# Completely $N^1$-Selective Palladium-Catalyzed Arylation of Unsymmetric Imidazoles: Application to the Synthesis of Nilotinib

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*Supporting Information*

**Complete Author Lists for Ref 1a**
Kantarjian, H.; Giles, F.; Wunderle, L.; Bhalla, K.; O'Brien, S.; Wassmann, B.; Tanaka, C.; Manley, P.; Rae, P.; Mietlowski, W.; Bochinski, K.; Hochhaus, A.; Griffin, J. D.; Hoelzer, D.; Albitar, M.; Dugan, M.; Cortes, J.; Alland, L.; Ottmann, O. G.

**Complete Author Lists for Ref 20d**
Lee, C. S.; Lee, T. H.; Yoon, S. K.; Choi, J. S.; Jang, Y. J.; Kim, S. W.; Chang, H. K.; Park, M. J.; Kim, T. H.; Ahn, Y. H.; Park, H. D.; Park, H. J.; Lim, D. C.; Lee, J. Y.; Lee, S. H.; Park, W. S.; Oh, Y. S.

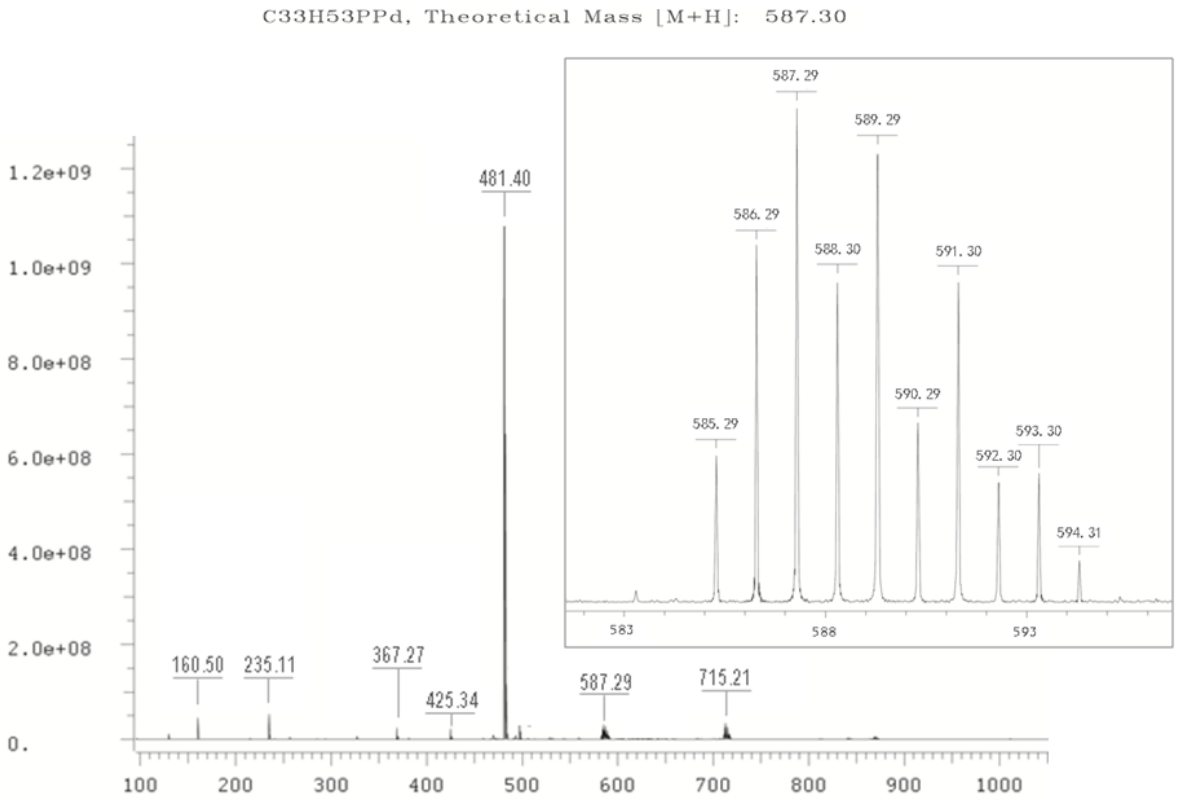
**General Procedures**
All reactions were carried out under an argon atmosphere. Toluene and THF were purchased from J.T. Baker in a CYCLE-TAINER solvent-deliver keg and vigorously purged with argon for 1 hour. The solvent was further purified by passing through successive alumina and Q5 reactant-packed columns on a solvent purification system. The 1,4-dioxane and *t*-BuOH were purchased from Aldrich Chemical Co. in Sure-Seal bottles and were used as received. $Pd_2(dba)_3$ was purchased from Strem Chemicals Inc. and aryl halides and imidazoles were purchased from Aldrich Chemical Co., Alfa Aesar, TCI America or Matrix Scientific and were used without further purification. Anhydrous tribasic potassium phosphate was purchased from Alfa Aesar and potassium *tert*-butoxide were purchased from Aldrich Chemical Co. and stored in a glovebox. Small portions were removed and stored in a desiccator for up to 2 weeks (All reactions were set-up outside of the glovebox). **L1**[1] and **L3** precatalyst[2] were prepared by literature procedures.
Reactions were monitored by GC and thin-layer chromatography (TLC) carried out on 0.25 mm E. Merck silica gel plates (60F-254) using UV light. Flash silica gel chromatography was performed using Silicycle SiliaFlashP60 (230-400 mesh) silica gel. All compounds were characterized by $^1$H NMR, $^{13}$C NMR and IR spectroscopy. Copies of the $^1$H and $^{13}$C NMR spectra can be found at the end of the Supporting Information. Nuclear Magnetic Resonance spectra were recorded on a Bruker 400 MHz instrument. All $^1$H NMR experiments are reported in δ units, parts per million (ppm), and were measured relative to the signals for residual chloroform (7.26 ppm) or dimethylsulfoxide-*d6* (2.50 ppm) in the deuterated solvent, unless otherwise stated. All $^{13}$C NMR spectra are reported in ppm relative to deuterochloroform (77.23 ppm) or dimethylsulfoxide-*d6* (39.52 ppm), unless otherwise stated, and all were obtained with $^1$H decoupling. All IR spectra were taken on a Perkin – Elmer 2000 FTIR. All GC analyses were performed on a Agilent 6890 gas chromatograph with an FID detectorusing a J & W DB-1 column (10 m, 0.1 mm I.D.). Elemental analyses were performed by Atlantic Microlabs Inc., Norcross, GA. ESI-MS spectra were recorded on a Bruker Daltonics APEXIV 4.7 Tesla Fourier Transform Ion Cyclotron Resonance Mass Spectrometer (FT-ICR-MS). The pure compounds are estimated to be ≥ 95% pure as determined by $^1$H NMR and GC analysis

**$^{31}$P NMR experiments (Figure 3)**
*Figure 3 (a)* An oven-dried vial was equipped with a magnetic stir bar and charged with $Pd_2(dba)_3$ (6.9 mg, 0.0075 mmol), **L1** (8.6 mg, 0.018 mol) and 4-methylimidazole (20 mg, 0.24 mmol). The vial was sealed with a screw-cap septum, and then evacuated and backfilled with argon (this process was repeated a total

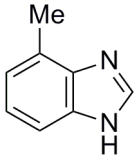
of 3 times). Anhydrous toluene (0.8 mL) was added via syringe. The resulting dark purple mixture was stirred at 120 °C for 3 min, at this point the color of the mixture turned to light green and Pd-black precipitate was observed. After cooling to room temperature the mixture was transferred to an Ar-filled septum-sealed NMR tube via syringe and $^{31}$P NMR spectrum was recorded.

*Figure 3 (b)* An oven-dried vial was equipped with a magnetic stir bar and charged with $Pd_2(dba)_3$ (6.9 mg, 0.0075 mmol) and **L1** (8.6 mg, 0.018 mol). The vial was sealed with a screw-cap septum, and then evacuated and backfilled with argon (this process was repeated a total of 3 times). Anhydrous toluene (0.8 mL) was added via syringe. The resulting dark purple mixture was stirred at 120 °C for 3 min, at this point the color of the mixture turned to red-brown. After cooling to room temperature the mixture was transferred to an Ar-filled septum-sealed NMR tube via syringe and $^{31}$P NMR spectrum was recorded.

**ESI-MS spectrum of the crude premixing solution (Figure 3, (b))**



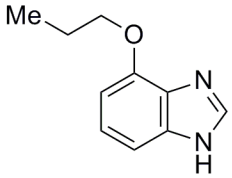

**4-Methylbenzimidazole**




A mixture of 2,3-diaminotoluene (2.44 g, 20 mmol), 95% formic acid (3.18 mL, 80 mmol) and $H_2O$ (12 mL) was stirred at 100 °C for 16 h in a sealed tube. After cooling to room temperature, the mixture was diluted with EtOAc and the organic phase was washed 3 times with sat. aq. $NaHCO_3$, dried over $MgSO_4$ and concentrated in vacuo. The crude product was purified via flash chromatography (EtOAc/MeOH, 40:1) to provide the title compound as a light brown solid (2.41 g, 90%), mp 139 °C. $^1$H NMR (400 MHz,

Acetone-$d_6$) δ 11.32 (br, 1H), 8.24 (s, 1H), 7.46 (d, $J$ = 8.0 Hz, 1H), 7.13 (t, $J$ = 7.6 Hz, 1H), 7.03 (d, $J$ = 7.2 Hz, 1H), 2.59 (s, 3H); $^{13}$C NMR (100 MHz, $CDCl_3$) δ 141.7, 126.5, 123.0, 113.2, 17.1 (Signals for C4 and bridgehead carbons were too weak to observed due to the tautomeric nature of the title compound); IR (film) νmax 2810, 1762, 1601, 1487, 1449, 1295, 1249, 1167, 946, 881, 746, 634 cm$^{-1}$; Anal. Calcd. For $C_8H_8N_2$: C, 72.70; H, 6.10. Found: C, 72.33; H, 6.05.

**4-Propoxybenzimidazole**




A mixture of 3-propoxybenzene-1,2-diamine[3] (3.0 g, 18 mmol), 95% formic acid (2.86 mL, 72 mmol) and $H_2O$ (10 mL) was stirred at 100 °C for 16 h in a sealed tube. After cooling to room temperature, the mixture was diluted with EtOAc and the organic phase was washed 3 times with sat. aq. $NaHCO_3$, dried over $MgSO_4$ and concentrated in vacuo. The crude product was purified via flash chromatography (EtOAc/MeOH, 30:1) to provide the title compound as a light brown solid (2.53 g, 80%), mp 160-161 °C. $^1$H NMR (400 MHz, DMSO-*d*6) δ 12.66 (br, 1H), 8.11 (s, 1H), 7.16 (br, 1H), 7.07 (t, $J$ = 7.6 Hz, 1H), 6.69 (br, 1H), 4.20-4.02 (m, 2H), 1.90-1.64 (m, 2H), 1.01 (t, $J$ = 7.6 Hz, 3H); $^{13}$C NMR (100 MHz, DMSO-*d*6) δ 140.7, 122.7, 108.0, 104.0, 69.6, 22.3, 10.5 (Signals for C4 and bridgehead carbons were too weak to observed due to the tautomeric nature of the title compound); IR (film) νmax 1597, 1450, 1308, 1252, 1093, 985, 954, 848, 723 cm$^{-1}$; Anal. Calcd. For $C_{10}H_{12}N_2O$: C, 68.16; H, 6.86. Found: C, 68.20; H, 6.87.

**General Procedure A: *N*-arylation of imidazoles (with >1.0 mol% Pd)**

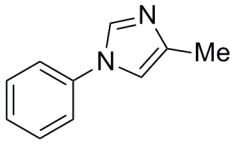
An oven-dried vial was equipped with a magnetic stir bar and charged with $Pd_2(dba)_3$ (0.005-0.0125 mmol) and the **L1** (0.01-0.025 mmol). The vial was sealed with a screw-cap septum, and then evacuated and backfilled with argon (this process was repeated a total of 3 times). Anhydrous toluene (0.83 mL) and anhydrous 1,4-dioxane (0.17 mL) were added via syringe. The resulting dark purple mixture was stirred at 120 °C for 3 min, at this point the color of the mixture turned to red-brown. A second oven-dried vial, which was equipped with stir bar, was charged with imidazole derivative (1.1-1.2 mmol) and $K_3PO_4$ (424 mg, 2.0 mmol) (aryl halides that were solid at room temperature were added at this point). The vial was sealed with a screw-cap septum, and then evacuated and backfilled with argon (this process was repeated a total of 3 times) and then aryl halide and the premixed catalyst solution were added by syringe to the second vial. The reaction mixture was heated at 120 °C for 5 h. The reaction was cooled to room temperature, diluted with EtOAc, washed with brine, dried over $MgSO_4$, concentrated in vacuo and purified via flash chromatography.

**General Procedure B: *N*-arylation of imidazoles (with 0.5 mol% Pd)**

An oven-dried vial was equipped with a magnetic stir bar and charged with $Pd_2(dba)_3$ (2.3 mg, 0.0025 mmol) and **L1** (2.4 mg, 0.005 mmol). The vial was sealed with a screw-cap septum, and then evacuated and backfilled with argon (this process was repeated a total of 3 times). Anhydrous toluene (0.3 mL) was added via syringe. The resulting dark purple mixture was stirred at 120 °C for 3 min, at this point the color of the mixture turned to red-brown. A second oven-dried vial, which was equipped with stir bar, was charged with imidazole derivative (1.2 mmol) and $K_3PO_4$ (424 mg, 2.0 mmol) (aryl halides that were solid at room temperature were added at this point). The vial was sealed with a screw-cap septum, and then evacuated and backfilled with argon (this process was repeated a total of 3 times) and then aryl halide and the premixed catalyst solution was added by syringe to the second vial, followed by addition of toluene (0.53 mL) and dioxane (0.17 mL) (total 1.0 mL solvent). The reaction mixture was heated at 120 °C for 5 h. The reaction was cooled to room temperature, diluted with EtOAc, washed with brine,

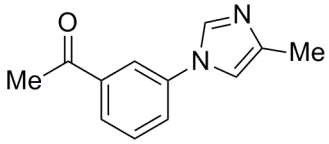
dried over $MgSO_4$, concentrated in vacuo and purified via flash chromatography.
*Note*: When using 0.5 mol% catalyst loading, premixing should be performed at 3.5 mM $Pd_2(dba)_3$ or higher concentration. Otherwise, lower catalytic activity was observed due to the formation of Pd-black during the premixing stage.

**4-Methyl-1-phenyl-1*H*-imidazole (Table 1, entry 13)**



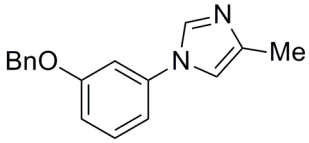

Following general procedure A, a mixture of bromobenzene (116 μL, 1.0 mmol), 4-methylimidazole (98 mg, 1.2 mmol), $K_3PO_4$ (424 mg, 2.0 mmol), $Pd_2(dba)_3$ (6.9 mg, 0.0075 mmol), **L1** (8.6 mg, 0.018 mmol) and toluene-dioxane (5:1, 1.0 mL) was heated to 120 °C for 5 h. The crude product was purified via flash chromatography (EtOAc/MeOH, 50:1) to provide the title compound as a light yellow solid (149 mg, 95%), mp 59-60 °C. $^1$H NMR (400 MHz, $CDCl_3$) δ 7.64 (d, *J* = 1.6 Hz, 1H), 7.36-7.29 (m, 2H), 7.25-7.17 (m, 3H), 6.89 (s, 1H), 2.20 (s, 3H); $^{13}$C NMR (100 MHz, $CDCl_3$) δ 139.4, 137.3, 134.4, 129.7, 126.9, 120.8, 114.4, 13.6; IR (film) νmax 3385, 3108, 2921, 1599, 1507, 1448, 1392, 1366, 1291, 1241, 1070, 1003, 969, 817, 759, 692 cm$^{-1}$; Anal. Calcd. For $C_{10}H_{10}N_2$: C, 75.92; H, 6.37. Found: C, 76.01; H, 6.30.

**1-[3-(4-Methyl-imidazol-1-yl)-phenyl]-ethanone (Table 2, entry 1)**



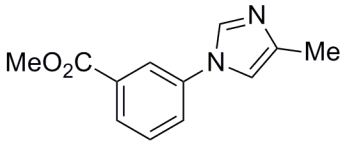

Following general procedure A, a mixture of 3'-bromoacetophenone (132 μL, 1.0 mmol), 4-methylimidazole (98 mg, 1.2 mmol), $K_3PO_4$ (424 mg, 2.0 mmol), $Pd_2(dba)_3$ (6.9 mg, 0.0075 mmol), **L1** (8.6 mg, 0.018 mmol) and toluene-dioxane (5:1, 1.0 mL) was heated to 120 °C for 5 h. The crude product was purified via flash chromatography (EtOAc/MeOH, 25:1) to provide the title compound as a white solid (1st run: 168 mg, 84%; 2nd run:166 mg, 83%), mp 84-86 °C. $^1$H NMR (400 MHz, $CDCl_3$) δ 7.87 (, 1H), 7.85-7.80 (m, 1H), 7.74 (d, *J* = 1.2 Hz, 1 H), 7.53-7.47 (m, 2H), 7.01-6.98 (m, 1H), 2.58 (s, 3H), 2.23 (s, 3H); $^{13}$C NMR (100 MHz, $CDCl_3$) δ 196.9, 140.0, 138.6, 137.9, 134.5, 130.3, 126.9, 125.1, 120.1, 114.4, 26.8, 13.8; IR (film) νmax 3360, 3119, 2926, 2359, 1685, 1593, 1502, 1452, 1364, 1261, 1163, 1073, 1032, 1005, 898, 798, 737, 687, 638, 590, 519 cm$^{-1}$ ; Anal. Calcd. For $C_{12}H_{12}N_2O$: C, 71.98; H, 6.04. Found: C, 71.88; H, 6.01.

**1-(3-(Benzyloxy)phenyl)-4-methyl-1*H*-imidazole (Table 2, entry 2)**



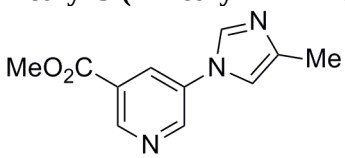

Following general procedure B, a mixture of 1-(benzyloxy)-3-bromobene (263 mg, 1.0 mmol), 4-methylimidazole (98 mg, 1.2 mmol), $K_3PO_4$ (424 mg, 2.0 mmol), $Pd_2(dba)_3$ (2.3 mg, 0.0025 mmol), **L1** (2.4 mg, 0.005 mmol) and toluene-dioxane (5:1, 1.0 mL) was heated to 120 °C for 5 h. The crude product was purified via flash chromatography (EtOAc/MeOH, 25:1) to provide the title compound as a pale yellow solid (1st run: 247 mg, 94%; 2nd run: 243 mg, 92%), mp 57-58 °C. $^1$H NMR (400 MHz, $CDCl_3$) δ 7.66 (d, *J* = 1.2 Hz, 1H), 7.39-7.29 (m, 4H), 7.29-7.22 (m, 2H), 6.92-6.82 (m, 4H), 5.00 (s, 2H), 2.24 (s, 3 H); $^{13}$C NMR (100 MHz, $CDCl_3$) δ 159.7, 139.3, 138.4, 136.2, 134.4, 130.6, 128.6, 128.1, 127.4, 114.4, 113.2, 112.9, 108.0, 70.1, 13.7; IR (film) νmax 3379, 3092, 3034, 2922, 1603, 1502, 1453, 1384, 1259, 1226, 1183, 1070, 1030, 988, 868, 838, 772, 741, 692, 626, 460 cm$^{-1}$; Anal. Calcd. For $C_{17}H_{16}N_2O$: C, 77.25; H, 6.10. Found: C, 76.92; H, 6.16.

**Methyl 5-(4-methyl-1*H*-imidazol-1-yl)benzoate (Table 2, entry 3)**



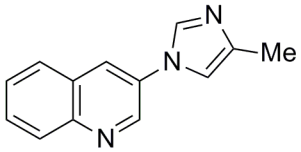

Following general procedure B, a mixture of methyl-3-bromobenzoate (215 mg, 1.0 mmol), 4-methylimidazole (98 mg, 1.2 mmol), $K_3PO_4$ (424 mg, 2.0 mmol), $Pd_2(dba)_3$ (2.3 mg, 0.0025 mmol), **L1** (2.4 mg, 0.005 mmol) and toluene-dioxane (5:1, 1.0 mL) was heated to 120 °C for 5 h. The crude product was purified via flash chromatography (EtOAc/MeOH, 20:1) to provide the title compound as a pale yellow solid (1st run: 194 mg, 90%; 2nd run: 198 mg, 92%), mp 50-51 °C. $^1$H NMR (400 MHz, $CDCl_3$) δ 7.88-7.80 (m, 2H), 7.68 (d, *J* = 1.6 Hz, 1H), 7.43-7.34 (m, 2H), 6.92 (s, 1H), 3.80 (s, 3H), 2.16 (s, 3H); $^{13}$C NMR (100 MHz, $CDCl_3$) δ 165.8, 139.8, 137.4, 134.3, 131.8, 129.8, 127.7, 124.7, 121.4, 114.2, 52.3, 13.6; IR (film) νmax 3385, 3115, 2953, 1723, 1593, 1503, 1456, 1293, 1114, 1072, 1004, 962, 816, 756, 683, 625, 522 cm$^{-1}$; Anal. Calcd. For $C_{12}H_{12}N_2O_2$: C, 66.65; H, 5.59. Found: C, 66.95; H, 5.68.

**Methyl 5-(4-methyl-1*H*-imidazol-1-yl)nicotinate (Table 2, entry 4)**




Following general procedure A, a mixture of methyl-3-bromonicotinate (216 mg, 1.0 mmol), 4-methylimidazole (98 mg, 1.2 mmol), $K_3PO_4$ (424 mg, 2.0 mmol), $Pd_2(dba)_3$ (6.9 mg, 0.0075 mmol), **L1** (8.6 mg, 0.018 mmol) and toluene-dioxane (5:1, 1.0 mL) was heated to 120 °C for 5 h. The crude product was purified via flash chromatography (EtOAc/MeOH, 15:1) to provide the title compound as a pale yellow solid (1st run: 180 mg, 83%; 2nd run: 186 mg, 86%), mp 124-126 °C. $^1$H NMR (400 MHz, $CDCl_3$) δ 9.09 (d, *J* = 1.6 Hz, 1H), 8.82 (d, *J* = 2.4 Hz, 1H), 8.22 (dd, *J* = 2.2, 1.6 Hz, 1H), 7.80 (d, *J* = 1.2 Hz, 1H), 7.03 (s, 1H), 3.93 (s, 3H), 2.24 (s, 3 H); $^{13}$C NMR (100 MHz, $CDCl_3$) δ 164.8, 148.9, 145.6, 140.9, 134.5, 133.9, 128.7, 126.9, 114.1, 52.9, 13.7; IR (film) νmax 3397, 3101, 2361, 1715, 1508, 1437, 1313, 1165, 1125, 1071, 1011, 954, 844, 764, 689 cm$^{-1}$; Anal. Calcd. For $C_{11}H_{11}N_3O_2$: C, 60.82; H, 5.10. Found: C, 60.84; H, 5.20.

**3-(4-Methyl-imidazol-1-yl)-quinoline (Table 2, entry 5)**

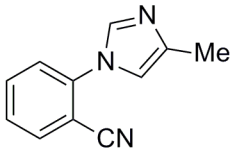



Following general procedure B, a mixture of 3-bromoquinoline (136 μL, 1.0 mmol), 4-methylimidazole (98 mg, 1.2 mmol), $K_3PO_4$ (424 mg, 2.0 mmol), $Pd_2(dba)_3$ (2.3 mg, 0.0025 mmol), **L1** (2.4 mg, 0.018 mmol) and toluene-dioxane (5:1, 1.0 mL) was heated to 120 °C for 5 h. The crude product was purified via flash chromatography (EtOAc/MeOH, 10:1) to provide the title compound as a white solid (1st run: 192 mg, 92%; 2nd run: 200 mg, 96%), mp 122-123 °C. $^1$H NMR (400 MHz, $CDCl_3$) δ 8.82 (d, *J* = 2.4 Hz, 1H), 7.98 (d, *J* = 8.4 Hz, 1H), 7.87 (d, *J* = 2.0 Hz, 1H), 7.75 (s, 1H), 7.68 (d, *J* = 8.0 Hz, 1H), 7.60-7.54 (m, 1H), 7.48-7.42 (m, 1H), 6.98 (s, 1H), 2.20 (s, 3H); $^{13}$C NMR (100 MHz, $CDCl_3$) δ 146.7, 144.0, 140.2, 134.6, 130.6, 129.5, 129.3, 127.8, 127.5, 127.4, 125.6, 114.3, 13.6; IR (film) νmax 3058, 2981, 2939, 2901, 1646, 1626, 1600, 1510, 1464, 1378, 1251, 1212, 1162, 1116, 1054, 981, 847, 809, 746, 625, 473 cm$^{-1}$; Anal. Calcd. For $C_{13}H_{11}N_3$: C, 74.62; H, 5.30. Found: C, 74.91; H, 5.36.

**1-(2-Fluorophenyl)-4-methyl-1*H*-imidazole (Table 2, entry 6)**

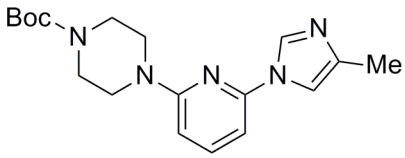

Following general procedure A, a mixture of 1-bromo-2-fluorobenzene (109 μL, 1.0 mmol), 4-methylimidazole (98 mg, 1.2 mmol), $K_3PO_4$ (424 mg, 2.0 mmol), $Pd_2(dba)_3$ (6.9 mg, 0.0075 mmol), **L1** (8.6 mg, 0.018 mmol) and toluene-dioxane (5:1, 1.0 mL) was heated to 120 °C for 5 h. The crude product was purified via flash chromatography (EtOAc/MeOH, 20:1) to provide the title compound as a pale yellow solid (1st run: 163 mg, 93%; 2nd run: 168 mg, 96%), mp 56-57 °C. $^1$H NMR (400 MHz, $CDCl_3$) δ 7.57 (t, *J* = 1.6 Hz, 1H), 7.24-7.12 (m, 2H), 7.11-7.03 (m, 2H), 6.82 (s, 1H), 2.15 (s, 3H); $^{13}$C NMR (100 MHz, $CDCl_3$) δ 154.7 (d, *J* = 249 Hz), 138.7, 136.1 (d, *J* = 5.0 Hz), 128.4 (d, *J* = 8.0 Hz), 125.4 (d, *J* = 11 Hz), 124.9, 124.8 (d, *J* = 29 Hz), 117.0 (d, *J* = 21 Hz), 115.7 (d, *J* = 3.0 Hz), 13.5; IR (film) νmax 3389, 2926, 1616, 1517, 1457, 1292, 1241, 1111, 1068, 972, 817, 759, 631, 470 cm$^{-1}$; Anal. Calcd. For $C_{10}H_9FN_2$: C, 68.17; H, 5.15. Found: C, 68.25; H, 5.09.

**2-(4-Methyl-1*H*-imidazol-1-yl)benzonitrile (Table 2, entry 7)**



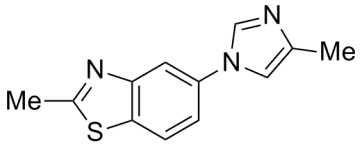

Following general procedure A, a mixture of 2-bromobenzonitrile (182 mg, 1.0 mmol), 4-methylimidazole (98 mg, 1.2 mmol), $K_3PO_4$ (424 mg, 2.0 mmol), $Pd_2(dba)_3$ (9.2 mg, 0.01 mmol), **L1** (10.6 mg, 0.022 mmol) and toluene-dioxane (5:1, 1.0 mL) was heated to 120 °C for 5 h. The crude product was purified via flash chromatography (EtOAc/MeOH, 25:1) to provide the title compound as a white solid (1st run: 161 mg, 88%; 2nd run: 159 mg, 87%), mp 128-129 °C. $^1$H NMR (400 MHz, $CDCl_3$) δ 7.75-7.62 (m, 3 H), 7.31 (t, *J* = 7.6 Hz, 1H), 7.37 (d, *J* = 8.0 Hz, 1 H), 7.01 (s, 1H), 2.23 (s, 3H); $^{13}$C NMR (100 MHz, $CDCl_3$) δ 139.8, 139.4, 135.8, 134.5, 134.4, 128.1, 125.4, 116.1, 115.9, 107.5, 13.7; IR (film) νmax 3376, 3235, 2361, 2226, 1670, 1598, 1504, 1452, 1320, 1291, 1240, 1067, 973, 824, 766, 631, 515 cm$^{-1}$; Anal. Calcd. For $C_{11}H_9N_3$: C, 72.11; H, 4.95. Found: C, 71.93; H, 4.93.

***tert*-Butyl 4-(6-(4-methyl-1*H*-imidazol-1-yl)pyridin-2-yl)piperazine-1-carboxylate (Table 2, entry 8)**



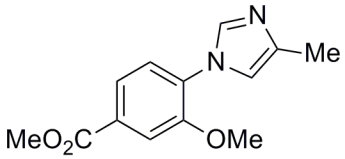

Following general procedure A, a mixture of *tert*-butyl 4-(6-bromopyridin-2-yl)piperazine-1-carboxylate[4] (342 mg, 1.0 mmol), 4-methylimidazole (98 mg, 1.2 mmol), $K_3PO_4$ (424 mg, 2.0 mmol), $Pd_2(dba)_3$ (9.2 mg, 0.01 mmol), **L1** (9.6 mg, 0.02 mmol) and toluene-dioxane (5:1, 1.0 mL) was heated to 120 °C for 5 h. The crude product was purified via flash chromatography (EtOAc-MeOH, 30:1) to provide the title compound as a white solid (1st run: 271 mg, 79%; 2nd run: 275 mg, 80%), mp 77-79 °C. $^1$H NMR (400 MHz, $CDCl_3$) δ 8.06 (d, *J* = 1.2 Hz, 1H), 7.44-7.38 (m, 1H), 7.16 (d, *J* = 1.2 Hz, 1H), 6.46 (d, *J* = 8.0 Hz, 1H), 6.37 (d, *J* = 7.6 Hz, 1H), 3.41 (s, 8H), 2.13 (s, 3H), 1.34 (s, 9H); $^{13}$C NMR (100 MHz, $CDCl_3$) δ 158.1, 154.6, 147.4, 140.0, 139.1, 134.0, 112.4, 104.1, 100.0, 79.9, 44.6, 42.9, 28.3, 13.7; IR (film) νmax 3373, 2976, 2860, 2362, 2205, 1695, 1606, 1457, 1242, 1128, 1080, 1003, 932, 780, 730, 614, 536 cm$^{-1}$.

**2-Methyl-5-(4-methyl-1*H*-imidazol-1-yl)benzothiazole (Table 2, entry 9)**



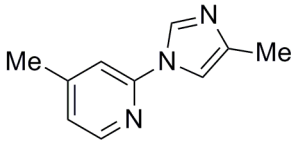
Following general procedure A, a mixture of 5-chloro-2-methylbenzothiazole (184 mg, 1.0 mmol), 4-methylimidazole (98 mg, 1.2 mmol), $K_3PO_4$ (424 mg, 2.0 mmol), $Pd_2(dba)_3$ (6.9 mg, 0.0075 mmol), **L1** (8.6 mg, 0.018 mmol) and toluene-dioxane (5:1, 1.0 mL) was heated to 120 °C for 5 h. The crude product was purified via flash chromatography (EtOAc/MeOH, 20:1) to provide the title compound as a white solid (1st run: 183 mg, 80%; 2nd run: 179 mg, 78%), mp 125-126 °C. $^1$H NMR (400 MHz, $CDCl_3$) δ 7.88-7.73 (m, 3H), 7.33-7.27 (m, 1H), 7.00 (d, *J* = 1.2 Hz, 1H), 2.80 (s, 3H), 2.27 (s, 3H); $^{13}$C NMR (100 MHz, $CDCl_3$) δ 169.7, 154.2, 139.8, 136.0, 134.9, 134.2, 122.5, 118.3, 115.0, 114.7, 20.4, 13.8; IR (film) νmax 3394, 3104, 2920, 1610, 1498, 1432, 1295, 1256, 1173, 1054, 864, 804, 737, 644, 620, 537, 435 cm$^{-1}$; Anal. Calcd. For $C_{12}H_{11}N_3S$: C, 62.86; H, 4.84. Found: C, 62.56; H, 4.80.

**3-Methoxy-4-(4-methyl-imidazol-1-yl)-benzoic acid methyl ester (Table 2, entry 10)**



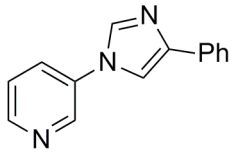
Following general procedure A, a mixture of 3-methoxy-4-trifluoromethanesulfonyloxy-benzoic acid methyl ester (312 mg, 1.0 mmol), 4-methylimidazole (98 mg, 1.2 mmol), $K_3PO_4$ (424 mg, 2.0 mmol), $Pd_2(dba)_3$ (6.9 mg, 0.0075 mmol), **L1** (8.6 mg, 0.018 mmol) and toluene-dioxane (5:1, 1.0 mL) was heated to 120 °C for 5 h. The crude product was purified via flash chromatography (EtOAc/MeOH, 20:1) to provide the title compound as a white solid (1st run: 188 mg, 77%; 2nd run: 186 mg, 76%), mp 85-87 °C. $^1$H NMR (400 MHz, dmso-*d6*) δ 7.92 (d, *J* = 1.6 Hz, 1H), 7.67 (d, *J* = 1.6 Hz, 1H), 7.64 (dd, *J* = 8.0, 1.6 Hz, 1H), 7.53 (d, *J* = 8.0 Hz, 1H), 7.23 (s, 1H), 3.90 (s, 3H), 3.89 (s, 3H), 2.16 (s, 3H); $^{13}$C NMR (100 MHz, dmso-*d6*) δ 166.1, 151.9, 137.2, 136.8, 130.4, 123.0, 124.6, 122.8, 116.6, 113.5, 56.3, 52.6, 13.2; IR (film) νmax 2593, 2362, 1719, 1520, 1457, 1293, 1240, 1157, 1030, 982, 879, 832, 763, 637 cm$^{-1}$.

**4-Methyl-2-(4-methyl-imidazol-1-yl)-pyridine (Table 2, entry 11)**



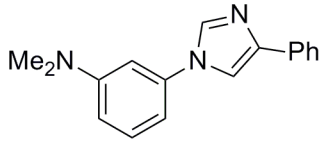
Following general procedure A, a mixture of trifluoromethanesulfonic acid 4-methoxy-pyridin-2-yl ester (170 μL, 1.0 mmol), 4-methylimidazole (98 mg, 1.2 mmol), $K_3PO_4$ (424 mg, 2.0 mmol), $Pd_2(dba)_3$ (6.9 mg, 0.0075 mmol), **L1** (8.6 mg, 0.018 mmol) and toluene-dioxane (5:1, 1.0 mL) was heated to 120 °C for 5 h. The crude product was purified via flash chromatography (EtOAc/MeOH, 20:1) to provide the title compound as pale yellow oil (1st run: 140 mg, 81%; 2nd run: 136 mg, 79%). $^1$H NMR (400 MHz, $CDCl_3$) δ 8.19 (d, *J* = 4.8 Hz, 1H), 8.15 (s, 1H), 7.24 (s, 1H), 7.01 (s, 1H), 6.92 (d, *J* = 4.4 Hz, 1H), 2.32 (s, 3H), 2.21 (s, 3H); $^{13}$C NMR (100 MHz, $CDCl_3$) δ 150.5, 149.3, 148.6, 139.7, 134.2, 122.7, 112.6, 112.5, 21.2, 13.8; IR (film) νmax 3402, 2926, 1613, 1489, 1447, 1291, 1174, 1079, 1032, 827, 728, 634, 521, 447 cm$^{-1}$.

**3-(4-Phenyl-1*H*-imidazol-1-yl)pyridine (Table 2, entry 12)**



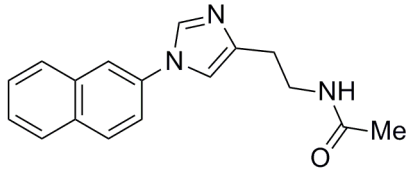
Following general procedure A, a mixture of 3-chloropyridine (95 μL, 1.0 mmol), 4-phenylimidazole (173 mg, 1.2 mmol), $K_3PO_4$ (424 mg, 2.0 mmol), $Pd_2(dba)_3$ (6.9 mg, 0.0075 mmol), **L1** (8.6 mg, 0.018 mmol) and toluene-dioxane (5:1, 1.0 mL) was heated to 120 °C for 5 h. The crude product was purified via flash chromatography (100% EtOAc) to provide the title compound as an off-white solid (1st run: 197 mg, 89%; 2nd run: 194 mg, 88%), mp 137-139 °C. $^1$H NMR (400 MHz, $CDCl_3$) δ 8.70 (d, *J* = 2.4 Hz, 1H), 8.54 (d, *J* = 4.8 Hz, 1H), 7.82 (s, 1H), 7.75 (d, *J* = 7.6 Hz, 2H), 7.67-7.61 (m, 1H), 7.48 (s, 1H), 7.38-7.26 (m, 3H), 7.25-7.17 (m, 1H); $^{13}$C NMR (100 MHz, $CDCl_3$) δ 148.8, 143.9, 142.6, 135.6, 133.7, 133.3, 128.8, 128.6, 127.4, 125.1, 124.3, 113.4; IR (film) νmax 3398, 3095, 2360, 1586, 1501, 1441, 1387, 1284, 1189, 1067, 965, 939, 803, 747, 694, 617 cm$^{-1}$; Anal. Calcd. For $C_{14}H_{11}N_3$: C, 76.00; H, 5.01. Found: C, 75.37; H, 5.09.

**Dimethyl-[3-(4-phenyl-imidazol-1-yl)-phenyl]-amine (Table 2, entry 13)**



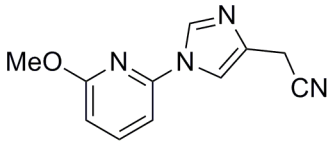
Following general procedure A, a mixture of 3-bromo-*N*,*N*-dimethylaniline (143 μL, 1.0 mmol), 4-phenylimidazole (173 mg, 1.2 mmol), $K_3PO_4$ (424 mg, 2.0 mmol), $Pd_2(dba)_3$ (6.9 mg, 0.0075 mmol), **L1** (8.6 mg, 0.018 mmol) and toluene-dioxane (5:1, 1.0 mL) was heated to 120 °C for 5 h. The crude product was purified via flash chromatography (EtOAc/hexanes, 1:1) to provide the title compound as an off-white solid (1st run: 256 mg, 97%; 2nd run: 250 mg, 95%), mp 104-105 °C. $^1$H NMR (400 MHz, $CDCl_3$) δ 7.89-7.84 (m, 3H), 7.53 (d, *J* = 1.2 Hz, 1H), 7.41-7.36 (m, 2H), 7.28-7.21 (m, 2H), 6.70-6.60 (m, 3H), 2.93 (s, 6H); $^{13}$C NMR (100 MHz, $CDCl_3$) δ 151.4, 142.6, 138.1, 135.7, 134.0, 130.2, 128.6, 126.8, 124.8, 114.0, 111.2, 108.9, 104.8, 40.2; IR (film) νmax 3386, 3073, 2892, 2356, 1604, 1502, 1349, 1244, 1173, 1063, 942, 828, 751, 691, 467 cm$^{-1}$; Anal. Calcd. For $C_{17}H_{17}N_3$: C, 77.54; H, 6.51. Found: C, 77.21; H, 6.38.

***N*-(2-(1-(Naphthalen-2-yl)-1*H*-imidazol-4-yl)ethyl)acetamide (Table 2, entry 14)**



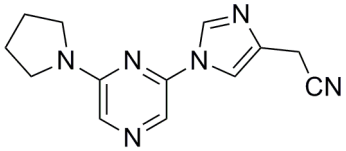
Following general procedure A, a mixture of 2-bromonaphthalene (207 mg, 1.0 mmol), *N*-acetylhistamine (184 mg, 1.2 mmol), $K_3PO_4$ (424 mg, 2.0 mmol), $Pd_2(dba)_3$ (6.9 mg, 0.0075 mmol), **L1** (8.6 mg, 0.018 mmol) and toluene-dioxane (5:1, 1.0 mL) was heated to 120 °C for 5 h. The crude product was purified via flash chromatography (EtOAc/MeOH, 9:1) to provide the title compound as a pale yellow solid (1st run: 264 mg, 95%; 2nd run: 262 mg, 94%), mp 137-138 °C. $^1$H NMR (400 MHz, $CDCl_3$) δ 7.89-7.74 (m, 4H), 7.69 (s, 1H), 7.52-7.37 (m, 3H), 7.15 (s, 1H), 6.97 (s, 1H), 3.57 (q, *J* = 6.4 Hz, 2H), 2.81 (t, *J* = 6.4 Hz, 2H), 1.96 (s, 3H); $^{13}$C NMR (100 MHz, $CDCl_3$) δ 170.3, 141.6, 135.1, 134.5, 133.5, 132.1, 130.1, 127.9, 127.8, 127.4, 126.5, 119.8, 118.6, 115.2, 39.4, 27.8, 23.4; IR (film) νmax 3274, 3068, 2361, 1652, 1558, 1514, 1373, 1274, 1067, 993, 855, 814, 751, 625, 474 cm$^{-1}$; Anal. Calcd. For $C_{14}H_{15}NO_2$: C, 73.10; H, 6.13. Found: C, 73.09; H, 6.13.

**2-(1-(6-Methoxypyridin-2-yl)-1*H*-imidazol-4-yl)acetonitrile (Table 2, entry 15)**



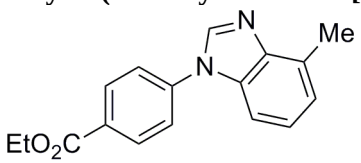
Following general procedure A (0.5 mmol scale), a mixture of 6-bromo-2-methoxypyridine (61 μL, 0.5 mmol), 4-cyanomethylimidazole (64 mg, 0.6 mmol), $K_3PO_4$ (212 mg, 1.0 mmol), $Pd_2(dba)_3$ (3.4 mg, 0.00375 mmol), **L1** (4.3 mg, 0.009 mmol) and toluene-dioxane (5:1, 0.5 mL) was heated to 120 °C for 5 h. The crude product was purified via flash chromatography (EtOAc) to provide the title compound as a white solid (1st run: 98 mg, 92%; 2nd run: 93 mg, 87%), mp 77-78 °C. $^1$H NMR (400 MHz, $CDCl_3$) δ 8.23 (d, *J* = 1.2 Hz, 1H), 7.63 (t, *J* = 8.0 Hz, 1H), 7.58 (d, *J* = 1.2 Hz, 1H), 6.84 (d, *J* = 7.6 Hz, 1H), 6.24 (d, *J* = 8.4 Hz, 1H), 3.91 (s, 3H), 3.73 (s, 2H); $^{13}$C NMR (100 MHz, $CDCl_3$) δ 163.8, 146.5, 141.2, 135.2, 133.0, 117.4, 114.0, 109.2, 103.6, 53.8, 17.9; IR (film) νmax 3745, 3132, 2941, 2361, 1614, 1582, 1475, 1420, 1255, 1150, 1090, 1034, 995, 862, 784, 726, 669, 607, 475 cm$^{-1}$; Anal. Calcd. For $C_{11}H_{10}N_4O$: C, 61.67; H, 4.71. Found: C, 61.41; H, 4.69.

