SUPPORTING INFORMATION

Synthesis of 1,2,4-Triazines and the Triazinoisoquinolinedione DEF Ring System of Noelaquinone

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General Information.

All reactions were performed under an N_2 atmosphere and all glassware was dried in an oven at 140 °C for 2 h prior to use. Reactions carried out at -78 °C employed a CO_2/acetone bath. THF and Et_2O were distilled over sodium/benzophenone ketyl, Et_3N was distilled from CaH_2, and CH_2Cl_2 and toluene were purified using an alumina column filtration system. All other reagents and solvents were used as received unless otherwise noted. Reactions were monitored by TLC analysis (pre-coated silica gel 60 F_{254} plates, 250 μm layer thickness) and visualization was accomplished with a 254 nm UV light and by staining with a PMA solution (5 g of phosphomolybdic acid in 100 mL of 95% EtOH), *p*-anisaldehyde solution (2.5 mL of *p*-anisaldehyde, 2 mL of AcOH, and 3.5 mL of conc. H_2SO_4 in 100 mL of 95% EtOH), Vaughn's reagent (4.8 g of $(NH_4)_6Mo_7O_{24}\cdot 4H_2O$ and 0.2 g of $Ce(SO_4)_2$ in 100 mL of a 3.5 N H_2SO_4 solution) or a $KMnO_4$ solution (1.5 g of $KMnO_4$ and 1.5 g of K_2CO_3 in 100 mL of a 0.1% NaOH solution). Flash chromatography on SiO_2 was used to purify the crude reaction mixtures.

NMR spectra were recorded using XWIN-NMR software. ^1H NMR spectra were obtained at 400 MHz in $CDCl_3$ unless otherwise noted. Chemical shifts were reported in parts per million with the residual solvent peak used as an internal standard. ^1H NMR spectra were obtained and are tabulated as follows: chemical shift, multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet), number of protons, and coupling constant(s). ^{13}C NMR spectra were run at 100 MHz using a proton-decoupled pulse sequence with a d_1 of 3 sec, and are tabulated by observed peak unless otherwise noted. Melting points were determined on a Mel-Temp II and are uncorrected. High-resolution mass spectrometry (HRMS) data (ESI/APCI technique) were recorded using a

Waters Q-Tof Ultima API-US instrument. HRMS data (EI technique) were recorded using a Micromass Autospec instrument. Mass spectrometry data were also recorded using an Applied Biosystems MDS SCIEX API 2000 LC/MS/MS system.

All organic azides and azide waste products should be considered toxic as well as potentially explosive and must be handled and stored with care. Avoid using halogenated solvents when performing reactions involving sodium azide, in addition to using halogenated solvents in reaction workup. Avoid quenching/manipulating/treating sodium azide reactions with acid, as generation of trace amounts of hydrazoic acid (HN_3) may result in an explosion. In general, a safety shield must be used when conducting reactions involving either sodium azide or organic azide derivatives.

Experimental Part.

2-(2-Azidoethylamino)isoindoline-1,3-dione (5). A solution of **3** (2.43 g, 15.0 mmol) in dichloromethane (150 mL) was treated with 4 Å MS (15.0 g), and freshly prepared α-bromoacetaldehyde **4** (15 mL of a 2 M solution in MeOH, 30 mmol) was added dropwise under vigorous stirring at room temperature. After 10 min, the mixture turned clear, and filtration and solvent removal under reduced pressure gave the crude α-bromohydrazone as a slightly yellow solid that was added into a 500 mL round bottom flask and dissolved in CH_3CN (150 mL) and AcOH (25 mL). $NaCNBH_3$ (9.45 g, 150.0 mmol) was added portionwise in 2 min, and the solution was stirred for 2 h. Solvent removal under reduced pressure at room temperature gave a viscous yellow oil that was transferred into a separatory funnel containing a saturated aqueous solution of $NaHCO_3$ (300 mL) and extracted with EtOAc (4×100 mL), and dried (Na_2SO_4). The combined organic layers were evaporated to give the crude bromoethyl derivative as a yellow oil that was rapidly purified by chromatography on SiO_2 (EtOAc:Hexanes, 2:3) to afford an oil (ca. 3 g) containing mostly the bromoethyl derivative. This oil was transferred into a 100 mL round bottom flask containing DMF (45 mL) and NaN_3 (2.93 g, 45.0 mmol) and the resulting solution was stirred at 60 °C for 15 h, poured into a separatory funnel containing H_2O (300 mL) and extracted with EtOAc (5 x 50 mL). The combined organic layers were washed with H_2O (4×30 mL), dried (Na_2SO_4) and concentrated to give a viscous yellow oil. Chromatography on SiO_2 (EtOAc:Hexanes, 1:9 to 2:8 to 3:7 to1:1) afforded **5** (1.62 g, 7.01 mmol, 47% from **3**) as a yellowish solid: Mp 56-57 °C (EtOAc/Hexanes); Rf 0.21 (EtOAc:Hexanes, 3:7); IR (neat) 3303, 2930, 2103, 1784, 1731, 1614, 1390, 1298, 1194, 884 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.90-7.85 (m, 2 H), 7.79-7.73 (m, 2 H), 4.83 (t, *J* = 5.3 Hz, 1 H), 3.54 (t, *J* = 5.3 Hz, 2 H), 3.23 (q, *J* = 5.6 Hz, 2 H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 166.3 (2C), 133.9 (2C), 129.8 (2C), 123.0 (2C), 49.8, 49.4; MS (EI) *m/z* (rel intensity) 231 ($M^{+\bullet}$, 22), 176 (74), 175 (48), 148 (63), 131 (55), 76 (100); HRMS (EI) *m/z* calcd for $C_{10}H_9N_5O_2$ 231.0756, found 231.0756.

3,4-Dihydro-[1,2,4]triazino[3,2-*a*]isoindol-6(2*H*)-one (6). A solution of **5** (0.462 g, 2.00 mmol) in toluene (20 mL) on a 50 mL round bottom flask equipped with a reflux condenser was treated with PBu_3 (0.809 g, 4.00 mmol) and stirred at 100 °□ for 3.5 h. The solvent was removed under reduced pressure and the resulting yellow oil was purified by chromatography on SiO_2 ($MeOH:CH_2Cl_2$, 2:98) to afford **6** (326 mg, 1.74 mmol, 87%) as colorless solid: Mp 183-184 °C (EtOAc); Rf 0.26 ($MeOH:CHCl_3$, 5:95); IR (neat) 3183, 1723, 1668, 1384, 1193, 957, 695 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.84-7.78 (m, 2 H), 7.70-7.59 (m, 2 H), 4.42 (t, *J* = 7.1 Hz, 1 H), 3.82 (t, *J* = 5.0 Hz, 2 H), 3.37-3.35 (m, 2 H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 163.6, 148.5, 132.8, 132.4, 131.6, 129.2, 122.8, 120.5, 46.0, 42.9; MS (EI) *m/z* (rel intensity) 187 ($M^{+\bullet}$, 62), 160 (10), 130 (100), 102 (52); HRMS (EI) *m/z* calcd for $C_{10}H_9N_3O$ 187.0746, found 187.0736.

2-((2-Azidoethyl)(benzyl)amino)isoindoline-1,3-dione. A solution of **5** (1.50 g, 6.49 mmol) in DMF (15 mL) was treated with K_2CO_3 (3.58 g, 25.9 mmol), TBAHS (44.0 mg, 1.30 mmol) and BnBr (7.75 mL, 64.8 mmol). The heterogeneous mixture was stirred at 70 °C for 20 h, transferred into a separatory funnel containing H_2O (200 mL) and extracted with EtOAc (4 × 50 mL). The combined organic extracts were dried (Na_2SO_4) and concentrated under reduced pressure to give a yellow oil. Chromatography on SiO_2 (EtOAc:Hexanes, 1:9) afforded 2-((2-azidoethyl)(benzyl)amino)isoindoline-1,3-dione (2.05 g, 6.34 mmol, 98%) as a colorless solid: Mp 59-61 °C ($CHCl_3$); Rf 0.54 (EtOAc:Hexanes, 2:3); IR (neat) 3063, 2924, 2866, 2102, 1785, 1717, 1610, 1467, 1372 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.86-7.83 (m, 2 H), 7.79-7.75 (m, 2 H), 7.53-7.50 (m, 2 H), 7.35-7.29 (m, 3 H), 4.55 (s, 2 H), 3.60-3.56 (m, 2 H), 3.52-3.48 (m, 2 H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 166.8 (2C), 135.6, 133.9 (2C), 129.6, 129.4, 128.8 (2C), 128.0

(2C), 127.5, 122.9 (2C), 59.6, 53.3, 49.2; MS (EI) *m/z* (rel intensity) 321 ($M^{+\bullet}$, 2), 277 (17), 265 (28), 118 (43), 91 (100); HRMS (EI) *m/z* calcd for $C_{17}H_{15}N_5O$ 321.1226, found 321.1219.

4-Benzyl-3,4-dihydro-[1,2,4]triazino[3,2-*a*]isoindol-6(2*H*)-one (7). A solution of 2-((2-azidoethyl)(benzyl)amino)isoindoline-1,3-dione (642 mg, 2.00 mmol) and PBu_3 (809 mg, 4.00 mmol) in toluene (20 mL) in a 50 mL round bottom flask equipped with a reflux condenser was stirred at 100 °C for 8 h. The solvent was removed under reduced pressure and the resulting yellow oil was purified by chromatography on SiO_2 (EtOAc:Hexanes, 3:7) to afford **7** (538 mg, 1.94 mmol, 97%) as a yellowish solid: Mp 121-122 °C (EtOAc); Rf 0.08 (EtOAc:Hexanes, 3:7); IR (neat) 2935, 2853, 1733, 1667, 1385, 1182, 928 cm^{-1} ; ^1H NMR (300 MHz, $CDCl_3$) δ 7.87-7.84 (m, 2 H), 7.70-7.60 (m, 2 H), 7.51-7.48 (m, 2 H), 7.42-7.32 (m, 3 H), 4.13 (s, 2 H), 3.80 (t, *J* = 5.1 Hz, 2 H), 3.14 (t, *J* = 5.2 Hz, 2 H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 163.0, 149.2, 135.3, 133.1, 132.8, 131.9, 129.7 (2C), 129.3, 128.6 (2C), 128.2, 123.2, 120.7, 59.7, 44.6, 39.5; MS (EI) *m/z* (rel intensity) 277 ($M^{+\bullet}$, 19), 186 (26), 130 (52), 91 (100); HRMS (EI) *m/z* calcd for $C_{17}H_{15}N_3O$ 277.1215, found 277.1214.

(*E*)-Ethyl 2-(2-(2-hydroxyethyl)hydrazono)propanoate (11). To a solution of **10** (3.6 mL, 32 mmol) in THF (75 mL) was added a mixture of EtOH (1 mL) and 2-hydroxyethylhydrazine (3.0 mL, 45 mmol). The reaction mixture was heated at reflux for 5 h, concentrated *in vacuo* to a thick oil, and diluted with water (10 mL) and EtOAc (10 mL). The aqueous phase was extracted with EtOAc (3×10 mL), washed with brine, dried (Na_2SO_4), concentrated *in vacuo* and purified by chromatography on SiO_2 (EtOAc) to afford **11** (3.32 g, 19.1 mmol, 59%) as a pale oil: IR (ATR) 3308, 2938, 1697, 1561, 1442, 1369, 1313, 1144, 1054 cm^{-1}; ^1H NMR (600 MHz, $CDCl_3$) δ 6.08 (s, *J* = 4.0 Hz, 1 H), 4.27 (q, *J* = 7.2 Hz, 2 H), 3.85 (t, *J* = 4.8 Hz, 2 H), 3.59-3.56 (m, 2 H), 2.98 (bs, 1 H), 1.95 (s, 3 H), 1.32 (t, *J* = 7.2 Hz, 3 H); ^{13}C NMR(150 MHz, $CDCl_3$) δ 165.2, 132.8, 62.5, 61.3, 52.4, 14.5, 10.3; MS (EI) *m/z* 174 ($M^{+\bullet}$, 9), 128 (30), 117 (69), 73 (88), 61(100); HRMS (EI) *m/z* calcd for $C_7H_{14}N_2O_3$ 174.1004, found 174.1009.

(*E*)-Ethyl 2-(2-(2-azidoethyl)hydrazono)propanoate (12). To a solution of **11** (1.96 g, 11.3 mmol) and Et_3N (2.4 mL, 17 mmol) in THF (50 mL) was added MsCl (1.1 mL, 15 mmol). The reaction mixture was stirred at 25 °C for 15 min, quenched with satd. $NaHCO_3$ (20 mL), diluted with water (20 mL) and extracted with Et_2O (3×10 mL). The combined organic layers were dried (Na_2SO_4) and concentrated in vacuo to afford a yellow oil that was used without further purification. To the crude oil was added DMF (70 mL) followed by NaN_3 (2.19 g, 33.8 mmol). This mixture was heated to 50 °C behind a blast shield for 16 h when TLC analysis (Hexanes:EtOAc, 1:1) showed that the mesylate intermediate was completely consumed. The reaction mixture was cooled to 25 °C, diluted with water (50 mL) and brine (30 mL) and extracted with EtOAc (4×50 mL). The combined organic phases were washed with water (50 mL), brine (50 mL), dried (Na_2SO_4), concentrated in vacuo and purified by chromatography on SiO_2 (100% Hexanes to Hexanes:EtOAc, 2:1 with 1% Et_3N) to afford **12** (1.57 g, 7.88 mmol, 70% for 2 steps) as a yellow oil: IR (ATR) 3310, 2982, 2099, 1699, 1569, 1445, 1369, 1307, 1149 cm^{-1}; ^{1}H NMR (600 MHz, $CDCl_3$) δ 5.84 (s, 1 H), 4.29 (q, *J* = 7.2 Hz, 2 H), 3.63-3.60 (m, 2 H), 3.55-3.53 (m, 2 H), 1.96 (s, 3 H), 1.33 (t, *J* = 7.2 Hz, 3 H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 165.1, 134.0, 61.4, 51.5, 49.9, 14.6, 10.5; MS (EI) *m/z* 184 ($[M-CH_3]^{+\bullet}$, 46), 174 (42), 130 (53), 117 (100), 56 (75); HRMS (EI) *m/z* calcd for $C_6H_{10}N_5O_2$ 184.0833 ($M-CH_3$), found 184.0835.

(E)-Ethyl 2-(2-(2-azidoethyl)-2-benzylhydrazono)propanoate (13). To a solution of **12** (491 mg, 2.46 mmol) in DMF (10 mL) was added K_2CO_3 (681 mg, 4.92 mmol), NaI (369 mg, 2.46 mmol), and benzyl bromide (1.8 mL, 15 mmol). The reaction mixture was heated at 70 °C behind a blast shield for 17 h, cooled to 25 °C, quenched with water (10 mL) and extracted with EtOAc (3×15 mL). The combined organic layers were washed with water (20 mL), brine (20 mL), dried (Na_2SO_4), concentrated *in vacuo* and purified by chromatography on SiO_2 (100% Hexanes to Hexanes:EtOAc, 1:1 with 1% Et_3N) to afford **13** (510 mg, 1.76 mmol, 72%) as a golden oil: IR (ATR) 2982, 2100, 1710, 1585, 1496, 1453, 1364, 1301, 1150, 1129, 1027 cm^{-1}; ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.38-7.35 (m, 4 H), 7.32-7.29 (m, 1 H), 4.33 (q, *J* = 7.2 Hz, 2 H), 4.07 (s, 2 H), 3.34 (t, *J* = 5.4 Hz, 2 H), 3.23 (t, *J* = 5.4 Hz, 2 H), 2.22 (s, 3 H), 1.36 (t, *J* = 7.2 Hz, 3 H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 165.1, 153.2, 136.8, 128.9, 128.4, 127.9, 62.0, 61.1, 56.2, 49.9, 16.2, 14.5; MS (EI) *m/z* 289 ($M^{+\bullet}$, 27), 234 (62), 188 (54), 91 (71), 65 (100); HRMS (EI) *m/z* calcd for $C_{14}H_{19}N_5O_2$ ($M^{+\bullet}$) 289.1542, found 289.1539.

1-(2-Azidoethyl)-1-benzylhydrazine (14). To a solution of **13** (87 mg, 0.30 mmol) in THF (2.5 mL) and H_2O (500 µL) at 25 °C was added hydrazine dihydrochloride (96 mg, 0.90 mmol). The reaction mixture was stirred at 25 °C for 3 h, quenched with solid Na_2CO_3, and concentrated *in vacuo*. The resulting oil was immediately purified by chromatography on SiO_2 (Hexanes:EtOAc, 1:1 with 1% NEt_3) to afford **14** (36 mg, 0.19 mmol, 63%) as an unstable, colorless oil: IR (ATR) 3345, 2937, 2815, 2099, 1717, 1600, 1495, 1453, 1352, 1279 cm^{-1}; ^1H NMR (600 MHz, $CDCl_3$) δ 7.36-7.33 (m, 4 H), 7.31-7.27 (m, 1 H), 3.74 (s, 2 H), 3.48 (t, *J* = 5.4 Hz, 2 H), 2.75 (t, *J* = 6.0 Hz, 2H), 2.74 (br s, 2 H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 137.3, 129.2, 128.7, 127.7, 67.7, 58.8, 48.8; MS (ESI) *m/z* 192 ($[M+H]^+$); HRMS (ESI) *m/z* calcd for $C_9H_{14}N_5$ ($[M+H]^+$) 192.1249, found 192.1215.

N'-(2-Azidoethyl)-N'-benzyl-2-iodobenzohydrazide (16). To a solution of **15** (4.33 g, 16.3 mmol) in CH_2Cl_2 (20 mL) cooled to 0 °C was added a solution of **14** (3.27 g, 17.1 mmol) in CH_2Cl_2 (5 mL) via syringe, followed by Et_3N (3.0 mL, 22 mmol). The reaction mixture was allowed to warm to 25 °C overnight. After 12 h, the reaction was quenched with satd. $NaHCO_3$ (15 mL), and extracted with CH_2Cl_2 (3 ×10 mL). The combined organic phases were dried (Na_2SO_4), concentrated *in vacuo*, and recrystallized using Hexanes/EtOAc to afford **16** (4.74 g, 11.3 mol, 69%) as cream colored needles: Mp 100-101 °C; IR (ATR) 3232, 2871, 2104, 1658, 1580, 1513, 1461, 1278 cm^{-1}; ^1H NMR (600 MHz, $CDCl_3$) δ 7.82 (dd, *J* = 7.9, 0.8 Hz, 2 H), 7.45 (app d, *J* = 7.2 Hz, 2 H), 7.37 (app t, *J* = 7.2 Hz, 2 H), 7.33-7.27 (m, 2 H), 7.07 (td, *J* = 7.8 Hz, 1 H), 6.93 (dd, *J* = 7.5, 1.2 Hz, 1 H), 6.74 (s, 1 H), 4.35 (s, 2 H), 3.56 (t, *J* = 6.0 Hz, 2 H), 3.43 (t, *J* = 6.0 Hz, 2 H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 169.2, 140.5 140.1, 136.9, 131.6, 129.6, 128.8, 128.3, 128.3, 128.0, 92.8, 60.5, 54.8, 49.2; MS (ESI) *m/z* 422 ($[M+H]^+$); HRMS (ESI) *m/z* calcd for $C_{16}H_{17}IN_5O$ ($[M+H]^+$) 422.0478, found 422.0465.

2-((2-Azidoethyl)(benzyl)amino)isoquinoline-1,3(2*H*,4*H*)-dione (18). To a flame-dried 100 mL flask was added **16** (3.20 g, 7.60 mmol), CuI (72 mg, 0.37 mmol), 2-picolinic acid (94 mg, 0.76 mmol) and Cs_2CO_3 (7.42 g, 22.8 mmol). The reaction mixture was purged 3x with N_2 and diluted with anhydrous dioxane (36 mL). After addition of **17** (2.3 mL, 15 mmol), the flask was placed in a pre-heated oil bath at 70 °C under a N_2

atmosphere. TLC analysis (Hexanes:EtOAc, 6:1) after 3 h showed that the starting material was consumed. The mixture was cooled to 25 °C, quenched with satd. NH_4Cl (10 mL), extracted with EtOAc (3×10 mL), washed with brine (20 mL), and concentrated in vacuo. A solution of the resulting residue in toluene (13 mL) and H_2O (3 mL) was treated with *p*-TSA (28.0 mg, 0.147 mmol) and the pink colored mixture was heated at reflux for 20 h, cooled to 25 °C, and quenched with satd. $NaHCO_3$ (20 mL). The biphasic mixture was partitioned and the aqueous phase was extracted with EtOAc (3×10 mL). The crude mixture showed one spot by TLC (Hexanes:EtOAc, 5:1). The combined organic phases were washed with brine (10 mL), dried (Na_2SO_4), concentrated *in vacuo*, and purified by chromatography on SiO_2 (Hexanes:EtOAc, 10:1 to 5:1) to afford **18** (1.87 g, 5.57 mmol, 73%, 2 steps) as a light yellow-green oil: IR (ATR) 3063, 2924, 2098, 1730, 1683, 1605, 1462, 1345, 1228 cm^{-1}; ^1H NMR (600 MHz, $CDCl_3$) δ 8.17 (d, *J* = 7.2 Hz, 1 H), 7.55 (dd, *J* = 7.2, 1.2 Hz, 1 H), 7.47 (d, *J* = 7.2 Hz, 2 H), 7.42 (t, *J* = 7.8 Hz, 1 H), 7.28-7.26 (m, 2 H), 7.22-7.19 (m, 2 H), 4.45, 4.39 (AB, *J* = 12.6 Hz, 2 H), 4.00, 3.83 (AB, *J* = 22.2 Hz, 2 H), 3.50-3.38 (m, 4 H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 169.9, 165.2, 137.0, 133.9, 133.8, 129.5, 129.4, 128.5, 127.9, 127.9, 127.2, 125.9, 60.2, 53.0, 50.4, 37.9,, 23.6; MS(ESI) *m/z* 336 ($[M+H]^+$); HRMS (ESI) *m/z* calcd for $C_{18}H_{18}N_5O_2$ ($[M+H]^+$) 336.1461, found 336.1449.

2'-((2-Azidoethyl)(benzyl)amino)-1'*H*-spiro[[1,3]dithiane-2,4'-isoquinoline]-1',3'(2'*H*)-dione (20). To a solution of **18** (889 mg, 2.65 mmol) in CH_2Cl_2 (30 mL) at 25 °C was added Et_3N (760 µL, 5.45 mmol) followed by **19** (1.27 g, 3.05 mmol). The green-yellow solution was stirred at 25 °C under N_2 for 21 h, quenched with satd. NH_4Cl (10 mL), extracted with CH_2Cl_2 (2×10 mL), dried (Na_2SO_4), concentrated, and purified by chromatography on SiO_2 (Hexanes:EtOAc, 4:1) to afford **20** (989 mg, 2.25 mmol, 85%) as a light green oil: IR (ATR) 2921, 2099, 1724, 1684, 1598, 1455, 1337, 1224 cm^{-1}; ^1H NMR (600 MHz, $CDCl_3$) δ 8.18 (dd, *J* = 7.9, 1.8 Hz, 1 H), 8.10 (app d, *J* = 7.8 Hz, 1 H), 7.65 (app dt, *J* = 7.5 Hz, *J* = 1.8 Hz, 1 H), 7.52 (d, *J* = 7.8 Hz, 2 H), 7.53-7.49 (m, 1 H), 7.29 (app t, *J* = 7.2 Hz, 2 H), 7.22 (t, *J* = 7.3 Hz, 1 H), 4.44 (app q, *J* = 13.2 Hz, 2 H), 3.82 (td, *J* = 13.5, 2.5 Hz, 1 H), 3.76 (td, *J* = 13.5, 2.5 Hz, 1 H), 3.50-3.32 (m, 4 H), 2.76 (dt, *J* = 13.9, 3.7 Hz, 1 H), 2.68 (dt, *J* = 13.9, 3.7 Hz, 1 H), 2.34-2.31 (m, 1 H), 2.07-1.99 (m, 2 H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 170.0, 163.3, 137.2, 137.0, 134.4, 129.9, 129.7, 129.6, 129.3, 128.5, 127.9,125.1, 60.5, 52.5, 50.0, 49.6, 29.2, 29.1; MS (ESI) *m/z* 440 ($[M+H]^+$); HRMS (ESI) *m/z* calcd for $C_{21}H_{22}N_5O_2S_2$ ($[M+H]^+$) 440.1215, found 440.1212.

2-((2-Azidoethyl)(benzyl)amino)-4,4-dimethoxyisoquinoline-1,3(2*H*,4*H*)-dione (21). To a solution of **20** (343 mg, 0.780 mmol) in dry MeOH (8.0 mL) was added TFA (180 μL, 2.35 mmol) and PIFA (674 mg, 2.35 mmol) at 23 °C. The reaction mixture was stirred for 15 min when TLC analysis (Hexanes:EtOAc, 4:1) of an aliquot quenched with Na_2CO_3 showed that the starting material was converted to a slightly more polar spot. After 20 min, the mixture was quenched with solid Na_2CO_3 to pH>7, causing the yellow solution to change to a colorless one as the pH increased. The solution was extracted with CH_2Cl_2 (4×10 mL), dried ($MgSO_4$), concentrated, and purified by chromatography on SiO_2 (Hexanes:EtOAc, 5:1 to 3:1 with 1% Et_3N) to afford **21** (226 mg, 0.571 mmol, 73%) as a pale yellow oil: ^{1}H NMR (600 MHz, $CDCl_3$) δ 8.16 (d, *J* = 8.1 Hz, 1 H), 7.68-7.65 (m, 2 H), 7.59-7.56 (m, 1 H), 7.45 (d, *J* = 7.2 Hz, 2 H), 7.25 (t, *J* = 7.2 Hz, 2 H), 7.19 (t, *J* = 7.2 Hz, 1 H), 4.46, 4.37 (AB, *J* = 12.6 Hz, 2 H), 3.54-3.49 (m, 1 H), 3.42-3.37 (m, 3 H), 3.32 (s, 3 H), 3.08 (s, 3 H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 168.5, 164.0, 136.6, 135.7, 133.9, 130.4, 129.8, 129.3, 128.5, 128.0, 126.6, 126.6, 96.0, 60.3, 52.9, 52.4, 52.2, 49.8; HRMS (ESI) *m/z* calcd for $C_{20}H_{22}N_5O_4$ ($[M+H]^+$) 396.1672, found 396.1663.

4-Benzyl-11,11-dimethoxy-3,4-dihydro-2*H*-[1,2,4]triazino[2,3-*b*]isoquinolin-6(11*H*)-one (22). To a flame-dried microwave vial was added a solution of **21** (385 mg, 0.974 mmol) in distilled PhCl (2.5 mL), followed by a solution of PBu_3 (337 mg, 1.67 mmol) in distilled PhCl (580 μL). The green-brown reaction mixture was stirred at room temperature for 10 min, and then heated at 180 °C for 20 min. TLC analysis (CH_2Cl_2:EtOH, 50:1) showed that the starting material was converted to a single, streaky, more polar spot. The dark purple solution was concentrated under a stream of N_2 and purified by chromatography on SiO_2 (100% CH_2Cl_2 to CH_2Cl_2:EtOH, 100:1 to 25:1) to produce an oily wax that was triturated with hexanes and then recrystallized from Et_2O:Hexanes to afford **22** (210 mg, 0.597 mmol, 61%) as cream colored needles: Mp 127-128 °C; ^{1}H NMR (600 MHz, $CDCl_3$) δ 8.18 (dd, *J* = 7.8, 0.6 Hz, 1 H), 7.73 (d, *J* = 7.2 Hz, 1 H), 7.66 (dt, *J* = 7.2, 1.2 Hz, 1 H), 7.56-7.53 (m, 3 H), 7.36 (t, *J* = 7.2 Hz, 2 H), 7.31 (t, *J* = 7.2 Hz, 1 H), 4.07 (brs, 2 H), 3.80 (brs, 2 H), 3.39 (brs, 6 H), 3.18 (t, *J* = 6.0 Hz, 2 H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 159.8, 148.1, 135.8, 135.6, 133.0, 130.1, 130.0, 129.3, 128.7, 128.2, 127.3, 126.3, 96.4, 58.4, 45.7, 39.0; MS (ESI) *m/z* 352 ($[M+H]^+$); HRMS (ESI) *m/z* calcd for $C_{20}H_{22}N_3O_3$ ($[M+H]^+$) 352.1661, found 352.1643.

2-Benzyl-3,4-dihydro-2H-[1,2,4]triazino[4,3-b]isoquinoline-6,11-dione (23a); 4-benzyl-3,4-dihydro-2H-[1,2,4]triazino[2,3-b]isoquinoline-6,11-dione (23b). After concentrated H_2SO_4 (3.0 mL) was added dropwise into H_2O (3.0 mL), the mixture was stirred for 5 min, cooled to room temperature and treated with a solution of **22** (29.8 mg, 0.0848 mmol) in CH_2Cl_2 (6.0 mL). The reaction mixture was stirred at 40 °C for 12 h under argon, cooled to 25 °C, and solid Na_2CO_3 was added until pH>7. The mixture was extracted with CH_2Cl_2 (3×30 mL), dried (Na_2SO_4), concentrated, and purified by chromatography on SiO_2 (Hexanes:EtOAc, 1:1 with 1% Et_3N) to afford **23a** (3.5 mg, 0.011 mmol, 13%) as golden needles and **23b** (8.4 mg, 0.028 mmol, 32%) as yellow crystals. **23a**: Mp 171.1-174.5 °C; IR (neat) 2923, 2852, 1659, 1597, 1527, 1453, 1361, 1342, 1314, 1281, 1246, 1079, 1064, 1008 cm^{-1}; ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.33 (td, *J* = 7.0, 2.0 Hz, 1 H), 8.32 (t, *J* = 6.5 Hz, 1 H), 7.80 (dtd, *J* = 18, 7.5, 1.5 Hz, 2 H), 7.35-7.33 (m, 5 H), 4.73 (s, 2 H), 4.15 (t, *J* = 5.2 Hz, 2 H), 3.25 (t, *J* = 5.2 Hz, 2 H); ^{13}C NMR (125 MHz, $CDCl_3$; some rearrangement and decomposition occurred during the spectral data collection) δ 173.8, 157.7, 135.6, 134.4, 133.3, 130.3, 129.0, 128.8, 128.4, 127.2, 63.1, 43.8, 38.1; HRMS (ESI) *m/z* calcd for $C_8H_{16}N_3O_2$ ($[M+H]^+$) 306.1243, found 306.1250. **23b**: Mp 121.1-124.3 °C; IR (neat) 3068, 2917, 2974, 2924, 2865, 1698, 1597, 1489, 1376, 1355, 1226, 1084, 1070, 984, 690 cm^{-1}; ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.37 (dd, *J* = 7.5, 1.0 Hz, 1 H), 8.28 (dd, *J* = 7.5, 1.0 Hz, 1 H), 7.87 (td, *J* = 7.5, 1.0 Hz, 1 H), 7.79 (td, *J* = 7.5, 1.0 Hz, 1 H), 7.54 (d, *J* = 7.0 Hz, 2 H), 7.4-7.3 (m, 3 H), 4.10 (s, 2 H), 3.97 (t, *J* = 6.5 Hz, 2 H), 3.12 (t, *J* = 6.5 Hz, 2 H); ^{13}C NMR (125 MHz, $CDCl_3$; some rearrangement and decomposition occurred during the spectral data collection) δ 176.0, 157.5, 144.4, 135.6, 135.2, 133.8, 131.4, 130.1, 129.7, 129.5, 128.7, 128.3, 127.7, 58.7, 43.3, 40.6; HRMS (ESI) *m/z* calcd for $C_{18}H_{16}N_3O_2$ ($[M+H]^+$) 306.1237, found 306.1243.

