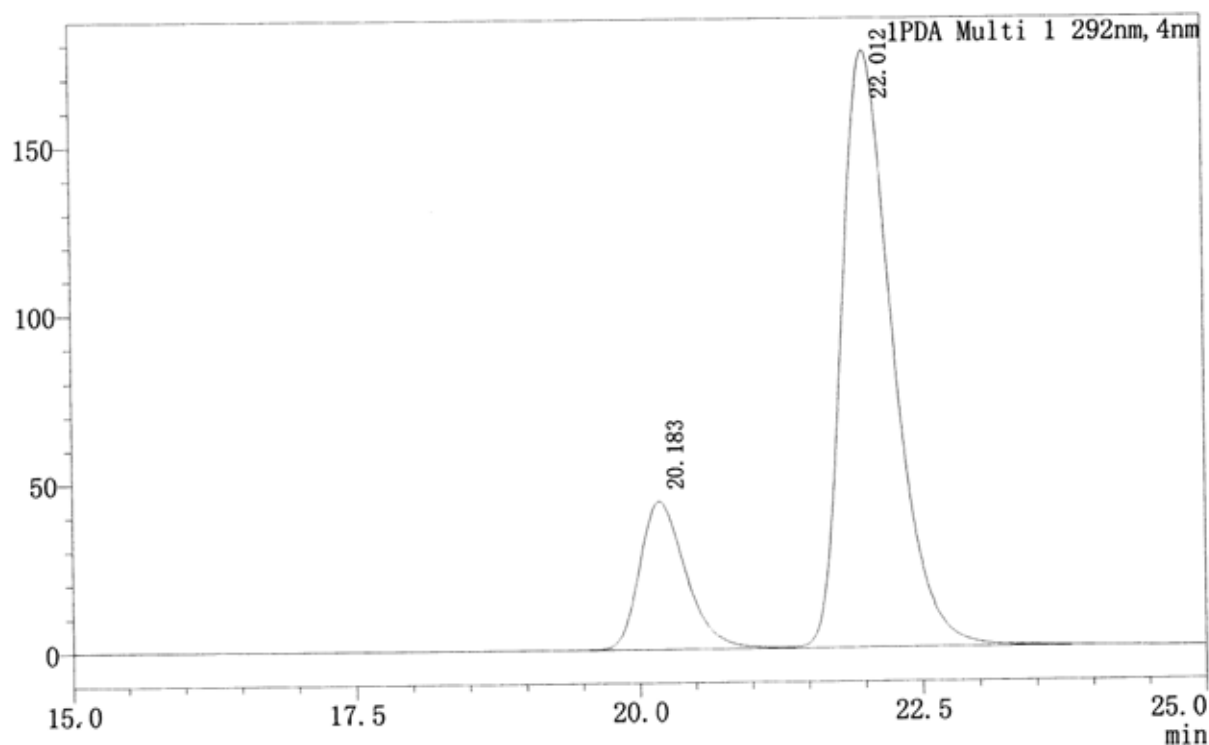
NO.	Ret. Time	Height (uAU)	Area (uAU*min)	Rel. Area %	Resolution(USP)
1	20.427	324851	8750552	49.847	--
2	22.208	298711	8804407	50.153	2.397



# Data Report

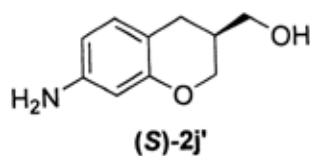
Sample Information  
 Sample Name : 003-P(NH<sub>2</sub>),AD-H,15%  
 Tray# : 1  
 Vial# : 47  
 Injection Volume : 7  
 Data File : 003-P(NH<sub>2</sub>),(AD-H,15%).lcd  
 Method File : 15%,30MIN.lcm  
 Report Format File : Templates.lsr  
 Sample Type : Unknown  
 Acquired by : System Administrator  
 Date Acquired : 2013/8/23 15:05:50  
 Date Processed : 2013/8/23 15:30:59

mAU



PDA Ch1 292nm

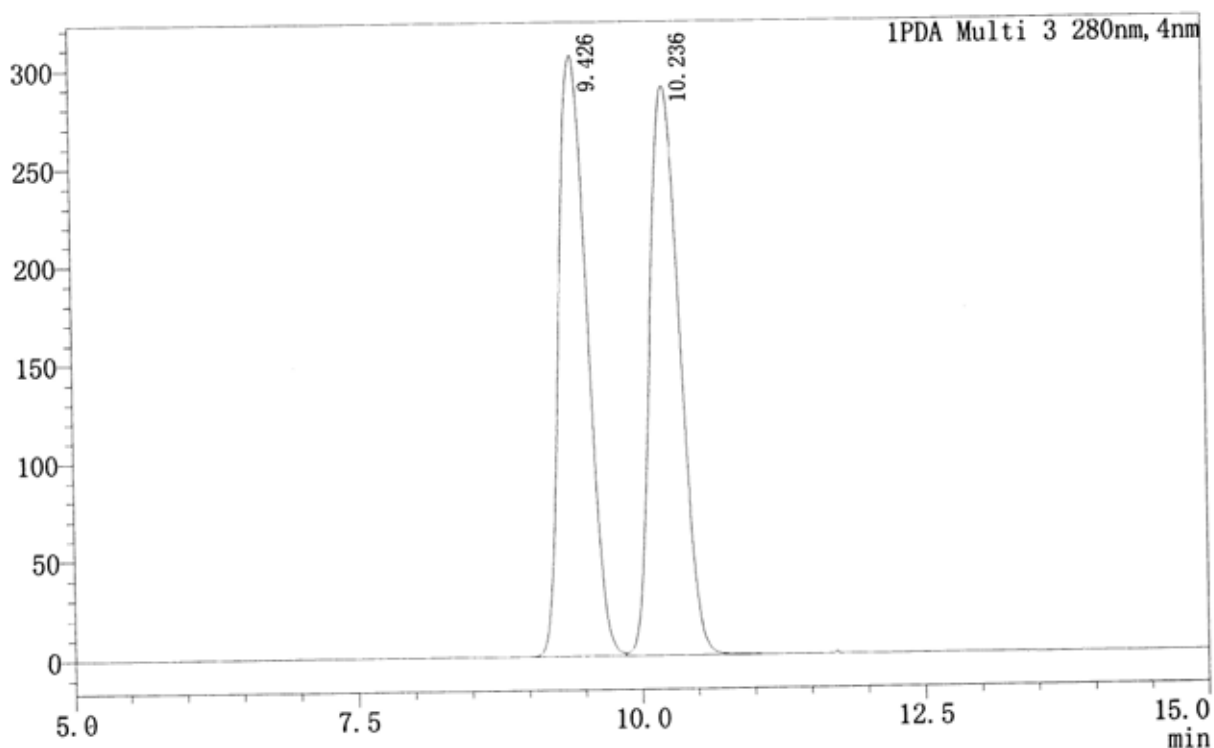
NO.	Ret. Time	Height (uAU)	Area (uAU*min)	Rel. Area %	Resolution (USP)
1	20.183	43717	1244427	18.425	--
2	22.012	176908	5509548	81.575	2.365



