Supporting Information

Synthesis of the Core Structure of Acutumine

Matthew D. Reeder, G. S. C. Srikanth, Spencer B. Jones, and Steven L. Castle*

Department of Chemistry and Biochemistry
Brigham Young University, Provo, Utah, 84602

General Experimental Details

Ether, methanol, methylene chloride, tetrahydrofuran, and toluene were dried by passage through a Glass Contour solvent drying system containing cylinders of activated alumina.[1] Flash chromatography was carried out using 60–230 mesh silica gel. ^{1}H NMR spectra were obtained on Varian 200, 300, or 500 MHz spectrometers with chloroform (7.27 ppm) or benzene (7.16 ppm) as internal reference. Signals are reported as follows: s (singlet), d (doublet), t (triplet), q (quartet), dd (doublet of doublets), dt (doublet of triplets), br s (broad singlet), m (multiplet). Coupling constants are reported in hertz (Hz). ^{13}C NMR spectra were obtained on Varian spectrometers operating at 50, 75, or 125 MHz with chloroform (77.23 ppm) or benzene (128.06 ppm) as internal reference. Infrared spectra were obtained on a Nicolet Avatar 360 FT-IR Spectrometer. Mass spectral data (EI, FAB) and were obtained from the Brigham Young University mass spectrometry facility. X-ray crystallographic data were obtained from the Brigham Young University X-ray crystallography facility.

[1] Pangborn, A. B.; Giardello, M. A.; Grubbs, R. H.; Rosen, R. K.; Timmers, F. J. *Organometallics* **1996**, *15*, 1518.

Methyl 3-(2,3,4-Trimethoxyphenyl)propionate (7). A solution of *trans*-2,3,4-trimethoxycinnamic acid (**6**) (5.50 g, 23.1 mmol) in anhydrous CH_3OH (80 mL) was treated with conc H_2SO_4 (0.8 mL) and stirred at reflux for 12 h. The resulting mixture was diluted with H_2O (300 mL) and extracted with $CHCl_3$ (4 × 78 mL). The combined organic layers were washed with satd aq $NaHCO_3$ (100 mL), dried ($MgSO_4$), and concentrated in vacuo. The resultant crude methyl ester was dissolved in anhydrous CH_3OH (22 mL) under Ar, treated with 10% Pd/C (0.11 g, 0.02 wt equiv), and stirred at rt under H_2 (1 atm) for 18 h. The mixture was filtered through a pad of Celite and concentrated in vacuo, affording **7** (5.49 g, 21.6 mmol, 94%) as an amber oil: ^{1}H NMR ($CDCl_3$, 300 MHz) δ 6.84 (d, *J* = 8.4 Hz, 1H), 6.59 (d, *J* = 8.4 Hz, 1H), 3.89 (s, 3H), 3.86 (s, 3H), 3.84 (s, 3H), 3.67 (s, 3H), 2.88 (t, *J* = 8.1 Hz, 2H), 2.58 (t, *J* = 8.4 Hz, 2H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 173.8, 152.6, 152.0, 142.3, 126.5 123.9, 107.2, 61.0, 60.8, 56.1, 51.7, 35.1, 25.6; IR (film) ν_{max} 2946, 1735, 1455, 1275, 1177, 1095 cm^{-1}; HRMS (FAB) *m/z* 277.1064 (MNa^+, $C_{13}H_{18}O_5Na$ requires 277.1052).

2-Methyl-4-(2,3,4-trimethoxyphenyl)-butan-2-ol (8). A flask charged with Mg turnings (cleaned by rinsing with 1 N HCl, H_2O, and acetone, then dried overnight at 110 °C, 1.89 g, 79 mmol) was treated with a trace amount of I_2, and the turnings were stirred vigorously until discoloration of Mg by the iodine occurred. Then, anhydrous Et_2O (50 mL) was added, after which methyl iodide (1.8 mL, 9.9 mmol) was added to the resulting light yellow solution at rt under N_2. When the solution began refluxing, the mixture was cooled to 0 °C. Methyl iodide was added every 5 min in 1.0 mL portions until a total of 4.8 mL (11.0 g, 79 mmol) had been added. The mixture was stirred at 0 °C for 30 min, after which a solution of **7** (5.2 g, 20 mmol) in anhydrous Et_2O (5 mL) was added in 0.25

mL portions every 3 min. The resulting mixture was then stirred at 0 °C for 20 min, and the reaction was quenched with 0.1 N HCl (35 mL), extracted with Et_2O (4 × 10 mL), dried ($MgSO_4$), and concentrated in vacuo. Flash chromatography (3% acetone–hexanes elution) yielded **8** (4.3 g, 17 mmol, 85%) as a white solid: ^{1}H NMR ($CDCl_3$, 300 MHz) δ 6.84 (d, *J* = 8.4 Hz, 1H), 6.61 (d, *J* = 8.4 Hz, 1H), 3.89 (s, 3H), 3.86 (s, 3H), 3.83 (s, 3H), 2.67–2.60 (m, 2H), 1.75–1.69 (m, 2H), 1.68–1.57 (br s, 1H), 1.28 (s, 6H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 152.2, 151.9, 142.5, 128.8, 123.8, 107.6, 71.1, 61.1, 60.9, 56.2, 45.3, 29.4 (2C), 24.9; IR (film) ν_{max} 3440, 2956, 1600, 1465, 1276, 1096 cm^{-1}; HRMS (FAB) *m/z* 277.1433 (MNa^+, $C_{14}H_{22}O_4Na$ requires 277.1415).

4,5,6-Trimethoxy-1,1-dimethylindan (9). A solution of **8** (3.00 g, 11.8 mmol) in anhydrous Et_2O (5 mL, syringe rinsed with an additional 0.5 mL) was added dropwise to conc H_2SO_4 (6.5 g, 66 mmol) at 0 °C. The resulting brown mixture was stirred at 0 °C for 1 h, then diluted with H_2O (20 mL, brown color disappeared) and Et_2O (10 mL). The layers were separated, and the aqueous layer was extracted with Et_2O (5 mL). The combined organic layers were washed with satd aq K_2CO_3 (5 mL) and H_2O (5 mL), then dried ($MgSO_4$) and concentrated in vacuo. Flash chromatography (10% acetone–hexanes elution) afforded **9** (2.36 g, 9.99 mmol, 85%) as a light yellow oil: ^{1}H NMR ($CDCl_3$, 200 MHz) δ 6.43 (s, 1H), 3.89 (s, 3H), 3.85 (s, 3H), 3.84 (s, 3H), 2.82 (t, *J* = 8.0 Hz, 2H), 1.90 (t, *J* = 8.0 Hz, 2H), 1.22 (s, 6H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 153.2, 149.6, 148.6, 140.6, 126.8, 101.5, 61.2, 60.5, 56.5, 44.7, 41.7, 28.8 (2C), 26.9; IR (film) ν_{max} 2944, 1592, 1465, 1328, 1207, 1113, 1042 cm^{-1}; HRMS (EI) *m/z* 236.1398 (MH^+, $C_{14}H_{20}O_3$ requires 236.1538).

5,6,7-Trimethoxy-3,3-dimethylindan-4-carbaldehyde (10). A solution of hexamethylenetetramine (0.42 g, 3.0 mmol) in trifluoroacetic acid (5 mL) was treated slowly with **9** (0.35 g, 1.5 mmol). The resulting mixture was refluxed for 12 h. Water (20 mL) was then added with stirring, and the mixture was stirred at 50 °C for 1 h and subsequently cooled to 10–20 °C. The mixture was extracted with EtOAc (2 × 50 mL), and the combined organic layers were washed with brine (25 mL), dried ($MgSO_4$), and concentrated in vacuo. Flash chromatography (3% acetone–hexanes elution) yielded **10** (0.32 g, 1.2 mmol, 82%) as a yellow oil: ^{1}H NMR ($CDCl_3$, 200 MHz) δ 10.36 (s, 1H), 4.01 (s, 3H), 3.94 (s, 3H), 3.87 (s, 3H), 2.81 (t, *J* = 8.0 Hz, 2H), 1.92 (t, *J* = 8.0 Hz, 2H), 1.37 (s, 6H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 192.3, 158.3, 154.8, 150.2, 143.6, 132.7, 122.2, 62.6, 61.1, 60.5, 47.0, 43.6, 27.0 (2C), 26.8; IR (film) ν_{max} 2945, 1780, 1685, 1564, 1463, 1317, 1173, 1111 cm^{-1}; HRMS (FAB) *m/z* 287.1346 (MNa^+, $C_{15}H_{20}O_4Na$ requires 287.1376).

6,7-Dimethoxy-3,3-dimethylindan-5-ol (5). A solution of **10** (1.32 g, 4.99 mmol) in anhydrous CH_2Cl_2 (25 mL) at –78 °C under N_2 was treated with BBr_3 (0.94 mL, 2.51 g, 10.0 mmol). The resulting mixture was stirred at –78 °C for 10 min, then poured into vigorously stirring satd aq $NaHCO_3$ (25 mL). The layers were separated, and the organic layer was dried ($MgSO_4$) then concentrated in vacuo. The crude phenol was then dissolved in anhydrous toluene (80 mL) and treated with propylene glycol (4.10 g, 53.9 mmol) and TsOH (1.28 g, 6.73 mmol). The resulting mixture was refluxed for 90 min, then the reaction was quenched with satd aq $NaHCO_3$ (50 mL) and diluted with EtOAc (10 mL). The layers were separated, and the organic layer was dried ($MgSO_4$) and concentrated in vacuo. Flash chromatography (10% acetone–hexanes elution)

afforded **5** (772 mg, 3.47 mmol, 70%) as a white solid: ^{1}H NMR ($CDCl_3$, 300 MHz) δ 6.51 (s, 1H), 5.62 (s, 1H), 3.90 (s, 3H), 3.89 (s, 3H), 2.84 (t, *J* = 7.2 Hz, 2H), 1.90 (t, *J* = 7.2 Hz, 2H), 1.21 (s, 6H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 149.5, 148.7, 148.5, 137.5, 125.8, 103.9, 61.3, 60.1, 44.4, 41.6, 28.7 (2C), 27.0; IR (film) ν_{max} 3410, 2949, 1585, 1462, 1316, 1151, 1090 cm^{-1}; HRMS (FAB) *m/z* 245.1153 (MNa^+, $C_{13}H_{18}O_3Na$ requires 245.1161).

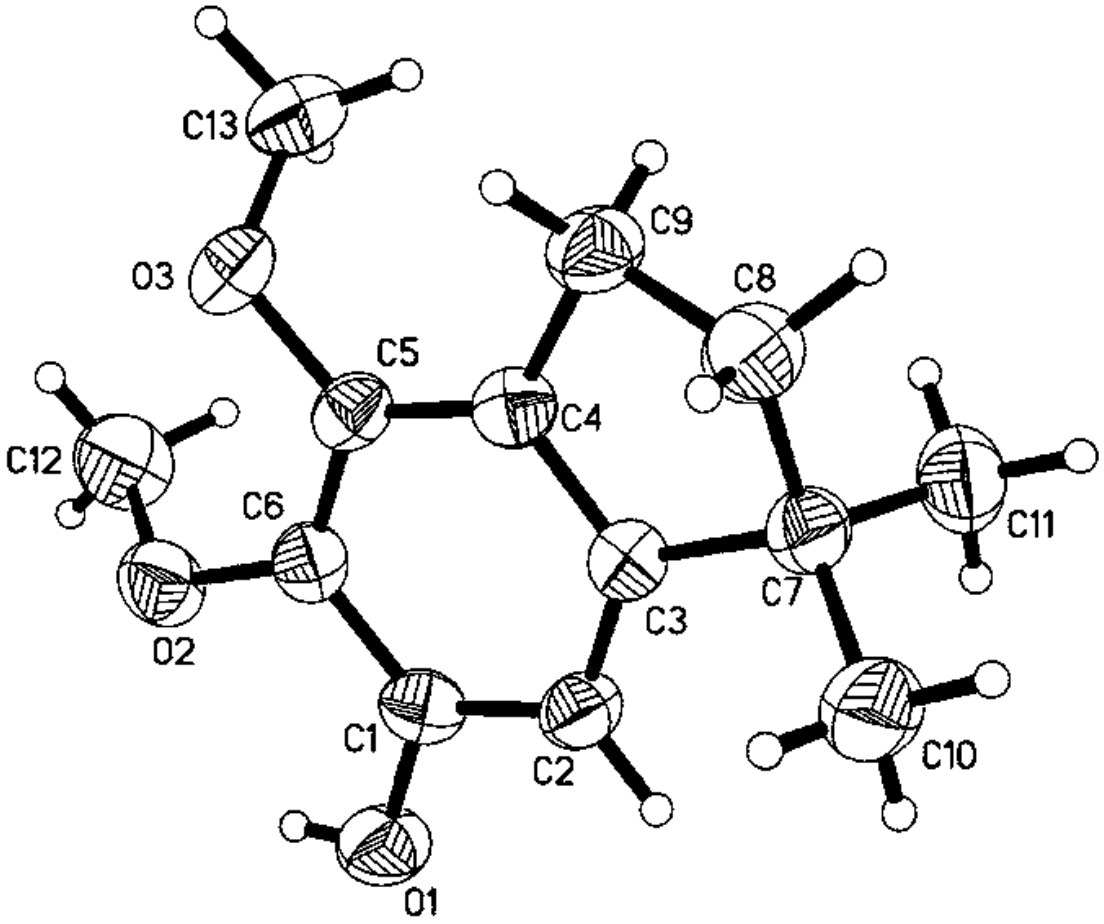


Figure S1. ORTEP Diagram of 5.

