Synthesis of new bridgehead substituted azabicyclo-[2.2.1]heptane and –[3.3.1]nonane derivatives as potent and selective α7 nicotinic ligands.

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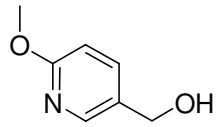
SUPPORTING INFORMATION

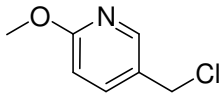
EXPERIMENTAL PROCEDURES

General. Unless otherwise indicated, all starting materials, reagents and catalysts were obtained from commercial suppliers and used without further purification. Anhydrous tetrahydrofuran (THF), toluene, dimethoxyethane (DME), methanol (MeOH), ethanol (EtOH), dichloromethane (CH_2Cl_2), chloroform ($CHCl_3$), ethylacetate (AcOEt) were purchased with quality standards for analysis from Carlo-Erba. Acetonitrile (MeCN) was purchased with quality standards for HPLC from Carlo-Erba. Prior to use, THF, toluène, and DME were subsequently dried over activated molecular sieves (4 Å). Reactions involving air or moisture sensitive reagents or intermediates were carried out under an inert atmosphere of nitrogen or argon in glassware that had been oven dried followed by nitrogen purge. Reaction temperatures are reported as the temperature of the bath surrounding the vessel, unless

otherwise indicated. Flash chromatography was conducted according to the established Still protocol[1] using Combiflash® Companion™ apparatus with the appropriate size Redisep® Rf disposable columns of normal phase silica with the indicated solvents. ^{1}H and ^{13}C (or J-MOD) NMR spectra were recorded on Brüker DPX400, AV400 and AV500. All NMR spectra were taken in deuterated chloroform ($CDCl_3$) or deuterated dimethylsulfoxide (DMSO-d_6) unless otherwise noted. Chemical shifts (δ) are expressed as ppm referenced to the residual solvent (i.e. chloroform, ^{1}H δ 7.24 ppm, ^{13}C δ 77.1 ppm and dimethylsulfoxide, ^{1}H δ 2.50 ppm, ^{13}C δ 39.5 ppm). Splitting patterns are expressed as s, singlet; d, doublet; t, triplet; q, quartet; p, pentet; m, multiplet; comp, overlapping multiplets of magnetically nonequivalent protons; br, broad; app, apparent. IR spectra were recorded on ThermoNicolet Avatar 330 FT IR on film or in solution with the indicated solvent.



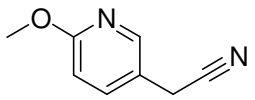
(6-Methoxy-pyridin-3-yl)-methanol (4). In a 2 liters three-necked flask equiped with an addition funnel, a thermometer and an argon entry, at -20°C, to a solution of 6-methoxy-nicotinic acid methyl ester (**3**) (45.29 g, 270.7 mmol) in dichloromethane (900 mL) was added dropwise diisobutylaluminium hydride 1.19 M solution in toluene (499 mL, 596 mmol) in a 3 hours period. The mixture was stirred 2 additionnal hours at -20°C, then, at 0°C, 150 mL of methanol were added dropwise followed by 300 mL of water. The resulting mixture was stirred overnight at room temperature. The layers were then separated, and the aqueous layer was washed with dichloromethane (3 x 200 mL). The combined organic layers were washed with brine (500 mL), dried over sodium sulfate and concentrated under reduced pressure. 35.88 g (95% yield) of desired compound **4** was thus isolated as a pale yellow oil and was engaged in the following step without purification. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.07 (d, *J* = 2.4 Hz, 1 H), 7.58 (dd, *J* = 8.5, 2.4 Hz, 1 H), 6.71 (d, *J* = 8.5 Hz, 1 H), 4.59 (s, 2 H), 3.90 (s, 3 H), 1.92 (br s, 1 H) ; J-MOD ^{13}C NMR (100 MHz, $CDCl_3$) δ 163.9, 145.7, 138.4, 129.0, 110.9, 62.5, 53.5 ; IR (film, cm^{-1}) 3346, 1611, 1574, 1496, 1393, 1291, 1026, 831 ; mass spectrum EI+ *m/z* 139.0628 [$C_7H_9NO_2$ (M+) requires 139.0633].



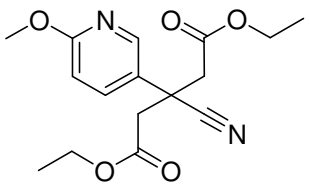
5-Chloromethyl-2-methoxy-pyridine (5). At 0°C, to a solution of (6-methoxy-pyridin-3-yl)-methanol **4** (58 g, 416.8 mmol) in toluene (480 mL), 39.5 mL (64.46 g, 541.8 mmol) of thionyl chloride

(1) Still, W. C. ; Kahn, M. ; Mitra, A. *J. Org. Chem.* **1978**, *43*, 2923-2925.

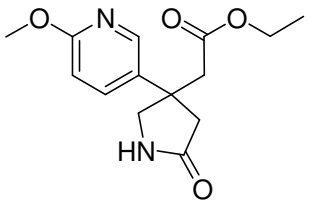
were added dropwise, maintaining temperature around 0°C. The resulting mixture was then stirred at room temperature overnight. After one night, the mixture was concentrated under reduced pressure and the resulting residue was diluted with 400 mL of dicholoromethane and washed carrefully with a saturated aqueous solution of potassium carbonate. The aqueous layer was then washed with dichloromethane (3 x 150 mL) and the combined organic layers were dried on sodium sulfate and concentrated under reduced pressure providing 65.7g (quantitative yield) of the desired product **5** as pale yellow oil and was engaged in the next step without purification. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.12 (d, *J* = 2.4 Hz, 1 H), 7.59 (dd, *J* = 8.5, 2.4 Hz, 1 H), 6.72 (d, *J* = 8.5 Hz, 1 H), 4.52 (s, 2 H), 3.92 (s, 3 H) ; J-MOD ^{13}C NMR (100 MHz, $CDCl_3$) δ 164.2, 146.7, 139.2, 126.1, 111.2, 53.6, 43.3 ; IR (film, cm^{-1}) 3015, 2983, 2947, 2900, 1613, 1573, 1495, 1391, 1311, 1293, 1258, 1025, 832, 757, 685 ; mass spectrum EI+ *m/z* 157.0302 [C_7H_8ClNO (M+) requires 157.02944].



(6-Methoxy-pyridin-3-yl)-acetonitrile (6). At room temperature, to a solution of 5-chloromethyl-2-methoxy-pyridine **5** (33 g, 209.4 mmol) in ethanol (280 mL) was added dropwise a solution of potassium cyanide (19.1 g ; 293.3 mmol) in water (140 mL). The resulting mixture was then stirred under reflux overnight (oil bath temperature : 100°C). After one night, the mixture was concentrated under reduced pressure and the resulting residue was diluted with a saturated aqueous solution of $NaHCO_3$ (300 mL). The aqueous layer was washed with dichloromethane (3 x 300 mL) and the combined organic layers were dried over sodium sulfate and concentrated under reduced pressure. The residue was purified *via* flash chromatography eluting with $CHCl_3$/MeOH/NH_4OH (in a 99/1/0.1 ratio) to yield 22.4 g (72%) of **6** as a colorless oil which was triturated in cyclohexane to give a white powder. ^{1}H NMR (400 MHz, DMSO-d_6) δ 8.13 (dd, *J* = 2.5, 0.6 Hz, 1 H), 7.69 (dd, *J* = 8.5, 2.5 Hz, 1 H), 6.85 (d, *J* = 8.5 Hz, 1 H), 3.97 (s, 2 H), 3.84 (s, 3 H) ; J-MOD ^{13}C NMR (100 MHz, DMSO-d_6) δ 163.1, 146.1, 139.1, 120.3, 119.0, 110.8, 53.2, 19.0 ; IR (CH_2Cl_2, cm^{-1}) 3055, 3015, 2943, 2845, 2255, 1612, 1575, 1493, 1394, 1291 ; mp 60-61°C ; mass spectrum CI+ *m/z* 149.0709 [$C_8H_9N_2O$ (M+1) requires 149.0715].