# Data Report

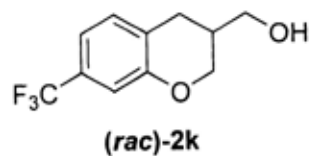
Sample Information  
Sample Name : 0026P(RS),AD-H,5%  
Tray# : 1  
Vial# : 83  
Injection Volume : 7  
Data File : 0026P(RS),AD-H,5%.lcd  
Method File : 5%,30MIN.lcm  
Report Format File : Templates.lsr  
Sample Type : Unknown  
Acquired by : System Administrator  
Date Acquired : 2013/9/24 13:20:55  
Date Processed : 2013/9/24 13:36:04

mAU



PDA Ch3 280nm

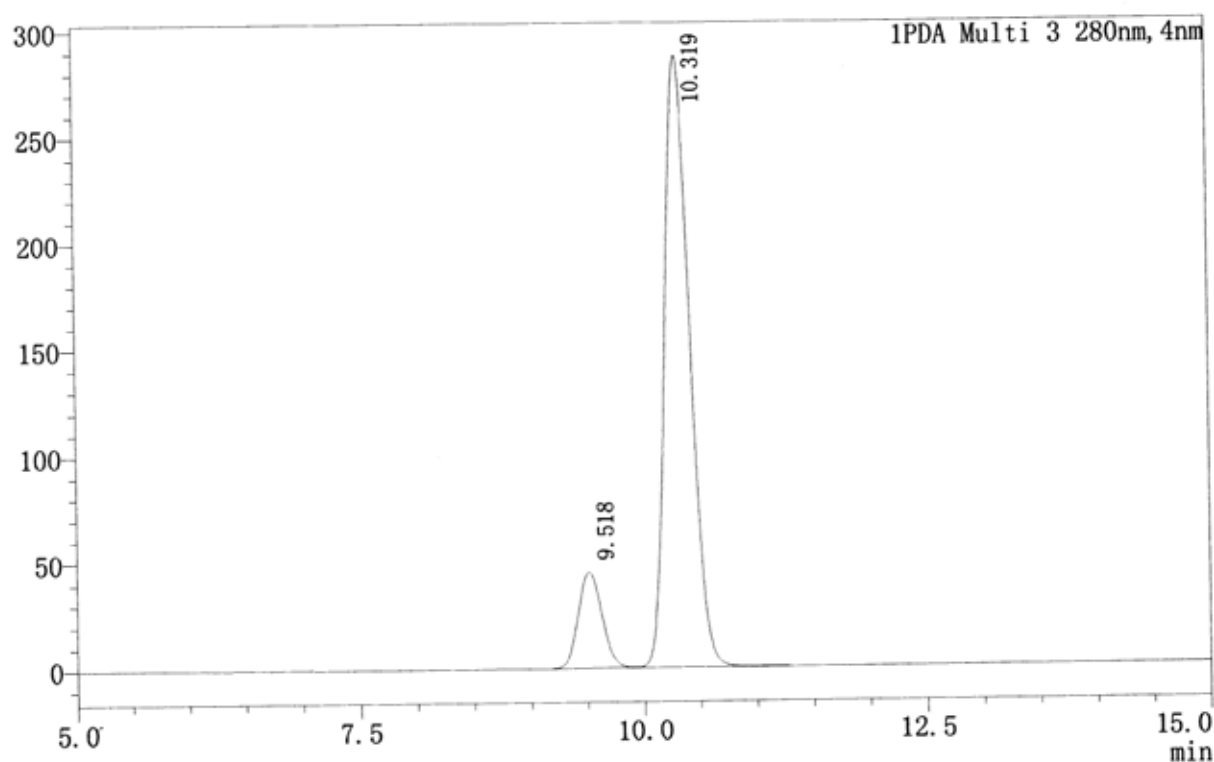
NO.	Ret. Time	Height (uAU)	Area (uAU*min)	Rel. Area %	Resolution (USP)
1	9.426	305649	5309699	49.925	—
2	10.236	289623	5325641	50.075	1.702



# Data Report

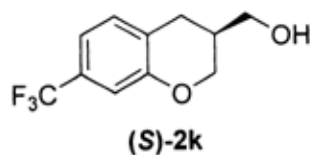
Sample Information  
Sample Name : 0026-P,AD-H,5%  
Tray# : 1  
Vial# : 3  
Injection Volume : 5  
Data File : 0026-P,AD-H,5%.lcd  
Method File : 5%,30MIN.lcm  
Report Format File : Templates.lsr  
Sample Type : Unknown  
Acquired by : System Administrator  
Date Acquired : 2013/9/24 18:23:35  
Date Processed : 2013/9/24 18:39:17

mAU



PDA Ch3 280nm

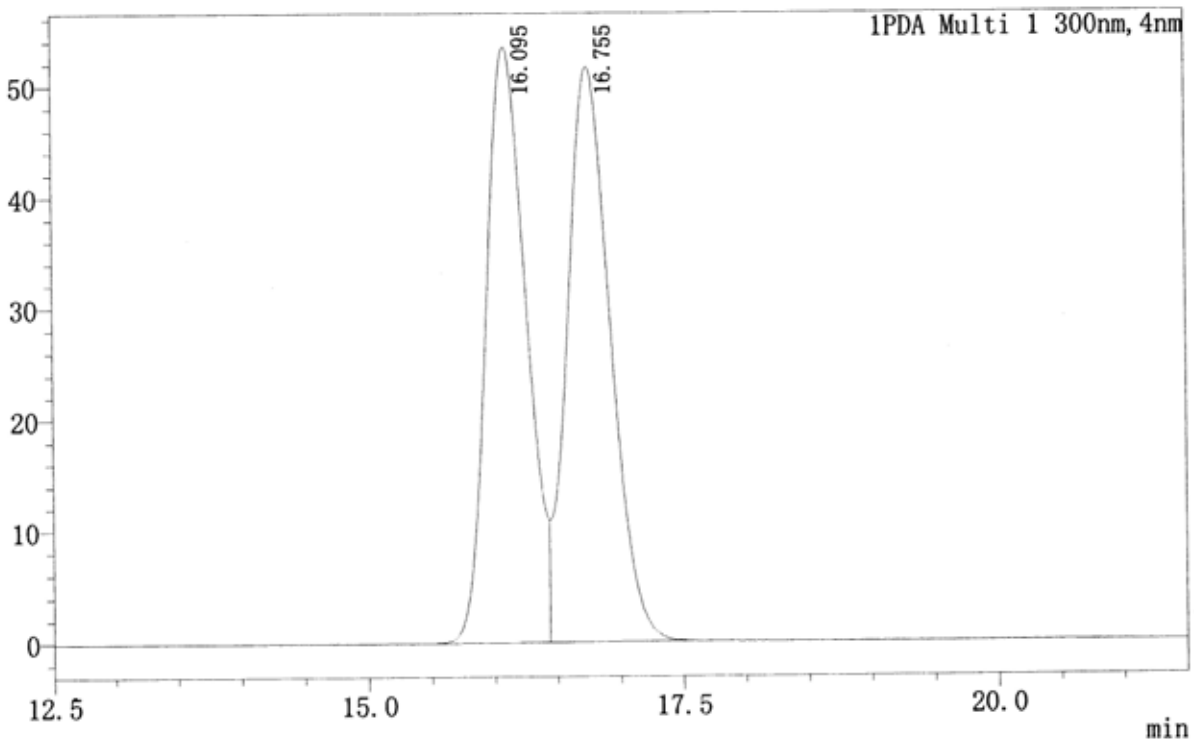
NO.	Ret. Time	Height (uAU)	Area (uAU*min)	Rel. Area %	Resolution(USP)
1	9.518	44814	650713	12.814	--
2	10.319	287363	4427242	87.186	1.983



