Structurally Simple Benzylidene-Type Photolabile Diol Protecting Groups

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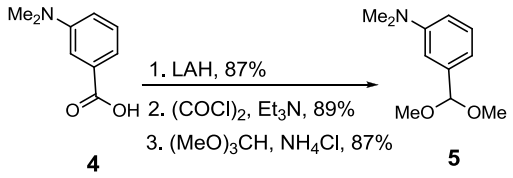
Experimental Section

General. Organic solutions were concentrated by rotary evaporation at ca. 12 Torr. Flash column chromatography was performed employing 230-400 mesh silica gel. Thin-layer chromatography was performed using glass plates pre-coated to a depth of 0.25 mm with 230-400 mesh silica gel impregnated with a fluorescent indicator (254 nm). Infrared (IR) data are presented as frequency of absorption (cm^{-1}). Proton and carbon-13 nuclear magnetic resonance (^{1}H NMR or ^{13}C NMR) spectra were recorded on 300, 400 and 700 MHz NMR spectrometers; Chemical shifts are expressed in parts per million (δ scale) downfield from tetramethylsilane and are referenced to residual protium in the NMR solvent ($CHCl_3$: δ 7.26). Data are presented as follows: chemical shift, multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet and/or multiple resonances), coupling constant in Hertz (Hz), integration.

Materials. Anhydrous solvents Tetrahydrofuran (THF), dimethylformamide (DMF), and dichloromethane (DCM) were used without distillation. Solvents for workup and column chromatography, such as petroleum ether (PE), hexanes (Hex), ethyl acetate (EA), methanol (MeOH), benzene, toluene (Tol) and triethylamine (TEA), camphor sulfonic acid (CAS), and other chemicals were obtained from commercial vendors and used without further purification.

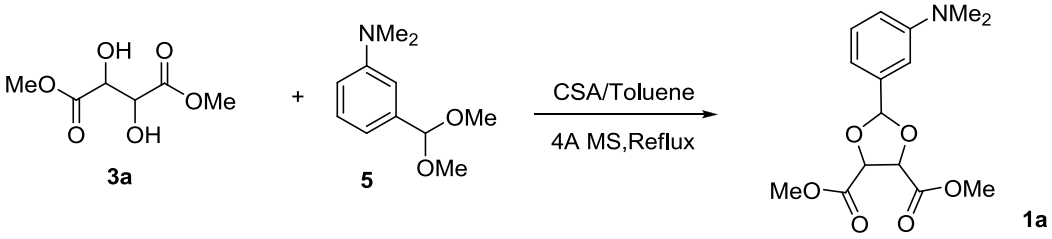


To a solution of 3-dimethylaminobenzoic acid **4** (5 g, 30.3 mmol) in THF (100.0 mL), $LiAlH_4$ (1.7 g, 45.5 mmol) was added at room temperature under nitrogen atmosphere. The reaction mixture was stirred for 19 h, quenched with statured solution of NH_4Cl, and filtered through sintered crucible. The filtrate was concentrated to provide the corresponding 3-dimethylaminobenzyl alcohol (3.9 g, 87%) as a brown liquid which was used for next step without further purification.

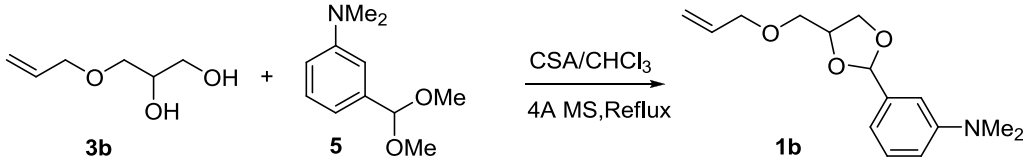
To a stirred solution of oxalyl chloride (4.3 mL, 51.7 mmol) in dry DCM (150.0 mL), DMSO (5.5 mL, 77.5 mmol) was added at -78 ^{o}C under nitrogen atmosphere. The reaction mixture was stirred for 15 min. followed by the addition of dimethylaminobenzyl alcohol (3.9 g, 25.8 mmol). After stirring for 1 h at -78 ^{o}C, triethyl amine (14.4 mL, 103.3 mmol) was added and reaction mixture was stirred at room temperature for additional 15 min, after which it was quenched with H_2O (100 mL). The organic phase was separated and the aqueous phase was extracted with DCM (2 x 50 mL), the combined organic phase was dried over anhydrous Na_2SO_4 and concentrated under reduced pressure to give the corresponding crude benzaldehyde. The crude product was purified by column chromatography (PE/EA 19:1) to afford the product as a yellow liquid (3.4 g, 89%): Rf = 0.5 (PE/EA 19:1); ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.96 (s, 1H), 7.39 (t, *J* = 7.5 Hz, 1H), 7.19 (m, 2H), 6.69-6.99 (m, 2H), 3.02 (s, 6H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 193.2, 150.7,

137.2, 129.6, 118.9, 118.3, 111.5, 40.44; IR (neat) 2808, 1696, 1600, 1498, 1356, 1205; HRMS (ESI) m/e calcd for $C_9H_{12}NO$ (M+H) 150.0919, found 150.0922.

To a solution of 3-dimethylaminobenzaldehyde (1.5 g, 10.1 mmol) and trimethylorthoformate (5.9 mL, 50.3 mmol) in methanol (25 mL) was added NH_4Cl (16 mg, 0.3 mmol) at room temperature. Reaction mixture was stirred at room temperature for 72 h and the reaction solution was concentrated. The crude mixture was purified by column chromatography (benzene/EA 95:5) to afford the acetal **5** (1.8 m, 87%) as a yellow liquid: Rf = 0.5 (benzene/EA 95:5); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.25 (t, *J* = 7.8 Hz, 1H), 6.83 (m, 2H), 6.69 (dd, *J* = 8.0, 2.5 Hz, 1H), 5.33 (s, 1H), 3.34 (s, 6H), 2.96 (s, 6H); ^{13}C NMR (75 MHz, $CDCl_3$) δ150.5, 138.7, 128.8, 115.0, 112.6, 110.6, 103.7, 52.7, 40.6; IR (neat) 2935, 2887, 2827, 1604, 1582, 1497, 1435, 1350; HRMS (ESI) m/e calcd for $C_{11}H_{18}NO_2$ (M+H) 196.1338, found 196.1342.

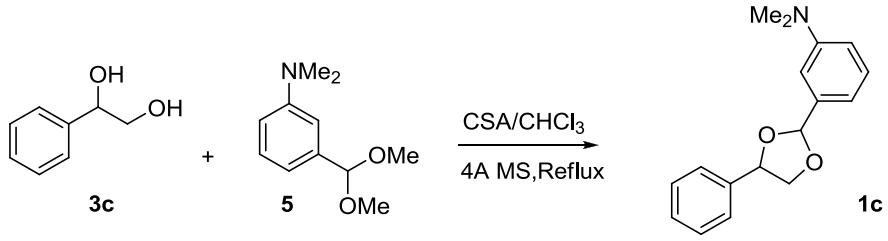


To a solution of the diol **3a** (134 mg, 0.75 mmol) and the PPG reagent **5** (293 mg, 1.50 mmol) in toluene (2 mL) was added a catalytic amount of CSA (35 mg, 0.15 mmol) and 4A molecular sieve (25 mg) at room temperature under N_2. The reaction mixture was refluxed for 24 h. After completion of reaction, the reaction solution was neutralized with TEA at room temperature and concentrated under reduced pressure. The crude reaction mixture was purified by column chromatography (PE/EA 10:1) to provide the desired product **1a** (165 mg, 71%) as a colorless liquid: Rf = 0.25 (PE/EA 10:1); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.26 (t, *J* = 7.8 Hz, 1H), 6.96 (s, 1H), 6.90 (d, *J* = 7.5 Hz, 1H), 6.77 (dd, *J* = 8.2, 2.0 Hz, 1H), 6.11 (s, 1H), 4.98 (d, *J* = 4.0 Hz, 1H), 4.86 (d, J = 3.7 Hz, 1H), 3.87 (s, 3H), 3.82 (s, 3H), 2.96 (s, 6H); ^{13}C NMR (176 MHz, $CDCl_3$) δ 170.2, 169.6, 150.7, 136.0, 129.2, 115.3, 114.2, 111.0, 107.3, 52.9, 40.6; IR (neat) 2954, 2905, 2803, 1754, 1677, 1609, 1585, 1503, 1438; HRMS (ESI) m/e calcd for $C_{15}H_{20}NO_6$ (M+H) 310.1291, found 310.1285.



To a solution of the diol **3b** (70 mg, 0.530 mmol) and the PPG reagent **5** (155 mg, 0.795 mmol) in $CHCl_3$(3 mL) was added a catalytic amount of CSA (25 mg, 0.106 mmol) and 4A molecular

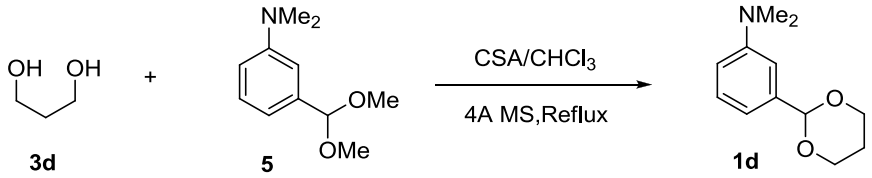
sieve (25 mg) at room temperature under N_2. The reaction mixture was refluxed for 4 h. After completion of the reaction, the reaction mixture was neutralized with TEA and then concentrated under reduced pressure. The crude product was purified by column chromatography (PE/EA 20:1) to provide the desired product **1b** (121 mg, 87%) as a colorless liquid: Rf =0.2 (PE/EA 20:1); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.22 (m, 1H), 6.84 (m, 2H), 6.72 (m,1H), 5.78-5.94 (m, 2H), 5.30-5.33 (m, 1H), 5.16-5.22 (m, 1H), 4.38-4.46 (m, 1H), 3.98-4.26 (m, 4H), 3.52-3.67 (m, 2H), 2.96 (s, 6H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 150.6, 138.6, 138.0, 134.4, 129.0, 117.3, 114.7, 114.5, 113.5, 113.3, 110.5, 110.2, 104.7, 104.1, 75.2, 74.8, 72.4, 70.9, 70.4, 67.9, 67.6, 40.6; IR (neat) 2872, 2804, 1680, 1607, 1584, 1500, 1440; HRMS (ESI) m/e calcd for $C_{15}H_{22}NO_3$ (M+H) 264.1600, found 264.1603.



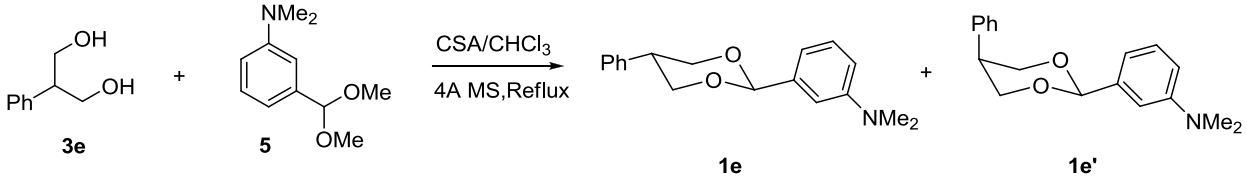
To a solution of the diol **3c** (69 mg, 0.50 mmol) and the PPG **5** (146 mg, 0.75 mmol) in $CHCl_3$ (3 mL) was added catalytic amount of CSA (23 mg, 0.10 mmol) and 4A MS (25 mg) at room temperature under N_2. The reaction mixture was refluxed for 5 h. After completion of reaction, the reaction solution was neutralized with TEA at room temperature and then concentrated under reduced pressure. The crude product was purified by column chromatography (PE/EA 100:1 to 20:1) to provide product **1c** (29 mg) and its diastereomer **1c'** (98 mg) as a colorless liquid with a combined 94% yield.

For **1c**, Rf = 0.5 (PE/EA 50:1); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.43-7.44 (m, 2H), 7.25-7.38 (m, 5H), 6.94 (m, 2H), 6.77 (m, 1H), 5.98 (s, 1H), 5.20 (t, *J* = 7.0 Hz, 1H), 4.37 (t, *J* = 7.0 Hz, 1H), 3.94 (t, *J* = 6.5 Hz, 1H), 2.96 (s, 6H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 150.7, 139.6, 139.0, 129.1, 128.6, 128.0, 126.1, 114.5, 113.4, 110.2, 104.9, 77.8, 72.7, 40.6; IR (neat) 3031, 2871, 2804, 1738, 1679, 1606, 1584, 1497; HRMS (ESI) m/e calcd for $C_{17}H_{20}NO_2$ (M+H) 270.1494, found 270.1495.

For **1c'**, Rf = 0.45 (PE/EA 50:1); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.26-7.43 (m. 6H), 6.90 (m, 2H), 6.75 (d, *J* = 7.3 Hz, 1H), 6.18 (s, 1H), 5.22 (t, *J* = 6.8 Hz, 1H), 4.52 (t, *J* = 7.0 Hz, 1H), 3.87 (t, *J* = 8.3 Hz, 1H), 2.97 (s, 6H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 150.6, 139.3, 138.2, 129.1, 128.5, 128.1, 126.4, 114.8, 113.5, 110.6, 105.0, 78.7, 72.1, 40.6; IR (neat) 3031, 2879, 2805, 1679, 1607, 1583, 1500, 1453; HRMS (ESI) m/e calcd for $C_{17}H_{20}NO_2$ (M+H) 270.1494, found 270.1497.



To a solution of the diol **3d** (45mg, 0.591 mmol) and the PPG reagent **5** (173 mg, 0.887 mmol) in $CHCl_3$ (3 mL) was added a catalytic amount of CSA (27 mg, 0.118mmol) and 4A molecular sieve (25 mg) under N_2 at room temperature. The reaction mixture was refluxed for 2 h, and was then neutralized with TEA at room temperature. The solution was concentrated under reduced pressure and the crude reaction mixture was purified by column chromatography (PE/EA 10:1) to provide **1d** (109 mg, 89%) as a colorless liquid: Rf = 0.25 (PE/EA 4:1); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.22 (t, *J* = 7.8 Hz, 1H), 6.83 (m, 2H), 6.72 (dd, *J* = 8.0, 2.5 Hz, 1H), 5.46 (s, 1H), 4.26 (dd, *J* = 10.8, 5.0 Hz, 2H), 3.98 (dt, *J* = 11.8, 2.0 Hz, 2H), 2.94 (s, 6H), 2.17-2.29 (m, 1H), 1.45 (d, *J* = 13.3 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 150.6, 139.4, 128.9, 114.3, 113.2, 109.9, 102.1, 67.3, 40.6, 25.7; IR (neat) 2985, 2922, 2850, 2802, 1680, 1608, 1584, 1499, 1438; HRMS (ESI) m/e calcd for $C_{12}H_{18}NO_2$ (M+H) 208.1338, found 208.1342.

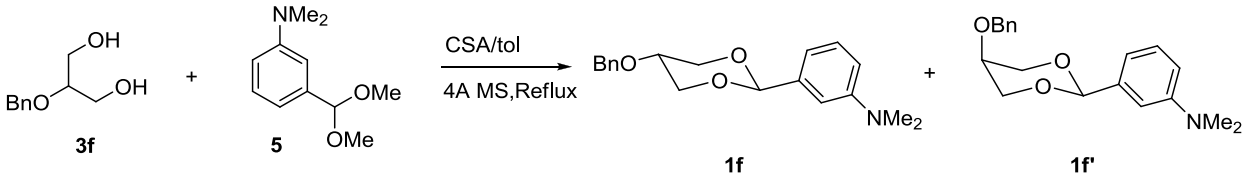


To a solution of the diol **3e** (99 mg, 0.650 mmol) and the PPG reagent **5** (190 mg, 0.975 mmol) in CH_3Cl (3 mL) was added a catalytic amount of CSA (30 mg, 0.130 mmol) and 4A MS (25 mg) at room temperature under N_2. The reaction mixture was refluxed for 2 h. After completion of reaction, the reaction mixture was neutralized with TEA at room temperature and then was concentrated under reduced pressure. The crude product was purified by column chromatography (PE/EA 50:1 to 20:1) to provide **1e** (115 mg) and its diastereomer **1e'** (42 mg) as colorless liquid in a combined 85% yield.

For **1e**, Rf = 0.4 (PE/EA 20:1); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.34 (m, 2H), 7.23-7.30 (m, 5 H), 6.93 (s, 1 H), 6.88 (d, *J* = 8.0 Hz, 1 H), 6.74 (dd, *J* = 8.0, 4.0 Hz, 1 H), 5.55 (s, 1H), 4.37 (dd, *J* = 12.0, 4.0 Hz, 2H), 4.04 (t, *J* = 12.0 Hz, 2H) 3.43-3.35 (m, 1H), 2.97 (s, 6H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 150.8, 139.0, 137.8, 129.1, 128.8, 127.7, 127.4, 114.5, 113.4, 110.1, 102.1, 72.4, 41.2, 40.7; IR (neat) 3029, 2959, 2917, 2850, 2802, 1679, 1605, 1583, 1495; HRMS (ESI) m/e calcd for $C_{18}H_{22}NO_2$ (M+H) 284.1651, found 284.1652.

For **1e'**, Rf = 0.35 (PE/EA 20:1); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.62 (d, J = 7.7, 2 H), 7.33 (t, J = 7.7, 2 H), 7.26-7.24 (m, 2 H), 6.91 (s, 1 H), 6.89 (d, *J* = 7.7 Hz, 1 H), 6.73 (dd, *J* = 8.4, 2.1 Hz, 1 H), 5.63 (s, 1 H), 4.39-4.34 (m, 4 H), 2.95 (s, 6 H), 2.76 (s, 1 H); ^{13}C NMR (176 MHz, $CDCl_3$)

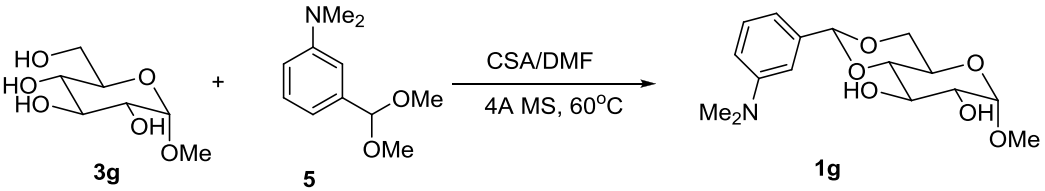
δ 150.8, 142.9, 139.3, 129.1, 128.6, 128.3, 126.5, 114.5, 113.3, 110.4, 102.1, 71.1, 40.7, 39.4; IR (neat); HRMS (ESI) m/e calcd for $C_{18}H_{22}NO_2$ (M+H) 284.1651, found 284.1648.



To a solution of the diol **3f** (120 mg, 0.659 mmol) and the PPG reagent **5** (193 mg, 0.988 mmol) in toluene (2 mL) was added a catalytic amount of CSA (31 mg, 0.132 mmol) and 4A MS (25 mg) at room temperature under N_2. The reaction mixture was refluxed for 3.5 h. After completion of reaction, the reaction mixture was neutralized with TEA at room temperature and then was concentrated under reduced pressure. The crude product was purified by column chromatography (PE/EA 20:1 to 5:1) to provide **1g** (138 mg) and its isomer **1g'** (50 mg) as a colorless liquid in a combined 91% yield.

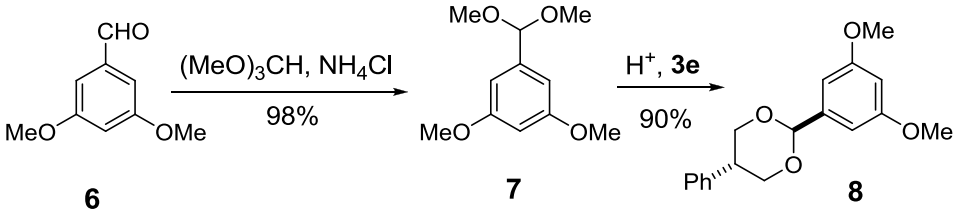
For **1f**, Rf = 0.2 (PE/EA 20:1); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.31-7.39 (m. 5H), 7.19-7.25 (t, *J* = 7.8 Hz, 1H), 6.81 (m, 2H), 6.70 (dd, *J* = 8.3, 2.7 Hz, 1H), 5.35 (s, 1H), 4.60 (s, 2H), 4.35 (dd, J = 10.6, 4.8 Hz, 2H), 3.79 (m, 1H), 3.62-3.67 (m, 2H), 2.94 (s, 6H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 150.6, 136.3, 137.9, 128.9, 128.5, 128.0, 127.7, 114.3, 113.3, 109.9, 101.8, 71.7, 70.1, 67.9, 40.6; IR (neat) 2858, 1678, 1608, 1100; HRMS (ESI) m/e calcd for $C_{19}H_{24}NO_3$ (M+H) 314.1756, found 314.1755.

For **1f'**, Rf = 0.5 (PE/EA 5:1); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.20-7.42 (m, 6H), 6.91 (s, 1H), 6.86 (d, *J* = 7.3 Hz, 1H), 6.71 (dd, *J* = 8.0, 2.2 Hz, 1H), 5.52 (s, 1h), 4.71 (s, 2H), 4.35 (d, *J* = 12.3 Hz, 2H), 4.02 (d, *J* = 12.6 Hz, 2H), 3.34 (s, 1H), 2.95 (s, 6H); ^{13}C NMR (75 MHz, $CDCl_3$) δ150.6, 138.8, 138.2, 128.8, 128.4, 127.7, 127.6, 114.6, 113.3, 110.2, 101.9, 70.2, 69.3, 68.9, 40.7; IR (neat) 2969; 2852, 1607, 14981343; HRMS (ESI) m/e calcd for $C_{19}H_{24}NO_3$ (M+H) 314.1756, found 314.1757.



To a solution of the methyl glucoside **3g** (97 mg, 0.5 mmol) and the PPG reagent **5** (146 mg, 0.75 mmol) in DMF (2 mL) was added a catalytic amount of CSA (23 mg, 0.1mmol) and 4A molecular sieve (25 mg) at room temperature under N_2. The reaction mixture was stirred at 60 ^{o}C for 2 h. After completion of reaction, the reaction mixture was neutralized with TEA and purified

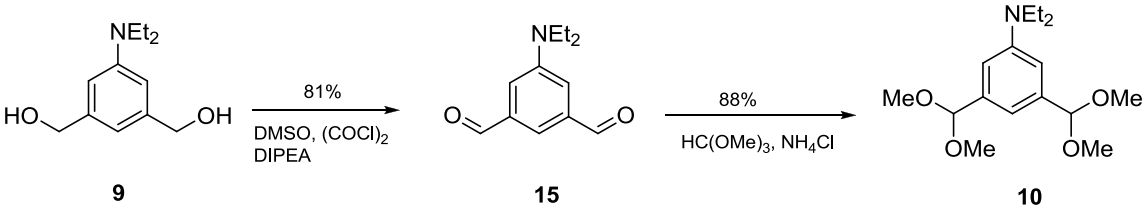
with column chromatography (DCM/MeOH 20:1) to produce **1g** as a colorless liquid which solidifies at 0 ^{o}C (138 mg, 85%). Rf = 0.4 (DCM/MeOH = 20:1); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.21 (t, *J* = 8.0 Hz, 1H), 6.82 (m, 2H), 6.72 (dd, J = 8.0, 2.5 Hz, 1H), 5.47 (s, 1H), 4.78 (d, *J* = 3.7 Hz, 1H), 4.29 (dd, *J* = 10.1, 4.8 Hz, 1H), 3.94 (t, *J* = 9.3 Hz, 1H), 3.80 (m, 1H), 3.72 (m, 1H), 3.63 (m, 1H), 3.46 (s, 3H), 2.95 (s, 6H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 150.6, 137.7, 129.0, 114.5, 113.6, 110.2, 102.6, 99.7, 81.0, 72.7, 71.7, 68.9, 62.3, 55.5, 40.6; IR (neat) 3433, 2912, 1608, 1501, 1073; HRMS (ESI) m/e calcd for $C_{16}H_{24}NO_6$ (M+H) 326.1604, found 326.1607.



6 **7** **8**

To a solution of the benzaldehyde **6** (2.0 g, 12.0 mmol) and and trimethylorthoformate (7.0 mL, 60.2 mmol) in methanol (20 mL) was added NH_4Cl (64.0 mg, 1.2 mmol). The reaction mixture was stirred for 24 h. After completion, the reaction mixture was concentrated under reduced pressure. The residue was purified by column chromatography (PE/EA 19:1) to provide the desired acetal **7** (2.4 g, 98%) as a colorless oil. Rf = 0.5 (PE/EA = 19:1); ^{1}H NMR (700 MHz, $CDCl_3$) δ 6.62 (s, 2H), 6.43 (s, 1H), 5.31 (s, 1H), 3.80 (s, 6H), 3.34 (s, 6H); ^{13}C NMR (176 MHz, $CDCl_3$) δ 160.8, 140.5, 104.5, 103.1, 100.7, 55.4, 52.8; IR (neat) 2943, 2833, 1599, 1460, 1356, 1298; HRMS (ESI) m/e calcd for $C_{11}H_{16}O_4Na$ (M+Na) 235.0946, found 235.0936.

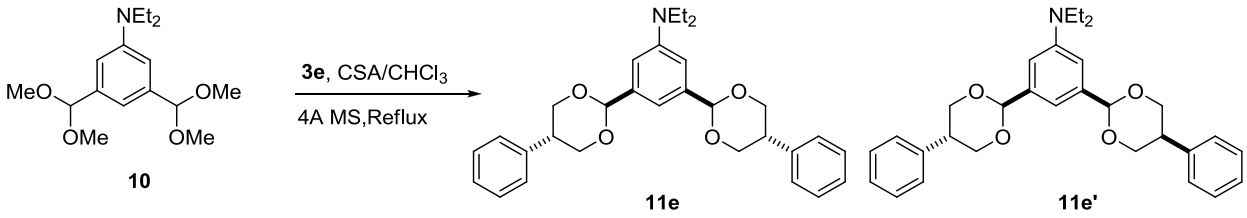
To a solution of the diol **3e** (100.0 mg, 0.66 mmol) and the PPG reagent **7** (278.0 mg, 1.31 mmol) in DCM (2 mL) was added a catalytic amount of *p*-TsOH (25 mg, 0.13 mmol) at room temperature under N_2. The reaction mixture was stirred for 7 h. After completion of reaction, the reaction mixture was concentrated and purified with column chromatography (PE/EA 39:1) to produce **8** (178 mg, 90%) as a white solid. Rf = 0.2 (PE/EA = 39:1); ^{1}H NMR (700 MHz, $CDCl_3$) δ 7.36 (t, *J* = 7.4 Hz, 2H), 7.30 (t, *J* = 7.4 Hz, 1H), 7.24 (d, *J* = 7.5 Hz, 2H), 6.72 (s, 2H), 6.46 (s, 1H), 5.53 (s, 1H), 4.37 (dd, *J* = 11.3, 4.5 Hz, 2H), 4.04 (t, *J* = 11.3 Hz, 1H), 3.82 (s, 6H), 3.38 (m, 1H); ^{13}C NMR (176 MHz, $CDCl_3$) δ 160.8, 140.4, 137.6, 128.9, 127.7, 127.5, 103.9, 101.6, 101.4, 72.4, 55.4, 41.1; IR (neat) 3007, 2964, 2940, 2854, 1597, 1461, 1378,1201; HRMS (ESI) m/e calcd for $C_{18}H_{21}O_4$ (M+H) 301.1440, found 301.1435.