**2-(1-(6-(Pyrrolidin-1-yl)pyrazin-2-yl)-1*H*-imidazol-4-yl)acetonitrile (Table 2, entry 16)**



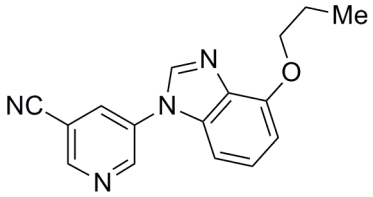
Following general procedure A, a mixture of 2-chloro-6-(pyrrolidin-1-yl)pyrazine (184 mg, 1.0 mmol), 4-cyanomethylimidazole (129 mg, 1.2 mmol), $K_3PO_4$ (424 mg, 2.0 mmol), $Pd_2(dba)_3$ (11.4 mg, 0.0125 mmol), **L1** (12 mg, 0.025 mmol) and toluene-dioxane (5:1, 1.0 mL) was heated to 130 °C for 5 h. The crude product was purified via flash chromatography (EtOAc-MeOH, 30:1) to provide the title compound as a white solid (1st run: 231 mg, 91%; 2nd run: 234 mg, 92%), mp 117-118 °C. $^1$H NMR (400 MHz, $CDCl_3$) δ 8.11 (d, *J* = 1.2 Hz, 1H), 7.75 (s, 1H), 7.63 (s, 1H), 7.48 (d, *J* = 1.2 Hz, 1H), 3.65 (s, 2H), 3.43-3.30 (m, 4H), 1.99-1.85 (m, 4H); $^{13}$C NMR (100 MHz, $CDCl_3$) δ 151.2, 142.6, 134.9, 132.8, 128.6, 118.3, 117.2, 113.5, 46.3, 25.1, 17.6; IR (film) νmax 3400, 3117, 2965, 2869, 2359, 2251, 1590, 1552, 1485, 1349, 1225, 1050, 985, 918, 830, 734, 615, 480 cm$^{-1}$.

**Ethyl 4-(4-methyl-1*H*-benzo[*d*]imidazol-1-yl)benzoate (Table 2, entry 17)**



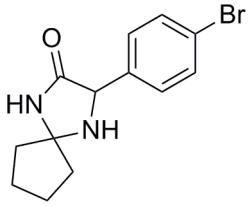
Following general procedure A, a mixture of ethyl 4-chlorobenzoate (156 μL, 1.0 mmol), 4-methylbenzimidazole (145 mg, 1.1 mmol), $K_3PO_4$ (424 mg, 2.0 mmol), $Pd_2(dba)_3$ (4.6 mg, 0.005 mmol), **L1** (4.8 mg, 0.01 mmol) and toluene-dioxane (5:1, 1.0 mL) was heated to 120 °C for 5 h. The crude product was purified via flash chromatography (Hexanes/EtOAc, 1:1) to provide the title compound as a pale yellow solid (1st run: 254 mg, 91%; 2nd run: 249 mg, 89%), mp 107-108 °C. Regiochemistry of the title compound was confirmed by strong NOE between $H^7$ proton of the benzimidazole ring and $H^{3/5}$ proton of the benzoate ring. $^1$H NMR (400 MHz, $CDCl_3$) δ 8.25-8.20 (m, 2H), 8.11 (s, 1H), 7.60-7.55 (m, 2H), 7.40-7.36 (m, 1H), 7.25-7.20 (m, 1H), 7.16-7.11 (m, 1H), 4.41 (q, *J* = 7.2 Hz, 2H), 2.70 (s, 3H), 1.40 (t, *J* = 7.6 Hz, 3H); $^{13}$C NMR (100 MHz, $CDCl_3$) δ 165.6, 143.6, 141.1, 140.3, 132.8, 131.6, 130.9, 129.7, 124.1, 123.6, 123.2, 108.0, 61.4, 16.8, 14.4; IR (film) νmax 3410, 2981, 1715, 1603, 1501, 1369, 1277, 1177, 1106, 1019, 855, 762, 706, 622 cm$^{-1}$; Anal. Calcd. For $C_{17}H_{16}N_2O_2$: C, 72.84; H, 5.75. Found: C, 72.88; H, 5.71.

**5-(4-Propoxy-1*H*-benzo[*d*]imidazol-1-yl)nicotinonitrile (Table 2, entry 18)**



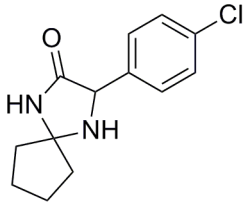

Following general procedure A, a mixture of 2-bromo-5-cyanopyridine (183 mg, 1.0 mmol), 4-propoxybenzimidazole (194 mg, 1.1 mmol), $K_3PO_4$ (424 mg, 2.0 mmol), $Pd_2(dba)_3$ (6.9 mg, 0.0075 mmol), **L1** (8.6 mg, 0.018 mmol) and toluene-dioxane (5:1, 1.0 mL) was heated to 120 °C for 5 h. The crude product was purified via flash chromatography (Hexanes/EtOAc, 2:3) to provide the title compound as an off-white solid (1st run: 239 mg, 86%; 2nd run: 236 mg, 85%), mp 121-122 °C. Regiochemistry of the title compound was confirmed by strong NOE between $H^7$ proton of the benzimidazole ring and $H^{4/6}$ proton of the pyridine ring. $^1$H NMR (400 MHz, $CDCl_3$) δ 9.04-9.01 (m, 1H), 8.90-8.80 (m, 1H), 8.16-8.13 (m, 1H), 8.01 (s, 1H), 7.28-7.22 (m, 1H), 7.03 (dd, *J* = 0.8, 7.2 Hz, 1H), 6.76 (dd, *J* = 0.8, 7.2 Hz, 1H), 4.15 (t, *J* = 6.8 Hz, 2H), 1.96-1.85 (m, 2H), 1.05 (t, *J* = 7.6 Hz, 3H); $^{13}$C NMR (100 MHz, $CDCl_3$) δ 151.8, 151.0, 148.2, 139.8, 134.4, 133.7, 133.6, 133.4, 125.8, 115.4, 111.0, 105.7, 102.0, 70.5, 22.5, 10.6; IR (film) νmax 3428, 2967, 1594, 1496, 1462, 1282, 1216, 1087, 1002, 900, 828, 779, 737, 699 cm$^{-1}$; Anal. Calcd. For $C_{16}H_{16}N_4O$: C, 69.05; H, 5.07. Found: C, 68.94; H, 5.10.

**3-(4-Bromophenyl)-1,4-diazaspiro[4.4]nonan-2-one**



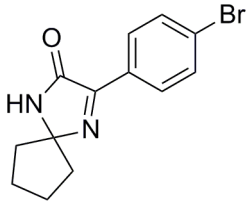

To a stirred solution of 4-bromobenzaldehyde (18.0 g 97.3 mmol) in EtOH (45 mL) in a 300 mL round-bottomed flask, were added a solution of $NH_4OAc$ (22.5 g, 292 mmol) and NaCN (5.24 g, 107 mmol) in $H_2O$ (16.4 mL) and 30% aq. $NH_4OH$ (38 mL) at room temperature. The reaction mixture was stirred at room temperature for 4 h and then TBME (*tert*-butyl methyl ether) (100 mL) was added. The mixture was transferred to a separatory funnel and the aqueous phase was removed. The organic phase was washed with $H_2O$ (80 mL, 2 times), brine (80 mL) and then, $H_2O$ (60 mL). The organic phase was dried over $MgSO_4$ and concentrated to ca. 30 mL. *n*-BuOH (180 mL) was added to the concentrated solution followed by cyclopentanone (21.5 mL, 243 mmol) and NaOEt (21 wt% in EtOH, 3.7 mL). The reaction mixture was stirred at 80 °C for 16 h. The dark-brown solution was concentrated to ca. 2/3 volume (white precipitate appeared at this point) and then cooled to 0 °C (ice bath). The precipitate was collected by filtration, washed 3-times with cold EtOH and dried in vacuo to give the title compound as an off-white solid (10.9 g, 38%), mp 178-179 °C. $^1$H NMR (400 MHz, dmso-*d6*) δ 8.55 (s, 1H), 7.52 (d, *J* = 8.4 Hz, 2H), 7.43 (d, *J* = 8.4 Hz, 2H), 4.52 (d, *J* = 8.4 Hz, 1H), 3.61 (d, *J* = 8.8 Hz, 1H), 1.89-1.55 (m, 8H); $^{13}$C NMR (100 MHz, dmso-*d6*) δ 173.4, 139.8, 130.7, 129.4, 120.1, 81.6, 61.1, 40.3, 38.7, 22.6, 22.4; IR (film) νmax 3853, 3647, 2362, 1699, 1652, 1558, 1540, 1386, 1071, 757, 668 cm$^{-1}$; Anal. Calcd. For $C_{13}H_{15}BrN_2O$: C, 52.90; H, 5.12. Found: C, 52.74; H, 5.19.

**3-(4-Chlorophenyl)-1,4-diazaspiro[4.4]nonan-2-one**



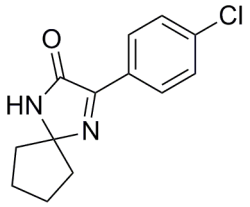
To a stirred solution of 4-chlorobenzaldehyde (14.1 g 100 mmol) in EtOH (50 mL) in a 300 mL round-bottomed flask, were added a solution of $NH_4OAc$ (23.1 g, 300 mmol) and NaCN (5.39 g, 110 mmol) in $H_2O$ (18 mL) and 30% aq. $NH_4OH$ (40 mL) at room temperature. The reaction mixture was stirred at room temperature for 4 h and then TBME (100 mL) was added. The mixture was transferred to a separatory funnel and the aqueous phase was removed. The organic phase was washed with $H_2O$ (80 mL, 2 times), brine (80 mL) and then, $H_2O$ (60 mL). The organic phase was dried over $MgSO_4$ and concentrated to ca. 30 mL. *n*-BuOH (180 mL) was added to the concentrated solution followed by cyclopentanone (22.1 mL, 250 mmol) and NaOEt (21 wt% in EtOH, 3.8 mL). The reaction mixture was stirred at 80 °C for 16 h. The dark-brown solution was concentrated to ca. 2/3 volume (white precipitate appeared at this point) and then cooled to 0 °C (ice bath). The precipitate was collected by filtration, washed 3-times with cold EtOH and dried in vacuo to give the title compound as an off-white solid (7.98 g, 32%), mp 174-176 °C. $^1$H NMR (400 MHz, dmso-*d6*) δ 8.55 (s, 1H), 7.49 (d, *J* = 8.4 Hz, 2H), 7.38 (d, *J* = 8.4 Hz, 2H), 4.54 (d, *J* = 8.8 Hz, 1H), 3.61 (d, *J* = 8.8 Hz, 1H), 1.89-1.56 (m, 8H); $^{13}$C NMR (100 MHz, dmso-*d6*) δ 173.4, 139.4, 131.5, 129.0, 127.8, 81.6, 61.0, 22.6, 22.4; IR (film) νmax 3854, 3745, 3214, 2336, 1699, 1652, 1558, 1540, 1457, 1386, 1092, 768, 668 cm$^{-1}$; Anal. Calcd. For $C_{13}H_{15}ClN_2O$: C, 62.28; H, 6.03. Found: C, 62.41; H, 6.03.

**3-(4-Bromophenyl)-1,4-diazaspiro[4.4]non-3-en-2-one (1a)**




An oven-dried 500 mL round-bottomed flask was equipped with a stir bar and charged with 3-(4-bromophenyl)-1,4-diazaspiro[4.4]nonan-2-one (10.0 g, 33.9 mmol) and DDQ (8.46 g, 37.3 mmol) and then EtOAc (200 mL) was added. The reaction mixture was stirred at 60 °C for 1 h. The reaction mixture was washed 2-times with sodium sulfite solution (20 wt% in $H_2O$, 140 mL and 70 ml) and then, $H_2O$ (70 mL). The organic phase was dried over $MgSO_4$, concentrated to ca. 80 mL and white precipitate that had formed was collected by filtration, washed 2 times with EtOAc and dried in vacuo to provide the title compound as an off-white solid (8.82 g, 89%), mp 184-185 °C. $^1$H NMR (400 MHz, dmso-*d6*) δ 10.08 (s, 1H), 8.26 (d, *J* = 8.8 Hz, 2H), 7.70 (d, *J* = 8.4 Hz, 2H), 2.00-1.75 (m, 8H); $^{13}$C NMR (100 MHz, dmso-*d6*) δ 163.9, 158.9, 131.6, 129.8, 125.2, 89.7, 37.0, 23.8 (aromatic C1 and C2 carbons are overlapped); IR (film) νmax 3853, 3745, 3153, 3056, 2955, 2361, 1707, 1652, 1558, 1476, 1381, 1264, 1002, 832, 744, 602, 525 cm$^{-1}$; Anal. Calcd. For $C_{13}H_{13}BrN_2O$: C, 53.62; H, 4.47. Found: C, 53.45; H, 4.49.

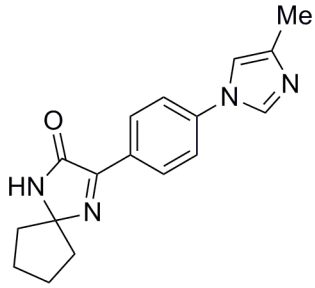
**3-(4-Chlorophenyl)-1,4-diazaspiro[4.4]non-3-en-2-one (1b)**




An oven-dried 500 mL round-bottomed flask was equipped with a stir bar and charged with 3-(4-chlorophenyl)-1,4-diazaspiro[4.4]nonan-2-one (6.7 g, 26.8 mmol) and DDQ (6.68 g, 29.4 mmol) and then, EtOAc (150 mL) was added. The reaction mixture was stirred at 60 °C for 1 h. The reaction mixture was washed 2-times with sodium sulfite solution (20 wt% in $H_2O$, 140 mL and 70 ml) and then, $H_2O$ (70 mL). The organic phase was dried over $MgSO_4$, concentrated to ca. 60 mL and hexanes (60 mL) was added. White precipitate that had formed was collected by filtration, washed 2 times with EtOAc-hexanes (1:1) and dried in vacuo to provide the title compound as an off-white solid (5.98 g, 90%), mp 174-176 °C.

$^{1}$H NMR (400 MHz, dmso-*d6*) δ 10.08 (s, 1H), 8.34 (d, *J* = 8.8 Hz, 2H), 7.56 (d, *J* = 8.8 Hz, 2H), 2.00-1.76 (m, 8H); $^{13}$C NMR (100 MHz, dmso-*d6*) δ 163.9, 158.8, 136.2, 129.6, 129.5, 128.7, 89.7, 37.0, 23.8; IR (film) νmax 3853, 3745, 3140, 3040, 2362, 1701, 1652, 1540, 1558, 1473, 1386, 1263, 1084, 838, 758, 668, 529 cm$^{-1}$; Anal. Calcd. For $C_{13}H_{13}ClN_2O$: C, 62.78; H, 5.27. Found: C, 62.63; H, 5.35.

**3-(4-(4-Methyl-1*H*-imidazol-1-yl)phenyl)-1,4-diazaspiro[4.4]non-3-en-2-one (2)**

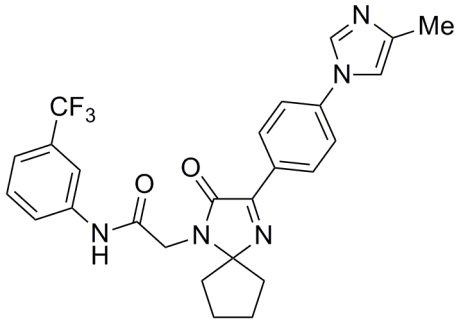



***From 1a***: An oven-dried vial was equipped with a magnetic stir bar and charged with $Pd_2(dba)_3$ (4.6 mg, 0.005 mmol) and **L1** (4.8 mg, 0.01 mmol). The vial was sealed with a screw-cap septum, and then evacuated and backfilled with argon (this process was repeated a total of 3 times). Then, toluene (0.6 mL) was added via syringe. This dark purple mixture was stirred at 130 °C for 3 min. A second oven-dried vial which was equipped with stir bar was charged with **1a** (298 mg, 1.0 mmol), 4-methylimidazole (164 mg, 2.0 mmol) and $K_3PO_4$ (424 mg, 2.0 mmol). The vial was sealed with a screw-cap septum, and then evacuated and backfilled with argon (this process was repeated a total of 3 times). 0.12 mL of the premixed catalyst solution was transferred to the second vial via syringe and then toluene (0.5 mL) and dioxane (0.5 mL) were added to the second vial. The reaction was heated at 130 °C for 6 h. The reaction mixture was cooled to room temperature, diluted with EtOAc, washed with brine, dried over $MgSO_4$, concentrated in vacuo and purified via flash chromatography (EtOAc-MeOH, 15:1) to provide the title compound as a white solid (273 mg, 93%), mp 194-195 °C.

***From 1b***: An oven-dried vial was equipped with a magnetic stir bar and charged with $Pd_2(dba)_3$ (4.6 mg, 0.005 mmol) and **L1** (4.8 mg, 0.01 mmol). The vial was sealed with a screw-cap septum, and then evacuated and backfilled with argon (this process was repeated a total of 3 times). Then, anhydrous toluene (0.6 mL) was added via syringe. This dark purple mixture was stirred at 130 °C for 3 min. A second oven-dried vial which was equipped with stir bar was charged with **1b** (249 mg, 1.0 mmol), 4-methylimidazole (164 mg, 2.0 mmol) and $K_3PO_4$ (424 mg, 2.0 mmol). The vial was sealed with a screw-cap septum, and then evacuated and backfilled with argon (this process was repeated a total of 3 times). 0.18 mL of the premixed catalyst solution was transferred to the second vial via syringe and then toluene (0.5 mL) and dioxane (0.5 mL) were added to the second vial. The reaction was heated at 130 °C for 6 h. The reaction mixture was cooled to room temperature, diluted with EtOAc, washed with brine, dried over $MgSO_4$, concentrated in vacuo and purified via flash chromatography (EtOAc-MeOH, 15:1) to provide the title compound as a white solid (270 mg, 92%), mp 195 °C.

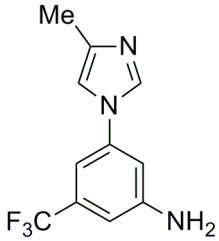
$^{1}$H NMR (400 MHz, dmso-*d6*) δ 10.09 (s, 1H), 8.42 (d, *J* = 8.8 Hz, 2H), 8.26 (d, *J* = 1.2 Hz, 1H), 7.74 (d, *J* = 8.8 Hz, 2H), 7.52 (s, 1H), 2.17 (s, 3H), 2.00-1.77 (m, 8H); $^{13}$C NMR (100 MHz, dmso-*d6*) δ 164.1, 158.8, 138.9, 138.8, 134.7, 129.4, 128.5, 119.3, 113.8, 89.6, 37.1, 23.9, 13.6; IR (film) νmax 3854, 3745, 3158, 3050, 2962, 2360, 1704, 1606, 1518, 1442, 1254, 1191, 1063, 963, 848, 752, 609, 540 cm$^{-1}$; Anal. Calcd. For $C_{17}H_{18}N_4O$: C, 69.37; H, 6.16. Found: C, 69.38; H, 6.11.

**2-(3-(4-(4-Methyl-1*H*-imidazol-1-yl)phenyl)-2-oxo-1,4-diazaspiro[4.4]non-3-en-1-yl)-*N*-(3-(trifluoro methyl)phenyl)acetamide (GSK2137305) (3)**



An oven-dried vial was equipped with a stir bar and charged with chloroacetyl chloride (175 μL, 2.3 mmol) and 3-aminobenzotrifluoride (287 μL, 2.3 mmol). NMP (2.0 mL) was added to the vial and stirred at room temperature for 15 min. The reaction mixture was transferred to a stirred solution of **2** (589 mg, 2.0 mmol) and 45 wt% aq. KOH (440 μL, 5 mmol) in NMP (1.5 mL) in a separate vial. The reaction mixture was stirred at room temperature for 6 h (white precipitate appeared at this point), then $H_2O$ (4.0 mL) was added. The reaction mixture was heated to 70 °C and then cooled to toom temperature. The white precipitate was collected by filtration, washed with $H_2O$ and isopropanol and dried in vacuo to provide the title compound as a white solid (812 mg, 82%), mp 244-246 °C. $^1$H NMR (400 MHz, dmso-*d6*) δ 10.60 (s, 1H), 8.45 (d, *J* = 8.8 Hz, 2H), 8.28 (d, *J* = 1.2 Hz, 1H), 8.11 (s, 1H), 7.82-7.74 (m, 3H), 7.61-7.53 (m, 2H), 7.42 (d, *J* = 8.0 Hz, 1H), 4.38 (s, 2H), 2.20-1.85 (m, 9H), 1.75-1.64 (m, 2H); $^{13}$C NMR (100 MHz, dmso-*d6*) δ 166.3, 162.6, 157.5, 139.4, 139.1, 138.9, 134.8, 130.1, 129.5 (q, *J* = 31 Hz), 129.4, 128.3, 124.1 (q, *J* = 271 Hz), 122.8, 119.9 (q, *J* = 4.0 Hz), 119.4, 115.2 (q, *J* = 3.0 Hz), 113.8, 93.3, 43.4, 34.4, 23.6, 13.6; IR (film) νmax 3854, 3746, 2362, 1884, 1772, 1678, 1653, 1616, 1559, 1516, 1417, 1312, 1251, 1160, 1118, 1065, 949, 888, 852, 793, 721, 691, 668, 623, 545, 466 cm$^{-1}$; Anal. Calcd. For $C_{26}H_{24}F_3N_5O_2$: C, 63.02; H, 4.88. Found: C, 62.58; H, 5.06.

**3-(4-Methyl-1*H*-imidazol-1-yl)-5-(trifluoromethyl)aniline (5)**

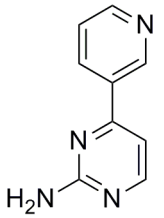


An oven-dried vial was equipped with a magnetic stir bar and charged with $Pd_2(dba)_3$ (2.3 mg, 0.0025 mmol) and **L1** (2.4 mg, 0.0025 mmol). The vial was sealed with a screw-cap septum, and then evacuated and backfilled with argon (this process was repeated a total of 3 times). Anhydrous toluene (0.5 mL) was added via syringe and the dark purple mixture was stirred at 120 °C for 3 min. The color of the mixture turns to red-brown after 3 min. A second oven-dried vial which was equipped with stir bar was charged with 3-amino-5-bromobenzotifluoride **4** (240 mg, 1.0 mmol), 4-methylimidazole (197 mg, 2.4 mmol) and $K_3PO_4$ (424 mg, 2.0 mmol). The vial was sealed with a screw-cap septum, and then evacuated and backfilled with argon (this process was repeated a total of 3 times). The premixed catalyst solution followed by anhydrous toluene (0.5 mL) and *t*BuOH (1.0 mL) were added via syringe to the second vial (total 2 mL of toluene-*t*BuOH 1:1 solution). The reaction was heated at 120 °C for 12 h. The reaction mixture was cooled to room temperature, diluted with EtOAc, washed with brine, dried over $MgSO_4$, concentrated in vacuo and purified via flash chromatography ($Et_2O$/EtOAc/MeOH, 125:125:1) to provide the title compound as a white solid (216 mg, 90%), mp 124-126 °C. $^1$H NMR (400 MHz, dmso-*d6*) δ 8.09 (d, *J* = 1.2 Hz, 1H), 7.36 (s, 1H), 6.98 (s, 1H), 6.96 (s, 1H), 6.84 (s, 1H), 5.91 (s, 2H), 2.15 (s, 3H); $^{13}$C NMR (100 MHz, dmso-*d6*) δ 150.9, 138.5, 134.8, 131.3 (q, *J* = 38 Hz), 124.1 (q, *J* = 272 Hz), 114.2,

107.9, 103.3 (q, *J* = 4 Hz), 13.5; IR (film) νmax 3854, 3745, 3414, 3215, 2362, 1620, 1509, 1412, 1328, 1293, 1254, 1199, 1158, 1115, 843, 807, 735, 691, 621 $cm^{-1}$; Anal. Calcd. For $C_{11}H_{10}F_3N_3$: C, 54.77; H, 4.18. Found: C, 54.69; H, 4.05.

*Note* : The use of an excess (> 2.0 eq.) amount of 4-methylimidazole, toluene/*t*BuOH mixed solvent and 0.5 M concentration of aryl bromoide (bromoaniline) was beneficial to suppress oligomerization of the bromoaniline.

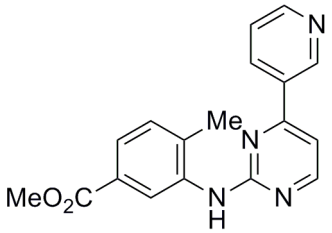
**3-(Dimethylamino)-1-(pyridin-3-yl)prop-2-en-1-one (6)**



In a 200 mL round-bottom flask equipped with a stirrer bar and reflux condenser were charged acetylpyridine (12.2 mL, 110 mmol), *N*,*N*-dimethylformamide dimethylacetal (14.9 mL, 110 mmol), and toluene (50 mL). The reaction mixture was heated to reflux for 18 h and then cooled to room temperature. The reaction mixture was concentrated in vacuo, then $Et_2O$ (50 ml) was added. The resulting yellow precipitate was collected by filtration, washed with cold $Et_2O$-hexanes (1:2) and dried in vacuo to produce 3-(Dimethylamino)-1-(pyridin-3-yl)prop-2-en-1-one (17.7 g, 91%) which was used next step without further purification.

In a 100 mL round-bottom flask equipped with a stirbar and reflux condenser were charged 3-(dimethylamino)-1-(pyridin-3-yl) prop-2-en-1-one (6.4 g, 36.3 mmol), guanidine hydrochloride (3.47 g, 36.3 mmol), NaOH (1.45 g, 36.3 mmol), and *n*BuOH (40 mL). The reaction was heated to reflux for 12 h and then cooled to room temperature. The precipitate that had formed was collected by filtration and was washed with cold water and dried in vacuo to produce the title compound as an off-white solid. (5.3 g, 85%), mp 187-189 °C. $^1$H NMR (400 MHz, dmso-*d6*) δ 9.23 (dd, *J* = 2.4, 0.8 Hz, 1H), 8.67 (dd, *J* = 4.8, 1.2 Hz, 1H), 8.40-8.36 (m, 2H), 7.53-7.48 (m, 1H), 7.20 (d, *J* = 5.2, 1H), 6.82 (s, 2H); $^{13}$C NMR (100 MHz, dmso-*d6*) δ 164.3, 162.1, 159.9, 151.6, 148.4, 134.6, 132.9, 124.2, 106.5; IR (film) νmax 3853, 3745, 3152, 2361, 1696, 1649, 1550, 1464, 668 $cm^{-1}$.

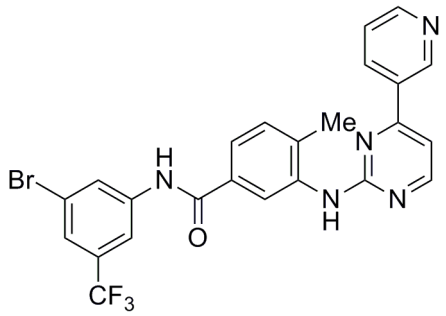
**Methyl 4-methyl-3-((4-(pyridin-3-yl)pyrimidin-2-yl)amino)benzoate (8)**



An oven-dried vial was equipped with a magnetic stir bar and charged with **L3** precatalyst (2.4 mg, 0.3 mol%), **6** (172 mg, 1.0 mmol) and $Cs_2CO_3$ (456 mg, 1.4 mmol). The vial was sealed with a screw-cap septum, and then evacuated and backfilled with argon (this process was repeated a total of 3 times). Then, **7** (176 μL, 1.1 mmol) and anhydrous 1,4-dioxane (2.0 mL) were added via syringe to the vial. The reaction mixture was heated at 120 °C for 1.5 h. The reaction solution was cooled to room temperature, diluted with EtOAc, washed with brine, dried over $MgSO_4$, concentrated in vacuo and purified via flash chromatography (EtOAc) to provide the title compound as an off-white solid (304 mg, 95%), mp 117 °C. $^1$H NMR (400 MHz, $CDCl_3$) δ 9.26 (d, *J* = 1.6 Hz, 1H), 8.95 (s, 1H), 8.69 (dd, *J* = 4.8, 1.6 Hz, 1H), 8.47 (d, *J* = 5.2 Hz, 1H), 8.40-8.35 (m, 1H), 7.67 (dd, *J* = 7.6, 1.6 Hz, 1H), 7.39 (dd, *J* = 8.0, 5.2 Hz, 1H), 7.29-7.15 (m, 3H), 3.91 (s, 3H), 2.37 (s, 3H); $^{13}$C NMR (100 MHz, $CDCl_3$) δ 167.2, 162.6, 160.6, 159.4, 151.7, 148.7, 137.7, 134.7, 133.4, 132.6, 130.6, 128.8, 124.6, 123.8, 122.4, 108.5, 52.3, 18.5; IR (film)

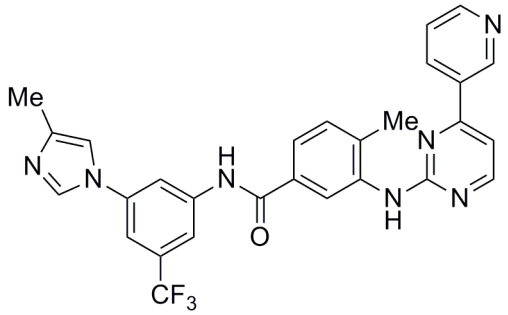
νmax 3360, 2361, 1711, 1557, 1445, 1414, 1297, 1230, 1110, 1011, 802, 754, 697, 602 $cm^{-1}$.
The title compound can also be prepared using $Pd_2(dba)_3$ (1 mol%) and XantPhos (2 mol%) instead of **L3** precatalyst to afford the title compound as an off-white solid (1.43 g, 90% yield).

***N*-(3-Bromo-5-(trifluoromethyl)phenyl)-4-methyl-3-((4-(pyridin-3-yl)pyrimidin-2-yl)amino)benzamide (10)**



An oven dried round-bottom flask was equipped with a magnetic stir bar and charged with **8** (800 mg, 2.5 mmol), **4** (630 mg, 2.63 mmol). The flask was equipped with a septum, and then evacuated and backfilled with argon (this process was repeated a total of 3 times), then, THF (12 mL) was added. The reaction solution was cooled to 0 °C and KO*t*Bu (1.55 g, 13.8 mmol) solution in THF (13 mL) was slowly added to the reaction mixture via syringe at 0 °C. After addition of the KO*t*Bu solution, the reaction was stirred at room temperature for 12 h. Brine (25 mL) was added to the reaction mixture and extracted with EtOAc (60 mL). The organic layer was dried over $MgSO_4$, concentrated in vacuo to a volume of approximately 10 mL. The white precipitate that had formed was collected by filtration, washed with cold $Et_2O$ to afford title compound as a white solid (1.12 g, 85%). mp 217 °C. $^1$H NMR (400 MHz, dmso-*d6*) δ 10.68 (s, 1H), 9.23 (s, 1H), 9.17 (s, 1H), 8.67 (d, *J* = 3.2 Hz, 1H), 8.53 (d, *J* = 5.2 Hz, 1H), 8.44-8.38 (m, 2H), 8.34 (s, 1H), 8.27 (s, 1H), 7.79 (d, *J* = 7.6 Hz, 1H), 7.62 (s, 1H), 7.54-7.39 (m, 3H), 2.35 (s, 3H); $^{13}$C NMR (100 MHz, dmso-*d6*) δ 165.8, 161.6, 161.1, 159.6, 151.4, 148.2. 141.6, 138.2, 136.9, 134.3, 132.1, 131.7, 131.1 (q, *J* = 32 Hz),130.4, 126.0, 124.6, 123.8, 123.7, 123.2 (q, *J* = 272 Hz), 122.3, 122.2, 115.4, 108.0, 18.3; IR (film) νmax 3854, 3747, 3628, 3650, 2361, 1699, 1675, 1652, 1558, 1540, 1456, 1436, 1179, 1125, 804, 668, 418 $cm^{-1}$.

**4-Methyl-*N*-(3-(4-methyl-1*H*-imidazol-1-yl)-5-(trifluoromethyl)phenyl)-3-((4-(pyridin-3-yl)pyrimidin-2-yl)amino)benzamide (9)**



*From **5** and **8***; An oven-dried vial was equipped with a magnetic stir bar and charged with **5** (120 mg, 0.5 mmol) and **8** (160 mg, 0.5 mmol). The vial was sealed with a screw-cap septum, and then evacuated and backfilled with argon (this process was repeated a total of 3 times), then, THF (3.0 mL) was added. The reaction solution was cooled to 0 °C and KO*t*Bu (309 mg, 2.75 mmol) solution in THF (3.0 mL) was slowly added to the reaction mixture at 0 °C. After addition of the KO*t*Bu solution, the reaction was stirred at room temperature for 12 h. Brine (15 mL) was added to the reaction mixture and extracted with EtOAc (25 mL). The organic layer was dried over $MgSO_4$, concentrated in vacuo. Isopropanol (5 ml) was added to the crude product and heated at 60 °C for 2 min, then cooled to room temperature. The white

precipitate that had formed was collected by filtration, washed with cold isopropanol (1st crop: 121 mg, 38%). The mother liquid was concentrated invacuo and purified by flash chromatography ($CH_2Cl_2$/MeOH, 15:2) to afford the title compound as a white solid (2nd crop: 165 mg, 52%); 90% combined isolated yield.

*From **10***; An oven-dried vial was equipped with a magnetic stir bar and charged with $Pd_2(dba)_3$ (2.7 mg, 1.0 mol%) and **L1** (3.2 mg, 2.2 mol%). The vial was sealed with a screw-cap septum, and then evacuated and backfilled with argon (this process was repeated a total of 3 times). Then, anhydrous 1,4-dioxane (0.6 mL) was added via syringe. This dark purple mixture was stirred at 120 °C for 3 min. A second oven-dried vial that was equipped with stir bar was charged with **10** (159 mg, 0.3 mmol), 4-methylimidazole (49 mg, 0.6 mmol) and $K_3PO_4$ (153 mg, 1.2 mmol). The vial was sealed with a screw-cap septum, and then evacuated and backfilled with argon (this process was repeated a total of 3 times). Anhydrous 1,4-dioxane (0.6 mL) and then, the premixed catalyst solution were added via syringe to the second vial (total 1.2 mL of 1,4-dioxane). The reaction was heated at 120 °C for 12 h. The reaction mixture was cooled to room temperature, diluted with EtOAc, washed with brine, dried over $MgSO_4$, concentrated in vacuo and purified via flash chromatography ($CH_2Cl_2$/MeOH, 15:2) to provide the title compound as a white solid (139 mg, 88%).
Mp 225-228 °C. $^1$H NMR (400 MHz, dmso-*d6*) δ 10.65 (s, 1H), 9.29 (s, 1H), 9.19 (s, 1H), 8.67 (d, *J* = 3.6 Hz, 1H), 8.54 (d, *J* = 5.2 Hz, 1H), 8.44 (d, *J* = 8.0 Hz, 1H), 8.37-8.30 (m, 2H), 8.23-8.15 (m, 2H), 8.78 (d, *J* = 7.6 Hz, 1H), 7.71 (s, 1H), 7.53-7.45 (m, 4H), 2.36 (s, 3H), 2.17 (s, 3H); $^{13}$C NMR (100 MHz, dmso-*d6*) δ 165.7, 161.7, 161.1, 159.6, 151.4, 148.2, 141.2, 138.9, 138.2, 137.9, 136.8, 135.0, 134.3, 132.1, 131.8, 130.8 (q, *J* = 32 Hz), 130.5, 124.3, 123.8, 123.7 (q, *J* = 271 Hz), 123.6, 114.9, 114.3, 114.2, 111.5, 108.0, 18.3, 13.6; IR (film) νmax 3853, 3819, 3649, 3628, 3363, 2361, 1734, 1699, 1652, 1617, 1577, 1540, 1494, 1474, 1437, 1399, 1317, 1194, 1165, 1107, 1075, 924, 852, 798, 746, 689, 668, 594, 546 cm$^{-1}$; Anal. Calcd. For $C_{28}H_{22}F_3N_7O$: C, 63.51; H, 4.19. Found: C, 62.98; H, 4.13.

**Computational Methods**

All calculations were carried out with Gaussian03 suite of computational programs.[5] Ground state geometry optimizations were evaluated using B3LYP[6] density functional method. For C, H, and N atoms, the 6-31G(d) basis set was used; while LANL2DZ effective core potentials of Hay and Wadt[7] with double-ζ basis sets were used for Pd and P atoms. Frequency calculations were performed on all optimized structures to verify that they have no negative frequencies. The Gibbs free energies were calculated at 298.15 K and 1 atm.

