Supporting Information For:

Concise Synthesis of the CDE Ring System of Tetrahydroisoquinoline Alkaloids Using Carbophilic Lewis Acid-Catalyzed Hydroamidation and Oxidative Friedel-Crafts Cyclization

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General procedure for Pt(II) and Au(I)-catalyzed 6-*exo* mode cyclization of 1a-d

To a solution of compound **1** (0.10 mmol) in CH_2Cl_2 (0.5 ml) were added $AuCl(PPh_3)$ (0.010 mmol) and $AgNTf_2$ (0.010 mmol) and the mixture was stirred at room temperature for 6 h. After being quenched with aqueous $NaHCO_3$ solution (0.5 ml), the mixture was extracted with $CHCl_3$ (3×1 ml) and the combined organic layers were dried over Na_2SO_4 and concentrated *in vacuo*. The residue was purified by column chromatography on silica gel (Hexane/AcOEt = 3/1 ~ 1/1) to give the desired product **2**.

2-(4-Nitro-*N*-(3-phenylprop-2-ynyl)phenylsulfonamido)-3-phenylpropanamide (1a): ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.98 (2H , d, J = 8.9 Hz), 7.69 (2H, d, J = 8.9 Hz), 7.38–7.07 (10 H , m), 6.36 (1H , br), 5.56 (1H, br),. 4.75–4.69 (2H, m), 4.39 (1H, d, J = 18.6 Hz), 3.42 (2H, dd, J = 14.6, 5.5 Hz), 2.98–2.94 (2H, m); ^{13}C NMR (126 MHz, $CDCl_3$) δ 171.2, 149.7, 145.2, 137.0, 131.3, 129.4, 129.1, 128.6, 128.5, 128.4, 126.8, 123.8, 121.7, 85.9, 83.5, 62.6, 60.3, 34.8, 34.5, 20.9, 14.1; IR ($CHCl_3$) 3492, 3381, 3222, 3108, 1697, 1654, 1607, 1590, 1525, 1496, 1455 cm^{-1}; MS (FAB) m/z 464 (MH^+, 100), 419 (14), 277 (47), 186 (12), 154 (35), 115 (97); HRMS (FAB) calcd for $C_{24}H_{22}N_3O_5S$ (MH^+) 464.1280, found 464.1269.

3-Phenyl-2-(3-phenylprop-2-ynylamino)propanamide (1b): ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.45–7.14 (5H, m), 7.14–6.93 (1H, br), 6.35–6.11 (1H, br), 3.68 (1H, dd, J = 9.8, 4.1 Hz), 3.61 (1H, d, J = 17.3 Hz), 3.46–3.39 (1H, m), 3.29 (1H, dd, J = 14.0, 4.0 Hz), 2.78 (1H, dd, J = 13.9, 9.8 Hz); ^{13}C NMR (125 MHz, $CDCl_3$) δ 176.5, 137.0, 131.5, 129.1, 128.8, 128.5, 128.2, 128.1, 127.4, 126.9, 122.7, 86.4, 83.8, 62.3, 39.3, 37.9; IR ($CHCl_3$) 3388, 3183, 2928, 2857, 1659, 1594 cm^{-1}; MS (FAB) m/z 279 (MH^+, 100), 234 (55), 154 (35), 136 (25), 115 (53), 95 (30), 83 (33); HRMS (FAB) calcd for $C_{18}H_{19}N_2O$ (MH^+) 279.1497, found 279.1497.

Methyl 1-Amino-1-oxo-3-phenylpropan-2-yl(3-phenylprop-2-ynyl)carbamate (1c): ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.38–7.23 (5H, m), 6.24 (1.0H, s), 5.78 (1H, s), 4.94–4.12 (2H, m), 3.94–3.90 (2H, m), 3.76 (3H, s), 3.37 (2H, m); ^{13}C NMR (126 MHz, $CDCl_3$) δ 181.5, 131.9, 129.3, 128.8, 128.5, 127.1, 84.5, 77.3, 63.1, 61.4, 54.3, 39.0, 36.6, 35.1, 34.5, 14.2; IR ($CHCl_3$) 3447, 3370, 2360, 1683 cm^{-1}; MS (FAB) m/z 337 (MH^+, 100), 292 (40), 190 (26), 154 (40), 136 (28), 115 (76); HRMS (FAB) calcd for $C_{20}H_{20}N_2O_3$ (MH^+) 337.1592, found 337.1562.

***N*-(4-Methoxybenzyl)-2-(4-nitro-*N*-(3-phenylprop-2-ynyl)phenylsulfonamido)-3-phenylpropanamide (1d):** ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.33–8.07 (1H, m), 7.64–7.55 (2H, m), 7.55–7.47 (1H, m), 7.33–7.11 (11.2H, m), 6.90 (2H, d, J = 7.0 Hz), 6.75 (2H, d, J = 7.0 Hz), 4.82 (2H, d, J = 18.9 Hz), 4.67 (1H, q, J = 8.8 Hz), 4.35 (1H, m), 3.76 (3H, s), 3.57–3.46 (1H, m), 3.23–3.13 (1H, m); ^{13}C NMR (126 MHz, $CDCl_3$) δ 169.5, 159.0, 136.5, 133.8, 131.9, 131.8, 131.5, 129.6, 129.4, 128.7, 128.6, 128.5, 128.2, 123.9, 114.0, 83.9, 61.6, 55.4, 42.3, 36.7, 34.6.; IR ($CHCl_3$) 3381, 1797, 1732, 1681, 1612, 1597, 1541, 1513, 1448 cm^{-1}; MS (FAB) m/z 584 (MH^+, 27), 397 (12), 307 (27), 289 (15), 154 (100), 136 (65), 115 (27); HRMS (FAB) calcd for $C_{32}H_{29}N_3O_6S$ (MH^+) 584.1855, found 584.1868.

(*Z*)-3-Benzyl-6-benzylidene-4-(4-nitrophenylsulfonyl)piperazin-2-one（2a): ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.20 (2H, d, J = 8.9 Hz), 7.88 (2H, d, J = 8.9 Hz), 7.39–7.19 (10H, m), 6.70 (1H, br), 5.39 (1H, t, J = 6.3 Hz), 5.02 (1H, dt, J = 6.7, 2.8 Hz), 4.36–4.32 (1H, m), 4.16–4.11 (1H, m), 3.31 (1H, dd, J = 14.4, 6.4 Hz), 3.21 (1H, dd, J = 14.4, 6.4 Hz); ^{13}C NMR (126 MHz, $CDCl_3$) δ 171.4, 150.1,

144.5, 137.1, 136.6, 135.1, 129.8, 129.1, 128.9, 128.8, 128.6, 127.4, 126.0, 124.1, 107.8, 63.6, 43.0, 35.8; IR ($CHCl_3$) 2927, 2360, 1667, 1529 cm^{-1}; MS (FAB) m/z 464 (MH^+, 18), 437 (15), 393 (15), 307 (32), 289 (17), 154 (100), 136 (63), 89 (20); HRMS (FAB) calcd for $C_{20}H_{20}N_2O_3$ (MH^+) 337.1592, found 337.1594.

(*Z*)-Methyl 2-Benzyl-5-benzylidene-3-oxopiperazine-1-carboxylate (2c): ^1H NMR (500 MHz, $CDCl_3$) δ 7.54–7.14 (5H, m), 6.85 (1H, br), 5.38 (1H, br), 4.93 (1H, br), 4.61–4.05 (2.0H, m), 4.05–3.54 (3H, s), 3.54–3.09 (2H, m); ^{13}C NMR (126 MHz, $CDCl_3$) δ 172.3, 138.2, 129.2, 128.9, 126.2, 110.3, 62.2, 53.4, 43.4, 34.8, 29.9; IR ($CHCl_3$) 3061, 3028, 1660, 1603, 1494, 1445 cm^{-1}; MS (FAB) m/z 337 (MH^+, 100), 292 (40), 190 (26), 154 (40), 136 (28), 115 (76); HRMS (FAB) calcd for $C_{20}H_{20}N_2O_3$ (MH^+) 337.1592, found 337.1594.

(*E*)-3-Benzyl-6-benzylidene-1-(4-methoxybenzyl)-4-(4-nitrophenylsulfonyl)piperazin-2-one (2d): ^1H NMR (500 MHz, $CDCl_3$) δ 7.96–7.89 (1H, m), 7.74–7.63 (2H, m), 7.63–7.56 (1H, m), 7.50–7.41 (3H, m), 7.41–7.16 (3H, m), 6.82 (2H, d, J = 8.1 Hz), 6.64 (2H, d, J = 8.1 Hz), 6.09–5.95 (1H, m), 5.13–5.07 (1H, m), 5.07–5.00 (1H, m), 4.03 (1H, dd, J = 8.6, 3.4 Hz), 3.91–3.78 (1H, m), 3.71 (3H, s), 3.33–3.23 (1H, m), 3.23–3.03 (1H, m); ^{13}C NMR (126 MHz, $CDCl_3$) δ 169.4, 159.2, 146.0, 136.2, 134.4, 133.6, 132.0, 131.3, 130.6, 130.2, 130.0, 129.3, 128.9, 128.7, 128.3, 127.4, 126.9, 123.9, 115.0, 113.5, 65.8, 55.0, 48.8, 44.0, 40.2 ; IR ($CHCl_3$) 3062, 3027, 2956, 2837, 2359, 1657, 1612, 1586, 1495, 1439 cm^{-1}; MS (FAB) m/z 584 (MH^+, 15), 549 (10), 154 (78), 121 (100), 69 (83); HRMS (FAB) calcd for $C_{32}H_{30}N_3O_6S$ (MH^+) 584.1855, found 584.1833.

Synthesis of alkynylaldehyde 5

3-(2,4,5-Trimethoxy-3-methylphenyl)propiolaldehyde (5). To a solution of compound **3** (3840 mg, 10 mmol) in Et_3N (20 ml) were added $PdCl_2(PPh_3)_2$ (350 mg, 0.50 mmol), CuI (143 mg, 1.0 mmol), and propargyl alcohol (624 mg, 12 mmol) and the resulting mixture was stirred under Ar at room temperature for 12 h. After filtration, Et_3N was removed *in vacuo* and the residue was purified by column chromatography on silica gel (Hexane/AcOEt = 1/1) to give alcohol (2010 mg, 85 %). To a solution of alcohol (920 mg, 3.90 mmol) in CH_2Cl_2 (15 ml) were added RuO_4N(*n*-Pr) (68 mg, 0.19 mmol), *N*-methylmorpholine *N*-oxide (684 mg, 5.85 mmol), and MS4A and the resulting mixture was stirred for 24 h under Ar. After filtration through Celite pad, the mixture was diluted with water (10 ml) and extracted with $CHCl_3$ (3×15 ml). The combined organic layers were dried over Na_2SO_4 and concentrated *in vacuo*. The residue was purified by column chromatography on silica gel (Hexane/AcOEt = 5/1) to give compound **5** (834 mg, 91 %). ^1H NMR (500 MHz, $CDCl_3$) δ 9.44 (s, 1H), 6.91 (s, 1H), 3.88 (s, 3H), 3.86 (s, 3H), 3.83 (s, 3H), 2.18 (s, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 176.2, 156.4, 151.3, 148.7, 126.0, 114.2, 107.1, 92.5, 91.8, 61.2, 60.1, 55.7, 9.1; IR ($CHCl_3$) 3586, 2938, 2851, 2183, 1715 cm^{-1}; MS (FAB) m/z 234 (M^+, 100), 219 (10), 206 (8), 176 (7), 154 (10); HRMS (FAB) calcd for $C_{13}H_{14}O_4$ (M^+) 234.0892, found 234.0913.

Synthesis of amino acid derivative 11

1-(Bromomethyl)-2,4,5-trimethoxy-3-methylbenzene (7). To a solution of **6** (2880 mg, 10.0 mmol) in CH_2Cl_2 (40 ml) were added PPh_3 (3140 mg, 12.0 mmol) and CBr_4 (3980 mg, 12.0 mmol) and the resulting mixture was stirred at room temperature for 8 h. The mixture was diluted with

water (15 ml) and extracted with $CHCl_3$ (3×20 ml). The combined organic layers were dried over Na_2SO_4 and concentrated *in vacuo*. The residue was purified by column chromatography on silica gel (Hexane/AcOEt = 3/1) to give compound **7** (2580 mg, 94 %). ^{1}H NMR (500 MHz, $CDCl_3$) δ 6.73 (s, 1H), 4.56 (s, 2H), 3.84 (d, *J* = 1.1 Hz, 3H), 3.81 (d, *J* = 1.1 Hz, 3H), 3.80 (s, 3H), 2.21 (s, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 150.9, 149.4, 148.6, 125.8, 125.8, 111.2, 61.0, 60.2, 55.9, 29.0, 9.5; IR ($CHCl_3$) 3565, 3280, 1282 cm^{-1}; MS (FAB) m/z 276 (10), 274 (M^+, 10), 226 (25), 195 (100), 149 (22), 136 (15); HRMS (FAB) calcd for $C_{11}H_{15}BrO_3$ (M^+) 274.0205, found 274.0225.

***tert*-Butyl 2-Amino-3-(2,4,5-trimethoxy-3-methylphenyl)propanoate (9).** To a stirred solution of **7** (3500 mg, 10 mmol), iminoester **8** (2360 mg, 8.00 mmol) and Bu_4NHSO_4 (2.0 mmol) in CH_2Cl_2 (20 ml) was slowly added 50 % KOH aq at 0 °C. After being stirred at room temperature for 4.5 h, the mixture was extracted with $CHCl_3$ (3×20 ml). The combined organic layers were dried over Na_2SO_4 and concentrated *in vacuo*. The residue was purified by column chromatography on silica gel (Hexane/AcOEt = 3/1) to give ester (3560 mg, 91 %). To a stirred solution of ester (2000 mg, 6.2 mmol) in THF (50 ml) was slowly added 15 % citric acid (17 ml) at 0 °C and the reaction mixture was stirred at room temperature for 8 h. After being basified with K_2CO_3, the mixture was extracted with AcOEt (3×30 ml). The combined organic layers were dried over Na_2SO_4 and concentrated *in vacuo*. The residue was purified by column chromatography on silica gel (Hexane/AcOEt = 1/1) to give compound **9** (1830 mg, 91 %). ^{1}H NMR (500 MHz, $CDCl_3$) δ 6.59 (1H, s), 3.84 (3H, s), 3.78 (3H, s), 3.69 (3H, s), 3.68–3.59 (1H, m), 3.00 (1H, dd, *J* = 13.5, 8.3 Hz), 2.77 (1H, dd, *J* = 13.5, 8.3 Hz), 2.20 (3H, s), 1.40 (9H, s); ^{13}C NMR (126 MHz, $CDCl_3$) δ 174.4, 151.0, 148.9, 146.6, 125.5, 125.4, 111.4, 80.9, 60.5, 60.1, 55.9, 55.6, 36.0, 27.9, 9.58; IR ($CHCl_3$) 3376, 3063, 3030, 2978, 2935, 2826, 1867, 1732 cm^{-1}; MS (FAB) m/z 326 (MH^+, 55), 270 (97), 224 (33), 195 (100), 181 (17), 57 (13); HRMS (FAB) calcd for $C_{17}H_{28}NO_5$ (MH^+) 326.1967, found 326.1974.

***tert*-Butyl 2-(Benzyloxycarbonylamino)-3-(2,4,5-trimethoxy-3-methylphenyl)propanoate (10).** To a stirred solution of **9** (2000 mg, 6.2 mmol) in CH_2Cl_2 (50 ml) were added Et_3N (1000 μl, 7.4 mmol) and CbzCl (1050 μl, 7.4 mmol) at 0 °C and the reaction mixture was stirred at room temperature for 8 h. After being diluted with water and extracted with AcOEt (3×30 ml), the combined organic layers were dried over Na_2SO_4 and concentrated *in vacuo*. The residue was purified by column chromatography on silica gel (Hexane/AcOEt = 1/1) to give **10** (2700 mg, 95 %). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.38–7.18 (m, 5H), 6.59 (s, 1H), 5.80 (d, *J* = 7.4 Hz, 1H), 5.11–5.02 (m, 2H), 4.50–4.41 (m, 1H), 3.77 (s, 3H), 3.74 (s, 3H), 3.66 (s, 3H), 3.05–2.93 (m, 2H), 2.19 (s, 3H), 1.39 (s, 9H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 170.8, 155.6, 150.7, 148.8, 146.8, 136.3, 128.1, 127.7, 125.0, 124.1, 111.3, 81.3, 77.3, 66.3, 60.3, 59.8, 55.6, 55.3, 32.6, 27.6, 9.4; IR ($CHCl_3$) 3350, 3064, 2977, 2937, 2838, 2604, 1752, 1593 cm^{-1}; MS (FAB) m/z 459 (M^+, 57), 404 (15), 360 (48), 195 (100), 91 (52); HRMS (FAB) calcd for $C_{25}H_{34}NO_7$ (MH^+) 460.2335, found 460.2363.

***tert*-Butyl 1-Amino-1-oxo-3-(2,4,5-trimethoxy-3-methylphenyl)propan-2-ylcarbamate (11).** To a stirred solution of **10** (3400 mg, 7.4 mmol) in CH_2Cl_2 (10 ml) was slowly added TFA (5 ml) at 0 °C and the reaction mixture was stirred at room temperature for 12 h. After the solvents were evaporated, the crude product was resolved in CH_2Cl_2 (30 ml). To the mixture were added ethyl

chloroformate (700 μg, 7.4 mmol) and Et_3N (1000 μl, 7.4 mmol) at 0 °C and the mixture was stirred for 30 min at the same temperature. After addition of 50 ml of aqueous NH_3, the resulting mixture was stirred at room temperature for 12 h, and extracted with AcOEt (3 × 15 ml). The extracts were washed with water and brine, and then concentrated *in vacuo*. The residue was purified by column chromatography on silica gel (Hexane/AcOEt = 1/2) to give compound **11** (2230 mg, 75 %). ^{1}H-NMR ($CDCl_3$) δ 7.34–7.25 (m, 5H), 6.61 (s, 1H), 6.12 (br, 2H), 5.66 (br, 1H), 5.06 (s, 2H), 4.32 (br, 1H), 3.78 (s, 3H), 3.77 (s, 3H), 3.68 (s, 3H), 3.04 (br, 2H), 2.20 (s, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 174.6, 156.4, 150.5, 149.2, 146.8, 136.2, 128.3, 127.9, 127.6, 125.1, 124.4, 111.3, 66.5, 60.4, 59.9, 56.2, 55.6, 32.4, 9.4; IR ($CHCl_3$) 3390, 3346, 3195, 3086, 3063, 3024, 2983, 2932, 2781, 1683, 1660 cm^{-1}; MS (FAB) m/z 402 (M^+, 13), 359 (15), 314 (22), 251 (11), 195 (100), 91 (72); HRMS (FAB) calcd for $C_{21}H_{26}N_2O_6$ (M^+) 402.1791, found 402.1833.

Synthesis of tricyclic fragment 17

3-(2,4,5-Trimethoxy-3-methylphenyl)-2-(3-(2,4,5-trimethoxy-3-methylphenyl)prop-2-ynylamino)propanamide (12). To a stirred solution of **11** (100 mg, 0.25 mmol) in MeOH (2 ml) was added $Pd(OH)_2$ (20 mg) and the reaction mixture was stirred at room temperature for 12 h under H_2 balloon. After being filtrated through a pad of Celite, the filtrate was concentrated *in vacuo*. The obtained residue was resolved in MeOH (2 ml) and then **5** (63 mg, 0.25 mmol) was added to the mixture. After the mixture was stirred at room temperature for 30 min, $NaBH_4$ (10 mg, 0.27 mmol) was slowly added at 0 °C over 1 h and the resulting mixture was stirred at room temperature for 9 h. After the solvents were removed under reduced pressure, water was added to the residue and the crude mixture was extracted with AcOEt (3×3 ml), The extracts were dried over $MgSO_4$ and concentrated *in vacuo*. The residue was purified by column chromatography on silica gel (Hexane/AcOEt = 1/2) to give **12** (86 mg, 71 %). ^{1}H NMR ($CDCl_3$) δ 6.69 (s, 1H), 6.68 (s, 1H), 3.81–3.65 (m, 20H), 3.53 (d, J = 17.2 Hz, 1H), 3.11 (d, J = 9.7 Hz, 1H), 2.96 (d, J = 9.7 Hz, 1H), 2.18 (s, 3H), 2.13 (s, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 176.8, 154.2, 150.9, 149.4, 148.8, 148.5, 146.8, 125.7, 125.3, 125.0, 113.4, 111.3, 111.0, 89.6, 80.0, 62.7, 60.7, 60.3, 60.1, 55.9, 55.8, 38.3, 33.6, 9.7, 9.3; IR ($CHCl_3$) 3387, 3334, 3184, 3058, 2909, 2859, 1659, 1594 cm^{-1}; MS (FAB) m/z 486 (M^+, 8), 425 (22), 368 (25), 312 (33), 219 (43), 195 (100), 154 (24), 136 (20); HRMS (FAB) calcd for $C_{26}H_{34}N_2O_7$ (M^+) 486.2366, found 486.2327.

Isopropyl

1-Amino-1-oxo-3-(2,4,5-trimethoxy-3-methylphenyl)propan-2-yl(3-(2,4,5-trimethoxy-3-methylphenyl)prop-2-ynyl)carbamate (13). To a solution of **12** (60 mg, 0.12 mmol) in CH_2Cl_2 (1 ml) were added diisopropylethylamine (132 μl, 0.76 mmol) and isopropyl chloroformate (43 μl, 0.35 mmol) and the mixture was stirred at same temperature for 6 h. After an aqueous $NaHCO_3$ solution (1 ml) was added, the mixture was extracted with $CHCl_3$ (3×1 ml) and the combined organic layers were dried over Na_2SO_4 and then concentrated *in vacuo*. The residue was purified by column chromatography on silica gel (Hexane/AcOEt = 3/1 ~ 1/1) to give **13** (61 mg, 88 %). ^{1}H NMR (500 MHz, $CDCl_3$) δ: 6.72 (s, 1H), 6.51 (s, 1H), 5.39 (s, 1H), 4.98 (s, 1H), 4.60 (m, 1H), 4.35 (m, 1H), 3.82–3.63 (m, 18H), 3.37 (m, 2H), 2.20 (s, 3H), 2.16 (s, 3H), 1.29 (m, 6H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 173.2, 173.0, 154.8, 154.7, 154.3, 154.1, 150.6, 149.0, 148.9, 148.7, 146.6, 146.5, 125.8,

125.6, 125.2, 113.0, 111.5, 111.2, 110.6, 110.5, 88.0, 87.7, 80.9, 80.3, 77.2, 69.8, 69.5, 61.0, 60.6, 60.5, 60.1, 60.0, 55.8, 55.7, 38.6, 37.2, 30.0, 29.5, 29.1, 21.9, 21.6, 9.4, 9.2; IR ($CHCl_3$) 2961, 2875, 2839, 1784, 1716 cm^{-1}; MS (FAB) m/z 573 (MH^+, 5), 529 (5), 485 (8), 346 (12), 321 (8), 271 (22), 209 (78), 195 (100), 91 (33); HRMS (FAB) calcd for $C_{30}H_{41}N_2O_9$ (MH^+) 573.2812, found 573.2789.