9 **15** **10**

To a stirred solution of oxalyl chloride (0.68 mL, 7.5 mmol) in dry DCM (50 mL) at -78 ^{o}C under nitrogen atmosphere, DMSO (1.065mL, 15 mmol) was added. The reaction mixture was stirred for 30 min before addition of the alcohol **9** (0.523 g, 2.5 mmol). After stirring for 1 h at -78 ^{o}C, DIPEA (3.48 mL, 20 mmol) was added and the reaction mixture was stirred for another 30 min. The reaction mixture was then gradually warmed up to 0 ^{o}C and was quenched with H_2O (25 mL). The organic phase was separated and the aqueous phase was extracted with DCM (1x25 mL). The combined organic phase was washed with water (3x15mL) and brine, dried over anhydrous Na_2SO_4, and concentrated under reduced pressure. The crude product was purified by column chromatography (PE/EA 30:1) to provide the desired dialdehyde **15** (418 mg, 81%) as a yellow solid. Rf = 0.2 (PE/EA 30:1); ^{1}H NMR (400 MHz, $CDCl_3$) δ 10.01 (s, 2H), 7.58 (s, 1H), 7.37 (s, 2H), 3.46 (q, *J* = 7.1 Hz, 4H), 1.22 (t, *J* = 7.1 Hz, 6H); ^{13}C NMR (176 MHz, $CDCl_3$) δ 192.2, 148.6, 138.0, 119.4, 116.0, 44.6, 12.3; IR (neat) 2972, 2833, 1687, 1592; HRMS (ESI) m/e calcd for $C_{12}H_{16}NO_2$ (M+H) 206.1181, found 206.1178.

To a solution of the dialdehyde (250 mg, 1.22 mmol) and trimethylorthoformate (1.1 mL, 9.736 mmol) in methanol (5 mL) was added NH_4Cl (13 mg, 0.24 mmol) at room temperature under N_2. The reaction mixture was refluxed and stirred for 24 h. After completion, the reaction mixture was concentrated under reduced pressure. The residue was purified by column chromatography (Hex/Tol/EA 15:15:1) to provide the desired diacetal **10** (319 mg, 88%) as a yellow solid. Rf = 0.2 (Hex/Tol/EA 15:15:1); ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.79 (s, 1H), 6.73 (s, 2H), 5.29 (s, 2H), 3.40-3.34 (m, 16H), 1.14 (t, *J* = 7.0 Hz, 6H); ^{13}C NMR (176 MHz, $CDCl_3$) δ 147.9, 138.9, 112.6, 110.0, 103.8, 52.9, 44.3, 12.6; IR (neat) 2967, 2933, 2894, 2827, 1602, 1465; HRMS (ESI) m/e calcd for $C_{16}H_{28}NO_4$ (M+H) 298.2018, found 298.2020.

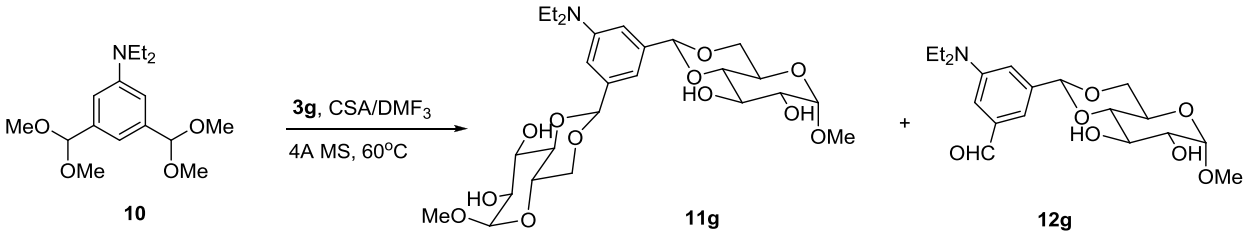


To a solution of diol **3e** (202 mg, 1.33 mmol) and the PPG reagent **10** (158 mg, 0.53 mmol) in CH_3Cl (3 mL) was added a catalytic amount of CSA (25 mg, 0.11 mmol) and 4A MS (25 mg) at room temperature under N_2. The reaction mixture was refluxed for 3 h. After completion of reaction, the reaction solution was neutralized with TEA at room temperature and then concentrated under reduced pressure. The crude product was purified by column chromatography (PE/EA 30:1 to 10:1) to provide the pure diacetal **11e** (95 mg), **11e'** (62mg), and a mixture of **11e** and **11e'** (58 mg) in a combined 86% yield.

For **11e**, Rf = 0.3 (PE/EA 10:1); ^{1}H NMR (700 MHz, $CDCl_3$) δ 7.35 (t, *J* = 7.5 Hz, 4H), 7.28 (t, *J* = 7.3 Hz, 2H), 7.24 (d, *J* = 7.2 Hz, 4H), 6.99 (s, 1H), 6.85 (s, 2H), 5.54 (s, 2H), 4.36 (dd, *J* = 11.5, 4.6 Hz, 4H), 4.02 (t, *J* = 11.5 Hz, 4H), 3.41-3.36 (m, 6H), 1.17 (t, *J* = 7.0 Hz, 6H); ^{13}C NMR (176 MHz, $CDCl_3$) δ 148.1, 139.3, 137.9, 128.8, 127.7, 111.2, 110.0, 101.9, 72.3, 44.2,

41.1, 12.7; IR (neat) 2966, 2921, 2872, 1598,1488, 1089; HRMS (ESI) m/e calcd for $C_{30}H_{36}NO_4$ (M+H) 474.2644, found 474.2635.

For **11e'**, Rf = 0.28 (PE/EA 10:1); ^{1}H NMR (700 MHz, $CDCl_3$) δ 7.64 (d, *J* = 7.0 Hz, 2H), 7.37 (t, *J* = 7.5 Hz, 2H), 7.34 (t, *J* = 7.8 Hz, 2H), 7.31 (t, *J* = 7.5 Hz, 1H), 7.26 (d, *J* = 7.5 Hz, 3H), 6.97 (s, 1H), 6.90 (s, 1H), 6.87 (s, 1H), 5.68 (s, 1H), 5.56 (s, 1H), 4.39-4.36 (m, 6H), 4.04 (t, *J* = 11.0 Hz, 2H), 3.43-3.40 (m, 5H), 1.19 (t, *J* = 7.0 Hz, 6H); ^{13}C NMR (176 MHz, $CDCl_3$) δ 148.1, 142.9, 139.5, 139.2, 137.9, 128.8, 128.6, 128.3, 127.7, 127.4, 126.5, 111.4, 110.1, 109.8, 102.0, 101.7, 72.4, 70.9, 44.4, 41.2, 39.6, 12.7; IR (neat) 2968, 2922, 2865, 1600, 1488, 1404; HRMS (ESI) m/e calcd for $C_{30}H_{36}NO_4$ (M+H) 474.2644, found 474.2633.



To a solution of **3g** (158 mg, 0.81 mmol) and the PPG reagent **10** (97 mg, 0.33 mmol) in DMF (3 mL) was added a catalytic amount of CSA (15 mg, 0.065mmol) and 4A molecular sieve (25 mg) at room temperature under N_2. The reaction mixture was heated at 60^{o}C for 4 h. After completion, the reaction mixture was neutralized with TEA and directly purified with column chromatography (DCM/MeOH 50:1) to provide **11g** (66 mg, 29%) and **12g** (54 mg, 35%).

For **11g**, Rf = 0.25 (DCM/MeOH 50:1); ^{1}H NMR (700 MHz, $CDCl_3$/CD_3CN 5:2) δ 6.85 (s, 1H), 6.74 (s, 2H), 5.44 (s, 2H), 4.74 (d, *J* = 3.8 Hz, 2H); 4.22 (dd, *J* = 10.2, 4.8 Hz, 2H); 3.80 (td, *J* = 9.1, 3.5 Hz, 2H); 3.74 (td, *J* = 9.8, 4.2 Hz, 2H); 3.69 (t, *J* = 9.2 Hz, 2H); 3.53 (td, *J* = 9.2, 4.2 Hz, 2H); 3.43 (s, 6H), 3.42 (t, *J* = 9.1 Hz, 2H); 3.38-3.35 (m, 6H), 2.88 (d, *J* = 8.4 Hz, 2H); 1.13 (t, *J* = 7.0 Hz, 3H); ^{13}C NMR (700 MHz, $CDCl_3$/CD_3CN 5:2) δ 152.9, 143.9, 116.8, 115.8, 107.54, 107.52, 105.5, 86.5, 78.2, 76.4, 74.1, 67.8, 60.6, 49.5, 17.6; IR (neat) 3347, 2926, 2867, 1606, 1364; HRMS (ESI) m/e calcd for $C_{26}H_{40}NO_{12}$ (M+H) 558.2551, found 558.2548.

For **12g**, Rf = 0.35 (DCM/MeOH 15:1); ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.92 (s, 1H), 7.27 (s, 1H), 7.12 (s, 1H), 7.00 (s, 1H), 5.52 (s, 1H), 4.81 (s, 1H), 4.31 (dd, *J* = 10.1, 4.8 Hz, 1H); 3.94 (t, *J* = 8.2 Hz, 1H); 3.83 (m, 1H), 3.75 (t, *J* = 10.3 Hz, 1H); 3.64 (m, 1H), 3.51 (t, *J* = 9.4 Hz, 1H); 3.47 (s, 3H), 3.44-3.36 (m, 5H), 2.88 (s, 1H), 2.39 (d, *J* = 14 Hz, 1H); 1.69 (s, 1H); 1.17 (t, *J* = 7.0 Hz, 3H); ^{13}C NMR (700 MHz, $CDCl_3$) δ 193.1, 148.2, 139.0, 137.6, 116.0, 115.5, 111.5, 101.8, 99.8, 81.0, 72.9, 71.8, 69.0, 62.3, 55.7, 44.4, 12.6; IR (neat) 3385, 3246, 2935, 2869, 1699, 1600, 1075; HRMS (ESI) m/e calcd for $C_{19}H_{28}NO_7$ (M+H) 382.1866, found 382.1859.

General procedure of photoreaction in NMR tubes: A 5.0 mM solution of **1g** (39 mg in 24 mL of MeOH) was equally distributed into 16 of 5 mm NMR tubes (1.5 mL x 16) in order to be irradiated under the same optimal conditions. (Typically, we first conducted photoreactions in NMR tubes to optimize the reaction conditions because we use ^{1}H NMR to monitor the reactions. Photo-deprotection reactions can also be carried out in other reaction vessels, for example, in a 250 mL reaction vessel in the regular setting of a Hanovia photoreactor.[1]) The NMR tubes were sealed and bound to the immersion well condenser of a Hanovia photoreactor with a 450 W medium pressure UV lamp. The reaction solution was irradiated with the UV light filtered through a Pyrex filter sleeve for 30 min. The reaction solutions in all NMR tubes were combined, concentrated and purified with column chromatography (DCM/MeOH 15:1 to 10:1) to provide the methyl glucoside **3g** (22 mg, 94%).

For the photo deprotection reaction of **1b**, the product **3b** was derivatized with TBSOTf/Et_3N, and isolated as the corresponding TBS-protected diol **S1** in 81% yield. Rf = 0.80 (PE/EA 19:1); ^{1}H NMR (400 MHz, $CDCl_3$) δ 5.81 (m, 1H), 5.18 (dd, *J* = 17.2, 1.6 Hz, 1H), 5.08 (dd, *J* = 10.4, 1.4 Hz, 1H), 3.91 (m, 2H), 3.75 (m, 1H), 3.50-3.41 (m, 3H), 3.30 (m, 1H), 0.8 (s, 18H), -0.03 (s, 12H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 135.0, 116.5, 72.7, 72.3, 65.1, 25.9, 18.3, 18.2; IR (neat) 2954, 2929, 2885, 2857, 1472, 1253; HRMS (ESI) m/e calcd for $C_{18}H_{41}O_3Si_2$ (M+H) 361.2594, found 361.2594.

Quantum yield determination: A 5.0 mM solution of **1** (in methanol) and a 5.0 mM solution of DEABn-protected ethylene glycol monoallyl ether (with known quantum yield of 0.26)[2] (CD_3CN/D_2O 4:1) in NMR tube were placed behind a standard 1 cm quartz UV cuvette contain the filter solution. Filtered light centered at 312 nm was obtained by passing light from the 450 W medium pressure mercury lamp through a solution of 2.0 mM K_2CrO_4 in a 5% K_2CO_3 aqueous solution.[3] The yields of photo reactions were determined by ^{1}H NMR analysis.

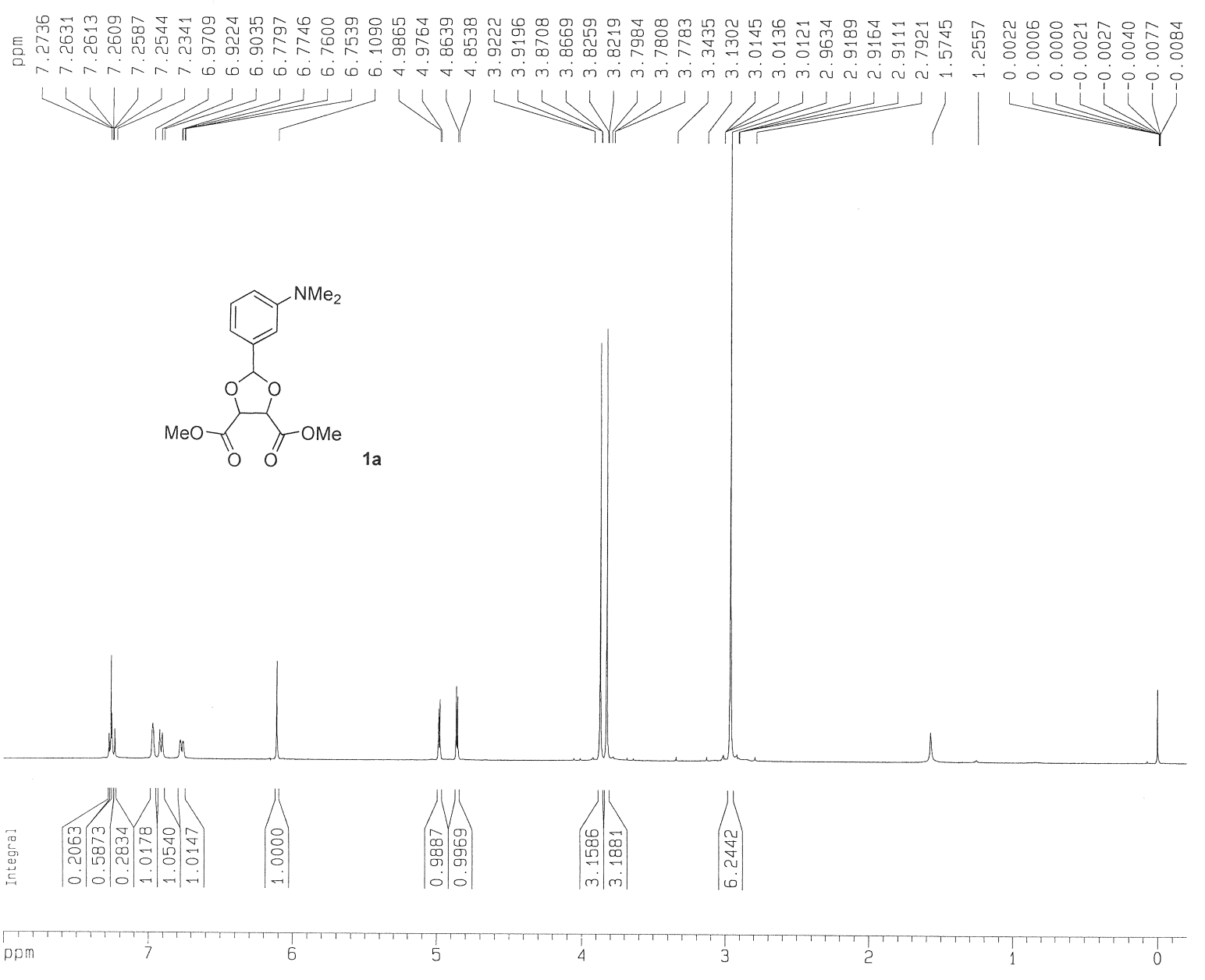
References:

[1] Wang, P.; Devalankar, D.; Lu, W. *J. Org. Chem*. **2016**, *81*, 6195.

[2] Wang, P.; Lu, W.; Devalankar, D. A.; Ding, Z. *Org. Lett*. **2015**, *17*, 2114.

[3] Filter solutions: (a) Zimmerman, H.; Nuss, J.; Tantillo, A. *J. Org. Chem*. **1988**, *53*, 3792. (b) Zimmerman, H. *Mol. Photochem*. **1971**, *3*, 281.

1H



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PROCNO	1

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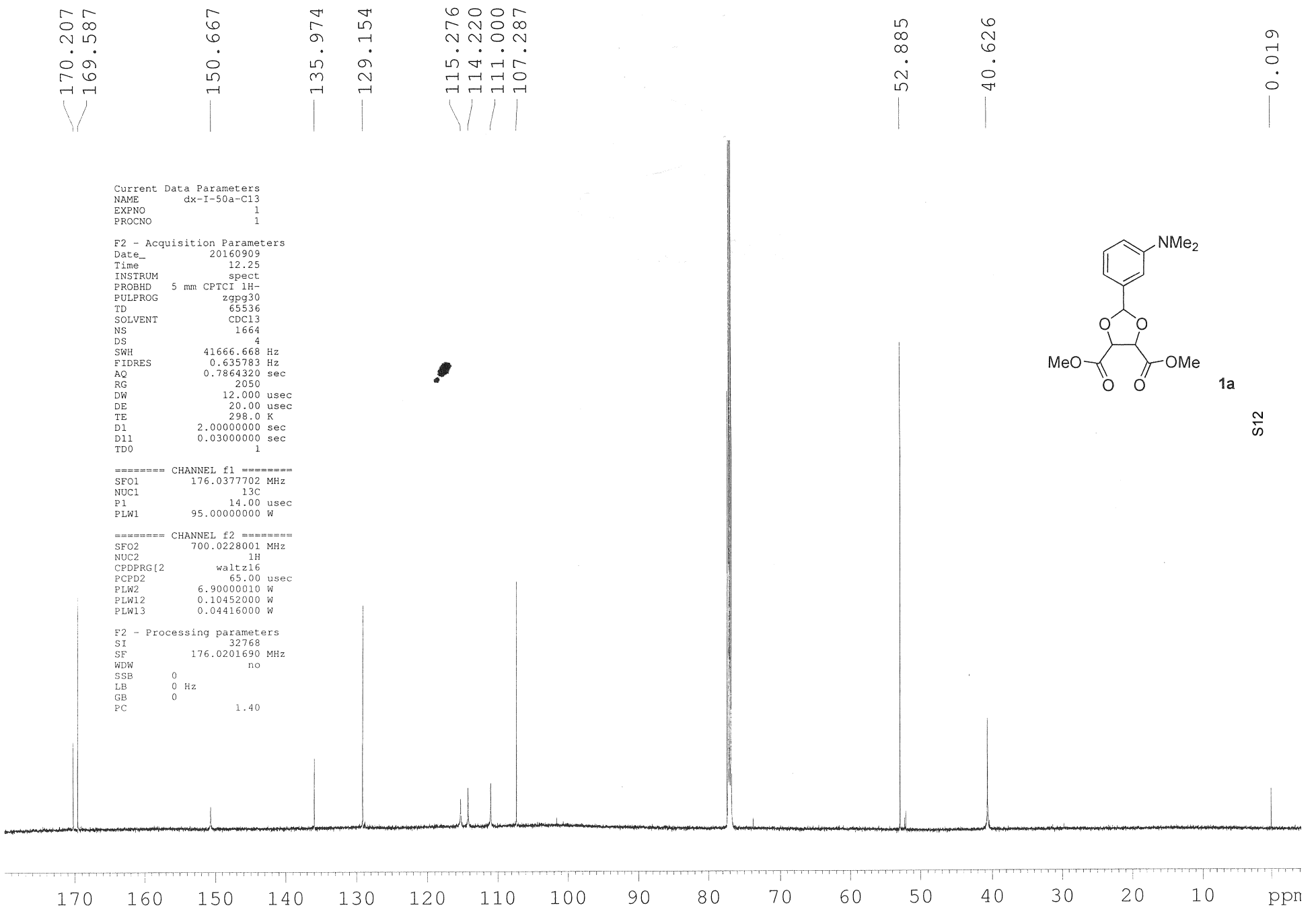
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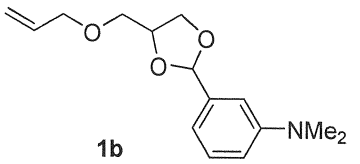
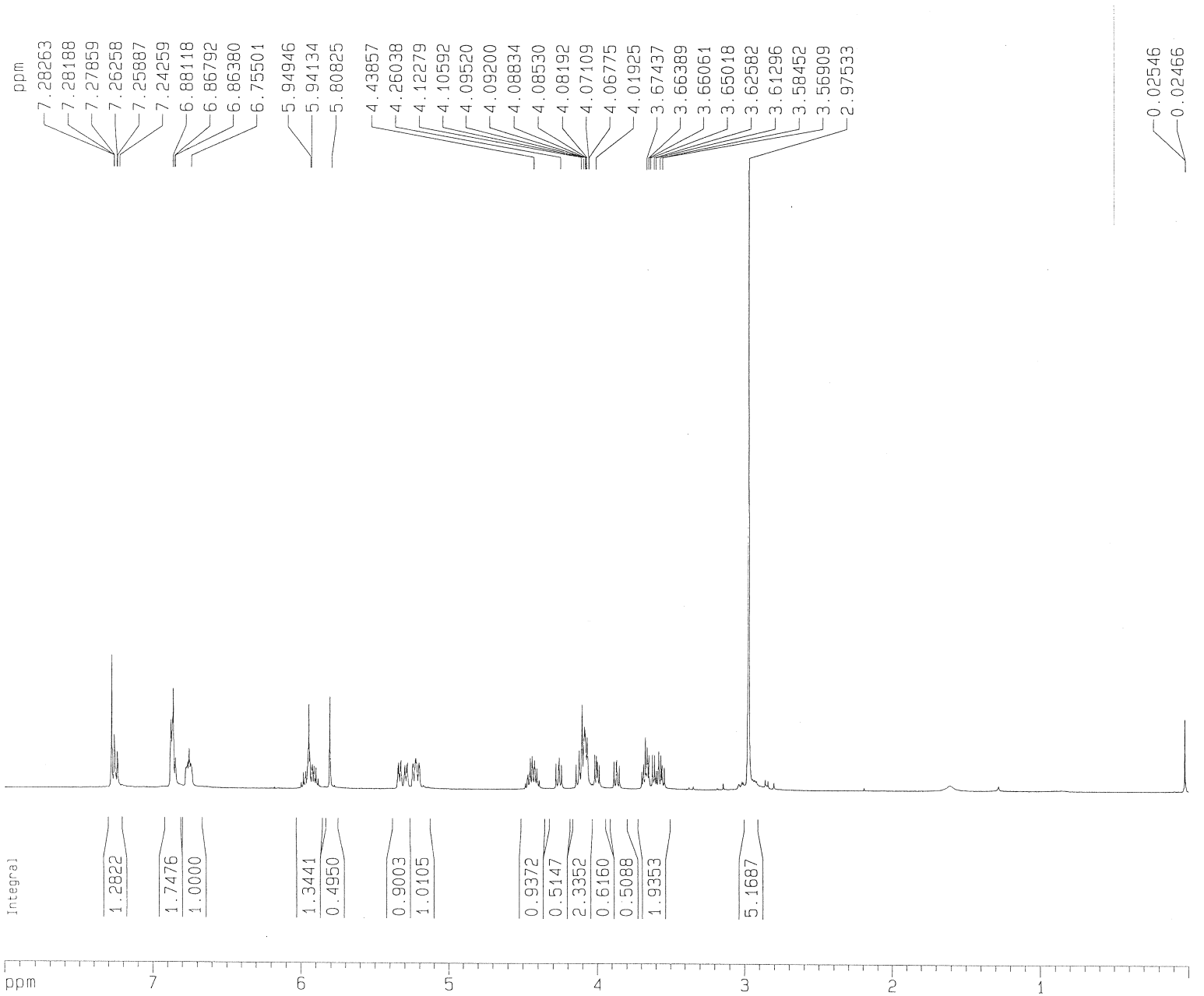
1H



1b

1b



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PROCNO	1

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TE	300.0 K
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1D NMR plot parameters

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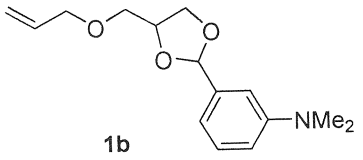

