Electronic Supplementary Material (ESI) for RSC Advances.

Supporting Information

Pd-catalyzed dehydrogenative cross-coupling of pyridine-*N*-oxides with uracils

Ebrahim Kianmehr,[a]* *Masoud Rezaeefard,*[a] *Mehran Rezazadeh Khalkhali*[a] *and Khalid Mohammed Khan*[b]

[a]*School of Chemistry, College of Science, University of Tehran, Tehran, Iran. E-mail: kianmehr@khayam.ut.ac.ir*

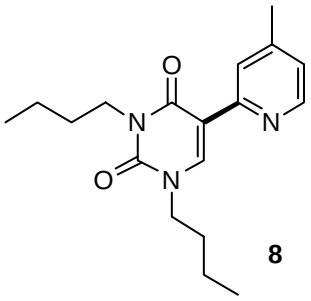
[b]*H. E. J. Research Institute of Chemistry, International Center for Chemical and Biological Sciences, University of Karachi, Karachi-75270, Pakistan*

General remarks:

Glassware was flame-dried. Solvents, Palladium acetate (98%), Ag_2CO_3, pyridine derivatives were purchased from Merck. All other commercially available reagents were utilized as received without purification. Thin layer chromatography (TLC) analysis was performed using Silicycle precoated TLC plates (silica gel 60 F_{254}). The products were purified by preparative column chromatography on silica gel (0.063-0.200 mm; Merck). IR Spectra: Shimadzu FT-IR-4300 spectrometer; in cm^{-1}. ^1H and ^{13}C-NMR Spectra: were recorded on Bruker DRX -500, 400, 300 and 250-Advance instrument; in $CDCl_3$ at 500.1, 400.1, 300.1, 250.1, 125.7, 100, 75.4 and 62.5 MHz, resp; δ in ppm, *J* in Hz. EI-MS (70 eV): HP 5973 GC-MS instrument; in m/z. Melting points: Electrothermal 9200 apparatus.

Reduction of 2-(1,3-dibutyl-2,4-dioxo-1,2,3,4-tetrahydropyrimidin-5-yl)-4-methylpyridine 1-oxide (7b) to 3-butyl-5-(4-methylpyridin-2-yl)-1-pentylpyrimidine-2,4(1*H*,3*H*)-dione (8)

To a stirred mixture of 7b (331 mg, 1.00 mmol) in toluene (10.0 mL) was added PCl_3 (175 μL, 2.00 mmol) dropwise. The reaction mixture was stirred for 120 min at room temperature. Saturated solution of $NaHCO_3$ (20 mL) was added and then stirred for additional 15 min. The aqueous layer was then washed with CH_2Cl_2 (20 mL × 3). The combined organic layers were dried over sodium sulphate and filtered, and concentrated in *vacuo*. Purification by column chromatography (n-hexane/EtOAc: 10/1→5/1) yielded 8 (300 mg, 95%) as a light yellow oil.



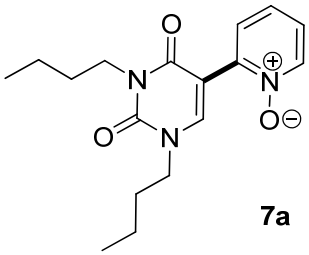
8

3-butyl-5-(4-methylpyridin-2-yl)-1-pentylpyrimidine-2,4(1*H*,3*H*)-dione (8)

^1H-NMR (250 MHz, $CDCl_3$): δ = 8.37 (d, J = 4.7 Hz, 1H), 8.32 (s, 1H), 8.21 (s, 1H), 7.01 (m, 1H), 4.05 (t, J = 7.5 Hz, 2H), 3.87 (t, J = 7.5 Hz, 2H), 2.38 (s, 3H), 1.76-1.67 (m, 4H), 1.46-1.39 (m, 4H), 0.97 (m, 6H). ^{13}C-NMR (62.89 MHz, $CDCl_3$): δ = 161.9 (Cq), 150.8 (Cq), 148.6 (CH), 147.8 (Cq), 143.6 (CH), 123.7 (CH), 123.2 (CH), 111.5 (Cq), 50.1 (CH_2), 41.6 (CH_2), 31.2 (CH_2), 29.7 (CH_2), 21.3 (CH_3), 20.3 (CH_2), 19.8 (CH_2), 13.8 (CH_3), 13.6 (CH_3). IR (KBr): 2954, 2926, 2866, 1698, 1647, 1599, 1477, 1450, 1371, 853, 759 cm-1. MS (EI) m/z (relative intensity): 329 (15) [M+], 315 (48), 300 (61), 245 (28), 217 (57), 159 (27), 119 (39), 92 (30). Anal. Calcd. for $C_{18}H_{25}N_3O_2$: C, 68.54; H, 7.99; N, 13.32; Found: C, 68.62; H, 7.96; N, 13.35.

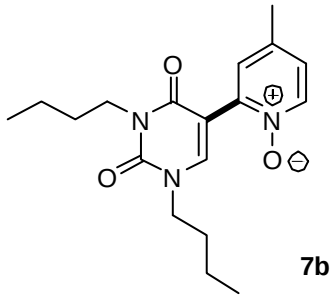
General Procedure: Pd-catalyzed dehydrogenative cross-coupling of pyridine-*N*-oxides with uracils

A 10 ml microwave vial equipped with a magnetic stirring bar and septum was flame-dried and then cooled . The vial was then charged with pyridine *N*-oxide (**5a**) (285 mg, 3 mmol), 1,3-dibutylpyrimidine-2,4(1*H*,3*H*)-dione (**6a**) (224 mg, 1.00 mmol), Ag_2CO_3 (827 mg, 3 mmol), $Pd(OAc)_2$ (23 mg, 10 mol %), pyridine (79 mg, 1 mmol) and 1,4-dioxane (4 mL). The vial was then sealed and immersed in an oil bath, which was preheated at 140 °C, for 18 h. After this time the reaction mixture was cooled to room temperature and then diluted with chloroform (10 mL). The mixture was washed with water (5 mL), and the aqueous phase was extracted with chloroform (10 mL). The organic extracts were dried over sodium sulphate and filtered. Concentration of the solution by rotary evaporation under reduced pressure gave a residue which was purified by using column chromatography (*n*-hexane/EtOAc: 1/1→1/4) yielded **3w** (272 mg, 82%) as a brown oil.



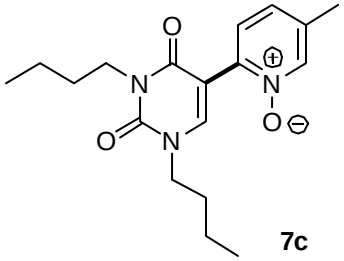
7a

2-(1,3-dibutyl-2,4-dioxo-1,2,3,4-tetrahydropyrimidin-5-yl)pyridine 1-oxide (7a)
^{1}H-NMR (300 MHz, $CDCl_3$): δ = 8.94 (s, 1H), 8.22 (d, J = 6.3 Hz, 1H), 7.93 (d, J = 8.1 Hz, 1H), 7.25 (t, J = 7.5 Hz, 1H), 7.12 (t, J = 7.5 Hz, 1H), 3.95 (t, J = 7.5 Hz, 2H), 3.80 (t, J = 7.5 Hz, 2H), 1.68 (quintet, J = 7.2 Hz, 2H), 1.57 (quintet, J = 8.1 Hz, 2H), 1.37-1.30 (m, 4H), 0.88 (m, 6H). ^{13}C-NMR (75 MHz, $CDCl_3$): δ = 161.3 (C_q), 150.4 (C_q), 146.5 (CH), 141.6 (C_q), 140.0 (CH), 128.3 (CH), 125.7 (CH), 123.8 (CH), 103.0 (C_q), 50.4 (CH_2), 41.8 (CH_2), 31.1 (CH_2), 29.5 (CH_2), 20.2 (CH_2), 19.7 (CH_2), 13.7 (CH_3), 13.6 (CH_3). IR (KBr): 2956, 2871, 1700, 1637, 1551, 1454, 1369, 1295, 1178, 855, 821, 761 cm^{-1}. MS (EI) *m/z* (relative intensity): 317 (50) [M^+], 300 (100), 275 (50), 258 (40), 219 (20), 201 (20), 159 (95), 131 (30), 119 (20), 92 (35), 78 (47). Anal. Calcd. for $C_{17}H_{23}N_3O_3$: C, 64.33; H, 7.30; N, 13.24; Found: C, 64.32; H, 7.28; N, 13.20.



7b

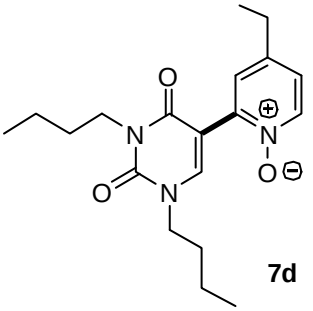
2-(1,3-dibutyl-2,4-dioxo-1,2,3,4-tetrahydropyrimidin-5-yl)-4-methylpyridine 1-oxide (7b): The general procedure A was followed using 1,3-dibutylpyrimidine-2,4(1*H*,3*H*)-dione **(6a)** (224 mg, 1.00 mmol) and 4-methylpyridine 1-oxide (**5b**) (327 mg, 3.00 mmol). Purification by column chromatography (*n*-hexane/EtOAc: 1/1→1/4) yielded **7b** (259 mg, 78%) as a brown solid (M.p.: 79–81 °C). ^{1}H-NMR (300 MHz, $CDCl_3$): δ = 8.90 (d, J = 5.7 Hz, 1H), 8.02 (t, J = 6.3 Hz, 1H), 7.67 (m, 1H), 6.85 (m, 1H), 3.86 (m, 2H), 3.71 (m, 2H), 2.22 (d, J = 4.5 Hz, 3H), 1.60 (m, 2H), 1.49 (m, 2H), 1.26 (m, 4H), 0.83 (m, 6H). ^{13}C-NMR (75 MHz, $CDCl_3$): δ = 161.3 (C_q), 150.3 (C_q), 146.5 (CH), 140.5 (C_q), 139.2 (CH), 137.0 (C_q), 128.5 (CH), 124.6 (CH), 103.0 (C_q), 50.2 (CH_2), 41.7 (CH_2), 31.1 (CH_2), 29.5 (CH_2), 20.4 (CH_3), 20.2 (CH_2), 19.6 (CH_2), 13.7 (CH_3), 13.6 (CH_3). IR (KBr): 2959, 2868, 1703, 1642, 1449, 1373, 1449, 1373, 1295, 1173, 825, 784, 760 cm^{-1}. MS (EI) *m*/*z* (relative intensity): 331 (13) [M^+], 314 (65), 289 (20), 272 (45), 258 (10), 233 (17), 173 (100), 159 (20), 145 (73), 133 (40), 106 (70), 92 (100), 77 (45). Anal. Calcd. for $C_{18}H_{25}N_3O_3$: C, 65.23; H, 7.60; N, 12.68; Found: C, 65.38; H, 7.62; N, 12.68.



7c

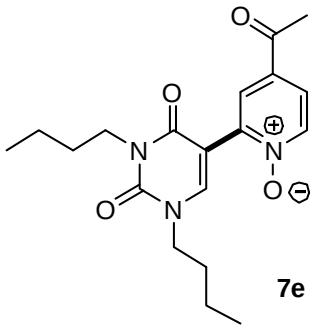
2-(1,3-dibutyl-2,4-dioxo-1,2,3,4-tetrahydropyrimidin-5-yl)-5-methylpyridine 1-oxide (7c): The general procedure A was followed using 1,3-dibutylpyrimidine-2,4(1*H*,3*H*)-dione **(6a)** (224 mg, 1.00 mmol) and 3-methylpyridine 1-oxide (**5c**) (327 mg, 3.00 mmol). Purification by column chromatography (*n*-hexane/EtOAc: 1/1→1/4) yielded **7c** (242 mg, 73%) as a orange solid (M.p.: 75–77 °C). ^{1}H-NMR (400 MHz, $CDCl_3$): δ = 8.97 (s, 1H), 8.13 (s, 1H), 7.87 (d, J = 8.4 Hz, 1H), 7.13 (d, J = 8.4 Hz, 1H), 4.02 (t, J = 7.2 Hz, 2H), 3.86 (t, J = 7.2 Hz, 2H), 2.32 (s, 3H), 1.74 (quintet, J = 7.2 Hz, 2H), 1.63 (quintet, J = 7.6 Hz, 2H), 1.39 (m, 4H), 0.95 (m, 6H). ^{13}C-NMR (100 MHz, $CDCl_3$): δ = 161.5 (C_q), 150.5 (C_q), 146.2 (CH), 139.7 (CH), 138.8 (C_q), 134.5 (C_q), 127.5 (CH), 127.1 (CH), 103.2 (C_q), 50.3 (CH_2), 41.8

(CH_2), 31.2 (CH_2), 29.6 (CH_2), 20.3(CH_3), 19.7 (CH_2), 18.1 (CH_2), 13.8 (CH_3), 13.7 (CH_3). IR (KBr): 3042, 2959, 2870, 1700, 1635, 1451, 1397, 1268, 1121, 1072, 815, 761 cm^{-1}. MS (EI) *m*/*z* (relative intensity): 331 (16) [M^+], 314 (50), 272 (24), 258 (7), 233 (24), 201 (27), 173 (100), 159 (17), 149 (90), 133 (25), 106 (42), 92 (48), 77 (50). Anal. Calcd. for $C_{18}H_{25}N_3O_3$: C, 65.23; H, 7.60; N, 12.68; Found: C, 65.05; H, 7.59; N, 12.71.



7d

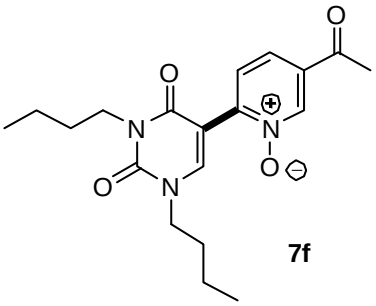
2-(1,3-dibutyl-2,4-dioxo-1,2,3,4-tetrahydropyrimidin-5-yl)-4-ethylpyridine 1-oxide (7d): The general procedure A was followed using 1,3-dibutylpyrimidine-2,4(1*H*,3*H*)-dione **(6a)** (224 mg, 1.00 mmol) and 4-ethylpyridine 1-oxide (**5d**) (327 mg, 3.00 mmol). Purification by column chromatography (*n*-hexane/EtOAc: 1/1→1/4) yielded **7d** (277 mg, 80%) as a brown oil. ^{1}H-NMR (300 MHz, $CDCl_3$): δ = 8.9 (s, 1H), 8.12 (d, J_H = 6.6 Hz, 1H), 7.73 (s, 1H), 6.94 (d, *J* = 6.6 Hz,1H), 3.93 (t, *J* = 7.2 Hz, 2H), 3.78 (t, *J* = 7.2 Hz, 2H), 2.58 (q, *J* = 7.5 Hz, 2H), 1.65 (m, 2H), 1.71-1.54 (m, 4H), 1.18 (t, *J* = 7.5 Hz, 3H), 0.90 (m, 6H). ^{13}C-NMR (75 MHz, $CDCl_3$): δ = 161.4 (C_q), 150.4 (C_q), 146.6 (CH), 143.4 (C_q), 140.8 (C_q), 139.4 (CH), 127.5 (CH), 123.4 (CH), 103.1 (C_q), 50.3 (CH_2), 41.8 (CH_2), 31.1 (CH_2), 29.5 (CH_2), 27.5 (CH_2), 20.2 (CH_2), 19.7 (CH_2), 14.2 (CH_3), 13.7 (CH_3), 13.6 (CH_3). IR (KBr): 2959, 2869, 1701, 1639, 1452, 1425, 1371, 1292, 1117, 832, 779, 758 cm^{-1}. MS (EI) *m*/*z* (relative intensity): 345 (45) [M^+], 328 (88), 303 (72), 286 (45), 272 (8), 187 (100), 173 (10), 159 (50), 91 (20). Anal. Calcd. for $C_{19}H_{27}N_3O_3$: C, 66.06; H, 7.88; N, 12.16; Found: C, 65.90; H, 7.90; N, 12.13.



7e

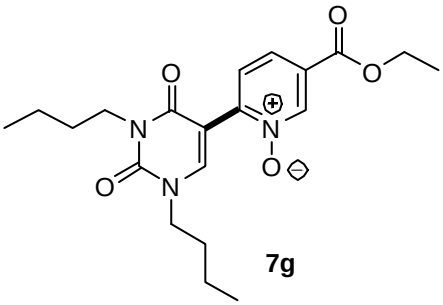
4-acetyl-2-(1,3-dibutyl-2,4-dioxo-1,2,3,4-tetrahydropyrimidin-5-yl)pyridine 1-oxide(7e): The general procedure A was followed using 1,3-dibutylpyrimidine-2,4(1*H*,3*H*)-dione **(6a)** (224 mg, 1.00 mmol) and 4-acetylpyridine 1-oxide (**5e**) (411 mg, 3.00 mmol). Purification by column chromatography (*n*-hexane/EtOAc: 1/1→1/4) yielded **7e** (263 mg, 73%) as a dark orange solid (M.p.: 95–97 °C). ^{1}H-NMR (500 MHz, $CDCl_3$): δ = 8.96 (s, 1H), 8.58 (s, 1H), 8.40 (d, *J* = 6.5 Hz, 1H), 7.76 (d, *J* = 5.3 Hz, 1H), 4.03 (t, *J* = 7.3 Hz, 2H), 3.89 (t, *J* = 7.1 Hz,

2H), 2.65 (s, 3H), 1.76 (m, 2H), 1.41 (m, 4H), 0.98 (m, 6H). ^{13}C-NMR (125 MHz, $CDCl_3$): δ = 194.1 (C_q), 161.1 (C_q), 150.2 (C_q), 146.9 (CH), 142.6 (C_q), 140.8 (CH), 134.0 (C_q), 127.9 (CH), 122.0 (CH), 102.1 (C_q), 50.6 (CH_2), 41.9 (CH_2), 31.1 (CH_2), 29.5 (CH_2), 26.4 (CH_3), 20.2 (CH_2), 19.7 (CH_2), 13.7 (CH_3), 13.6 (CH_3). IR (KBr): 3043, 2956, 2869, 1696, 1657, 1459, 1424, 1272, 1237, 857, 524, 776 cm^{-1}. MS (EI) *m*/*z* (relative intensity): 359 (75) [M^+], 342 (100), 328 (8), 300 (40), 286 (26), 243 (30), 201 (82), 120 (15). Anal. Calcd. for $C_{19}H_{25}N_3O_4$: C, 63.49; H, 7.01; N, 11.69; Found: C, 63.38; H, 7.03; N, 11.64.



7f

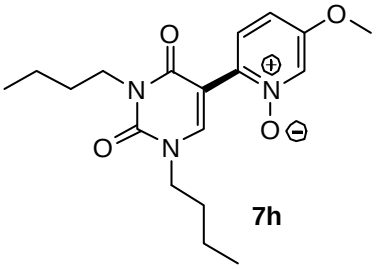
5-acetyl-2-(1,3-dibutyl-2,4-dioxo-1,2,3,4-tetrahydropyrimidin-5-yl)pyridine 1-oxide (7f): The general procedure A was followed using 1,3-dibutylpyrimidine-2,4(1*H*,3*H*)-dione **(6a)** (224 mg, 1.00 mmol) and 3-acetylpyridine 1-oxide (**5f**) (411 mg, 3.00 mmol). Purification by column chromatography (*n*-hexane/EtOAc: 1/1→1/4) yielded **7f** (245 mg, 68%) as a Brown solid (M.p.: 102–103 °C). ^{1}H-NMR (400 MHz, $CDCl_3$): δ = 9.31 (s, 1H), 8.78 (d, *J* = 1.6 Hz, 1H), 8.21 (d, *J* = 8.4 Hz, 1H), 7.76 (dd, *J* = 8.4, 2.0 Hz, 1H), 4.02 (t, *J* = 7.2 Hz, 2H), 3.88 (t, *J* = 7.6 Hz, 2H), 2.61 (s, 3H), 1.75 (quintet, *J* = 7.6 Hz, 2H), 1.63 (quintet, *J* = 7.6 Hz, 2H), 1.45-1.35 (m, 4H), 0.96 (m, 6H). ^{13}C-NMR (100 MHz, $CDCl_3$): δ = 193.8 (C_q), 161.2 (C_q), 150.2 (C_q), 147.2 (CH), 144.8 (C_q), 140.2 (CH), 132.6 (C_q), 127.8 (CH), 124.3 (CH), 102.3 (C_q), 50.6 (CH_2), 41.9 (CH_2), 31.2 (CH_2), 29.5 (CH_2), 26.7 (CH_3), 20.2 (CH_2), 19.7 (CH_2), 13.8 (CH_3), 13.6 (CH_3). IR (KBr): 2961, 2872, 1691, 1644, 1501, 1456, 1381, 1288, 1223, 912, 936, 776 cm^{-1}. MS (EI) *m*/*z* (relative intensity): 395 (22) [M^+], 342 (80), 328 (5), 300 (34), 286 (15), 243 (17), 201 (100), 120 (15). Anal. Calcd. for $C_{19}H_{25}N_3O_4$: C, 63.49; H, 7.01; N, 11.69; Found: C, 63.56; H, 6.98; N, 11.71.