(*Z*)-Isopropyl 3-Oxo-2-(2,4,5-trimethoxy-3-methylbenzyl)-5-(2,4,5-trimethoxy-3-methylbenzylidene)piperazine-1-carboxylate (14). To a solution of **13** (60 mg, 0.10 mmol) in CH_2Cl_2 (0.5 ml) were added $AuCl(PPh_3)$ (5.0 mg, 0.010 mmol) and $AgNTf_2$ (4.8 mg, 0.010 mmol) and the mixture was stirred at room temperature for 6 h. After being quenched with an aqueous $NaHCO_3$ solution (0.5 ml), the mixture was extracted with $CHCl_3$ (3×1 ml) and the combined organic layers were dried over Na_2SO_4 and then concentrated *in vacuo*. The residue was purified by column chromatography on silica gel (Hexane/AcOEt = 3/1 ~ 1/1) to give **14** (48 mg, 85 %). ^1H NMR (500 MHz, $CDCl_3$) δ 6.67 (s, 1H), 6.61 (s, 1H), 5.27 (s, 1H), 4.96–4.86 (m, 2H), 4.18–4.00 (m, 2H), 3.98–3.84 (m, 4H), 3.81 (s, 3H), 3.79 (s, 3H), 3.78 (s, 3H), 3.68 (s, 3H), 3.66 (s, 3H), 3.33 (s, 3H), 2.21 (s, 3H), 2.19 (s, 3H), 1.34-1.26 (m, 6H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 173.2, 155.7, 150.8, 149.1, 149.0, 148.3, 146.7, 133.5, 126.9, 126.2, 125.2, 124.9, 111.5, 110.9, 110.5, 77.2, 69.6, 61.3, 60.7, 60.4, 60.2, 60.1, 56.0, 55.8, 44.8, 29.5, 22.0, 9.5, 9.3; IR ($CHCl_3$) 3235, 2983, 2840, 1772, 1700, 1635 cm^{-1}; MS (FAB) m/z 573 (MH^+, 8), 529 (5), 485 (10), 346 (10), 321 (10), 271 (15), 219 (55), 195 (100), 181 (17), 165 (15), 91 (25); HRMS (FAB) calcd for $C_{30}H_{41}N_2O_9$ (MH^+) 573.2812, found 573.2766.

(*Z*)-Isopropyl 4-Benzyl-3-oxo-2-(2,4,5-trimethoxy-3-methylbenzyl)-5-(2,4,5-trimethoxy-3-methylbenzylidene)piperazine-1-carboxylate (16). To a solution of **14** (60 mg, 0.10 mmol) in DMF (0.5 ml) were added BnBr (5.0 mg, 0.010 mmol) and NaH (4.8 mg, 0.010 mmol) at 0 °C and the mixture was stirred at the same temperature for 1 h. After being quenched with an aqueous $NaHCO_3$ solution (0.5 ml), the mixture was extracted with $CHCl_3$ (3×1 ml) and the combined organic layers were dried over Na_2SO_4 and then concentrated *in vacuo*. The residue was purified by column chromatography on silica gel (Hexane/AcOEt = 3/1 ~ 1/1) to give **16** (63 mg, 95 %). ^1H NMR (500 MHz, $CDCl_3$) δ 7.22–7.13 (m, 3H), 7.00 (d, J = 6.9 Hz, 2H), 6.71 (s, 1H), 6.19 (s, 1H), 5.73 (t, J = 5.2 Hz, 1H), 5.06 (t, J = 6.9 Hz, 1H), 4.86–4.78 (m, 1H), 4.65–4.47 (m, 3H), 3.95–3.82 (m, 4H), 3.80 (s, 3H), 3.63 (s, 3H), 3.59 (s, 3H), 3.44 (s, 3H), 3.39 (s, 3H), 3.36–3.25 (m, 1H), 3.35–3.23 (m, 1H), 2.20 (s, 3H), 2.18 (s, 3H), 1.22 (d, J = 6.3 Hz, 3H), 1.08 (br, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 171.2, 155.2, 150.7, 148.9, 148.8, 148.4, 146.5, 140.0, 137.7, 128.2, 127.9, 127.1, 126.2, 126.0, 125.0, 124.8, 118.4, 111.4, 111.0, 77.3, 69.3, 61.9, 60.4, 60.3, 60.1, 60.1, 55.9, 55.7, 48.9, 44.6, 30.9, 22.1, 21.8, 9.7, 9.6; IR ($CHCl_3$) 2979, 1698, 1650 cm^{-1}; MS (FAB) m/z 663 (MH^+, 45), 571 (27), 467 (27), 381 (16), 326 (100), 195 (50), 154 (47), 136 (35), 91 (65); HRMS (FAB) calcd for $C_{37}H_{47}N_2O_9$ (MH^+) 663.3238, found 663.3285.

(*E*)-3-Benzyl-2-[(2,4,5-trimethoxy-3-methylphenyl)-methylene]-7,9,10-trimethoxy-8-methyl-4-oxo-1,2,3,4,5,6-hexahydro-1,5-imino-3-benzazocine-11-carboxylic acid isopropyl ester (17). To a solution of **16** (33 mg, 0.05 mmol) in MeCN (0.10 ml) was added NBS (11 mg, 0.06 mmol) and the mixture was stirred at 60 °C for 15 min. After being quenched with an aqueous $NaHCO_3$

solution (0.5 ml), the mixture was extracted with $CHCl_3$ (3×1 ml) and the combined organic layers were dried over Na_2SO_4 and then concentrated *in vacuo*. The residue was purified by column chromatography on silica gel (Hexane/AcOEt = 3/1 ~ 1/1) to give compound **17** (23 mg, 70 %). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.52 (s, 1H), 7.11–6.99 (m, 3H), 6.79 (s, 1H), 6.72–6.62 (m, 2H), 6.08 (s, 1H), 5.71 (d, *J* = 14.9 Hz, 1H), 5.33–5.19 (m, 1H), 5.12–4.93 (m, 1H), 4.55 (d, *J* = 15.5 Hz, 1H), 3.99 (s, 3H), 3.79 (s, 3H), 3.69 (s, 3H), 3.45 (s, 3H), 3.39 (d, *J* = 17.2 Hz, 1H), 3.14–3.03 (m, 1H), 2.98 (s, 3H), 2.88 (s, 3H), 2.19 (s, 3H), 2.17 (s, 3H), 1.33 (d, *J* = 5.7 Hz, 3H), 1.28 (d, *J* = 5.7 Hz, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 168.3, 152.9, 152.7, 150.7, 150.3, 149.2, 146.8, 146.4, 136.3, 134.6, 128.4, 126.7, 126.1, 125.4, 125.2, 125.1, 124.7, 121.7, 110.2, 107.6, 77.2, 69.6, 60.3, 60.1, 59.9, 59.5, 59.0, 56.5, 53.4, 45.8, 43.7, 28.2, 22.2, 9.3, 9.2; IR ($CHCl_3$) 2936, 2830, 1698, 1672, 1639 cm^{-1}; MS (FAB) m/z 661 (M^+, 100), 575 (5), 278 (22), 234 (33), 204 (15), 91 (22); HRMS (FAB) calcd for $C_{37}H_{45}N_2O_9$ (M^+) 660.3047, found 660.3027.

(*Z*)-4-Benzyl-1-[(isopropyloxy)carbonyl]-6-[(2,4,5-trimethoxy-3-methylphenyl)methyl]-3-[(2,4, 5-trimethoxy-3-methylphenyl)methylene]-2,5-piperazinedione (18). To a solution of **16** (33 mg, 0.05 mmol) in MeOH (0.10 ml) was added CAN (11 mg, 0.06 mmol) and the mixture was stirred at room temperature for 1 h. After being quenched with an aqueous $NaHCO_3$ solution (0.2 ml), the mixture was extracted with $CHCl_3$ (3×1 ml) and dried over Na_2SO_4 and then concentrated *in vacuo*. The residue was resolved in HCO_2H (0.20 ml) and the mixture was stirred at 60 ºC for 1h. The reaction mixture was diluted with water and extracted with $CHCl_3$ (3×1 ml). The extracts were dried over Na_2SO_4 and concentrated *in vacuo*. The residue was purified by column chromatography on silica gel (Hexane/AcOEt = 3/1 ~ 1/1) to give **18** (5.08 mg, 15%) and **17** (4.95 mg, 15%). **18**: ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.30 (s, 2H), 6.95–6.87 (m, 2H), 6.82 (s, 2H), 6.52 (s, 1H), 5.31 (d, *J* = 14.9 Hz, 1H), 5.22 (t, *J* = 7.1 Hz, 1H), 5.11–4.94 (m, 1H), 4.19 (d, *J* = 14.9 Hz, 1H), 3.95 (s, 3H), 3.88 (s, 3H), 3.81 (s, 3H), 3.65 (s, 3H), 3.64 (s, 3H), 3.48 (s, 3H), 3.34–3.21 (m, 2H), 2.19 (s, 3H), 2.03 (s, 3H), 1.32 (d, *J* = 6.1 Hz, 3H), 1.26 (d, *J* = 6.1 Hz, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 166.7, 161.8, 152.5, 151.5, 150.9, 149.5, 149.0, 148.8, 147.4, 135.8, 128.4, 128.3, 127.4, 127.4, 125.5, 125.4, 122.4, 120.9, 120.0, 111.9, 110.6, 77.2, 71.6, 61.6, 60.6, 60.2, 60.0, 59.7, 56.0, 55.8, 47.5, 32.4, 21.4, 21.4, 9.5, 9.2; IR ($CHCl_3$) 2959, 2874, 2836, 1775, 1722, 1689, 1615 cm^{-1}; MS (FAB) m/z 677 (MH^+, 18), 591 (12), 195 (100), 154 (30), 136 (25), 91 (52); HRMS (FAB) calcd for $C_{37}H_{45}N_2O_{10}$ (MH^+) 677.3074, found 677.3062.

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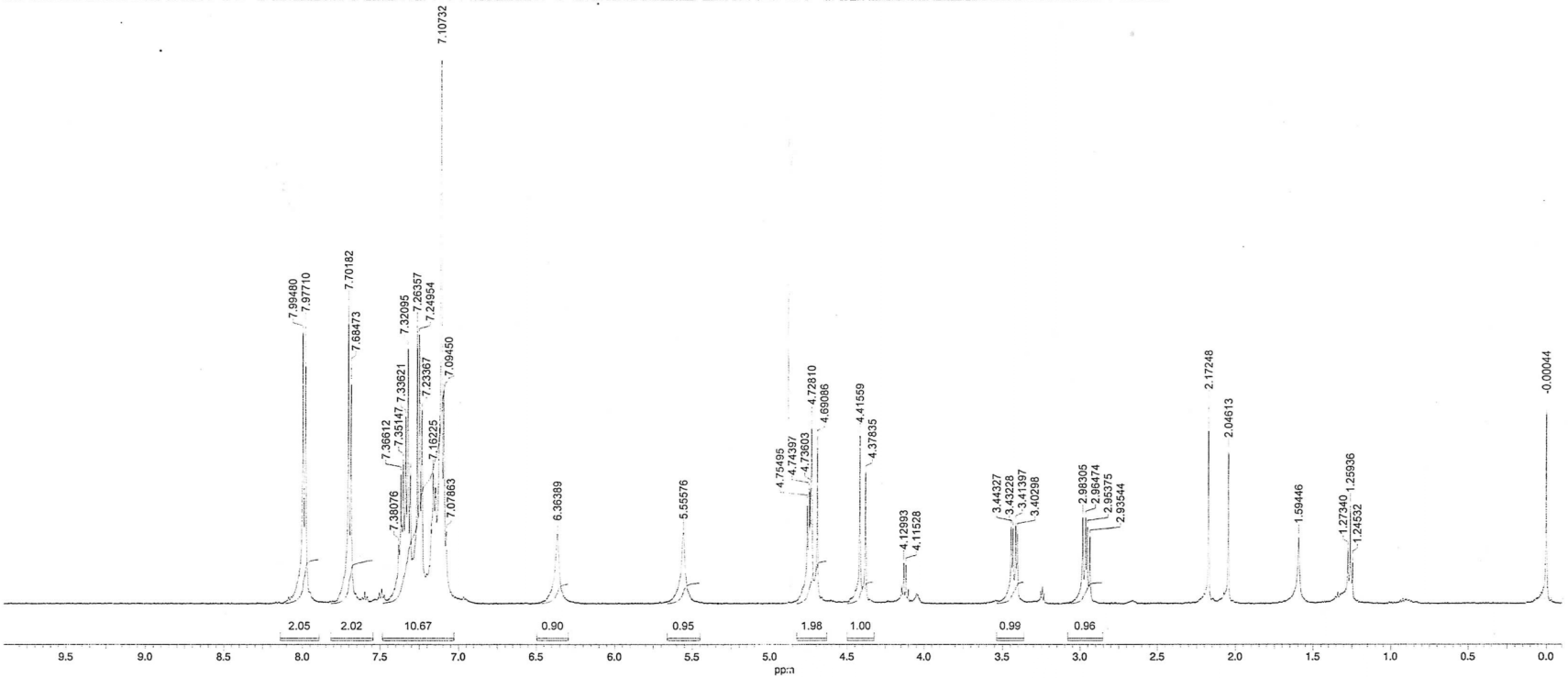

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3	1.26	629.7	0.1772	19	4.42	2207.8	0.3062	35	7.23	3616.8	0.3545
4	1.27	636.7	0.1005	20	4.69	2345.4	0.3058	36	7.25	3624.8	0.4922
5	1.59	797.2	0.1205	21	4.73	2364.0	0.3394	37	7.26	3631.8	0.5099
6	2.05	1023.1	0.2780	22	4.74	2368.0	0.2155	38	7.31	3653.5	0.2405
7	2.17	1086.2	0.3665	23	4.74	2372.0	0.2095	39	7.32	3660.5	0.4681
8	2.94	1467.7	0.1227	24	4.75	2377.5	0.1870	40	7.34	3668.1	0.3429
9	2.95	1476.9	0.1402	25	5.56	2777.9	0.1294	41	7.35	3675.7	0.2629
10	2.96	1482.4	0.1572	26	6.36	3181.9	0.1268	42	7.36	3680.3	0.1444
11	2.98	1491.5	0.1572	27	7.08	3539.3	0.1319	43	7.37	3683.1	0.2386
12	3.40	1701.5	0.1347	28	7.09	3547.3	0.3912	44	7.38	3690.4	0.1087
13	3.41	1707.0	0.1489	29	7.11	3553.7	1.0000	45	7.68	3842.4	0.4378
14	3.43	1716.1	0.1370	30	7.12	3561.3	0.3283	46	7.70	3850.9	0.5503
15	3.44	1721.6	0.1400	31	7.15	3572.6	0.1955	47	7.98	3988.5	0.5090
16	4.12	2057.6	0.0719	32	7.15	3574.4	0.2113	48	7.99	3997.4	0.4977

No.	(ppm)	Value	Absolute Value
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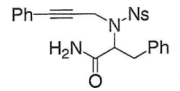
3	[4.32 .. 4.50]	1.000	8.45528e+2
4	[4.63 .. 4.82]	1.978	1.67248e+3
5	[5.45 .. 5.66]	0.951	8.03832e+2
6	[6.30 .. 6.50]	0.904	7.64460e+2
7	[7.03 .. 7.48]	10.670	9.02148e+3
8	[7.54 .. 7.82]	2.018	1.70628e+3
9	[7.89 .. 8.14]	2.047	1.73057e+3



1a

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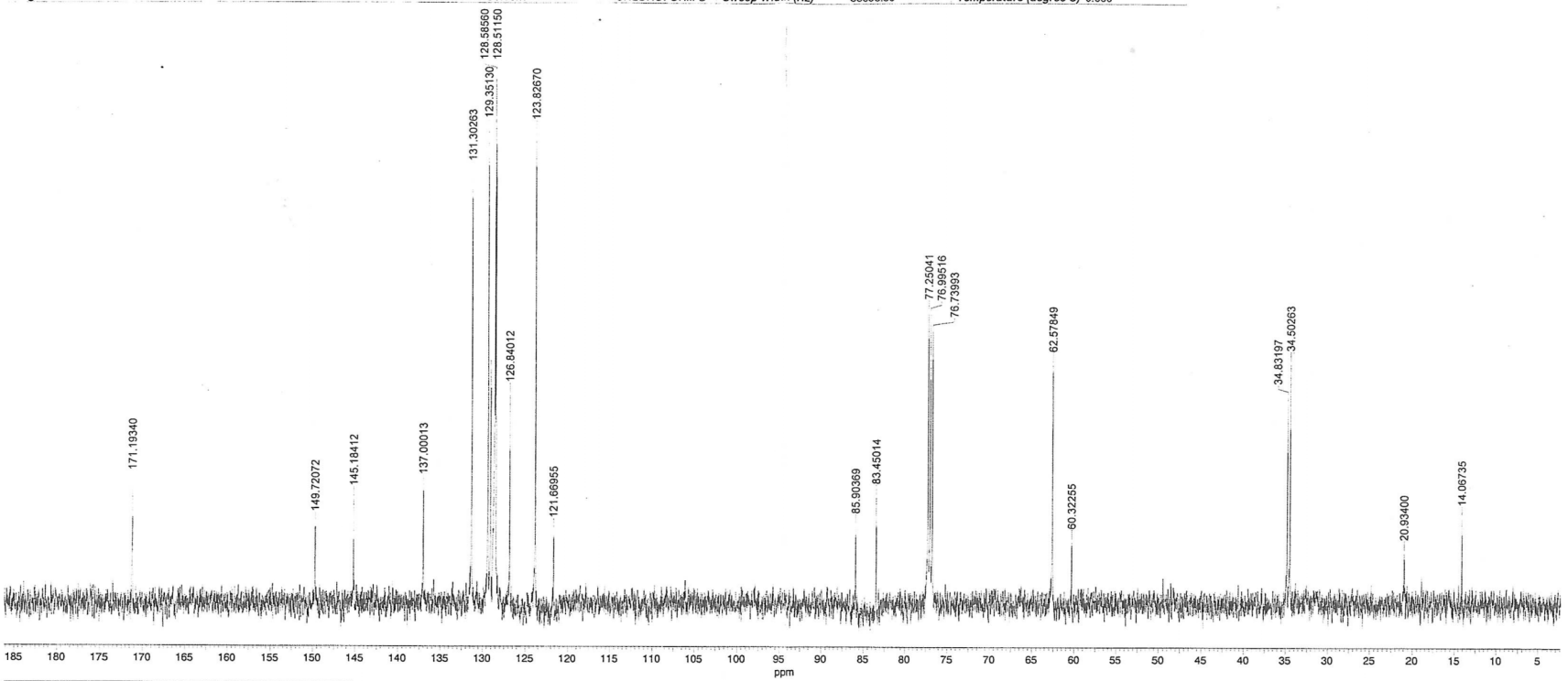

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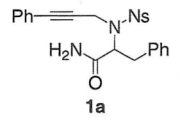
3	34.50	4335.3	0.4096	15	128.45	16139.2	0.7829
4	34.83	4376.6	0.3526	16	128.51	16147.5	0.9131
5	60.32	7579.5	0.1053	17	128.59	16156.8	1.0000
6	62.58	7863.0	0.4070	18	129.05	16214.7	0.3982
7	76.74	9642.4	0.4657	19	129.35	16253.0	0.8042
8	77.00	9674.4	0.4940	20	131.30	16498.2	0.7313
9	77.25	9706.5	0.4966	21	137.00	17214.1	0.1959
10	83.45	10485.5	0.1809	22	145.18	18242.4	0.1758
11	85.90	10793.8	0.1304	23	149.72	18812.4	0.1323
12	121.67	15287.8	0.1225	24	171.19	21510.5	0.2017