1b

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BRUKER

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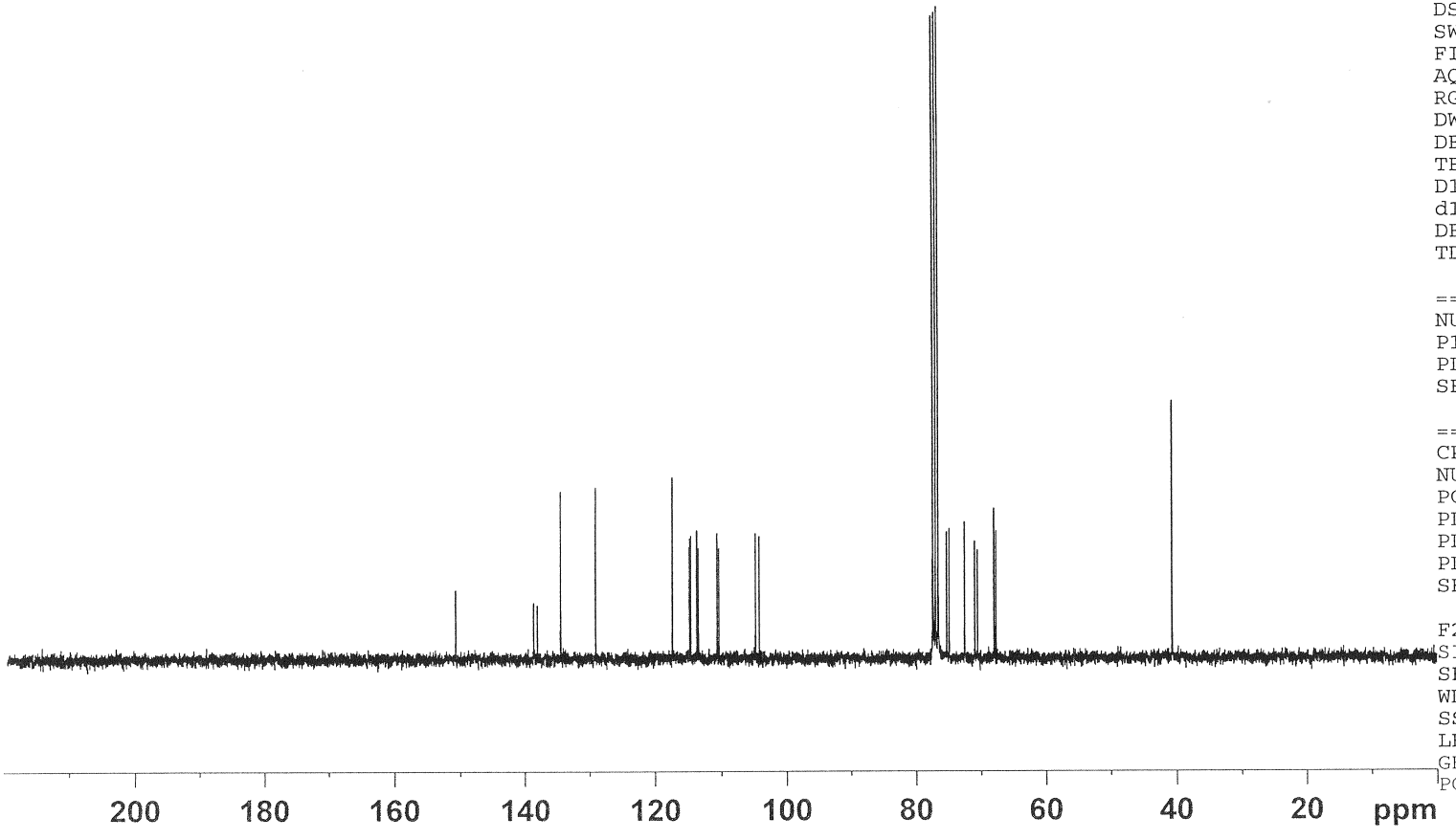

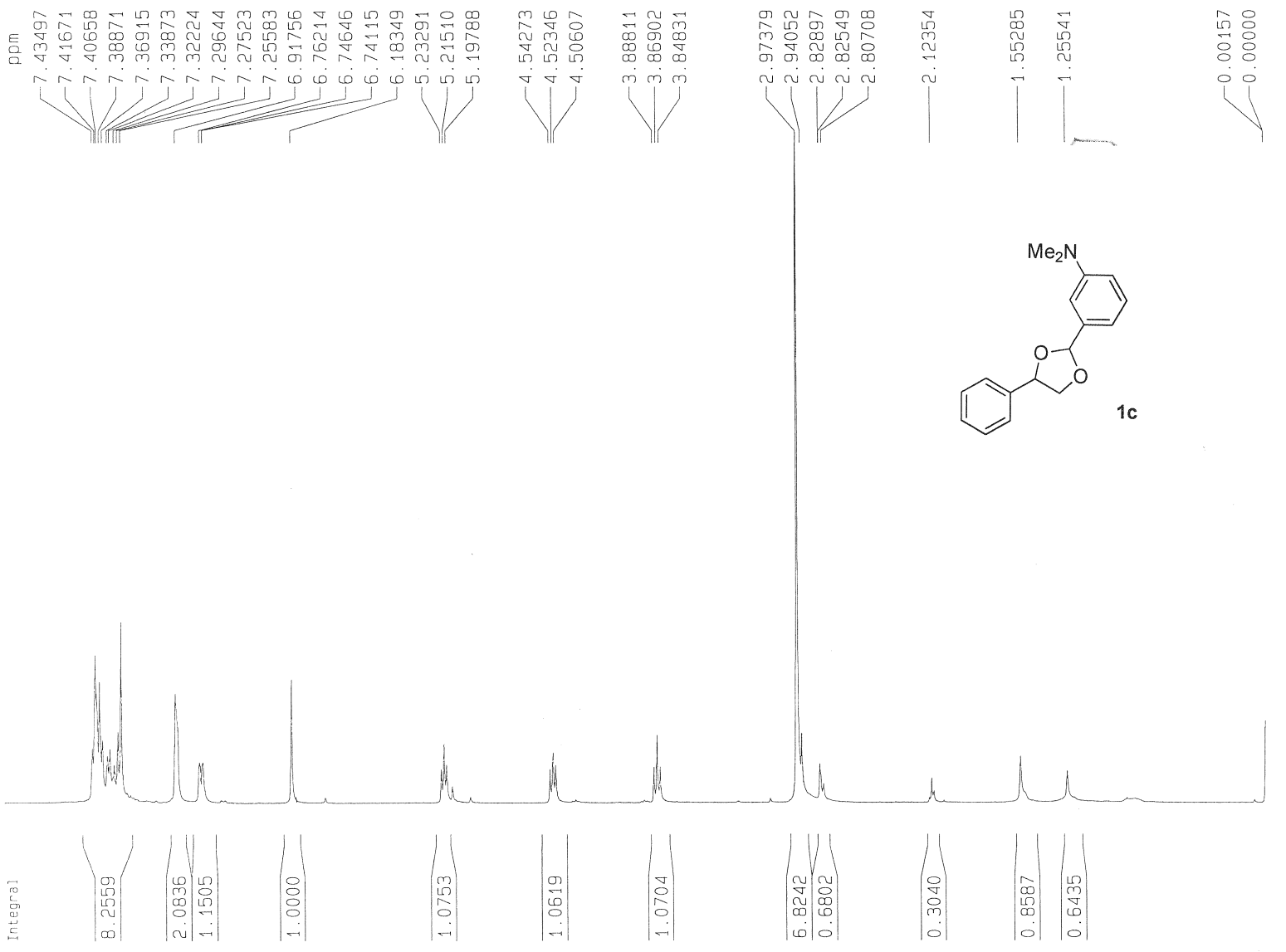

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PROCNO	1

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1D NMR plot parameters

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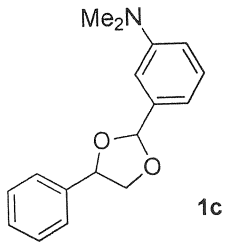

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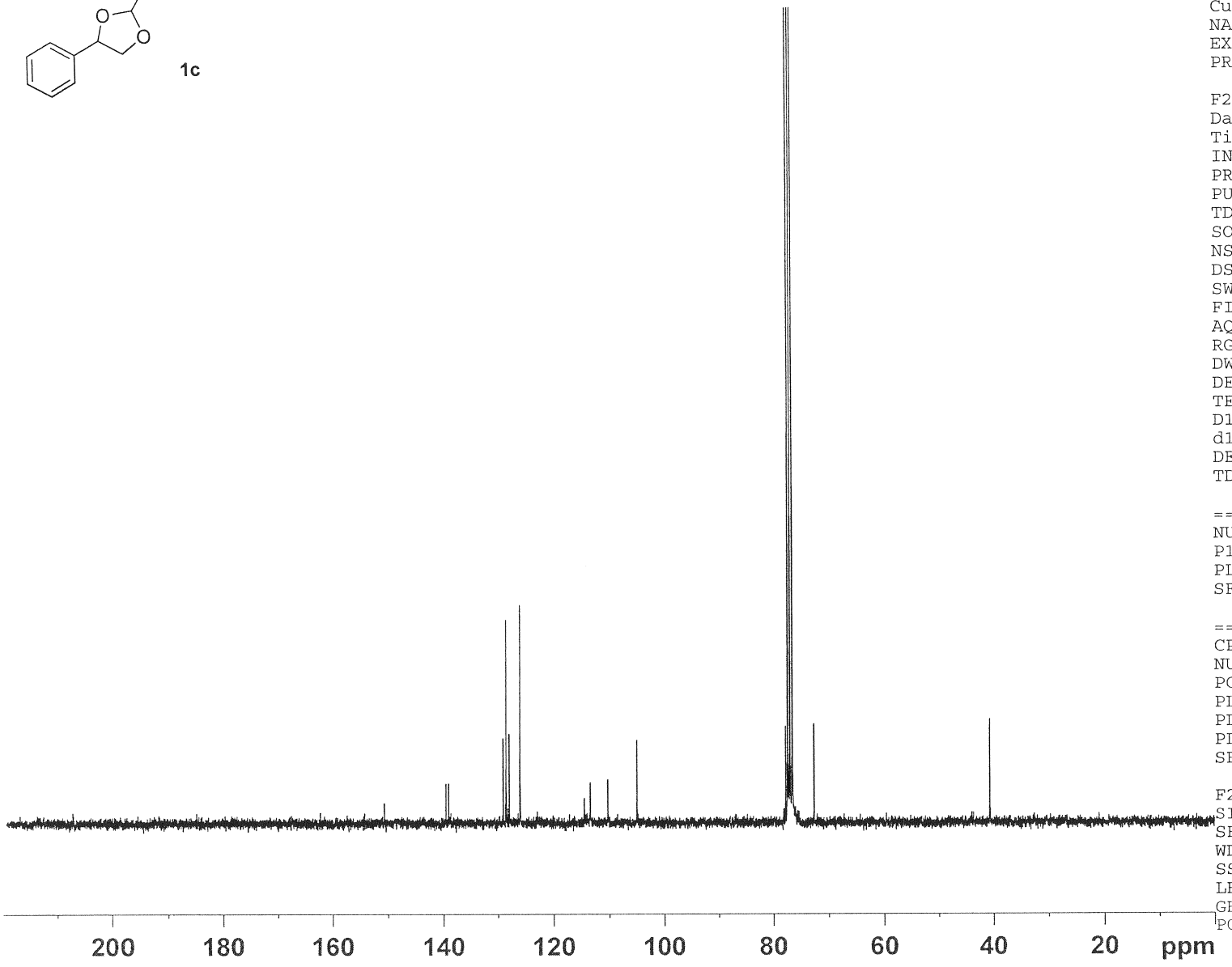

BRUKER

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TE 300.0 K
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d11 0.03000000 sec
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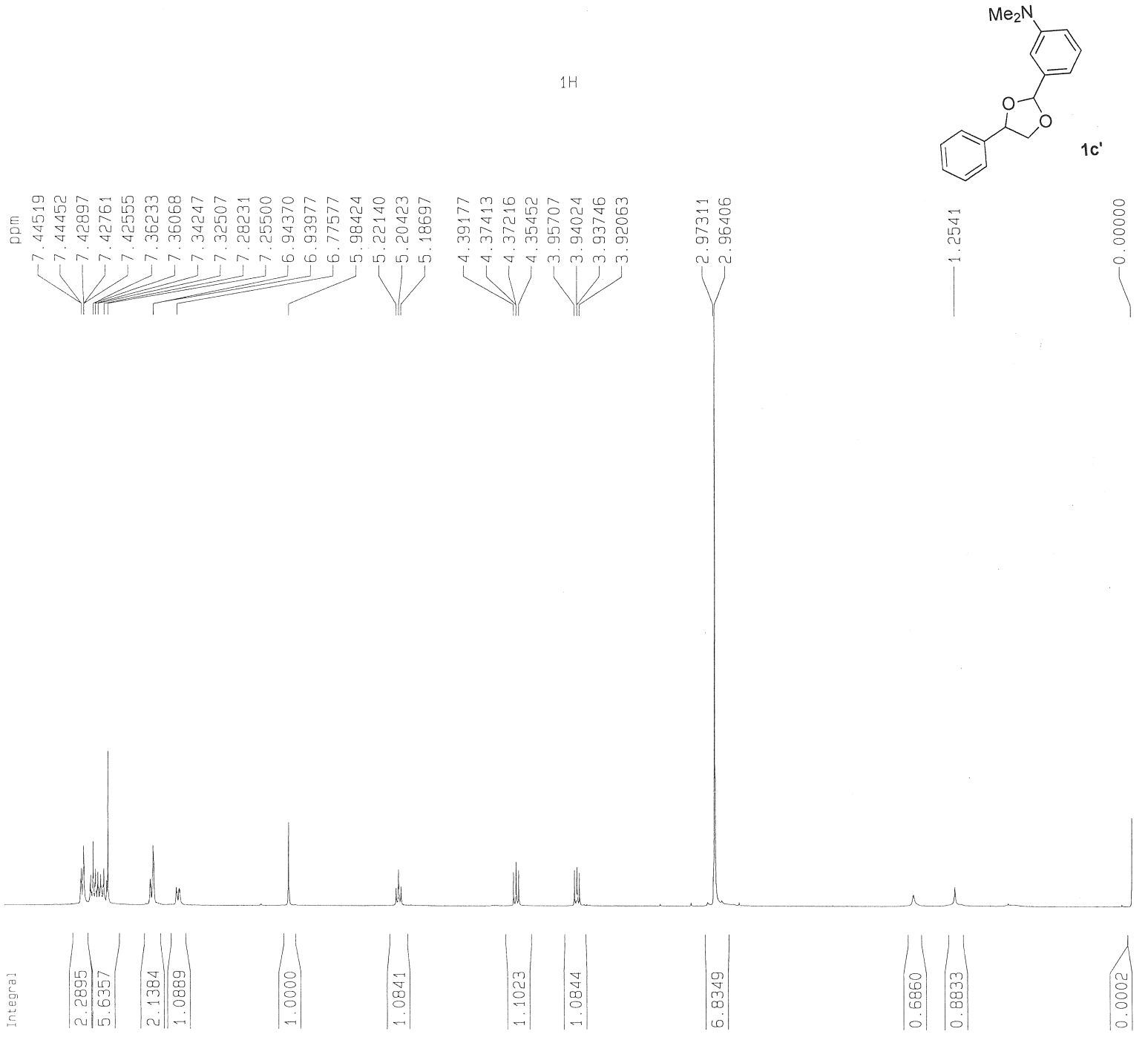

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TE	300.0 K
D1	1.00000000 sec

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1D NMR plot parameters

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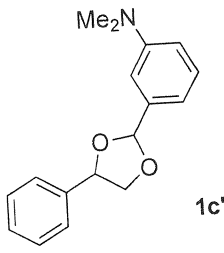
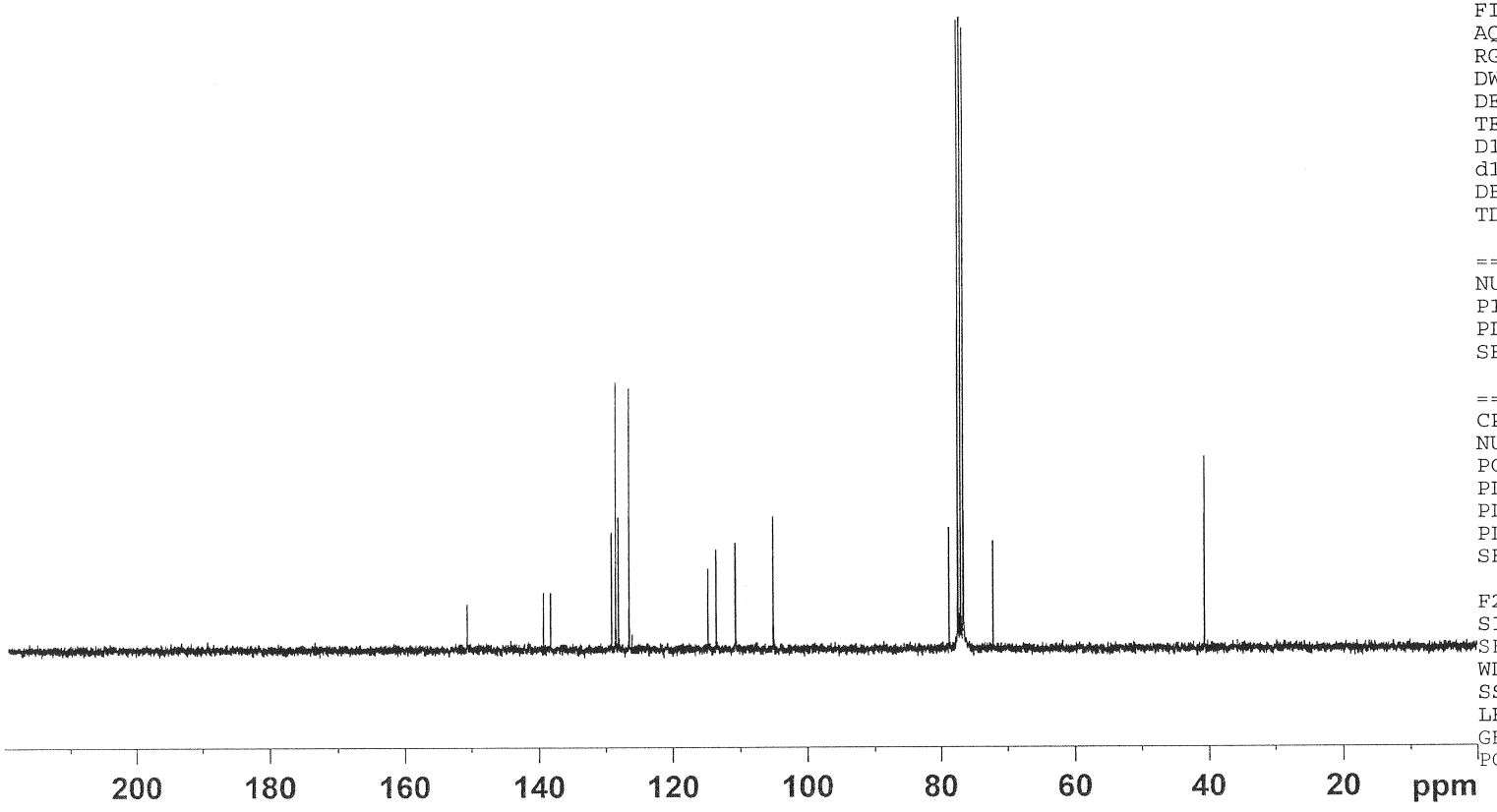
PPMCM	0.40000 ppm/cm
HZCM	160.05200 Hz/cm



13C

150.70
139.25
138.29
129.17
128.64
128.55
128.14
126.52
114.84
113.57
110.70
105.11
78.60
77.45
77.22
77.02
76.60
72.30



BRUKER

Current Data Parameters
NAME dd-4-78-spot2-13C
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20160404
Time 16.21
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 32768
DW 27.800 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1

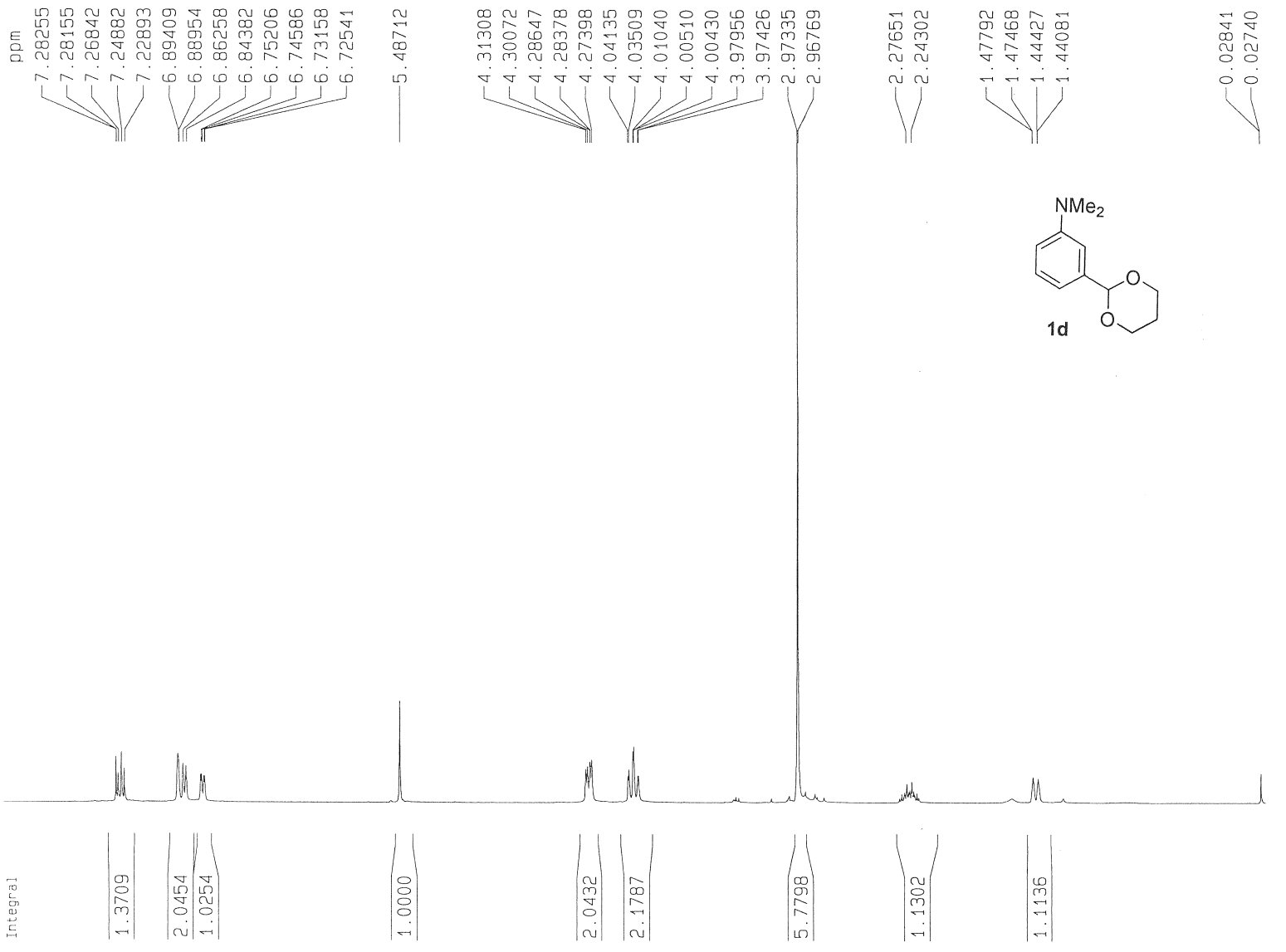
======== CHANNEL f1 ========
NUC1 13C
P1 8.00 usec
PL1 -1.80 dB
SFO1 75.4124265 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 75.00 usec
PL2 -2.50 dB
PL12 11.48 dB
PL13 12.00 dB
SFO2 299.8811995 MHz

F2 - Processing parameters
SI 32768
SF 75.4048860 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1d

1H



Current Data Parameters

NAME	dd-4-64pure
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

Date_	20160314
Time	7.33
INSTRUM	drx400
PROBHD	5 mm Multinu
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	64
DS	2
SWH	8278.146 Hz
FIDRES	0.126314 Hz
AQ	3.9584243 sec
RG	228.1
DW	60.400 usec
DE	6.00 usec
TE	300.0 K
D1	1.00000000 sec

========== CHANNEL f1 ========

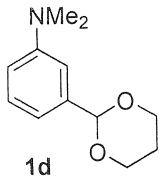
NUC1	1H
P1	6.50 usec
PL1	0.00 dB
SFO1	400.1324710 MHz

F2 - Processing parameters

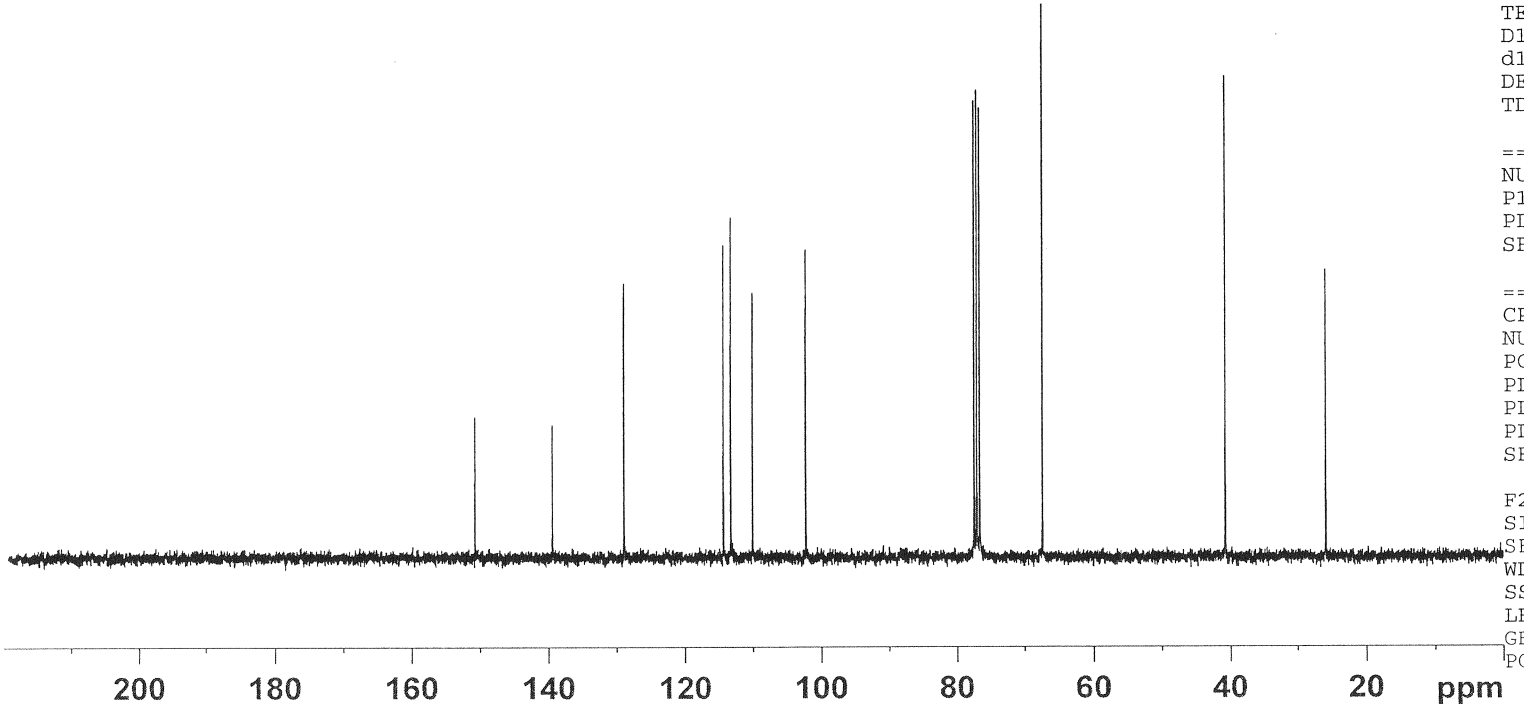
SI	131072
SF	400.1300000 MHz
WDW	no
SSB	0
LB	0.00 Hz
GB	0
PC	1.00

