

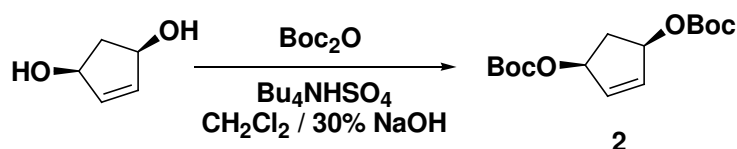
# New Nucleophiles for Palladium-Catalyzed Asymmetric Allylic Alkylation. Total Synthesis of Agelastatin A

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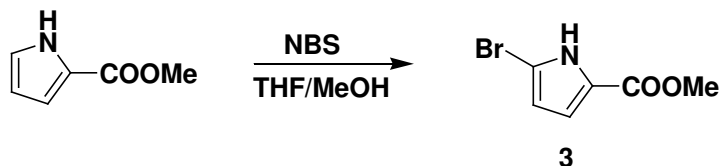
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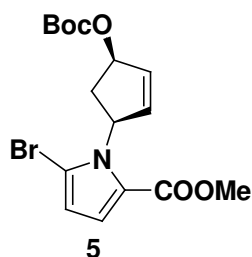
## Supporting Information



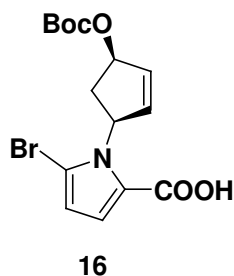
**Compound 2:** A 30% aqueous NaOH solution (8 ml) was added to a solution of cyclopent-4-ene-1,3-diol (1 g, 10 mmol),  $\text{Boc}_2\text{O}$  (5.45 g, 25 mmol) and  $(n\text{-Bu})_4\text{NHSO}_4$  (340 mg, 1 mmol) at 0 °C. After stirring vigorously for 12 hr, 1.5 g  $\text{Boc}_2\text{O}$  and 3 ml 30% aqueous NaOH were added at rt, then stirring continued for another 12 hr. Brine (20 ml) was added and the mixture was diluted with  $\text{CH}_2\text{Cl}_2$ . The organic layer was separated and dried over  $\text{MgSO}_4$ . After evaporation of the solvent, the product was purified by silica gel flash column chromatography (petroleum ether / diethyl ether = 25/1, then 10/1). A white solid was obtained (2.4 g, 80%): mp: 59-60 °C;  $R_f$ : 3/7 (petroleum ether / ethyl acetate = 20/1);  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 500 MHz)  $\delta$  6.11 (s, 2H), 6.38 (dd,  $J = 7.5, 4.0$  Hz, 2H), 2.94-2.88 (m, 1H), 1.87 (dt,  $J = 15, 4.0$  Hz, 1H), 1.47 (s, 18H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 75MHz)  $\delta$  153.0, 134.5, 82.2, 78.8, 37.0, 27.7; IR (film): 2981, 1738, 1459, 1395, 1370, 1339, 1272, 1253, 1159, 1087, 850  $\text{cm}^{-1}$ ; ESI ( $\text{C}_{15}\text{H}_{24}\text{O}_6$ ): 323.2  $[\text{M}+\text{Na}]^+$ .



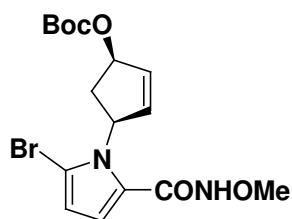
**Compound 3:** NBS (recrystallized, 0.49 g) was added to a solution of pyrrole-2-carboxylate methyl ester in THF (160 ml) and MeOH (80 ml) at 0 °C. After the solution was stirred at 0 °C for 30 min, a second portion of NBS (recrystallized, 0.63 g) was added and after 40 min, a third portion of NBS (recrystallized, 0.51 g) was added. After stirring for 2 hr, the solvent was removed under vacuum. Compound **3** was purified via silica gel flash column chromatography (petroleum ether / diethyl ether = 20/1, then 8/1) to give a white floppy solid (1.6 g, 50%): mp: 101-103 °C;  $R_f$ : 0.35 (petroleum ether / ether = 4/1);  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz):  $\delta$  9.1 (br s, 1H), 6.82 (dd,  $J = 3.0, 3.5$  Hz, 1H), 6.21 (dd,  $J = 3.0, 3.5$  Hz, 1H), 3.86 (s, 3H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz)  $\delta$  160.8, 123.7, 116.8, 112.7, 105.2, 51.8; IR (film): 3250, 2924, 2853, 1702, 1552, 1449, 1415, 1389, 1327, 1207  $\text{cm}^{-1}$ ; HRMS ( $\text{C}_6\text{H}_6\text{NO}_2\text{Br}$ ): Calc'd. 202.958190 ( $\text{M}^+$ ), Found 202.958096.



**Compound 5:** (eg. Table 1, entry 5) A solution of  $[\text{Pd}(\text{C}_3\text{H}_5\text{Cl})_2]$  (18.3 mg, 0.05 mmol) and (*R,R*)-**4** (103.5 mg, 0.15 mmol) in 15 ml degassed  $\text{CH}_2\text{Cl}_2$ , which had been stirring at rt for 10 min, was added to a mixture of compound **2** (750 mg, 2.5 mmol), compound **3** (512.5 mg, 2.5 mmol) and  $\text{Cs}_2\text{CO}_3$  (815 mg, 2.5 mmol) under Ar. The mixture was stirred at rt for 2 to 3 hr, then filtered through a celite cake. After removing the solvent under vacuum, compound **5** was purified via silica gel flash column chromatography (petroleum ether / diethyl ether = 25/1) to give a light yellow oil (846 mg, 88%, 87% ee by HPLC OD column, 98:2 heptane: *i*-propanol, 1 ml/min).  $R_f$ : 0.5 (petroleum ether / ethyl acetate = 8/1);  $[\alpha]_D^{22}$ : -2.64 ( $\text{CH}_2\text{Cl}_2$ , c 0.94);  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 500 MHz)  $\delta$  6.93 (d,  $J$  = 4.0 Hz, 1H), 6.58-6.54 (m, 1H), 6.19 (d,  $J$  = 4.0 Hz, 1H), 6.11 (ddd,  $J$  = 2.0, 2.0, 5.5 Hz, 1H), 6.01 (ddd,  $J$  = 2.0, 2.5, 5.5 Hz, 1H), 5.57-5.54 (m, 1H), 3.80 (s, 3H), 3.10 (ddd,  $J$  = 8.0, 8.0, 13.5 Hz, 1H), 2.29 (ddd,  $J$  = 6.5, 8.0, 13.5 Hz, 1H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 125 MHz)  $\delta$  160.8, 153.1, 135.7, 130.8, 123.8, 119.1, 113.5, 109.7, 82.3, 79.7, 60.2, 51.3, 38.2, 27.8; IR (film): 2981, 1736, 1708, 1522, 1441, 1410, 1369, 1339, 1275, 1255, 1224, 1156, 1131, 1094  $\text{cm}^{-1}$ ; HRMS ( $\text{C}_{16}\text{H}_{20}\text{NO}_5\text{Br}$ ): Calc'd. 385.052484 ( $\text{M}^+$ ), Found 385.051845.

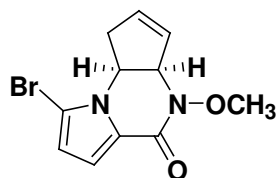


**Compound 16:** A solution of LiOH (1N, 4.7 ml) was added to a solution of compound **5** (605 mg, 1.57 mmol) in THF (15 ml) and  $\text{H}_2\text{O}$  (0.3 ml). The solution was stirred at rt for 48 hrs. The THF was removed under vacuum, and the aqueous phase was diluted with water and acidified to pH = 1-2 by aqueous HCl (3 N). Preformed white precipitate was collected and washed with 2 ml water twice, then dried under high vacuum. Compound **16** was obtained as a white powder (500 mg, 86%): mp: 119-121  $^\circ\text{C}$ ;  $R_f$ : 0.5 (DCM / MeOH = 8/1);  $[\alpha]_D$ : +19.31 ( $\text{CH}_2\text{Cl}_2$ , c 0.72);  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 500 MHz)  $\delta$  7.11 (d,  $J$  = 4.4 Hz, 1H), 6.54 (td,  $J$  = 8.0, 2.0 Hz, 1H), 6.24 (d,  $J$  = 4.4 Hz, 1H), 6.14-6.12 (m, 1H), 6.04-6.01 (m, 1H), 5.57 (td,  $J$  = 1.6, 8.0 Hz, 1H), 3.17-3.10 (m, 1H), 2.33-2.26 (m, 1H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 125 MHz)  $\delta$  164.6, 153.2, 135.5, 131.0, 122.9, 121.2, 114.1, 111.3, 82.3, 79.6, 60.4, 38.2, 27.8; IR (film): 3200, 2981, 2926, 1736, 1670, 1523, 1448, 1403, 1276, 1254, 1162  $\text{cm}^{-1}$ ; HRMS ( $\text{C}_{15}\text{H}_{18}\text{NO}_5\text{Br}$ ): Calc'd. 371.036834 ( $\text{M}^+$ ), Found 371.036488.



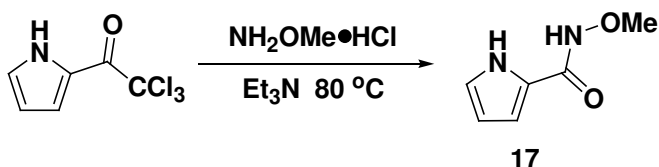
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**Compound 6:** To a solution of compound **16** (100 mg, 0.27 mmol) in THF (2.7 ml),  $(\text{COCl})_2$  (36.8 mg, 0.29 mmol) and one drop of DMF were added. The resulting solution named A was then stirred for 1 hr at rt. In another separate operation, water (0.3 ml) was added to a mixture of  $\text{NH}_2\text{OMe}\cdot\text{HCl}$  (225.5 mg, 2.7 mmol) and  $\text{K}_2\text{CO}_3$  (372.6 mg, 2.7 mmol). The resulting mixture was stirred at rt for 0.5 hr, before it was added to the above solution A. The resulting solution was stirred for 12 hr at rt and then extracted with ethyl acetate (10 ml) three times. The combined the organic phase was washed once with brine (10 ml) and then dried over  $\text{MgSO}_4$ . Compound **6** was purified by silica gel flash column chromatography ( $\text{CH}_2\text{Cl}_2$ , then  $\text{CH}_2\text{Cl}_2 / \text{MeOH} = 40/1$ ) to give a yellow solid (87.7 mg, 85%): mp: 58-60 °C;  $R_f$ : 0.6 ( $\text{CH}_2\text{Cl}_2 / \text{MeOH} = 9/1$ );  $[\alpha]_D$ : +1.55 ( $\text{CH}_2\text{Cl}_2$ , c 0.74);  $^1\text{H NMR}$  ( $\text{CDCl}_3$ , 500 MHz)  $\delta$  8.47 (br, 1H), 6.50 (d,  $J = 4$  Hz, 1H), 6.22-6.25 (m, 1H), 6.13 (d,  $J = 4$  Hz, 1H), 6.11 (dt,  $J = 6, 2$  Hz, 1H), 6.00 (dt,  $J = 6, 2$  Hz, 1H), 5.49-5.23 (m, 1H), 3.81 (s, 3H), 3.14-3.08 (ddd,  $J = 13.5, 7.5$  Hz, 1H), 2.30-2.25 (ddd,  $J = 13.5, 7.5, 6.5$  Hz, 1H), 1.47 (s, 9H);  $^{13}\text{C NMR}$  ( $\text{CDCl}_3$ , 125 MHz)  $\delta$  160.9, 153.1, 135.5, 131.2, 124.7, 113.7, 112.8, 108.4, 82.3, 79.6, 64.4, 60.5, 38.2, 27.8; IR (film): 3244, 2979, 2933, 1738, 1651, 1530, 1416, 1340, 1277, 1161, 1096, 1055  $\text{cm}^{-1}$ ; HRMS ( $\text{C}_{16}\text{H}_{21}\text{N}_2\text{O}_5\text{Br}$ ): Calc'd. 409.063383 ( $\text{M}^+$ ), Found 400.064848.