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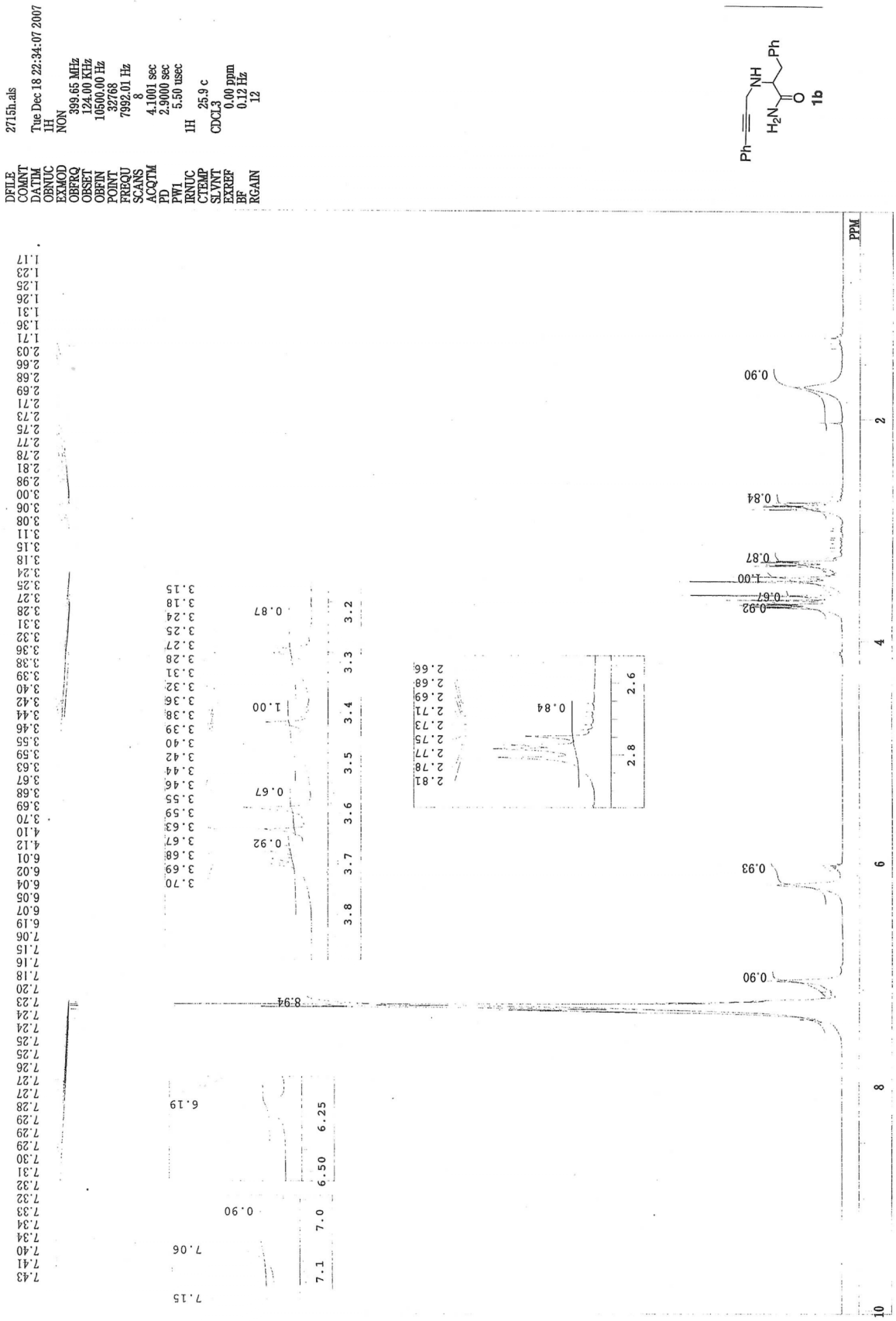
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25.9 c
CDCl3
0.00 ppm
0.12 Hz
12
Ph
NH
Ph
O
H_2N
1b
PPM
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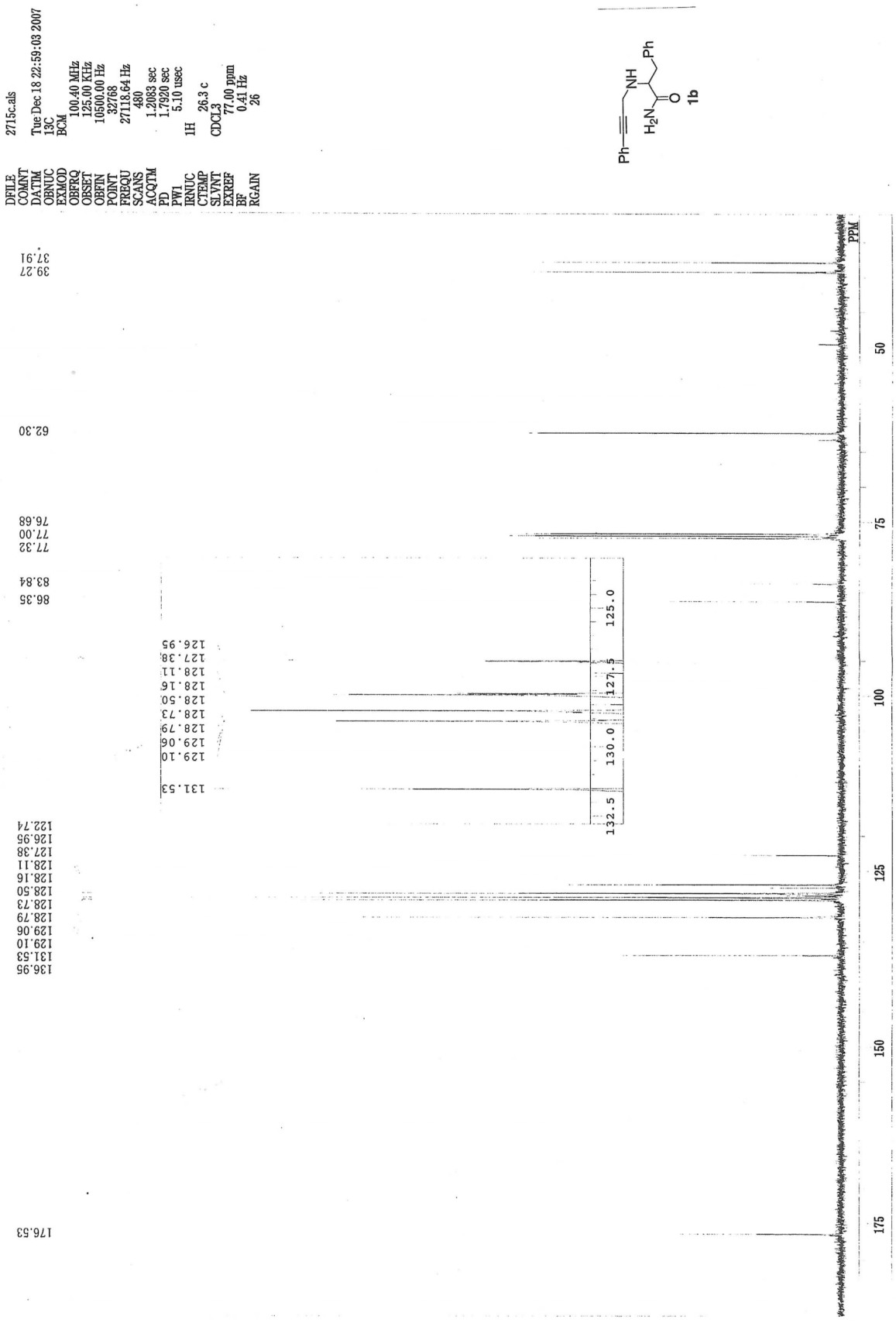
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Ph
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O
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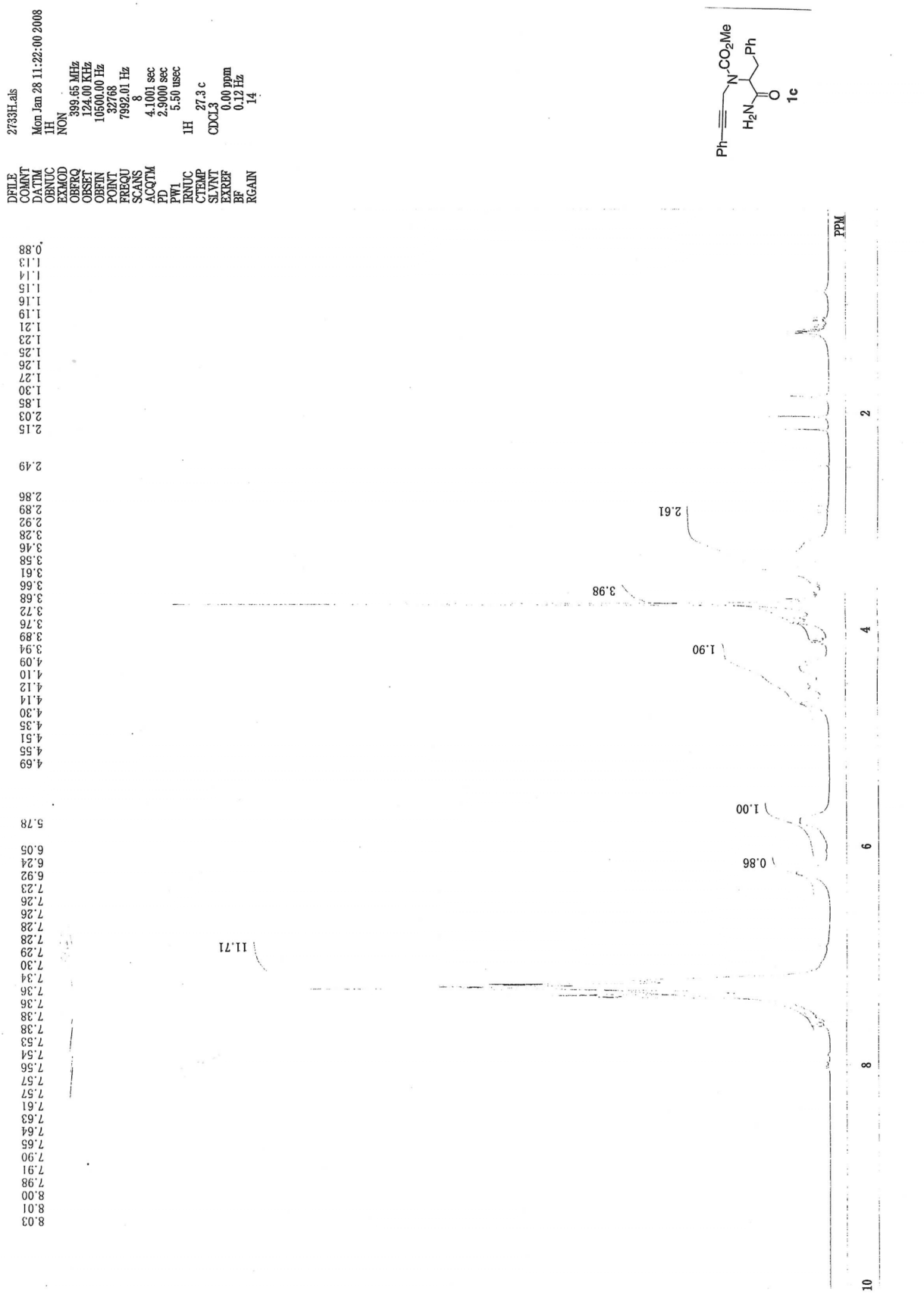
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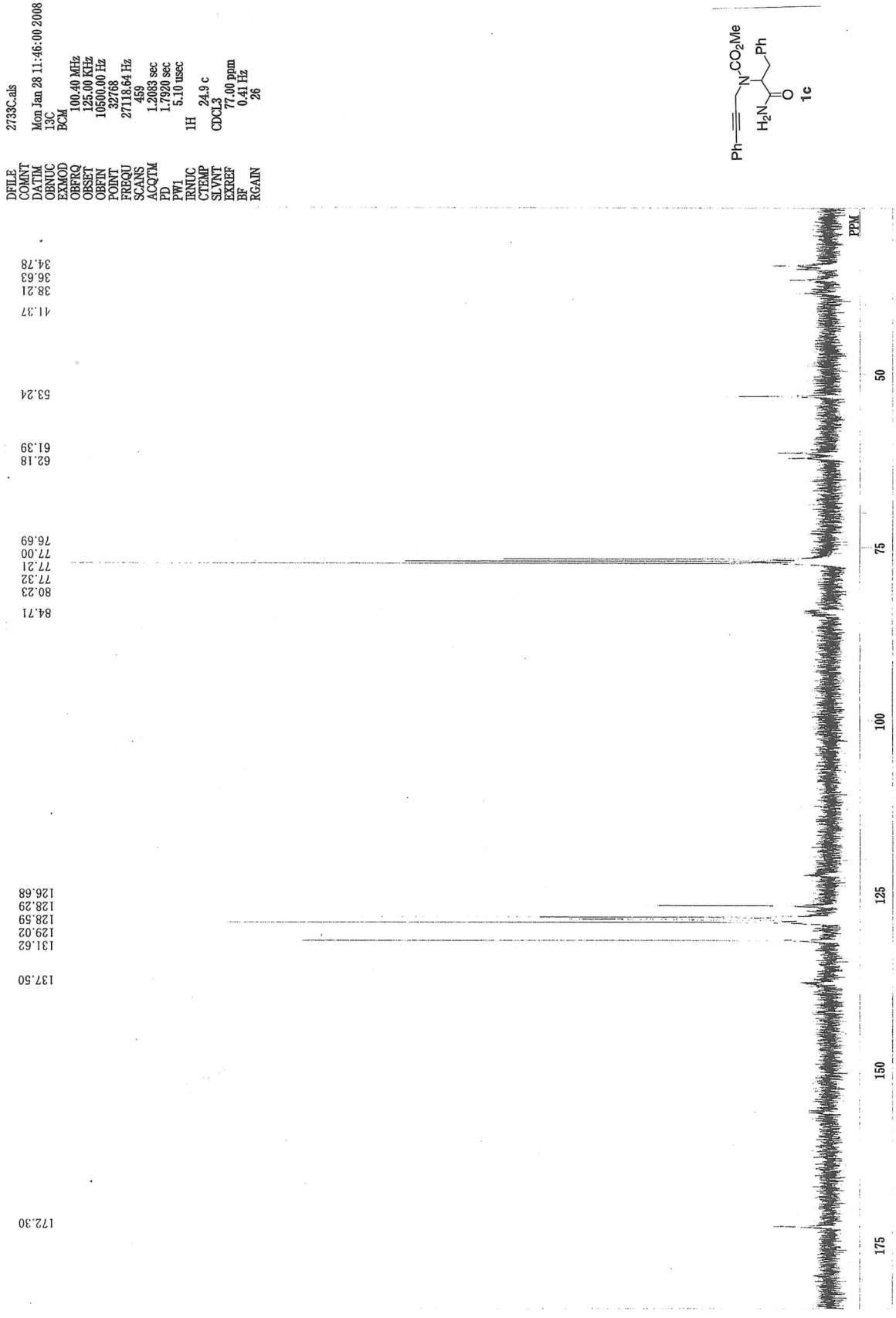
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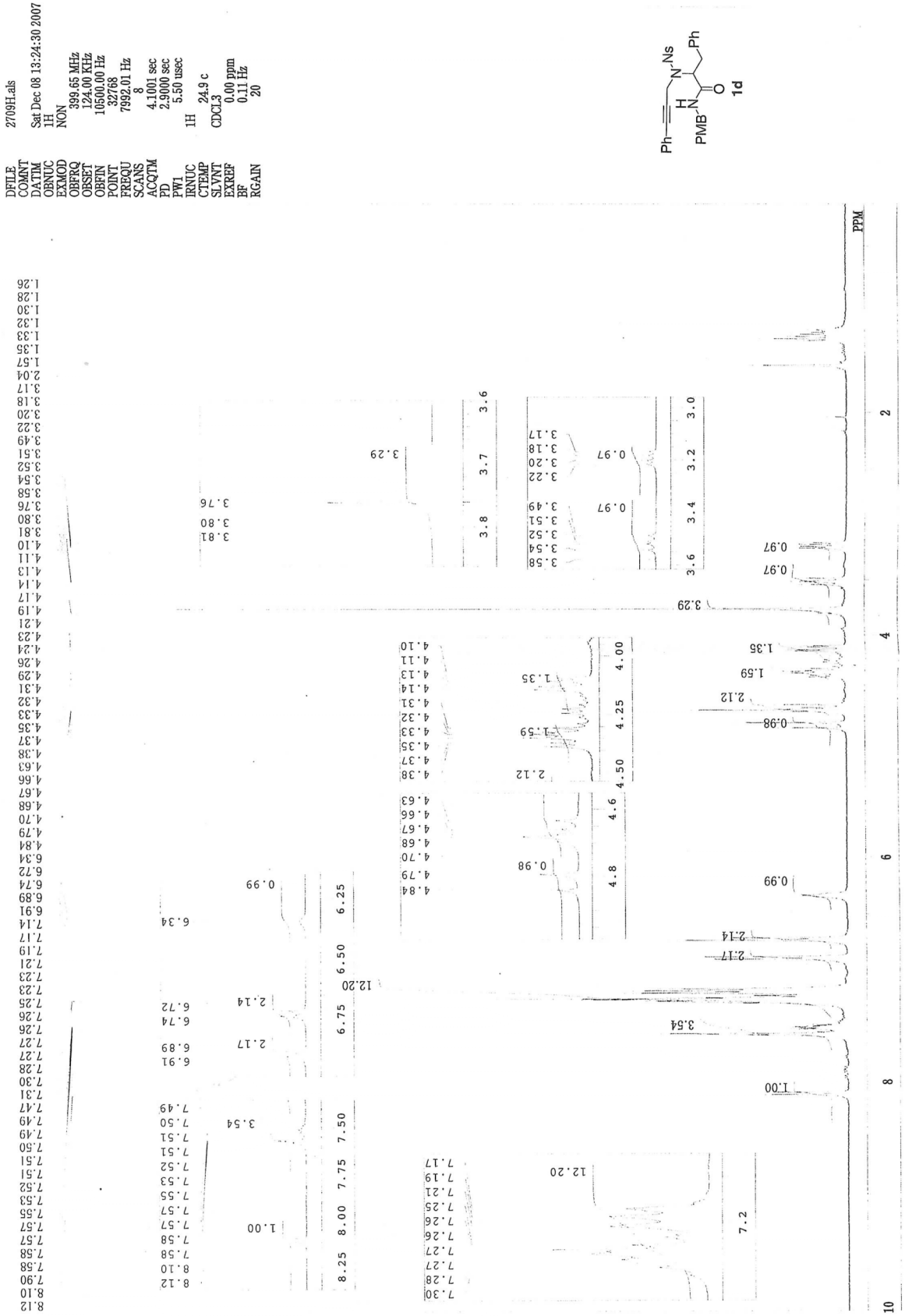
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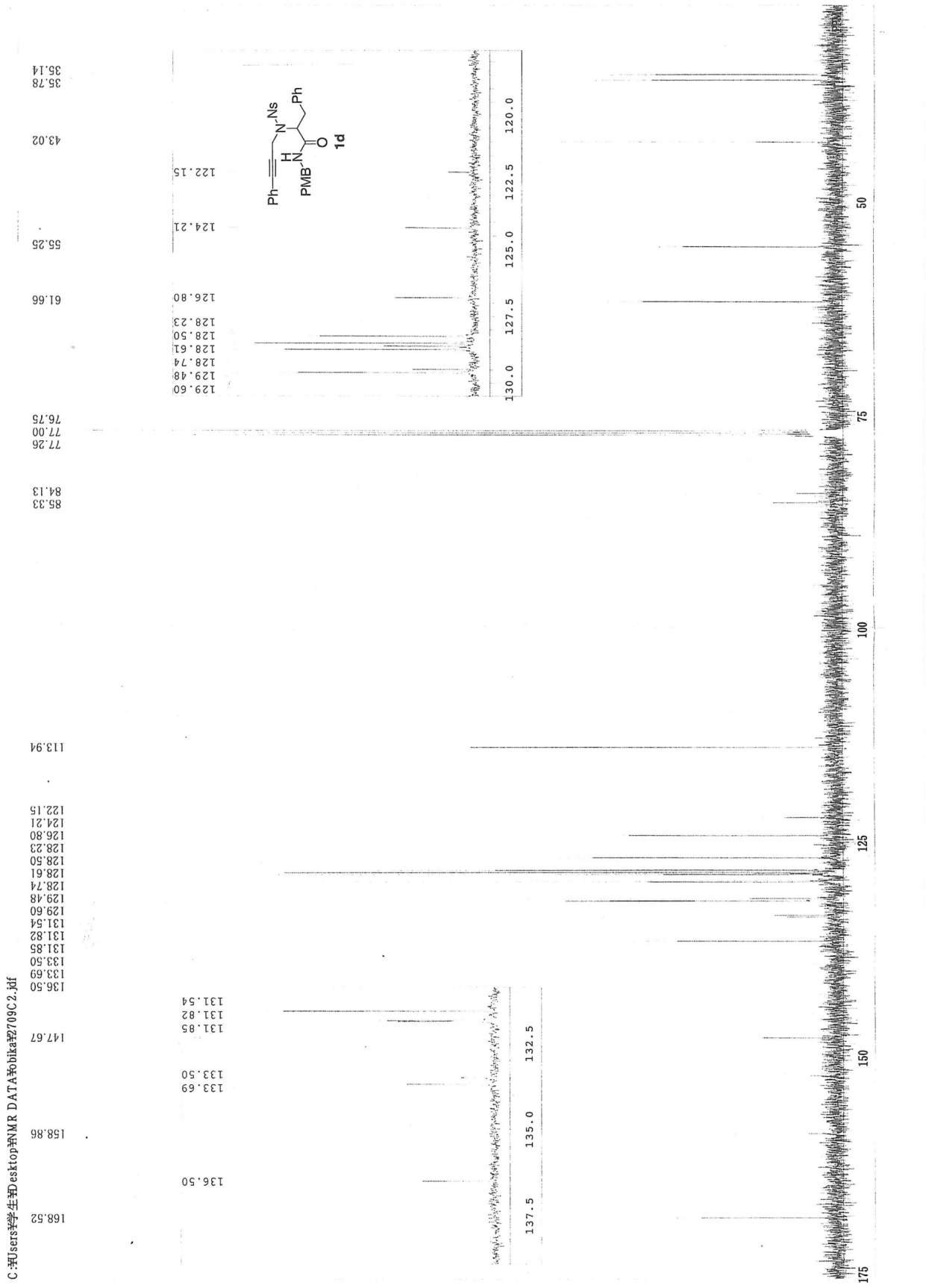
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168.52
ppm

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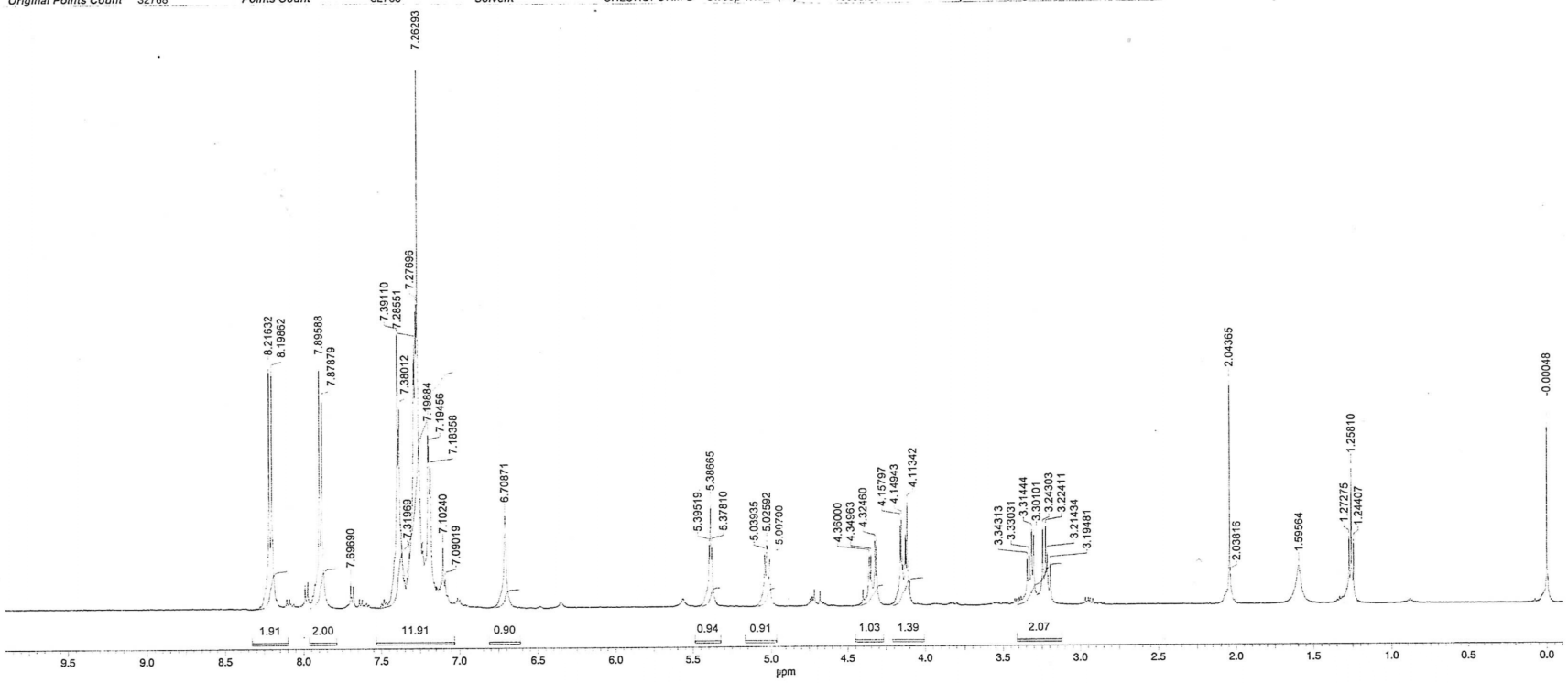
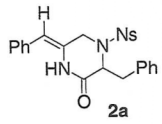
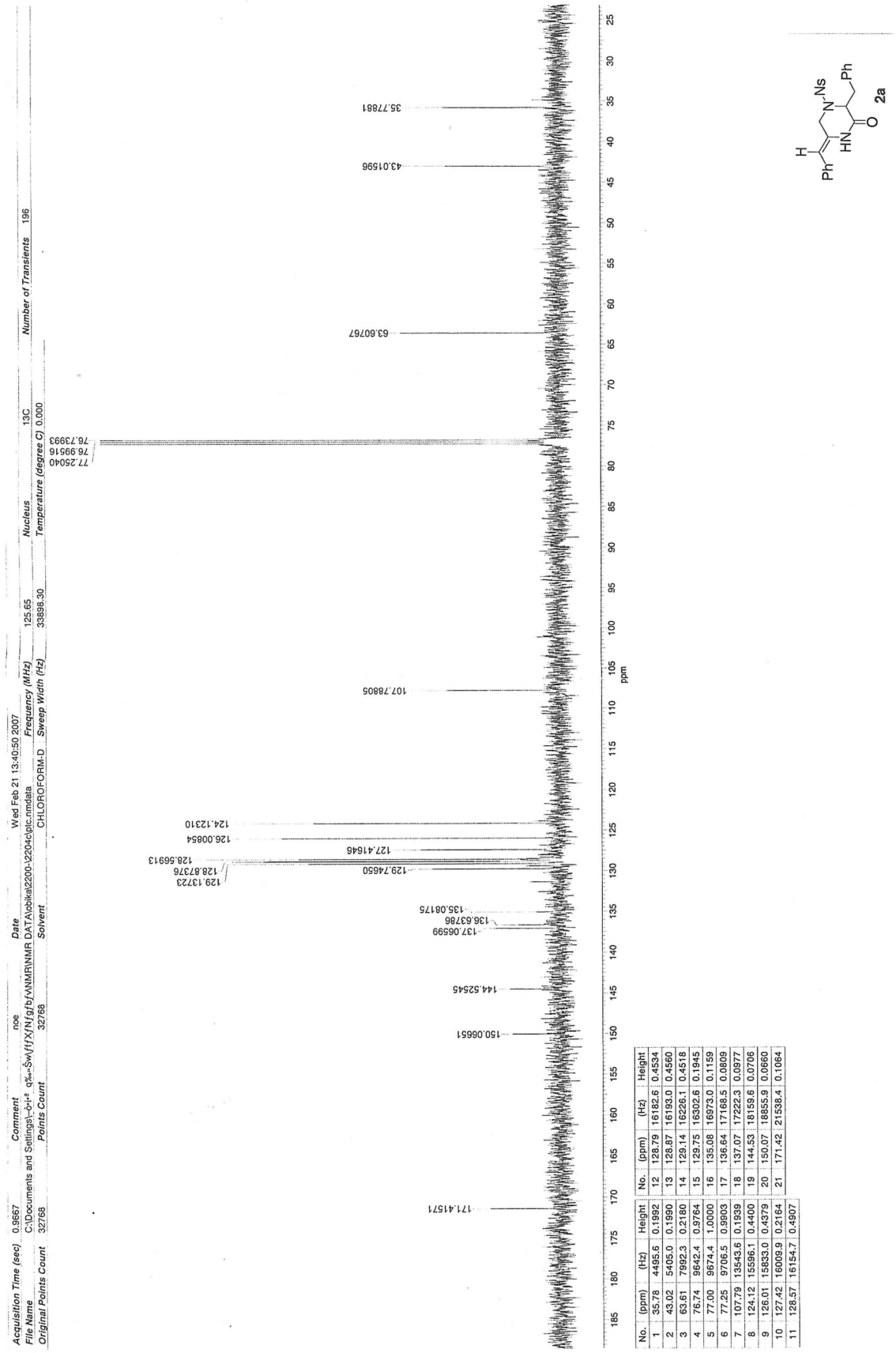