1D NMR plot parameters

CX	20.00 cm
F1P	8.000 ppm
F1	3201.04 Hz
F2P	0.000 ppm
F2	0.00 Hz
PPMCM	0.40000 ppm/cm
HZCM	160.05200 Hz/cm



150.76
139.46
128.00
114.40
113.22
110.05
102.35
77.51
77.08
76.66
67.41
40.77
25.82



Current Data Parameters
NAME dd-4-64-13c
EXPNO 1
PROCNO 1

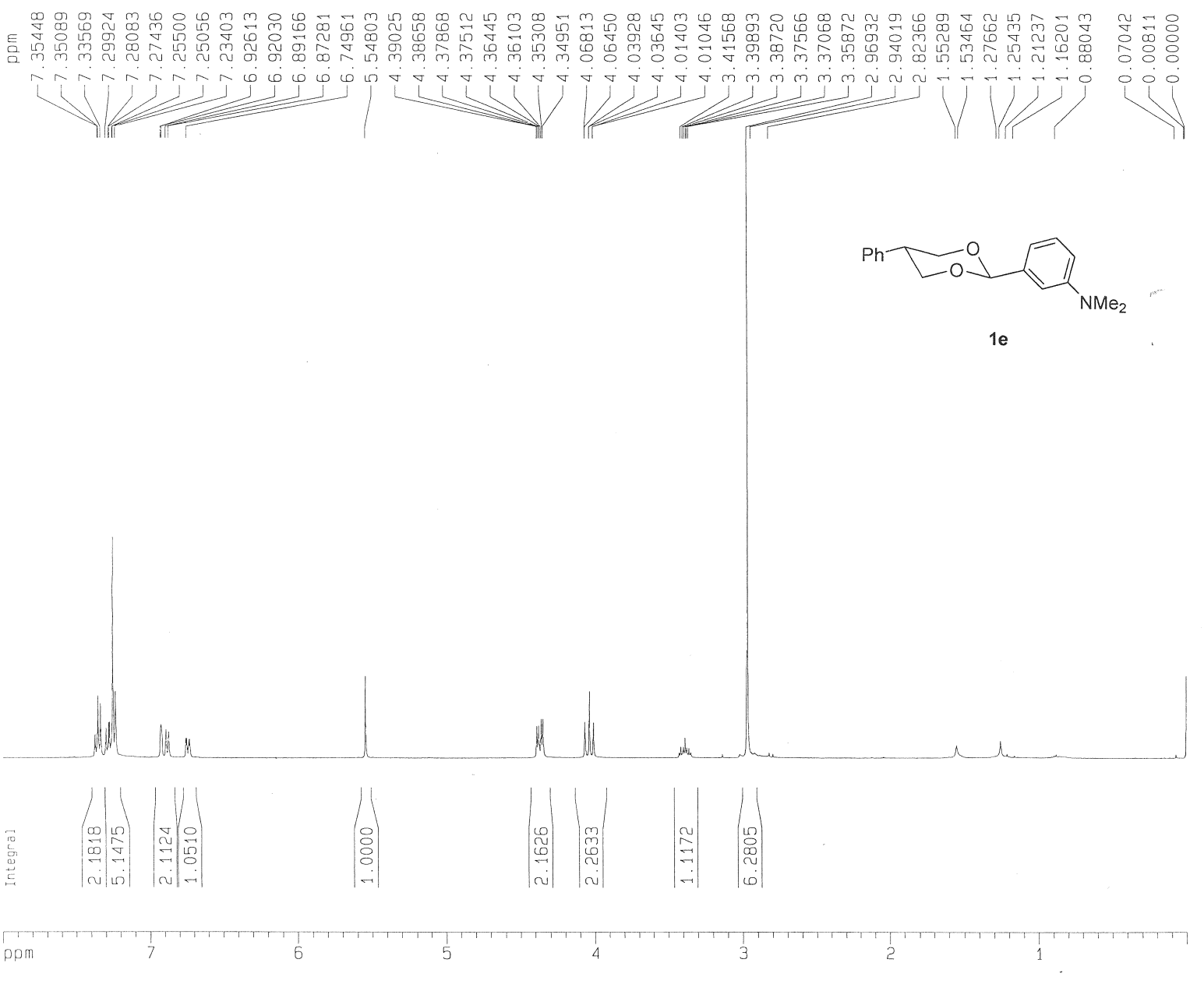
F2 - Acquisition Parameters
Date_ 20160329
Time 17.34
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 295
DS 4
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 32768
DW 27.800 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 8.00 usec
PL1 -1.80 dB
SFO1 75.4124265 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 75.00 usec
PL2 -2.50 dB
PL12 11.48 dB
PL13 12.00 dB
SFO2 299.8811995 MHz

F2 - Processing parameters
SI 32768
SF 75.4048860 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1H



1e

Current Data Parameters

NAME	dd-4-69-1spot
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

Date_	20160321
Time	16.33
INSTRUM	drx400
PROBHD	5 mm Multinu
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	64
DS	2
SWH	8278.146 Hz
FIDRES	0.126314 Hz
AQ	3.9584243 sec
RG	406.4
DW	60.400 usec
DE	6.00 usec
TE	300.0 K
D1	1.00000000 sec

========== CHANNEL f1 ========

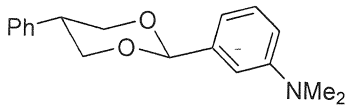
NUC1	1H
P1	6.50 usec
PL1	0.00 dB
SFO1	400.1324710 MHz

F2 - Processing parameters

SI	131072
SF	400.1300109 MHz
WDW	no
SSB	0
LB	0.00 Hz
GB	0
PC	1.00

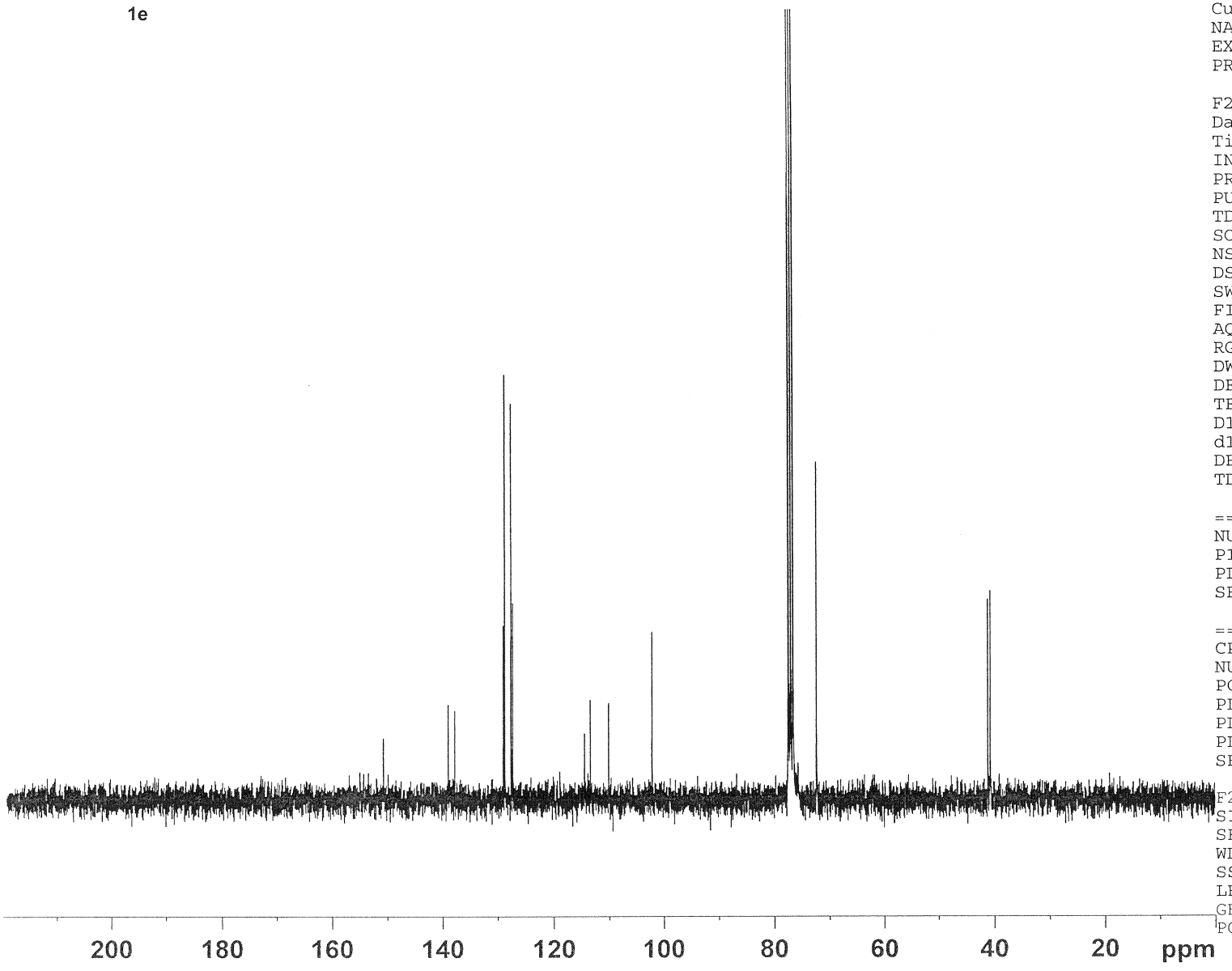
1D NMR plot parameters

CX	20.00 cm
F1P	8.000 ppm
F1	3201.04 Hz
F2P	0.000 ppm
F2	0.00 Hz
PPMCM	0.40000 ppm/cm
HZCM	160.05200 Hz/cm



1e

150.77
134.99
133.79
129.07
128.80
127.71
117.41
114.47
113.44
110.06
102.11
77.44
77.02
76.59
72.39
41.16
40.72



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Current Data Parameters
NAME dd-4-69spot1-13c
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20160330
Time 13.59
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 32768
DW 27.800 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 8.00 usec
PL1 -1.80 dB
SFO1 75.4124265 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 75.00 usec
PL2 -2.50 dB
PL12 11.48 dB
PL13 12.00 dB
SFO2 299.8811995 MHz

F2 - Processing parameters
SI 32768
SF 75.4048860 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

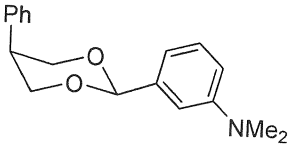
7.332
7.321
7.261
7.249
7.245
7.243
7.238
6.908
6.891
6.880
6.739
6.736
6.727
6.724
5.633
4.386
4.382
4.370
4.365
4.354
4.337
2.950
2.948
2.761
1.561
1.254
-0.000

```
Current Data Parameters
NAME      dx-I-66-2-Hconfirm
EXPNO                      2
PROCNO                     1

F2 - Acquisition Parameters
Date_            20160713
Time                18.54
INSTRUM             spect
PROBHD   5 mm CPTCI 1H-
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             14097.744 Hz
FIDRES           0.215115 Hz
AQ               2.3243434 sec
RG                  22.6
DW                 35.467 usec
DE                 10.00 usec
TE                 298.0 K
D1              1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1          700.0243229 MHz
NUC1                  1H
P1                  8.00 usec
PLW1          6.90000010 W

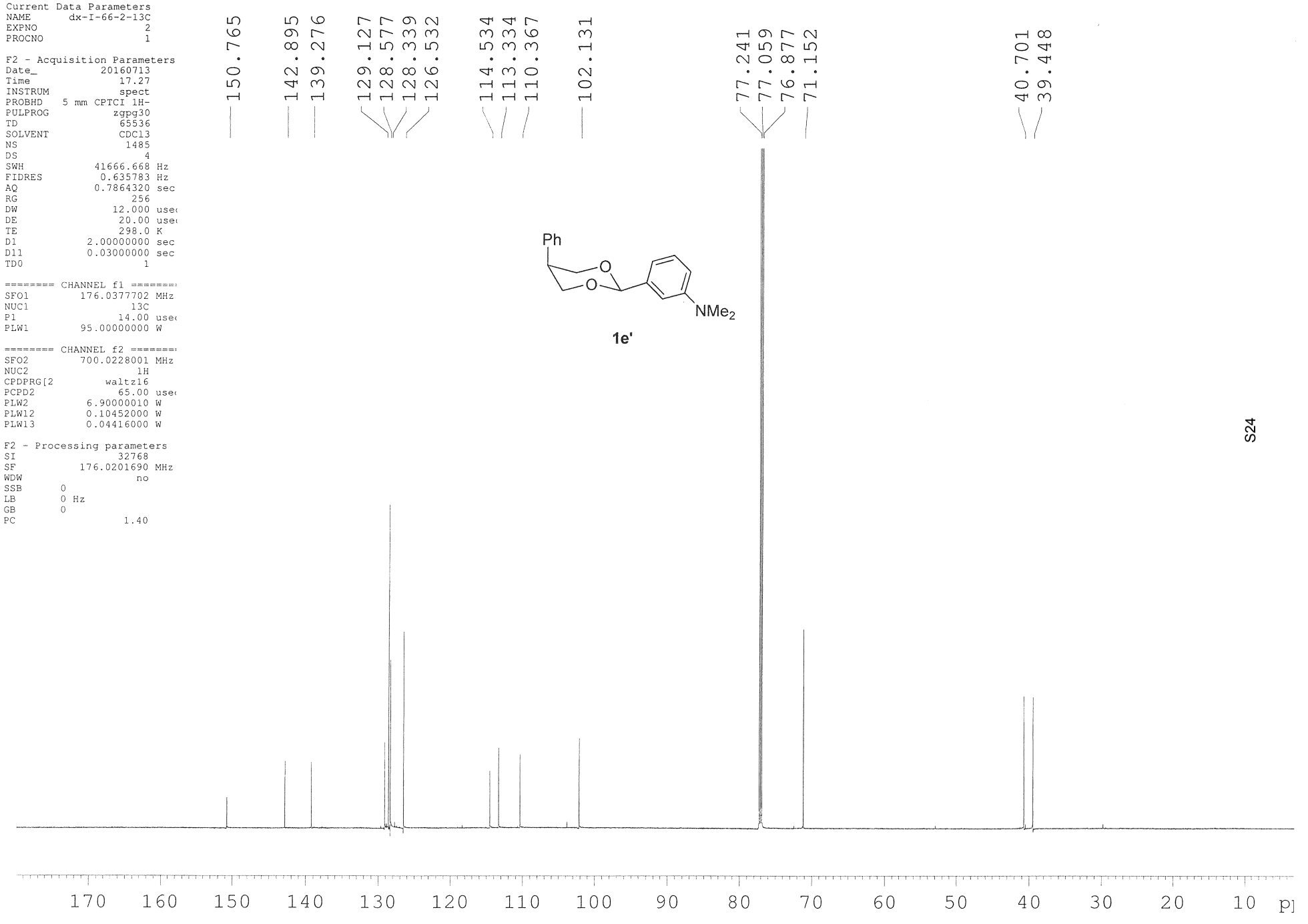
F2 - Processing parameters
SI                 65536
SF            700.0200283 MHz
WDW                   no
SSB       0
LB        0 Hz
GB        0
PC                 1.00
```

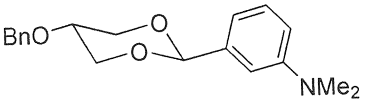


1e'

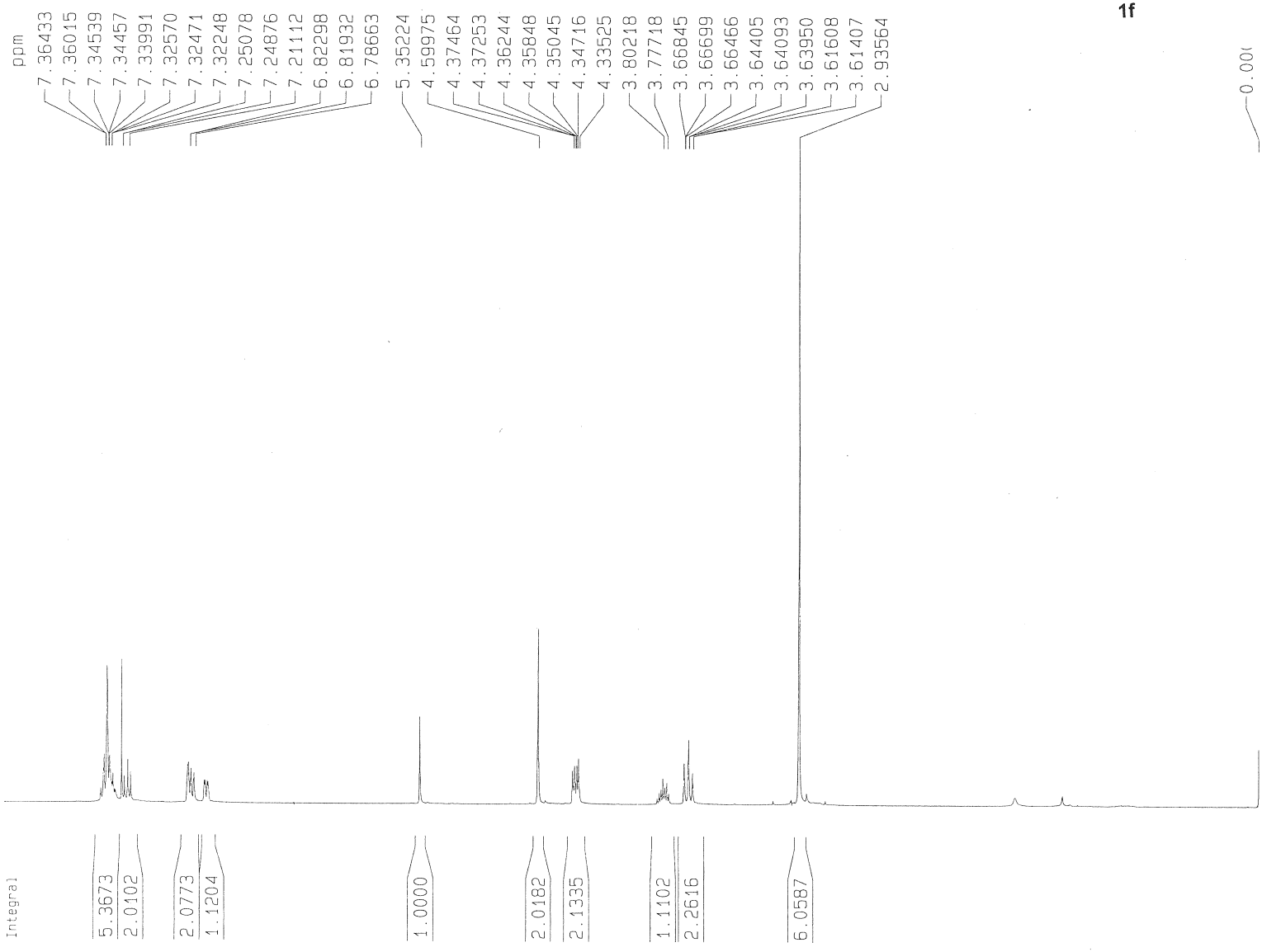
7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm

2.006 2.045 2.526 2.010 1.008 1.000 4.111 6.118 1.008


Current Data Parameters
NAME dx-I-66-2-13C
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160713
Time 17.27
INSTRUM spect
PROBHD 5 mm CPTCI 1H-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1485
DS 4
SWH 41666.668 Hz
FIDRES 0.635783 Hz
AQ 0.7864320 sec
RG 256
DW 12.000 use
DE 20.00 use
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 176.0377702 MHz
NUC1 13C
P1 14.00 use
PLW1 95.00000000 W
======== CHANNEL f2 ========
SFO2 700.0228001 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 65.00 use
PLW2 6.90000010 W
PLW12 0.10452000 W
PLW13 0.04416000 W
F2 - Processing parameters
SI 32768
SF 176.0201690 MHz
WDW no
SSB 0
LB 0 Hz
GB 0
PC 1.40
150.765
142.895
139.276
129.127
128.577
128.339
126.532
114.534
113.334
110.367
102.131
77.241
77.059
76.877
71.152
40.701
39.448
Ph
O
O
NMe2
1e'
S24
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm



1f



Current Data Parameters

NAME	dd-4-84spot1
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

Date_	20160406
Time	8.44
INSTRUM	drx400
PROBHD	5 mm Multinu
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	8278.146 Hz
FIDRES	0.126314 Hz
AQ	3.9584243 sec
RG	228.1
DW	60.400 usec
DE	6.00 usec
TE	300.0 K
D1	1.00000000 sec

========== CHANNEL f1 ========

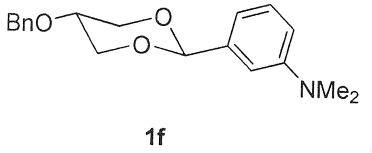
NUC1	1H
P1	6.50 usec
PL1	0.00 dB
SFO1	400.1324710 MHz

F2 - Processing parameters

SI	131072
SF	400.1300130 MHz
WDW	no
SSB	0
LB	0.00 Hz
GB	0
PC	1.00

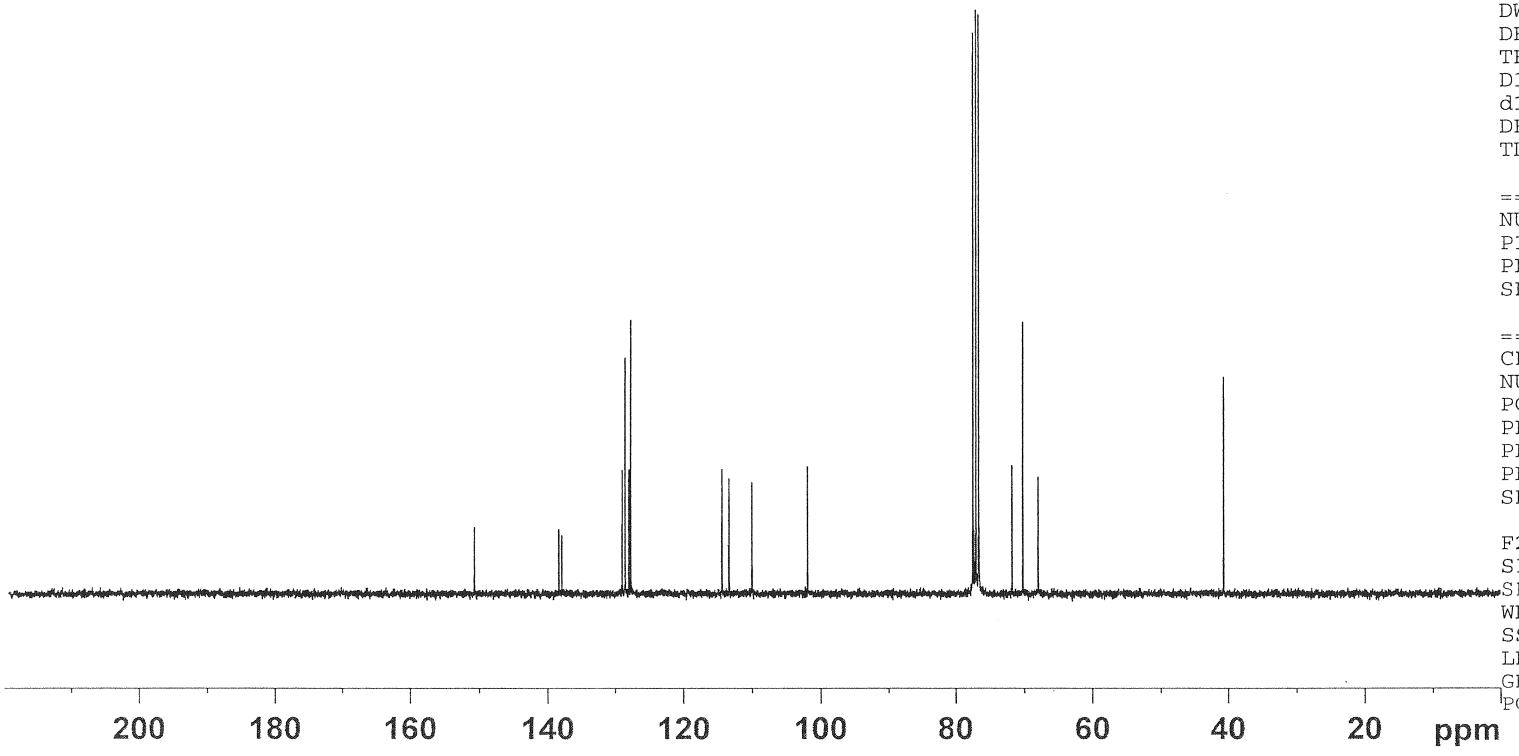
1D NMR plot parameters

CX	20.00 cm
F1P	8.000 ppm
F1	3201.04 Hz
F2P	0.000 ppm
F2	0.00 Hz
PPMCM	0.40000 ppm/cm
HZCM	160.05200 Hz/cm



1f

150.71
138.38
137.95
129.03
128.56
126.03
127.77
114.37
113.37
110.02
101.41
77.46
77.24
77.04
76.61
71.81
70.22
67.98
40.66



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```
Current Data Parameters
NAME      dd-4-70spot1-13c
EXPNO                    1
PROCNO                   1

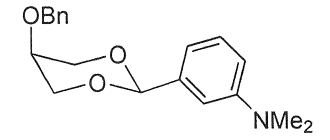
F2 - Acquisition Parameters
Date_             20160401
Time                 11.26
INSTRUM              spect
PROBHD   5 mm DUL 13C-1
PULPROG           zgpg30
TD                65536
SOLVENT           CDCl3
NS                 1024
DS                    4
SWH           17985.611 Hz
FIDRES         0.274439 Hz
AQ             1.8219508 sec
RG                32768
DW                27.800 usec
DE                 6.00 usec
TE                300.0 K
D1           2.00000000 sec
d11          0.03000000 sec
DELTA        1.89999998 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 13C
P1                  8.00 usec
PL1                -1.80 dB
SFO1         75.4124265 MHz

