

## Supporting information

### **An Entry to a Chiral Dihydropyrazole Scaffold: Enantioselective [3+2] Cycloaddition of Nitrile Imines**

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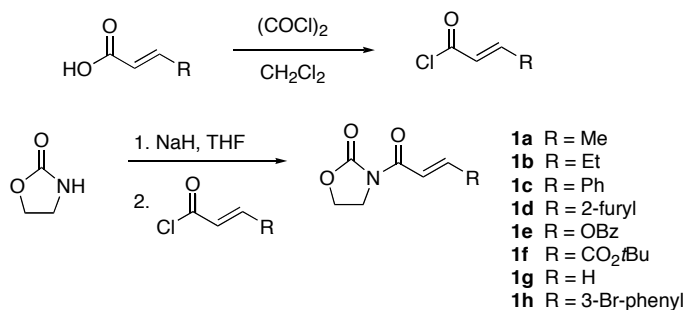
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**General:** Dichloromethane was distilled from calcium hydride under nitrogen prior to use. Tetrahydrofuran was distilled from sodium benzophenone ketyl under nitrogen. Magnesium perchlorate, magnesium iodide, copper(II) triflate, zinc iodide, and nickel perchlorate were purchased from Aldrich chemicals. Magnesium triflimide and zinc triflimide were prepared according to literature procedure. Cyclopropyl bisoxazoline ligand {3aR-[2(3'aS, 8'aR), 3aa, 8aa]}-2,2'-(cyclopropylidene)-bis{3a,8a-dihydro-8H-indeno[1,2d]-oxazole} was prepared according to literature procedure. Flash chromatography was performed using EM Science silica gel 60 (230-400 mesh) or on an ISCO<sup>TM</sup> CombiFlash Companion with AnaLogix<sup>TM</sup> RS-4 columns. All glassware was oven dried, assembled hot and cooled under a stream of nitrogen before use. Reactions with air sensitive materials were carried out by standard syringe techniques.

Melting points were measured with a Fisher-Johns melting points apparatus and are uncorrected. <sup>1</sup>H-NMR were recorded on a Varian Unity/Inova-500 NB (500 MHz), Varian Unity/Inova-400 NB (400 MHz), or Varian Mercury-300 (300 MHz). Chemical shifts are reported in parts per million (ppm) down field from TMS, using residual CDCl<sub>3</sub> (7.27 ppm) as an internal standard. Data are reported as follows: Chemical shift, multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet), coupling constant and integration. <sup>13</sup>C-NMR was recorded on Varian Unity/Inova-500 NB (125 MHz), Varian Unity/Inova-400 NB (100 MHz), and Varian Mercury-300 (75MHz) spectrometers, using broadband proton decoupling. Chemical shifts are reported in parts per million (ppm) downfield from TMS, using the middle resonance of CDCl<sub>3</sub> (77.23) as an internal standard. HPLC analyses were carried out on Waters 515 HPLC pump and a 2487 dual λ absorbance detector connected to a PC with Empower workstation.

Rotations were recorded on a JASCO-DIP-370 instrument. FT-IR spectra were recorded on a Mettler-Toledo ReactIR-4000. High Resolution Mass Spectra (HRMS) (ESI+) were obtained from the Mass Spectrometry Laboratory, North Dakota State University, Fargo, North Dakota.

**General procedure for the synthesis of 3-(-2-Alkenoyl)-2-oxazolidinones (1a-1h):**

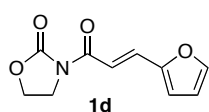


To a solution of  $\alpha,\beta$ -unsaturated acid (11.0 mmol) in 15 mL of dichloromethane at 0 °C, under nitrogen, was added oxalyl chloride (12.1 mmol) followed by one drop of DMF. After 10 minutes the solution was allowed to warm to room temperature and stir for two hours. The solvent and excess oxalyl chloride were removed *in vacuo* to give crude acid chloride, which was used without further purification.

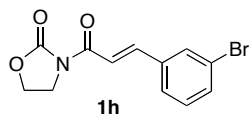
In a separate flask 2-oxazolidinone (11.0 mmol) was dissolved in THF (45 mL) and cooled to 0 °C under nitrogen. To this solution was added sodium hydride (15.0 mmol, 60% dispersion in mineral oil) in portions. This suspension was stirred at 0 °C for 10 min, then allowed to warm to room temperature and stir for an additional 45 minutes. Crude acid chloride was then added dropwise and the mixture allowed to stir for 4 hours at room temperature. The reaction was quenched with water (2 mL) and the THF was removed *in vacuo*. The reaction mixture was extracted with EtOAc (3 x 20 mL). The combined organic extracts were then washed with water (2 x 10 mL), and 1 M NaOH (2

x 10 mL), dried over MgSO<sub>4</sub> and concentrated. The crude product was purified by flash column silica gel chromatography using hexane/EtOAc as eluent.

Analytical characteristics for compounds **1a**<sup>1</sup>, **1b**<sup>2</sup>, **1c**<sup>3</sup>, **1e**<sup>4</sup>, **1f**<sup>5</sup>, and **1g**<sup>6</sup> were in agreement with previously reported spectra and properties.

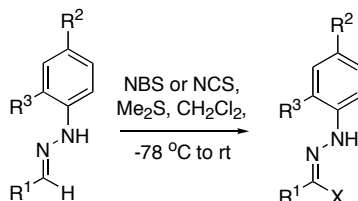


**3-(3-Furan-2-yl-acryloyl)-oxazolidin-2-one (1d):** This compound was obtained as an off white solid in 74% yield; m.p. = 135-137 °C; <sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz) δ 4.11 (t, J = 8.0 Hz, 2H), 4.44 (t, J = 8.0 Hz, 2H), 6.47 (dd, J = 1.6 Hz, 3.6 Hz, 1H), 6.70 (d, J = 3.6 Hz, 1H), 7.51 (d, J = 1.6 Hz, 1H), 7.60 (d, J = 15.2 Hz, 1H), 7.72 (d, J = 15.2 Hz, 1H). <sup>13</sup>C NMR (CDCl<sub>3</sub>, 100 MHz) δ 43.0, 62.2, 112.7, 114.3, 116.2, 132.5, 145.5, 151.5, 153.7, 165.5; IR (neat, cm<sup>-1</sup>): 3110, 2997, 2912, 1762, 1667, 1611, 1555, 1339; HRMS Exact mass calcd. for C<sub>10</sub>H<sub>9</sub>NO<sub>4</sub>Na<sup>+</sup> 230.0424 found 230.0416.



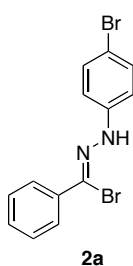
**3-[3-(3-Bromo-phenyl)-acryloyl]-oxazolidin-2-one (1h):** This compound was obtained as a white solid in 68% yield; m.p. = 153-155 °C; <sup>1</sup>H NMR (CDCl<sub>3</sub>, 500 MHz) δ 4.13 (t, J = 6.4 Hz, 2H), 4.46 (t, J = 6.4 Hz, 2H), 7.26 (t, J = 6.0 Hz, 1H), 7.51 (t, J = 6.4 Hz, 2H), 7.72 (s, 1H), 7.75 (d, J = 12.8 Hz, 1H), 7.87 (d, J = 12.8 Hz, 1H). <sup>13</sup>C NMR (CDCl<sub>3</sub>, 125 MHz) δ 43.0, 62.3, 118.2, 123.2, 127.3, 130.6, 131.4, 133.6, 136.8, 144.6, 153.7, 165.1; IR (neat, cm<sup>-1</sup>): 3091, 2923, 1764, 1676, 1621, 1555, 1474; HRMS Exact mass calcd. for C<sub>12</sub>H<sub>10</sub>BrNO<sub>3</sub>Na<sup>+</sup> 317.9736 found 317.9740.

#### General procedure for the synthesis of hydrazoneyl halides (2a-2e):

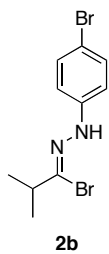


To NBS or NCS (73.2 mmol) in CH<sub>2</sub>Cl<sub>2</sub> (100 mL) at 0 °C was added methyl sulfide (147 mmol) over 5 minutes. After stirring for 15 minutes, the reaction was further cooled to –

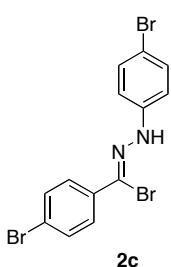
78 °C. Then the corresponding hydrazone (24.4 mmol) in CH<sub>2</sub>Cl<sub>2</sub> (75 mL) was added. The reaction was allowed to stir at –78 °C for 1 h, then slowly allowed to warm to room temperature over 3 h. The reaction was quenched by addition of 200 mL of cold water. The organic layer was then washed with 200 mL cold water, 50 mL brine, 50 mL sat. aq. Na<sub>2</sub>SO<sub>3</sub>, and 100 mL water. The organic layer was dried over MgSO<sub>4</sub>, filtered, and concentrated. Crude product was purified by flash column silica gel chromatography eluting first with 19:1 hexanes/EtOAc, then 9:1 hexanes/EtOAc to give the corresponding hydrazoneoyl halides. Chromatography should be performed quickly as hydrazoneoyl halides decompose over time on silica gel. Note: hydrazoneoyl bromides **2b**, **2c**, and **8** were unstable and should be used immediately after preparation.



**N-4-Bromophenylbenzohydrazoneoyl bromide (2a).** Obtained according to the general procedure in 95% yield as a red solid, m.p. = 94-96 °C; <sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz) δ 7.07 (m, 2H), 7.35-7.43 (m, 5H), 7.89 (m, 2H), 8.02 (br s, 1H). <sup>13</sup>C NMR (CDCl<sub>3</sub>, 100 MHz) δ 113.5, 115.4, 120.5, 127.9, 128.6, 129.8, 132.5, 135.7, 142.4; IR (neat, cm<sup>-1</sup>): 3305, 3060, 3033, 1590, 1495, 1310; HRMS Exact mass calcd. for C<sub>26</sub>H<sub>19</sub>Br<sub>2</sub>N<sub>4</sub><sup>+</sup> (nitrile imine dimer + H<sup>+</sup>) 544.9971 found 544.9995.

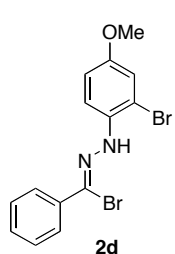


**N-4-Bromophenylisopropohydrazoneoyl bromide (2b).** Obtained according to the general procedure in 46% yield as a red oil. <sup>1</sup>H NMR (CDCl<sub>3</sub>, 500 MHz) δ 1.26 (d, J = 6.5 Hz, 6H), 2.89 (sept, J = 6.5 Hz, 1H), 6.93 (dt, J = 9 Hz, 2.5 Hz, 2H), 7.34 (dt, J = 9 Hz, 2.5 Hz, 2H), 7.57 (br s, 1H). <sup>13</sup>C NMR and high resolution mass spectroscopy were not performed due to the instability of this compound.



**N-4-Bromophenyl-4-bromobenzohydrazoneoyl bromide (2c).** Obtained according to the general procedure in 92% yield as a purple solid, m.p. = 143-146 °C. <sup>1</sup>H NMR (CDCl<sub>3</sub>, 300 MHz) δ 7.04 (d, J = 8.7 Hz, 2H), 7.40 (d, J = 8.7 Hz, 2H), 7.51 (d, J = 8.7 Hz, 2H), 7.74 (d, J = 8.7 Hz, 2H), 8.01 (br s, 1H). <sup>13</sup>C NMR (CDCl<sub>3</sub>, 75 MHz) δ 113.7,

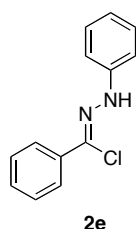
115.4, 119.2, 124.0, 129.1, 131.8, 132.5, 134.6, 142.1; IR (neat,  $\text{cm}^{-1}$ ): 3303, 3072, 1590, 1497, 1395; HRMS Exact mass calcd for  $\text{C}_{26}\text{H}_{17}\text{Br}_2\text{N}_4^+$  (nitrile imine dimer +  $\text{H}^+$ ) 700.8181 found 700.8146.



***N*-2-Bromo-4-methoxyphenylbenzohydrazonoyl bromide (2d).**

Obtained according to the general procedure in 42% yield as a red oil.  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 500 MHz)  $\delta$  3.79 (s, 3H), 6.90 (dd,  $J$  = 9 Hz, 2.5 Hz, 1H), 7.08 (d,  $J$  = 2.5 Hz, 1H), 7.38-7.42 (m, 3H), 7.49 (d,  $J$  = 9.0 Hz, 1H), 7.91 (m, 2H), 8.42 (br s, 1H).  $^{13}\text{C}$  NMR and high resolution mass spectroscopy

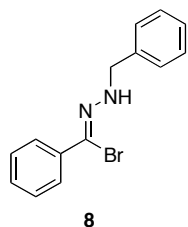
were not performed due to the instability of this compound.



***N*-Phenylbenzohydrazonoyl chloride (2e).**

**2e** has been previously prepared.<sup>7</sup> Obtained according to the general procedure in 88% yield as a yellow-red solid, m.p. = 127-128  $^{\circ}\text{C}$  (lit. 129-131  $^{\circ}\text{C}$ ).  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz)  $\delta$  6.93 (t,  $J$  = 7.2 Hz, 1H), 7.17 (d,  $J$  = 7.6 Hz, 2H), 7.30-7.42 (m, 5H), 7.92 (d,  $J$  = 7.2 Hz, 2H), 8.03 (br s, 1H). IR (neat,  $\text{cm}^{-1}$ ): 3309, 1606,

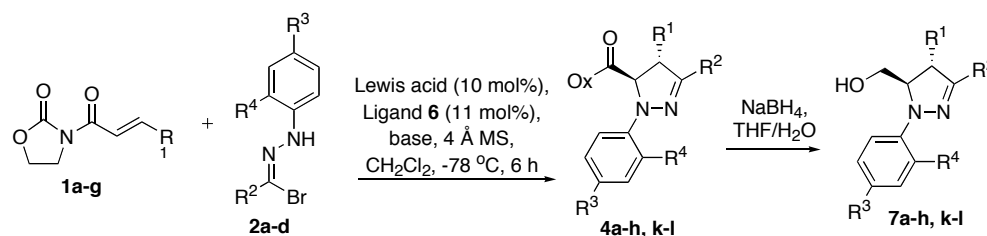
1505, 1436. HRMS Exact mass calcd for  $\text{C}_{26}\text{H}_{20}\text{N}_4^+$  (nitrile imine dimer) 388.1682 found 388.1688.



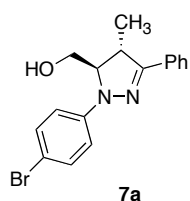
***N*-Benzylbenzohydrazonoyl bromide (8).**

Obtained according to the general procedure in 58% yield as a red oil.  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz)  $\delta$  4.59 (s, 2H), 6.19 (br s, 1H), 7.30-7.42 (m, 4H), 7.84-7.87 (m, 2H), 7.30-7.42 (m, 4H).  $^{13}\text{C}$  NMR and high resolution mass spectroscopy were not performed due to the instability of this compound.

**General Procedure for Enantioselective Nitrile Imine Cycloadditions (With Hydrazonoyl Bromides (2a-d) as Nitrile Imine Precursors):**

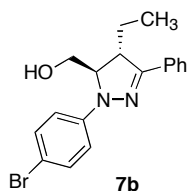


A flame-dried flask containing 4 Å molecular sieves (150 mg), Lewis acid (0.020 mmol), ligand **6** (0.022 mmol), and substrate **1a-g** (0.2 mmol) in CH<sub>2</sub>Cl<sub>2</sub> (2 mL) was stirred for 30 minutes at room temperature. The flask was then cooled to -78 °C and stirred for 5 minutes. Then hydrazonoyl bromide **2a-d** (0.3 mmol) in CH<sub>2</sub>Cl<sub>2</sub> (1 mL) was added to the reaction. After 5 minutes amine base (0.3 mmol) was added to the reaction. After stirring for 6 h at -78 °C, the reaction was quenched with 0.5 M HCl (20 mL) and extracted with diethyl ether (10 mL). The organic layer was filtered through a pad of silica gel (1 g) and MgSO<sub>4</sub> (1 g). The separatory funnel was rinsed with diethyl ether (10 mL), which was used to wash the silica gel-MgSO<sub>4</sub> pad. The combined organic layers were concentrated to yield crude **4a-h, k-l**, which was analyzed by <sup>1</sup>H NMR to determine diastereomeric ratios. Crude **4a-h, k-l** was then reconcentrated and dissolved in THF (5 mL). To this solution was added NaBH<sub>4</sub> (1.32 mmol) in H<sub>2</sub>O (1 mL). After 3 h at room temperature, the reaction was quenched with 1 M HCl (10 mL) and extracted with diethyl ether (10 mL). The organic layer was filtered through a pad of silica gel (1 g) and MgSO<sub>4</sub> (1 g). The separatory funnel was rinsed with diethyl ether (10 mL), which was used to wash the silica gel-MgSO<sub>4</sub> pad. The combined organic layers were concentrated to yield crude **7a-h, k-l**, which were purified by flash column silica gel chromatography on an ISCO<sup>TM</sup> CombiFlash Companion with AnaLogix<sup>TM</sup> RS-4 columns.



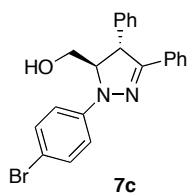
**(4*S*,5*R*)-1-(4-Bromo-phenyl)-4,5-dihydro-5-hydroxymethyl-4-methyl-3-phenyl-1*H*-pyrazole (7a):** 64 mg, 93% yield (light green foam), m.p. = 67-69 °C; The enantiomeric purity was determined by

HPLC (254 nm, 25 °C)  $t_R$  13.9 min (major);  $t_R$  19.5 min (minor) [Chiracel AD (0.46 cm x 25 cm)(from Daicel Chemical Ind., Ltd.) hexane *i*-PrOH, 95/5, 1.0 mL/min] as 99% ee.  $[\alpha]_D^{25} = +501.6$  ( $c$  0.30,  $\text{CHCl}_3$ );  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz)  $\delta$  1.32 (d,  $J = 7.2$  Hz, 3H), 1.79 (br s, 1H), 3.61 (dq,  $J = 3.2$  Hz, 7.2 Hz, 1H), 3.73 (m, 2H), 4.03 (m, 1H), 7.07 (m, 2H), 7.32-7.42 (m, 5H), 7.76 (m, 2H).  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz)  $\delta$  19.0, 43.2, 61.7, 69.4, 111.3, 114.9, 126.4, 128.9, 129.1, 131.7, 132.2, 144.0, 154.0; IR (neat,  $\text{cm}^{-1}$ ): 3500-3300 (OH), 3058, 2968, 2931, 2873, 1590, 1486, 1393; HRMS Exact mass calcd. for  $\text{C}_{17}\text{H}_{17}\text{BrN}_2\text{OH}^+$  345.0597 found 345.0580.



**(4S,5R)-1-(4-Bromo-phenyl)-4,5-dihydro-4-ethyl-5-hydroxymethyl-3-phenyl-1H-pyrazole (7b):** 67 mg, 93% yield (yellow oil); The enantiomeric purity was determined before reduction by HPLC of the oxazolidinone derivative (**4b**) (254 nm, 25 °C)  $t_R$  23.3 min (major);  $t_R$

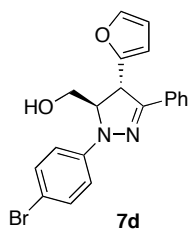
49.6 min (minor) [Chiralcel AD (0.46 cm x 25 cm) (from Daicel Chemical Ind., Ltd.) hexane/*i*-PrOH, 90/10, 1.0 mL/min] as 99% ee.  $[\alpha]_D^{25} = +430.8$  ( $c$  0.57,  $\text{CHCl}_3$ );  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 500 MHz)  $\delta$  0.90 (t,  $J = 7.5$  Hz, 3H), 1.55-1.82 (m, 3H,  $\text{CH}_2\text{-CH}_3$  + OH), 3.53 (app dt,  $J = 8.0$  Hz, 3.5 Hz, 1H), 3.75 (m, 2H), 4.13 (app q,  $J = 4.0$  Hz, 1H), 7.09 (d,  $J = 8.5$  Hz, 2H), 7.34-7.42 (m, 5H), 7.70 (d,  $J = 8.5$  Hz, 2H).  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 125 MHz)  $\delta$  10.6, 24.9, 50.0, 62.5, 66.5, 111.2, 114.8, 126.5, 128.9, 129.1, 131.9, 132.2, 143.9, 152.6; IR (neat,  $\text{cm}^{-1}$ ): 3500-3300 (OH), 3058, 2962, 2931, 2875, 1590, 1488; HRMS Exact mass calcd. for  $\text{C}_{18}\text{H}_{19}\text{BrN}_2\text{ONa}^+$  381.0573 found 381.0584.