**Cartersian Coordinates for all Calculated Complexes:**

**Complex B**

| | | | |
|---|---|---|---|
| C | 1.531468 | -2.517732 | 1.140088 |
| C | 2.176371 | -2.870997 | -0.046035 |
| C | 1.573765 | -2.479806 | -1.244724 |
| C | 0.350111 | -1.808139 | -1.289802 |
| C | -0.333535 | -1.497404 | -0.066852 |
| C | 0.294206 | -1.866260 | 1.167493 |
| H | 1.996580 | -2.797899 | 2.081625 |
| H | 2.067574 | -2.717735 | -2.180651 |
| C | -1.848855 | -1.346638 | -0.088398 |
| C | -2.589408 | -0.133619 | 0.060653 |
| C | -2.536277 | -2.584057 | -0.224050 |
| C | -4.006027 | -0.218827 | 0.139484 |
| C | -3.943278 | -2.620034 | -0.281765 |
| C | -4.673954 | -1.446872 | -0.050593 |
| P | -1.616990 | 1.503483 | 0.138785 |
| C | -0.392155 | -1.706287 | 2.525358 |
| H | -1.330147 | -1.170845 | 2.362661 |
| C | 3.447606 | -3.710103 | 0.017768 |
| H | 4.005847 | -3.359620 | 0.896288 |
| C | 4.377255 | -3.572285 | -1.196411 |
| H | 3.904867 | -3.939980 | -2.116445 |
| H | 4.687251 | -2.532997 | -1.339117 |
| H | 5.278024 | -4.176481 | -1.036768 |
| C | 3.078287 | -5.191185 | 0.257968 |
| H | 3.983122 | -5.797975 | 0.381938 |
| H | 2.460143 | -5.315101 | 1.154466 |
| H | 2.516507 | -5.593897 | -0.594275 |
| C | -0.231940 | -1.485305 | -2.668499 |
| H | -1.130446 | -0.883402 | -2.516952 |
| C | 0.751852 | -0.660879 | -3.523808 |
| H | 0.259059 | -0.314691 | -4.440234 |
| H | 1.123053 | 0.211301 | -2.978665 |
| H | 1.621616 | -1.256305 | -3.823766 |
| C | -0.655040 | -2.742827 | -3.456199 |
| H | -0.995348 | -2.459952 | -4.459780 |
| H | 0.182057 | -3.440737 | -3.572699 |
| H | -1.474354 | -3.274433 | -2.966485 |
| C | 0.458162 | -0.890176 | 3.517958 |
| H | -0.089482 | -0.742606 | 4.456889 |
| H | 1.395769 | -1.401911 | 3.761435 |
| H | 0.716178 | 0.092618 | 3.110615 |
| C | -0.760367 | -3.069004 | 3.149511 |
| H | 0.132435 | -3.675915 | 3.338052 |
| H | -1.270592 | -2.918753 | 4.108642 |
| H | -1.426645 | -3.644795 | 2.500883 |
| Pd | 0.770462 | 0.865608 | 0.047596 |
| C | -2.147705 | 2.592949 | -1.390706 |
| C | -2.032401 | 2.441403 | 1.830160 |
| C | -3.643926 | 2.874318 | -1.622854 |
| H | -4.216987 | 1.960252 | -1.793248 |
| H | -4.109753 | 3.444150 | -0.815597 |
| H | -3.728340 | 3.482948 | -2.533067 |
| C | -1.418187 | 3.951759 | -1.305505 |
| H | -0.349783 | 3.847984 | -1.123241 |
| H | -1.541498 | 4.469247 | -2.266225 |
| H | -1.839455 | 4.605094 | -0.538104 |
| C | -1.642486 | 1.837785 | -2.634182 |
| H | -1.797077 | 2.466540 | -3.520790 |
| H | -0.579450 | 1.602976 | -2.569546 |
| H | -2.197581 | 0.907399 | -2.789155 |
| C | -3.057779 | 3.598271 | 1.801114 |
| H | -4.084772 | 3.289737 | 1.603374 |
| H | -3.061023 | 4.056470 | 2.798695 |
| H | -2.791422 | 4.385638 | 1.094820 |
| C | -2.479533 | 1.418282 | 2.891124 |
| H | -2.621905 | 1.943933 | 3.844665 |
| H | -3.419737 | 0.922090 | 2.642626 |
| H | -1.721966 | 0.651207 | 3.055932 |
| C | -0.700954 | 3.058286 | 2.303241 |
| H | 0.076241 | 2.298906 | 2.414587 |
| H | -0.325074 | 3.815332 | 1.611101 |
| H | -0.856254 | 3.537254 | 3.279409 |
| C | -1.810011 | -3.922039 | -0.266342 |
| H | -2.254097 | -4.614932 | 0.457782 |
| H | -1.891177 | -4.404584 | -1.247101 |
| H | -0.750710 | -3.844978 | -0.036804 |
| C | -4.894407 | 0.950583 | 0.484342 |
| H | -5.697124 | 1.089160 | -0.248058 |

| | | | |
|---|---|---|---|
| H | -5.383406 | 0.791864 | 1.454860 |
| H | -4.345206 | 1.876464 | 0.544581 |
| C | -6.188402 | -1.482502 | 0.036350 |
| H | -6.579693 | -2.499118 | 0.052248 |
| H | -6.547749 | -0.989585 | 0.946172 |
| H | -6.660026 | -0.961478 | -0.808215 |
| C | -4.662218 | -3.929056 | -0.551923 |
| H | -5.539198 | -3.779304 | -1.187411 |
| H | -4.021985 | -4.650694 | -1.059938 |
| H | -5.010988 | -4.404634 | 0.375660 |
| C | 1.669148 | 2.661400 | -0.293371 |
| C | 2.195152 | 3.445683 | 0.739348 |
| C | 1.905687 | 3.054550 | -1.617309 |
| C | 2.883534 | 4.630467 | 0.455576 |
| H | 2.094430 | 3.134735 | 1.773470 |
| C | 2.597479 | 4.237947 | -1.900947 |
| H | 1.552812 | 2.447589 | -2.446102 |
| C | 3.081519 | 5.036586 | -0.864738 |
| H | 3.279382 | 5.226144 | 1.275286 |
| H | 2.762536 | 4.525073 | -2.937106 |
| H | 3.622899 | 5.953316 | -1.083265 |
| N | 2.749512 | 0.304451 | 0.158661 |
| C | 4.800857 | -0.084129 | 0.937688 |
| N | 4.873478 | -0.151420 | -0.437398 |
| C | 3.503064 | 0.207240 | 1.311065 |
| C | 3.635790 | 0.102270 | -0.849395 |
| H | 3.343942 | 0.141571 | -1.890365 |
| H | 3.065737 | 0.334619 | 2.291482 |
| C | 6.002311 | -0.310319 | 1.805554 |
| H | 6.798764 | 0.410888 | 1.580308 |
| H | 5.747279 | -0.207617 | 2.866776 |
| H | 6.430338 | -1.310779 | 1.657196 |

**Complex B-TS**

| | | | |
|---|---|---|---|
| C | 1.361207 | 2.642169 | -1.047160 |
| C | 1.957009 | 3.025387 | 0.156085 |
| C | 1.328578 | 2.615463 | 1.334226 |
| C | 0.126252 | 1.898179 | 1.341222 |
| C | -0.503858 | 1.566327 | 0.103425 |
| C | 0.145900 | 1.950158 | -1.108514 |
| H | 1.841417 | 2.937886 | -1.976862 |
| H | 1.774946 | 2.881876 | 2.286723 |
| C | -1.995012 | 1.254653 | 0.081619 |
| C | -2.602446 | -0.036136 | -0.053431 |
| C | -2.812292 | 2.415555 | 0.176303 |
| C | -4.019483 | -0.102536 | -0.116896 |
| C | -4.217097 | 2.306721 | 0.203102 |
| C | -4.816257 | 1.052348 | 0.028785 |
| P | -1.463977 | -1.563202 | -0.174366 |
| C | -0.485992 | 1.751649 | -2.488177 |
| H | -1.402609 | 1.172291 | -2.354104 |
| C | 3.181510 | 3.934875 | 0.128344 |
| H | 3.793976 | 3.613367 | -0.724813 |
| C | 4.072121 | 3.862910 | 1.376181 |
| H | 3.543398 | 4.197734 | 2.277536 |
| H | 4.447122 | 2.847709 | 1.529584 |
| H | 4.937227 | 4.523996 | 1.248088 |
| C | 2.738371 | 5.390779 | -0.138003 |
| H | 3.610435 | 6.049136 | -0.232951 |
| H | 2.148661 | 5.471591 | -1.058043 |
| H | 2.120965 | 5.765177 | 0.688215 |
| C | -0.484251 | 1.556449 | 2.703096 |
| H | -1.319294 | 0.873111 | 2.532433 |
| C | 0.527453 | 0.844251 | 3.624533 |
| H | 0.019844 | 0.448758 | 4.512370 |
| H | 1.021160 | 0.013812 | 3.111913 |
| H | 1.307123 | 1.530889 | 3.974050 |
| C | -1.050383 | 2.789561 | 3.437519 |
| H | -1.417760 | 2.501154 | 4.430147 |
| H | -0.280725 | 3.557963 | 3.577111 |
| H | -1.883610 | 3.240004 | 2.893352 |
| C | 0.434313 | 0.969852 | -3.444776 |
| H | -0.074786 | 0.784456 | -4.398764 |
| H | 1.353346 | 1.524898 | -3.663749 |
| H | 0.725704 | 0.007773 | -3.012305 |
| C | -0.894068 | 3.091953 | -3.135189 |
| H | -0.023017 | 3.737803 | -3.295203 |
| H | -1.361456 | 2.914070 | -4.111450 |
| H | -1.608483 | 3.640880 | -2.515205 |
| Pd | 0.848702 | -0.833361 | -0.092094 |
| C | -1.803655 | -2.749498 | 1.339424 |
| C | -1.823456 | -2.525725 | -1.863987 |
| C | -3.228007 | -3.269186 | 1.608698 |
| H | -3.926541 | -2.461974 | 1.841676 |
| H | -3.636875 | -3.867007 | 0.790916 |
| H | -3.184537 | -3.922550 | 2.490719 |
| C | -0.862950 | -3.964583 | 1.184821 |
| H | 0.171307 | -3.667884 | 1.011878 |
| H | -0.890328 | -4.547233 | 2.115287 |
| H | -1.168830 | -4.636182 | 0.378406 |
| C | -1.368929 | -1.960537 | 2.587070 |
| H | -1.426532 | -2.615715 | 3.466605 |
| H | -0.341406 | -1.604294 | 2.495276 |
| H | -2.024508 | -1.103089 | 2.769056 |
| C | -2.740718 | -3.770583 | -1.838087 |
| H | -3.794168 | -3.545486 | -1.666871 |
| H | -2.682987 | -4.245205 | -2.826574 |
| H | -2.424878 | -4.519529 | -1.110074 |
| C | -2.377677 | -1.540134 | -2.909200 |
| H | -2.480900 | -2.062769 | -3.869743 |
| H | -3.359167 | -1.143912 | -2.638701 |

| | | | |
|---|---|---|---|
| H | -1.703525 | -0.697327 | -3.069256 |
| C | -0.441877 | -3.015716 | -2.351678 |
| H | 0.262435 | -2.185759 | -2.451784 |
| H | 0.003306 | -3.742441 | -1.666804 |
| H | -0.556382 | -3.497999 | -3.332480 |
| C | -2.225297 | 3.821552 | 0.209376 |
| H | -2.674781 | 4.439501 | -0.576914 |
| H | -2.430399 | 4.329173 | 1.158913 |
| H | -1.148511 | 3.841113 | 0.064800 |
| C | -4.776962 | -1.378106 | -0.387166 |
| H | -5.524986 | -1.583294 | 0.387147 |
| H | -5.324091 | -1.314071 | -1.337261 |
| H | -4.122975 | -2.233530 | -0.443153 |
| C | -6.325446 | 0.915238 | -0.040572 |
| H | -6.835167 | 1.877763 | -0.015897 |
| H | -6.639810 | 0.410926 | -0.962076 |
| H | -6.718589 | 0.315331 | 0.791120 |
| C | -5.085197 | 3.538617 | 0.388270 |
| H | -5.883556 | 3.355924 | 1.114406 |
| H | -4.520049 | 4.397459 | 0.747773 |
| H | -5.568248 | 3.839888 | -0.551479 |
| C | 2.319350 | -2.313045 | 0.331331 |
| C | 2.592903 | -3.336050 | -0.596857 |
| C | 2.491195 | -2.586140 | 1.704382 |
| C | 2.945558 | -4.610950 | -0.154301 |
| H | 2.521934 | -3.141163 | -1.661291 |
| C | 2.846979 | -3.865415 | 2.130804 |
| H | 2.338910 | -1.803129 | 2.440174 |
| C | 3.070854 | -4.891931 | 1.209495 |
| H | 3.130417 | -5.390699 | -0.890012 |
| H | 2.952475 | -4.055310 | 3.196645 |
| H | 3.356276 | -5.884429 | 1.545552 |
| N | 2.961481 | -0.648307 | -0.175872 |
| C | 4.677528 | 0.326573 | -1.206615 |
| N | 4.755096 | 0.672318 | 0.133903 |
| C | 3.591213 | -0.487030 | -1.408487 |
| C | 3.729194 | 0.076052 | 0.703204 |
| H | 3.503429 | 0.115772 | 1.758882 |
| H | 3.219090 | -0.965007 | -2.301774 |
| C | 5.686021 | 0.815118 | -2.200025 |
| H | 6.700576 | 0.512562 | -1.912308 |
| H | 5.482501 | 0.414571 | -3.198974 |
| H | 5.686652 | 1.910720 | -2.267423 |

**Complex B’**

| | | | |
|---|---|---|---|
| C | -1.219337 | 2.682229 | 1.247457 |
| C | -1.885495 | 3.108258 | 0.096508 |
| C | -1.363603 | 2.699724 | -1.133760 |
| C | -0.208545 | 1.919875 | -1.244095 |
| C | 0.486274 | 1.518798 | -0.057380 |
| C | -0.040996 | 1.929160 | 1.209694 |
| H | -1.610647 | 2.989031 | 2.214485 |
| H | -1.869513 | 3.003805 | -2.043259 |
| C | 1.963833 | 1.167012 | -0.147003 |
| C | 2.535022 | -0.137659 | -0.025870 |
| C | 2.807400 | 2.300776 | -0.311189 |
| C | 3.952873 | -0.248505 | -0.024126 |
| C | 4.201736 | 2.144330 | -0.433510 |
| C | 4.771936 | 0.876540 | -0.256086 |
| P | 1.357600 | -1.632310 | 0.127283 |
| C | 0.721158 | 1.730186 | 2.523529 |
| H | 1.601680 | 1.121002 | 2.307548 |
| C | -3.097360 | 4.026116 | 0.232817 |
| H | -3.727441 | 3.588094 | 1.019111 |
| C | -3.963182 | 4.142263 | -1.028919 |
| H | -3.413534 | 4.609737 | -1.856488 |
| H | -4.335193 | 3.163763 | -1.346824 |
| H | -4.829692 | 4.780168 | -0.819276 |
| C | -2.648612 | 5.424705 | 0.711488 |
| H | -3.519742 | 6.067787 | 0.883663 |
| H | -2.074909 | 5.374653 | 1.643668 |
| H | -2.016818 | 5.908457 | -0.044168 |
| C | 0.300799 | 1.595678 | -2.653714 |
| H | 1.157677 | 0.924461 | -2.552885 |
| C | -0.762908 | 0.879408 | -3.509197 |
| H | -0.326168 | 0.559450 | -4.462807 |
| H | -1.162473 | -0.002020 | -3.003004 |
| H | -1.605862 | 1.540432 | -3.738997 |
| C | 0.786634 | 2.844951 | -3.419637 |
| H | 1.076139 | 2.567711 | -4.440471 |
| H | -0.005092 | 3.599530 | -3.491891 |
| H | 1.653234 | 3.309536 | -2.945056 |
| C | -0.105917 | 1.003812 | 3.598774 |
| H | 0.512116 | 0.806055 | 4.483221 |
| H | -0.958336 | 1.609038 | 3.926771 |
| H | -0.498452 | 0.051680 | 3.231454 |
| C | 1.234845 | 3.069218 | 3.095448 |
| H | 0.403992 | 3.739841 | 3.343322 |
| H | 1.804830 | 2.890515 | 4.015352 |
| H | 1.887854 | 3.591294 | 2.391745 |
| Pd | -0.970472 | -0.755892 | 0.100990 |
| C | 1.663253 | -2.781830 | -1.427371 |
| C | 1.753955 | -2.593659 | 1.812231 |
| C | 3.104930 | -3.188871 | -1.779588 |
| H | 3.743810 | -2.326105 | -1.981040 |
| H | 3.574723 | -3.815755 | -1.018509 |
| H | 3.063999 | -3.784171 | -2.701496 |
| C | 0.833973 | -4.075745 | -1.275653 |
| H | -0.183289 | -3.894580 | -0.933204 |
| H | 0.766787 | -4.558812 | -2.259349 |

| | | | |
|---|---|---|---|
| H | 1.310794 | -4.792637 | -0.603668 |
| C | 1.125023 | -1.978428 | -2.625565 |
| H | 1.177498 | -2.602260 | -3.527434 |
| H | 0.086857 | -1.680621 | -2.481246 |
| H | 1.724114 | -1.081298 | -2.808808 |
| C | 2.575442 | -3.901394 | 1.735922 |
| H | 3.595154 | -3.785470 | 1.367618 |
| H | 2.657896 | -4.295411 | 2.757302 |
| H | 2.080176 | -4.670206 | 1.141620 |
| C | 2.434933 | -1.635915 | 2.807881 |
| H | 2.543671 | -2.153761 | 3.769937 |
| H | 3.427880 | -1.315476 | 2.489986 |
| H | 1.832645 | -0.745130 | 2.986212 |
| C | 0.387243 | -2.993501 | 2.402691 |
| H | -0.267448 | -2.129574 | 2.543305 |
| H | -0.135378 | -3.708789 | 1.764550 |
| H | 0.545678 | -3.466310 | 3.381527 |
| C | 2.267253 | 3.725217 | -0.311709 |
| H | 2.795035 | 4.329263 | 0.435556 |
| H | 2.421103 | 4.223273 | -1.275082 |
| H | 1.204946 | 3.784529 | -0.089787 |
| C | 4.696823 | -1.525964 | 0.278967 |
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| H | 5.281516 | -1.420945 | 1.202438 |
| H | 4.033865 | -2.366349 | 0.407115 |
| C | 6.278500 | 0.697244 | -0.269218 |
| H | 6.810769 | 1.642612 | -0.365832 |
| H | 6.633737 | 0.223401 | 0.653512 |
| H | 6.604481 | 0.051725 | -1.095649 |
| C | 5.092858 | 3.340964 | -0.713905 |
| H | 5.830756 | 3.111129 | -1.488879 |
| H | 4.530720 | 4.208014 | -1.058088 |
| H | 5.650908 | 3.650992 | 0.180197 |
| C | -2.028056 | -2.456017 | -0.261094 |
| C | -2.511988 | -3.280915 | 0.760071 |
| C | -2.388719 | -2.750524 | -1.583235 |
| C | -3.282013 | -4.411788 | 0.460928 |
| H | -2.307833 | -3.052575 | 1.799503 |
| C | -3.157888 | -3.879973 | -1.881238 |
| H | -2.081906 | -2.103469 | -2.398919 |
| C | -3.600549 | -4.722568 | -0.860618 |
| H | -3.641774 | -5.040498 | 1.272542 |
| H | -3.419779 | -4.088598 | -2.916081 |
| H | -4.202891 | -5.597260 | -1.091247 |
| N | -2.896772 | 0.005744 | 0.112241 |
| C | -4.986114 | 0.585194 | 0.559367 |
| N | -4.834446 | 0.725096 | -0.800608 |
| C | -3.818828 | 0.130630 | 1.140471 |
| C | -3.580715 | 0.352514 | -1.014878 |
| H | -3.124140 | 0.326418 | -1.993353 |
| C | -3.548017 | -0.167865 | 2.579490 |
| H | -4.476780 | -0.073560 | 3.153374 |
| H | -3.172547 | -1.187422 | 2.733387 |
| H | -2.816959 | 0.517423 | 3.024051 |
| H | -5.923985 | 0.817052 | 1.051775 |

**Complex B'-TS**

| | | | |
|---|---|---|---|
| C | -0.739784 | 2.942611 | 1.193310 |
| C | -1.303921 | 3.461811 | 0.025637 |
| C | -0.839517 | 2.946804 | -1.185999 |
| C | 0.183576 | 1.993511 | -1.261527 |
| C | 0.790737 | 1.517606 | -0.062727 |
| C | 0.301252 | 2.007427 | 1.184237 |
| H | -1.093626 | 3.321163 | 2.149776 |
| H | -1.271781 | 3.313476 | -2.111018 |
| C | 2.175297 | 0.886863 | -0.115862 |
| C | 2.496541 | -0.505081 | 0.003449 |
| C | 3.218595 | 1.843106 | -0.260951 |
| C | 3.869649 | -0.870777 | 0.022527 |
| C | 4.563063 | 1.434439 | -0.361258 |
| C | 4.888567 | 0.084769 | -0.176828 |
| P | 1.068843 | -1.769672 | 0.132342 |
| C | 0.954058 | 1.659613 | 2.523728 |
| H | 1.703623 | 0.886759 | 2.338352 |
| C | -2.313251 | 4.601783 | 0.126817 |
| H | -2.977054 | 4.360251 | 0.968363 |
| C | -3.200292 | 4.792428 | -1.110968 |
| H | -2.612543 | 5.073886 | -1.994248 |
| H | -3.769814 | 3.883933 | -1.326093 |
| H | -3.914233 | 5.604598 | -0.929658 |
| C | -1.579347 | 5.916220 | 0.474780 |
| H | -2.297417 | 6.731751 | 0.623425 |
| H | -0.981997 | 5.818372 | 1.388190 |
| H | -0.901469 | 6.207156 | -0.337513 |
| C | 0.638375 | 1.553569 | -2.656162 |
| H | 1.338728 | 0.724254 | -2.532162 |
| C | -0.541190 | 1.049982 | -3.512054 |
| H | -0.172148 | 0.591615 | -4.437323 |
| H | -1.133786 | 0.305962 | -2.972994 |
| H | -1.211359 | 1.869327 | -3.797407 |
| C | 1.378980 | 2.664566 | -3.429216 |
| H | 1.623905 | 2.320411 | -4.441625 |
| H | 0.759845 | 3.564509 | -3.523586 |
| H | 2.313711 | 2.946883 | -2.939761 |
| C | -0.058762 | 1.105863 | 3.543190 |
| H | 0.452569 | 0.801495 | 4.464808 |
| H | -0.807883 | 1.857271 | 3.816296 |
| H | -0.591723 | 0.239242 | 3.140460 |
| C | 1.698789 | 2.866785 | 3.132538 |
| H | 1.011485 | 3.695845 | 3.337729 |

| | | | |
|---|---|---|---|
| H | 2.171249 | 2.582096 | 4.080768 |
| H | 2.481606 | 3.238165 | 2.465315 |
| Pd | -1.091503 | -0.636640 | 0.115674 |
| C | 1.162829 | -2.963219 | -1.409667 |
| C | 1.252133 | -2.813990 | 1.805397 |
| C | 2.450193 | -3.759307 | -1.695328 |
| H | 3.298325 | -3.107404 | -1.916140 |
| H | 2.728860 | -4.445282 | -0.892054 |
| H | 2.272698 | -4.370575 | -2.590549 |
| C | -0.007088 | -3.963013 | -1.289603 |
| H | -0.959924 | -3.466199 | -1.110442 |
| H | -0.092006 | -4.515607 | -2.234683 |
| H | 0.150556 | -4.702886 | -0.500661 |
| C | 0.909340 | -2.069995 | -2.637803 |
| H | 0.824900 | -2.702470 | -3.531732 |
| H | -0.017230 | -1.502379 | -2.536819 |
| H | 1.734754 | -1.370216 | -2.804052 |
| C | 1.885292 | -4.223675 | 1.738932 |
| H | 2.957541 | -4.224506 | 1.540268 |
| H | 1.754183 | -4.694950 | 2.722261 |
| H | 1.398828 | -4.874570 | 1.009962 |
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| H | 2.037945 | -2.532127 | 3.798242 |
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| H | 1.527277 | -1.024466 | 3.039035 |
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| H | -0.784170 | -3.636719 | 1.650158 |
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| C | 2.954017 | 3.343631 | -0.272634 |
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| H | 3.170263 | 3.792752 | -1.248841 |
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| H | -2.699238 | -0.624822 | 2.931222 |
| H | -5.319303 | 1.916663 | 1.633637 |

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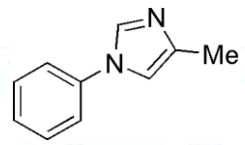


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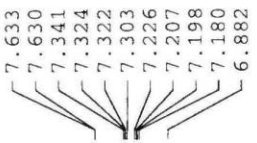



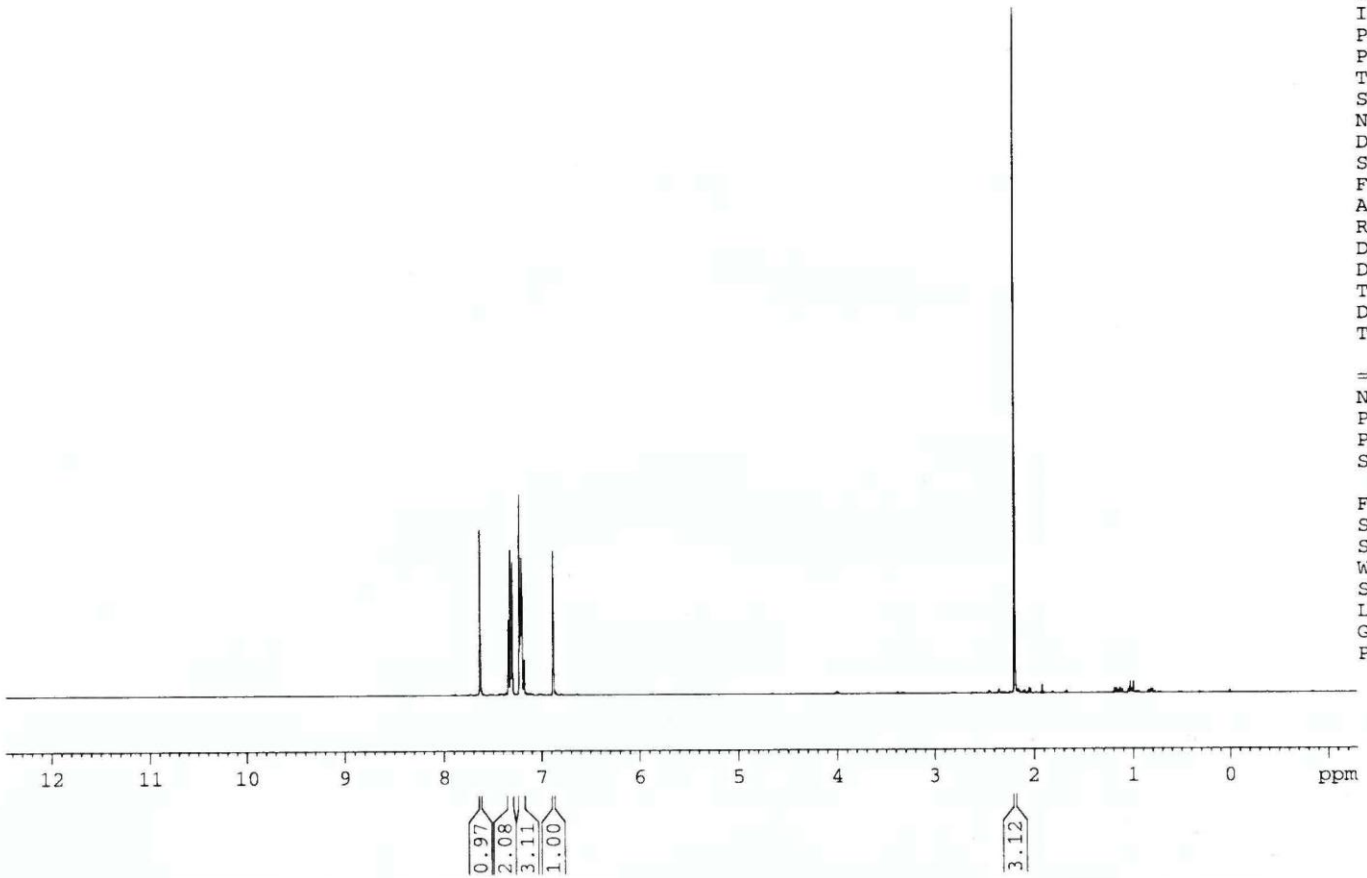


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SOLVENT                   CDCl3
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DS                            2
SWH                    8278.146 Hz
FIDRES                 0.126314 Hz
AQ                     3.9584243 sec
RG                           57
DW                        60.400 usec
DE                         6.00 usec
TE                        295.2 K
D1                   1.00000000 sec
TD0                           1

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PL1                        0.00 dB
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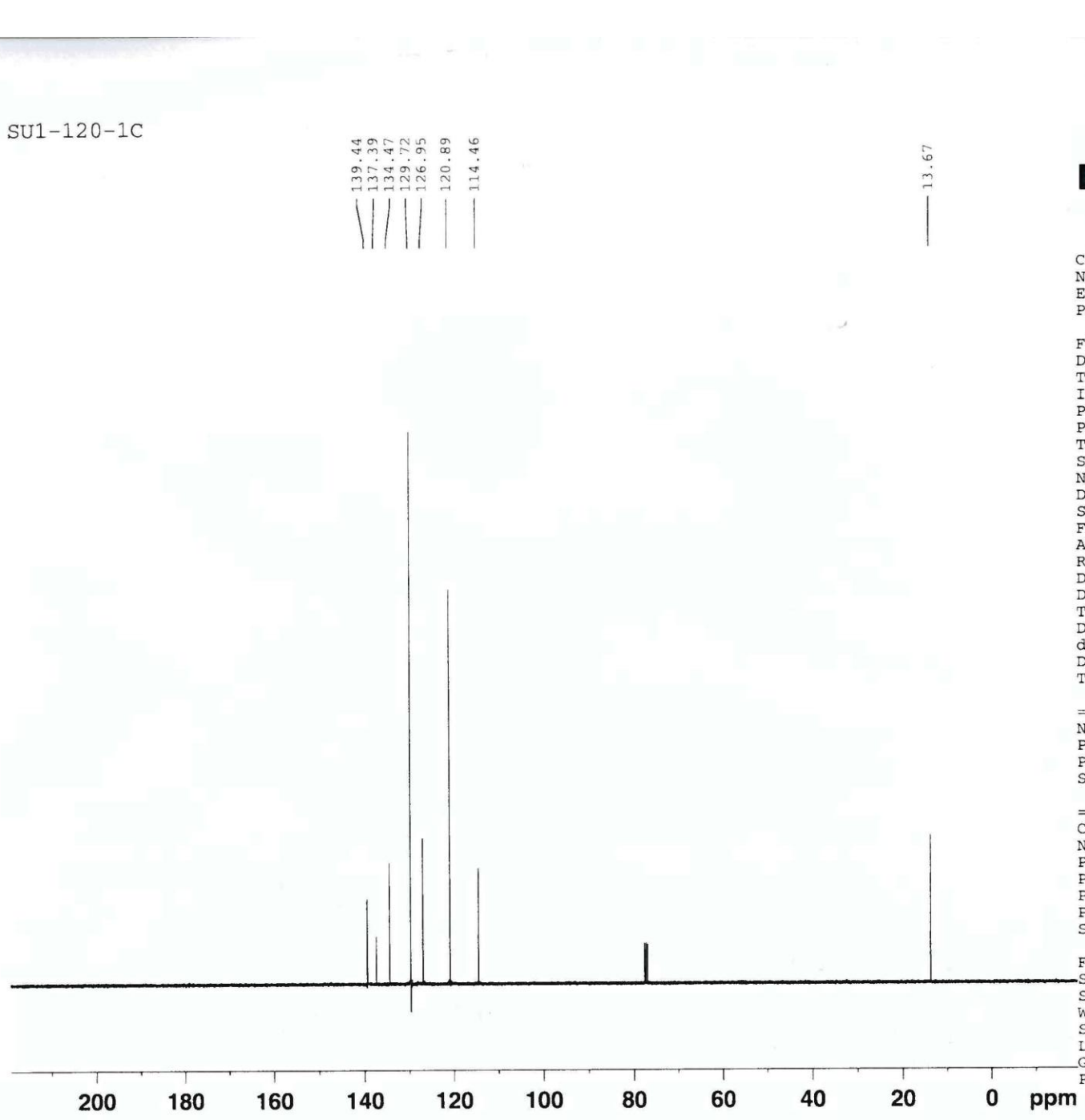


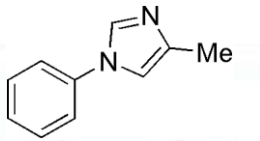


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TD 65536
SOLVENT CDCl3
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FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 4597.6
DW 20.850 usec
DE 6.00 usec
TE 296.2 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
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PL13 19.00 dB
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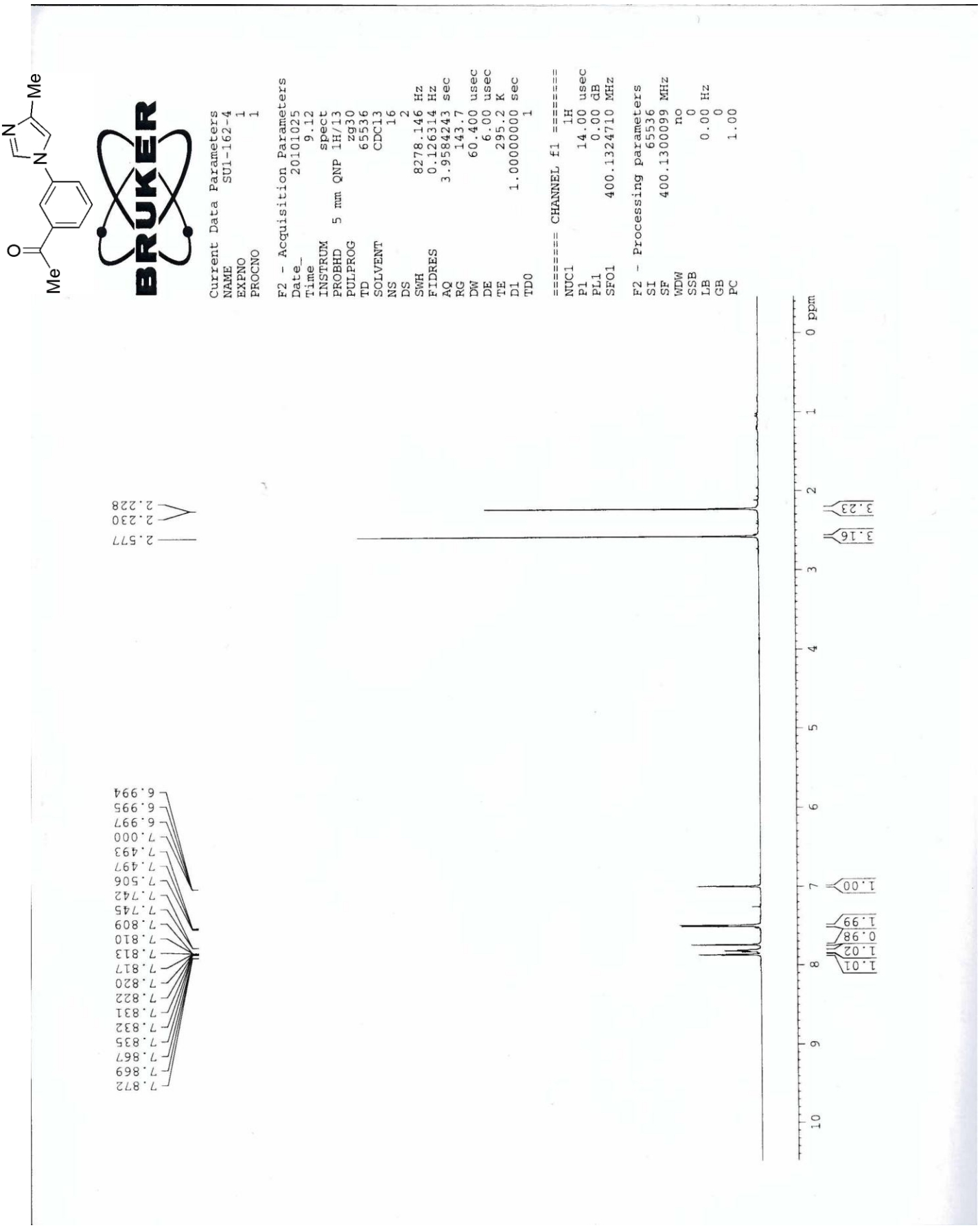
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PULPROG zg30
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SOLVENT CDCl3
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DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 143.7
DW 60.400 usec
DE 6.00 usec
TE 295.2 K
D1 1.00000000 sec
TD0 1
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7.742
7.506
7.497
7.493
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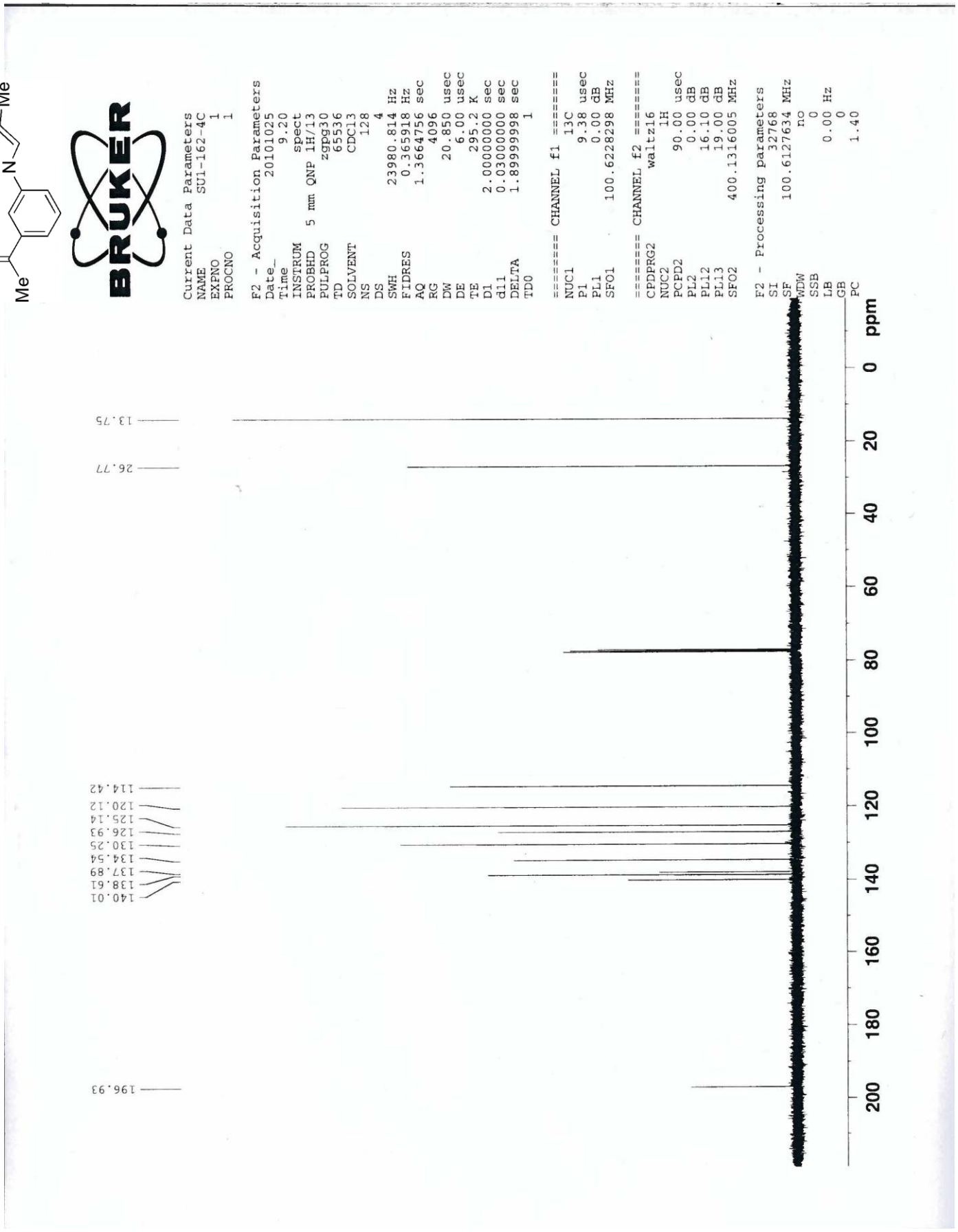
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FIDRES 0.365918 Hz
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RG 4096
DW 20.850 usec
DE 6.00 usec
TE 295.2 K
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d11 0.03000000 sec
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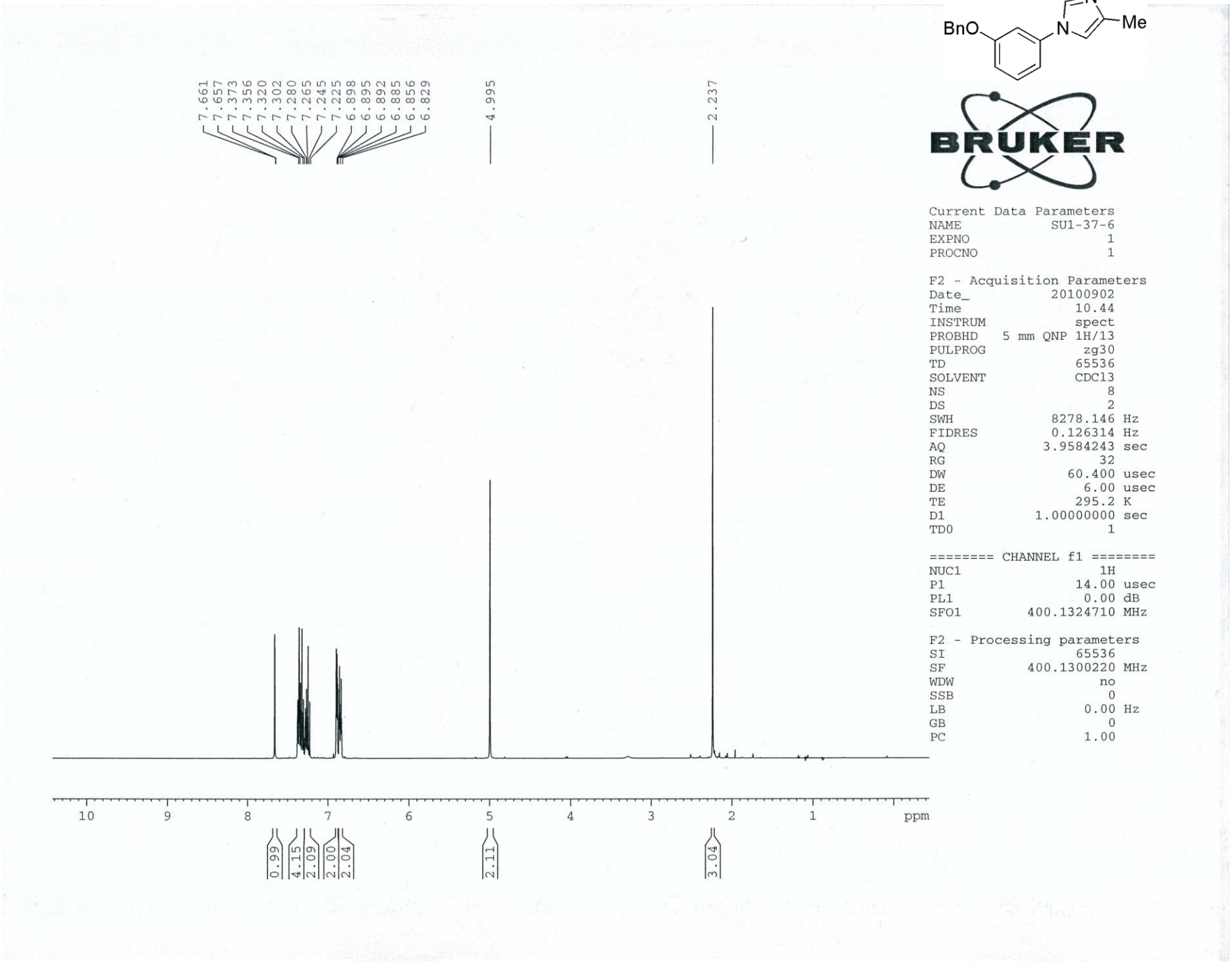
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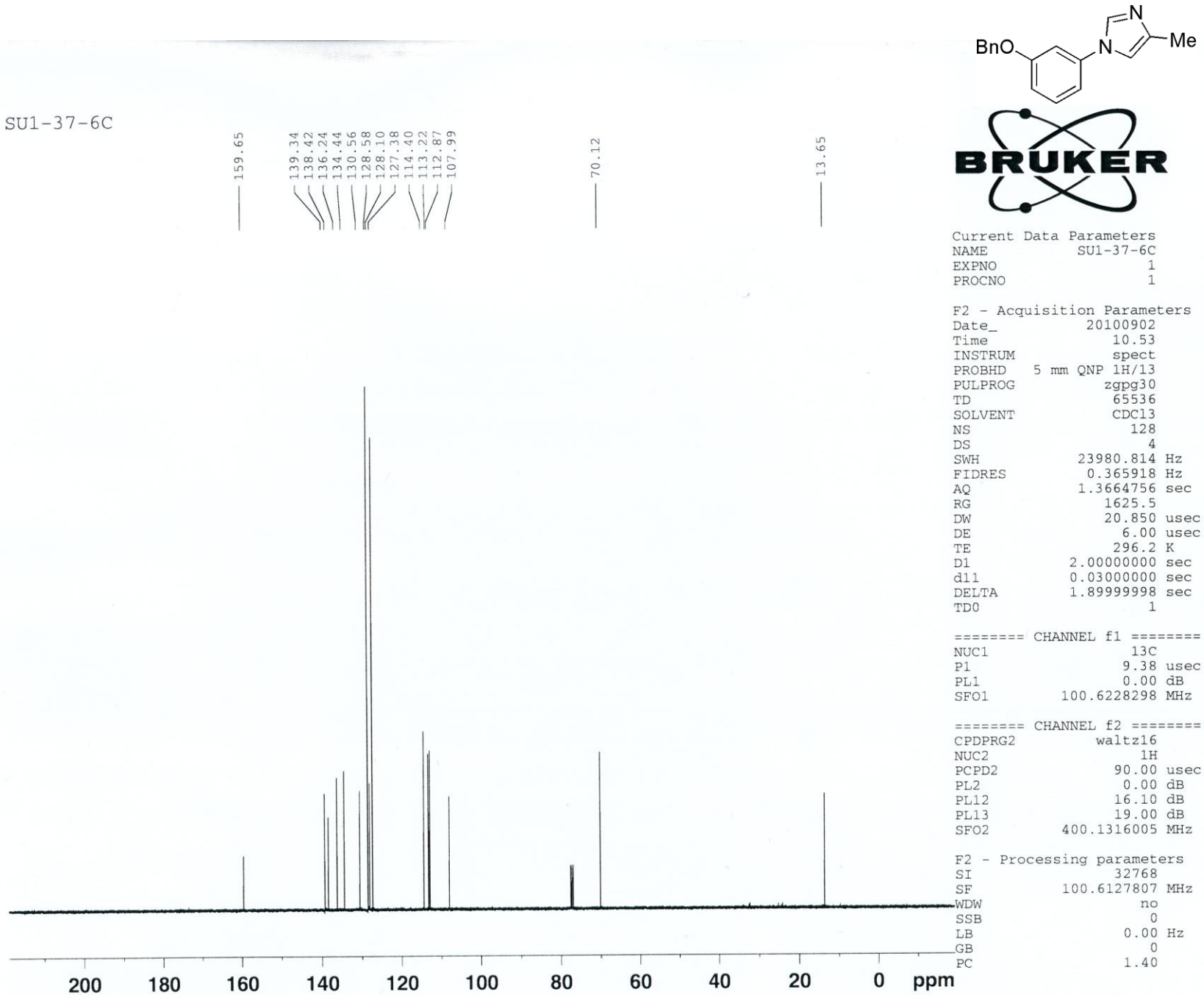
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d11 0.03000000 sec
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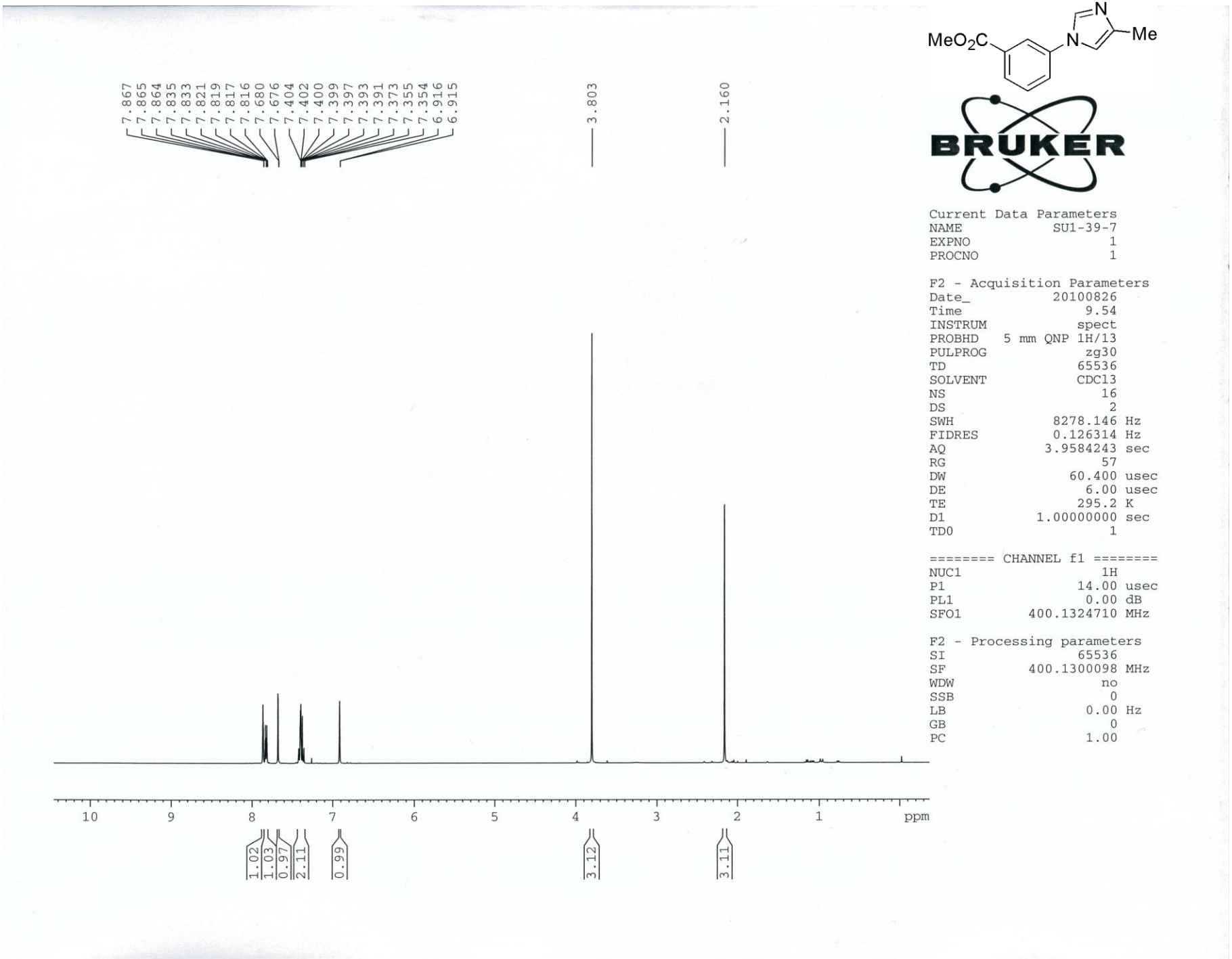
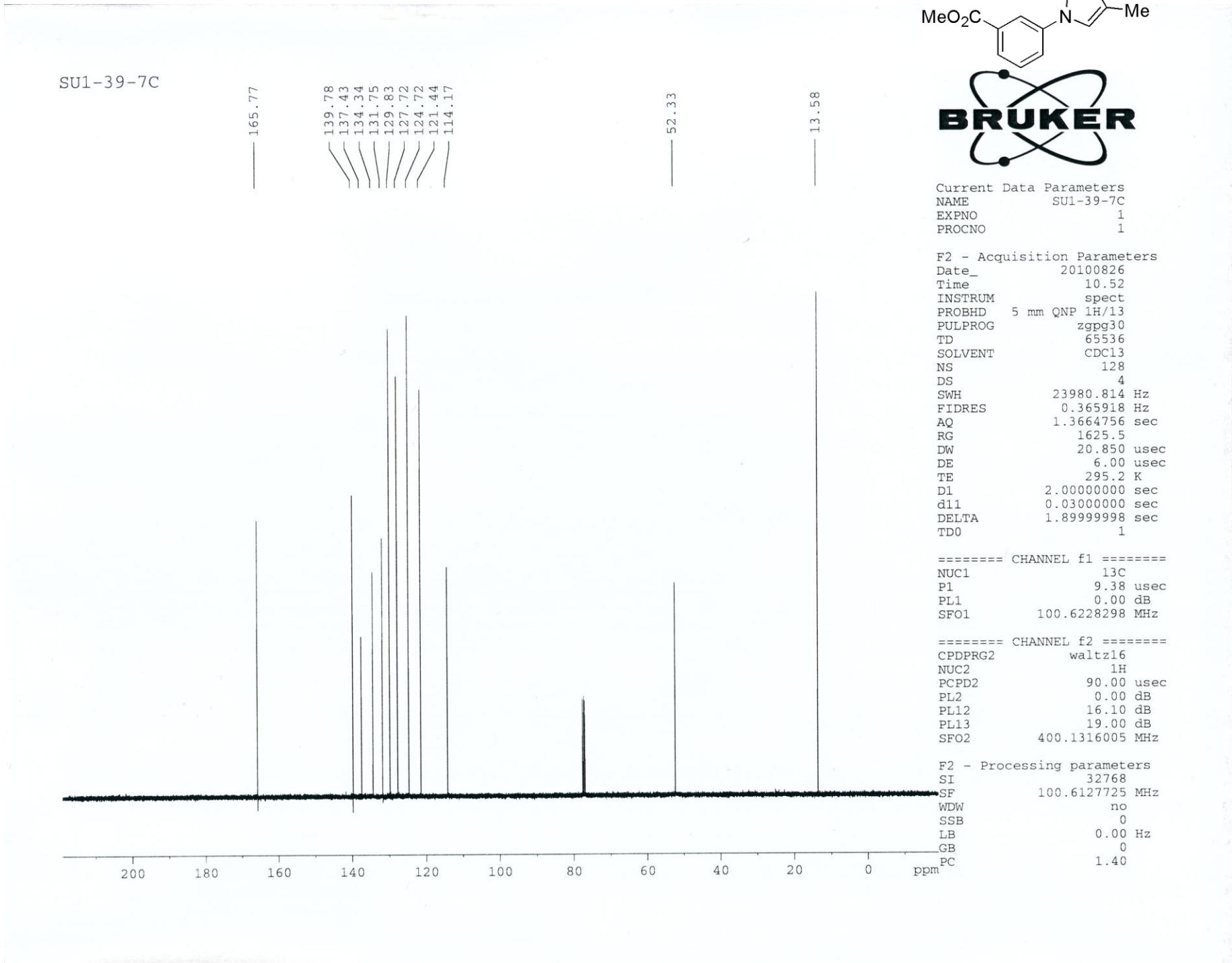
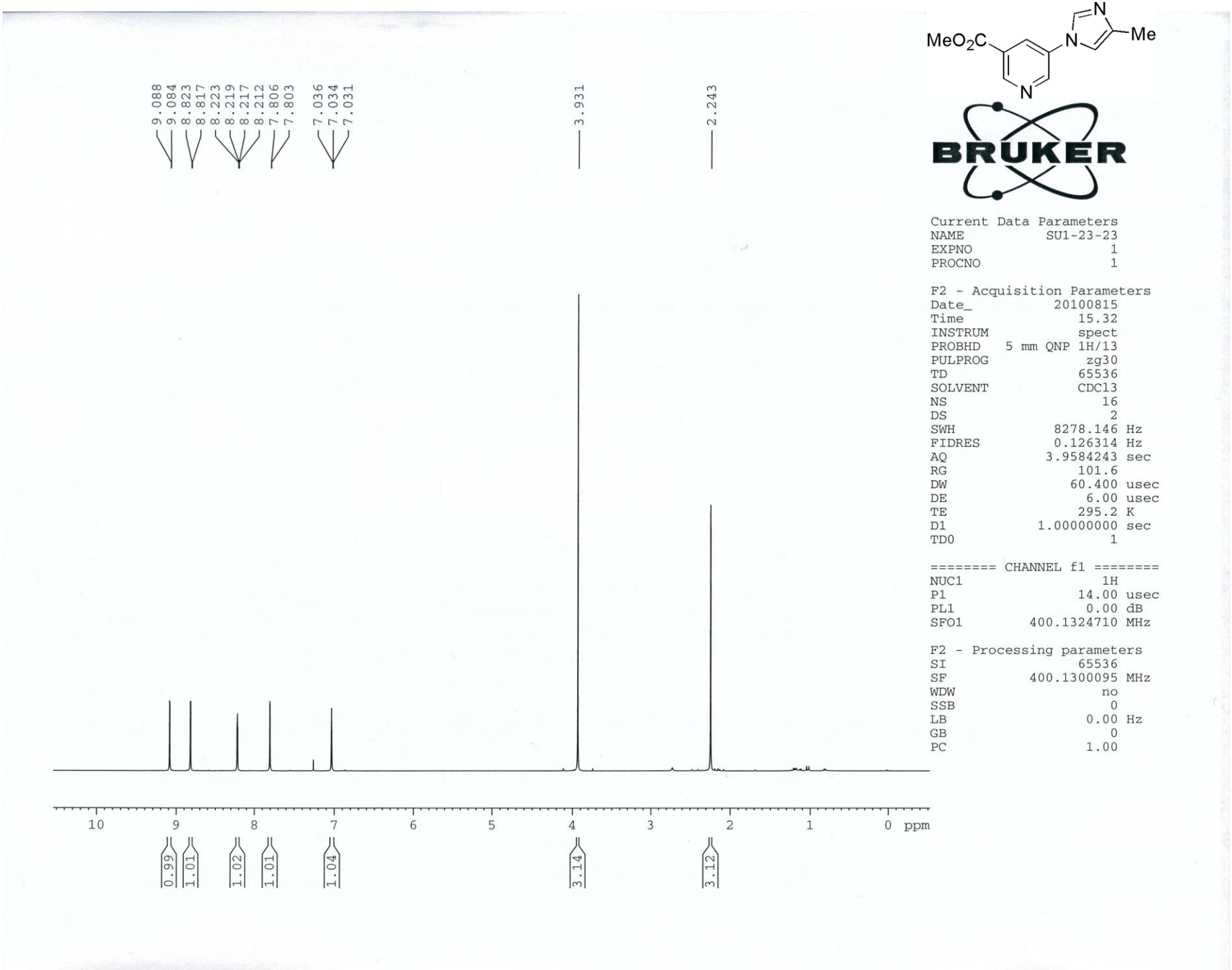
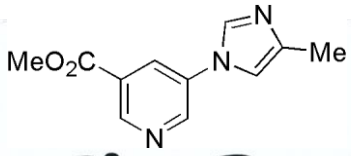
======== CHANNEL f1 ========
NUC1 13C
P1 9.38 usec
PL1 0.00 dB
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 0.00 dB
PL12 16.10 dB
PL13 19.00 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127807 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.40
200
180
160
140
120
100
80
60
40
20
0
ppm