No.	(ppm)	(Hz)	Height	No.	(ppm)	(Hz)	Height	No.	(ppm)	(Hz)	Height
1	-0.00	-0.2	0.3522	22	4.31	2157.1	0.1125	43	7.28	3638.5	0.5605
2	1.24	622.0	0.1168	23	4.32	2162.3	0.1190	44	7.29	3642.8	0.4880
3	1.26	629.1	0.2489	24	4.35	2174.8	0.0938	45	7.29	3644.6	0.4479
4	1.27	636.4	0.1225	25	4.36	2180.0	0.0917	46	7.32	3659.8	0.1034
5	1.60	797.8	0.0694	26	5.01	2503.5	0.0876	47	7.33	3667.2	0.0569
6	2.04	1019.1	0.0543	27	5.02	2510.2	0.1098	48	7.35	3675.1	0.0527
7	2.04	1021.8	0.4089	28	5.03	2513.0	0.1118	49	7.36	3682.4	0.1001
8	3.19	1597.4	0.0778	29	5.04	2519.7	0.0957	50	7.37	3685.5	0.1439
9	3.21	1607.2	0.0960	30	5.38	2689.1	0.1146	51	7.37	3687.0	0.1556
10	3.22	1612.1	0.1447	31	5.39	2693.3	0.1875	52	7.38	3690.1	0.3670
11	3.24	1621.5	0.1447	32	5.40	2697.6	0.1134	53	7.39	3692.8	0.3113
12	3.30	1650.5	0.1286	33	6.71	3354.4	0.1684	54	7.39	3695.6	0.5019
13	3.31	1657.2	0.1370	34	7.09	3545.1	0.0544	55	7.39	3697.4	0.5017
14	3.33	1665.2	0.0942	35	7.10	3551.2	0.1124	56	7.70	3848.4	0.0503
15	3.34	1671.6	0.0846	36	7.18	3591.8	0.2567	57	7.88	3939.4	0.3830
16	4.11	2056.7	0.1919	37	7.19	3597.3	0.2970	58	7.90	3947.9	0.4410
17	4.12	2061.0	0.1324	38	7.20	3599.4	0.3173	59	8.19	4096.9	0.1275
18	4.13	2063.7	0.1304	39	7.23	3616.2	0.1052	60	8.20	4099.3	0.4363
19	4.14	2071.1	0.0759	40	7.24	3618.6	0.1038	61	8.22	4108.2	0.4405
20	4.15	2074.7	0.1620	41	7.26	3631.5	1.0000				
21	4.16	2079.0	0.1572	42	7.27	3634.8	0.5579				

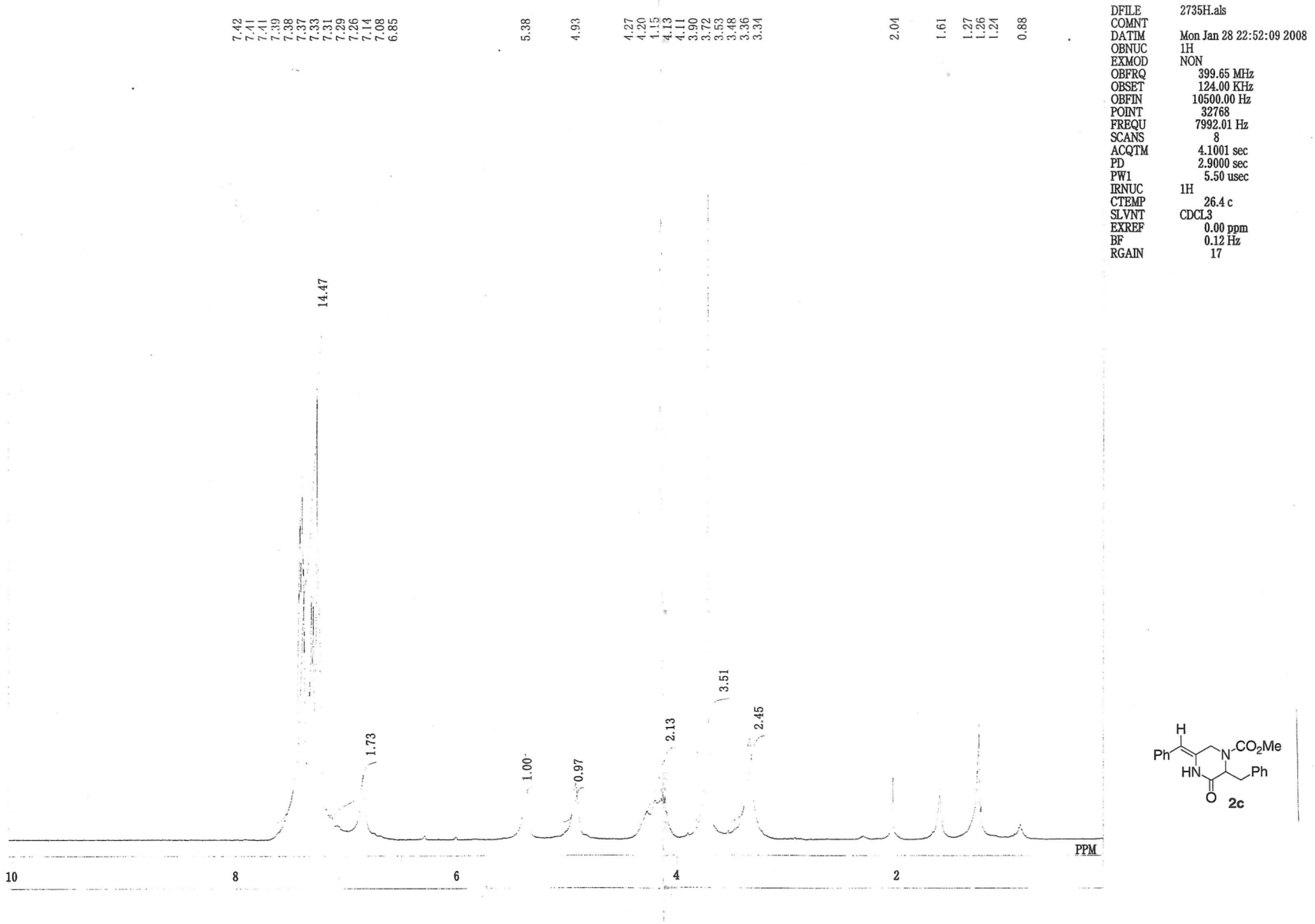


2a



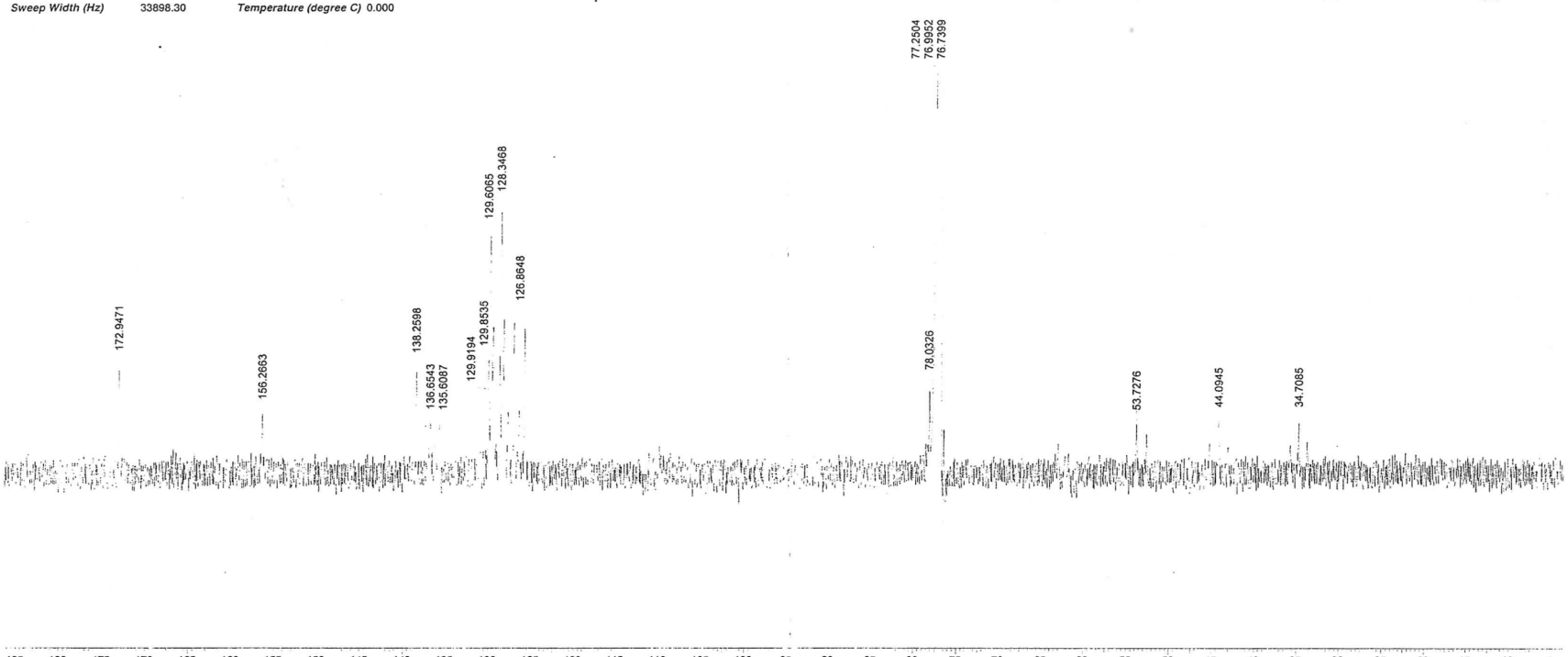
No.	(ppm)	(Hz)	Height
1	35.78	4495.6	0.1992
2	43.02	5405.0	0.1990
3	63.61	7992.3	0.2180
4	76.74	9642.4	0.9764
5	77.00	9674.4	1.0000
6	77.25	9706.5	0.9903
7	107.79	13543.6	0.1939
8	124.12	15596.1	0.4400
9	126.01	15833.0	0.4379
10	127.42	16009.9	0.2164
11	128.57	16154.7	0.4907

No.	(ppm)	(Hz)	Height
12	128.79	16182.6	0.4534
13	128.87	16193.0	0.4560
14	129.14	16226.1	0.4518
15	129.75	16302.6	0.1945
16	135.08	16973.0	0.1159
17	136.64	17168.5	0.0809
18	137.07	17222.3	0.0977
19	144.53	18159.6	0.0706
20	150.07	18855.9	0.0660
21	171.42	21538.4	0.1064

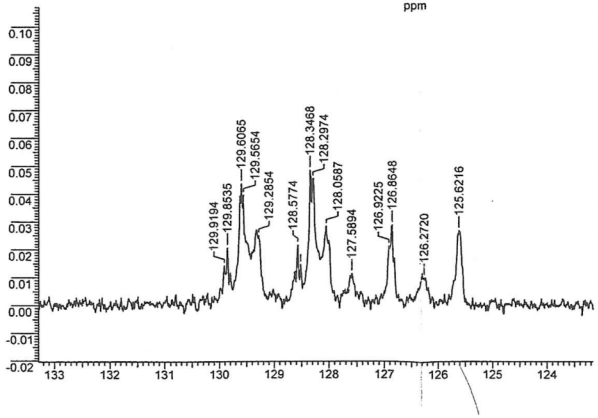

7.42
7.41
7.41
7.39
7.38
7.37
7.33
7.31
7.29
7.26
7.14
7.08
6.85
5.38
4.93
4.27
4.20
4.15
4.13
4.11
3.90
3.72
3.53
3.48
3.36
3.34
2.04
1.61
1.27
1.26
1.24
0.88
14.47
1.73
1.00
0.97
2.13
3.51
2.45
PPM
10
8
6
4
2
DFILE 2735H.als
COMNT
DATIM Mon Jan 28 22:52:09 2008
OBNUC 1H
EXMOD NON
OBFRQ 399.65 MHz
OBSET 124.00 KHz
OBFIN 10500.00 Hz
POINT 32768
FREQU 7992.01 Hz
SCANS 8
ACQTM 4.1001 sec
PD 2.9000 sec
PW1 5.50 usec
IRNUC 1H
CTEMP 26.4 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 17
H
Ph
N
CO2Me
HN
Ph
O
2c

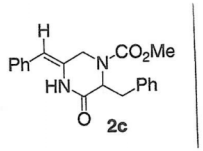
29 Jan 2008

Acquisition Time (sec)	0.9667	*Comment*	noe	*Date*	Tue Jan 29 09:01:43 2008	*File Name*	C:\Users\Šw ĩ\Desktop\NMR\NMR DATA\obika\2700-\2735C\co2me-kanka-c.nmdata
Frequency (MHz)	125.65	*Nucleus*	13C	*Number of Transients*	10430 *Original Points Count* 32768	*Points Count*	32768 *Solvent* CHLOROFORM-D
Sweep Width (Hz)	33898.30	*Temperature (degree C)*	0.000				

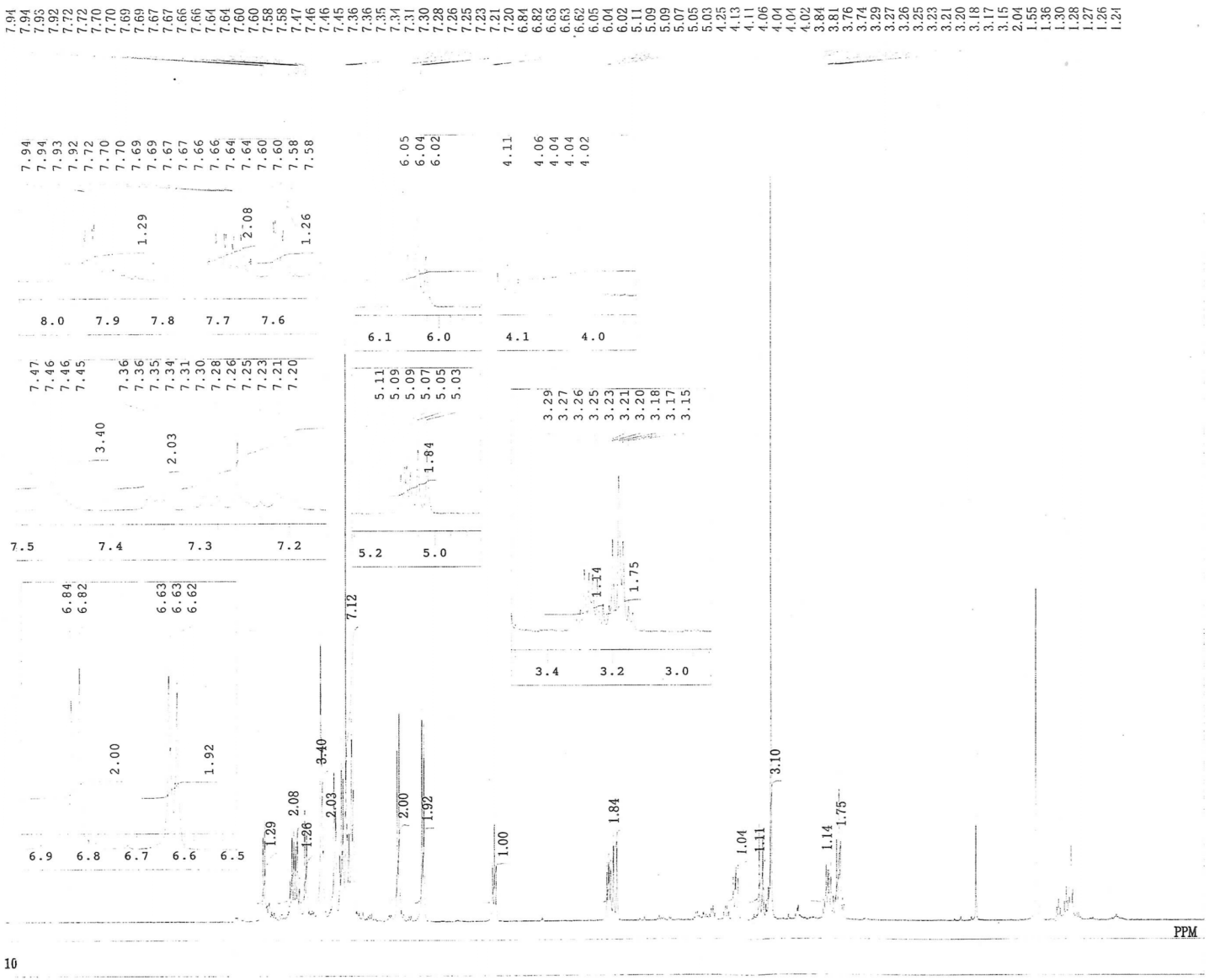


No.	(ppm)	(Hz)	Height	No.	(ppm)	(Hz)	Height
1	34.71	4361.1	0.0097	13	128.58	16155.7	0.0217
2	44.09	5540.5	0.0097	14	129.29	16244.7	0.0251
3	53.73	6750.9	0.0091	15	129.57	16279.9	0.0396
4	76.74	9642.4	0.9544	16	129.61	16285.1	0.0435
5	77.00	9674.4	1.0000	17	129.85	16316.1	0.0206
6	77.25	9706.5	0.9942	18	129.92	16324.4	0.0142
7	78.03	9804.8	0.0163	19	135.61	17039.2	0.0090
8	126.86	15940.6	0.0288	20	136.65	17170.6	0.0092
9	126.92	15947.8	0.0211	21	138.26	17372.3	0.0194
10	128.30	16120.6	0.0456	22	156.27	19634.9	0.0110
11	128.35	16126.8	0.0485	23	172.95	21730.8	0.0198
12	128.52	16148.5	0.0147				