**(4S,5R)-1-(4-Bromo-phenyl)-4,5-dihydro-3,4-diphenyl-5-hydroxymethyl-1H-pyrazole (7c):** 77 mg, 95% yield (light green foam), m.p. = 78-79 °C; The enantiomeric purity was determined by HPLC (254 nm, 25 °C)  $t_R$  14.6 min (minor);  $t_R$  20.6 min (major)

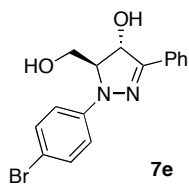
[Chiracel AD (0.46 cm x 25 cm)(from Daicel Chemical Ind., Ltd.) hexane/*i*-PrOH, 95/5, 1.0 mL/min] as 97% ee.  $[\alpha]_D^{25} = +268.6$  ( $c$  0.52,  $\text{CHCl}_3$ );  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz)  $\delta$  1.76 (br s, 1H), 3.85 (m, 2H), 4.21 (app q,  $J = 4.0$  Hz, 1H), 4.65 (d,  $J = 4.0$  Hz, 1H), 7.08 (m, 2H), 7.16 (m, 2H), 7.22-7.29 (m, 6H), 7.36 (m, 2H), 7.65 (m, 2H).  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz)  $\delta$  55.2, 62.1, 71.5, 111.8, 115.2, 126.8, 127.6, 127.7, 128.7, 128.9, 129.5,

131.8, 132.3, 140.7, 143.8, 151.2; IR (neat,  $\text{cm}^{-1}$ ): 3500-3300 (OH), 3062, 3027, 2931, 2877, 1588, 1488, 1391; HRMS Exact mass calcd. for  $\text{C}_{22}\text{H}_{19}\text{BrN}_2\text{OH}^+$  407.0735 found 407.0728.



**(4S,5R)-1-(4-Bromo-phenyl)-4,5-dihydro-4-furan-2-yl-5-hydroxymethyl-1H-pyrazole (7d):** 75 mg, 94% yield (light green solid), m.p. = 156-158 °C; The enantiomeric purity was determined by HPLC (254 nm, 25 °C)  $t_R$  31.7 min (minor);  $t_R$  51.8 min (major) [Chiracel OD-H (0.46 cm x 25 cm)(from Daicel Chemical Ind., Ltd.)

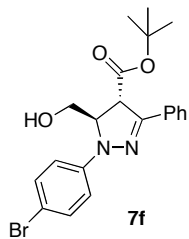
hexane/*i*-PrOH, 95/5, 1.0 mL/min] as 99% ee.  $[\alpha]_D^{25} = +352.4$  ( $c$  0.34,  $\text{CHCl}_3$ );  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 500 MHz)  $\delta$  1.88 (br s, 1H), 3.83 (br d,  $J = 11.5$  Hz, 1H), 3.91 (dd,  $J = 11.5$  Hz, 5.0 Hz, 1H), 4.40 (dd,  $J = 8.0$  Hz, 4.5 Hz, 1H), 4.86 (d,  $J = 4.5$  Hz, 1H), 6.08 (d,  $J = 3.5$  Hz, 1H), 6.26 (dd,  $J = 3.0$  Hz, 2.0 Hz, 1H), 7.09 (d,  $J = 9.0$  Hz, 2H), 7.29-7.34 (m, 4H), 7.38 (d,  $J = 9.0$  Hz, 2H), 7.72 (dd,  $J = 8.0$  Hz, 1.5 Hz, 2H).  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 125 MHz)  $\delta$  48.2, 61.7, 68.6, 107.4, 110.9, 112.1, 115.3, 126.5, 128.7, 129.1, 131.7, 132.2, 142.6, 143.7, 148.3, 152.5; IR (neat,  $\text{cm}^{-1}$ ): 3500-3300 (OH), 3060, 2933, 2879, 1590, 1488, 1391; HRMS Exact mass calcd. for  $\text{C}_{20}\text{H}_{17}\text{BrN}_2\text{O}_2\text{Na}^+$  419.0366 found 419.0363.



**(4S,5R)-1-(4-Bromo-phenyl)-4,5-dihydro-5-hydroxymethyl-3-phenyl-1H-pyrazol-4-ol (7e):** 67 mg, 97% yield (light green foam), m.p. = 82-83 °C; The enantiomeric purity was determined by HPLC (254 nm, 25 °C)  $t_R$  18.8 min (major);  $t_R$  23.3 min (minor) [Chiracel OD-H (0.46 cm x

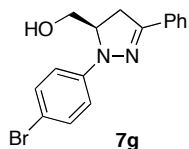
25 cm)(from Daicel Chemical Ind., Ltd.) hexane/*i*-PrOH, 93/7, 1.0 mL/min] as 96% ee.  $[\alpha]_D^{25} = +364.6$  ( $c$  0.55,  $\text{CHCl}_3$ );  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 500 MHz)  $\delta$  1.97 (br s, 1H), 2.77 (br s, 1H), 3.63 (dd,  $J = 11.5$  Hz, 6.5 Hz, 1H), 3.83 (dd,  $J = 11.5$  Hz, 3.0 Hz, 1H), 4.16 (app quintet,  $J = 3.0$  Hz, 1H), 5.32 (br s, 1H), 7.01 (d,  $J = 9.0$  Hz, 2H), 7.33-7.39 (m, 5H), 7.82 (d,  $J = 7.0$  Hz, 2H).  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 125 MHz)  $\delta$  60.3, 70.0, 77.5, 112.4, 115.2, 126.3, 129.0, 129.3, 131.0, 132.4, 143.0, 149.4; IR (neat,  $\text{cm}^{-1}$ ): 3500-3200 (OH), 2952, 2890, 1592, 1490, 1396; HRMS Exact mass calcd. for  $\text{C}_{16}\text{H}_{15}\text{BrN}_2\text{O}_2\text{Na}^+$  369.0209 found 369.0222.





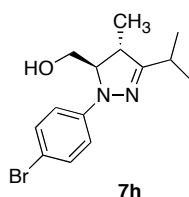
**(4*S*,5*R*)-1-(4-Bromo-phenyl)-4,5-dihydro-5-hydroxymethyl-3-phenyl-1*H*-pyrazole-4-carboxylic acid *tert*-butyl ester (7f):** 77 mg,

90% yield (light green solid), m.p. =155-157 °C. The enantiomeric purity was determined by HPLC (254 nm, 25 °C)  $t_R$  21.8 min (major);  $t_R$  42.2 min (minor) [Chiracel AD (0.46 cm x 25 cm)(from Daicel Chemical Ind., Ltd.) hexane/*i*-PrOH, 95/5, 1.0 mL/min] as 93% ee.  $[\alpha]_D^{25} = +222.6$  ( $c$  1.1, CHCl<sub>3</sub>); <sup>1</sup>H NMR (CDCl<sub>3</sub>, 500 MHz)  $\delta$  1.32 (s, 9H), 1.88 (br s, 1H), 3.78 (br d,  $J$  = 11.5 Hz, 1H), 3.90 (dd,  $J$  = 11.5 Hz, 4.5 Hz, 1H), 4.34 (d,  $J$  = 4.5 Hz, 1H), 4.66 (dd,  $J$  = 8.5 Hz, 4.5Hz, 1H), 7.09 (m, 2H), 7.34-7.40 (m, 5H), 7.81 (m, 2H). <sup>13</sup>C NMR (CDCl<sub>3</sub>, 125 MHz)  $\delta$  27.9, 55.7, 62.1, 66.3, 82.8, 112.1, 115.4, 126.8, 128.5, 129.2, 131.7, 132.2, 143.5, 146.8, 169.4; IR (neat, cm<sup>-1</sup>): 3500-3300 (OH); 2979, 2933, 1727, 1588, 1490, 1393; HRMS Exact mass calcd. for C<sub>21</sub>H<sub>23</sub>BrN<sub>2</sub>O<sub>3</sub>Na<sup>+</sup> 453.0784 found 453.0757.



**(5*R*)-1-(4-Bromo-phenyl)-4,5-dihydro-5-hydroxymethyl-3-phenyl-1*H*-pyrazole (7g):** 63 mg, 95% yield (light green solid), m.p. = 156-159

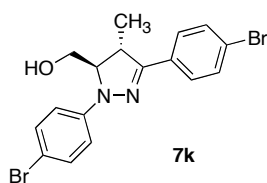
°C; The enantiomeric purity was determined by HPLC (254 nm, 25 °C)  $t_R$  20.7 min (major);  $t_R$  26.4 min (minor) [Chiracel AD (0.46 cm x 25 cm) (from Daicel Chemical Ind., Ltd.) hexane *i*-PrOH, 95/5, 1.0 mL/min] as 84% ee.  $[\alpha]_D^{25} = +343.7$  ( $c$  0.57, CHCl<sub>3</sub>); <sup>1</sup>H NMR (CDCl<sub>3</sub>, 500 MHz)  $\delta$  1.60 (br s, 1H), 3.31 (dd,  $J$  = 5.5 Hz, 17.0 Hz, 1H), 3.47 (dd,  $J$  = 7.5 Hz, 17.0 Hz, 1H), 3.75 (dd,  $J$  = 2.5 Hz, 11.0 Hz, 1H), 3.90 (dd,  $J$  = 5.0 Hz, 11.0 Hz, 1H), 4.44-4.49 (m, 1H), 7.09 (d,  $J$  = 9.0 Hz, 2H), 7.34-7.41 (m, 5H), 7.73 (d,  $J$  = 7.0 Hz, 2H). <sup>13</sup>C NMR (CDCl<sub>3</sub>, 125 MHz)  $\delta$  36.4, 61.5, 62.7, 111.7, 115.1, 126.2, 128.8, 129.3, 132.2, 132.5, 144.3, 149.8; IR (neat, cm<sup>-1</sup>): 3500-3300 (OH), 3060, 2929, 1590, 1488, 1397; HRMS Exact mass calcd. for C<sub>16</sub>H<sub>15</sub>BrN<sub>2</sub>ONa<sup>+</sup> 353.0260 found 353.0242.



**(4*S*,5*R*)-1-(4-Bromo-phenyl)-4,5-dihydro-5-hydroxymethyl-3-isopropyl-4-methyl-1*H*-pyrazole (7h):** 61 mg, 98% yield (pink oil);

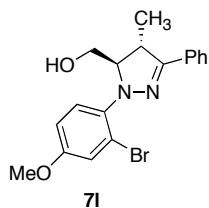
The enantiomeric purity was determined by HPLC (254 nm, 25 °C)  $t_R$  35.1 min (major);  $t_R$  40.5 min (minor) [Chiracel AD (0.46 cm x 25 cm)(from Daicel Chemical Ind., Ltd.) hexane/*i*-PrOH, 95/5, 1.0 mL/min] as 99% ee.

$[\alpha]_D^{25} = +255.4$  (*c* 0.74, CHCl<sub>3</sub>); <sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz)  $\delta$  1.17 (d, *J* = 7.2 Hz, 3H), 1.21 (d, *J* = 7.2 Hz, 3H), 1.26 (d, *J* = 6.8 Hz, 3H), 1.77 (br s, 1H) 2.65 (sept, *J* = 7.2 Hz, 1H), 3.17 (overlapping dq, *J* = 5.6 Hz, 6.8 Hz, 1H), 3.62-3.67 (m, 2H), 3.80 (m, 1H), 6.95 (d, *J* = 8.8 Hz, 2H), 7.31 (d, *J* = 8.8 Hz, 2H). <sup>13</sup>C NMR (CDCl<sub>3</sub>, 100 MHz)  $\delta$  18.0, 19.9, 21.2, 28.0, 44.8, 62.4, 70.0, 111.3, 115.2, 132.0, 146.0, 163.4; IR (neat, cm<sup>-1</sup>): 3500-3300 (OH), 2968, 2931, 2875, 1590, 1490, 1333; HRMS Exact mass calcd. for C<sub>14</sub>H<sub>19</sub>BrN<sub>2</sub>ONa<sup>+</sup> 333.0573 found 333.0570.



**(4*S*,5*R*)-1,3-Bis-(4-bromo-phenyl)-4,5-dihydro-5-hydroxymethyl-4-methyl-1*H*-pyrazole (7k):** 80 mg, 95% yield (yellow-green foam), m.p. = 65-66 °C; The enantiomeric purity was determined by HPLC (254 nm, 25 °C) *t<sub>R</sub>* 19.7 min (major); *t<sub>R</sub>*

28.6 min (minor) [Chiracel OD-H (0.46 cm x 25 cm)(from Daicel Chemical Ind., Ltd.) hexane/*i*-PrOH, 1.0 mL/min] as 95% ee.  $[\alpha]_D^{25} = +380.5$  (*c* 0.68, CHCl<sub>3</sub>); <sup>1</sup>H NMR (CDCl<sub>3</sub>, 500 MHz)  $\delta$  1.28 (d, *J* = 7.5 Hz, 3H), 2.09 (br s, 1H), 3.55 (dq, *J* = 3.0 Hz, 7.5 Hz, 1H), 3.68 (d, *J* = 5.0 Hz, 2H), 4.01 (dd, *J* = 7.5 Hz, 5.0 Hz, 1H), 7.04 (d, *J* = 9.0 Hz, 2H), 7.36 (d, *J* = 9.0 Hz, 2H), 7.50 (d, *J* = 8.5 Hz, 2H), 7.60 (d, *J* = 8.5 Hz, 2H). <sup>13</sup>C NMR (CDCl<sub>3</sub>, 125 MHz)  $\delta$  18.9, 42.9, 61.5, 69.3, 111.4, 114.9, 122.9, 127.8, 130.6, 132.0, 132.2, 143.5, 152.7; IR (neat, cm<sup>-1</sup>): 3500-3300 (OH), 3062, 3029, 2931, 2877, 1590, 1486, 1385; HRMS Exact mass calcd. for C<sub>17</sub>H<sub>16</sub>Br<sub>2</sub>N<sub>2</sub>ONa<sup>+</sup> 444.9522 found 444.9523.



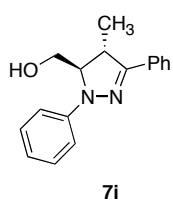
**(4*S*,5*R*)-1-(2-Bromo-4-methoxy-phenyl)-4,5-dihydro-5-hydroxymethyl-4-methyl-3-phenyl-1*H*-pyrazole (7l):** 72 mg, 96% yield (clear oil); The enantiomeric purity was determined by HPLC (254 nm, 25 °C) *t<sub>R</sub>* 24.4 min (major); *t<sub>R</sub>* 28.7 min (minor) [Chiracel AD

(0.46 cm x 25 cm)(from Daicel Chemical Ind., Ltd.) hexane/*i*-PrOH, 95/5, 1.0 mL/min] as 96% ee.  $[\alpha]_D^{25} = -99.6$  (*c* 0.45, CHCl<sub>3</sub>); <sup>1</sup>H NMR (CDCl<sub>3</sub>, 500 MHz)  $\delta$  1.40 (d, *J* = 7.0 Hz, 3H), 1.77 (br s, 1H), 3.51 (dd, *J* = 12.0 Hz, 3.5 Hz, 1H), 3.57 (dd, *J* = 12.0 Hz, 5 Hz, 1H), 3.74 (app. quintet, *J* = 7.0 Hz, 1H), 3.81 (s, 3H), 4.10 (app. quartet, *J* = 5.0 Hz, 1H), 6.87 (dd, *J* = 8.5 Hz, 2.5Hz, 1H), 7.15 (d, *J* = 2.5 Hz, 1H), 7.34-7.41 (m, 4H), 7.72 (d, *J* =

8.0 Hz, 2H).  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 125 MHz)  $\delta$  17.6, 42.6, 55.9, 61.2, 74.4, 114.6, 118.3, 119.2, 126.5, 126.8, 128.7, 128.9, 132.1, 138.3, 155.4, 157.8; IR (neat,  $\text{cm}^{-1}$ ): 3500-3300 (OH), 3058, 2966, 2937, 2836, 1600, 1567, 1488; HRMS Exact mass calcd for  $\text{C}_{18}\text{H}_{19}\text{BrN}_2\text{O}_2\text{Na}^+$  397.0522 found 397.0530.

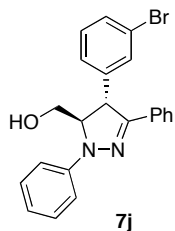
**General Procedure for Enantioselective Nitrile Imine Cycloadditions (With Hydrazonoyl Chlorides (**2e**) as the Nitrile Imine Precursor):** A flame-dried flask containing 4 Å molecular sieves (150 mg), Lewis acid (0.060 mmol), ligand **6** (0.066 mmol), and substrate **1a** or **1h** (0.2 mmol) in  $\text{CH}_2\text{Cl}_2$  (2 mL) was stirred for 30 minutes at room temperature. The flask was then cooled to  $-20\text{ }^\circ\text{C}$  and stirred for 5 minutes. Then hydrazonoyl chloride **2e** (0.3 mmol) in  $\text{CH}_2\text{Cl}_2$  (1 mL) was added to the reaction. After 5 minutes  $\text{NEt}_3$  (0.3 mmol) was added to the reaction. After stirring for 48 h at  $-20\text{ }^\circ\text{C}$ , the reaction was quenched with 0.5 M HCl (20 mL) and extracted with diethyl ether (10 mL). The organic layer was filtered through a pad of silica gel (1 g) and  $\text{MgSO}_4$  (1g). The separatory funnel was rinsed with diethyl ether (10 mL), which was used to wash the silica gel- $\text{MgSO}_4$  pad. The combined organic layers were concentrated to yield crude **4i** or **4j**, which was analyzed by  $^1\text{H}$  NMR to determine diastereomeric ratios. Crude **4i** or **4j** was then reconcentrated and dissolved in THF (5 mL). To this solution was added  $\text{NaBH}_4$  (1.32 mmol) in  $\text{H}_2\text{O}$  (1 mL). After 3 h at room temperature, the reaction was quenched with 1 M HCl (10 mL) and extracted with diethyl ether (10 mL). The organic layer was filtered through a pad of silica gel (1 g) and  $\text{MgSO}_4$  (1g). The separatory funnel was rinsed with diethyl ether (10 mL), which was used to wash the silica gel- $\text{MgSO}_4$  pad. The combined organic layers were concentrated to yield crude **7i** or **7j**, which were purified by flash column silica chromatography on an ISCO<sup>TM</sup> CombiFlash Companion with AnaLogix<sup>TM</sup> RS-4 columns.

Note: The temperature of reactions using **2e** as the nitrile imine could be lowered to  $-78\text{ }^\circ\text{C}$  if (tert-butylimino)tris(dimethylamino) phosphorane was used as the base.



**(4*S*,5*R*)-4,5-dihydro-1,3-diphenyl-5-hydroxymethyl-4-methyl-1*H*-pyrazole (**7i**):** 49 mg, 92% yield (light green foam or solid), m.p. = 90-91  $^\circ\text{C}$ ; The enantiomeric purity was determined by HPLC (254 nm, 25  $^\circ\text{C}$ )  $t_R$

18.5 min (major);  $t_R$  25.4 min (minor) [Chiracel AD (0.46 cm x 25 cm)(from Daicel Chemical Ind., Ltd.) hexane/*i*-PrOH, 97/3, 1.0 mL/min] as 95% ee.  $[\alpha]_D^{25} = +582.0$  (*c* 0.34, CHCl<sub>3</sub>); <sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz)  $\delta$  1.33 (d, *J* = 7.2 Hz, 3H), 1.72 (br s, 1H), 3.63 (dq, *J* = 3.2 Hz, 7.2 Hz, 1H), 3.77 (d, *J* = 4.8 Hz, 2H), 4.09 (m, 1H), 6.86 (t, *J* = 7.2 Hz, 1H), 7.21 (d, *J* = 7.6 Hz, 2H), 7.29-7.35 (m, 3H), 7.40 (t, *J* = 7.2 Hz, 2H), 7.78 (d, *J* = 7.6 Hz, 2H). <sup>13</sup>C NMR (CDCl<sub>3</sub>, 100 MHz)  $\delta$  19.1, 43.1, 61.9, 69.4, 113.4, 119.4, 126.4, 128.78, 128.82, 129.5, 132.0, 145.0, 153.3; IR (neat, cm<sup>-1</sup>): 3500-3300 (OH), 3058, 2966, 2929, 2873, 1596, 1493, 1381, 1138; HRMS Exact mass calcd. for C<sub>17</sub>H<sub>18</sub>N<sub>2</sub>ONa<sup>+</sup> 289.1311 found 289.1315.