7g

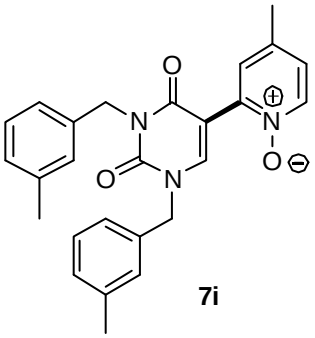
2-(1,3-dibutyl-2,4-dioxo-1,2,3,4-tetrahydropyrimidin-5-yl)-5 (ethoxycarbonyl)pyridine 1-oxide (7g): The general procedure A was followed using 1,3-dibutylpyrimidine-2,4(1*H*,3*H*)-dione **(6a)** (224 mg, 1.00 mmol) and 3-(ethoxycarbonyl)pyridine 1-oxide (**5g**) (411 mg, 3.00

mmol). Purification by column chromatography (*n*-hexane/EtOAc: 1/1→1/4) yielded **7g** (247 mg, 66%) as a brown solid (M.p.: 124–126 °C). ^{1}H-NMR (500 MHz, $CDCl_3$): δ = 9.25 (s, 1H), 8.82 (s, J = 1.5 Hz, 1H), 8.15 (d, J = 9.0 Hz, 1H), 7.83 (dd, J = 9, 1.7 Hz, 1H), 4.39 (q, J = 7.1 Hz, 2), 3.99 (t, J = 7.5 Hz, 2H), 3.85 (t, J = 7.5 Hz, 2H), 1.75-1.67 (m, 2H), 1.62-1.57 (m, 2H), 1.37 (m, 7H), 0.93 (m, 6H). ^{13}C-NMR (125 MHz, $CDCl_3$): δ = 162.8 (C_q), 161.1 (C_q), 150.2 (C_q), 147.2 (CH), 144.8 (C_q), 141.1 (CH), 127.6 (CH), 127.0 (C_q), 125.9 (CH), 102.3 (C_q), 62.1 (CH_2), 50.6 (CH_2), 41.9 (CH_2), 31.1 (CH_2), 29.5 (CH_2), 20.2 (CH_2), 19.7 (CH_2), 14.1 (CH_3), 13.7 (CH_3), 13.6 (CH_3). IR (KBr): 2931, 2049, 1731, 1645, 1607, 1568, 1370, 1020, 945, 772 cm^{-1}. MS (EI) *m*/*z* (relative intensity): 389 (10) [M^+], 372 (30), 358 (5), 330 (17), 231 (45), 167 (70), 149 (100). Anal. Calcd. for $C_{20}H_{27}N_3O_5$: C, 61.68; H, 6.99; N, 10.79; Found: C, 61.46; H, 7.01; N, 10.83.



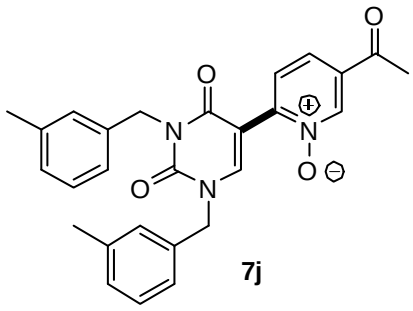
7h

2-(1,3-dibutyl-2,4-dioxo-1,2,3,4-tetrahydropyrimidin-5-yl)-5-methoxypyridine 1-oxide (7h): The general procedure A was followed using 1,3-dibutylpyrimidine-2,4(1*H*,3*H*)-dione **(6a)** (224 mg, 1.00 mmol) and 3-methoxypyridine 1-oxide (**5h**) (375 mg, 3.00 mmol). Purification by column chromatography (*n*-hexane/EtOAc: 1/1→1/4) yielded **7h** (296 mg, 85%) as a drak orange solid (M.p.: 70–72 °C). ^{1}H-NMR (500 MHz, $CDCl_3$): δ = 8.80 (s, 1H), 8.04 (s, 1H), 7.86 (d, J = 9.0 Hz, 1H), 6.96 (d, J = 7.5 Hz, 1H), 4.01 (t, J = 7.5, Hz, 2H), 3.86 (s, 3H), 3.85 (t, J = 7.5 Hz, 2H), 1.74 (m, 2H), 1.62 (m, 2H), 1.40 (m, 4H), 0.96 (m, 6H). ^{13}C-NMR (125 MHz, $CDCl_3$): δ = 161.5 (C_q), 156.1 (C_q), 150.5 (C_q), 145.8 (CH), 135.0 (C_q), 127.9 (CH), 127.6 (CH), 113.8 (CH), 103.1 (C_q), 56.2 (CH_3), 50.3 (CH_2), 41.8 (CH_2), 31.2 (CH_2), 29.6 (CH_2), 20.2 (CH_2), 19.7 (CH_2), 13.7 (CH_3), 13.6 (CH_3). IR (KBr): 3063, 2959, 2930, 1705, 1635, 1513, 1452, 1337, 1300, 1178, 827, 779, 759 cm^{-1}. MS (EI) *m*/*z* (relative intensity): 347 (80) [M^+], 330 (100), 288 (45), 274 (10), 231 (17), 189 (85), 175 (10), 161 (36), 108 (20). Anal. Calcd. for $C_{18}H_{25}N_3O_4$: C, 62.23; H, 7.25; N, 12.10; Found: C, 62.27; H, 7.22; N, 12.09.



7i

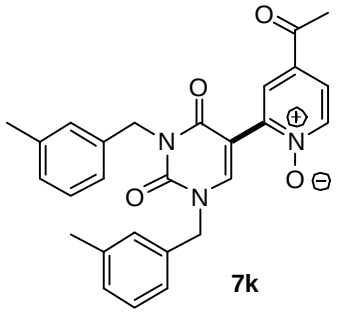
2-(1,3-bis(3-methylbenzyl)-2,4-dioxo-1,2,3,4-tetrahydropyrimidin-5-yl)-4-methylpyridine 1-oxide (7i): The general procedure A was followed using 1,3-bis(3-methylbenzyl)pyrimidine-2,4(1*H*,3*H*)-dione **(6b)** (320 mg, 1.00 mmol) and 4-methylpyridine 1-oxide (**5b**) (327 mg, 3.00 mmol). Purification by column chromatography (*n*-hexane/EtOAc: 1/1→1/4) yielded **7i** (320 mg, 77%) as a yellow solid (M.p.: 118–119 °C). ^{1}H-NMR (400 MHz, $CDCl_3$): δ = 9.06 (s, 1H), 8.19 (d, J = 6.4 Hz, 1H), 7.74 (d, J = 2.0 Hz, 1H), 7.30-6.98 (m, 8H), 6.97(d, J = 2.0 Hz, 1H), 5.19 (s, 2H), 5.01 (s, 2H), 2.37 (s, 3H), 2.35 (s, 3H), 2.33 (s, 3H). ^{13}C-NMR (100 MHz, $CDCl_3$): δ = 161.3 (C_q), 150.8 (C_q), 146.6 (CH), 140.7 (C_q), 139.3 (CH), 138.8 (C_q), 138.1 (C_q), 137.9 (C_q), 136.3 (C_q), 135.1 (C_q), 129.4 (CH), 129.3 (CH), 128.99 (CH), 128.96 (CH), 128.8 (CH), 128.5 (CH), 128.3 (CH), 126.0 (CH), 125.3 (CH), 124.9 (CH), 103.6 (C_q), 53.3 (CH_2), 45.1 (CH_2), 21.42 (CH_3), 21.41 (CH_3), 20.5 (CH_3). IR (KBr): 2919, 1738, 1698, 1652, 1445, 1371, 1217, 883, 827, 790 cm^{-1}. MS (EI) *m*/*z* (relative intensity):427 (65) [M^+], 411 (50), 172 (5), 159 (25), 146 (33), 105 (100), 91 (20). Anal. Calcd. for $C_{26}H_{25}N_3O_3$: C, 73.05; H, 5.89; N, 9.83; Found: C, 73.16; H, 5.90; N, 9.79.



7j

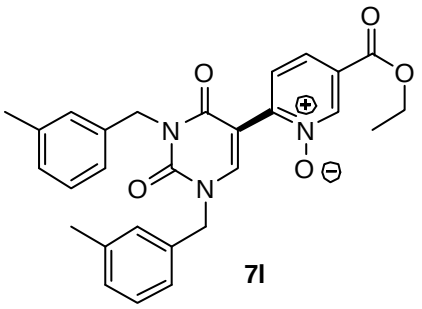
5-acetyl-2-(1,3-bis(3-methylbenzyl)-2,4-dioxo-1,2,3,4-tetrahydropyrimidin-5-yl)pyridine 1-oxide (7j): The general procedure A was followed using 1,3-bis(3-methylbenzyl)pyrimidine-2,4(1*H*,3*H*)-dione **(6b)** (320 mg, 1.00 mmol) and 3-acetylpyridine 1-oxide (**5f**) (411 mg, 3.00 mmol). Purification by column chromatography (*n*-hexane/EtOAc: 1/1→1/4) yielded **7j** (286 mg, 65%) as a yellow solid (M.p.: 129–131 °C). ^{1}H-NMR (500 MHz, $CDCl_3$): δ = 9.36 (s, 1H), 8.82 (s, 1H), 8.19 (d, J = 8.5 Hz, 1H), 7.83 (m, 1H), 7.29-7.16 (m, 6H), 7.09 (m, 1H), 5.20 (s, 2H), 5.04 (s, 2H), 2.61 (s, 3H), 2.36 (s, 3H), 2.34 (s, 3H). ^{13}C-NMR (125 MHz, $CDCl_3$): δ = 193.5 (C_q), 161.0 (C_q), 150.6 (C_q), 147.3 (CH), 140.5 (CH), 139.0 (C_q), 138.2 (C_q), 136.1 (C_q), 134.7 (C_q), 132.7 (C_q), 129.6 (CH), 129.1 (CH), 129.0 (CH), 128.6 (CH), 128.3 (CH), 128.1 (CH), 126.0 (CH), 125.4 (CH), 103.0 (C_q), 53.6 (CH_2), 45.2 (CH_2), 26.7 (CH_3), 21.4 (CH_3). IR (KBr): 3032, 29.22, 1696, 1652, 1586, 1445, 1388,

1284, 1220, 1114, 885, 830, 793 cm^{-1}. MS (EI) *m*/*z* (relative intensity): 445 (45) [M^+], 438 (35), 291 (10), 215 (35), 187 (15), 146 (75), 120 (37), 105 (100), 91 (15), 79 (34). Anal. Calcd. for $C_{27}H_{25}N_3O_4$: C, 71.19; H, 5.53; N, 9.22; Found: C, 71.21; H, 5.54; N, 9.18.



7k

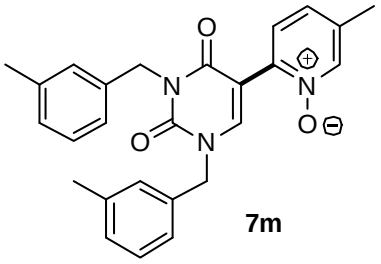
4-acetyl-2-(1,3-bis(3-methylbenzyl)-2,4-dioxo-1,2,3,4-tetrahydropyrimidin-5-yl)pyridine 1-oxide (7k): The general procedure A was followed using 1,3-bis(3-methylbenzyl)pyrimidine-2,4(1*H*,3*H*)-dione **(6b)** (320 mg, 1.00 mmol) and 4-acetylpyridine 1-oxide (**5e**) (411 mg, 3.00 mmol). Purification by column chromatography (*n*-hexane/EtOAc: 1/1→1/4) yielded **7k** (308 mg, 70%) as a orange solid (M.p.: 125–126 °C). ^{1}H-NMR (250 MHz, $CDCl_3$): δ = 8.94 (s, 1H), 8.46 (d, J = 2.5 Hz, 1H), 8.24 (d, J = 5.0 Hz, 1H), 7.66 (m, 1H), 7.27-7.07 (m, 8H), 5.20 (s, 2H), 5.01 (s, 2H), 2.59 (s, 3H), 2.34 (s, 3H), 2.32 (s, 3H). ^{13}C-NMR (62.89 MHz, $CDCl_3$) δ = 194.3 (C_q), 161.2 (C_q), 150.7 (C_q), 146.1 (CH), 140.4 (C_q), 139.0 (C_q), 138.2 (C_q), 136.2 (C_q), 134.8 (C_q), 132.1 (C_q), 129.51 (CH), 129.47 (CH), 129.03 (CH), 128.94 (CH), 128.6 (CH), 128.4 (CH), 127.7 (CH), 126.0 (CH), 125.3 (CH), 122.1 (CH), 103.4 (C_q), 53.4 (CH_2), 45.2 (CH_2), 26.4 (CH_3), 21.4 (CH_3). IR (KBr): 2918, 1702, 1644, 1607, 1545, 1446, 1358, 1221, 950, 667 cm^{-1}. MS (EI) *m*/*z* (relative intensity): 455 (45) [M^+], 438 (50), 291 (12), 215 (41), 146 (70), 105 (100), 91 (21), 77 (44). Anal. Calcd. for $C_{27}H_{25}N_3O_4$: C, 71.19; H, 5.53; N, 9.22; Found: C, 71.36; H, 5.53; N, 9.25.



7l

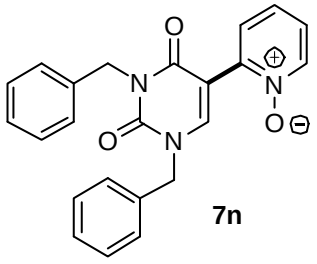
2-(1,3-bis(3-methylbenzyl)-2,4-dioxo-1,2,3,4-tetrahydropyrimidin-5-yl)-5-(ethoxycarbonyl)pyridine 1-oxide (7l): The general procedure A was followed using 1,3-bis(3-methylbenzyl)pyrimidine-2,4(1*H*,3*H*)-dione **(6b)** (320 mg, 1.00 mmol) and 3-(ethoxycarbonyl)pyridine 1-oxide (**5g**) (501 mg, 3.00 mmol). Purification by column chromatography (*n*-hexane/EtOAc: 1/1→1/4) yielded **7l** (292 mg, 60%) as a orange solid (M.p.: 127–129 °C). ^{1}H-NMR (500 MHz, $CDCl_3$): δ = 9.36 (s, 1H), 8.84 (s, 1H), 8.13 (d, J = 8.5 Hz, 1H), 7.83 (d, J = 8.5 Hz, 1H), 7.28-7.15 (m, 7H), 7.10 (s, J = 7.5 Hz, 1H), 5.20 (s,

2H), 5.03 (s, 2H), 4.41 (q, J = 7.0 Hz, 2H), 2.36 (s, 3H), 2.34 (s, 3H), 1.41 (t, J = 7.0 Hz, 3H). ^{13}C-NMR (125 MHz, $CDCl_3$): δ = 163.2 (C_q), 161.1 (C_q), 150.7 (C_q), 146.9 (CH), 144.6 (C_q), 141.1 (CH), 139.0 (C_q), 138.2 (C_q), 136.2 (C_q), 134.8 (C_q), 129.5 (CH), 129.0 (CH), 128.6 (CH), 128.4 (CH), 127.7 (CH), 127.2 (C_q), 126.0 (CH), 125.8 (CH), 125.4 (CH), 103.0 (C_q), 62.2 (CH_2), 53.5 (CH_2), 45.2 (CH_2), 21.4 (CH_3), 14.2 (CH_3). IR (KBr): 2986, 1732, 1703, 1657, 1606, 1548, 1398, 1358, 1218, 1020, 966, 818 cm^{-1}. MS (EI) *m*/*z* (relative intensity): 485 (20) [M^+], 468 (13), 245 (10), 217 (17), 146 (64), 105 (100), 91 (33), 77 (41). Anal. Calcd. for $C_{28}H_{27}N_3O_5$: C, 69.26; H, 5.61; N, 8.65; Found: C, 69.18; H, 5.62; N, 8.63.



7m

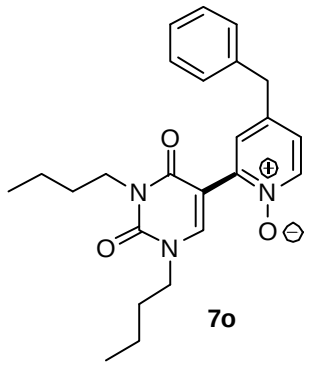
2-(1,3-bis(3-methylbenzyl)-2,4-dioxo-1,2,3,4-tetrahydropyrimidin-5-yl)-5-methylpyridine 1-oxide (7m): The general procedure A was followed using 1,3-bis(3-methylbenzyl)pyrimidine-2,4(1*H*,3*H*)-dione **(6b)** (320 mg, 1.00 mmol) and 3-methylpyridine 1-oxide (**5c**) (327 mg, 3.00 mmol). Purification by column chromatography (*n*-hexane/EtOAc: 1/1→1/4) yielded **7m** (325 mg, 76%) as a dark orange solid (M.p.: 149–150 °C). ^{1}H-NMR (250 MHz, $CDCl_3$): δ = 9.01 (s, 1H), 8.10 (s, 1H), 7.82 (d, J = 7.5 Hz, 1H), 7.26-7.05 (m, 8H), 5.17 (s, 2H), 4.99 (s, 2H), 2.33 (s, 3H), 2.32 (s, 3H), 2.29 (s, 3H). ^{13}C-NMR (62.98 MHz, $CDCl_3$): δ = 161.2 (C_q), 150.1 (C_q), 145.9 (CH), 139.7 (C_q), 138.9 (C_q), 138.1 (C_q), 136.4 (C_q), 135.0 (C_q), 134.7 (C_q), 129.5 (CH), 129.3 (CH), 129.1 (CH), 128.4 (CH), 128.3 (CH), 127.6 (CH), 127.0 (CH), 126.0 (CH), 125.3 (CH), 103.3 (C_q), 53.3 (CH_2), 45.1 (CH_2), 21.4 (CH_3), 18.0 (CH_3). IR (KBr): 3042, 2921, 1701, 1643, 1589, 1499, 1330, 1207, 781, 759 cm^{-1}. MS (EI) *m*/*z* (relative intensity): 427 (51) [M^+], 410 (32), 313 (62), 200 (31), 146 (17), 109 (100), 92 (27), 77 (35). Anal. Calcd. for $C_{26}H_{25}N_3O_3$: C, 73.05; H, 5.89; N, 9.83; Found: C, 72.87; H, 5.91; N, 9.87.



7n

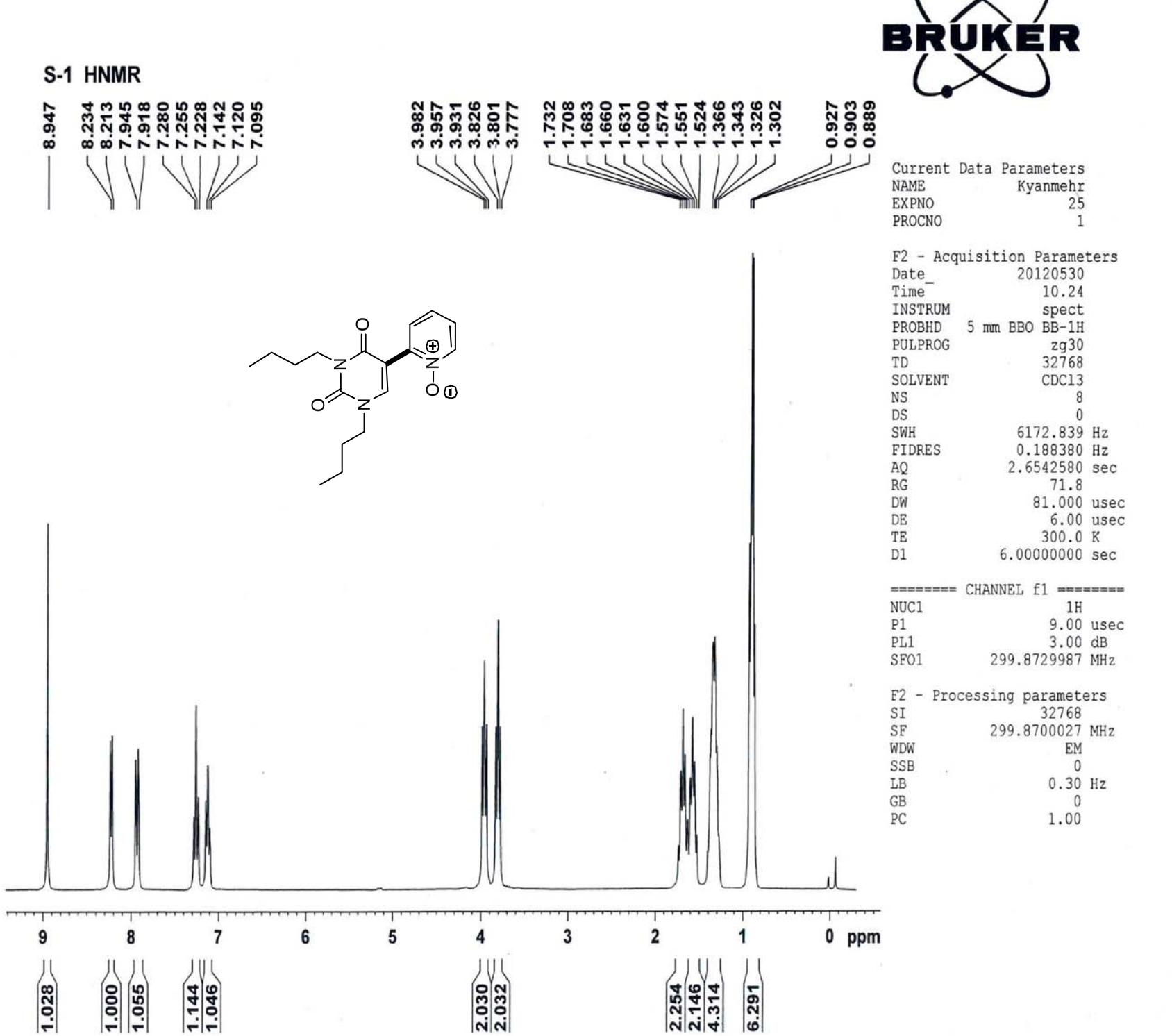
2-(1,3-dibenzyl-2,4-dioxo-1,2,3,4-tetrahydropyrimidin-5-yl)pyridine 1-oxide (7n): The general procedure A was followed using 1,3-dibenzylpyrimidine-2,4(1*H*,3*H*)-dione **(6c)** (292 mg, 1.00 mmol) and pyridine 1-oxide (**5a**) (285 mg, 3.00 mmol). Purification by column chromatography (*n*-hexane/EtOAc: 1/1→1/4) yielded **7n** (301 mg, 78%) as a dark orange solid (M.p.: 118–120 °C). ^{1}H-NMR (250 MHz, $CDCl_3$): δ = 9.18 (s, 1H), 8.23 (d, J =

5.0 Hz, 1H), 7.93 (m, 1H), 7.54-7.11 (m, 12H), 5.21 (s, 2H), 5.04 (s, 2H). ^{13}C-NMR (62.89 MHz, $CDCl_3$): δ = 161.2 (C_q), 150.8 (C_q), 146.2 (CH), 140.0 (CH), 136.4 (C_q), 135.0 (C_q), 129.13 (CH), 129.10 (CH), 128.6 (CH), 128.4 (CH), 128.3 (CH), 127.8 (CH), 125.5 (CH), 123.9 (CH), 103.8 (C_q), 53.4 (CH_2), 45.1 (CH_2). IR (KBr): 3416, 3066, 1700, 1650, 1694, 1449, 1428, 1375, 1212, 760, 694 cm^{-1}. MS (EI) *m*/*z* (relative intensity): 385 (54) [M^+], 368 (40), 235 (11), 132 (37), 91 (100), 78 (13), 65 (26). Anal. Calcd. for $C_{23}H_{19}N_3O_3$: C, 71.67; H, 4.97; N, 10.90; Found: C, 71.81; H, 4.99; N, 10.87.