6,6,7-Trimethoxy-3,3-dimethyl-1,2,3,6-tetrahydroinden-5-one (4). A solution of **5** (2.20 g, 9.90 mmol) in anhydrous CH_3OH (80 mL) was added to a mixture of $KHCO_3$ (2.38 g, 23.7 mmol), $PhI(OAc)_2$ (3.51 g, 10.9 mmol), and anhydrous CH_3OH (80

mL) at 0 °C under N_2. The resulting yellow-orange mixture was stirred for 10 min, diluted with CH_2Cl_2 (100 mL), and washed with brine (200 mL). The layers were separated, and the organic layer was dried ($MgSO_4$) and concentrated in vacuo, affording **4** (2.40 g, 9.51 mmol, 96%) as a yellow oil: ^{1}H NMR ($CDCl_3$, 300 MHz) δ 5.72 (s, 1H), 4.03 (s, 3H), 3.29 (s, 6H), 2.68 (t, *J* = 8.0 Hz, 2H), 1.70 (t, *J* = 8.0 Hz, 2H), 1.16 (s, 6H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 194.4, 175.5, 153.6, 119.3, 111.4, 94.1, 58.9, 50.9 (2C), 43.8, 39.0, 31.5, 26.8, 25.1; IR (film) ν_{max} 2955, 1724, 1452, 1217, 1096 cm^{-1}; HRMS (FAB) *m/z* 275.1274 (MNa^+, $C_{14}H_{20}O_4Na$ requires 275.1259).

5-Allyl-6,6,7-trimethoxy-3,3-dimethyl-2,3,5,6-tetrahydro-1*H*-inden-5-ol (11). A solution of **4** (3.84 g, 15.2 mmol) in anhydrous Et_2O (100 mL) at 0°C under N_2 was treated dropwise with allylmagnesium chloride (2.0 M soln in THF, 11.4 mL, 22.8 mmol) The resulting mixture was stirred for 15 min, and the reaction was quenched by the addition of H_2O (30 mL). The layers were separated, and the organic layer was dried ($MgSO_4$) and concentrated in vacuo. This afforded crude **11** (4.41 g, 15.0 mmol) that was used directly in the subsequent transformation: ^{1}H NMR (Benzene-d_6, 300 MHz) δ 6.36–6.13 (m, 1H), 5.30 (s, 1H), 5.14–5.11 (m, 1H), 5.05 (t, *J* = 1.5 Hz, 1H), 3.60 (s, 3H), 3.45 (s, 3H), 3.25 (s, 3H), 3.13 (s, 1H), 2.68 (d, *J* = 11.1 Hz, 2H), 2.51–2.36 (m, 1H), 2.35–2.18 (m, 1H), 1.39 (t, *J* = 11.1 Hz, 2H), 1.00 (s, 3H), 0.98 (s, 3H); ^{13}C NMR (Benzene-d_6, 75 MHz) δ 148.8, 146.6, 135.4, 126.4, 122.8, 117.1, 102.2, 81.2, 60.1, 51.4, 50.7, 41.2, 40.6, 40.4, 28.1, 27.5, 24.9; IR (film) ν_{max} 3504, 2949, 1636, 1452, 1210, 1053, 908 cm^{-1}; HRMS (FAB) *m/z* 317.1742 (MNa^+, $C_{17}H_{26}O_4Na$ requires 317.1729).

3a-Allyl-6,6,7-trimethoxy-3,3-dimethyl-1,2,3,3a,4,6-hexahydroinden-5-one (12). A mixture of 18-crown-6 (12.3 g, 46.5 mmol), KO*t*-Bu (5.05 g, 45.0 mmol), and

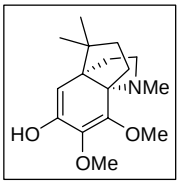
anhydrous THF (250 mL) at 0°C under N_2 was stirred for 15 min, then treated with a solution of **11** (4.41 g, 15.0 mmol) in anhydrous THF (50 mL, added over 3 min). The resulting mixture was stirred at 0°C under N_2 for 1 h. The reaction was quenched by the addition of H_2O (100 mL) and diluted with Et_2O (200 mL). The layers were separated, and the organic layer was dried ($MgSO_4$) and concentrated in vacuo. Flash chromatography (1% Et_3N in 10% EtOAc–hexanes elution) afforded **12** (2.68 g, 9.10 mmol, 60% over two steps) as a colorless oil: ^{1}H NMR (Benzene-d_6, 500 MHz) δ 5.81–5.68 (m, 1H), 4.96 (s, 1H), 4.93 (d, J = 6.0 Hz, 1H), 3.71 (s, 3H), 3.45 (s, 3H), 3.35 (s, 3H), 2.64 (d, J = 13.5 Hz, 1H), 2.54–2.46 (m, 1H), 2.27 (d, J = 13.0 Hz, 1H), 2.19–2.12 (m, 1H), 1.98 (d, J = 7.5 Hz, 2H), 1.56–1.49 (m, 1H), 1.24–1.18 (m, 1H), 0.73 (s, 3H), 0.71 (s, 3H); ^{13}C NMR (Benzene-d_6, 125 MHz) δ 202.5, 146.0, 136.1, 135.4, 118.1, 98.0, 60.1, 52.1, 51.2, 51.0, 44.2, 42.7, 41.8, 36.6, 26.6, 23.6, 22.0; IR (film) ν_{max} 2952, 1732, 1450, 1063 cm^{-1}; HRMS (FAB) *m/z* 317.1714 (MNa^+, $C_{17}H_{26}O_4Na$ requires 317.1729).

6,6,7-Trimethoxy-3,3-dimethyl-3a(2-methylaminoethyl)-1,2,3,3a,4,6-hexahydroinden-5-one (3). A solution of **12** (50 mg, 0.17 mmol) in EtOAc (28 mL, 0.006 M) at –78 °C was treated with O_3 (bubbled for 1.25 min). Et_3N (71 μL, 52 mg, 0.51 mmol) was then added and the resulting mixture was stirred at rt for 16 h then concentrated in vacuo. This procedure was repeated twice.

The three crude mixtures of aldehyde and **12** (derived from 150 mg of **12**) were combined and dissolved in anhydrous CH_3OH (200 μL), treated with powdered 4Å molecular sieves (30 mg) and CH_3NH_2 (2.0 M in CH_3OH, 410 μL, 0.82 mmol), and stirred at rt under N_2 for 30 min. $NaBH_3CN$ (26 mg, 0.41 mmol) was then added and the resulting mixture was stirred at rt under N_2 for 16 h. It was then diluted with EtOAc (3

mL), washed with aq KOH (10 M, 1 mL), and the aqueous layer was extracted with EtOAc (2 × 4 mL). The combined organic layers were dried (Na_2SO_4) and concentrated in vacuo. Flash chromatography (SiO_2, 1% Et_3N in 5–10% EtOAc–hexanes gradient elution) afforded recovered **12** (73 mg, 49% recovery) and **3** (59.4 mg, 0.191 mmol, 37%, 73% based on recovered **12**) as a colorless oil that solidified on standing: ^{1}H NMR (Benzene-d_6, 500 MHz) δ 3.64 (s, 3H), 3.53 (s, 3H), 3.38 (s, 3H), 3.03 (dt, *J* = 3.5, 12.5 Hz, 1H), 2.88 (s, 3H), 2.74 (ddd, *J* = 2.0, 5.0, 12.0 Hz, 1H), 2.44–2.36 (m, 1H), 2.06 (ddd, *J* = 1.5, 10.5, 18.0 Hz, 1H), 1.76 (dd, *J* = 3.0, 11.5 Hz, 1H), 1.55 (dt, *J* = 5.0, 12.5 Hz, 1H), 1.49 (d, *J* = 11.5 Hz, 1H), 1.49–1.43 (m, 1H), 1.41–1.28 (m, 2H), 1.25 (ddd, *J* = 1.5, 9.0, 13.0 Hz, 1H), 0.79 (s, 3H), 0.71 (s, 3H); ^{13}C NMR (Benzene-d_6, 125 MHz) δ 190.6, 149.2, 134.6, 100.5, 61.6, 52.8, 52.2, 49.8, 49.1, 42.7, 39.0, 37.4, 30.6, 27.9, 25.4, 23.5, 22.0; IR (film) ν_{max} 3460, 2940, 1728, 1460, 1112 cm^{-1}; HRMS (EI) *m/z* 334.1984 (MNa^+, $C_{17}H_{29}O_4NNa$ requires 334.1985).

4,5-Dimethoxy-7,10,10-trimethyl-7-azatricyclo[4.3.3.0^{1,6}] dodeca-2,4-dien-3-ol (13). A solution of **3** (20 mg, 0.064 mmol) in anhydrous CH_2Cl_2 (0.6 mL) at –10 °C was treated with powdered 4Å molecular sieves (32 mg) followed by TMSOTf (12 μL, 13.8 mg, 0.062 mmol). The resulting mixture was stirred at –10 °C for 10 min, treated with additional TMSOTf (12 μL, 13.8 mg, 0.062 mmol), and stirred for 5 min. The mixture was concentrated in vacuo, and all volatile materials were removed under high vacuum. The residue was purified by column chromatography (SiO_2, 2 × 15 cm, 1% Et_3N in 0–5% EtOAc–hexanes gradient elution) to afford **13** (8.9 mg, 0.032 mmol, 50%) as a colorless oil along with the O-



demethylated byproduct **14** (1.9 mg, 0.0072 mmol, 11%) as a pale yellow oil that solidified on standing.

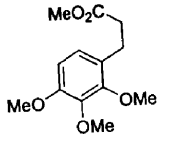
For **13**: ^{1}H NMR (Benzene-d_6, 500 MHz) δ 5.67 (s, 1H), 3.76 (s, 3H), 3.60 (s, 3H), 2.71 (dd, *J* = 5.5, 12.0 Hz, 1H), 2.41 (s, 3H), 2.27 (d, *J* = 16.5 Hz, 1H), 2.24 (dt, *J* = 3.5, 11.5 Hz, 1H), 2.04 (dd, *J* = 2.5, 11.0 Hz, 1H), 1.88 (dd, *J* = 3.5, 16.5 Hz, 1H), 1.69 (d, *J* = 10.5 Hz, 1H), 1.51 (dt, *J* = 5.0, 13.0 Hz, 1H), 1.21 (d, *J* = 11.5 Hz, 1H), 0.88 (s, 3H), 0.86 (s, 3H); ^{13}C NMR (Benzene-d_6, 125 MHz) δ 142.4, 126.9, 118.3, 89.3, 60.7, 60.1, 53.6, 50.7, 46.2, 44.1, 41.8, 38.5, 33.1, 30.6, 26.0, 22.8; IR (film) ν_{max} 3421, 2920, 2854, 1662, 1460, 1254, 1064, 842 cm^{-1}; HRMS (FAB) *m/z* 302.1733 (MNa^+, $C_{16}H_{25}NO_3Na$ requires 302.1732); 2D ^{1}H–^{1}H COSY NMR (Benzene-d_6, 500 MHz) 5.67/1.88 (w, H-2/H-11), 2.71/2.23 (s, H-8/H-8), 2.71/1.51 (w, H-8/H-9), 2.27/1.88 (s, H-11/H-11), 2.23/1.51 (w, H-8/H-9), 2.04/1.69 (s, H-12/H-12), 1.51/1.21 (m, H-9/H-9); 2D ^{1}H–^{13}C HMQC (Benzene-d_6, 500 MHz) 5.67/118.3 (m, H-2/C-2), 3.76/60.1 (s, OCH_3), 3.60/60.7 (s, OCH_3), 2.71/50.7 (m, H-8/C-8), 2.41/38.5 (s, NCH_3), 2.27/46.2 (m, H-11/C-11), 2.23/50.7 (m, H-8/C-8), 2.04/41.8 (m, H-12/C-12), 1.88/46.2 (m, H-11/C-11), 1.69/41.8 (m, H-12/C-12), 1.21/33.1 (w, H-9/C-9) 0.88/26.0 (s, CCH_3), 0.86/22.8 (s, CCH_3).