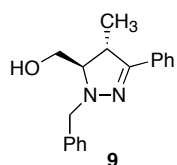


**(4*S*,5*R*)-4-(3-Bromo-phenyl)-4,5-dihydro-1,3-diphenyl-5-hydroxymethyl-1*H*-pyrazole (7j):** 77mg, 95% yield (light green foam), m.p. = 73-75 °C; The enantiomeric purity was determined by HPLC (254 nm, 25 °C)  $t_R$  7.7 min (major);  $t_R$  16.3 min (minor) [Chiracel AD (0.46 cm x 25 cm)(from Daicel Chemical Ind., Ltd.) hexane/*i*-PrOH, 90/10, 1.0

mL/min] as 97% ee.  $[\alpha]_D^{25} = +203.8$  (*c* 0.50, CHCl<sub>3</sub>); <sup>1</sup>H NMR (CDCl<sub>3</sub>, 500 MHz)  $\delta$  1.84 (br s, 1H), 3.88 (d, *J* = 4.25 Hz, 2H), 4.27 (q, *J* = 4.25 Hz, 1H), 4.67 (d, *J* = 4.25 Hz, 1H), 6.90 (t, *J* = 7.5 Hz, 1H), 7.10 (d, *J* = 7.0 Hz, 1H), 7.14 (t, *J* = 7.5 Hz, 1H), 7.22 (d, *J* = 8.0 Hz, 2H), 7.26-7.33 (m, 5H), 7.37 (d, *J* = 8.0 Hz, 1H), 7.42 (s, 1H), 7.67 (d, *J* = 7.5 Hz, 2H). <sup>13</sup>C NMR (CDCl<sub>3</sub>, 125 MHz)  $\delta$  54.4, 61.8, 71.2, 113.7, 112.0, 123.4, 126.3, 126.6, 128.7, 128.8, 129.6, 130.6, 130.8, 131.1, 131.8, 143.2, 144.4, 149.6; IR (neat, cm<sup>-1</sup>): 3500-3300 (OH), 3058, 2935, 2879, 1606, 1501, 1397; HRMS Exact mass calcd. for C<sub>22</sub>H<sub>19</sub>BrN<sub>2</sub>ONa<sup>+</sup> 429.0573 found 429.0578.

**General Procedure for Enantioselective Nitrile Imine Cycloadditions (With *N*-Benzylbenzohydrazonoyl bromide (8) as the Nitrile Imine Precursors):** A flame-dried flask containing 4 Å molecular sieves (150 mg), Lewis acid (0.060 mmol), ligand **6** (0.066 mmol), and substrate **1a** (0.2 mmol) in CH<sub>2</sub>Cl<sub>2</sub> (2 mL) was stirred for 30 minutes at room temperature. The flask was then cooled to -78 °C and stirred for 5 minutes. Then hydrazonoyl bromide **8** (0.3 mmol) in CH<sub>2</sub>Cl<sub>2</sub> (1 mL) was added to the reaction. After 5 minutes DBU or (tert-butylimino)tris(dimethylamino) phosphorane (0.3 mmol)

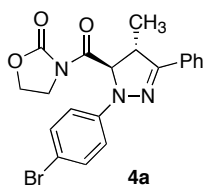
was added to the reaction. After stirring for 6 h at  $-78\text{ }^{\circ}\text{C}$ , the reaction was quenched with 0.5 M HCl (20 mL) and extracted with diethyl ether (10 mL). The organic layer was filtered through a pad of silica gel (1 g) and  $\text{MgSO}_4$  (1 g). The separatory funnel was rinsed with diethyl ether (10 mL), which was used to wash the silica gel- $\text{MgSO}_4$  pad. The combined organic layers were concentrated to yield crude oxazolidinone intermediate, which was analyzed by  $^1\text{H}$  NMR to determine diastereomeric ratios. The crude oxazolidinone intermediate was then reconcentrated and dissolved in THF (5 mL). To this solution was added  $\text{NaBH}_4$  (1.32 mmol) in  $\text{H}_2\text{O}$  (1 mL). After 3 h at room temperature, the reaction was quenched with 1 M HCl (10 mL) and extracted with diethyl ether (10 mL). The organic layer was filtered through a pad of silica gel (1 g) and  $\text{MgSO}_4$  (1 g). The separatory funnel was rinsed with diethyl ether (10 mL), which was used to wash the silica gel- $\text{MgSO}_4$  pad. The combined organic layers were concentrated to yield crude **9**, which was purified by flash column silica chromatography on an ISCO<sup>TM</sup> CombiFlash Companion with AnaLogix<sup>TM</sup> RS-4 columns.



**(4S,5R)-1-Benzyl-4,5-dihydro-5-hydroxymethyl-4-methyl-3-phenyl-1H-pyrazole (9):** 32 mg, 57% yield (tan oil); The enantiomeric purity was determined by HPLC (254 nm,  $25\text{ }^{\circ}\text{C}$ )  $t_R$  52.6 min (major);  $t_R$  60.0 min (minor) [Chiracel OD-H (1 cm x 25 cm)(from Daicel Chemical Ind., Ltd.) hexane/*i*-

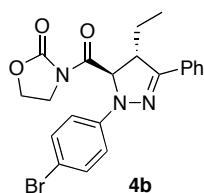
PrOH, 93/7, 1.0 mL/min] as 94% ee.  $[\alpha]_D^{25} = +56.8$  (*c* 0.42,  $\text{CHCl}_3$ );  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz)  $\delta$  1.21 (d,  $J = 6.8\text{ Hz}$ , 3H), 2.20 (br s, 1H), 3.03 (dt,  $J = 10.0\text{ Hz}$ , 3.0 Hz, 1H), 3.51-3.59 (m, 3H), 4.40 (br s, 2H), 7.28-7.40 (m, 8H), 7.61 (m, 2H).  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz)  $\delta$  18.2, 42.8, 58.7, 61.0, 73.5, 126.7(2C), 127.8, 128.6 (4C), 128.7, 129.5(2C), 132.6, 137.1, 155.1; IR (neat,  $\text{cm}^{-1}$ ): 3500-3300 (OH), 3062, 3029, 2962, 2929, 2873, 1663, 1453; HRMS Exact mass calcd. for  $\text{C}_{18}\text{H}_{20}\text{N}_2\text{ONa}^+$  303.1468 found 303.1462.

### Supporting Information for Intermediates 4a-l



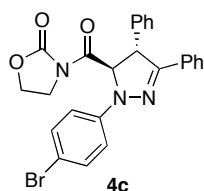
**(4S,5R)-1-(4-Bromo-phenyl)-4,5-dihydro-4-methyl-5-(oxazolidin-2-one-3-carbonyl)-3-phenyl-1H-pyrazole (4a):** Isolated as a yellow solid, m.p. =  $221\text{--}225\text{ }^{\circ}\text{C}$ .  $[\alpha]_D^{25} = +207.8$  (*c* 0.41,  $\text{CHCl}_3$ ), 99% ee;

$^1\text{H}$  NMR ( $\text{CDCl}_3$ , 500 MHz)  $\delta$  1.50 (d,  $J$  = 7.0 Hz, 3H), 3.61 (dq,  $J$  = 2.0 Hz, 7 Hz, 1H), 3.87-3.99 (m, 2H), 4.45 (app. dt,  $J$  = 2.0 Hz, 8.5 Hz, 2H), 5.68 (d,  $J$  = 2.0 Hz, 1H), 6.95 (d,  $J$  = 9.0 Hz, 2H), 7.32-7.40 (m, 5H), 7.40 (d, 7.5 Hz, 2H).  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 125 MHz)  $\delta$  18.2, 42.6, 46.7, 63.2, 68.3, 111.6, 114.7, 126.3, 128.8, 129.1, 131.1, 132.2, 143.7, 152.1, 154.1, 169.0; IR (neat,  $\text{cm}^{-1}$ ): 3058, 2979, 2927, 1776, 1706, 1590, 1492, 1387; HRMS Exact mass calcd. for  $\text{C}_{20}\text{H}_{18}\text{BrN}_3\text{O}_3\text{Na}^+$  450.0424 found 450.0426.



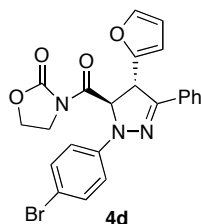
**(4S,5R)-1-(4-Bromo-phenyl)-4,5-dihydro-4-ethyl-5-(oxazolidin-2-one-3-carbonyl)-3-phenyl-1H-pyrazole (4b):** Isolated as a white foam, m.p. = 77-79 °C.  $[\alpha]_{\text{D}}^{25} = +124.2$  ( $c$  0.39,  $\text{CHCl}_3$ ), 99% ee;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 500 MHz)  $\delta$  0.89 (t,  $J$  = 7.0 Hz, 3H), 1.85 (m, 1H), 2.04

(m, 1H), 3.69 (m, 1H), 3.89-4.06 (m, 2H), 4.45-4.52 (m, 2H), 5.88 (d,  $J$  = 2.5 Hz, 1H), 6.95 (d,  $J$  = 8.5 Hz, 2H), 7.31-7.39 (m, 5H), 7.74 (d,  $J$  = 7.5 Hz, 2H).  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 125 MHz)  $\delta$  9.5, 24.6, 42.8, 52.6, 63.1, 65.4, 111.6, 114.5, 126.4, 128.8, 129.1, 131.6, 132.2, 143.3, 150.3, 154.0, 169.0; IR (neat,  $\text{cm}^{-1}$ ): 3058, 2968, 2933, 2877, 1771, 1704, 1590, 1490, 1385; HRMS Exact mass calcd. for  $\text{C}_{21}\text{H}_{20}\text{BrN}_3\text{O}_3\text{Na}^+$  464.0580 found 464.0566.



**(4S,5R)-1-(4-Bromo-phenyl)-4,5-dihydro-3,4-diphenyl-5-(oxazolidin-2-one-3-carbonyl)-1H-pyrazole (4c):** Isolated as a yellow foam, m.p. = 107-110 °C.  $[\alpha]_{\text{D}}^{25} = -47.7$  ( $c$  1.46,  $\text{CHCl}_3$ ), 97% ee;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 500 MHz)  $\delta$  3.90-4.01 (m, 2H), 4.45 (t,  $J$  = 8.5

Hz, 2H), 4.63 (d,  $J$  = 2.0 Hz, 1H), 5.99 (d,  $J$  = 2.0 Hz, 1H), 7.03 (d,  $J$  = 9.0 Hz, 2H), 7.25-7.30 (m, 8H), 7.38 (d,  $J$  = 9.0 Hz, 2H), 7.67 (dd,  $J$  = 8.0 Hz, 1.5 Hz, 2H).  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 125 MHz)  $\delta$  42.7, 57.5, 63.1, 69.4, 112.0, 115.0, 126.5, 128.0, 128.2, 128.6, 129.2, 131.3, 132.3, 137.6, 143.5, 146.4, 150.6, 153.7, 168.6; IR (neat,  $\text{cm}^{-1}$ ): 3060, 3031, 2989, 2923, 1775, 1700, 1588, 1490, 1387; HRMS Exact mass calcd. for  $\text{C}_{25}\text{H}_{20}\text{BrN}_3\text{O}_3\text{Na}^+$  512.0580 found 512.0580.



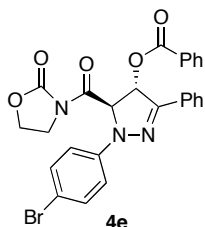
**(4*S*,5*R*)-1-(4-Bromo-phenyl)-4,5-dihydro-4-furan-2-yl-5-**

**(oxazolidin-2-one-3-carbonyl)-1*H*-pyrazole (4d):**

Isolated as a yellow foam, m.p. = 105-107 °C.  $[\alpha]_D^{25} = -7.9$  (*c* 0.58, CHCl<sub>3</sub>), 99% ee;

<sup>1</sup>H NMR (CDCl<sub>3</sub>, 500 MHz)  $\delta$  3.96-4.07 (m, 2H), 4.50 (t, *J* = 8.5 Hz, 2H), 4.82 (d, *J* = 3.0 Hz, 1H), 6.19 (d, *J* = 3.0 Hz, 1H), 6.23 (d, *J* = 3.0

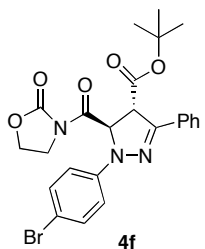
Hz, 1H), 6.30 (dd, *J* = 2.0 Hz, 3.0 Hz, 1H), 7.00 (d, *J* = 9.0 Hz, 2H), 7.29-7.38 (m, 6H), 7.70 (d, *J* = 7.0 Hz, 2H). <sup>13</sup>C NMR (CDCl<sub>3</sub>, 125 MHz)  $\delta$  42.8, 50.7, 63.1, 67.2, 108.2, 111.0, 112.3, 115.0, 126.4, 128.7, 129.2, 131.3, 132.3, 142.7, 143.3, 147.2, 150.6, 153.7, 168.7; IR (neat, cm<sup>-1</sup>): 3058, 2989, 2923, 1771, 1704, 1590, 1490, 1387; HRMS Exact mass calcd. for C<sub>23</sub>H<sub>18</sub>BrN<sub>3</sub>O<sub>4</sub>Na<sup>+</sup> 502.0373 found 502.0370.



**(4*S*,5*R*)-Benzoic acid 1-(4-bromo-phenyl)-4,5-dihydro-5-(oxazolidin-2-one-3-carbonyl)-3-phenyl-1*H*-pyrazol-4-yl ester (4e):**

Isolated as a yellow foam, m.p. = 101-103 °C.  $[\alpha]_D^{25} = -46.5$  (*c* 0.72, CHCl<sub>3</sub>), 96% ee; <sup>1</sup>H NMR (CDCl<sub>3</sub>, 500 MHz)  $\delta$  4.07-4.13 (m,

2H), 4.49-4.58 (m, 2H), 6.38 (d, *J* = 3.0 Hz, 1H), 6.80 (d, *J* = 3.0 Hz, 1H), 7.04 (d, *J* = 9.0 Hz, 2H), 7.31-7.43 (m, 7H), 7.57 (t, *J* = 7.5 Hz, 1H), 7.79 (d, *J* = 7.5 Hz, 2H), 8.03 (dd, *J* = 8.0 Hz, 1.0 Hz, 2H). <sup>13</sup>C NMR (CDCl<sub>3</sub>, 125 MHz)  $\delta$  43.0, 63.0, 66.9, 78.4, 113.0, 114.9, 126.1, 128.7, 128.9, 129.0, 129.4, 130.3, 130.4, 132.4, 134.0, 142.3, 144.4, 153.7, 165.9, 167.7; IR (neat, cm<sup>-1</sup>): 3062, 2989, 2923, 1775, 1706, 1590, 1492, 1451, 1389; HRMS Exact mass calcd. for C<sub>26</sub>H<sub>20</sub>BrN<sub>3</sub>O<sub>5</sub>Na<sup>+</sup> 556.0479 found 556.0490.



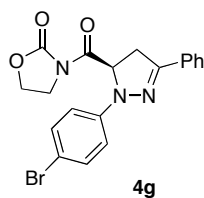
**(4*S*,5*R*)-1-(4-Bromo-phenyl)-4,5-dihydro-5-(oxazolidin-2-one-3-carbonyl)-3-phenyl-1*H*-pyrazole-4-carboxylic acid *tert*-butyl ester (4f):**

Isolated as a yellow solid, m.p. = 115-118 °C.  $[\alpha]_D^{25} = +50.6$  (*c* 0.63, CHCl<sub>3</sub>), 93% ee; <sup>1</sup>H NMR (CDCl<sub>3</sub>, 500 MHz)  $\delta$  1.37 (s, 9H),

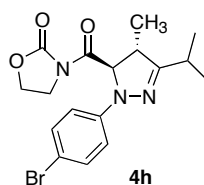
3.97-4.10 (m, 2H), 4.29 (d, *J* = 4.0 Hz, 1H), 4.54 (t, *J* = 8.0 Hz, 2H),

6.20 (d, *J* = 4.0 Hz, 1H), 6.93 (d, *J* = 9.0 Hz, 2H), 7.32-7.38 (m, 5H), 7.86 (d, *J* = 8.0 Hz, 2H). <sup>13</sup>C NMR (CDCl<sub>3</sub>, 125 MHz)  $\delta$  27.9, 42.7, 57.7, 63.2, 65.6, 83.4, 112.0, 114.7, 126.8, 128.5, 129.2, 131.1, 132.2, 142.6, 144.3, 153.7, 167.6, 168.2; IR (neat, cm<sup>-1</sup>):

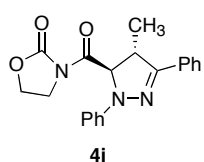
3060, 2979, 2931, 1775, 1710, 1590, 1492, 1380; HRMS Exact mass calcd. for  $C_{24}H_{24}BrN_3O_5Na^+$  536.0792 found 536.0785.



**(5R)-1-(4-Bromo-phenyl)-4,5-dihydro-5-(oxazolidin-2-one-3-carbonyl)-3-phenyl-1H-pyrazole (4g):** Isolated as a off-white solid, m.p. = 240-243 °C.  $[\alpha]_D^{25} = +274.5$  (c 0.57,  $CHCl_3$ ), 84% ee;  $^1H$  NMR ( $CDCl_3$ , 500 MHz)  $\delta$  3.28 (dd, J = 5.5 Hz, 17.5 Hz, 1H), 3.90 (dd, J = 12.5 Hz, 17.5 Hz, 1H), 4.01-4.14 (m, 2H), 4.52-4.60 (m, 2H), 5.94 (dd, J = 5.5 Hz, 12.5 Hz, 1H), 6.20 (d, J = 9.0 Hz, 2H), 7.34-7.41 (m, 5H), 7.70 (d, J = 7.0 Hz, 2H).  $^{13}C$  NMR ( $CDCl_3$ , 125 MHz)  $\delta$  39.0, 42.7, 61.4, 63.2, 111.8, 114.7, 126.1, 128.8, 129.3, 132.0, 132.2, 143.5, 147.3, 153.8, 169.8; IR (neat,  $cm^{-1}$ ): 2921, 1787, 1700, 1590, 1503, 1387; HRMS Exact mass calcd. for  $C_{19}H_{16}BrN_3O_3Na^+$  436.0267 found 436.0270.



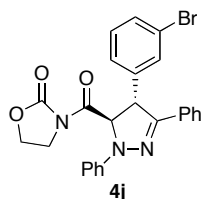
**(4S,5R)-1-(4-Bromo-phenyl)-4,5-dihydro-3-isopropyl-4-methyl-5-(oxazolidin-2-one-3-carbonyl)-1H-pyrazole (4h):** Isolated as a pink foam, m.p. = 74-75 °C.  $[\alpha]_D^{25} = +12.1$  (c 0.69,  $CHCl_3$ ), 99% ee;  $^1H$  NMR ( $CDCl_3$ , 500 MHz)  $\delta$  1.14 (d, J = 7.0 Hz, 3H), 1.27 (d, J = 7.0 Hz, 3H), 1.38 (d, J = 7.0 Hz, 3H), 2.64 (sept, J = 7.0 Hz, 1H), 3.14 (dq, J = 2.5 Hz, 7.0 Hz, 1H), 3.94-4.05 (m, 2H), 4.50 (t, J = 8.0 Hz, 2H), 5.44 (d, J = 2.5 Hz, 1H), 6.08 (d, J = 9.0 Hz, 2H), 7.29 (d, J = 9.0 Hz, 2H).  $^{13}C$  NMR ( $CDCl_3$ , 125 MHz)  $\delta$  17.6, 20.0, 21.4, 28.0, 42.6, 48.3, 63.1, 67.5, 110.8, 114.3, 132.1, 144.5, 154.0, 160.7, 169.9; IR (neat,  $cm^{-1}$ ): 2970, 2933, 2875, 1773, 1706, 1592, 1403, 1387; HRMS Exact mass calcd. for  $C_{17}H_{20}BrN_3O_3Na^+$  416.0580 found 416.0577.