# Data Report

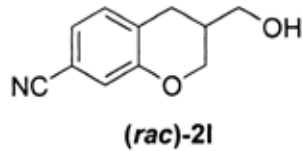
Sample Information  
Sample Name : 008P(RS),AD-H,10%  
Tray# : 1  
Vial# : 79  
Injection Volume : 6  
Data File : 008P(RS),AD-H,10%,15'.lcd  
Method File : 10%,20min.lcm  
Report Format File : Templates.lsr  
Sample Type : Unknown  
Acquired by : System Administrator  
Date Acquired : 2013/8/7 11:17:09  
Date Processed : 2013/8/7 11:38:45

mAU



PDA Ch1 300nm

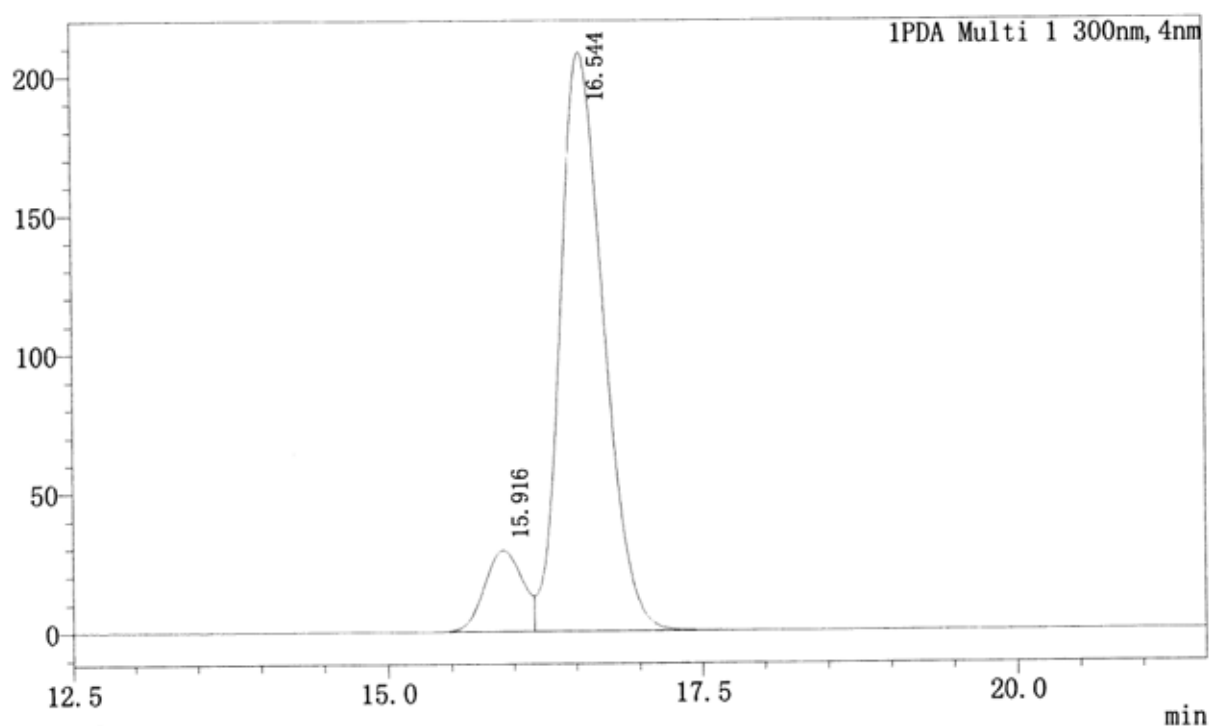
NO.	Ret. Time	Height (uAU)	Area (uAU*min)	Rel. Area %	Resolution (USP)
1	16.095	53565	1185836	49.152	--
2	16.755	51669	1226747	50.848	1.066



# Data Report

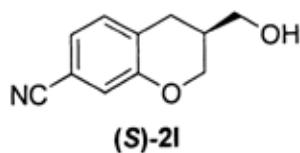
Sample Information  
Sample Name : 008-P,AD-H,10%  
Tray# : 1  
Vial# : 16  
Injection Volume : 7  
Data File : 008-P,AD-H,10%,15'.lcd  
Method File : 10%,25MIN.lcm  
Report Format File : Templates.lsr  
Sample Type : Unknown  
Acquired by : System Administrator  
Date Acquired : 2013/8/7 14:16:00  
Date Processed : 2013/8/7 14:38:09

mAU



PDA Ch1 300nm

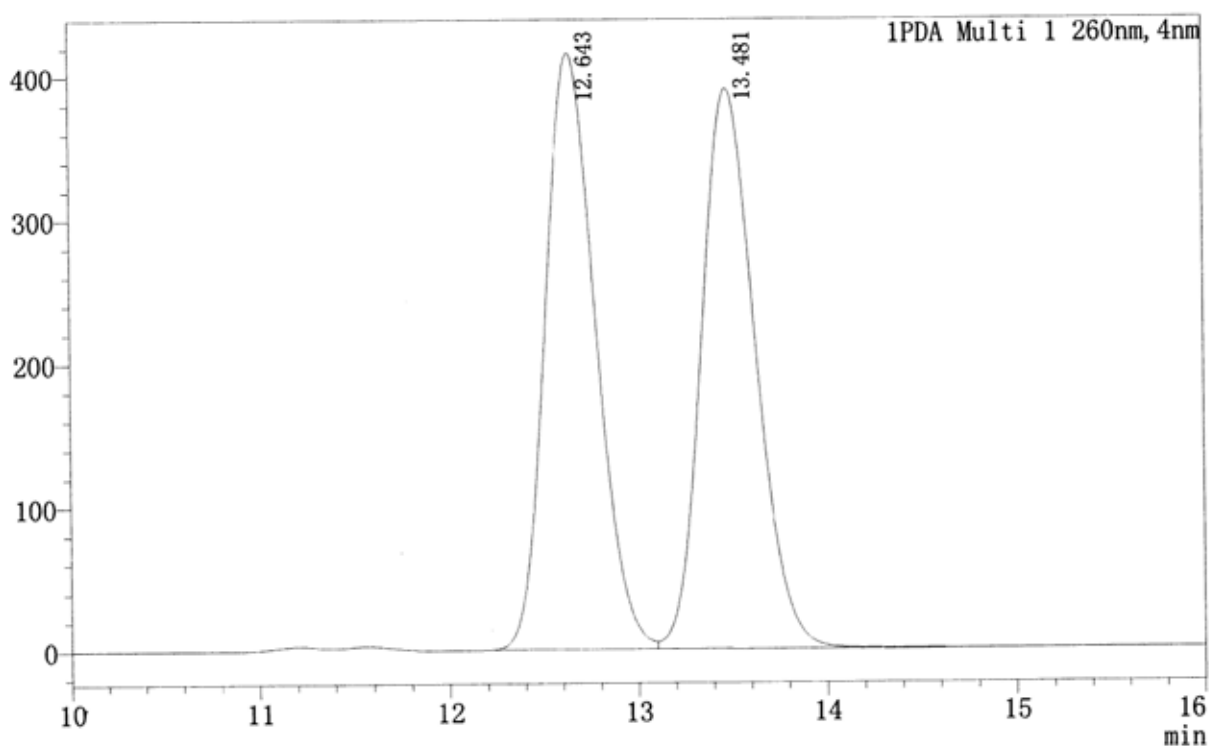
NO.	Ret. Time	Height (uAU)	Area (uAU*min)	Rel. Area %	Resolution(USP)
1	15.916	29012	613085	10.931	--
2	16.544	208166	4995578	89.069	1.002