For **14**: ^{1}H NMR (Benzene-d_6, 500 MHz) δ 3.64 (s, 3H), 2.68 (dd, *J* = 5.5, 12.0 Hz, 1H), 2.44–2.30 (m, 1H), 2.26 (s, 3H), 2.21–2.13 (m, 2H), 1.84 (dd, *J* = 3.0, 12.0 Hz, 1H), 1.79 (d, *J* = 12.0 Hz, 1H), 1.47 (dt, *J* = 5.0, 13.0 Hz, 1H), 1.30–1.24 (m, 1H), 1.08 (dd, *J* = 8.5, 12.5 Hz, 1H), 0.77 (d, *J* = 16 Hz, 1H), 0.61 (s, 3H), 0.44 (s, 3H); ^{13}C NMR (Benzene-d_6, 75 MHz) δ 191.3, 157.6, 149.3, 84.8, 60.0, 50.5, 49.4, 43.3, 40.8, 36.3,

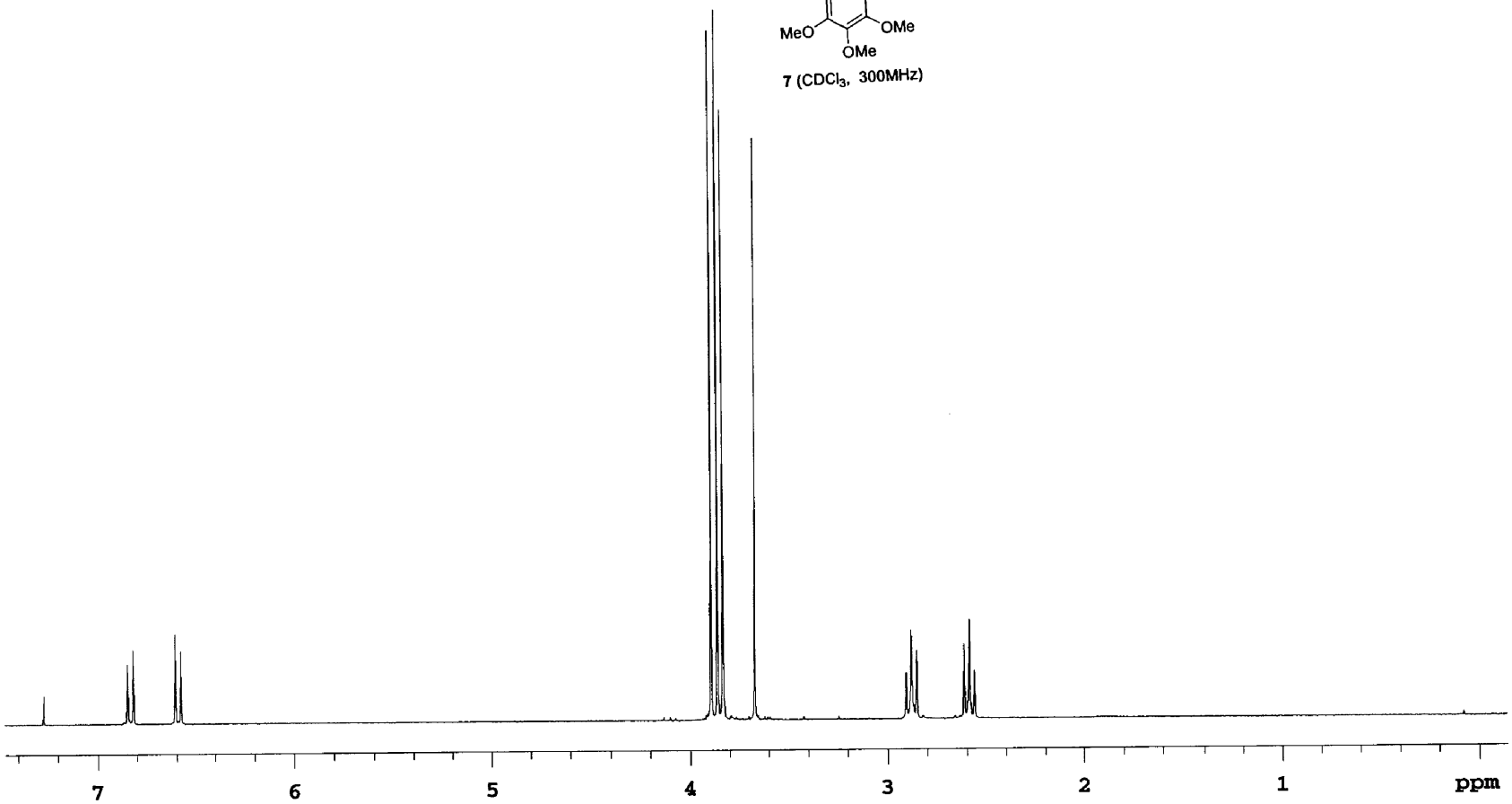
30.6, 29.2, 25.7, 24.5, 21.7; IR (film) ν_{max} 3389, 2931, 1660, 1473, 1374, 1288, 1064 cm^{-1}; HRMS (EI) *m/z* 266.1762 (M^+, $C_{15}H_{23}NO_3$ requires 266.1756).

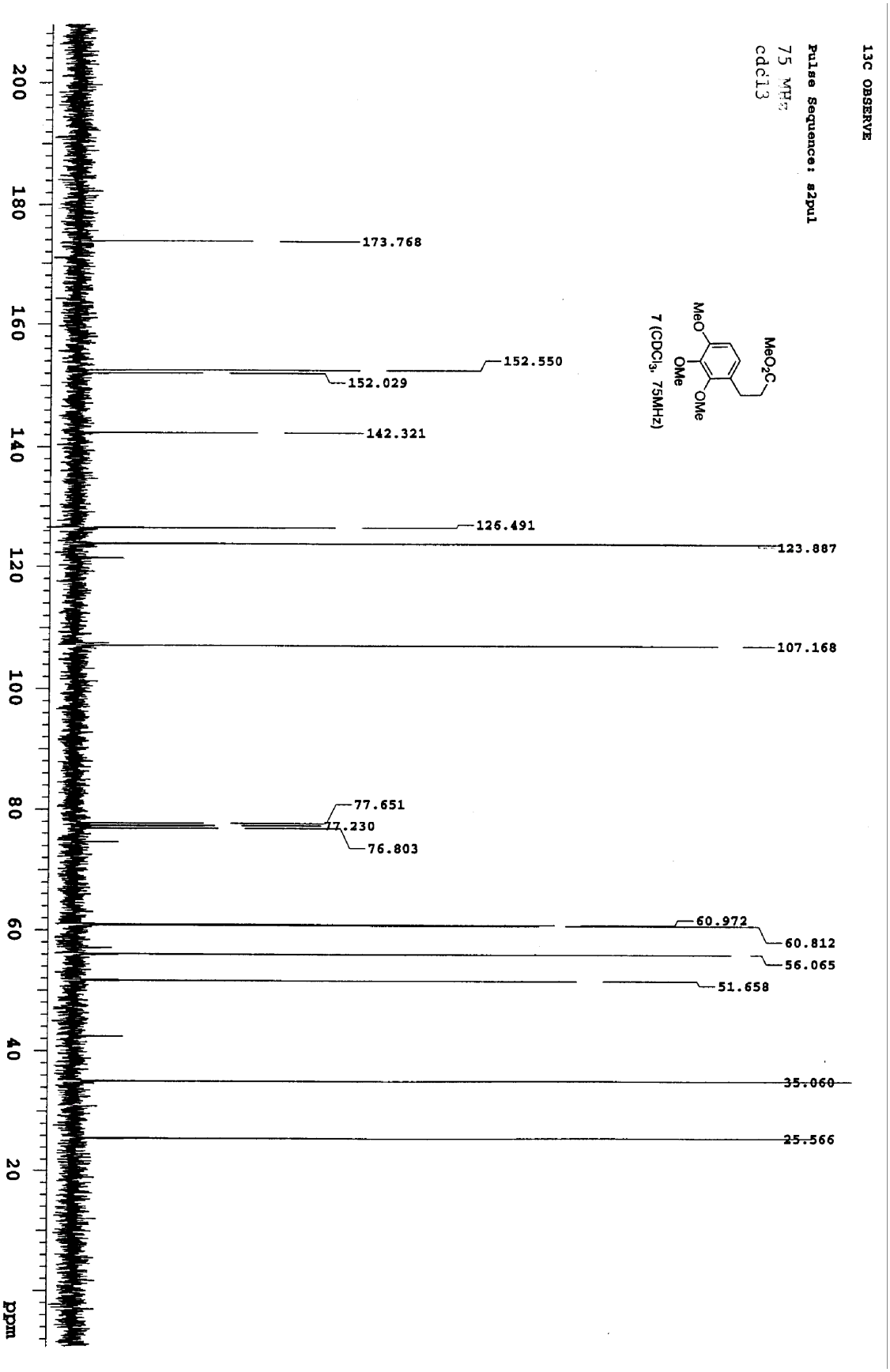
SC-MDR-9 (CDCL3, 300 MHz)

Pulse Sequence: s2pul



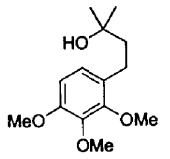
7 ($CDCl_3$, 300MHz)



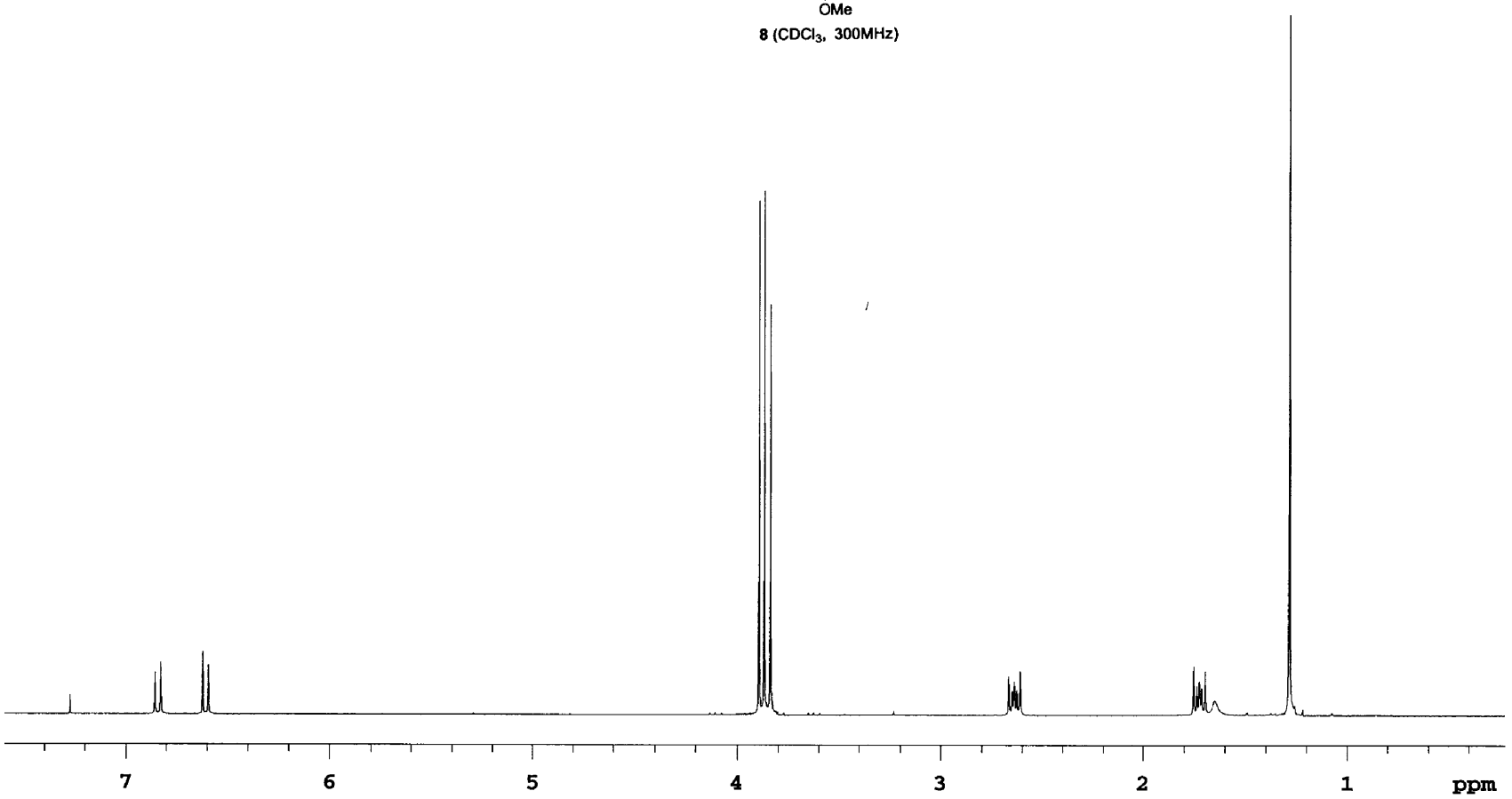

13C OBSERVE
Pulse Sequence: s2pul
75 MHz
cdcl3
7 (CDCl3, 75MHz)
MeO
OMe
OMe
MeO2C
173.768
152.550
152.029
142.321
126.491
123.887
107.168
77.651
77.230
76.803
60.972
60.812
56.065
51.658
35.060
25.566
200
180
160
140
120
100
80
60
40
20
ppm