**(4S,5R)-4,5-dihydro-4-methyl-1,3-diphenyl-5-(oxazolidin-2-one-3-carbonyl)-1H-pyrazole (4i):** Isolated as a yellow foam, m.p. = 96-98 °C.  $[\alpha]_D^{25} = +235.9$  (c 0.57,  $CHCl_3$ ), 95% ee;  $^1H$  NMR ( $CDCl_3$ , 500 MHz)  $\delta$  1.50 (d, J = 7.5 Hz, 3H), 3.60 (dq, J = 2.0 Hz, 7.5 Hz, 1H), 3.89-4.01 (m, 2H), 4.46 (m, 2H), 5.72 (d, J = 2.0 Hz, 1H), 6.85 (t, J = 7.0 Hz, 1H), 7.07 (d, J = 7.5 Hz, 2H), 7.26-7.33 (m, 3H), 7.38 (t, J = 7.0 Hz, 2H), 7.74 (d, J = 7.5 Hz, 2H).  $^{13}C$  NMR ( $CDCl_3$ , 125 MHz)  $\delta$  18.2, 42.6, 46.4, 63.2, 68.3, 113.0, 119.7, 126.2, 128.8,



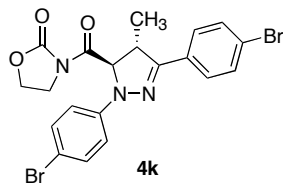
128.9, 129.5, 131.5, 144.5, 151.4, 154.2, 169.4; IR (neat,  $\text{cm}^{-1}$ ): 3058, 3031, 2977, 2929, 1771, 1704, 1598, 1505, 1387; HRMS Exact mass calcd. for  $\text{C}_{20}\text{H}_{19}\text{N}_3\text{O}_3\text{Na}^+$  372.1319 found 372.1320.



**(4*S*,5*R*)-4-(3-Bromo-phenyl)-4,5-dihydro-1,3-diphenyl-5-(oxazolidin-2-one-3-carbonyl)-1*H*-pyrazole (4j):** Isolated as a yellow

foam, m.p. = 118-121 °C.  $[\alpha]_{\text{D}}^{25} = -93.1$  ( $c$  0.64,  $\text{CHCl}_3$ ), 97% ee;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 500 MHz)  $\delta$  3.97-4.07 (m, 2H), 4.52 (m, 2H), 4.58 (d,  $J$

= 2.0 Hz, 1H), 6.01 (d,  $J$  = 2.0 Hz, 1H), 6.92 (t,  $J$  = 7.5 Hz, 1H), 7.15 (d,  $J$  = 8.0 Hz, 2H), 7.19 (t,  $J$  = 8.0 Hz, 1H), 7.26-7.34 (m, 7 H), 7.41 (dt,  $J$  = 8.0 Hz, 1.0 Hz, 1H), 7.48 (t,  $J$  = 1.5 Hz, 1H), 7.67 (dd,  $J$  = 8.0 Hz, 1.0 Hz, 2H).  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 125 MHz)  $\delta$  42.7, 56.7, 63.2, 69.3, 113.5, 120.3, 123.2, 126.4, 126.9, 128.7, 129.0, 129.6, 130.8, 131.0, 131.3, 131.4, 140.1, 144.2, 149.3, 153.8, 168.5; IR (neat,  $\text{cm}^{-1}$ ): 3058, 2989, 2923, 1776, 1704, 1598, 1503, 1387; HRMS Exact mass calcd. for  $\text{C}_{25}\text{H}_{21}\text{BrN}_3\text{ONa}^+$  512.0580 found 512.0586.

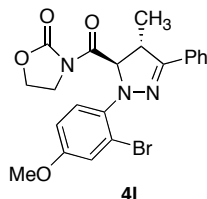


**(4*S*,5*R*)-1,3-Bis-(4-bromo-phenyl)-4,5-dihydro-4-methyl-5-**

**(oxazolidin-2-one-3-carbonyl)-1*H*-pyrazole (4k):** Isolated as a yellow solid, m.p. = 215-217 °C.  $[\alpha]_{\text{D}}^{25} = +202.9$  ( $c$  0.62,  $\text{CHCl}_3$ ),

95% ee;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 500 MHz)  $\delta$  1.50 (d,  $J$  = 7.5 Hz, 3H),

3.58 (dq,  $J$  = 2.0 Hz, 7.5 Hz, 1H), 3.95-4.07 (m, 2H), 4.51-4.55 (m, 2H), 5.69 (d,  $J$  = 2.0 Hz, 1H), 6.50 (d,  $J$  = 9.0 Hz, 2H), 7.37 (d,  $J$  = 9.0 Hz, 2H), 7.51 (d,  $J$  = 9.0 Hz, 2H), 7.60 (d,  $J$  = 9.0 Hz, 2H).  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 125 MHz)  $\delta$  18.1, 42.6, 46.5, 63.3, 68.4, 112.1, 114.8, 123.1, 127.7, 130.1, 132.0, 132.3, 143.5, 151.1, 154.1, 168.9; IR (neat,  $\text{cm}^{-1}$ ): 3056, 2977, 2925, 1771, 1704, 1594, 1788; HRMS Exact mass calcd. for  $\text{C}_{20}\text{H}_{17}\text{Br}_2\text{N}_3\text{O}_3\text{Na}^+$  527.9529 found 527.9525.



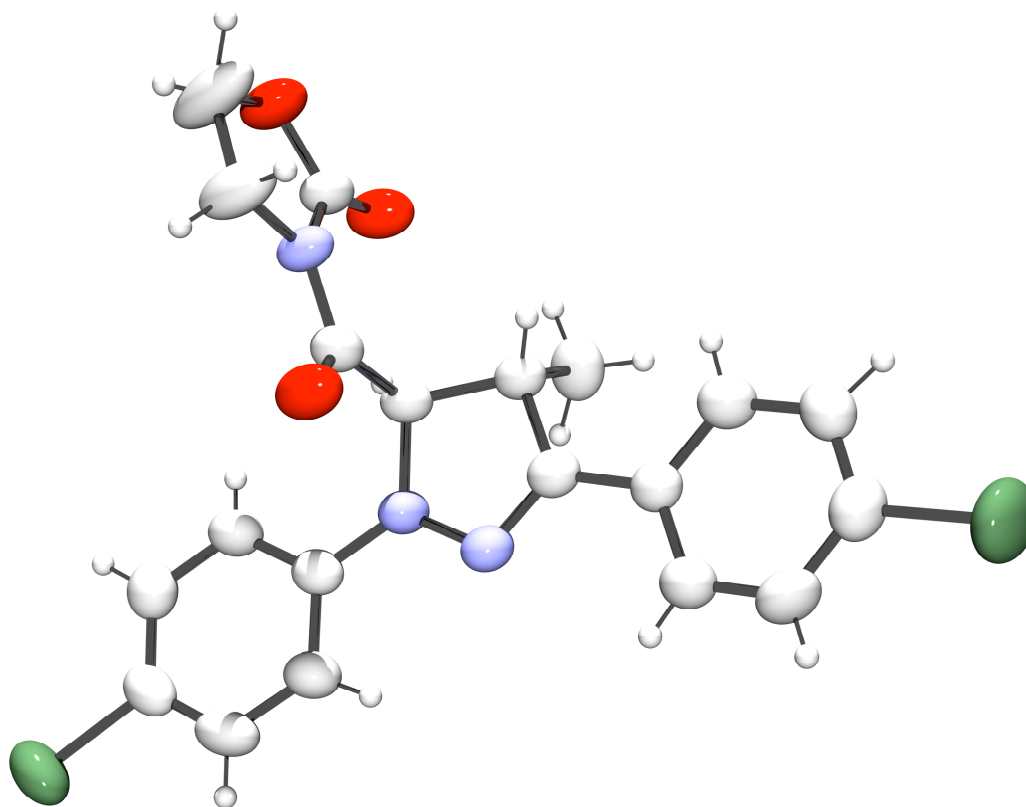
**(4*S*,5*R*)-1-(2-Bromo-4-methoxy-phenyl)-4,5-dihydro-4-methyl-5-(oxazolidin-2-one-3-carbonyl)-3-phenyl-1*H*-pyrazole (4l):** Isolated

as a off-white foam, m.p. = 74-76 °C.  $[\alpha]_{\text{D}}^{25} = +7.34$  ( $c$  0.29,  $\text{CHCl}_3$ ),

96% ee;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 500 MHz)  $\delta$  1.57 (d,  $J$  = 7.0 Hz, 3H), 3.66 (dq,  $J$  = 1.5 Hz, 7.0 Hz, 1H), 3.79 (s, 3H), 3.90 (t,  $J$  = 8.0 Hz, 2H), 4.39-4.47 (m, 2H), 6.13 (d,  $J$  = 1.5 Hz, 1H), 6.87 (m, 2H), 7.33 (t,  $J$  = 7.5 Hz, 1H), 7.39 (t,  $J$  = 7.5 Hz, 2H), 7.72-7.76 (m, 3H).  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 500 MHz)  $\delta$  17.4, 42.6, 47.4, 55.9, 63.0, 71.4, 113.7, 114.9, 124.5, 126.1, 126.3, 128.8, 129.0, 131.4, 136.6, 152.4, 153.0, 156.4, 170.0; IR (neat,  $\text{cm}^{-1}$ ): 3058, 2975, 2838, 1771, 1706, 1605, 1493, 1387; HRMS Exact mass calcd. for  $\text{C}_{21}\text{H}_{20}\text{BrN}_3\text{O}_4\text{Na}^+$  480.0529 found 480.0527.

### Crystal Structure for Intermediate 4k

-(Crystal data and structure refinement for 4k can be found in the attached CIF file)

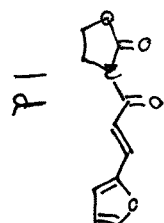
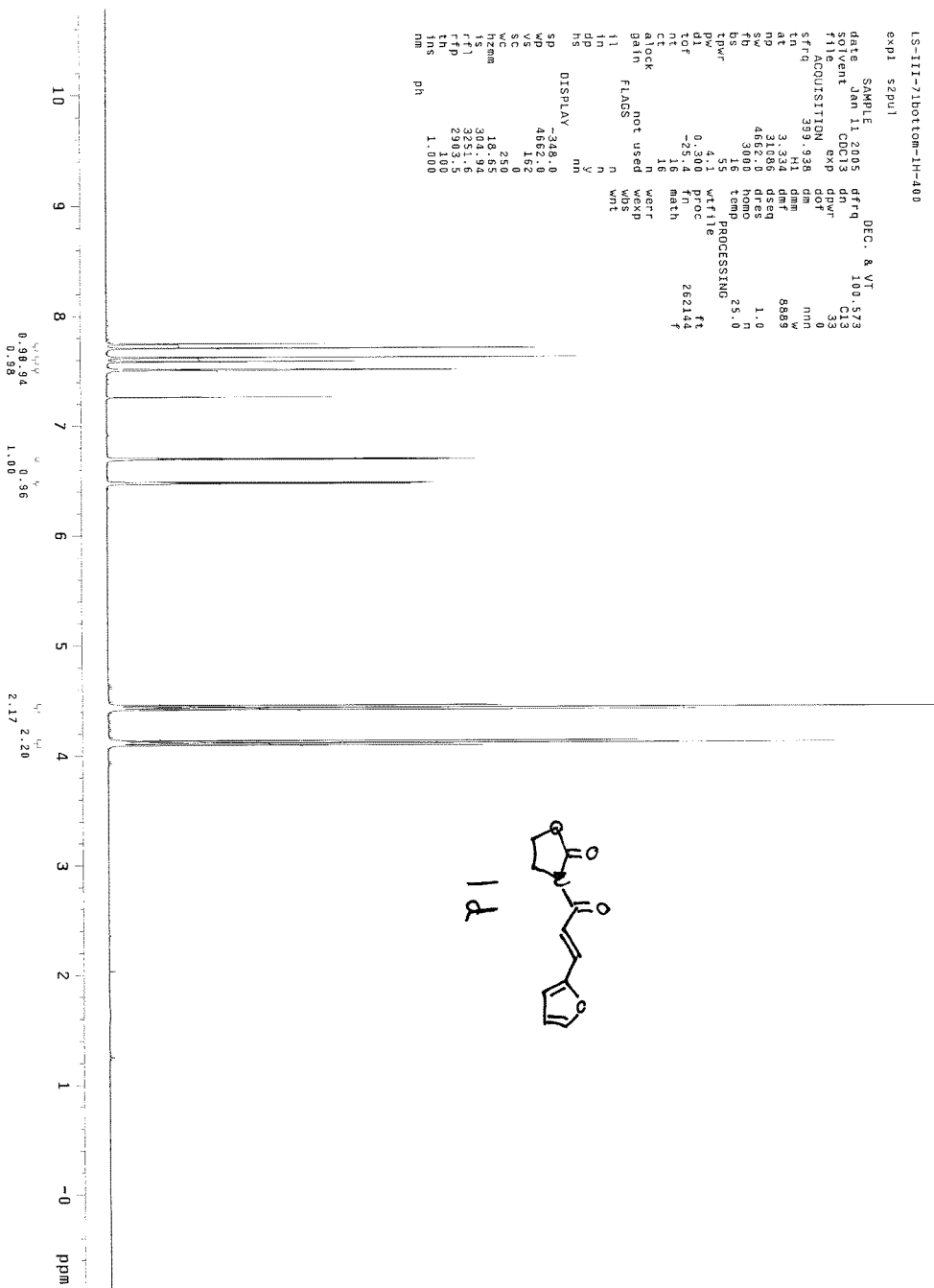


### References:

- 1) Kanemasa, S.; Kanai, T. *J. Am. Chem. Soc.* **2000**, *122*, 10710-10711.
- 2) Cai, C.; Soloshonok, V. A.; Hruby, V. J. *J. Org. Chem.* **2001**, *66*, 1339-1350.
- 3) Sibi, M. P.; Chen, J. *J. Am. Chem. Soc.* **2000**, *123*, 9472-9473.

- 4) Sibi, M. P.; Zimmerman, J.; Rheault, T. *Angew. Chem., Int. Ed.* **2003**, *42*, 4521-4523.
- 5) Sibi, M. P.; Hasegawa, H. *Org. Lett.* **2002**, *4*, 3347-3349.
- 6) Narasaka, K.; Iwasawa, N.; Inoue, M.; Yamada, T.; Nakashima, M.; Sugimori, J. *J. Am. Chem. Soc.* **1989**, *111*, 5340-5345.
- 7) Patel, H. V.; Vyas, K. A.; Pandey, S. P.; Fernandes, P. S. *Tetrahedron* **1996**, *52*, 661-668.

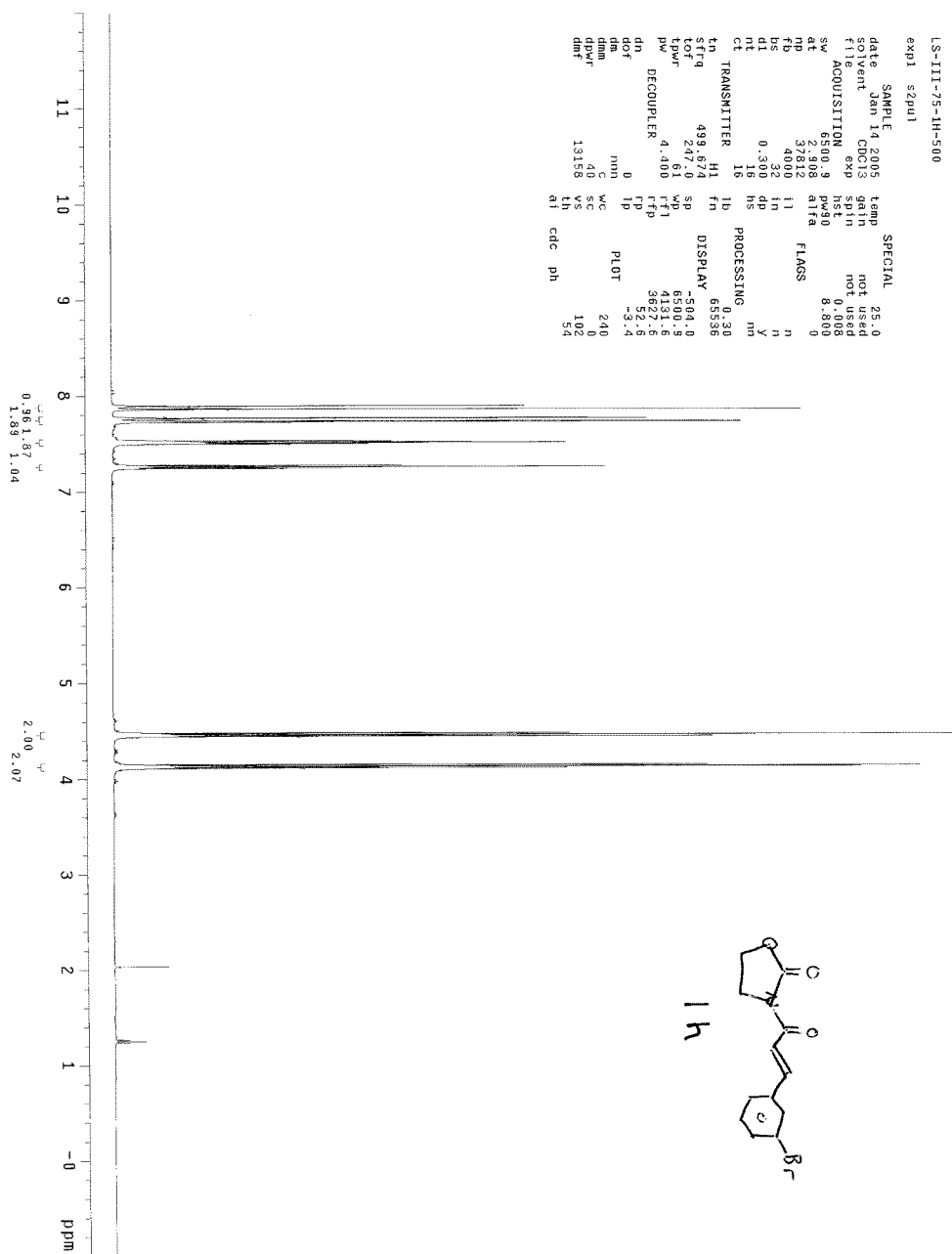
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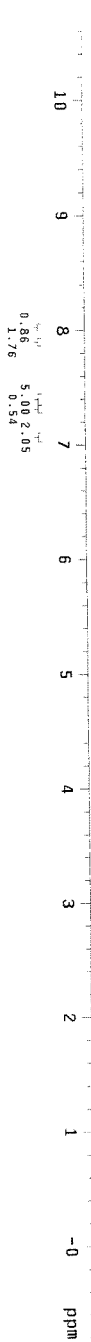
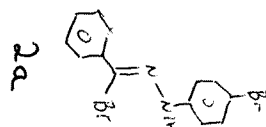
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LS-N-bromophenylhydrazonylbromide

expt1 s2pui

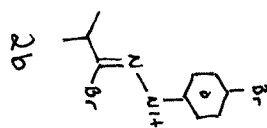
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LS-II-291-1-H-500

expi s2pu1

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sfreq	499.674	sp	65536
tofr	247.0	sp	DISPLAY -507.6
tpwr	61	wp	6500.9
pw	4.400	rfp	507.0
DECOUPLER	0	fp	58.4
dof	0	tp	-7.6
dm	nm	WC	PLOT
dmr	40	sc	240
dwr	13158	vs	0
dnt	13158	th	33
		at	19
		cd	ph

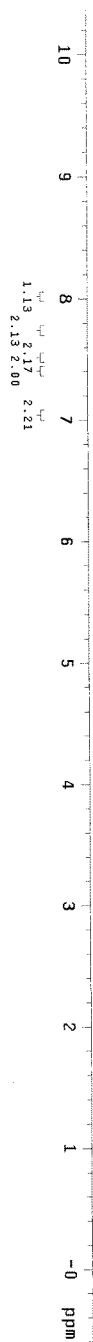
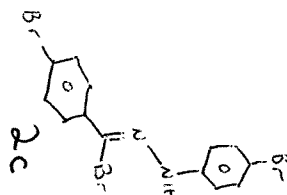


STANDARD 1H OBSERVE

```

exp1 std1h
SAMPLE
date Feb 1 2005 dfrq DEC. 8 VI
solvent cdcl3 dn H1
file COUSSION exp dnm H1
sfrq 300.139 dm H1
at 1.997 dmf 7400
nd 33184 1b PROCESSING 0.10
fb 1800 wtfle
bs 16 proc
tpwr 758 fn not used
pw 1.000 weff
d1 0 wepd
tof 16 wbs
ct 16 wnt
back not used
Bath FLAG
11 n
1n n
op DISPLAY y
sp -188.5
wp 3301.4
vs 13.1
wc 250
h2mm 13.21
1s1 5.12.73
f1p 2374.3
th 2.000
ins nm cdc ph