# Data Report

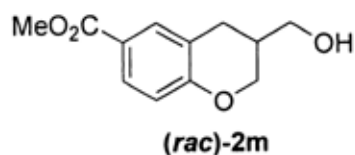
Sample Information  
Sample Name : 0013P(RS),AD-H,10%  
Tray# : 1  
Vial# : 1  
Injection Volume : 8  
Data File : 0013P(RS),AD-H,10%.lcd  
Method File : 10%,60MIN.lcm  
Report Format File : Templates.lsr  
Sample Type : Unknown  
Acquired by : System Administrator  
Date Acquired : 2013/8/29 15:57:50  
Date Processed : 2013/8/29 16:19:22

mAU



PDA Ch1 260nm

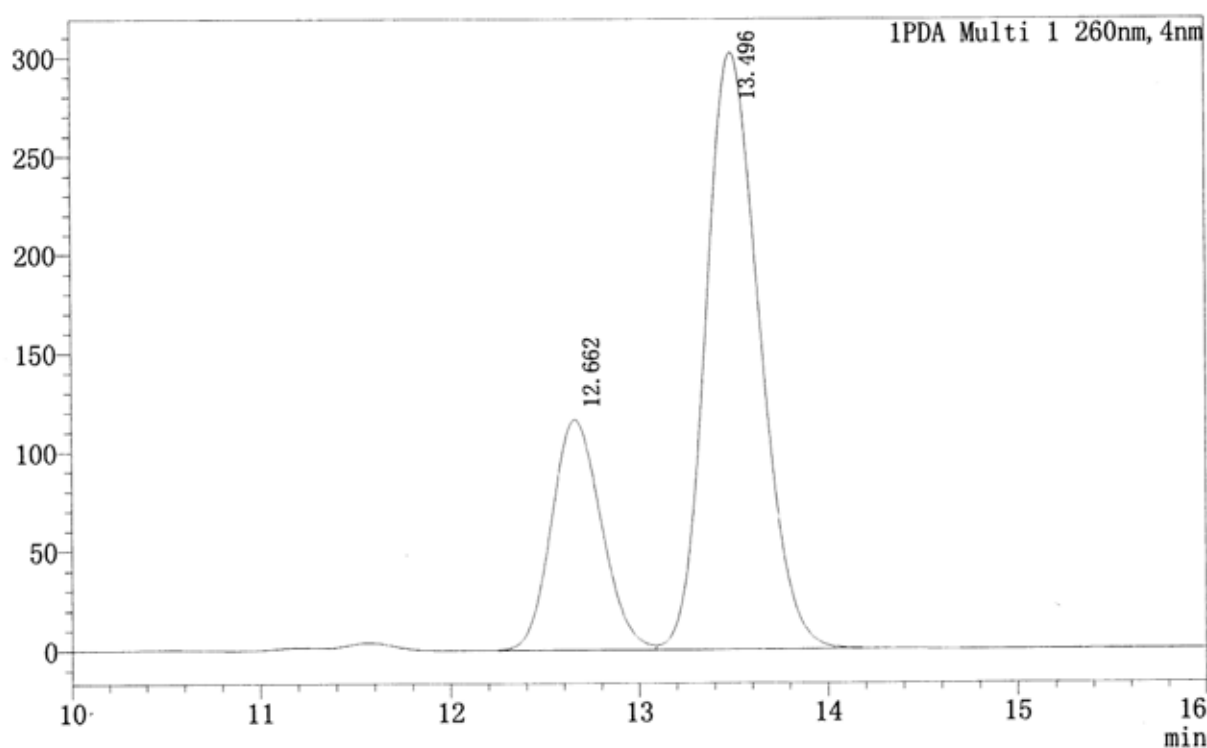
NO.	Ret. Time	Height (uAU)	Area (uAU*min)	Rel. Area %	Resolution (USP)
1	12.643	415719	7700314	49.876	--
2	13.481	390710	7738692	50.124	1.632



# Data Report

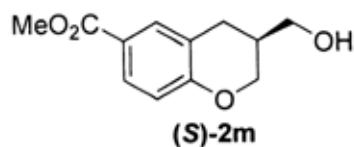
Sample Information  
Sample Name : 21057A-P,AD-H,10%  
Tray# : 1  
Vial# : 2  
Injection Volume : 8  
Data File : 0013-P,AD-H,10%.lcd  
Method File : 10%,60MIN.lcm  
Report Format File : Templates.lsr  
Sample Type : Unknown  
Acquired by : System Administrator  
Date Acquired : 2013/8/29 16:20:43  
Date Processed : 2013/8/29 16:40:51

mAU



PDA Ch1 260nm

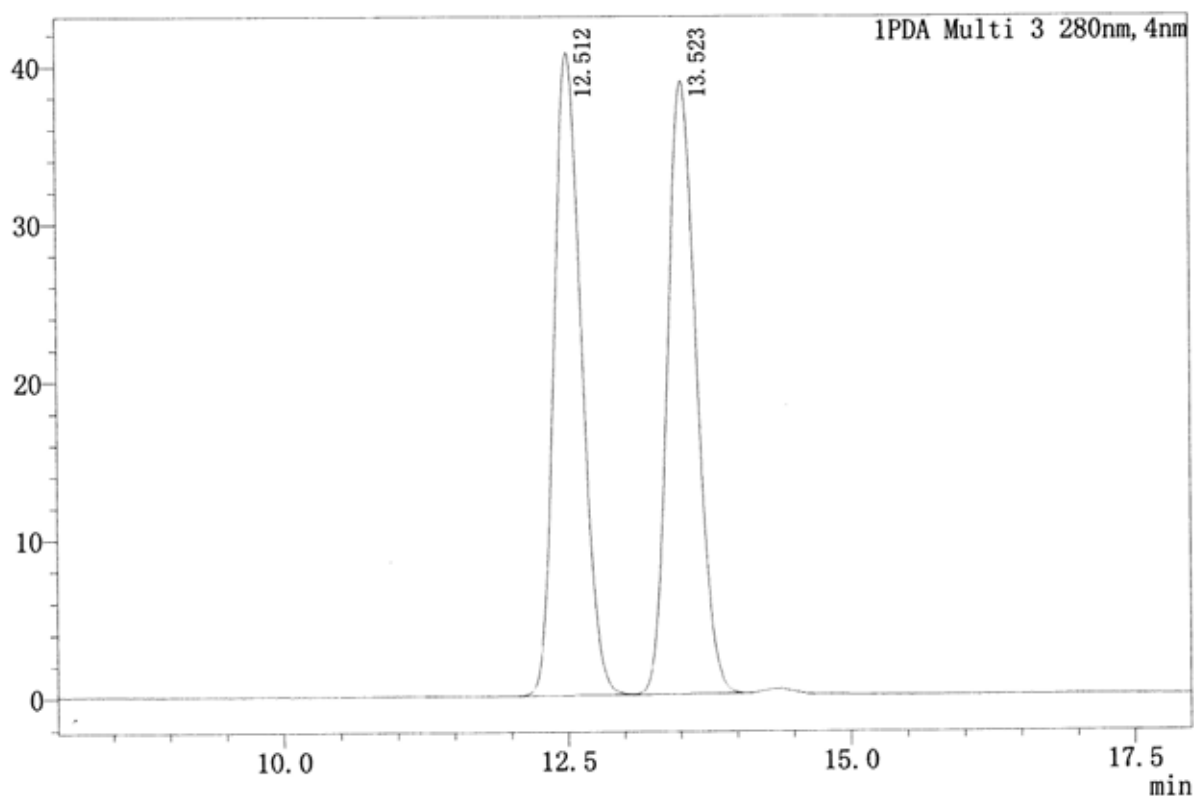
NO.	Ret. Time	Height (uAU)	Area (uAU*min)	Rel. Area %	Resolution (USP)
1	12.662	116079	2138388	26.357	--
2	13.496	302147	5974740	73.643	1.631