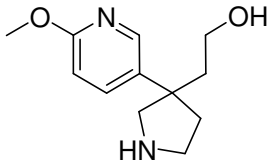


3-Cyano-3-(6-methoxy-pyridin-3-yl)-pentanedioic acid diethyl ester (7). In a 1 liter three-necked flask equiped with an addition funnel, a thermometer and an argon entry, at -78°C, to 400 mL of anhydrous THF were added 202.5 mL of a 2 M solution of LDA (405 mmol,) in THF, followed by a solution of (6-methoxy-pyridin-3-yl)-acetonitrile **6** (20 g, 135 mmol) in THF (50 mL) dropwise. The resulting mixture was warmed up to 0°C and then stirred 1 hour at 0°C. After one hour, the mixture was cooled to -78°C and pure ethyl bromoacetate (45 mL, 67.63 g, 405 mmol) was added dropwise. The whole mixture was slowly warmed up to room temperature and stirred overnight. After one night, the mixture was slowly hydrolyzed with 250 mL of a saturated aqueous solution of ammonium chloride, transferred in a separating funnel, diluted with 800 mL of diethylether. The aqueous phase was basified with solid Na_2CO_3 and washed with diethylether (2 x 300 mL). The combined organic layers were dried over sodium sulfate and concentrated under reduced pressure. The residue was purified *via* flash chromatography eluting with AcOEt/cyclohexane (5-20%) to yield 34 g (79%) of **7** as a brown oil. ^{1}H NMR (400 MHz, DMSO-d_6) δ 8.30 (dd, *J* = 2.8, 0.7 Hz, 1 H), 7.85 (dd, *J* = 8.8, 2.8 Hz, 1 H), 6.85 (dd, *J* = 8.8, 0.7 Hz, 1 H), 3.97 (qd, *J* = 7.1, 1.8 Hz, 4 H), 3.85 (s, 3 H), 3.28 (AB, *J* = 50.2, 16.6 Hz, 4 H), 1.05 (t, *J* = 7.1 Hz, 6 H) ; J-MOD ^{13}C NMR (100 MHz, DMSO-d_6) δ 168.3 (2 C), 163.0, 144.7, 137.0, 126.2, 120.3, 110.1, 60.4 (2 C), 53.3, 42.5 (2 C), 38.7, 13.8 (2 C) ; IR (film, cm^{-1}) 2981, 2942, 2899, 2844, 2241, 1733, 1604, 1565, 1491, 1377, 1291, 1189, 1017, 837 ; mass spectrum CI+ *m/z* 321.1437 [$C_{16}H_{21}N_2O_5$ (M+1) requires 321.1450].

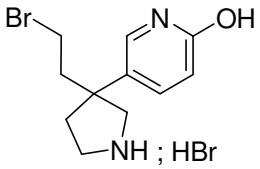


[3-(6-Methoxy-pyridin-3-yl)-5-oxo-pyrrolidin-3-yl]-acetic acid ethyl ester (8). A solution of 3-cyano-3-(6-methoxy-pyridin-3-yl)-pentanedioic acid diethyl ester **7** (12.1 g, 37.77 mmol) and Raney®-Nickel (slurry in water, 3 spatulas) in ethanol (300 mL) was submitted to hydrogenation in a Parr apparatus at 70°C, under 7 bars of hydrogen, for 6 hours. After 6 hours, the catalyst was removed by filtration on Celite, and the resulting mixture was concentrated under reduced pressure to furnish 9.6 g (92%) of the desired compound **8** as an orange oil. ^{1}H NMR (400 MHz, DMSO-d_6) δ 8.05 (dd, *J* = 2.6, 0.5 Hz, 1 H), 7.71 (br s, 1 H), 7.63 (dd, *J* = 8.7, 2.6 Hz, 1 H), 6.77 (dd, *J* = 8.7, 0.6 Hz, 1 H), 3.87 (q, *J* = 7.1 Hz, 2 H), 3.82 (s, 3 H), 3.64 (dd, *J* = 9.8, 0.6 Hz, 1 H), 3.48 (d, *J* = 9.8 Hz, 1 H), 2.79 (AB, 2 H), 2.60 (AB, 2 H), 0.98 (t, *J* = 7.1 Hz, 3 H) ; ^{13}C NMR (100 MHz, DMSO-d_6) δ 174.9, 170.2, 162.2, 144.4, 137.5, 132.7, 109.9, 59.7, 53.0, 51.6, 44.1, 43.4, 42.2, 13.8 ; IR (film, cm^{-1}) 3371, 3245, 3114, 2980,

2941, 2905, 1723, 1696, 1605, 1569, 1490, 1463, 1443, 1380, 1332, 1289, 1261, 1198, 1178, 1020, 830 ; mass spectrum CI+ *m/z* 279.1350 [$C_{14}H_{19}N_2O_4$ (M+1) requires 279.1345].

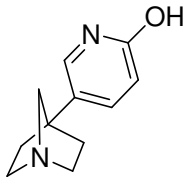


2-[3-(6-Methoxy-pyridin-3-yl)-pyrrolidin-3-yl]-ethanol (9). In a 1 liter three-necked flask equiped with a reflux condenser, a thermometer and an argon entry, at 0°C, to a solution of [3-(6-methoxy-pyridin-3-yl)-5-oxo-pyrrolidin-3-yl]-acetic acid ethyl ester **8** (9.12 g, 32.77 mmol) in anhydrous THF (540 mL) was added portionwise lithium aluminium hydride (12.44 g, 327.7 mmol). When the addition was complete, the mixture was stirred overnight at 50°C. After one night, the mixture was cooled to 0°C, slowly hydrolyzed with a saturated aqueous solution of Na_2SO_4 until white flakes appeared, and then filtrated on Celite. The Celite cake was washed with dichloromethane (3 x 200 mL) and the combined filtrates were concentrated under reduced pressure. The residue was purified *via* flash chromatography eluting with CH_2Cl_2/MeOH/NH_4OH (in a 90/10/1 to 80/20/2 ratio) to yield 7.3 g (79%) of **9** as a colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.01 (d, *J* = 2.4 Hz, 1 H), 7.43 (dd, *J* = 8.6, 2.6 Hz, 1 H), 6.69 (d, *J* = 8.6 Hz, 1 H), 3.89 (s, 3 H), 3.59 (m, 1 H), 3.51 (m, 1 H), 3.38 (d, *J* = 9.9 Hz, 1 H), 3.24 (m, 1 H), 3.07 (m, 2 H), 2.97 (br s, 2 H), 2.15 (m, 2 H), 1.84 (m, 2 H); ^{13}C NMR (100 MHz, DMSO-d_6) δ 161.9, 144.6, 138.0, 133.9, 109.8, 57.9, 56.6, 52.9, 47.1, 44.3, 42.2, 36.7 ; IR (CH_2Cl_2 ; cm^{-1}) 3352, 3048, 2946, 2920, 2882, 2847, 1602, 1567, 1496, 1373, 1288, 1136, 1069, 1023, 828 ; mass spectrum CI+ *m/z* 223.1445 [$C_{12}H_{19}N_2O_2$ (M+1) requires 223.14465].

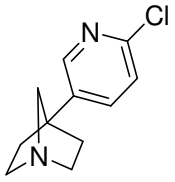


5-[3-(2-Bromo-ethyl)-pyrrolidin-3-yl]-pyridin-2-ol hydrobromide (10) . A solution of 2-[3-(6-methoxy-pyridin-3-yl)-pyrrolidin-3-yl]-ethanol **9** (5.76 g, 25.9 mmol) in hydrobromic acid (130 mL, 48 wt. % in water) was distributed in 4 sealed tubes (pressure vessels, 4 x 50 mL). The tubes were then stirred at 150°C overnight. After one night, the tubes were cooled to room temperature, opened, and the combined reaction mixtures were concentrated under reduced pressure. The residue was diluted with 150 mL of toluene, concentrated under reduced pressure (this operation was repeated twice) to furnish 9.12 g (quantitative yield) of the desired compound **10** as a browm powder and was engaged in the next step without purification. ^{1}H NMR (400 MHz, D_2O) δ 7.92 (d, *J* = 8.9 Hz, 1 H), 7.69 (s, 1 H), 6.86 (d, *J*

= 9.0 Hz, 1 H), 3.75 (AB, *J* = 11.8 Hz, 1 H), 3.57 (m, 2 H), 3.43 (m, 1 H), 3.33 (m, 1 H), 3.22 (m, 1 H), 2.56 (m, 1 H), 2.40 (m, 3 H) ; J-MOD ^{13}C NMR (100 MHz, DMSO-d_6) δ 161.4, 141.1, 133.3, 119.5, 119.1, 52.8, 48.0, 43.2, 40.7, 33.7, 29.1 ; mass spectrum (free base) ESI+ *m/z* 271.0437 [$C_{11}H_{16}BrN_2O$ (M+1) requires271.0441] ; mp 218-220°C.

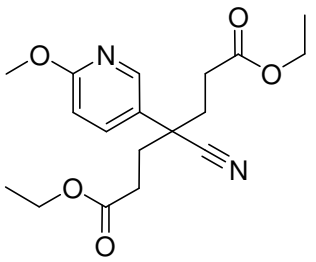


5-(1-Aza-bicyclo[2.2.1]hept-4-yl)-pyridin-2-ol (11). To a suspension of 5-[3-(2-bromo-ethyl)-pyrrolidin-3-yl]-pyridin-2-ol hydrobromide **10** (9.12 g; 25.9 mmol) in chloroform (530 mL) was added portionwise potassium carbonate (25.83 g, 186.9 mmol) and 20 mL of water. The resulting mixture was stirred at 50°C overnight. After one night, the layers were separated and the aqueous layer was washed with chloroform (3 x 150 mL). The combined organic layers were dried over sodium sulfate and concentrated under reduced pressure. The residue was then triturated in diethylether, filtrated to yield 3.63 g (74%) of **11** as a brown powder. ^{1}H NMR (400 MHz, DMSO-d_6) δ 11.42 (br s, 1 H), 7.49 (dd, *J* = 9.4, 2.7 Hz, 1 H), 7.12 (d, *J* = 2.5 Hz, 1 H), 6.30 (d, *J* = 9.4 Hz, 1 H), 2.86 (m, 2 H), 2.55 (m, 2 H), 2.43 (s, 2 H) 1.64 (m, 2 H), 1.44 (m, 2 H) ; ^{13}C NMR (100 MHz, DMSO-d_6) δ 161.7, 141.0, 131.3, 119.7, 119.0, 63.2, 55.0 (2 C), 49.8, 36.6 (2 C) ; IR (CH_2Cl_2 , cm^{-1}) 3386, 3037; 2957, 2930, 1679, 1662, 1624, 1545, 1469, 1313, 1237, 1002, 961 ; mp 170-172°C ; mass spectrum CI+ *m/z* 191.1229 [$C_{11}H_{15}N_2O$ (M+1) requires 191.1184].