```

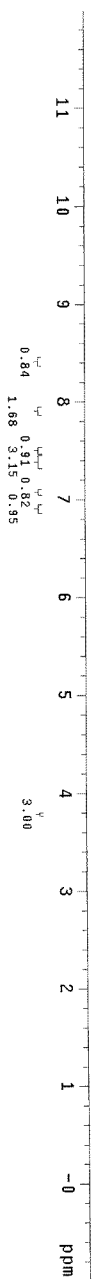
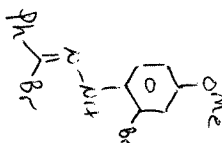




LS-II-246-1H-500

exp1 s2pu1

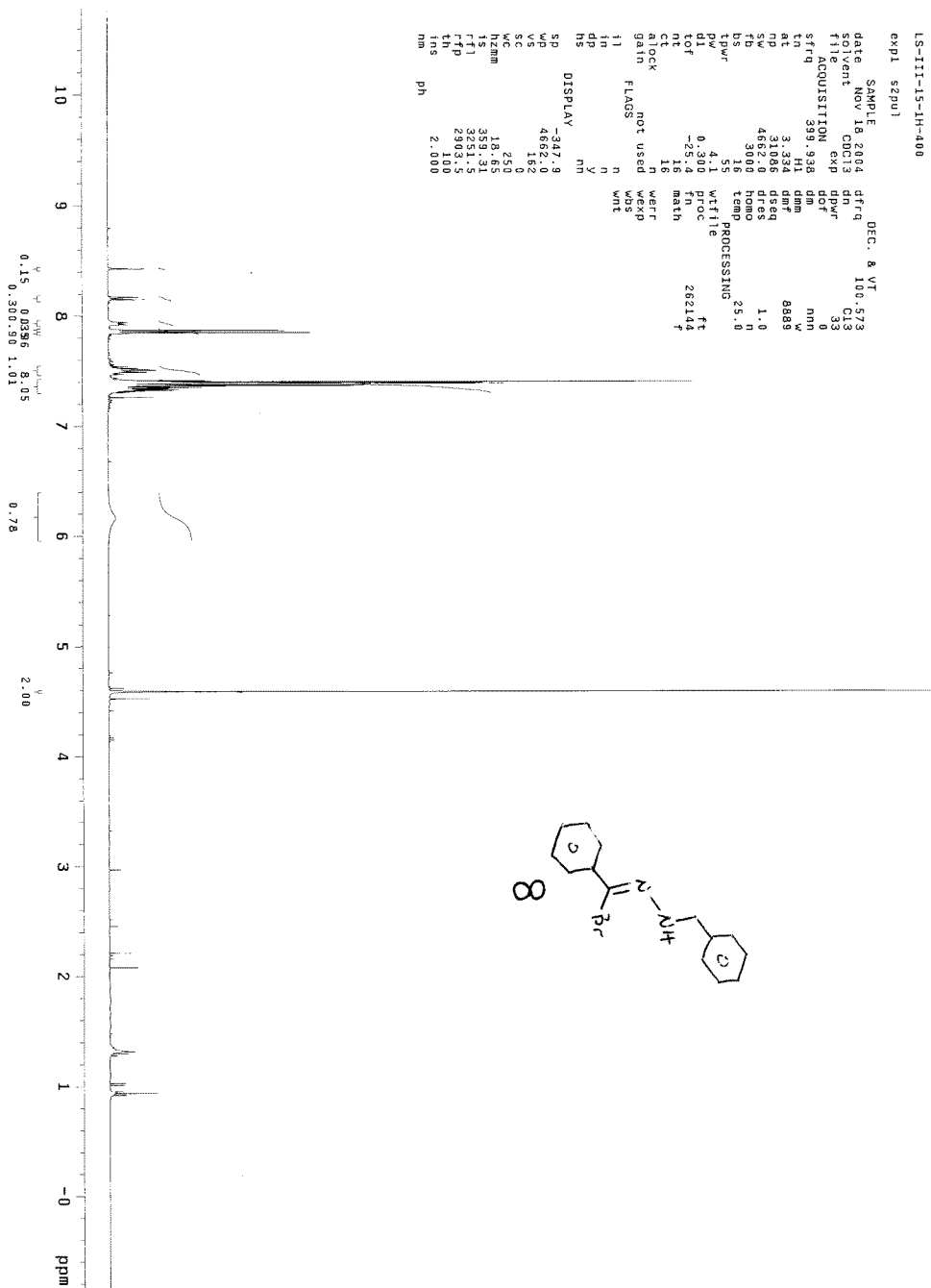
SAMPLE SPECIAL  
 date Nov 2004 temp 25.0  
 solvent CDCl3 gain not used  
 file CDCl3 not used  
 ACQUISITION exp 0.800  
 at 2.908 0.800  
 nt 3.7812 0.800  
 np 4000 11  
 fb 4000 11  
 ds 0.32 11  
 nt 0.32 11  
 ct 16 16  
 TRANSMITTER H1  
 16 16  
 SFR 499.674 fm  
 tof 247.0 sp  
 tpr 61 wp  
 pw 4.400 fpi  
 DECOUPLER 0 fpi  
 dof 0 tp  
 dm 0 wc  
 dm 4 wc  
 dm 4 wc  
 dm 13158 vs  
 dm 13158 th  
 al cdc ph



exp1 szput

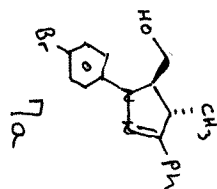
```

date      SAMPLE  DEC. 8  VT
Nov 16 2004  100.5729388
solvent    CHCl3  dn
f1) ACQUISITION  exp  dof
sfrq       399.938  dm
t1         3.81  dnm
n1         3.81  dnm
n2         3.81  dnm
n3         3.81  dnm
n4         3.81  dnm
n5         3.81  dnm
n6         3.81  dnm
n7         3.81  dnm
n8         3.81  dnm
n9         3.81  dnm
n10        3.81  dnm
n11        3.81  dnm
n12        3.81  dnm
n13        3.81  dnm
n14        3.81  dnm
n15        3.81  dnm
n16        3.81  dnm
n17        3.81  dnm
n18        3.81  dnm
n19        3.81  dnm
n20        3.81  dnm
n21        3.81  dnm
n22        3.81  dnm
n23        3.81  dnm
n24        3.81  dnm
n25        3.81  dnm
n26        3.81  dnm
n27        3.81  dnm
n28        3.81  dnm
n29        3.81  dnm
n30        3.81  dnm
n31        3.81  dnm
n32        3.81  dnm
n33        3.81  dnm
n34        3.81  dnm
n35        3.81  dnm
n36        3.81  dnm
n37        3.81  dnm
n38        3.81  dnm
n39        3.81  dnm
n40        3.81  dnm
n41        3.81  dnm
n42        3.81  dnm
n43        3.81  dnm
n44        3.81  dnm
n45        3.81  dnm
n46        3.81  dnm
n47        3.81  dnm
n48        3.81  dnm
n49        3.81  dnm
n50        3.81  dnm
n51        3.81  dnm
n52        3.81  dnm
n53        3.81  dnm
n54        3.81  dnm
n55        3.81  dnm
n56        3.81  dnm
n57        3.81  dnm
n58        3.81  dnm
n59        3.81  dnm
n60        3.81  dnm
n61        3.81  dnm
n62        3.81  dnm
n63        3.81  dnm
n64        3.81  dnm
n65        3.81  dnm
n66        3.81  dnm
n67        3.81  dnm
n68        3.81  dnm
n69        3.81  dnm
n70        3.81  dnm
n71        3.81  dnm
n72        3.81  dnm
n73        3.81  dnm
n74        3.81  dnm
n75        3.81  dnm
n76        3.81  dnm
n77        3.81  dnm
n78        3.81  dnm
n79        3.81  dnm
n80        3.81  dnm
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n82        3.81  dnm
n83        3.81  dnm
n84        3.81  dnm
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n87        3.81  dnm
n88        3.81  dnm
n89        3.81  dnm
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n92        3.81  dnm
n93        3.81  dnm
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n100       3.81  dnm
n101       3.81  dnm
n102       3.81  dnm
n103       3.81  dnm
n104       3.81  dnm
n105       3.81  dnm
n106       3.81  dnm
n107       3.81  dnm
n108       3.81  dnm
n109       3.81  dnm
n110       3.81  dnm
n111       3.81  dnm
n112       3.81  dnm
n113       3.81  dnm
n114       3.81  dnm
n115       3.81  dnm
n116       3.81  dnm
n117       3.81  dnm
n118       3.81  dnm
n119       3.81  dnm
n120       3.81  dnm
n121       3.81  dnm
n122       3.81  dnm
n123       3.81  dnm
n124       3.81  dnm
n125       3.81  dnm
n126       3.81  dnm
n127       3.81  dnm
n128       3.81  dnm
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n131       3.81  dnm
n132       3.81  dnm
n133       3.81  dnm
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n136       3.81  dnm
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n149       3.81  dnm
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n151       3.81  dnm
n152       3.81  dnm
n153       3.81  dnm
n154       3.81  dnm
n155       3.81  dnm
n156       3.81  dnm
n157       3.81  dnm
n158       3.81  dnm
n159       3.81  dnm
n160       3.81  dnm
n161       3.81  dnm
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n164       3.81  dnm
n165       3.81  dnm
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n168       3.81  dnm
n169       3.81  dnm
n170       3.81  dnm
n171       3.81  dnm
n172       3.81  dnm
n173       3.81  dnm
n174       3.81  dnm
n175       3.81  dnm
n176       3.81  dnm
n177       3.81  dnm
n178       3.81  dnm
n179       3.81  dnm
n180       3.81  dnm
n181       3.81  dnm
n182       3.81  dnm
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n185       3.81  dnm
n186       3.81  dnm
n187       3.81  dnm
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n189       3.81  dnm
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n195       3.81  dnm
n196       3.81  dnm
n197       3.81  dnm
n198       3.81  dnm
n199       3.81  dnm
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n201       3.81  dnm
n202       3.81  dnm
n203       3.81  dnm
n204       3.81  dnm
n205       3.81  dnm
n206       3.81  dnm
n207       3.81  dnm
n208       3.81  dnm
n209       3.81  dnm
n210       3.81  dnm
n211       3.81  dnm
n212       3.81  dnm
n213       3.81  dnm
n214       3.81  dnm
n215       3.81  dnm
n216       3.81  dnm
n217       3.81  dnm
n218       3.81  dnm
n219       3.81  dnm
n220       3.81  dnm
n221       3.81  dnm
n222       3.81  dnm
n223       3.81  dnm
n224       3.81  dnm
n225       3.81  dnm
n226       3.81  dnm
n227       3.81  dnm
n228       3.81  dnm
n229       3.81  dnm
n230       3.81  dnm
n231       3.81  dnm
n232       3.81  dnm
n233       3.81  dnm
n234       3.81  dnm
n235       3.81  dnm
n236       3.81  dnm
n237       3.81  dnm
n238       3.81  dnm
n239       3.81  dnm
n240       3.81  dnm
n241       3.81  dnm
n242       3.81  dnm
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n245       3.81  dnm
n246       3.81  dnm
n247       3.81  dnm
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n252       3.81  dnm
n253       3.81  dnm
n254       3.81  dnm
n255       3.81  dnm
n256       3.81  dnm
n257       3.81  dnm
n258       3.81  dnm
n259       3.81  dnm
n260       3.81  dnm
n261       3.81  dnm
n262       3.81  dnm
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n264       3.81  dnm
n265       3.81  dnm
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n268       3.81  dnm
n269       3.81  dnm
n270       3.81  dnm
n271       3.81  dnm
n272       3.81  dnm
n273       3.81  dnm
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n275       3.81  dnm
n276       3.81  dnm
n277       3.81  dnm
n278       3.81  dnm
n279       3.81  dnm
n280       3.81  dnm
n281       3.81  dnm
n282       3.81  dnm
n283       3.81  dnm
n284       3.81  dnm
n285       3.81  dnm
n286       3.81  dnm
n287       3.81  dnm
n288       3.81  dnm
n289       3.81  dnm
n290       3.81  dnm
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n292       3.81  dnm
n293       3.81  dnm
n294       3.81  dnm
n295
```



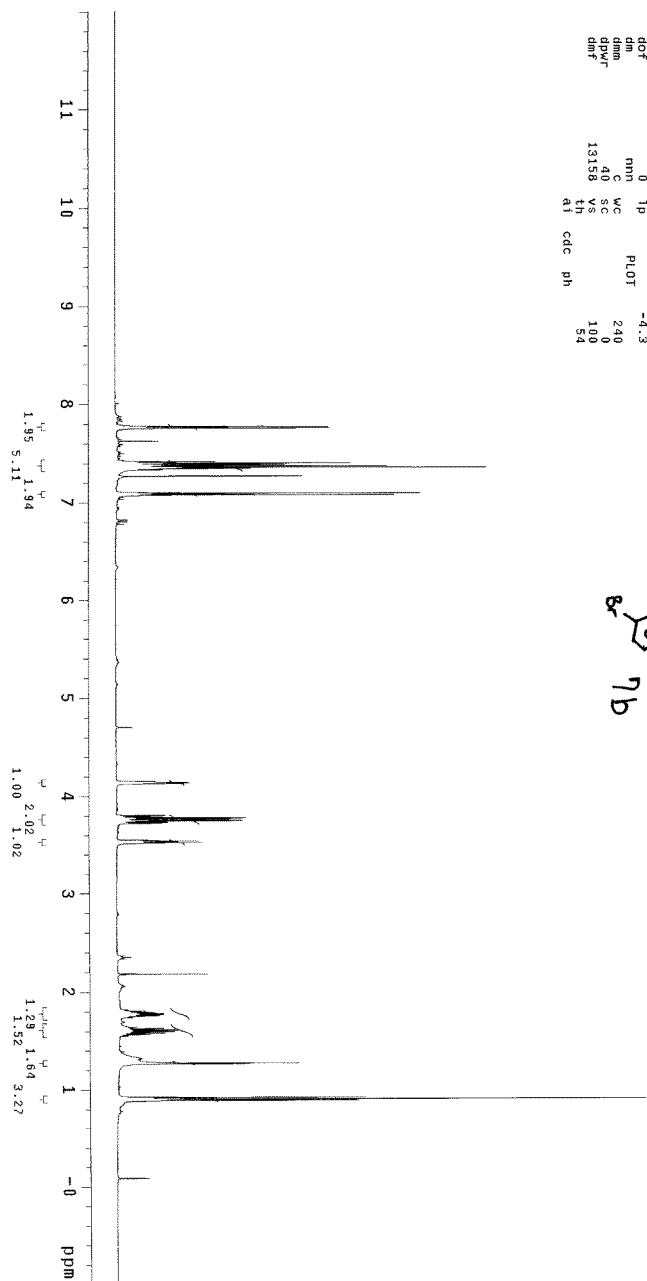
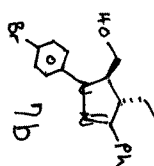
LS-III-264C  
exp2 s2put1

SAMPLE		SPECIAL	
date	Apr 21 2005	temp	25.0
solvent	CDCl3	gain	not used
file	ACQUISITION	exp	not used
sw	6500.9	pw90	8.800
at	2.908	atfa	0
np	37812	11	0
nt	16	hs	0
ct	TRANSMITTER	16	hs
to	499.674	4n	65338
tof	247.0	sp	-500.6
tpwr	61	wp	6500.9
pw	4.400	rfp	3632.6
decoupler	4.400	rfp	3632.6
dnf	0	lp	-5.8
dm	nm	mc	240
dmw	40	sc	0
dmw	13158	vs	60
dmf	41	cdc	54
	ph		



exp2 s2put

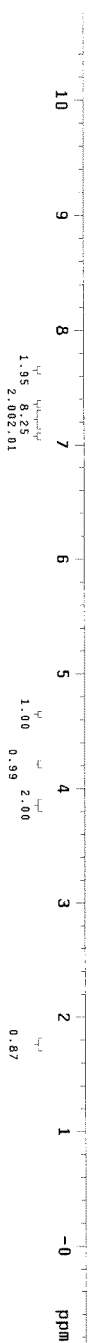
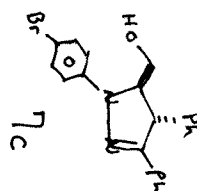
SAMPLE										SPECIAL	
date	Apr 21, 2005	temp	25.0								
solvent	CDCl <sub>3</sub>	gain	not used								
f1	ACQUISITION	exp	hst	0.008							
		pw	8.800								
sw	6500.9	pw40	0								
at	2.300										
ft	32000										
fb	3000										
td	1	n	n								
ct	1	n	n								
	0.300	dp	dp								
	16		y								
PROCESSING											
ct	16	fb	0.30								
TRANSMITTER											
tn	11	fb	65.56								
freq	488.58	SP	DISPLAY	500.6							
tpwr	2.07	wd		500.9							
tpwr	61	wp		4132.4							
pw	4.400	fft		583.1							
DECOUPLER											
dn	1p	fb	-4.3								
dof	nm										
dm	nm										
dmr	4.0	SC	2.00								
dmr	4.90	SC	0								
dmr	13158	VS	100.00								
ti	cdc	ph	54								



LS-II-272A-1H-400

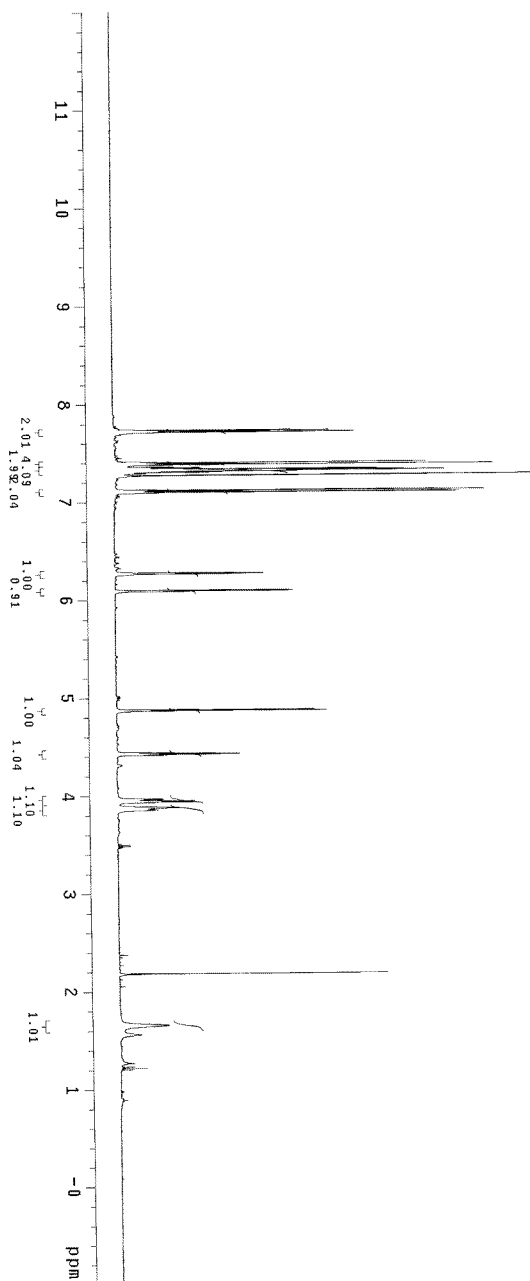
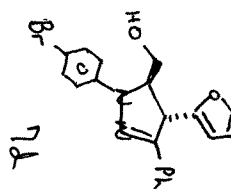
exp1 szpu1

SAMPLE Nov 22 2004 dfrc DEC. & VI  
 solvent CDC13 dn C13  
 file ACQUISITION exp  
 stfrq 399.938 dm  
 tn H1 dmm  
 at 3.334 dmf  
 nd 32.086 dsrq 8889  
 sb 48.454 dsrq 1.0  
 fb 3600 homo  
 bs 16 temp 25.0  
 tpwr 55 wtf file  
 pw 4.1 proc  
 sh 0.300 fnc 262144  
 tof -25.4 fn math f  
 nt 16  
 ct 16  
 atock not used  
 gain flags n wgt  
 i1 n  
 in n  
 sp n  
 ps n  
 DISPLAY nm  
 sp -356.5  
 wp 4662.0  
 v 155  
 sc 0  
 wc 250  
 hznm 18.65  
 f31 325.09  
 f31 325.09  
 rfd 0  
 th 25  
 ins 1.000  
 nm ph