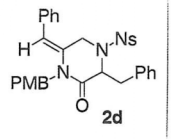




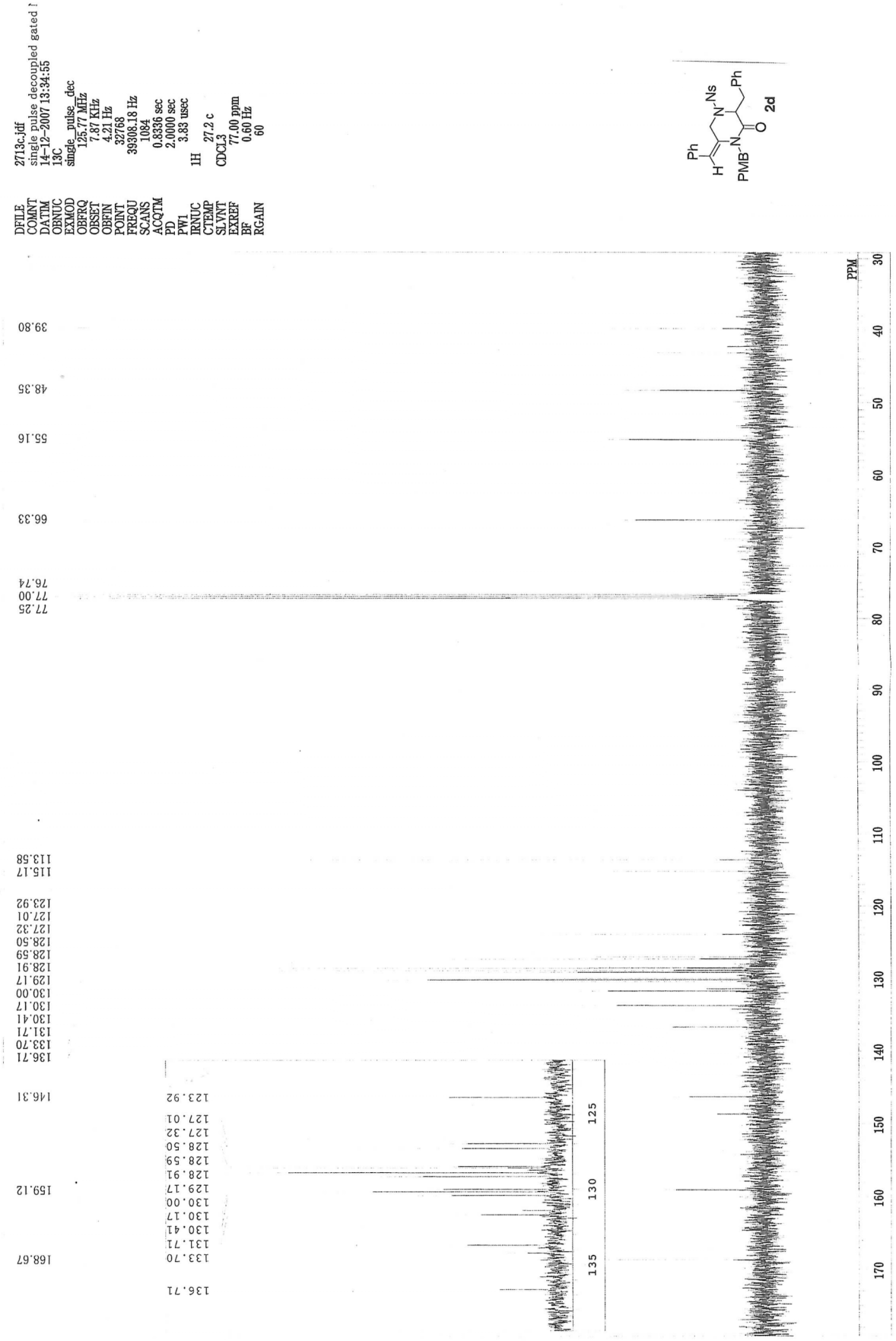
2c

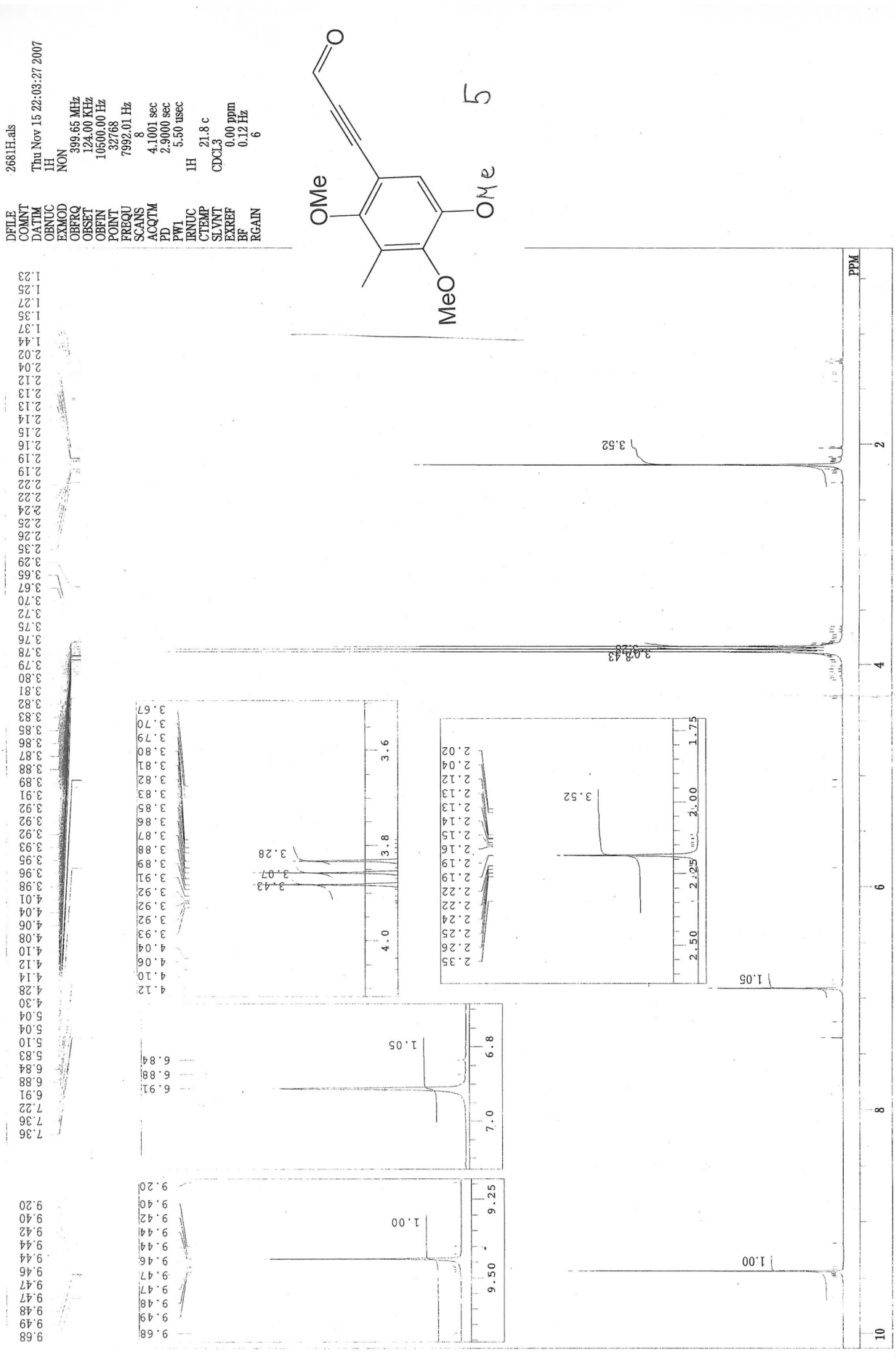


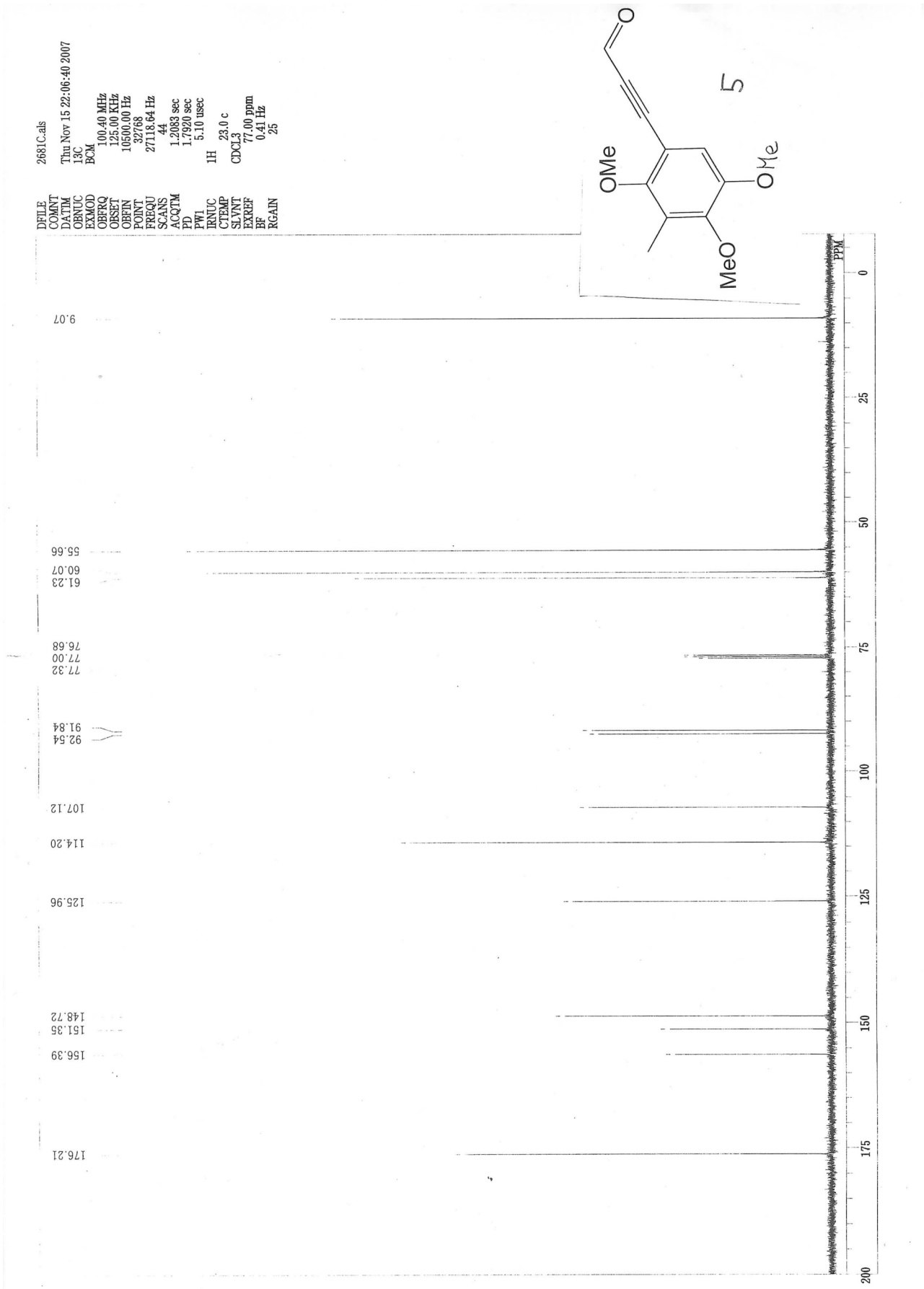
DFILE	2713h2.jdf
COMNT	single_pulse
DATIM	14-12-2007 12:42:21
OBNUC	1H
EXMOD	single_pulse.ex2
OBFRQ	500.16 MHz
OBSET	2.41 KHz
OBFIN	6.01 Hz
POINT	16384
FREQU	9384.38 Hz
SCANS	8
ACQTM	1.7459 sec
PD	5.0000 sec
PW1	6.05 usec
IRNUC	1H
CTEMP	26.5 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.28 Hz
RGAIN	46

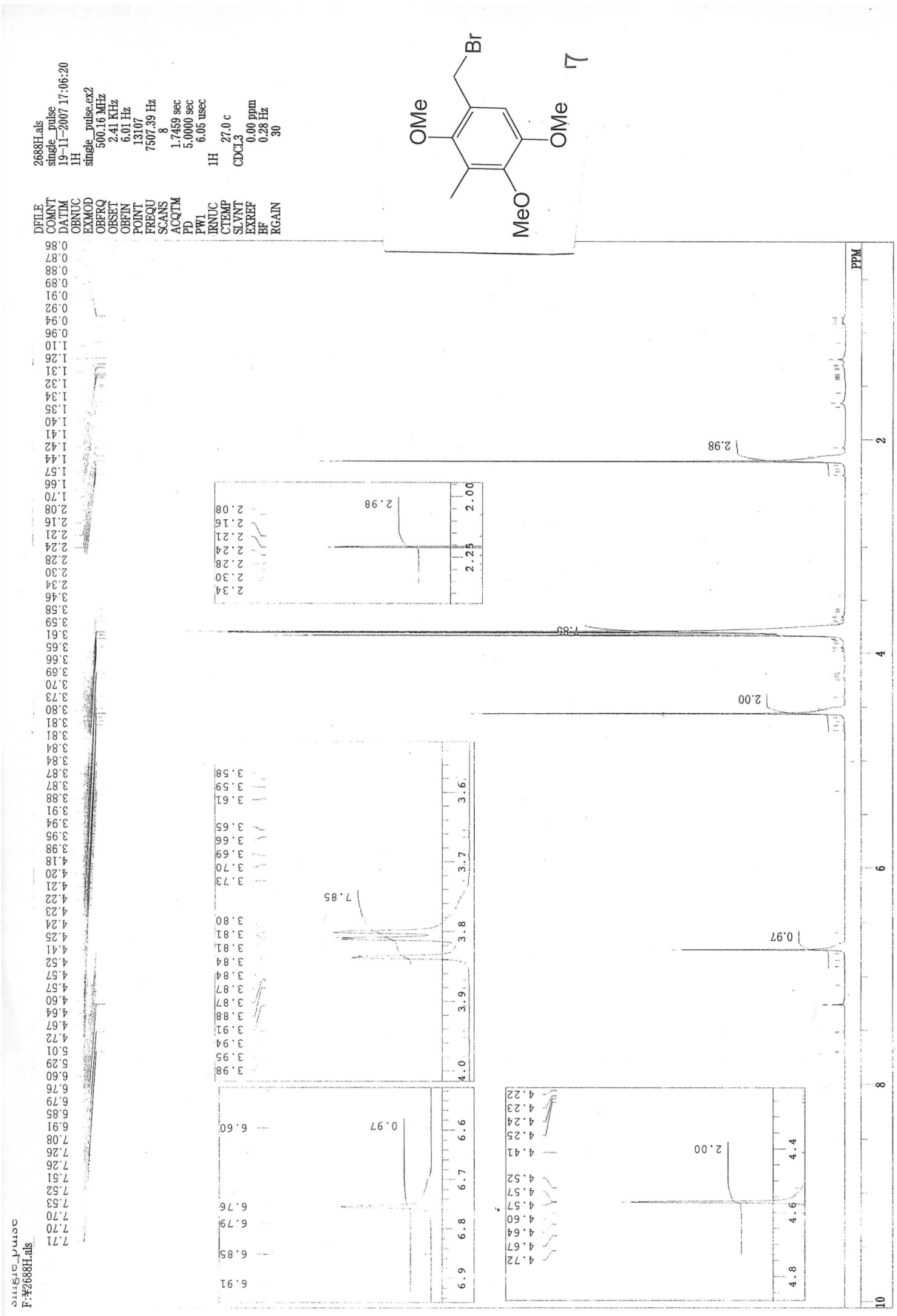


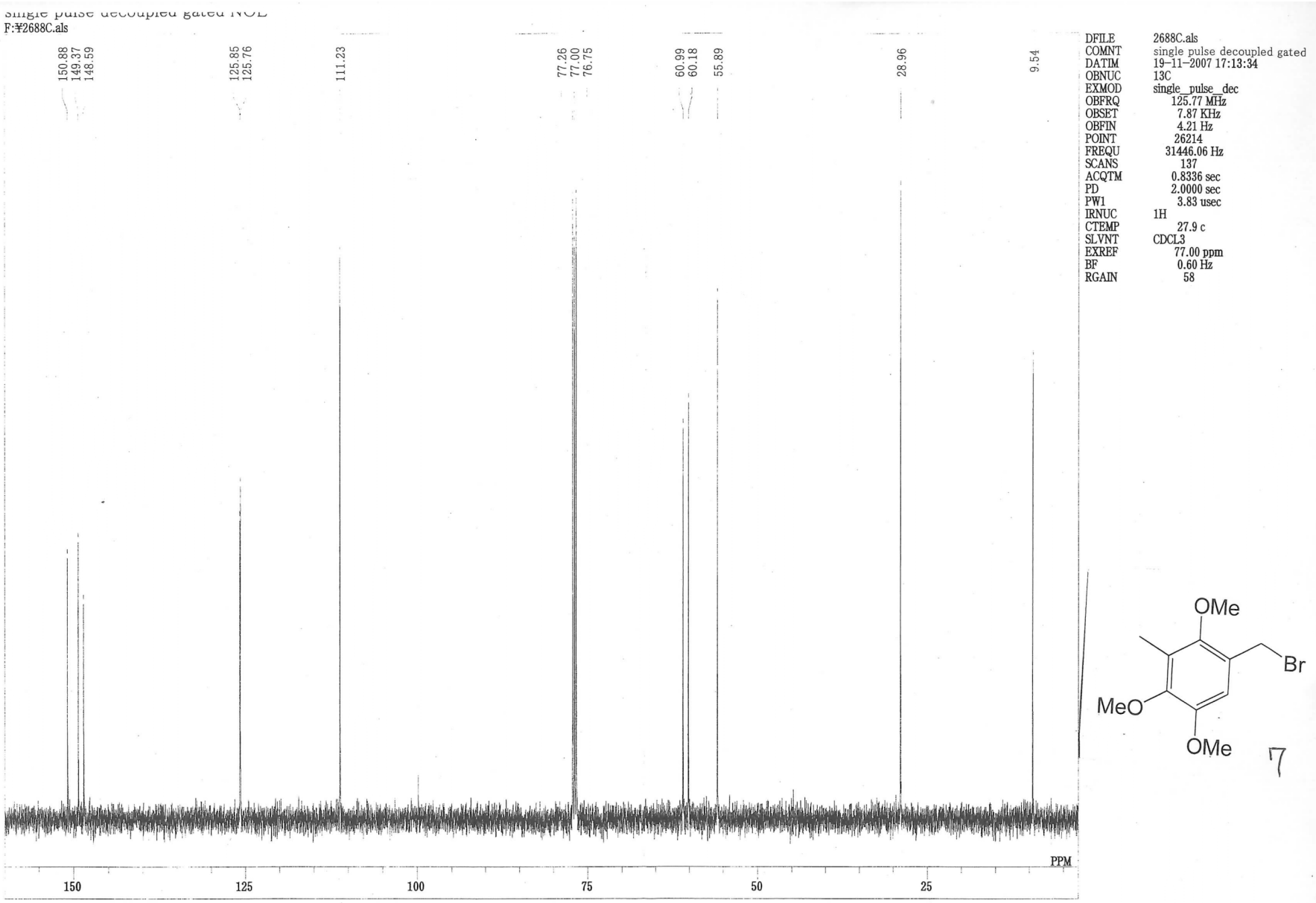
2d

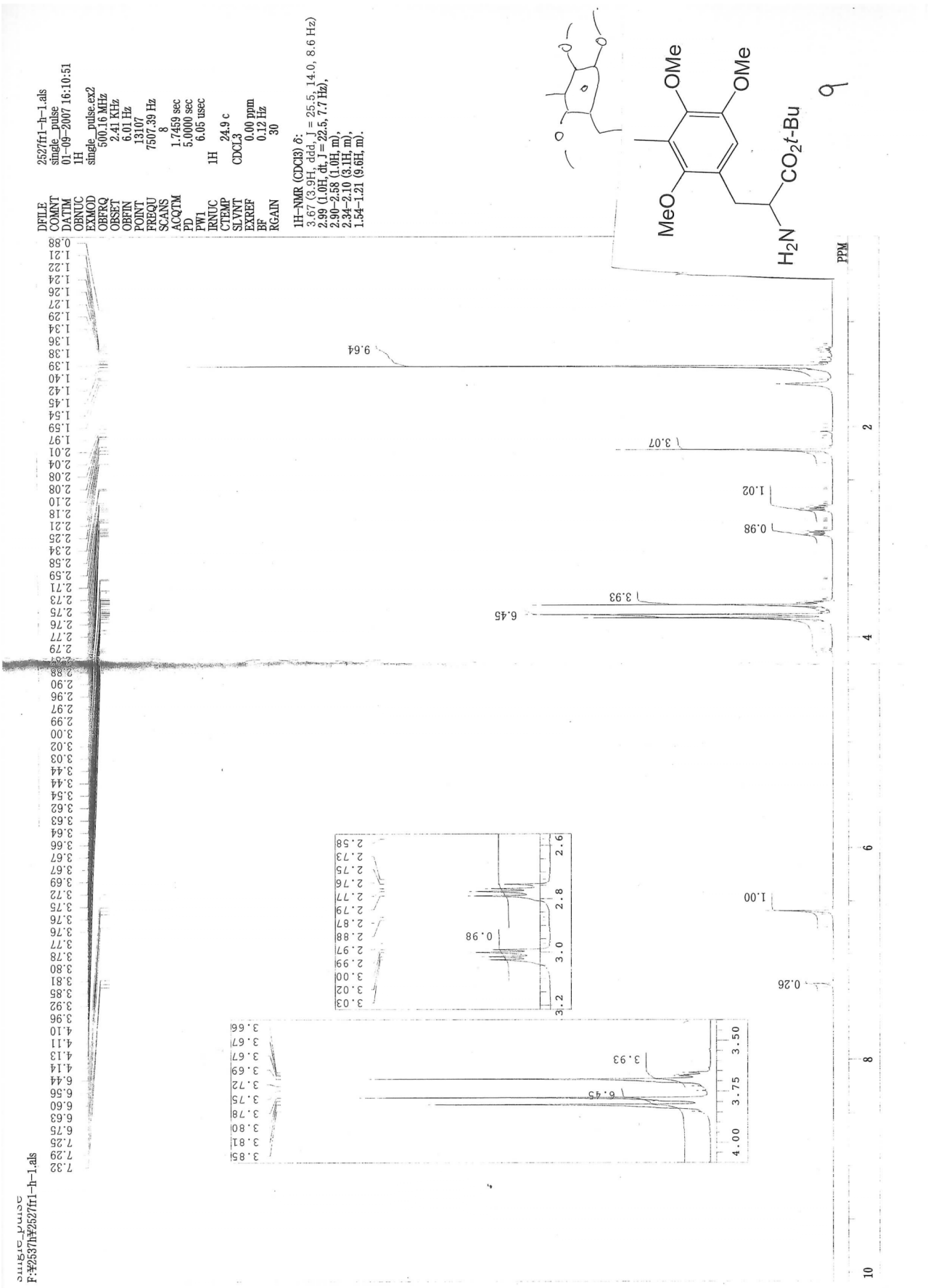

2713c.jdf
single pulse decoupled gated
14-12-2007 13:34:55
13C
single_pulse_dec
125.77 MHz
7.87 KHz
4.21 Hz
32768
39308.18 Hz
1084
0.8336 sec
2.0000 sec
3.83 usec
1H
27.2 c
CDCl3
77.00 ppm
0.60 Hz
60
DFILE
COMNT
DATIM
OBNUC
EXMOD
OBFRQ
OBSET
OBFIN
POINT
FREQU
SCANS
ACQTM
PD
PW1
IRNUC
CTEMP
SLVNT
EXREF
BF
RGAIN
Ph
H
PMB
N
Ns
Ph
O
2d
39.80
48.35
55.16
66.33
76.74
77.00
77.25
113.58
115.17
123.92
127.01
127.32
128.50
128.59
128.91
129.17
130.00
130.17
130.41
131.71
133.70
136.71
146.31
159.12
168.67
PPM

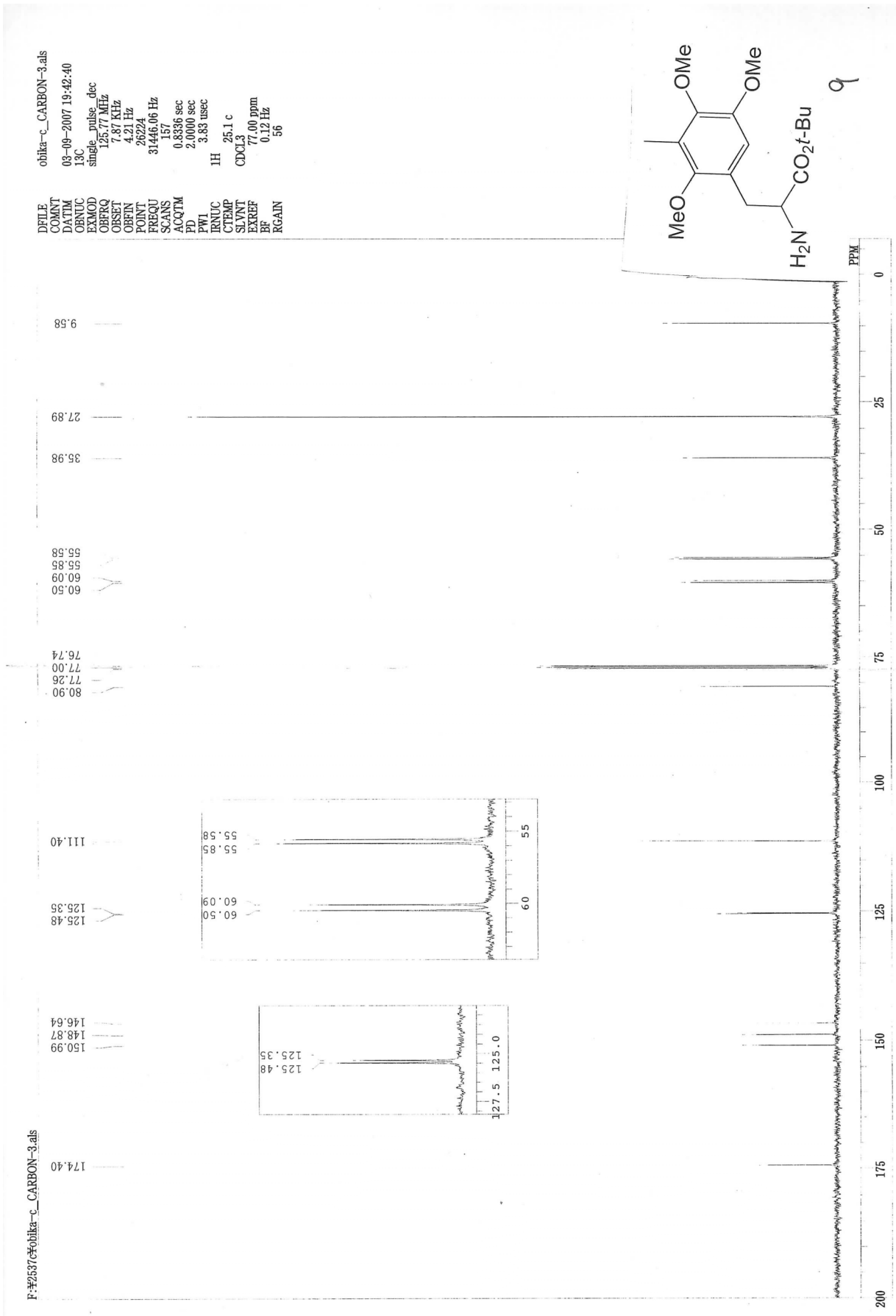

2681H.als
Thu Nov 15 22:03:27 2007
1H
NON
399.65 MHz
124.00 KHz
10500.00 Hz
32768
7992.01 Hz
8
4.1001 sec
2.9000 sec
5.50 usec
1H
21.8 c
CDCl3
0.00 ppm
0.12 Hz
6
DFILE
COMNT
DATIM
OBNUC
EXMOD
OBFRQ
OBSET
OBFIN
POINT
FREQU
SCANS
ACQTM
PD
PW1
IRNUC
CTEMP
SLVNT
EXREF
BF
RGAIN
OMe
OMe
MeO
5
3.52
1.05
1.00
PPM