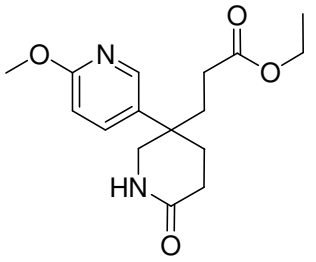


4-(6-Chloro-pyridin-3-yl)-1-aza-bicyclo[2.2.1]heptane (1). A solution of 5-(1-aza-bicyclo[2.2.1]hept-4-yl)-pyridin-2-ol **11** (2.24 g , 11.77 mmol) in $POCl_3$ (32.9 mL, 54.16 g, 353.2 mmol) was equally distributed in two sealed tube. The tubes were then stirred at 140°C for 45 minutes. After 45 minutes, the tubes were cooled to room temperature and the combined reaction mixtures were slowly poured in crushed ice. The whole mixture was carrefully basified to pH=10 with a saturated aqueous solution of Na_2CO_3 and then stirred one additionnal hour. After one hour, this aqueous layer was washed with dichloromethane (4 x 150 mL) and the combined organic layers were dried over sodium sulfate and concentrated under reduced pressure. The residue was diluted once with 200 mL of EtOH, concentrated under reduced pressure, then with 200 mL of toluene and concentrated under reduced pressure to

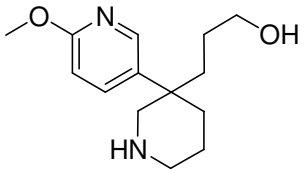
dryness to yield 2.23 g (91%) of **1** as a brown gum. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.32 (d, *J* = 2.4 Hz, 1 H), 7.58 (dd, *J* = 8.2, 2.5 Hz, 1 H), 7.27 (d, *J* = 8.2 Hz, 1 H), 3.29 (td, *J* = 10.7, 5.0 Hz, 2 H), 2.93 (s, 2 H), 2.89 (m, 2 H), 1.95 (td, *J* = 10.6, 4.7 Hz, 2 H), 1.80 (m, 2 H) ; J-MOD ^{13}C NMR (100 MHz, DMSO-d_6) δ 148.3, 148.2, 138.4, 137.2, 123.8, 62.6, 54.5 (2 C), 50.3, 36.4 (2 C) ; mp 70-72°C ; mass spectrum CI+ *m/z* 209.08509 [$C_{11}H_{14}ClN_2$ (M+1) requires 209.08455].



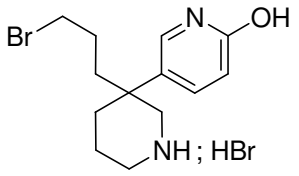
4-Cyano-4-(6-methoxy-pyridin-3-yl)-heptanedioic acid diethyl ester (12). To a solution of (6-methoxy-pyridin-3-yl)-acetonitrile **6** (7.375 g, 49.78 mmol) in acetonitrile (200 mL) was added Triton® B (40% w/w in methanol, 2.3 mL, 4.98 mmol). The mixture was refluxed and a solution of ethyl acrylate (27.28 mL, 25.04 g, 248.9 mmol) in acetonitrile (65 mL) was added dropwise. When the addition was complete, the resulting mixture was stirred under reflux overnight. After one night, the mixture was cooled to room temperature, concentrated under reduced pressure. The residue was diluted with chloroform (250 mL), washed with a saturated aqueous solution of $NaHCO_3$. The aqueous layer was washed with chloroform (2 x 200 mL) and the combined organic layers were dried over sodium sulfate and concentrated under reduced pressure. The residue was purified *via* flash chromatography eluting with CH_2Cl_2/[CH_2Cl_2/MeOH/NH_4OH 80/20/2] (in a 100/0 to 90/10 ratio) to yield 17.22 g (99%) of **12** as a yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.23 (d, *J* = 2.6 Hz, 1 H), 7.52 (dd, *J* = 8.7, 2.6 Hz, 1 H), 6.75 (d, *J* = 8.7 Hz, 1 H), 4.05 (m, 4 H), 3.92 (s, 3 H), 2.47 (m, 2 H), 2.33 (m, 2 H), 2.18 (m, 4 H), 1.19 (t, *J* = 7.2 Hz, 6 H) ; J-MOD ^{13}C NMR (100 MHz, DMSO-d_6) δ 171.2 (2 C), 163.4, 144.7, 136.9, 125.2, 120.7, 111.0, 60.1 (2 C), 53.3, 44.3, 34.2 (2 C), 30.0 (2 C), 13.9 (2 C) ; IR (film, cm^{-1}) 2978, 2937, 2902, 2837, 2239, 1733, 1602, 1568, 1495, 1460, 1380, 1291, 1260, 1190, 1023, 832 ; mass spectrum EI+ *m/z* 348.1683 [$C_{18}H_{24}N_2O_5$ (M+) requires 348.1685].



3-(6'-Methoxy-6-oxo-1,4,5,6-tetrahydro-2H-[3,3']bipyridinyl-3-yl)-propionic acid ethyl ester (13). The procedure described for the synthesis of **8** was followed using 12.8 g of **12** (36.73 mmol) in 200 mL of ethanol and Raney®-Nickel (slurry in water, 3 spatulas) to yield 10.7 g (95%) of **13** as a colorless oil. ^{1}H NMR (400 MHz, DMSO-d_6) δ 8.12 (d, *J* = 2.5 Hz, 1 H), 7.72 (dd, *J* = 8.7, 2.5 Hz, 1 H), 7.49 (d, *J* = 2.2 Hz, 1 H), 6.80 (d, *J* = 8.7 Hz, 1 H), 3.92 (d, *J* = 7.1 Hz, 2 H), 3.84 (s, 3 H), 3.55 (d, *J* = 12.4 Hz, 1 H), 3.24 (d, *J* = 12.4 Hz, 1 H), 2.15 (m, 2 H), 1.90 (m, 6 H), 1.10 (t, *J* = 7.1 Hz, 3 H) ; ^{13}C NMR (100 MHz, DMSO-d_6) δ 172.5, 169.5, 162.2, 145.4, 137.8, 129.9, 110.2, 59.8, 54.9, 53.0, 37.6, 34.6, 30.6, 28.6, 28.1, 13.9 ; IR (CH_2Cl_2 ; cm^{-1}) 3396, 3044, 2979, 2946, 2870, 1727, 1665, 1603, 1491, 1375, 1295, 1219, 1186, 1023, 830 ; mass spectrum EI+ *m/z* 306.15835 [$C_{16}H_{22}N_2O_4$ (M+) requires 306.15796].

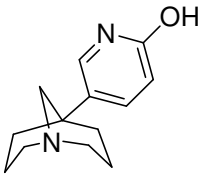


3-(6'-Methoxy-1,4,5,6-tetrahydro-2H-[3,3']bipyridinyl-3-yl)-propan-1-ol (14). The procedure described for the synthesis of **9** was followed using 10.7 g of **13** (34.93 mmol) in 580 mL of THF and 13.26 g (349.3 mmol) of lithium aluminium hydride. to yield 8.5 g (97%) of **14** as a pale yellow oil. ^{1}H NMR (200 MHz, $CDCl_3$) δ 8.07 (d, *J* = 2.5 Hz, 1 H), 7.50 (dd, *J* = 8.7, 2.6 Hz, 1 H), 6.70 (d, *J* = 8.7 Hz, 1 H), 3.89 (s, 3 H), 3.45 (t, *J* = 6.5 Hz, 2 H), 3.43 (s, 1 H), 3.19 (d, *J* = 12.8 Hz, 1 H), 2.83 (d, *J* = 12.8 Hz, 1 H), 2.76 (m, 2 H), 2.05 (m, 1 H), 1.65 (m, 5 H + H_2O), 1.45 (m, 1 H), 1.19 (m, 2 H); J-MOD ^{13}C NMR (100 MHz, DMSO-d_6) δ 161.4, 145.3, 137.9, 134.3, 109.6, 61.3, 55.1, 52.8, 46.5, 38.3, 35.9, 34.6, 26.7, 22.4 ; IR (film ; cm^{-1}) 3281, 2935, 2852, 2816, 2729, 1601, 1569, 1498, 1454, 1378, 1280, 1256, 1168, 1129, 1057, 1022, 894, 823 ; mass spectrum CI+ *m/z* 251.17568 [$C_{14}H_{23}N_2O_2$ (M+1) requires 251.17595].