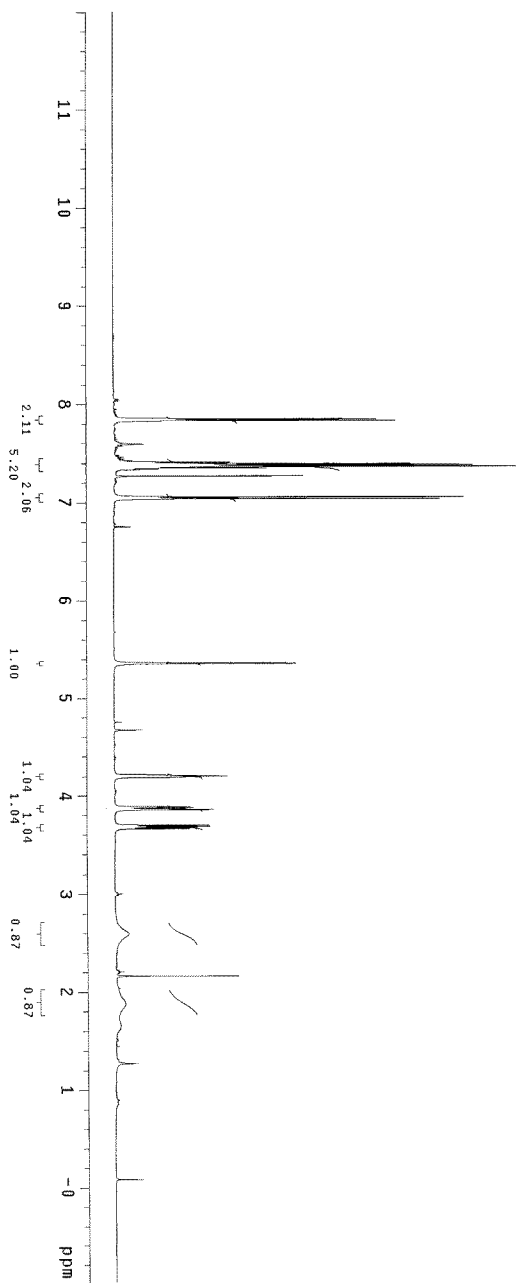
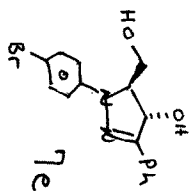
exp2 s2put

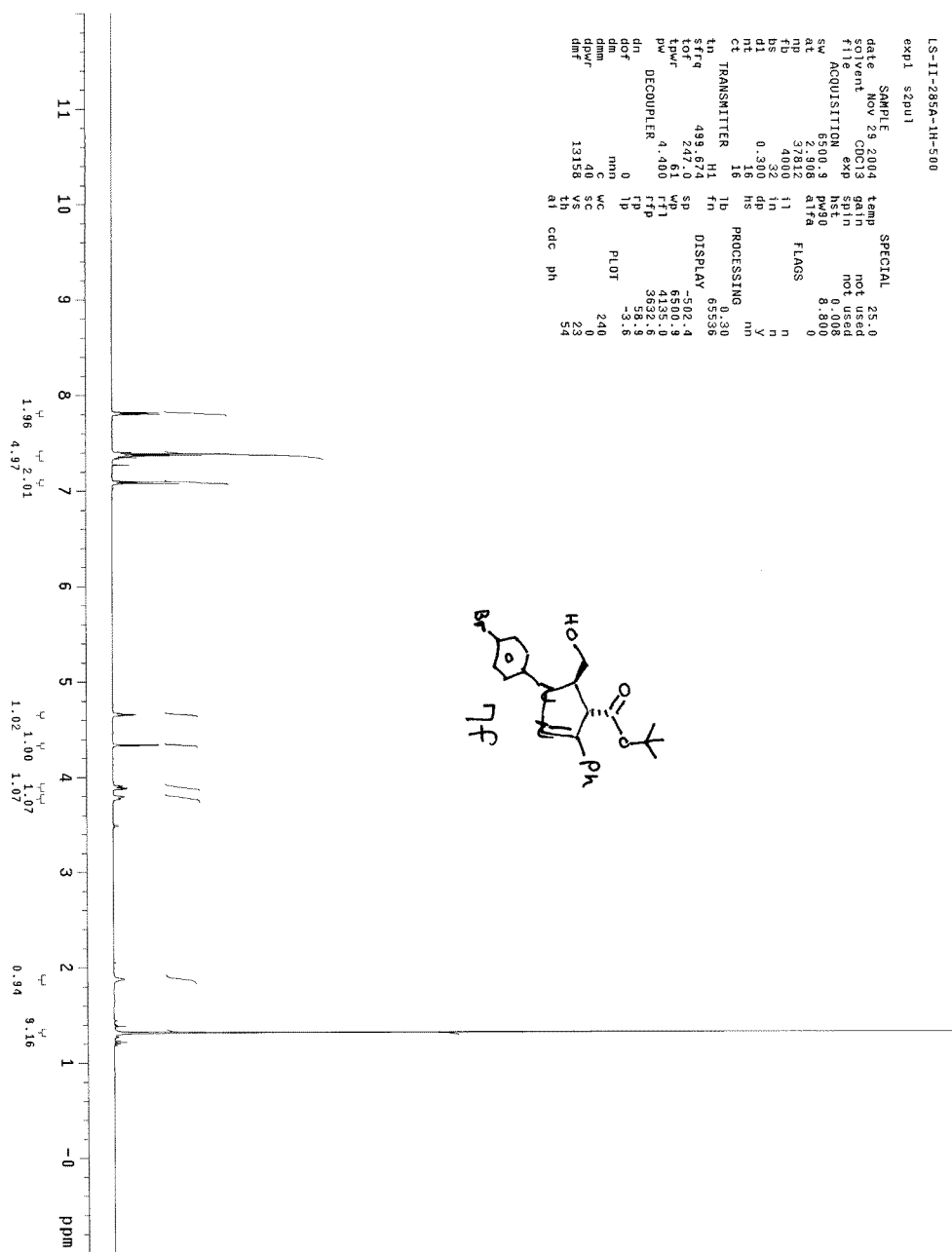
date	SAMPLE	Apr 21 2005	temp	25.0
solvent	CDC13	gain	not used	
f1	ACQUISITION	exp	0.008	
sw	6500.3	pw300	8.800	
at	2.3005		0	
td	3.0000			
fb	3.0000			
cl	0.300	dp		
ci	16			
PROCESSING				
ln	11	lb	0.30	
tp	489.58			
tpwr	247.0	display	85536	
pw	61	sp	501.0	
DECOUPLER	4.400	wd	6500.9	
		fft	4132.8	
		fft	97.3	
			-7.4	
dof	nm	Plot	2.0	
dms	nm			
dim	40	SC	60	
dmf	13158	VS	20	
		th	54	
		cdc	ph	



LS-III-297C  
exp2 s2put

SAMPLE		SPECIAL	
date	Apr 21 2005	temp	25.0
solvent	CDCl3	gain	not used
file	ACQUISITION	exp	not used
sw	65008.9	pw90	8.800
at	2.908	alfa	0
np	37812	11	0
nt	16	hs	nm
ct	TRANSMITTER	16	hs
tn	499.674	fn	65538
stf	247.0	sp	-501.0
tpw	61	wp	6500.8
pw	4.400	rfp	3632.6
decoupler	0	tp	-1.3
dmf	13158	vs	125
at	cdc	ph	54



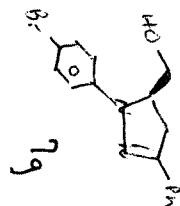




LS-III-284B-1H-500

exp2 sz2pu1

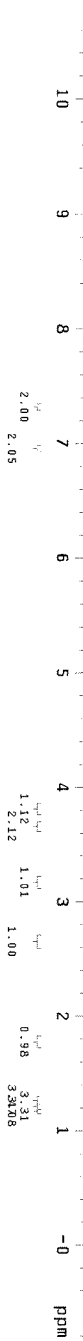
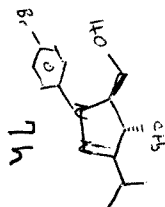
SAMPLE		SPECIAL	
date	Feb 4 2005	temp	25.0
solvent	CDCl3	gain	not used
file	exp	spin	not used
ACQUISITION		ns	8.800
sw	500.9	atfa	0
at	2.308	flaos	n
fb	37812	flaos	n
bs	4000	flaos	n
nt	0.302	flaos	y
nt	16	flaos	hs
ct	16	flaos	hs
TRANSMITTER		PROCESSING	
h1	h1	h1	0.30
sfreq	499.674	h1	6536
tof	247.0	sp	-507.2
tdwr	61	wp	6500.2
pw	4.400	ft1	507.2
DECOUPLER		ft2	59.6
do	0	tp	-1.7
dof	0	plot	240
dm	mm	vc	240
dmr	c	sc	0
dmr	40	vs	233
dmr	13158	th	43
dmr	th	at	cdc ph



LS-IT-293C-1H-400

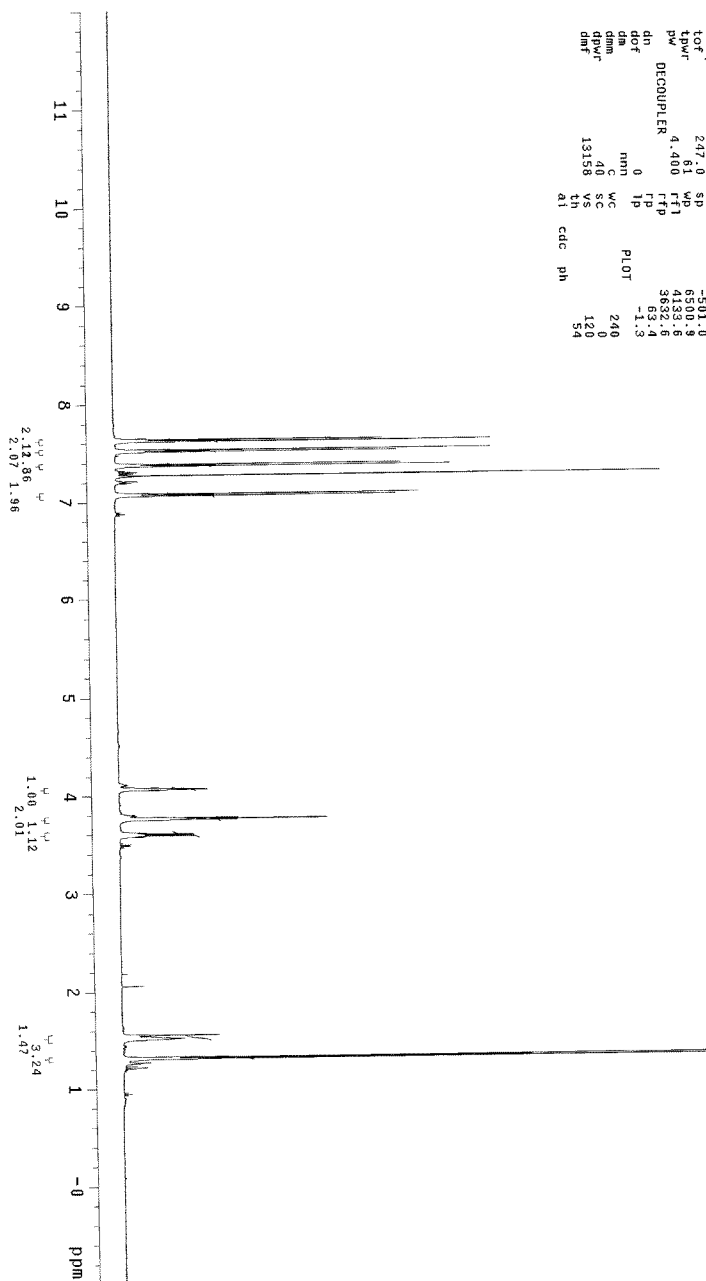
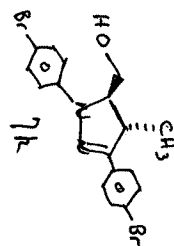
exp1 52pu1

SAMPLE DEC. & VT  
date Jan 31, 2005 dfpq 100.573  
solvent CDCl3 dn C13  
f1 ACQUISITION exp 30  
f2 30  
sfrq 399.338 dm  
tn H1 dm  
at 3.334 dm 8889  
n1 3.334 dm  
sw 4662.0 dres 1.0  
fb 3000 homo  
ds 16 temp 25.0  
tdw 15 PROCESSING  
n1 4.1 wfile  
d1 0.360 proc ft  
tof -25.4 tn 262144  
nt 16 math  
st 16  
a1 lock not used  
gain not used  
11 FLAOS n  
12 n  
13 n  
14 n  
15 n  
16 n  
17 n  
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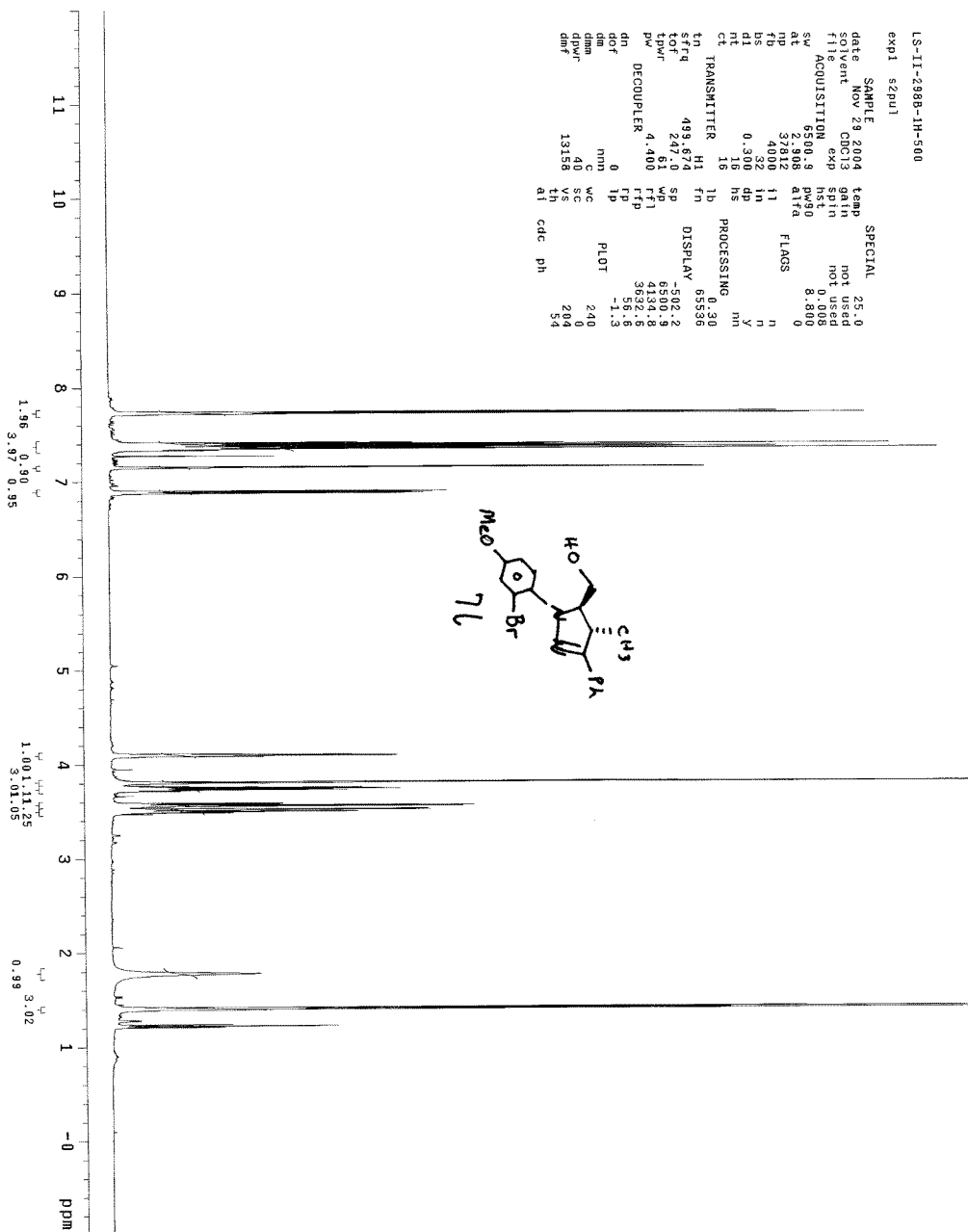
exp2 s2pu1

date	SAMPLE	temp	25.0
Apr 21, 2005	CDC13	gain	not used
solvent	CDC13	mod	used
file	ACQUISITION	exp	0.008
sw	6500.3	pw	8.600
at	4.208	dt	0
rb	3000.0	flags	
fb	4000		
bs	0.300	in	n
nt	0.300	nd	n
ct	15	yp	y
tr	15		
cn	TRANSMITTER	PROCESSING	0.550
in	439.41	lb	6.386
ft	439.41	DISPLAY	6.386
strg	2.470	sp	-501.0
tdwv	61	wd	6500.3
pw	4.400	fft	4132.6
DECOUPLER		fft	5832.4
do-f	nm	Plot	-1.3
dim	0		240
dim	4	SC	0
dim	4	VS	120
diff	13158	at	54
		ct	
		cdc	ph



exp1 szput

date	SAMPLE	Nov 29 2004	temp	SPECIAL	25.0
solvent	CDCI3	gain	not used		
f1	ACQUISITION	exp	hist	0.068	
at	6500.8	pw	8.800		
sw	2.3065	alwa	0		
ft	30000				
fb	3000				
bs	32				
ci	0.300	dp			
ct	15				
cs	15				
TRANSMITTER	1b				
tn	409.8	H1			
sfreq	2.9974	SP	DISPLAY	-502.2	
tpwr	2.971	WD		6500.9	
tpv	61			4133.8	
DECOUPLER	4.400	FTF		3656.8	
dn					
dof	0				
dmn	mmh				
dinv	C	WC		2400	
dmv	13188	W		2040	
at	cdc	ph		54	

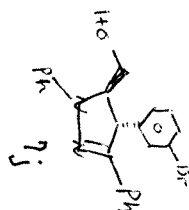


LS-II-820-1H-400  
 exp1 s2pu1  
 SAMPLE DEC. & VT  
 date Jan 31 2005 dfreq 100.573  
 solvent CDCl3 dn C13  
 f1 ACQUISITION exp dm 30  
 tn 399.938 dm nm  
 at 3.334 dm w  
 f2 3.134 dm 8889  
 f3 4662.0 dres 1.0  
 f4 3000 homo 25.0  
 bs 16 temp  
 tpmr 55 wffile  
 d1 0.300 proc ft  
 tof -25.4 fn 262144  
 nt 16 math f  
 atlock 16 weff  
 gain not used wexp  
 flags not used wbs  
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LS-III-868-1H-500

exp2 s2put

SAMPLE		SPECIAL	
date	Jan 31 2005	tamp	25.0
solvent	CDCl3	gain	not used
file	exp	spin	not used
ACQUISITION	exp	not used	not used
sw	6500.9	pw	8.800
at	2.808	atfa	0
np	37812	flacs	n
fb	4000	fl	n
ds	52.170	fl	n
d1	0.300	dp	y
nt	16	hs	nn
ct	16	PROCESSING	6536
td	h1	fn	6536
stfq	499.674	sp	-597.0
tof	247.0	wp	620.9
tdwr	61	wp	597.0
pw	4.400	ffp	62.7
DECOUPLER	0	tp	-11.6
dn	mn	PLOT	240
dm	mn	vc	0
dmu	40	sc	0
tdwr	40	vs	341
dmf	13158	th	54
		ai	cuc ph