7o

4-benzyl-2-(1,3-dibutyl-2,4-dioxo-1,2,3,4-tetrahydropyrimidin-5-yl)pyridine 1-oxide (7o): The general procedure A was followed using **(6a)** (224 mg, 1.00 mmol) and 4-benzylpyridine 1-oxide (**5i**) (555 mg, 3.00 mmol). Purification by column chromatography (*n*-hexane/EtOAc: 1/1→1/4) yielded **7o** (327 mg, 80%) as a yellow oil. ^{1}H-NMR (250 MHz, $CDCl_3$): δ = 9.01 (s, 1H), 8.15 (d, *J* = 6.5 Hz, 1H), 7.87 (d, *J* = 2.5 Hz, 1H), 7.35-7.17 (m, 5H), 6.90 (m, 1H), 4.01 (m, 4H), 3.84 (t, *J* = 7.5 Hz, 2H), 1.76-1.61 (m, 4H), 1.46-1.31 (m, 4H), 0.95 (t, *J* = 7.2 Hz, 6H). ^{13}C-NMR (62.89 MHz, $CDCl_3$): δ = 161.4 (Cq), 150.4 (Cq), 146.6 (CH), 140.9 (Cq), 140.1 (CH), 139.7 (CH), 138.4 (Cq), 129.0 (CH), 128.9 (CH), 128.3 (CH), 126.9 (CH), 124.1 (CH), 103.1 (Cq), 50.4 (CH_2), 41.8 (CH_2), 40.5 (CH_2), 31.2 (CH_2), 29.6 (CH_2), 20.3 (CH_2), 19.7 (CH_2), 13.8 (CH_3), 13.6 (CH3). IR (KBr): 2958, 1699, 1648, 1456, 1430, 1354, 1245, 1223, 809, 700 cm^{-1}. MS (EI) *m*/*z* (relative intensity): 407 (17) [M^+], 390 (22), 329 (41), 252 (30), 215 (30), 143 (100), 105 (47), 77 (32)**.** Anal. Calcd. for $C_{30}H_{25}N_3O_3$: C, 75.77; H, 5.30; N, 8.84; Found: C, 75.62; H, 5.27; N, 8.87.



BRUKER

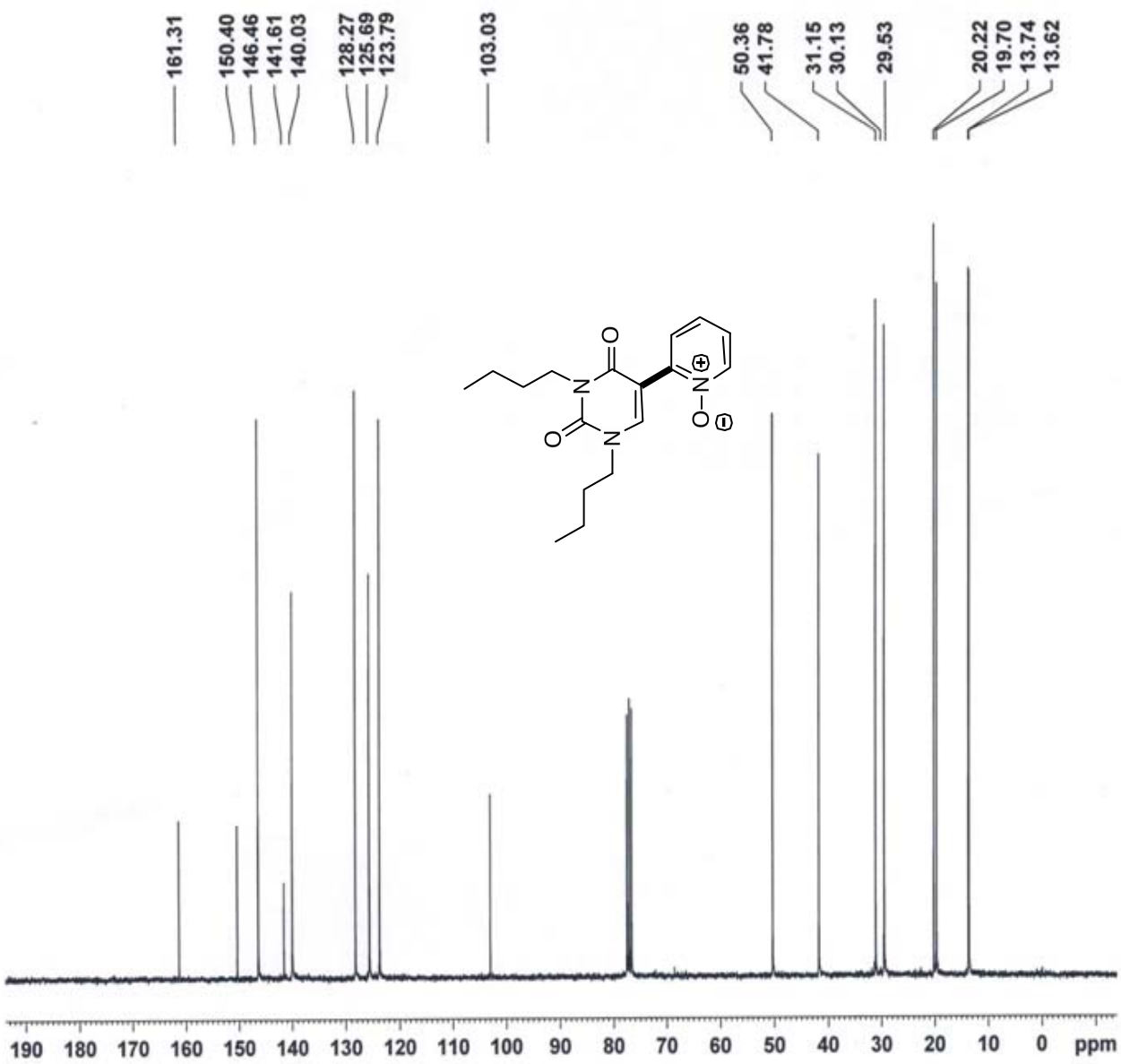
Current Data Parameters
NAME Kyanmehr
EXPNO 25
PROCNO 1

F2 - Acquisition Parameters
Date_ 20120530
Time 10.24
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 8
DS 0
SWH 6172.839 Hz
FIDRES 0.188380 Hz
AQ 2.6542580 sec
RG 71.8
DW 81.000 usec
DE 6.00 usec
TE 300.0 K
D1 6.00000000 sec

======== CHANNEL f1 ========
NUC1 1H
P1 9.00 usec
PL1 3.00 dB
SFO1 299.8729987 MHz

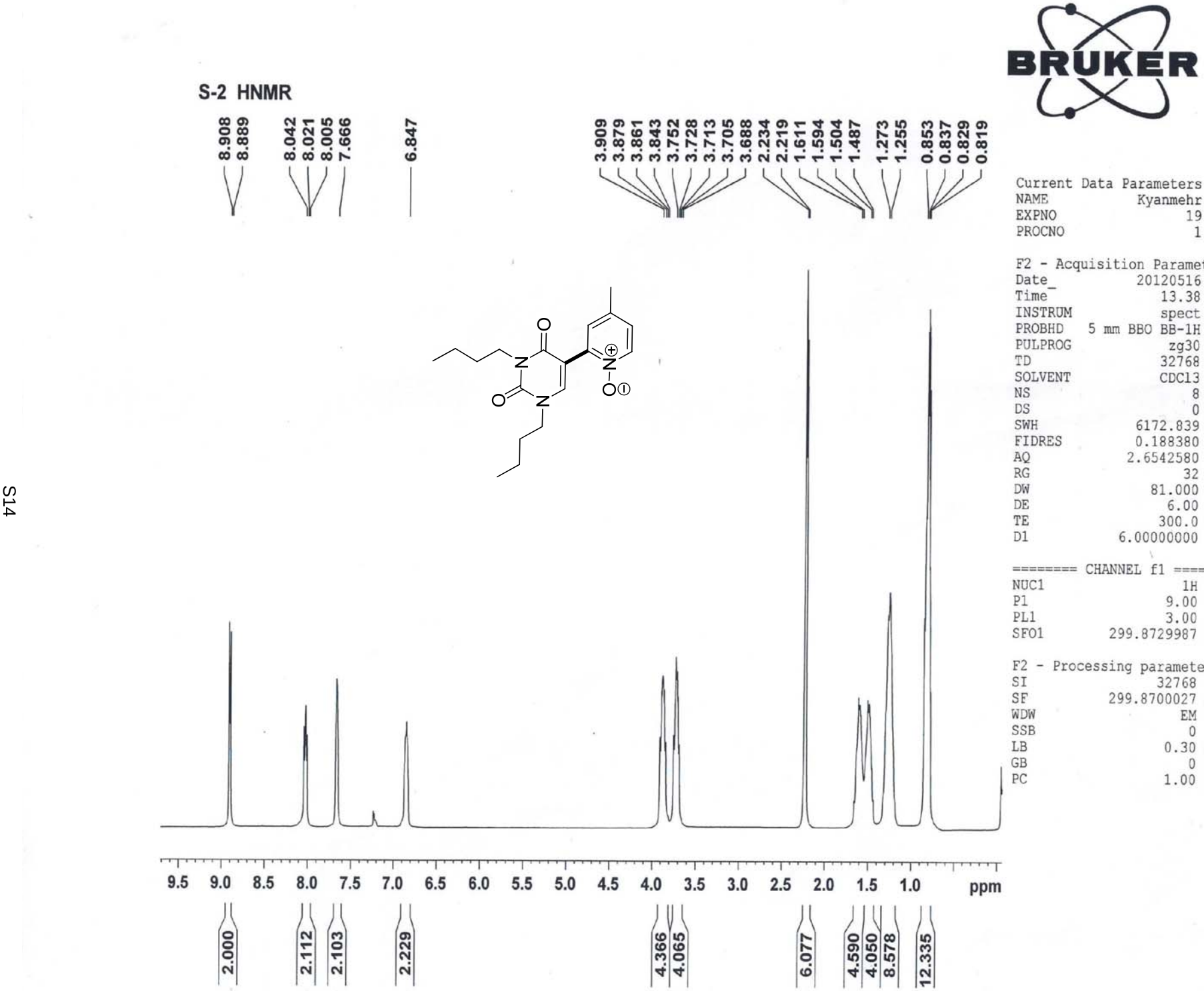
F2 - Processing parameters
SI 32768
SF 299.8700027 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

S-1 CNMR

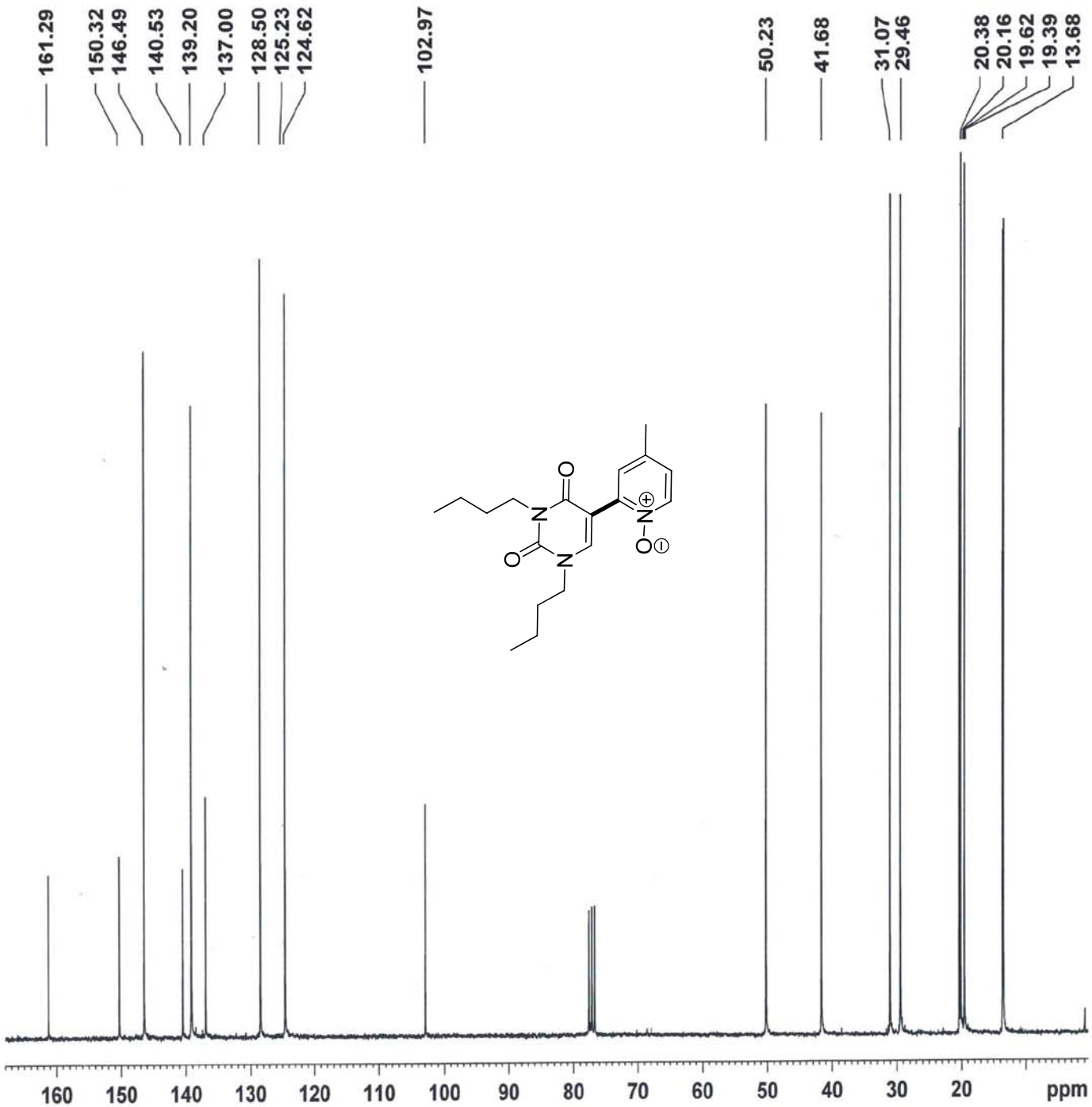


BRUKER

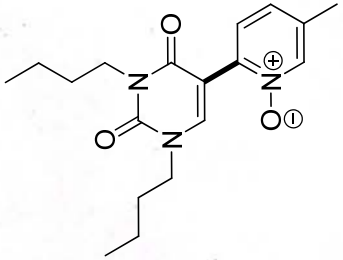
```
Current Data Parameters
NAME                  Kyanmehr
EXPNO                       26
PROCNO                       1

F2 - Acquisition Parameters
Date_                 20120530
Time                     11.52
INSTRUM                  spect
PROBHD     5 mm BBO BB-1H
PULPROG                 zgpg30
TD                       65536
SOLVENT                  CDCl3
NS                         850
DS                           2
SWH                  17985.611 Hz
FIDRES                0.274439 Hz
AQ                    1.8219508 sec
RG                       32768
DW                      27.800 usec
DE                       6.00 usec
TE                      300.0 K
D1                    4.00000000 sec
d11                   0.03000000 sec
d12                   0.00002000 sec

======== CHANNEL f1 ========
NUC1                       13C
P1                      17.00 usec
PL1                      -6.00 dB
SFO1                75.4099117 MHz

======== CHANNEL f2 ========
CPDPRG2                waltz16
NUC2                       1H
PCPD2                   80.00 usec
PL2                      3.00 dB
PL12                    23.00 dB
PL13                    23.00 dB
SFO2               299.8711995 MHz

F2 - Processing parameters
SI                       32768
SF                  75.4023710 MHz
WDW                         EM
SSB                          0
LB                       1.00 Hz
GB                           0
PC                        1.40
```


S-2 HNMR
BRUKER
8.908
8.889
8.042
8.021
8.005
7.666
6.847
3.909
3.879
3.861
3.843
3.752
3.728
3.713
3.705
3.688
2.234
2.219
1.611
1.594
1.504
1.487
1.273
1.255
0.853
0.837
0.829
0.819
Current Data Parameters
NAME Kyanmehr
EXPNO 19
PROCNO 1
F2 - Acquisition Parameters
Date_ 20120516
Time 13.38
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 8
DS 0
SWH 6172.839 Hz
FIDRES 0.188380 Hz
AQ 2.6542580 sec
RG 32
DW 81.000 usec
DE 6.00 usec
TE 300.0 K
D1 6.00000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 9.00 usec
PL1 3.00 dB
SFO1 299.8729987 MHz
F2 - Processing parameters
SI 32768
SF 299.8700027 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 ppm
2.000
2.112
2.103
2.229
4.366
4.065
6.077
4.590
4.050
8.578
12.335

S-2 CNMR




```
Current Data Parameters
NAME                 Kyanmehr
EXPNO                      18
PROCNO                      1

F2 - Acquisition Parameters
Date_                20120516
Time                    12.17
INSTRUM                 spect
PROBHD    5 mm BBO BB-1H
PULPROG                zgpg30
TD                      65536
SOLVENT                 CDCl3
NS                        850
DS                          2
SWH                 17985.611 Hz
FIDRES                0.274439 Hz
AQ                    1.8219508 sec
RG                      32768
DW                     27.800 usec
DE                      6.00 usec
TE                      300.0 K
D1                  2.00000000 sec
d11                 0.03000000 sec
d12                 0.00002000 sec

======== CHANNEL f1 ========
NUC1                       13C
P1                      17.00 usec
PL1                     -6.00 dB
SFO1                75.4099117 MHz

======== CHANNEL f2 ========
CPDPRG2                waltz16
NUC2                       1H
PCPD2                  80.00 usec
PL2                     3.00 dB
PL12                   23.00 dB
PL13                   23.00 dB
SFO2               299.8711995 MHz

F2 - Processing parameters
SI                      32768
SF                 75.4023710 MHz
WDW                        EM
SSB                         0
LB                      1.00 Hz
GB                          0
PC                       1.40
```

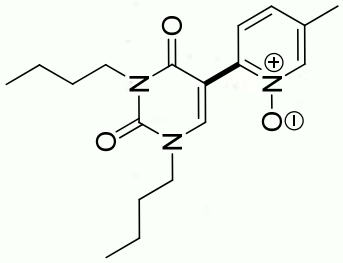


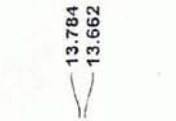
S6 1H

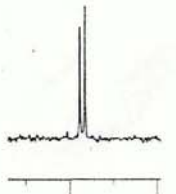
NAME	
EXPNO	1
PROCNO	1
Date_	20121017
Time	10.35
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg
TD	32768
SOLVENT	CDCl3
NS	8
DS	0
SWH	6009.615 Hz
FIDRES	0.183399 Hz
AQ	2.7263477 sec
RG	80.6
DW	83.200 usec
DE	6.50 usec
TE	292.8 K
D1	6.00000000 sec
TD0	1