7.867
7.865
7.864
7.835
7.833
7.821
7.819
7.817
7.816
7.680
7.676
7.404
7.402
7.400
7.399
7.397
7.393
7.391
7.373
7.355
7.354
6.916
6.915
3.803
2.160
MeO2C
N
N
Me
BRUKER
Current Data Parameters
NAME SU1-39-7
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20100826
Time 9.54
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 57
DW 60.400 usec
DE 6.00 usec
TE 295.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 14.00 usec
PL1 0.00 dB
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 65536
SF 400.1300098 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
10
9
8
7
6
5
4
3
2
1
ppm
1.02
1.03
0.97
2.11
0.99
3.12
3.11

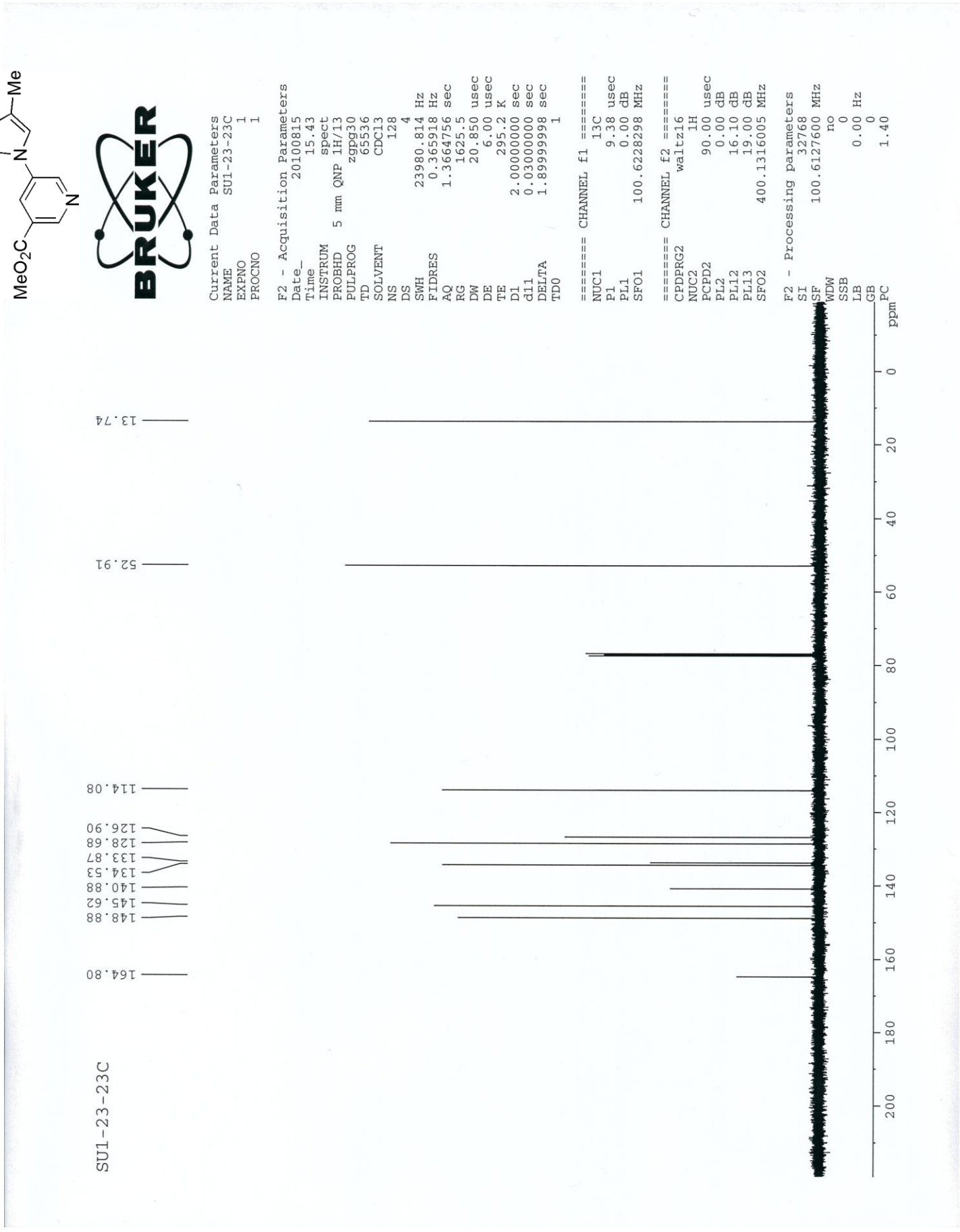

SU1-39-7C
165.77
139.78
137.43
134.34
131.75
129.83
127.72
124.72
121.44
114.17
52.33
13.58
MeO2C
N
N
Me
BRUKER
Current Data Parameters
NAME SU1-39-7C
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20100826
Time 10.52
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 128
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 1625.5
DW 20.850 usec
DE 6.00 usec
TE 295.2 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.38 usec
PL1 0.00 dB
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 0.00 dB
PL12 16.10 dB
PL13 19.00 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127725 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.40
200
180
160
140
120
100
80
60
40
20
0
ppm

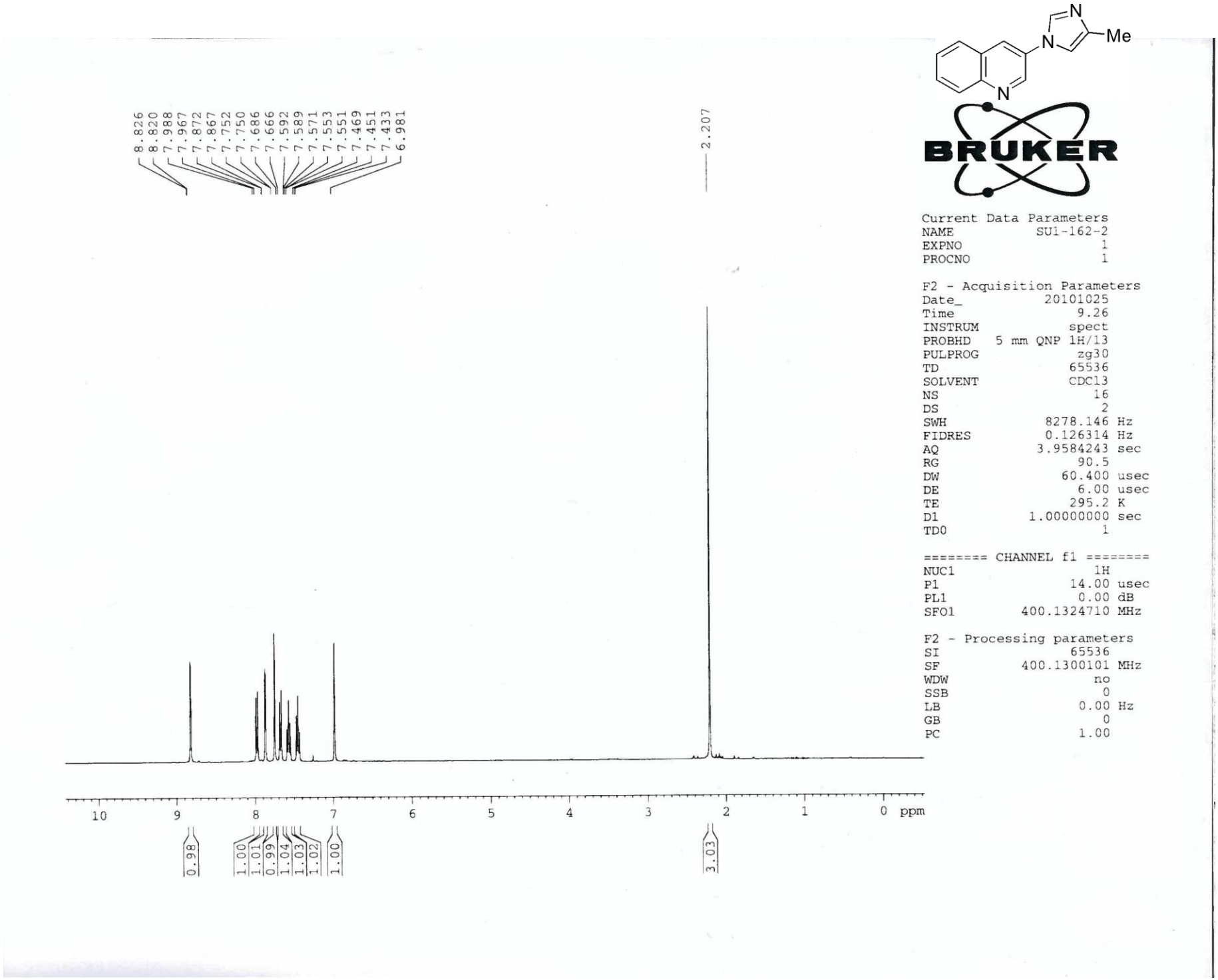


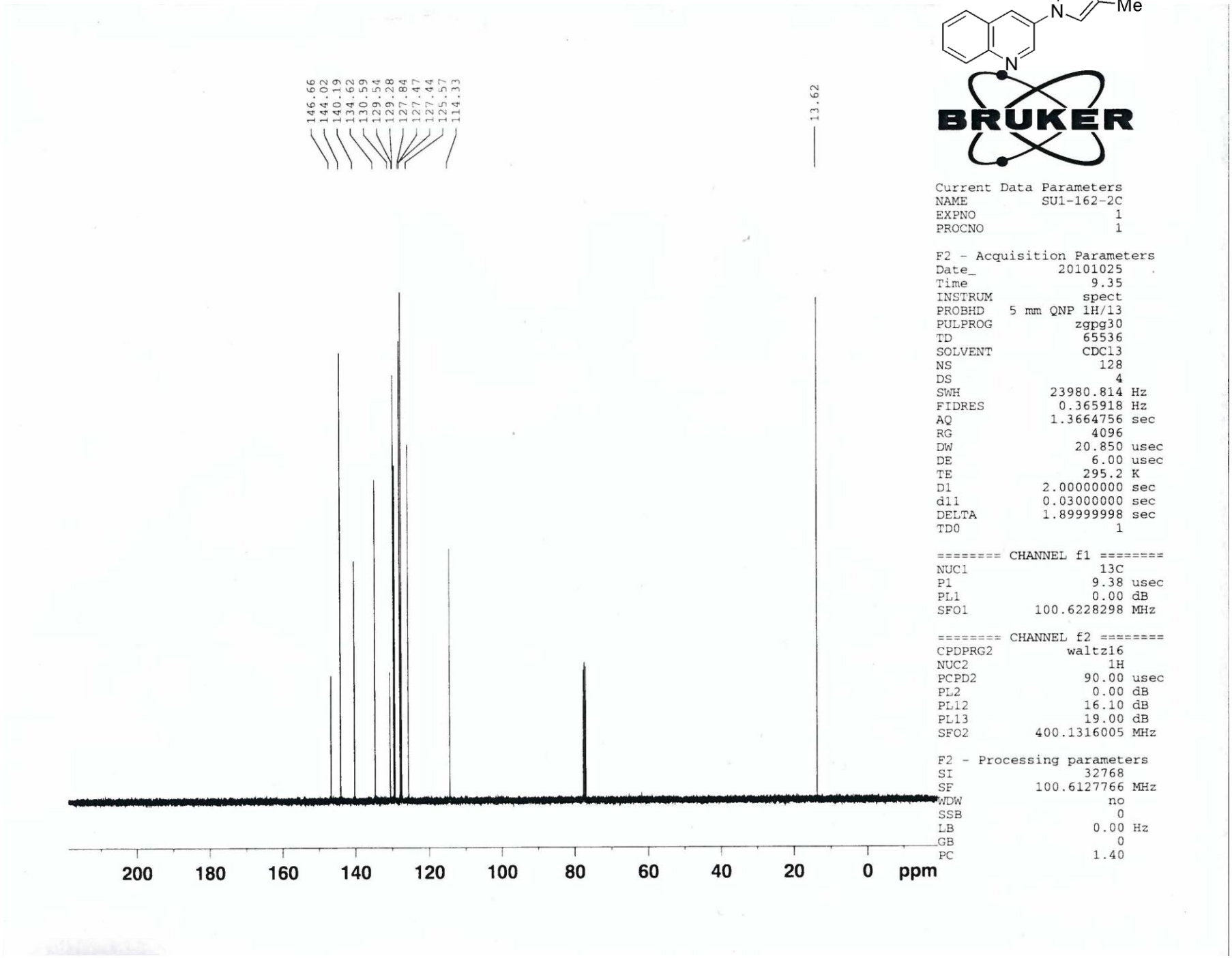



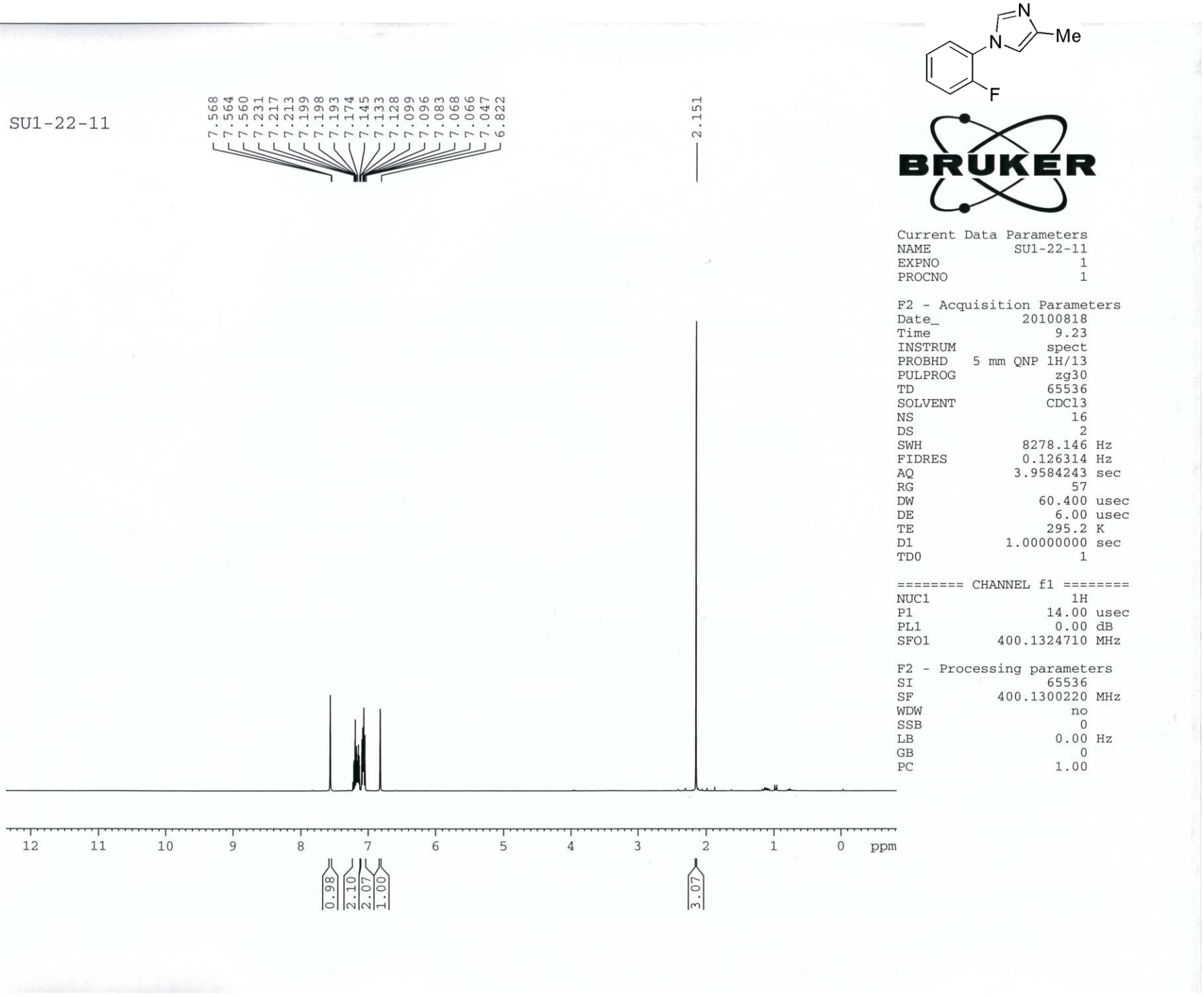

BRUKER

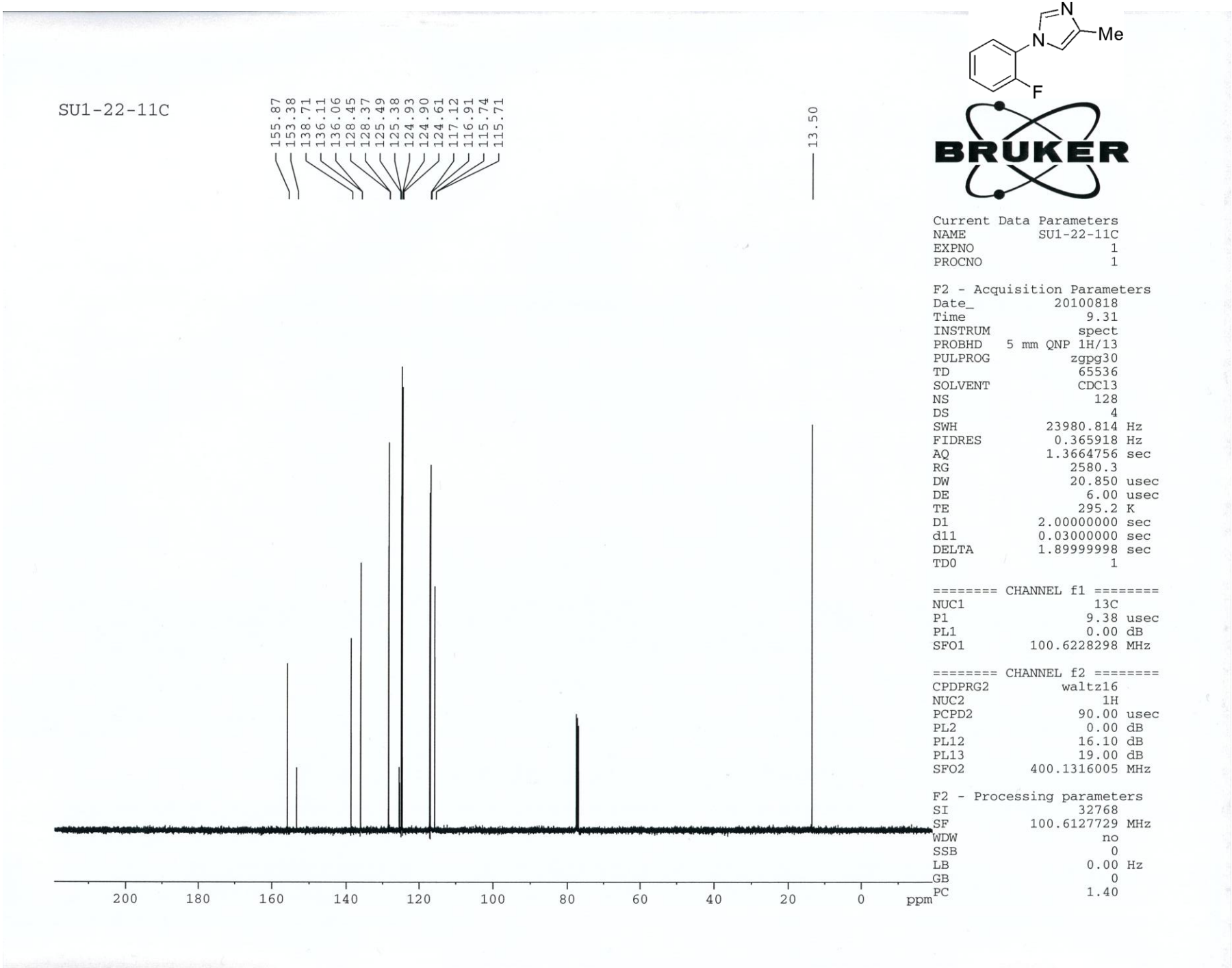
```
Current Data Parameters
NAME                  SU1-23-23
EXPNO                         1
PROCNO                        1

F2 - Acquisition Parameters
Date_                  20100815
Time                      15.32
INSTRUM                   spect
PROBHD   5 mm QNP 1H/13
PULPROG                   zg30
TD                       65536
SOLVENT                  CDCl3
NS                          16
DS                           2
SWH                   8278.146 Hz
FIDRES                0.126314 Hz
AQ                    3.9584243 sec
RG                        101.6
DW                       60.400 usec
DE                        6.00 usec
TE                       295.2 K
D1                  1.00000000 sec
TD0                           1

======== CHANNEL f1 ========
NUC1                         1H
P1                       14.00 usec
PL1                       0.00 dB
SFO1                400.1324710 MHz

F2 - Processing parameters
SI                       65536
SF                  400.1300095 MHz
WDW                         no
SSB                          0
LB                        0.00 Hz
GB                           0
PC                        1.00
```

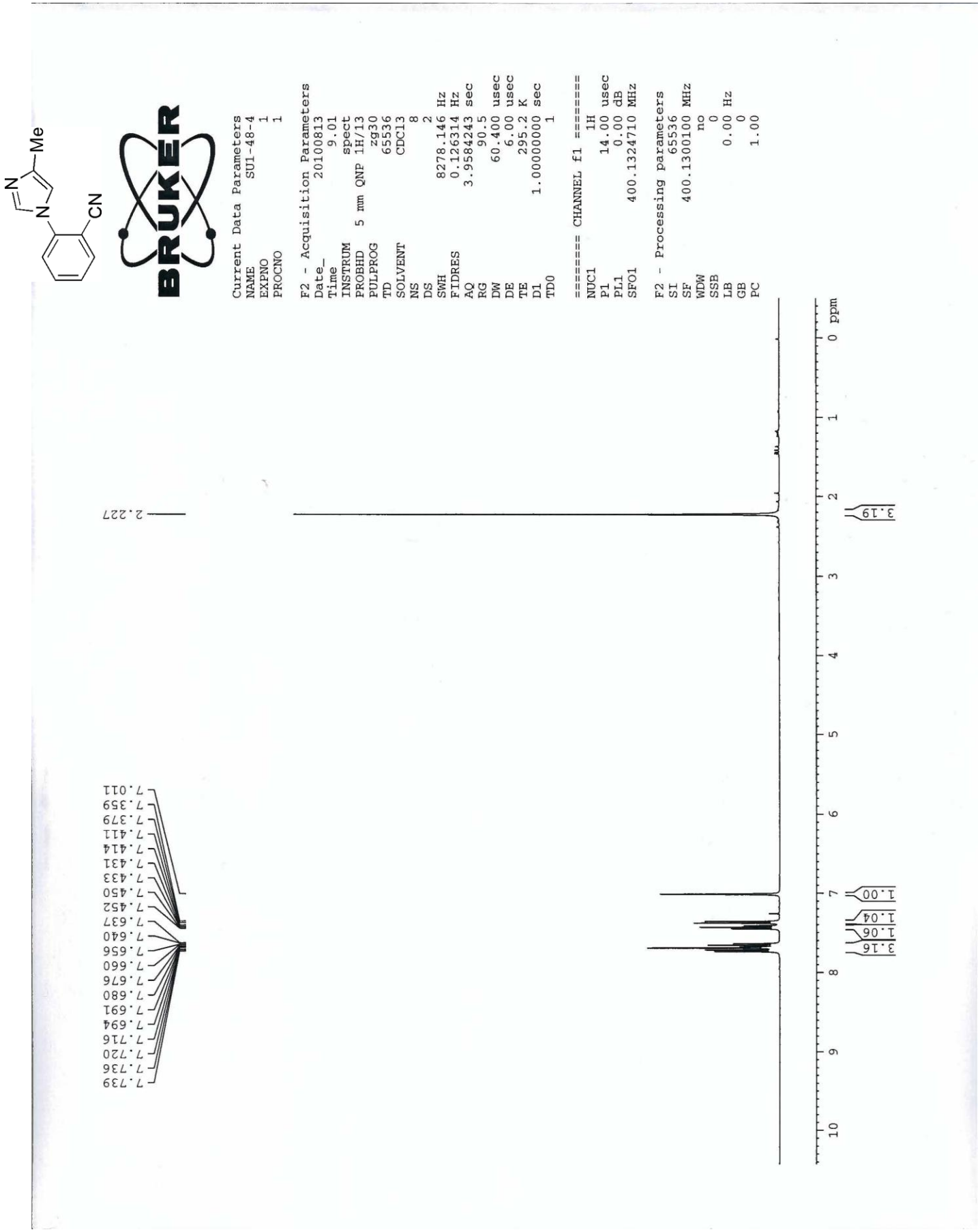

Me
N
N
N
MeO2C
BRUKER
Current Data Parameters
NAME SU1-23-23C
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20100815
Time 15.43
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 128
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 1625.5
DW 20.850 usec
DE 6.00 usec
TE 295.2 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.38 usec
PL1 0.00 dB
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 0.00 dB
PL12 16.10 dB
PL13 19.00 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127600 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.40
13.74
52.91
114.08
126.90
128.68
133.87
134.53
140.88
145.62
148.88
164.80
SU1-23-23C
ppm
200
180
160
140
120
100
80
60
40
20
0

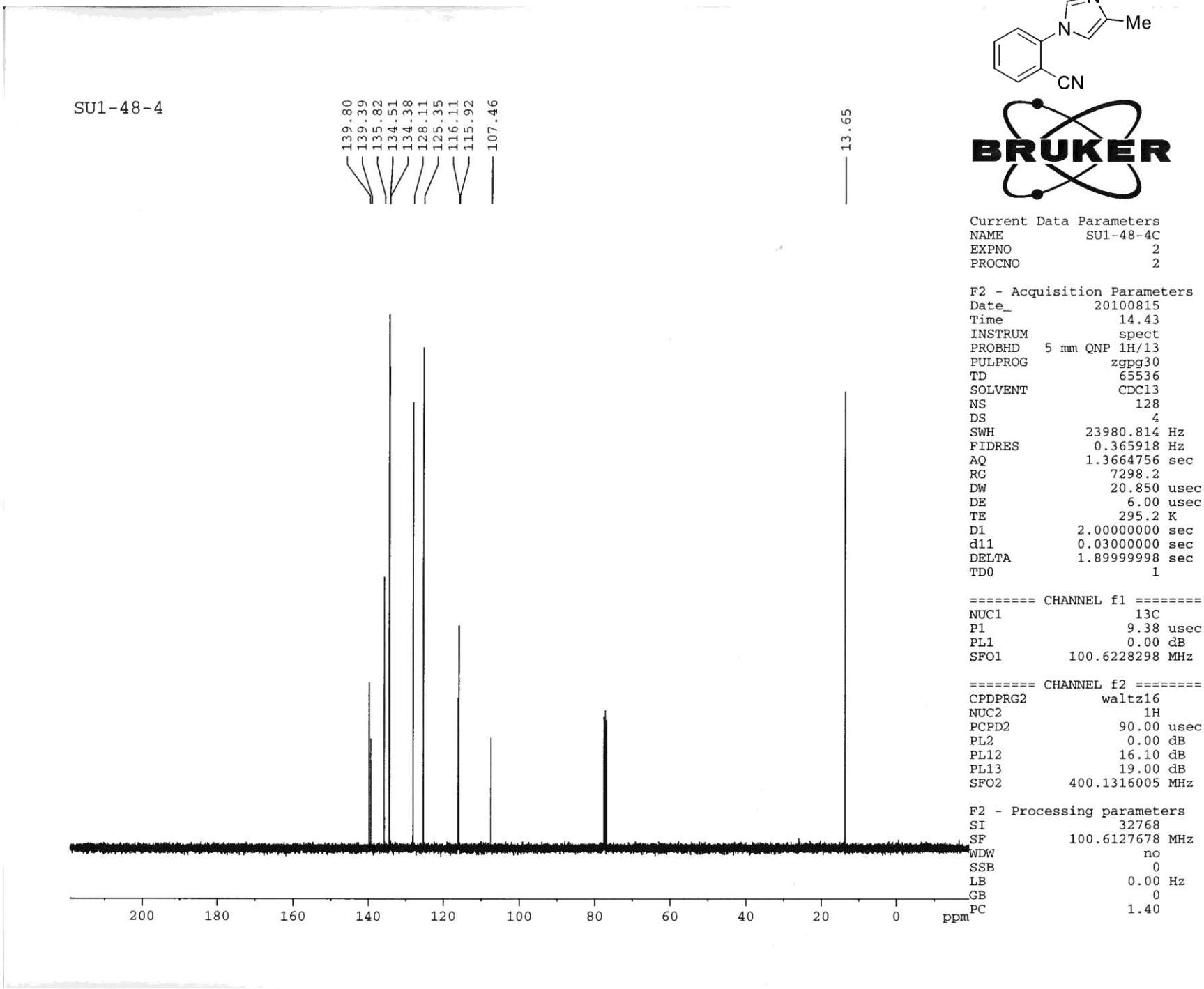

8.826
8.820
7.988
7.967
7.872
7.867
7.752
7.750
7.686
7.666
7.592
7.589
7.571
7.553
7.551
7.469
7.451
7.433
6.981
2.207
10
9
8
7
6
5
4
3
2
1
0 ppm
0.98
1.00
1.01
0.99
1.04
1.03
1.02
1.00
3.03
N
N
Me
N
BRUKER
Current Data Parameters
NAME SU1-162-2
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20101025
Time 9.26
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 90.5
DW 60.400 usec
DE 6.00 usec
TE 295.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 14.00 usec
PL1 0.00 dB
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 65536
SF 400.1300101 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