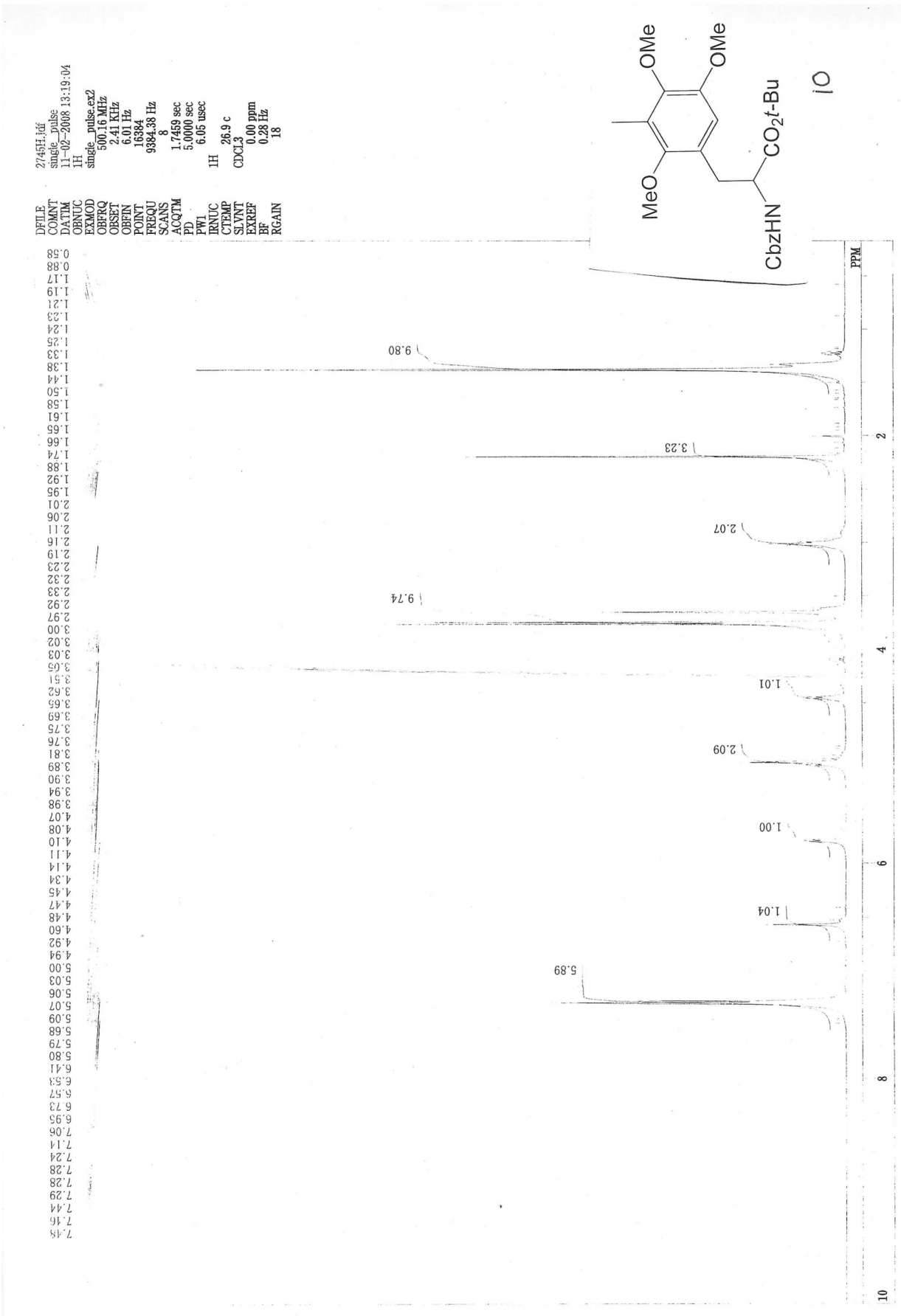

2681C.als
Thu Nov 15 22:06:40 2007
13C
BCM
100.40 MHz
125.00 KHz
10500.00 Hz
32768
27118.64 Hz
44
1.2083 sec
1.7920 sec
5.10 usec
1H
23.0 c
CDCL3
77.00 ppm
0.41 Hz
25
DFILE
COMNT
DATIM
OBNUC
EXMOD
OBFRQ
OBSET
OBFIN
POINT
FREQU
SCANS
ACQTM
PD
PW1
IRNUC
CTEMP
SLVNT
EXREF
BF
RGAIN
OMe
OMe
MeO
5
9.07
55.66
60.07
61.23
76.68
77.00
77.32
91.84
92.54
107.12
114.20
125.96
148.72
151.35
156.39
176.21
PPM
0
25
50
75
100
125
150
175
200

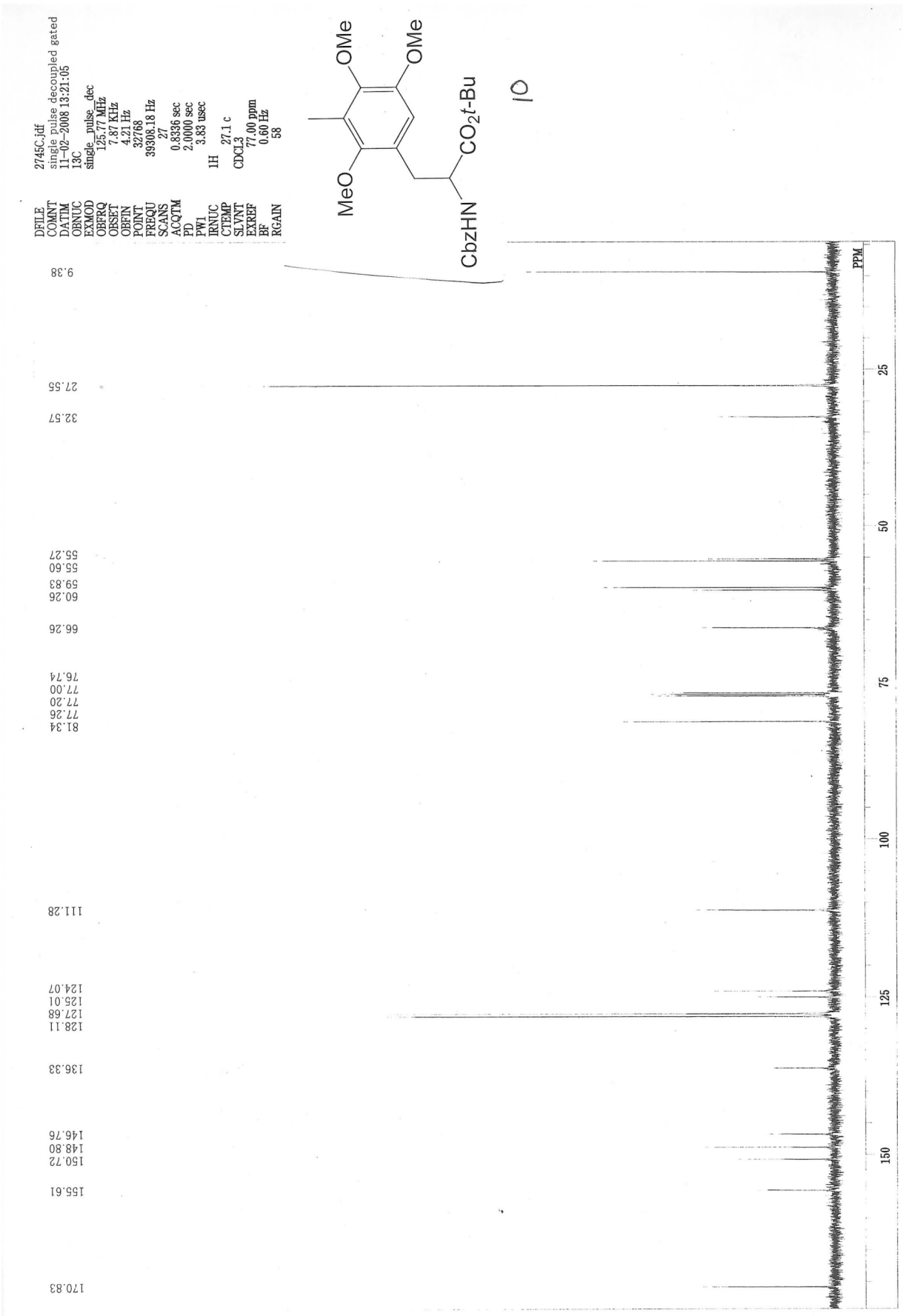

Br
OMe
OMe
MeO
17
2688H.als
single_pulse
19-11-2007 17:06:20
1H
single_pulse.ex2
500.16 MHz
2.41 KHz
6.01 Hz
13107
7507.39 Hz
8
1.7459 sec
5.0000 sec
6.05 usec
1H
27.0 c
CDCL3
0.00 ppm
0.28 Hz
30
DFILE
COMNT
DATIM
OBNUC
EXMOD
OBFRQ
OBSET
OBFIN
POINT
FREQU
SCANS
ACQTM
PD
PW1
IRNUC
CTEMP
SLVNT
EXREF
BF
RGAIN
PPM
2.98
7.85
2.00
0.97


single pulse decoupled gated NOE
F:¥2688C.als
150.88
149.37
148.59
125.85
125.76
111.23
77.26
77.00
76.75
60.99
60.18
55.89
28.96
9.54
DFILE 2688C.als
COMNT single pulse decoupled gated
DATIM 19-11-2007 17:13:34
OBNUC 13C
EXMOD single_pulse_dec
OBFRQ 125.77 MHz
OBSET 7.87 KHz
OBFIN 4.21 Hz
POINT 26214
FREQU 31446.06 Hz
SCANS 137
ACQTM 0.8336 sec
PD 2.0000 sec
PW1 3.83 usec
IRNUC 1H
CTEMP 27.9 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.60 Hz
RGAIN 58
OMe
Br
MeO
OMe
7
PPM
150
125
100
75
50
25


OMe
OMe
MeO
H_2N
CO_2t-Bu
9
PPM
9.64
3.07
1.02
0.98
3.93
6.45
1.00
0.26
0.98
3.93
6.45

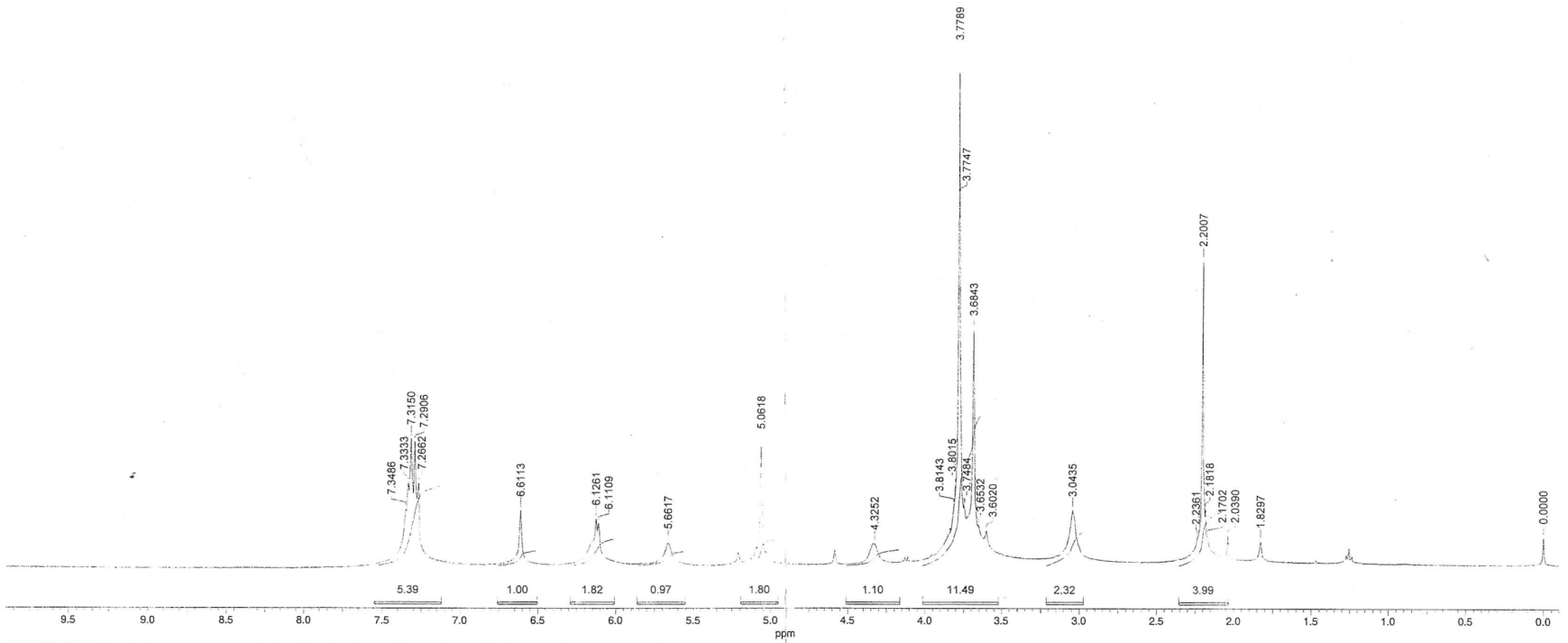

F:¥2537¥obika-c_CARBON-3.als
DFILE obika-c_CARBON-3.als
COMNT
DATIM 03-09-2007 19:42:40
OBNUC 13C
EXMOD single_pulse_dec
OBFRQ 125.77 MHz
OBSET 7.87 KHz
OBFIN 4.21 Hz
POINT 26224
FREQU 31446.06 Hz
SCANS 157
ACQTM 0.8336 sec
PD 2.0000 sec
PW1 3.83 usec
IRNUC 1H
CTEMP 25.1 c
SLVNT CDCl3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 56
OMe
OMe
MeO
CO2t-Bu
H2N
9
PPM
9.58
27.89
35.98
55.58
55.85
60.09
60.50
76.74
77.00
77.26
80.90
111.40
125.35
125.48
146.64
148.87
150.99
174.40


2745H.fid
single_pulse
11-02-2008 13:19:04
1H
single_pulse.ex2
500.16 MHz
2.41 KHz
6.01 Hz
16384
9384.38 Hz
8
1.7459 sec
5.0000 sec
6.05 usec
1H
26.9 c
CDCl3
0.00 ppm
0.28 Hz
18
DFILE
COMNT
DATIM
OBNUC
EXMOD
OBFRQ
OBSET
OBFIN
POINT
FREQU
SCANS
ACQTM
PD
PW1
IRNUC
CTEMP
SLVNT
EXREF
BF
RGAIN
OMe
OMe
MeO
CO2t-Bu
CbzHN
iO
9.80
3.23
2.07
9.74
1.01
2.09
1.00
1.04
5.89
PPM
2
4
6
8
10


DFILE 2745C.jdf
COMNT single pulse decoupled gated
DATIM 11-02-2008 13:21:05
OBNUC 13C
EXMOD single_pulse_dec
OBFRQ 125.77 MHz
OBSET 7.87 KHz
OBFIN 4.21 Hz
POINT 32768
FREQU 39308.18 Hz
SCANS 27
ACQTM 0.8336 sec
PD 2.0000 sec
PW1 3.83 usec
IRNUC 1H
CTEMP 27.1 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.60 Hz
RGAIN 58
OMe
OMe
MeO
CO2t-Bu
CbzHN
10
9.38
27.55
32.57
55.27
55.60
59.83
60.26
66.26
76.74
77.00
77.20
77.26
81.34
111.28
124.07
125.01
127.68
128.11
136.33
146.76
148.80
150.72
155.61
170.83
PPM
25
50
75
100
125
150

23 Jan 2008

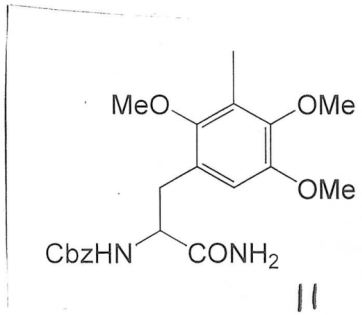
Acquisition Time (sec)	4.1001	*Date*	23 Jan 2008 11:05:00			*File Name*	C:\Users\Sw ¶\Desktop\NMR\NMR DATA\obika\2700-\2730H.als	*Frequency (MHz)*	399.65
Nucleus	1H	*Number of Transients*	8	*Original Points Count*	32768	*Points Count*	32768	*Pulse Sequence*	NON	*Solvent*	CHLOROFORM-D
Sweep Width (Hz)	7992.01	*Temperature (degree C)*	24.900						



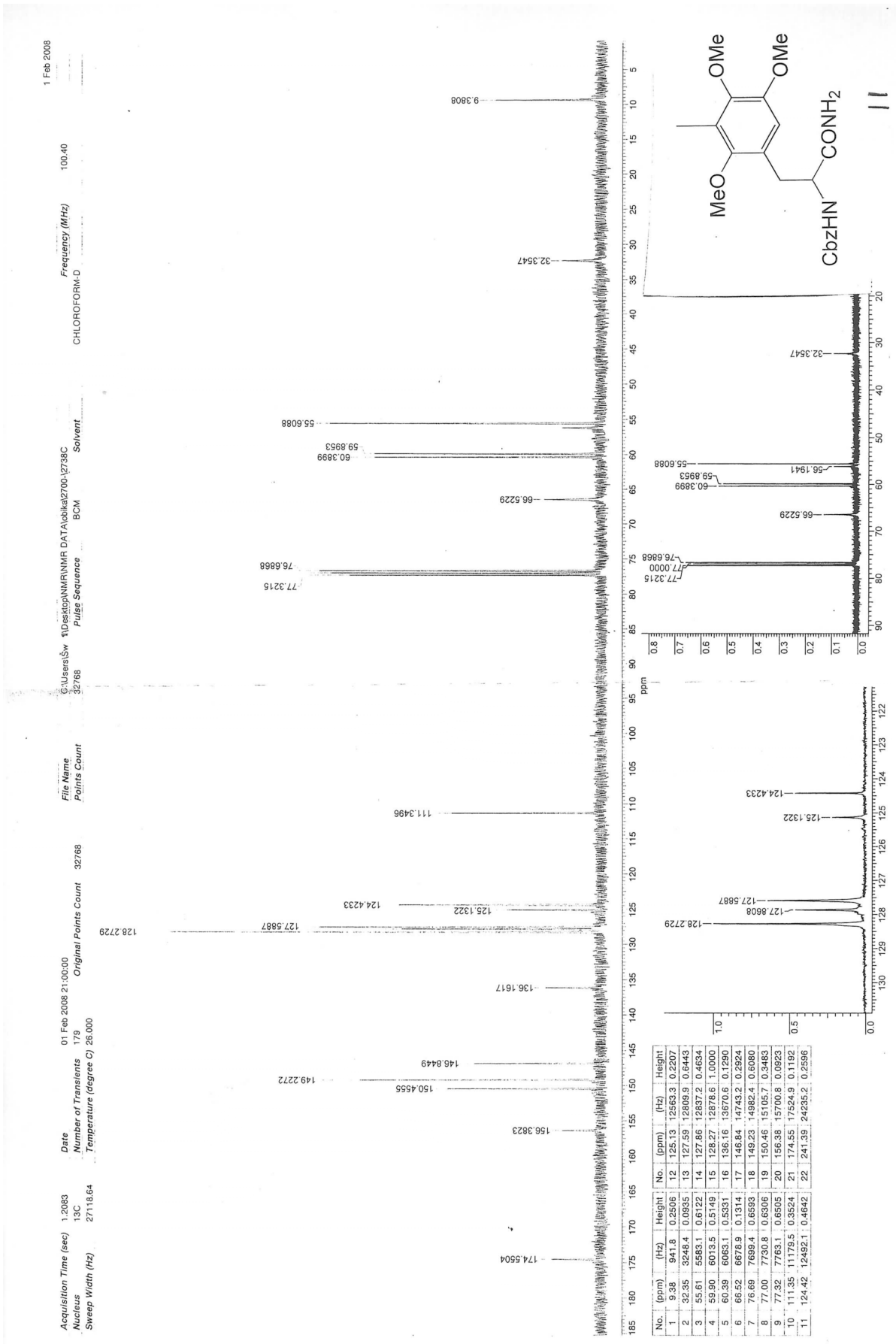
No.	(ppm)	(Hz)	Height
1	-0.00	-0.0	0.0525
2	1.83	731.2	0.0432
3	2.04	814.9	0.0531
4	2.17	867.3	0.0530
5	2.18	872.0	0.0994
6	2.20	879.5	0.5383
7	2.24	893.7	0.0502
8	2.24	895.4	0.0457
9	3.04	1216.4	0.0982
10	3.60	1439.5	0.0621
11	3.65	1460.0	0.0712
12	3.68	1472.4	0.4145
13	3.74	1496.1	0.1039
14	3.75	1498.1	0.1077
15	3.77	1508.5	0.6518
16	3.78	1510.3	0.8976

No.	(ppm)	(Hz)	Height
17	3.80	1519.3	0.1414
18	3.81	1524.4	0.1067
19	4.33	1728.5	0.0390
20	5.06	2022.9	0.2159
21	5.66	2262.7	0.0408
22	6.11	2442.2	0.0755
23	6.13	2448.3	0.0824
24	6.61	2642.2	0.0971
25	7.27	2903.9	0.1449
26	7.27	2905.6	0.1354
27	7.29	2913.7	0.2174
28	7.31	2923.4	0.2228
29	7.33	2930.8	0.1439
30	7.35	2936.8	0.1000
31	15.43	6167.8	1.0000

No.	(ppm)	Value	Absolute Value
1	[2.04 .. 2.36]	3.991	1.15038e+3
2	[2.97 .. 3.21]	2.325	6.70090e+2
3	[3.52 .. 4.02]	11.494	3.31302e+3
4	[4.16 .. 4.51]	1.099	3.16738e+2
5	[4.95 .. 5.19]	1.798	5.18273e+2
6	[5.55 .. 5.86]	0.967	2.78793e+2
7	[6.01 .. 6.29]	1.818	5.23915e+2
8	[6.50 .. 6.76]	1.000	2.88247e+2
9	[7.12 .. 7.55]	5.389	1.55322e+3