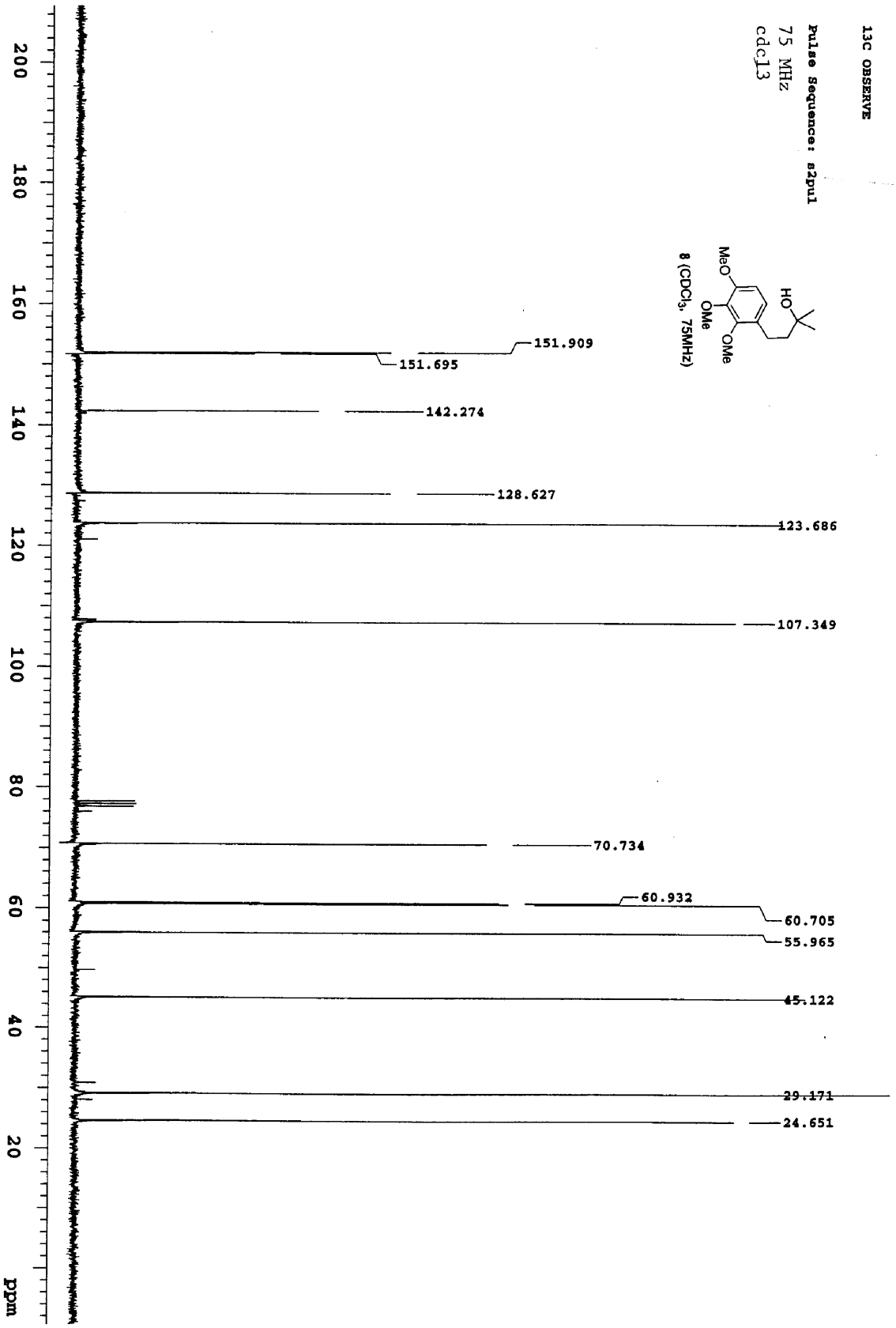
MDR-10-2 (CDCl3, 300 MHz)