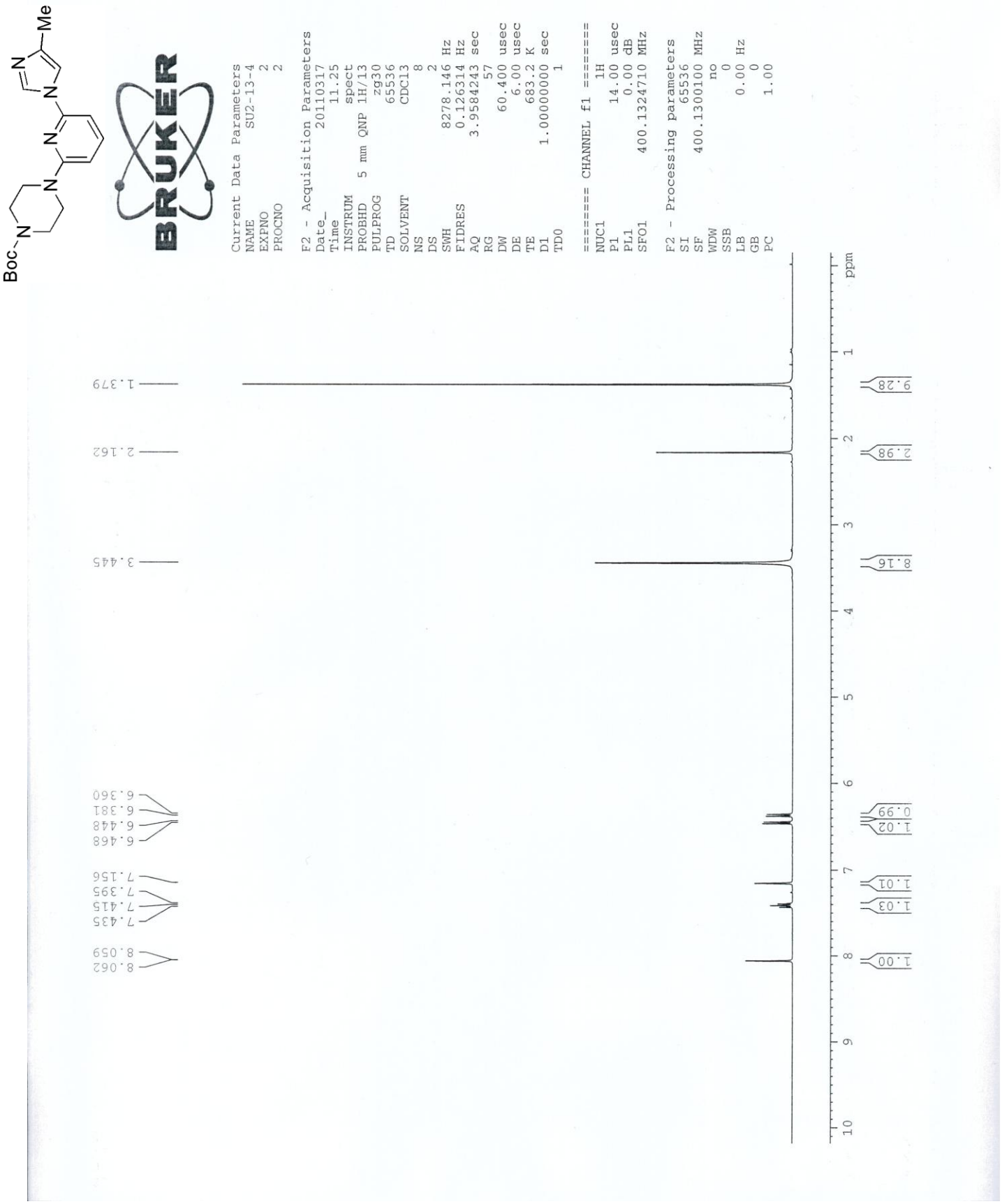

146.66
144.02
140.19
134.62
130.59
129.54
129.28
127.84
127.47
127.44
125.57
114.33
13.62
200
180
160
140
120
100
80
60
40
20
0
ppm
N
N
Me
N
BRUKER
Current Data Parameters
NAME SU1-162-2C
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20101025
Time 9.35
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 128
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 4096
DW 20.850 usec
DE 6.00 usec
TE 295.2 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.38 usec
PL1 0.00 dB
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 0.00 dB
PL12 16.10 dB
PL13 19.00 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127766 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.40

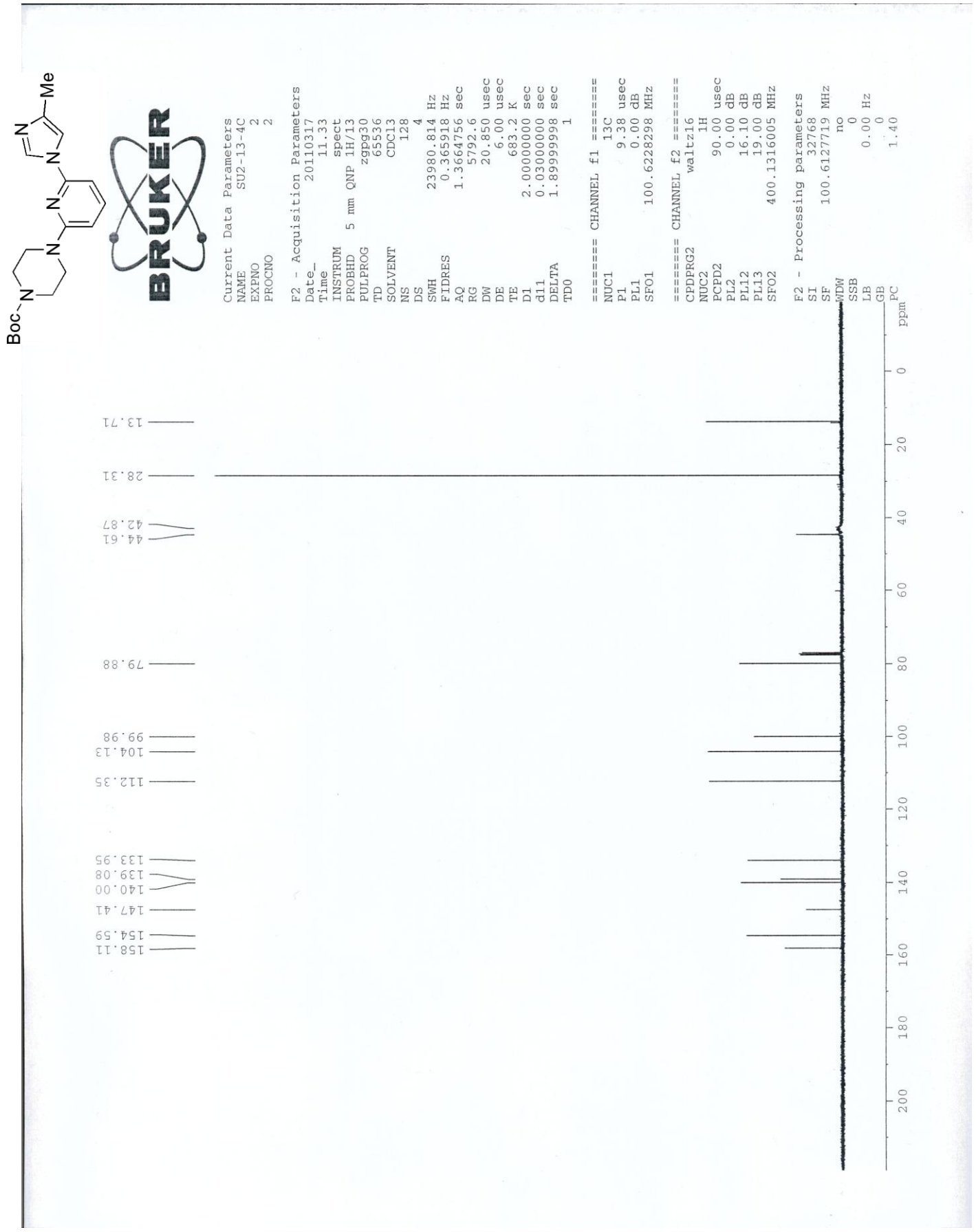

SU1-22-11
7.568
7.564
7.560
7.231
7.217
7.213
7.199
7.198
7.193
7.174
7.145
7.133
7.128
7.099
7.096
7.083
7.068
7.066
7.047
6.822
2.151
N
N
Me
F
BRUKER
Current Data Parameters
NAME SU1-22-11
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20100818
Time 9.23
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 57
DW 60.400 usec
DE 6.00 usec
TE 295.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 14.00 usec
PL1 0.00 dB
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 65536
SF 400.1300220 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
12
11
10
9
8
7
6
5
4
3
2
1
0
ppm
0.98
2.10
2.07
1.00
3.07

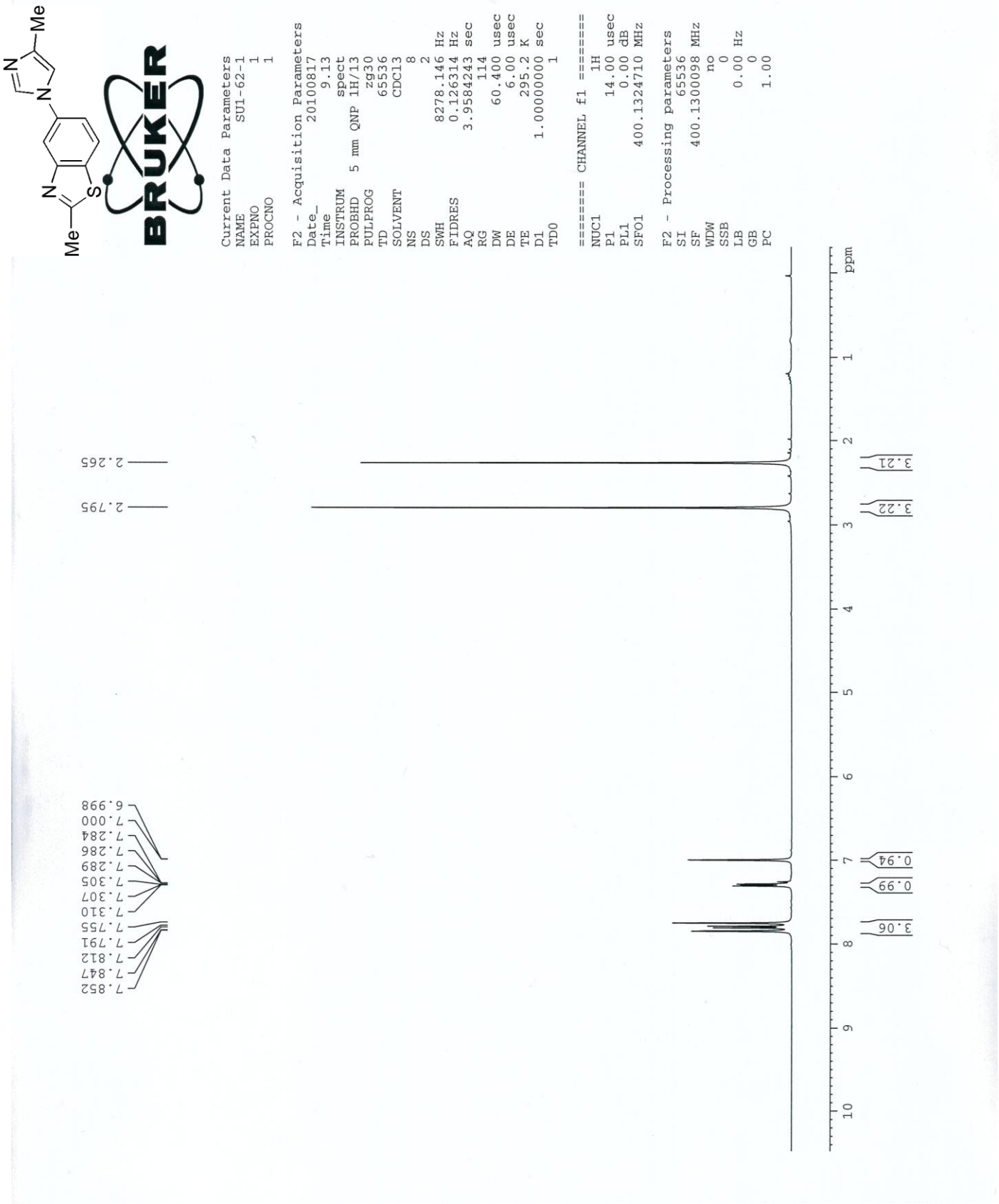

SU1-22-11C
155.87
153.38
138.71
136.11
136.06
128.45
128.37
125.49
125.38
124.93
124.90
124.61
117.12
116.91
115.74
115.71
13.50
N
N
Me
F
BRUKER
Current Data Parameters
NAME SU1-22-11C
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20100818
Time 9.31
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 128
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 2580.3
DW 20.850 usec
DE 6.00 usec
TE 295.2 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.38 usec
PL1 0.00 dB
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 0.00 dB
PL12 16.10 dB
PL13 19.00 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127729 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.40
200
180
160
140
120
100
80
60
40
20
0
ppm

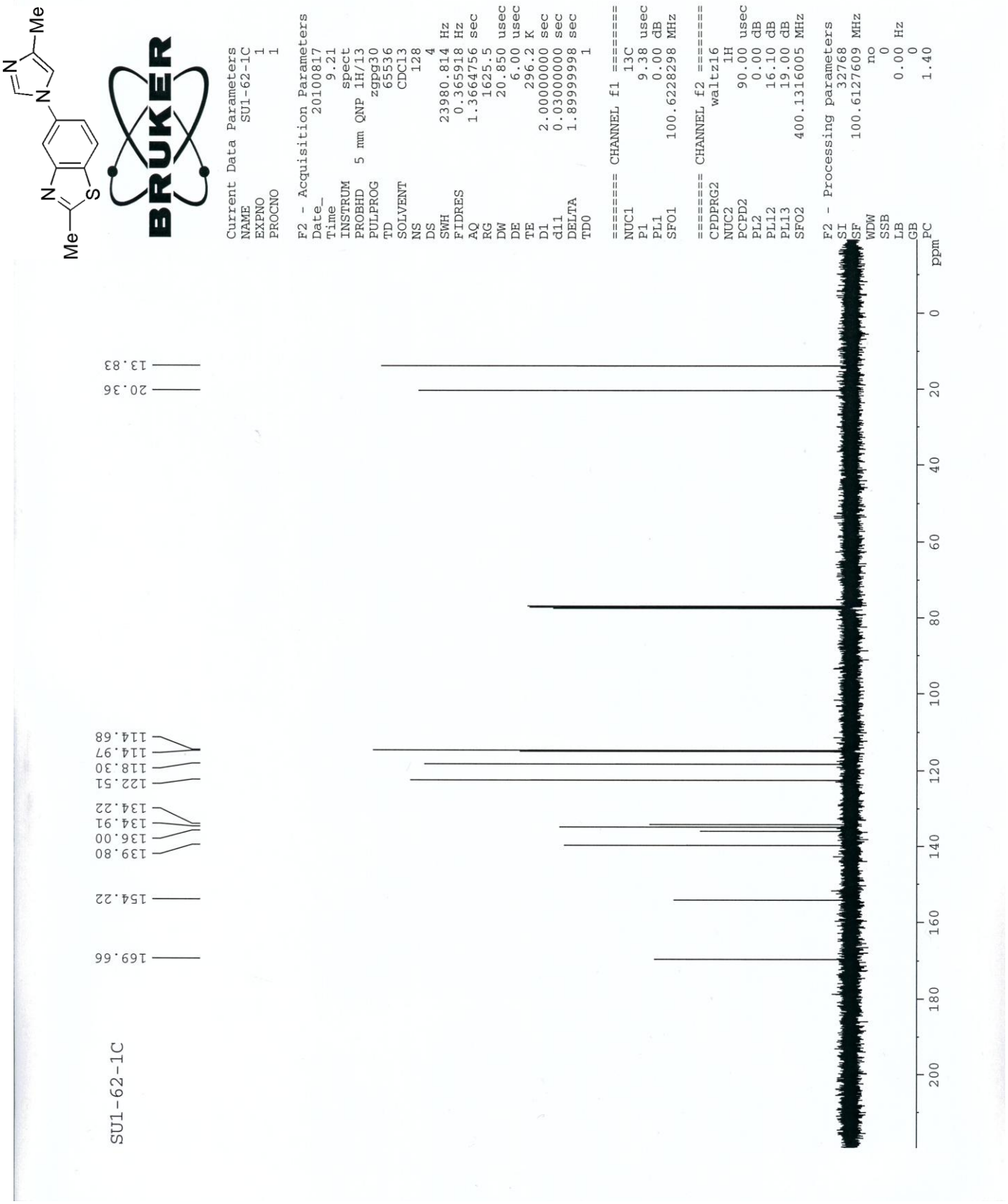

Me
N
N
CN
BRUKER
Current Data Parameters
NAME SU1-48-4
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20100813
Time 9.01
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 90.5
DW 60.400 usec
DE 6.00 usec
TE 295.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 14.00 usec
PL1 0.00 dB
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 65536
SF 400.1300100 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
2.227
7.011
7.359
7.379
7.411
7.414
7.431
7.433
7.450
7.452
7.637
7.640
7.656
7.660
7.676
7.680
7.691
7.694
7.716
7.720
7.736
7.739
3.19
3.16
1.06
1.04
1.00
ppm

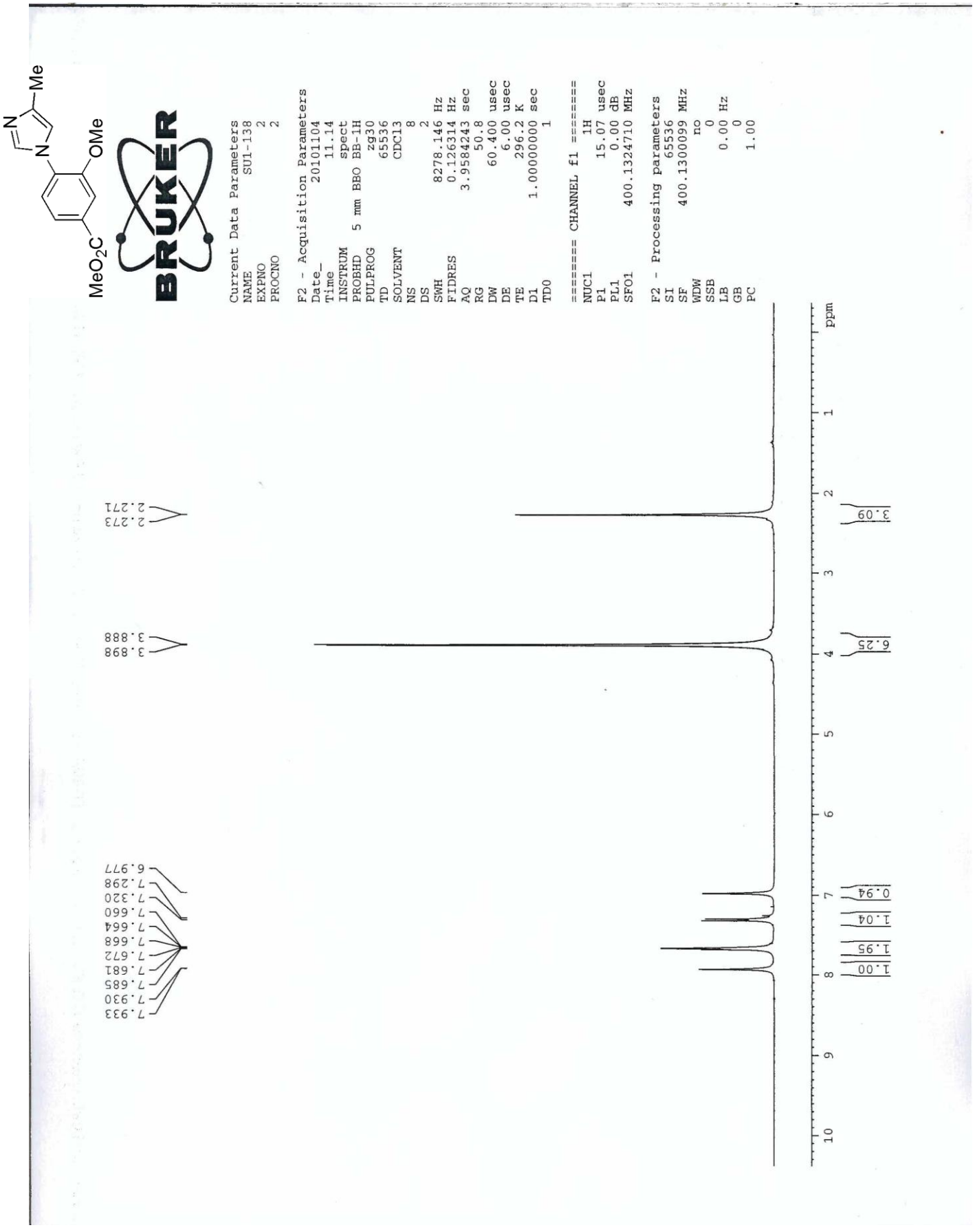

SU1-48-4
139.80
139.39
135.82
134.51
134.38
128.11
125.35
116.11
115.92
107.46
13.65
N
N
Me
CN
BRUKER
Current Data Parameters
NAME SU1-48-4C
EXPNO 2
PROCNO 2
F2 - Acquisition Parameters
Date_ 20100815
Time 14.43
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 128
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 7298.2
DW 20.850 usec
DE 6.00 usec
TE 295.2 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.38 usec
PL1 0.00 dB
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 0.00 dB
PL12 16.10 dB
PL13 19.00 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127678 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.40
200
180
160
140
120
100
80
60
40
20
0
ppm

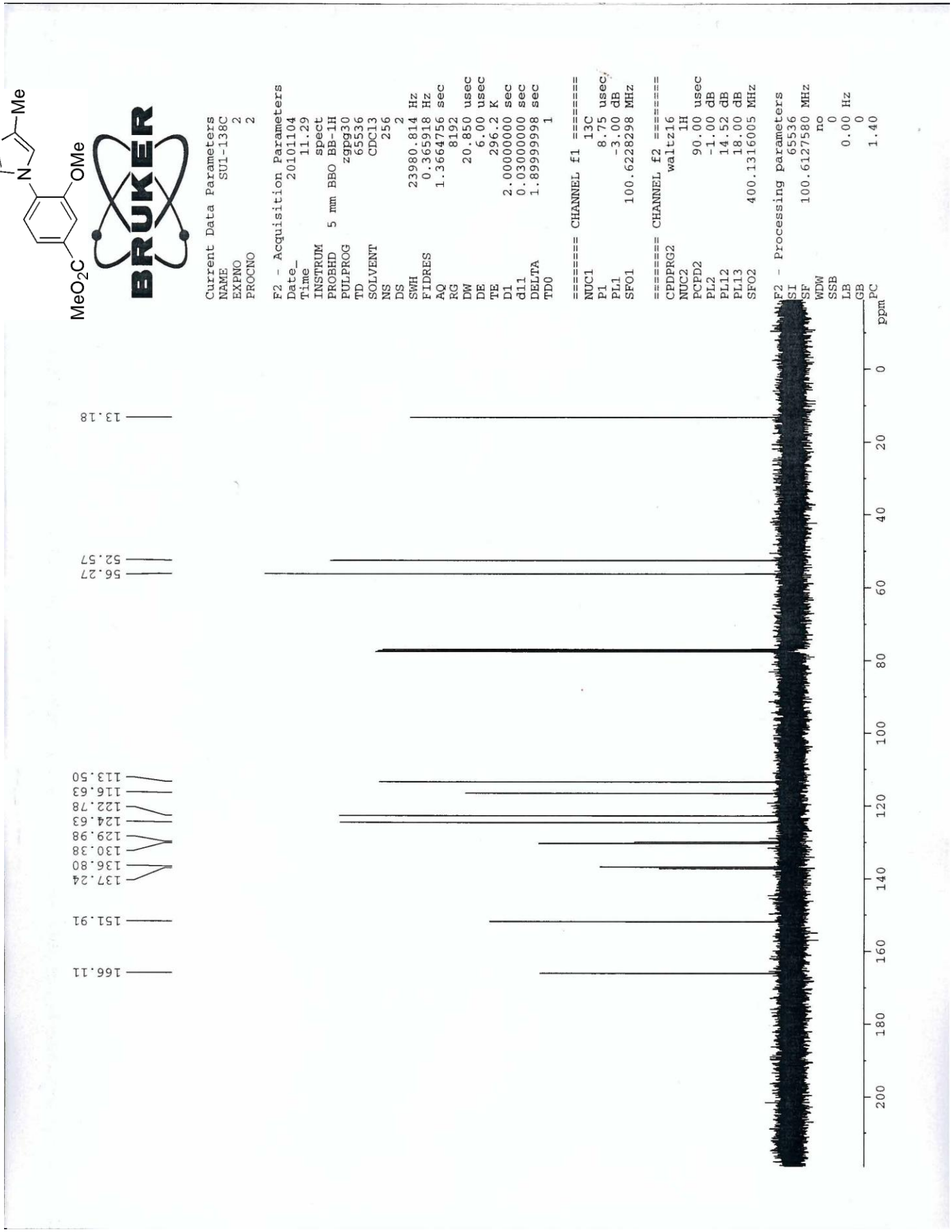

Boc
N
N
N
N
Me
BRUKER
Current Data Parameters
NAME SU2-13-4
EXPNO 2
PROCNO 2
F2 - Acquisition Parameters
Date_ 20110317
Time 11.25
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 57
DW 60.400 usec
DE 6.00 usec
TE 683.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 14.00 usec
PL1 0.00 dB
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 65536
SF 400.1300100 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
1.379
2.162
3.445
6.360
6.381
6.448
6.468
7.156
7.395
7.415
7.435
8.059
8.062
9.28
2.98
8.16
0.99
1.02
1.01
1.03
1.00
ppm


Boc
Me
BRUKER
Current Data Parameters
NAME SU2-13-4C
EXPNO 2
PROCNO 2
F2 - Acquisition Parameters
Date_ 20110317
Time 11.33
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 128
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 5792.6
DW 20.850 usec
DE 6.00 usec
TE 683.2 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.38 usec
PL1 0.00 dB
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 0.00 dB
PL12 16.10 dB
PL13 19.00 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127719 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.40
13.71
28.31
42.87
44.61
79.88
99.98
104.13
112.35
133.95
139.08
140.00
147.41
154.59
158.11
ppm
0
20
40
60
80
100
120
140
160
180
200


Me
N
N
S
Me
BRUKER
Current Data Parameters
NAME SU1-62-1
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20100817
Time 9.13
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 114
DW 60.400 usec
DE 6.00 usec
TE 295.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 14.00 usec
PL1 0.00 dB
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 65536
SF 400.1300098 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
2.265
2.795
6.998
7.000
7.284
7.286
7.289
7.305
7.307
7.310
7.755
7.791
7.812
7.847
7.852
3.21
3.22
0.94
0.99
3.06
ppm


Me
N
N
N
S
Me
BRUKER
Current Data Parameters
NAME SU1-62-1C
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20100817
Time 9.21
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 128
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 1625.5
DW 20.850 usec
DE 6.00 usec
TE 296.2 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.38 usec
PL1 0.00 dB
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 0.00 dB
PL12 16.10 dB
PL13 19.00 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127609 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
1.40
PC
ppm
13.83
20.36
114.68
114.97
118.30
122.51
134.22
134.91
136.00
139.80
154.22
169.66
SU1-62-1C


Me
N
N
OMe
MeO2C
BRUKER
Current Data Parameters
NAME SU1-138
EXPNO 2
PROCNO 2
F2 - Acquisition Parameters
Date_ 20101104
Time 11.14
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 50.8
DW 60.400 usec
DE 6.00 usec
TE 296.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 15.07 usec
PL1 0.00 dB
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 65536
SF 400.1300099 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
2.271
2.273
3.888
3.898
6.977
7.298
7.320
7.660
7.664
7.668
7.672
7.681
7.685
7.930
7.933
3.09
6.25
0.94
1.04
1.95
1.00
ppm


Me
N
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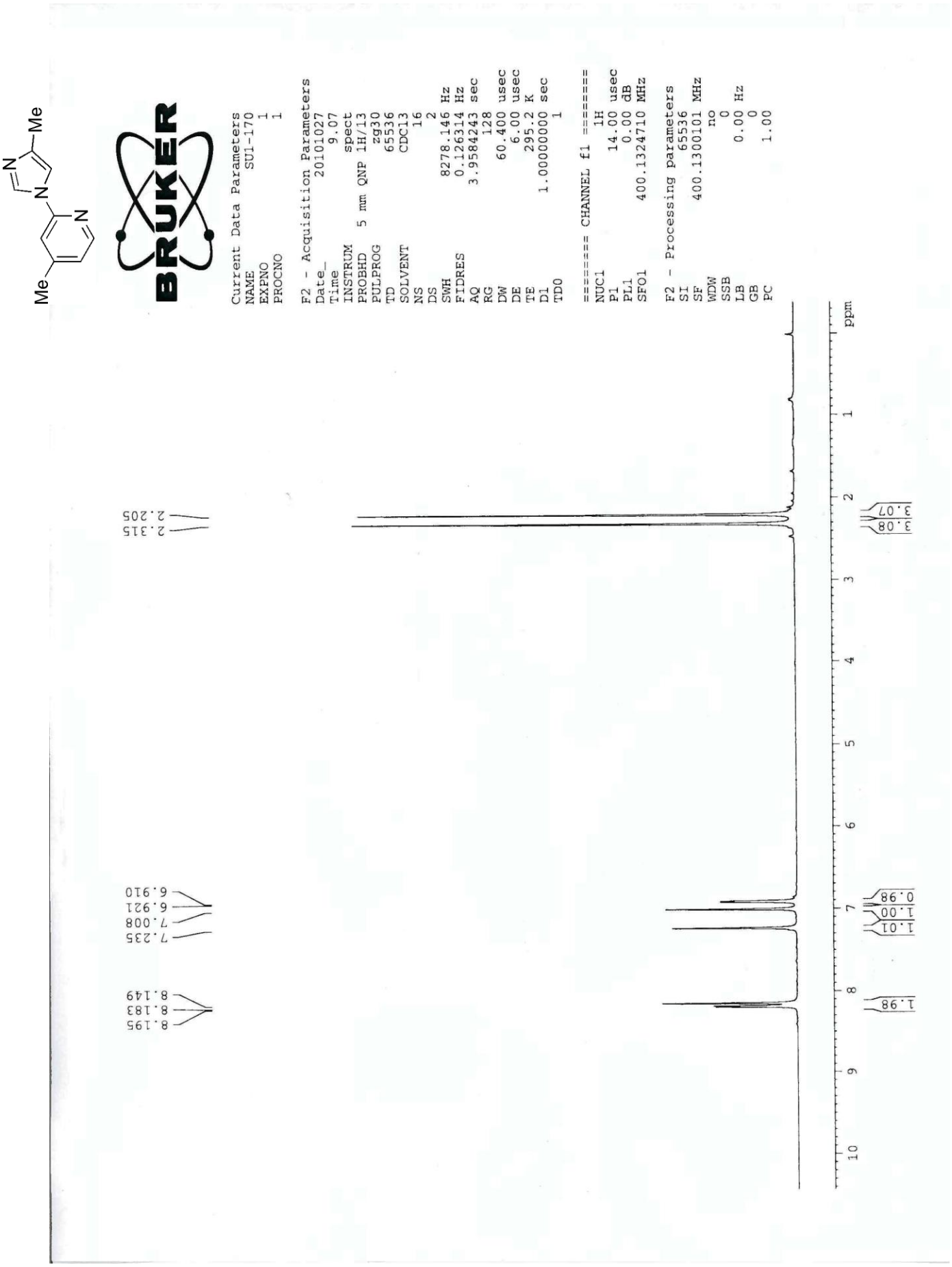
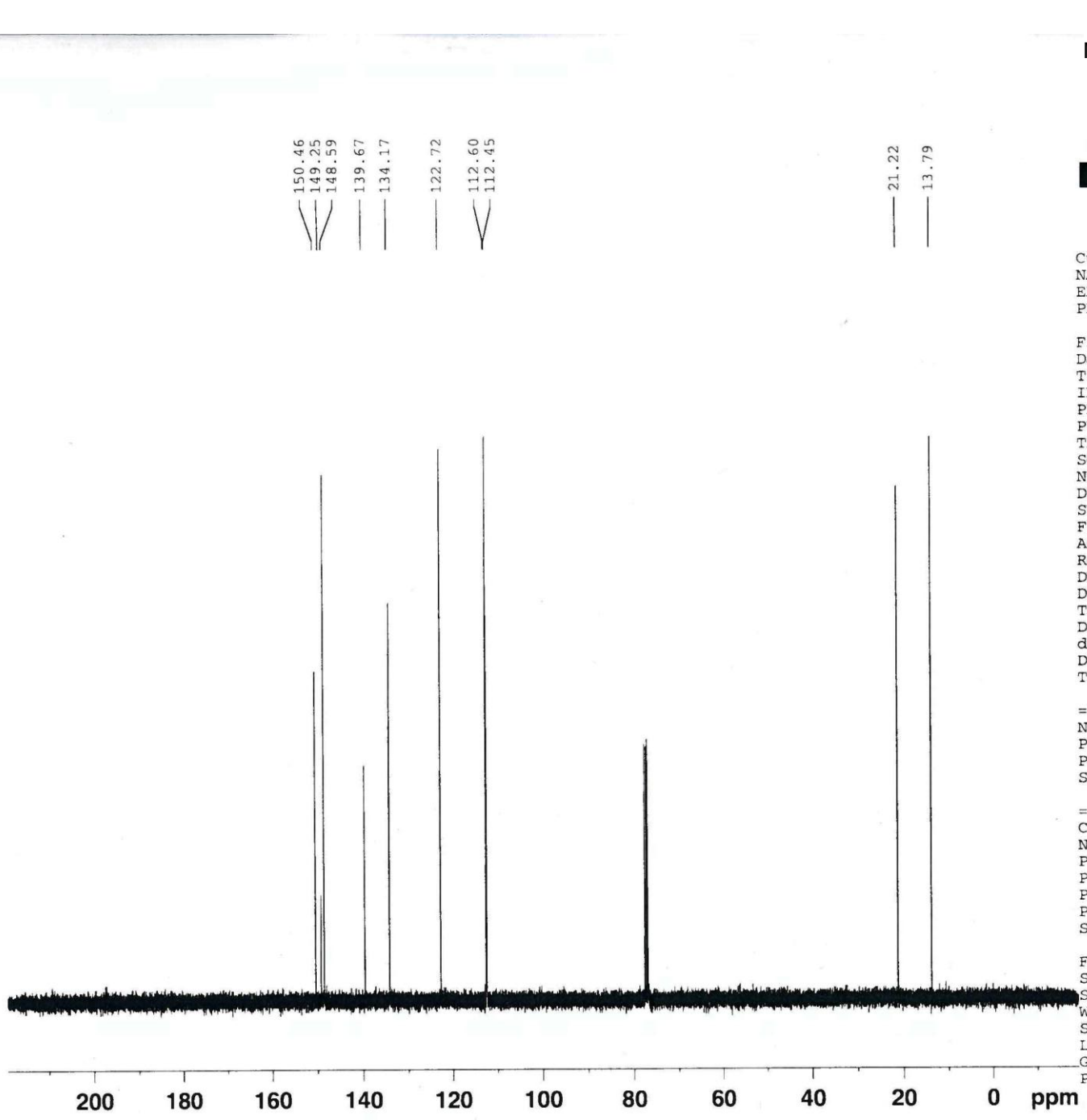
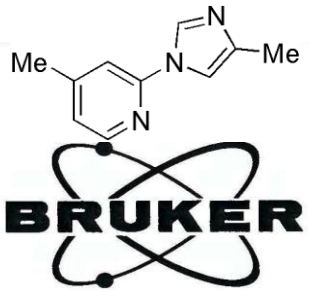
OMe
MeO2C
BRUKER
Current Data Parameters
NAME SU1-138C
EXPNO 2
PROCNO 2
F2 - Acquisition Parameters
Date_ 20101104
Time 11.29
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 256
DS 2
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 8192
DW 20.850 usec
DE 6.00 usec
TE 296.2 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 8.75 usec
PL1 -3.00 dB
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -1.00 dB
PL12 14.52 dB
PL13 18.00 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 65536
SF 100.6127580 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.40
13.18
52.57
56.27
113.50
116.63
122.78
124.63
129.98
130.38
136.80
137.24
151.91
166.11
200
180
160
140
120
100
80
60
40
20
0
ppm

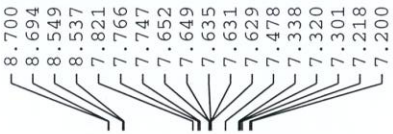

Me
Me
N
N
N
BRUKER
Current Data Parameters
NAME SU1-170
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20101027
Time 9.07
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDC13
NS 16
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 128
DW 60.400 usec
DE 6.00 usec
TE 295.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 14.00 usec
PL1 0.00 dB
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 65536
SF 400.1300101 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
2.315
2.205
7.235
7.008
6.921
6.910
8.195
8.183
8.149
3.08
3.07
1.98
1.01
1.00
0.98
ppm

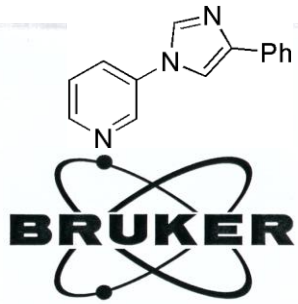



```
Current Data Parameters
NAME                  SU1-170C
EXPNO                        1
PROCNO                       1

F2 - Acquisition Parameters
Date_                 20101027
Time                      9.15
INSTRUM                  spect
PROBHD    5 mm QNP 1H/13
PULPROG                 zgpg30
TD                       65536
SOLVENT                  CDC13
NS                         128
DS                           4
SWH                23980.814 Hz
FIDRES             0.365918 Hz
AQ                 1.3664756 sec
RG                    1625.5
DW                   20.850 usec
DE                    6.00 usec
TE                    295.2 K
D1                 2.00000000 sec
d11                0.03000000 sec
DELTA              1.89999998 sec
TD0                          1

======== CHANNEL f1 ========
NUC1                       13C
P1                        9.38 usec
PL1                       0.00 dB
SFO1              100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2                waltz16
NUC2                       1H
PCPD2                    90.00 usec
PL2                       0.00 dB
PL12                     16.10 dB
PL13                     19.00 dB
SFO2              400.1316005 MHz

F2 - Processing parameters
SI                         32768
SF                100.6127641 MHz
WDW                           no
SSB                            0
LB                         0.00 Hz
GB                             0
PC                          1.40
```

SU1-23-18



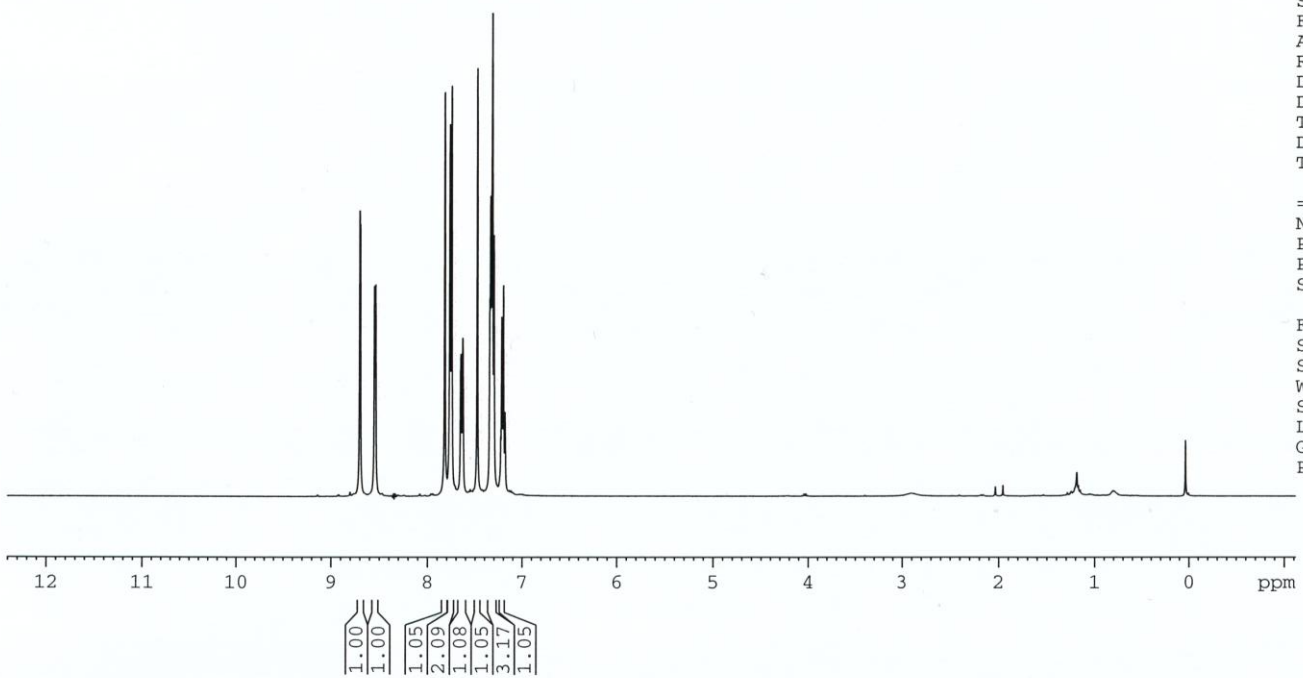




Current Data Parameters
NAME SU1-23-18
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20100813
Time 9.30
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 57
DW 60.400 usec
DE 6.00 usec
TE 295.2 K
D1 1.00000000 sec
TD0 1

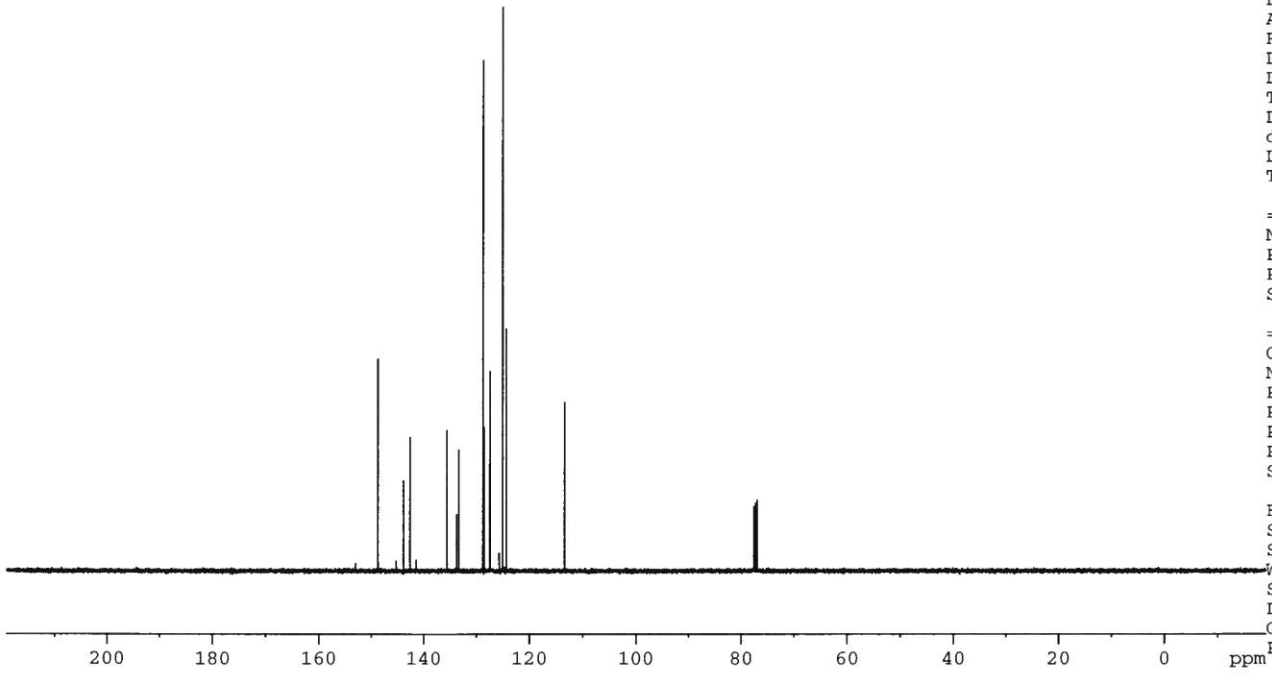
======== CHANNEL f1 ========
NUC1 1H
P1 14.00 usec
PL1 0.00 dB
SFO1 400.1324710 MHz