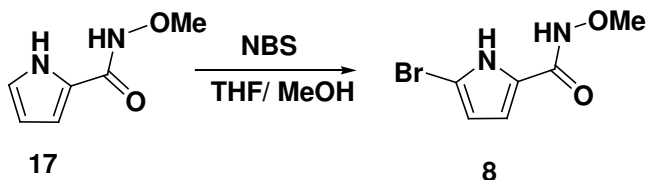


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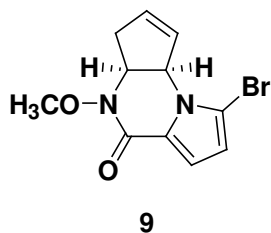
**Compound 7:** A solution of  $\text{Pd}_2(\text{dba})_3\text{CHCl}_3$  (2.29 mg, 0.00025 mmol) and *(R,R)*-**4** (5.18 mg, 0.00075 mmol) in  $\text{CH}_2\text{Cl}_2$  (1 ml), which had been stirred at rt for 10 min, was added to a mixture of compound **6** (20 mg, 0.05 mmol) and  $\text{Cs}_2\text{CO}_3$  (16.3 mg, 0.05 mmol) under Ar. The mixture was stirred at rt for 12 hr, then filtered through a celite cake. After removing the solvent under vacuum, compound **7** was purified via silica gel flash column chromatography (petroleum ether / ether = 8/1, then  $\text{CH}_2\text{Cl}_2 / \text{MeOH} = 40/1$ ) to give a white solid (12.9 mg, 91.5%): mp: 107-109 °C;  $R_f$ : 0.6 ( $\text{CH}_2\text{Cl}_2 / \text{MeOH} = 9/1$ );  $[\alpha]_D$ : +164.7 ( $\text{CH}_2\text{Cl}_2$ , c 0.34);  $^1\text{H NMR}$  ( $\text{CDCl}_3$ , 500 MHz)  $\delta$  6.94 (dd,  $J = 1, 4$  Hz, 1H), 6.30 (dd,  $J = 1, 4$  Hz, 1H), 6.22-6.20 (m, 1H), 6.13-6.12 (m, 1H), 4.84-4.79 (m, 2H), 3.85 (s, 3H), 3.13-3.07 (m, 1H), 2.63-2.58 (m, 1H);  $^{13}\text{C NMR}$  ( $\text{CDCl}_3$ , 125 MHz)  $\delta$  156.3, 135.5, 128.6, 123.7, 116.1, 106.8, 101.6, 64.1, 64.0, 57.2, 38.2; IR (film): 2927, 2853, 1668, 1545, 1418, 1358, 1334, 1310, 1027  $\text{cm}^{-1}$ ; HRMS ( $\text{C}_{11}\text{H}_{11}\text{N}_2\text{O}_2\text{Br}$ ): Calc'd 282.000389 ( $\text{M}^+$ ), Found 281.999714.



**Compound 17:** 2,2,2-Trichloro-1-(1*H*-pyrrol-2-yl)-ethanone (1.08 g, 5 mmol),  $\text{NH}_2\text{OMe}\cdot\text{HCl}$  (625 mg, 7.5 mmol) and  $\text{Et}_3\text{N}$  (2.1 ml) were sealed in a tube. After the mixture was stirred at 80 °C overnight,  $\text{Et}_3\text{N}$  was removed under vacuum. The residue was then diluted with  $\text{CH}_2\text{Cl}_2$ . Compound **17** was purified by silica gel flash column chromatography (petroleum ether / ethyl acetate = 3/2, then 1/1) to give an off-white solid (687.5 mg, 98%): mp: 96-98 °C;  $R_f$ : 0.25 (petroleum ether / ethyl acetate = 1/1);  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz)  $\delta$  10.28 (br, 1H), 9.90 (br, 1H), 6.93 (s, 1H), 6.82 (s, 1H), 6.19 (d,  $J = 2.4$  Hz, 1H), 3.80 (s, 3H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz)  $\delta$  161.4, 122.9, 122.4, 112.2, 110.0, 64.7; IR (film): 3240 (br), 2980, 1628, 1554, 1507, 1439, 1405, 1330, 1096, 1047  $\text{cm}^{-1}$ ; HRMS ( $\text{C}_6\text{H}_8\text{N}_2\text{O}_2$ ): Calc'd. 140.058578 ( $\text{M}^+$ ), Found 140.058374.

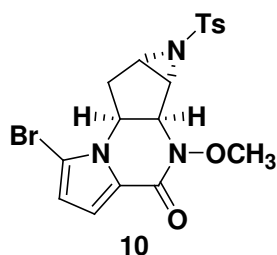


**Compound 8:** NBS (225 mg, 1.26 mmol) was added to a solution of compound **17** (446 mg, 3.19 mmol) in THF (35 ml) and MeOH (17 ml) at 0 °C. After the mixture was stirred for 30 min, another portion of NBS (314 mg, 1.76 mmol) was added. The resulting solution was allowed to warm to room temperature and stirred overnight. After removal of the solvent, compound **8** was purified by silica gel flash column chromatography (petroleum ether / ethyl acetate = 7/3, then 3/2) to give an off-white solid (662 mg, 95%): mp: 125-126 °C;  $R_f$ : 0.3 (petroleum ether / ethyl acetate = 1/1);  $^1\text{H}$  NMR ( $\text{CD}_3\text{OD}$ , 400 MHz)  $\delta$  6.64 (d,  $J = 4.0$  Hz, 1H), 6.12 (d,  $J = 4.0$  Hz, 1H), 3.75 (s, 3H);  $^{13}\text{C}$  NMR ( $\text{CD}_3\text{OD}$ , 100 MHz)  $\delta$  161.4, 125.5, 113.3, 112.5, 105.2, 64.6; IR (film): 3207 (br), 3002, 1622, 1553, 1505, 1411, 1327, 1046  $\text{cm}^{-1}$ ; HRMS ( $\text{C}_6\text{H}_7\text{N}_2\text{O}_2\text{Br}$ ): Calc'd. 217.969089 ( $\text{M}^+$ ), Found 217.969212.

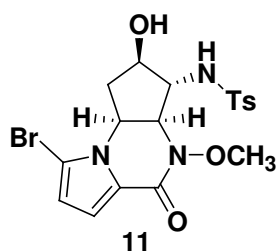


**Compound 9:** (eg. Table 2, entry 9) A solution of  $\text{Pd}_2(\text{dba})_3\text{CHCl}_3$  (5.2 mg, 0.005 mmol) and (*R,R*)-**4** (10.4 mg, 0.015 mmol) in degassed  $\text{CH}_2\text{Cl}_2$  (0.5 ml), which had been stirring at 0 °C for 10 min, was added to a mixture of compound **8** (21.9 mg, 0.1 mmol), compound **2** (45 mg, 0.15 mmol) and HOAc (10  $\mu\text{l}$ , 1M solution in  $\text{CH}_2\text{Cl}_2$ ) under Ar. The mixture was stirred at rt for 3.5 hr, and then a solution of  $\text{Pd}_2(\text{dba})_3\text{CHCl}_3$  (5.2 mg, 0.005 mmol) and *Rac*-**4** (10.4 mg, 0.015 mmol) in degassed  $\text{CH}_2\text{Cl}_2$  (0.5 ml), which had been stirring at rt for 10 min, was added. The resulting solution was stirred at rt for 3 hr. Compound **9** was purified via silica gel flash column chromatography (petroleum ether / ethyl acetate = 4/1, then 3/1) to give a colorless

solid (23.0 mg, 82%, 97.5% ee by HPLC OD column, 90:10 heptane: *i*-propanol, 0.8 ml/min): mp: 111-113 °C;  $R_f$ : 0.2 (petroleum ether / ethyl acetate = 4/1);  $[\alpha]_D$ : -120.2 (c 1.0, CH<sub>2</sub>Cl<sub>2</sub>); <sup>1</sup>H NMR (CDCl<sub>3</sub>, 500 MHz)  $\delta$  6.91 (d,  $J$  = 5.0 Hz, 1H), 6.28 (d,  $J$  = 5.0 Hz, 1H), 6.02 (m, 1H), 5.95 (m, 1H), 5.29 (d,  $J$  = 8.0 Hz, 1H), 4.65 (ddd,  $J$  = 8.0, 6.5, 2.5 Hz, 1H), 3.85 (s, 3H), 2.92 (d,  $J$  = 21.5 Hz, 1H), 2.71 (ddd,  $J$  = 21.5, 6.5, 2.5 Hz, 1H); <sup>13</sup>C NMR (CDCl<sub>3</sub>, 125 MHz)  $\delta$  157.3, 132.0, 129.0, 123.8, 114.7, 113.6, 104.8, 63.0, 61.6, 60.7, 36.4; IR (film): 2925, 2854, 1667, 1545, 1417, 1320, 1028 cm<sup>-1</sup>; HRMS (C<sub>11</sub>H<sub>11</sub>N<sub>2</sub>O<sub>2</sub>Br): Calc'd. 282.000389 (M<sup>+</sup>), Found 281.999491.

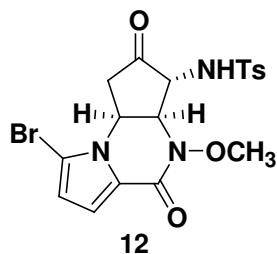


**Compound 10:** Benzene (1 ml) was added to a mixture of 4 Å molecular sieves (75 mg), compound **5** (20 mg, 0.071 mmol), PhI=NTs (132 mg, 0.355 mmol), and catalyst **14** (17.3 mg, 0.0355 mmol) under N<sub>2</sub>. The resulting solution was stirred at rt for 4 hr, then filtered through a silica gel cake, and the cake was rinsed with ethyl acetate. After removing the solvent under vacuum, compound **10** was purified via alumina (neutral, activity 3) flash column chromatography (petroleum ether / ethyl acetate = 8/1, 4/1, then 3/1) to give a colorless foam (16.6 mg, 52%):  $R_f$ : 0.5 (petroleum ether / ethyl acetate=3/2);  $[\alpha]_D$ : +21.8 (c 0.85, CHCl<sub>3</sub>); <sup>1</sup>H NMR (CDCl<sub>3</sub>, 500 MHz):  $\delta$  7.86 (dd,  $J$  = 1.5, 6.5 Hz, 2H), 7.40 (dd,  $J$  = 0.5, 8.5 Hz, 2H), 6.94 (d,  $J$  = 4.0 Hz, 1H), 6.26 (d,  $J$  = 4.5 Hz, 1H), 4.50-4.44 (m, 2H), 3.97 (s, 3H), 3.88 (d,  $J$  = 5 Hz, 1H), 3.61 (dd,  $J$  = 2.5, 5.0 Hz, 1H), 2.77 (dd,  $J$  = 7.0, 14 Hz, 1H), 2.48 (s, 3H), 1.93 (ddd,  $J$  = 14.5, 9.0, 2.5 Hz, 1H); <sup>13</sup>C NMR (CDCl<sub>3</sub>, 125 MHz)  $\delta$  158.2, 145.5, 134.8, 130.3, 128.2, 122.8, 116.2, 114.1, 106.2, 64.5, 61.3, 53.3, 44.7, 44.1, 32.9, 22.0; IR (film): 2925, 2855, 1682, 1543, 1433, 1416, 1325, 1163 cm<sup>-1</sup>; HRMS (C<sub>18</sub>H<sub>21</sub>N<sub>3</sub>O<sub>4</sub>SBr): Calc'd. 451.020139 (M<sup>+</sup>), Found 451.020921.



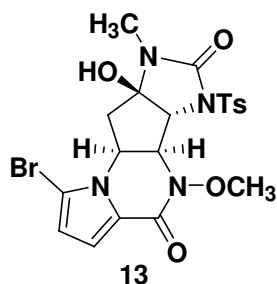
**Compound 11:** TFA (30  $\mu$ l) was added to a solution of compound **10** (18 mg, 0.04 mmol) in dioxane (0.9 ml) and water (0.6 ml) under N<sub>2</sub>. The resulting solution was heated in a microwave at 150 °C for 2.5 hr. The residue was diluted with ethyl acetate, washed with saturated NaHCO<sub>3</sub>, washed with brine, and finally dried over MgSO<sub>4</sub>. Compound **11** was purified via silica gel flash column chromatography (petroleum ether / ethyl acetate = 4/1, then 1/1) to give a colorless foam:  $R_f$ : 0.2 (petroleum ether / ethyl acetate = 1/1);  $[\alpha]_D$ : +36.2 (c 0.90, CH<sub>2</sub>Cl<sub>2</sub>); <sup>1</sup>H NMR (CDCl<sub>3</sub>, 500 MHz)  $\delta$  7.80 (d,  $J$  = 8 Hz, 2H), 7.35 (d,  $J$  = 8 Hz, 2H), 6.91 (d,  $J$  = 4 Hz, 1H), 6.28 (d,  $J$  = 4 Hz, 1H), 5.68 (d,  $J$  = 3.5 Hz, 1H), 4.76-4.71 (m, 1H), 4.33 (dd,  $J$  = 7.5, 14 Hz, 1H), 4.23

(dd,  $J = 3.5, 7.0$  Hz, 1H), 3.63 (dd, 1H), 3.54 (br, 1H), 2.89-2.84 (m, 1H), 2.44 (s, 3H), 2.10-2.04 (m, 1H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 125 MHz)  $\delta$  157.2, 144.5, 135.3, 130.0, 127.4, 122.8, 115.7, 114.1, 105.8, 75.8, 65.7, 64.3, 63.0, 53.3, 38.6, 21.6; IR (film): 3435, 3238, 2828, 1652, 1540, 1415, 1338, 1160, 2093  $\text{cm}^{-1}$ ; HRMS ( $\text{C}_{18}\text{H}_{20}\text{N}_3\text{O}_5\text{SBr}$ ): Calc'd. 471.028658 ( $\text{M}^+$ ), Found 471.028806.