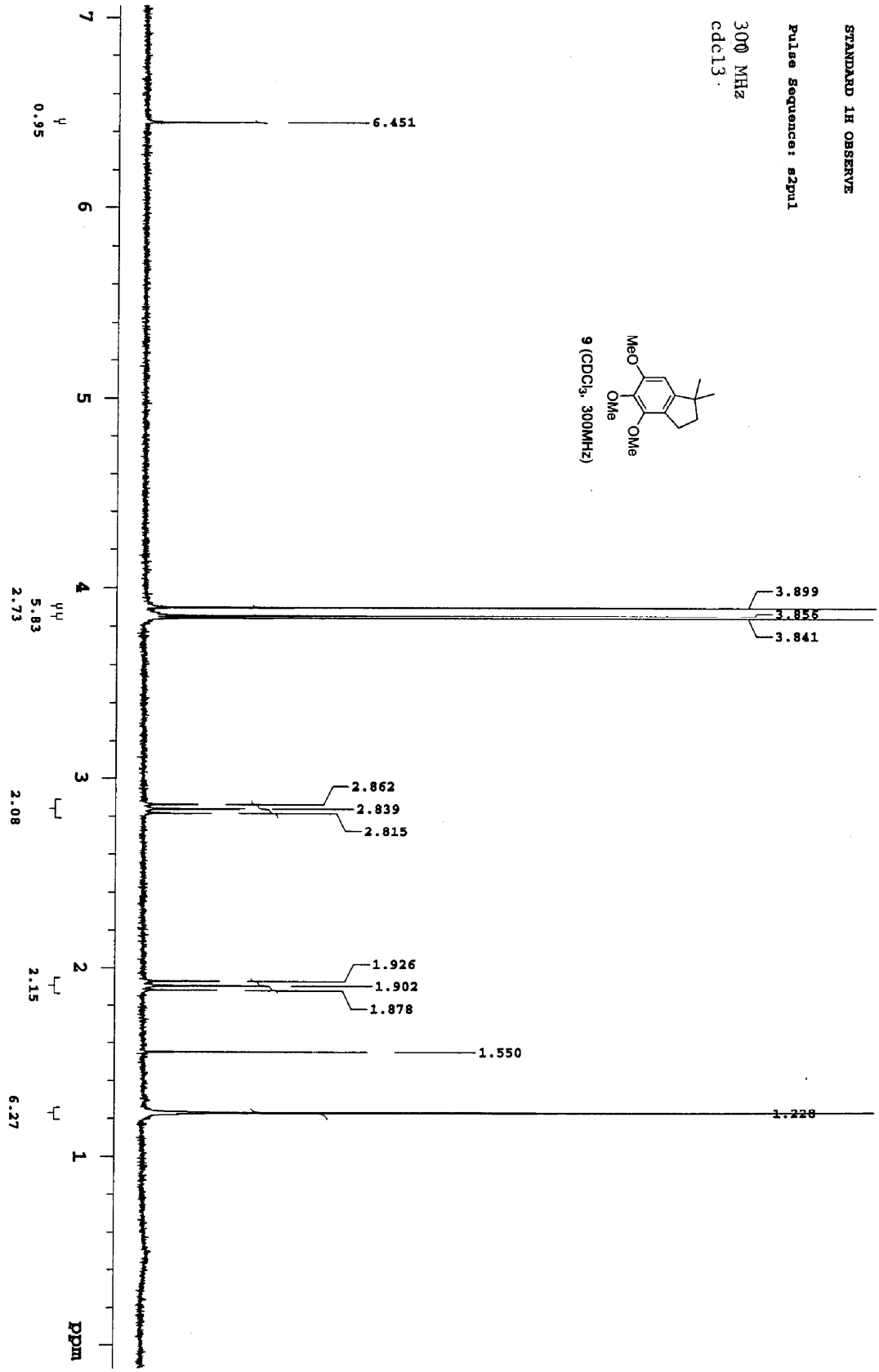
Pulse Sequence: s2pul

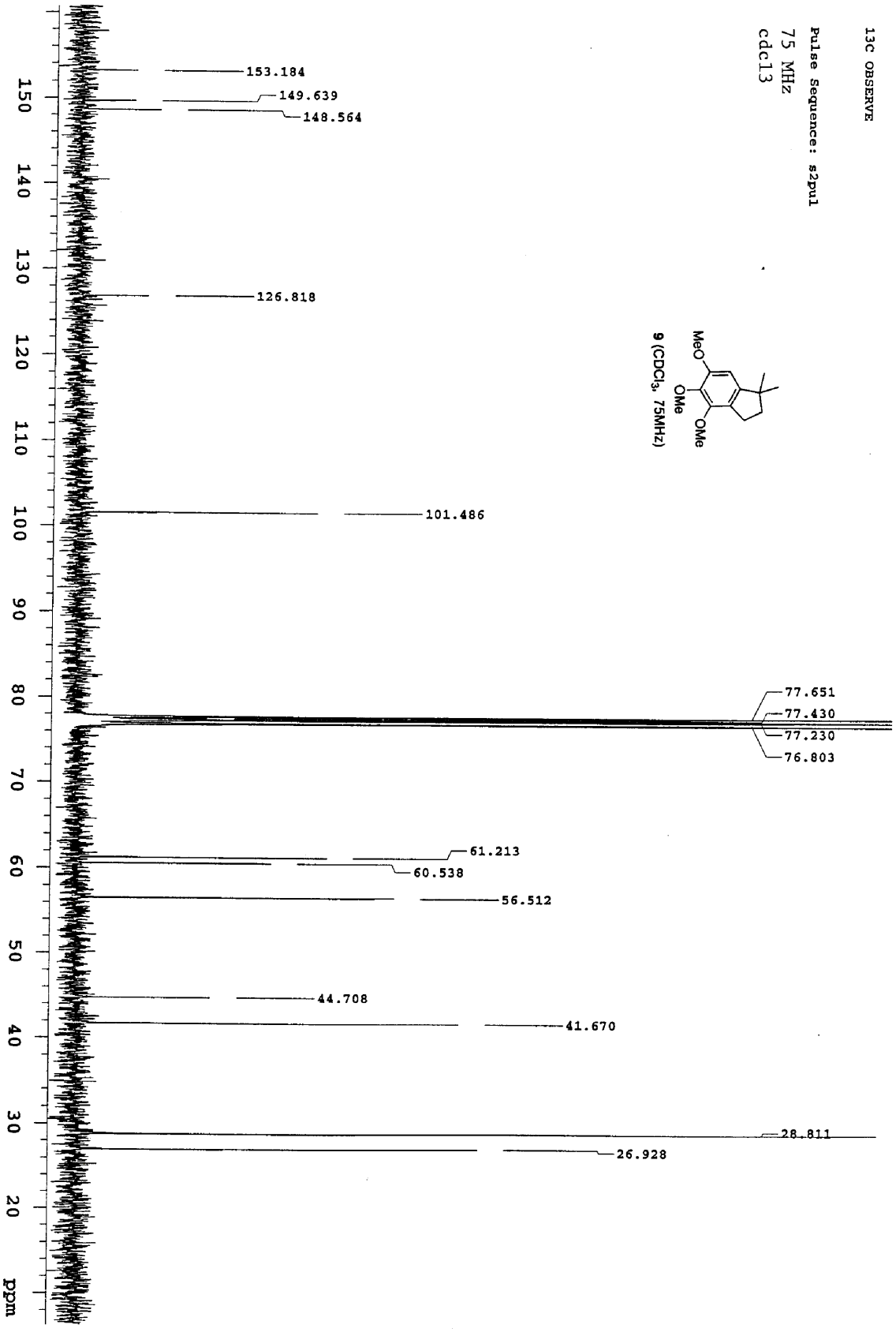


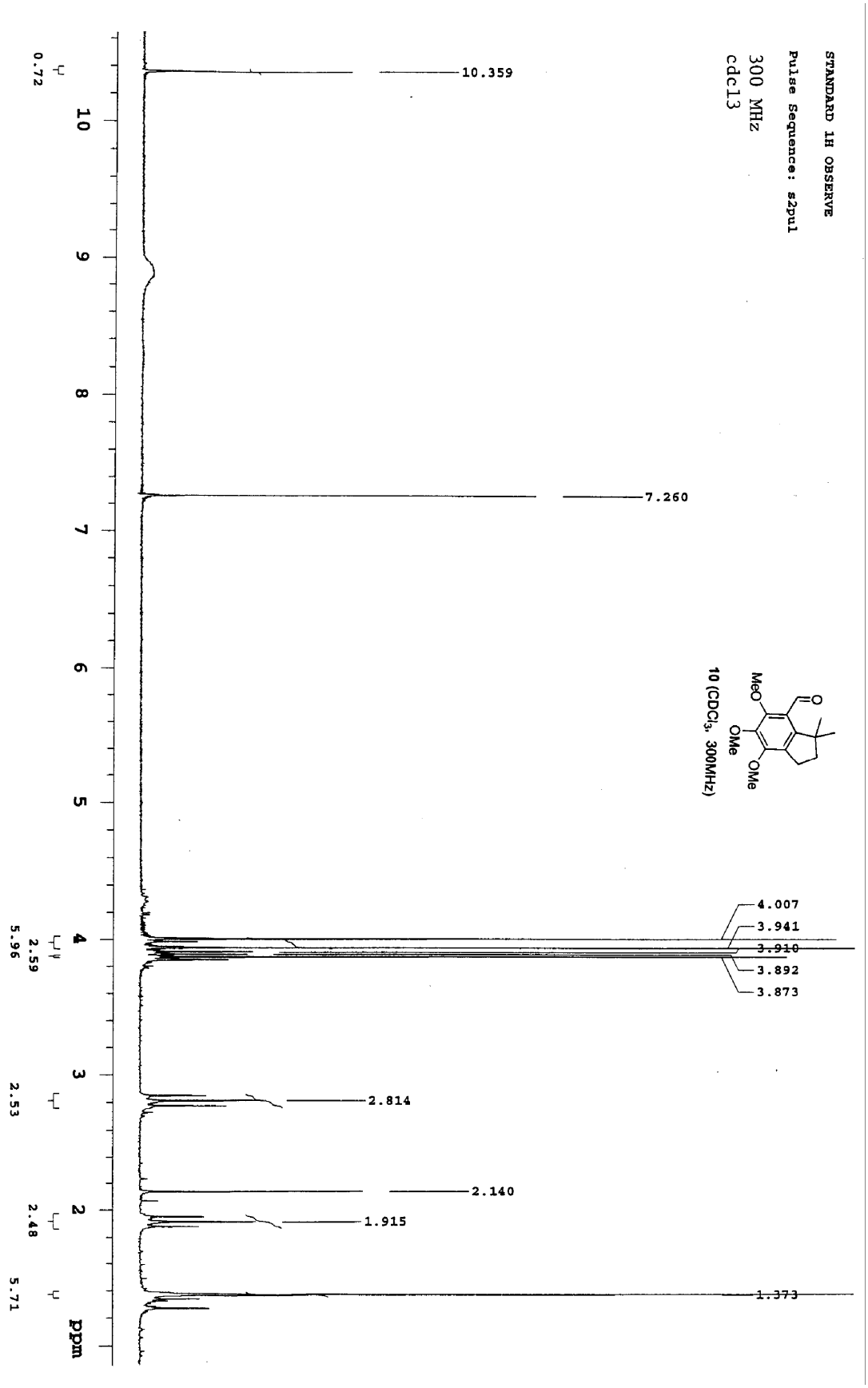
8 ($CDCl_3$, 300MHz)

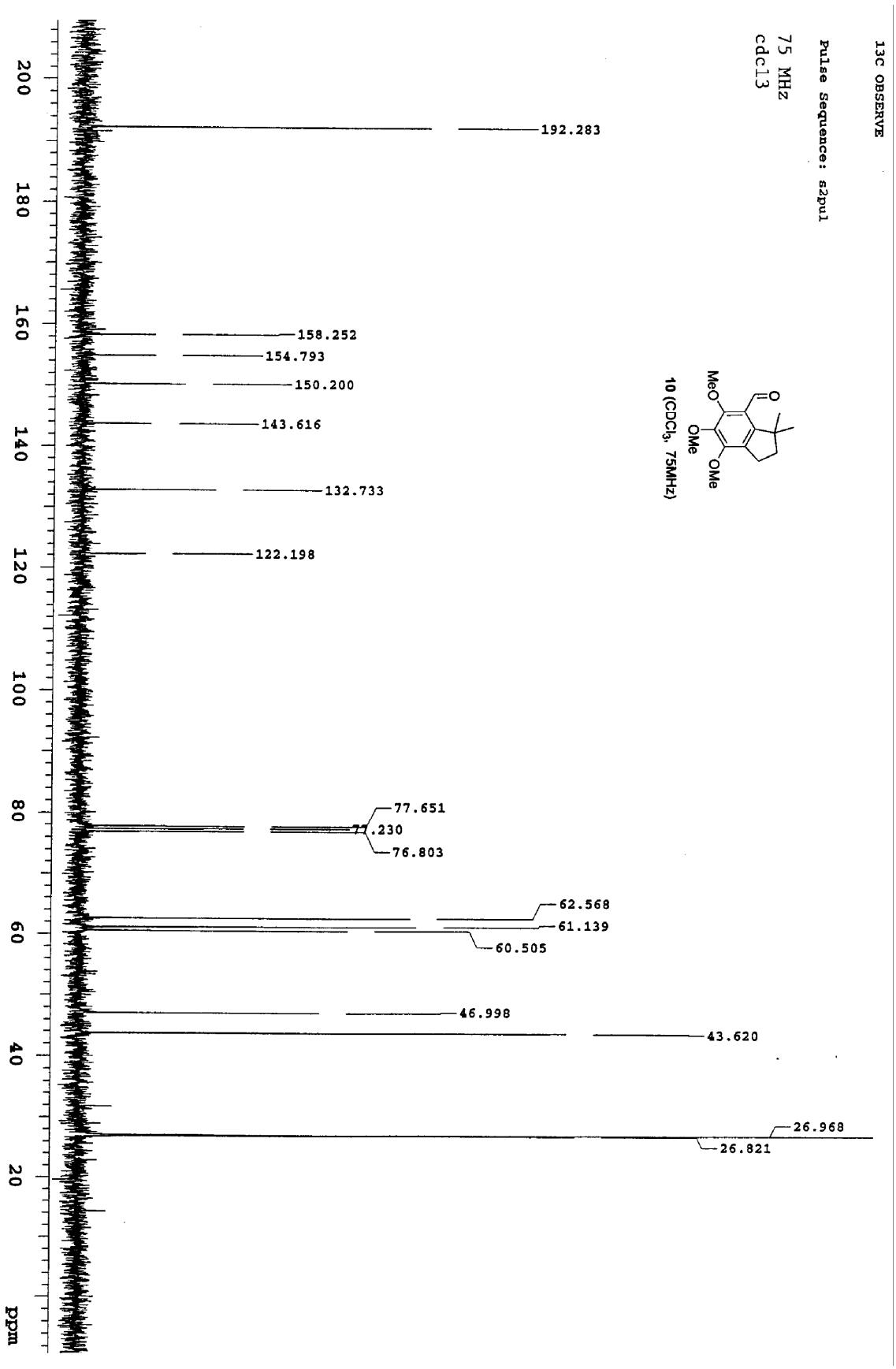


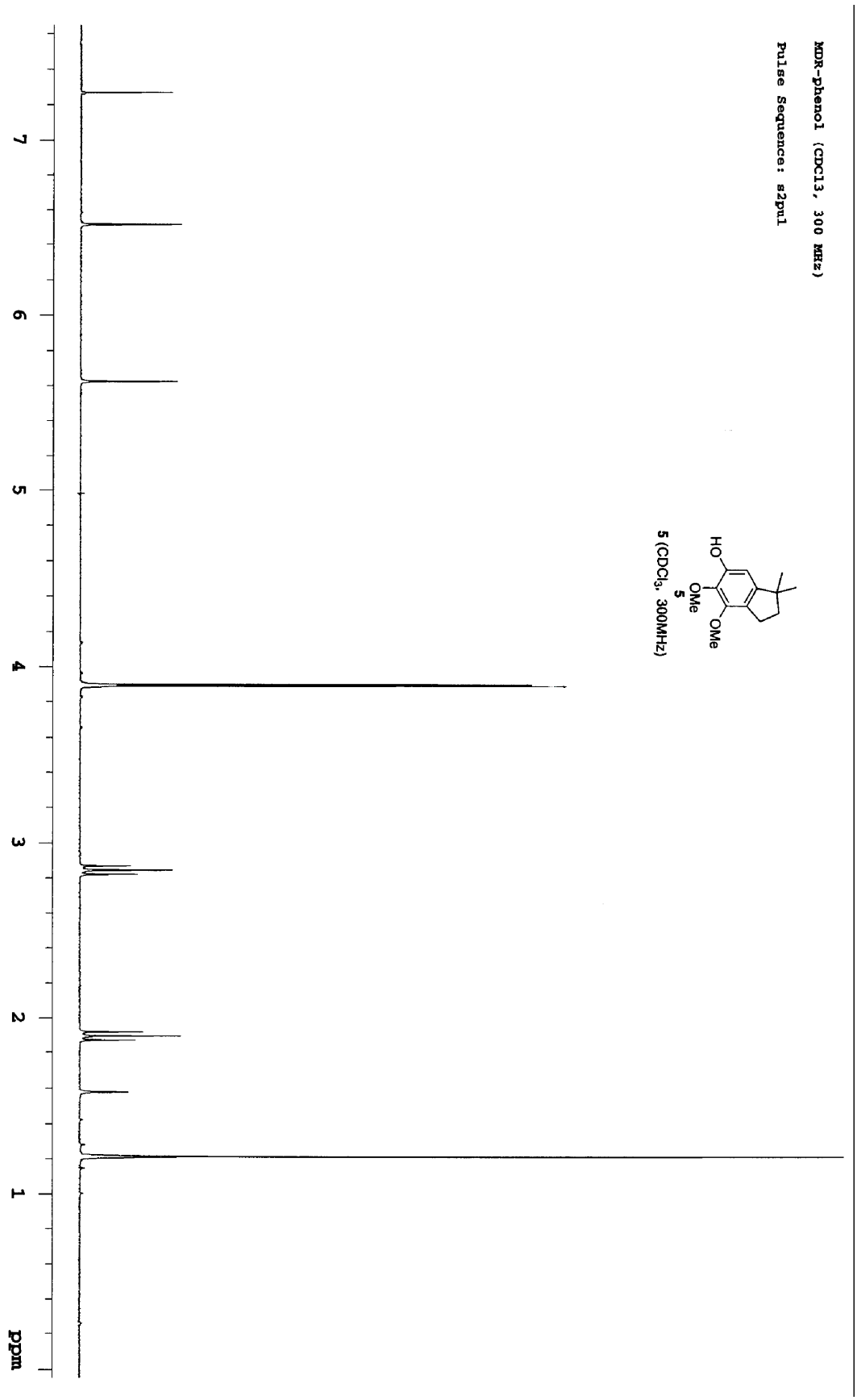

13C OBSERVE
Pulse Sequence: s2pul
75 MHz
cdcl3
8 (CDCl3, 75MHz)
MeO
OMe
OMe
HO
151.909
151.695
142.274
128.627
123.686
107.349
70.734
60.932
60.705
55.965
45.122
29.171
24.651
200
180
160
140
120
100
80
60
40
20
ppm