N'-(2-Azidoethyl)-2-iodo-N'-(4-methoxybenzyl)benzohydrazide. To a solution of **15** (259 mg, 0.972 mmol) in CH_2Cl_2 (20 mL) cooled to 0 °C was added a solution of **24** (226 mg, 1.02 mmol) in CH_2Cl_2 (5 mL) via syringe, followed by Et_3N (0.20 mL, 1.5 mmol). After addition, the vessel was removed from the ice bath and the solution was allowed to warm to 25 °C overnight. After 12 h, the reaction mixture was quenched with satd. $NaHCO_3$ (15 mL) and extracted with CH_2Cl_2 (3 ×10 mL). The combined organic phases were dried (Na_2SO_4), concentrated *in vacuo*, and purified by chromatography on SiO_2 (Hexanes:EtOAc, 4:1 with 1% Et_3N) to afford N'-(2-azidoethyl)-2-iodo-N'-(4-methoxybenzyl)benzohydrazide (304 mg, 0.675 mmol, 69%) as colorless flake-like crystals: IR (neat) 3215.2, 3047.4, 2907.6, 2093.1, 1653.2, 1610.4, 1509.7, 1459.4, 1297.2, 1245.1, 1172.4, 1030.7, 820.1 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$) δ 7.69 (dd, *J* = 8.0, 1.0 Hz, 1 H), 7.40 (s, 1 H), 7.28 (dt, *J*= 8.5, 2.0 Hz, 2 H), 7.18 (td, *J*= 7.5, 1.0 Hz, 1 H), 6.97 (td, *J*= 7.5, 1.0 Hz, 1 H), 6.86 (dd, *J* = 7.5, 1.5 Hz, 1 H), 6.82 (dt, *J*= 9.0, 2.0 Hz, 2 H), 4.10 (s, 2 H), 3.73 (s, 3 H), 3.44 (t, *J* = 6.0 Hz, 2 H), 3.18 (t, *J* = 6.0 Hz, 2 H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 168.5, 158.9, 140.3, 139.4, 131.0, 130.4, 128.6, 127.8, 113.6, 92.6, 59.8, 55.1, 54.4, 48.6; HRMS (ESI) *m/z* calcd for $C_{17}H_{19}IN_5O_2$ ($[M+H]^+$) 452.0584, found 452.0564.

2-((2-Azidoethyl)(4-methoxybenzyl)amino)isoquinoline-1,3(2*H*,4*H*)-dione (25). To a flame-dried flask was added N'-(2-azidoethyl)-2-iodo-N'-(4-methoxybenzyl)benzohydrazide (215 mg, 0.476 mmol), CuI (9.1 mg, 0.048 mmol), 2-picolinic acid (11.7 mg, 0.0951 mmol) and Cs_2CO_3 (465 mg, 1.43 mmol). The reaction mixture was purged 3x with N_2, and diluted with anhydrous dioxane (19 mL). After addition of **17** (94 mg, 0.59 mmol), the reaction flask was placed in a pre-heated oil bath at 70 °C under a N_2 atmosphere. TLC analysis (Hexanes:EtOAc, 6:1) after 3 h showed that the starting material was consumed. The mixture was cooled to 25 °C, quenched with satd. NH_4Cl (10 mL), extracted with EtOAc (3×10 mL), washed with brine (20 mL), and concentrated in vacuo. The residue was dissolved in toluene (13 mL) and H_2O (3 mL), and *p*-TSA (45.2 mg, 0.238 mmol) was added. The pink colored mixture was heated at reflux for 20 h, cooled to 25 °C, and quenched with satd. $NaHCO_3$ (20 mL). The biphasic mixture was partitioned and then the aqueous phase was extracted with EtOAc (3x10 mL). The crude mixture showed one spot by TLC (Hexanes: EtOAc, 5:1). The combined organic phases were washed with brine (10 mL), dried (Na_2SO_4), concentrated in vacuo, and purified by chromatography on SiO_2 (Hexanes:EtOAc, 10:1 to 5:1) to afford **25** (109 mg, 0.297 mmol, 63%) as a bright yellow oil: IR (neat) 3068, 2947, 2097, 1726, 1681, 1609, 1510, 1459, 1342, 1245, 1171, 1033, 757 cm^{-1}; ^{1}H NMR (500 MHz, CDCl$_3$) δ 8.13 (d, *J* = 8.0 Hz, 1 H), 7.52 (t, *J* = 7.5 Hz, 1 H), 7.39 (t, *J* = 7.5 Hz, 1 H), 7.34 (dt, *J* = 8.5, 2.0 Hz, 2 H), 7.16 (d, *J* = 7.5 Hz, 1 H), 6.76 (dt, *J* = 8.5, 2.0 Hz, 2 H), 4.35, 4.30 (AB, *J* = 12.0 Hz, 2 H), 3.96, 3.80 (AB, *J* = 22.0 Hz, 2 H), 3.71 (s, 3 H), 3.43-3.31 (m, 4 H); ^{13}C NMR (125 MHz, CDCl$_3$) δ 169.7, 165.0, 159.1, 133.7, 130.6, 129.2, 128.8, 127.7, 127.0, 125.7, 113.6, 59.2, 55.2, 52.7, 50.1, 37.6; HRMS (ESI) *m/z* calcd for $C_{17}H_{20}N_5O_3$ ([M+H]$^+$) 366.1566, found 366.1589.

2'-((2-Azidoethyl)(4-methoxybenzyl)amino)-1'*H*-spiro[[1,3]dithiane-2,4'-isoquinoline]-1',3'(2'*H*)-dione. To a solution of **25** (109 mg, 0.297 mmol) in CH_2Cl_2 (30 mL) at 25 °C was added Et_3N (91.9 µL, 0.654 mmol) followed by **19** (156 mg, 0.357 mmol). The green-yellow solution was stirred at 25 °C under N_2 for 21 h, quenched with satd. NH_4Cl (10 mL), extracted with CH_2Cl_2 (2×10 mL), dried (Na_2SO_4), concentrated, and purified by chromatography on SiO_2 (Hexanes:EtOAc, 4:1) to afford 2'-((2-azidoethyl)(4-methoxybenzyl)amino)-1'*H*-spiro[[1,3]dithiane-2,4'-isoquinoline]-1',3'(2'*H*)-dione (92.1 mg, 0.196 mmol, 66%) as a colorless oil: IR (neat) 3009, 2927, 2871, 2097, 1723, 1681, 1611, 1512, 1335, 1243, 1171,751, 699 cm^{-1}; ^{1}H NMR (500 MHz, CDCl$_3$) δ 8.16 (dd, *J* = 8.0, 1.0 Hz, 1 H), 8.10 (dd, *J* = 8.0, 1.0 Hz, 1 H), 7.63 (td, *J* = 8.0, 1.0 Hz, 1 H), 7.48 (td, *J* = 8.0, 1.0 Hz, 1 H), 7.42 (dt, *J* = 8.0, 2.0 Hz, 2 H), 6.81 (dt, *J* = 8.0, 2.0 Hz, 2 H), 4.38, 4.34 (AB, *J* = 7.0 Hz, 2 H), 3.80 (td, *J* = 13.5, 2.0 Hz, 1 H), 3.75 (td, *J* =13.5, 2.0 Hz, 1 H), 3.74 (s, 3 H), 3.45-3.30 (m, 4 H), 2.73 (dt, *J* = 13.5, 3.5 Hz, 1 H), 2.66 (dt, *J* = 13.5, 3.5 Hz, 1 H), 2.32-2.29 (m, 1 H), 2.05-1.97 (m, 1 H); ^{13}C NMR (125 MHz, CDCl$_3$) δ 169.8, 163.1, 159.2, 136.9, 134.2, 130.7, 129.7, 129.5, 129.0, 125.0, 113.7, 59.6, 55.3, 52.2, 49.8, 49.4, 29.0, 28.9, 23.5; HRMS (ESI) *m/z* calcd for $C_{22}H_{23}N_5O_3S_2Na$ ([M+Na]$^+$) 492.1140, found 492.1170.

2-((2-Azidoethyl)(4-methoxybenzyl)amino)-4,4-dimethoxyisoquinoline-1,3(2*H*,4*H*)-dione. To a solution of 2'-((2-azidoethyl)(4-methoxybenzyl)amino)-1'*H*-spiro[[1,3]dithiane-2,4'-isoquinoline]-1',3'(2'*H*)-dione (92.1 mg, 0.196 mmol) in dry MeOH (8.0 mL) was added TFA (43.7 µL, 0.588 mmol) and PIFA (288 mg, 0.670 mmol) at 23 °C. The mixture was stirred for 15 min when TLC analysis (Hexanes:EtOAc, 4:1) of an aliquot neutralized with Na_2CO_3 showed that the starting material was converted to a slightly more polar spot. After 20 min, the reaction was quenched with solid Na_2CO_3 until pH>7, causing the yellow solution to change to colorless as the pH increased. The mixture was extracted with CH_2Cl_2 (4×10 mL), dried ($MgSO_4$), concentrated, and purified by chromatography on SiO_2 (Hexanes:EtOAc, 5:1 to 3:1 with 1% Et_3N) to afford 2-((2-azidoethyl)(4-methoxybenzyl)amino)-4,4-dimethoxyisoquinoline-1,3(2*H*,4*H*)-dione (56.6 mg, 0.133 mmol, 68%) as a pale yellow oil: IR (neat) 2992, 2936, 2863, 2097, 1739, 1691, 1739, 1512, 1458, 1333, 1282, 1243, 1172, 1083, 764 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$) δ 8.13 (d, *J* = 12.0 Hz, 1 H), 7.65 (d, *J* = 4.0 Hz, 2 H), 7.56-7.53 (m, 1 H), 7.33 (d, *J* = 8.5 Hz, 2 H), 6.75 (d, *J* = 8.5 Hz, 2 H), 4.38, 4.29 (AB, *J* = 12.5 Hz, 2 H), 3,71 (s, 3 H), 3.50-3.45 (m, 1 H), 3.37-3.33 (m, 3 H), 3.30 (s, 3 H), 3.11 (s, 3 H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 168.5, 163.8, 159.3, 135.7, 133.6, 130.9, 130.3, 129.2, 128.6, 126.6, 126.5, 113.7, 95.9, 59.5, 55.3, 52.7, 52.2, 52.1, 49.7; HRMS (ESI) *m/z* calcd for $C_{21}H_{23}N_5O_5Na$ ($[M+Na]^+$) 448.1597, found 448.1629.

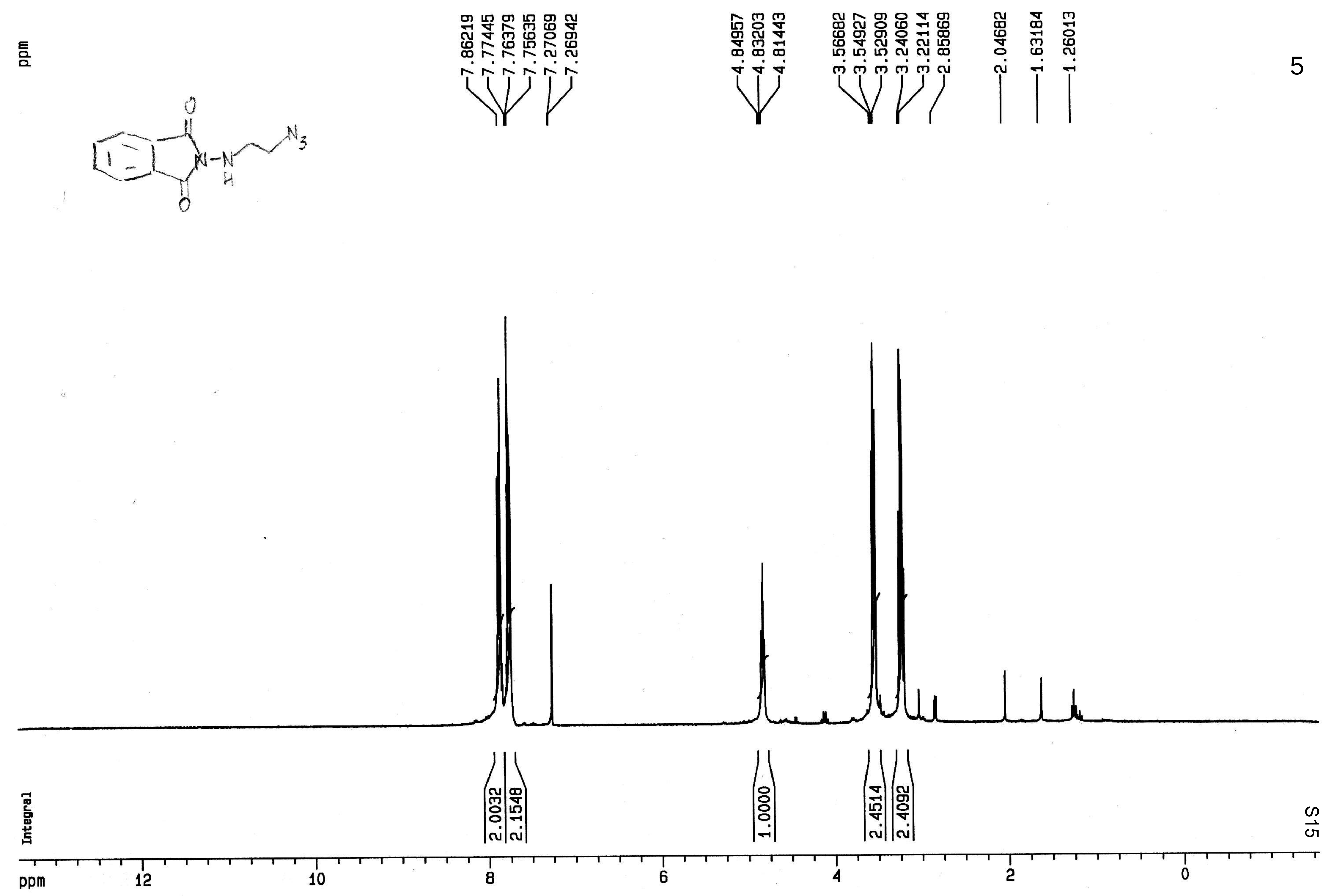
11,11-Dimethoxy-4-(4-methoxybenzyl)-3,4-dihydro-2*H*-[1,2,4]triazino[2,3-b]isoquinolin-6(11*H*)-one (26). To a flame-dried microwave vial was added a solution of 2-((2-azidoethyl)(4-methoxybenzyl)amino)-4,4-dimethoxyisoquinoline-1,3(2*H*,4*H*)-dione (56.6 mg, 0.133 mmol) in distilled PhCl (2.5 mL), followed by a solution of PBu_3 (46.0 mg, 0.228 mmol) in distilled PhCl (580 µL). The green-brown mixture was stirred at room temperature for 10 min then heated at 180 °C in the microwave reactor for 20 min. TLC analysis (EtOAc:Hexanes, 4:1) showed that the starting material was converted to a much more polar spot. The dark purple solution was concentrated under a stream of N_2 and the residue was purified by chromatography on SiO_2 (EtOAc:Hexanes, 4:1 with 1% Et_3N) to produce **26** (44.5 mg, 0.117 mmol, 88%) as a dark brown oil: IR (neat) 3096, 3046, 2990, 2936, 2872, 1735, 1692, 1646, 1586, 1512, 1443, 1363, 1351, 1297, 1277, 1234, 1172, 1073, 1033, 938 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$) δ 8.14 (dd, *J* = 8.0, 1.0 Hz, 1 H), 7.69 (dd, *J* = 7.5, 1.0 Hz, 1 H), 7.61 (td, *J* = 7.5, 1.5 Hz, 1 H), 7.50 (td, *J* = 7.5, 1.5 Hz, 1 H), 7.42 (dt, *J* = 9.5, 2.5 Hz, 2 H), 6.85 (dt, *J* = 9.5, 2.5 Hz, 2 H), 3.97 (brs, 2 H), 3.76 (s, 3 H), 3.77-3.72 (m, 2 H), 3.35 (brs, 6 H), 3.14 (t, *J* = 6.0 Hz, 2 H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 159.6, 159.4, 148.1, 135.6, 132.7, 131.0. 129.8, 129.0, 127.8, 127.3, 126.1, 113.9, 96.2, 57.6, 55.2, 52.4, 45.4, 38.9; HRMS (ESI) *m/z* calcd for $C_{21}H_{23}N_3O_4$ (M^+) 381.1689, found 381.1718.

11,11-Dimethoxy-3,4-dihydro-2H-[1,2,4]triazino[2,3-*b*]isoquinolin-6(11H)-one (27). Concentrated H_2SO_4 (3 mL) was added dropwise into H_2O (3 mL), and the acid mixture was stirred for 5 min and cooled to 4 °C. A solution of **26** (15.5 mg, 0.0406 mmol) in CH_2Cl_2 (10 mL) was added into the acid solution at 4 °C. The reaction mixture was immediately cooled to -20 °C, stirred at -20 °C for 12 h, and quenched with solid Na_2CO_3 at -20 - 4 °C until pH>7. The mixture was diluted with H_2O (20 mL), extracted with CH_2Cl_2 (3×30 mL), dried (Na_2SO_4), concentrated, and purified by chromatography on SiO_2 (EtOAc:Hexanes, 4:1 with 1% Et_3N) to afford **27** (7.6 mg, 0.0291 mmol, 72%) as a light yellow solid: IR (neat) 3276.7, 3271.1, 3066.0, 2926.3, 2849.8, 2875.9, 2831.2, 1675.6, 1638.3, 1584.3, 1552.6, 1364.3, 1284.2, 1237.6, 1036.3 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$) δ 8.16 (dd, *J* = 8.0, 1.0 Hz, 1 H), 7.73 (dd, *J* = 7.5, 1.0 Hz, 1 H), 7.66 (td, *J* = 7.5, 1.0 Hz, 1 H), 7.55 (td, *J* = 7.5, 1.0 Hz, 1 H), 6.52 (br s, 1 H), 3.89 (t, *J* = 5.0 Hz, 2 H), 3.34 (s, 6 H), 3.28 (t, *J* = 5.0 Hz, 2 H); ^{13}C NMR (75 MHz, $CDCl_3$) δ157.7, 145.5, 136.1, 133.2, 130.0, 128.4, 126.4, 126.1, 96.0, 52.3, 46.5, 43.9; HRMS (ESI) *m/z* calcd for $C_{13}H_{16}N_3O_3$($[M+H]^+$) 262.1192, found 262.1200;

3,4-Dihydro-2H-[1,2,4]triazino[2,3-*b*]isoquinoline-6,11-dione (28). Concentrated H_2SO_4 (3 mL) was added dropwise into H_2O (3 mL), and the acid mixture was stirred for 5 min and cooled to room temperature. A solution of **27** (18.0 mg, 0.0689 mmol) in CH_2Cl_2 (10 mL) was added into the acid solution. The reaction mixture was stirred for 6 h 15 min at room temperature, and quenched with solid Na_2CO_3 to pH>7. The mixture was diluted with H_2O (20 mL), extracted with CH_2Cl_2 (3×20 mL), dried (Na_2SO_4), and concentrated *in vacuo*. The crude ^{1}HNMR showed that the reaction was complete. The product was purified by chromatography on SiO_2 (EtOAc:Hexanes, 4:1 with 1% Et_3N) to afford **28** (9.2 mg, 0.0427 mmol, 62%) as yellow powder: IR 3282, 2924, 2867, 2850, 1694, 1661, 1609, 1596, 1581, 1458, 1437, 1379, 1297, 1241, 1215, 1102, 1010 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$) δ 8.28 (dd, *J* = 7.5, 1.0 Hz, 1 H), 8.25 (dd, *J* = 7.5, 1.0 Hz, 1 H), 7.85 (td, *J* = 7.5, 1.0 Hz, 1 H), 7.76 (td, *J* = 7.5, 1.0 Hz, 1 H), 6.64 (br s, 1 H), 4.05 (td, *J* = 5.0, 1.0 Hz, 2 H), 3.35-3.33 (m, 2 H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 175.4, 156.0, 141.8, 135.7, 133.8, 131.1, 129.2, 128.8, 128.0, 48.1, 42.6; HRMS (ESI) *m/z* calcd for $C_{11}H_{10}N_3O_2$([M+H]$^+$) 216.0773, found 216.0759.

5

DAm-Azide (CDCl3): Azidoethylcompound



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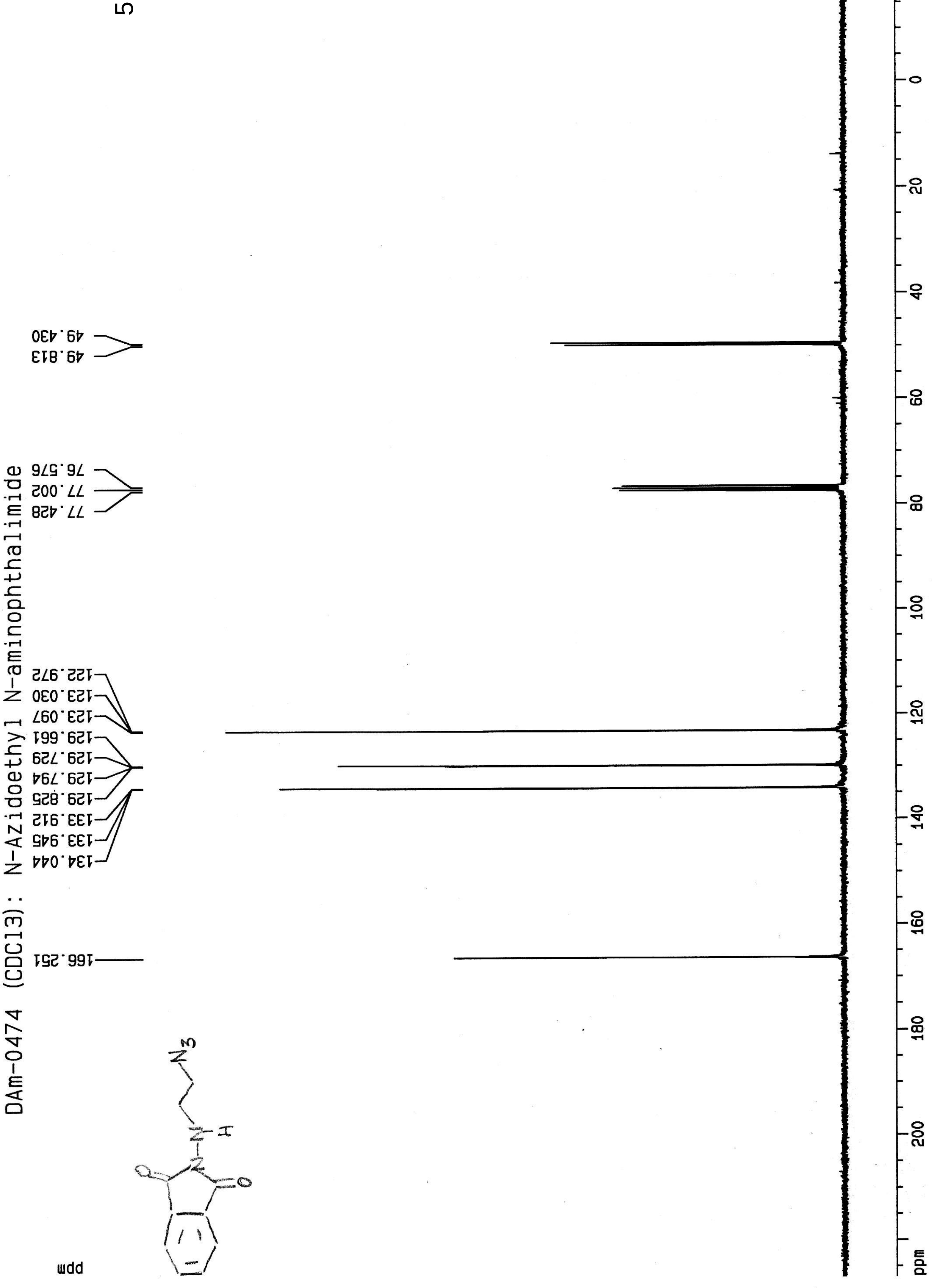
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123.097
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ppm
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N
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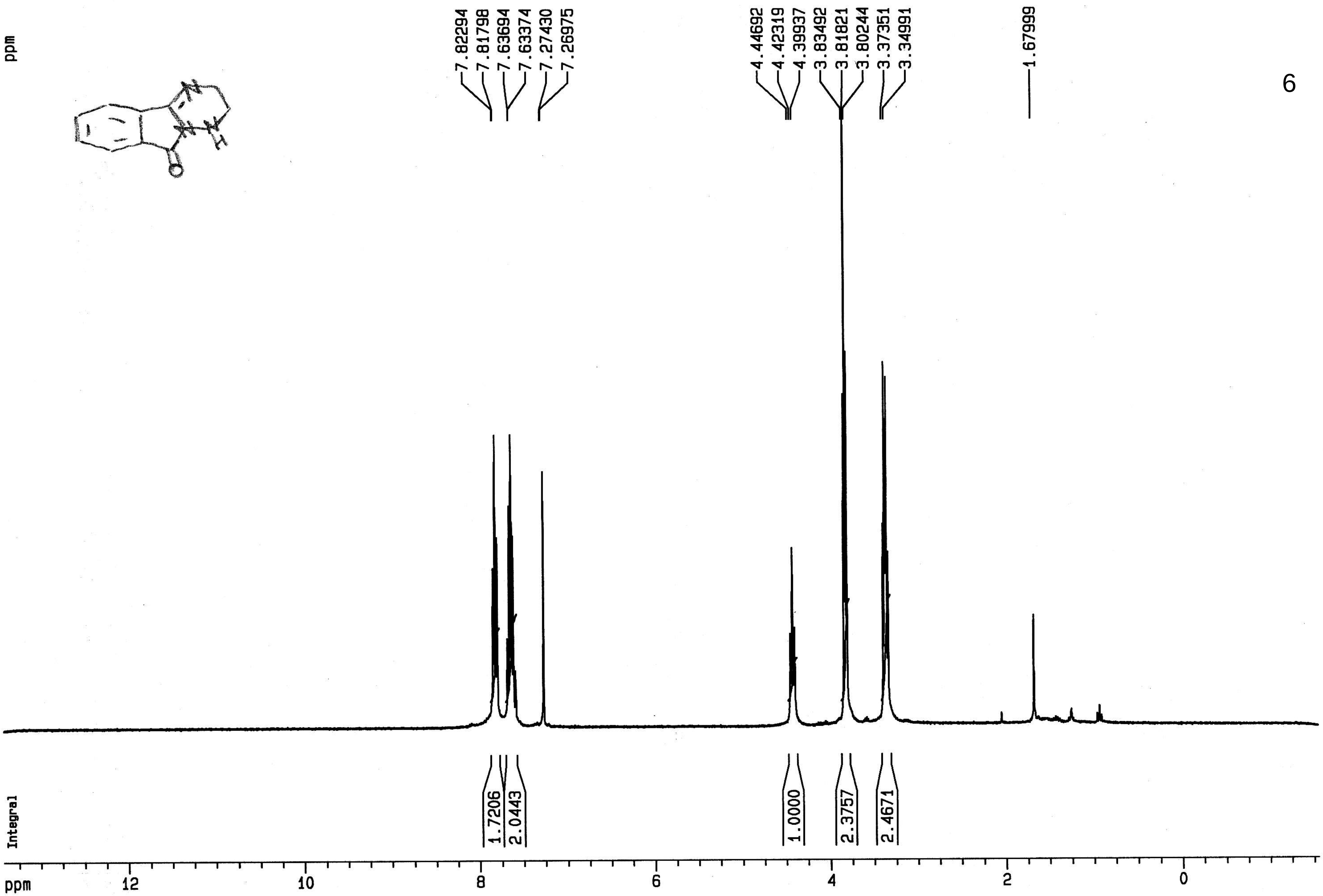
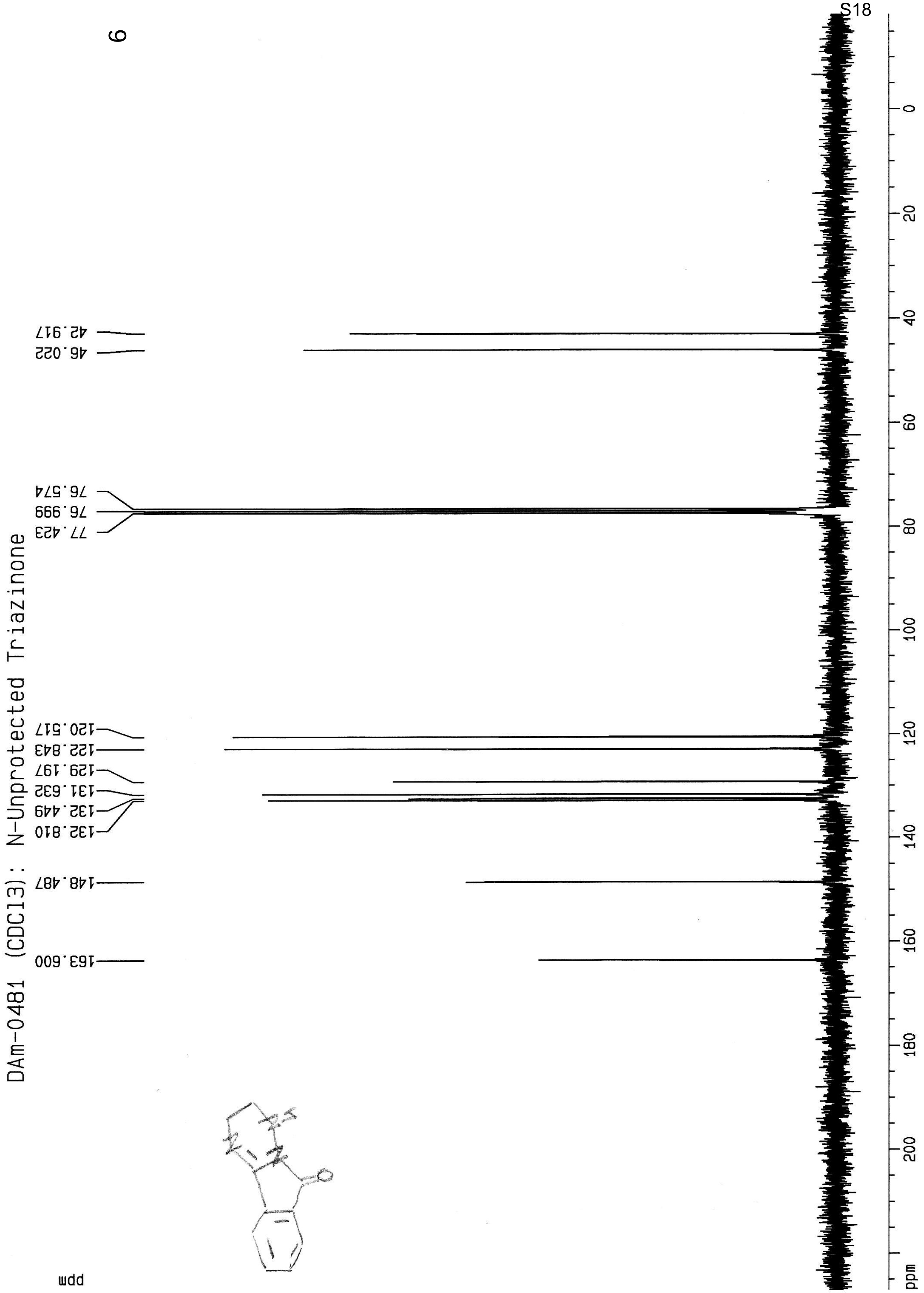
160
140
120
100
80
60
40
20
0