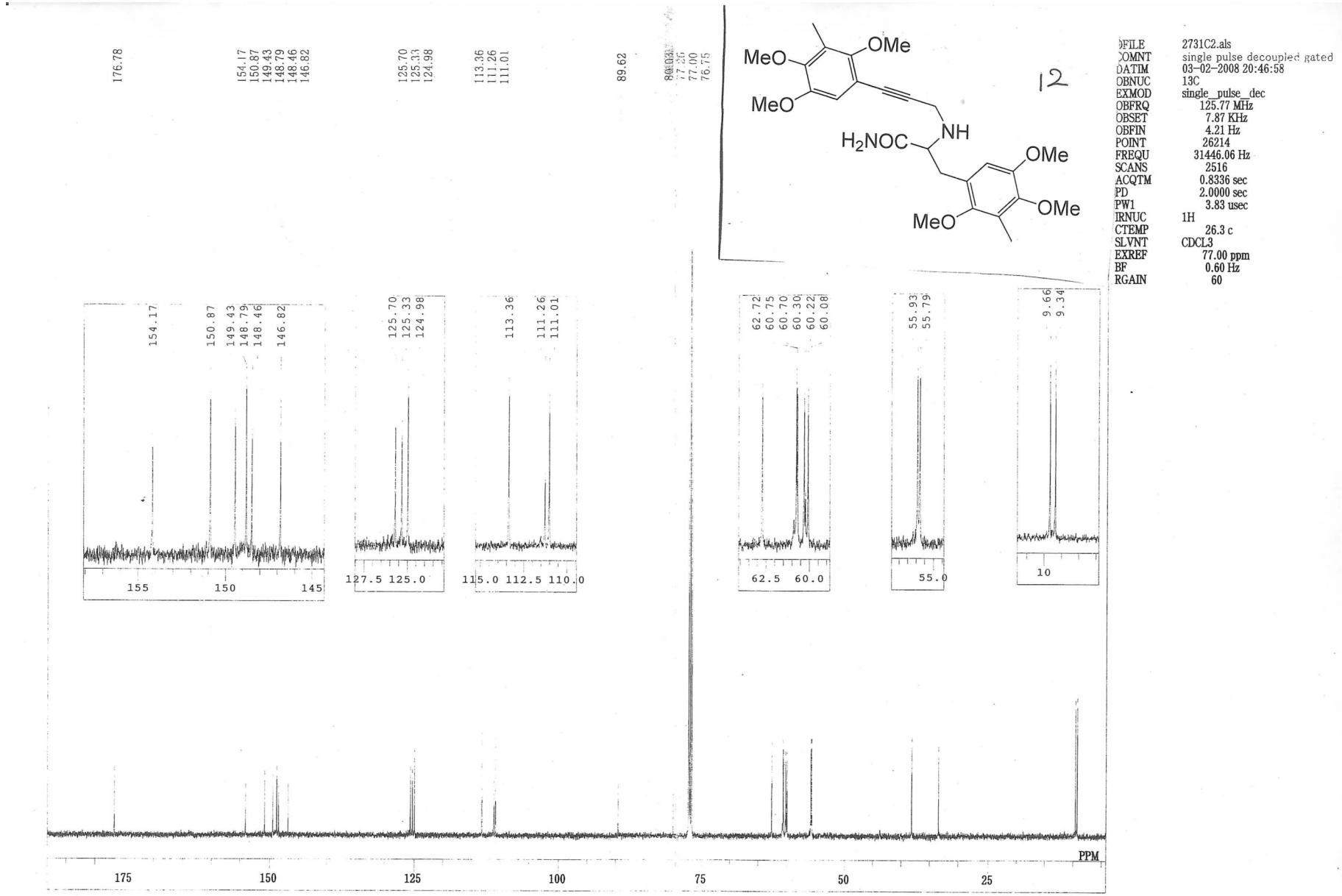
11



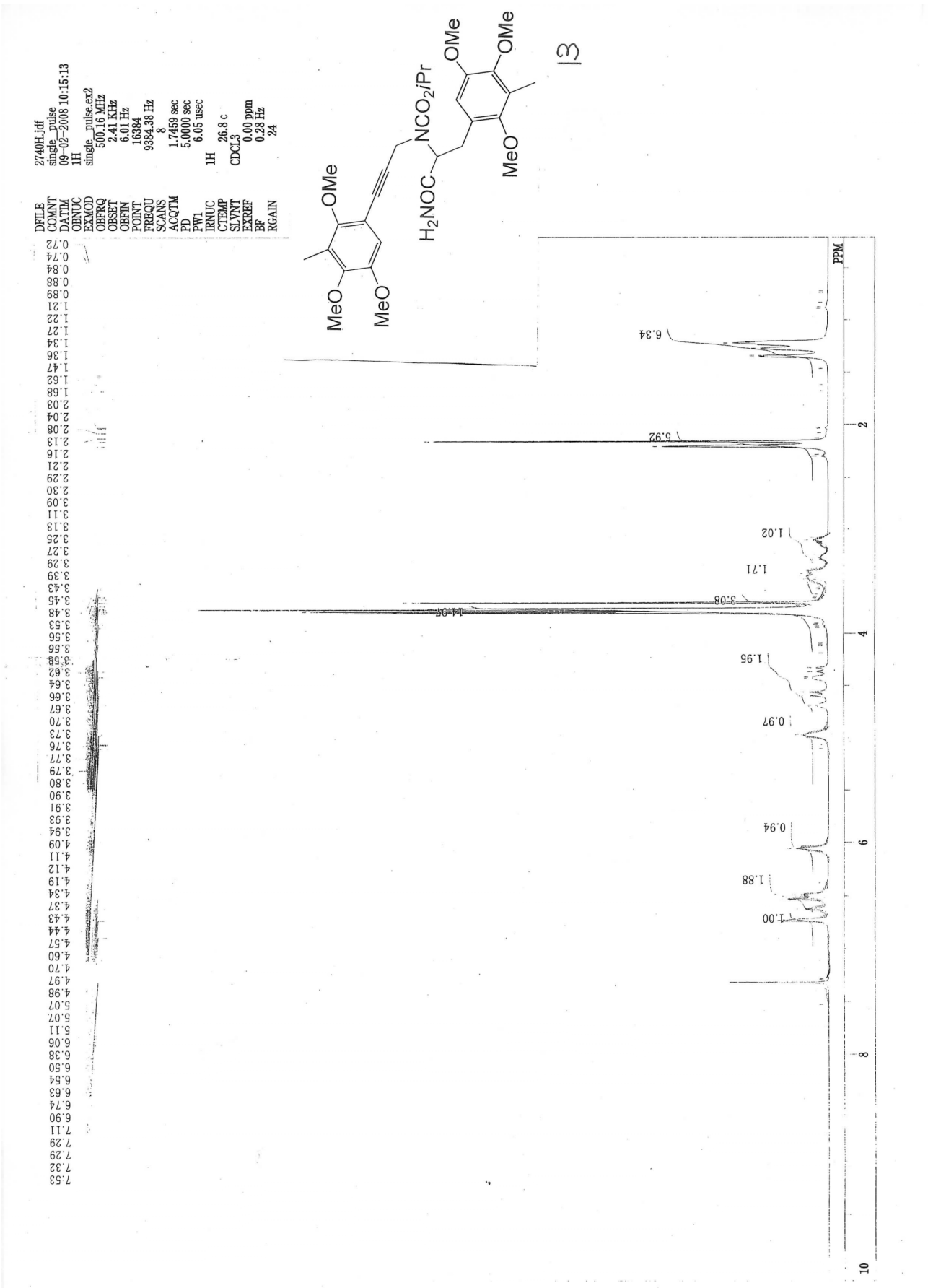
No.	(ppm)	(Hz)	Height
1	9.38	941.8	0.2506
2	32.35	3248.4	0.0935
3	55.61	5583.1	0.6122
4	59.90	6013.5	0.5149
5	60.39	6063.1	0.5331
6	66.52	6678.9	0.1314
7	76.69	7699.4	0.6593
8	77.00	7730.8	0.6306
9	77.32	7763.1	0.6505
10	111.35	11179.5	0.3524
11	124.42	12492.1	0.4642

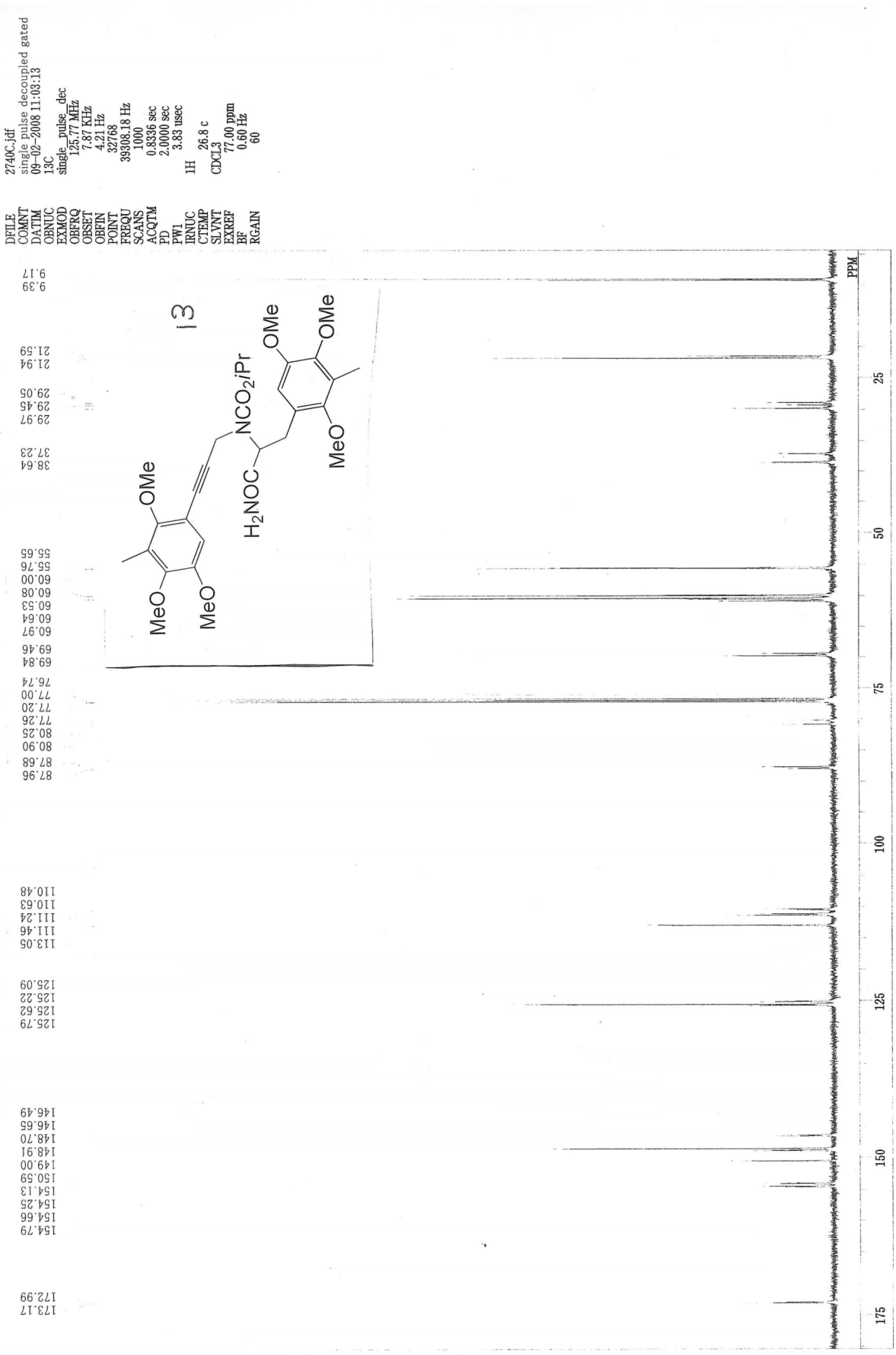
No.	(ppm)	(Hz)	Height
12	125.13	12563.3	0.2207
13	127.59	12809.9	0.6443
14	127.86	12837.2	0.4634
15	128.27	12878.6	1.0000
16	136.16	13670.6	0.1290
17	146.84	14743.2	0.2924
18	149.23	14982.4	0.6080
19	150.46	15105.7	0.3483
20	156.38	15700.8	0.0923
21	174.55	17524.9	0.1192
22	241.39	24235.2	0.2596

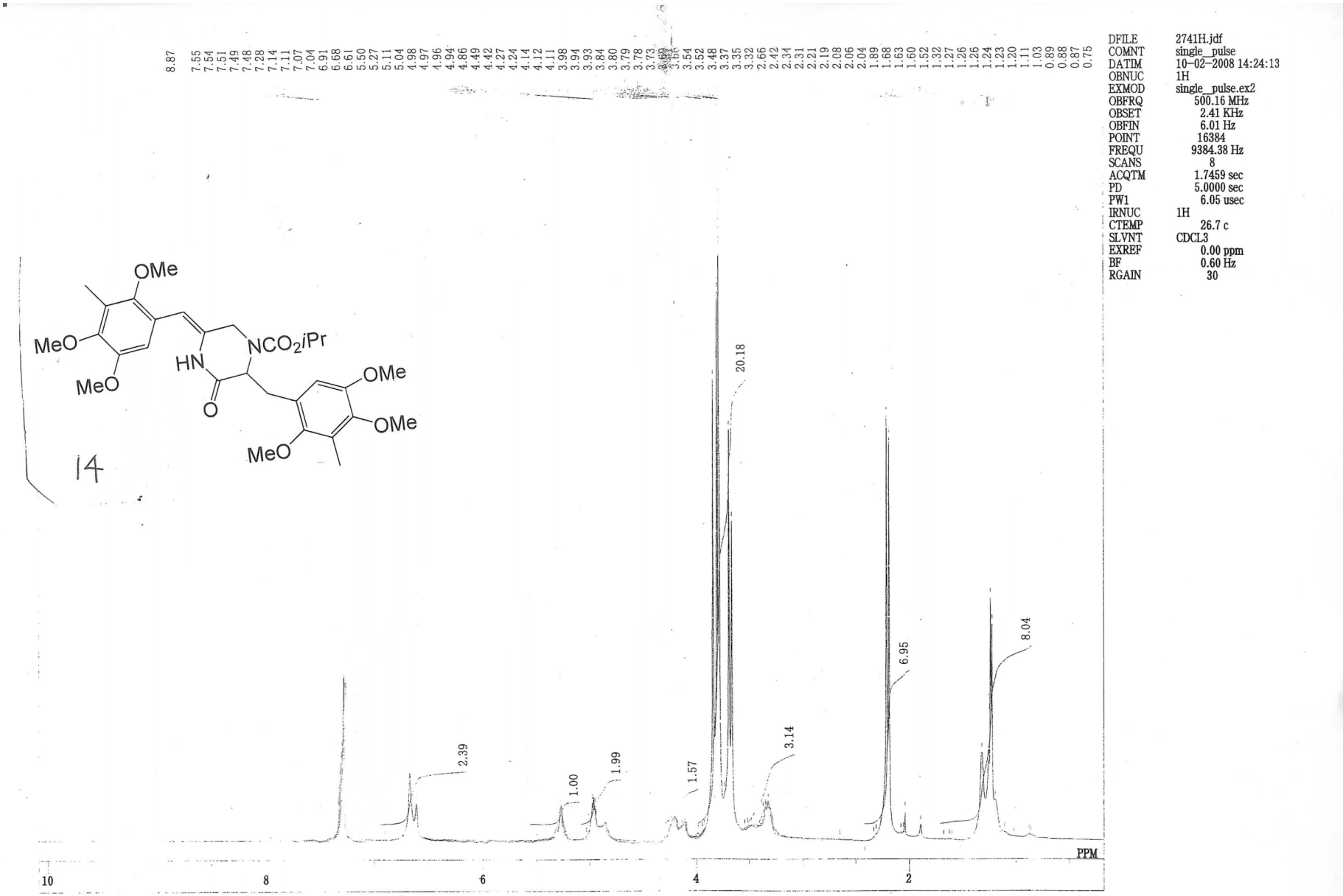

12
OMe
OMe
MeO
MeO
NH
H_2NOC
MeO
2731H.jdf
single_pulse
27-01-2008 14:50:33
1H
single_pulse.ex2
500.16 MHz
2.41 KHz
6.01 Hz
16384
9384.38 Hz
8
1.7459 sec
5.0000 sec
6.05 usec
1H
26.4 c
CDCL3
0.00 ppm
0.28 Hz
36
PPM

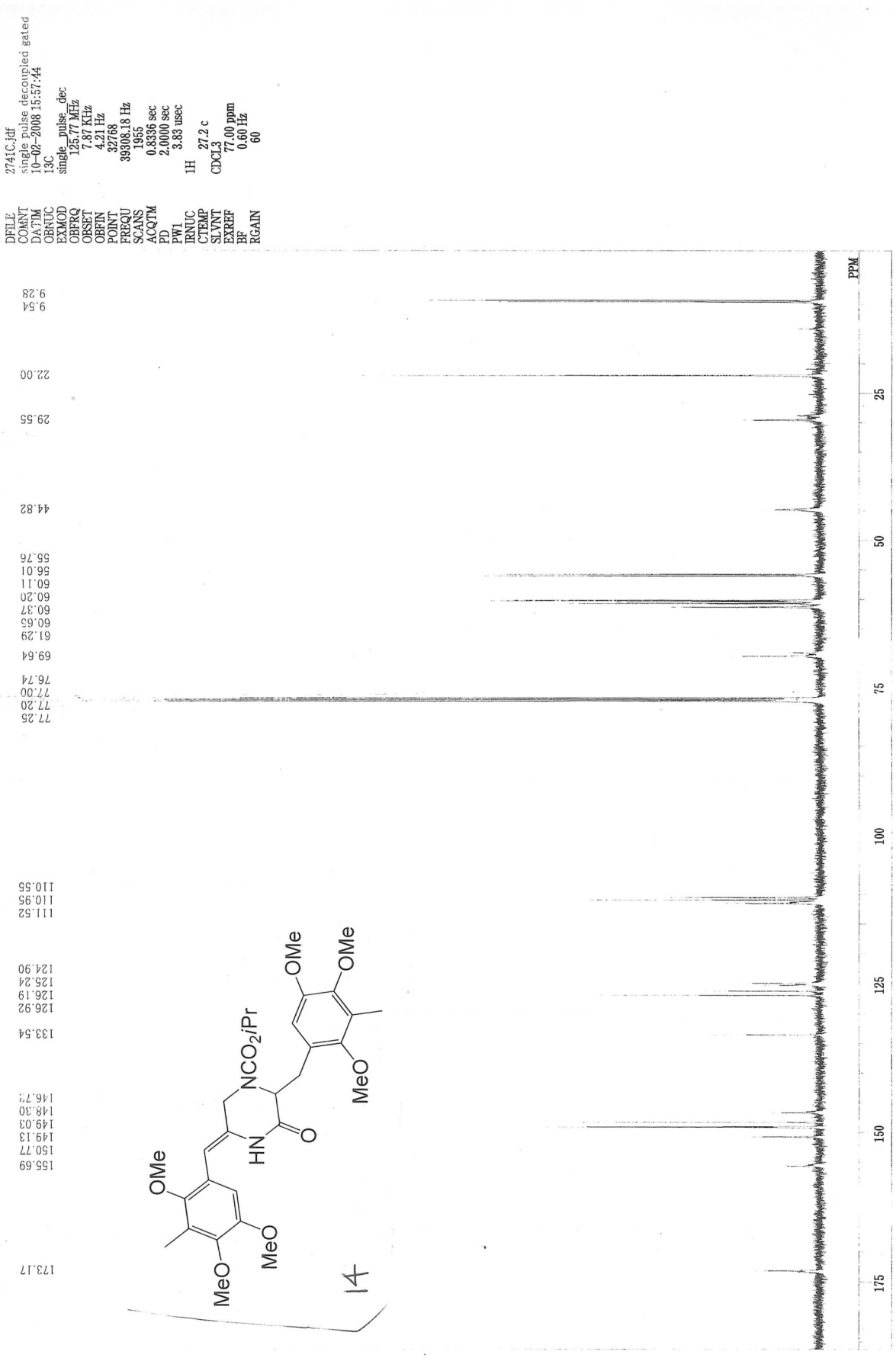


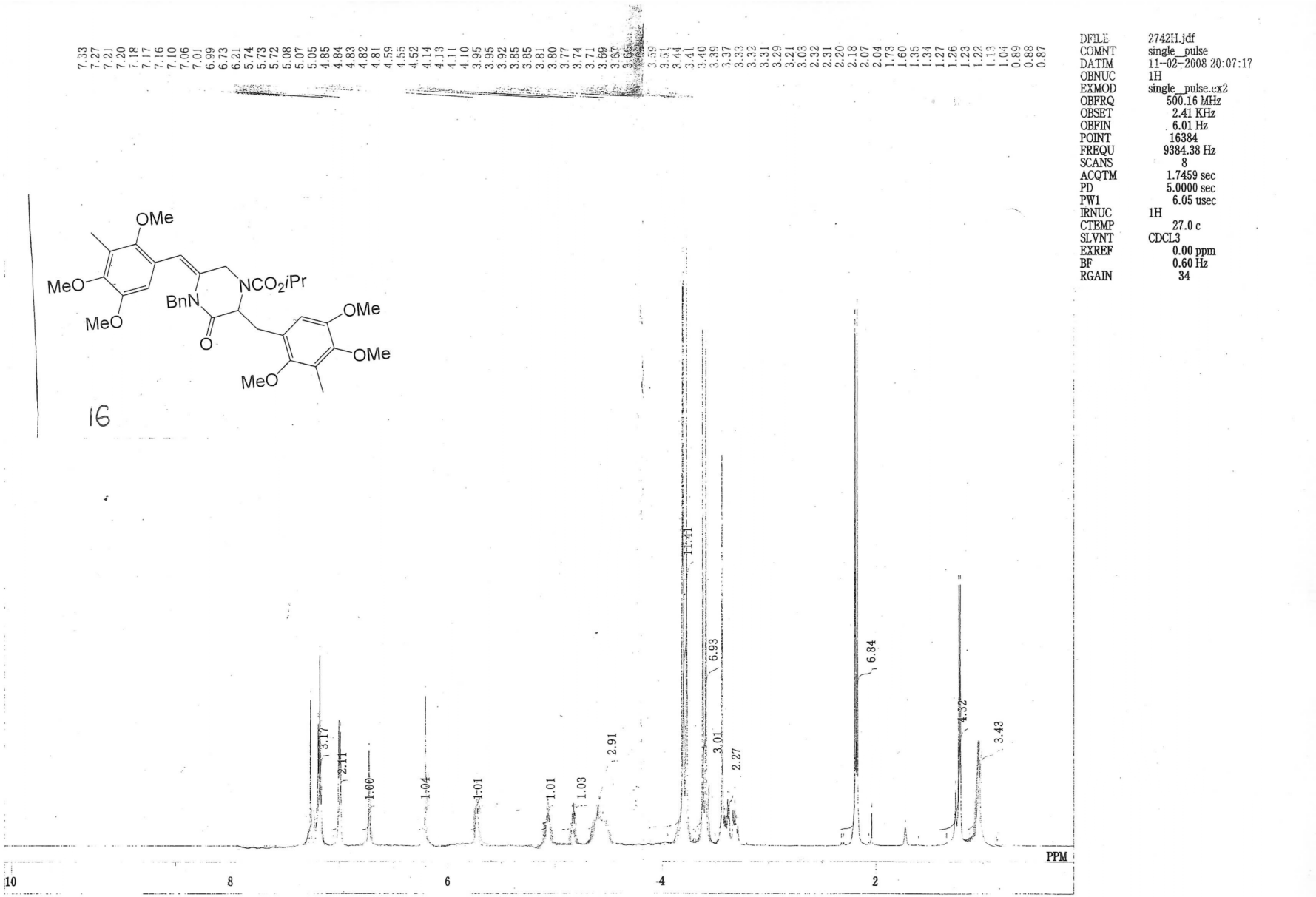
DFILE	2731C2.als
COMNT	single pulse decoupled gated
DATIM	03-02-2008 20:46:58
OBNUC	13C
EXMOD	single_pulse_dec
OBFRQ	125.77 MHz
OBSET	7.87 KHz
OBFIN	4.21 Hz
POINT	26214
FREQU	31446.06 Hz
SCANS	2516
ACQTM	0.8336 sec
PD	2.0000 sec
PW1	3.83 usec
IRNUC	1H
CTEMP	26.3 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.60 Hz
RGAIN	60

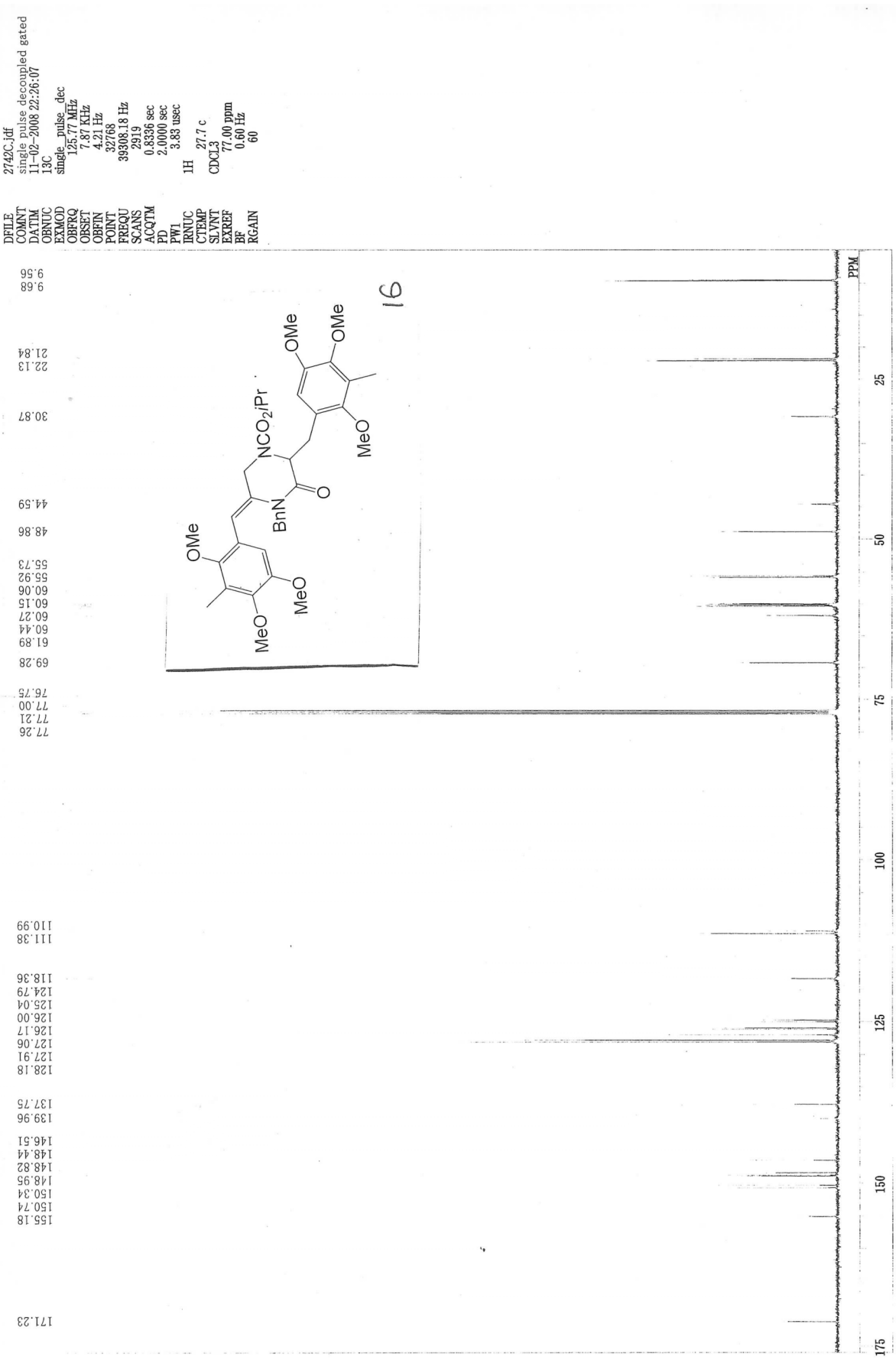

2740H.idf
single_pulse
09-02-2008 10:15:13
1H
single_pulse.ex2
500.16 MHz
2.41 KHz
6.01 Hz
16384
9384.38 Hz
8
1.7459 sec
5.0000 sec
6.05 usec
1H
26.8 c
CDCl3
0.00 ppm
0.28 Hz
24
MeO
MeO
OMe
NCO2iPr
H2NOC
OMe
OMe
MeO
3
6.34
5.92
1.02
1.71
3.08
14.97
1.95
0.97
0.94
1.88
1.00
PPM