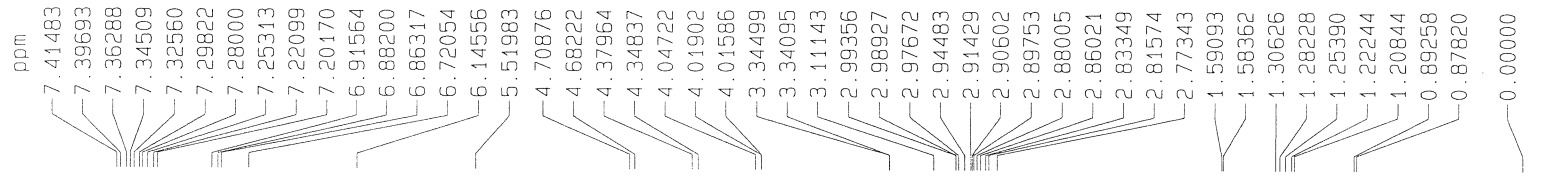
======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2              75.00 usec
PL2                -2.50 dB
PL12               11.48 dB
PL13               12.00 dB
SFO2        299.8811995 MHz

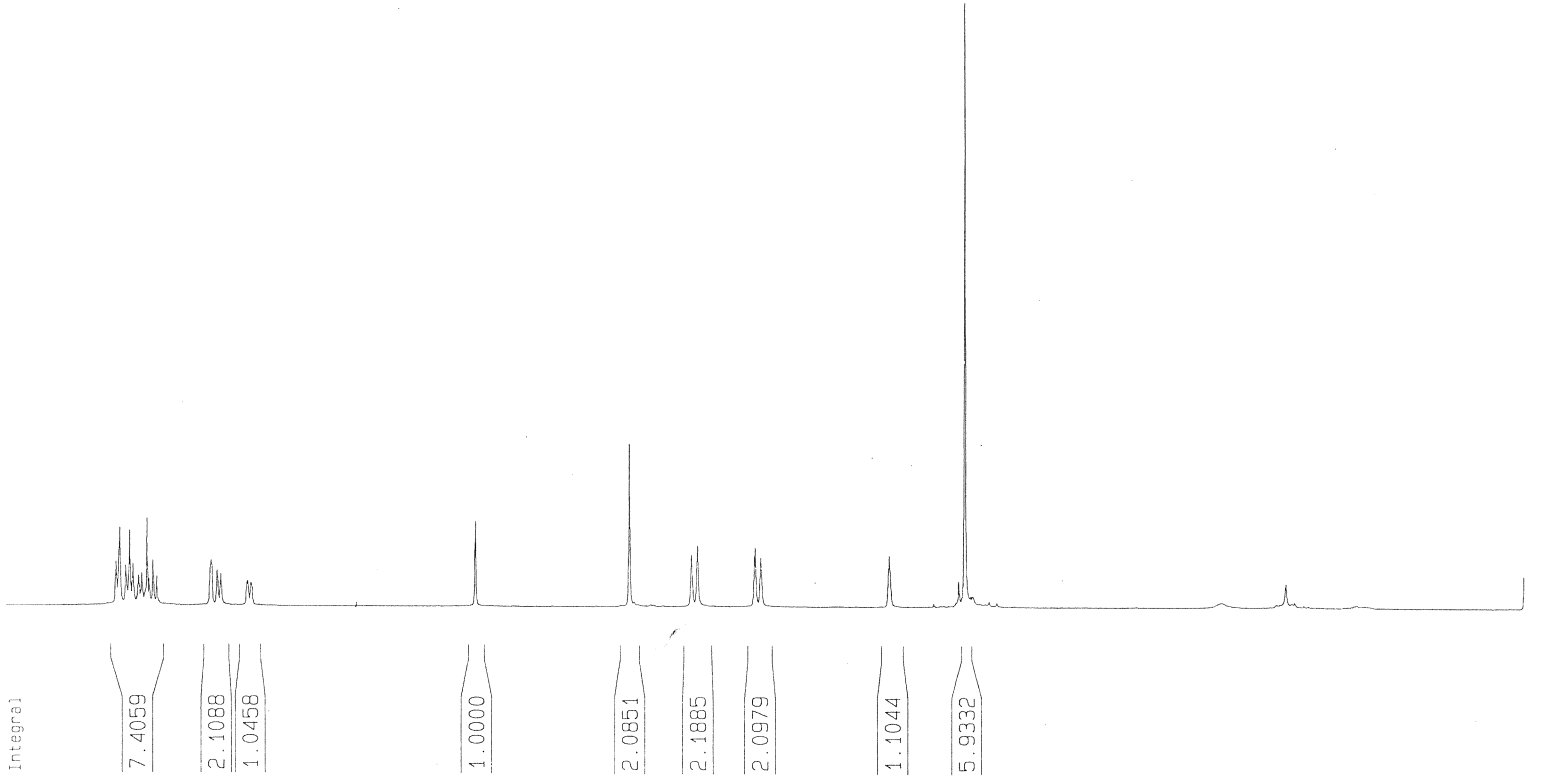
F2 - Processing parameters
SI                32768
SF          75.4048860 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```

1H



1f'





Current Data Parameters

NAME	dd-4-84spot2
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

Date_	20160405
Time	14.39
INSTRUM	drx400
PROBHD	5 mm Multinu
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	8278.146 Hz
FIDRES	0.126314 Hz
AQ	3.9584243 sec
RG	322.5
DW	60.400 usec
DE	6.00 usec
TE	300.0 K
D1	1.00000000 sec

========== CHANNEL f1 ========

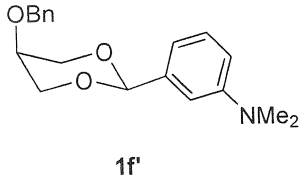
NUC1	1H
P1	6.50 usec
PL1	0.00 dB
SF01	400.1324710 MHz

F2 - Processing parameters

SI	131072
SF	400.1300118 MHz
WDW	no
SSB	0
LB	0.00 Hz
GB	0
PC	1.00

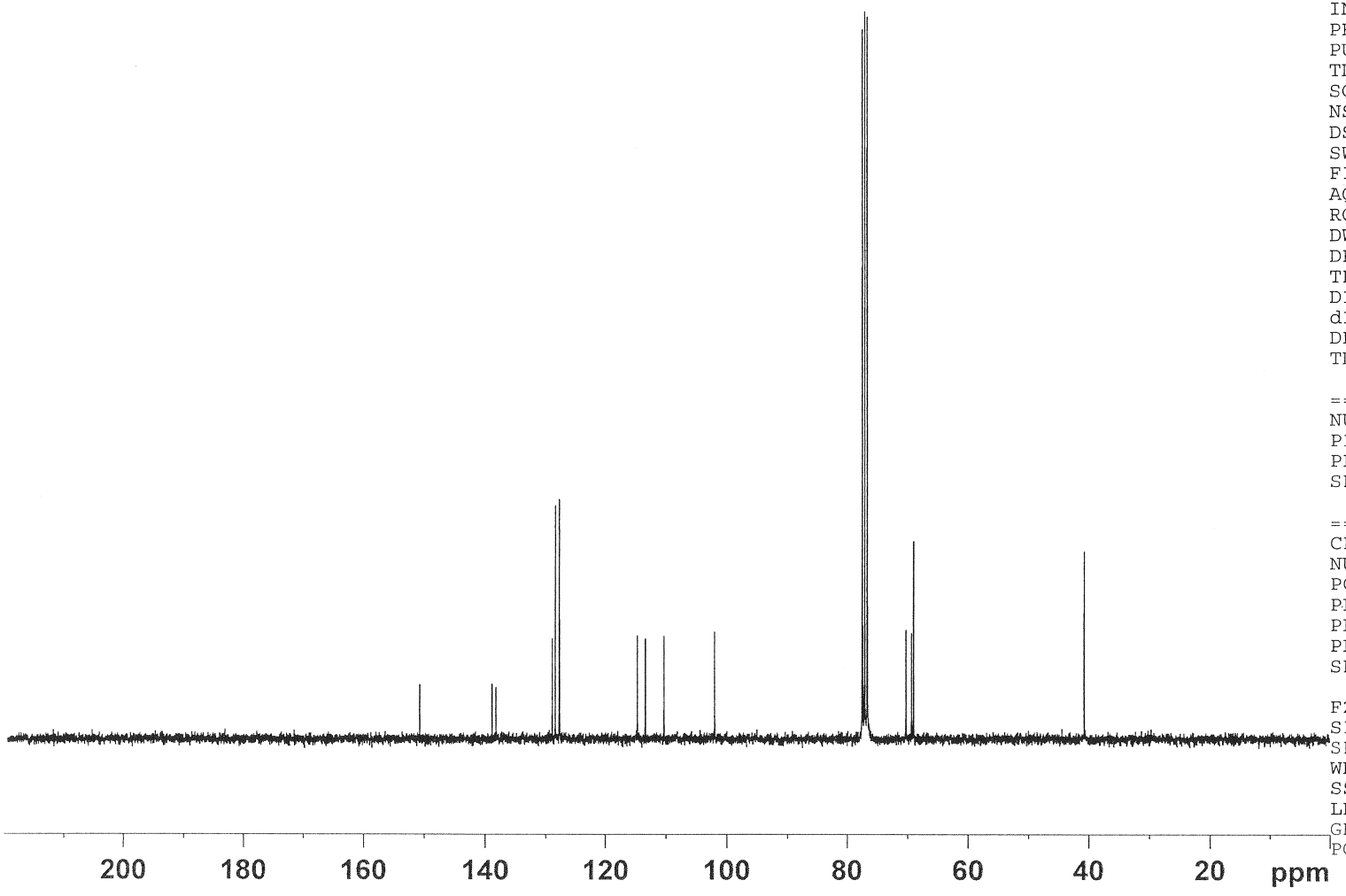
1D NMR plot parameters

CX	20.00 cm
F1P	8.000 ppm
F1	3201.04 Hz
F2P	0.000 ppm
F2	0.00 Hz
PPMCM	0.40000 ppm/cm
HZCM	160.05200 Hz/cm



1f'

150.72
138.86
138.24
128.60
128.42
127.73
127.65
114.64
113.36
110.31
101.95
77.45
77.23
77.03
76.60
70.33
69.33
68.99
40.73



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```
Current Data Parameters
NAME      dd-4-84-spot2-13C
EXPNO                    1
PROCNO                   1

F2 - Acquisition Parameters
Date_             20160405
Time                 14.57
INSTRUM              spect
PROBHD    5 mm DUL 13C-1
PULPROG            zgpg30
TD                  65536
SOLVENT             CDCl3
NS                   1024
DS                      4
SWH           17985.611 Hz
FIDRES         0.274439 Hz
AQ             1.8219508 sec
RG                  32768
DW                27.800 usec
DE                 6.00 usec
TE                300.0 K
D1           2.00000000 sec
d11          0.03000000 sec
DELTA        1.89999998 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   13C
P1                  8.00 usec
PL1                -1.80 dB
SFO1         75.4124265 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                   1H
PCPD2              75.00 usec
PL2                -2.50 dB
PL12               11.48 dB
PL13               12.00 dB
SFO2        299.8811995 MHz

F2 - Processing parameters
SI                  32768
SF          75.4048860 MHz
WDW                    EM
SSB                     0
LB                 1.00 Hz
GB                      0
PC                   1.40
```

1g

7.087 7.075 7.064 6.827 6.825 6.743 6.732 6.665 6.662 6.654 6.650

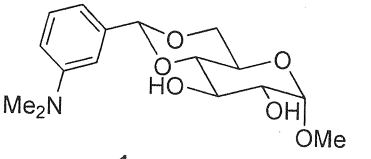
5.403 4.788 4.621 4.615 4.108 4.104 4.100 4.097 3.725 3.712 3.698 3.637 3.634 3.631 3.626 3.622 3.416 3.410 3.402 3.397 3.329 2.817

```
Current Data Parameters
NAME          dx-I-57-1H
EXPNO                  3
PROCNO                 1

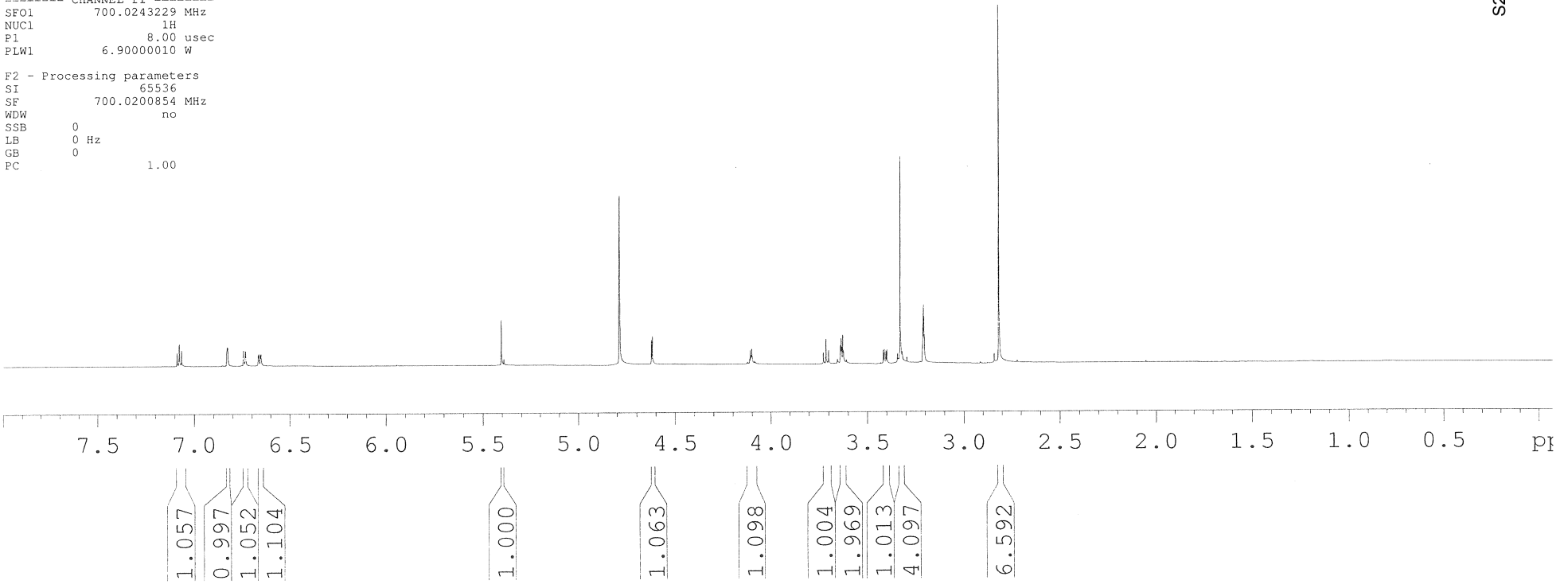
F2 - Acquisition Parameters
Date_           20160719
Time               17.17
INSTRUM            spect
PROBHD   5 mm CPTCI 1H-
PULPROG             zg30
TD                 65536
SOLVENT             MeOD
NS                   128
DS                     2
SWH           14097.744 Hz
FIDRES         0.215115 Hz
AQ             2.3243434 sec
RG                  20.2
DW                35.467 usec
DE                10.00 usec
TE                298.0 K
D1            1.00000000 sec
TD0                    1

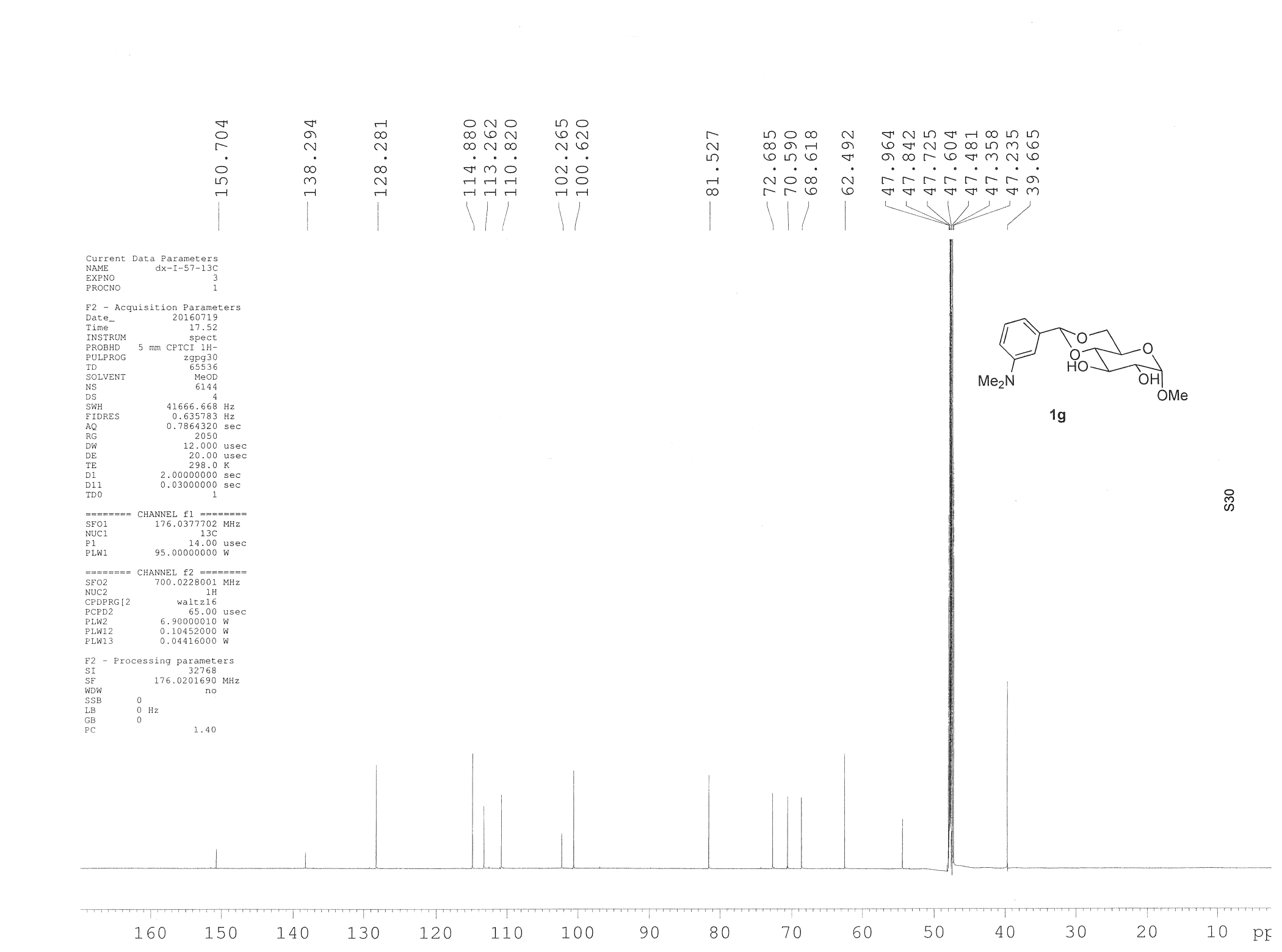
======== CHANNEL f1 ========
SFO1        700.0243229 MHz
NUC1                   1H
P1                  8.00 usec
PLW1         6.90000010 W