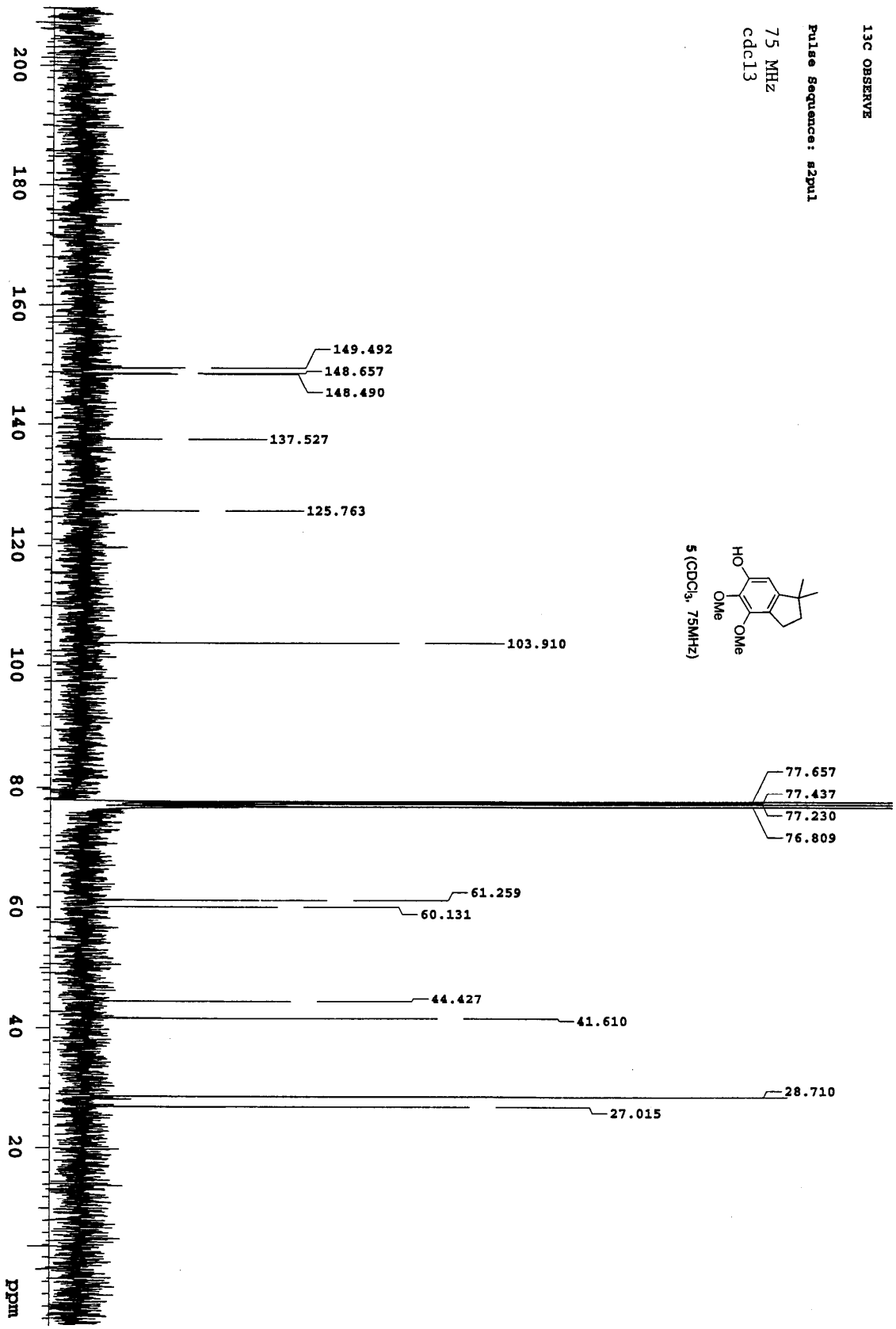

STANDARD 1H OBSERVE
Pulse Sequence: s2pul
300 MHz
cdcl3
MeO
OMe
OMe
9 (CDCl3, 300MHz)
6.451
3.899
3.856
3.841
2.862
2.839
2.815
1.926
1.902
1.878
1.550
1.228
0.95
5.83
2.73
2.08
2.15
6.27
ppm

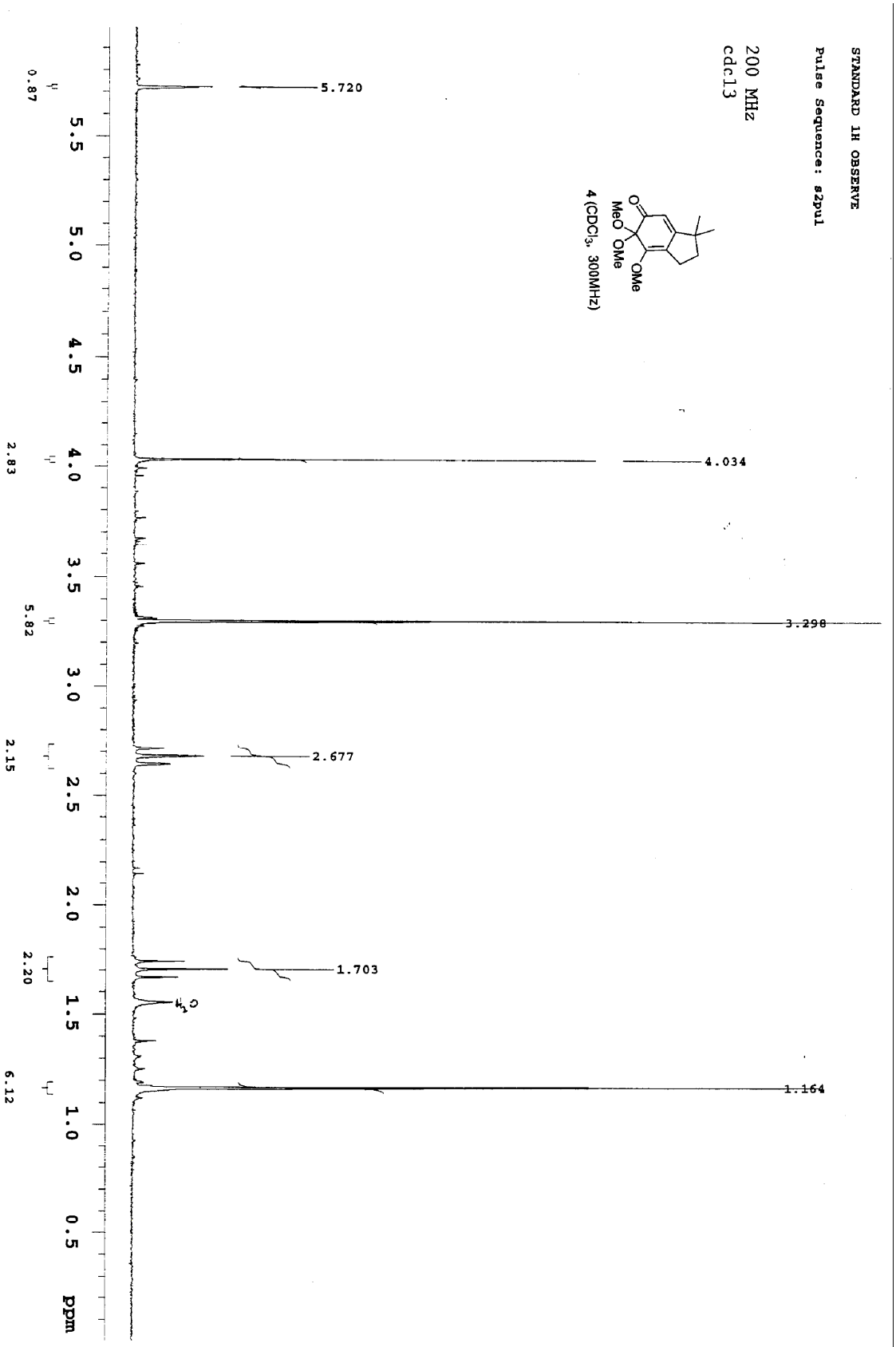

13C OBSERVE
Pulse Sequence: s2pul
75 MHz
cdcl3
MeO
OMe
OMe
9 (CDCl3, 75MHz)
153.184
149.639
148.564
126.818
101.486
77.651
77.430
77.230
76.803
61.213
60.538
56.512
44.708
41.670
28.811
26.928
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm

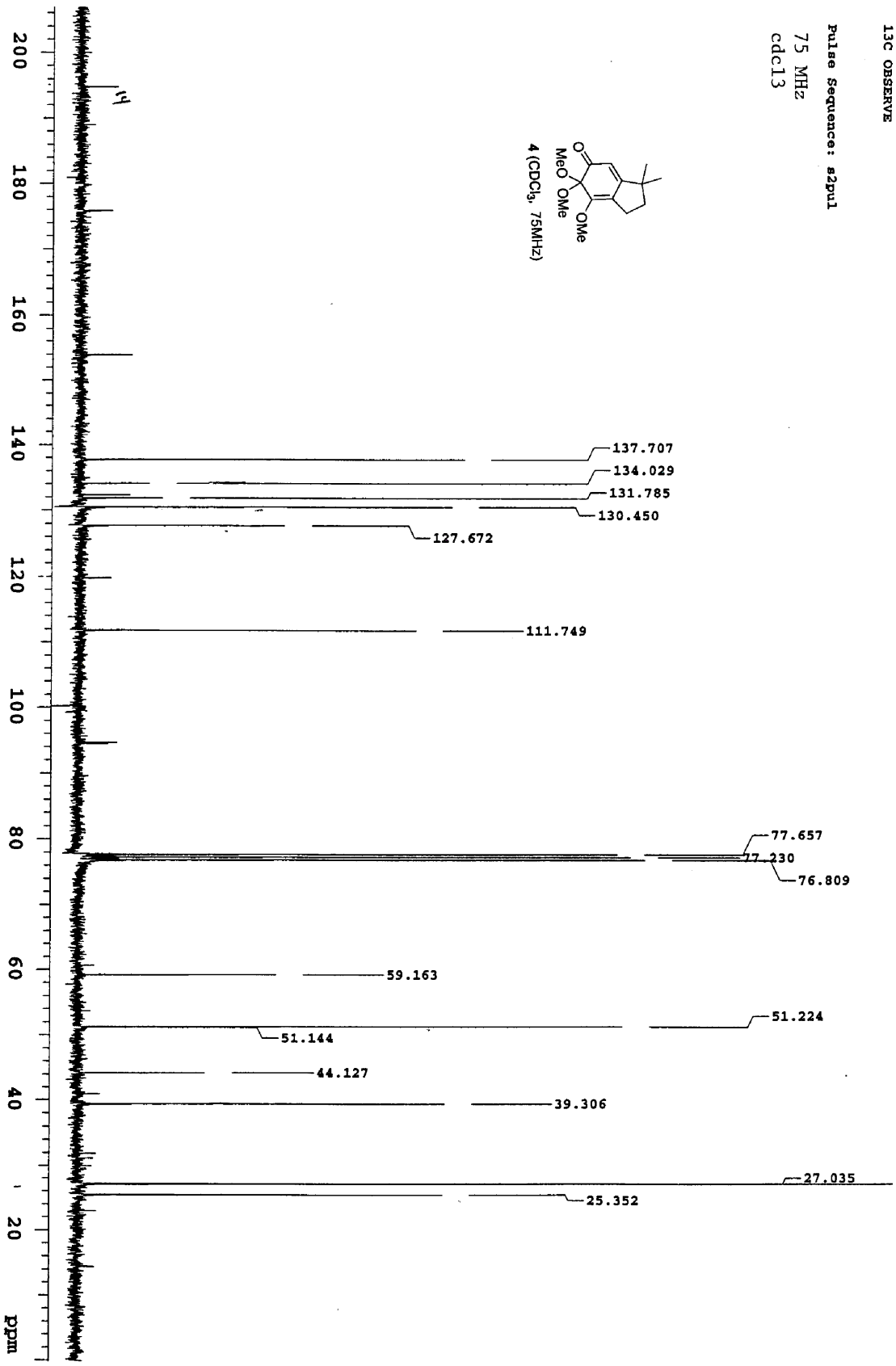

STANDARD 1H OBSERVE
Pulse Sequence: s2pul
300 MHz
cdcl3
10 (CDCl3, 300MHz)
MeO
OMe
OMe
O
10.359
7.260
4.007
3.941
3.910
3.892
3.873
2.814
2.140
1.915
1.373
0.72
5.96
2.59
2.53
2.48
5.71
ppm