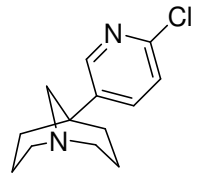


3'-(3-Bromo-propyl)-1',2',3',4',5',6'-hexahydro-[3,3']bipyridinyl-6-ol hydrobromide (15). The procedure described for the synthesis of **10** was followed using 8.5 g of **14** (33.95 mmol) in 120 mL of hydrobromic acid (48 wt. % in water) to yield 12.9 g (quantitative yield) of **15** as a brown powder. ^{1}H NMR (400 MHz, D_2O) δ 7.93 (d, *J* = 9.3 Hz, 1 H), 7.66 (s, 1 H), 6.85 (d, *J* = 9.5 Hz, 1 H), 3.68 (d, *J* = 13.2 Hz, 1 H), 3.44 (s, 2 H), 3.33 (d, *J* = 13.9 Hz, 1 H), 3.23 (m, 2 H), 2.35 (m, 1 H), 1.88 (m, 6 H), 1.54

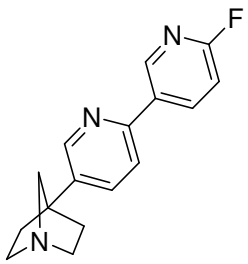
(m, 1 H) ; J-MOD ^{13}C NMR (100 MHz, DMSO-d_6) δ 161.1, 141.6, 134.2, 121.0, 118.7, 49.7, 43.4, 37.5, 37.3, 34.9, 29.6, 26.4, 18.4 ; mass spectrum (free base) ESI+ *m/z* 299.0755 [$C_{13}H_{19}BrN_2O$ (M+1) requires 299.07535]. ; mp 236-238°C.



5-(1-Aza-bicyclo[3.3.1]non-5-yl)-pyridin-2-ol (16). The procedure described for the synthesis of **11** was followed using 12.9 g of **15** (33.95 mmol) in 340 mL of chloroform and 23.45 g (169.7 mmol) of potassium carbonate to yield 4.5 g (61%) of **16** as an orange powder. ^{1}H NMR (400 MHz, DMSO-d_6) δ 11.31 (br s, 1 H), 7.54 (dd, *J* = 9.6, 2.6 Hz, 1 H), 6.98 (d, *J* = 2.3 Hz, 1 H), 6.29 (d, *J* = 9.6 Hz, 1 H), 2.91 (m, 4 H), 2.86 (s, 2 H), 2.02 (m, 4 H), 1.64 (m, 2 H), 1.43 (m, 2 H) ; J-MOD ^{13}C NMR (100 MHz, DMSO-d_6) δ 161.4, 139.4, 129.7, 126.9, 119.8, 57.8, 51.2 (2 C), 35.4 (2 C), 29.6, 23.2 (2 C) ; IR ($CHCl_3$; cm^{-1}) 3387 (weak), 2993, 2932, 2853, 1662, 1619, 1547, 1464, 1085, 881, 838 ; mass spectrum CI+ *m/z* 219.1498 [$C_{13}H_{19}N_2O$ (M+1) requires 219.1497] ; mp 191-192°C.

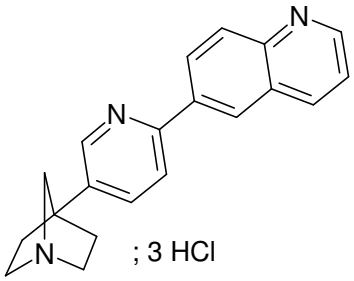


5-(6-Chloro-pyridin-3-yl)-1-aza-bicyclo[3.3.1]nonane (2). The procedure described for the synthesis of **1** was followed using 0.7 g of **16** (3.21 mmol) in 9 mL of $POCl_3$ to yield 0.68 g (90%) of **2** as a brown powder. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.30 (d, *J* = 2.4 Hz, 1 H), 7.54 (dd, *J* = 8.4, 2.6 Hz, 1 H), 7.26 (d, *J* = 8.6 Hz, 1 H), 3.21 (s, 2 H), 3.19 (m, 4 H), 2.23 (m, 4 H), 1.82 (m, 2 H), 1.68 (m, 2 H) ; ^{13}C NMR (100 MHz, DMSO-d_6) δ 147.6, 147.0, 145.4, 136.6, 123.7, 57.8, 51.2 (2 C), 36.0 (2 C), 30.8, 23.3 (2 C) ; IR ($CHCl_3$; cm^{-1}) 2985, 2934, 2880, 2852, 1577, 1566, 1480, 1459, 1365, 1111, 1079, 1028, 874 ; mass spectrum EI+ *m/z* 236.1106 [$C_{13}H_{17}ClN_2$ (M+) requires 236.1080] ; mp 103-104°C.



5-(1-Aza-bicyclo[2.2.1]hept-4-yl)-6'-fluoro-[2,3']bipyridinyl (17a).

General Procedure A : To a solution of 4-(6-chloro-pyridin-3-yl)-1-aza-bicyclo[2.2.1]heptane **1** (50 mg, 0.24 mmol) in a 2/1 mixture of DME and water (8 mL & 4 mL) was added 2-fluoro-5-pyridylboronic acid (84 mg, 0.6 mmol) and potassium carbonate (83 mg, 0.6 mmol). The resulting mixture was bubbled for 30 minutes while stirring with an argon flush. After 30 minutes, $PdCl_2(PPh_3)_2$ (45 mg, 0.07 mmol) was added and the resulting mixture was bubbled for 30 additionnal minutes while stirring with an argon flush. After 30 minutes, the resulting mixture was stirred under reflux for 3 hours. After 3 hours, 30 mL of a saturated aqueous solution of Na_2CO_3 was added, and the whole mixture was washed with chloroform (3 x 50 mL). The combined organic layers were dried over sodium sulfate and concentrated under reduced pressure. The residue was purified *via* flash chromatography eluting with $CHCl_3$/MeOH/NH_4OH (in a 95/5/0.5 ratio) to yield 35 mg (54%) of **17a** as a white powder. ^{1}H NMR (400 MHz, DMSO-d_6) δ 8.90 (d, *J* = 2.0 Hz, 1 H), 8.68 (d, *J* = 1.8 Hz, 1 H), 8.61 (td, *J* = 8.2, 2.4 Hz, 1 H), 7.98 (d, *J* = 8.2 Hz, 1 H), 7.87 (dd, *J* = 8.2, 2.3 Hz, 1 H), 7.29 (dd, *J* = 8.6, 2.8 Hz, 1 H), 2.94 (td, *J* = 10.4, 4.9 Hz, 2 H), 2.65 (s, 2 H), 2.62 (m, 2 H), 1.80 (m, 2 H), 1.66 (m, 2 H) ; J-MOD ^{13}C NMR (100 MHz, DMSO-d_6) δ 163.2 (d, *J* = 235.7 Hz, 1 C), 150.6, 148.3, 145.6 (d, *J* = 15.5 Hz, 1 C), 139.9 (d, *J* = 8.2 Hz, 1 C), 138.1, 135.9, 132.8 (d, *J* = 4.4 Hz, 1 C), 120.0, 109.6 (d, *J* = 37.6 Hz, 1 C), 63.3, 55.1 (2 C), 51.2, 37.4 (2 C) ; ^{19}F NMR (188 MHz, DMSO-d_6) δ -69.5 ; mass spectrum CI+ *m/z* 270.13993 [$C_{16}H_{17}FN_3$ (M+1) requires270.14065] ; mp 108-109°C.

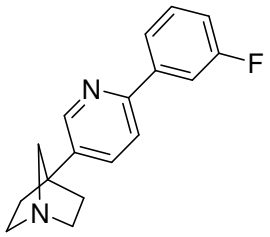


; 3 HCl

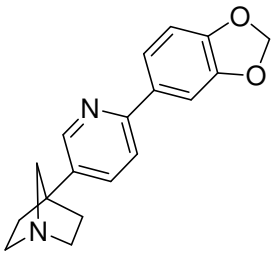
6-[5-(1-Aza-bicyclo[2.2.1]hept-4-yl)-pyridin-2-yl]-quinoline hydrochloride 1/3 (17b).