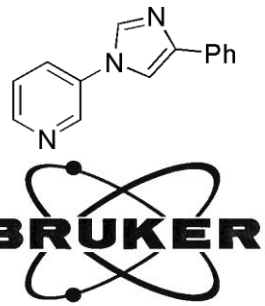
F2 - Processing parameters
SI 65536
SF 400.1300220 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00



SU1-23-18

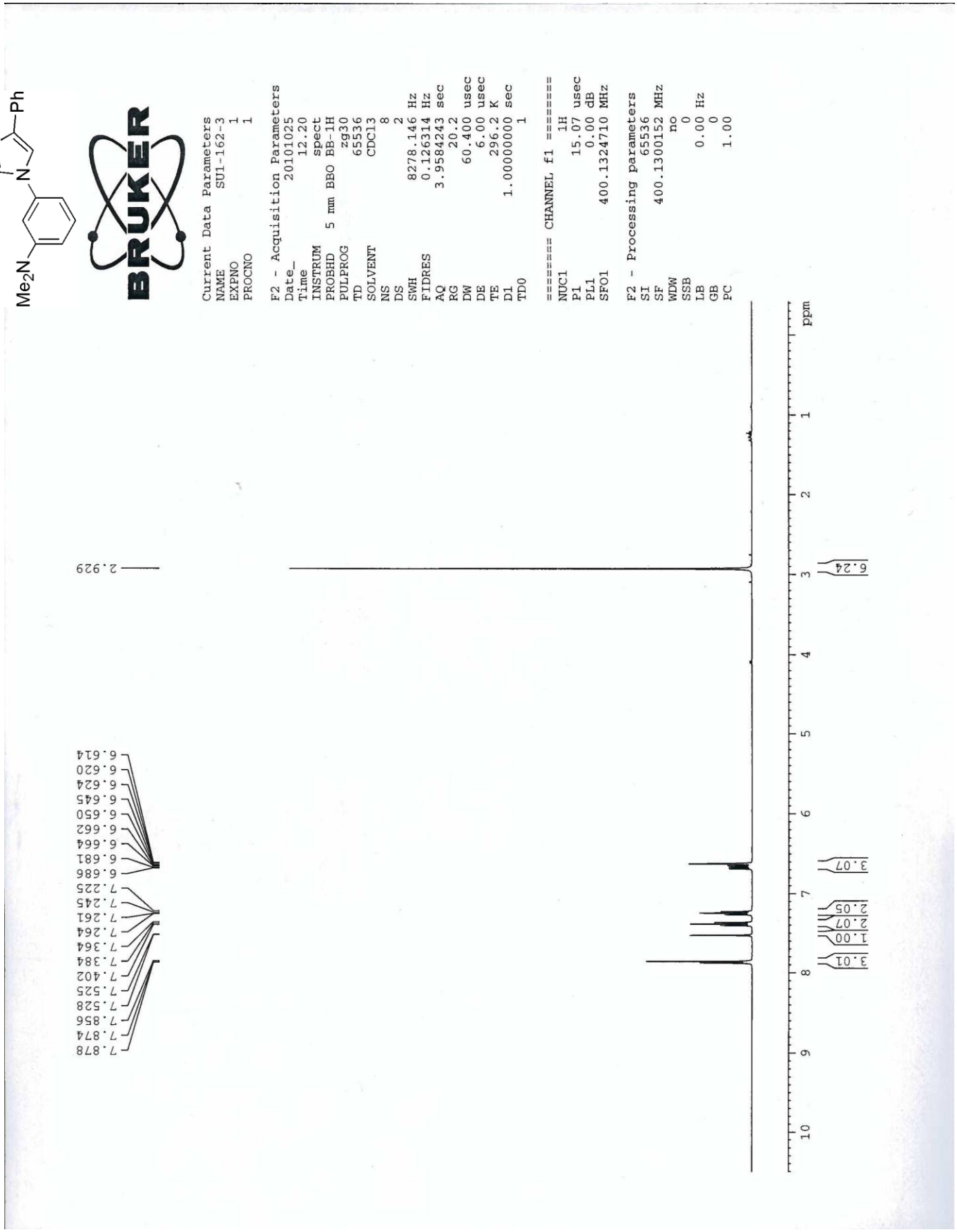
148.77
143.87
142.62
135.58
133.74
133.32
128.75
128.56
127.44
125.05
124.33
113.39

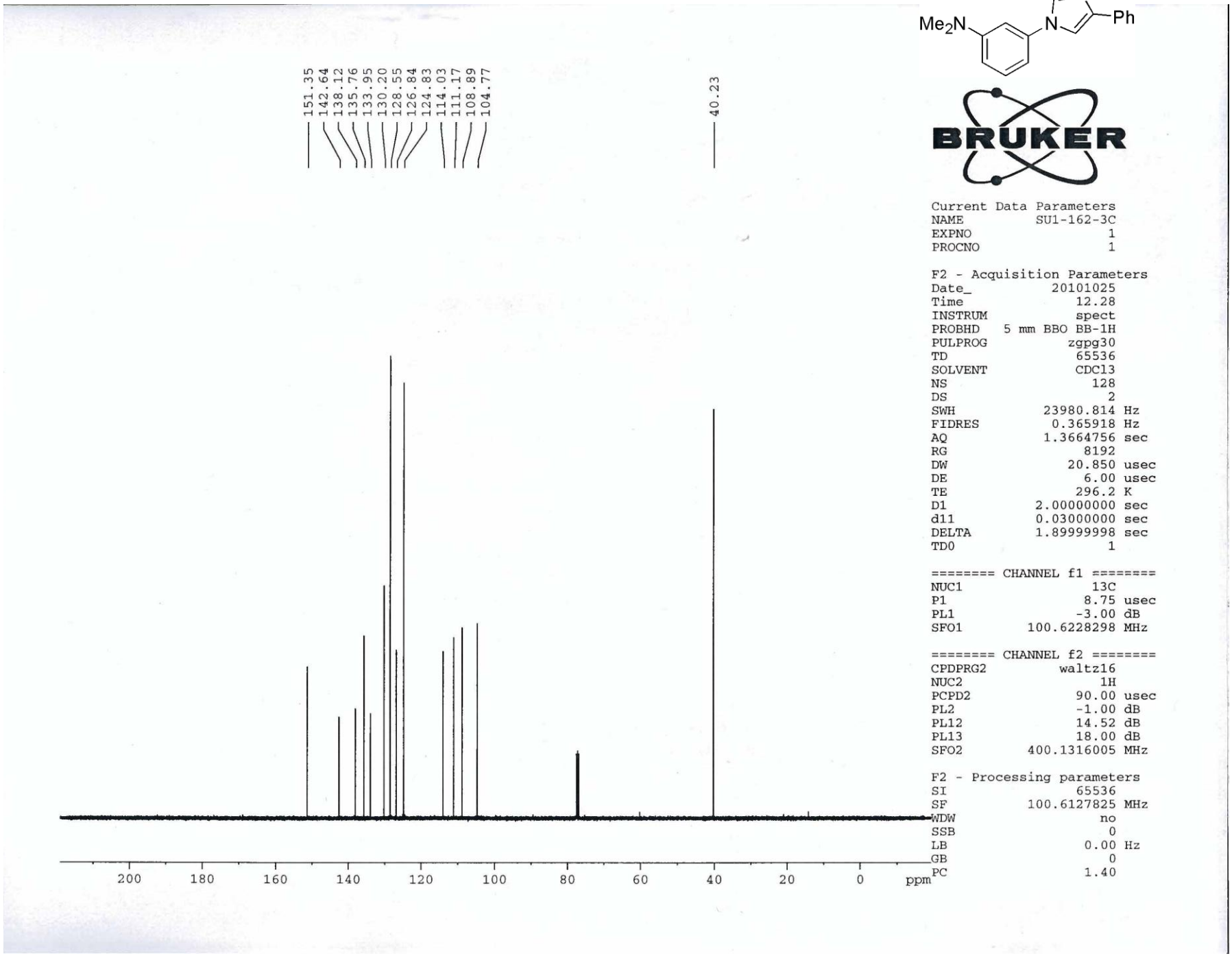


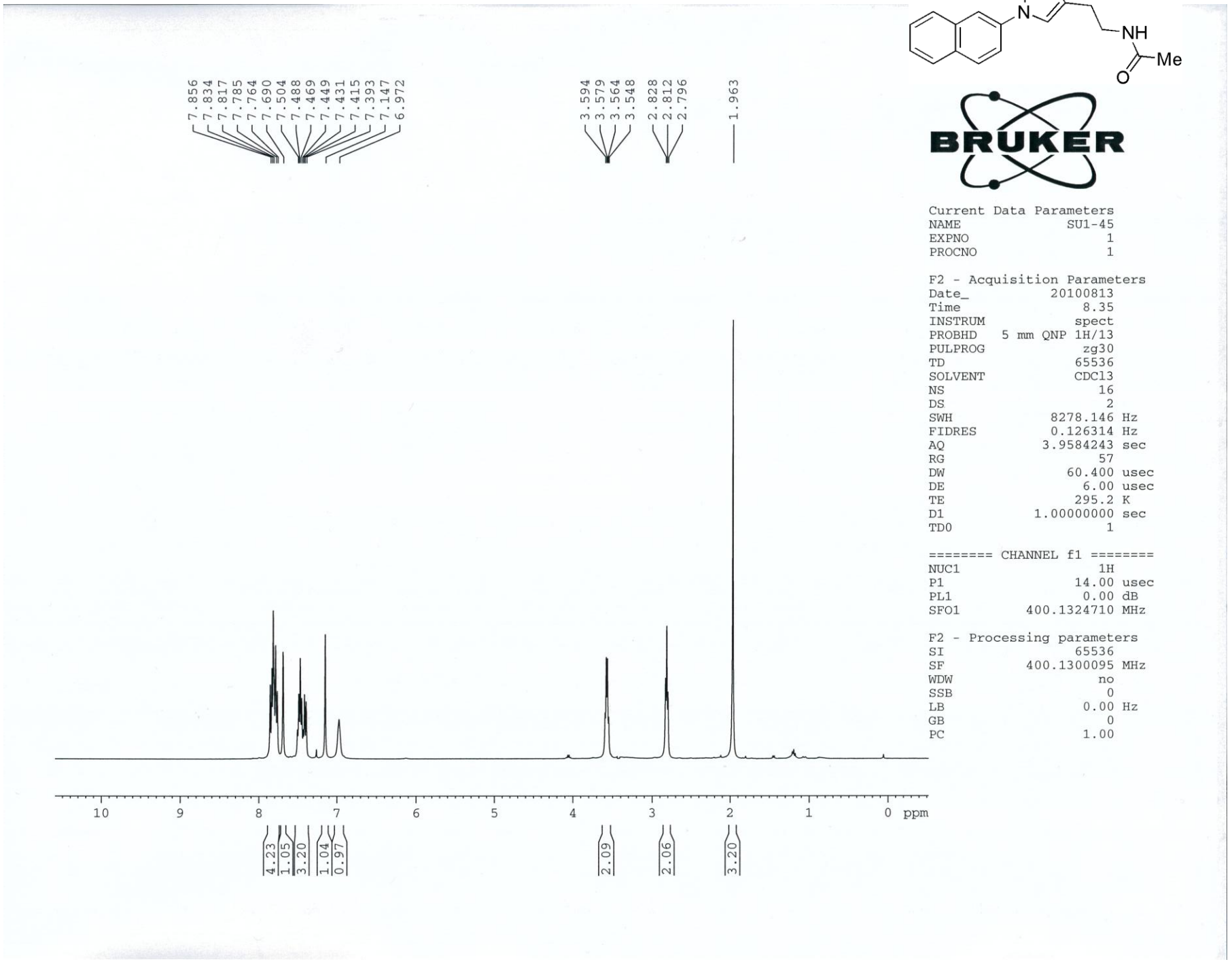



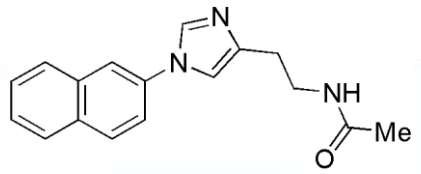

BRUKER

```
Current Data Parameters
NAME                  SU1-23-18
EXPNO                         2
PROCNO                        2

F2 - Acquisition Parameters
Date_                  20100815
Time                      15.24
INSTRUM                   spect
PROBHD    5 mm QNP 1H/13
PULPROG                  zgpg30
TD                        65536
SOLVENT                   CDCl3
NS                          128
DS                            4
SWH                   23980.814 Hz
FIDRES                 0.365918 Hz
AQ                     1.3664756 sec
RG                        1625.5
DW                        20.850 usec
DE                         6.00 usec
TE                        295.2 K
D1                    2.00000000 sec
d11                   0.03000000 sec
DELTA                 1.89999998 sec
TD0                           1

======== CHANNEL f1 ========
NUC1                        13C
P1                         9.38 usec
PL1                        0.00 dB
SFO1                100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2                 waltz16
NUC2                        1H
PCPD2                     90.00 usec
PL2                        0.00 dB
PL12                      16.10 dB
PL13                      19.00 dB
SFO2                400.1316005 MHz

F2 - Processing parameters
SI                          32768
SF                 100.6127669 MHz
WDW                            no
SSB                             0
LB                         0.00 Hz
GB                              0
PC                         1.40
```

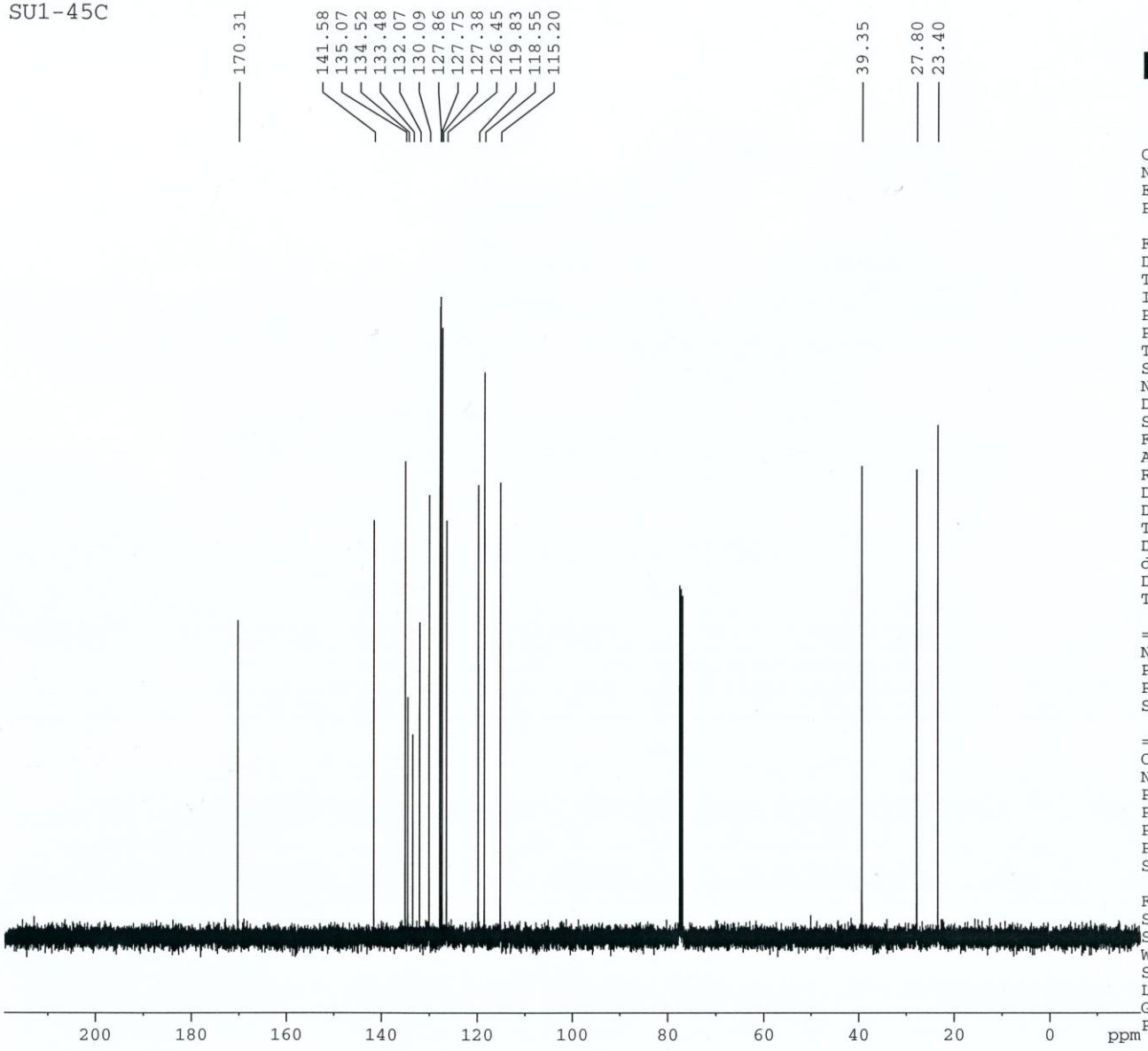

Ph
N
N
Me2N
BRUKER
Current Data Parameters
NAME SU1-162-3
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20101025
Time 12.20
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 20.2
DW 60.400 usec
DE 6.00 usec
TE 296.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 15.07 usec
PL1 0.00 dB
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 65536
SF 400.1300152 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
2.929
6.614
6.620
6.624
6.645
6.650
6.662
6.664
6.681
6.686
7.225
7.245
7.261
7.264
7.364
7.384
7.402
7.525
7.528
7.856
7.874
7.878
6.24
3.07
2.05
2.07
1.00
3.01
ppm


151.35
142.64
138.12
135.76
133.95
130.20
128.55
126.84
124.83
114.03
111.17
108.89
104.77
40.23
200
180
160
140
120
100
80
60
40
20
0
ppm
Me2N
N
N
Ph
BRUKER
Current Data Parameters
NAME SU1-162-3C
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20101025
Time 12.28
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 128
DS 2
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 8192
DW 20.850 usec
DE 6.00 usec
TE 296.2 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 8.75 usec
PL1 -3.00 dB
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -1.00 dB
PL12 14.52 dB
PL13 18.00 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 65536
SF 100.6127825 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.40


7.856
7.834
7.817
7.785
7.764
7.690
7.504
7.488
7.469
7.449
7.431
7.415
7.393
7.147
6.972
3.594
3.579
3.564
3.548
2.828
2.812
2.796
1.963
10
9
8
7
6
5
4
3
2
1
0 ppm
4.23
1.05
3.20
1.04
0.97
2.09
2.06
3.20
N
N
NH
Me
O
BRUKER
Current Data Parameters
NAME SU1-45
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20100813
Time 8.35
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 57
DW 60.400 usec
DE 6.00 usec
TE 295.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 14.00 usec
PL1 0.00 dB
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 65536
SF 400.1300095 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00




SU1-45C




BRUKER

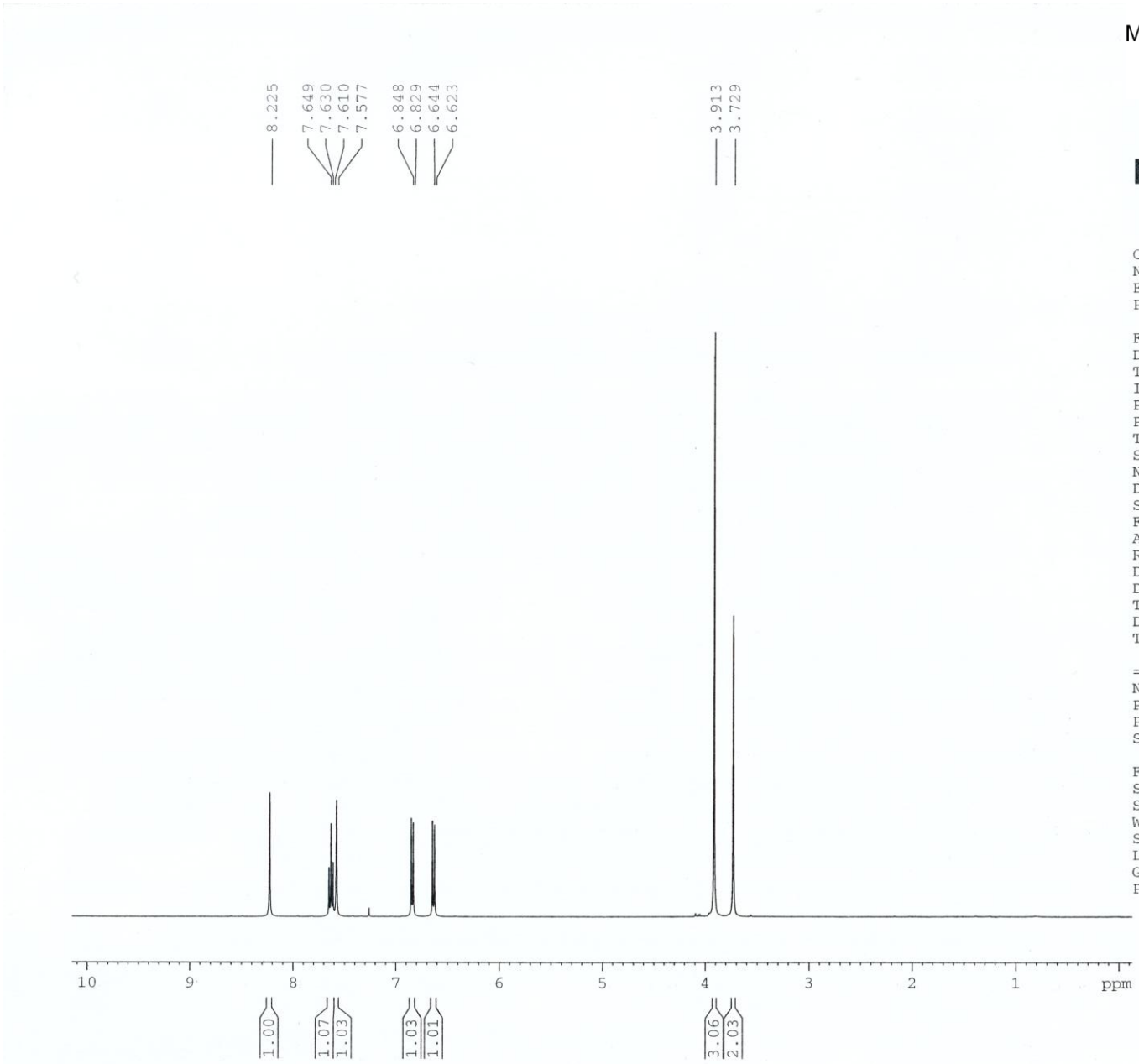
Current Data Parameters
NAME SU1-45C
EXPNO 1
PROCNO 1

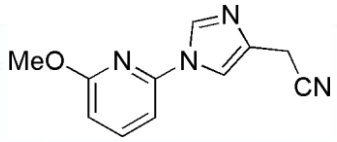
F2 - Acquisition Parameters
Date_ 20100813
Time 8.46
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 128
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 1625.5
DW 20.850 usec
DE 6.00 usec
TE 295.2 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 9.38 usec
PL1 0.00 dB
SFO1 100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 0.00 dB
PL12 16.10 dB
PL13 19.00 dB
SFO2 400.1316005 MHz

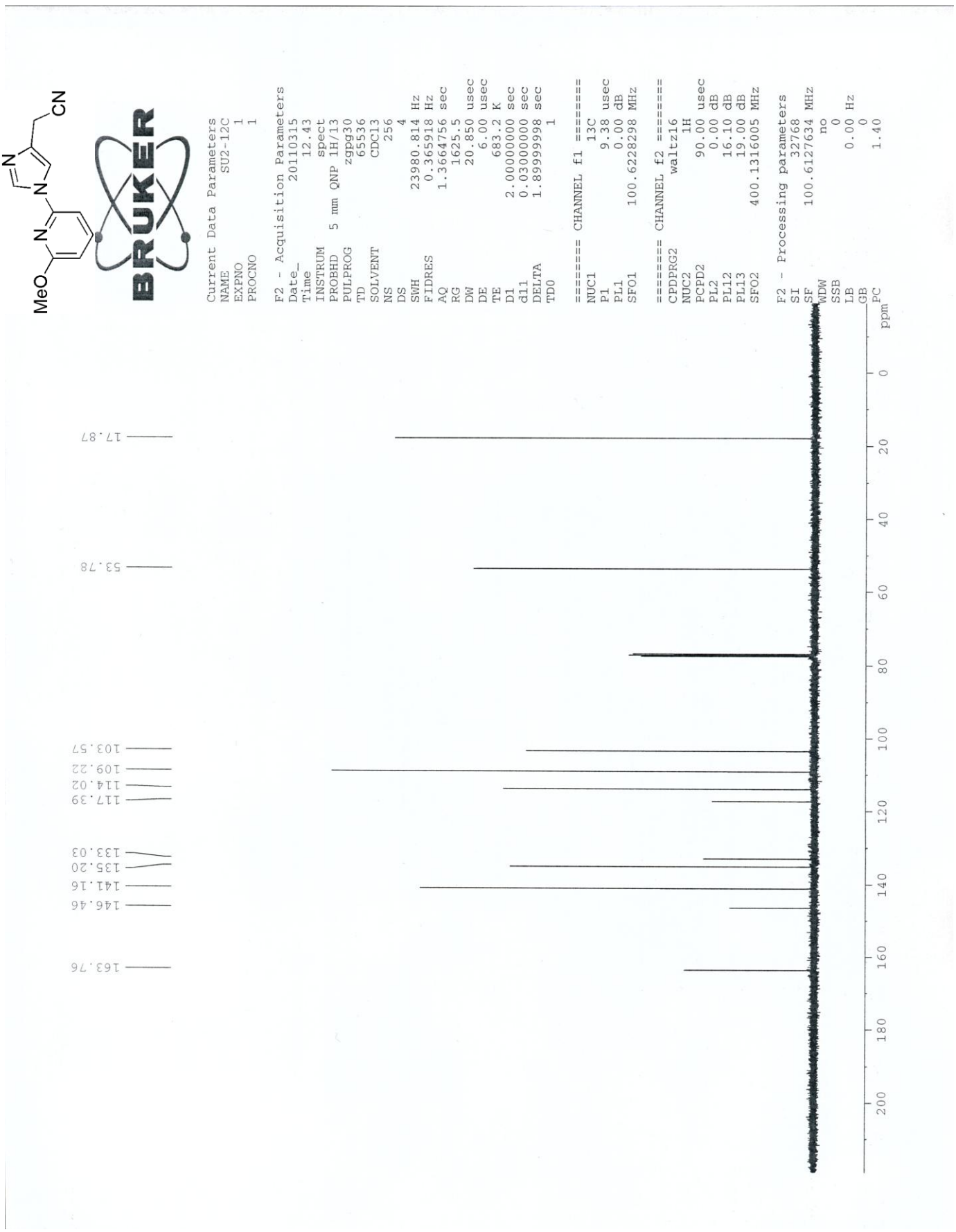
F2 - Processing parameters
SI 32768
SF 100.6127681 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.40

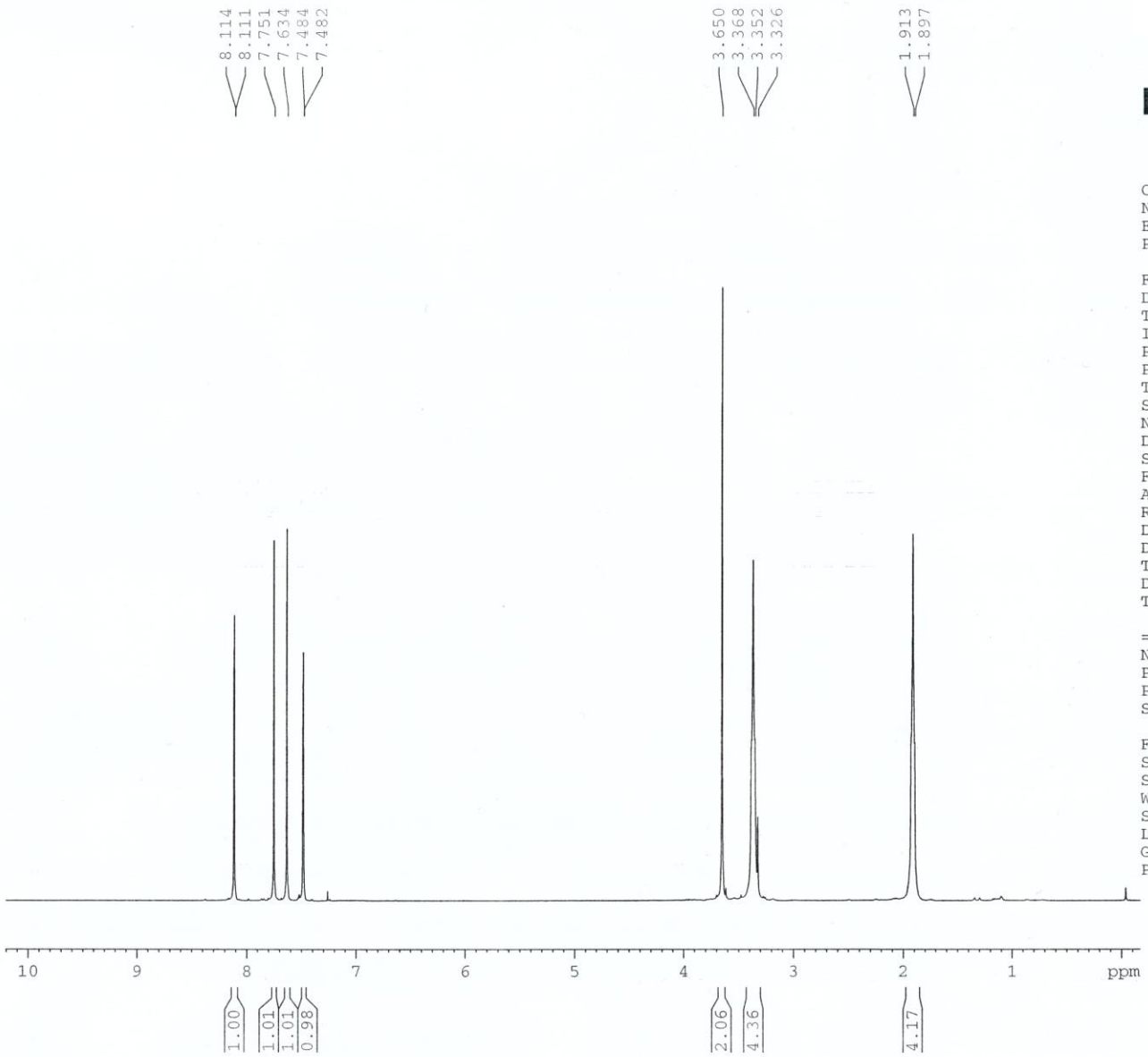


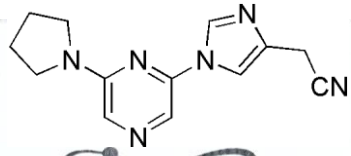




BRUKER

```
Current Data Parameters
NAME                  SU2-12
EXPNO                      1
PROCNO                     1

F2 - Acquisition Parameters
Date_              20110315
Time                  12.28
INSTRUM               spect
PROBHD    5 mm QNP 1H/13
PULPROG                zg30
TD                    65536
SOLVENT               CDCl3
NS                       16
DS                        2
SWH                8278.146 Hz
FIDRES             0.126314 Hz
AQ                 3.9584243 sec
RG                      114
DW                   60.400 usec
DE                    6.00 usec
TE                   683.2 K
D1                1.00000000 sec
TD0                        1

======== CHANNEL f1 ========
NUC1                      1H
P1                     14.00 usec
PL1                     0.00 dB
SFO1           400.1324710 MHz

F2 - Processing parameters
SI                        65536
SF             400.1300098 MHz
WDW                         no
SSB                          0
LB                      0.00 Hz
GB                           0
PC                        1.00
```

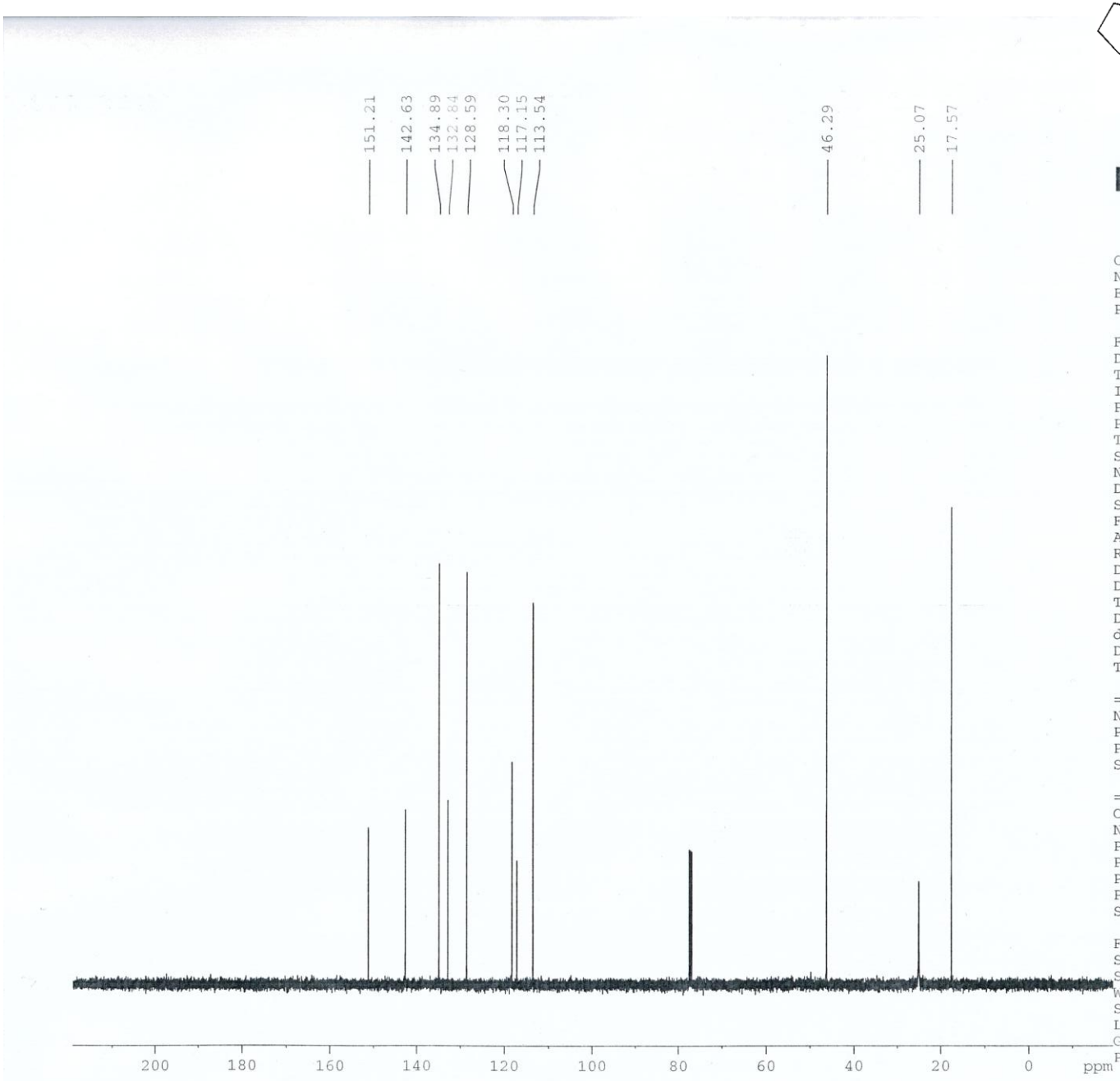

MeO
N
N
N
CN
BRUKER
Current Data Parameters
NAME SU2-12C
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20110315
Time 12.43
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 256
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 1625.5
DW 20.850 usec
DE 6.00 usec
TE 683.2 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.38 usec
PL1 0.00 dB
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 0.00 dB
PL12 16.10 dB
PL13 19.00 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127634 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.40
17.87
53.78
103.57
109.22
114.02
117.39
133.03
135.20
141.16
146.46
163.76
ppm
200
180
160
140
120
100
80
60
40
20
0

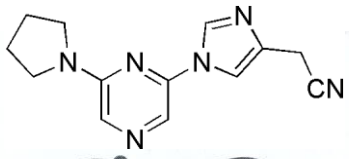



BRUKER

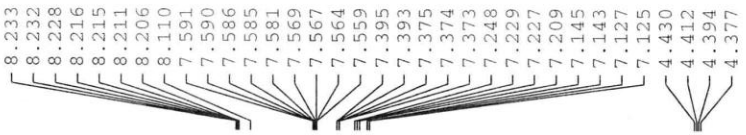
```
Current Data Parameters
NAME                  SU2-13-5
EXPNO                        1
PROCNO                       1

F2 - Acquisition Parameters
Date_                 20110318
Time                     17.07
INSTRUM                  spect
PROBHD    5 mm QNP 1H/13
PULPROG                  zg30
TD                      65536
SOLVENT                  CDCl3
NS                           8
DS                           2
SWH                 8278.146 Hz
FIDRES              0.126314 Hz
AQ                  3.9584243 sec
RG                          57
DW                     60.400 usec
DE                      6.00 usec
TE                     300.0 K
D1                  1.00000000 sec
TD0                          1

======== CHANNEL f1 ========
NUC1                        1H
P1                     14.00 usec
PL1                     0.00 dB
SFO1               400.1324710 MHz

F2 - Processing parameters
SI                      65536
SF                 400.1300098 MHz
WDW                        no
SSB                         0
LB                      0.00 Hz
GB                          0
PC                       1.00
```



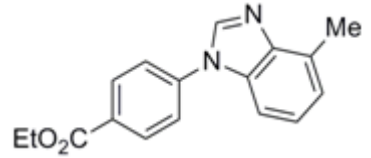

BRUKER

```
Current Data Parameters
NAME                  SU2-13-5C
EXPNO                         1
PROCNO                        1

F2 - Acquisition Parameters
Date_                  20110318
Time                      17.11
INSTRUM                   spect
PROBHD    5 mm QNP 1H/13
PULPROG                  zgpg30
TD                        65536
SOLVENT                   CDCl3
NS                           64
DS                            4
SWH                  23980.814 Hz
FIDRES                0.365918 Hz
AQ                    1.3664756 sec
RG                       1625.5
DW                       20.850 usec
DE                        6.00 usec
TE                       683.2 K
D1                   2.00000000 sec
d11                  0.03000000 sec
DELTA                1.89999998 sec
TD0                           1

======== CHANNEL f1 ========
NUC1                        13C
P1                         9.38 usec
PL1                        0.00 dB
SFO1               100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2                 waltz16
NUC2                        1H
PCPD2                     90.00 usec
PL2                        0.00 dB
PL12                      16.10 dB
PL13                      19.00 dB
SFO2               400.1316005 MHz

F2 - Processing parameters
SI                        32768
SF                 100.6127841 MHz
WDW                          no
SSB                           0
LB                         0.00 Hz
GB                            0
PC                         1.40
```