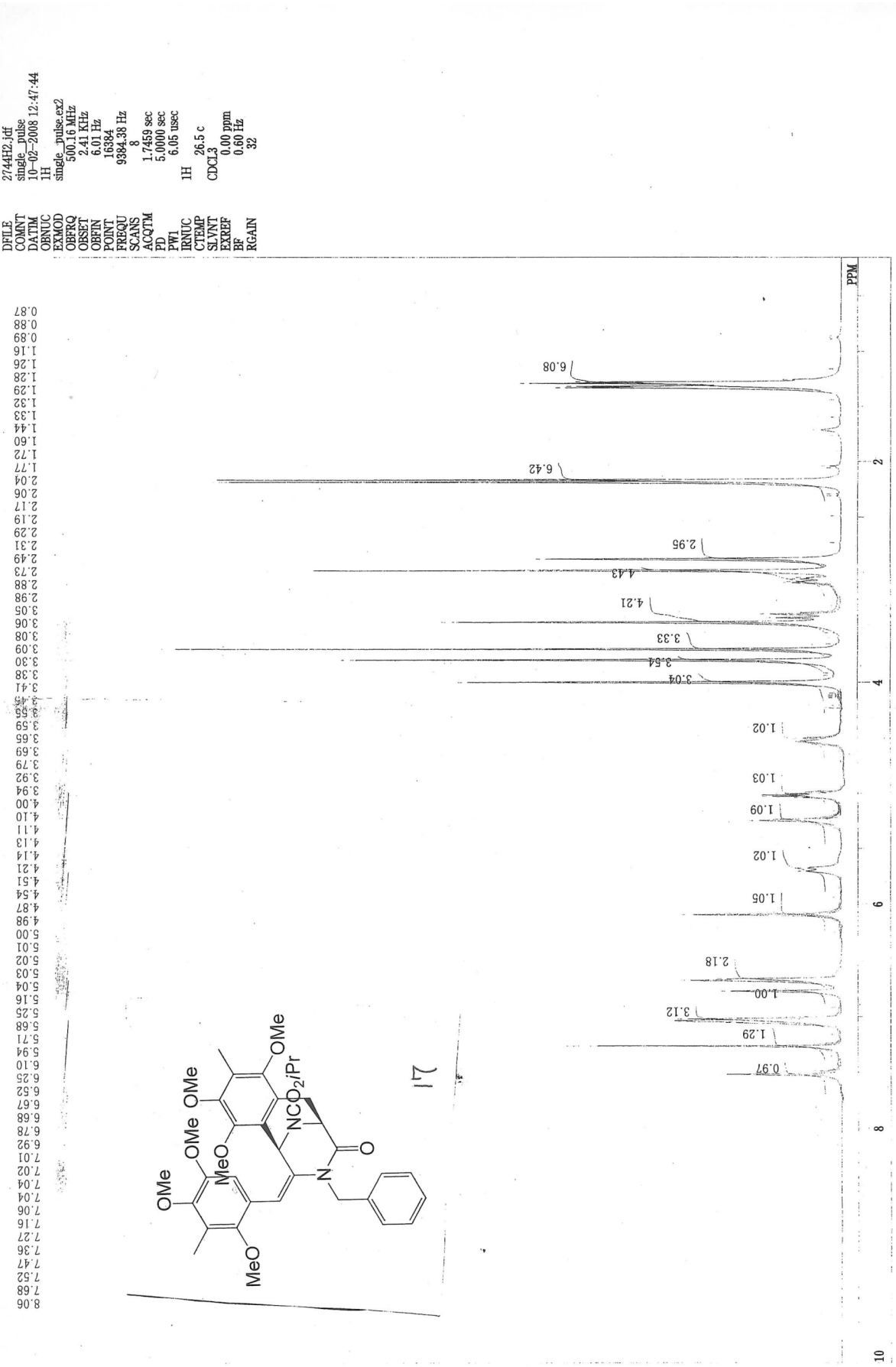

2740c.jdf
single pulse decoupled gated
09-02-2008 11:03:13
13C
single_pulse_dec
125.77 MHz
7.87 KHz
4.21 Hz
32768
39308.18 Hz
1000
0.8336 sec
2.0000 sec
3.83 usec
1H
26.8 c
CDCl3
77.00 ppm
0.60 Hz
60
DFILE
COMNT
DATIM
OBNUC
EXMOD
OBFRQ
OBSET
OBFIN
POINT
FREQU
SCANS
ACQTM
PD
PW1
IRNUC
CTEMP
SLVNT
EXREF
BF
RGAIN
13
OMe
MeO
MeO
H2NOC
NCO2iPr
OMe
OMe
MeO
PPM

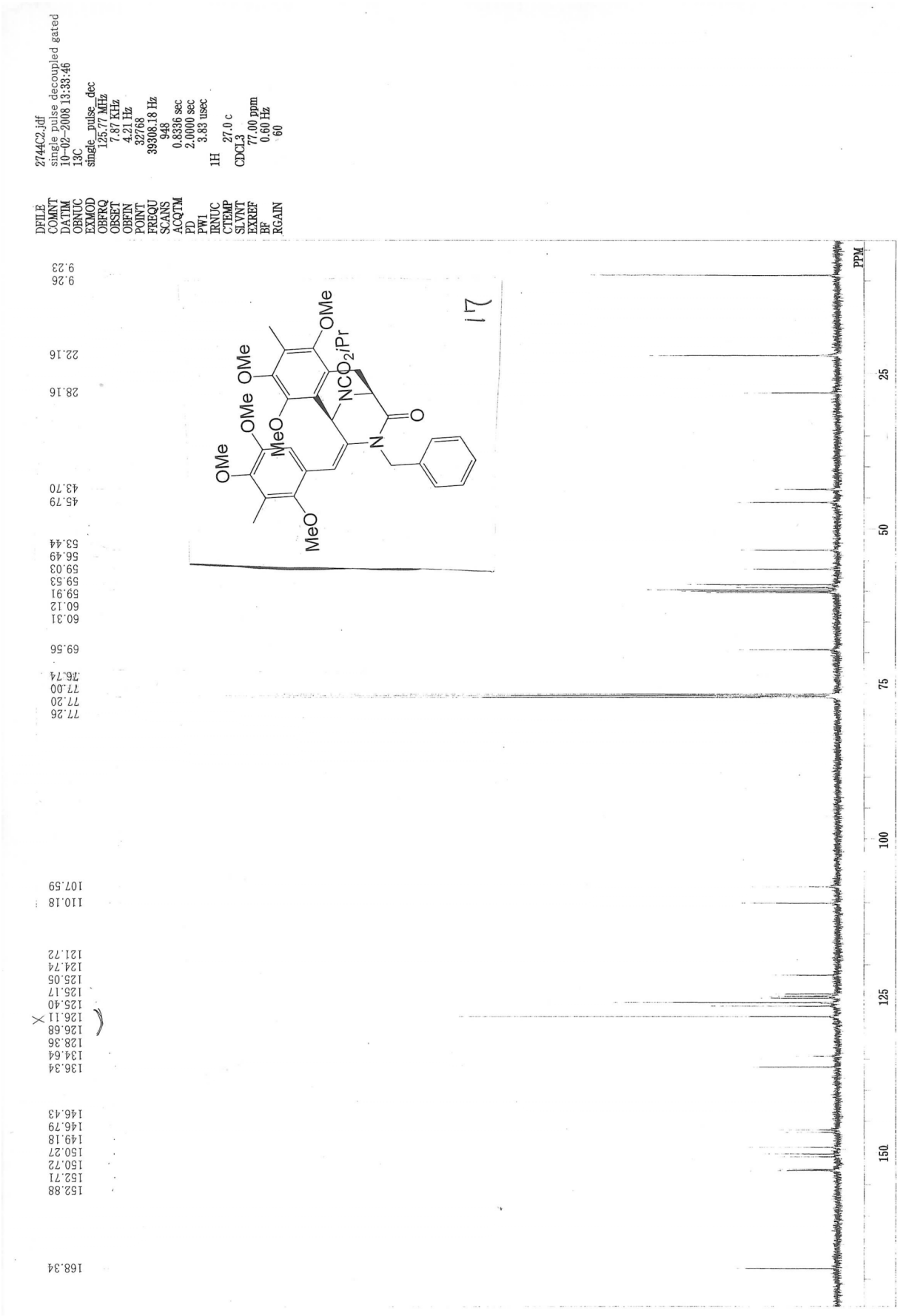

8.87
7.55
7.54
7.51
7.49
7.48
7.28
7.14
7.11
7.07
7.04
6.91
6.68
6.61
5.50
5.27
5.11
5.04
4.98
4.97
4.96
4.94
4.86
4.49
4.42
4.27
4.24
4.14
4.12
4.11
3.98
3.94
3.93
3.84
3.80
3.79
3.78
3.73
3.69
3.66
3.54
3.52
3.48
3.37
3.35
3.32
2.66
2.42
2.34
2.31
2.21
2.19
2.08
2.06
2.04
1.89
1.68
1.63
1.60
1.52
1.32
1.27
1.26
1.26
1.24
1.23
1.20
1.11
1.03
0.89
0.88
0.87
0.75
DFILE 2741H.jdf
COMNT single_pulse
DATIM 10-02-2008 14:24:13
OBNUC 1H
EXMOD single_pulse.ex2
OBFRQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 16384
FREQU 9384.38 Hz
SCANS 8
ACQTM 1.7459 sec
PD 5.0000 sec
PW1 6.05 usec
IRNUC 1H
CTEMP 26.7 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.60 Hz
RGAIN 30
OMe
MeO
MeO
HN
O
NCO2iPr
OMe
OMe
MeO
14
2.39
1.00
1.99
1.57
20.18
3.14
6.95
8.04
PPM
10
8
6
4
2

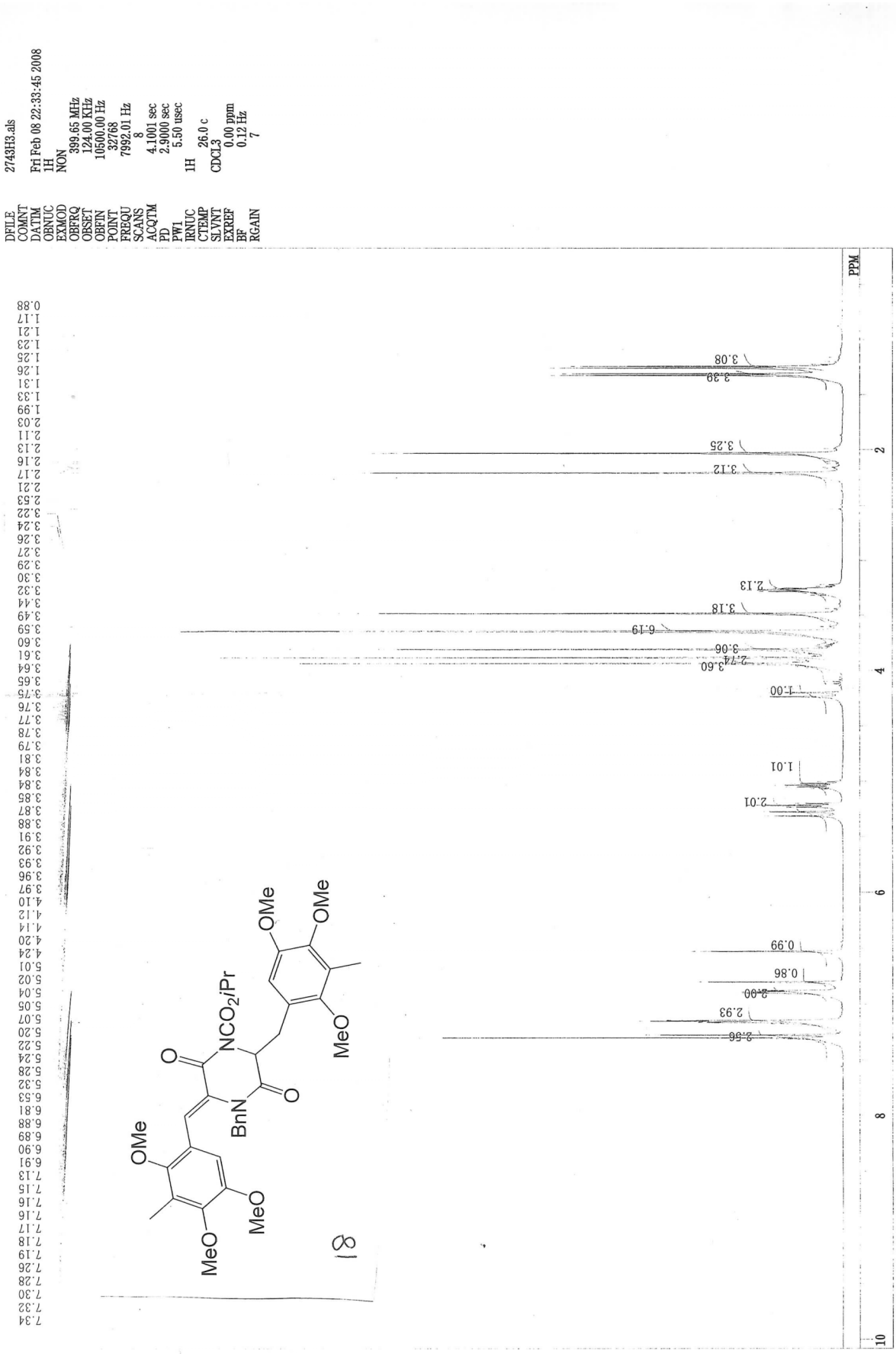

DFILE 2741C.jdf
COMNT single pulse decoupled gated
DATIM 10-02-2008 15:57:44
OBNUC 13C
EXMOD single_pulse_dec
OBFRQ 125.77 MHz
OBSET 7.87 KHz
OBFIN 4.21 Hz
POINT 32768
FREQU 39308.18 Hz
SCANS 1955
ACQTM 0.8336 sec
PD 2.0000 sec
PW1 3.83 usec
IRNUC 1H
CTEMP 27.2 c
SLVNT CDCl3
EXREF 77.00 ppm
BF 0.60 Hz
RGAIN 60
9.28
9.54
22.00
29.55
44.82
55.76
56.01
60.11
60.20
60.37
60.65
61.29
69.64
76.74
77.00
77.20
77.25
110.55
110.95
111.52
124.90
125.24
126.19
126.92
133.54
146.73
148.30
149.03
149.13
150.77
155.69
173.17
PPM
25
50
75
100
125
150
175
OMe
MeO
MeO
HN
O
NCO2iPr
OMe
OMe
MeO
14

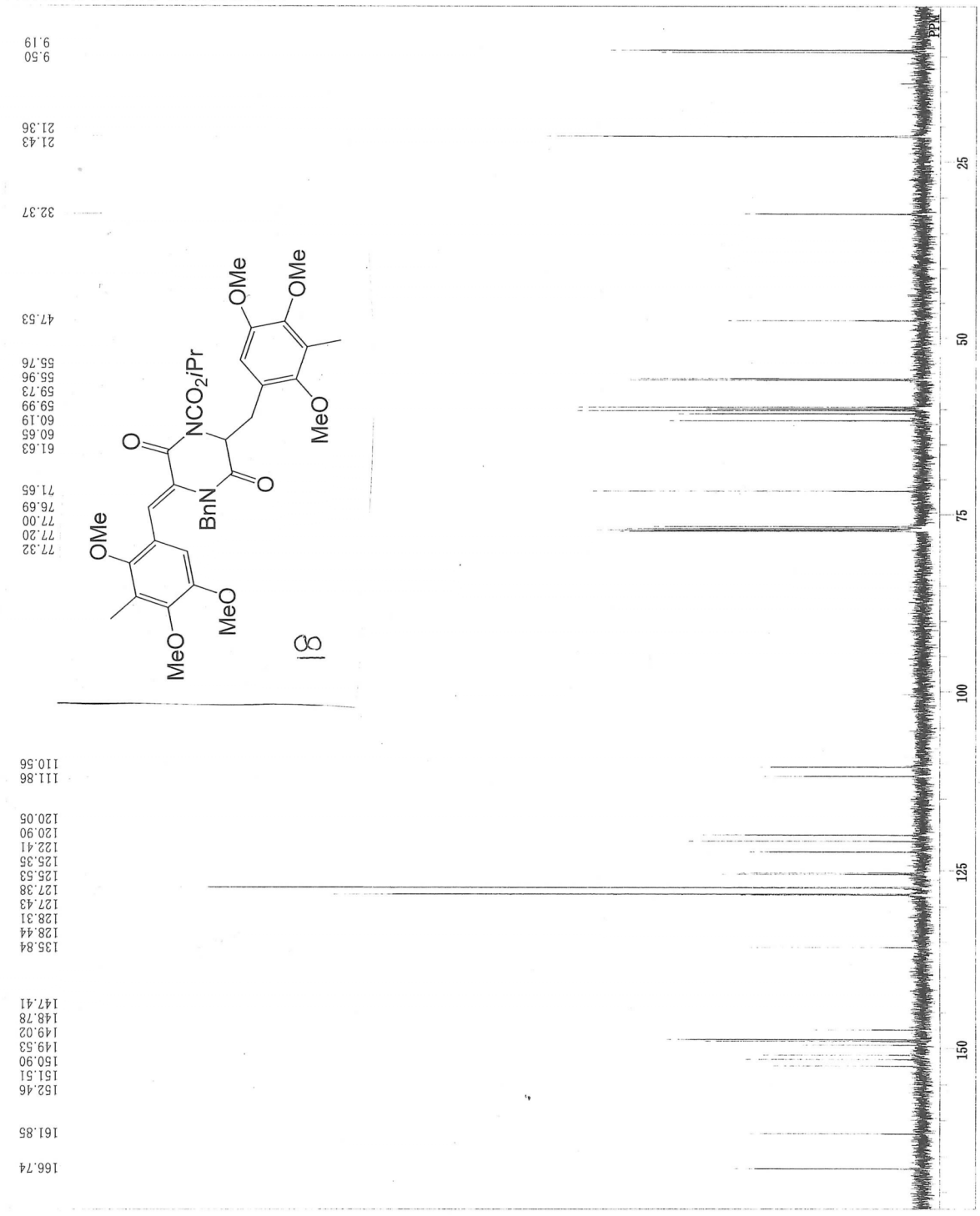

DFILE 2742H.jdf
COMNT single_pulse
DATIM 11-02-2008 20:07:17
OBNUC 1H
EXMOD single_pulse.ex2
OBFRQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 16384
FREQU 9384.38 Hz
SCANS 8
ACQTM 1.7459 sec
PD 5.0000 sec
PW1 6.05 usec
IRNUC 1H
CTEMP 27.0 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.60 Hz
RGAIN 34
OMe
MeO
MeO
BnN
NCO2iPr
O
OMe
OMe
MeO
16
3.17
2.11
1.00
1.04
1.01
1.01
1.03
2.91
11.41
6.93
3.01
2.27
6.84
4.32
3.43
PPM
10
8
6
4
2


2742C.idf
single pulse decoupled gated
11-02-2008 22:26:07
13C
single_pulse_dec
125.77 MHz
7.87 KHz
4.21 Hz
32768
393308.18 Hz
2919
0.8336 sec
2.0000 sec
3.83 usec
1H
27.7 c
CDCL3
77.00 ppm
0.60 Hz
60
DFILE
COMNT
DATIM
OBNUC
EXMOD
OBFRQ
OBSET
OBFIN
POINT
FREQU
SCANS
ACQTM
PD
PW1
IRNUC
CTEMP
SLVNT
EXREF
BF
RGAIN
9.56
9.68
21.84
22.13
30.87
44.59
48.86
55.73
55.92
60.06
60.15
60.27
60.44
61.89
69.28
76.75
77.00
77.21
77.26
110.99
111.38
118.36
124.79
125.04
126.00
126.17
127.06
127.91
128.18
137.75
139.96
146.51
148.44
148.82
148.95
150.34
150.74
155.18
171.23
PPM
25
50
75
100
125
150
175
16
OMe
OMe
MeO
NCO2iPr
BnN
O
OMe
MeO
MeO


2744H2.idf
single_pulse
10-02-2008 12:47:44
1H
single_pulse.ex2
500.16 MHz
2.41 KHz
6.01 Hz
16384
9384.38 Hz
8
1.7459 sec
5.0000 sec
6.05 usec
1H 26.5 c
CDCL3
0.00 ppm
0.60 Hz
32
DFILE
COMNT
DATIM
OBNUC
EXMOD
OBFRQ
OBSET
OBFIN
POINT
FREQU
SCANS
ACQTM
PD
PW1
IRNUC
CTEMP
SLVNT
EXREF
BF
RGAIN
PPM
0.97
1.29
3.12
1.00
2.18
1.05
1.02
1.09
1.03
1.02
3.04
3.54
3.33
4.21
4.43
2.95
6.42
6.08
OMe
OMe
OMe
MeO
MeO
NCO2iPr
O
N
17


2744C2.jdf
single pulse decoupled gated
10-02-2008 13:33:46
13C
single_pulse_dec
125.77 MHz
7.87 KHz
4.21 Hz
32768
393308.18 Hz
948
0.8336 sec
2.0000 sec
3.83 usec
1H
27.0 c
CDCl3
77.00 ppm
0.60 Hz
60
DFILE
COMNT
DATIM
OBNUC
EXMOD
OBFRQ
OBSET
OBFIN
POINT
FREQU
SCANS
ACQTM
PD
PW1
IRNUC
CTEMP
SLVNT
EXREF
BF
RGAIN
OMe
OMe
OMe
MeO
MeO
NCO2iPr
N
O
17
PPM


DFILE 2743H3.als
COMNT
DATIM Fri Feb 08 22:33:45 2008
OBNUC 1H
EXMOD NON
OBFRQ 399.65 MHz
OBSET 124.00 KHz
OBFIN 10500.00 Hz
POINT 32768
FREQU 7992.01 Hz
SCANS 8
ACQTM 4.1001 sec
PD 2.9000 sec
PW1 5.50 usec
IRNUC 1H
CTEMP 26.0 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 7
PPM
18
NCO2iPr
BnN
OMe
MeO


DFILE 2743C2.als
COMNT
DATIM Fri Feb 08 22:37:32 2008
OBNUC 13C
EXMOD BCM
OBFRQ 100.40 MHz
OBSET 125.00 KHz
OBFIN 10500.00 Hz
POINT 32768
FREQU 27118.64 Hz
SCANS 58
ACQTM 1.2083 sec
PD 1.7920 sec
PW1 5.10 usec
IRNUC 1H
CTEMP 27.5 c
SLVNT CDCl3
EXREF 77.00 ppm
BF 0.41 Hz
RGAIN 25
9.19
9.50
21.36
21.43
32.37
47.53
55.76
55.96
59.73
59.99
60.19
60.65
61.63
71.65
76.69
77.00
77.20
77.32
110.56
111.86
120.05
120.90
122.41
125.35
125.53
127.38
127.43
128.31
128.44
135.84
147.41
148.78
149.02
149.53
150.90
151.51
152.46
161.85
166.74
PPM
25
50
75
100
125
150
OMe
OMe
MeO
NCO2iPr
O
O
BnN
OMe
MeO
MeO
18