F2 - Processing parameters
SI                 65536
SF          700.0200854 MHz
WDW                   no
SSB       0
LB        0 Hz
GB        0
PC                  1.00
```

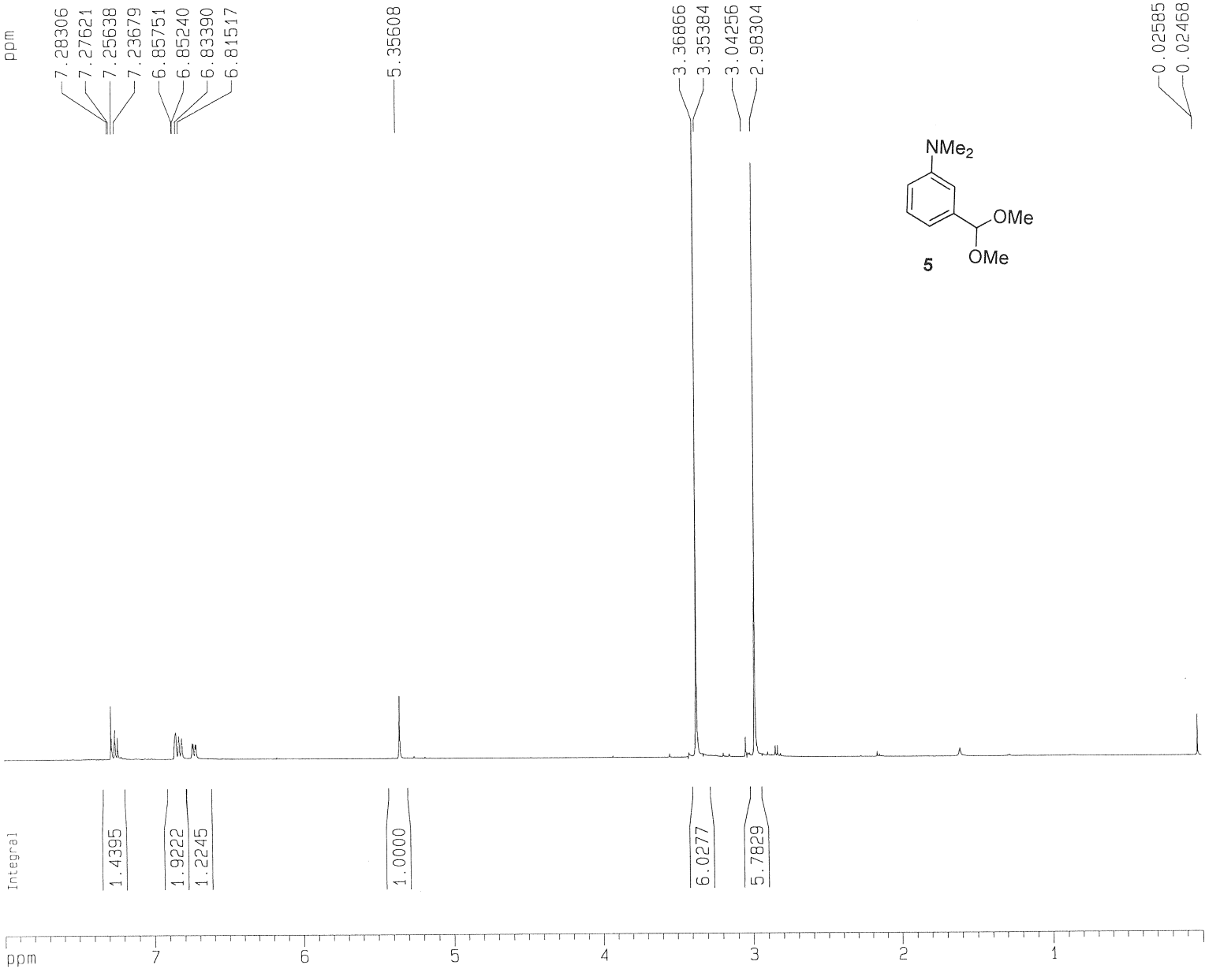


1g




150.704
138.294
128.281
114.880
113.262
110.820
102.265
100.620
81.527
72.685
70.590
68.618
62.492
47.964
47.842
47.725
47.604
47.481
47.358
47.235
39.665
Current Data Parameters
NAME dx-I-57-13C
EXPNO 3
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160719
Time 17.52
INSTRUM spect
PROBHD 5 mm CPTCI 1H-
PULPROG zgpg30
TD 65536
SOLVENT MeOD
NS 6144
DS 4
SWH 41666.668 Hz
FIDRES 0.635783 Hz
AQ 0.7864320 sec
RG 2050
DW 12.000 usec
DE 20.00 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 176.0377702 MHz
NUC1 13C
P1 14.00 usec
PLW1 95.00000000 W
======== CHANNEL f2 ========
SFO2 700.0228001 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 65.00 usec
PLW2 6.90000010 W
PLW12 0.10452000 W
PLW13 0.04416000 W
F2 - Processing parameters
SI 32768
SF 176.0201690 MHz
WDW no
SSB 0
LB 0 Hz
GB 0
PC 1.40
Me2N
HO
OH
OMe
1g
S30
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

1H



Current Data Parameters

NAME	dd-4-65pure
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

Date_	20160310
Time	8.35
INSTRUM	drx400
PROBHD	5 mm Multinu
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	8278.146 Hz
FIDRES	0.126314 Hz
AQ	3.9584243 sec
RG	362
DW	60.400 usec
DE	6.00 usec
TE	300.0 K
D1	1.00000000 sec

========== CHANNEL f1 ========

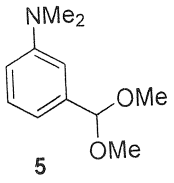
NUC1	1H
P1	6.50 usec
PL1	0.00 dB
SF01	400.1324710 MHz

F2 - Processing parameters

SI	131072
SF	400.1300000 MHz
WDW	no
SSB	0
LB	0.00 Hz
GB	0
PC	1.00

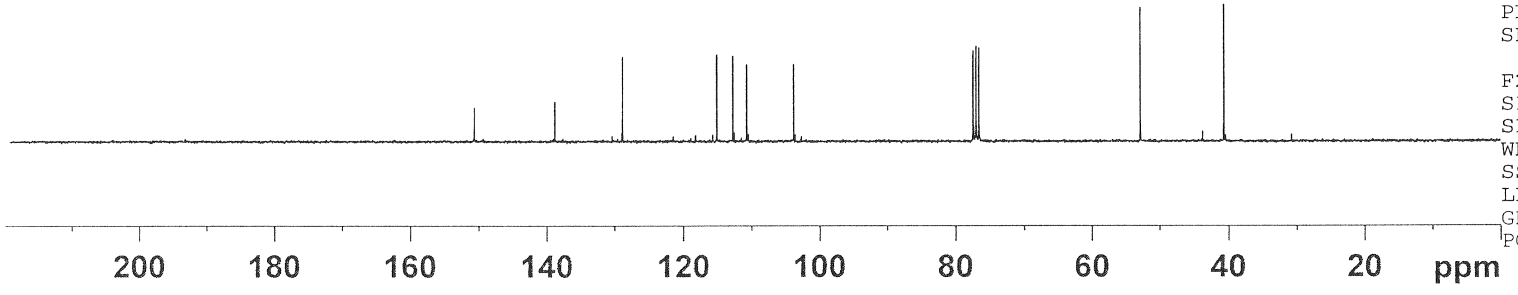
1D NMR plot parameters

CX	20.00 cm
F1P	8.000 ppm
F1	3201.04 Hz
F2P	0.000 ppm
F2	0.00 Hz
PPMCM	0.40000 ppm/cm
HZCM	160.05200 Hz/cm



5

150.86
139.86
129.80
115.09
112.72
110.70
102.82
77.50
77.08
76.65
50.66
40.66



BRUKER

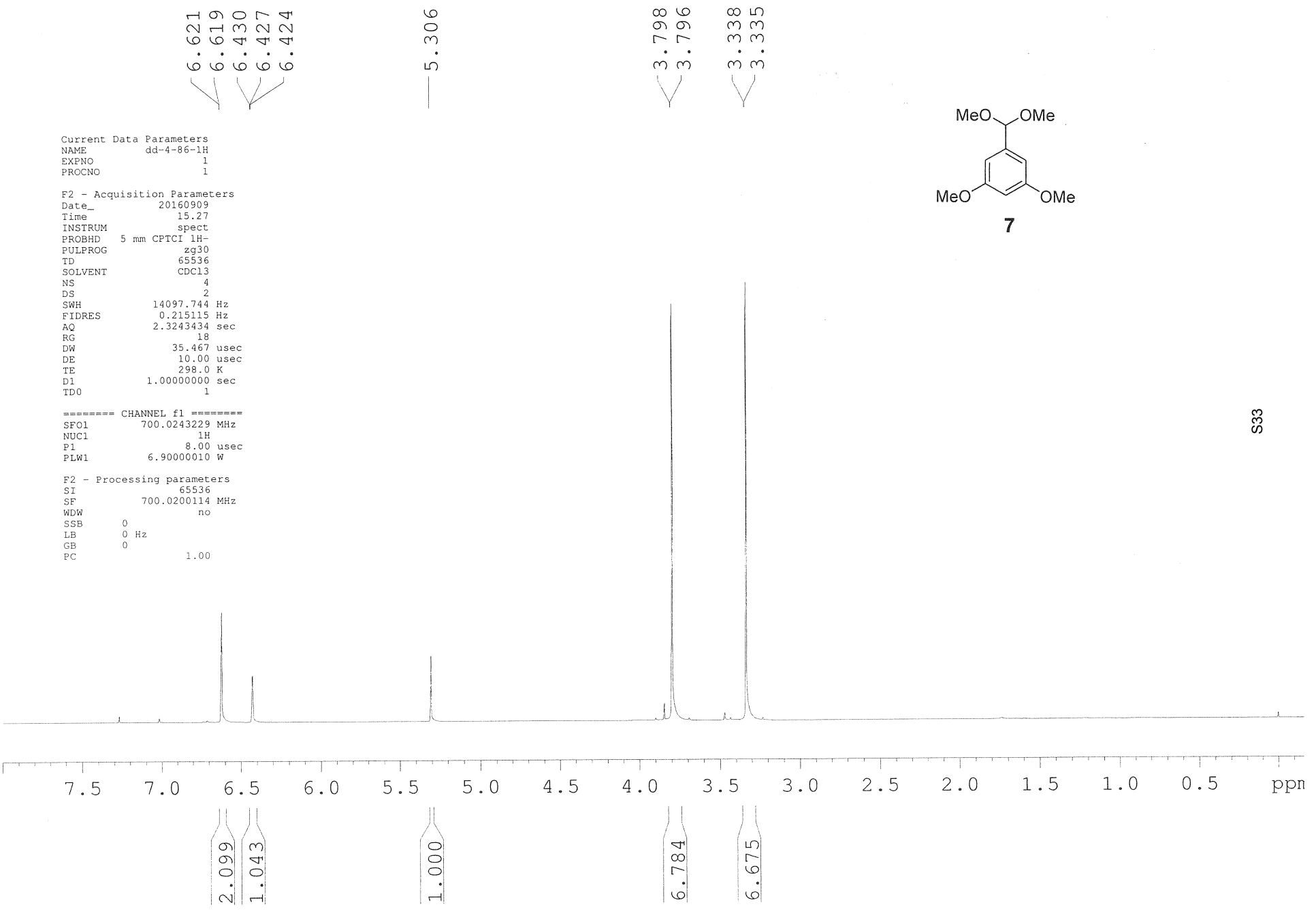
Current Data Parameters
NAME dd-4-67c
EXPNO 1
PROCNO 1

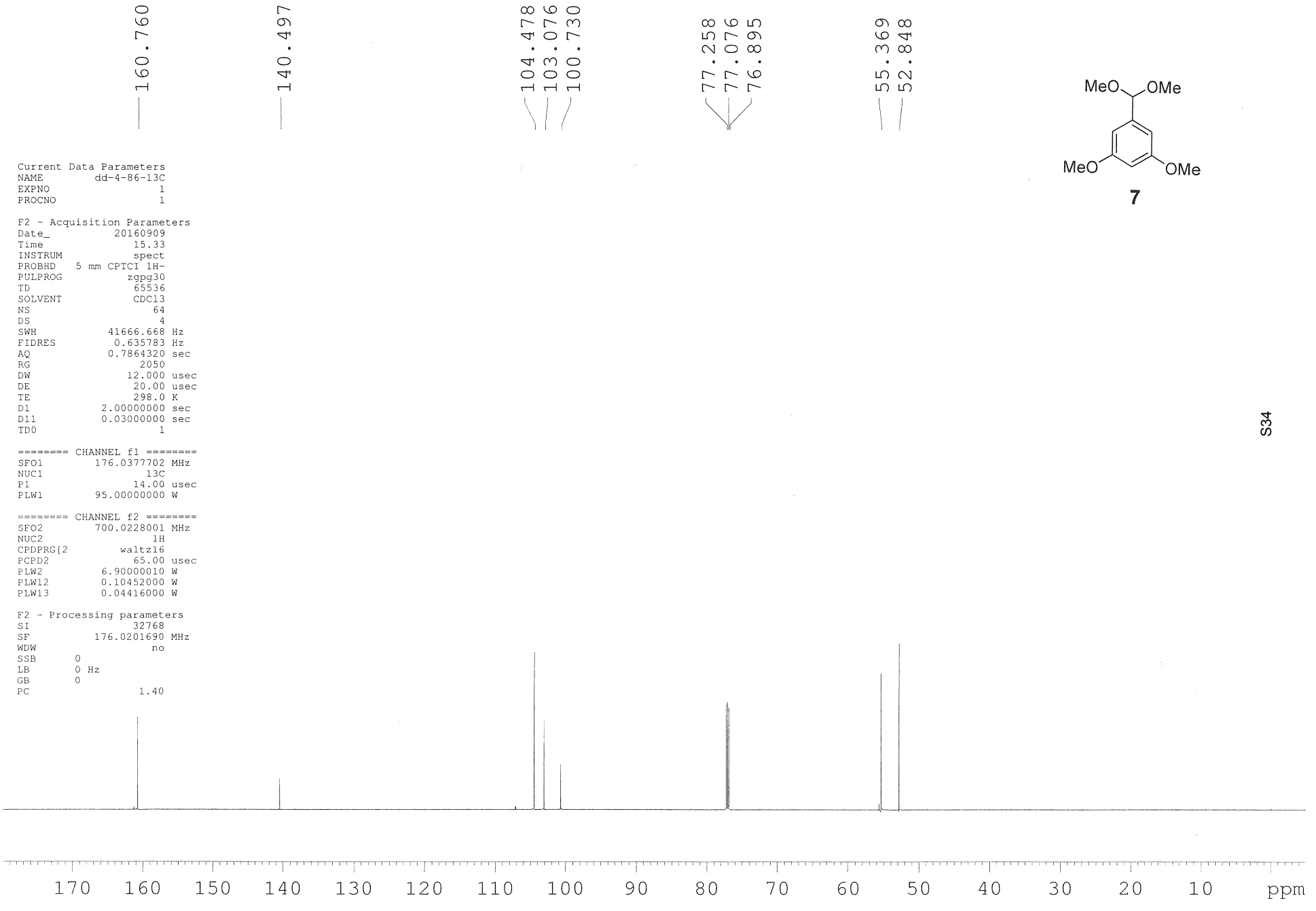
F2 - Acquisition Parameters
Date_ 20160427
Time 10.51
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 32768
DW 27.800 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1

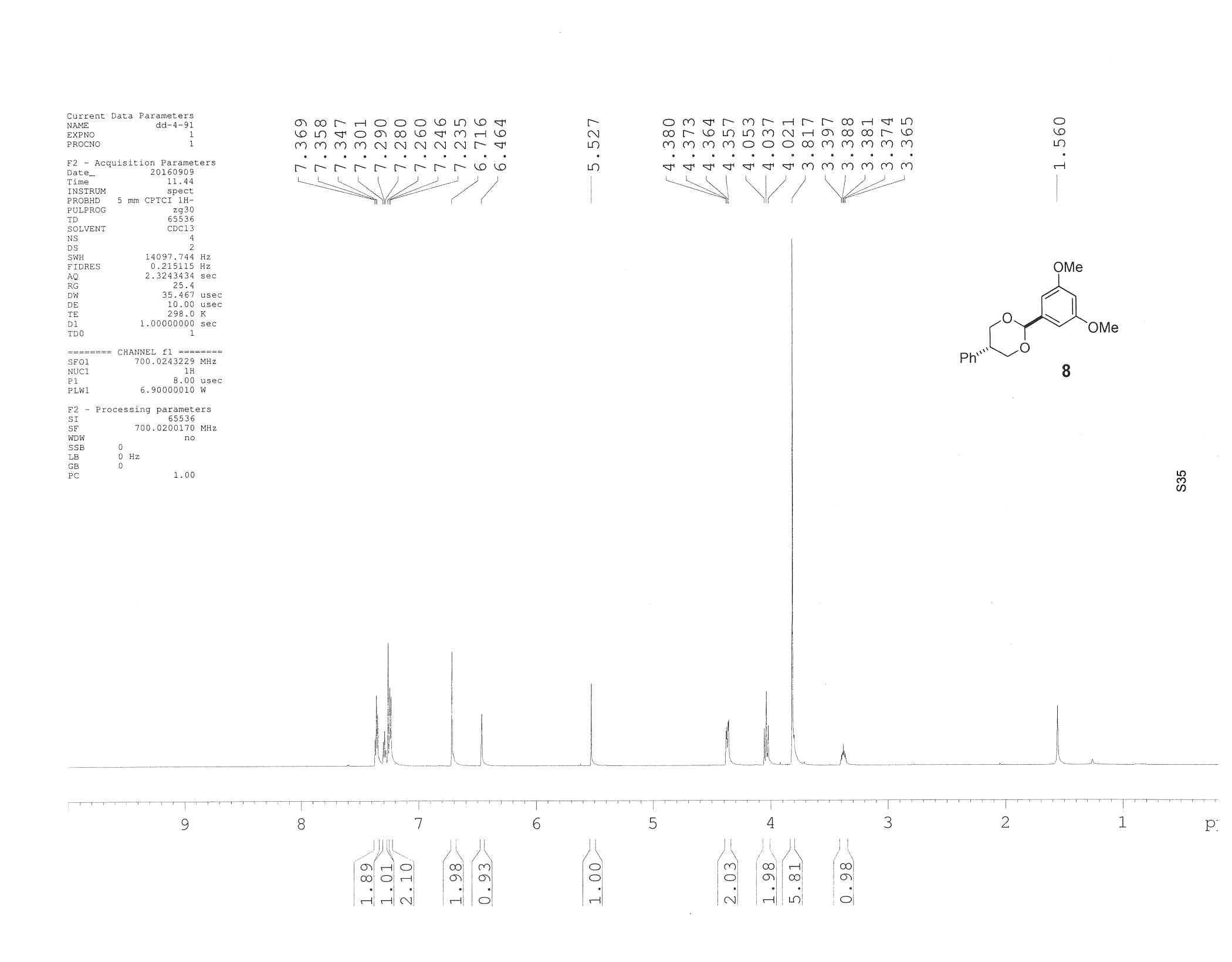
======== CHANNEL f1 ========
NUC1 13C
P1 8.00 usec
PL1 -1.80 dB
SFO1 75.4124265 MHz

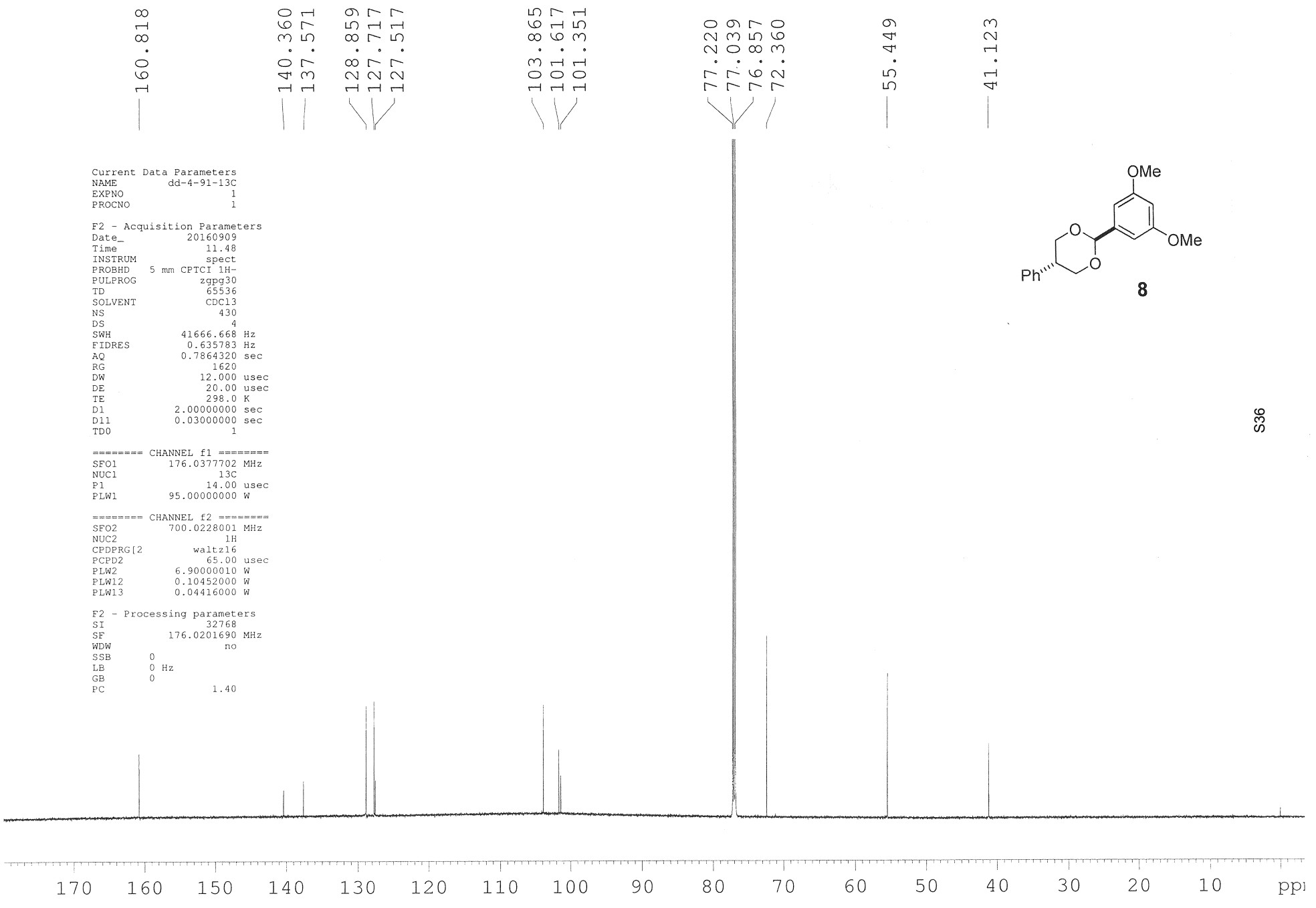
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 75.00 usec
PL2 -2.50 dB
PL12 11.48 dB
PL13 12.00 dB
SFO2 299.8811995 MHz

F2 - Processing parameters
SI 32768
SF 75.4048860 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

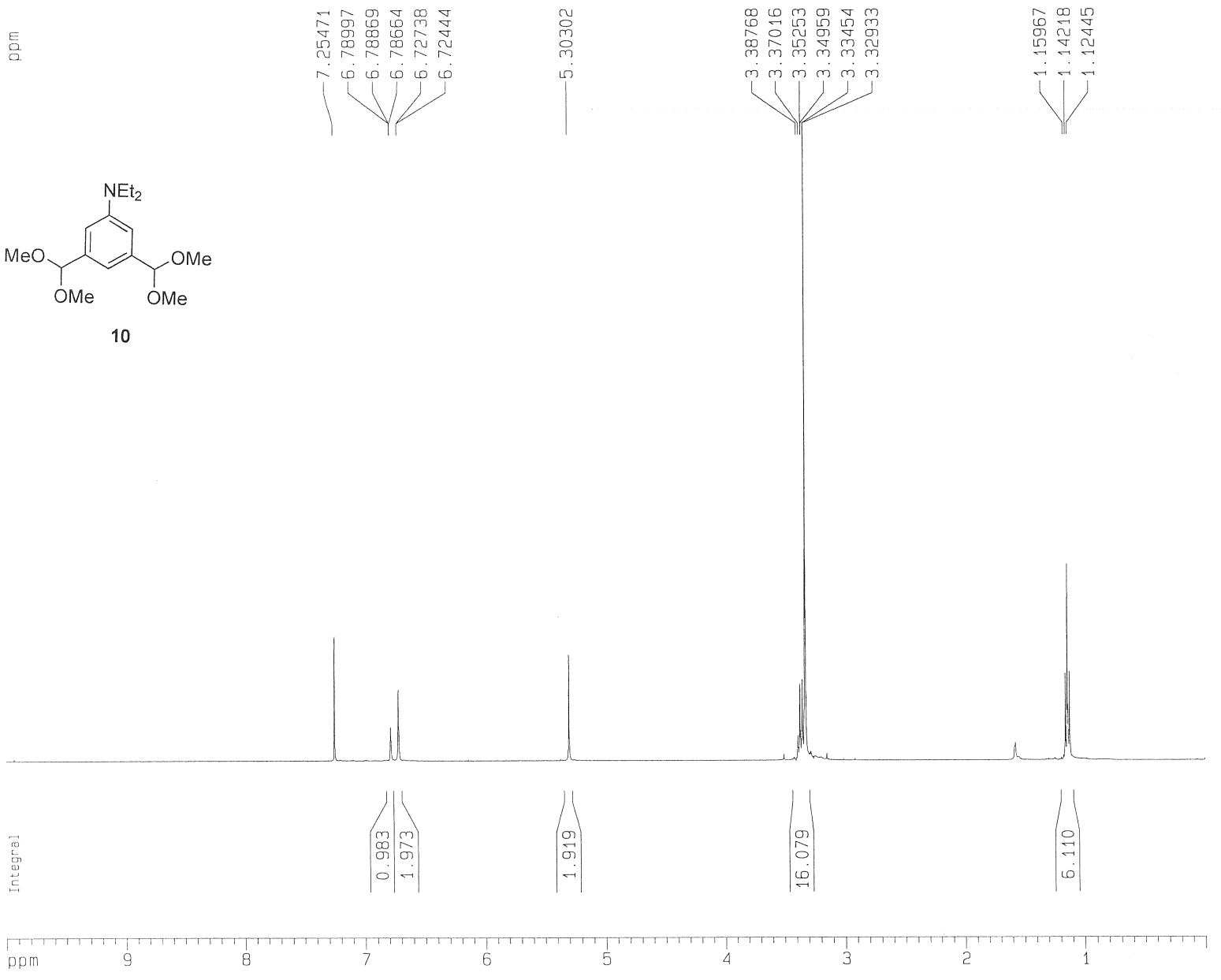

6.621
6.619
6.430
6.427
6.424
5.306
3.798
3.796
3.338
3.335
Current Data Parameters
NAME dd-4-86-1H
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160909
Time 15.27
INSTRUM spect
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 4
DS 2
SWH 14097.744 Hz
FIDRES 0.215115 Hz
AQ 2.3243434 sec
RG 18
DW 35.467 usec
DE 10.00 usec
TE 298.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 700.0243229 MHz
NUC1 1H
P1 8.00 usec
PLW1 6.90000010 W
F2 - Processing parameters
SI 65536
SF 700.0200114 MHz
WDW no
SSB 0
LB 0 Hz
GB 0
PC 1.00
MeO
OMe
MeO
OMe
7
7.5
7.0
6.5
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
2.099
1.043
1.000
6.784
6.675


160.760
140.497
104.478
103.076
100.730
77.258
77.076
76.895
55.369
52.848
MeO
OMe
MeO
OMe
7
Current Data Parameters
NAME dd-4-86-13C
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160909
Time 15.33
INSTRUM spect
PROBHD 5 mm CPTCI 1H-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 64
DS 4
SWH 41666.668 Hz
FIDRES 0.635783 Hz
AQ 0.7864320 sec
RG 2050
DW 12.000 usec
DE 20.00 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 176.0377702 MHz
NUC1 13C
P1 14.00 usec
PLW1 95.00000000 W
======== CHANNEL f2 ========
SFO2 700.0228001 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 65.00 usec
PLW2 6.90000010 W
PLW12 0.10452000 W
PLW13 0.04416000 W
F2 - Processing parameters
SI 32768
SF 176.0201690 MHz
WDW no
SSB 0
LB 0 Hz
GB 0
PC 1.40
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm


Current Data Parameters
NAME dd-4-91
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160909
Time 11.44
INSTRUM spect
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 4
DS 2
SWH 14097.744 Hz
FIDRES 0.215115 Hz
AQ 2.3243434 sec
RG 25.4
DW 35.467 usec
DE 10.00 usec
TE 298.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 700.0243229 MHz
NUC1 1H
P1 8.00 usec
PLW1 6.90000010 W
F2 - Processing parameters
SI 65536
SF 700.0200170 MHz
WDW no
SSB 0
LB 0 Hz
GB 0
PC 1.00
7.369
7.358
7.347
7.301
7.290
7.280
7.260
7.246
7.235
6.716
6.464
5.527
4.380
4.373
4.364
4.357
4.053
4.037
4.021
3.817
3.397
3.388
3.381
3.374
3.365
1.560
OMe
OMe
O
O
O
Ph
8
9
8
7
6
5
4
3
2
1
1.89
1.01
2.10
1.98
0.93
1.00
2.03
1.98
5.81
0.98


160.818
140.360
137.571
128.859
127.717
127.517
103.865
101.617
101.351
77.220
77.039
76.857
72.360
55.449
41.123
Current Data Parameters
NAME dd-4-91-13C
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160909
Time 11.48
INSTRUM spect
PROBHD 5 mm CPTCI 1H-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 430
DS 4
SWH 41666.668 Hz
FIDRES 0.635783 Hz
AQ 0.7864320 sec
RG 1620
DW 12.000 usec
DE 20.00 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 176.0377702 MHz
NUC1 13C
P1 14.00 usec
PLW1 95.00000000 W
======== CHANNEL f2 ========
SFO2 700.0228001 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 65.00 usec
PLW2 6.90000010 W
PLW12 0.10452000 W
PLW13 0.04416000 W
F2 - Processing parameters
SI 32768
SF 176.0201690 MHz
WDW no
SSB 0
LB 0 Hz
GB 0
PC 1.40
OMe
OMe
O
O
Ph
8
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

1H



Current Data Parameters

NAME	dx-I-61
EXPNO	2
PROCNO	1

F2 - Acquisition Parameters

Date_	20160627
Time	9.37
INSTRUM	drx400
PROBHD	5 mm Multinu
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	8278.146 Hz
FIDRES	0.126314 Hz
AQ	3.9584243 sec
RG	362
DW	60.400 usec
DE	6.00 usec
TE	300.0 K
D1	1.00000000 sec

========== CHANNEL f1 ========

NUC1	1H
P1	6.50 usec
PL1	0.00 dB
SFO1	400.1324710 MHz

F2 - Processing parameters

SI	131072
SF	400.1300114 MHz
WDW	no
SSB	0
LB	0.00 Hz
GB	0
PC	1.00

1D NMR plot parameters

CX	20.00 cm
F1P	10.000 ppm
F1	4001.30 Hz
F2P	0.000 ppm
F2	0.00 Hz
PPMCM	0.50000 ppm/cm
HZCM	200.06500 Hz/cm

```
Current Data Parameters
NAME         DX-I-61-C13
EXPNO                  1
PROCNO                 1

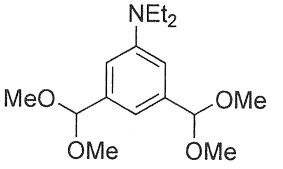
F2 - Acquisition Parameters
Date_           20160627
Time               13.08
INSTRUM            spect
PROBHD   5 mm CPTCI 1H-
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 1635
DS                    4
SWH           41666.668 Hz
FIDRES         0.635783 Hz
AQ             0.7864320 sec
RG                  256
DW                12.000 usec
DE                20.00 usec
TE                298.0 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1         176.0377702 MHz
NUC1                13C
P1                14.00 usec
PLW1         95.00000000 W

======== CHANNEL f2 ========
SFO2         700.0228001 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             65.00 usec
PLW2          6.90000010 W
PLW12         0.10452000 W
PLW13         0.04416000 W