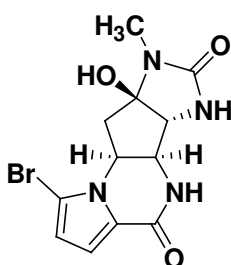
**Compound 12:**

- a) From Compound **11**: Dess-Martin periodinane (13.5 mg, 0.032 mmol) was added to a solution of compound **11** (10 mg, 0.0213 mmol) and  $\text{NaHCO}_3$  (5.4 mg, 0.0639 mmol) in 0.25 ml  $\text{CH}_2\text{Cl}_2$ . The resulting solution was stirred at rt for 30 min before being quenched with sat. aq.  $\text{Na}_2\text{S}_2\text{O}_3$ . Compound **12** was purified via silica gel flash column chromatography (petroleum ether / ethyl acetate = 3/1, then 3/2) to give a white solid (6-7 mg 70-80%).
- b) From Compound **10**: DMSO (1 ml) was added to a mixture of compound **10** (30 mg, 0.0665 mmol) and  $\text{In}(\text{OTf})_3$  (26 mg, 0.0462 mmol) at rt under  $\text{N}_2$ . The resulting solution was heated at 80  $^\circ\text{C}$  for 6 hr, before being diluted with ethyl acetate (30 ml). The solution was washed with brine. The aqueous phases were combined, and extracted with ethyl acetate. Finally, the organic phases were combined and dried over  $\text{MgSO}_4$ . After the solvent was removed under vacuum, compound **12** was purified via silica gel flash column chromatography (petroleum ether / ethyl acetate = 3/1, then 3/2) to give a white solid (28.3 mg, 91%): mp: 100-102  $^\circ\text{C}$ ,  $R_f$ : 0.3 (petroleum ether / ethyl acetate = 1/1);  $[\alpha]_D$ : +57.8 (c 0.97,  $\text{CH}_2\text{Cl}_2$ );  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 500 MHz)  $\delta$  7.82 (d,  $J = 8.0$  Hz, 2H), 7.37 (d,  $J = 8.0$  Hz, 2H), 7.03 (d,  $J = 4.0$  Hz, 1H), 6.35 (d,  $J = 4.0$  Hz, 1H), 5.63 (d,  $J = 7.5$  Hz, 1H), 5.23-5.18 (m, 1H), 5.00 (d,  $J = 2.5$  Hz, 1H), 4.98 (d,  $J = 5.5$  Hz, 1H), 4.00 (br, 1H), 3.84 (s, 3H), 3.02 (dd,  $J = 7.5, 18$  Hz, 1H), 2.68 (dd,  $J = 11, 18$  Hz, 1H), 2.46 (s, 3H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 125 MHz) TBD; IR (film): 3252, 2924, 2854, 1768, 1652, 1548, 1416, 1338, 1162, 1093, 1025  $\text{cm}^{-1}$ ; HRMS ( $\text{C}_{18}\text{H}_{18}\text{N}_3\text{O}_5\text{SBr}$ ): Calc'd. 469.013008 ( $\text{M}^+$ ), Found 469.013016.



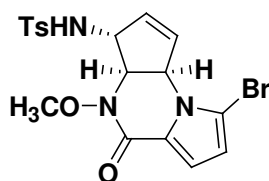
**Compound 13:** To a mixture of compound **12** (15 mg, 0.034 mmol) and  $\text{Cs}_2\text{CO}_3$  (2.2 mg, 0.0068 mmol) in  $\text{CH}_2\text{Cl}_2$  (0.5 ml) was added a solution of  $\text{CH}_3\text{NCO}$  (2.3

mg, in 0.85 ml benzene) during a period of 2 hr at room temperature. The resulting solution was stirred for another 2 hr before being quenched with  $\text{KH}_2\text{PO}_4$  (5 ml, 1.0 M). The mixture was extracted with 5 ml  $\text{CHCl}_3$  twice and then extracted with 5 ml ethyl acetate twice. The organic phases were combined, and dried over  $\text{MgSO}_4$ . Compound **13** was purified via silica gel column chromatography (petroleum ether / ethyl acetate = 4/1, then 2/3, then 1/1) to give a foam-like colorless solid (9.0 mg, 53%):  $R_f$ : 0.4 (petroleum ether / ethyl acetate = 2/3);  $[\alpha]_D$ : + 9.67 (c 0.73,  $\text{CHCl}_3$ );  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 500 MHz)  $\delta$  7.98 (d,  $J$  = 8.0 Hz, 2H), 7.37 (d,  $J$  = 8.0 Hz, 2H), 6.96 (d,  $J$  = 4.0 Hz, 1H), 6.29 (d,  $J$  = 4.0 Hz, 1H), 5.76 (s, 1H), 5.04 (s, 1H), 4.65 (m, 2H), 3.95 (s, 3H), 2.88 (s, 3H), 2.67 (dd,  $J$  = 7.5, 13 Hz, 1H), 2.43 (s, 3H), 2.32 (dd,  $J$  = 13, 13 Hz, 1H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 125 MHz)  $\delta$  156.6, 152.8, 145.6, 135.3, 130.1, 128.2, 122.6, 116.9, 114.0, 106.3, 91.6, 66.4, 65.5, 62.0, 53.4, 38.9, 25.1, 21.8; IR (film): 3290 (br), 2924, 1743, 1651, 1545, 1414, 1344, 1305, 1175, 1092, 1026  $\text{cm}^{-1}$ ; HRMS ( $\text{C}_{20}\text{H}_{21}\text{N}_4\text{O}_6\text{SBr}$ ): Calc'd. 524.036518 ( $\text{M}^+$ ), Found 524.036512.



(+)-Agelastatin A (1)

**(+)-Agelastatin A (1)**: Freshly made  $\text{SmI}_2$  (1.6 ml, 0.1 M in THF) was added to compound **13** (10.0 mg, 0.019 mmol) under argon at 0 °C. The resulting blue solution was allowed to gradually warm to rt and stirred for 2 hr before another 0.5 ml  $\text{SmI}_2$  (0.1 M in THF) was added. After the solution was stirred at rt for 15 min, THF was removed under vacuum. The residue was first purified by silica gel chromatography (10% to 15% MeOH in  $\text{CH}_2\text{Cl}_2$ ). A yellow solid was obtained, which was then dissolved in 15% MeOH in  $\text{CH}_2\text{Cl}_2$  and filtered through charcoal. The (+)-Agelastatin A was further purified by another silica gel chromatography (10% to 15% MeOH in  $\text{CH}_2\text{Cl}_2$ ) to give an off-white solid (6.0 mg, 88%): mp: 195 °C (decomposed);  $R_f$ : 0.25 (10% MeOH in  $\text{CH}_2\text{Cl}_2$ );  $[\alpha]_D$ : + 53.2 (c 0.13, MeOH);  $^1\text{H}$  NMR ( $\text{CD}_3\text{OD}$ , 500 MHz):  $\delta$  6.91 (d,  $J$  = 4.0 Hz, 1H), 6.33 (d,  $J$  = 4.0 Hz, 1H), 4.60 (m, 1H), 4.09 (d,  $J$  = 5.5 Hz, 1H), 3.89 (s, 1H), 2.81 (s, 3H), 2.65 (dd,  $J$  = 6.5, 13 Hz, 1H), 2.10 (dd,  $J$  = 13, 13 Hz, 1H);  $^{13}\text{C}$  NMR ( $\text{CD}_3\text{OD}$ , 150 MHz)  $\delta$  161.4, 161.1, 124.1, 116.0, 113.8, 107.2, 95.7, 67.4, 62.2, 54.4, 40.0, 24.2; IR (film): 3346 (br), 2923, 1685, 1644, 1555, 1424, 1380  $\text{cm}^{-1}$ ; ESI ( $\text{C}_{12}\text{H}_{13}\text{N}_4\text{O}_3\text{Br}$ ): 341.0 [ $\text{M}+\text{H}$ ] $^+$ , 363.0 [ $\text{M}+\text{Na}$ ] $^+$ .

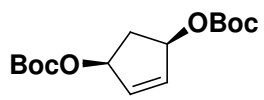


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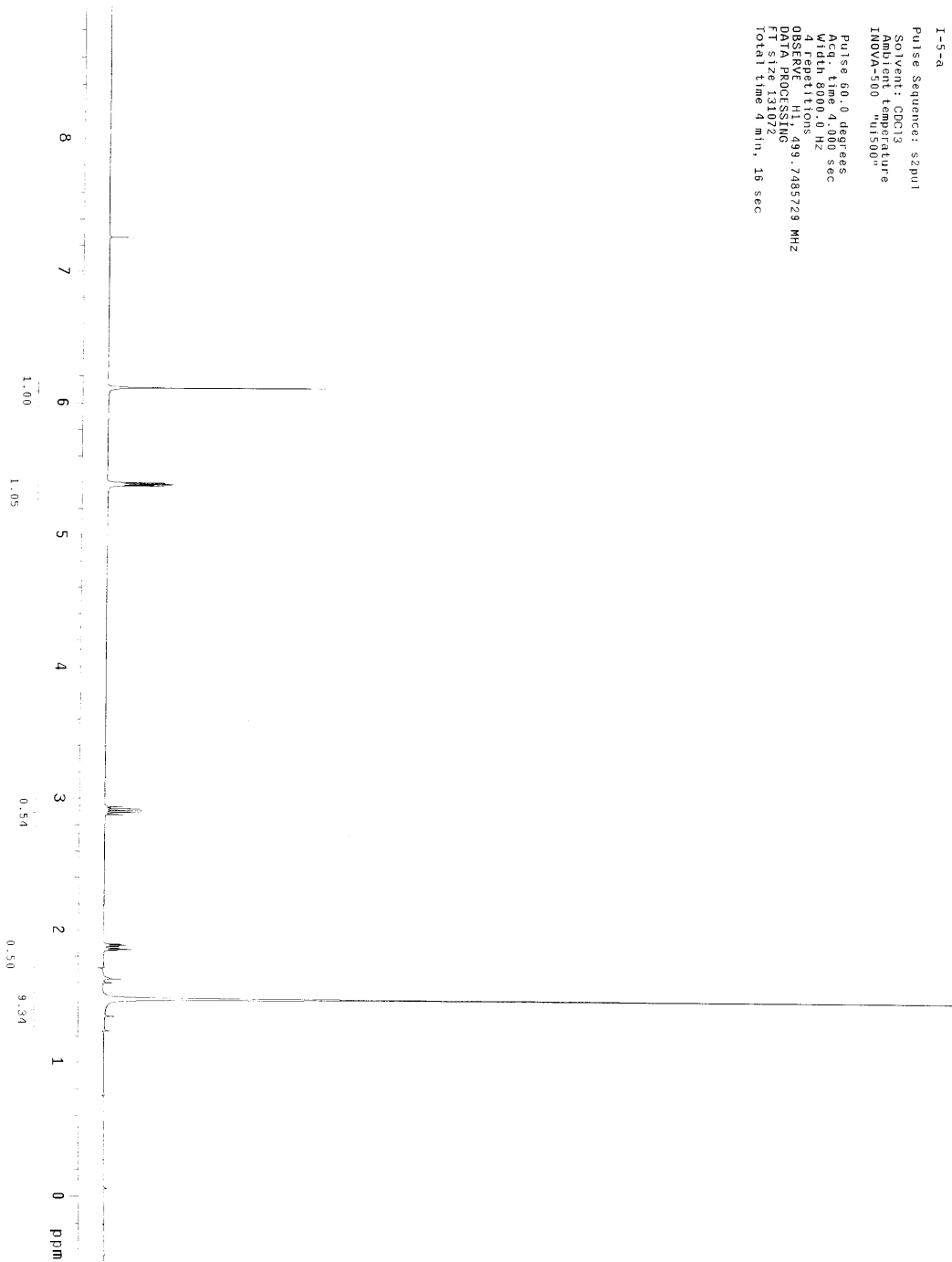
**Compound 15**: A solution of dry pyridine (0.001 ml) in dry benzene (0.01 ml) was added to a solution of *N*-sulfinyl-*p*-toluenesulfonamide (0.11 g) in dry benzene (0.19 ml) at rt. The resulting mixture was stirred at rt for 1 hr, and then it was diluted with 1

ml of toluene. The resulting suspension was added to compound **9** (22 mg) portionwise at rt. The suspension was heated at 100 °C for 40 hr. The solvents were removed under vacuum. Methanol (1.5 ml) and trimethyl phosphite (0.03 ml, 0.25 mmol) were added to the residue and the mixture was stirred at rt for 1 hr. The solvents were removed under vacuum and the residue was purified via silica gel flash column chromatography (petroleum ether / ethyl acetate = 4/1, then 3/1, then 2/1) to give an off-white foam (15 mg, 43%):  $[\alpha]_D$ : -111.7 (c 0.90, CH<sub>2</sub>Cl<sub>2</sub>);  $R_f$ : 0.2 (petroleum ether / ethyl acetate = 4/1); <sup>1</sup>H NMR (CDCl<sub>3</sub>, 500 MHz)  $\delta$  7.79 (d,  $J$  = 8.0 Hz, 2H), 7.33 (d,  $J$  = 8.0 Hz, 2H), 6.89 (d,  $J$  = 4.0 Hz, 1H), 6.29 (d,  $J$  = 4.0 Hz, 1H), 6.02 (d,  $J$  = 5.5 Hz, 1H), 5.85(m, 1H), 5.55(d,  $J$  = 6.5 Hz, 1H), 4.91 (d,  $J$  = 6 Hz, 1H), 4.69 (m, 1H), 4.62 (d,  $J$  = 6.5 Hz, 1H), 3.85 (s, 3H), 2.43 (s, 3H); <sup>13</sup>C NMR (CDCl<sub>3</sub>, 125 MHz)  $\delta$  155.4, 144.4, 136.4, 134.5, 130.9, 130.1, 127.4, 123.3, 115.3, 114.0, 105.4, 64.7, 62.3, 62.0, 59.5, 21.7; IR (film): 3189, 2926, 1651, 1548, 1428, 1334, 1161, 1094 cm<sup>-1</sup>; HRMS (C<sub>18</sub>H<sub>18</sub>N<sub>3</sub>O<sub>4</sub>SBr): Calc'd. 451. 020139 (M<sup>+</sup>), Found 451.020526.