DAm-0481 (CDCl3): Unprotected Triazinone



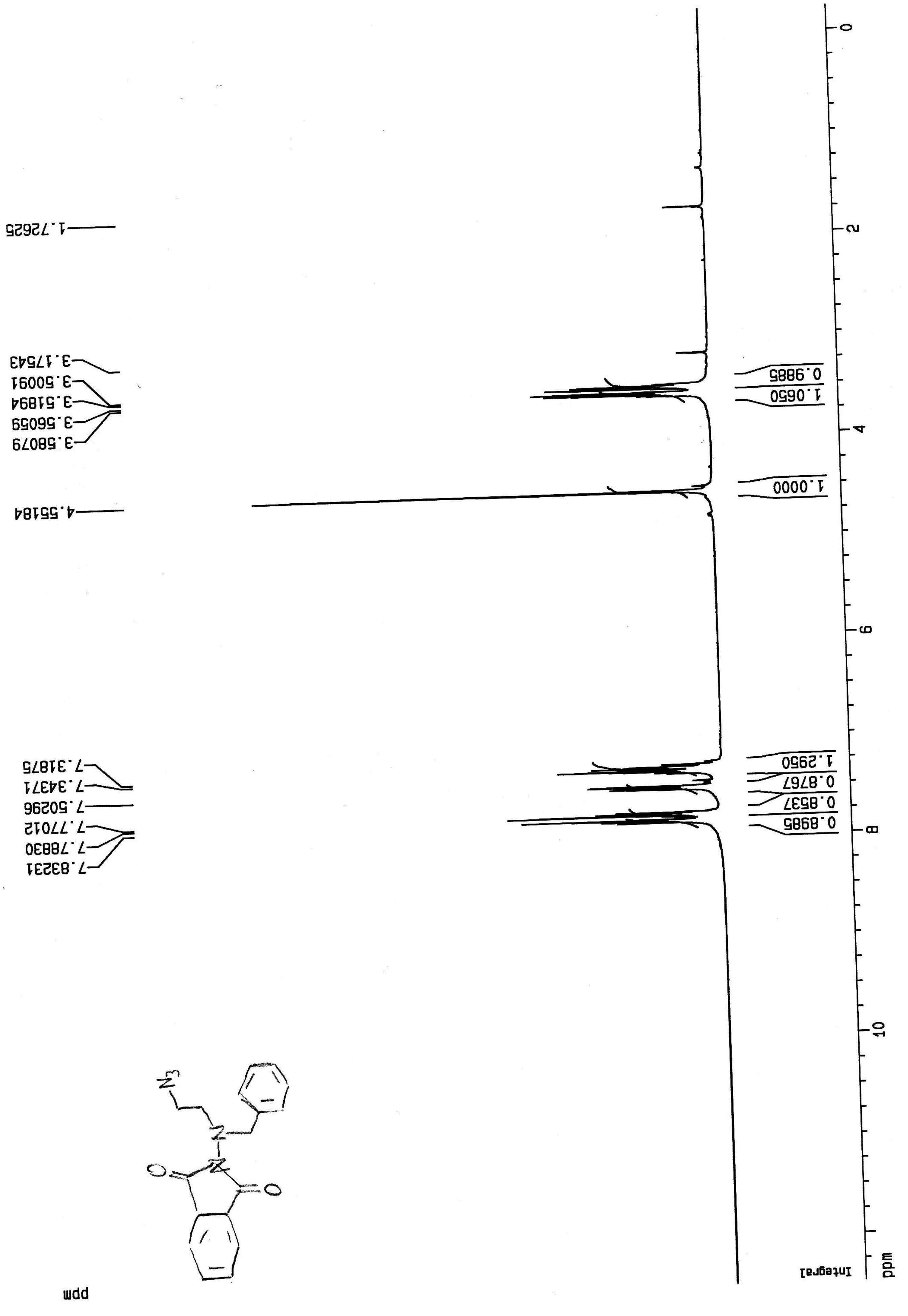
6

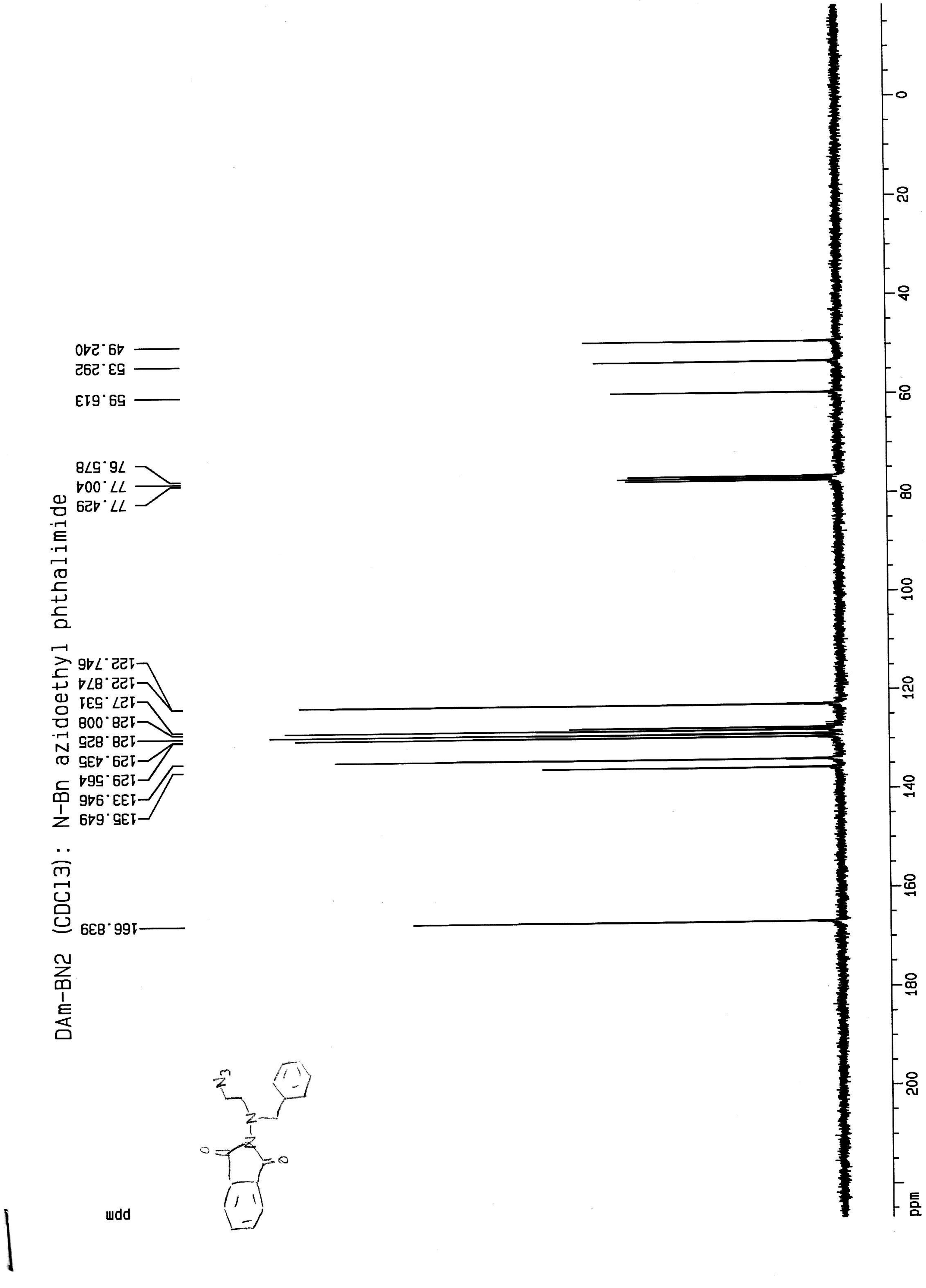


6

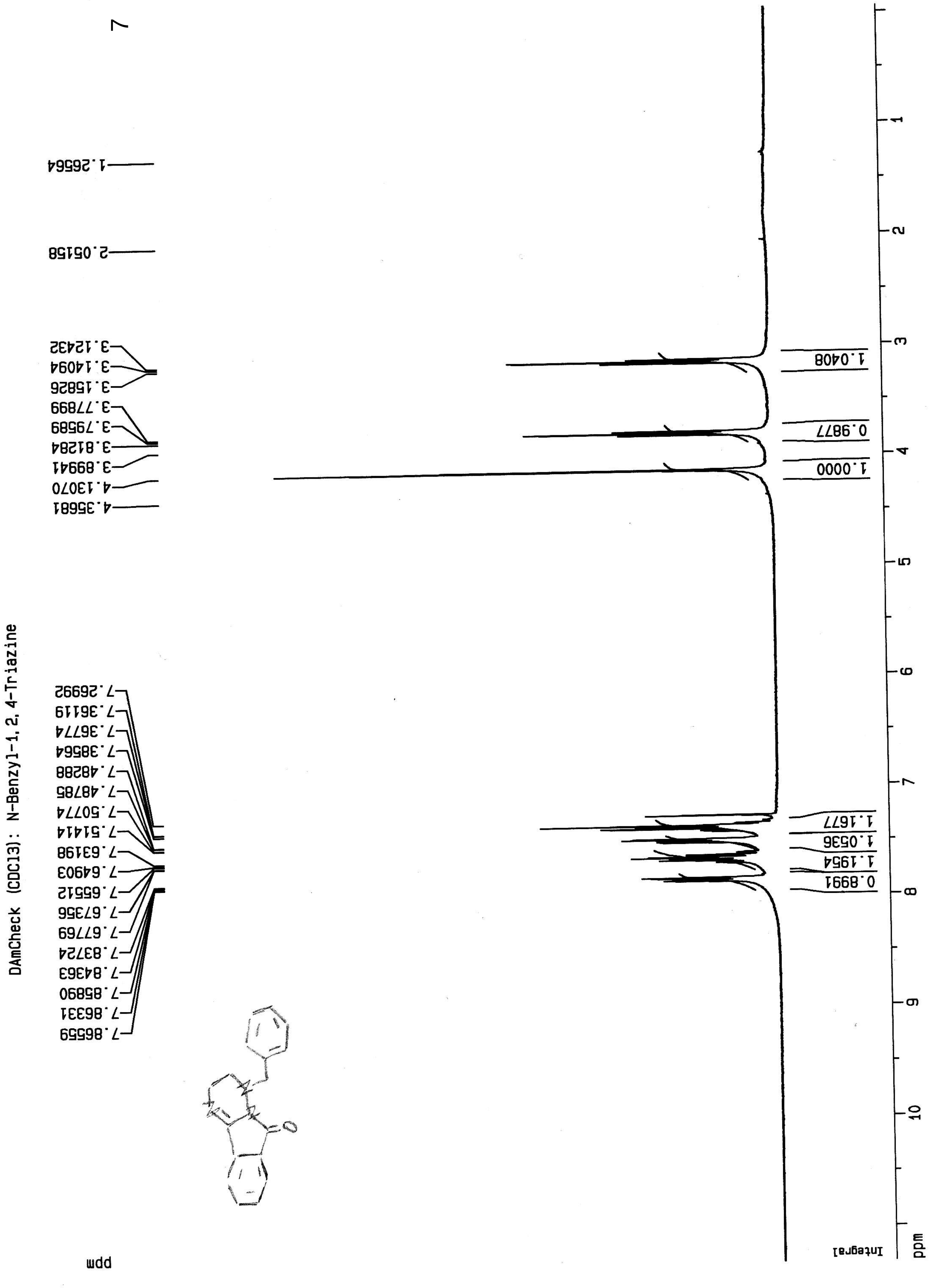
DAm-0481 (CDCl3): N-Unprotected Triazinone
42.917
46.022
76.574
76.999
77.423
120.517
122.843
129.197
131.632
132.449
132.810
148.487
163.600
ppm
0
20
40
60
80
100
120
140
160
180
200
ppm

DAm-BN1 (CDCl3): N-Bn azidoethyl phthalimide

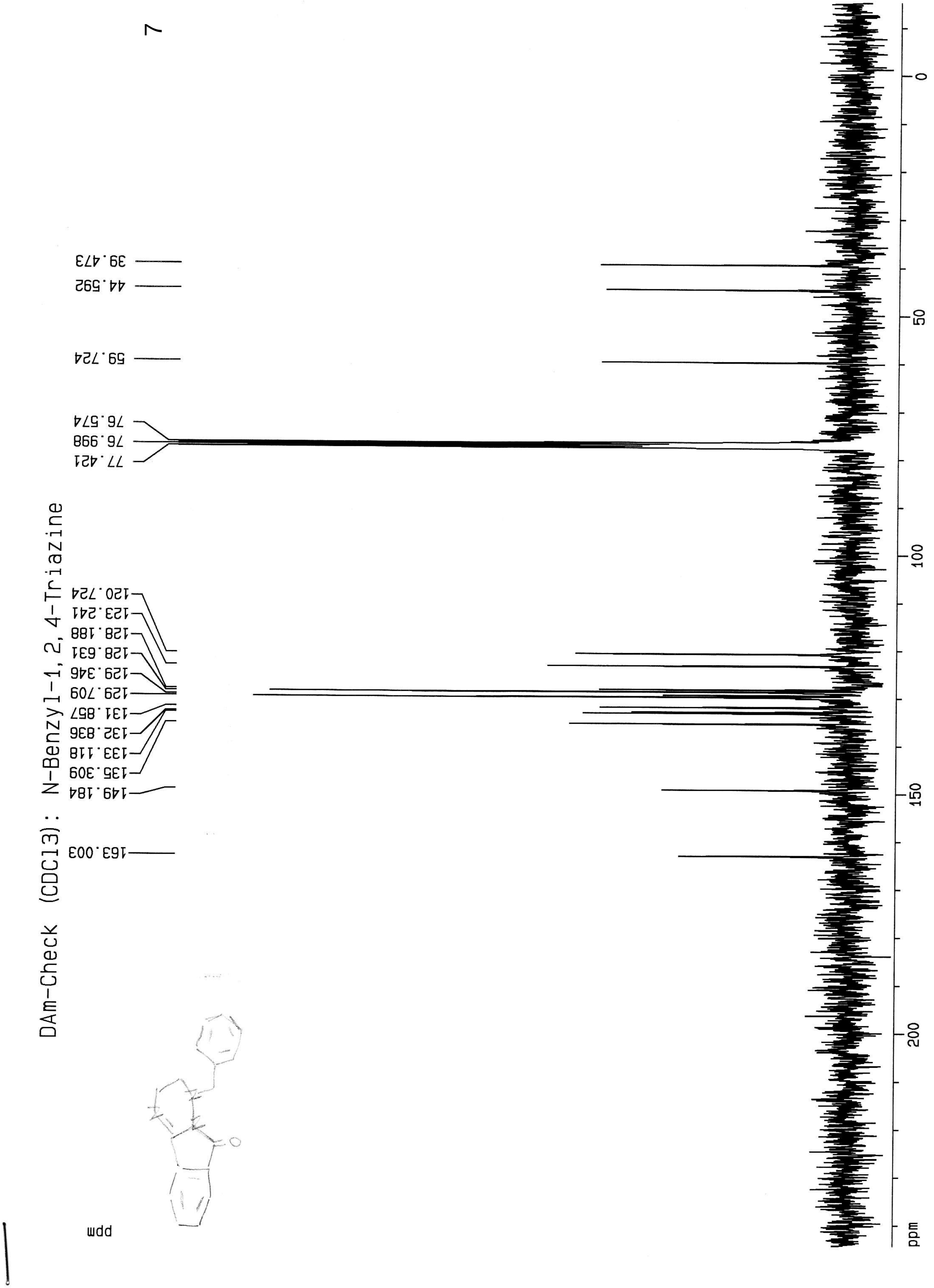


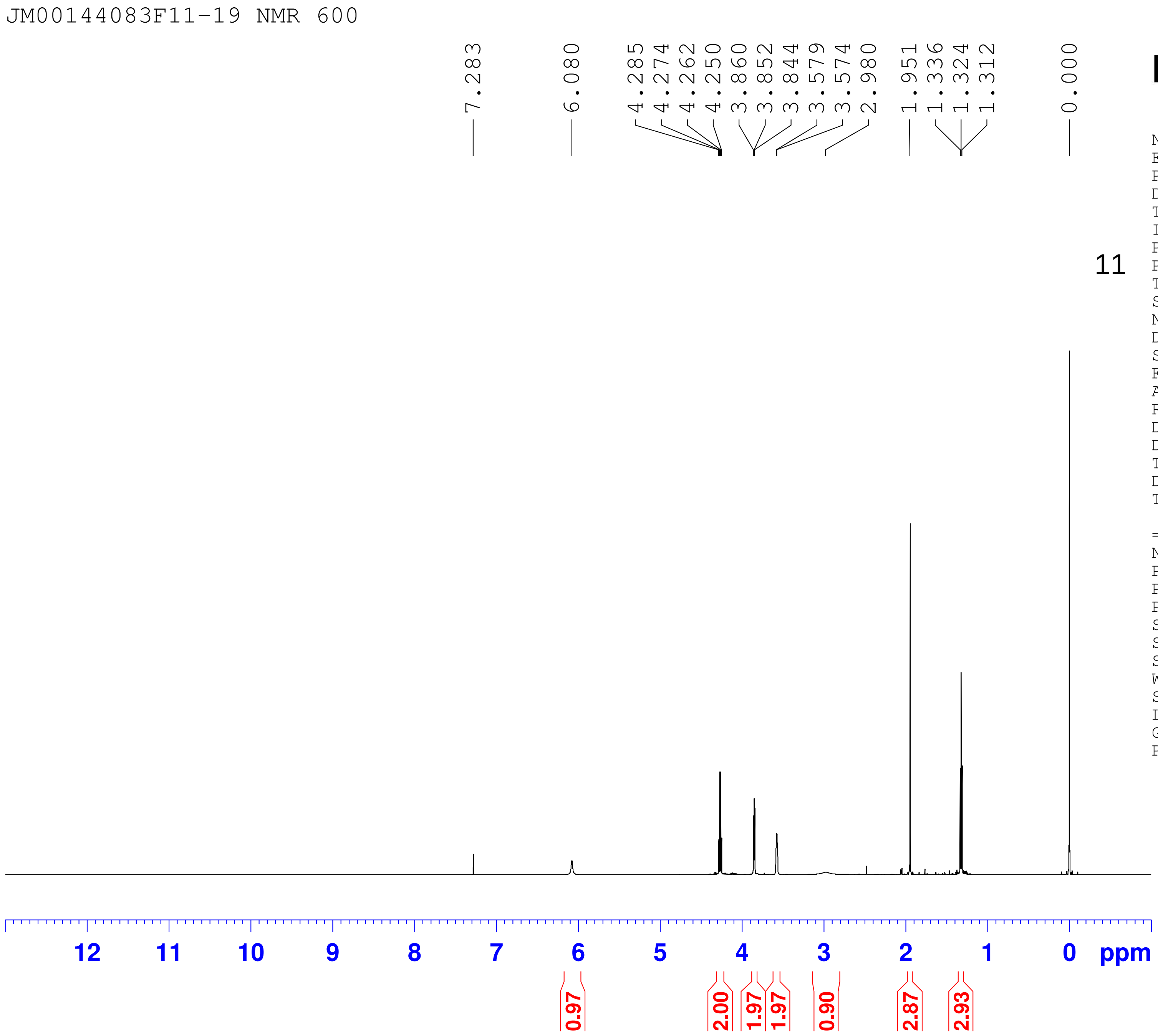

DAm-BN2 (CDCl3): N-Bn azidoethyl phthalimide
49.240
53.292
59.613
76.578
77.004
77.429
122.746
122.874
127.531
128.008
128.825
129.435
129.564
133.946
135.649
166.839
ppm
0
20
40
60
80
100
120
140
160
180
200
ppm
N3
N
O
O

7


DAmCheck (CDCl3): N-Benzyl-1,2,4-Triazine
1.26564
2.05158
3.12432
3.14094
3.15826
3.77899
3.79589
3.81284
3.89941
4.13070
4.35681
7.26992
7.36119
7.36774
7.38564
7.48288
7.48785
7.50774
7.51414
7.63198
7.64903
7.65512
7.67356
7.67769
7.83724
7.84363
7.85890
7.86331
7.86559
1.0408
0.9977
1.0000
1.1677
1.0536
1.1954
0.8991
Integral
1
2
3
4
5
6
7
8
9
10
ppm
ppm

7

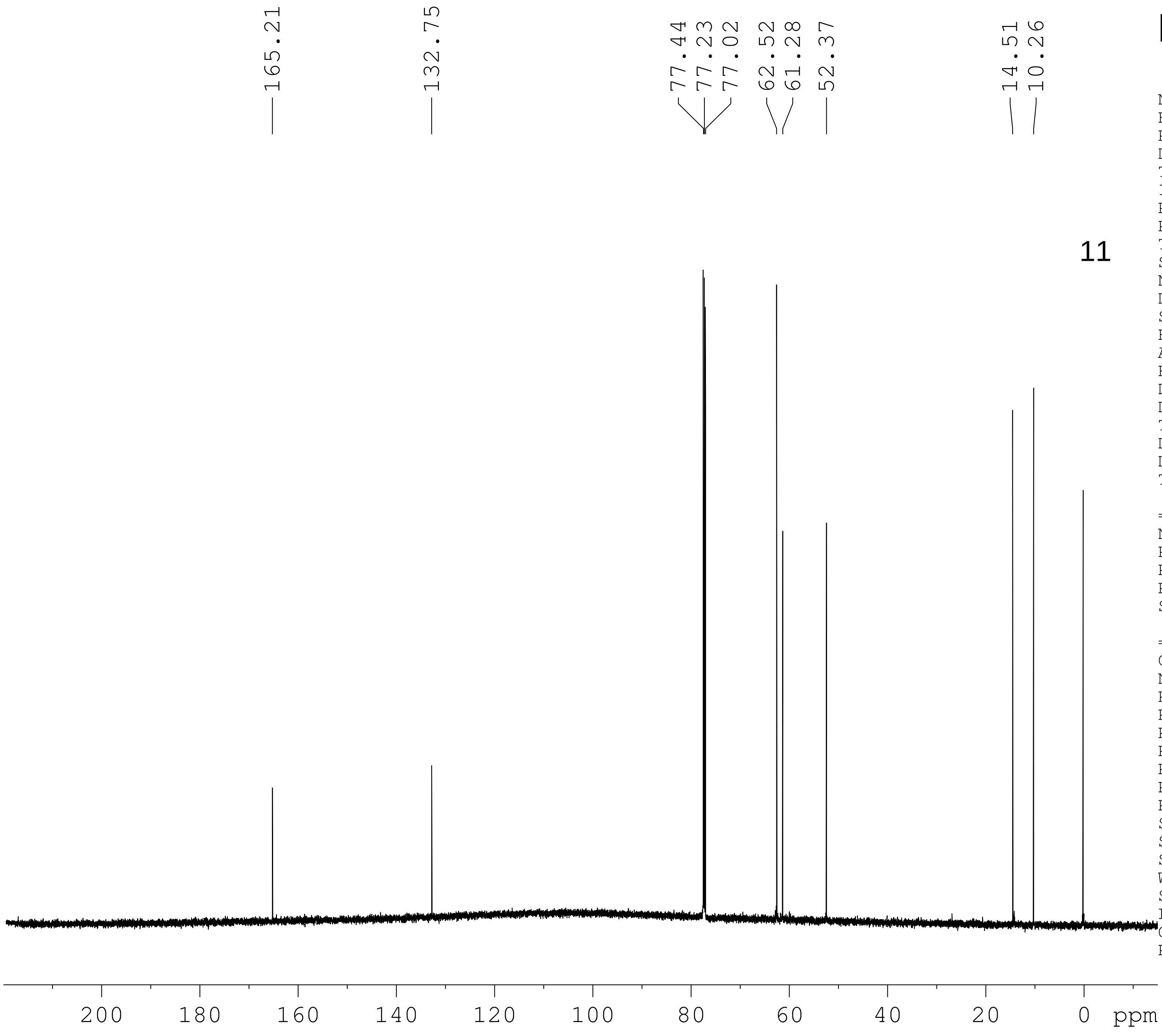

DAm-Check (CDCl3): N-Benzyl-1, 2, 4-Triazine
39.473
44.592
59.724
76.574
76.998
77.421
120.724
123.241
128.188
128.631
129.346
129.709
131.857
132.836
133.118
135.309
149.184
163.003
ppm
0
50
100
150
200
ppm



11

```
NAME       JM00144083F11-19
EXPNO                     1
PROCNO                    1
Date_              20090103
Time                  18.07
INSTRUM               spect
PROBHD     5 mm CPTCI 1H-
PULPROG                zg30
TD                    65536
SOLVENT               CDCl3
NS                       32
DS                        2
SWH               12376.237 Hz
FIDRES             0.188846 Hz
AQ                2.6477449 sec
RG                     28.5
DW                   40.400 usec
DE                     6.50 usec
TE                    298.0 K
D1               1.00000000 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                     1H
P1                     8.00 usec
PL1                    4.00 dB
PL1W             7.00000000 W
SFO1            600.1337060 MHz
SI                    32768
SF              600.1299969 MHz
WDW                      EM
SSB                       0
LB                     0.30 Hz
GB                        0
PC                     1.00
```