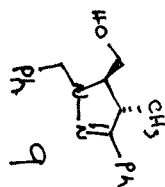


LS-II-45-1H-400

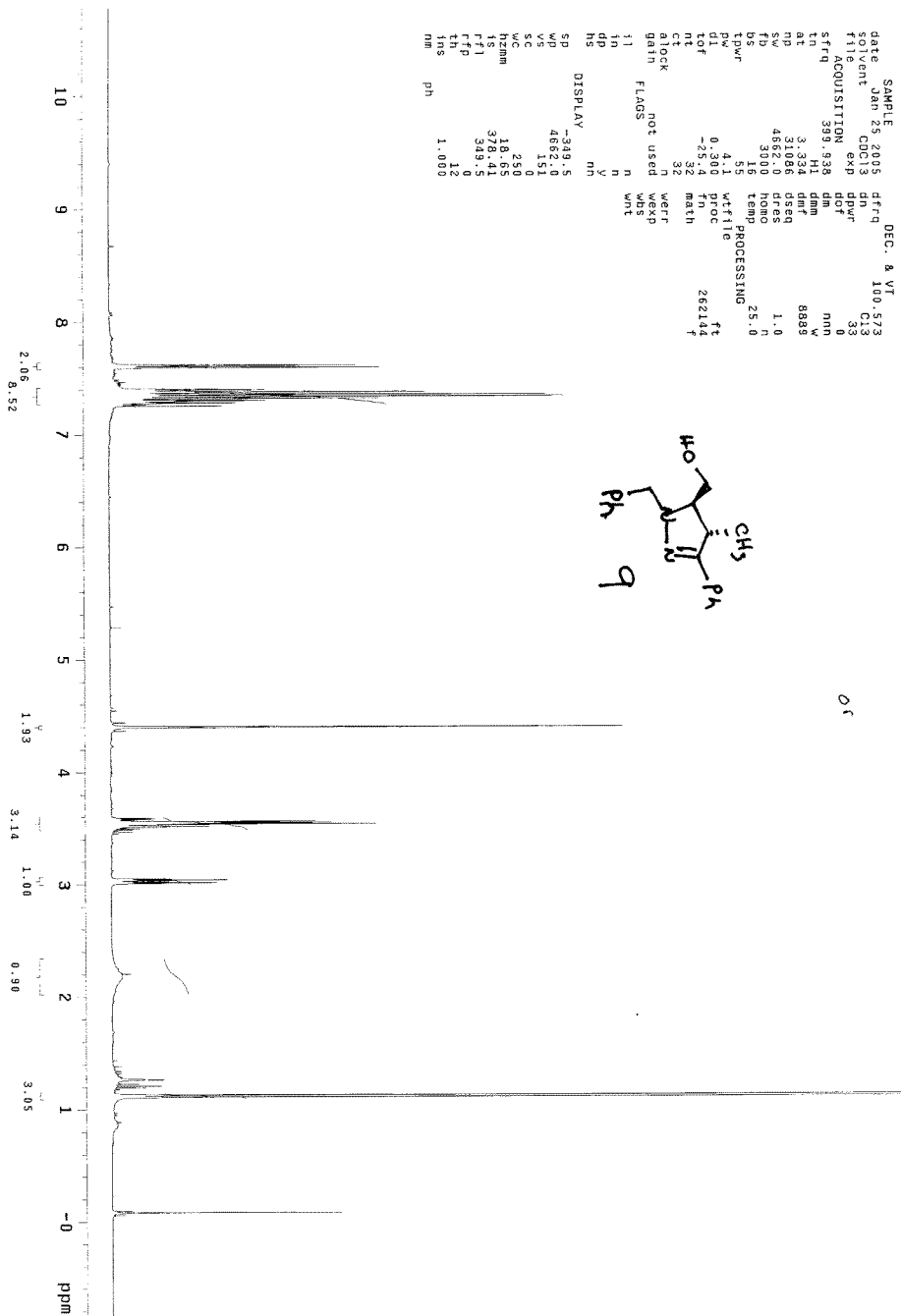
exp1 s2pu1

SAMPLE DEC. & VI  
date Jan 25 2005 dfreq 100.573  
solvent CDCl3 dn C13  
f1h1e 0.000 dmf 3.0  
ACQUISITION exp 389.938 dm nmh  
stfrq 389.938 dm nmh  
at 3.334 dmf 8885  
pd 46526 dfeq 1.0  
fb 3000 homo n  
bs 16 temp PROCESSING 25.0  
tpwr 55 wt f16  
pw 4.300 pproc 262144  
si 0.300 fno  
tof -25.4 math f  
nt 32 math f  
ct 32 weff  
clock not used wbs  
gain not used wbs  
11 n  
1n n  
dn n  
hs n  
hs n

DISPLAY  
sp -349.5  
wp 4682.1  
wc 1.0  
sc 0  
hzm 250  
hzm 318.65  
f1 349.5  
f2 0  
th 1.000  
nm



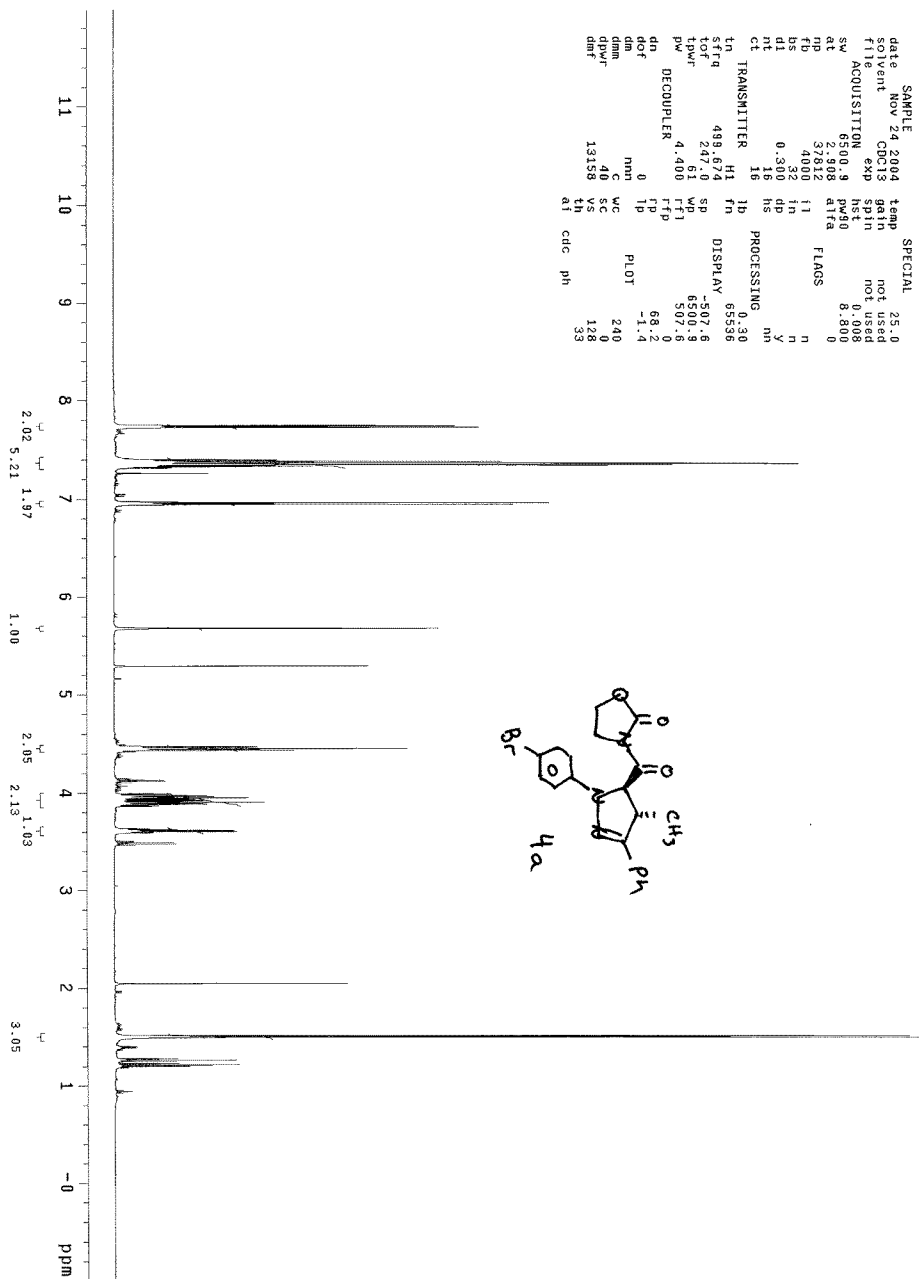
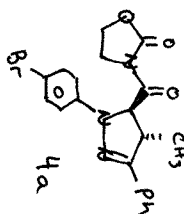
0.1



LS-III-10A-1H-500

expt szpul

SAMPLE		SPECIAL	
date	Nov 24 2004	temp	25.0
solvent	CDCl3	gain	not used
flavor	exp	spin	not used
ACQUISITION	exp	not used	not used
sw	6500.9	pw90	8.800
at	2.908	atfa	0
np	37812	11	n
bs	4032	in	n
dl	0.300	dp	y
nt	16	hs	nm
ct	TRANSMITTER	16	1b
to	499.674	hi	fn
stfq	247.0	sp	65536
pw	61	wp	-507.6
pw	4.400	rff	6507.8
DECOUPLER	0	fp	507.0
do	0	1p	68.2
dm	nm	mc	-1.4
dm	40	sc	240
dpwr	13158	vs	0
dmf	13158	vs	128
	at	cdc	ph
			33

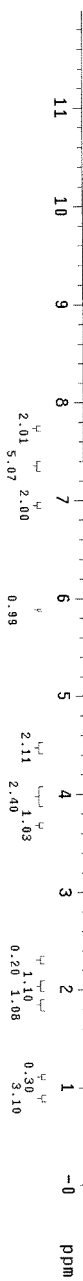
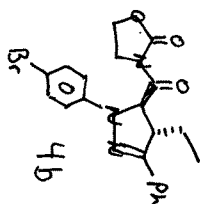




LS-IT-2828-IH-500

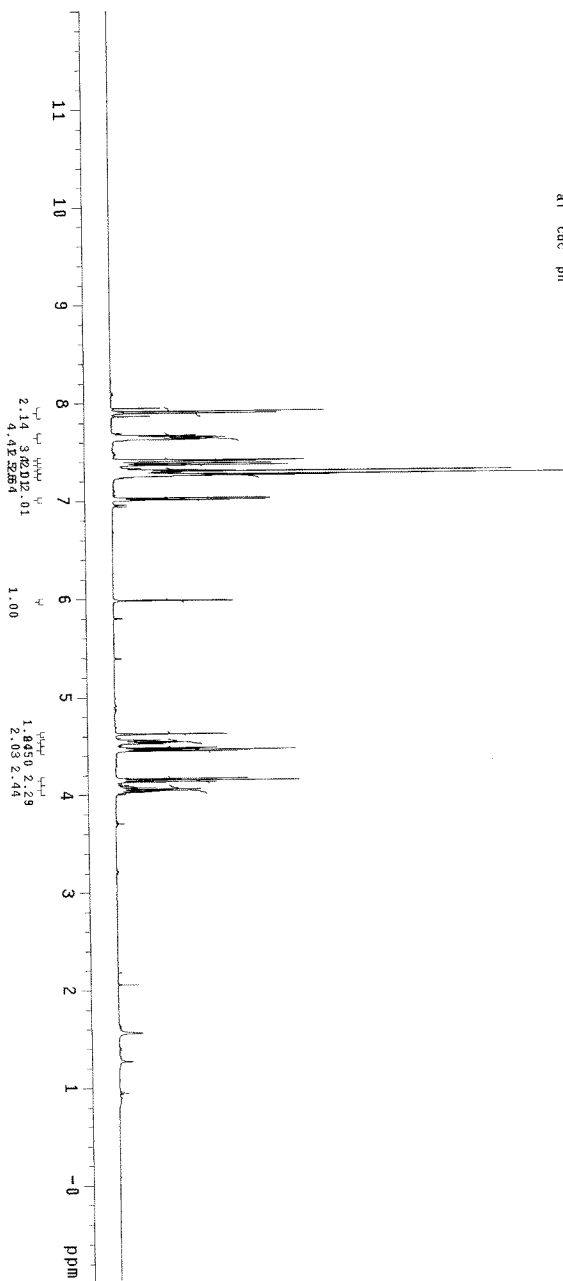
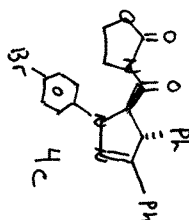
exp1 s2pul


SAMPLE		SPECIAL	
date	Nov 29 2004	temp	25.0
solvent	CDCl3	gain	not used
file	exp	spin	not used
sw	65.00.9	pw	8.800
at	2.508	atfa	0
np	37812	flags	n
ns	4000		n
ds	0.300		y
dl	0.300		nm
nl	16		
ct	16		
TRANSMITTER		PROCESSING	
td	h1	fn	6538
stf	493.674	sp	-507.2
tpw	247.0	wp	6500.9
pw	4.400	fft	3827.6
decoupler	0	tp	60.1
dof	0	plot	-1.9
dm	mm	uc	240
dmf	40	sc	0
dmf	13158	vs	112
	th	cdc	54
	ph		

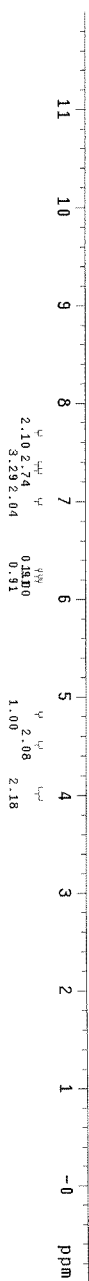


LS-III-19A  
exp2 s2pu1

SAMPLE		SPECIAL	
date	Apr 21 2005	temp	25.0
solvent	CDCl3	gain	not used
file	exp	spin	not used
ACQUISITION	exp	psd	8.800
sv	8500.9	psd	8.800
at	2.908	alfa	0
np	37812	11	0
fb	4000	11	0
hs	0.300	dp	0
dt	0.300	dp	0
nt	16	hs	0
ct	16	hs	0
TRANSMITTER		h1	fb
tp	499.574	ftn	65536
sfreq	247.0	sp	-501.0
tofr	61	wp	6500.9
tpwr	4.400	ftn	3632.6
DECOUPLER		tp	67.6
dn	0	tp	-9.2
dof	0	PLOT	
dm	mm	vc	240
dmw	40	sc	0
dmf	13158	vs	75
at	th	cdc	ph

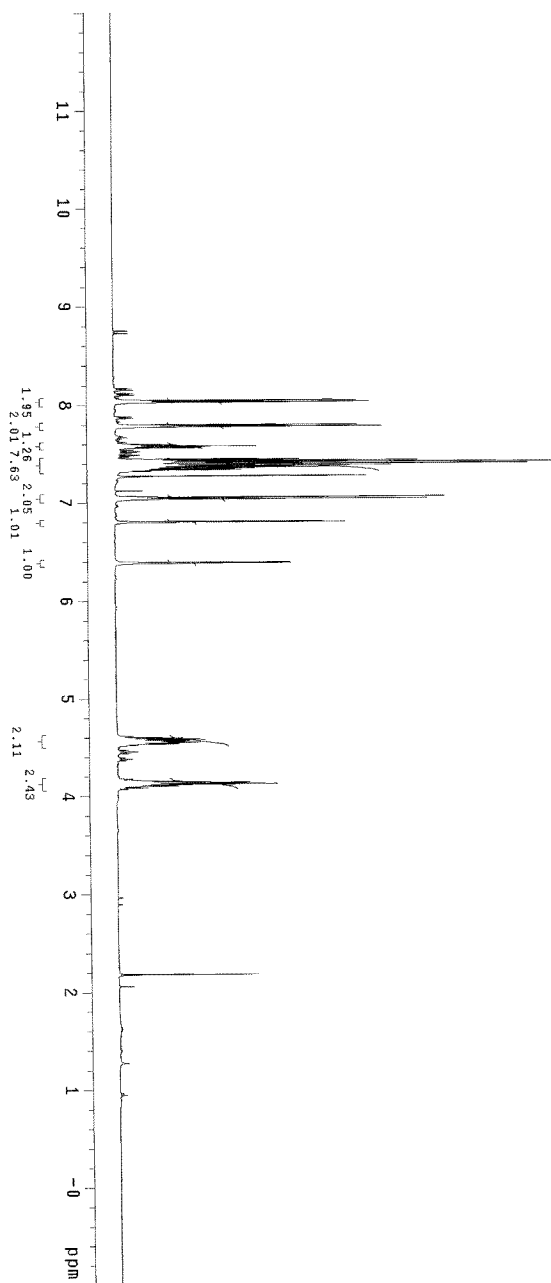
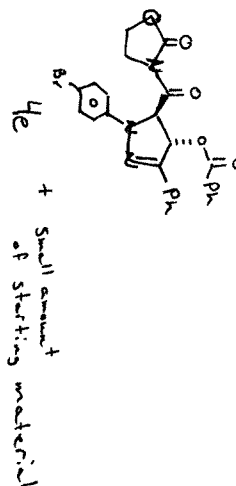



  
 + small amount  
 of starting material



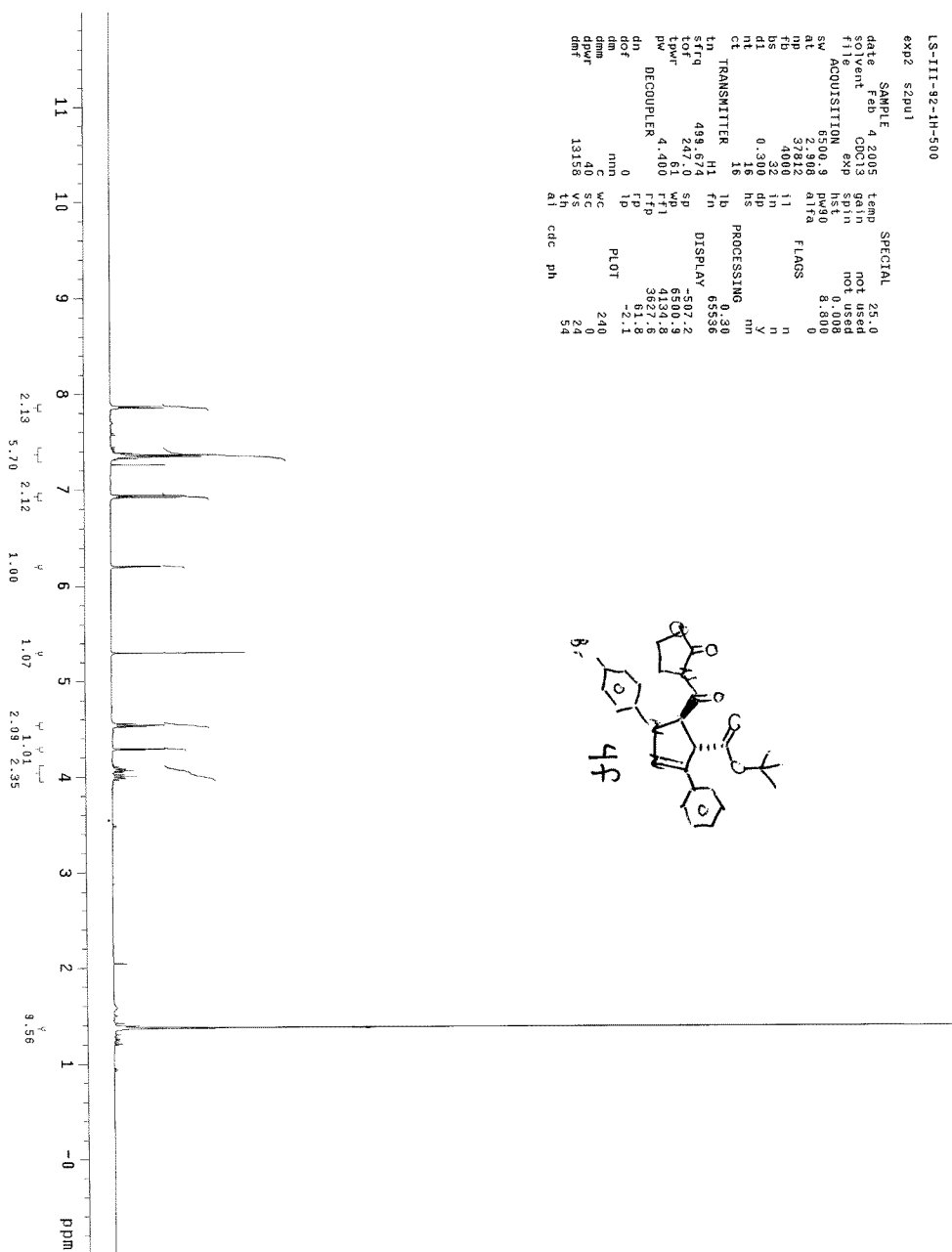
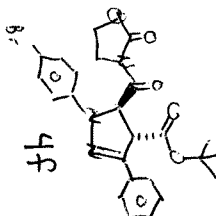
exp2 s2put

SAMPLE										SPECIAL	
date	Apr 21, 2005	temp	25.0								
solvent	CDCl3	gain	not used								
f1	ACQUISITION	exp	hst	0.008							
			8.800								
			0								
sw	6500.9	pw	0								
at	2.908										
sf	3.000										
td	3.000										
fb	32	in	n								
fl	0.300	dp	n								
cl	16										
ct	16										
TRANSMITTER											
tn	11	tb	0.300								
tf	488.8	DISPLAY	6535.8								
tp	247.0	sp	501.0								
tlpr	61	sr	6500.9								
pw	4.480	fft	501.0								
DECOUPLER											
dn		fft	74.6								
dof		1p	-11.6								
dm	nmh										
dmm	nmh										
d4	4	uc	240.0								
d5	5	uc	150.0								
dmy	13158	at	54								
		cd									
		ph									