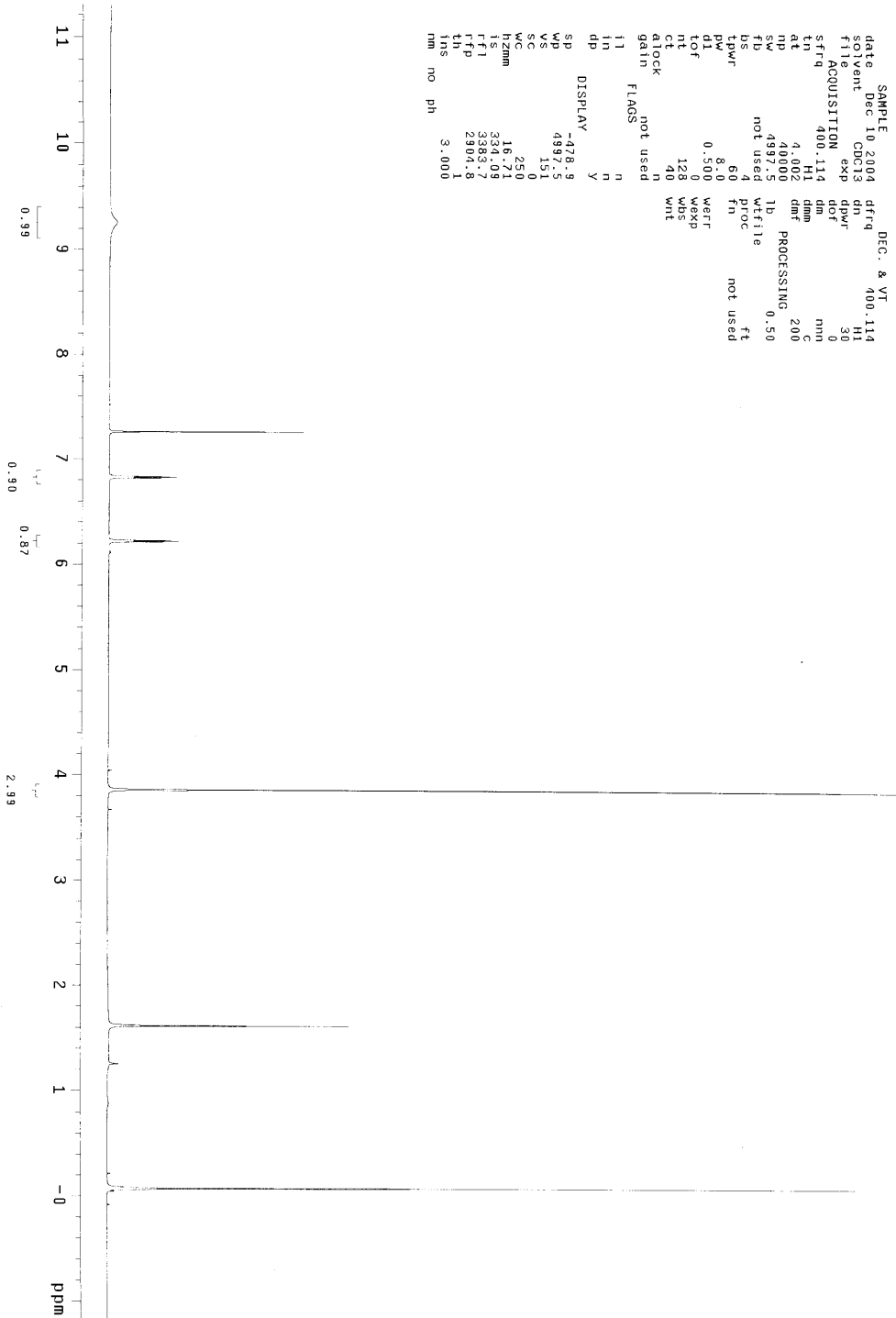
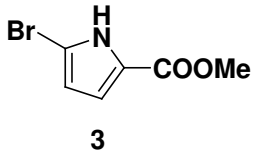




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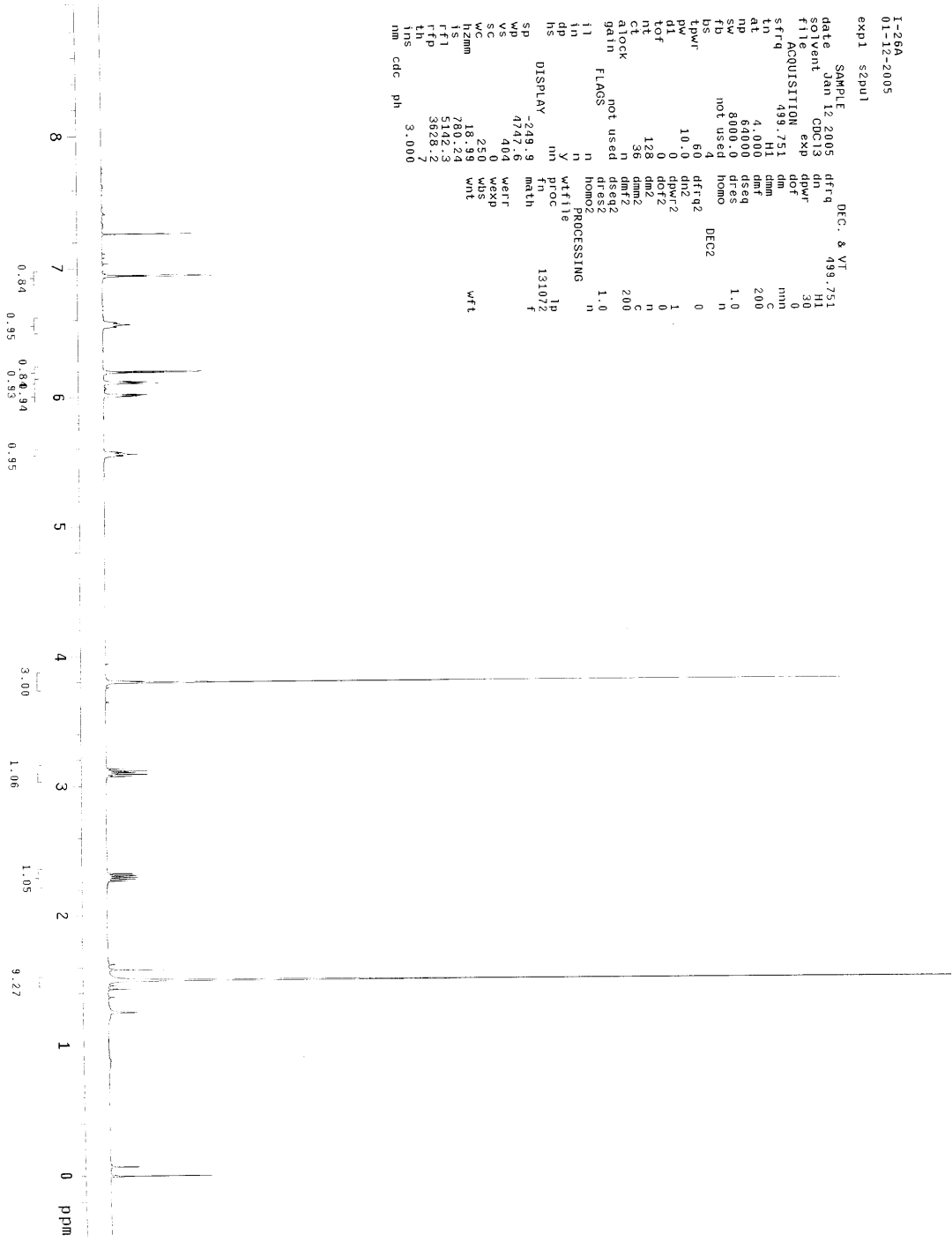
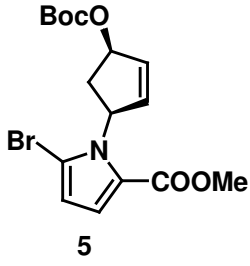
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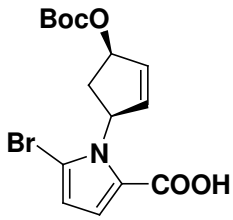


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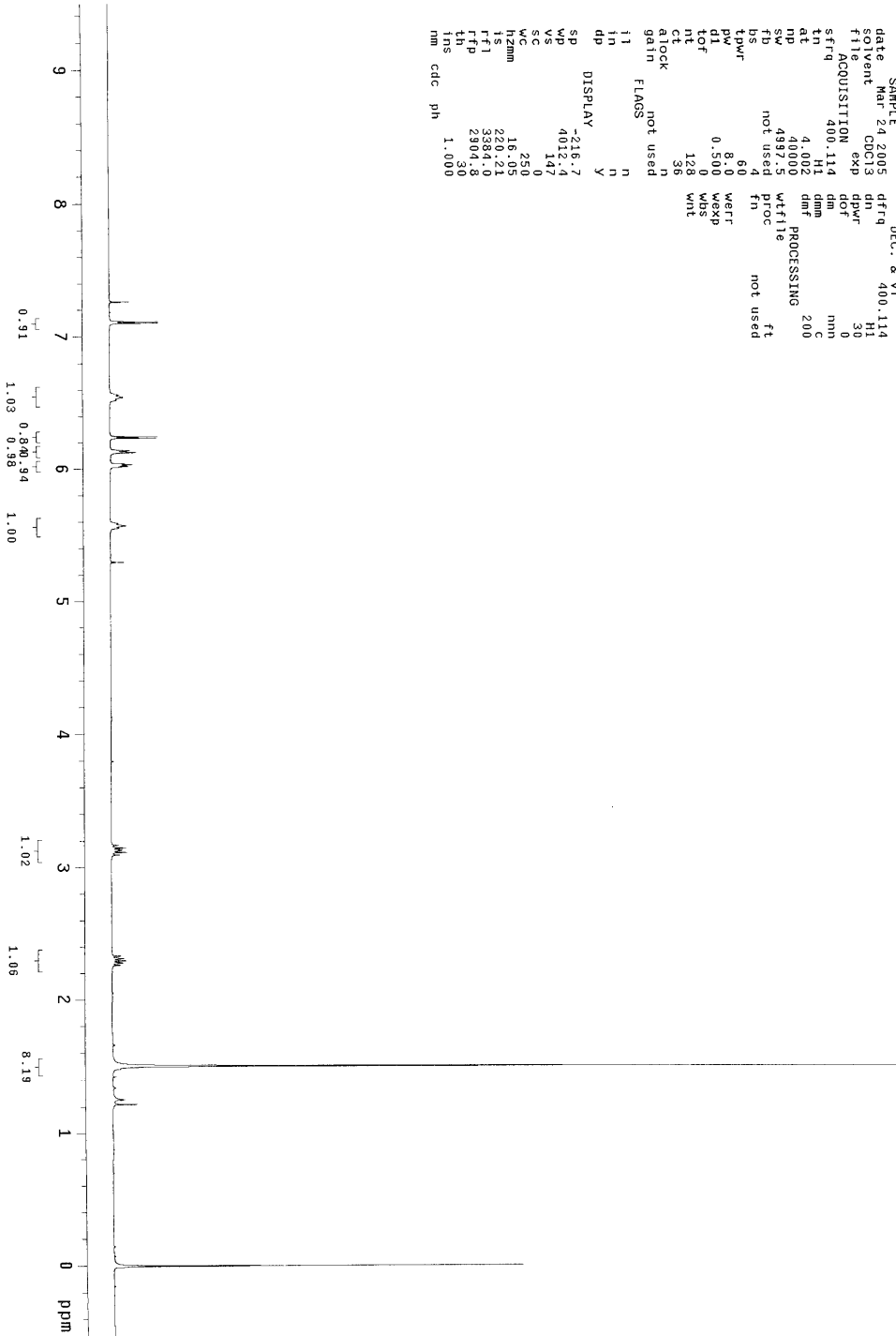
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l693       n
l694       n
l695       n
l696       n
l697       n
l698       n
l699       n
l700       n
l701       n
l702       n
l703       n
l704       n
l705       n
l706       n
l707       n
l708       n
l709       n
l710       n
l711       n
l712       n
l713       n
l714       n
l715       n
l716       n
l717       n
l718       n
l719       n
l720       n
l721       n
l722       n
l723       n
l724       n
l725       n
l726       n
l727       n
l728       n
l729       n
l730       n
l731       n
l732       n
l733       n
l734       n
l735       n
l736       n
l737       n
l738       n
l739       n
l740       n
l741       n
l742       n
l743       n
l744       n
l745       n
l746       n
l747       n
l748       n
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l758       n
l759       n
l760       n
l761       n
l762       n
l763       n
l764       n
l765       n
l766       n
l767       n
l768       n
l769       n
l770       n
l771       n
l772       n
l773       n
l774       n
l775       n
l776       n
l777       n
l778       n
l779       n
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l782       n
l783       n
l784       n
l785       n
l786       n
l787       n
l788       n
l789       n
l790       n
l791       n
l792       n
l793       n
l794       n
l795       n
l796       n
l797       n
l798       n
l799       n
l800       n
l801       n
l802       n
l803       n
l804       n
l805       n
l806       n
l807       n
l808       n
l809       n
l810       n
l811       n
l812       n
l813       n
l814       n
l815       n
l816       n
l817       n
l818       n
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l820       n
l821       n
l822       n
l823       n
l824       n
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l828       n
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l835       n
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l839       n
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l842       n
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l845       n
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l847       n
l848       n
l849       n
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l852       n
l853       n
l854       n
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l859       n
l860       n
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l869       n
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l873       n
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l877       n
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l889       n
l890       n
l891       n
l892       n
l893       n
l894       n
l895       n
l896       n
l897       n
l898       n
l899       n
l900       n
l901       n
l902       n
l903       n
l904       n
l905       n
l906       n
l907       n
l908       n
l909       n
l910       n
l911       n
l912       n
l913       n
l914       n
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l916       n
l917       n
l918       n
l919       n
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l955       n
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l962       n
l963       n
l964       n
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l967       n
l968       n
l969       n
l970       n
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l972       n
l973       n
l974       n
l975       n
l976       n
l977       n
l978       n
l979       n
l980       n
l981       n
l982       n
l983       n
l984       n
l985       n
l986       n
l987       n
l988       n
l989       n
l990       n
l991       n
l992       n
l993       n
l994       n
l995       n
l996       n
l997       n
l998       n
l999       n
1000      n
nm no ph  3.000
  
```



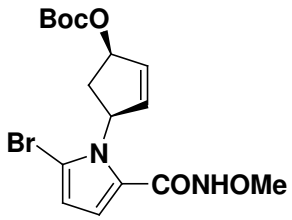


16

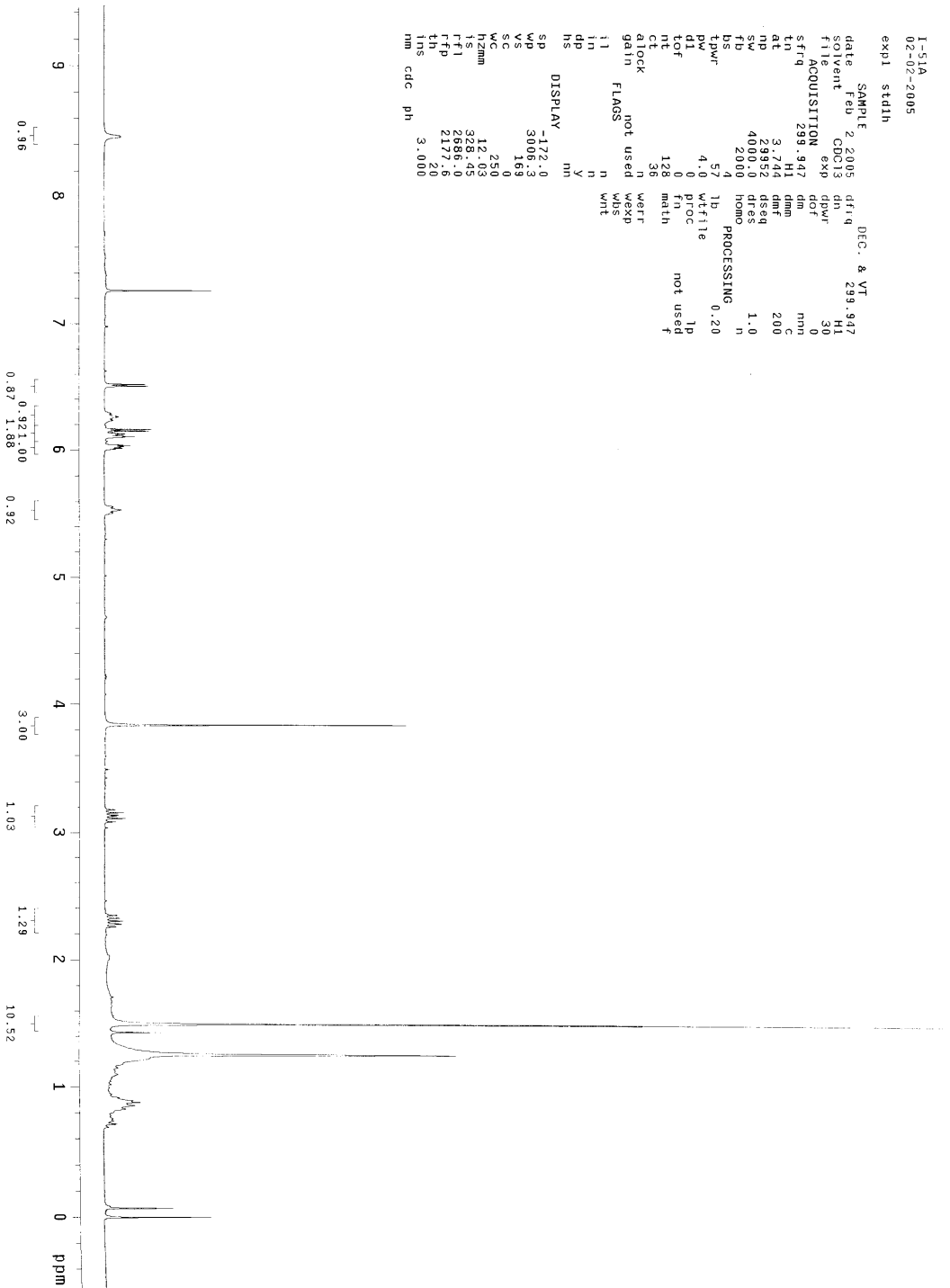