JM00144083F11-19 NMR 600

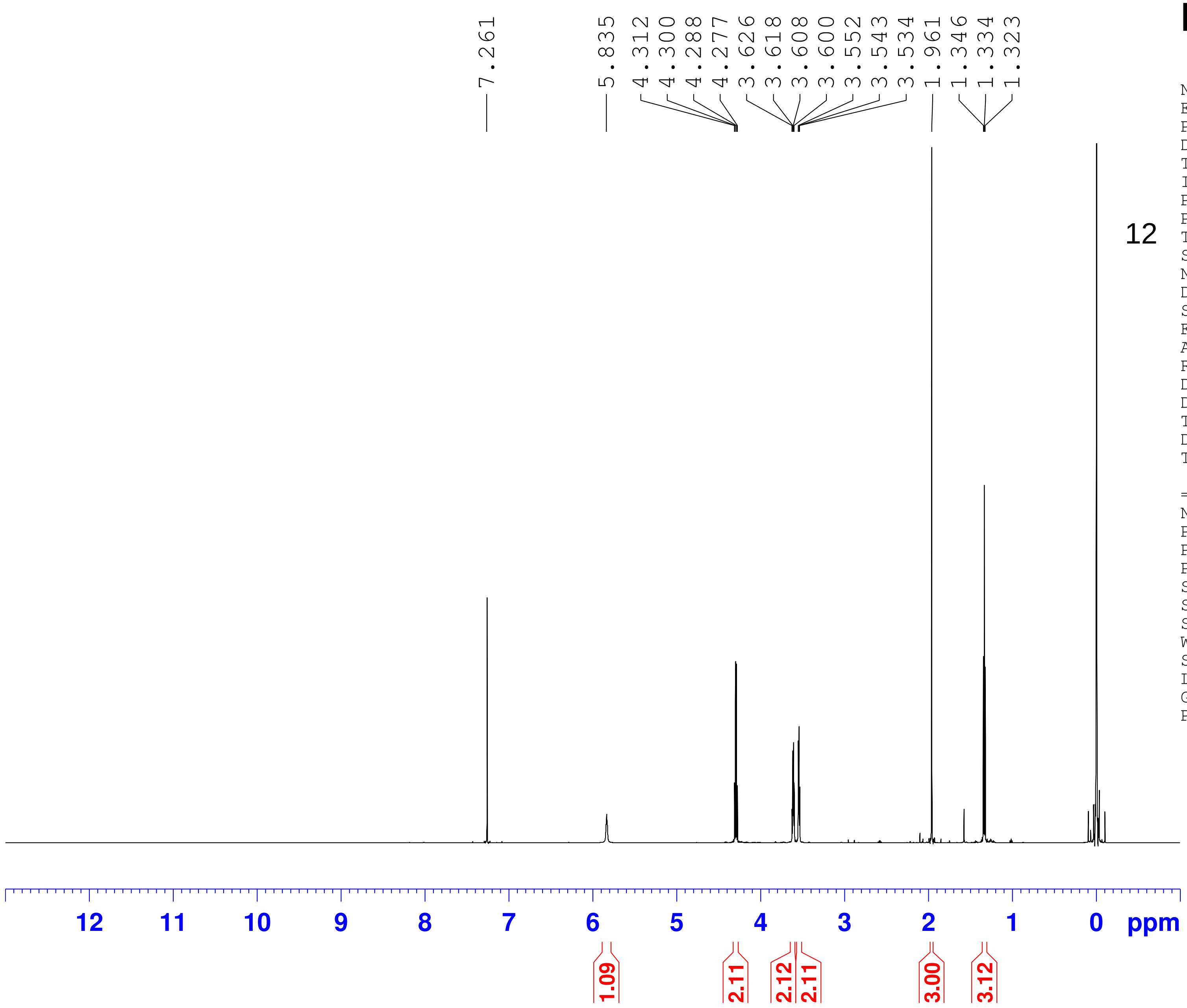


11

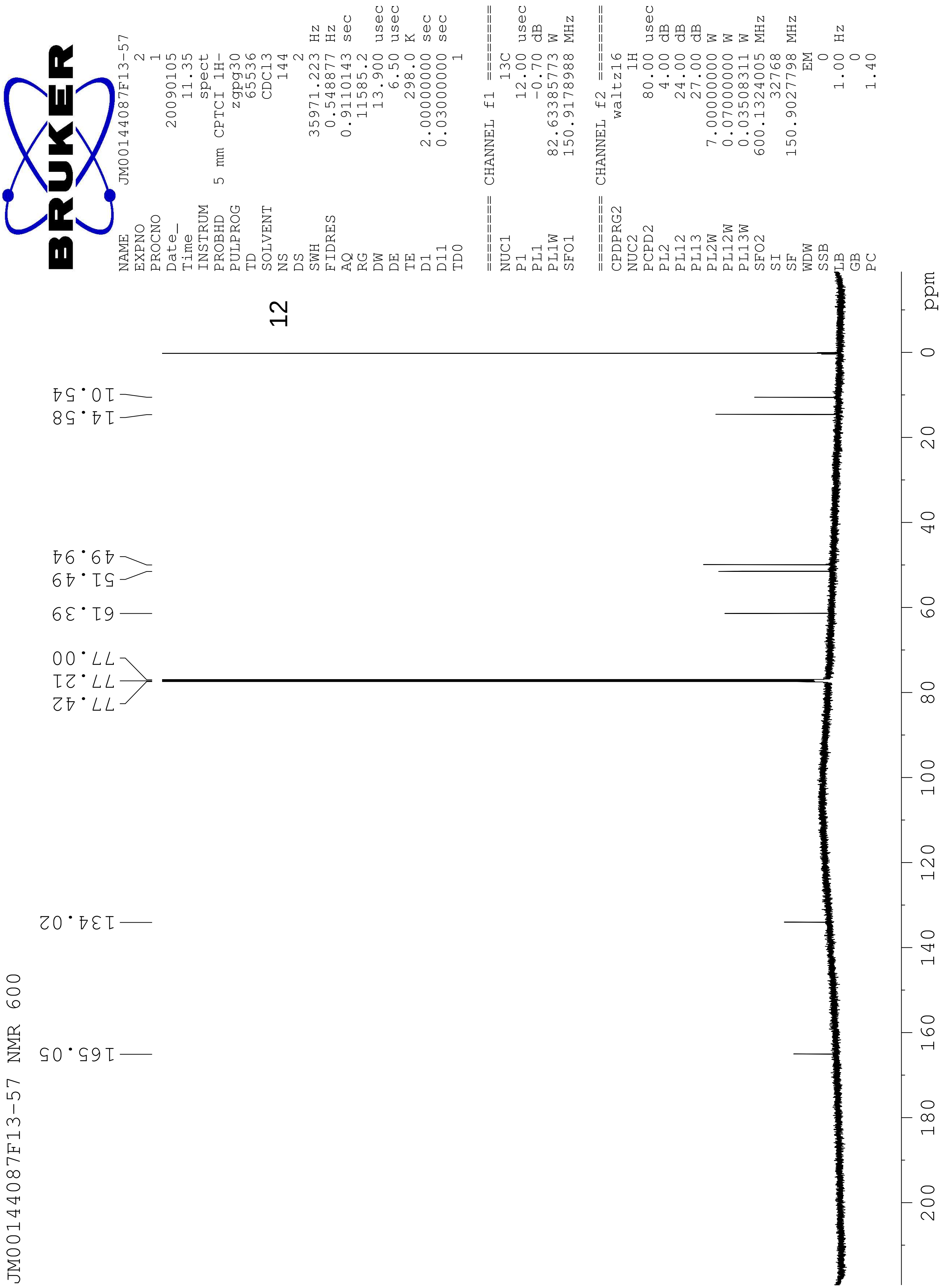
```
NAME       JM00144083F11-19
EXPNO                     2
PROCNO                    1
Date_              20090103
Time                  18.14
INSTRUM               spect
PROBHD     5 mm CPTCI 1H-
PULPROG             zgpg30
TD                    65536
SOLVENT               CDCl3
NS                       73
DS                        2
SWH               35971.223 Hz
FIDRES             0.548877 Hz
AQ                0.9110143 sec
RG                    13004
DW                   13.900 usec
DE                     6.50 usec
TE                    298.0 K
D1               2.00000000 sec
D11              0.03000000 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                    13C
P1                    12.00 usec
PL1                   -0.70 dB
PL1W            82.63385773 W
SFO1            150.9178988 MHz

======== CHANNEL f2 ========
CPDPRG2             waltz16
NUC2                     1H
PCPD2                 80.00 usec
PL2                    4.00 dB
PL12                  24.00 dB
PL13                  27.00 dB
PL2W             7.00000000 W
PL12W            0.07000000 W
PL13W            0.03508311 W
SFO2            600.1324005 MHz
SI                    32768
SF              150.9027801 MHz
WDW                      EM
SSB                       0
LB                     1.00 Hz
GB                        0
PC                     1.40
```

JM00144087F13-57 NMR 600

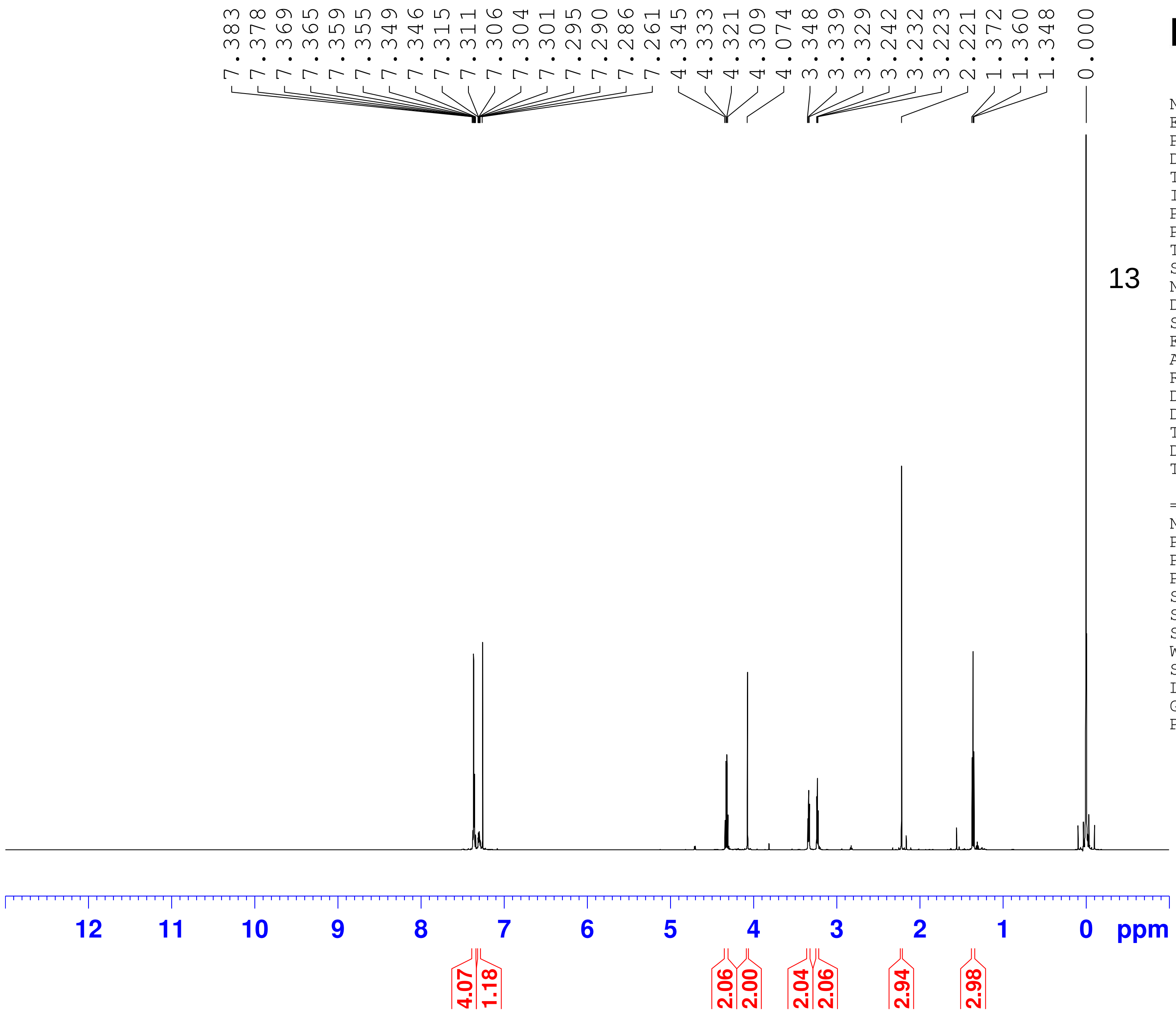
12



```
NAME       JM00144087F13-57
EXPNO                     1
PROCNO                    1
Date_              20090105
Time                  11.25
INSTRUM               spect
PROBHD   5 mm CPTCI 1H-
PULPROG                zg30
TD                    65536
SOLVENT               CDCl3
NS                       48
DS                        2
SWH            12376.237 Hz
FIDRES          0.188846 Hz
AQ             2.6477449 sec
RG                       64
DW               40.400 usec
DE                 6.50 usec
TE                298.0 K
D1           1.00000000 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                     1H
P1                 8.00 usec
PL1                4.00 dB
PL1W         7.00000000 W
SFO1        600.1337060 MHz
SI                    32768
SF          600.1300102 MHz
WDW                      EM
SSB                       0
LB                 0.30 Hz
GB                        0
PC                     1.00
```

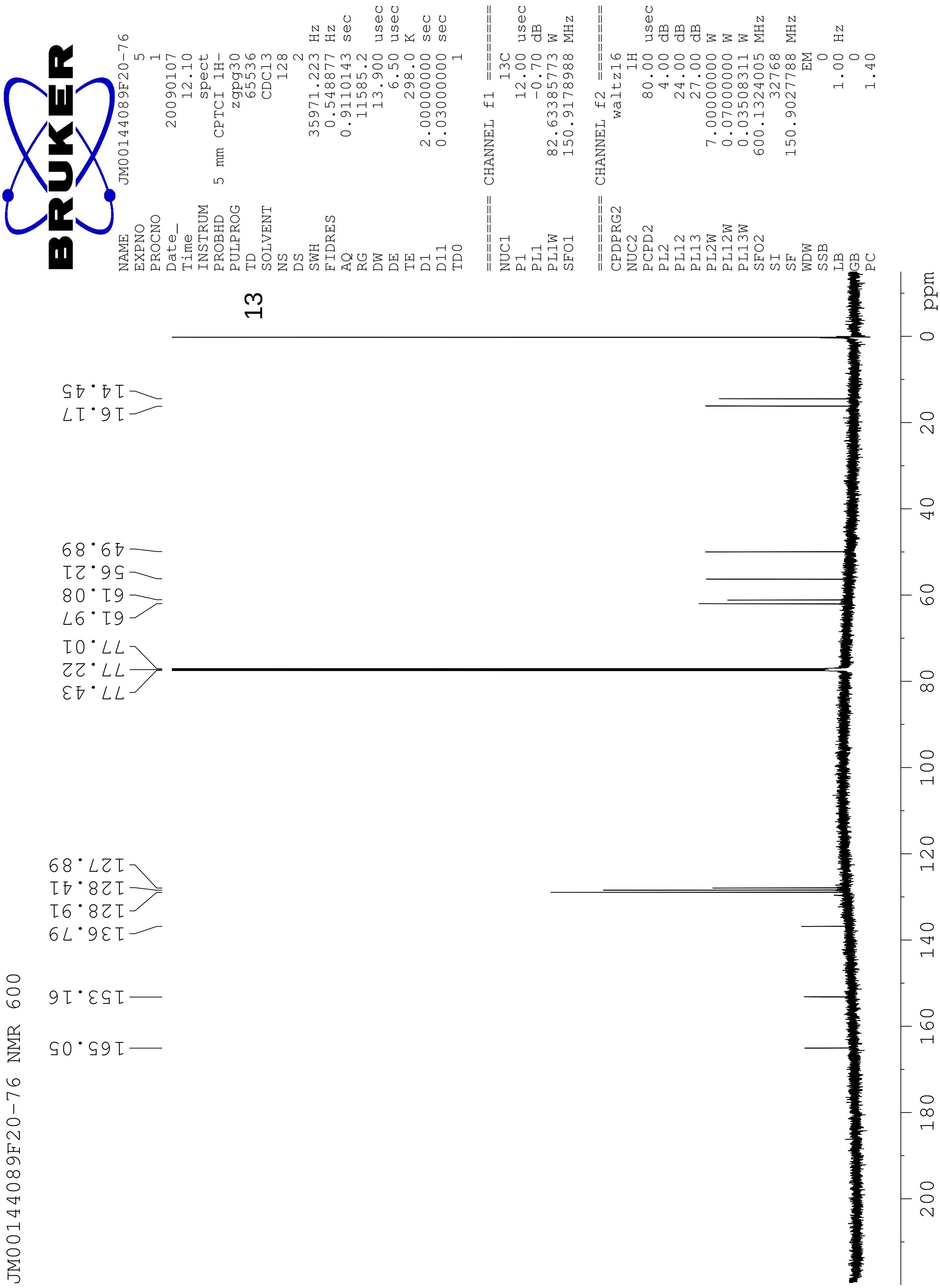

NAME JM00144087F13-57
EXPNO 2
PROCNO 1
Date_ 20090105
Time 11.35
INSTRUM spect
PROBHD 5 mm CPTCI 1H-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 144
DS 2
SWH 35971.223 Hz
FIDRES 0.548877 Hz
AQ 0.9110143 sec
RG 11585.2
DW 13.900 usec
DE 6.50 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 12.00 usec
PL1 -0.70 dB
PL1W 82.63385773 W
SFO1 150.9178988 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 4.00 dB
PL12 24.00 dB
PL13 27.00 dB
PL2W 7.00000000 W
PL12W 0.07000000 W
PL13W 0.03508311 W
SFO2 600.1324005 MHz
SI 32768
SF 150.9027798 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
12
10.54
14.58
49.94
51.49
61.39
77.00
77.21
77.42
134.02
165.05
ppm
0
20
40
60
80
100
120
140
160
180
200
JM00144087F13-57 NMR 600

JM00144089F20-76 NMR 600



13

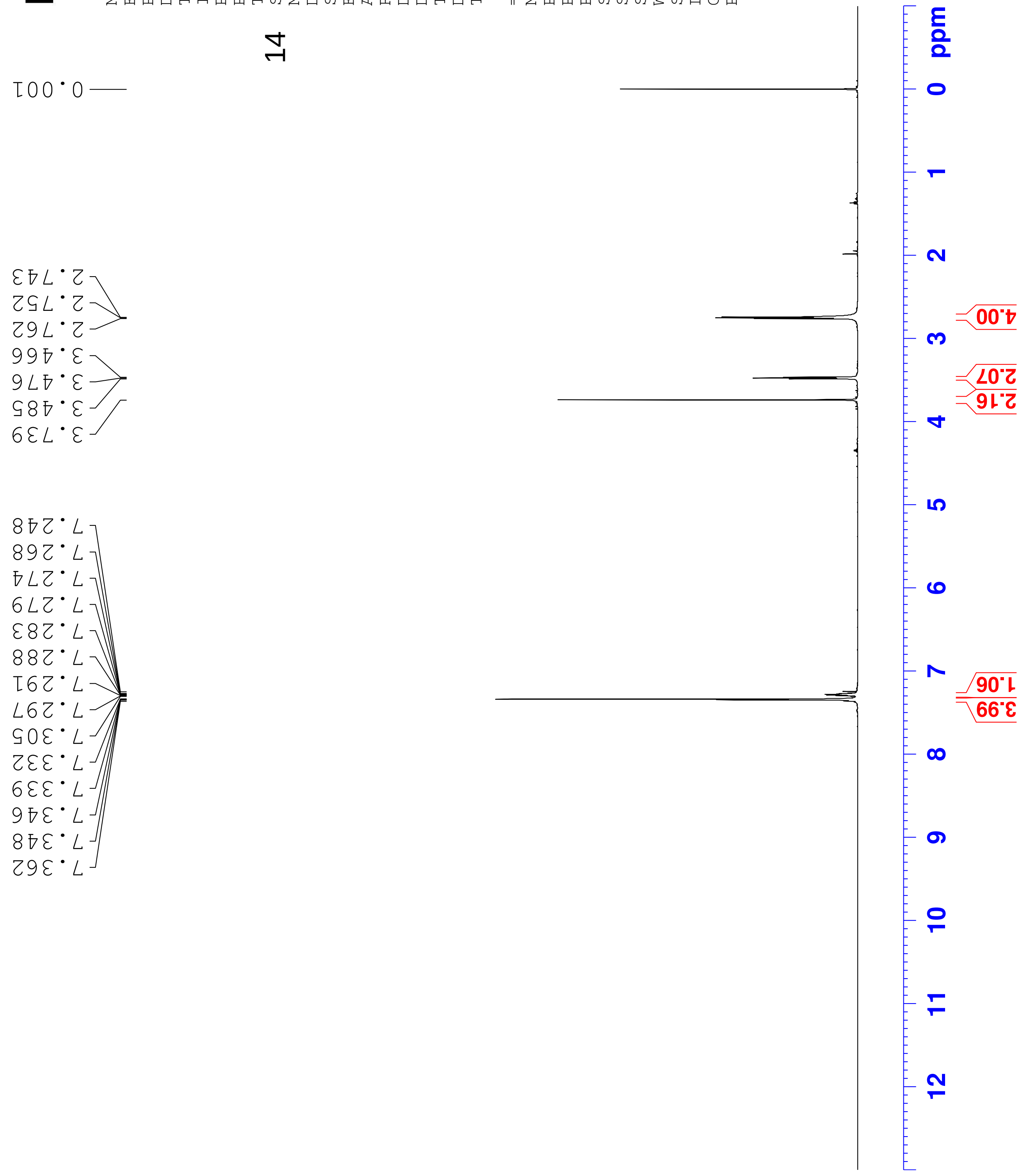
```
NAME          JM00144089F20-76
EXPNO                    4
PROCNO                   1
Date_             20090107
Time                 12.01
INSTRUM              spect
PROBHD     5 mm CPTCI 1H-
PULPROG               zg30
TD                   65536
SOLVENT              CDCl3
NS                      32
DS                       2
SWH              12376.237 Hz
FIDRES            0.188846 Hz
AQ               2.6477449 sec
RG                      64
DW                  40.400 usec
DE                    6.50 usec
TE                   298.0 K
D1              1.00000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                    1H
P1                    8.00 usec
PL1                   4.00 dB
PL1W            7.00000000 W
SFO1           600.1337060 MHz
SI                   32768
SF             600.1300103 MHz
WDW                     EM
SSB                      0
LB                    0.30 Hz
GB                       0
PC                    1.00
```

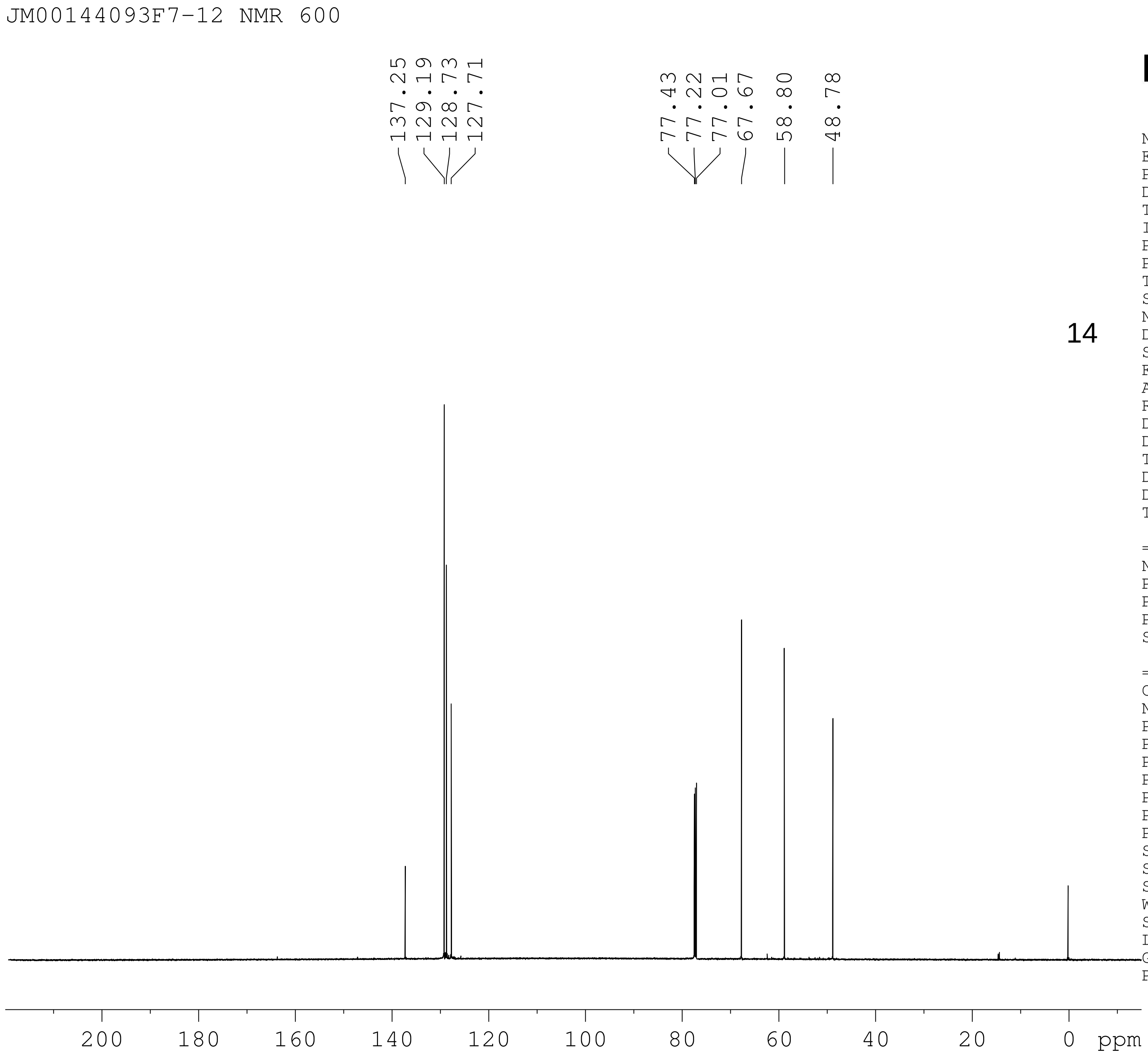

JM00144089F20-76 NMR 600
13
14.45
16.17
49.89
56.21
61.08
61.97
77.01
77.22
77.43
127.89
128.41
128.91
136.79
153.16
165.05
NAME JM00144089F20-76
EXPNO 5
PROCNO 1
Date_ 20090107
Time 12.10
INSTRUM spect
PROBHD 5 mm CPTCI 1H-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 128
DS 2
SWH 35971.223 Hz
FIDRES 0.548877 Hz
AQ 0.9110143 sec
RG 11585.2
DW 13.900 usec
DE 6.50 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 12.00 usec
PL1 -0.70 dB
PL1W 82.63385773 W
SFO1 150.9178988 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 4.00 dB
PL12 24.00 dB
PL13 27.00 dB
PL2W 7.00000000 W
PL12W 0.07000000 W
PL13W 0.03508311 W
SFO2 600.1324005 MHz
SI 32768
SF 150.9027788 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
ppm
0
20
40
60
80
100
120
140
160
180
200

JM00144093F7-12 NMR 600

NAME	JM00144093F7-12
EXPNO	1
PROCNO	1
Date_	20090108
Time	16.12
INSTRUM	spect
PROBHD	5 mm CPTCI 1H-
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	48
DS	2
SWH	12376.237 Hz
FIDRES	0.188846 Hz
AQ	2.6477449 sec
RG	16
DW	40.400 usec
DE	6.50 usec
TE	298.0 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	8.00 usec
PL1	4.00 dB
PL1W	7.00000000 W
SFO1	600.1337060 MHz
SI	32768
SF	600.1300180 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

14

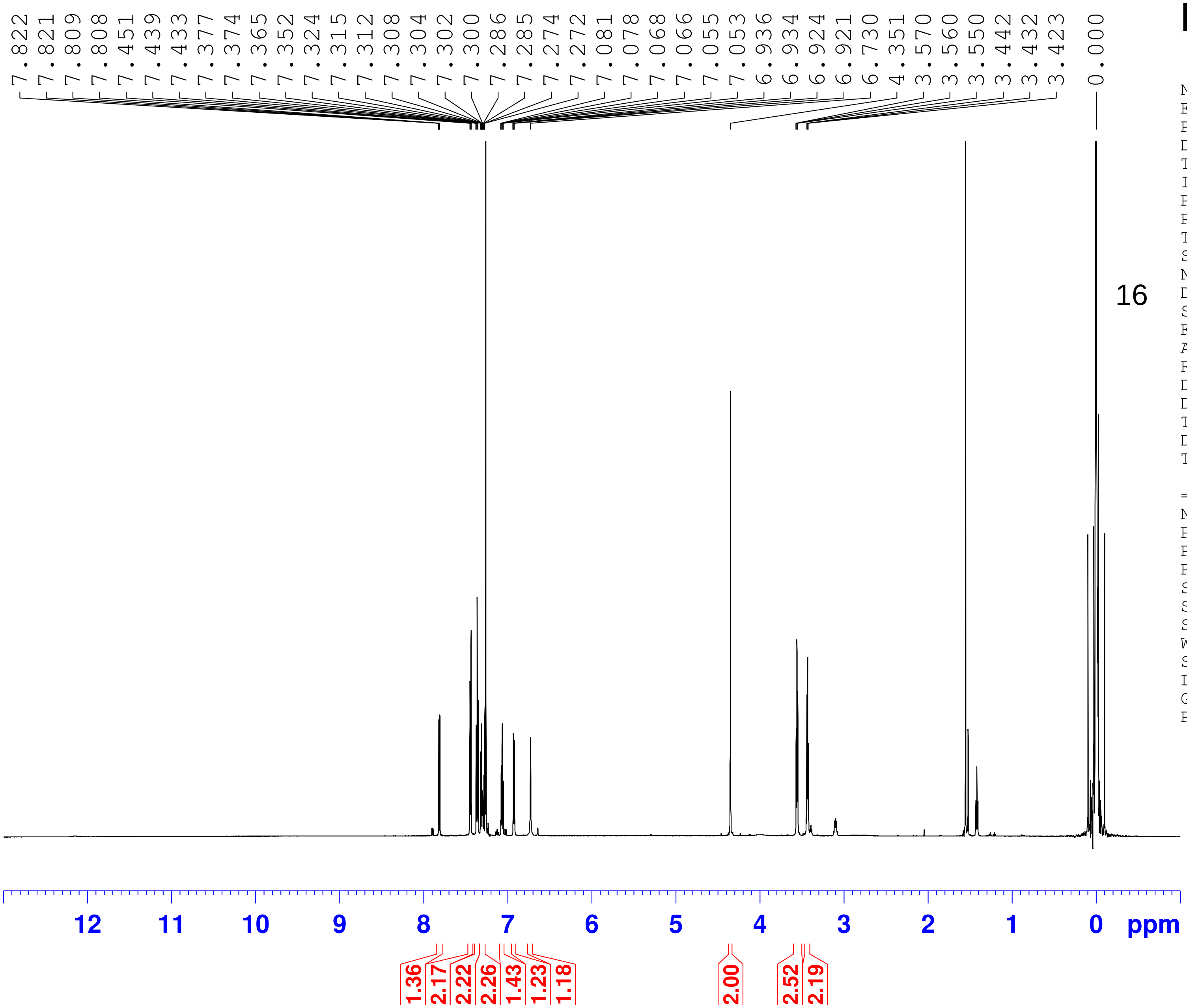




14

```
NAME       JM00144093F7-12
EXPNO                    2
PROCNO                   1
Date_             20090108
Time                 16.22
INSTRUM              spect
PROBHD     5 mm CPTCI 1H-
PULPROG            zgpg30
TD                   65536
SOLVENT              CDCl3
NS                     150
DS                       2
SWH              35971.223 Hz
FIDRES            0.548877 Hz
AQ               0.9110143 sec
RG                  9195.2
DW                  13.900 usec
DE                    6.50 usec
TE                   298.0 K
D1              2.00000000 sec
D11             0.03000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                   13C
P1                   12.00 usec
PL1                  -0.70 dB
PL1W           82.63385773 W
SFO1           150.9178988 MHz

======== CHANNEL f2 ========
CPDPRG2            waltz16
NUC2                    1H
PCPD2                80.00 usec
PL2                   4.00 dB
PL12                 24.00 dB
PL13                 27.00 dB
PL2W            7.00000000 W
PL12W           0.07000000 W
PL13W           0.03508311 W
SFO2           600.1324005 MHz
SI                   32768
SF             150.9027910 MHz
WDW                     EM
SSB                      0
LB                    1.00 Hz
GB                       0
PC                    1.40
```