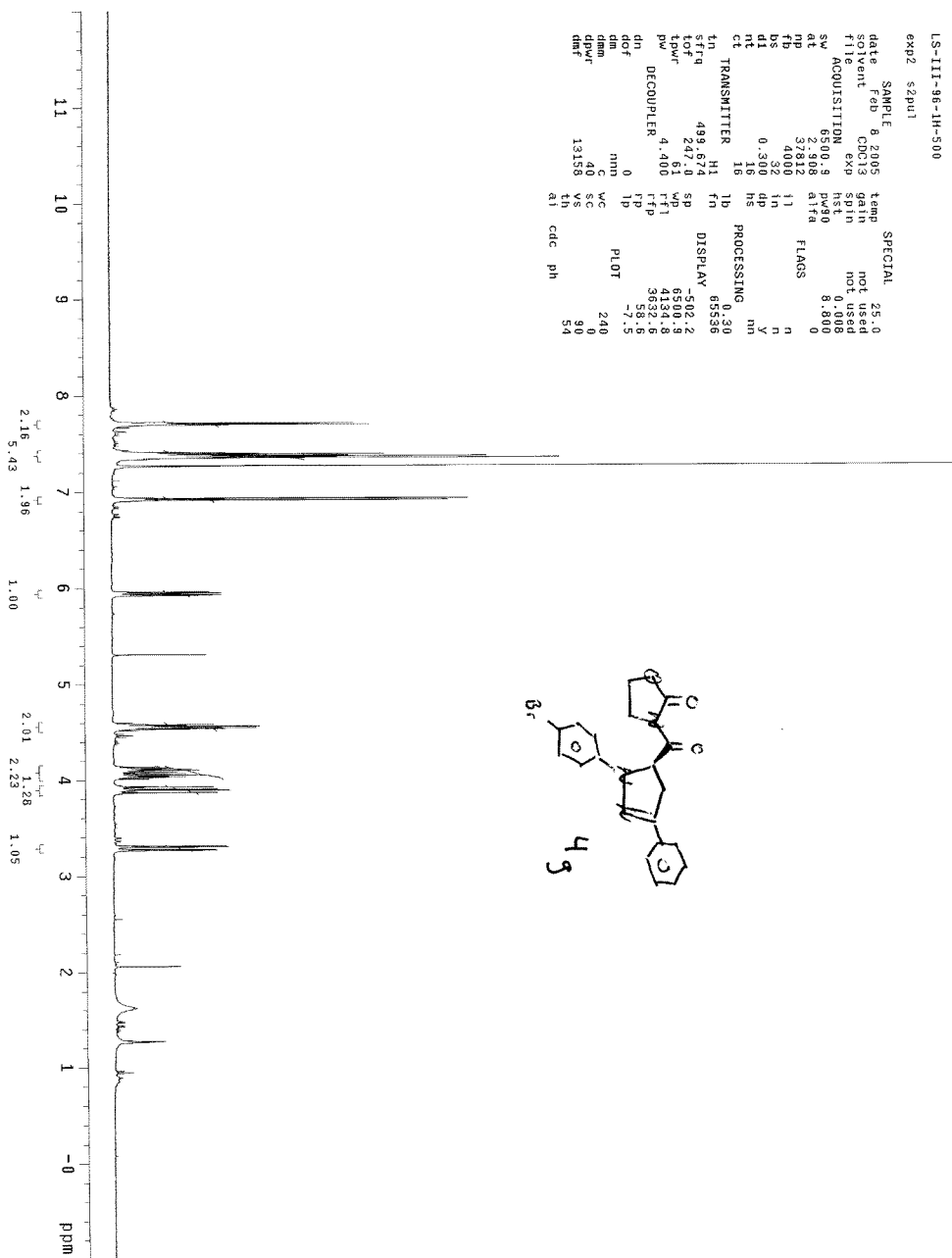
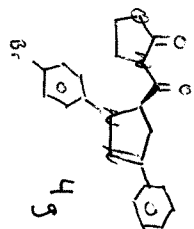


exp2 s2pu1

date	4 2005	temp	25.0	SPECIAL
solvent	CDC13	gain	not used	
f1	ACQUISITION	exp	0.008	
sw	6500.3	pw	8.800	
at	2.986	afw	0	
ft	3060	flags		
fp	3000			
bs	32	n	n	
bs	32	n	n	
cl	0.300	dp	y	
cl	16			
TRANSMITTER				
tn	11	lb	0.30	
trf	408.61	DISPLAY	65536	
trf	267.0	SP	507.2	
tpwr	61	wp	4130.8	
pw	4.400	fft	4134.8	
DECOUPLER				
dcf	0	rf	3651.8	
dcf	0	lp	-2.1	
dmn	nn	PLOT		
dmn	nn		24.0	
dmn	nn	WC	4	
dmf	13158	C	24	
at	cdc	ph	54	



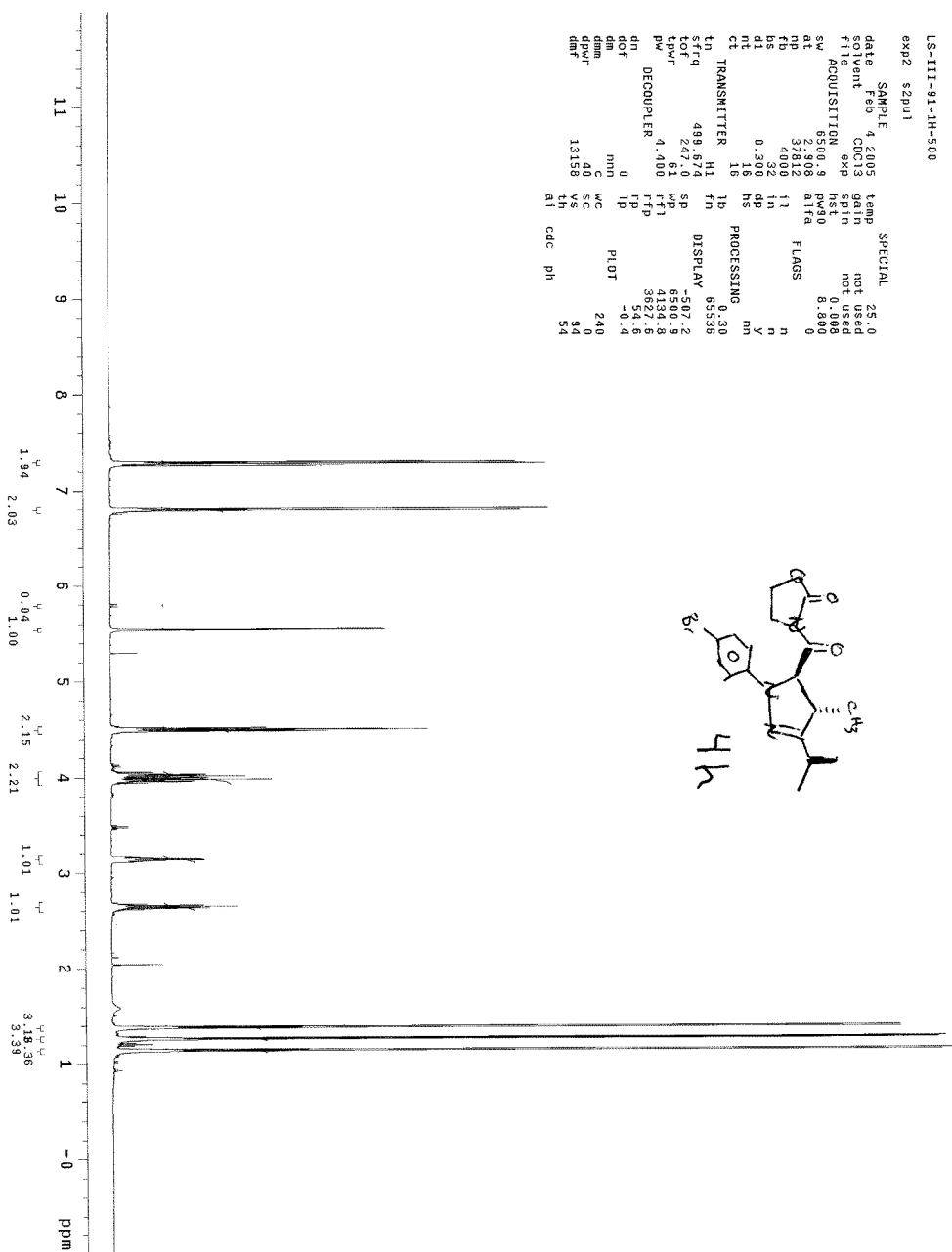
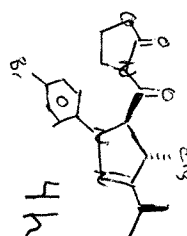
LS-II-96-1H-500  
 exp2 szpul  
 SAMPLE  
 date Feb 8 2005 temp 25.0  
 solvent CDCl3 gain not used  
 T1 1.000 s delay not used  
 T1 ACQUISITION exp 0.008  
 sw 6500.9 pw90 8.800  
 at 2.908 a1fa 0  
 f1 3.400 i1  
 ds 4032 in n  
 d1 0.300 dp y  
 nt 18 hs  
 ct TRANSMITTER H1 fn  
 tn 1b  
 sfreq 499.674 DISPLAY 65536  
 tof 247.0 sp 6500.2  
 idwr 4123.8 w 4123.8  
 pw 4.400 rfp 3632.6  
 DECOUPLER rfp 58.6  
 dn 0 tp  
 dnf mm PLOT 240  
 dnm C wc 90  
 dpm 40 sc 0  
 dmf 13158 vs 54  
 at cdc ph



LS-III-91-1H-500

exp2 s2put1

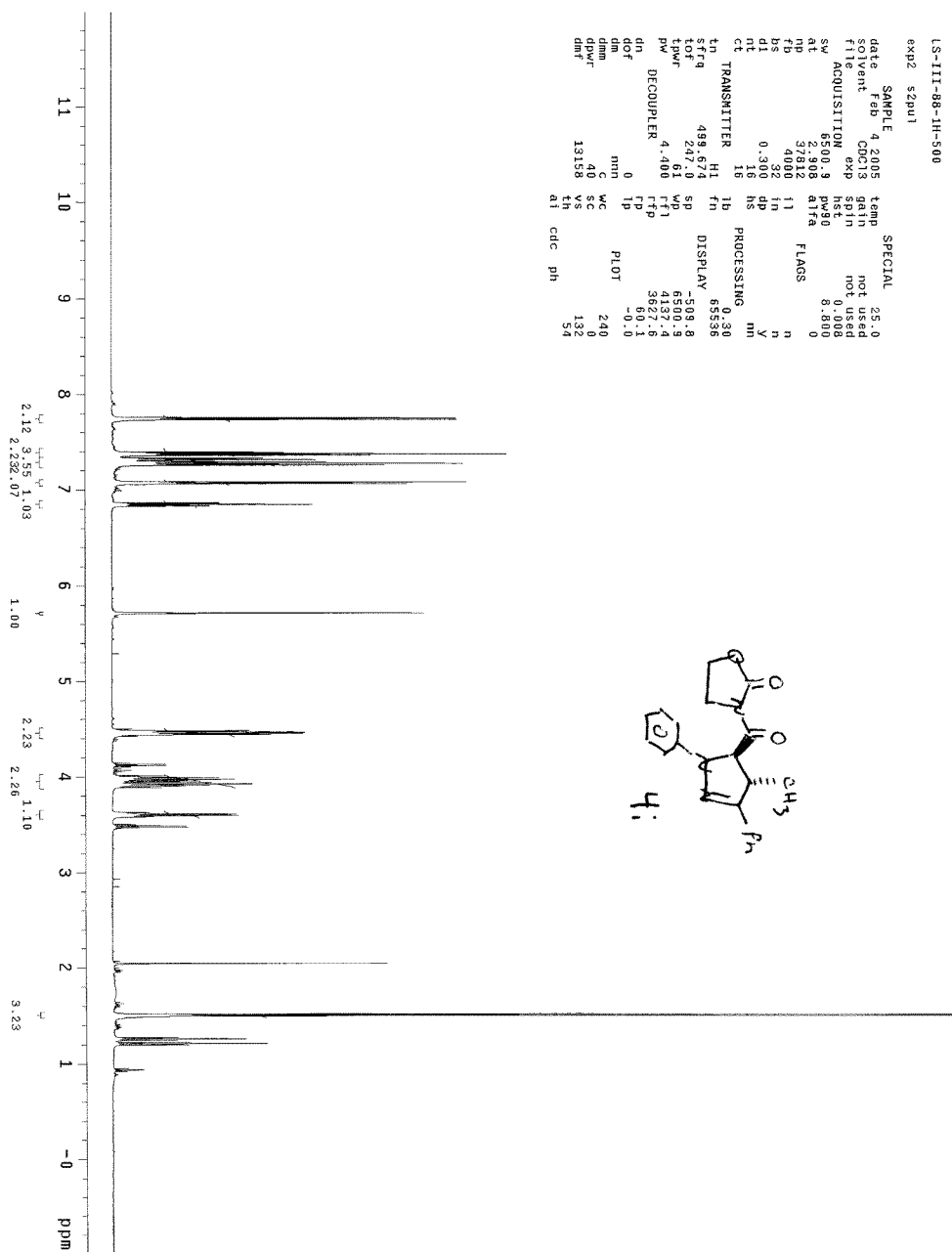
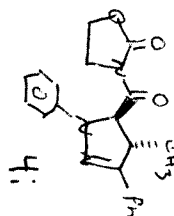
SAMPLE 4 2005 temp SPECIAL 25.0  
date Feb CDC13 gain not used  
solvent CDCl3 spin not used  
file ACQUISITION exp 8.000  
sw 6500.9 pw40  
at 2.908 a1f4 0  
np 37812 11  
nb 4000 11  
h1 1633 11  
d1 0.300 d0  
nt 16 hs  
ct TRANSMITTER H1 16  
th 70  
sfrq 489.674 DISPLAY 65538  
lof 247.0 sp -507.2  
tdw 61 wp 6500.9  
pw 4.400 rfp 3627.6  
DECOUPLER rfp 54.6  
dn 0 tp -0.4  
dm 0 mm WC PLOT 240  
dms 40 SC 0  
dpr 13158 VS 94  
dmf th at cdc ph 54



LS-III-88-1H-500

exp2 sapul

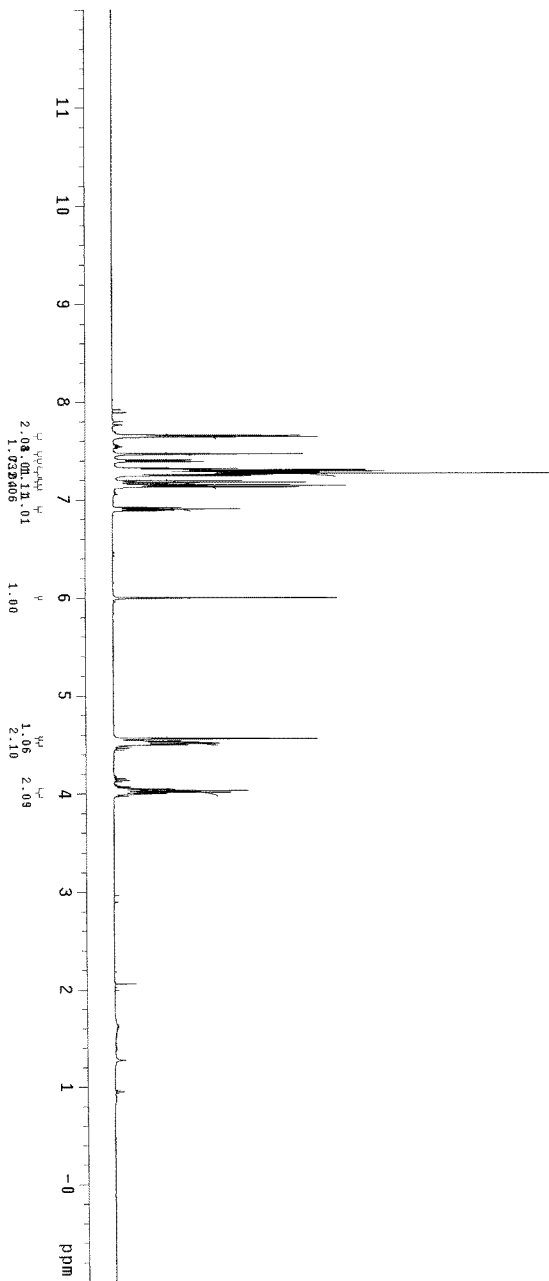
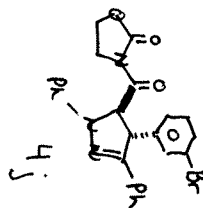
SAMPLE SPECIAL  
date Feb 4 2005 temp 25.0  
solvent CDCl3 gain not used  
F1 300.134 MHz NO 0.008  
ACQUISITION exp hst 8.800  
sw 6500.9 pw90 0  
at 2.808 alfa 0  
np 3.232 11 FLACS n  
bs 7.43 34000 92 in n  
di 0.300 dp y  
nt 16 ns PROCESSING mh  
ct TRANSMITTER H1 fn 65536  
tn 499.674 SP DISPLAY 500.8  
sfrq 247.8 100 500.8  
fwhz 4.406 rfi 4132.4  
pwr 4.406 rfp 3627.6  
DECOUPLER 0 tp 60.1  
dnf 0 1P PLOT -0.0  
dnc 0 1P 240  
dmm C WC 0  
dpmr 40 SC 132  
dmr 13158 V5 134  
at cdc ph





LS-III-87  
exp2 s2pul1

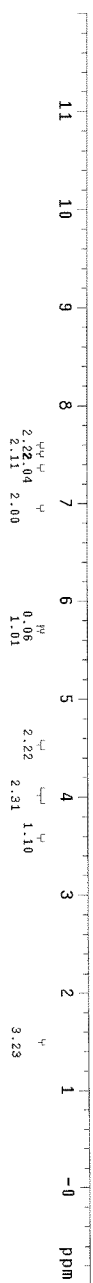
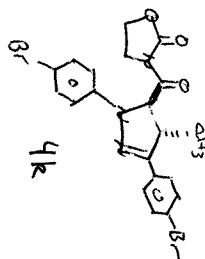
SAMPLE		SPECIAL	
date	Apr 21 2005	temp	25.0
solvent	CDCl3	gain	not used
flavor	exp	not used	
ACQUISITION	exp	not used	
sw	6500.9	pw90	8.800
at	2.908	atfa	0
dp	37812	11	n
fd	4032	in	n
hl	0.300	dp	y
nl	16	hs	PROCESSING 0.30
ct	TRANSMITTER	h1	fn
in	489.674	sp	DISPLAY 65536
tof	247.0	wp	-501.0
dpwr	4.400	rff	3622.5
DECOUPLER	0	fp	-4.5
do	0	tp	
dmf	13158	vs	54



LS-111-83-1H-500

exp2 szpul1

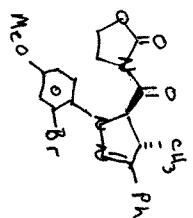
SAMPLE  
 date Feb 4 2005 temp 25.0  
 solvent CDCl3 gain not used  
 file CDC13 spin not used  
 ACQUISITION exp 18.000  
 sw 6500.9 pw90 8.800  
 at 2.908 atfa 0  
 dp 37812 f1  
 fd 4032 f2  
 hz 400 f3  
 d1 0.300 dp  
 nt 16 hs  
 ct TRANSMITTER H1 f0  
 tn 16 PROCESSING 0.30  
 sfreq 499.674 H1 f0  
 tof 247.0 sp  
 fwhz 61.3  
 pw 4.400 rfp  
 DECOUPLER  
 dof 0 f1  
 dm 0 mm WC  
 dnm 40 SC  
 dpr 13158 VS  
 dmf 54  
 at cdc ph



LS-III-94

exp2 s2put1

SAMPLE SPECIAL 25.0  
 date Apr 21 2005 temp  
 solvent CDCl3 gain not used  
 file ACQUISITION exp not used  
 SW 6500.9 pw90 8.800  
 at 2.908 atfa 0  
 np 37812 11 flags  
 nt 16 hs  
 ct TRANSMITTER 16 1b PROCESSING 6.30  
 stfq 489.674 f0 DISPLAY -500.8  
 tof 247.0 sp 6500.9  
 lpw 61 wpi 4532.9  
 pw 4.400 ffp 3632.6  
 DECOUPLER 1p TP 3632.6  
 dn 0 1p PLOT -1.7  
 dm 0 mm WC 240  
 dnm 40 sc 0  
 dpwr 13158 v6 30  
 dmf 54 at cdc ph



4L