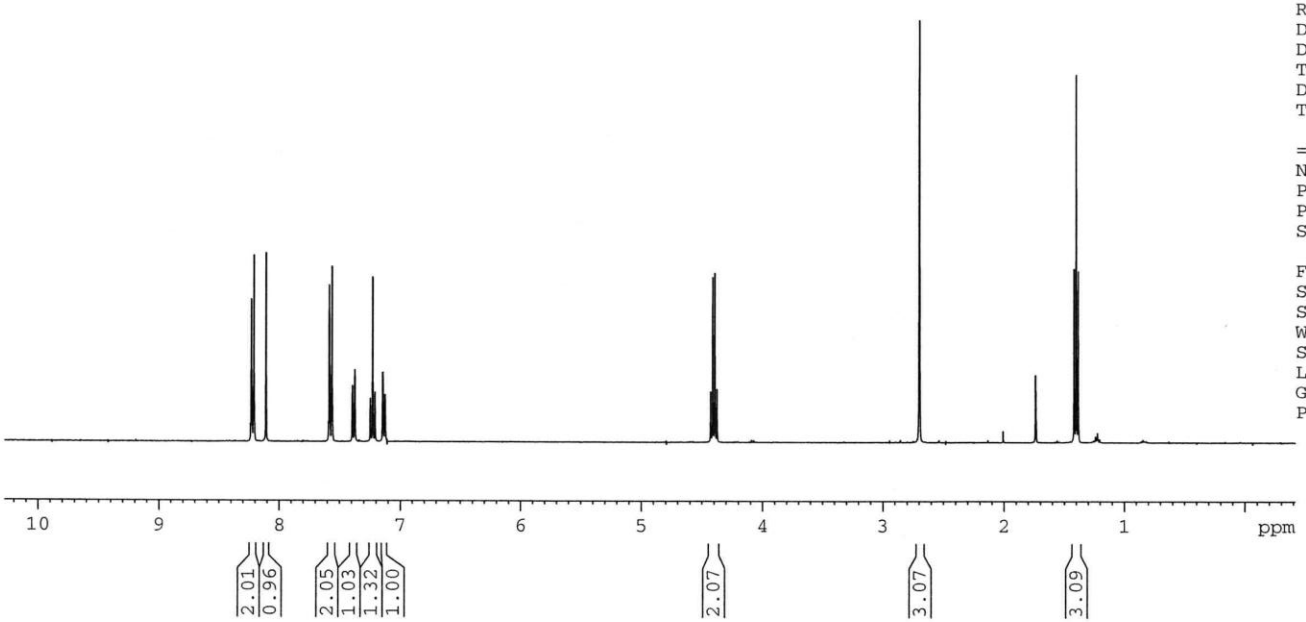


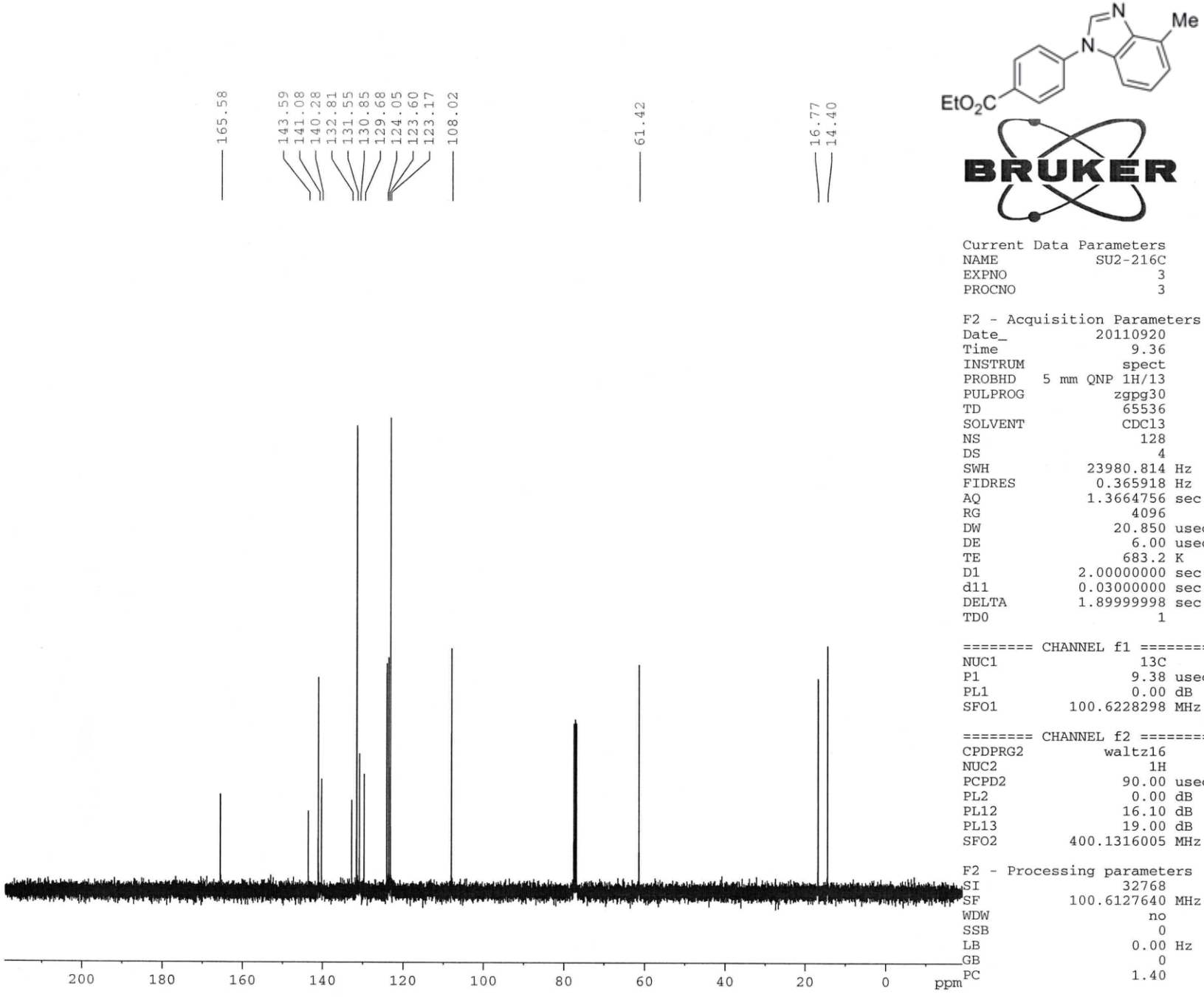




```
Current Data Parameters
NAME                  SU2-216
EXPNO                       1
PROCNO                      1

F2 - Acquisition Parameters
Date_              20110919
Time                  11.02
INSTRUM               spect
PROBHD    5 mm BBO BB-1H
PULPROG               zg30
TD                    65536
SOLVENT               CDCl3
NS                        8
DS                        2
SWH                8278.146 Hz
FIDRES             0.126314 Hz
AQ                 3.9584243 sec
RG                      128
DW                    60.400 usec
DE                     6.00 usec
TE                    297.2 K
D1                 1.00000000 sec
TD0                         1

======== CHANNEL f1 ========
NUC1                       1H
P1                      15.07 usec
PL1                      0.00 dB
SFO1            400.1324710 MHz

F2 - Processing parameters
SI                        65536
SF              400.1300212 MHz
WDW                         no
SSB                          0
LB                        0.00 Hz
GB                           0
PC                        1.00
```






BRUKER

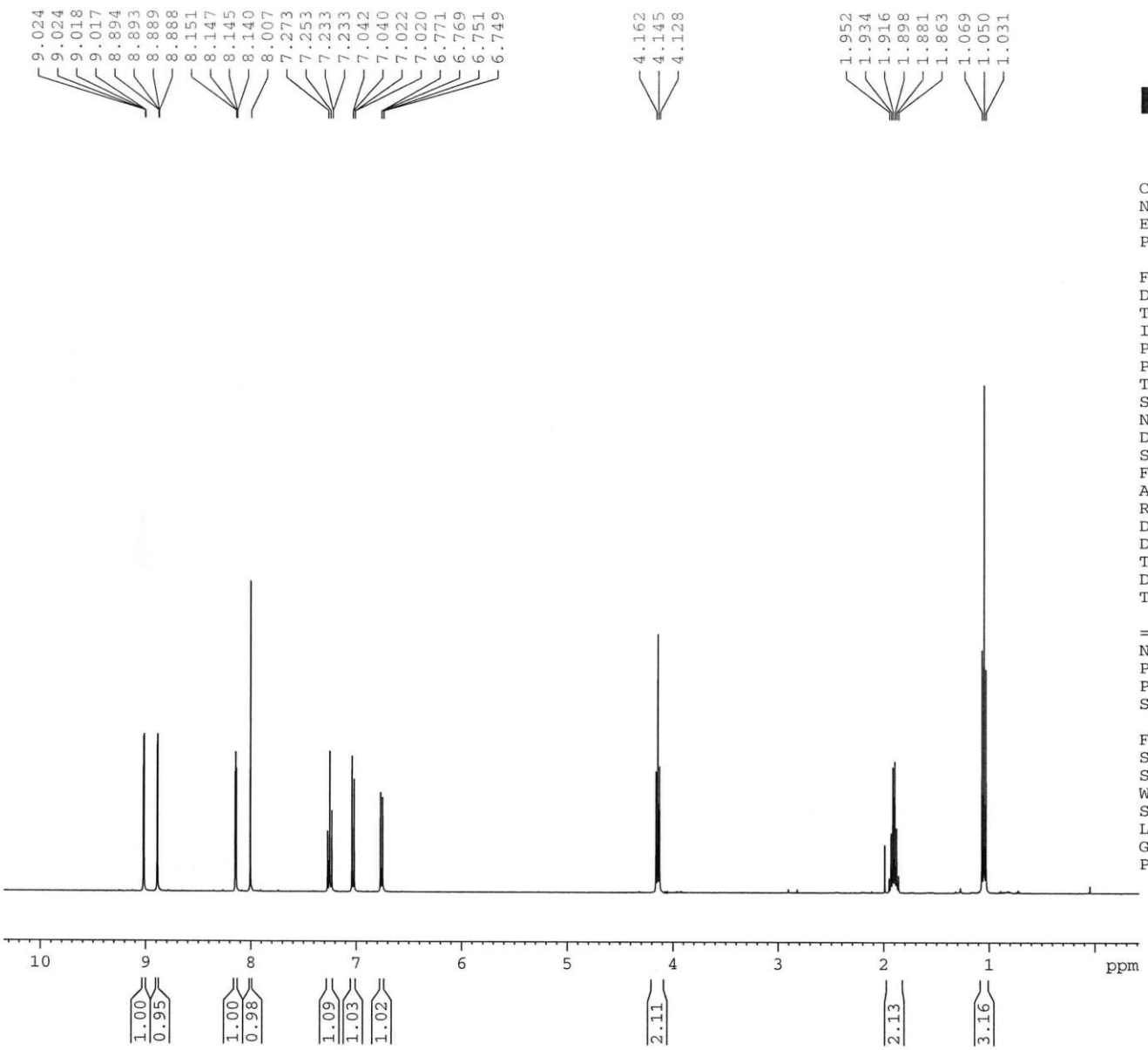
Current Data Parameters
NAME SU2-216C
EXPNO 3
PROCNO 3

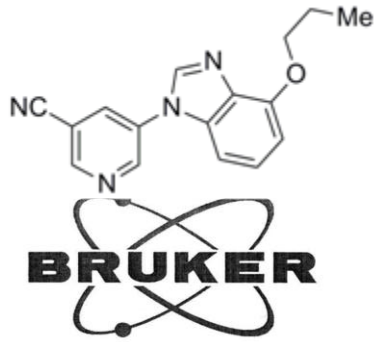
F2 - Acquisition Parameters
Date_ 20110920
Time 9.36
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 128
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 4096
DW 20.850 usec
DE 6.00 usec
TE 683.2 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 9.38 usec
PL1 0.00 dB
SFO1 100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 0.00 dB
PL12 16.10 dB
PL13 19.00 dB
SFO2 400.1316005 MHz

F2 - Processing parameters
SI 32768
SF 100.6127640 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.40




Current Data Parameters

| | |
|---|---|
| NAME | SU2-219 |
| EXPNO | 1 |
| PROCNO | 1 |

F2 - Acquisition Parameters

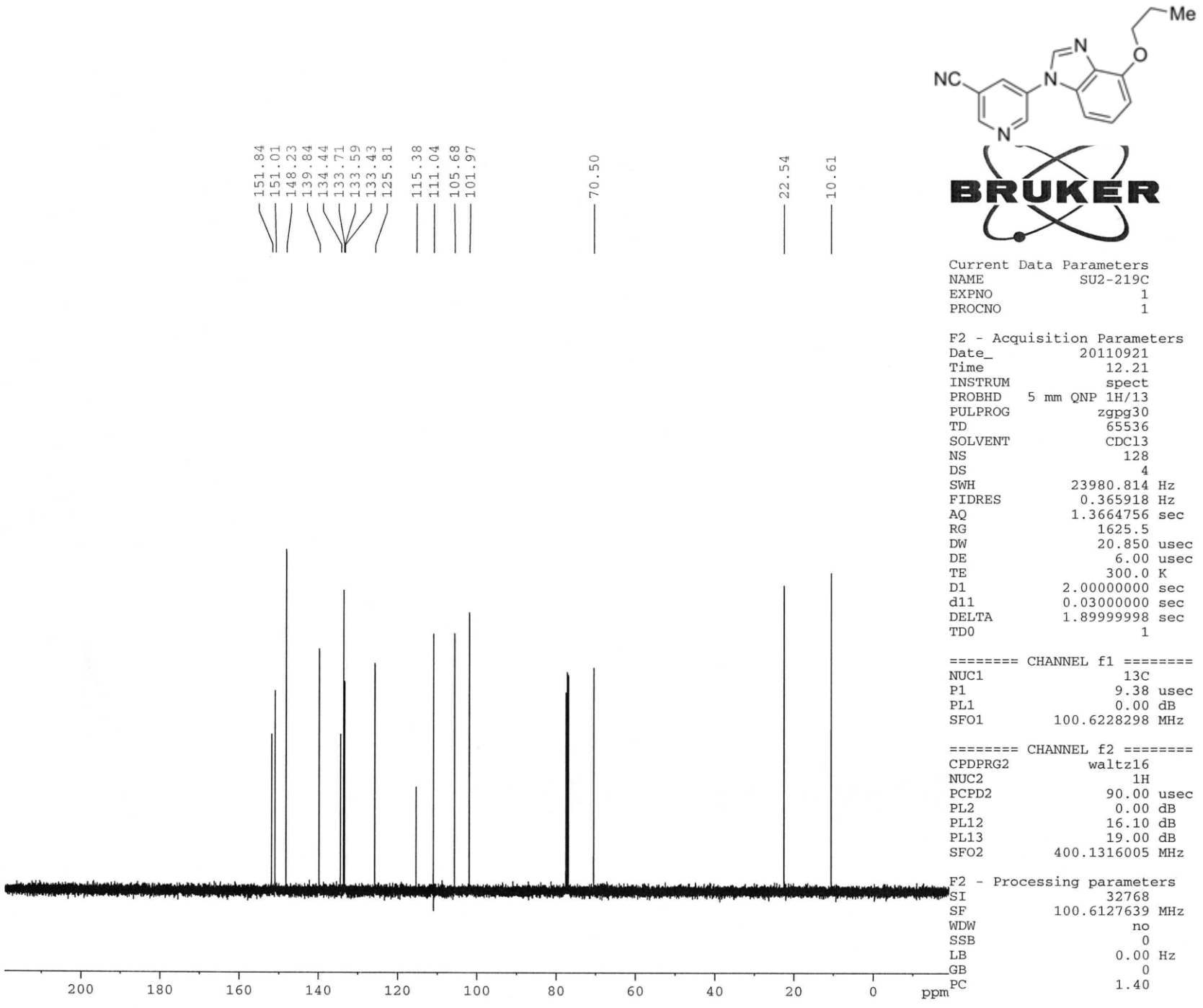
| | |
|---|---|
| Date_ | 20110921 |
| Time | 12.13 |
| INSTRUM | spect |
| PROBHD | 5 mm QNP 1H/13 |
| PULPROG | zg30 |
| TD | 65536 |
| SOLVENT | CDCl3 |
| NS | 8 |
| DS | 2 |
| SWH | 8278.146 Hz |
| FIDRES | 0.126314 Hz |
| AQ | 3.9584243 sec |
| RG | 1625.5 |
| DW | 60.400 usec |
| DE | 6.00 usec |
| TE | 683.2 K |
| D1 | 1.00000000 sec |
| TD0 | 1 |

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

| | |
|---|---|
| NUC1 | 1H |
| P1 | 14.00 usec |
| PL1 | 0.00 dB |
| SFO1 | 400.1324710 MHz |

F2 - Processing parameters

| | |
|---|---|
| SI | 65536 |
| SF | 400.1300099 MHz |
| WDW | no |
| SSB | 0 |
| LB | 0.00 Hz |
| GB | 0 |
| PC | 1.00 |



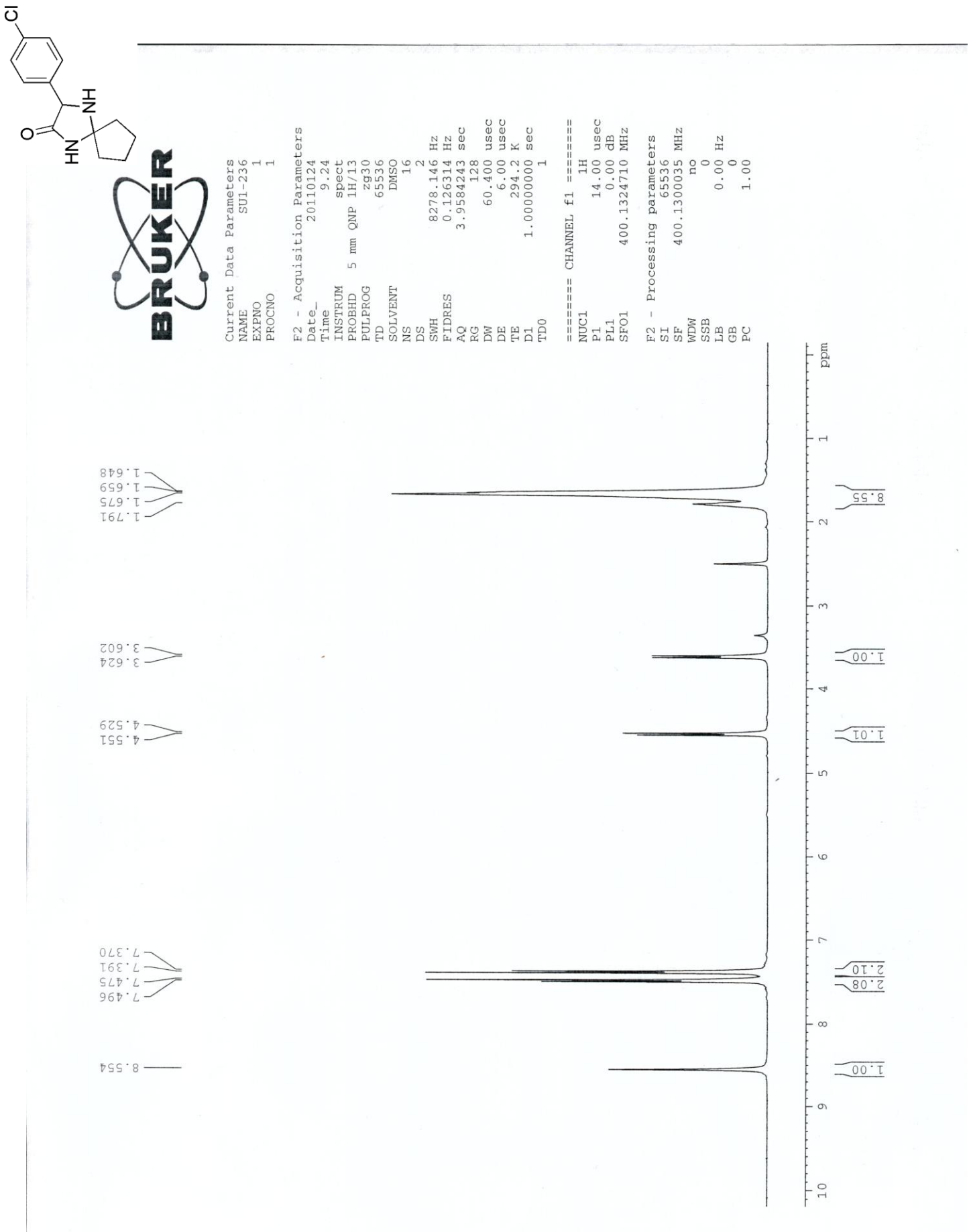

Current Data Parameters
NAME SU2-219C
EXPNO 1
PROCNO 1

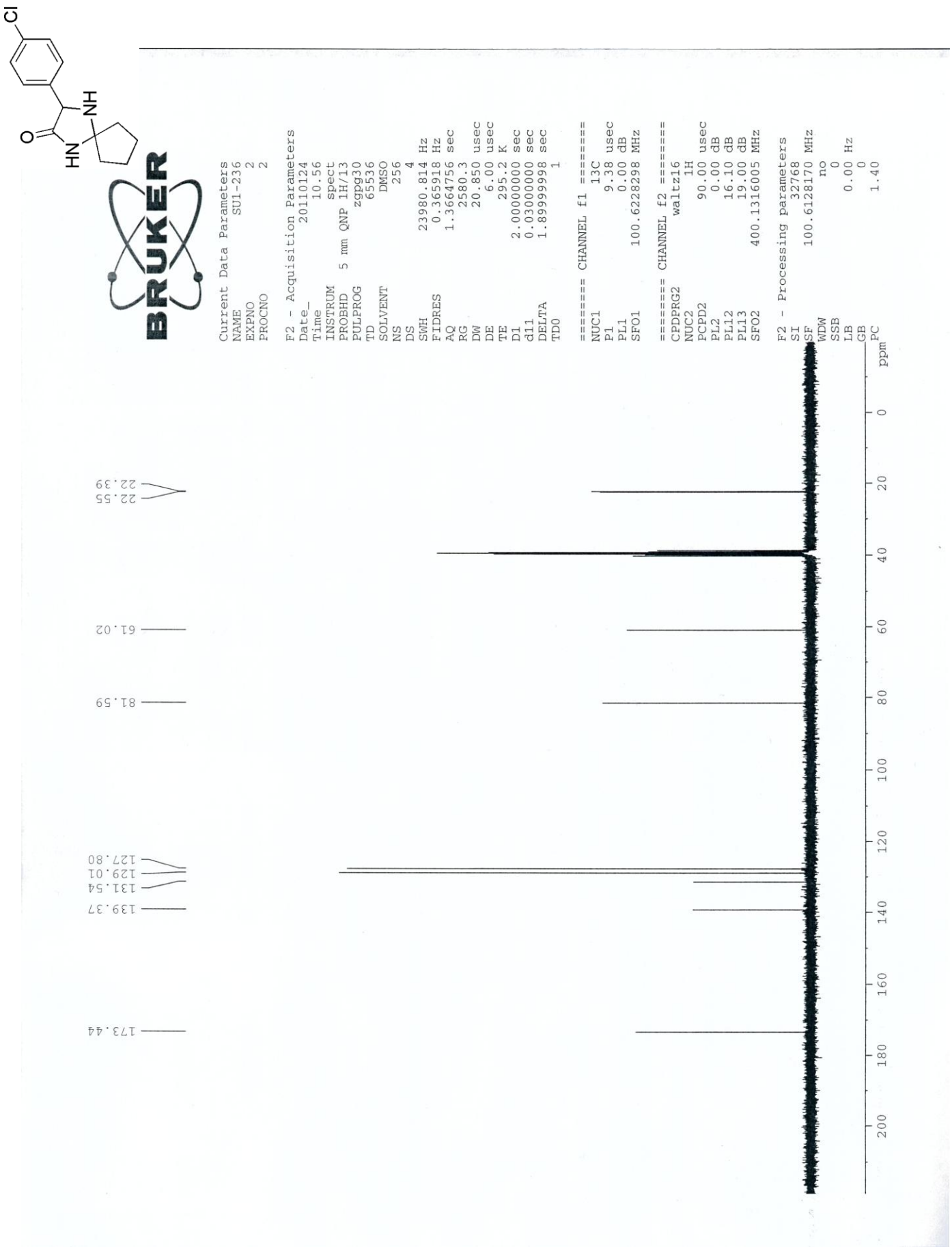
F2 - Acquisition Parameters
Date_ 20110921
Time 12.21
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 128
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 1625.5
DW 20.850 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1

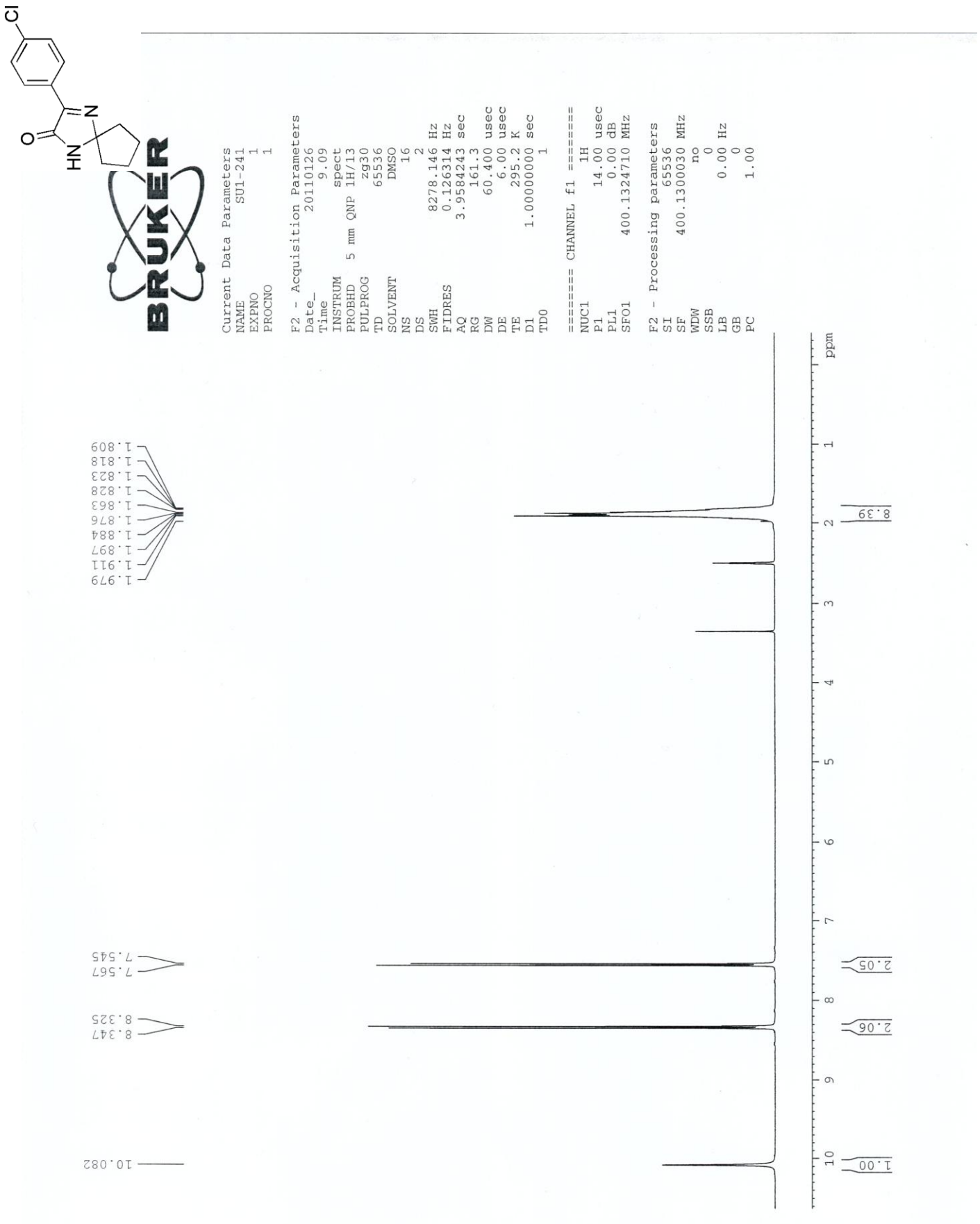
======== CHANNEL f1 ========
NUC1 13C
P1 9.38 usec
PL1 0.00 dB
SFO1 100.6228298 MHz

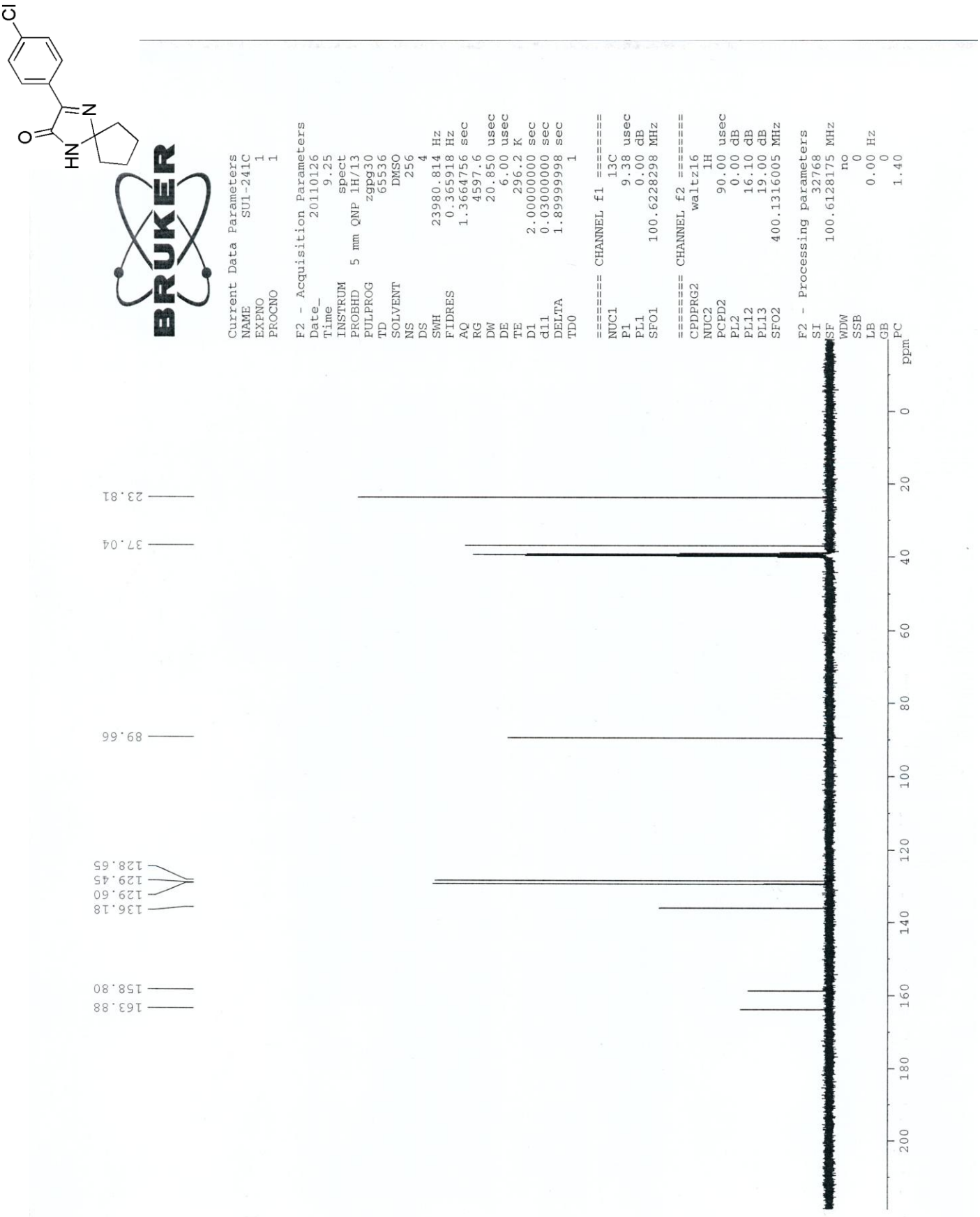
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 0.00 dB
PL12 16.10 dB
PL13 19.00 dB
SFO2 400.1316005 MHz

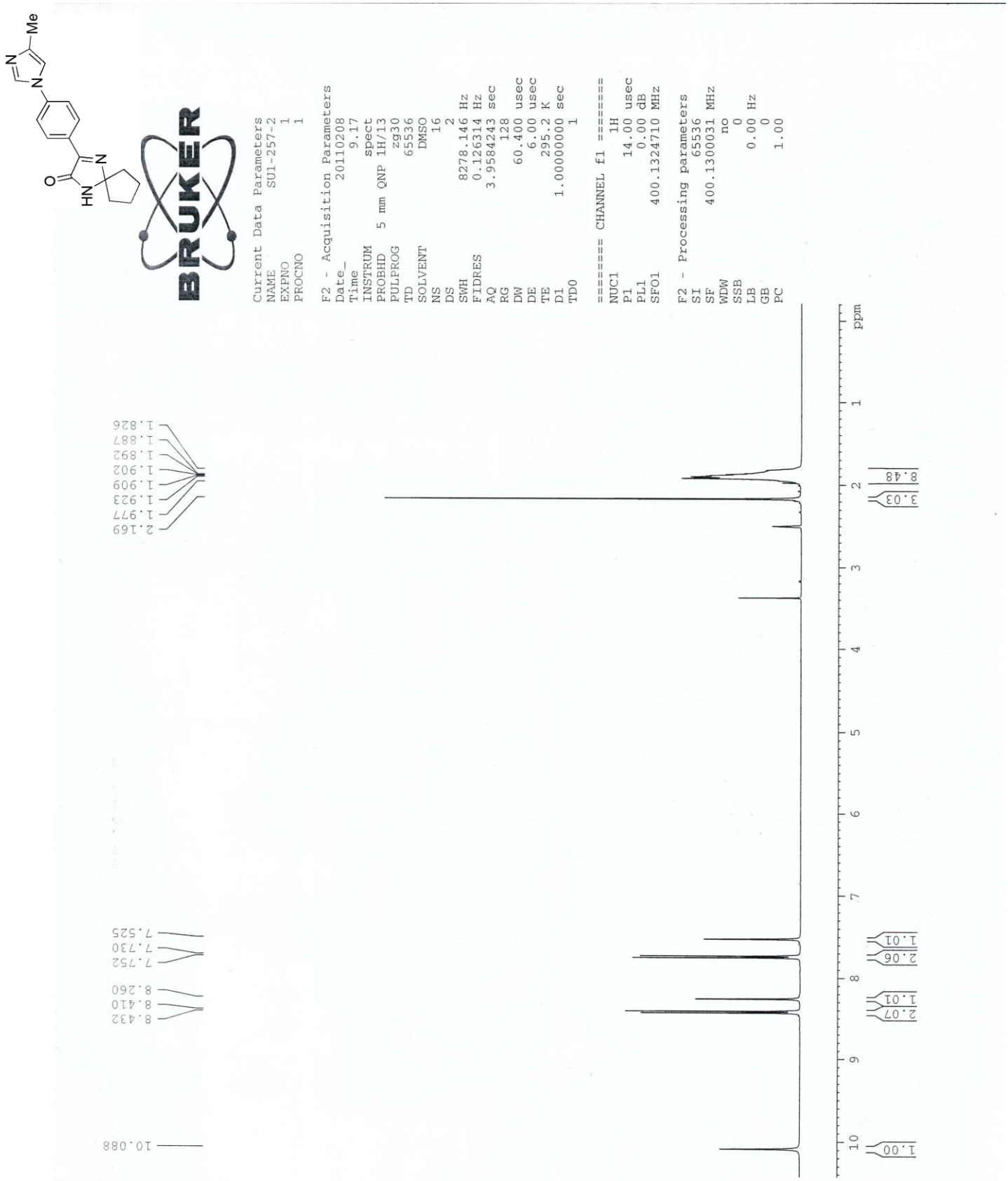
F2 - Processing parameters
SI 32768
SF 100.6127639 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.40

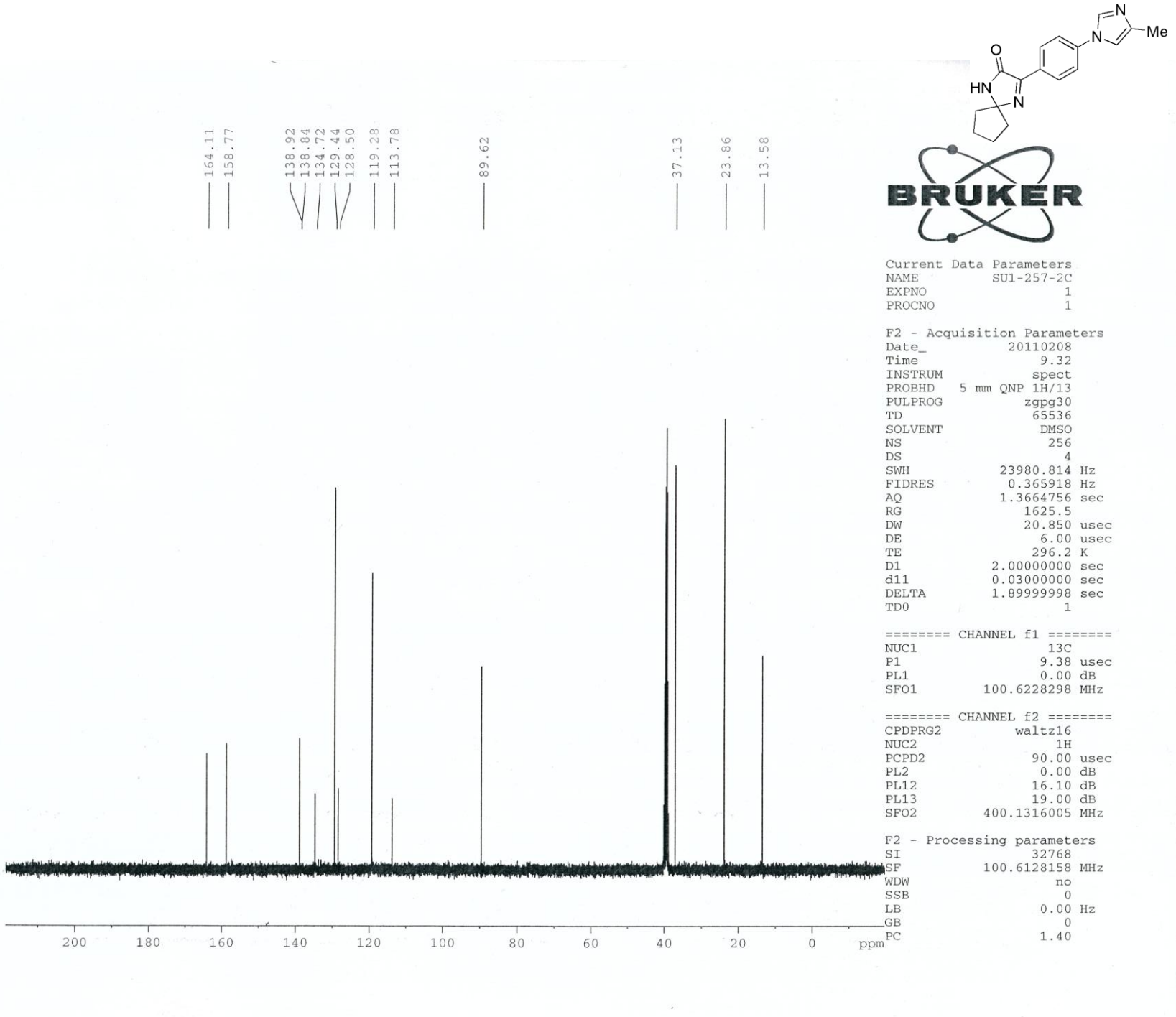

Cl
NH
HN
O
NH
BRUKER
Current Data Parameters
NAME SU1-236
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20110124
Time 9.24
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 16
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 128
DW 60.400 usec
DE 6.00 usec
TE 294.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 14.00 usec
PL1 0.00 dB
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 65536
SF 400.1300035 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
1.648
1.659
1.675
1.791
3.602
3.624
4.529
4.551
7.370
7.391
7.475
7.496
8.554
8.55
1.00
1.01
1.00
2.08
2.10
1.00
ppm

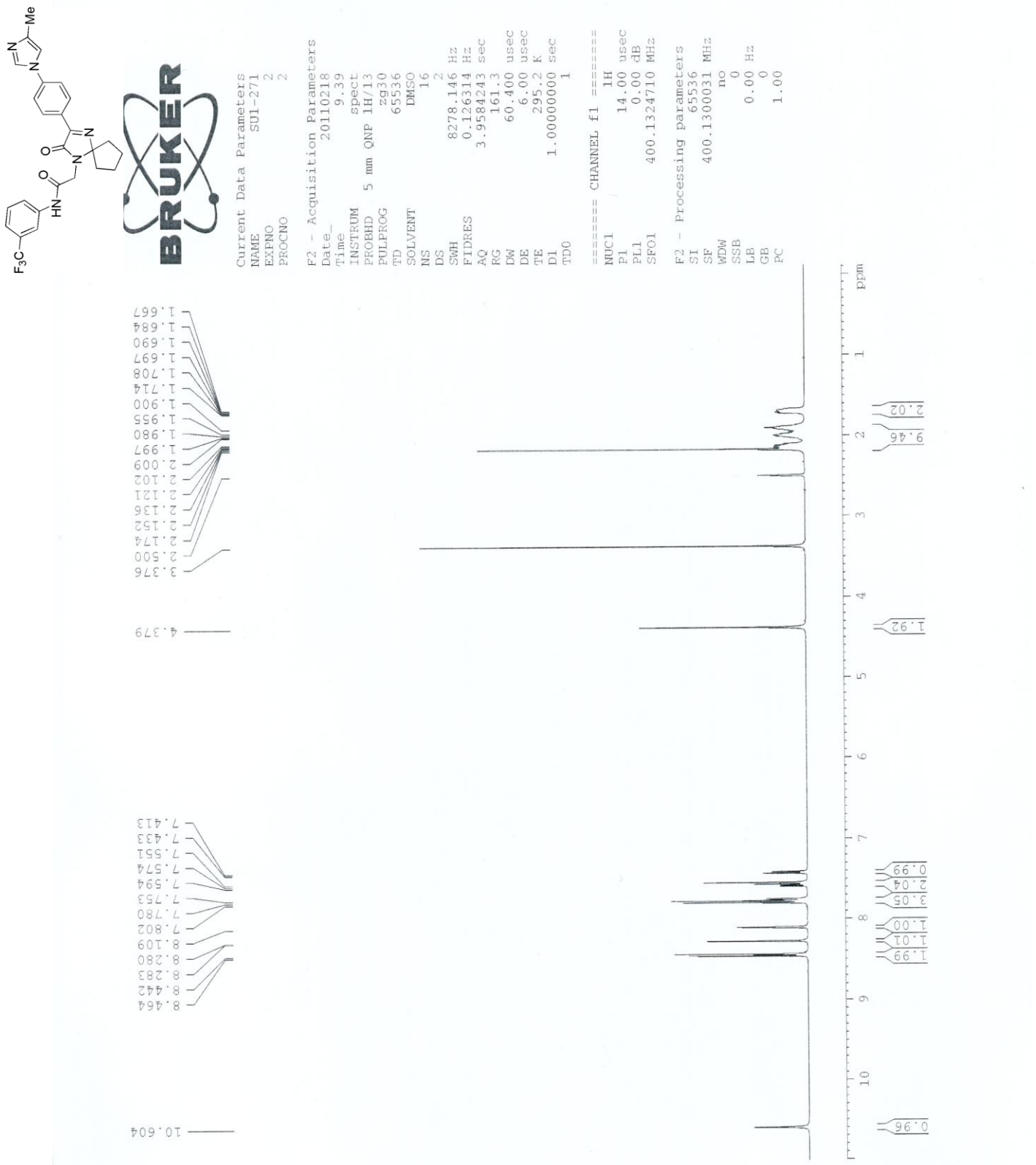

Cl
NH
O
HN
BRUKER
Current Data Parameters
NAME SU1-236
EXPNO 2
PROCNO 2
F2 - Acquisition Parameters
Date_ 20110124
Time 10.56
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 256
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 2580.3
DW 20.850 usec
DE 6.00 usec
TE 295.2 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.38 usec
PL1 0.00 dB
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 0.00 dB
PL12 16.10 dB
PL13 19.00 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6128170 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.40
22.39
22.55
61.02
81.59
127.80
129.01
131.54
139.37
173.44
ppm
200
180
160
140
120
100
80
60
40
20
0