```

I-59A
03-24-2005
expt stdIn
SAMPLE
date Mar 24 2005 dfrq 400.114
solvent CDCl3 dn
file ACQUISITION exp dpr 30
sfreq 400.114 det nmn
tn H1 dmf 200
at 4.002 wtfile
np 40000 PROCESSING
sw 4997.5 not used ft
fb not used fn
ps 6.0 not used
pwr 8.0 weff
d1 0.500 wexp
tof 0 wbs
nt 128 wnt
ct 36
atlock not used
gain not used
f1 n
f2 n
f3 n
f4 n
f5 n
f6 n
f7 n
f8 n
f9 n
f10 n
f11 n
f12 n
f13 n
f14 n
f15 n
f16 n
f17 n
f18 n
f19 n
f20 n
f21 n
f22 n
f23 n
f24 n
f25 n
f26 n
f27 n
f28 n
f29 n
f30 n
nm cdc ph 1.000
  
```



6

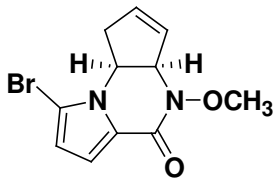


```

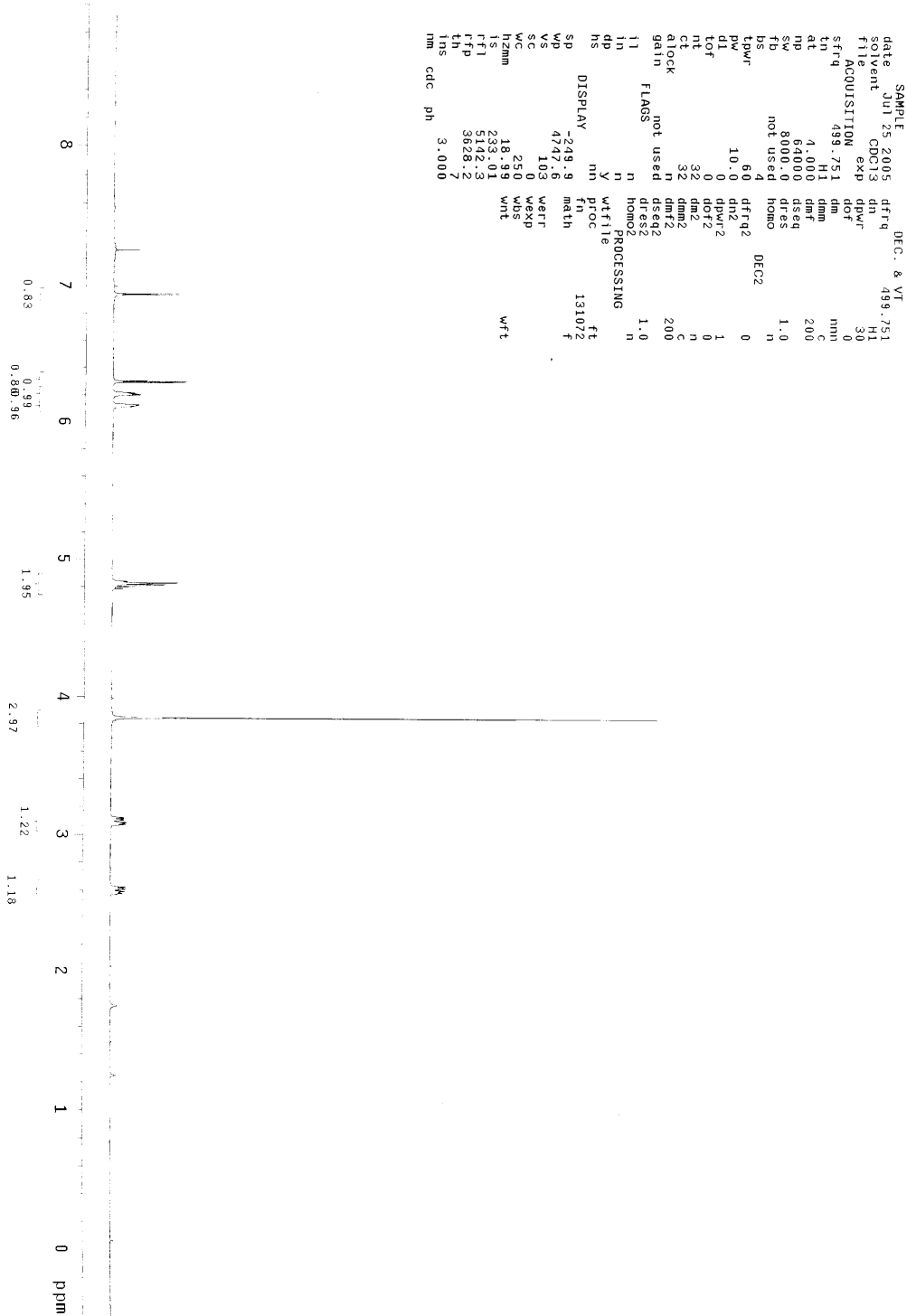
I-51A
02-02-2005
expt stdth

SAMPLE 2.2005 DFC. & VI
date Feb 02 2005 dfreq 299.947
solvent CDCl3 dn H1
file exp dpwr 30
ACQUISITION exp dpwr 0
sfrq 299.947 dm rnm
at 3.744 dmf 200
np 29952 dres 1.0
sw 4000.0 dres n
fb 2000 homo n
ds 57 1b PROCESSING 0.20
tpwr 4.0 wfc 1p
pw 0 wfc 1p
tdf 0 fn not used
nt 128 math f
ct 36
atlock n werr
gain not used wexp
flags n wds
il n wts
in n
dp y
hs n

DISPLAY -172.0
SP 3109.3
WD 1.63
SC 0
WC 250
hzmm 12.03
ts 388.45
rfi 2995.0
rfp 2177.20
tfs 3.000
nm cdc ph
  
```



7



```

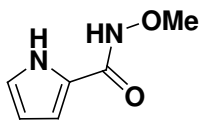
II-75A
07-25-2005
exp1 szput

SAMPLE          CDC13
date Jul 25 2005
solvent CDCl3
file ACQUISITION exp
sfreq 493.751
in H1
at 4.000
np 64000
sw 8000.0
fb not used
ds 4
cpwr 60
dv 10.0
tof 0
nt 32
ct 32
alock n
gain not used
11 n
12 n
13 n
14 n
15 n
16 n
17 n
18 n
19 n
20 n
21 n
22 n
23 n
24 n
25 n
26 n
27 n
28 n
29 n
30 n
31 n
32 n
33 n
34 n
35 n
36 n
37 n
38 n
39 n
40 n
41 n
42 n
43 n
44 n
45 n
46 n
47 n
48 n
49 n
50 n
51 n
52 n
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76 n
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78 n
79 n
80 n
81 n
82 n
83 n
84 n
85 n
86 n
87 n
88 n
89 n
90 n
91 n
92 n
93 n
94 n
95 n
96 n
97 n
98 n
99 n
100 n