General Procedure B : To a solution of 4-(6-Chloro-pyridin-3-yl)-1-aza-bicyclo[2.2.1]heptane **1** (55 mg, 0.26 mmol) in a 2/1 mixture of DME and water (7 mL & 3 mL) was added 6-quinolineboronic acid pinacol ester (168 mg, 0.66 mmol) and potassium carbonate (91 mg, 0.66 mmol). The resulting mixture was bubbled for 30 minutes while stirring with an argon flush. After 30 minutes, $PdCl_2(PPh_3)_2$ (56 mg, 0.08 mmol) was added and the resulting mixture was bubbled for 30 additionnal minutes while stirring with an argon flush. After 30 minutes, the resulting mixture was stirred under reflux for 3 hours. After 3 hours, 30 mL of a saturated aqueous solution of Na_2CO_3 was added, and the whole mixture was washed with chloroform (3 x 50 mL). The combined organic layers were dried over sodium sulfate and concentrated under reduced pressure. The residue was purified *via* flash chromatography eluting with

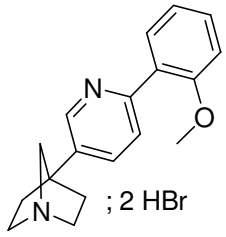
$CHCl_3$/MeOH/NH_4OH (in a 85/15/1.5 ratio). The purified fraction was concentrated under reduced pressure and the resulting residue was diluted with 3.5 mL of a 5-6 N solution of HCl in i-PrOH. The mixture was stirred at room temperature for 3 hours. After 3 hours, a precipitate was formed, which was collected by filtration, washed with diethylether and dried under vacuum at 90°C one night to yield 165 mg (61%) of **17b** as a white powder. ^{1}H NMR (400 MHz, DMSO-d_6) δ 11.41 (br s, 1 H), 9.30 (dd, *J* = 5.0, 1.4 Hz, 1 H), 9.23 (d, *J* = 8.6 Hz, 1 H), 8.90 (d, *J* = 1.8 Hz, 1 H), 8.52 (d, *J* = 8.5 Hz, 1 H), 8.20 (dd, *J* = 8.5, 7.4 Hz, 1 H), 8.16 (dd, *J* = 8.2, 2.4 Hz, 1 H), 8.09 (dd, *J* = 7.2, 0.8 Hz, 1 H), 8.01 (dd, *J* = 8.7, 5.0 Hz, 1 H), 7.93 (d, *J* = 8.2 Hz, 1 H), 3.67 (s, 2 H), 3.57 (m, 2 H), 3.49 (m, 2 H), 2.26 (m, 4 H) ; J-MOD ^{13}C NMR (100 MHz, DMSO-d_6) δ 154.3, 147.3, 146.2, 142.6, 140.3, 137.7, 136.7, 134.3, 132.9, 130.1, 126.2, 125.2, 123.3, 122.4, 60.0, 52.4 (2 C), 49.1, 33.6 (2 C) ; mass spectrum (free base) CI+ *m/z* 302.16443 [$C_{20}H_{20}N_3$ (M+1) requires 302.16572] ; mp 327-328°C.



4-[6-(3-Fluoro-phenyl)-pyridin-3-yl]-1-aza-bicyclo[2.2.1]heptane (17c). General procedure A described for the synthesis of **17a** was followed using 55 mg of **1** (0.26 mmol), 92 mg of 3-fluorophenylboronic acid (0.66 mmol), 91 mg of potassium carbonate (0.66 mmol) and 37 mg of $PdCl_2(PPh_3)_2$ (0.05 mmol) in a 2/1 mixture of DME/water (respectively 7 mL & 3 mL) to yield 48 mg (68%) of **17c** as a white powder. ^{1}H NMR (400 MHz, DMSO-d_6) δ 8.67 (d, *J* = 1.8 Hz, 1 H), 7.88 (m, 4 H), 7.52 (AB, *J* = 14.2, 8.0 Hz, 1 H), 7.25 (td, *J* = 8.6, 2.1 Hz, 1 H), 2.95 (td, *J* = 10.4, 4.8 Hz, 2 H), 2.66 (s, 2 H), 2.63 (m, 2 H), 1.81 (td, *J* = 10.2, 4.5 Hz, 2 H), 1.67 (m, 2 H) ; J-MOD ^{13}C NMR (100 MHz, DMSO-d_6) δ 162.7 (d, *J* = 241.2 Hz, 1 C), 152.4, 148.1, 141.2 (d, *J* = 7.7 Hz, 1 C), 138.0, 135.8, 130.7 (d, *J* = 8.3 Hz, 1 C), 122.2 (d, *J* = 2.4 Hz, 1 C), 120.0, 115.5 (d, *J* = 21.1 Hz, 1 C), 112.8 (d, *J* = 22.6 Hz, 1 C), 63.3, 55.1 (2 C), 51.1, 37.3 (2 C) ; ^{19}F NMR (188 MHz, DMSO-d_6) δ -112.9 ; mass spectrum CI+ *m/z* 269.14497 [$C_{17}H_{18}FN_2$ (M+1) requires 269.14540] ; mp 88-90°C.

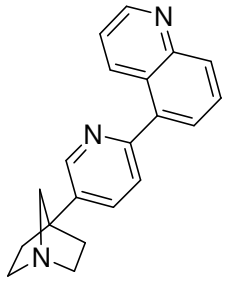


4-(6-Benzo[1,3]dioxol-5-yl-pyridin-3-yl)-1-aza-bicyclo[2.2.1]heptane (17d) . General procedure A described for the synthesis of **17a** was followed using 55 mg of **1** (0.26 mmol), 109 mg of 3,4-(methylenedioxy)phenylboronic acid (0.66 mmol), 91 mg of potassium carbonate (0.66 mmol) and 28 mg of $PdCl_2(PPh_3)_2$ (0.04 mmol) in a 2/1 mixture of DME/water (respectively 8 mL & 4 mL) to yield 61 mg (79%) of **17d** as a white powder. ^{1}H NMR (400 MHz, DMSO-d_6) δ 8.63 (d, *J* = 1.7 Hz, 1 H), 7.88 (d, *J* = 8.3 Hz, 1 H), 7.83 (dd, *J* = 8.3, 2.2 Hz, 1 H), 7.63 (m, 2 H), 7.01 (d, *J* = 8.7 Hz, 1 H), 6.08 (s, 2 H), 3.33 (m, 2 H), 3.28 (s, 2 H), 3.17 (m, 2 H), 2.03 (m, 4 H) ; ^{13}C NMR (100 MHz, DMSO-d_6) δ 154.2, 148.1, 147.9, 147.6, 135.5, 133.7, 132.8, 120.5, 119.3, 108.4, 106.5, 101.3, 61.1, 53.3 (2 C), 49.6, 34.7 (2 C) ; mass spectrum EI+ *m/z* 294.13705 [$C_{18}H_{18}N_2O_2$ (M+) requires 294.13683] ; mp 256-258°C.

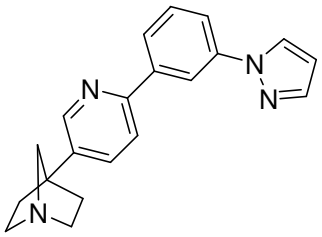


4-[6-(2-Methoxy-phenyl)-pyridin-3-yl]-1-aza-bicyclo[2.2.1]heptane hydrobromide 1/2 (17e). General procedure C : To a solution of 4-(6-chloro-pyridin-3-yl)-1-aza-bicyclo[2.2.1]heptane **1** (50 mg, 0.24 mmol) in a 2/1 mixture of DME/water (8 mL & 4 mL) was added 2-methoxyphenylboronic acid (91 mg, 0.6 mmol) and potassium carbonate (83 mg, 0.6 mmol). The resulting mixture was bubbled for 30 minutes while stirring with an argon flush. After 30 minutes, $PdCl_2(PPh_3)_2$ (50 mg, 0.07 mmol) was added and the resulting mixture was bubbled for 30 additionnal minutes while stirring with an argon flush. After 30 minutes, the resulting mixture was stirred under reflux for 3 hours. After 3 hours, 30 mL of a saturated aqueous solution of Na_2CO_3 was added, and the whole mixture was washed with chloroform (3 x 50 mL). The combined organic layers were dried over sodium sulfate and concentrated under reduced pressure. The residue was purified *via* flash chromatography eluting with $CHCl_3$/MeOH/NH_4OH (in a 90/10/1 ratio). The purified fraction was concentrated under reduced pressure and the resulting residue was diluted with 3 mL of isopropyl alcohol and 0.15 mL of a 5.7 M solution of hydrobromic acid in acetic acid was added. The mixture was stirred at room temperature for 30 minutes, concentrated under reduced pressure. The residue was diluted with methanol, concentrated under reduced pressure. This operation was repeated 3 times. The resulting residue was then triturated in diethylether and the precipitate formed was collected by filtration, washed with diethylether and dried under vacuum at 60°C for one night to yield 58 mg (55%) of **17e** as a brown powder. ^{1}H NMR (400 MHz, DMSO-d_6) δ 10.35 (br s, 1 H), 8.87 (d, *J* = 1.9 Hz, 1 H), 8.44 (d, *J* = 8.1 Hz, 1 H), 8.14 (d, *J* = 8.4 Hz, 1 H), 7.68 (dd, *J* = 7.6, 1.7 Hz, 1 H), 7.59 (td, *J* = 7.9, 1.7 Hz, 1 H), 7.27 (d, *J* = 8.3 Hz, 1 H), 7.17

(t, *J* = 7.5 Hz, 1 H), 3.86 (s, 3 H), 3.71 (s, 2 H), 3.54 (m, 4 H), 2.27 (m, 4 H) ; ^{13}C NMR (100 MHz, DMSO-d_6) δ 156.7, 150.5, 142.6, 141.2, 135.3, 132.5, 131.6, 126.8, 122.3, 121.0, 112.2, 60.1, 55.9, 52.7 (2 C), 48.9, 33.2 (2 C) ; mass spectrum (free base) EI+ *m/z* 280.15807 [$C_{18}H_{20}N_2O$ (M+) requires 208.15756] ; mp 114-116°C.