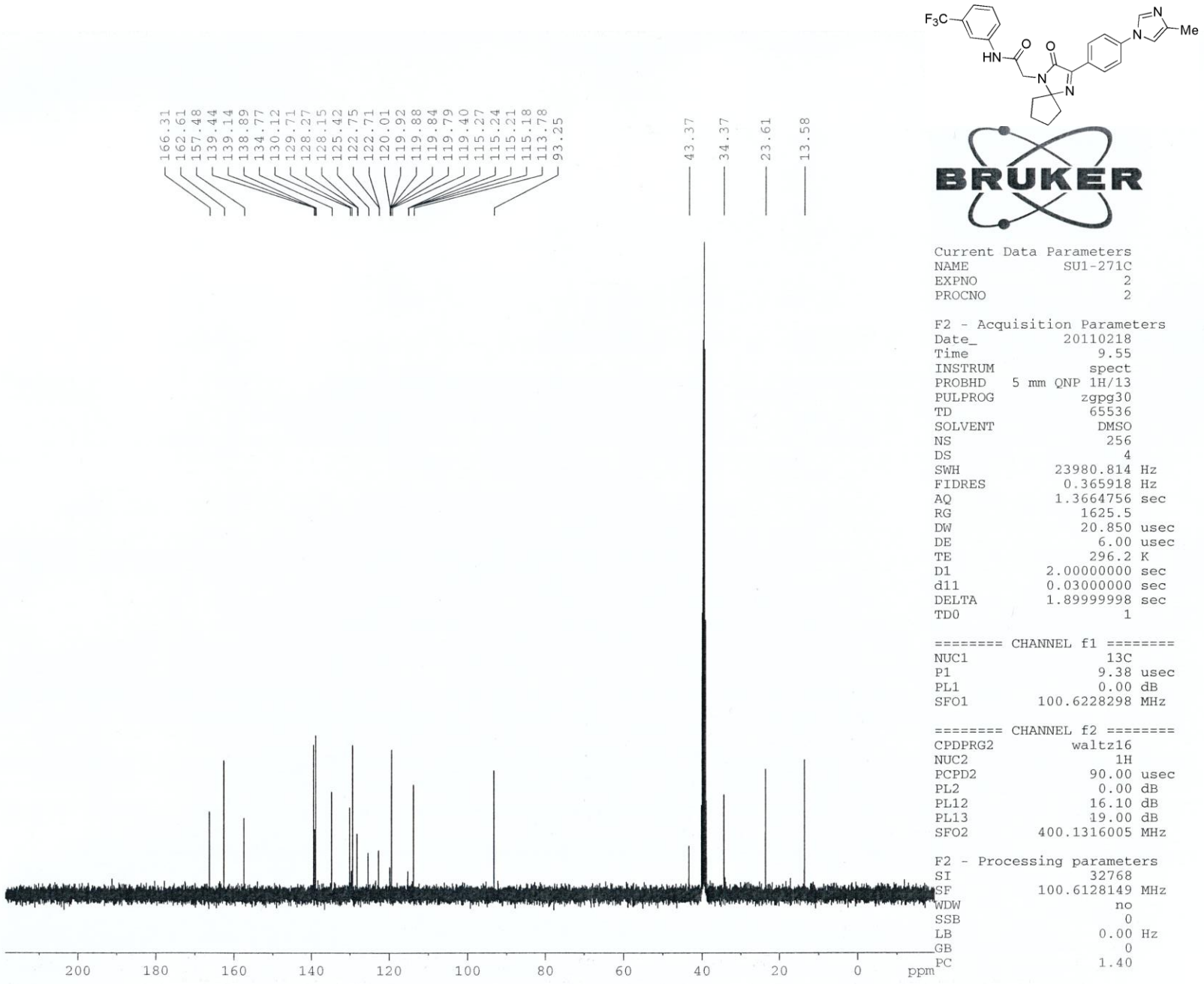

Cl
N
O
HN
BRUKER
Current Data Parameters
NAME SU1-241
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20110126
Time 9.09
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 16
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 161.3
DW 60.400 usec
DE 6.00 usec
TE 295.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 14.00 usec
PL1 0.00 dB
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 65536
SF 400.1300030 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
1.809
1.818
1.823
1.828
1.863
1.876
1.884
1.897
1.911
1.979
7.545
7.567
8.325
8.347
10.082
8.39
2.05
2.06
1.00
ppm


Cl
O
HN
N
BRUKER
Current Data Parameters
NAME SU1-241C
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20110126
Time 9.25
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 256
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 4597.6
DW 20.850 usec
DE 6.00 usec
TE 296.2 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.38 usec
PL1 0.00 dB
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 0.00 dB
PL12 16.10 dB
PL13 19.00 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6128175 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.40
23.81
37.04
89.66
128.65
129.45
129.60
136.18
158.80
163.88
ppm
0
20
40
60
80
100
120
140
160
180
200


Me
N
N
O
HN
N
BRUKER
Current Data Parameters
NAME SU1-257-2
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20110208
Time 9.17
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 16
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 128
DW 60.400 usec
DE 6.00 usec
TE 295.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 14.00 usec
PL1 0.00 dB
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 65536
SF 400.1300031 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
2.169
1.977
1.923
1.909
1.902
1.892
1.887
1.826
7.525
7.730
7.752
8.260
8.410
8.432
10.088
1.00
2.07
1.01
2.06
1.01
3.03
8.48
ppm


164.11
158.77
138.92
138.84
134.72
129.44
128.50
119.28
113.78
89.62
37.13
23.86
13.58
200
180
160
140
120
100
80
60
40
20
0
ppm
N
N
Me
O
HN
N
BRUKER
Current Data Parameters
NAME SU1-257-2C
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20110208
Time 9.32
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 256
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 1625.5
DW 20.850 usec
DE 6.00 usec
TE 296.2 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.38 usec
PL1 0.00 dB
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 0.00 dB
PL12 16.10 dB
PL13 19.00 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6128158 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.40


Me
N
N
O
N
O
HN
F3C
BRUKER
Current Data Parameters
NAME SU1-271
EXPNO 2
PROCNO 2
F2 - Acquisition Parameters
Date_ 20110218
Time 9.39
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 16
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 161.3
DW 60.400 usec
DE 6.00 usec
TE 295.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 14.00 usec
PL1 0.00 dB
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 65536
SF 400.1300031 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
10.604
8.464
8.442
8.283
8.280
8.109
7.802
7.780
7.753
7.594
7.574
7.551
7.433
7.413
4.379
3.376
2.500
2.174
2.152
2.136
2.121
2.102
2.009
1.997
1.980
1.955
1.900
1.714
1.708
1.697
1.690
1.684
1.667
0.96
1.99
1.01
1.00
3.05
2.04
0.99
1.92
9.46
2.02
ppm







BRUKER

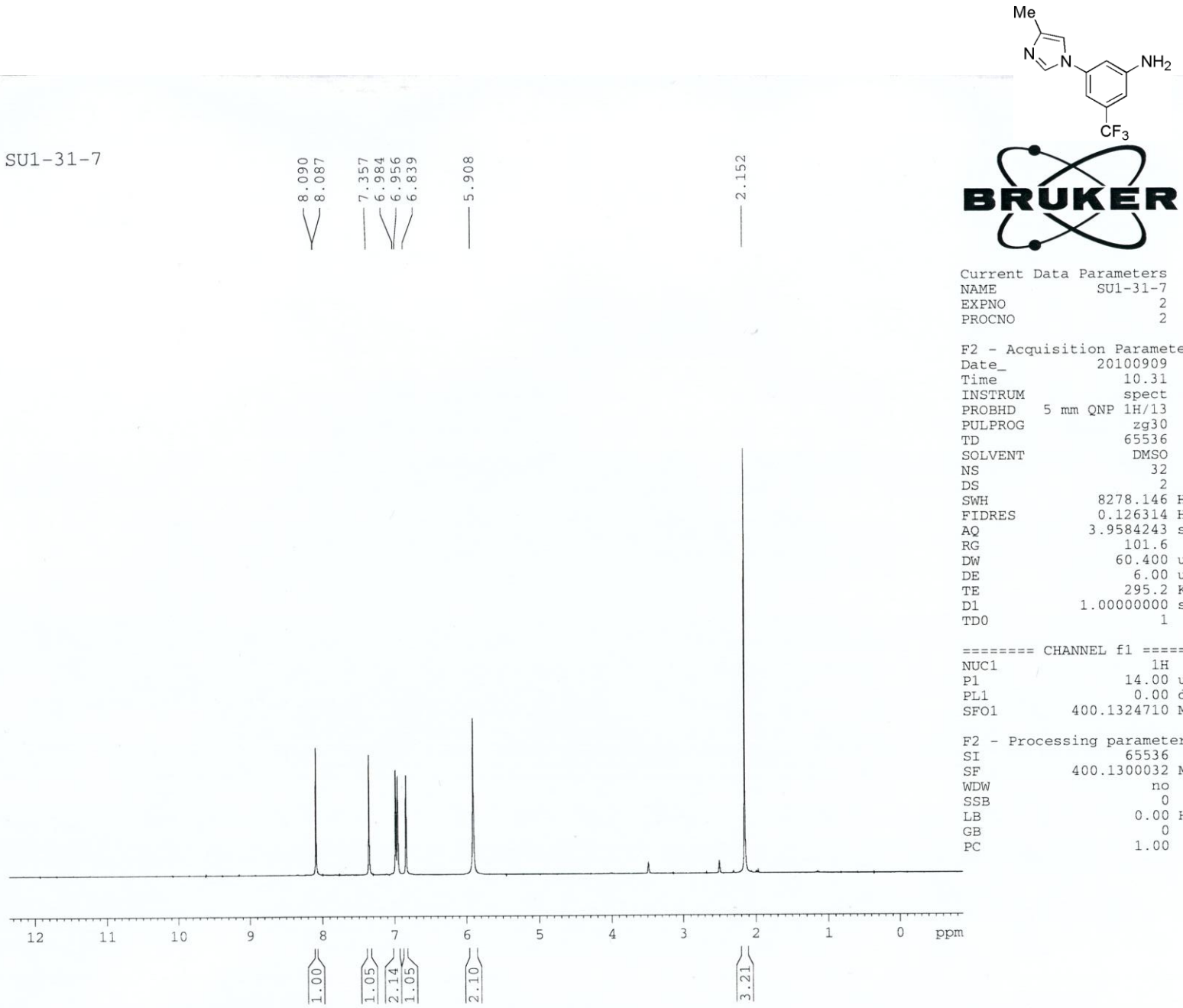
Current Data Parameters
NAME SU1-271C
EXPNO 2
PROCNO 2

F2 - Acquisition Parameters
Date_ 20110218
Time 9.55
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 256
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 1625.5
DW 20.850 usec
DE 6.00 usec
TE 296.2 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 9.38 usec
PL1 0.00 dB
SFO1 100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 0.00 dB
PL12 16.10 dB
PL13 19.00 dB
SFO2 400.1316005 MHz

F2 - Processing parameters
SI 32768
SF 100.6128149 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.40



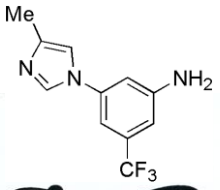

BRUKER

Current Data Parameters
NAME SU1-31-7
EXPNO 2
PROCNO 2

F2 - Acquisition Parameters
Date_ 20100909
Time 10.31
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 32
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 101.6
DW 60.400 usec
DE 6.00 usec
TE 295.2 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 14.00 usec
PL1 0.00 dB
SFO1 400.1324710 MHz

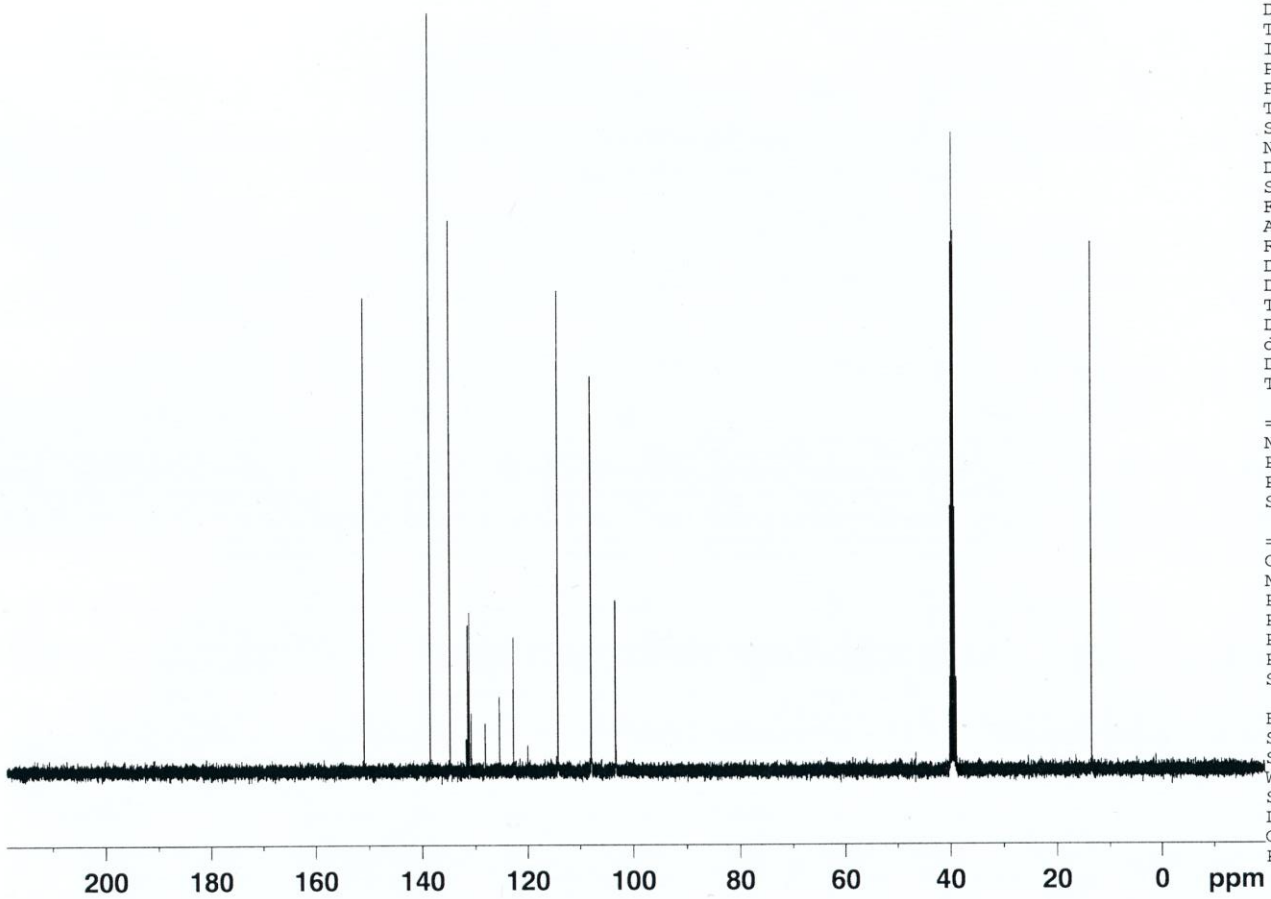
F2 - Processing parameters
SI 65536
SF 400.1300032 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00




SU1-31-7C

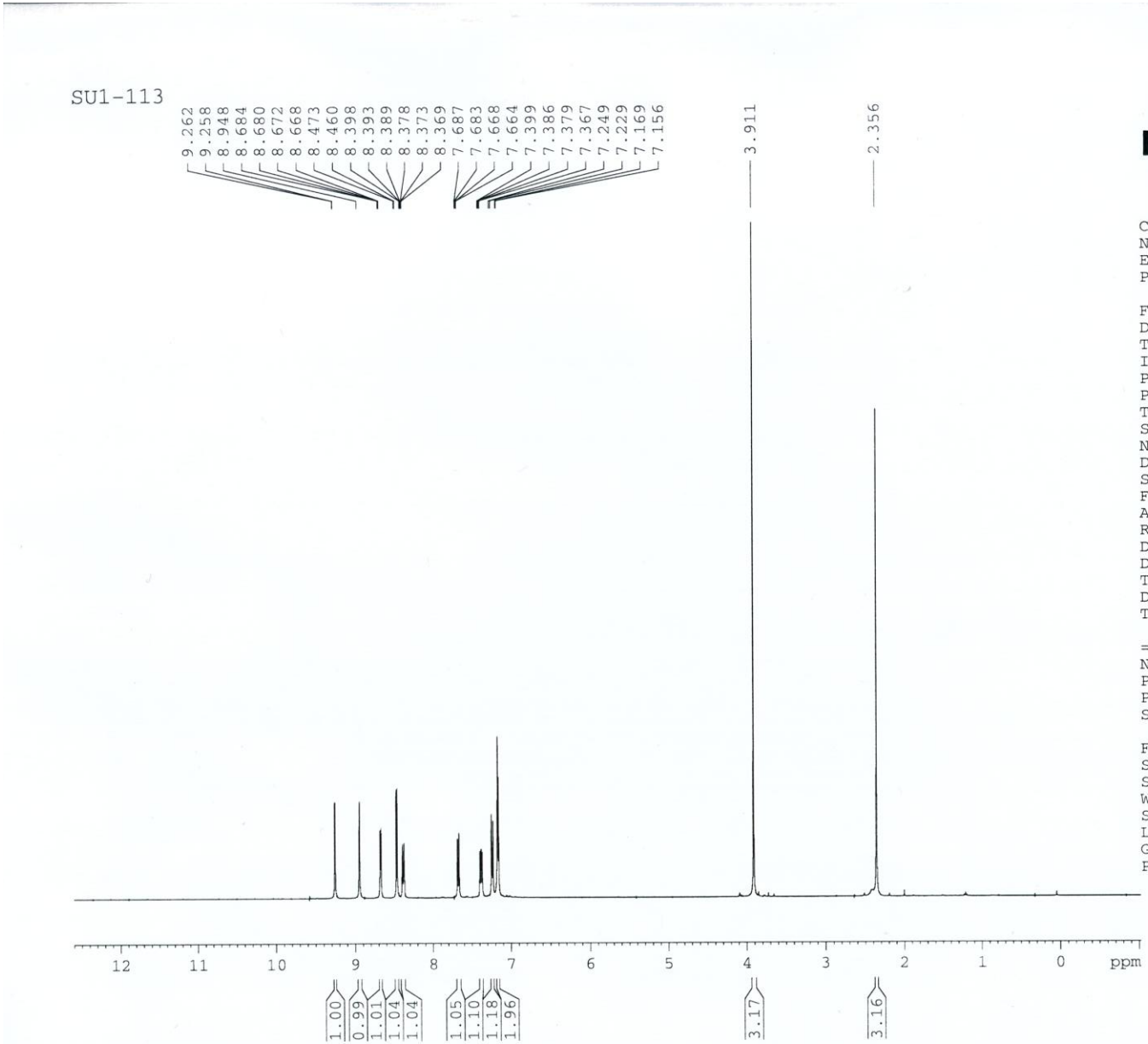
150.90
138.51
134.78
131.77
131.45
131.14
130.82
128.13
125.42
122.71
120.00
114.21
107.91
103.34
103.31
103.27
103.23

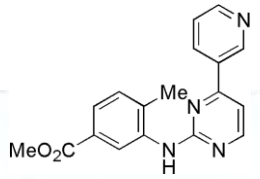
13.52




BRUKER

```
Current Data Parameters
NAME                  SU1-31-7C
EXPNO                         2
PROCNO                        2

F2 - Acquisition Parameters
Date_                  20100909
Time                      10.47
INSTRUM                   spect
PROBHD    5 mm QNP 1H/13
PULPROG                 zgpg30
TD                        65536
SOLVENT                   DMSO
NS                          256
DS                            4
SWH                 23980.814 Hz
FIDRES                0.365918 Hz
AQ                  1.3664756 sec
RG                         6502
DW                       20.850 usec
DE                        6.00 usec
TE                       296.2 K
D1                  2.00000000 sec
d11                 0.03000000 sec
DELTA               1.89999998 sec
TD0                           1

======== CHANNEL f1 ========
NUC1                        13C
P1                        9.38 usec
PL1                       0.00 dB
SFO1               100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2                waltz16
NUC2                        1H
PCPD2                    90.00 usec
PL2                       0.00 dB
PL12                     16.10 dB
PL13                     19.00 dB
SFO2               400.1316005 MHz

F2 - Processing parameters
SI                        32768
SF                 100.6128090 MHz
WDW                          no
SSB                           0
LB                        0.00 Hz
GB                            0
PC                         1.40
```



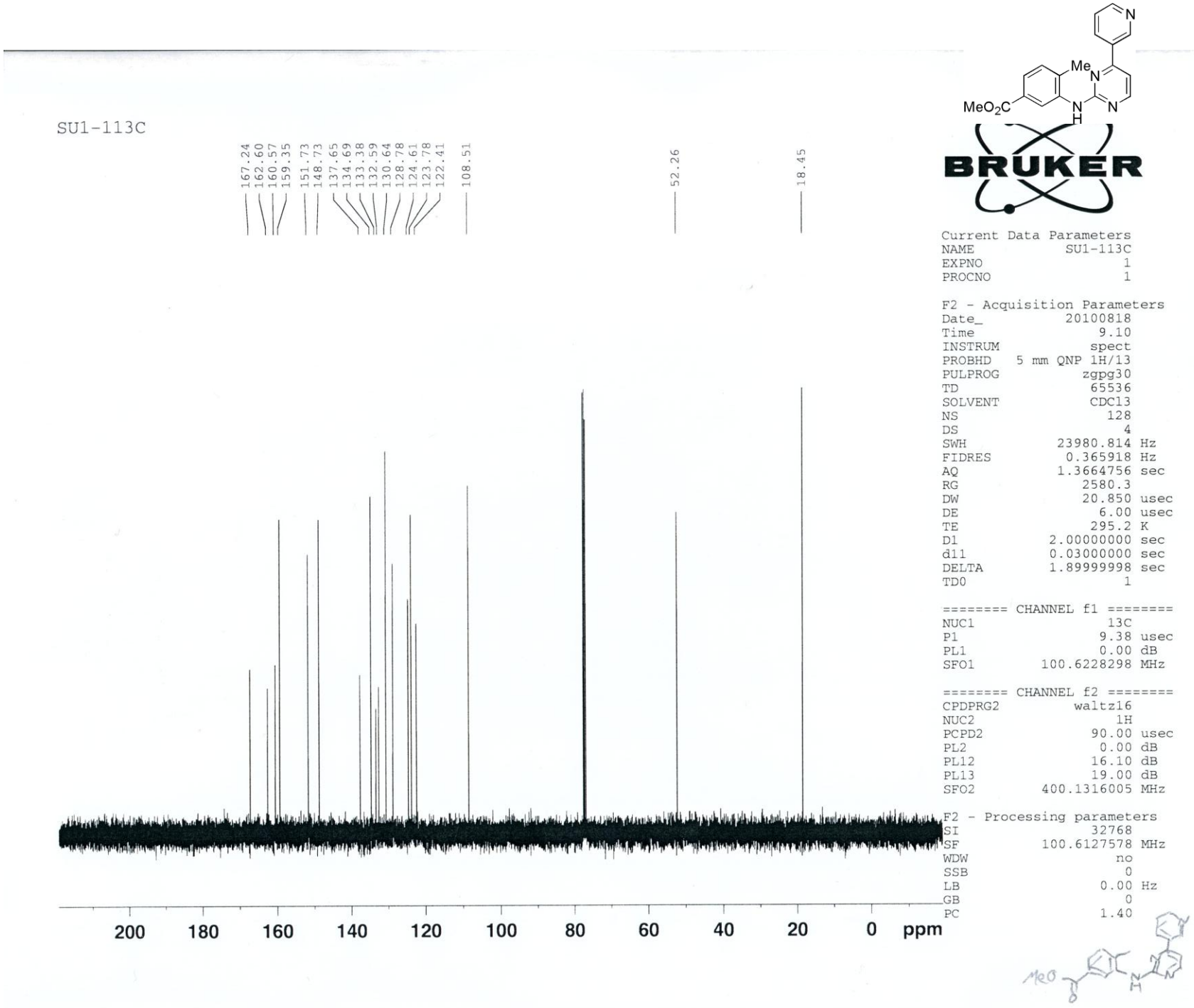

BRUKER

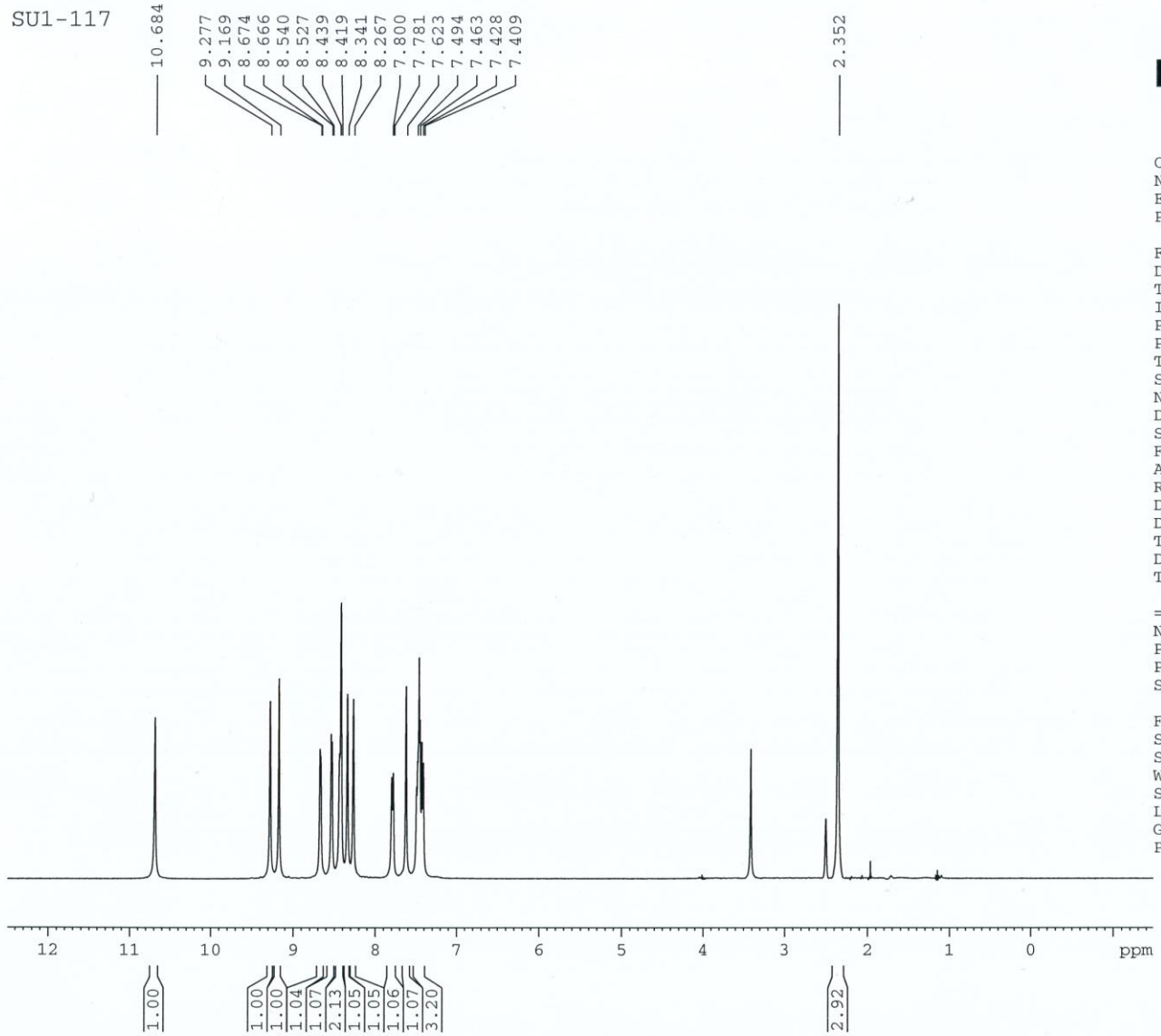
Current Data Parameters
NAME SU1-113
EXPNO 3
PROCNO 3

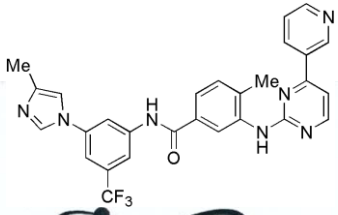
F2 - Acquisition Parameters
Date_ 20100818
Time 9.37
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 32
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 143.7
DW 60.400 usec
DE 6.00 usec
TE 295.2 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 14.00 usec
PL1 0.00 dB
SFO1 400.1324710 MHz

F2 - Processing parameters
SI 65536
SF 400.1300179 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

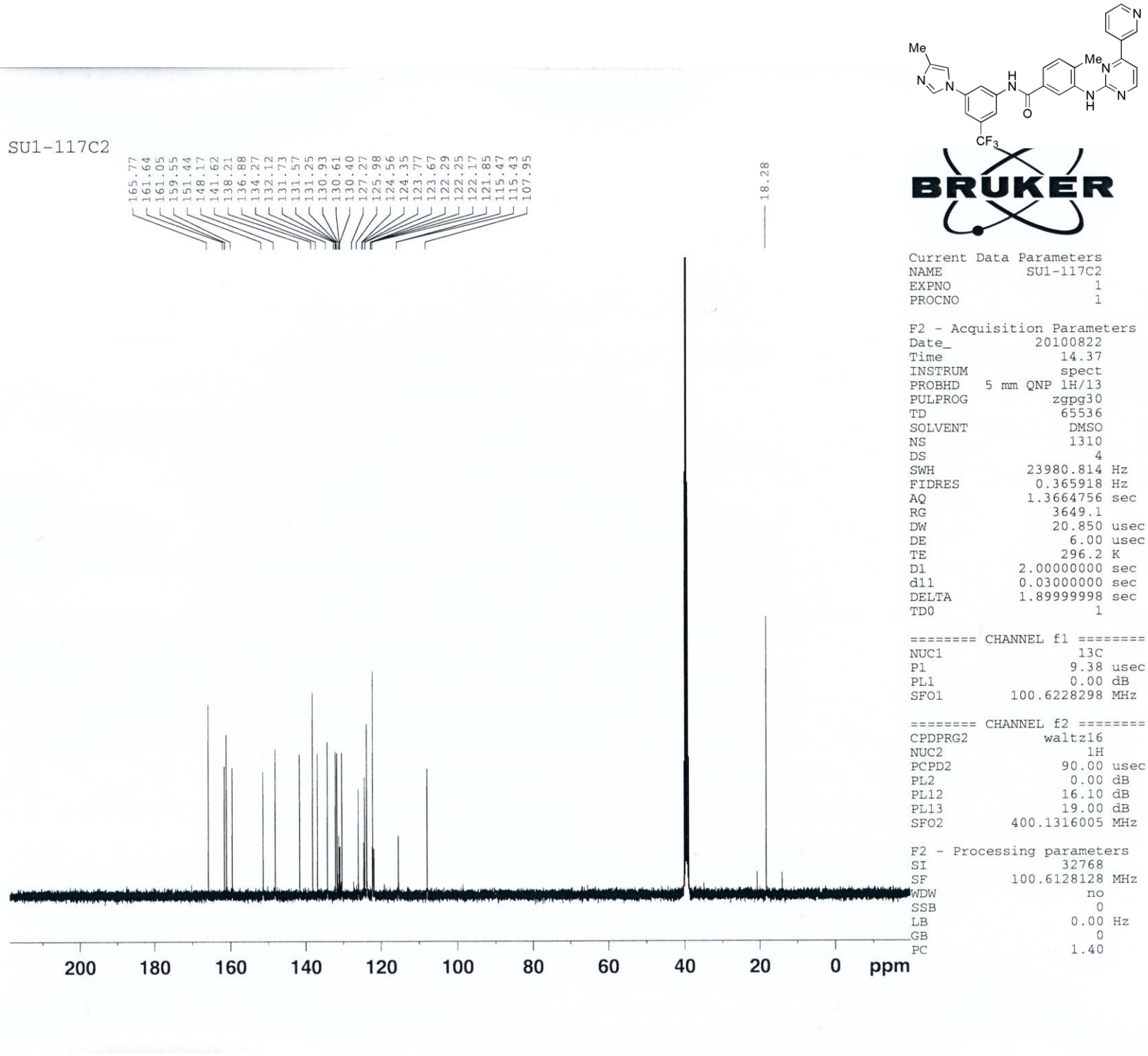

SU1-113C
167.24
162.60
160.57
159.35
151.73
148.73
137.65
134.69
133.38
132.59
130.64
128.78
124.61
123.78
122.41
108.51
52.26
18.45
200
180
160
140
120
100
80
60
40
20
0
ppm
Me
N
MeO2C
N
H
N
N
BRUKER
Current Data Parameters
NAME SU1-113C
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20100818
Time 9.10
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDC13
NS 128
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 2580.3
DW 20.850 usec
DE 6.00 usec
TE 295.2 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.38 usec
PL1 0.00 dB
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 0.00 dB
PL12 16.10 dB
PL13 19.00 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127578 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.40

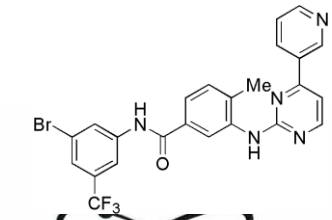


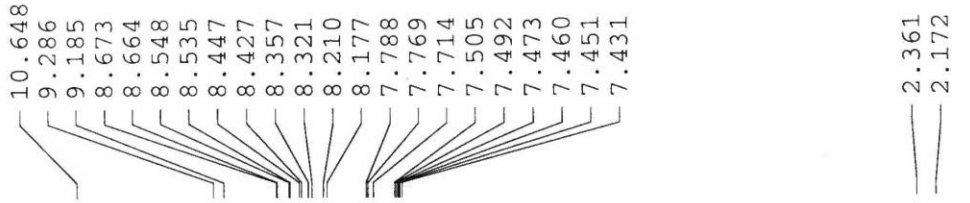

BRUKER

```
Current Data Parameters
NAME                  SU1-117
EXPNO                       1
PROCNO                      1

F2 - Acquisition Parameters
Date_                20100822
Time                    13.20
INSTRUM                 spect
PROBHD    5 mm QNP 1H/13
PULPROG                  zg30
TD                      65536
SOLVENT                  DMSO
NS                         16
DS                          2
SWH                  8278.146 Hz
FIDRES               0.126314 Hz
AQ                 3.9584243 sec
RG                      143.7
DW                     60.400 usec
DE                      6.00 usec
TE                     295.2 K
D1                1.00000000 sec
TD0                         1

======== CHANNEL f1 ========
NUC1                       1H
P1                     14.00 usec
PL1                     0.00 dB
SFO1             400.1324710 MHz

F2 - Processing parameters
SI                      65536
SF               400.1300037 MHz
WDW                        no
SSB                         0
LB                      0.00 Hz
GB                          0
PC                      1.00
```


SU1-117C2
165.77
161.64
161.05
159.55
151.44
148.17
141.62
138.21
136.88
134.27
132.12
131.73
131.57
131.25
130.93
130.61
130.40
127.27
125.98
124.56
124.35
123.77
123.67
122.29
122.25
122.17
121.85
115.47
115.43
107.95
18.28
200
180
160
140
120
100
80
60
40
20
0
ppm
Me
Me
N
N
N
N
H
N
O
N
H
N
CF3
BRUKER
Current Data Parameters
NAME SU1-117C2
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20100822
Time 14.37
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 1310
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 3649.1
DW 20.850 usec
DE 6.00 usec
TE 296.2 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.38 usec
PL1 0.00 dB
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 0.00 dB
PL12 16.10 dB
PL13 19.00 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6128128 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.40

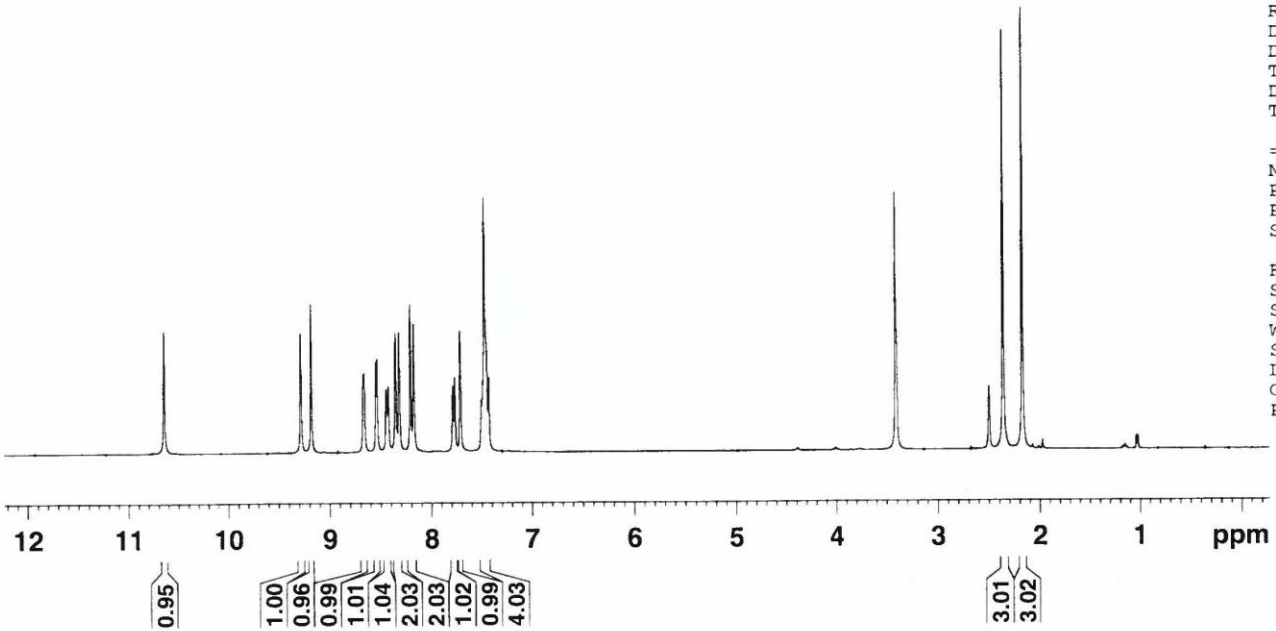


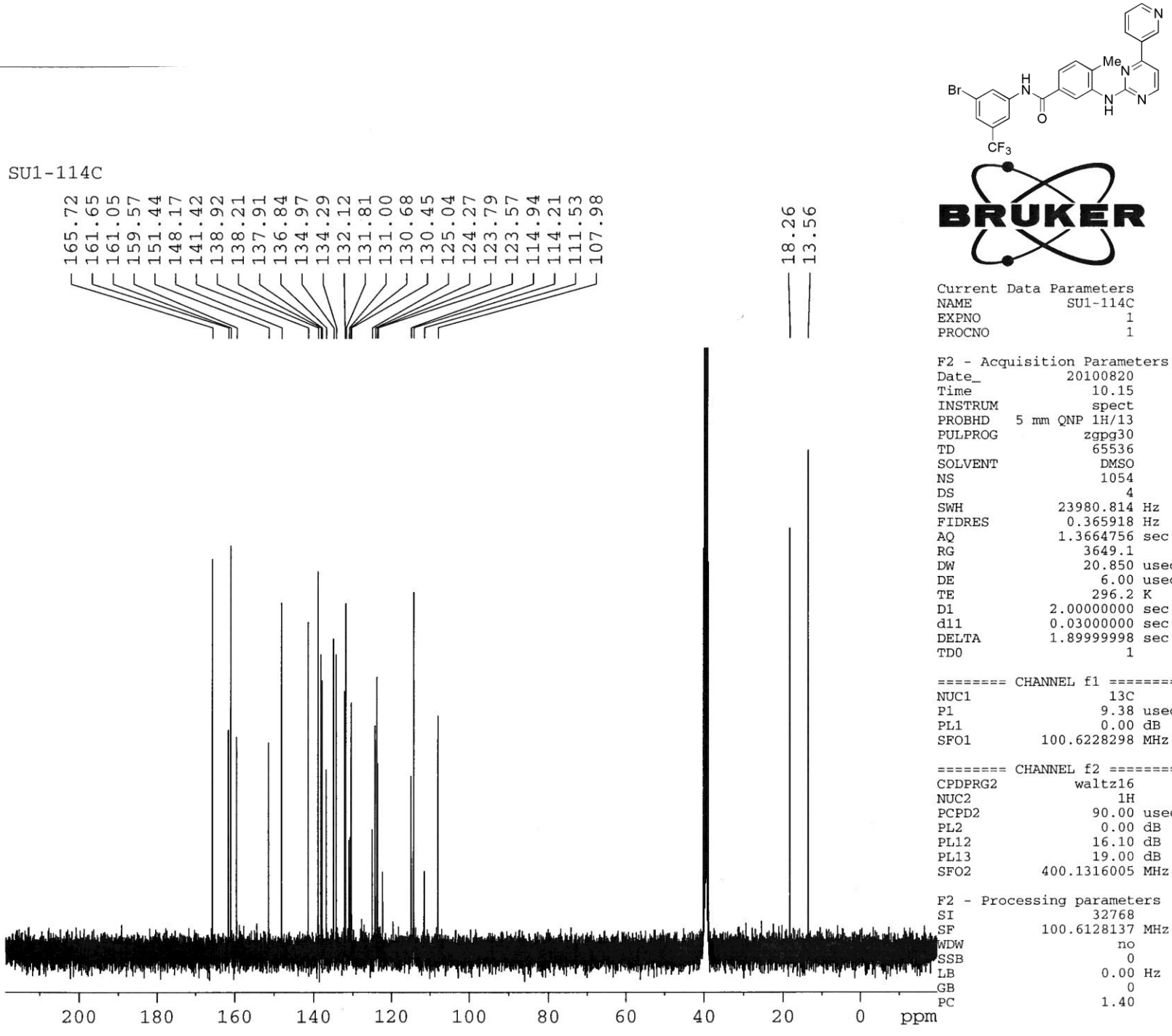

SU1-114




BRUKER

```
Current Data Parameters
NAME                  SU1-114
EXPNO                       1
PROCNO                      1

F2 - Acquisition Parameters
Date_              20100820
Time                   8.45
INSTRUM               spect
PROBHD   5 mm QNP 1H/13
PULPROG                zg30
TD                    65536
SOLVENT                DMSO
NS                       64
DS                        2
SWH                8278.146 Hz
FIDRES            0.126314 Hz
AQ                3.9584243 sec
RG                    143.7
DW                   60.400 usec
DE                    6.00 usec
TE                   295.2 K
D1               1.00000000 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                     1H
P1                   14.00 usec
PL1                   0.00 dB
SFO1          400.1324710 MHz

F2 - Processing parameters
SI                    65536
SF            400.1300036 MHz
WDW                      no
SSB                       0
LB                    0.00 Hz
GB                        0
PC                    1.00
```




SU1-114C
165.72
161.65
161.05
159.57
151.44
148.17
141.42
138.92
138.21
137.91
136.84
134.97
134.29
132.12
131.81
131.00
130.68
130.45
125.04
124.27
123.79
123.57
114.94
114.21
111.53
107.98
18.26
13.56
200
180
160
140
120
100
80
60
40
20
0
ppm
Br
H
N
O
Me
N
N
H
N
N
CF3
BRUKER
Current Data Parameters
NAME SU1-114C
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20100820
Time 10.15
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 1054
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 3649.1
DW 20.850 usec
DE 6.00 usec
TE 296.2 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.38 usec
PL1 0.00 dB
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 0.00 dB
PL12 16.10 dB
PL13 19.00 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6128137 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.40