# Data Report

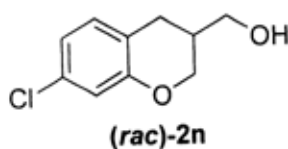
Sample Information  
Sample Name : 0041P(RS),AD-H,5%  
Tray# : 1  
Vial# : 80  
Injection Volume : 5  
Data File : 0041P(RS),AD-H,5%.lcd  
Method File : 5%,30MIN.lcm  
Report Format File : Templates.lsr  
Sample Type : Unknown  
Acquired by : System Administrator  
Date Acquired : 2013/9/16 11:20:58  
Date Processed : 2013/9/16 11:49:50

mAU



PDA Ch3 280nm

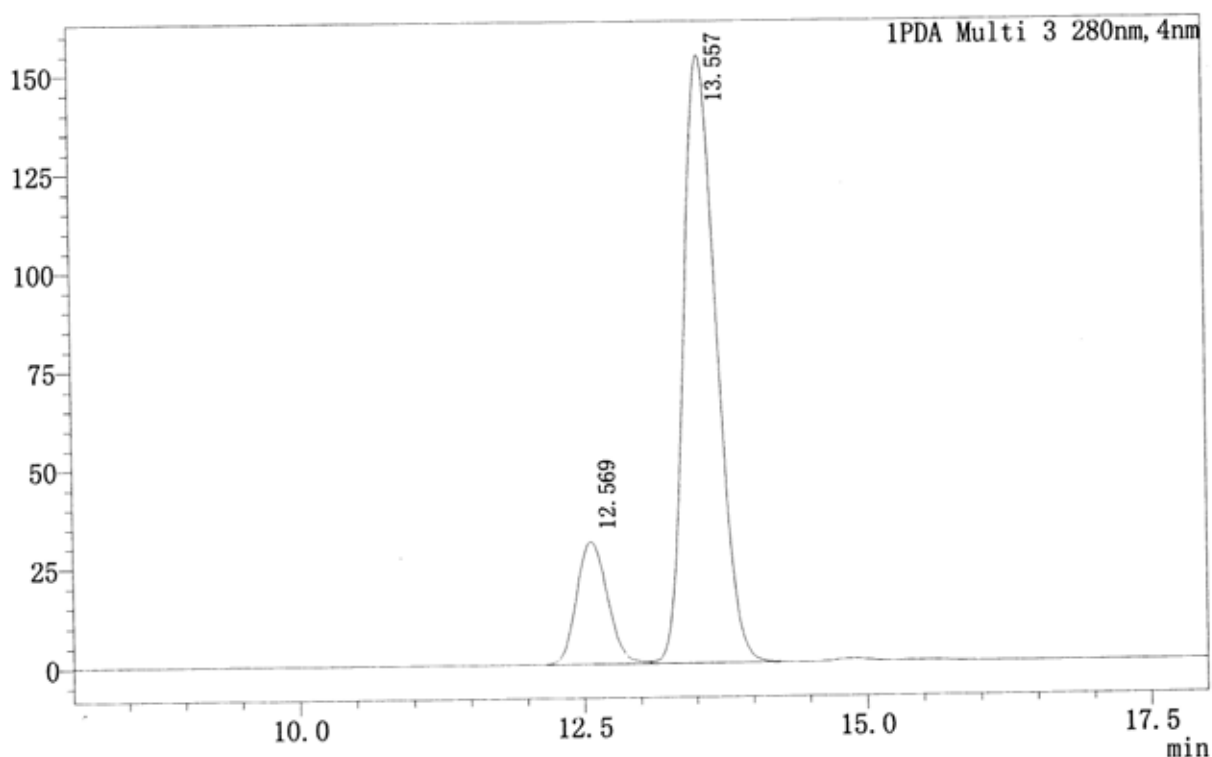
NO.	Ret. Time	Height (uAU)	Area (uAU*min)	Rel. Area %	Resolution (USP)
1	12.512	40725	683377	49.825	--
2	13.523	38855	688182	50.175	2.180



# Data Report

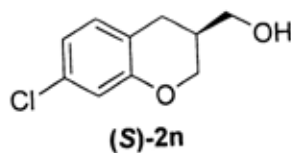
Sample Information  
Sample Name : 0041-P,AD-H,5%  
Tray# : 1  
Vial# : 1  
Injection Volume : 8  
Data File : 0041-P,AD-H,5%.lcd  
Method File : 5%,30MIN.lcm  
Report Format File : Templates.lsr  
Sample Type : Unknown  
Acquired by : System Administrator  
Date Acquired : 2013/9/16 12:09:34  
Date Processed : 2013/9/16 12:27:56