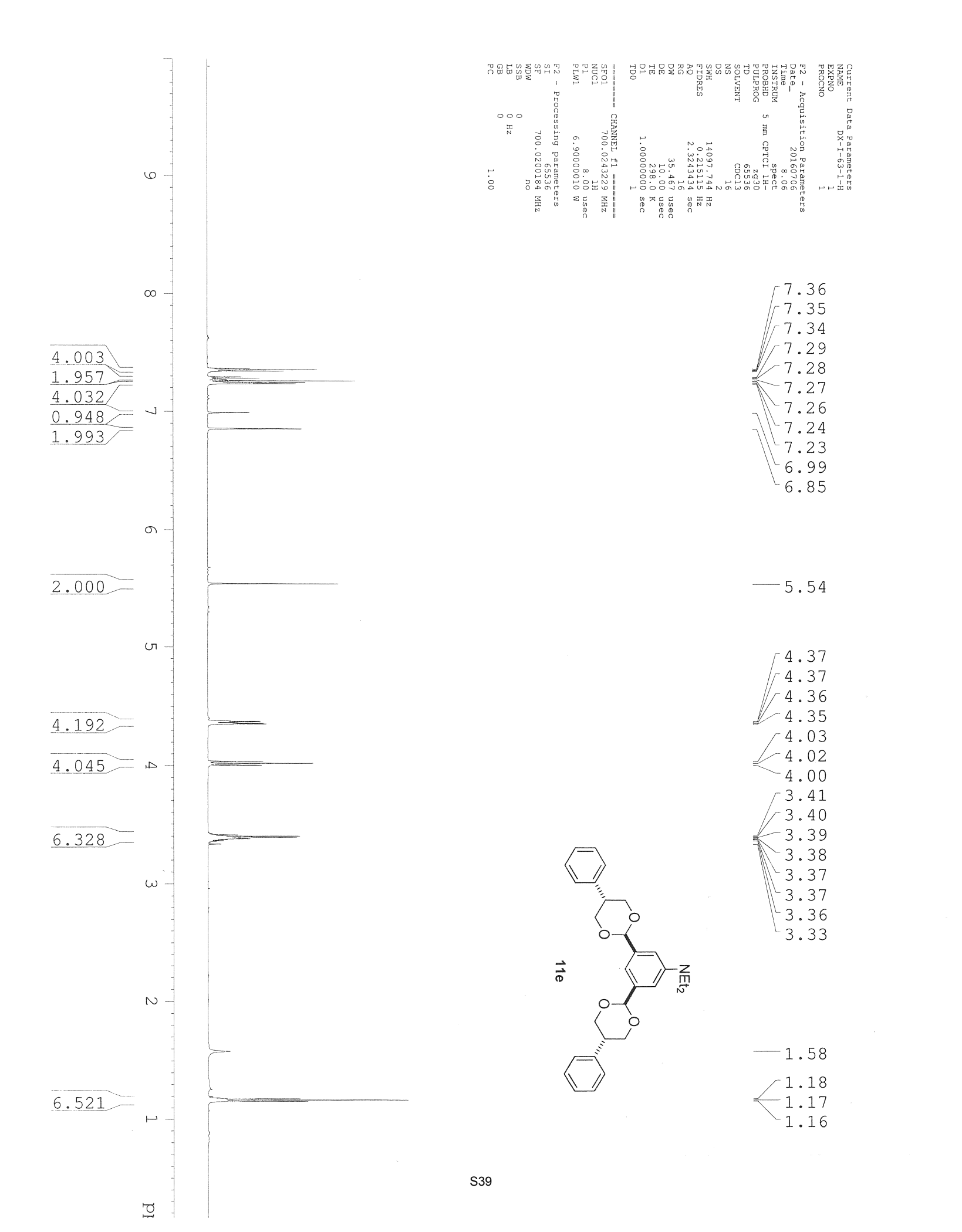
F2 - Processing parameters
SI                 32768
SF           176.0201690 MHz
WDW                   no
SSB       0
LB        0 Hz
GB        0
PC                  1.40
```

147.927
138.911
112.601
110.028
103.824
77.202
77.021
76.839
52.887
44.325
12.585
0.001



10

200 180 160 140 120 100 80 60 40 20 0 p


Current Data Parameters
NAME DX-I-63-1-H
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160706
Time 8.06
INSTRUM spect
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 14097.744 Hz
FIDRES 0.215115 Hz
AQ 2.3243434 sec
RG 16
DW 35.467 usec
DE 10.00 usec
TE 298.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 700.0243229 MHz
NUC1 1H
P1 8.00 usec
PLW1 6.90000010 W
F2 - Processing parameters
SI 65536
SF 700.0200184 MHz
WDW no
SSB 0
LB 0 Hz
GB 0
PC 1.00
7.36
7.35
7.34
7.29
7.28
7.27
7.26
7.24
7.23
6.99
6.85
5.54
4.37
4.37
4.36
4.35
4.03
4.02
4.00
3.41
3.40
3.39
3.38
3.37
3.37
3.36
3.33
1.58
1.18
1.17
1.16
4.003
1.957
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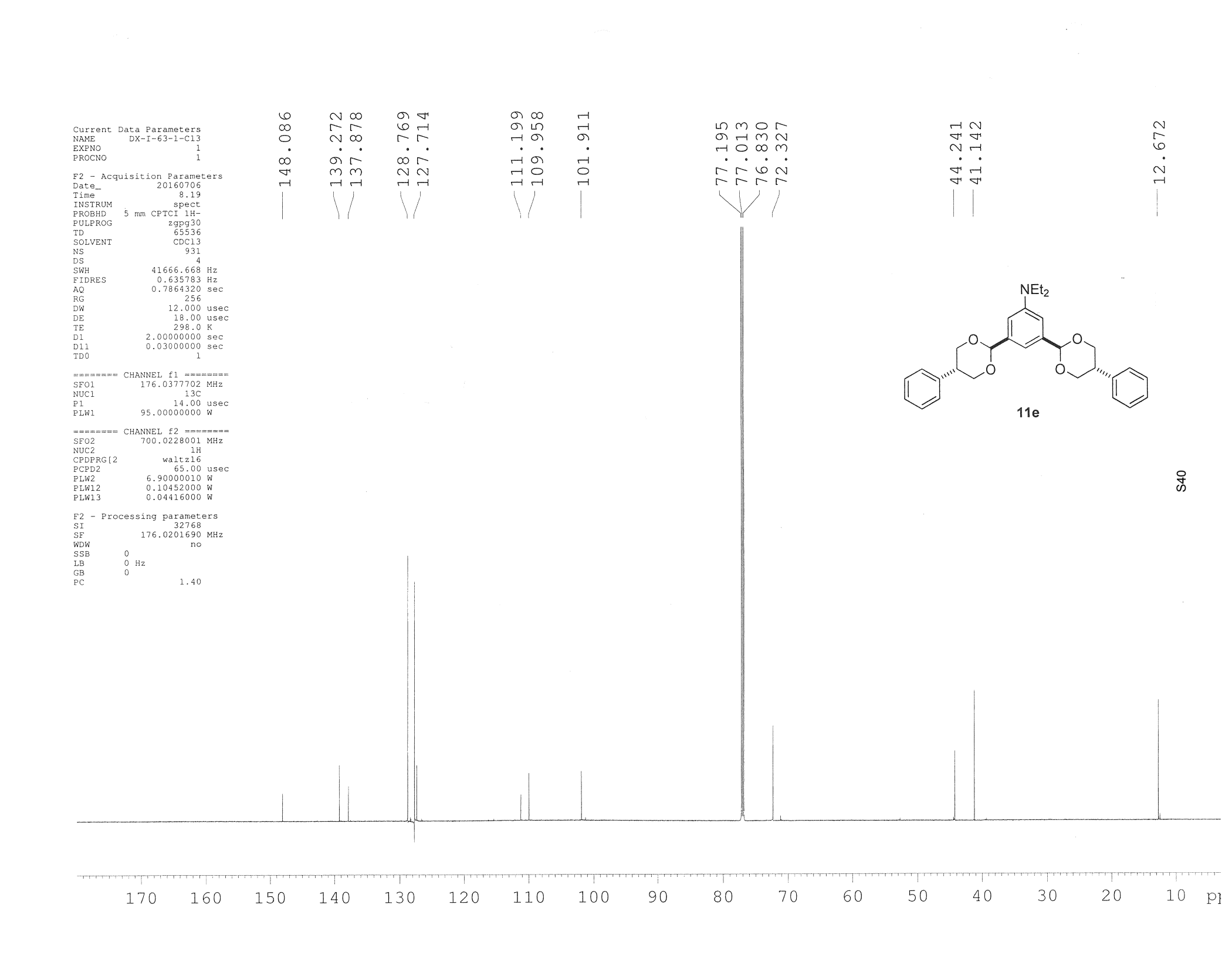
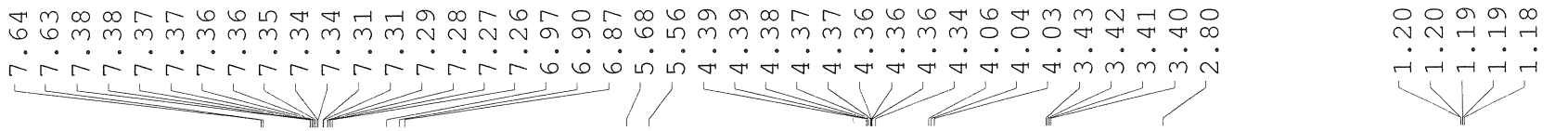
0.948
1.993
2.000
4.192
4.045
6.328
6.521
11e
NEt2


Current Data Parameters
NAME DX-I-63-1-C13
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160706
Time 8.19
INSTRUM spect
PROBHD 5 mm CPTCI 1H-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 931
DS 4
SWH 41666.668 Hz
FIDRES 0.635783 Hz
AQ 0.7864320 sec
RG 256
DW 12.000 usec
DE 18.00 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 176.0377702 MHz
NUC1 13C
P1 14.00 usec
PLW1 95.00000000 W
======== CHANNEL f2 ========
SFO2 700.0228001 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 65.00 usec
PLW2 6.90000010 W
PLW12 0.10452000 W
PLW13 0.04416000 W
F2 - Processing parameters
SI 32768
SF 176.0201690 MHz
WDW no
SSB 0
LB 0 Hz
GB 0
PC 1.40
148.086
139.272
137.878
128.769
127.714
111.199
109.958
101.911
77.195
77.013
76.830
72.327
44.241
41.142
12.672
NEt2
O
O
O
O
11e
S40
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

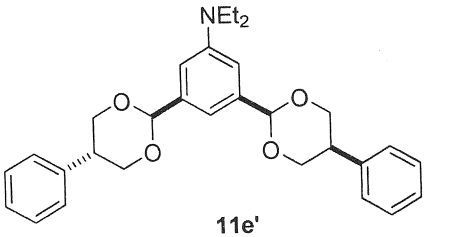



```
Current Data Parameters
NAME          dx-I-63-2H
EXPNO                  1
PROCNO                 1

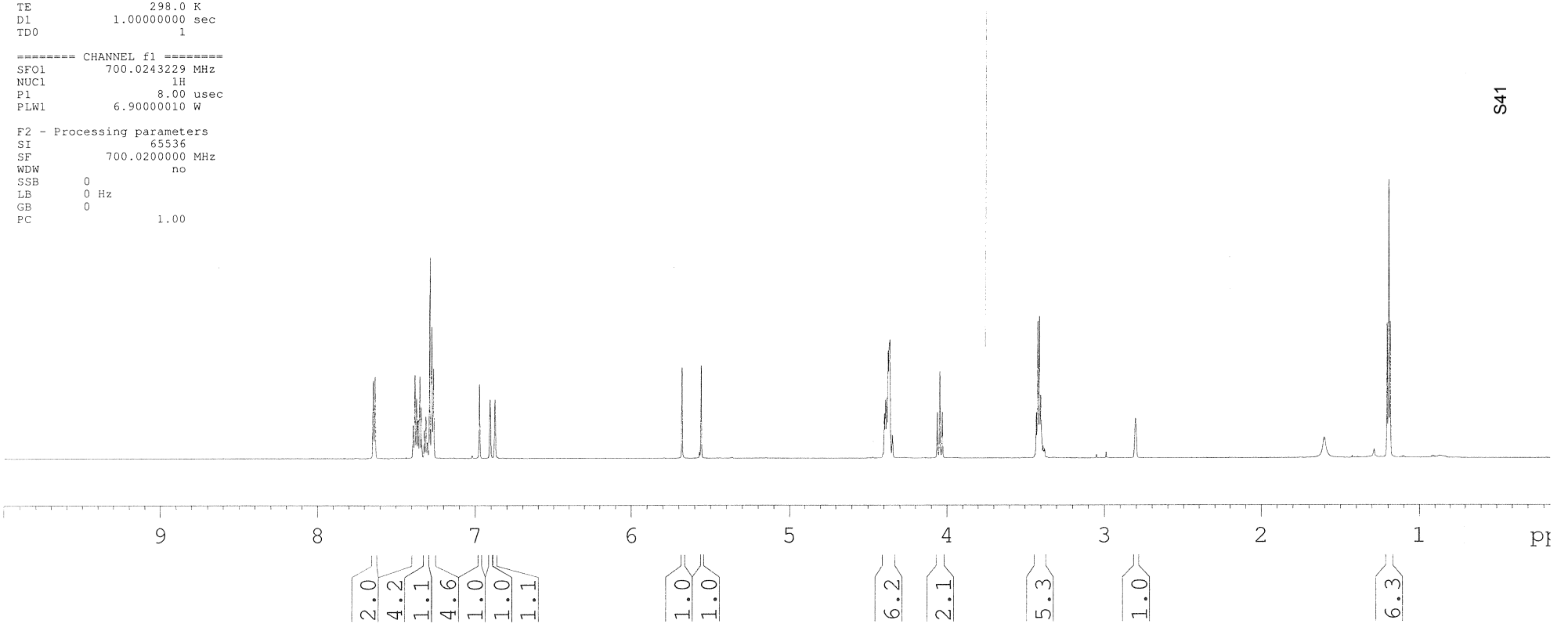
F2 - Acquisition Parameters
Date_           20160712
Time              14.24
INSTRUM           spect
PROBHD   5 mm CPTCI 1H-
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    2
SWH           14097.744 Hz
FIDRES         0.215115 Hz
AQ            2.3243434 sec
RG                   32
DW               35.467 usec
DE               10.00 usec
TE               298.0 K
D1           1.00000000 sec
TD0                   1

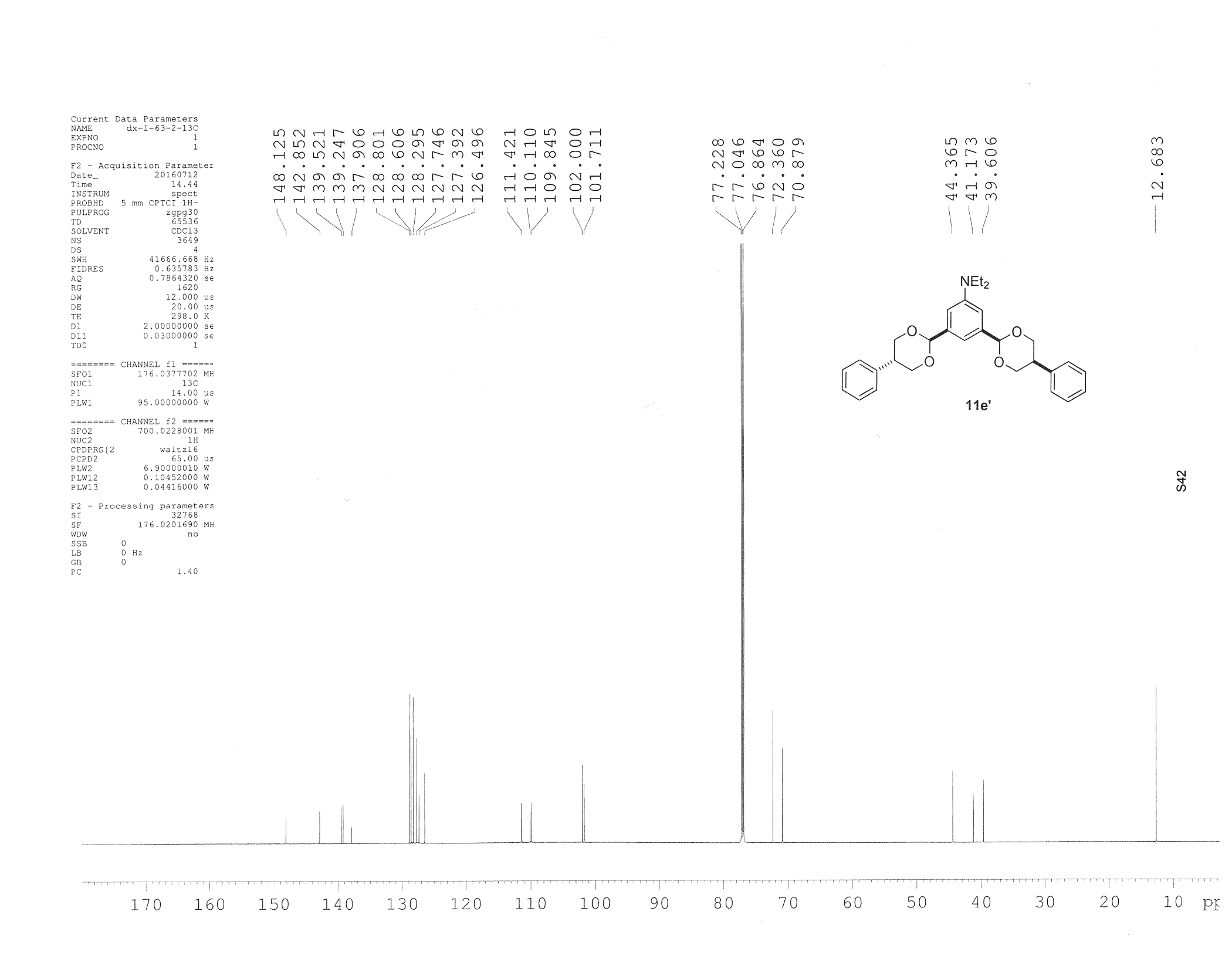
======== CHANNEL f1 ========
SFO1        700.0243229 MHz
NUC1                 1H
P1                8.00 usec
PLW1         6.90000010 W