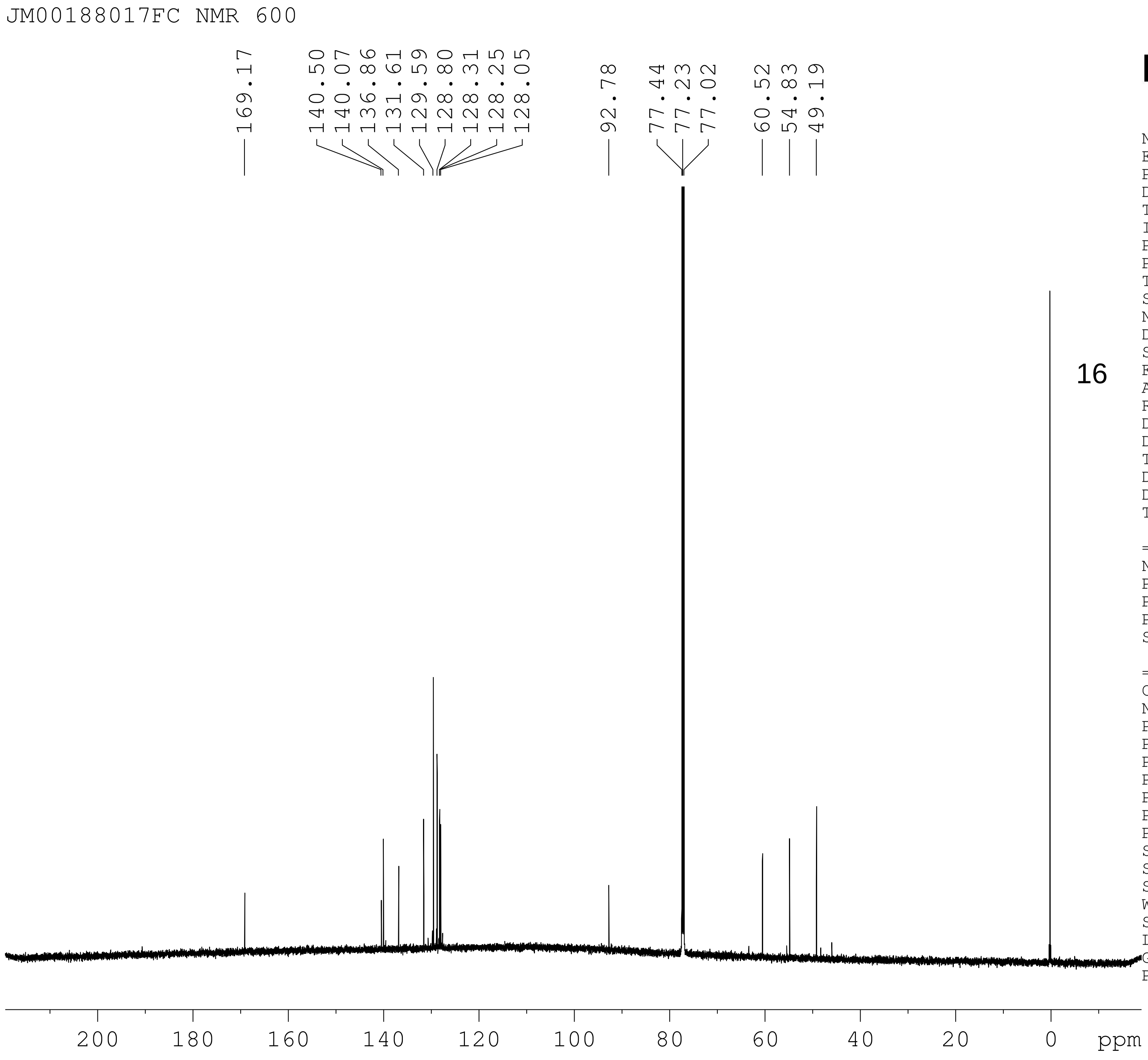
jm00188017fc nmr 600



16

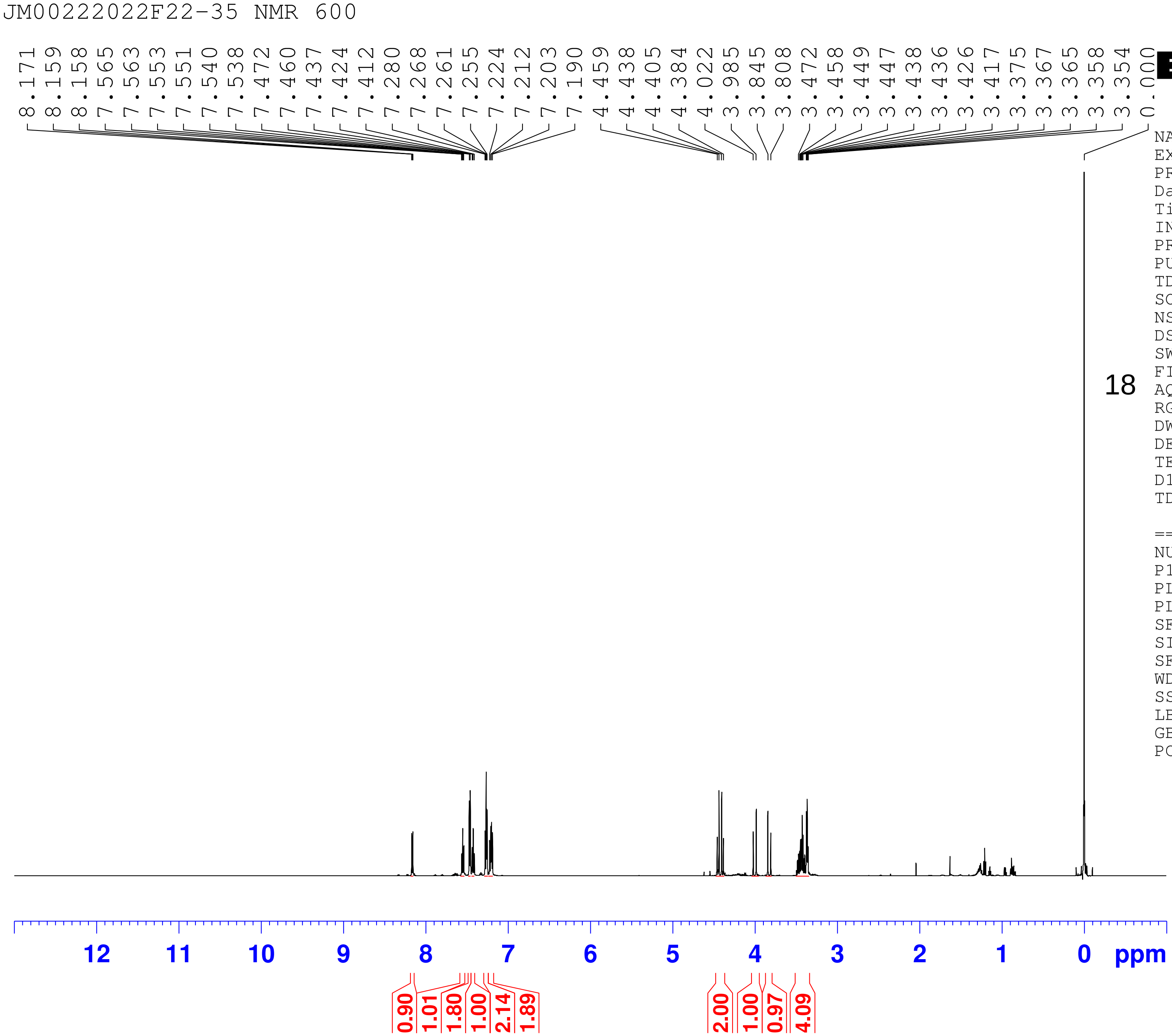
```
NAME          jm00188017fc
EXPNO                    1
PROCNO                   1
Date_             20090520
Time                 11.01
INSTRUM              spect
PROBHD   5 mm CPTCI 1H-
PULPROG               zg30
TD                   65536
SOLVENT              CDCl3
NS                      22
DS                       0
SWH              12376.237 Hz
FIDRES            0.188846 Hz
AQ               2.6477449 sec
RG                    71.8
DW                  40.400 usec
DE                    6.50 usec
TE                   298.0 K
D1              2.00000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                    1H
P1                    8.00 usec
PL1                   4.00 dB
PL1W            7.00000000 W
SFO1           600.1337060 MHz
SI                   32768
SF             600.1300098 MHz
WDW                     EM
SSB                      0
LB                    0.30 Hz
GB                       0
PC                    1.00
```

JM00188017FC NMR 600



16

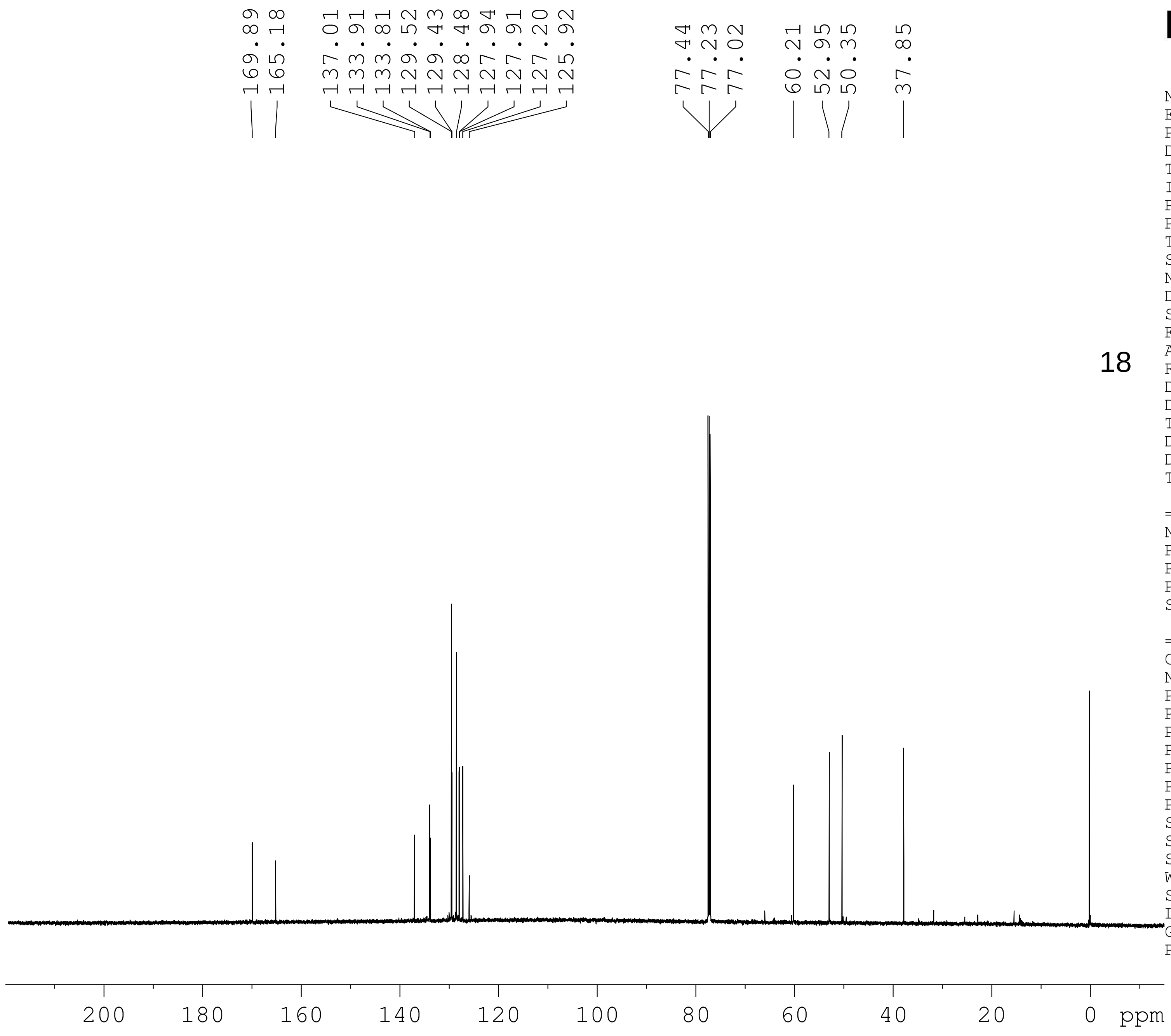
```
NAME         jm00188017fc
EXPNO                   5
PROCNO                  1
Date_            20090805
Time                11.41
INSTRUM             spect
PROBHD   5 mm CPTCI 1H-
PULPROG            zgpg30
TD                  65536
SOLVENT             CDCl3
NS                    300
DS                      2
SWH             35971.223 Hz
FIDRES           0.548877 Hz
AQ              0.9110143 sec
RG                  16384
DW                 13.900 usec
DE                   6.50 usec
TE                  298.0 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                  13C
P1                  12.00 usec
PL1                 -0.70 dB
PL1W          82.63385773 W
SFO1          150.9178988 MHz

======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                   1H
PCPD2               80.00 usec
PL2                  4.00 dB
PL12                24.00 dB
PL13                27.00 dB
PL2W           7.00000000 W
PL12W          0.07000000 W
PL13W          0.03508311 W
SFO2          600.1324005 MHz
SI                  32768
SF            150.9027784 MHz
WDW                    EM
SSB                     0
LB                   1.00 Hz
GB                      0
PC                   1.40
```



18

```
NAME       JM00222022F22-35
EXPNO                     3
PROCNO                    1
Date_              20090930
Time                  11.42
INSTRUM               spect
PROBHD     5 mm CPTCI 1H-
PULPROG                zg30
TD                    65536
SOLVENT               CDCl3
NS                       24
DS                        2
SWH               12376.237 Hz
FIDRES             0.188846 Hz
AQ                2.6477449 sec
RG                     35.9
DW                   40.400 usec
DE                     6.50 usec
TE                    298.0 K
D1               1.00000000 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                     1H
P1                     8.00 usec
PL1                    4.00 dB
PL1W             7.00000000 W
SFO1            600.1337060 MHz
SI                    32768
SF              600.1300098 MHz
WDW                      EM
SSB                       0
LB                     0.30 Hz
GB                        0
PC                     1.00
```

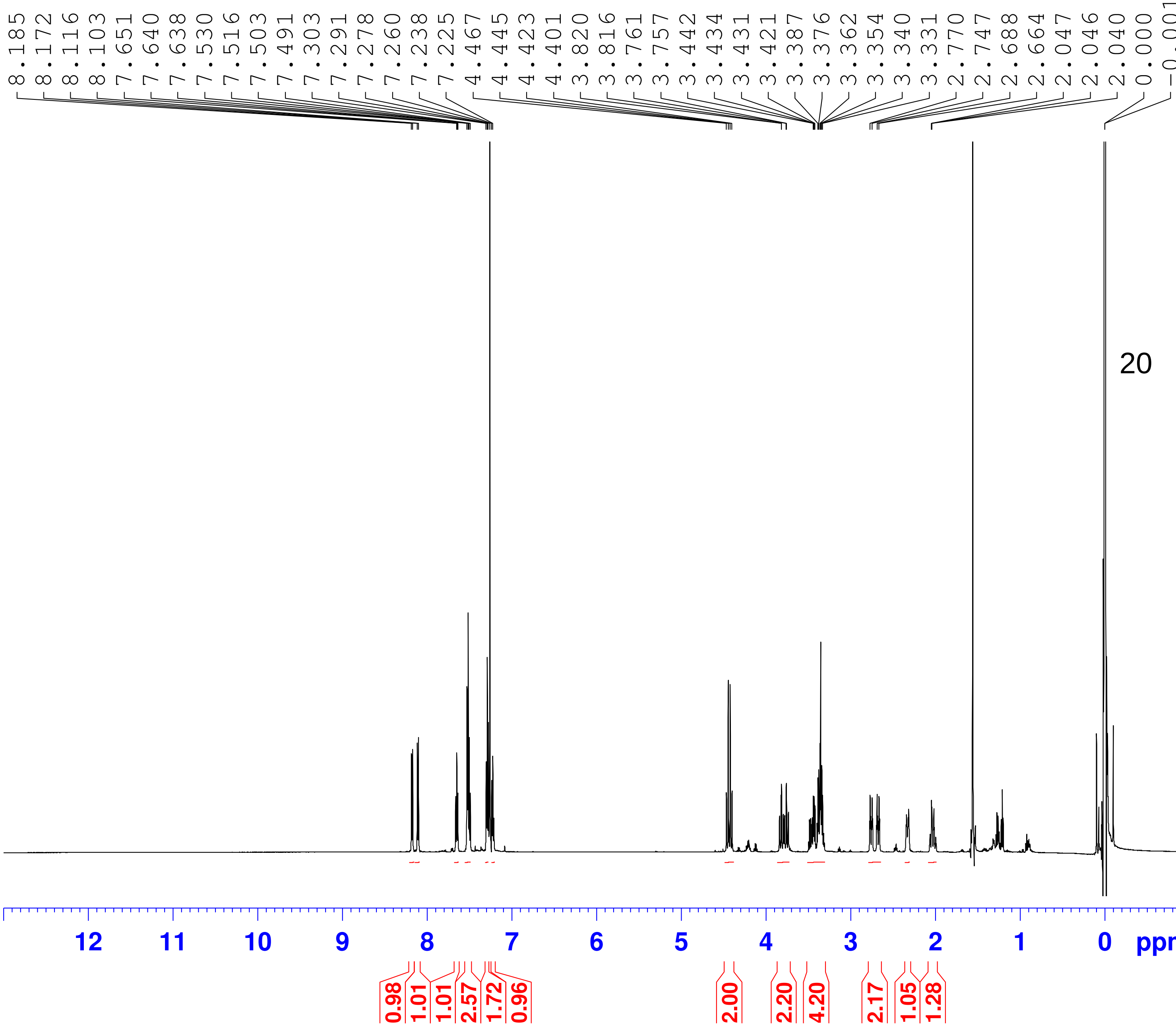
JM00222022F22-35 NMR 600



18

```
NAME       JM00222022F22-35
EXPNO                     4
PROCNO                    1
Date_              20090930
Time                  11.47
INSTRUM               spect
PROBHD   5 mm CPTCI 1H-
PULPROG             zgpg30
TD                    65536
SOLVENT               CDCl3
NS                      110
DS                        2
SWH              35971.223 Hz
FIDRES            0.548877 Hz
AQ               0.9110143 sec
RG                  11585.2
DW                   13.900 usec
DE                     6.50 usec
TE                    298.0 K
D1               2.00000000 sec
D11              0.03000000 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                    13C
P1                    12.00 usec
PL1                   -0.70 dB
PL1W            82.63385773 W
SFO1            150.9178988 MHz

======== CHANNEL f2 ========
CPDPRG2             waltz16
NUC2                     1H
PCPD2                 80.00 usec
PL2                    4.00 dB
PL12                  24.00 dB
PL13                  27.00 dB
PL2W             7.00000000 W
PL12W            0.07000000 W
PL13W            0.03508311 W
SFO2            600.1324005 MHz
SI                    32768
SF              150.9027805 MHz
WDW                      EM
SSB                       0
LB                     1.00 Hz
GB                        0
PC                     1.40
```

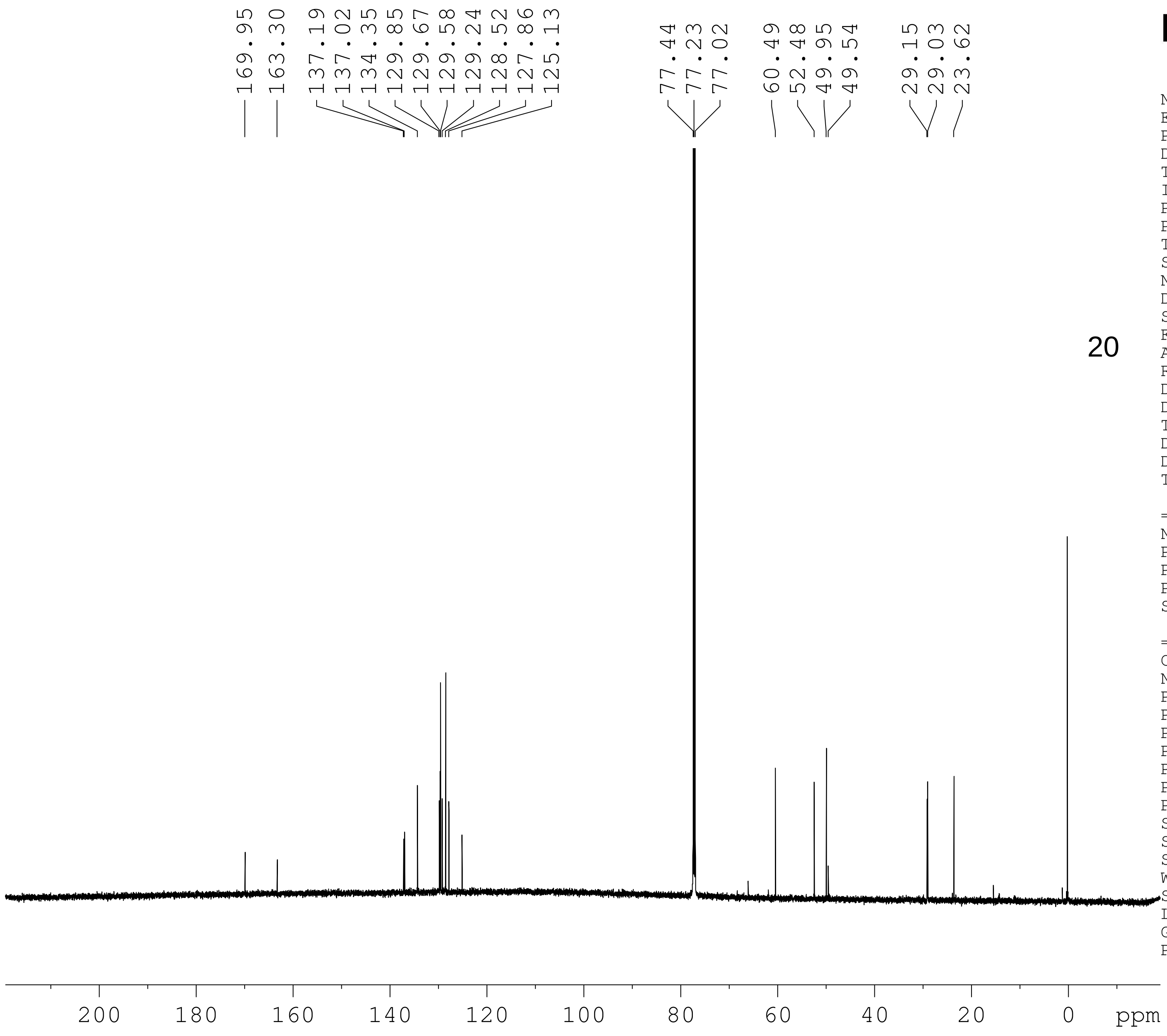
jm00188068f2-14 nmr 600



20

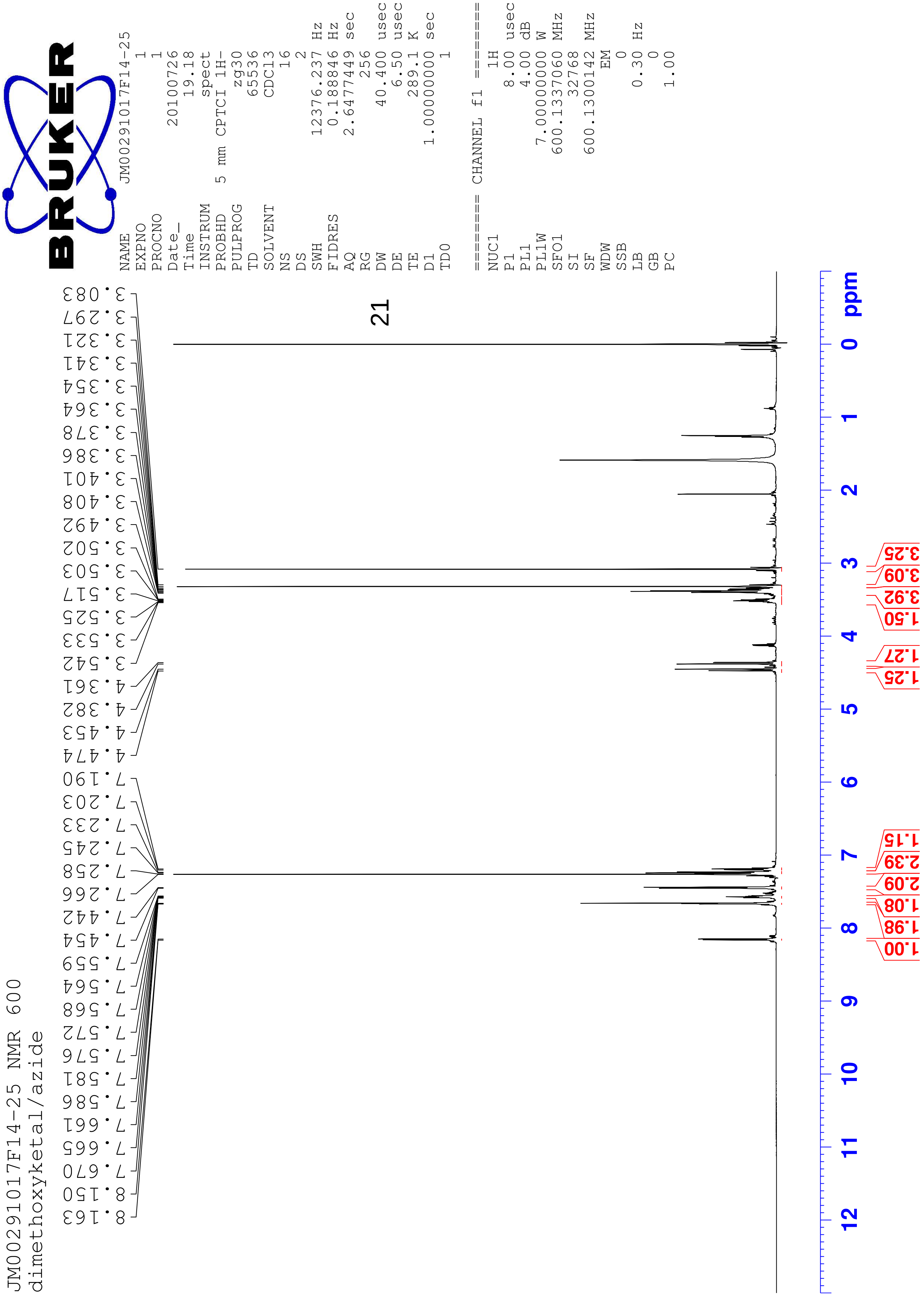
```
NAME      jm00188068f2-14
EXPNO                   1
PROCNO                  1
Date_            20090715
Time                 8.30
INSTRUM             spect
PROBHD     5 mm CPTCI 1H-
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                     32
DS                      2
SWH             12376.237 Hz
FIDRES           0.188846 Hz
AQ              2.6477449 sec
RG                   90.5
DW                 40.400 usec
DE                   6.50 usec
TE                  298.0 K
D1             1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   1H
P1                   8.00 usec
PL1                  4.00 dB
PL1W           7.00000000 W
SFO1          600.1337060 MHz
SI                  32768
SF            600.1300099 MHz
WDW                    EM
SSB                     0
LB                   0.30 Hz
GB                      0
PC                   1.00
```

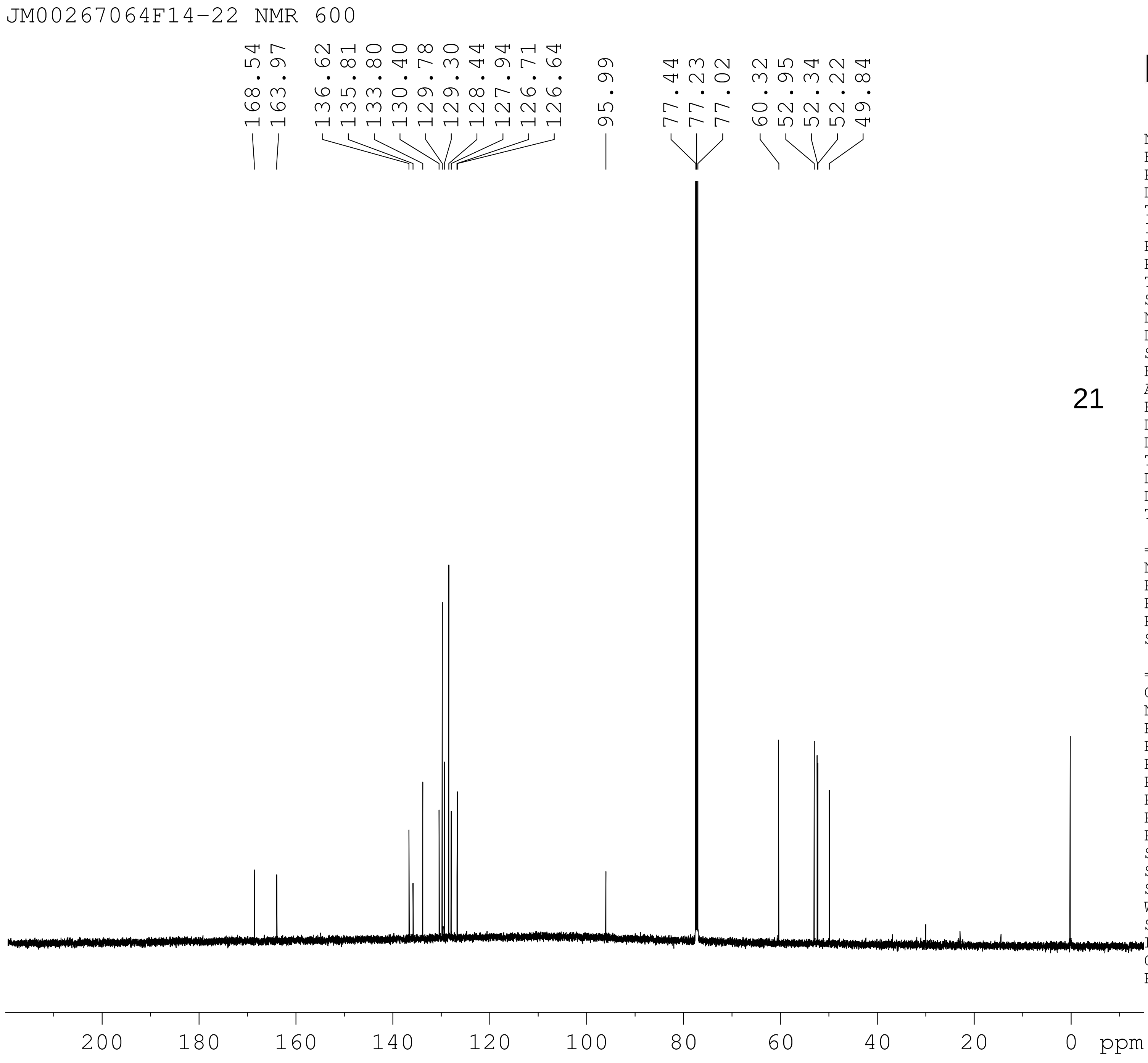
jm00188068f2-14 nmr 600



20

```
NAME       jm00188068f2-14
EXPNO                    3
PROCNO                   1
Date_             20090805
Time                 13.17
INSTRUM              spect
PROBHD     5 mm CPTCI 1H-
PULPROG            zgpg30
TD                   65536
SOLVENT              CDCl3
NS                     200
DS                       2
SWH              35971.223 Hz
FIDRES            0.548877 Hz
AQ               0.9110143 sec
RG                   13004
DW                  13.900 usec
DE                    6.50 usec
TE                   298.0 K
D1              2.00000000 sec
D11             0.03000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                   13C
P1                   12.00 usec
PL1                  -0.70 dB
PL1W           82.63385773 W
SFO1           150.9178988 MHz

======== CHANNEL f2 ========
CPDPRG2            waltz16
NUC2                    1H
PCPD2                80.00 usec
PL2                   4.00 dB
PL12                 24.00 dB
PL13                 27.00 dB
PL2W            7.00000000 W
PL12W           0.07000000 W
PL13W           0.03508311 W
SFO2           600.1324005 MHz
SI                   32768
SF             150.9027782 MHz
WDW                     EM
SSB                      0
LB                    1.00 Hz
GB                       0
PC                    1.40
```