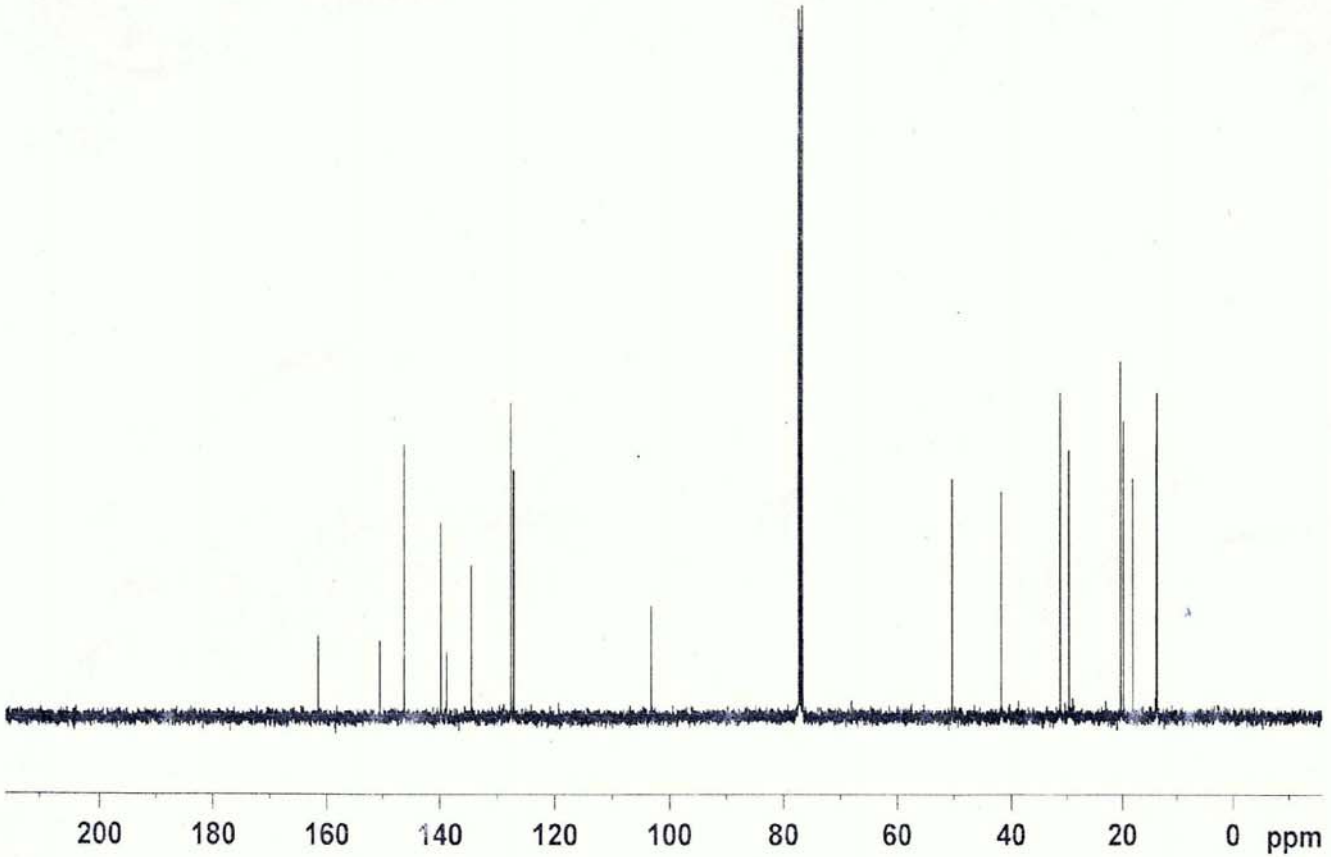
======== CHANNEL f1 ========

NUC1	1H
P1	13.50 usec
PL1	0.00 dB
PL1W	11.30348873 W
SFO1	400.1329410 MHz
SI	32768
SF	400.1300000 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00







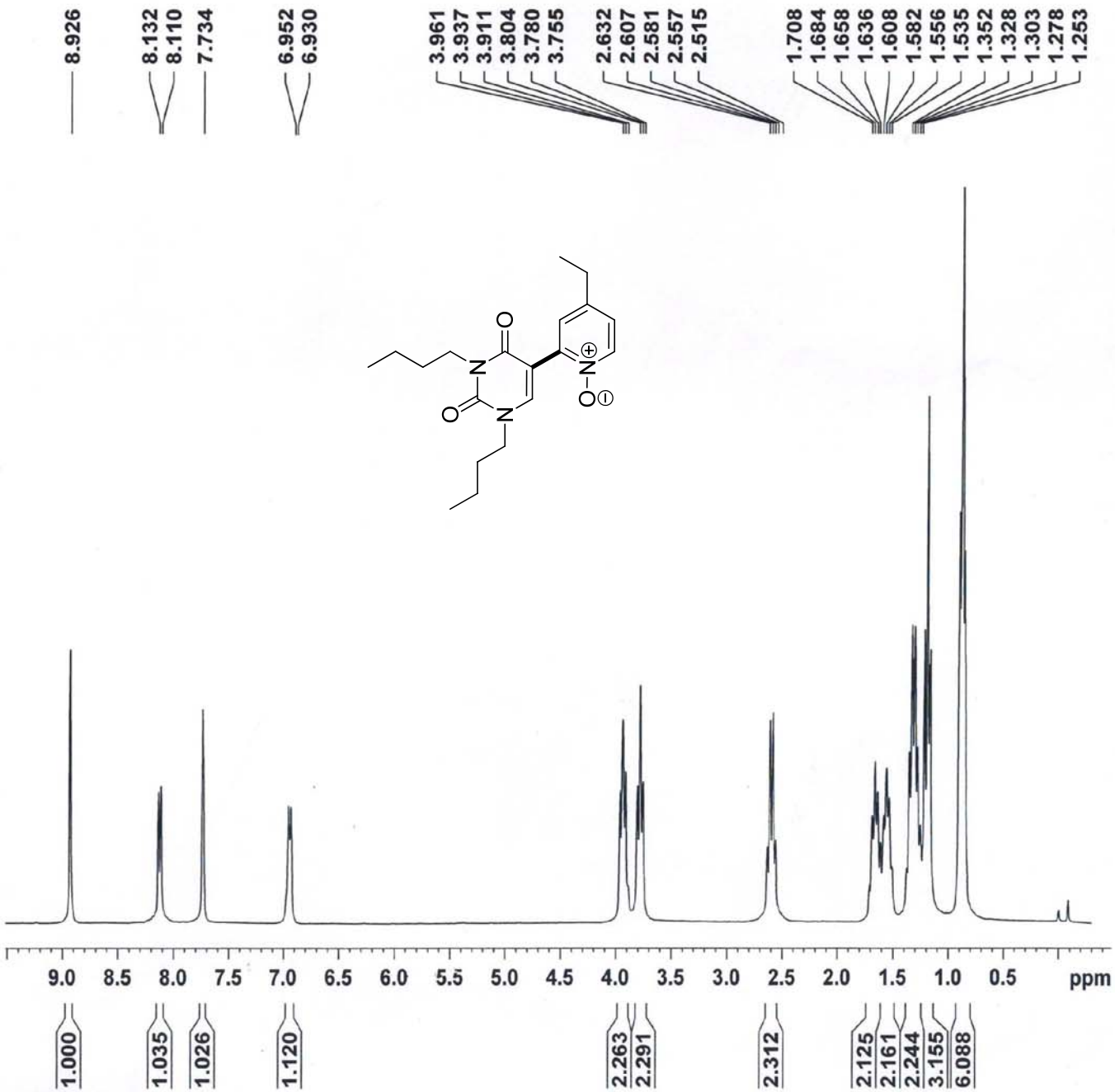


NAME S6 13C
EXPNO 1
PROCNO 1
Date_ 20121017
Time 10.59
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg
TD 65536
SOLVENT CDCl3
NS 328
DS 0
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 203
DW 20.800 usec
DE 6.50 usec
TE 293.5 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 7.60 usec
PL1 -2.00 dB
PL1W 56.92932510 W
SFO1 100.6228298 MHz

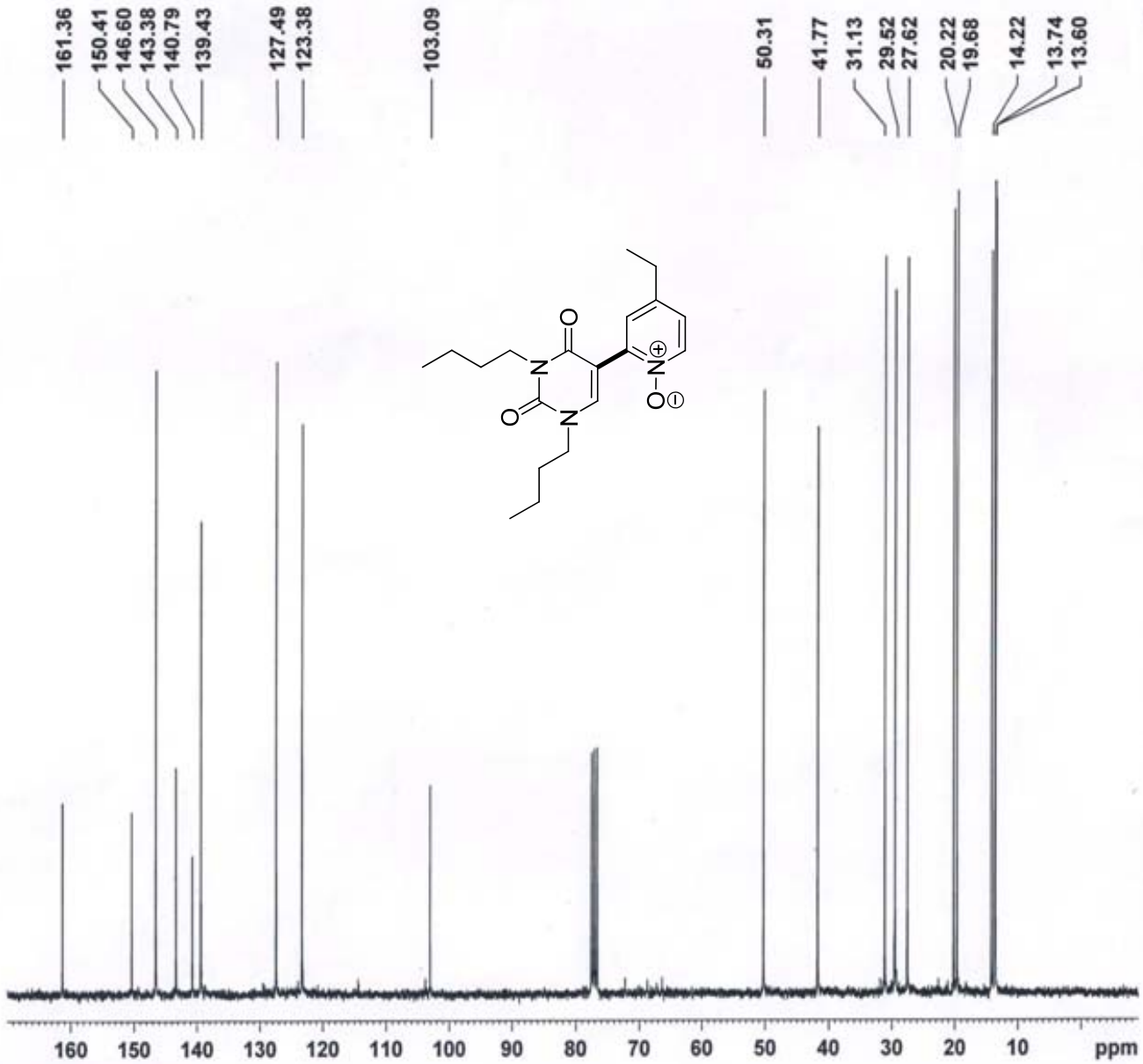
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 85.00 usec
PL2 0.00 dB
PL12 16.00 dB
PL13 16.00 dB
PL2W 11.30348873 W
PL12W 0.28393081 W
PL13W 0.28393081 W
SFO2 400.1316005 MHz
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

S-4 HNMR




```
Current Data Parameters
NAME                  Kyanmehr
EXPNO                       28
PROCNO                       1

F2 - Acquisition Parameters
Date_                 20120530
Time                     13.50
INSTRUM                  spect
PROBHD    5 mm BBO BB-1H
PULPROG                  zg30
TD                      32768
SOLVENT                 CDCl3
NS                          8
DS                          0
SWH                6172.839 Hz
FIDRES             0.188380 Hz
AQ                 2.6542580 sec
RG                      71.8
DW                 81.000 usec
DE                  6.00 usec
TE                  300.0 K
D1                 6.00000000 sec

======== CHANNEL f1 ========
NUC1                       1H
P1                   9.00 usec
PL1                  3.00 dB
SFO1           299.8729987 MHz

F2 - Processing parameters
SI                      32768
SF             299.8700027 MHz
WDW                        EM
SSB                         0
LB                   0.30 Hz
GB                          0
PC                       1.00
```

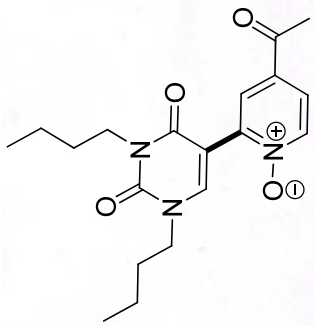
S-4 CNMR



BRUKER

```
Current Data Parameters
NAME                  Kyanmehr
EXPNO                       27
PROCNO                       1

F2 - Acquisition Parameters
Date_                 20120530
Time                     13.47
INSTRUM                  spect
PROBHD     5 mm BBO BB-1H
PULPROG                 zgpg30
TD                       65536
SOLVENT                  CDCl3
NS                         500
DS                           2
SWH                17985.611 Hz
FIDRES             0.274439 Hz
AQ                 1.8219508 sec
RG                       32768
DW                    27.800 usec
DE                     6.00 usec
TE                     300.0 K
D1                 4.00000000 sec
d11                0.03000000 sec
d12                0.00002000 sec

======== CHANNEL f1 ========
NUC1                       13C
P1                    17.00 usec
PL1                    -6.00 dB
SFO1              75.4099117 MHz

======== CHANNEL f2 ========
CPDPRG2                waltz16
NUC2                       1H
PCPD2                 80.00 usec
PL2                    3.00 dB
PL12                  23.00 dB
PL13                  23.00 dB
SFO2             299.8711995 MHz

F2 - Processing parameters
SI                       32768
SF                75.4023710 MHz
WDW                         EM
SSB                          0
LB                     1.00 Hz
GB                           0
PC                        1.40
```

Mr Rezaieefar S7

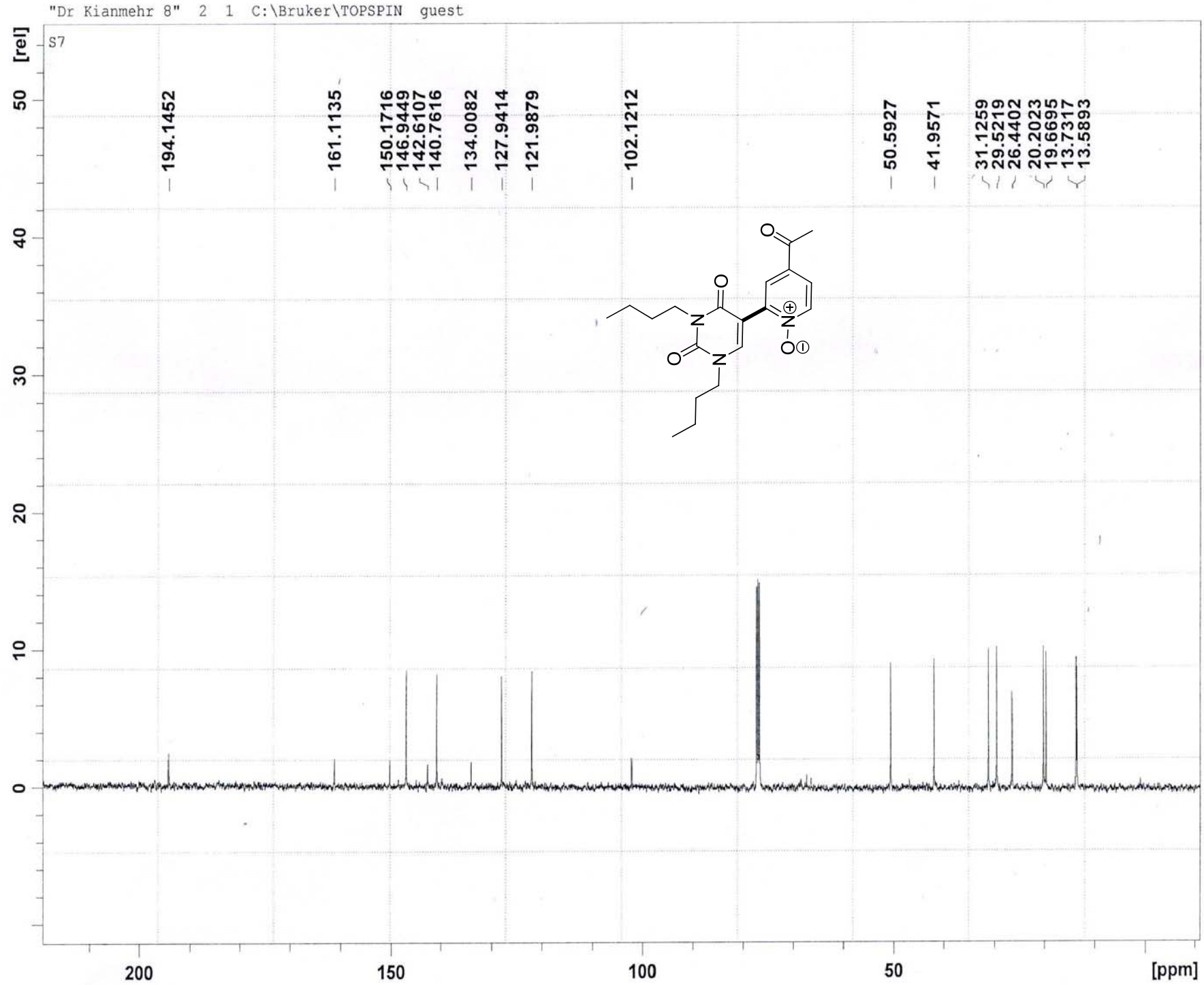


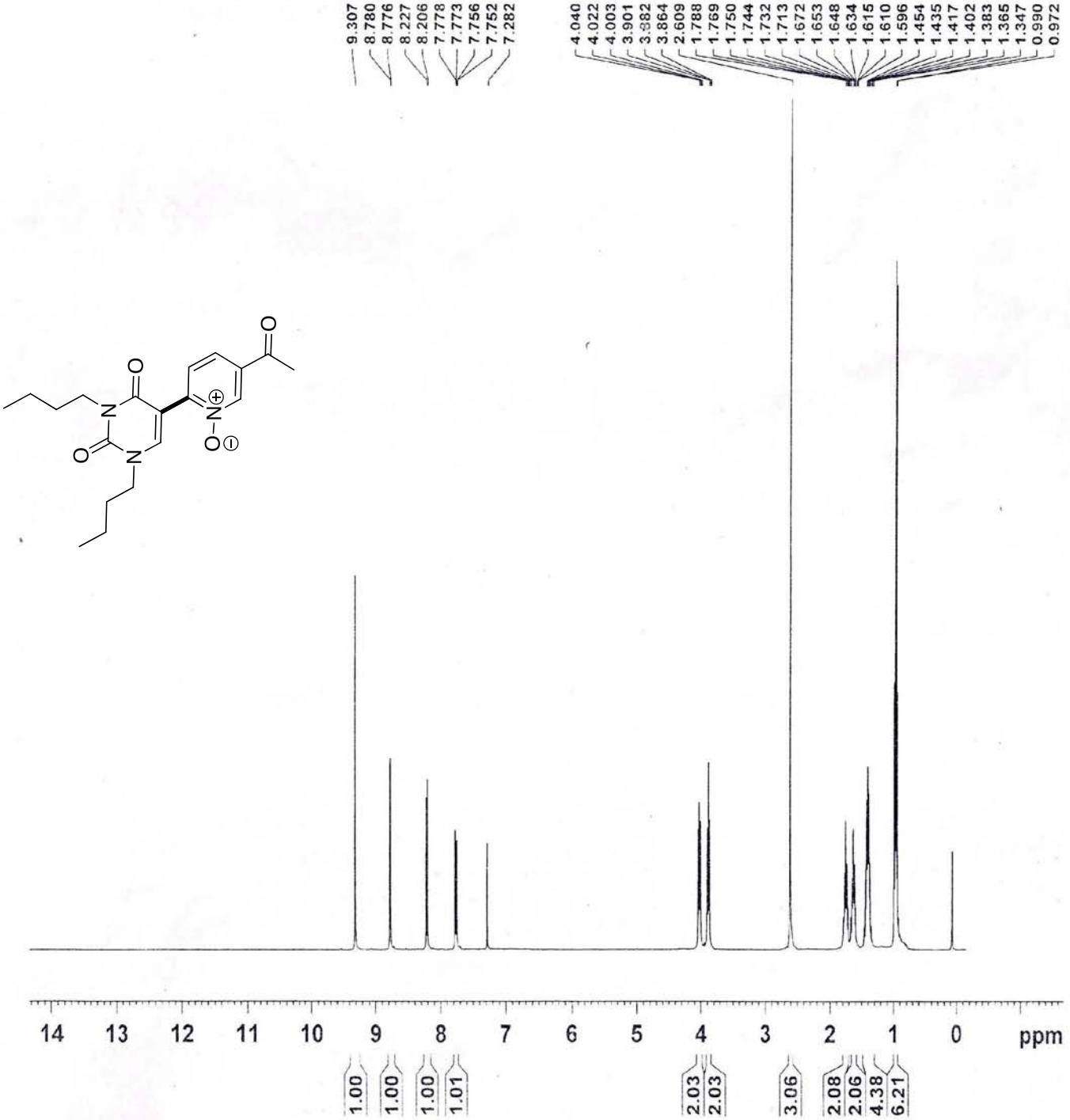
Current Data Parameters
NAME Dr Kianmehr 8
EXPNO 1
PROCNO 1