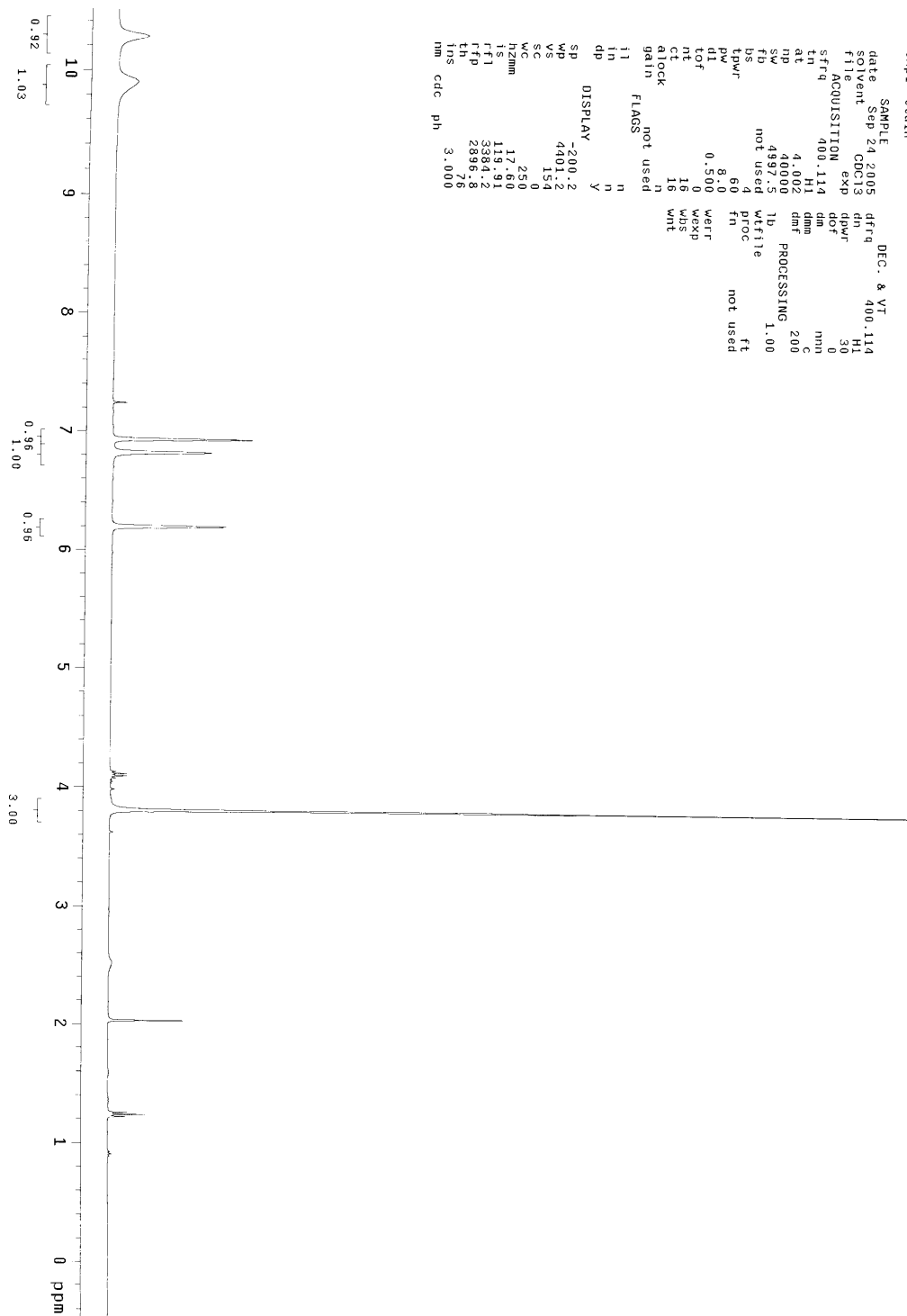
DISPLAY -249.9 nm
          4747.6 warr
          103 wexp
          0 wbs
          18.93 wlt
          239.91
          5142.3
          3628.2
          7
          3.000 nm cdc ph

DEC. & VI
499.751
H1
30
0
nm
nm
200
1.0
n
0
DEC2
0
1
n
n
C
200
1.0
n
n
PROCESsing
n
ft
131072
f
math

```



17

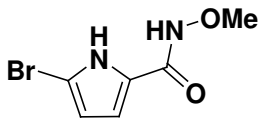


```

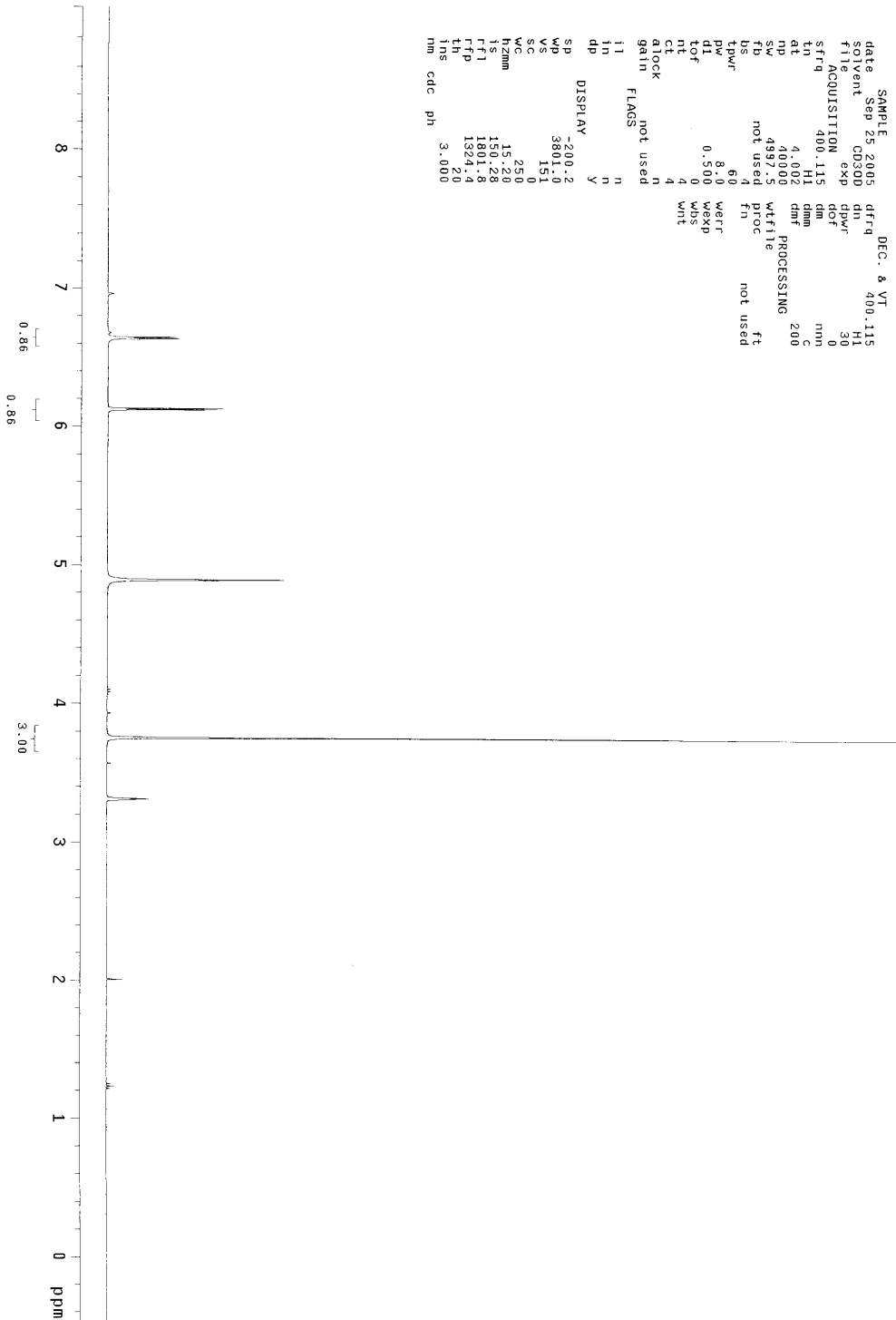
111-73A
09-24-2005
expi sidlh

SAMPLE
date Sep 24 2005 DEC. & VT
solvent CDC13 dh 400.114
file CDC13 exp dpwr H1
ACQUISITION exp dpwr 30
sfrq 400.114 dof 0
tn H1 nm
at 4.002 dmf PROCESSING 200
np 400000 fd 1.00
sw 4997.5 lb wifile
fd not used wifile
uswr 4 proc ft
pw 80 fn not used
dl 0.500 weft
tof 0 wexp
nt 16 wbs
ct 16 wnt
alock n
gain not used
fl n
in n
dp y

DISPLAY
sp -200.2
wp 4401.2
s 154
wc 0
h2mm 17.60
rs 119.91
rfi 3384.2
rfp 2896.8
tn 76
nm 3.000
cdc ph
  
```



8

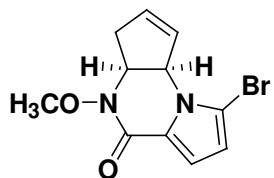


```

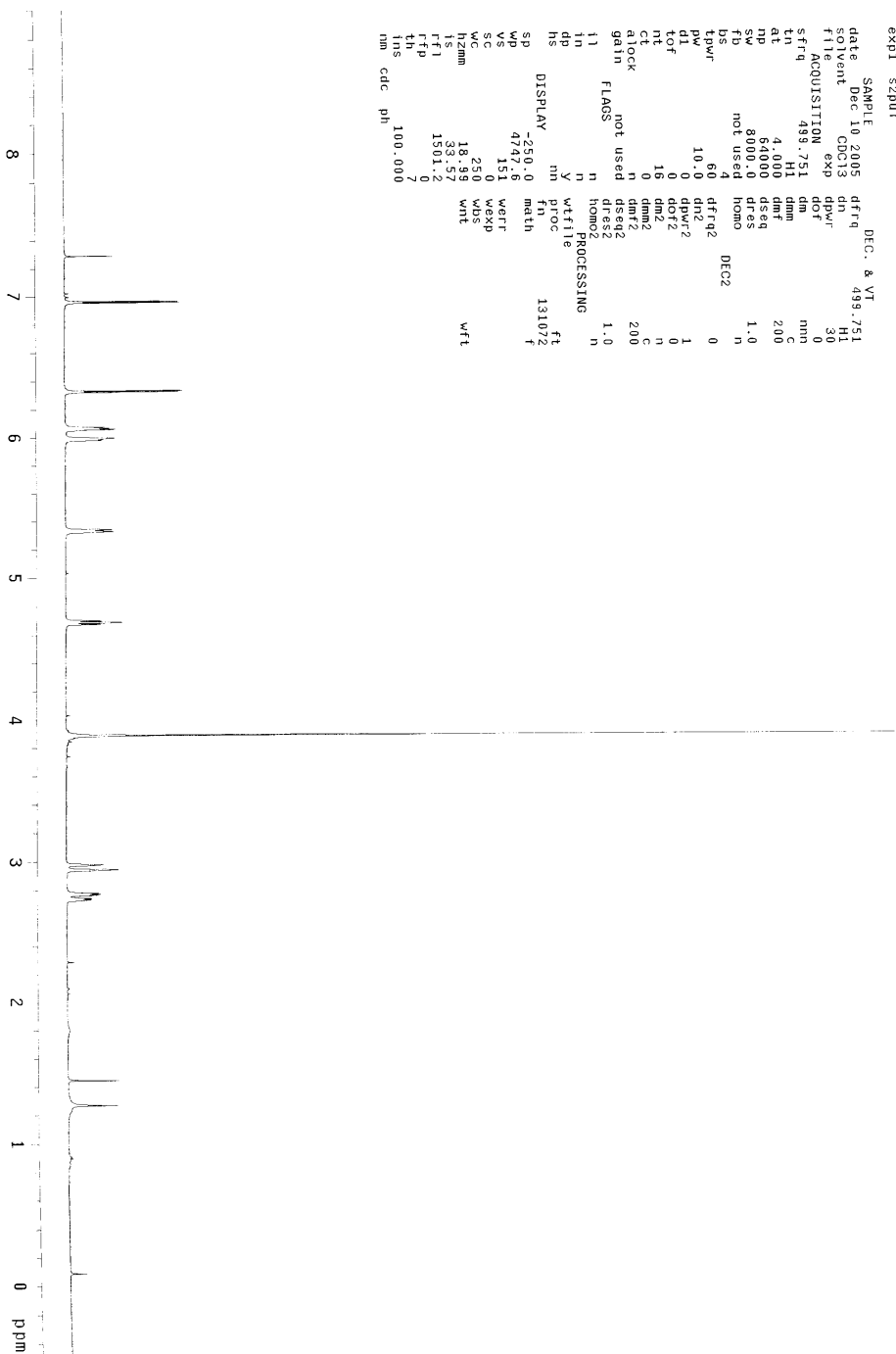
III-74A-Me00
09-25-2005
expt std/h

SAMPLE          DEC. & VT
date Sep 25 2005 dfrq 400.115
solvent CD300 dn H1
file OD300 exp dpwr 30
ACQUISITION    exp dof 0
sfrq 400.115 dm mn C
dn 4.011 dmm 200
nt 40000 wfile PROCESSING
sw 4997.5 wtfile ft
fb not used proc fn not used
bs 60 tpwr 8.0 wefr
pw 8.0 wefr
ql 0.500 wefr
nt 4 wnt
ct 4
atlock not used
gain not used
FLAGS          n
i1 n
i2 n
i3 n
i4 n
i5 n
i6 n
i7 n
i8 n
i9 n
i10 n
i11 n
i12 n
i13 n
i14 n
i15 n
i16 n
i17 n
i18 n
i19 n
i20 n
i21 n
i22 n
i23 n
i24 n
i25 n
i26 n
i27 n
i28 n
i29 n
i30 n
i31 n
i32 n
i33 n
i34 n
i35 n
i36 n
i37 n
i38 n
i39 n
i40 n
i41 n
i42 n
i43 n
i44 n
i45 n
i46 n
i47 n
i48 n
i49 n
i50 n
i51 n
i52 n
i53 n
i54 n
i55 n
i56 n
i57 n
i58 n
i59 n
i60 n
i61 n
i62 n
i63 n
i64 n
i65 n
i66 n
i67 n
i68 n
i69 n
i70 n
i71 n
i72 n
i73 n
i74 n
i75 n
i76 n
i77 n
i78 n
i79 n
i80 n
i81 n
i82 n
i83 n
i84 n
i85 n
i86 n
i87 n
i88 n
i89 n
i90 n
i91 n
i92 n
i93 n
i94 n
i95 n
i96 n
i97 n
i98 n
i99 n
i100 n
nm cdc ph 3.000
  
```





9

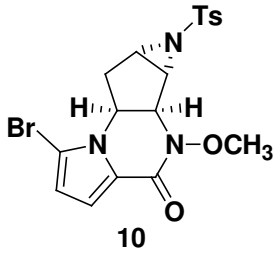