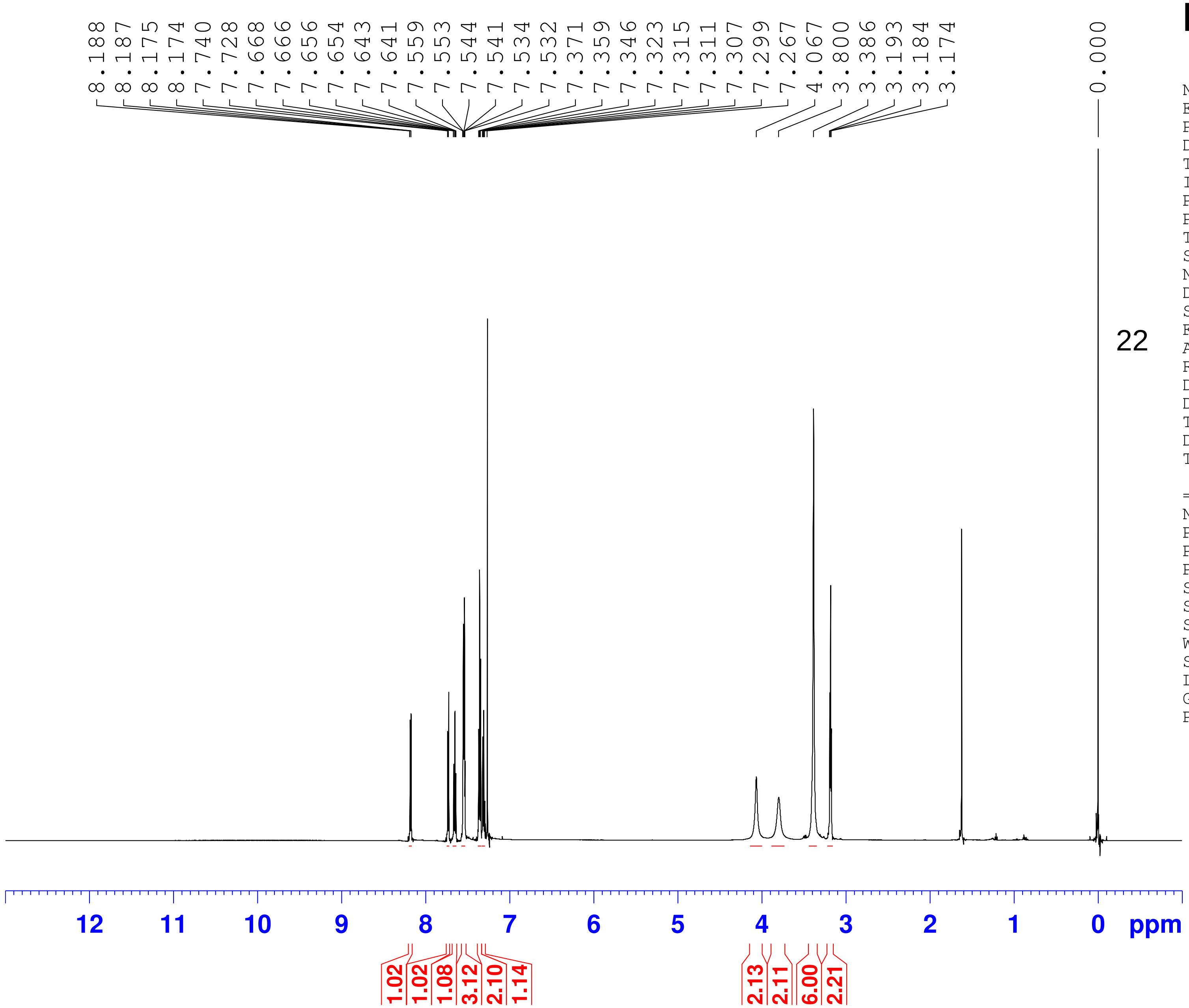

JM0029101 7F14-25 NMR 600
dimethoxyketal/azide
21
3.083
3.297
3.321
3.341
3.354
3.364
3.378
3.386
3.401
3.408
3.492
3.502
3.503
3.517
3.525
3.533
3.542
4.361
4.382
4.453
4.474
7.190
7.203
7.233
7.245
7.258
7.266
7.442
7.454
7.559
7.564
7.568
7.572
7.576
7.581
7.586
7.661
7.665
7.670
8.150
8.163
NAME JM0029101 7F14-25
EXPNO 1
PROCNO 1
Date_ 20100726
Time 19.18
INSTRUM spect
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 12376.237 Hz
FIDRES 0.188846 Hz
AQ 2.6477449 sec
RG 256
DW 40.400 usec
DE 6.50 usec
TE 289.1 K
D1 1.0000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 8.00 usec
PL1 4.00 dB
PL1W 7.00000000 W
SFO1 600.1337060 MHz
SI 32768
SF 600.1300142 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
3.25
3.09
3.92
1.50
1.27
1.25
1.15
2.39
2.09
1.08
1.98
1.00
ppm
0
1
2
3
4
5
6
7
8
9
10
11
12



21

```
NAME       JM00267064F14-22
EXPNO                     2
PROCNO                    1
Date_              20100528
Time                  18.51
INSTRUM               spect
PROBHD     5 mm CPTCI 1H-
PULPROG              zgpg30
TD                    65536
SOLVENT               CDCl3
NS                       93
DS                        2
SWH               35971.223 Hz
FIDRES             0.548877 Hz
AQ                0.9110143 sec
RG                    16384
DW                   13.900 usec
DE                     6.50 usec
TE                    298.0 K
D1               2.00000000 sec
D11              0.03000000 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                    13C
P1                    12.00 usec
PL1                   -0.70 dB
PL1W            82.63385773 W
SFO1            150.9178988 MHz

======== CHANNEL f2 ========
CPDPRG2             waltz16
NUC2                     1H
PCPD2                 80.00 usec
PL2                    4.00 dB
PL12                  24.00 dB
PL13                  27.00 dB
PL2W             7.00000000 W
PL12W            0.07000000 W
PL13W            0.03508311 W
SFO2            600.1324005 MHz
SI                    32768
SF              150.9027785 MHz
WDW                      EM
SSB                       0
LB                     1.00 Hz
GB                        0
PC                     1.40
```

JM00291032F30-47 NMR 600
recrystallized

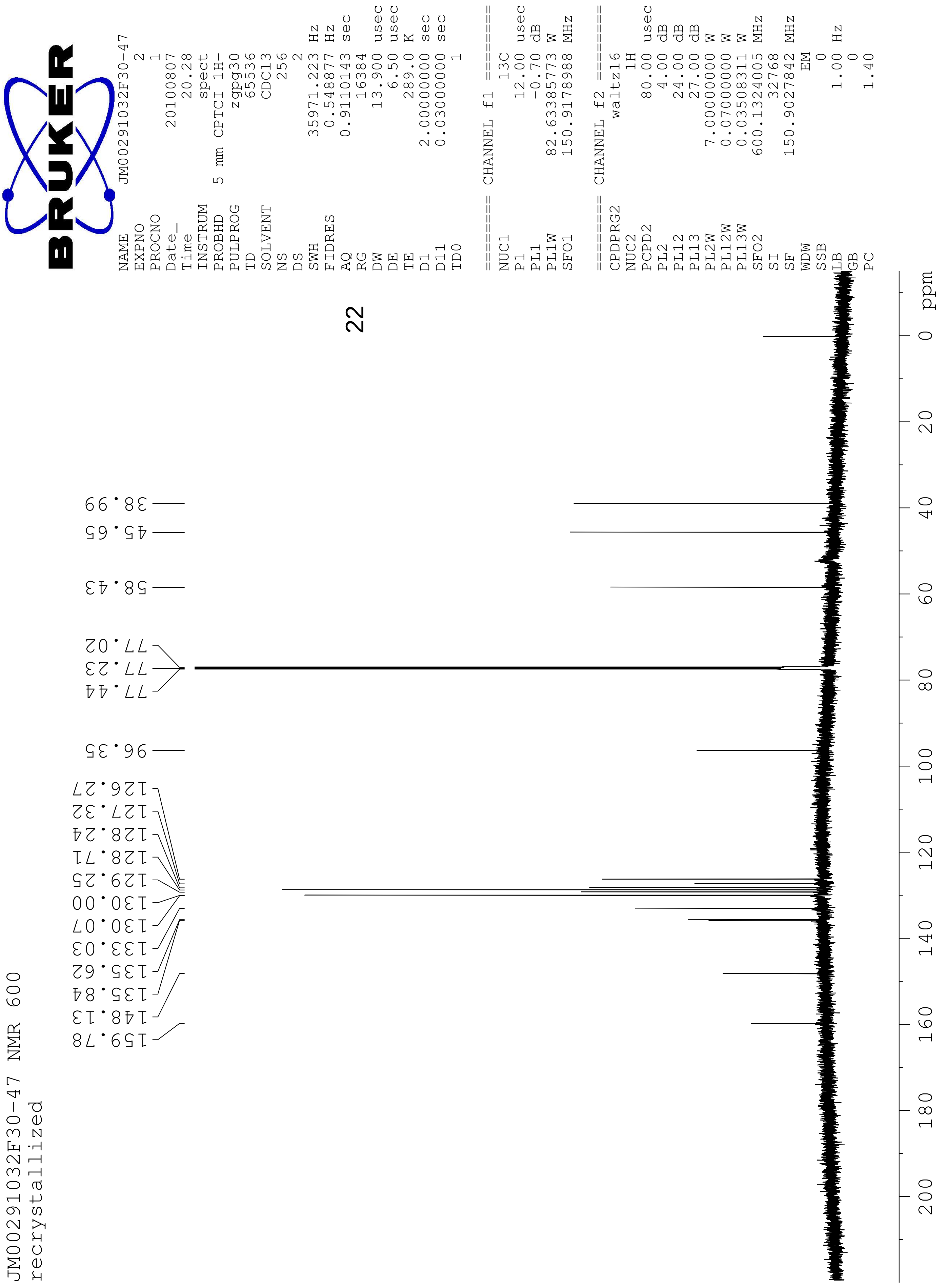


22

NAME	JM00291032F30-47
EXPNO	1
PROCNO	1
Date_	20100807
Time	20.23
INSTRUM	spect
PROBHD	5 mm CPTCI 1H-
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	12376.237 Hz
FIDRES	0.188846 Hz
AQ	2.6477449 sec
RG	114
DW	40.400 usec
DE	6.50 usec
TE	289.0 K
D1	1.00000000 sec
TD0	1

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

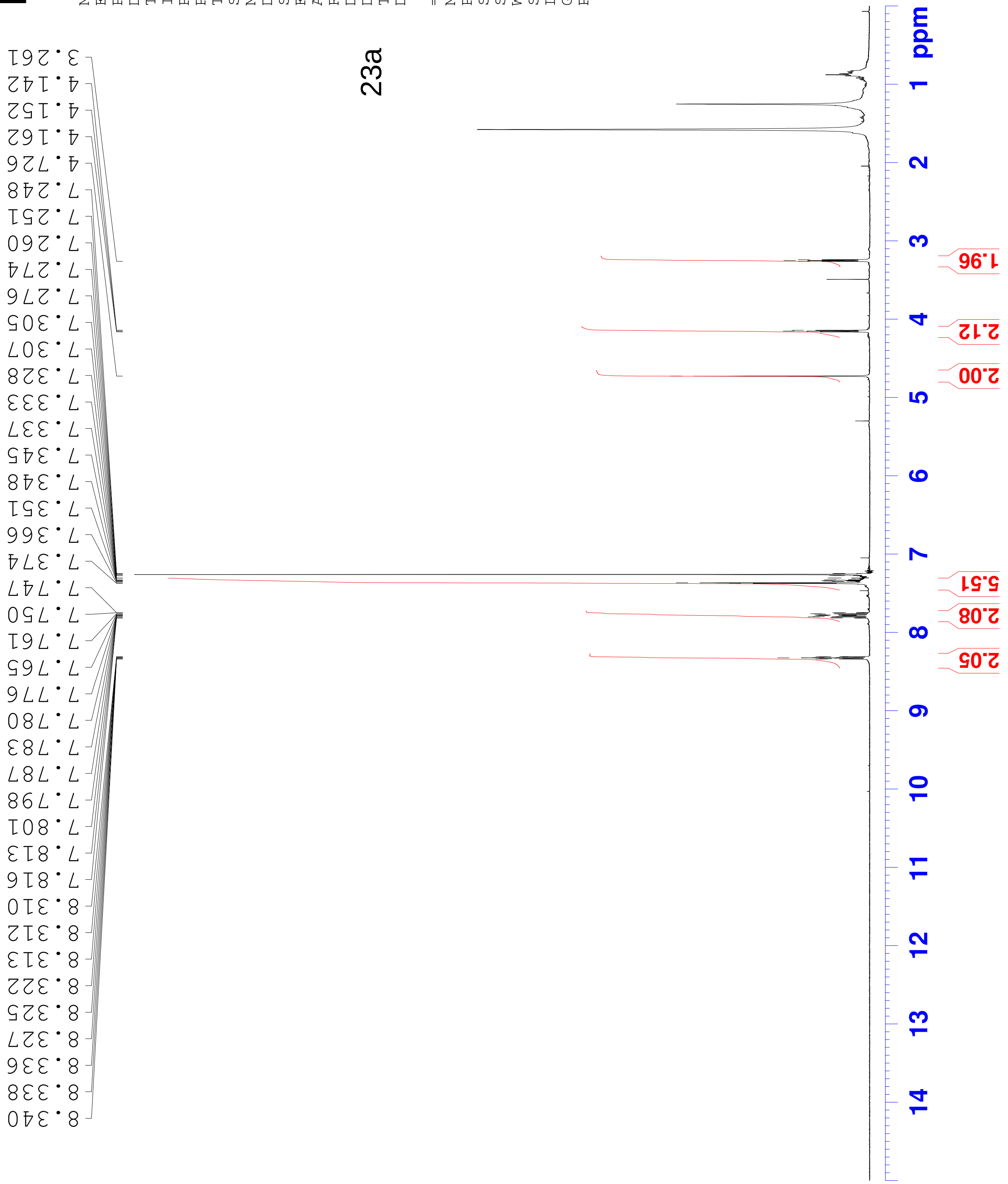
NUC1	1H
P1	8.00 usec
PL1	4.00 dB
PL1W	7.00000000 W
SFO1	600.1337060 MHz
SI	32768
SF	600.1300134 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00


22
JM00291032F30-47 NMR 600
recrystallized
38.99
45.65
58.43
77.02
77.23
77.44
96.35
126.27
127.32
128.24
128.71
129.25
130.00
130.07
133.03
135.62
135.84
148.13
159.78
ppm
0
20
40
60
80
100
120
140
160
180
200
NAME JM00291032F30-47
EXPNO 2
PROCNO 1
Date_ 20100807
Time 20.28
INSTRUM spect
PROBHD 5 mm CPTCI 1H-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 256
DS 2
SWH 35971.223 Hz
FIDRES 0.548877 Hz
AQ 0.9110143 sec
RG 16384
DW 13.900 usec
DE 6.50 usec
TE 289.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 12.00 usec
PL1 -0.70 dB
PL1W 82.63385773 W
SFO1 150.9178988 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 4.00 dB
PL12 24.00 dB
PL13 27.00 dB
PL2W 7.00000000 W
PL12W 0.07000000 W
PL13W 0.03508311 W
SFO2 600.1324005 MHz
SI 32768
SF 150.9027842 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

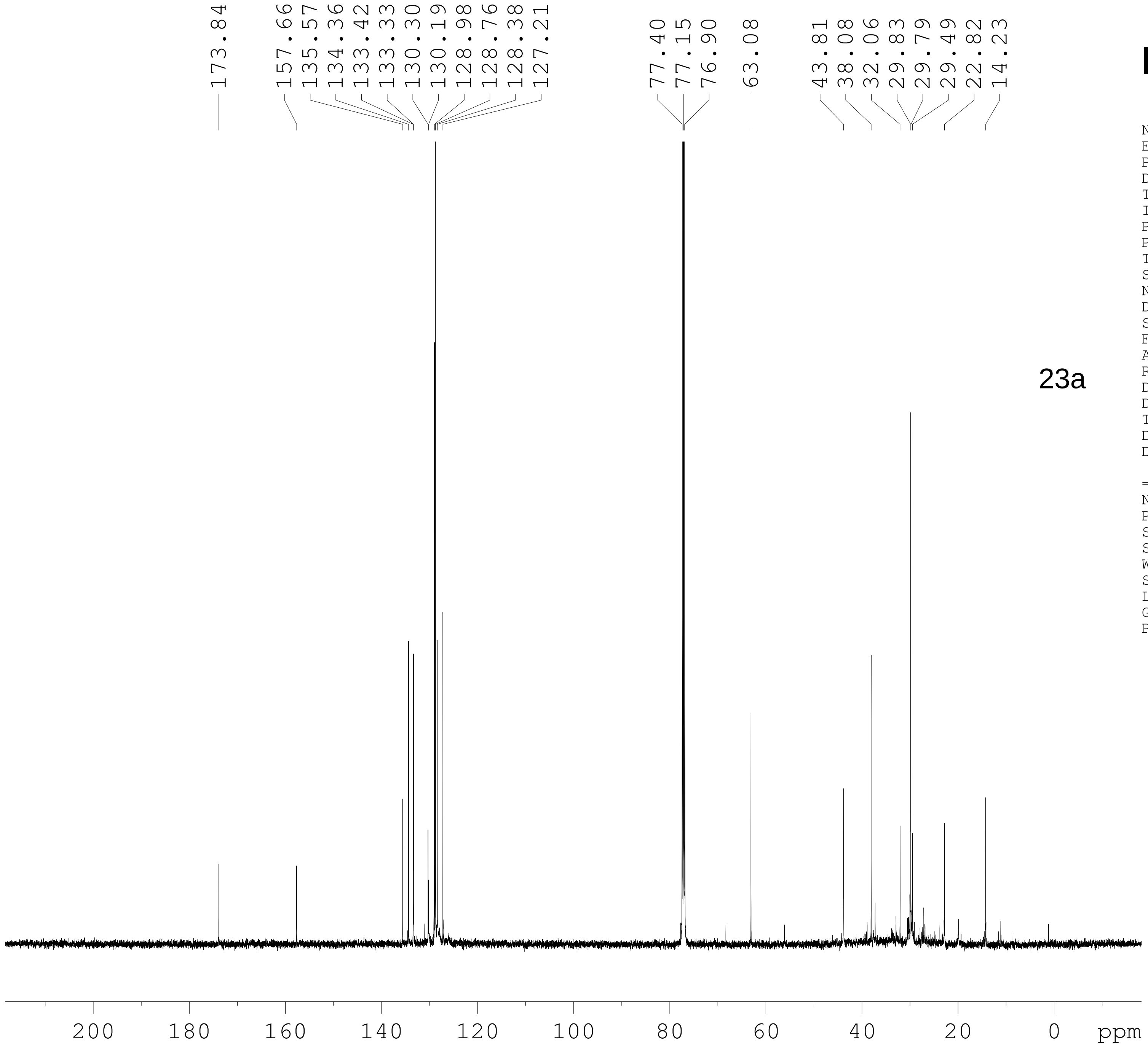
CLM-ketal deprotection HCl H2SO4

```
NAME     CLM-ketal deprotection
EXPNO                 1
PROCNO                1
Date_          20110828
Time              17.20
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    2
SWH           10330.578 Hz
FIDRES         0.157632 Hz
AQ            3.1719923 sec
RG                  203
DW               48.400 usec
DE                 6.50 usec
TE                296.4 K
D1           1.00000000 sec

======== CHANNEL f1 ========
NUC1                 1H
P1                10.20 usec
SI                65536
SF          500.1600119 MHz
WDW                  EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.00
```

23a

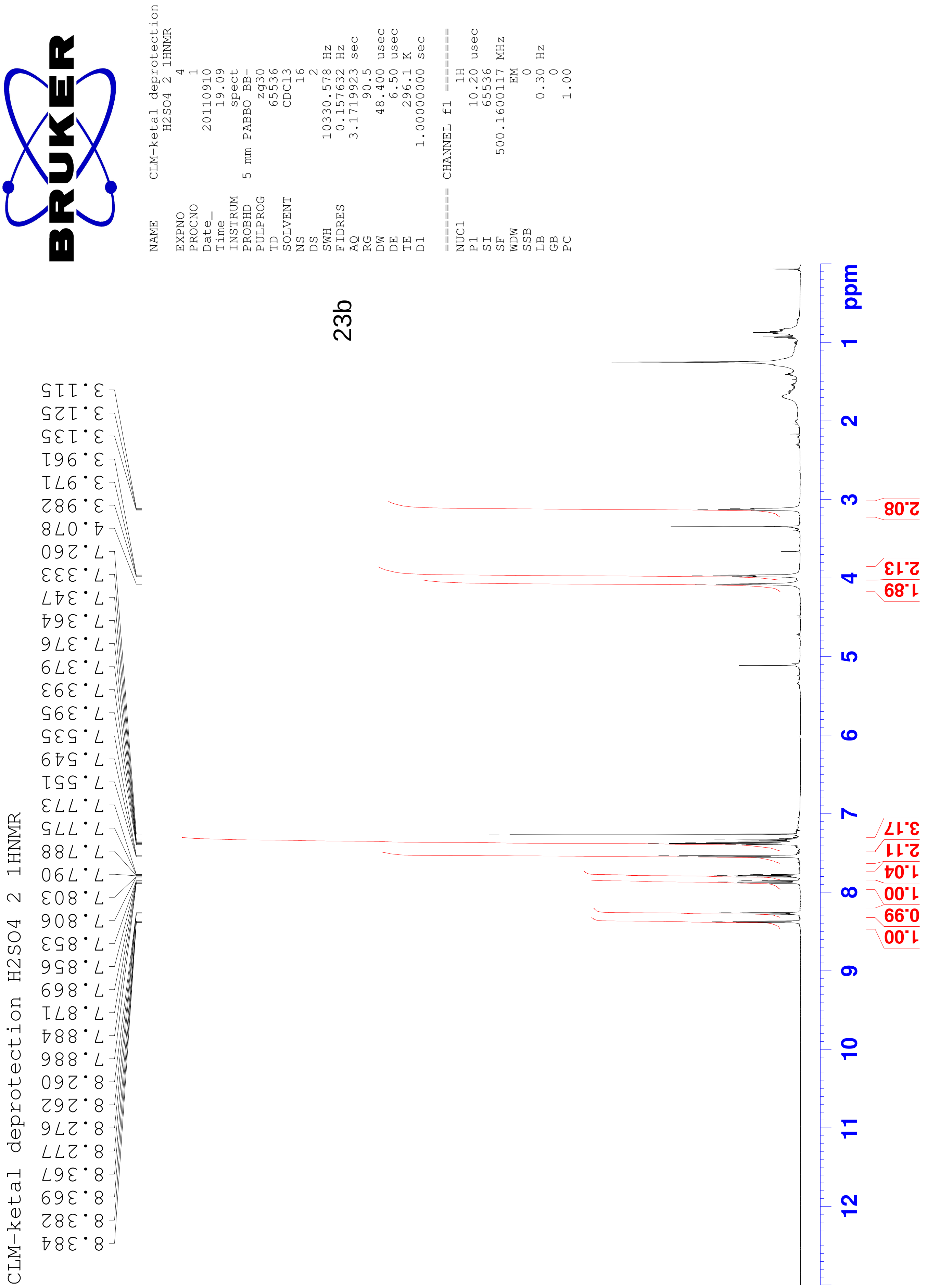


CLM-ketal deprotection 13C NMR

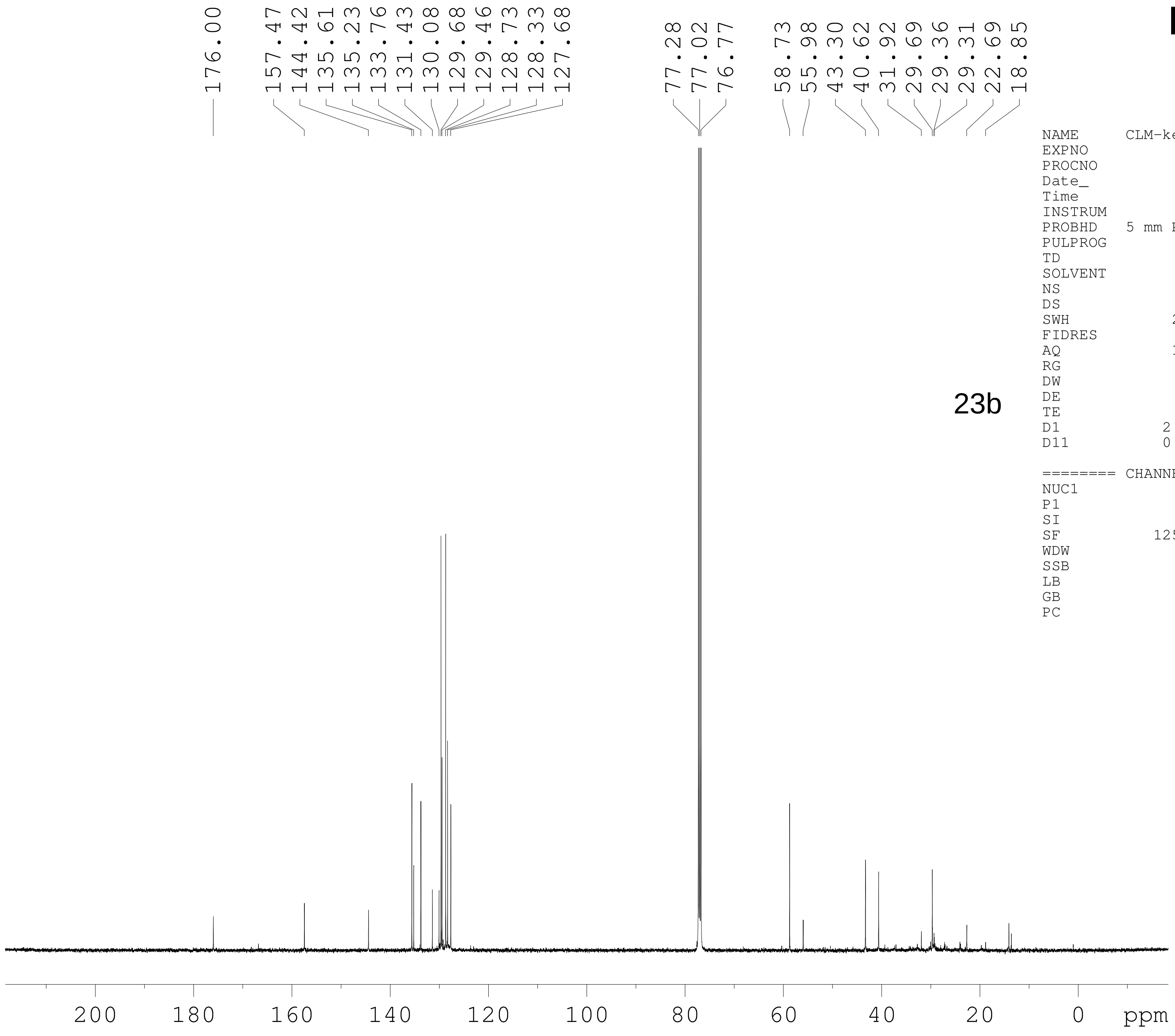


23a

```
NAME       CLM-ketal deprotection
EXPNO                   3
PROCNO                  1
Date_            20110904
Time                 4.17
INSTRUM             spect
PROBHD   5 mm PABBO BB-
PULPROG            zgpg30
TD                  65536
SOLVENT             CDCl3
NS                   8192
DS                      4
SWH            29761.904 Hz
FIDRES          0.454131 Hz
AQ             1.1010548 sec
RG                    203
DW                 16.800 usec
DE                   6.50 usec
TE                  299.7 K
D1             2.00000000 sec
D11            0.03000000 sec

======== CHANNEL f1 ========
NUC1                  13C
P1                   8.80 usec
SI                  32768
SF            125.7653147 MHz
WDW                    EM
SSB                     0
LB                   1.00 Hz
GB                      0
PC                   1.40
```


CLM-ketal deprotection H2SO4 2 1HNMR
3.115
3.125
3.135
3.961
3.971
3.982
4.078
7.260
7.333
7.347
7.364
7.376
7.379
7.393
7.395
7.535
7.549
7.551
7.773
7.775
7.788
7.790
7.803
7.806
7.853
7.856
7.869
7.871
7.884
7.886
8.260
8.262
8.276
8.277
8.367
8.369
8.382
8.384
BRUKER
NAME CLM-ketal deprotection
H2SO4 2 1HNMR
EXPNO 4
PROCNO 1
Date_ 20110910
Time 19.09
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1719923 sec
RG 90.5
DW 48.400 usec
DE 6.50 usec
TE 296.1 K
D1 1.00000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 10.20 usec
SI 65536
SF 500.1600117 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
23b
2.08
2.13
1.89
3.17
2.11
1.04
1.00
0.99
1.00
12
11
10
9
8
7
6
5
4
3
2
1
ppm