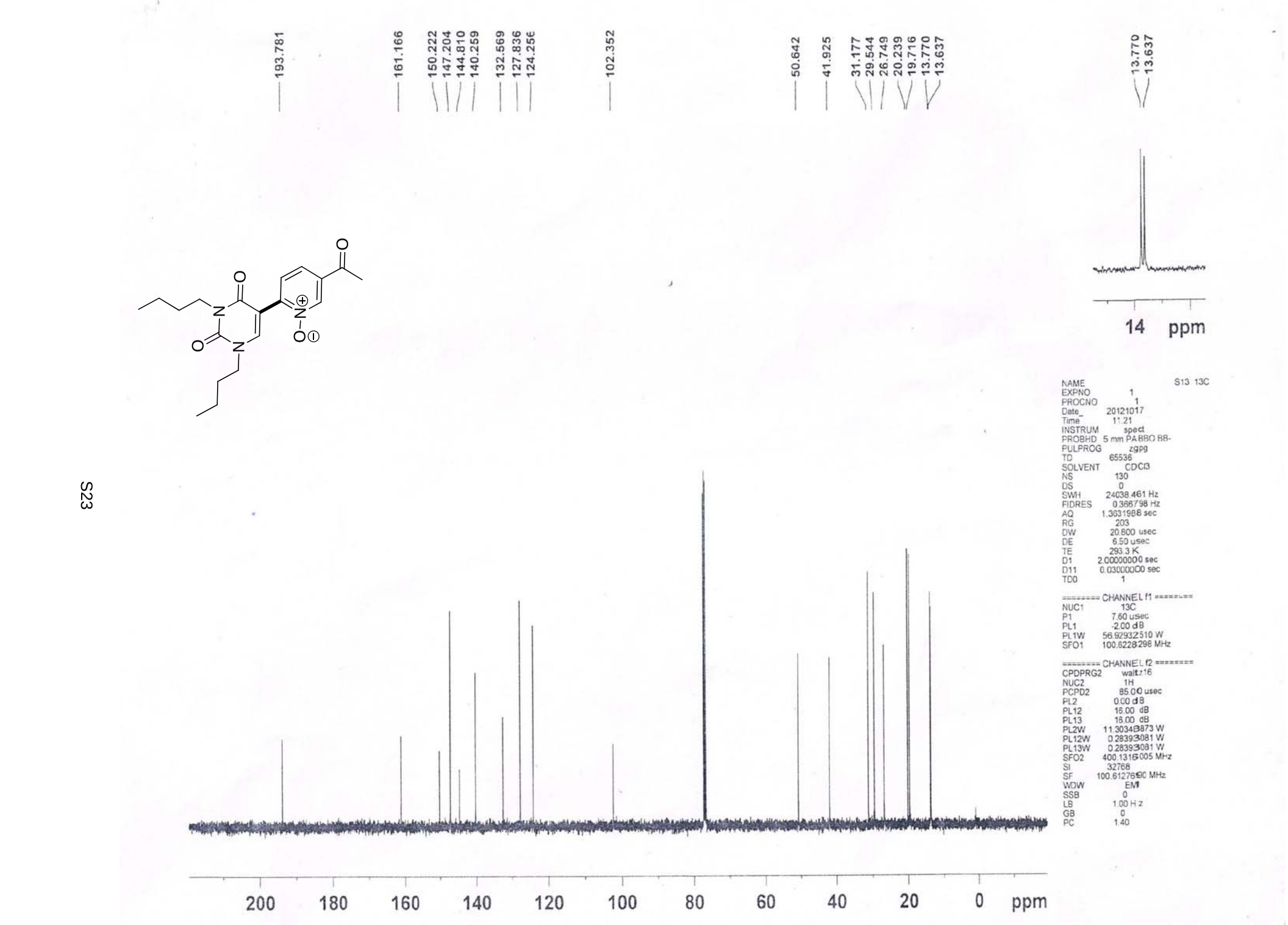
F2 - Acquisition Parameters
Date_ 20120520
Time 11.44
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 6
DS 0
SWH 8503.401 Hz
FIDRES 0.259503 Hz
AQ 1.9268084 sec
RG 181
DW 58.800 usec
DE 6.50 usec
TE 673.2 K
D1 1.00000000 sec
TD0 1

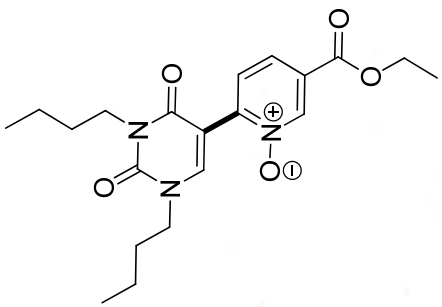
======== CHANNEL f1 ========
NUC1 1H
P1 9.50 usec
PL1 3.00 dB
SFO1 500.1335009 MHz

F2 - Processing parameters
SI 32768
SF 500.1300000 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 12.00


"Dr Kianmehr 8" 2 1 C:\Bruker\TOPSPIN guest
S7
[rel]
194.1452
161.1135
150.1716
146.9449
142.6107
140.7616
134.0082
127.9414
121.9879
102.1212
50.5927
41.9571
31.1259
29.5219
26.4402
20.2023
19.6695
13.7317
13.5893
200
150
100
50
[ppm]


9.307
8.780
8.776
8.227
8.206
7.778
7.773
7.756
7.752
7.282
4.040
4.022
4.003
3.901
3.882
3.864
2.609
1.788
1.769
1.750
1.744
1.732
1.713
1.672
1.653
1.648
1.634
1.615
1.610
1.596
1.454
1.435
1.417
1.402
1.383
1.365
1.347
0.990
0.972
NAME S13 1H
EXPNO 1
PROCNO 1
Date_ 20121017
Time 11.05
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 8
DS 0
SWH 6009.615 Hz
FIDRES 0.183399 Hz
AQ 2.7263477 sec
RG 80.6
DW 83.200 usec
DE 6.50 usec
TE 292.8 K
D1 6.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 13.50 usec
PL1 0.00 dB
PL1W 11.30348873 W
SFO1 400.1329410 MHz
SI 32768
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 ppm
1.00
1.00
1.00
1.01
2.03
2.03
3.06
2.08
2.06
4.38
6.21


193.781
161.166
150.222
147.204
144.810
140.259
132.569
127.836
124.256
102.352
50.642
41.925
31.177
29.544
26.749
20.239
19.716
13.770
13.637
13.770
13.637
14 ppm
NAME S13 13C
EXPNO 1
PROCNO 1
Date_ 20121017
Time 11.21
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg
TD 65536
SOLVENT CDCl3
NS 130
DS 0
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 203
DW 20.800 usec
DE 6.50 usec
TE 293.3 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 7.60 usec
PL1 -2.00 dB
PL1W 56.92932510 W
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 85.00 usec
PL2 0.00 dB
PL12 16.00 dB
PL13 16.00 dB
PL2W 11.30343873 W
PL12W 0.28393081 W
PL13W 0.28393081 W
SFO2 400.1316005 MHz
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200 180 160 140 120 100 80 60 40 20 0 ppm



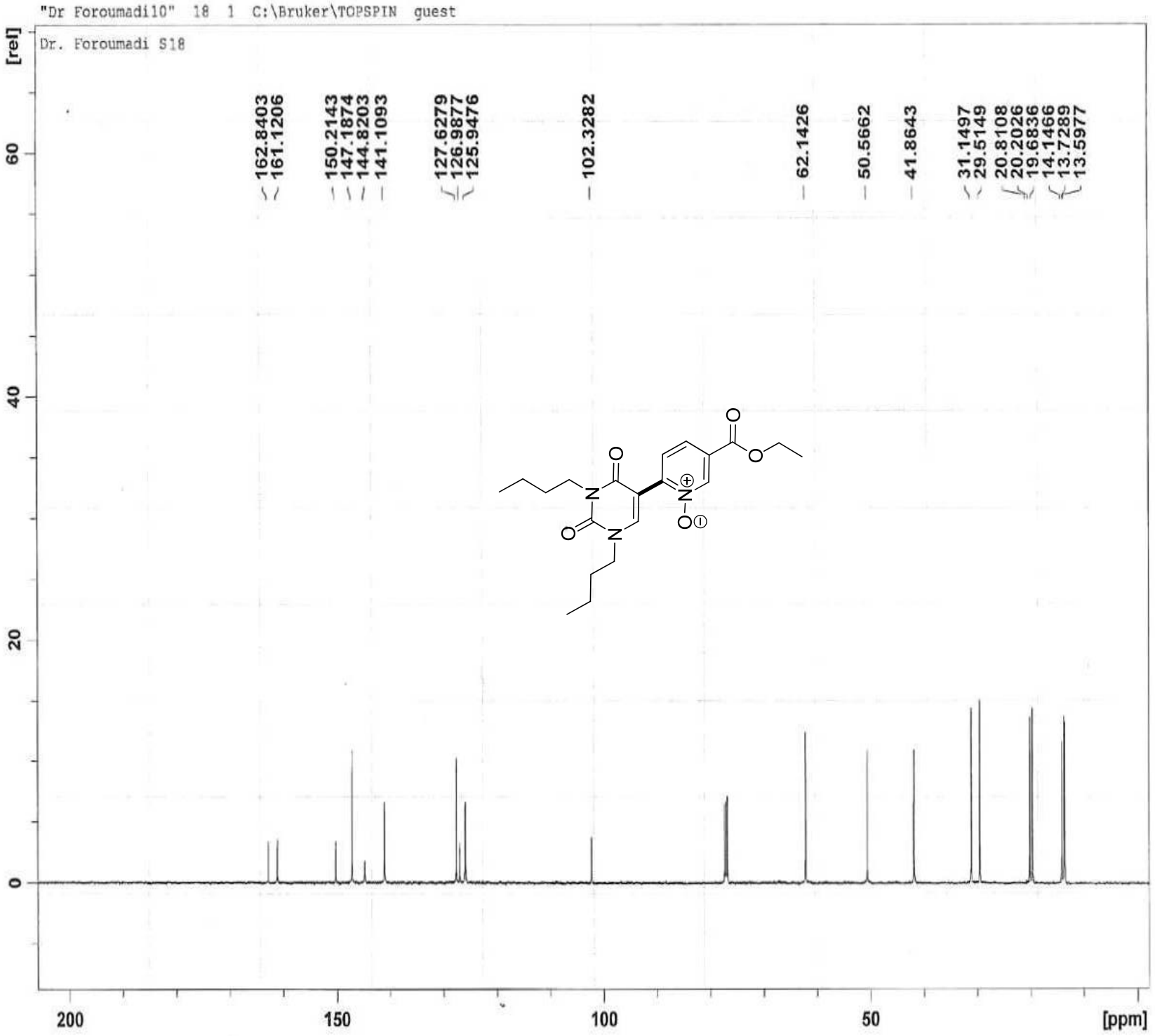
BRUKER

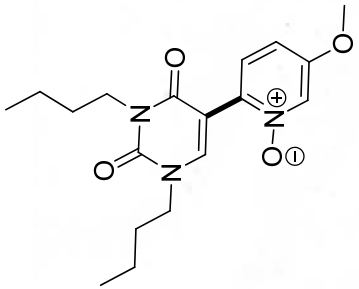
Current Data Parameters
NAME
EXPNO 7
PROCNO 1

F2 - Acquisition Parameters
Date_ 20121128
Time 13.00
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 8
DS 0
SWH 9014.423 Hz
FIDRES 0.275098 Hz
AQ 1.8175818 sec
RG 90.5
DW 55.467 usec
DE 6.50 usec
TE 295.8 K
D1 1.50000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 4.00 usec
PL1 3.00 dB
SFO1 500.1335009 MHz

F2 - Processing parameters
SI 32768
SF 500.1300000 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 150.00


"Dr Foroumadi10" 18 1 C:\Bruker\TOPSPIN guest
Dr. Foroumadi S18
[rel]
60
40
20
0
162.8403
161.1206
150.2143
147.1874
144.8203
141.1093
127.6279
126.9877
125.9476
102.3282
62.1426
50.5662
41.8643
31.1497
29.5149
20.8108
20.2026
19.6836
14.1466
13.7289
13.5977
200
150
100
50
[ppm]



8.803
8.044
7.878
7.860
7.276
6.971
6.956
4.030
4.015
3.999
3.870
3.855
3.840
1.757
1.742
1.727
1.636
1.621
1.412
1.403
1.388
0.985
0.971

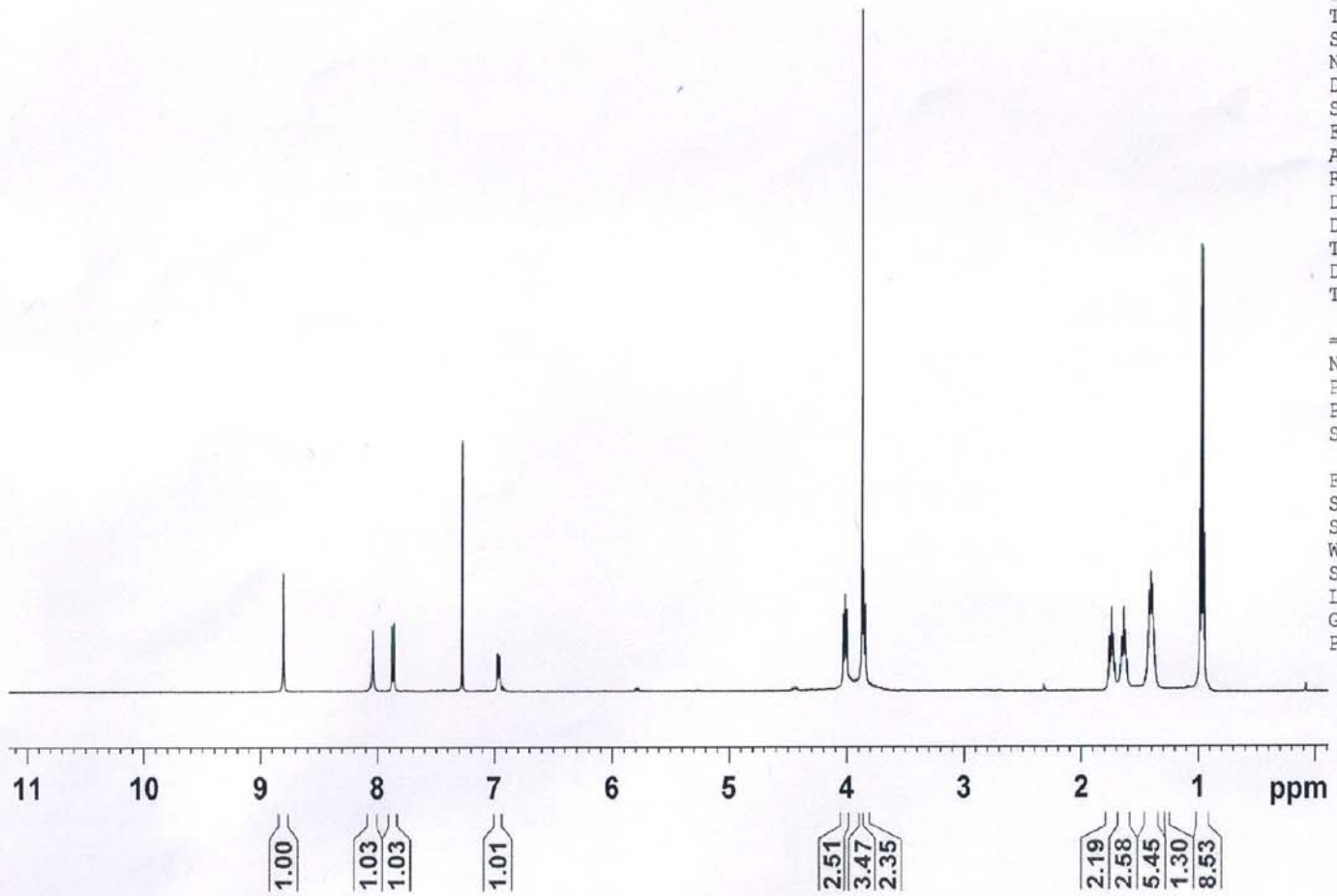
BRUKER

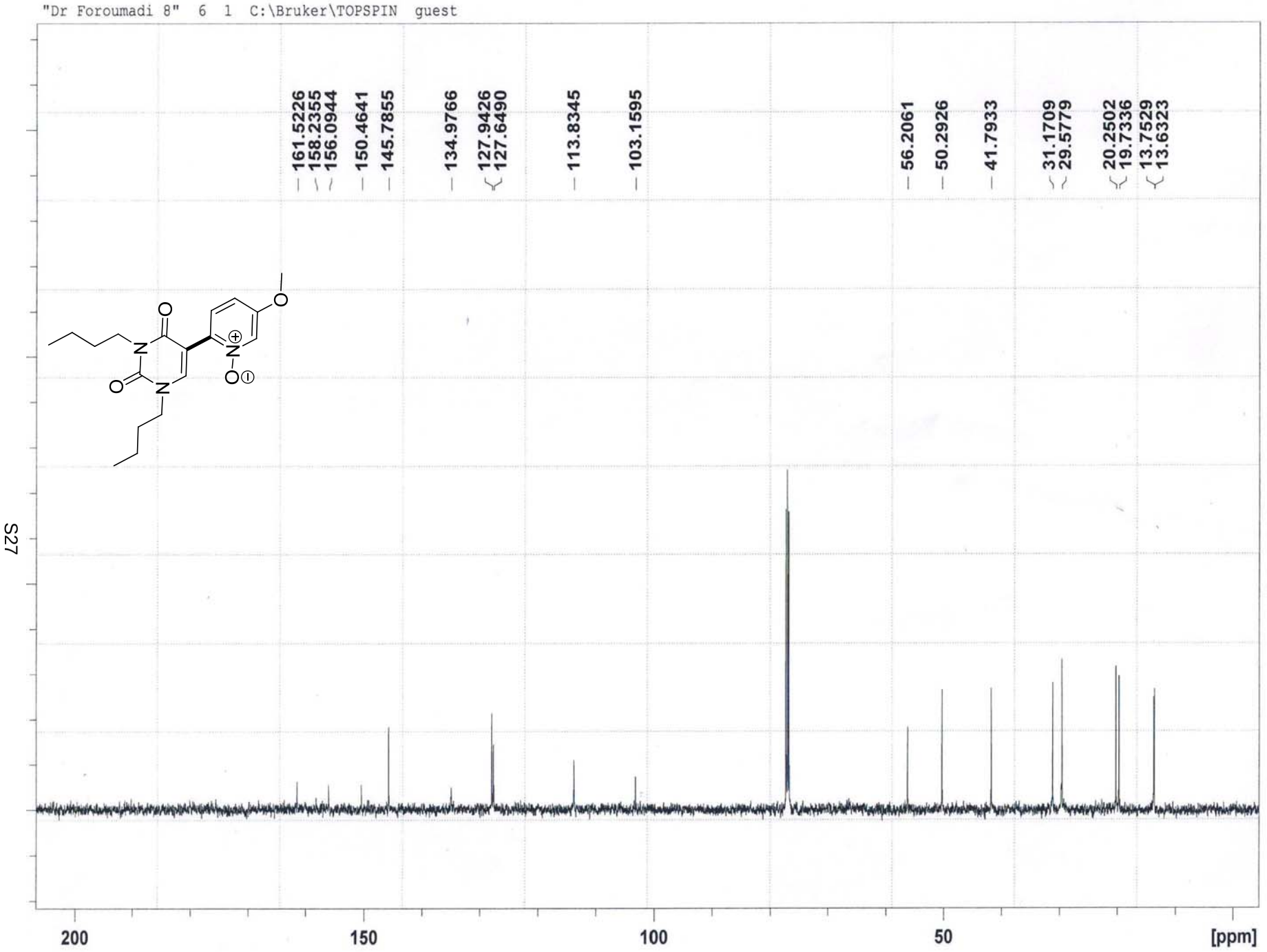
Current Data Parameters
NAME
EXPNO 102
PROCNO 1

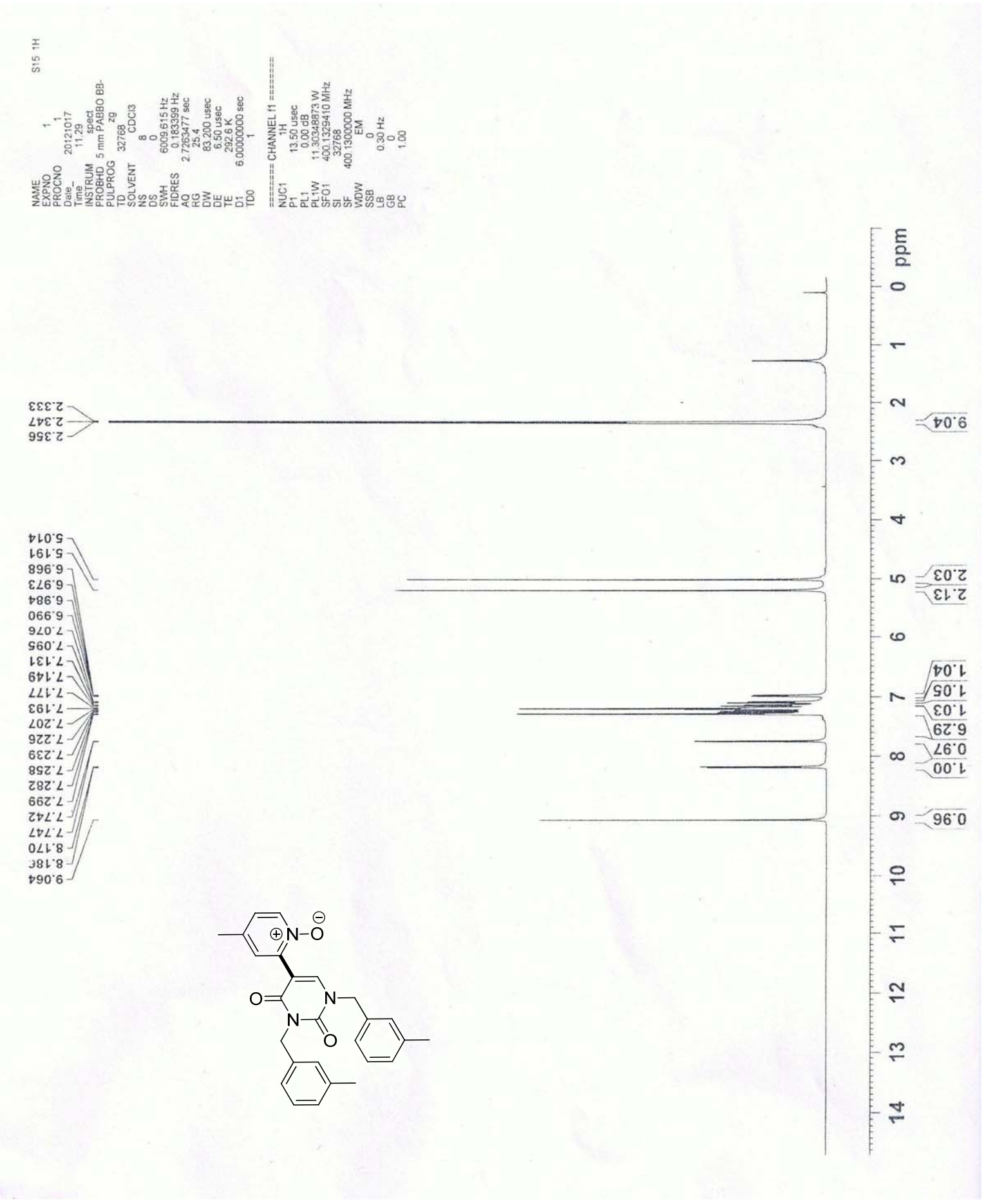
F2 - Acquisition Parameters
Date_ 20121128
Time 12.14
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 8
DS 0
SWH 9014.423 Hz
FIDRES 0.275098 Hz
AQ 1.8175818 sec
RG 90.5
DW 55.467 usec
DE 6.50 usec
TE 295.2 K
D1 1.50000000 sec
TD0 1