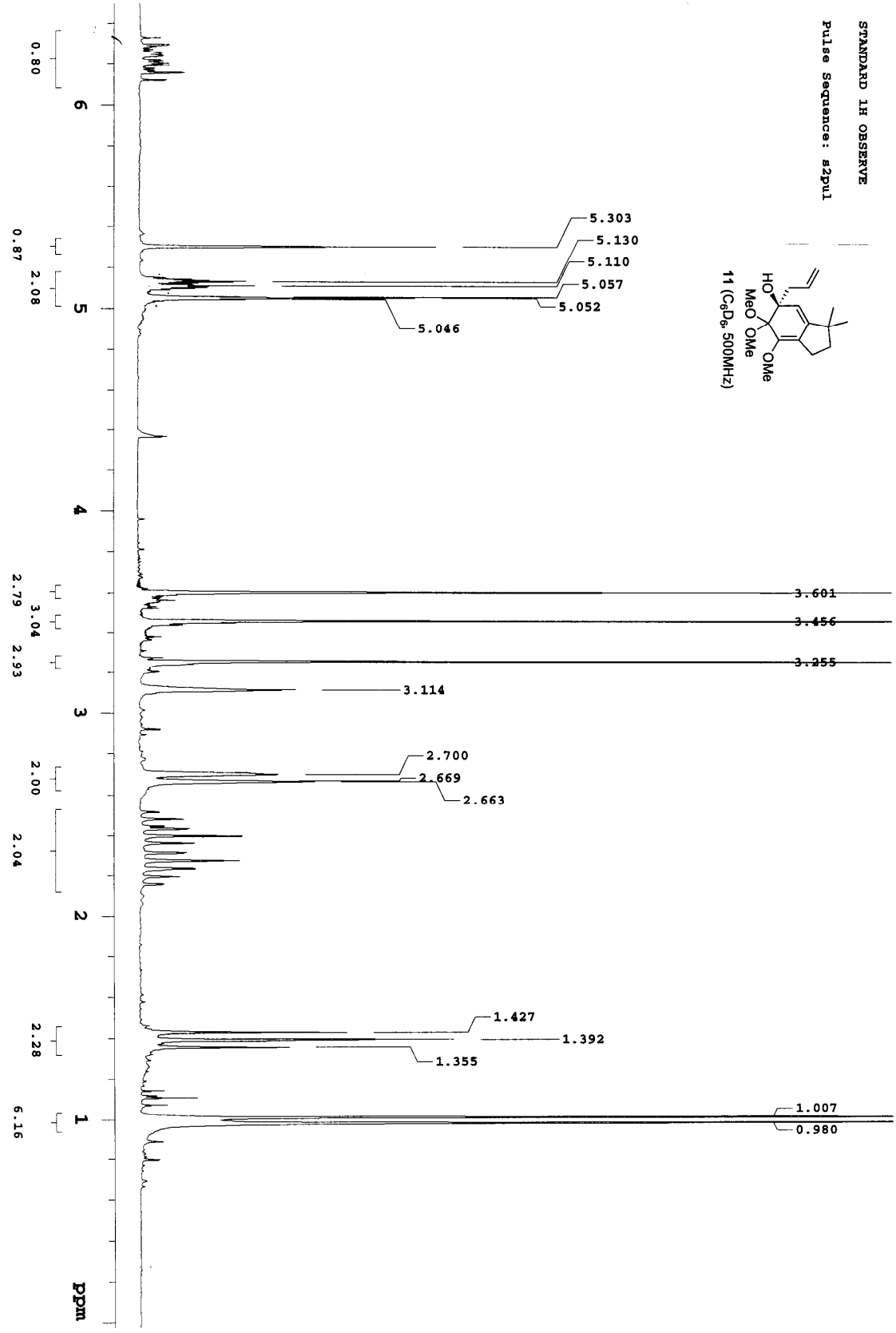

13C OBSERVE
Pulse Sequence: s2pul
75 MHz
cdc13
10 (CDCl3, 75MHz)
MeO
OMe
OMe
O
192.283
158.252
154.793
150.200
143.616
132.733
122.198
77.651
77.230
76.803
62.568
61.139
60.505
46.998
43.620
26.968
26.821
200
180
160
140
120
100
80
60
40
20
ppm

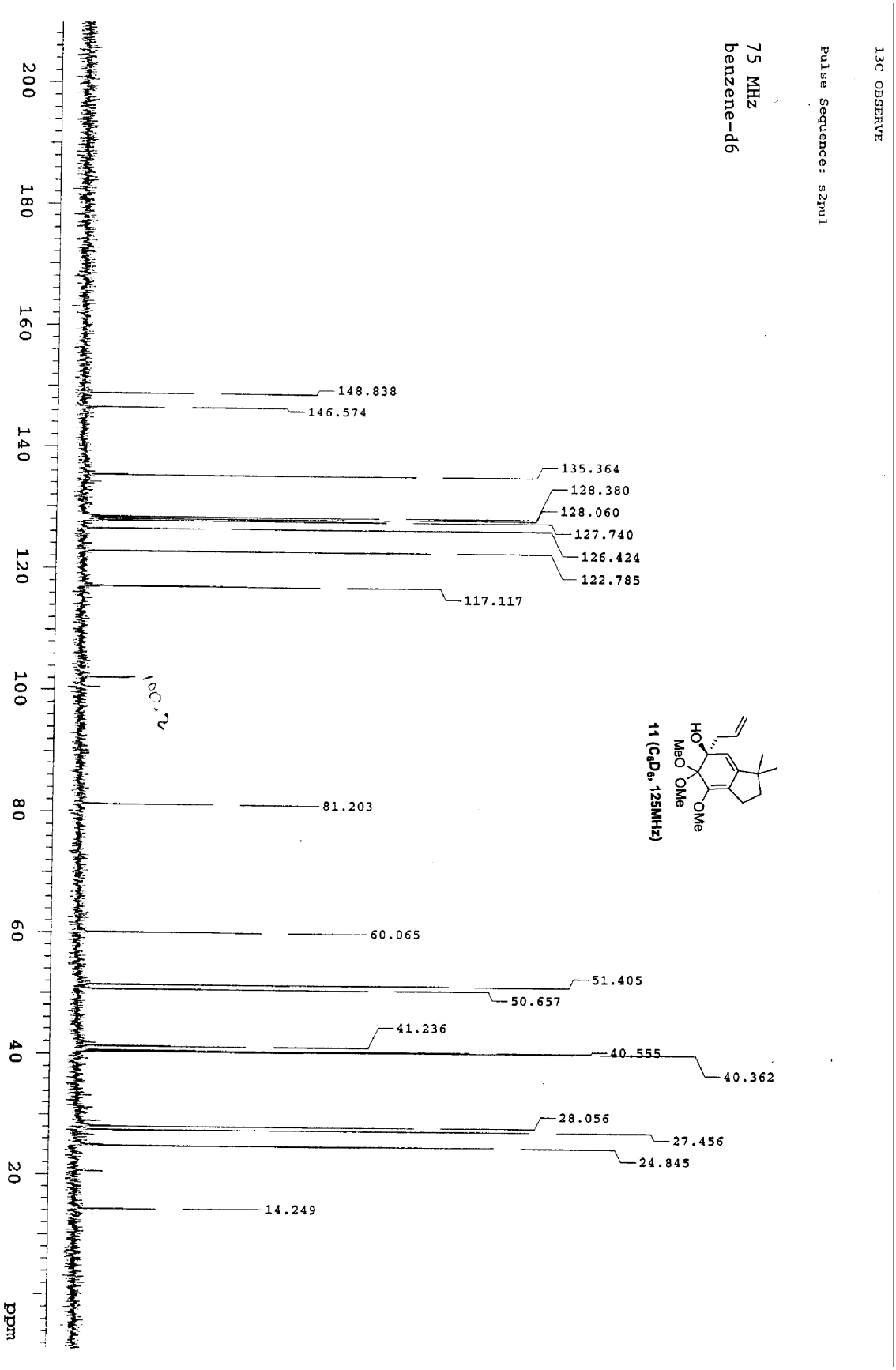

MDR-phenol (CDCl3, 300 MHz)
Pulse Sequence: s2pul
HO
OMe
OMe
5
5 (CDCl3, 300MHz)
7
6
5
4
3
2
1
ppm

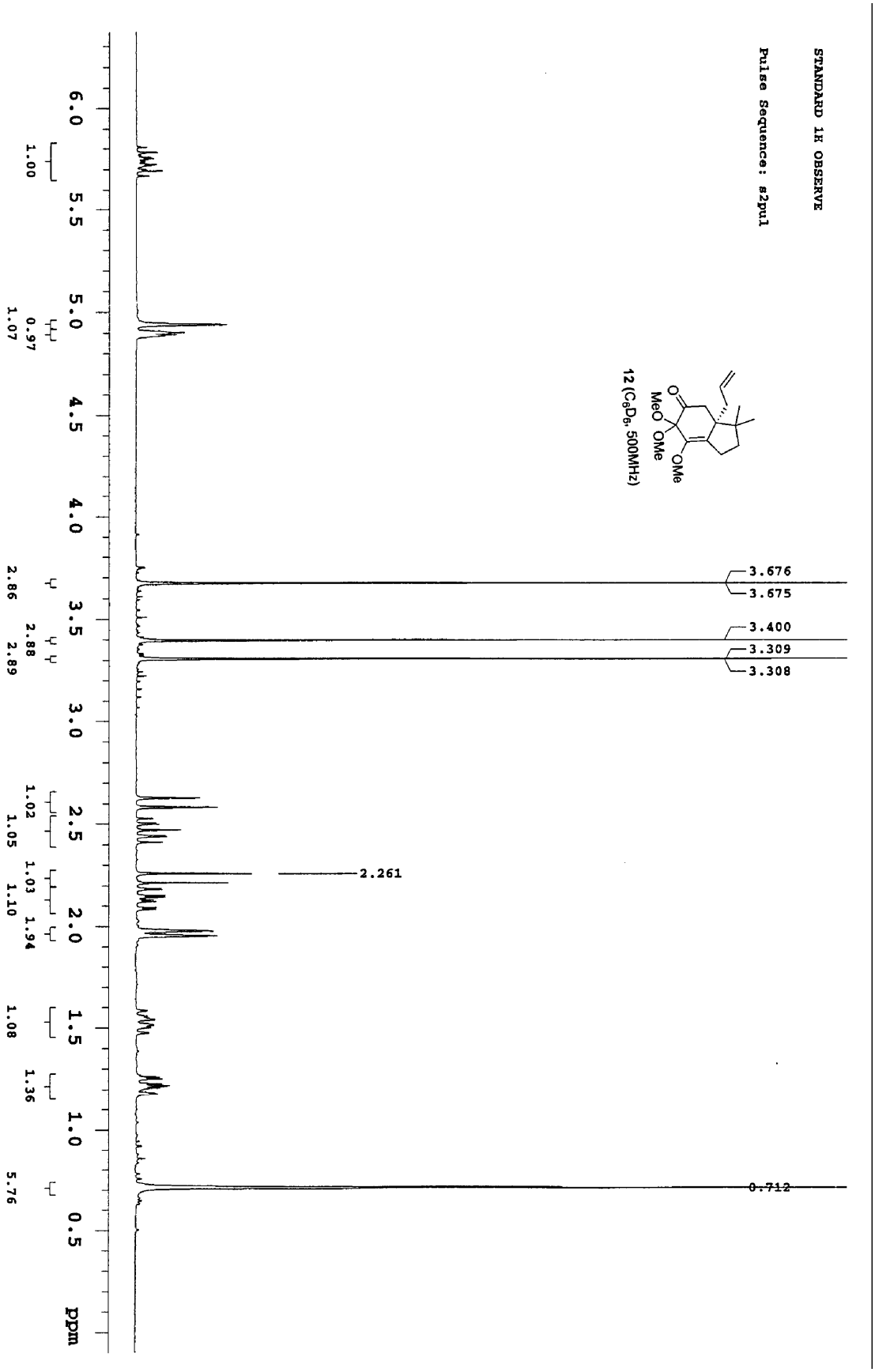

13C OBSERVE
Pulse Sequence: s2pul
75 MHz
cdc13
5 (CDCl3, 75MHz)
HO
OMe
OMe
149.492
148.657
148.490
137.527
125.763
103.910
77.657
77.437
77.230
76.809
61.259
60.131
44.427
41.610
28.710
27.015
200
180
160
140
120
100
80
60
40
20
ppm