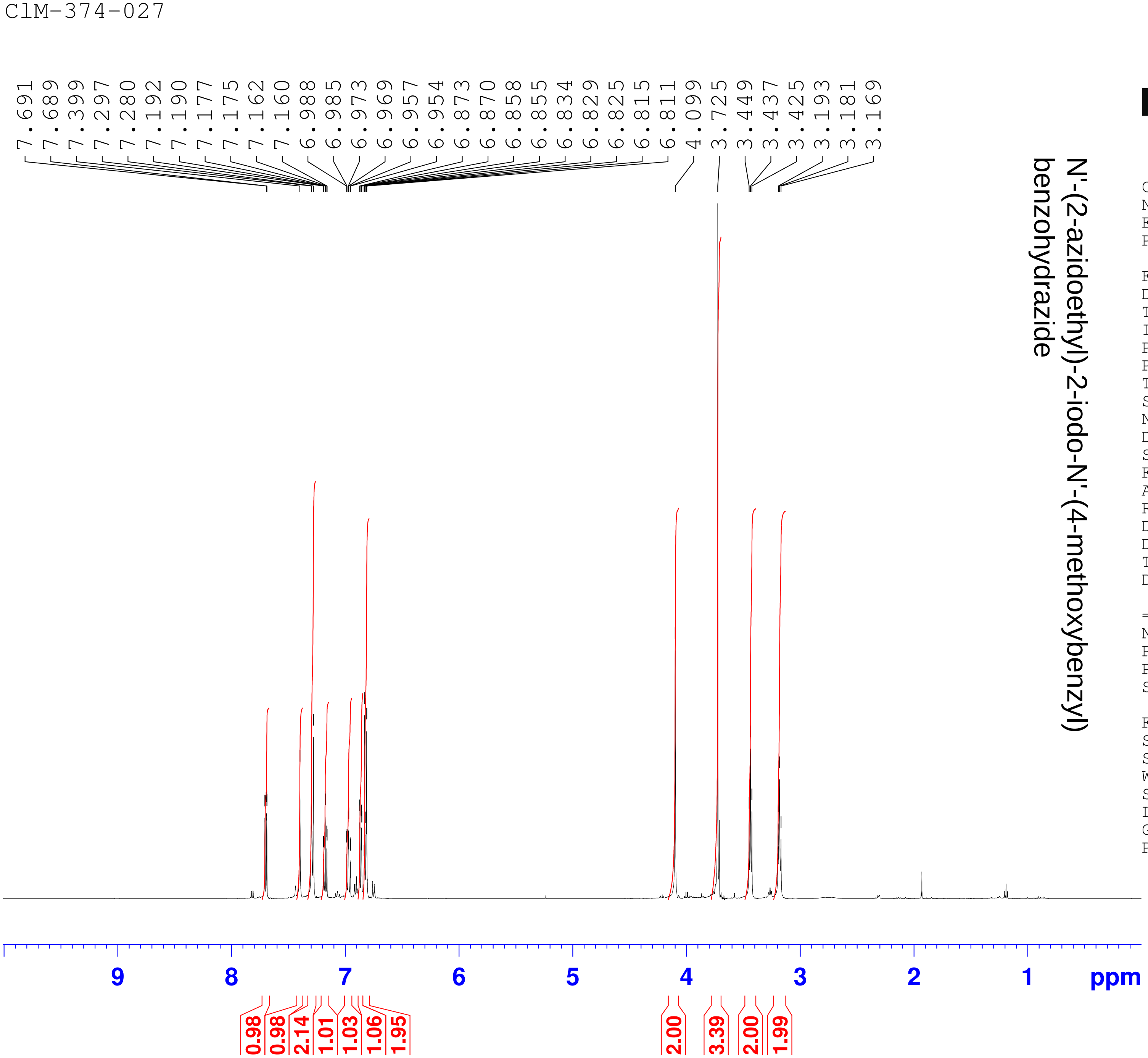
CLM-ketal deprotection H2SO4 2 13C NMR



23b

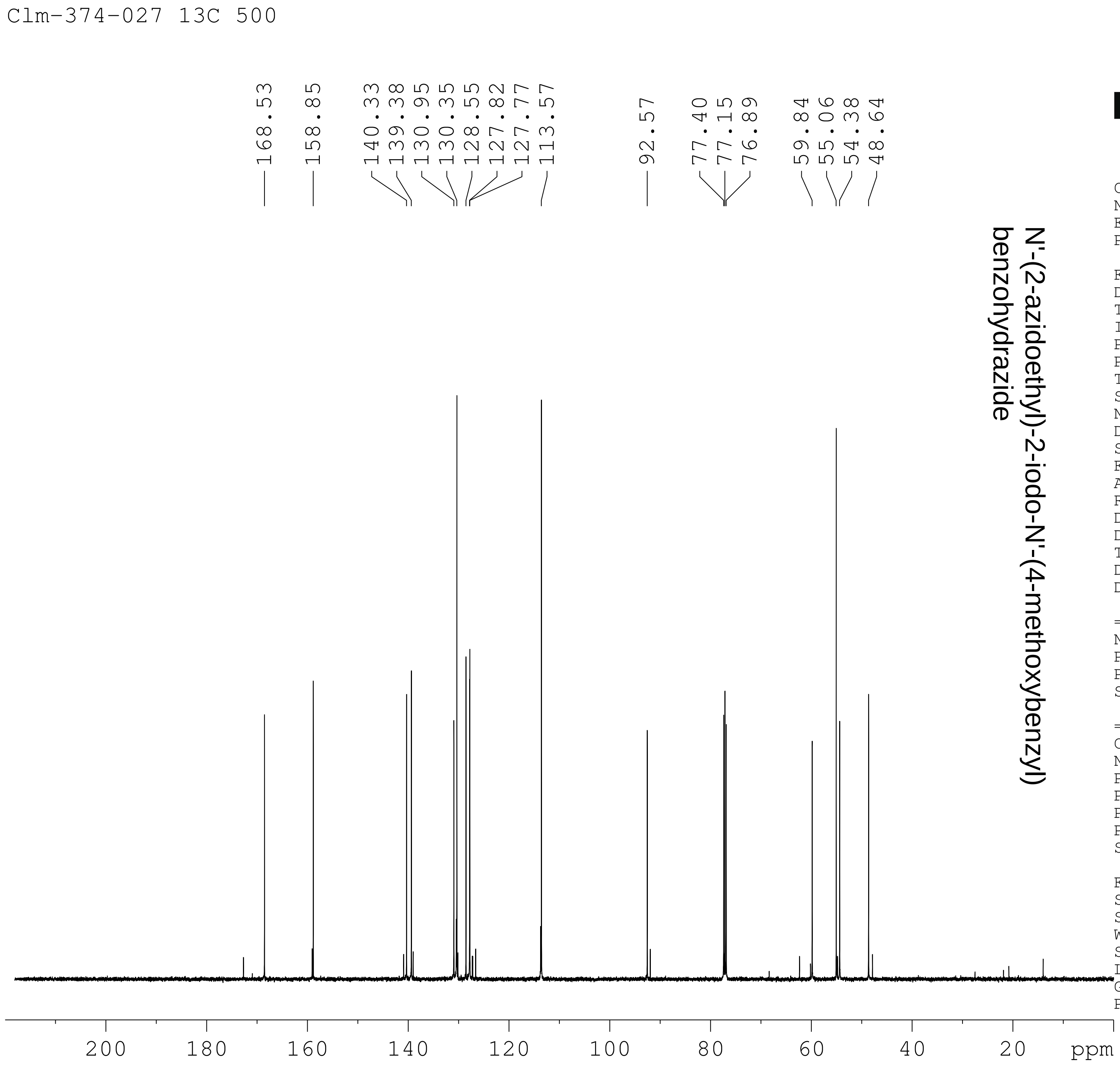
```
NAME     CLM-ketal deprotection H2SO4 2 13C NMR
EXPNO                     5
PROCNO                    1
Date_              20110911
Time                   7.38
INSTRUM               spect
PROBHD   5 mm PABBO BB-
PULPROG             zgpg30
TD                    65536
SOLVENT               CDCl3
NS                    14000
DS                        4
SWH             29761.904 Hz
FIDRES           0.454131 Hz
AQ              1.1010548 sec
RG                      203
DW                   16.800 usec
DE                     6.50 usec
TE                    296.3 K
D1               2.00000000 sec
D11              0.03000000 sec

======== CHANNEL f1 ========
NUC1                    13C
P1                     8.80 usec
SI                    32768
SF             125.7653333 MHz
WDW                      EM
SSB                       0
LB                     1.00 Hz
GB                        0
PC                     1.40
```

N'-(2-azidoethyl)-2-iodo-N'-(4-methoxybenzyl)
benzohydrazide



```
Current Data Parameters
NAME           CLM-374-027
EXPNO                    2
PROCNO                   1

F2 - Acquisition Parameters
Date_             20111104
Time                 22.09
INSTRUM              spect
PROBHD   5 mm PABBO BB-
PULPROG               zg30
TD                   65536
SOLVENT              CDCl3
NS                      16
DS                       2
SWH              10330.578 Hz
FIDRES            0.157632 Hz
AQ               3.1719923 sec
RG                      32
DW                  48.400 usec
DE                    6.50 usec
TE                   293.7 K
D1              1.00000000 sec

======== CHANNEL f1 ========
NUC1                    1H
P1                   10.20 usec
PLW1           18.74300003 W
SFO1           500.1630887 MHz

F2 - Processing parameters
SI                   65536
SF             500.1600000 MHz
WDW                     EM
SSB      0
LB                    0.30 Hz
GB       0
PC                    1.00
```

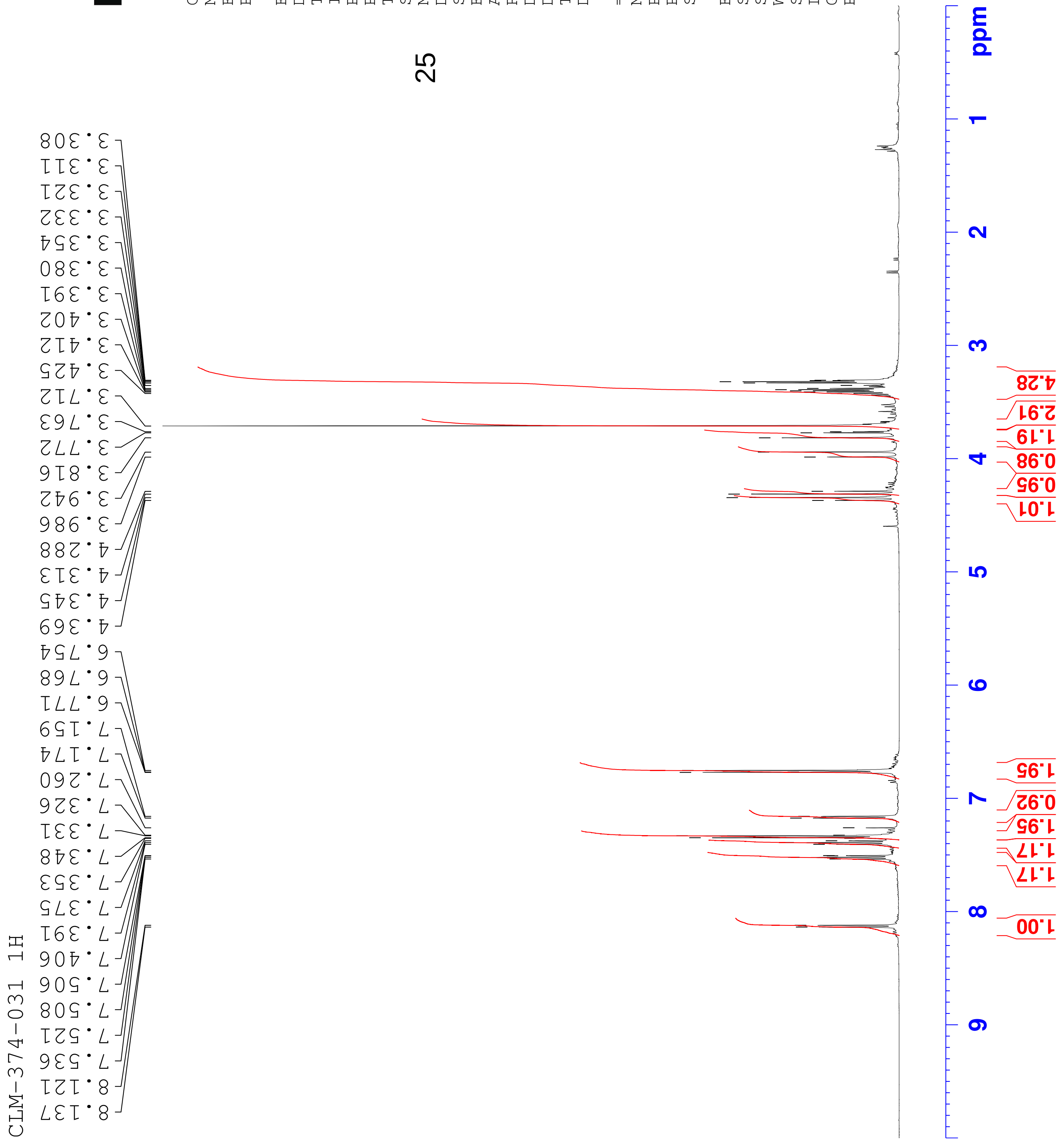
N'-(2-azidoethyl)-2-iodo-N'-(4-methoxybenzyl) benzohydrazide



```
Current Data Parameters
NAME          CLM-374-027
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_            20111104
Time                22.06
INSTRUM             spect
PROBHD   5 mm PABBO BB-
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                   250
DS                     4
SWH            29761.904 Hz
FIDRES          0.454131 Hz
AQ             1.1010548 sec
RG                   203
DW                16.800 usec
DE                  6.50 usec
TE                 294.0 K
D1            2.00000000 sec
D11           0.03000000 sec

======== CHANNEL f1 ========
NUC1                 13C
P1                  8.80 usec
PLW1        112.58000183 W
SFO1         125.7779080 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2              80.00 usec
PLW2         18.74300003 W
PLW12         0.36736000 W
PLW13     0 W
SFO2         500.1620006 MHz

F2 - Processing parameters
SI                 32768
SF           125.7653639 MHz
WDW                   EM
SSB       0
LB                  1.00 Hz
GB        0
PC                  1.40
```

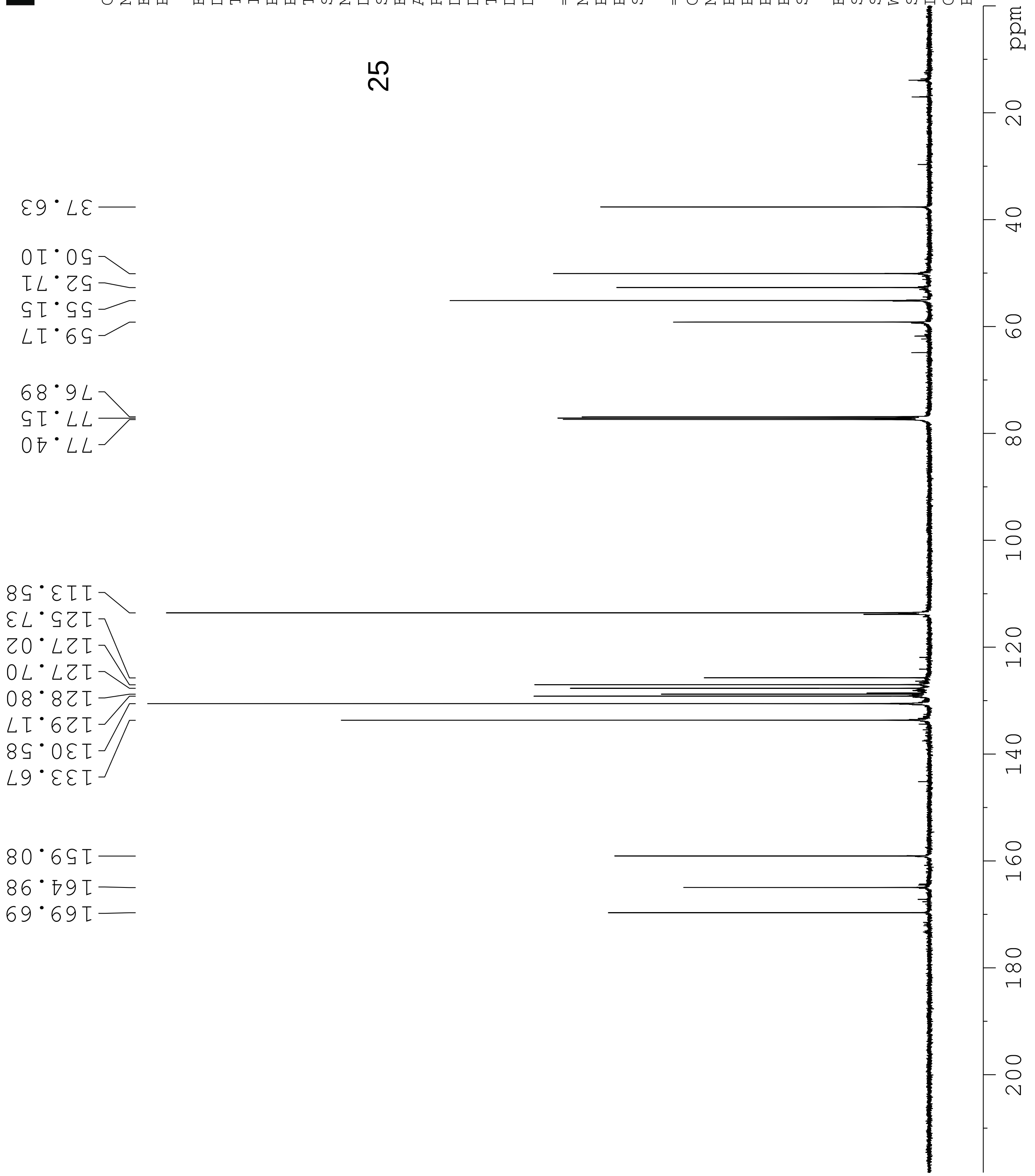
```
Current Data Parameters
NAME        CLM-374-031
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20111113
Time              12.35
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    2
SWH           10330.578 Hz
FIDRES         0.157632 Hz
AQ            3.1719923 sec
RG                 25.4
DW               48.400 usec
DE                 6.50 usec
TE                294.5 K
D1           1.00000000 sec

======== CHANNEL f1 ========
NUC1                 1H
P1                10.20 usec
PLW1        18.74300003 W
SFO1       500.1630887 MHz

F2 - Processing parameters
SI                65536
SF          500.1600112 MHz
WDW                  EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.00
```

25



```
Current Data Parameters
NAME          CLM-374-031
EXPNO                   2
PROCNO                  1

F2 - Acquisition Parameters
Date_            20111113
Time                13.38
INSTRUM             spect
PROBHD   5 mm PABBO BB-
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                  1024
DS                     4
SWH            29761.904 Hz
FIDRES          0.454131 Hz
AQ             1.1010548 sec
RG                   203
DW                16.800 usec
DE                  6.50 usec
TE                 295.1 K
D1            2.00000000 sec
D11           0.03000000 sec

======== CHANNEL f1 ========
NUC1                 13C
P1                  8.80 usec
PLW1        112.58000183 W
SFO1         125.7779080 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2              80.00 usec
PLW2         18.74300003 W
PLW12         0.36736000 W
PLW13                  0 W
SFO2         500.1620006 MHz

F2 - Processing parameters
SI                 32768
SF           125.7653330 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```

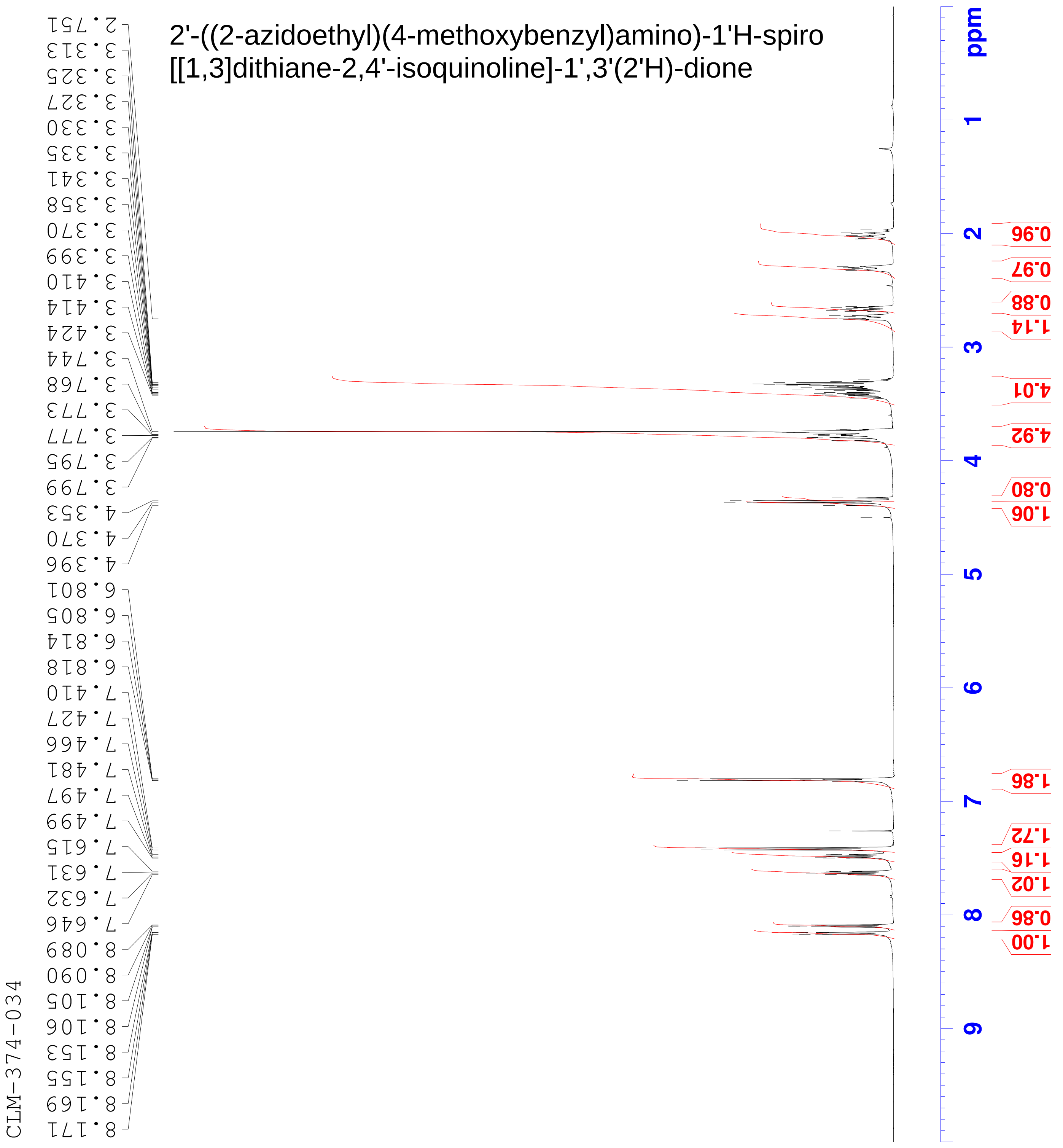
25



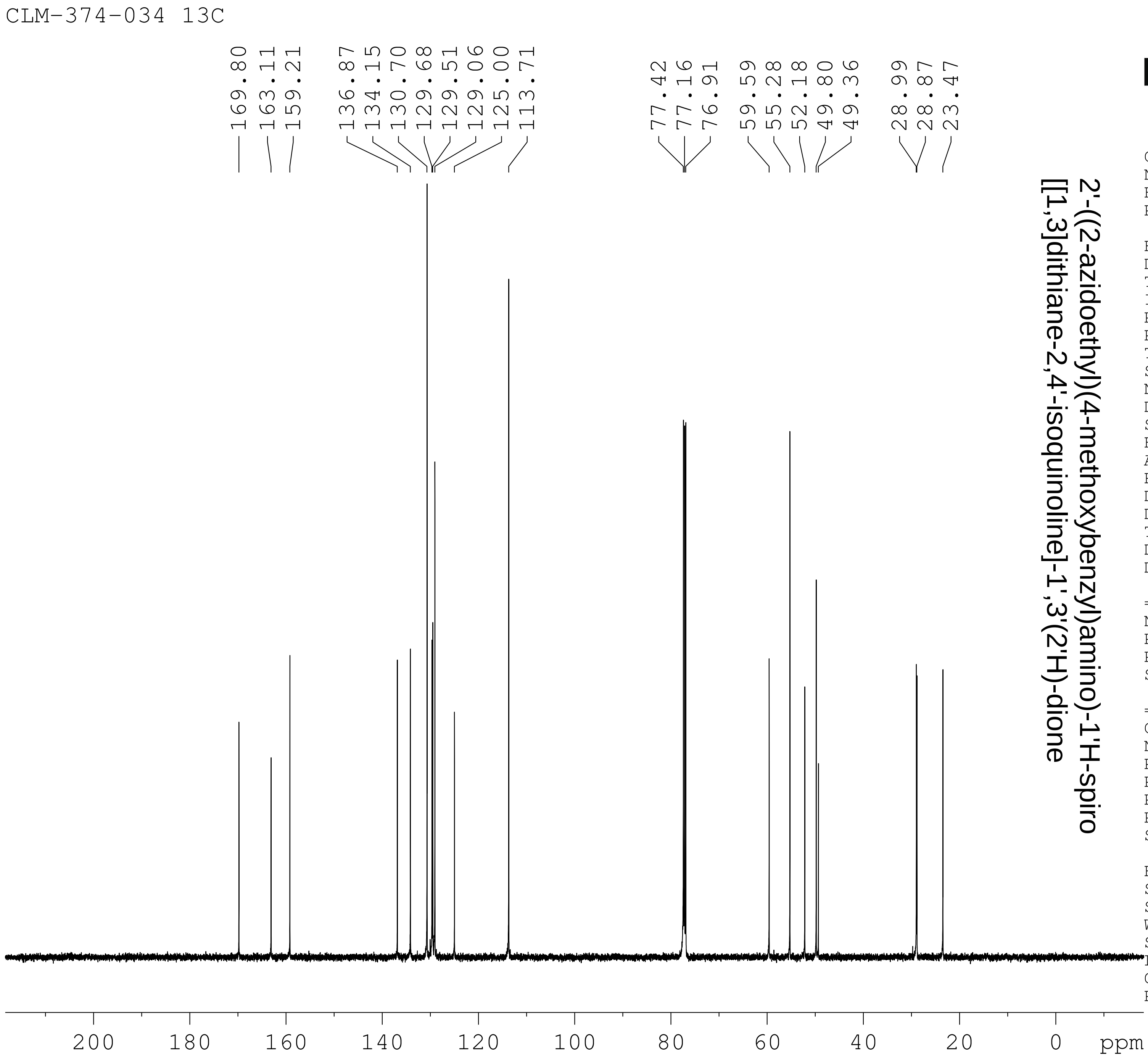
CLM-374-031 13C

NAME	CLM-374-034
EXPNO	1
PROCNO	1
Date_	20111118
Time	18.45
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	10330.578 Hz
FIDRES	0.157632 Hz
AQ	3.1719923 sec
RG	32
DW	48.400 usec
DE	6.50 usec
TE	293.8 K
D1	1.00000000 sec
========	CHANNEL f1 ========
NUC1	1H
P1	10.20 usec
SI	65536
SF	500.1600114 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

2'-((2-azidoethyl)(4-methoxybenzyl)amino)-1'H-spiro[[1,3]dithiane-2,4'-isoquinoline]-1',3'(2'H)-dione



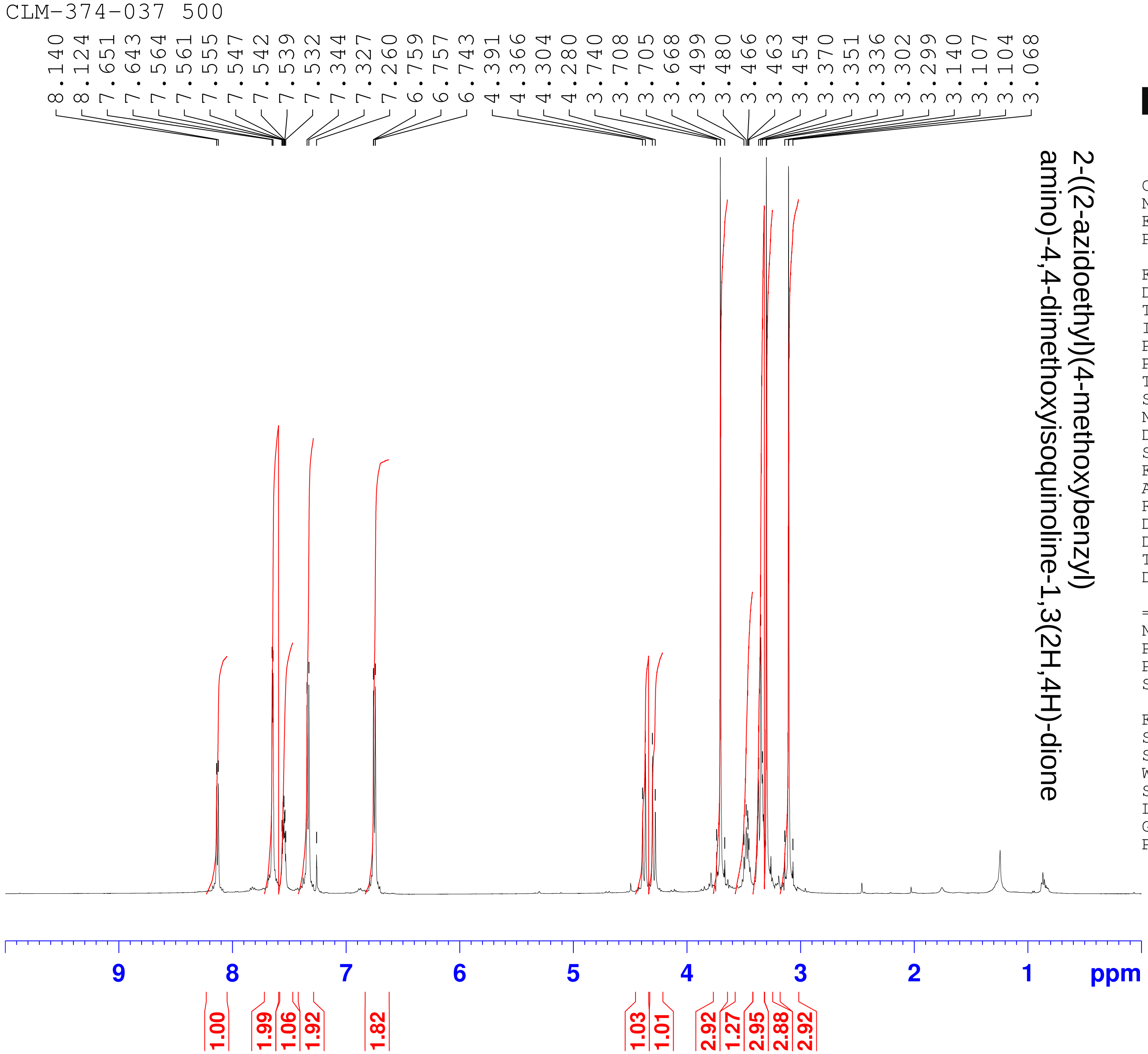
CLM-374-034



2'-((2-azidoethyl)(4-methoxybenzyl)amino)-1'H-spiro
[[1,3]dithiane-2,4'-isoquinoline]-1',3'(2'H)-dione

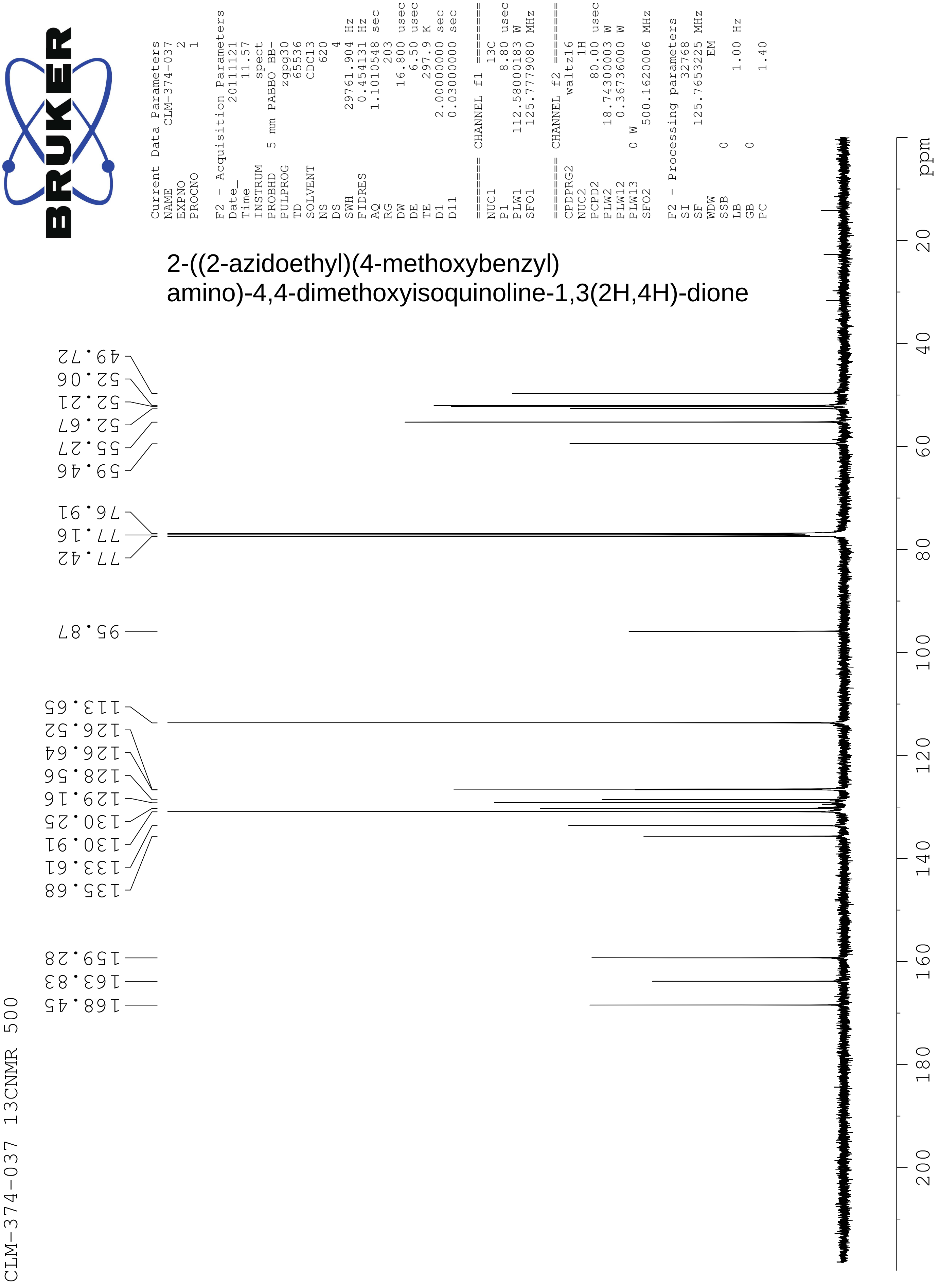
```
Current Data Parameters
NAME           CLM-374-034
EXPNO                    2
PROCNO                   1