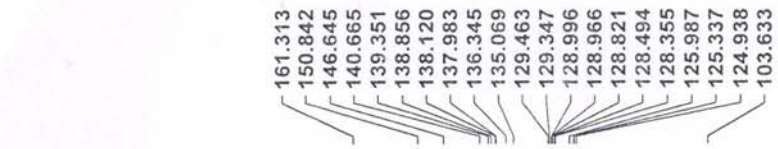
======== CHANNEL f1 ========
NUC1 1H
P1 4.00 usec
PL1 3.00 dB
SFO1 500.1335009 MHz

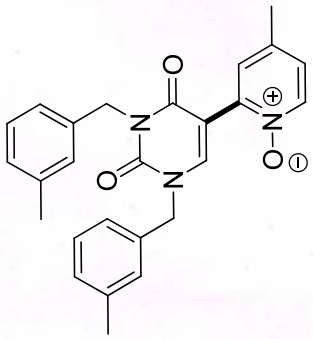
F2 - Processing parameters
SI 32768
SF 500.1300000 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 20.00



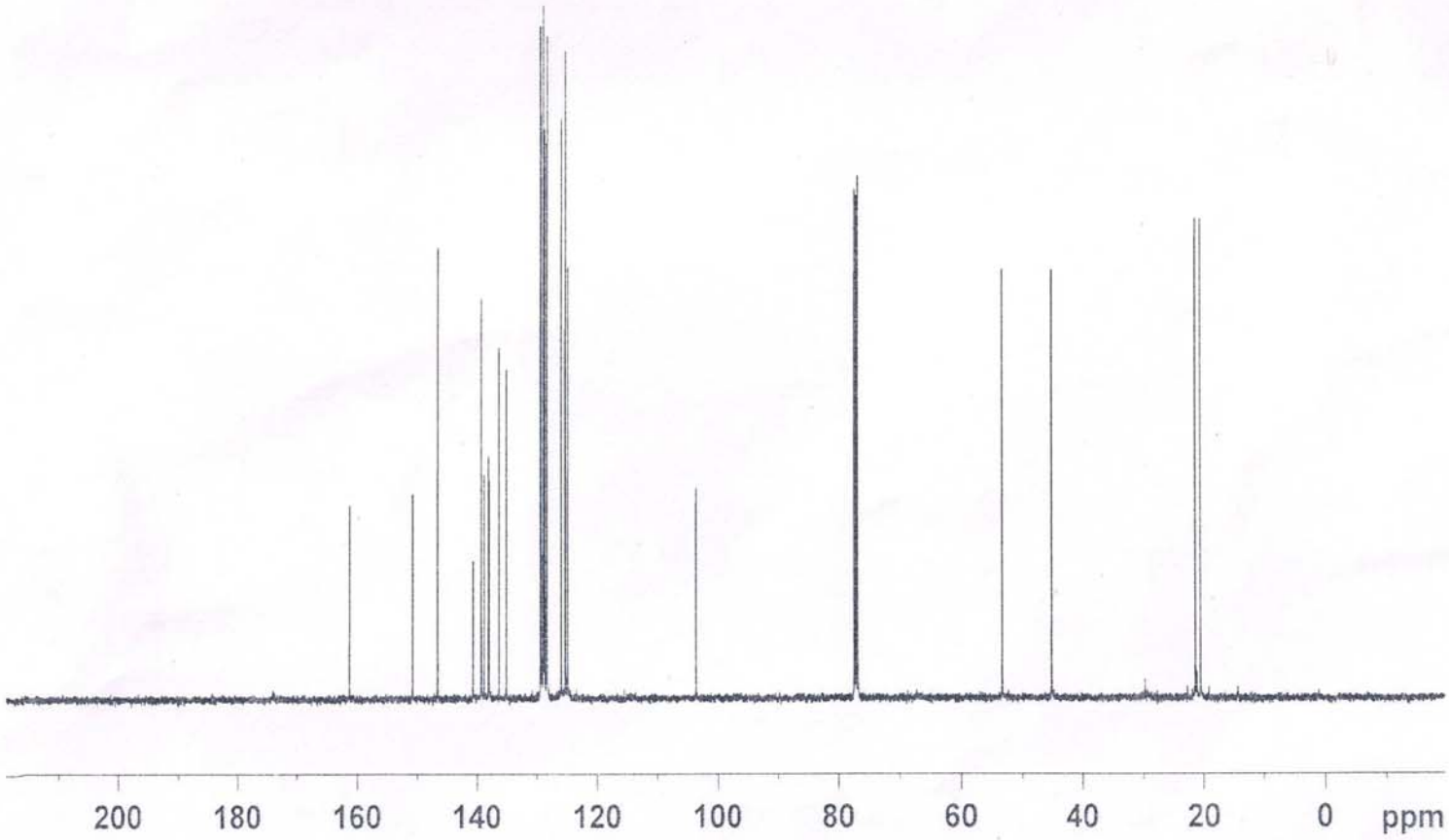

"Dr Foroumadi 8" 6 1 C:\Bruker\TOPSPIN guest
161.5226
158.2355
156.0944
150.4641
145.7855
134.9766
127.9426
127.6490
113.8345
103.1595
56.2061
50.2926
41.7933
31.1709
29.5779
20.2502
19.7336
13.7529
13.6323
200
150
100
50
[ppm]


S15 1H
NAME 1
EXPNO 1
PROCNO 1
Date_ 20121017
Time_ 11.29
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 8
DS 0
SWH 6009.615 Hz
FIDRES 0.183399 Hz
AQ 2.7263477 sec
RG 25.4
DW 83.200 usec
DE 6.50 usec
TE 292.6 K
D1 6.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 13.50 usec
PL1 0.00 dB
PL1W 11.30348873 W
SFO1 400.1329410 MHz
SI 32768
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
2.333
2.347
2.356
5.014
5.191
6.868
6.973
6.984
6.990
7.076
7.095
7.131
7.149
7.177
7.193
7.207
7.226
7.239
7.258
7.282
7.299
7.742
7.747
8.170
8.186
9.064
9.04
2.03
2.13
1.04
1.05
1.03
6.29
0.97
1.00
0.96
ppm
O
N
O
N
N
O