mAU



PDA Ch3 280nm

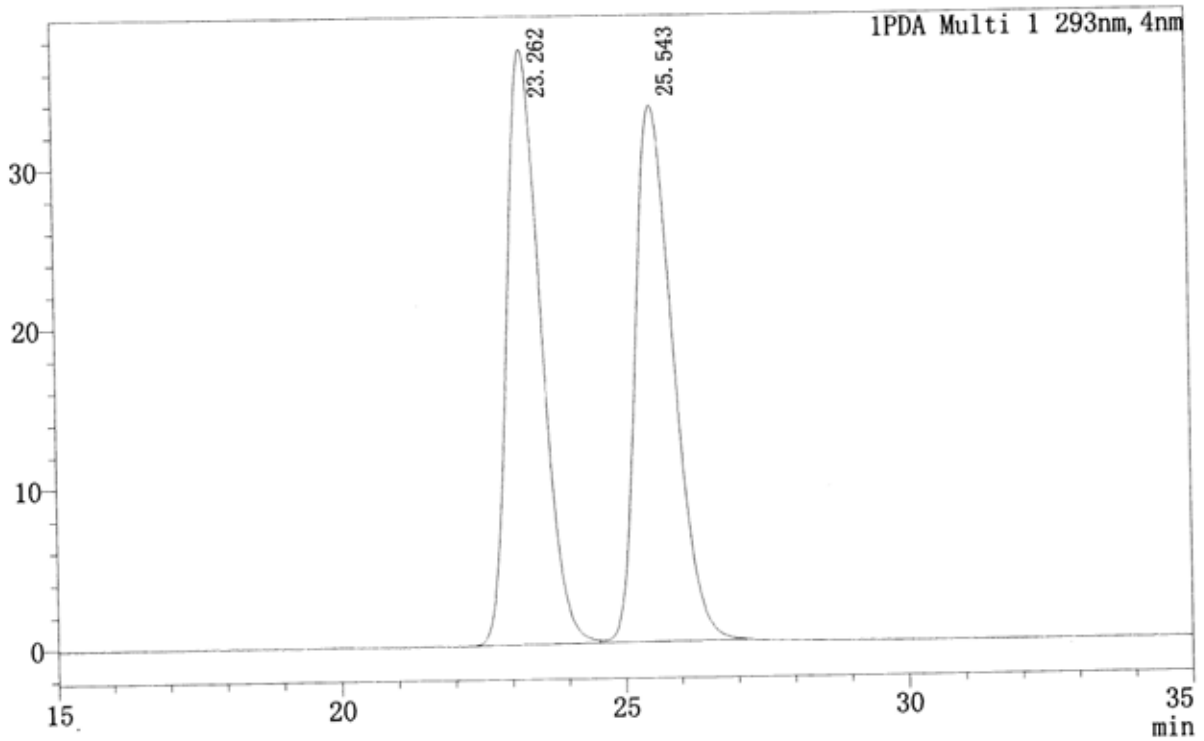
NO.	Ret. Time	Height (uAU)	Area (uAU*min)	Rel. Area %	Resolution (USP)
1	12.569	30866	588640	15.996	—
2	13.557	154223	3091271	84.004	1.895



# Data Report

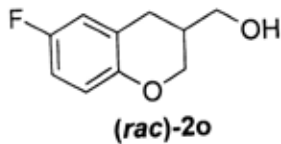
Sample Information  
Sample Name : 0033P(RS),OD-H,3%  
Tray# : 1  
Vial# : 78  
Injection Volume : 6  
Data File : 0033P(RS),OD-H,3%.lcd  
Method File : 3%,35MIN.lcm  
Report Format File : Templates.lsr  
Sample Type : Unknown  
Acquired by : System Administrator  
Date Acquired : 2013/8/26 15:32:15  
Date Processed : 2013/8/26 16:07:38

mAU



PDA Ch1 293nm

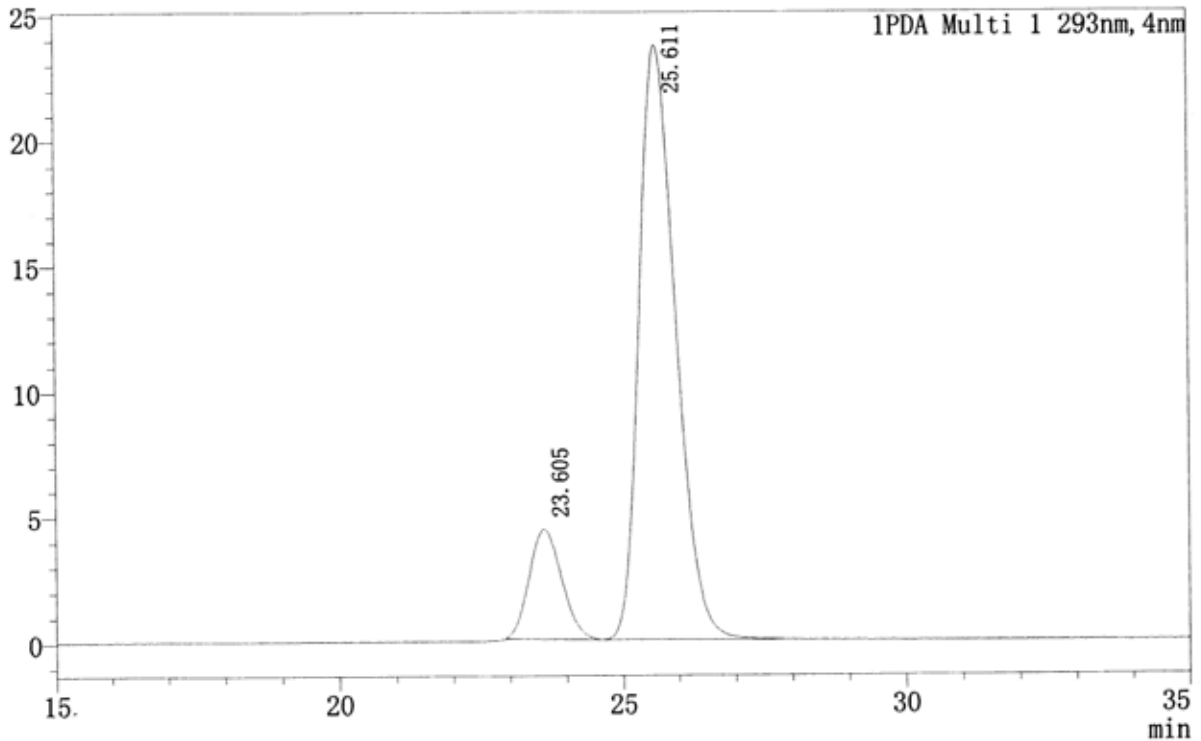
NO.	Ret. Time	Height (uAU)	Area (uAU*min)	Rel. Area %	Resolution (USP)
1	23.262	37309	1558019	49.992	—
2	25.543	33624	1558529	50.008	1.964



# Data Report

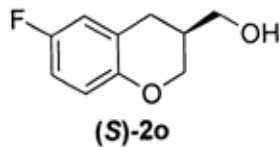
Sample Information  
Sample Name : 21057B-P,OD-H,3%  
Tray# : 1  
Vial# : 61  
Injection Volume : 6  
Data File : 21057B-P,OD-H,3%.lcd  
Method File : 3%,35MIN.lcm  
Report Format File : Templates.lsr  
Sample Type : Unknown  
Acquired by : System Administrator  
Date Acquired : 2013/8/26 17:24:24  
Date Processed : 2013/8/26 18:05:43