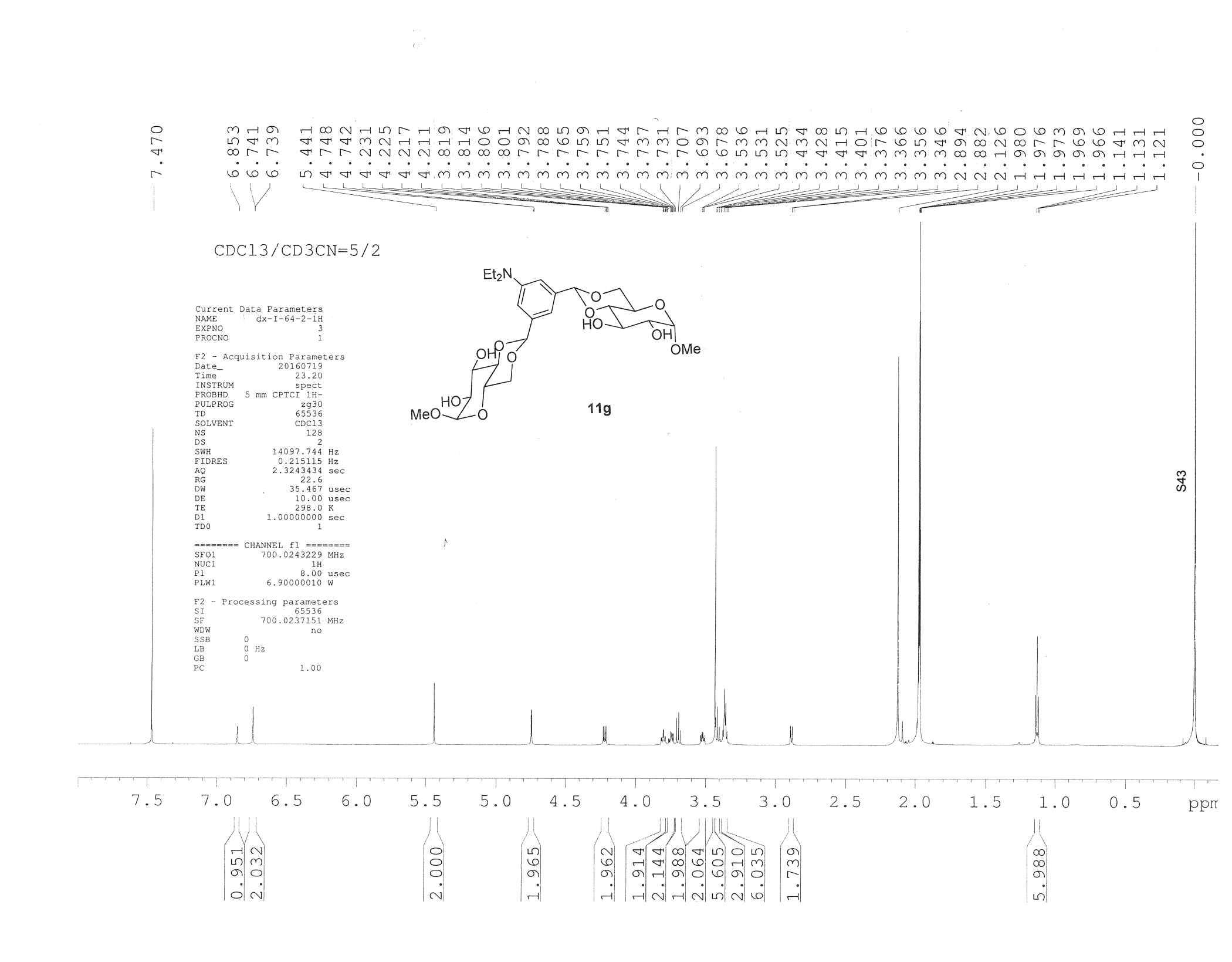
F2 - Processing parameters
SI                65536
SF           700.0200000 MHz
WDW                  no
SSB       0
LB        0 Hz
GB        0
PC                1.00
```

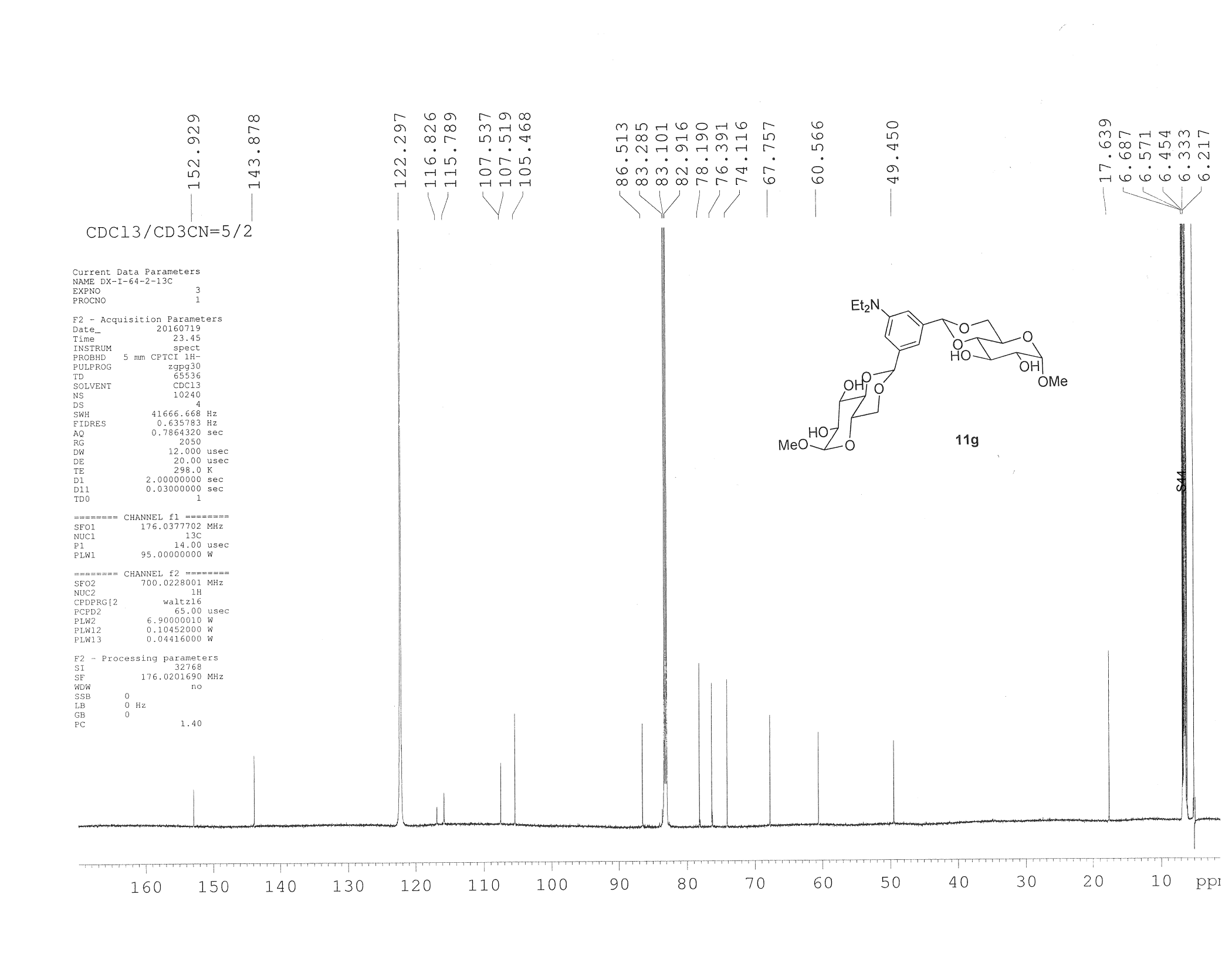


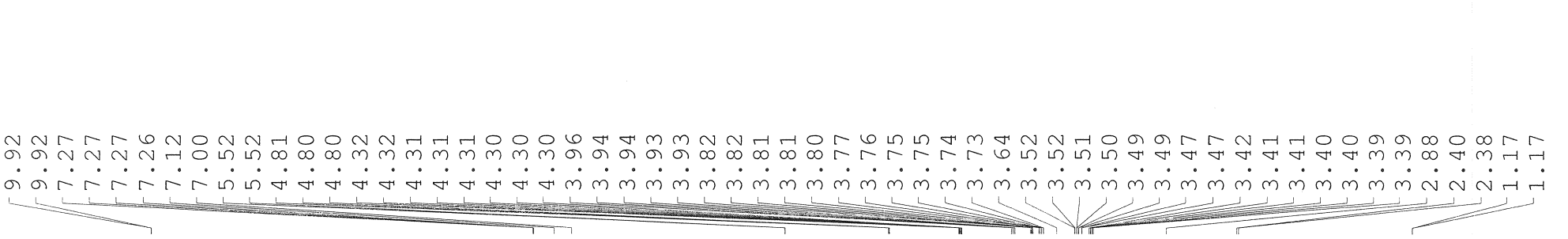
11e'




Current Data Parameters
NAME dx-I-63-2-13C
EXPNO 1
PROCNO 1
F2 - Acquisition Parameter
Date_ 20160712
Time 14.44
INSTRUM spect
PROBHD 5 mm CPTCI 1H-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 3649
DS 4
SWH 41666.668 Hz
FIDRES 0.635783 Hz
AQ 0.7864320 se
RG 1620
DW 12.000 us
DE 20.00 us
TE 298.0 K
D1 2.00000000 se
D11 0.03000000 se
TD0 1
======== CHANNEL f1 ======
SFO1 176.0377702 MH
NUC1 13C
P1 14.00 us
PLW1 95.00000000 W
======== CHANNEL f2 ======
SFO2 700.0228001 MH
NUC2 1H
CPDPRG[2 waltz16
PCPD2 65.00 us
PLW2 6.90000010 W
PLW12 0.10452000 W
PLW13 0.04416000 W
F2 - Processing parameters
SI 32768
SF 176.0201690 MH
WDW no
SSB 0
LB 0 Hz
GB 0
PC 1.40
148.125
142.852
139.521
139.247
137.906
128.801
128.606
128.295
127.746
127.392
126.496
111.421
110.110
109.845
102.000
101.711
77.228
77.046
76.864
72.360
70.879
44.365
41.173
39.606
12.683
NEt2
O
O
O
O
11e'
S42
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm


7.470
6.853
6.741
6.739
5.441
4.748
4.742
4.231
4.225
4.217
4.211
3.819
3.814
3.806
3.801
3.792
3.788
3.765
3.759
3.751
3.744
3.737
3.731
3.707
3.693
3.678
3.536
3.531
3.525
3.434
3.428
3.415
3.401
3.376
3.366
3.356
3.346
2.894
2.882
2.126
1.980
1.976
1.973
1.969
1.966
1.141
1.131
1.121
-0.000
CDCl3/CD3CN=5/2
Current Data Parameters
NAME dx-I-64-2-1H
EXPNO 3
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160719
Time 23.20
INSTRUM spect
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 128
DS 2
SWH 14097.744 Hz
FIDRES 0.215115 Hz
AQ 2.3243434 sec
RG 22.6
DW 35.467 usec
DE 10.00 usec
TE 298.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 700.0243229 MHz
NUC1 1H
P1 8.00 usec
PLW1 6.90000010 W
F2 - Processing parameters
SI 65536
SF 700.0237151 MHz
WDW no
SSB 0
LB 0 Hz
GB 0
PC 1.00
Et2N
OH
HO
MeO
OMe
11g
S43
7.5
7.0
6.5
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
0.951
2.032
2.000
1.965
1.962
1.914
2.144
1.988
2.064
5.605
2.910
6.035
1.739
5.988


152.929
143.878
122.297
116.826
115.789
107.537
107.519
105.468
86.513
83.285
83.101
82.916
78.190
76.391
74.116
67.757
60.566
49.450
17.639
6.687
6.571
6.454
6.333
6.217
CDCl3/CD3CN=5/2
Current Data Parameters
NAME DX-I-64-2-13C
EXPNO 3
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160719
Time 23.45
INSTRUM spect
PROBHD 5 mm CPTCI 1H-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 10240
DS 4
SWH 41666.668 Hz
FIDRES 0.635783 Hz
AQ 0.7864320 sec
RG 2050
DW 12.000 usec
DE 20.00 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 176.0377702 MHz
NUC1 13C
P1 14.00 usec
PLW1 95.00000000 W
======== CHANNEL f2 ========
SFO2 700.0228001 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 65.00 usec
PLW2 6.90000010 W
PLW12 0.10452000 W
PLW13 0.04416000 W
F2 - Processing parameters
SI 32768
SF 176.0201690 MHz
WDW no
SSB 0
LB 0 Hz
GB 0
PC 1.40
Et2N
OH
HO
MeO
OMe
11g
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

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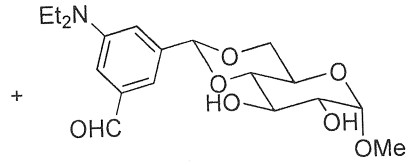
Current Data Parameters
NAME      dx-I-64-1confirm
EXPNO                    2
PROCNO                   1

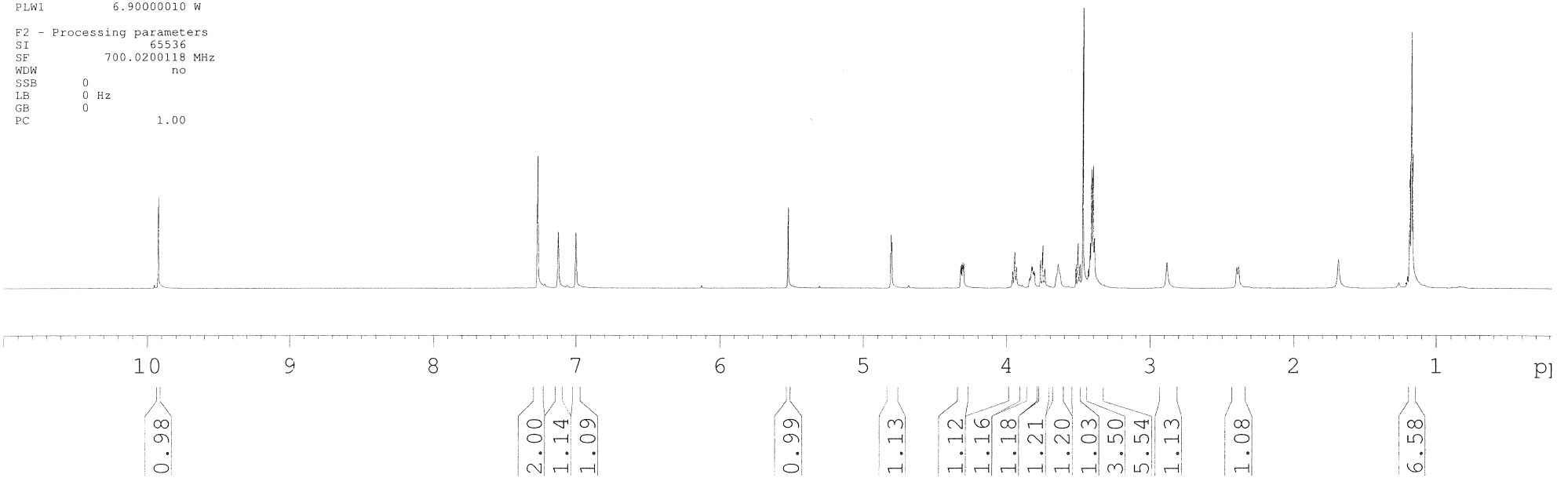
F2 - Acquisition Parameters
Date_           20160713
Time               12.08
INSTRUM            spect
PROBHD   5 mm CPTCI 1H-
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH            14097.744 Hz
FIDRES          0.215115 Hz
AQ             2.3243434 sec
RG                  22.6
DW                35.467 usec
DE                10.00 usec
TE                298.0 K
D1            1.00000000 sec
TD0                    1

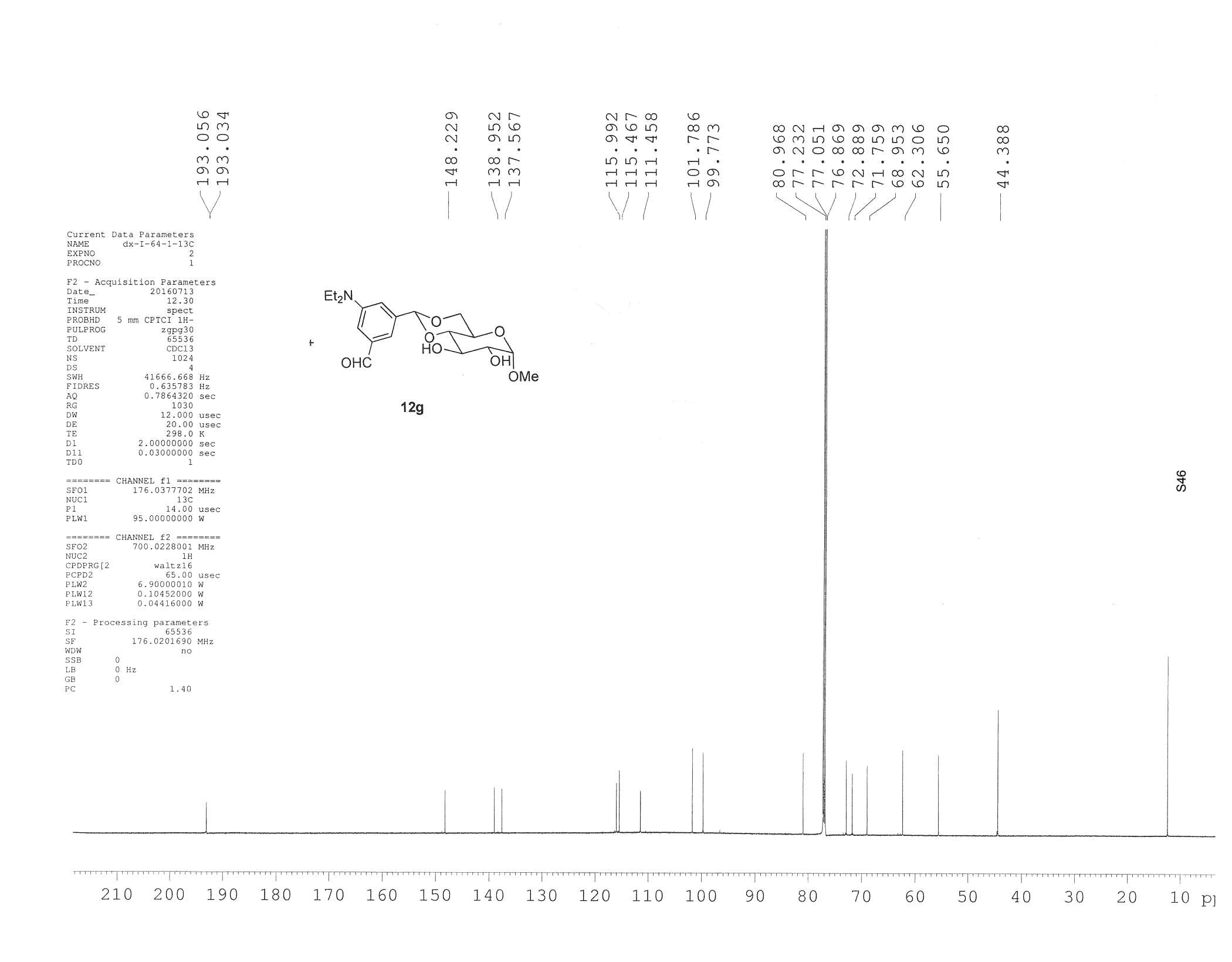
======== CHANNEL f1 ========
SFO1         700.0243229 MHz
NUC1                   1H
P1                  8.00 usec
PLW1          6.90000010 W

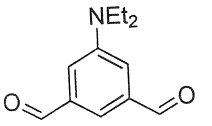
F2 - Processing parameters
SI                 65536
SF            700.0200118 MHz
WDW                   no
SSB       0
LB        0 Hz
GB        0
PC                  1.00
```



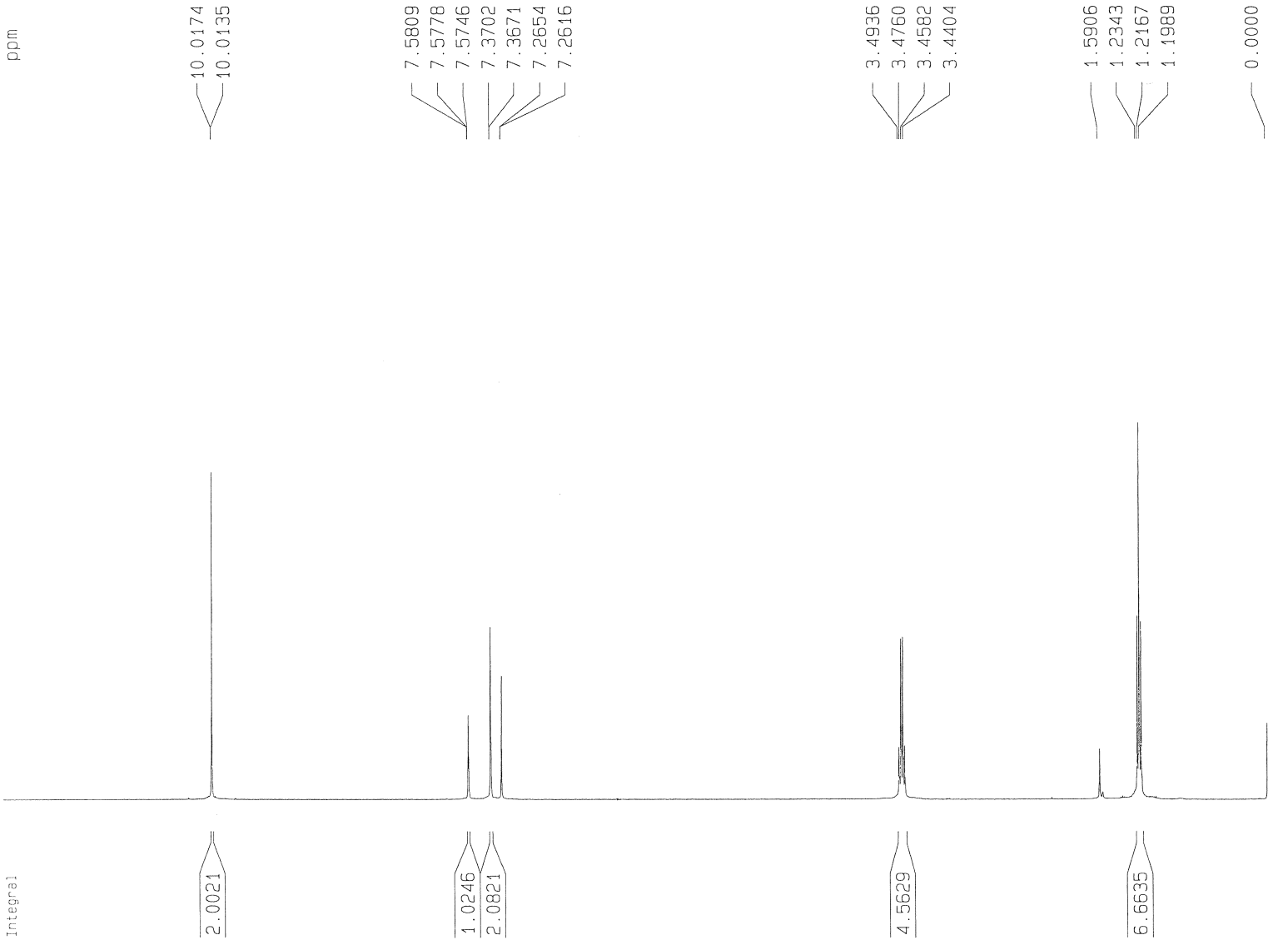
12g




193.056
193.034
148.229
138.952
137.567
115.992
115.467
111.458
101.786
99.773
80.968
77.232
77.051
76.869
72.889
71.759
68.953
62.306
55.650
44.388
Current Data Parameters
NAME dx-I-64-1-13C
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160713
Time 12.30
INSTRUM spect
PROBHD 5 mm CPTCI 1H-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 41666.668 Hz
FIDRES 0.635783 Hz
AQ 0.7864320 sec
RG 1030
DW 12.000 usec
DE 20.00 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 176.0377702 MHz
NUC1 13C
P1 14.00 usec
PLW1 95.00000000 W
======== CHANNEL f2 ========
SFO2 700.0228001 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 65.00 usec
PLW2 6.90000010 W
PLW12 0.10452000 W
PLW13 0.04416000 W
F2 - Processing parameters
SI 65536
SF 176.0201690 MHz
WDW no
SSB 0
LB 0 Hz
GB 0
PC 1.40
Et2N
OHC
HO
OH
OMe
12g
S46
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm



15



Current Data Parameters

NAME	dx-I-59spot2
EXPNO	2
PROCNO	1

F2 - Acquisition Parameters

Date_	20160622
Time	10.12
INSTRUM	drx400
PROBHD	5 mm Multinu
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	8278.146 Hz
FIDRES	0.126314 Hz
AQ	3.9584243 sec
RG	512
DW	60.400 usec
DE	6.00 usec
TE	300.0 K
D1	1.00000000 sec

========== CHANNEL f1 ========

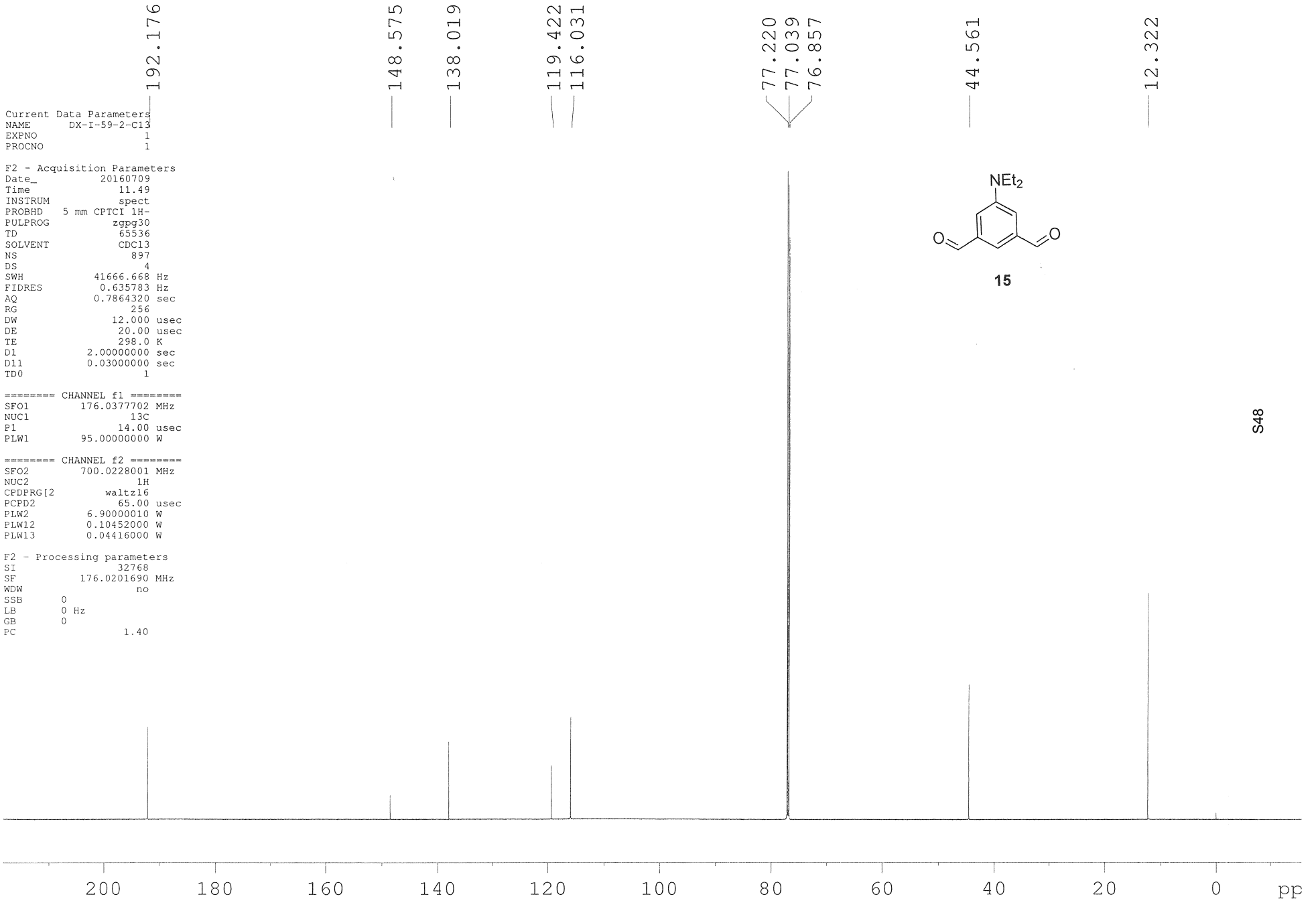
NUC1	1H
P1	6.50 usec
PL1	0.00 dB
SFO1	400.1324710 MHz

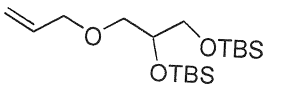
F2 - Processing parameters

SI	131072
SF	400.1300071 MHz
WDW	no
SSB	0
LB	0.00 Hz
GB	0
PC	1.00

1D NMR plot parameters

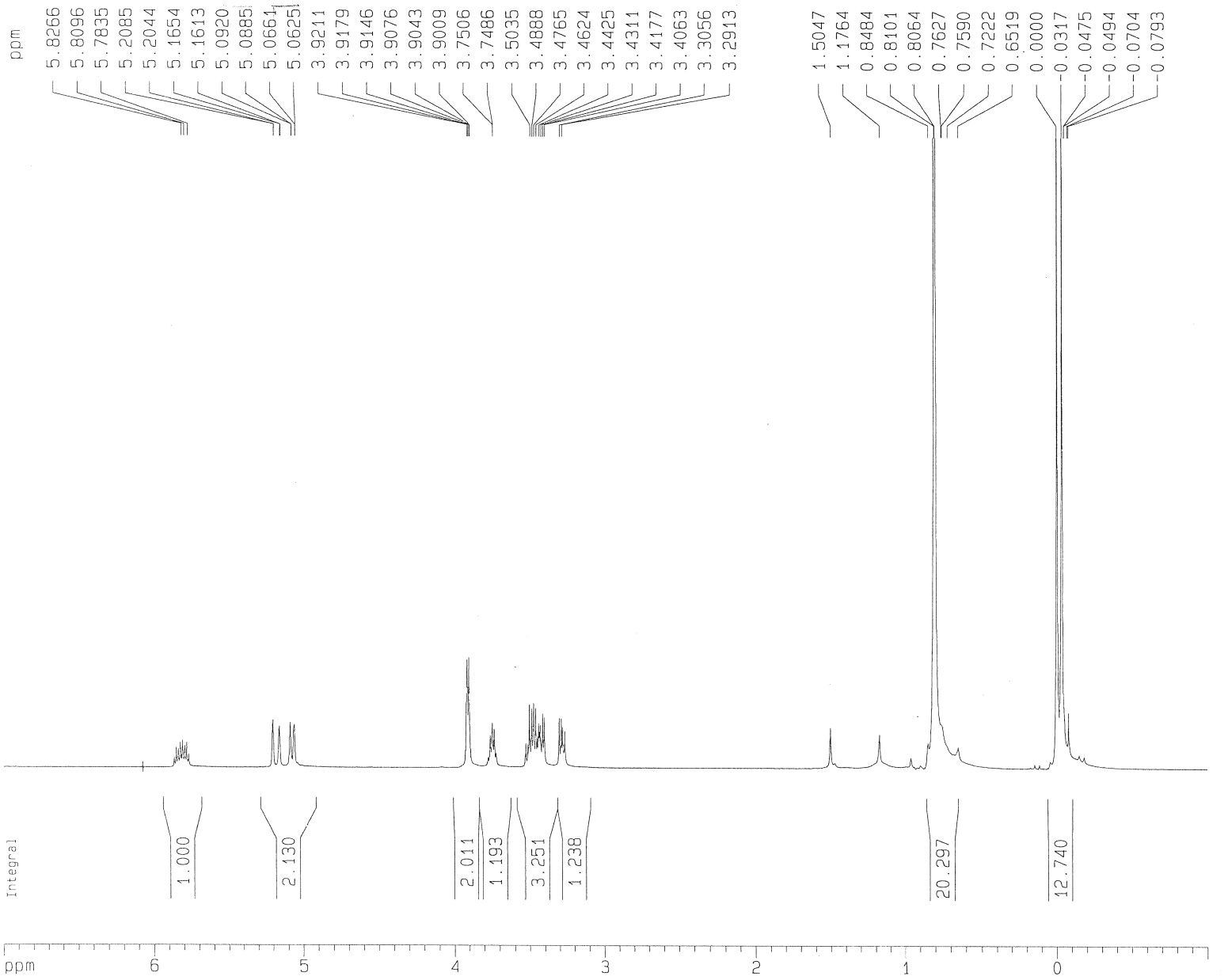
CX	20.00 cm
F1P	12.000 ppm
F1	4801.56 Hz
F2P	0.000 ppm
F2	0.00 Hz
PPMCM	0.60000 ppm/cm
HZCM	240.07800 Hz/cm


192.176
148.575
138.019
119.422
116.031
77.220
77.039
76.857
44.561
12.322
Current Data Parameters
NAME DX-I-59-2-C13
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160709
Time 11.49
INSTRUM spect
PROBHD 5 mm CPTCI 1H-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 897
DS 4
SWH 41666.668 Hz
FIDRES 0.635783 Hz
AQ 0.7864320 sec
RG 256
DW 12.000 usec
DE 20.00 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 176.0377702 MHz
NUC1 13C
P1 14.00 usec
PLW1 95.00000000 W
======== CHANNEL f2 ========
SFO2 700.0228001 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 65.00 usec
PLW2 6.90000010 W
PLW12 0.10452000 W
PLW13 0.04416000 W
F2 - Processing parameters
SI 32768
SF 176.0201690 MHz
WDW no
SSB 0
LB 0 Hz
GB 0
PC 1.40
NEt2
O
O
15
200
180
160
140
120
100
80
60
40
20
0
ppm



S1

1H



Current Data Parameters

NAME	dd-4-85repure
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

Date_	20160404
Time	8.18
INSTRUM	drx400
PROBHD	5 mm Multinu
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	8278.146 Hz
FIDRES	0.126314 Hz
AQ	3.9584243 sec
RG	181
DW	60.400 usec
DE	6.00 usec
TE	300.0 K
D1	1.00000000 sec

========== CHANNEL f1 ========

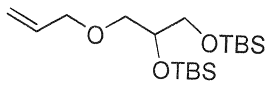
NUC1	1H
P1	6.50 usec
PL1	0.00 dB
SFO1	400.1324710 MHz

F2 - Processing parameters

SI	131072
SF	400.1300399 MHz
WDW	no
SSB	0
LB	0.00 Hz
GB	0
PC	1.00

1D NMR plot parameters

CX	20.00 cm
F1P	7.000 ppm
F1	2800.91 Hz
F2P	-1.000 ppm
F2	-400.13 Hz
PPMCM	0.40000 ppm/cm
HZCM	160.05202 Hz/cm



S1

135.00
116.53
77.43
77.00
76.58
72.71
72.28
72.20
65.08
25.94
25.87
18.34
18.21

BRUKER

```
Current Data Parameters
NAME          dd-4-85-13C
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_            20160405
Time                17.25
INSTRUM             spect
PROBHD   5 mm DUL 13C-1
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                   897
DS                     4
SWH           17985.611 Hz
FIDRES         0.274439 Hz
AQ             1.8219508 sec
RG                 32768
DW                27.800 usec
DE                 6.00 usec
TE                300.0 K
D1           2.00000000 sec
d11          0.03000000 sec
DELTA        1.89999998 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  13C
P1                 8.00 usec
PL1               -1.80 dB
SFO1         75.4124265 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2             75.00 usec
PL2               -2.50 dB
PL12              11.48 dB
PL13              12.00 dB
SFO2        299.8811995 MHz

F2 - Processing parameters
SI                 32768
SF          75.4048860 MHz
WDW                   EM
SSB                    0
LB                1.00 Hz
GB                     0
PC                 1.40
```