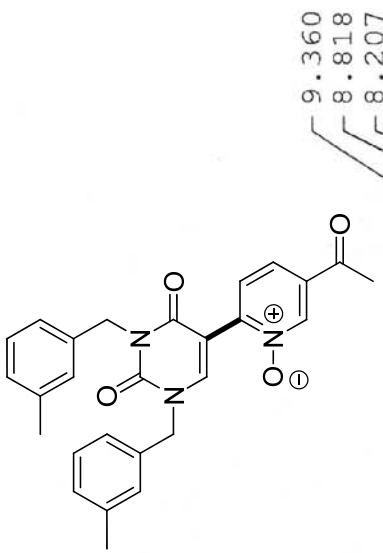




```
NAME                                S15 13C
EXPNO                 1
PROCNO                1
Date_              20121017
Time                12.03
INSTRUM             spect
PROBHD     5 mm PABBO BB-
PULPROG             zgpg
TD                 65536
SOLVENT            CDCl3
NS                   512
DS                     0
SWH            24038.461 Hz
FIDRES          0.366798 Hz
AQ              1.3631988 sec
RG                   203
DW                20.800 usec
DE                 6.50 usec
TE                 293.3 K
D1              2.00000000 sec
D11             0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                7.60 usec
PL1               -2.00 dB
PL1W          56.92932510 W
SFO1          100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                 1H
PCPD2            85.00 usec
PL2               0.00 dB
PL12             16.00 dB
PL13             16.00 dB
PL2W          11.30348873 W
PL12W          0.28393081 W
PL13W          0.28393081 W
SFO2          400.1316005 MHz
SI                 32768
SF            100.6127690 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```



Dr. Foroumadi S25



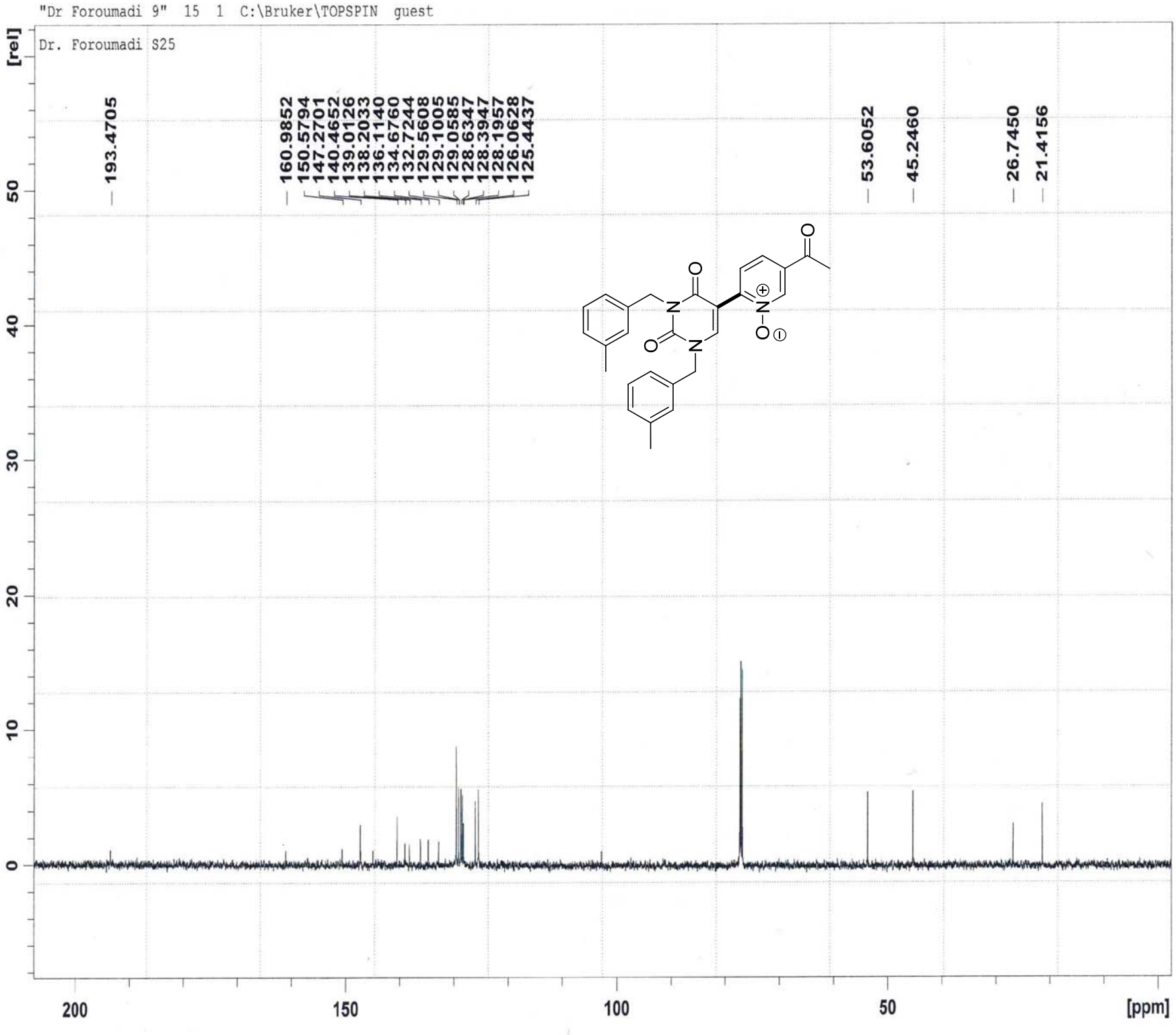
BRUKER

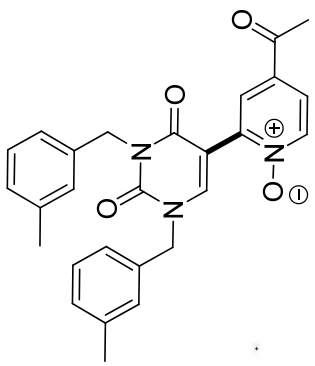
Current Data Parameters
NAME Dr Foroumadi 9
EXPNO 14
PROCNO 1

F2 - Acquisition Parameters
Date_ 20121230
Time 10.23
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 8
DS 0
SWH 9014.423 Hz
FIDRES 0.275098 Hz
AQ 1.8175818 sec
RG 181
DW 55.467 usec
DE 6.50 usec
TE 291.1 K
D1 2.50000000 sec
TD0 1

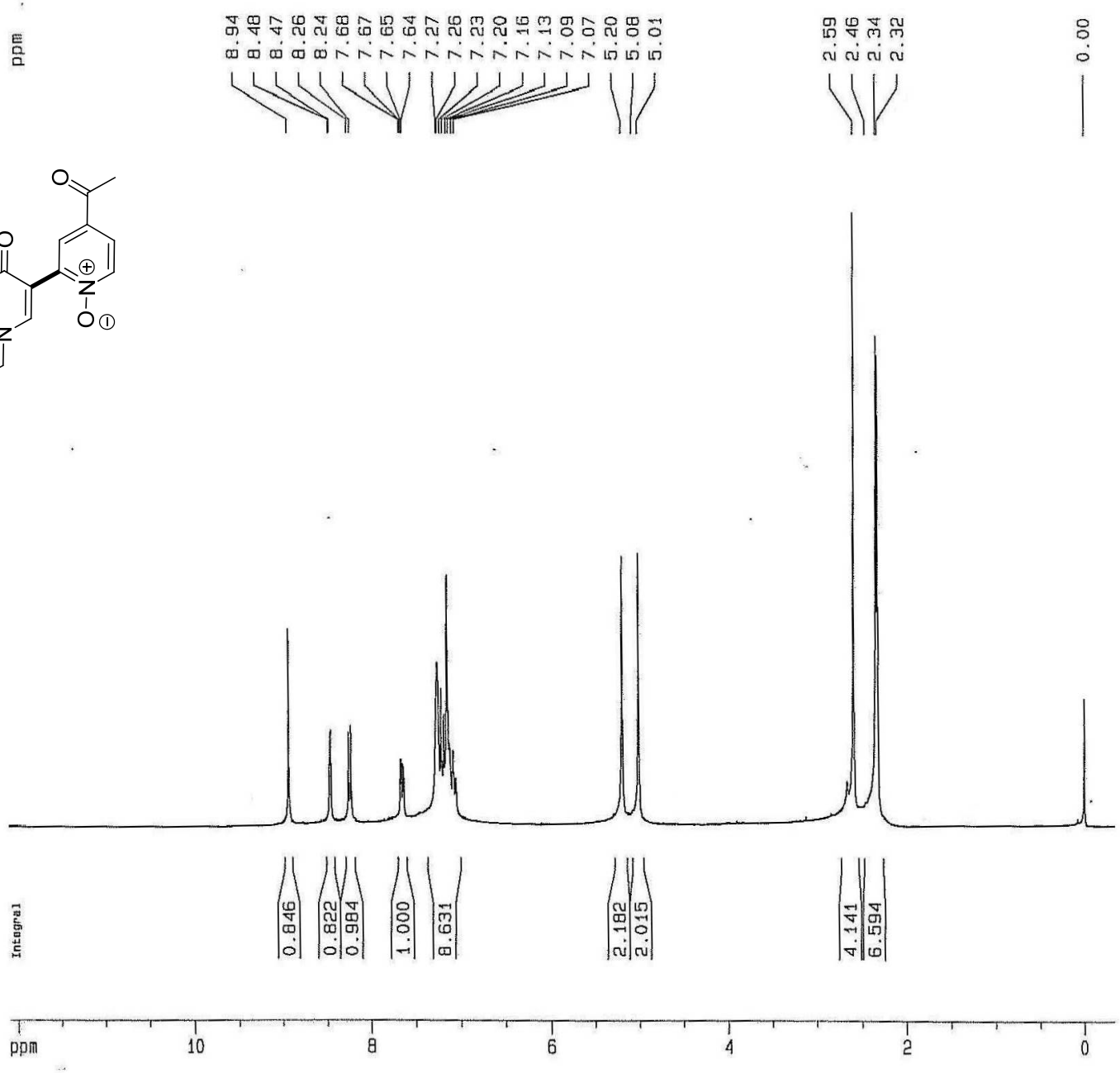
======== CHANNEL f1 ========
NUC1 1H
P1 4.00 usec
PL1 3.00 dB
SFO1 500.1335009 MHz

F2 - Processing parameters
SI 32768
SF 500.1300000 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 28.00


"Dr Foroumadi 9" 15 1 C:\Bruker\TOPSPIN guest
Dr. Foroumadi S25
[rel]
193.4705
160.9852
150.5794
147.2701
140.4652
139.0126
138.2033
136.1140
134.6760
132.7244
129.5608
129.1005
129.0585
128.6347
128.3947
128.1957
126.0628
125.4437
53.6052
45.2460
26.7450
21.4156
0
10
20
30
40
50
200
150
100
50
[ppm]



S32



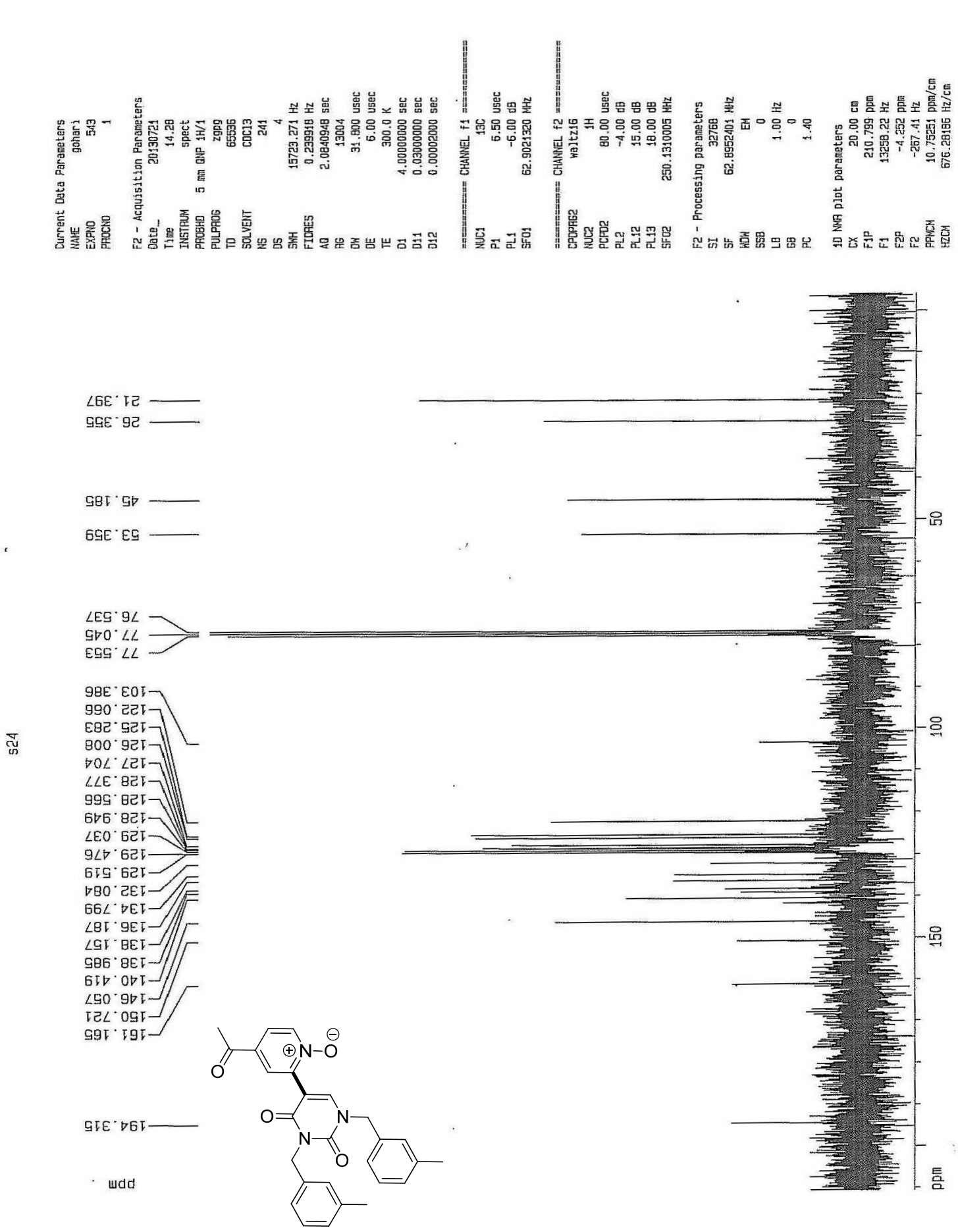
Current Data Parameters
NAME gohari
EXPNO 542
PROCNO 1

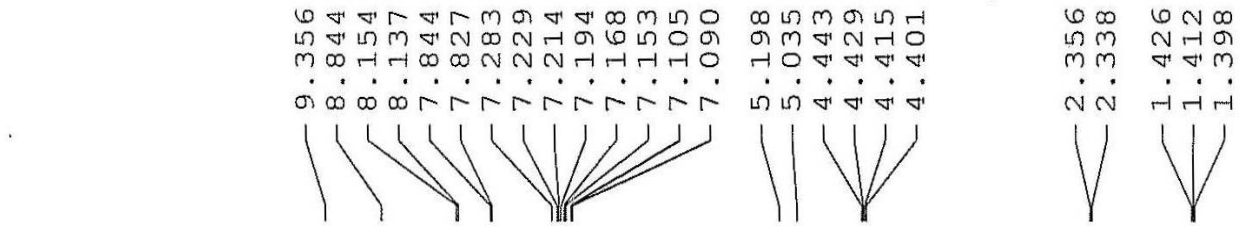
F2 - Acquisition Parameters
Date_ 20130721
Time 14.18
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zg
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 6265.664 Hz
FIDRES 0.095606 Hz
AQ 5.2298226 sec
RG 228.1
DW 79.800 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec

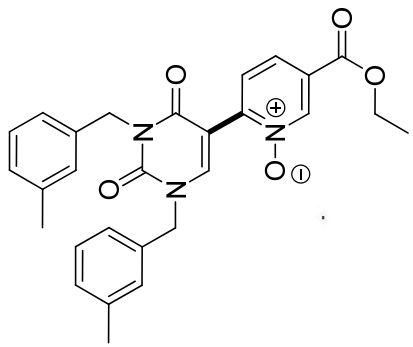
========== CHANNEL f1 ==========
NUC1 1H
P1 10.50 usec
PL1 -4.00 dB
SFO1 250.1315371 MHz

F2 - Processing parameters
SI 32768
SF 250.1300072 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
F1P 12.104 ppm
F1 3027.47 Hz
F2P -0.349 ppm
F2 -87.31 Hz
PPMCM 0.62263 ppm/cm
HZCM 155.73877 Hz/cm


Current Data Parameters
NAME gohari
EXPNO 543
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130721
Time 14.28
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zgpg
TD 65536
SOLVENT CDCl3
NS 241
DS 4
SWH 15723.271 Hz
FIDRES 0.239918 Hz
AQ 2.0840948 sec
RG 13004
DW 31.800 usec
DE 6.00 usec
TE 300.0 K
D1 4.000000000 sec
D11 0.030000000 sec
D12 0.000020000 sec
CHANNEL f1
NUC1 13C
P1 6.50 usec
PL1 -6.00 dB
SFO1 62.9021320 MHz
CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -4.00 dB
PL12 15.00 dB
PL13 18.00 dB
SFO2 250.1310005 MHz
F2 - Processing parameters
SI 32768
SF 62.8952401 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
1D NMR plot parameters
CX 20.00 cm
F1P 210.799 ppm
F1 13258.22 Hz
F2P -4.252 ppm
F2 -267.41 Hz
PPMCM 10.75251 ppm/cm
HZCM 676.28186 Hz/cm
21.397
26.355
45.185
53.359
76.537
77.045
77.553
103.386
122.066
125.283
126.008
127.704
128.377
128.566
128.949
129.037
129.476
129.519
132.084
134.799
136.187
138.157
138.985
140.419
146.057
150.721
161.165
194.315
ppm
50
100
150
ppm
O
N
O
O
N
N
O
s24





Current Data Parameters
NAME Dr Foroumadi10
EXPNO 15
PROCNO 1

F2 - Acquisition Parameters

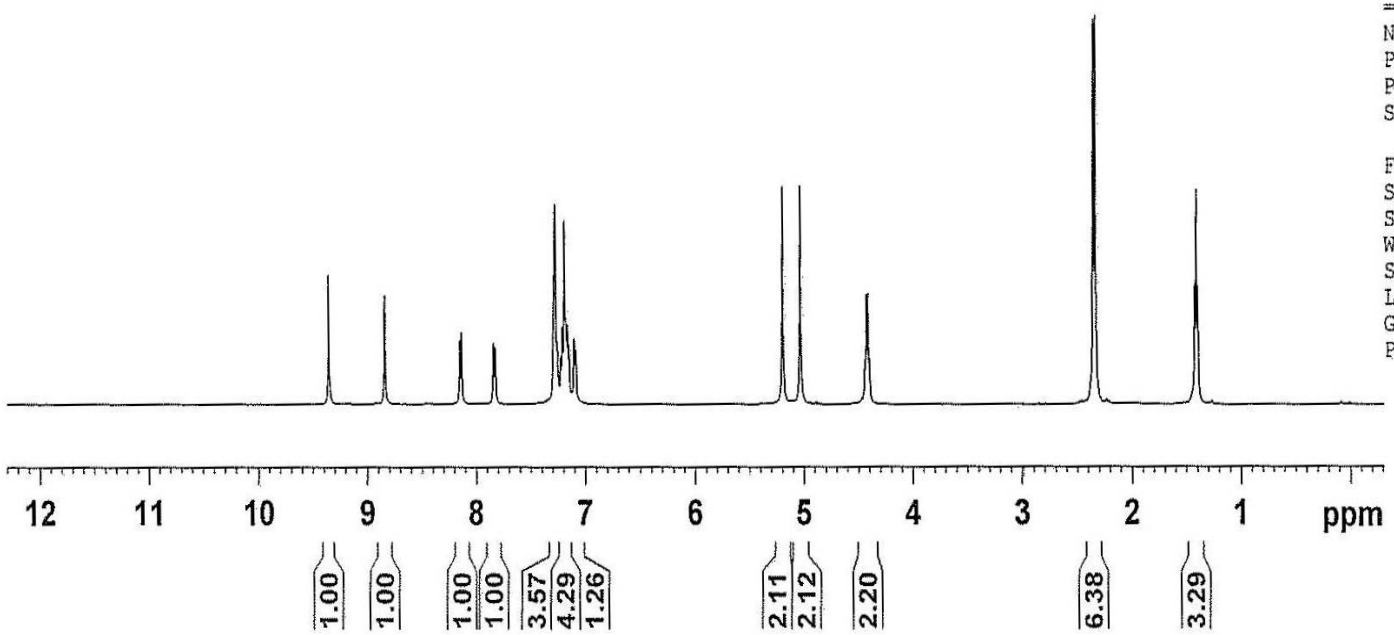
Date_	20130213
Time	8.21
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg
TD	32768
SOLVENT	CDCl3
NS	8
DS	0
SWH	9014.423 Hz
FIDRES	0.275098 Hz
AQ	1.8175818 sec
RG	181
DW	55.467 usec
DE	6.50 usec
TE	292.9 K
D1	2.50000000 sec
TD0	1

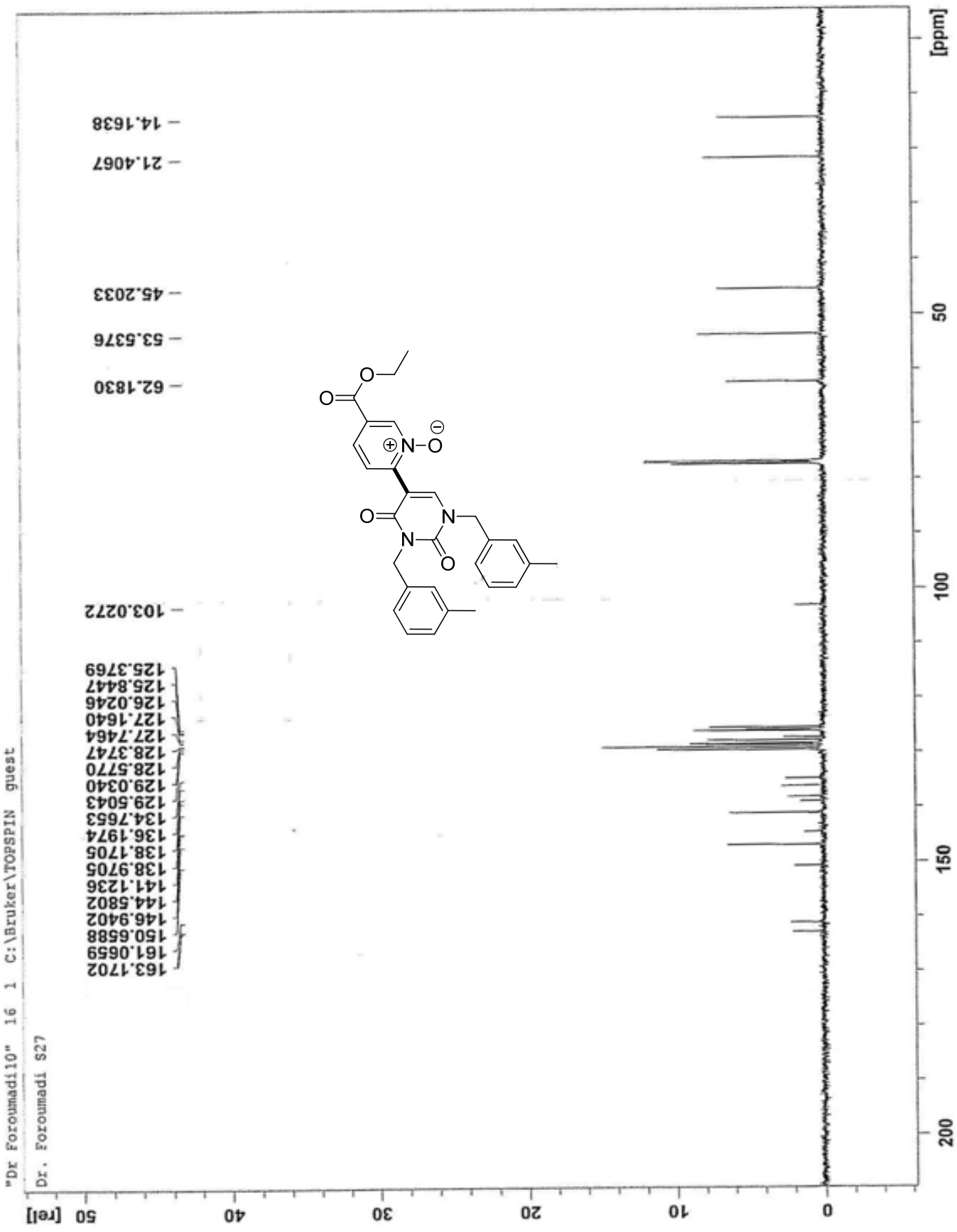
======== CHANNEL f1 ========

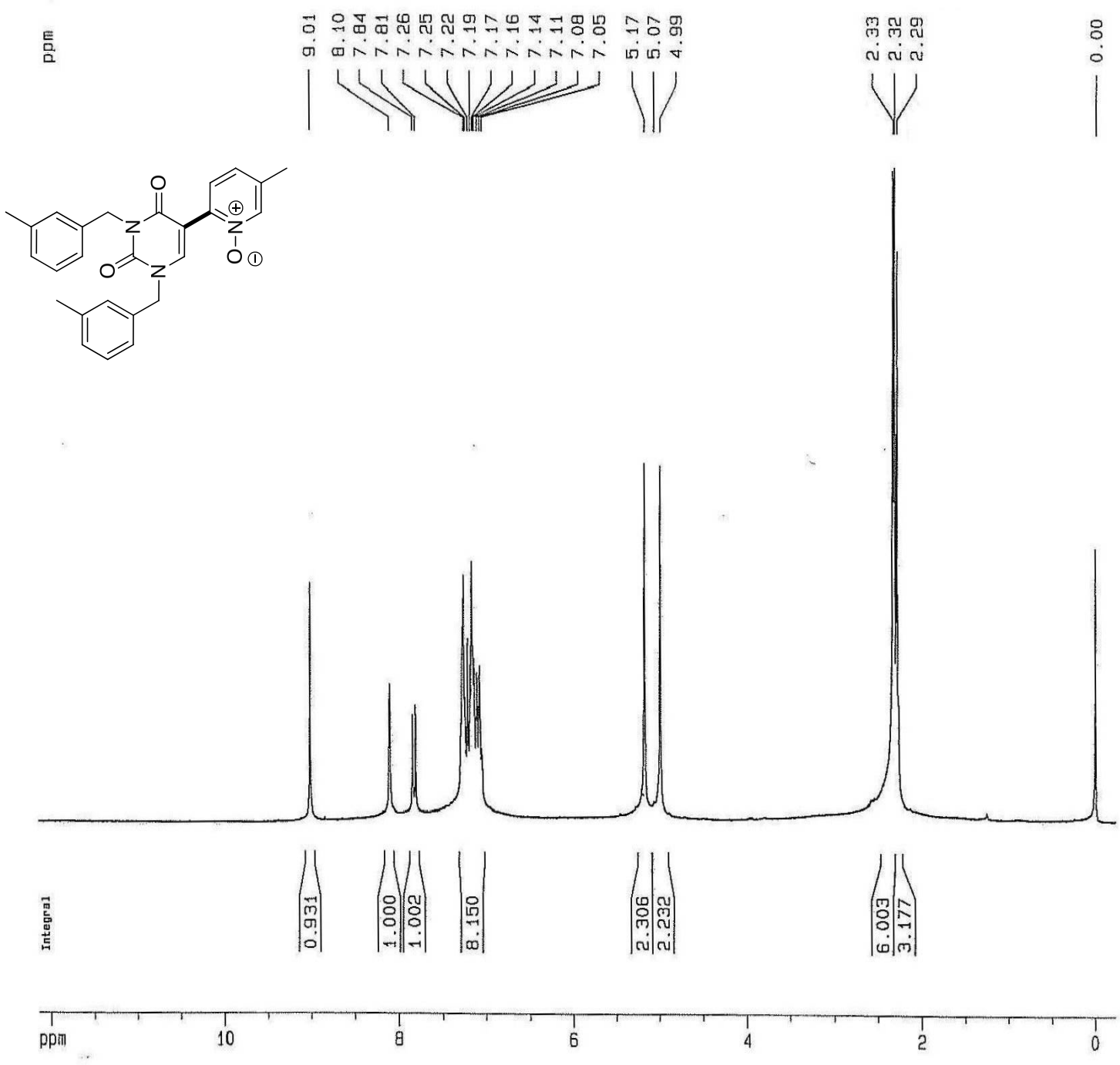
NUC1	1H
P1	4.00 usec
PL1	3.00 dB
SFO1	500.1335009 MHz

F2 - Processing parameters

SI	32768
SF	500.1300000 MHz
WDW	EM
SSB	0
LB	3.00 Hz
GB	0
PC	20.00




"Dr Foroumadi10" 16 1 C:\Bruker\TOPSPIN guest
Dr. Foroumadi S27
14.1638
21.4067
45.2033
53.5376
62.1830
103.0272
125.3769
125.8447
126.0246
127.1640
127.7464
128.3747
128.5770
129.0340
129.5043
134.7653
136.1974
138.1705
138.9705
141.1236
144.5802
146.9402
150.6588
161.0659
163.1702
[rel]
[ppm]



Current Data Parameters

NAME	gohari
EXPNO	544
PROCNO	1

F2 - Acquisition Parameters

Date_	20130721
Time	15.42
INSTRUM	spect
PROBHD	5 mm QNP 1H/1
PULPROG	zg
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	6265.664 Hz
FIDRES	0.095606 Hz
AQ	5.2298226 sec
RG	456.1
DW	79.800 usec
DE	6.00 usec
TE	300.0 K
D1	1.00000000 sec

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

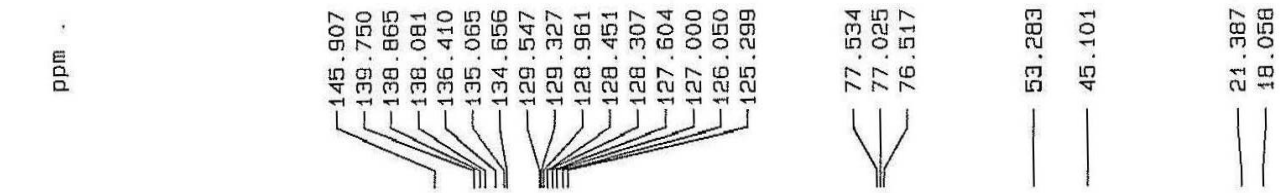
NUC1	1H
P1	10.50 usec
PL1	-4.00 dB
SFO1	250.1315371 MHz

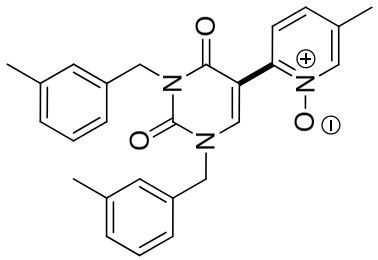
F2 - Processing parameters

SI	32768
SF	250.1300073 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

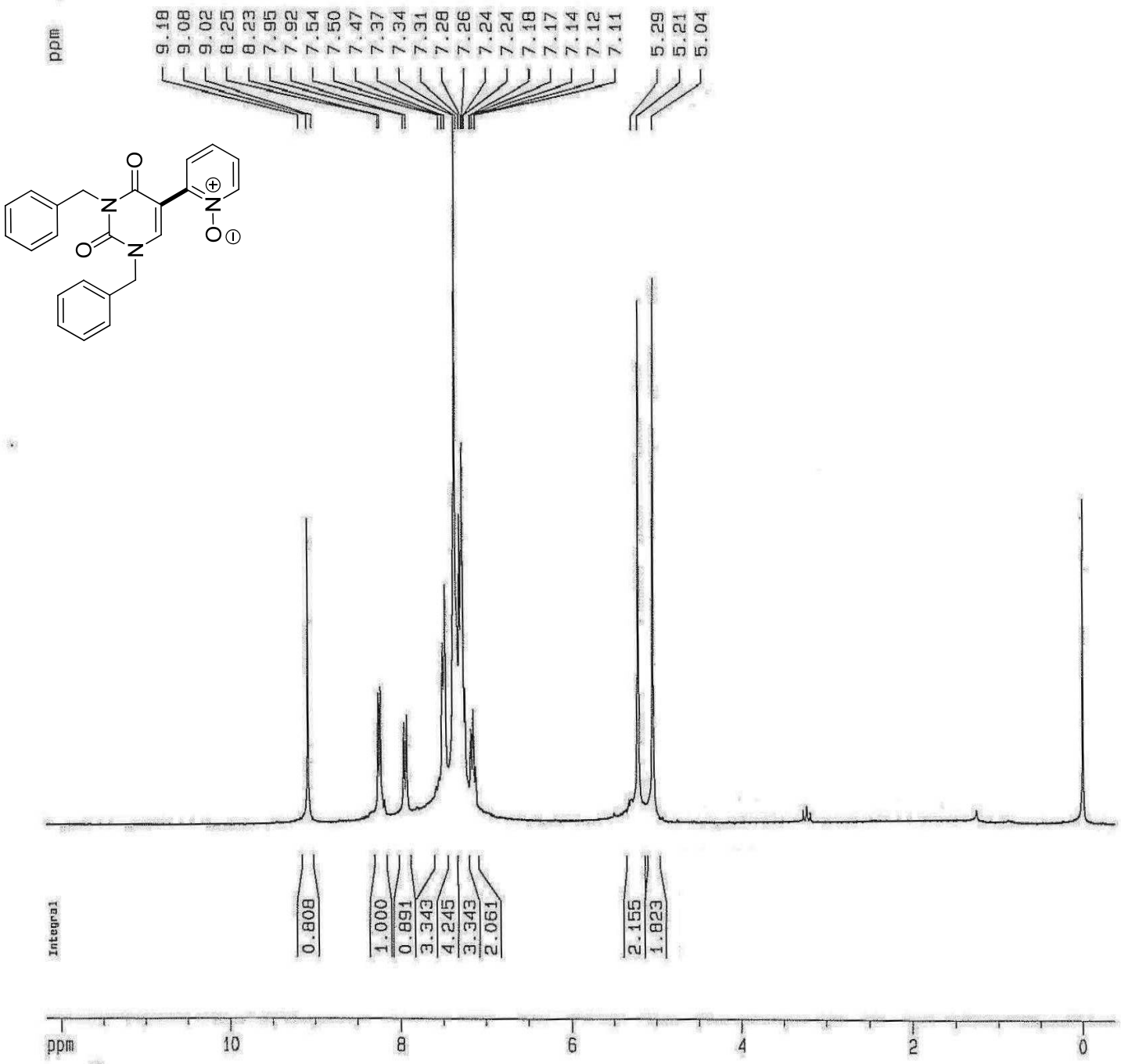
1D NMR plot parameters

CX	20.00 cm
F1P	12.143 ppm
F1	3037.44 Hz
F2P	-0.237 ppm
F2	-59.28 Hz
PPMCM	0.61902 ppm/cm
HZCM	154.83594 Hz/cm






```
Current Data Parameters
NAME                  gohari
EXPNO                    545
PROCNO                     1