```

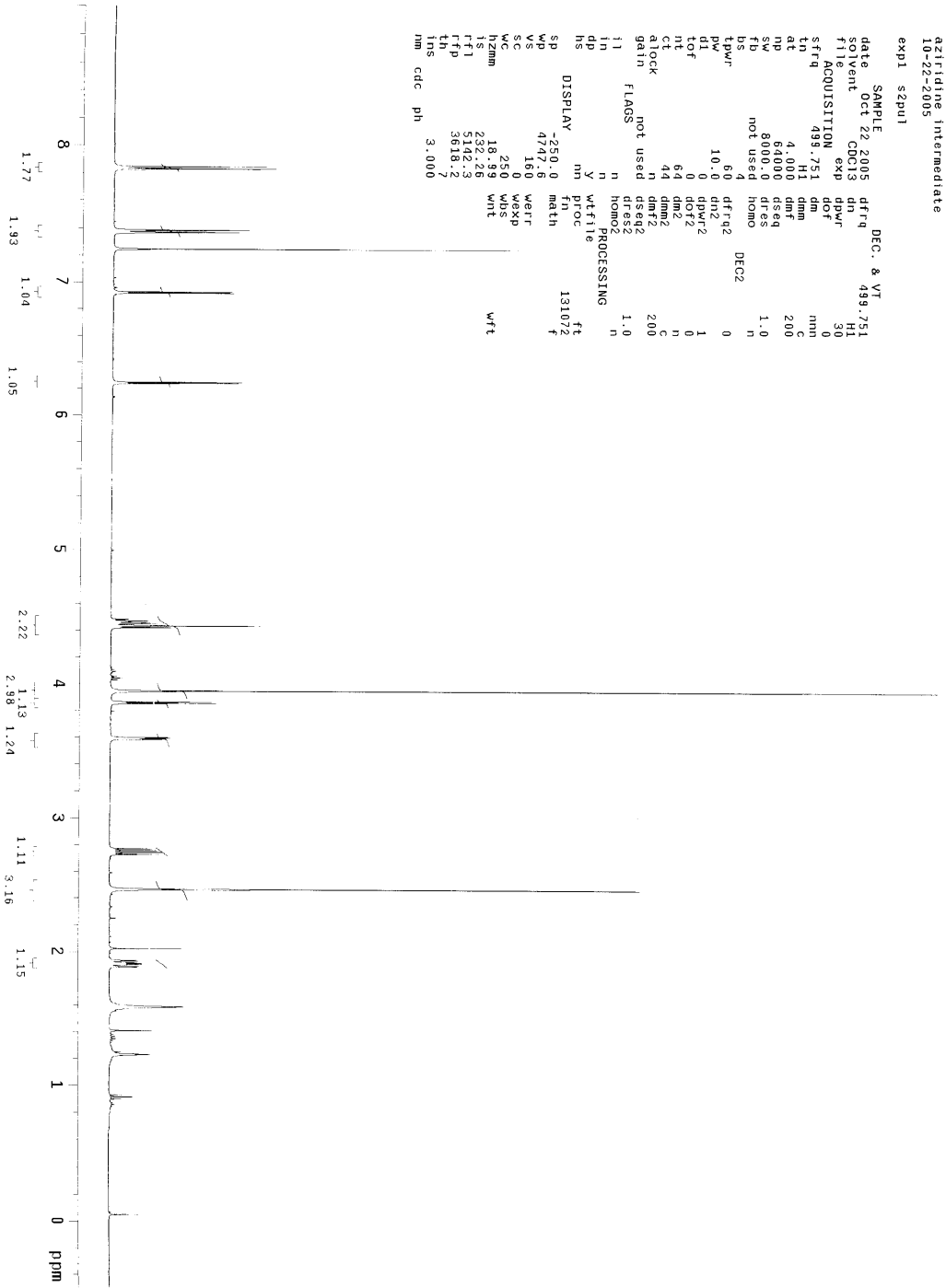
IV-93A
12-10-2005
expt1 szpwt1

SAMPLE DEC: & VT
date Dec 10 2005 dfrq 499.751
solvent CDCl3 499.751 H1
F1 ACQUISITION exp dof 30
sfrq 499.751 dm 0
tn H1 dnm 200
at 4.000 dmf
mp 4.000 dfrs
sw 8000.0 dres 1.0
fb not used home n
bs 4 dfrq2 DEC2 0
tpwr 60 dnt 1
pw 10.0 dnf2 0
d1 0 dnf2 0
scf 0 dnf2 0
ncr 16 dnm2 n
ct 0 dnm2 n
atlock not used dseq2 1.0
gain not used dres2 n
f1 n home PROCESSING n
f2 n wfile n
f3 n m proc n
f4 n m file n
f5 n m file n
hs DISPLAY 250.0 math 131072
sp 250.0
wp 474.51 warr ft
ws 150 wexp 1
sc 0 wds wht
wc 250 wht
hznam 18.99
ls 33.52
ff 1301.6
ftd 7
th 7
ins 100.000
nm cdc ph

```



10

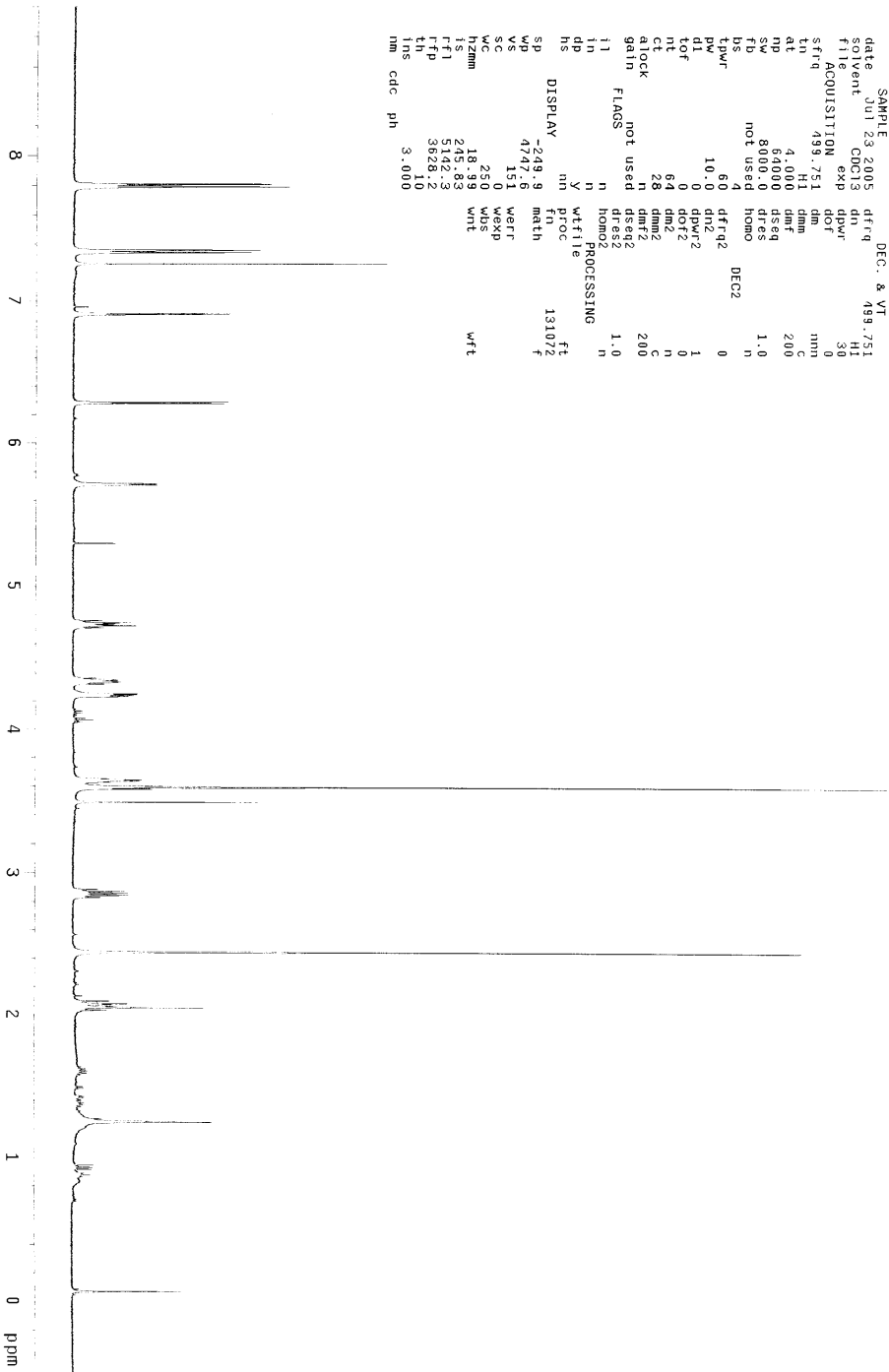
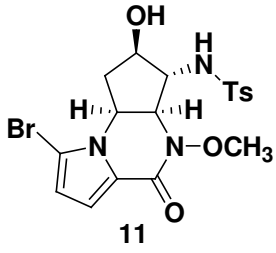


```

SAMPLE      DEC. & VI
date      Oct 22 2005      dfrq      499.751
solvent    CDCl3           dmf2      1
file       G013          dpwrf     30
ACQUISITION exp          dof        0
sfrq      499.751      dm          mm
tn        4.000      dmf2       C
dt        4.000      dseq      200
td        4.000      dseq      200
sw        8000.0      dseq      1.0
fb        not used    homo
bs        not used    homo
tpwr      60          dfrq2     0
pw        10.0      dmf2     1
d1        0          dmf2     1
d2        0          dmf2     1
d3        0          dmf2     1
d4        64         dmf2     1
ct        44         dmf2     C
atlock    gain      n         C
gain      not used   dseq2    200
f1        n         dseq2    1.0
f2        n         dseq2    1.0
f3        n         dseq2    1.0
f4        n         dseq2    1.0
f5        n         dseq2    1.0
f6        n         dseq2    1.0
f7        n         dseq2    1.0
f8        n         dseq2    1.0
f9        n         dseq2    1.0
f10       n         dseq2    1.0
f11       n         dseq2    1.0
f12       n         dseq2    1.0
f13       n         dseq2    1.0
f14       n         dseq2    1.0
f15       n         dseq2    1.0
f16       n         dseq2    1.0
f17       n         dseq2    1.0
f18       n         dseq2    1.0
f19       n         dseq2    1.0
f20       n         dseq2    1.0
f21       n         dseq2    1.0
f22       n         dseq2    1.0
f23       n         dseq2    1.0
f24       n         dseq2    1.0
f25       n         dseq2    1.0
f26       n         dseq2    1.0
f27       n         dseq2    1.0
f28       n         dseq2    1.0
f29       n         dseq2    1.0
f30       n         dseq2    1.0
f31       n         dseq2    1.0
f32       n         dseq2    1.0
f33       n         dseq2    1.0
f34       n         dseq2    1.0
f35       n         dseq2    1.0
f36       n         dseq2    1.0
f37       n         dseq2    1.0
f38       n         dseq2    1.0
f39       n         dseq2    1.0
f40       n         dseq2    1.0
f41       n         dseq2    1.0
f42       n         dseq2    1.0
f43       n         dseq2    1.0
f44       n         dseq2    1.0
f45       n         dseq2    1.0
f46       n         dseq2    1.0
f47       n         dseq2    1.0
f48       n         dseq2    1.0
f49       n         dseq2    1.0
f50       n         dseq2    1.0
f51       n         dseq2    1.0
f52       n         dseq2    1.0
f53       n         dseq2    1.0
f54       n         dseq2    1.0
f55       n         dseq2    1.0
f56       n         dseq2    1.0
f57       n         dseq2    1.0
f58       n         dseq2    1.0
f59       n         dseq2    1.0
f60       n         dseq2    1.0
f61       n         dseq2    1.0
f62       n         dseq2    1.0
f63       n         dseq2    1.0
f64       n         dseq2    1.0
f65       n         dseq2    1.0
f66       n         dseq2    1.0
f67       n         dseq2    1.0
f68       n         dseq2    1.0
f69       n         dseq2    1.0
f70       n         dseq2    1.0
f71       n         dseq2    1.0
f72       n         dseq2    1.0
f73       n         dseq2    1.0
f74       n         dseq2    1.0
f75       n         dseq2    1.0
f76       n         dseq2    1.0
f77       n         dseq2    1.0
f78       n         dseq2    1.0
f79       n         dseq2    1.0
f80       n         dseq2    1.0
f81       n         dseq2    1.0
f82       n         dseq2    1.0
f83       n         dseq2    1.0
f84       n         dseq2    1.0
f85       n         dseq2    1.0
f86       n         dseq2    1.0
f87       n         dseq2    1.0
f88       n         dseq2    1.0
f89       n         dseq2    1.0
f90       n         dseq2    1.0
f91       n         dseq2    1.0
f92       n         dseq2    1.0
f93       n         dseq2    1.0
f94       n         dseq2    1.0
f95       n         dseq2    1.0
f96       n         dseq2    1.0
f97       n         dseq2    1.0
f98       n         dseq2    1.0
f99       n         dseq2    1.0
f100      n         dseq2    1.0
  
```

aziridine intermediate  
10-22-2005

expi s2pui



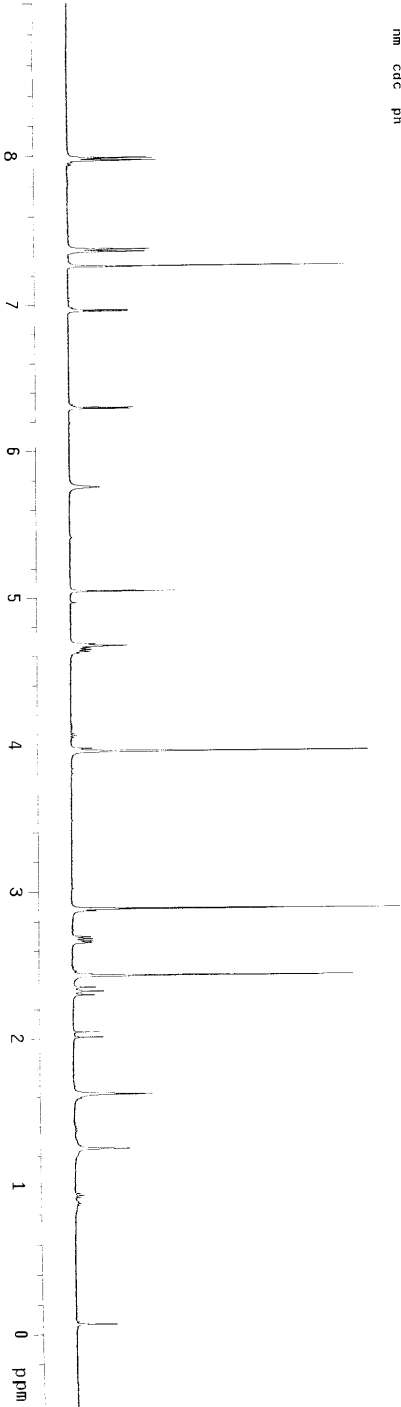
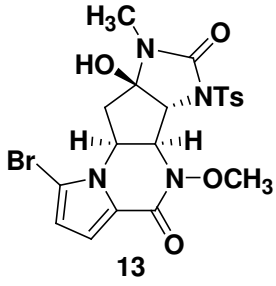
```