mAU



PDA Ch1 293nm

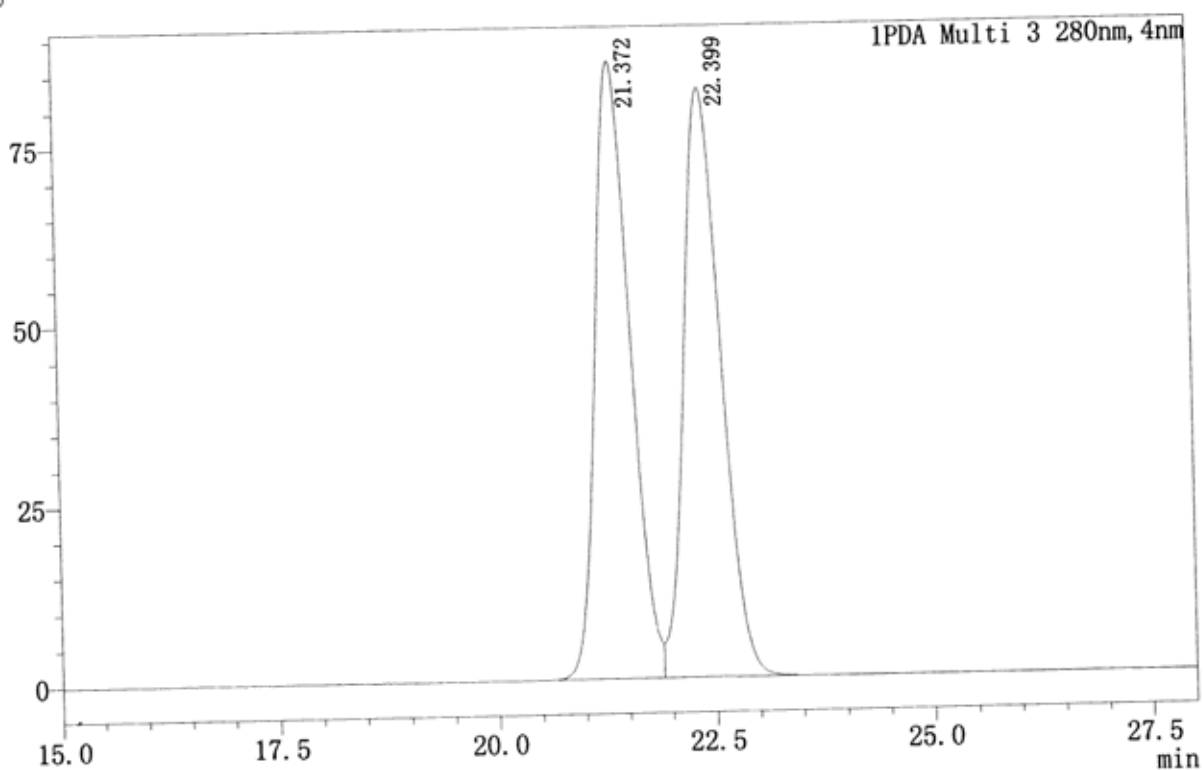
NO.	Ret. Time	Height (uAU)	Area (uAU*min)	Rel. Area %	Resolution (USP)
1	23.605	4329	173071	13.967	--
2	25.611	23654	1066105	86.033	1.777



# Data Report

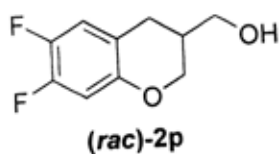
Sample Information  
Sample Name : 0039P(RS),AD-H,3%  
Tray# : 1  
Vial# : 79  
Injection Volume : 6  
Data File : 0039P'(RS),AD-H,3%.lcd  
Method File : 5%,30MIN.lcm  
Report Format File : Templates.lsr  
Sample Type : Unknown  
Acquired by : System Administrator  
Date Acquired : 2013/9/22 16:12:41  
Date Processed : 2013/9/22 16:42:45

mAU



PDA Ch3 280nm

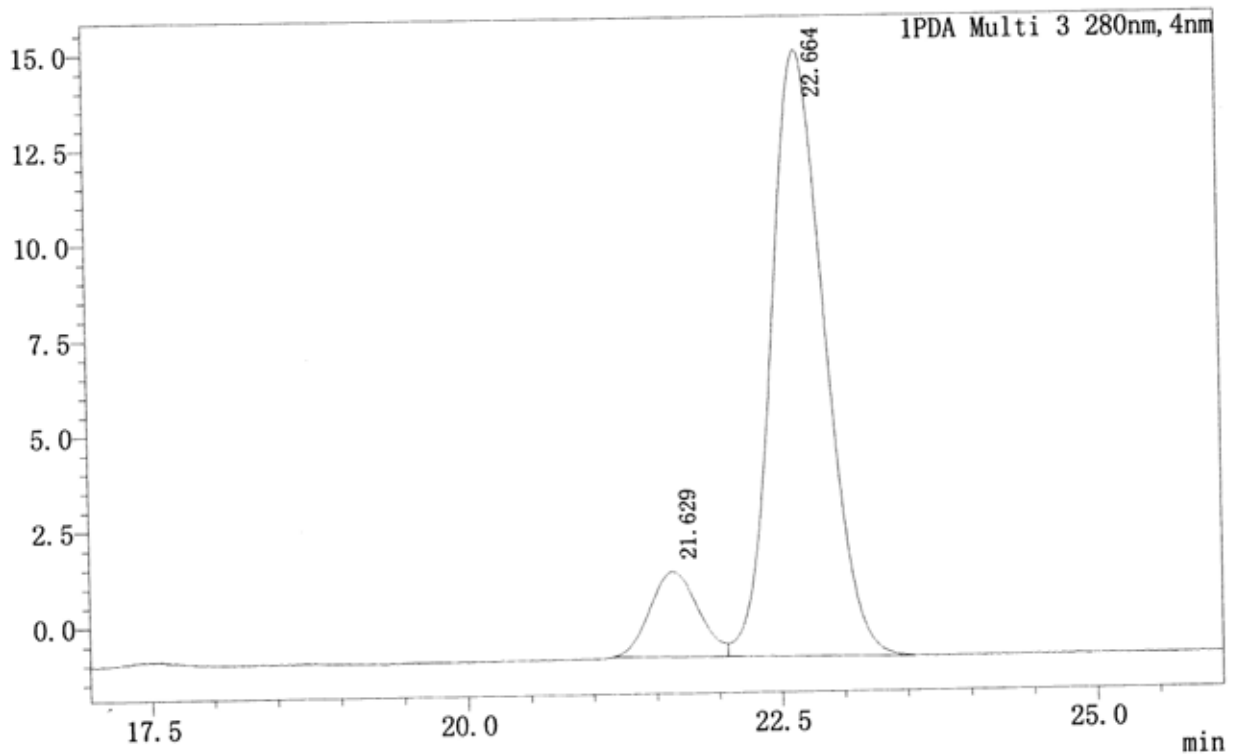
NO.	Ret. Time	Height (uAU)	Area (uAU*min)	Rel. Area %	Resolution (USP)
1	21.372	86189	2404983	49.852	—
2	22.399	82291	2419293	50.148	1.348



# Data Report

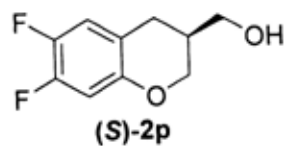
Sample Information  
Sample Name : 21082-P,AD-H,3%  
Tray# : 1  
Vial# : 4  
Injection Volume : 6  
Data File : 21082-P,AD-H,3%.lcd  
Method File : 5%,30MIN.lcm  
Report Format File : Templates.lsr  
Sample Type : Unknown  
Acquired by : System Administrator  
Date Acquired : 2013/9/22 16:43:49  
Date Processed : 2013/9/22 17:15:54

mAU



PDA Ch3 280nm

NO.	Ret. Time	Height (uAU)	Area (uAU*min)	Rel. Area %	Resolution (USP)
1	21.629	2223	59887	11.388	—
2	22.664	15907	465975	88.612	1.382