F2 - Acquisition Parameters
Date_                20130721
Time                    14.52
INSTRUM                 spect
PROBHD     5 mm QNP 1H/1
PULPROG                 zgpg
TD                     65536
SOLVENT                CDCl3
NS                       469
DS                         4
SWH                15723.271 Hz
FIDRES             0.239918 Hz
AQ                 2.0840948 sec
RG                     13004
DW                    31.800 usec
DE                     6.00 usec
TE                    300.0 K
D1                 4.00000000 sec
D11                0.03000000 sec
D12                0.00002000 sec

========== CHANNEL f1 ==========
NUC1                     13C
P1                     6.50 usec
PL1                   -6.00 dB
SFO1              62.9021320 MHz

========== CHANNEL f2 ==========
CPDPRG2               waltz16
NUC2                      1H
PCPD2                 80.00 usec
PL2                   -4.00 dB
PL12                  15.00 dB
PL13                  18.00 dB
SFO2             250.1310005 MHz

F2 - Processing parameters
SI                       32768
SF                62.8952401 MHz
WDW                         EM
SSB                          0
LB                    1.00 Hz
GB                           0
PC                        1.40

1D NMR plot parameters
CX                    20.00 cm
F1P                  206.116 ppm
F1                 12963.69 Hz
F2P                   -4.612 ppm
F2                  -290.07 Hz
PPMCM                10.53638 ppm/cm
HZCM                662.68829 Hz/cm
```



Current Data Parameters

NAME	gohari
EXPNO	548
PROCNO	1

F2 - Acquisition Parameters

Date_	20130721
Time	18.00
INSTRUM	spect
PROBHD	5 mm GNP 1H/1
PULPROG	zg
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	6265.664 Hz
FIDRES	0.095606 Hz
AQ	5.2298226 sec
RG	574.7
DW	79.800 usec
DE	6.00 usec
TE	300.0 K
D1	1.00000000 sec

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

NUC1	1H
P1	10.50 usec
PL1	-4.00 dB
SFO1	250.1315371 MHz

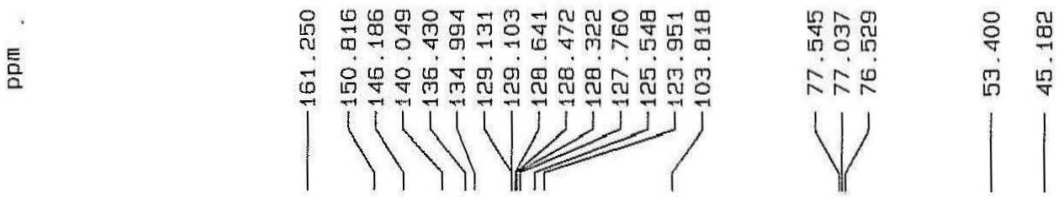
F2 - Processing parameters

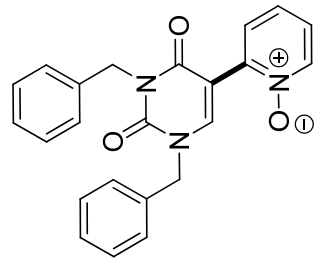
SI	32768
SF	250.1300072 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

1D NMR plot parameters

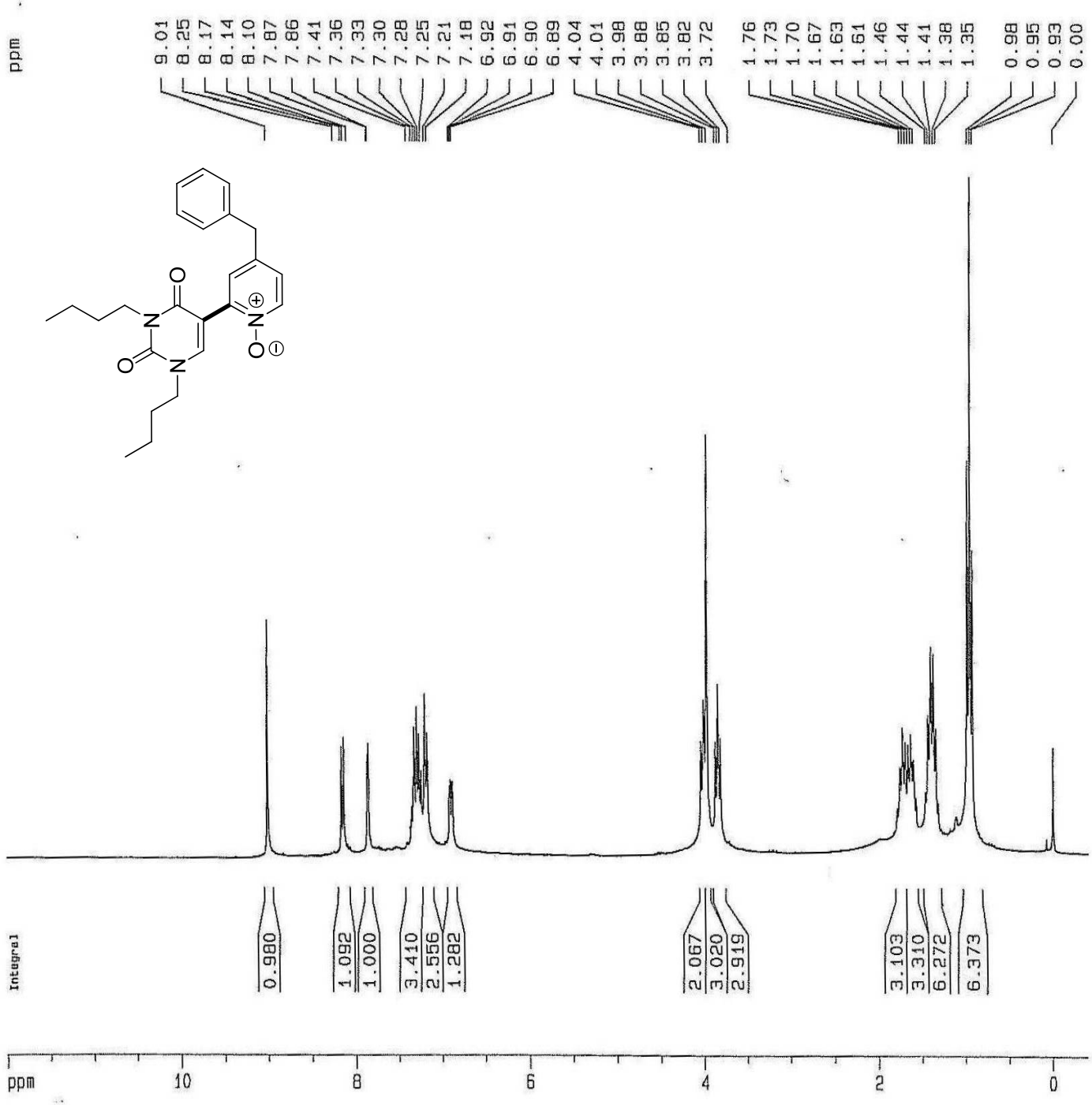
CX	20.00 cm
F1P	12.176 ppm
F1	3045.53 Hz
F2P	-0.385 ppm
F2	-96.33 Hz
PPMCM	0.62805 ppm/cm
HZCM	157.09302 Hz/cm

519






```
Current Data Parameters
NAME                  gohari
EXPNO                    549
PROCNO                     1

F2 - Acquisition Parameters
Date_              20130721
Time                  17.09
INSTRUM               spect
PROBHD     5 mm QNP 1H/1
PULPROG                zgpg
TD                    65536
SOLVENT               CDCl3
NS                      550
DS                        4
SWH               15723.271 Hz
FIDRES            0.239918 Hz
AQ                2.0840948 sec
RG                    13004
DW                  31.800 usec
DE                   6.00 usec
TE                  300.0 K
D1                4.00000000 sec
D11               0.03000000 sec
D12               0.00002000 sec

========== CHANNEL f1 ==========
NUC1                    13C
P1                   6.50 usec
PL1                 -6.00 dB
SFO1              62.9021320 MHz

========== CHANNEL f2 ==========
CPDPRG2              waltz16
NUC2                    1H
PCPD2               80.00 usec
PL2                 -4.00 dB
PL12                15.00 dB
PL13                18.00 dB
SFO2             250.1310005 MHz

F2 - Processing parameters
SI                     32768
SF                52.8952396 MHz
WDW                       EM
SSB                        0
LB                    1.00 Hz
GB                         0
PC                     1.40

1D NMR plot parameters
CX                   20.00 cm
F1P                 210.078 ppm
F1                13212.91 Hz
F2P                 -3.171 ppm
F2                 -199.45 Hz
PPMCM               10.66245 ppm/cm
HZCM               670.61786 Hz/cm
```



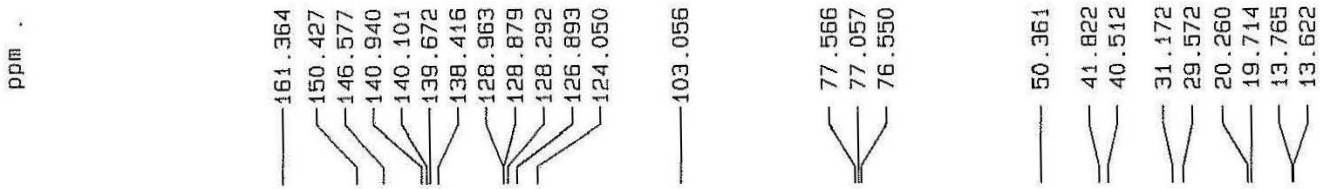
Current Data Parameters
NAME gohari
EXPNO 545
PROCNO 1

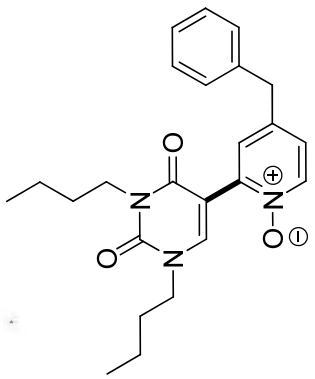
F2 - Acquisition Parameters
Date_ 20130721
Time 15.56
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zg
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 6265.664 Hz
FIDRES 0.095606 Hz
AQ 5.2298226 sec
RG 181
DW 79.800 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec

========== CHANNEL f1 ==========
NUC1 1H
P1 10.50 usec
PL1 -4.00 dB
SFO1 250.1315371 MHz

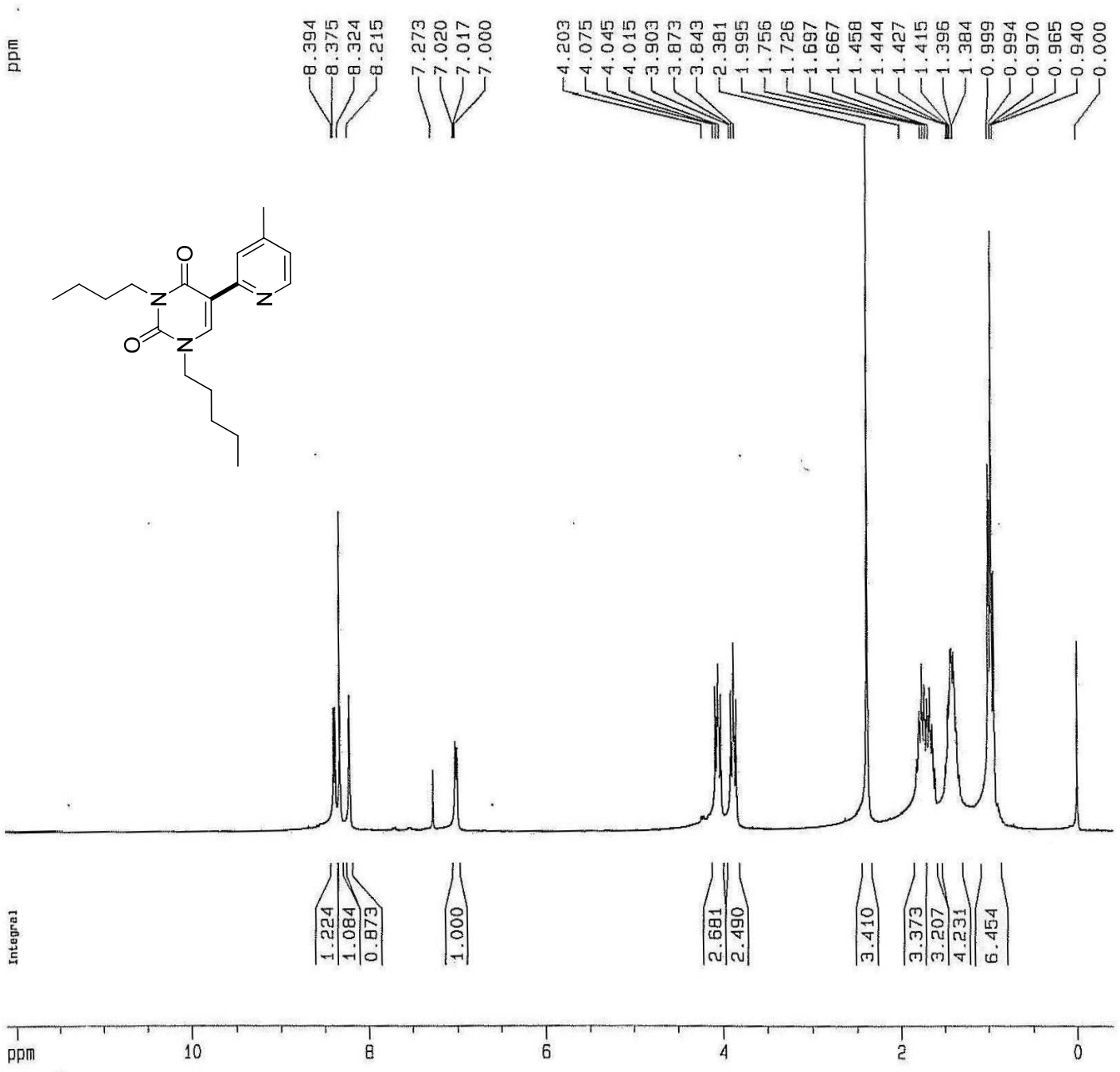
F2 - Processing parameters
SI 32768
SF 250.1300033 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
F1P 12.015 ppm
F1 3005.34 Hz
F2P -0.401 ppm
F2 -100.41 Hz
PPMCM 0.62083 ppm/cm
HZCM 155.28735 Hz/cm






```
Current Data Parameters
NAME                  gohari
EXPNO                    547
PROCNO                     1

F2 - Acquisition Parameters
Date_              20130721
Time                  16.54
INSTRUM               spect
PROBHD     5 mm QNP 1H/1
PULPROG               zgpg
TD                    65536
SOLVENT               CDCl3
NS                      551
DS                        4
SWH               15723.271 Hz
FIDRES            0.239918 Hz
AQ                2.0840948 sec
RG                    13004
DW                   31.800 usec
DE                    6.00 usec
TE                   300.0 K
D1                4.00000000 sec
D11               0.03000000 sec
D12               0.00002000 sec

========== CHANNEL f1 ==========
NUC1                    13C
P1                    6.50 usec
PL1                  -6.00 dB
SFO1             62.9021320 MHz

========== CHANNEL f2 ==========
CPDPRG2              waltz16
NUC2                    1H
PCPD2                80.00 usec
PL2                  -4.00 dB
PL12                 15.00 dB
PL13                 18.00 dB
SFO2            250.1310005 MHz

F2 - Processing parameters
SI                      32768
SF               62.8952401 MHz
WDW                       EM
SSB                        0
LB                    1.00 Hz
GB                         0
PC                     1.40

1D NMR plot parameters
CX                   20.00 cm
F1P                202.874 ppm
F1                 12759.79 Hz
F2P                 -3.531 ppm
F2                 -222.10 Hz
PPMCM              10.32025 ppm/cm
HZCM               649.09473 Hz/cm
```



Current Data Parameters

NAME	gohari
EXPNO	540
PROCNO	1

F2 - Acquisition Parameters

Date_	20130721
Time	15.48
INSTRUM	spect
PROBHD	5 mm GNP 1H/1
PULPROG	zg
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	6265.664 Hz
FIDRES	0.095606 Hz
AQ	5.2298226 sec
RG	203.2
DW	79.800 usec
DE	6.00 usec
TE	300.0 K
D1	1.00000000 sec

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

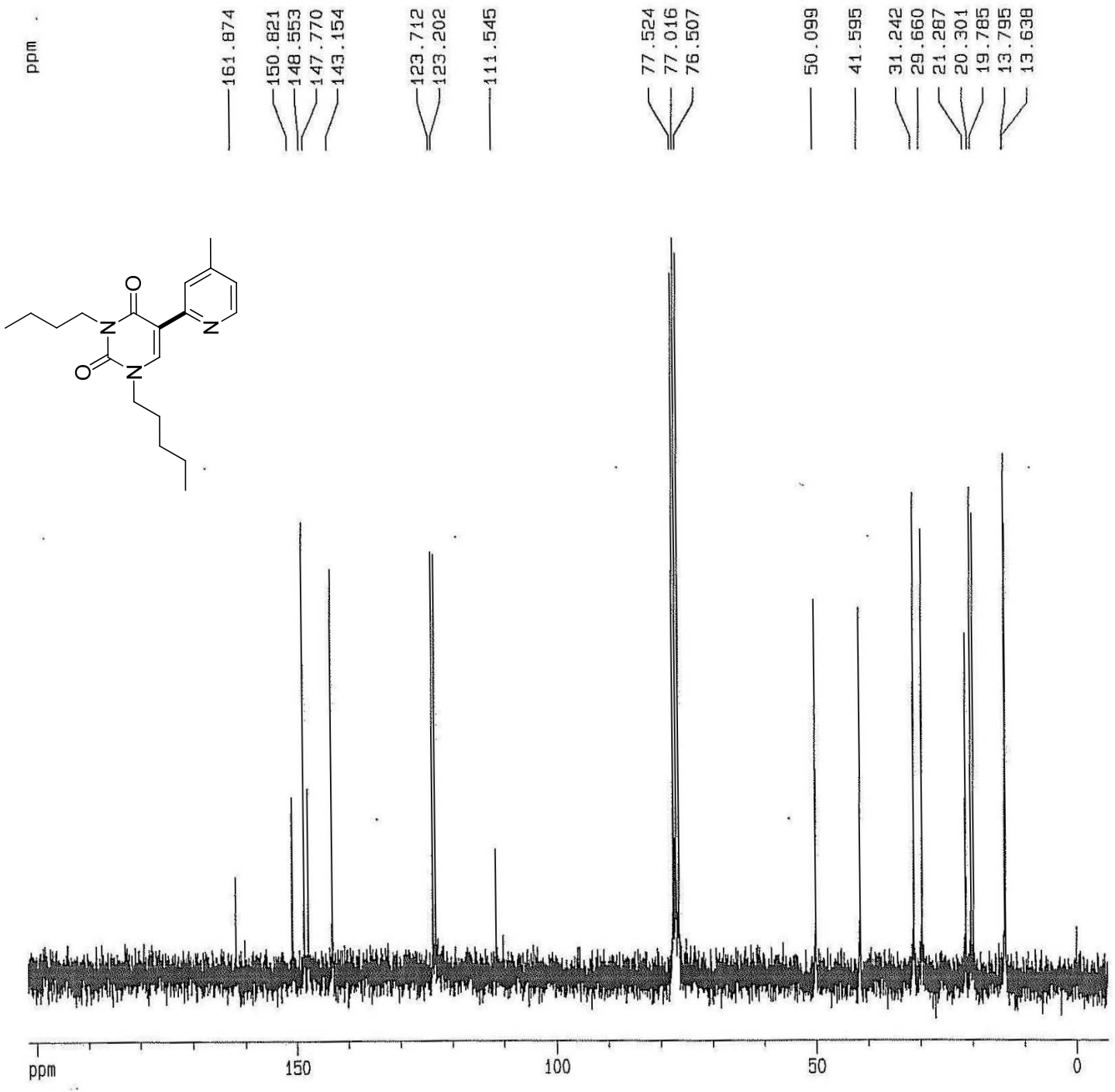
NUC1	1H
P1	10.50 usec
PL1	-4.00 dB
SFO1	250.1315371 MHz

F2 - Processing parameters

SI	32768
SF	250.1300043 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

1D NMR plot parameters

CX	20.00 cm
F1P	12.120 ppm
F1	3031.47 Hz
F2P	-0.405 ppm
F2	-101.37 Hz
PPMCM	0.62624 ppm/cm
HZCM	156.64160 Hz/cm



Current Data Parameters

NAME	gohari
EXPNO	541
PROCNO	1

F2 - Acquisition Parameters

Date_	20130721
Time	13.53
INSTRUM	spect
PROBHD	5 mm QNP 1H/1
PULPROG	zgpg
TD	65536
SOLVENT	CDCl3
NS	523
DS	4
SWH	15723.271 Hz
FIDRES	0.239918 Hz
AQ	2.0840948 sec
RG	13004
DW	31.800 usec
DE	6.00 usec
TE	300.0 K
D1	4.00000000 sec
D11	0.03000000 sec
D12	0.00002000 sec

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

NUC1	13C
P1	6.50 usec
PL1	-6.00 dB
SFO1	62.9021320 MHz

========== CHANNEL f2 ==========

CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	-4.00 dB
PL12	15.00 dB
PL13	18.00 dB
SFO2	250.1310005 MHz

F2 - Processing parameters

SI	32768
SF	62.8952401 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

1D NMR plot parameters

CX	20.00 cm
F1P	201.793 ppm
F1	12691.82 Hz
F2P	-6.053 ppm
F2	-380.69 Hz
PPMCM	10.39229 ppm/cm
HZCM	653.62592 Hz/cm