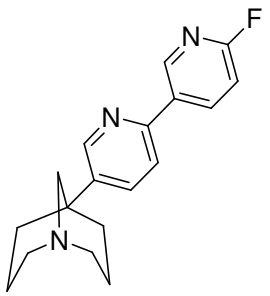


5-[5-(1-Aza-bicyclo[2.2.1]hept-4-yl)-pyridin-2-yl]-quinoline (17f). General procedure A described for the synthesis of **17a** was followed using 50 mg of **1** (0.24 mmol), 104 mg of 5-quinolineboronic acid (0.6 mmol), 83 mg of potassium carbonate (0.6 mmol) and 50 mg of $PdCl_2(PPh_3)_2$ (0.05 mmol) in a 2/1 mixture of DME/water (respectively 8 mL & 4 mL) to yield 72 mg (72%) of **17f** as a white powder. ^{1}H NMR (400 MHz, DMSO-d_6) δ 8.94 (dd, *J* = 4.1, 1.6 Hz, 1 H), 8.78 (d, *J* = 2.1 Hz, 1 H), 8.60 (d, *J* = 8.5 Hz, 1 H), 8.10 (d, *J* = 8.5 Hz, 1 H), 7.95 (dd, *J* = 8.1, 2.3 Hz, 1 H), 7.85 (t, *J* = 7.3 Hz, 1 H), 7.75 (d, *J* = 7.1 Hz, 1 H), 7.67 (d, *J* = 8.1 Hz, 1 H), 7.53 (dd, *J* = 8.6, 4.1 Hz, 1 H), 2.99 (td, *J* = 10.5, 4.8 Hz, 2 H), 2.72 (s, 2 H), 2.67 (m, 2 H), 1.87 (td, *J* = 10.0, 4.3 Hz, 2 H), 1.72 (m, 2 H) ; ^{13}C NMR (100 MHz, DMSO-d_6) δ 155.1, 150.4, 148.1, 147.8, 138.2, 137.3, 135.8, 134.1, 129.6, 128.9, 127.6, 125.7, 124.3, 121.6, 63.4, 55.0 (2 C), 51.1, 37.4 (2 C) ; mass spectrum EI+ *m/z* 301.15715 [$C_{20}H_{19}N_3$ (M+) requires 301.15790] ; mp 129-131°C.

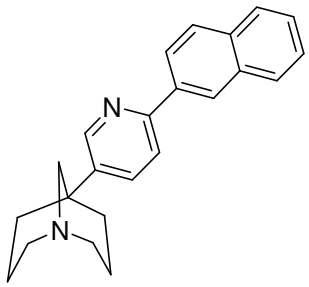


4-[6-(3-Pyrazol-1-yl-phenyl)-pyridin-3-yl]-1-aza-bicyclo[2.2.1]heptane (17g). General procedure A described for the synthesis of **17a** was followed using 100 mg of **1** (0.48 mmol), 324 mg of 1-[3-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)phenyl]-1H-pyrazole (1.2 mmol), 166 mg of potassium carbonate (1.2 mmol) and 101 mg of $PdCl_2(PPh_3)_2$ (0.14 mmol) in a 2/1 mixture of DME/water (respectively 16 mL & 8 mL) to yield 64 mg (48%) of **17g** as a white powder. ^{1}H NMR (400 MHz, DMSO-d_6) δ 8.70 (dd, *J* = 2.3, 0.6 Hz, 1 H), 8.60 (d, *J* = 2.3 Hz, 1 H), 8.52 (t, *J* = 1.8 Hz, 1 H), 8.00 (m, 2 H), 7.88 (m, 2 H), 7.78 (d, *J* = 1.6 Hz, 1 H), 7.60 (t, *J* = 7.9 Hz, 1 H), 6.57 (td, *J* = 1.8, 0.6

Hz, 1 H), 2.95 (m, 2 H), 2.67 (s, 2 H), 2.64 (m, 2 H), 1.82 (m, 2 H), 1.68 (m, 2 H) ; J-MOD ^{13}C NMR (100 MHz, DMSO-d_6) δ 153.1, 148.1, 141.0, 140.2, 140.0, 137.7, 135.8, 129.9, 127.9, 124.0, 120.0, 118.7, 116.2, 107.9, 63.3, 55.0 (2 C), 51.1, 37.3 (2 C) ; IR (CH_2Cl_2 , cm^{-1}) 3041, 2957, 2884, 1610, 1593, 1555, 1516, 1478, 1397, 1334, 1194, 1044, 967, 946, 841, 817 ; mass spectrum EI+ *m/z* 316.1679 [$C_{20}H_{20}N_4$ (M+) requires 316.1688] ; mp 114-116°C.

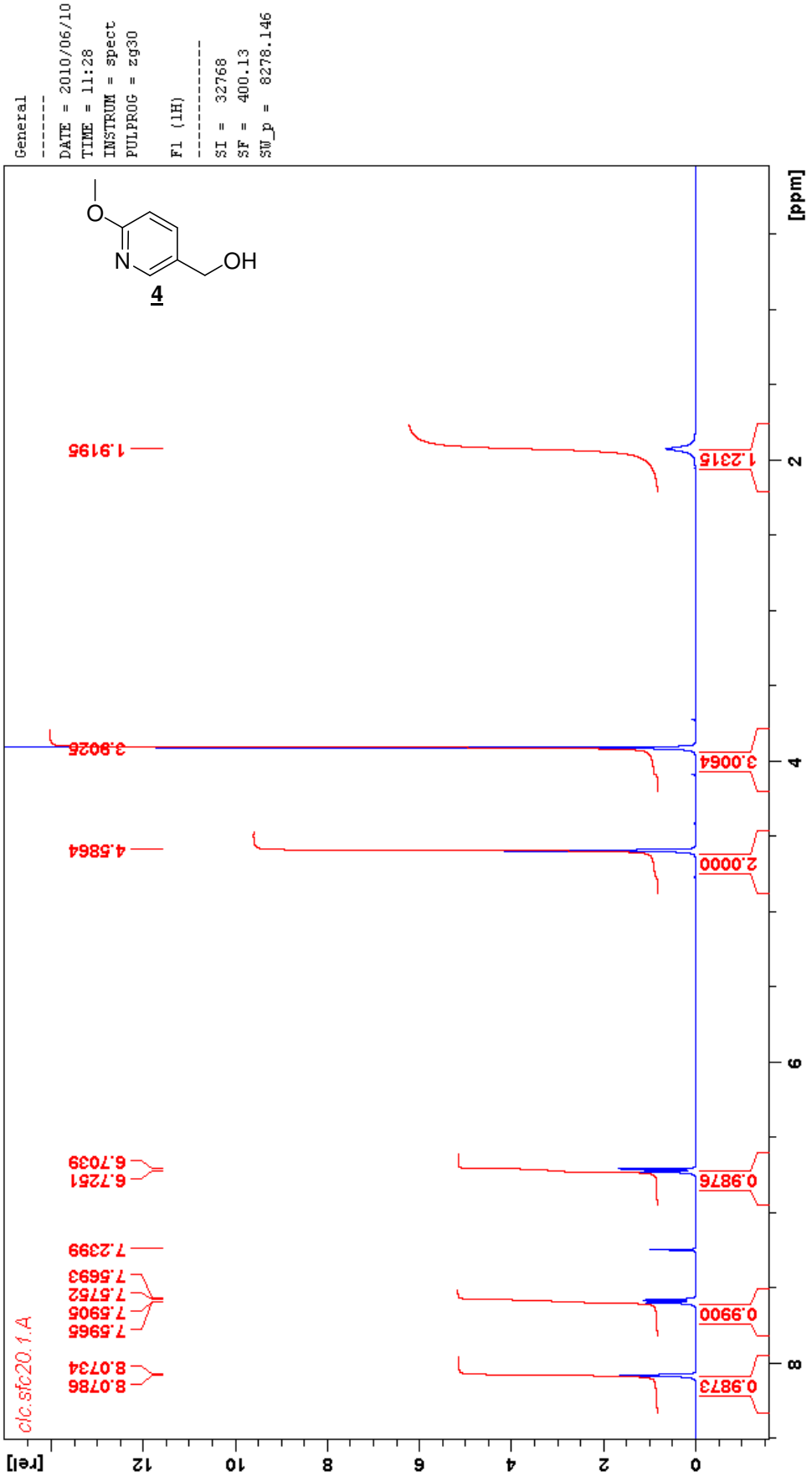


5-(1-Aza-bicyclo[3.3.1]non-5-yl)-6'-fluoro-[2,3']bipyridinyl (18a). General procedure A described for the synthesis of **17a** was followed using 50 mg of **2** (0.21 mmol), 76 mg of 2-fluoro-5-pyridylboronic acid (0.53 mmol), 73 mg of potassium carbonate (0.53 mmol) and 42 mg of $PdCl_2(PPh_3)_2$ (0.06 mmol) in a 2/1 mixture of DME/water (respectively 8 mL & 4 mL) to yield 23 mg (37%) of **18a** as a white powder. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.75 (br s, 1 H), 8.65 (br s, 1 H), 8.40 (t, *J* = 7.5 Hz, 1 H), 7.64 (m, 2 H), 7.00 (d, *J* = 6.7 Hz, 1 H), 3.21 (s, 2 H), 3.12 (m, 4 H), 2.22 (m, 4 H), 1.88 (m, 2 H), 1.59 (m, 2 H) ; J-MOD ^{13}C NMR (100 MHz, DMSO-d_6) δ 163.2 (d, *J* = 235.6 Hz, 1 C), 150.0, 146.8, 145.6 (d, *J* = 15.5 Hz, 1 C), 145.3, 139.9 (d, *J* = 8.2 Hz, 1 C), 133.9, 132.7 (d, *J* = 4.0 Hz, 1 C), 119.9, 109.5 (d, *J* = 37.6 Hz, 1 C), 58.0, 51.2 (2 C), 36.0 (2 C), 31.0, 23.4 (2 C) ; ^{19}F NMR (188 MHz, DMSO-d_6) δ -69.5 ; IR ($CHCl_3$; cm^{-1}) 2985, 2932, 2896, 2850, 1590, 1473, 1392, 1082, 824 ; mass spectrum CI+ *m/z* 298.17289 [$C_{18}H_{21}FN_3$ (M+1) requires 298.17195] ; mp 121-122°C.