F2 - Acquisition Parameters
Date_             20111118
Time                 19.26
INSTRUM              spect
PROBHD    5 mm PABBO BB-
PULPROG            zgpg30
TD                  65536
SOLVENT             CDCl3
NS                    680
DS                      4
SWH            29761.904 Hz
FIDRES          0.454131 Hz
AQ             1.1010548 sec
RG                    203
DW                 16.800 usec
DE                   6.50 usec
TE                  294.3 K
D1            2.00000000 sec
D11           0.03000000 sec

======== CHANNEL f1 ========
NUC1                  13C
P1                   8.80 usec
PLW1         112.58000183 W
SFO1          125.7779080 MHz

======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                   1H
PCPD2               80.00 usec
PLW2          18.74300003 W
PLW12          0.36736000 W
PLW13      0 W
SFO2          500.1620006 MHz

F2 - Processing parameters
SI                  32768
SF            125.7653270 MHz
WDW                    EM
SSB        0
LB                   1.00 Hz
GB         0
PC                   1.40
```

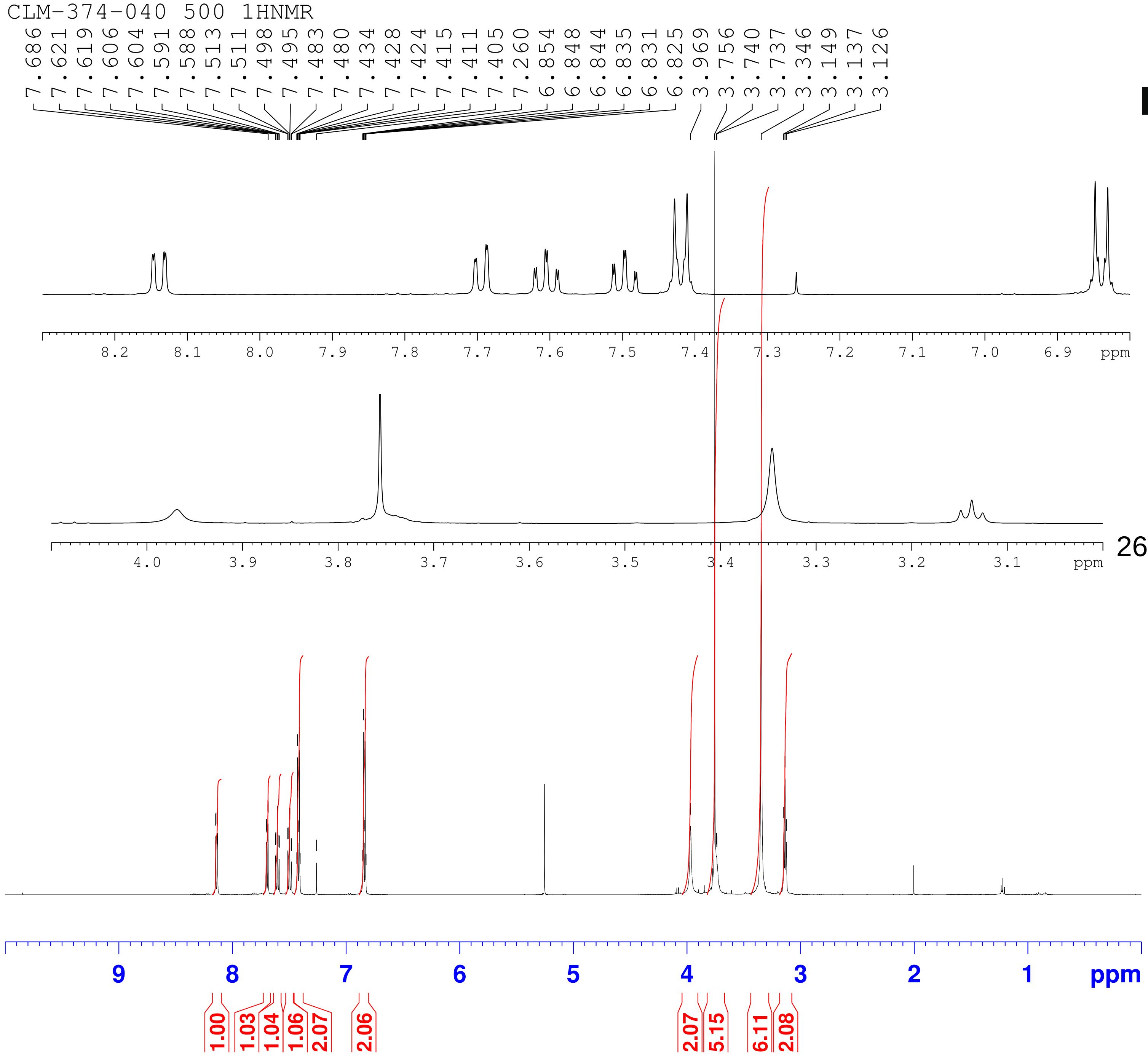
2-((2-azidoethyl)(4-methoxybenzyl)
amino)-4,4-dimethoxyisoquinoline-1,3(2H,4H)-dione



```
Current Data Parameters
NAME          CLM-374-037
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_            20111121
Time                11.17
INSTRUM             spect
PROBHD     5 mm PABBO BB-
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                     16
DS                      2
SWH             10330.578 Hz
FIDRES           0.157632 Hz
AQ              3.1719923 sec
RG                     32
DW                 48.400 usec
DE                   6.50 usec
TE                  294.6 K
D1             1.00000000 sec

======== CHANNEL f1 ========
NUC1                   1H
P1                  10.20 usec
PLW1          18.74300003 W
SFO1          500.1630887 MHz

F2 - Processing parameters
SI                  65536
SF            500.1600121 MHz
WDW                    EM
SSB      0
LB                   0.30 Hz
GB       0
PC                   1.00
```

2-((2-azidoethyl)(4-methoxybenzyl)amino)-4,4-dimethoxyisoquinoline-1,3(2H,4H)-dione

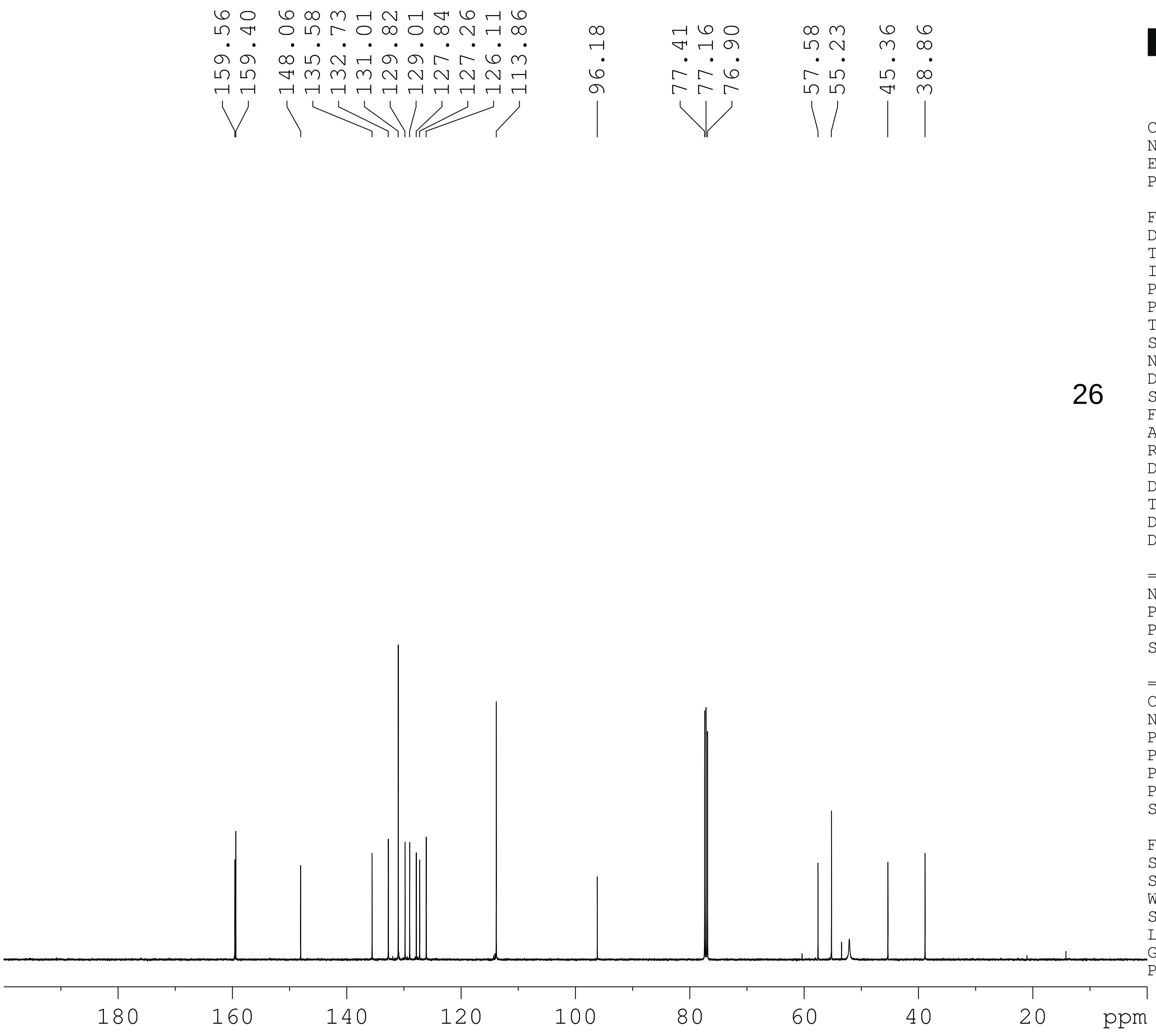




26

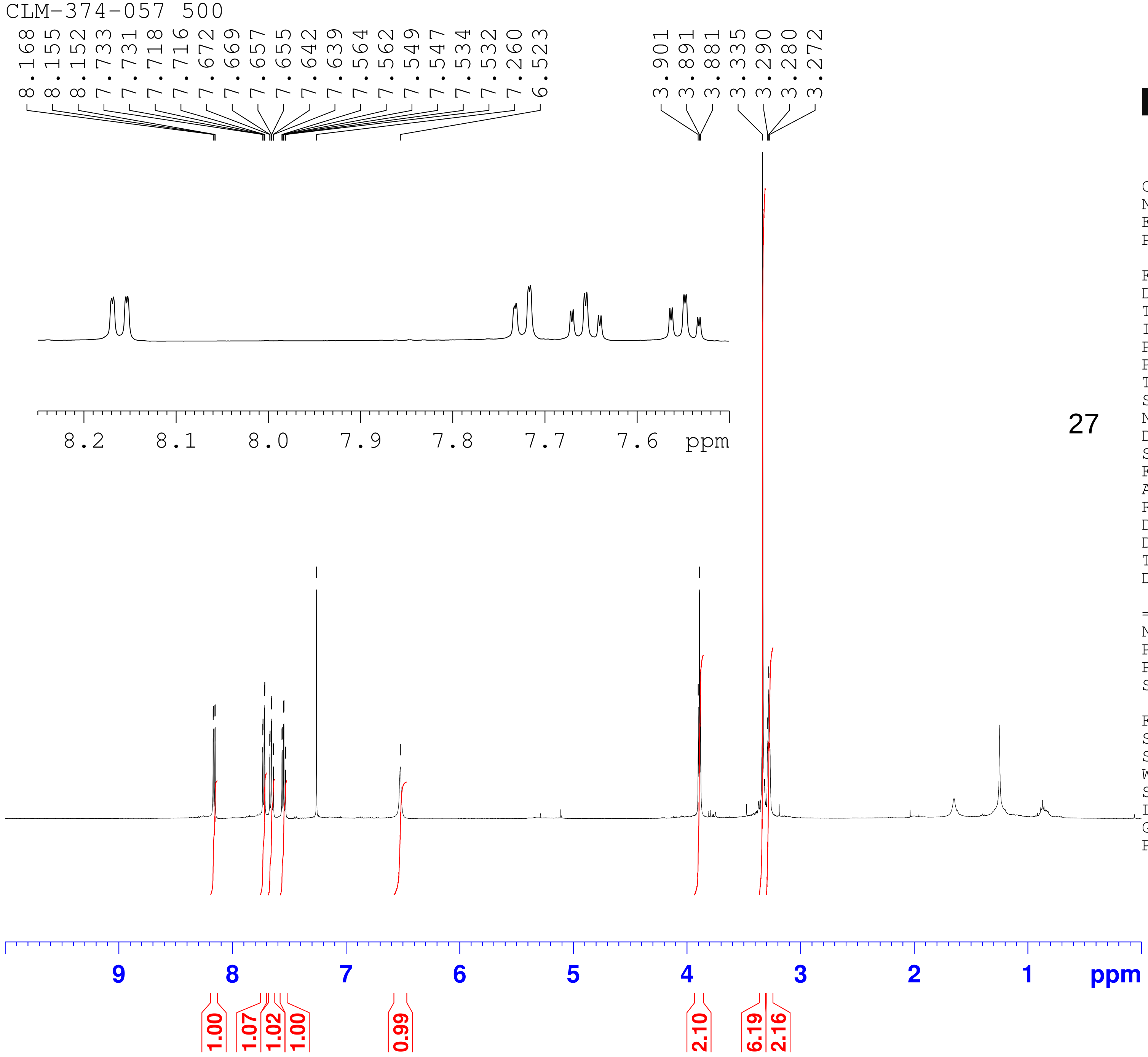
```
Current Data Parameters
NAME             CLM-374-040
EXPNO                      3
PROCNO                     1

F2 - Acquisition Parameters
Date_               20120119
Time                   12.53
INSTRUM                spect
PROBHD     5 mm PABBO BB-
PULPROG                 zg30
TD                     65536
SOLVENT                CDCl3
NS                        16
DS                         2
SWH             10330.578 Hz
FIDRES           0.157632 Hz
AQ              3.1719923 sec
RG                        32
DW                48.400 usec
DE                  6.50 usec
TE                 298.0 K
D1            1.00000000 sec

======== CHANNEL f1 ========
NUC1                      1H
P1                 10.20 usec
PLW1         18.74300003 W
SFO1         500.1630887 MHz

F2 - Processing parameters
SI                     65536
SF           500.1600114 MHz
WDW                       EM
SSB      0
LB                      0.30 Hz
GB       0
PC                      1.00
```

CLM-374-040 13C 500



26

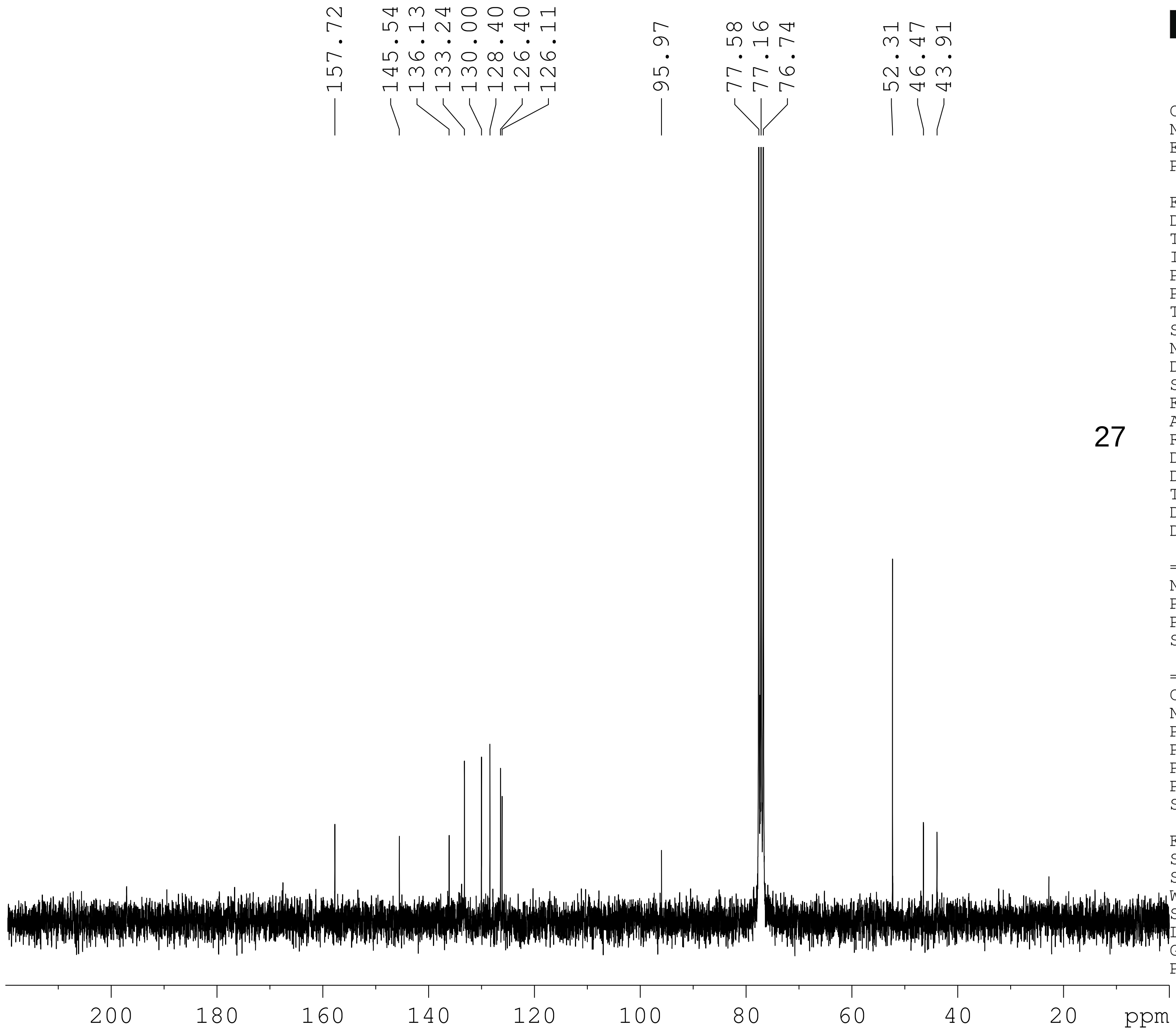
```
Current Data Parameters
NAME          CLM-374-040
EXPNO                   5
PROCNO                  1

F2 - Acquisition Parameters
Date_            20120119
Time                20.57
INSTRUM             spect
PROBHD   5 mm PABBO BB-
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                   800
DS                     4
SWH            29761.904 Hz
FIDRES          0.454131 Hz
AQ             1.1010548 sec
RG                   203
DW                16.800 usec
DE                  6.50 usec
TE                 298.0 K
D1            2.00000000 sec
D11           0.03000000 sec

======== CHANNEL f1 ========
NUC1                 13C
P1                  8.80 usec
PLW1        112.58000183 W
SFO1         125.7779080 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2              80.00 usec
PLW2         18.74300003 W
PLW12         0.36736000 W
PLW13    0 W
SFO2         500.1620006 MHz

F2 - Processing parameters
SI                 32768
SF           125.7653279 MHz
WDW                   EM
SSB      0
LB                  1.00 Hz
GB       0
PC                  1.40
```



27

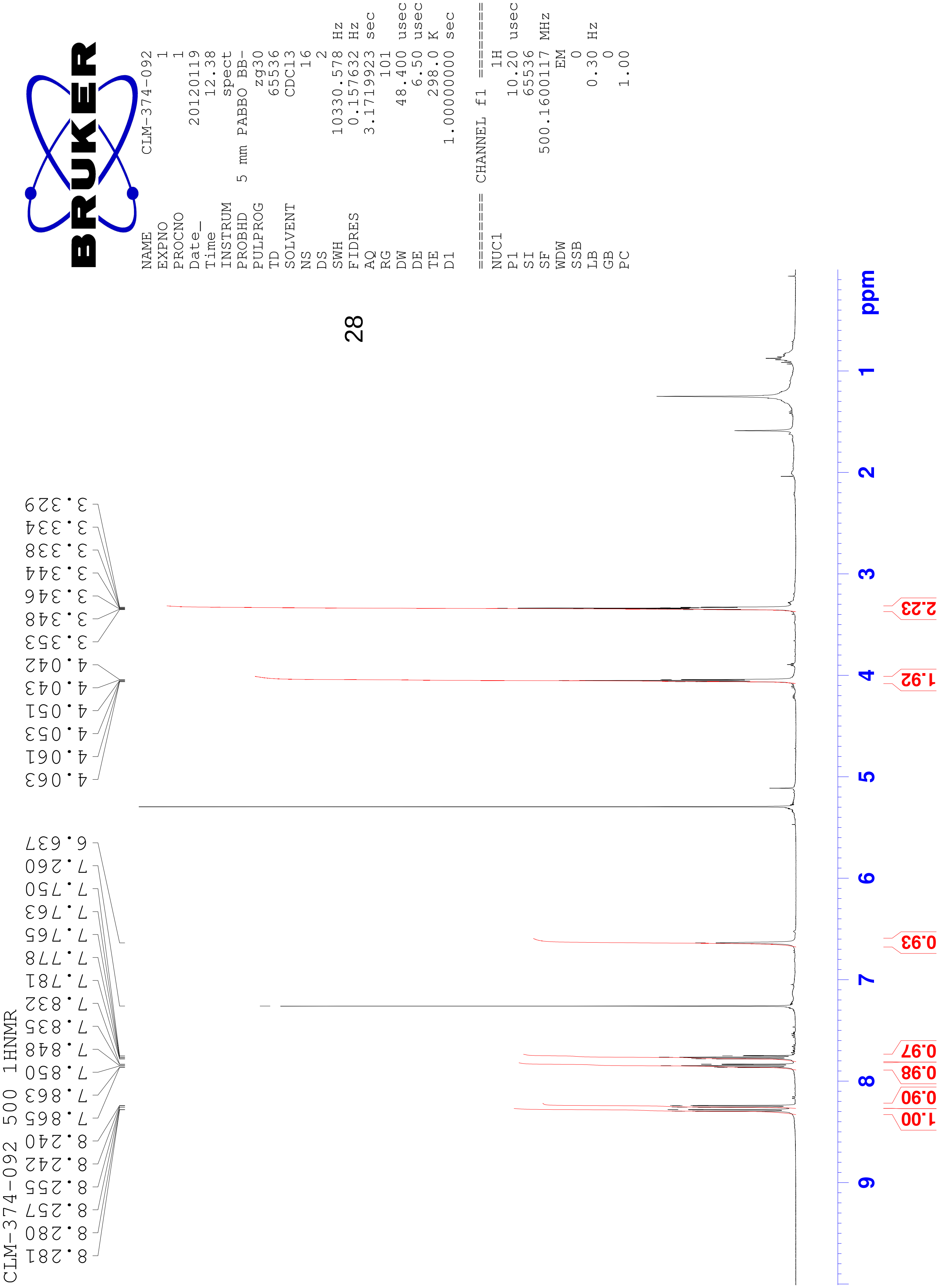
```
Current Data Parameters
NAME          CLM-374-057
EXPNO                   2
PROCNO                  1

F2 - Acquisition Parameters
Date_            20120117
Time                21.04
INSTRUM             spect
PROBHD   5 mm PABBO BB-
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                     16
DS                      2
SWH             10330.578 Hz
FIDRES           0.157632 Hz
AQ              3.1719923 sec
RG                   80.6
DW                 48.400 usec
DE                   6.50 usec
TE                  298.0 K
D1             1.00000000 sec

======== CHANNEL f1 ========
NUC1                   1H
P1                  10.20 usec
PLW1          18.74300003 W
SFO1          500.1630887 MHz

F2 - Processing parameters
SI                  65536
SF            500.1600117 MHz
WDW                    EM
SSB      0
LB                   0.30 Hz
GB       0
PC                   1.00
```

CLM-374-057 13C 300



27

```
Current Data Parameters
NAME          CLM-374-057
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_            20111218
Time                16.58
INSTRUM             spect
PROBHD   5 mm Multinucl
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                  2500
DS                     4
SWH            18028.846 Hz
FIDRES          0.275098 Hz
AQ             1.8175818 sec
RG                  1620
DW                27.733 usec
DE                  6.50 usec
TE                 293.6 K
D1            2.00000000 sec
D11           0.03000000 sec

======== CHANNEL f1 ========
NUC1                 13C
P1                 12.50 usec
PLW1         16.99006081 W
SFO1          75.5004428 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2             100.00 usec
PLW2         18.20941925 W
PLW12         0.31717300 W
PLW13    0 W
SFO2         300.2312009 MHz

F2 - Processing parameters
SI                 32768
SF            75.4928845 MHz
WDW                   EM
SSB      0
LB                  1.00 Hz
GB       0
PC                  1.40
```


BRUKER
NAME CLM-374-092
EXPNO 1
PROCNO 1
Date_ 20120119
Time 12.38
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1719923 sec
RG 101
DW 48.400 usec
DE 6.50 usec
TE 298.0 K
D1 1.00000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 10.20 usec
SI 65536
SF 500.1600117 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
28
CLM-374-092 500 1HNMR
3.329
3.334
3.338
3.344
3.346
3.348
3.353
4.042
4.043
4.051
4.053
4.061
4.063
6.637
7.260
7.750
7.763
7.765
7.778
7.781
7.832
7.835
7.848
7.850
7.863
7.865
8.240
8.242
8.255
8.257
8.280
8.281
2.23
1.92
0.93
0.97
0.98
0.90
1.00
ppm
1
2
3
4
5
6
7
8
9

Current Data Parameters	
NAME	CLM-374-086
EXPNO	1
PROCNO	1
F2 - Acquisition Parameters	
Date_	20120111
Time	11.58
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	1024
DS	4
SWH	29761.904 Hz
FIDRES	0.454131 Hz
AQ	1.1010548 sec
RG	203
DW	16.800 usec
DE	6.50 usec
TE	298.0 K
D1	2.00000000 sec
D11	0.03000000 sec
======== CHANNEL f1 ========	
NUC1	13C
P1	8.80 usec
PLW1	112.58000183 W
SFO1	125.7779080 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PLW2	18.74300003 W
PLW12	0.36736000 W
PLW13	0 W
SFO2	500.1620006 MHz
F2 - Processing parameters	
SI	32768
SF	125.7653152 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

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CLM-374-086 13C