II-69A
07-25-2005
expt1 szpu1

SAMPLE          date       Jul 23 2005
SOLVENT         CDCl3
F1 ACQUISITION exp 1
SFRQ            499.751 dm
IN              HI dm
AT              4.000 dmf
MP              8000.0 dseq
FW              not used
BS              not used
PW              60 dfrq2
DL              10.0 dh2
DQ              0 dpwr2
RG              0 dpr2
CT              28 dm2
ALOCK           not used
GAIN            not used
I1             n
I2             n
I3             n
I4             n
I5             n
I6             n
I7             n
I8             n
I9             n
I10            n
I11            n
I12            n
I13            n
I14            n
I15            n
I16            n
I17            n
I18            n
I19            n
I20            n
I21            n
I22            n
I23            n
I24            n
I25            n
I26            n
I27            n
I28            n
I29            n
I30            n
I31            n
I32            n
I33            n
I34            n
I35            n
I36            n
I37            n
I38            n
I39            n
I40            n
I41            n
I42            n
I43            n
I44            n
I45            n
I46            n
I47            n
I48            n
I49            n
I50            n
I51            n
I52            n
I53            n
I54            n
I55            n
I56            n
I57            n
I58            n
I59            n
I60            n
I61            n
I62            n
I63            n
I64            n
I65            n
I66            n
I67            n
I68            n
I69            n
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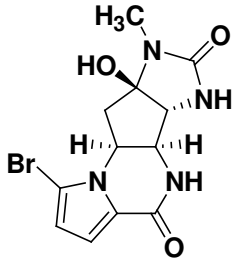


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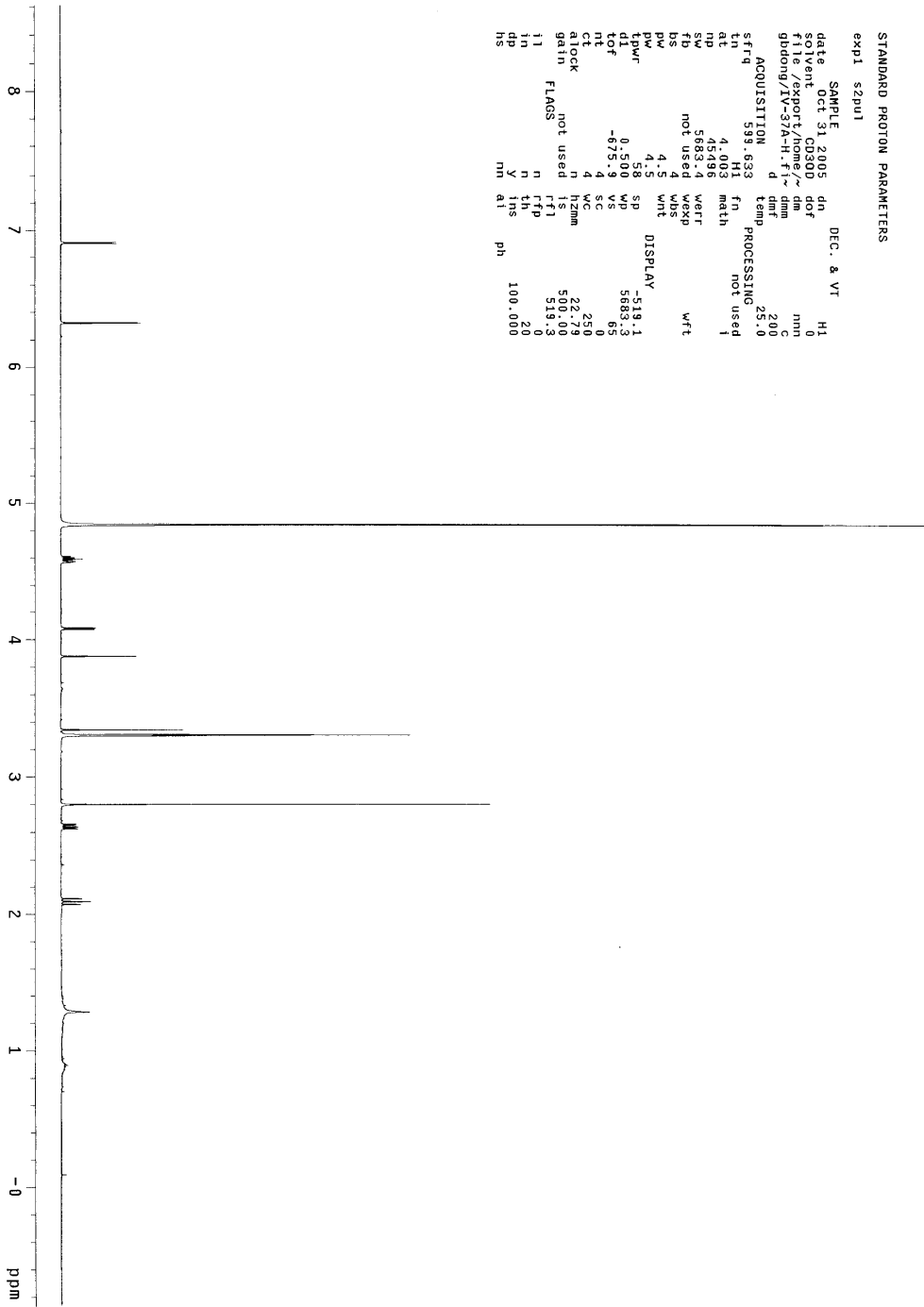
VT-1A-pure
10-10-2005
exp1 s2pul1

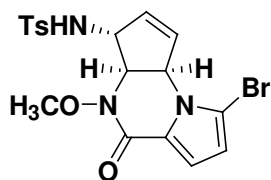
SAMPLE DEC: & VT
date Oct 11 2005 dfrq 499.751
solvent CDC13 nm 401
F1 ACQUISITION exp 0
F2 ACQUISITION exp 0
sfrq 499.751 dm
tn H1 dnm
at 4.000 dnmf
np 64000 dseq
sw 8000.0 dres
TD not used homo
DECO2
tpr 60 dfrq2
pw 10.0 dn2
d1 0 dpr2 1
tof 0 dcf2 0
nt 64 dmf 0
ct 64 dmf2 0
clock gain not used dseq2 200
dres2 1.0
i1 n homo2
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nm cdc ph
  
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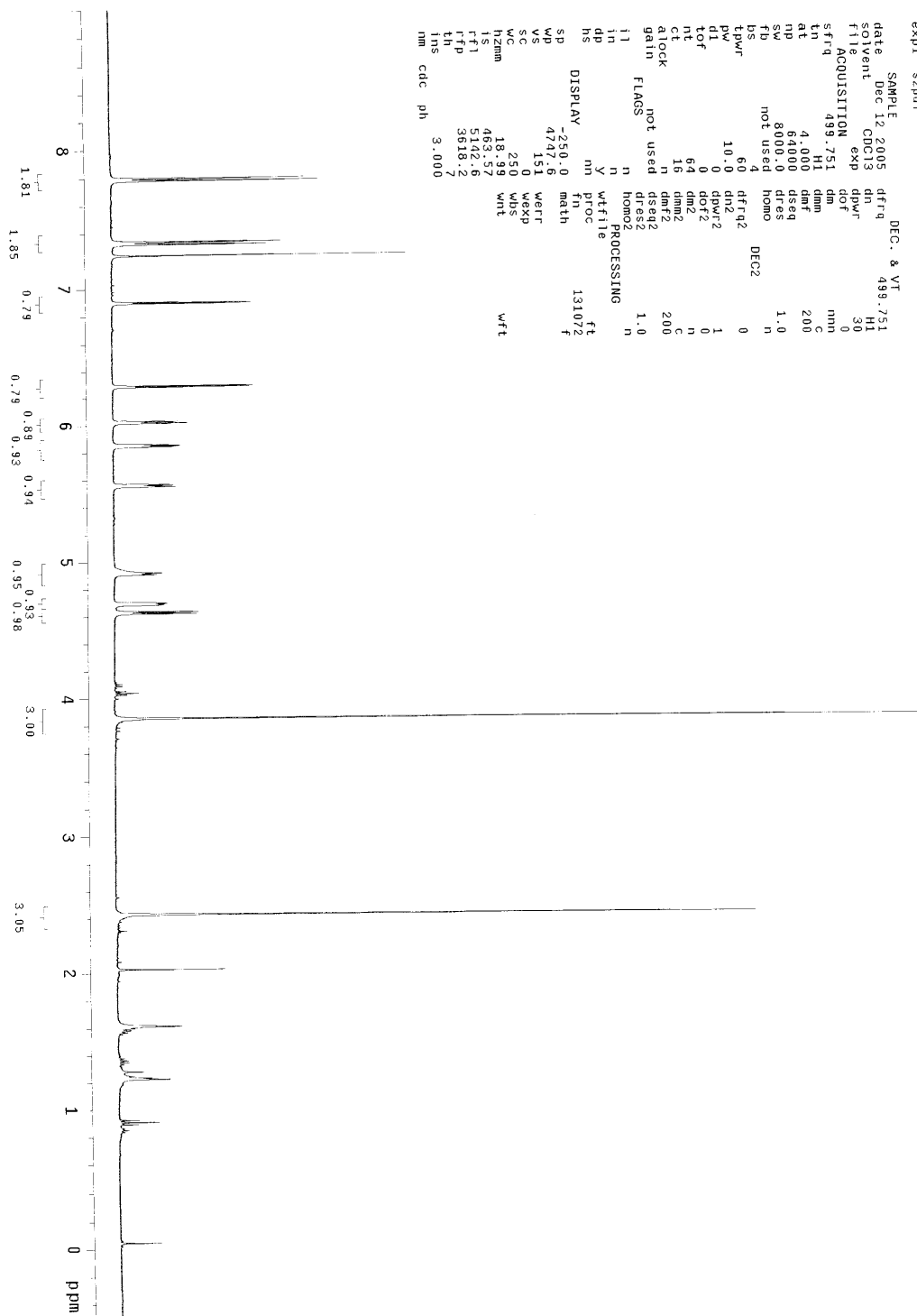


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V-7A  
12-12-2005

exp1 s2pu1

SAMPLE Dec 12 2005 dfc: 8 vt  
date Dec 12 2005 dfc: 8 vt  
solvent CDCl3 dn H1  
f1 ACQUISITION exp dpwr 30  
f1 ACQUISITION dof 0  
sfrq 499.751 dm nmh  
tn H1 dmm C  
at 4.000 dmf 200  
np 64000 dseg  
sw 8000 dres  
fd not used homo 1.0  
fb 4  
tpwr 60 dfrc2 DEC2  
pw 10.0 dn2 0  
d1 0 dpwr2 1  
tof 0 dof2 0  
nt 64 dmm2 n  
ct 16 dmm2 n  
atlock n dseg2 200  
gain not used dres2 1.0  
f1 n homo2 n  
in y wfile PROCESSING  
dp y wfile  
hs nm proc 131072  
DISPLAY -250.0 math ft  
wp 474.51 wpr 1  
s 151 wexp  
wc 250 wbs  
h2mm 18.39 wnt wfl  
ts 463.57  
f1 5142.6  
f1p 3618.2  
th 3.000  
ims cdc ph