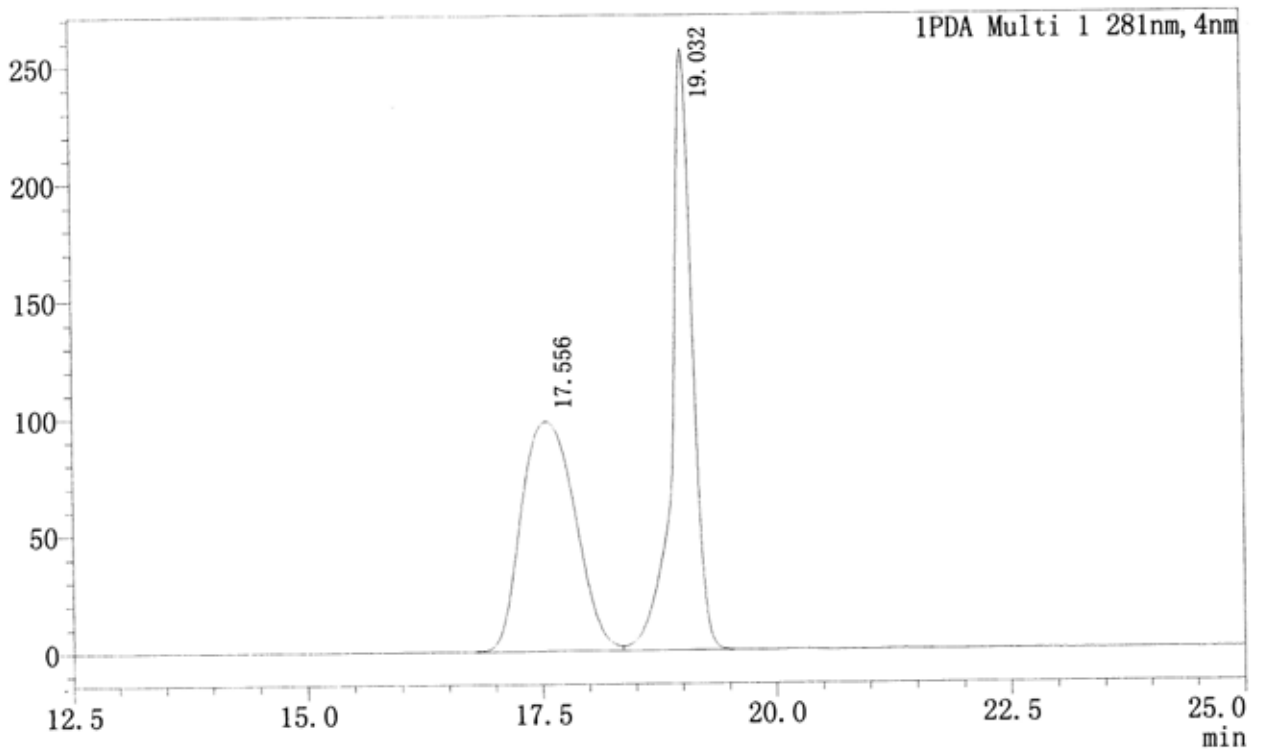


# Data Report

## Sample Information

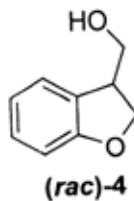
Sample Name : 21022P(RS),AD-H,3%,25'  
Tray# : 1  
Vial# : 84  
Injection Volume : 7  
Data File : 21022P(RS),AD-H,3%,25'.lcd  
Method File : 5%,60MIN.lcm  
Report Format File : Templates.lsr  
Sample Type : Unknown  
Acquired by : System Administrator  
Date Acquired : 2013/9/29 11:40:44  
Date Processed : 2013/9/29 12:08:28

mAU



PDA Ch1 281nm

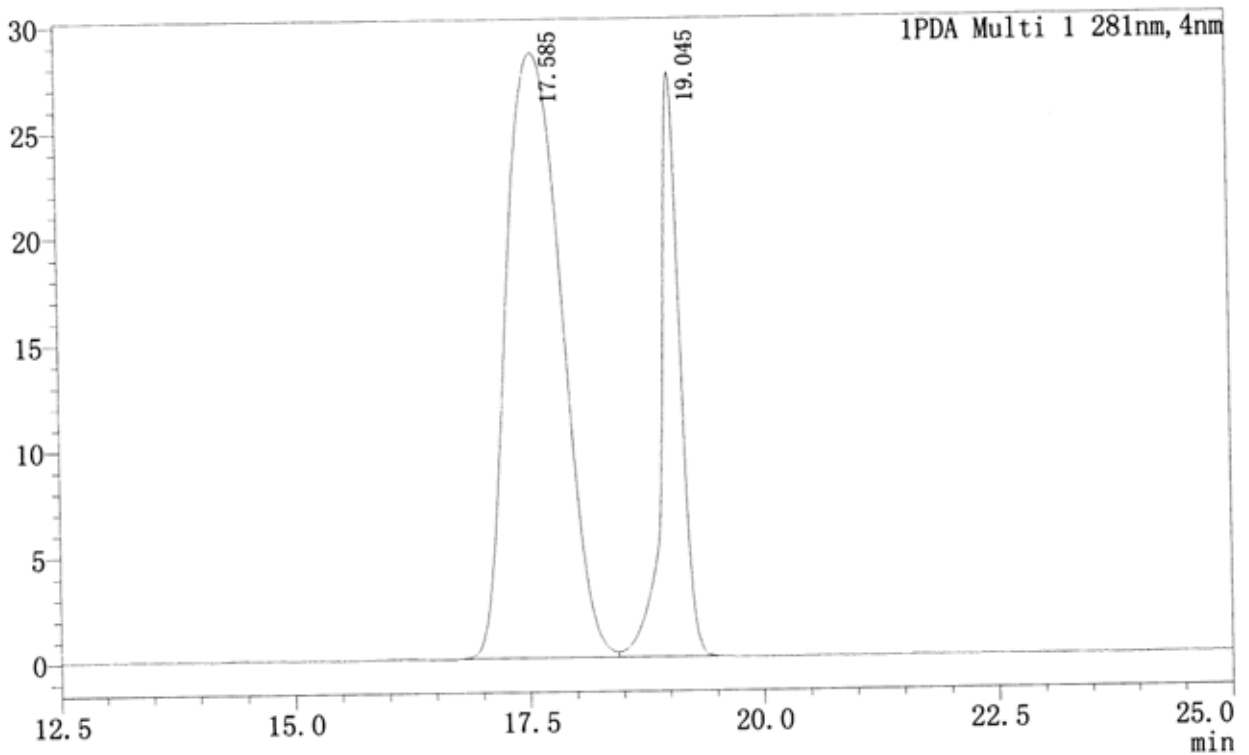
NO.	Ret. Time	Height (uAU)	Area (uAU*min)	Rel. Area %	Resolution (USP)
1	17.556	98000	3933501	50.096	--
2	19.032	256508	3918364	49.904	2.132



# Data Report

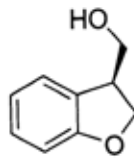
Sample Information  
Sample Name : 21022A-P,AD-H,3%,25'  
Tray# : 1  
Vial# : 4  
Injection Volume : 7  
Data File : 21022A-P,AD-H,3%,25'.lcd  
Method File : 5%,60MIN.lcm  
Report Format File : Templates.lsr  
Sample Type : Unknown  
Acquired by : System Administrator  
Date Acquired : 2013/9/29 12:11:41  
Date Processed : 2013/9/29 12:37:35

mAU



PDA Ch1 281nm

NO.	Ret. Time	Height (uAU)	Area (uAU*min)	Rel. Area %	Resolution (USP)
1	17.585	28500	1165215	74.685	--
2	19.045	27521	394963	25.315	2.098



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