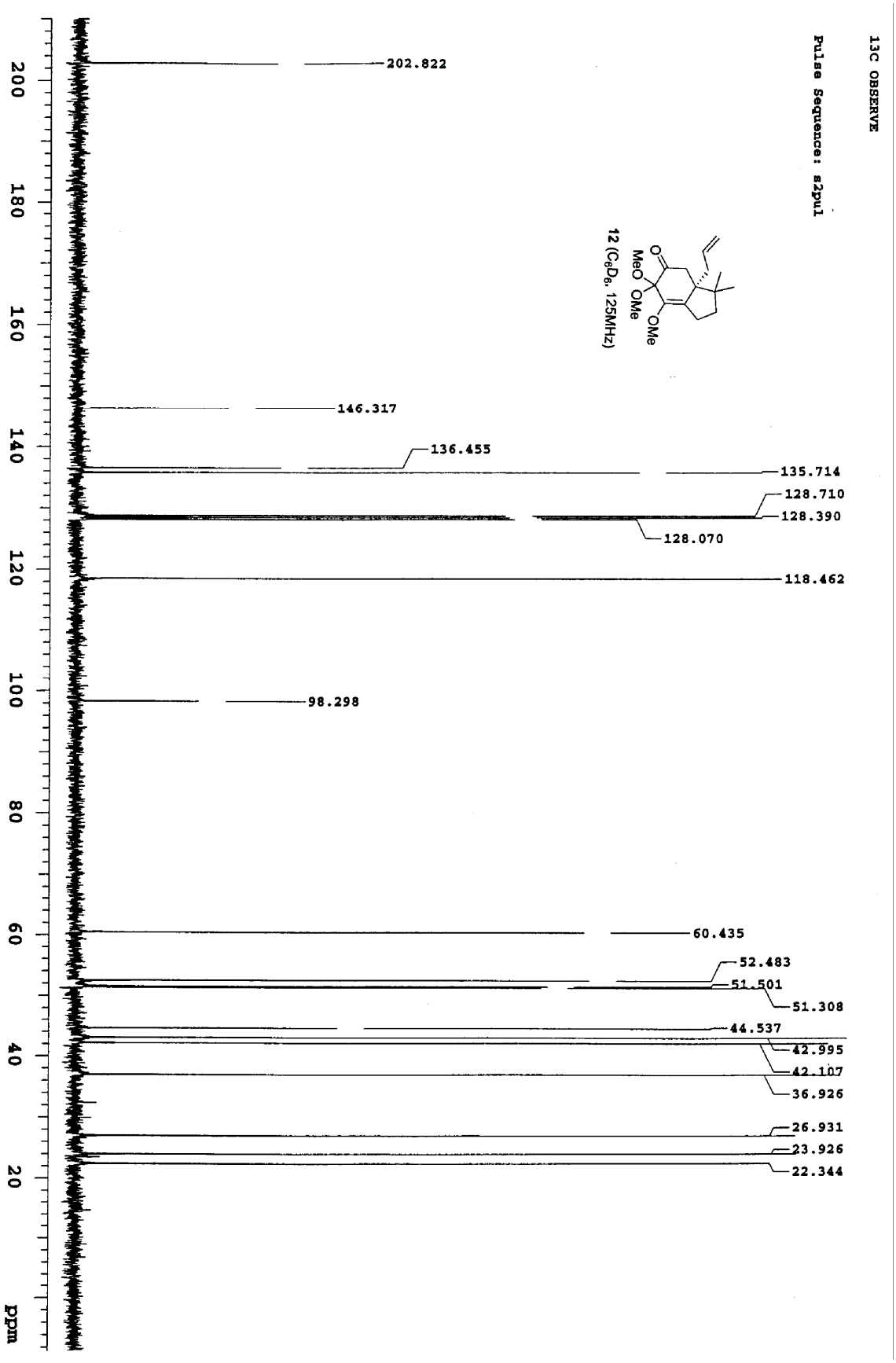

STANDARD 1H OBSERVE
Pulse Sequence: s2pul
200 MHz
cdcl3
4 ($CDCl_3$, 300MHz)
OMe
MeO OMe
5.720
4.034
3.298
2.677
1.703
1.164
H_2O
0.87
2.83
5.82
2.15
2.20
6.12
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm


13C OBSERVE
Pulse Sequence: s2pul
75 MHz
cdcl3
4 (CDCl3, 75MHz)
MeO OMe
OMe
137.707
134.029
131.785
130.450
127.672
111.749
77.657
77.230
76.809
59.163
51.224
51.144
44.127
39.306
27.035
25.352
ppm


STANDARD 1H OBSERVE
Pulse Sequence: s2pul
11 (C_6D_6, 500MHz)
HO
MeO
OMe
OMe
5.303
5.130
5.110
5.057
5.052
5.046
3.601
3.456
3.255
3.114
2.700
2.669
2.663
1.427
1.392
1.355
1.007
0.980
0.80
0.87
2.08
2.79
3.04
2.93
2.00
2.04
2.28
6.16
ppm

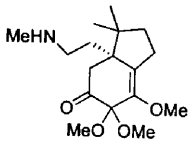

13C OBSERVE
Pulse Sequence: s2pul
75 MHz
benzene-d6
148.838
146.574
135.364
128.380
128.060
127.740
126.424
122.785
117.117
81.203
60.065
51.405
50.657
41.236
40.555
40.362
28.056
27.456
24.845
14.249
ppm
HO
MeO
OMe
OMe
11 (C6D6, 125MHz)


STANDARD 1H OBSERVE
Pulse Sequence: s2pul
12 (C6D6, 500MHz)
MeO
OMe
OMe
3.676
3.675
3.400
3.309
3.308
2.261
0.712
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
1.00
0.97
1.07
2.86
2.88
2.89
1.02
1.05
1.03
1.10
1.94
1.08
1.36
5.76


13C OBSERVE
Pulse Sequence: s2pul
12 (C6D6, 125MHz)
MeO
OMe
OMe
O
202.822
146.317
136.455
135.714
128.710
128.390
128.070
118.462
98.298
60.435
52.483
51.501
51.308
44.537
42.995
42.107
36.926
26.931
23.926
22.344
200
180
160
140
120
100
80
60
40
20
ppm

amine matts project

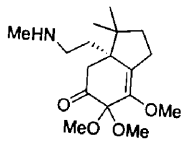
Pulse Sequence: s2pul



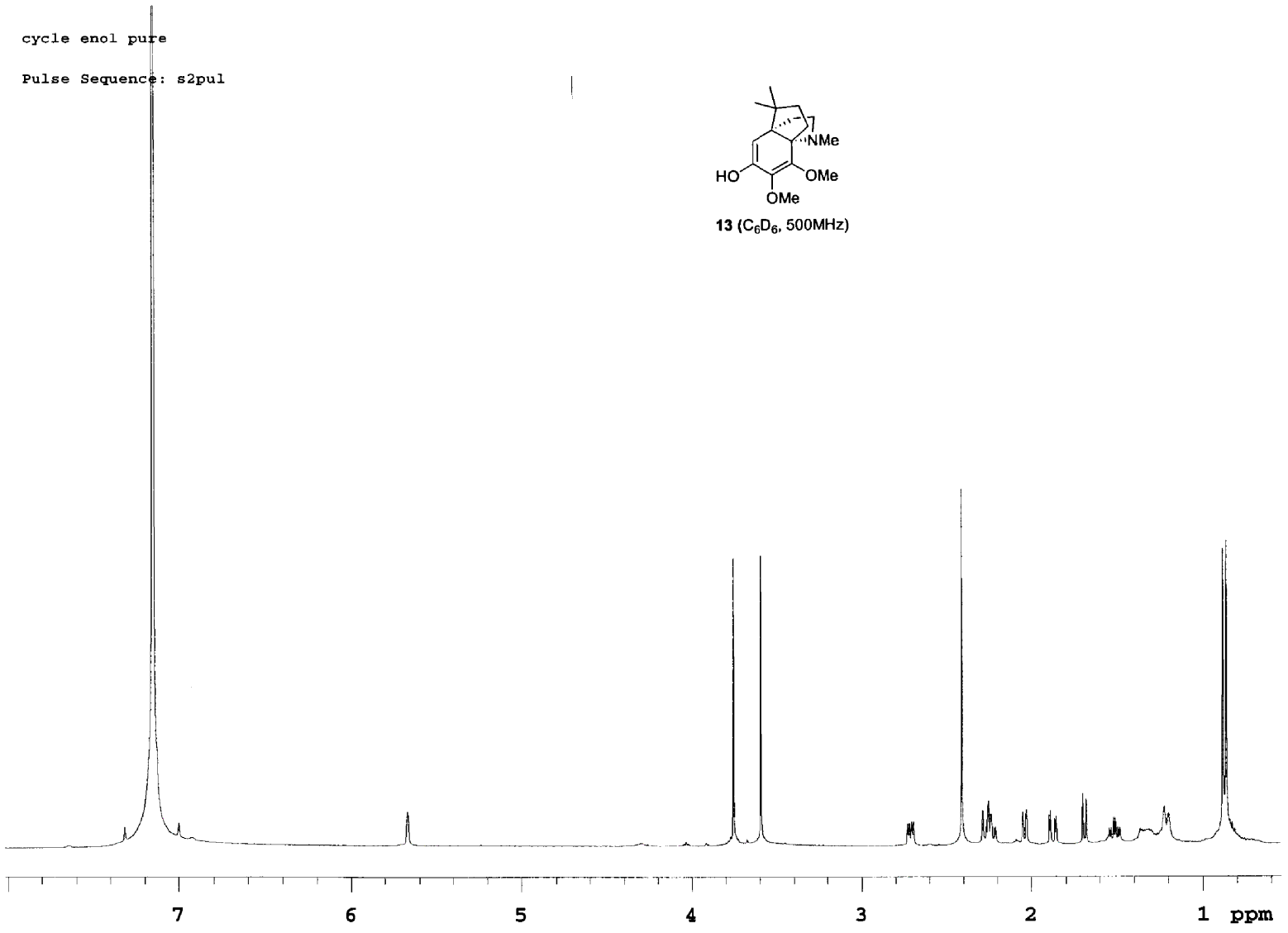
3 (C_6D_6, 500MHz)

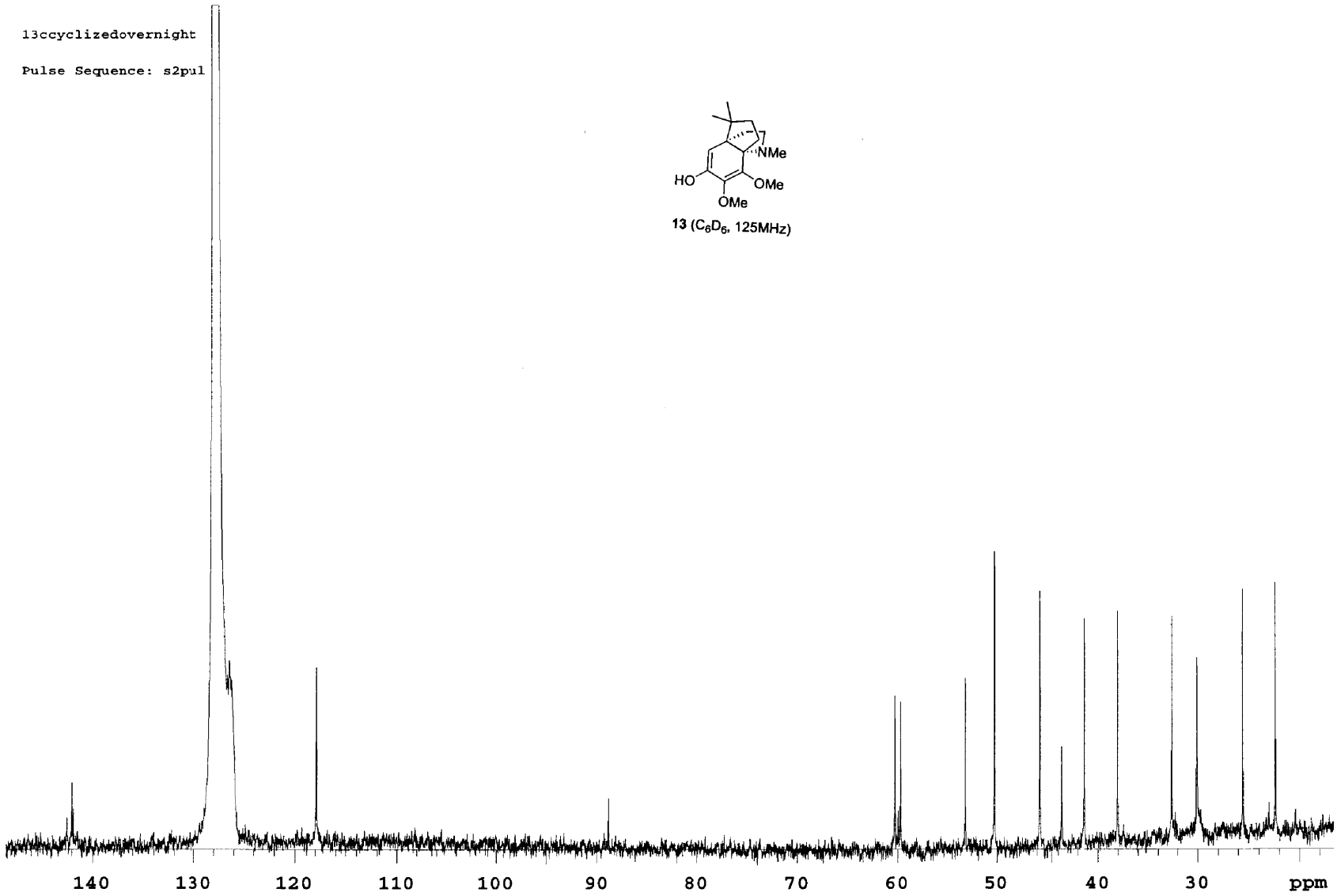
amine13C

Pulse Sequence: s2pul



3 (C_6D_6, 125MHz)


cycle enol pure
Pulse Sequence: s2pul
NMe
HO
OMe
OMe
13 (C6D6, 500MHz)
7
6
5
4
3
2
1
ppm


13ccyclizedovernight
Pulse Sequence: s2pul
NMe
HO
OMe
OMe
13 (C_6D_6, 125MHz)
140
130
120
110
100
90
80
70
60
50
40
30
ppm