5-(6-Naphthalen-2-yl-pyridin-3-yl)-1-aza-bicyclo[3.3.1]nonane (18h). General procedure A described for the synthesis of **17a** was followed using 50 mg of **2** (0.21 mmol), 91 mg of 2-naphthylboronic acid (0.53 mmol), 73 mg of potassium carbonate (0.53 mmol) and 45 mg of $PdCl_2(PPh_3)_2$ (0.06 mmol) in a 2/1 mixture of DME/water (respectively 8 mL & 4 mL) to yield 32 mg (46%) of **18h** as a white powder. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.70 (d, *J* = 2.1 Hz, 1 H), 8.45 (s, 1 H),

8.11 (d, *J* = 8.6 Hz, 1 H), 7.92 (m, 2 H), 7.84 (m, 1 H), 7.81 (d, *J* = 8.4 Hz, 1 H), 7.67 (dd, *J* = 8.4, 2.3 Hz, 1 H), 7.47 (m, 2 H), 3.24 (s, 2 H), 3.14 (m, 4 H), 2.23 (m, 4 H), 1.91 (m, 2 H), 1.60 (m, 2 H) ; J-MOD ^{13}C NMR (100 MHz, DMSO-d_6) δ 153.2, 146.6, 144.5, 135.9, 133.7, 133.1, 133.0, 128.5, 128.2, 127.5, 126.5, 126.4, 125.4, 124.2, 119.9, 57.9, 51.2 (2 C), 36.0 (2 C), 31.0, 23.3 (2 C) ; IR ($CHCl_3$; cm^{-1}) 3057, 3042, 2931, 2878, 2849, 1589, 1553, 1482, 1460, 1449, 1443, 1346, 1081, 1024, 821 ; mass spectrum EI+ *m*/*z* 328.1953 [$C_{23}H_{24}N_2$ (M+) requires 328.1940] ; mp 163-165°C.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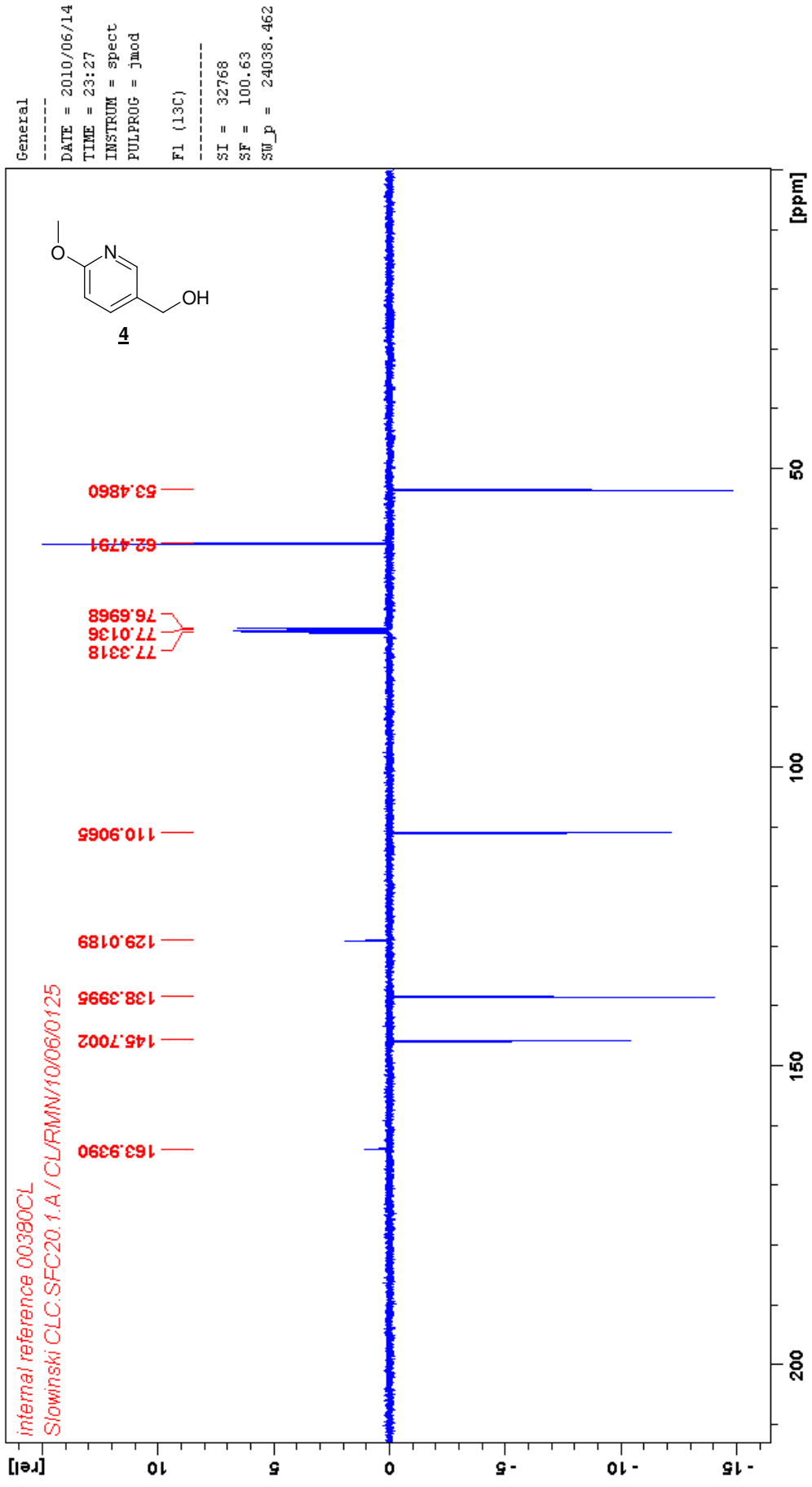
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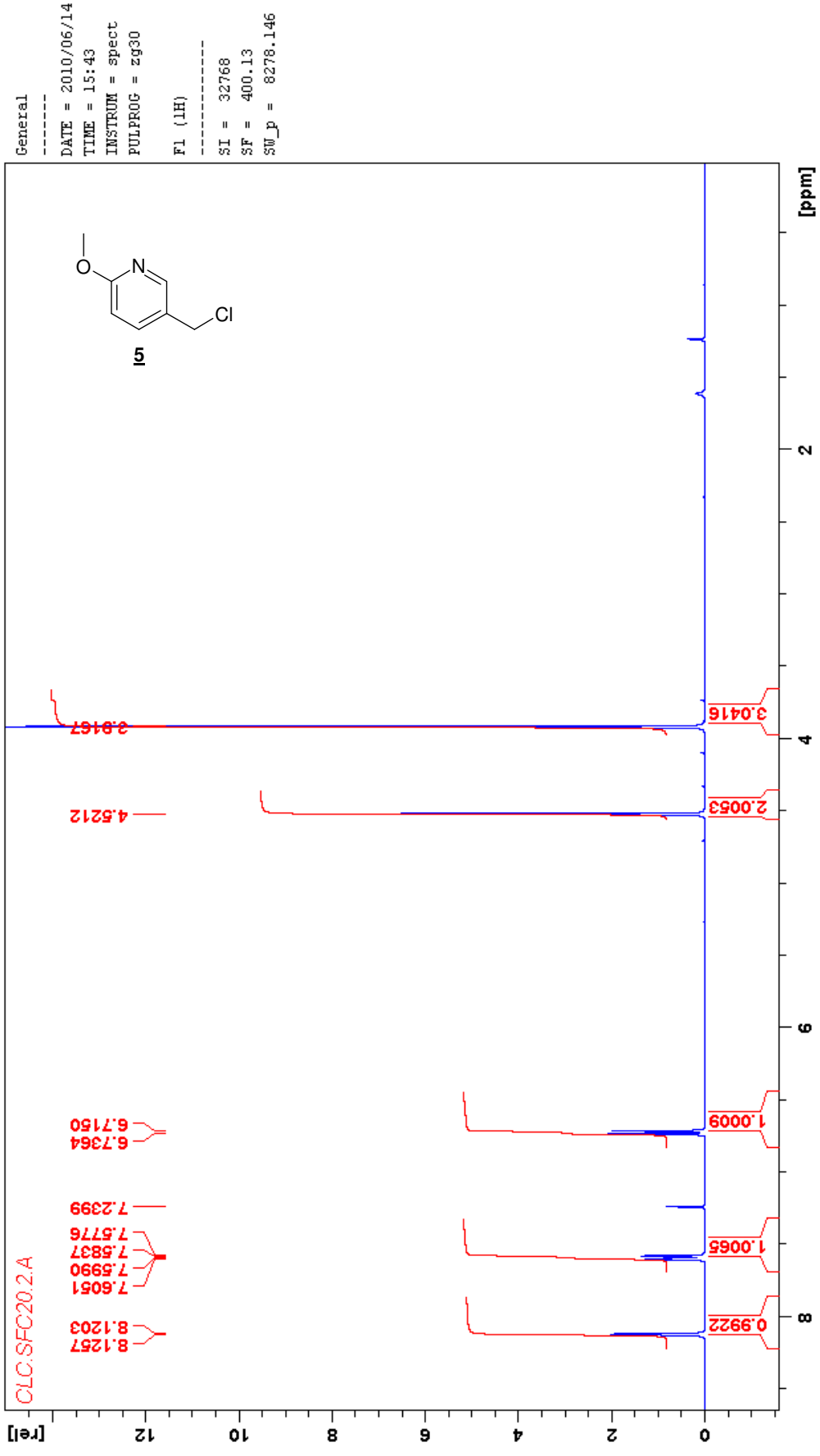
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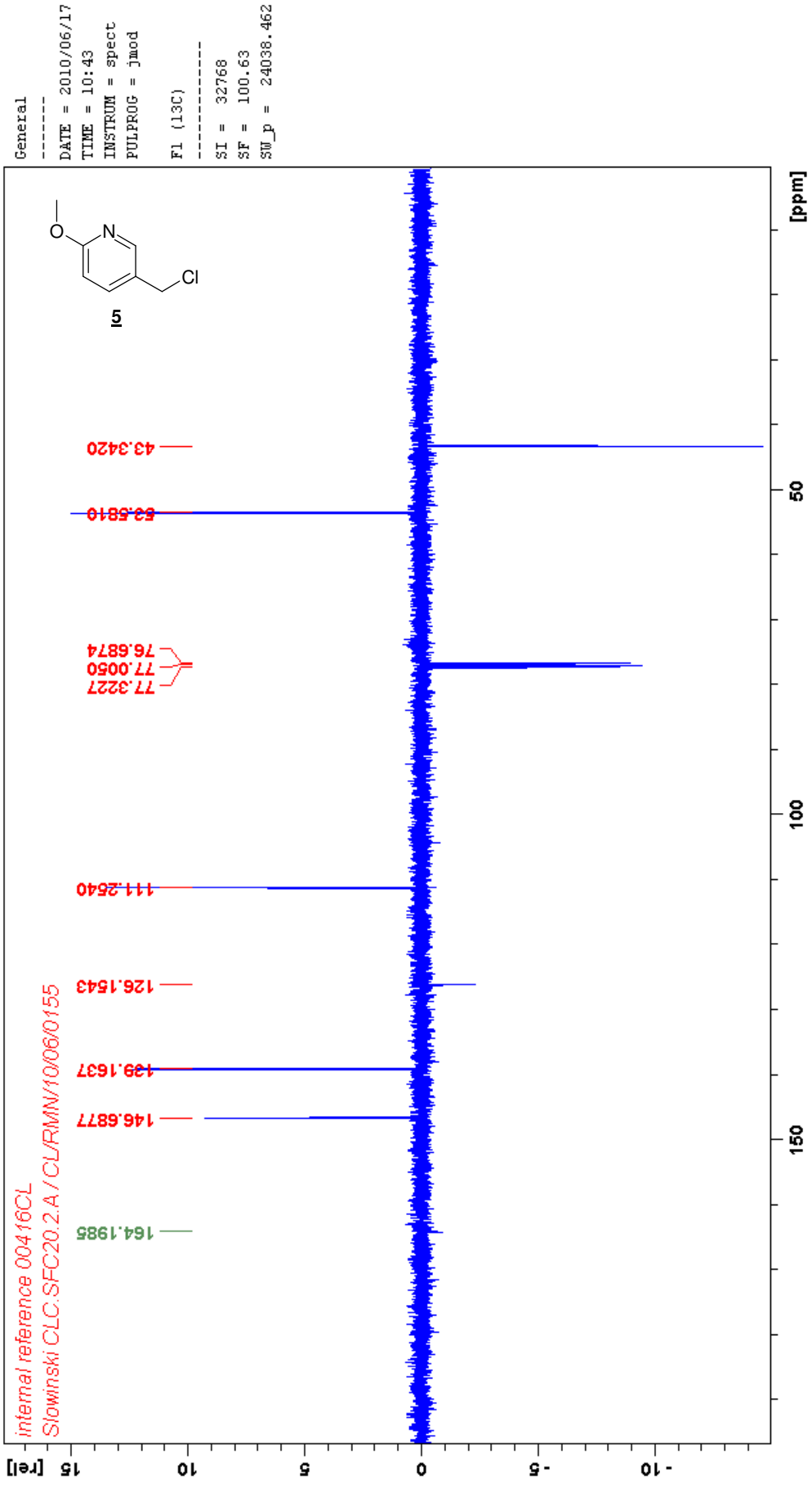
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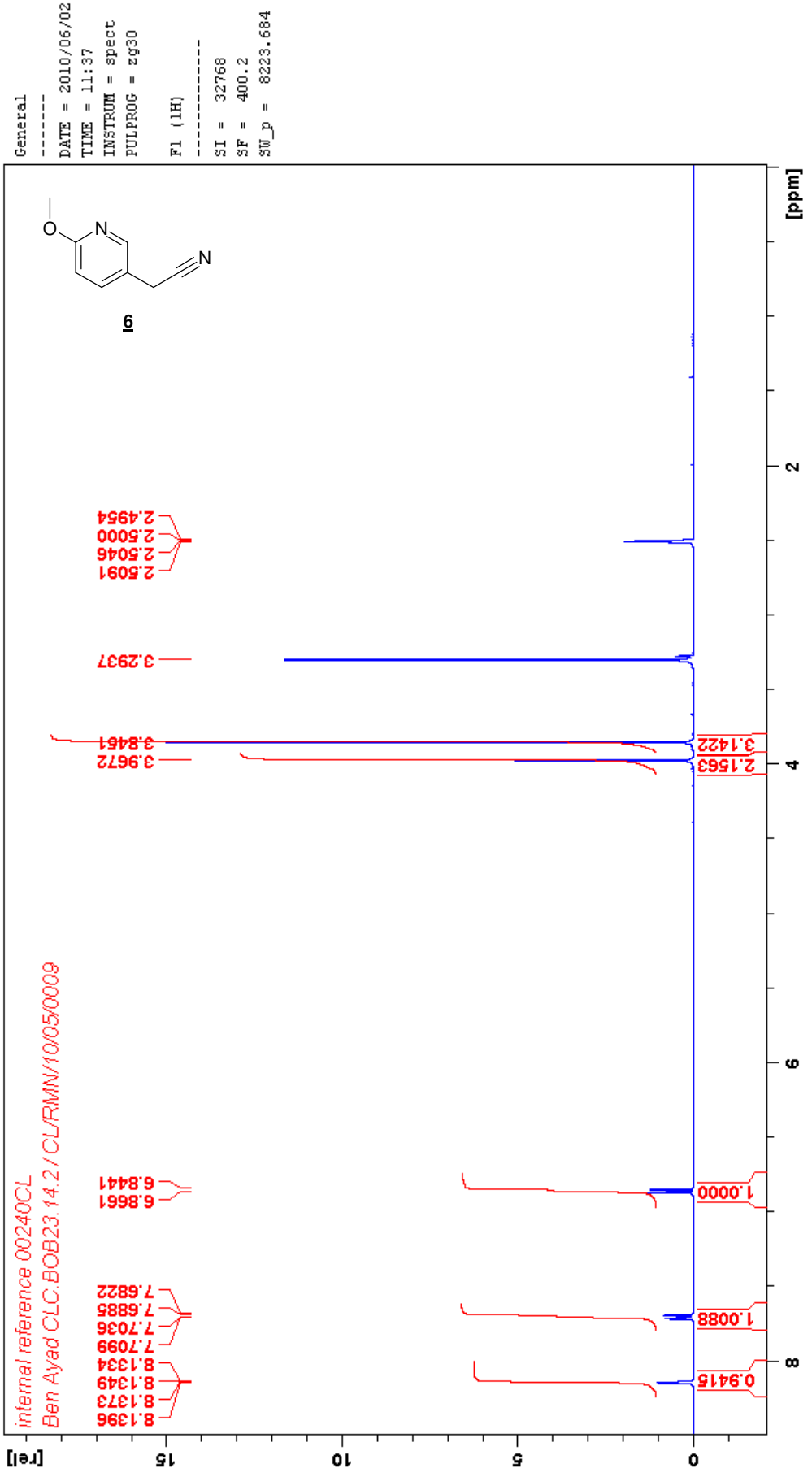
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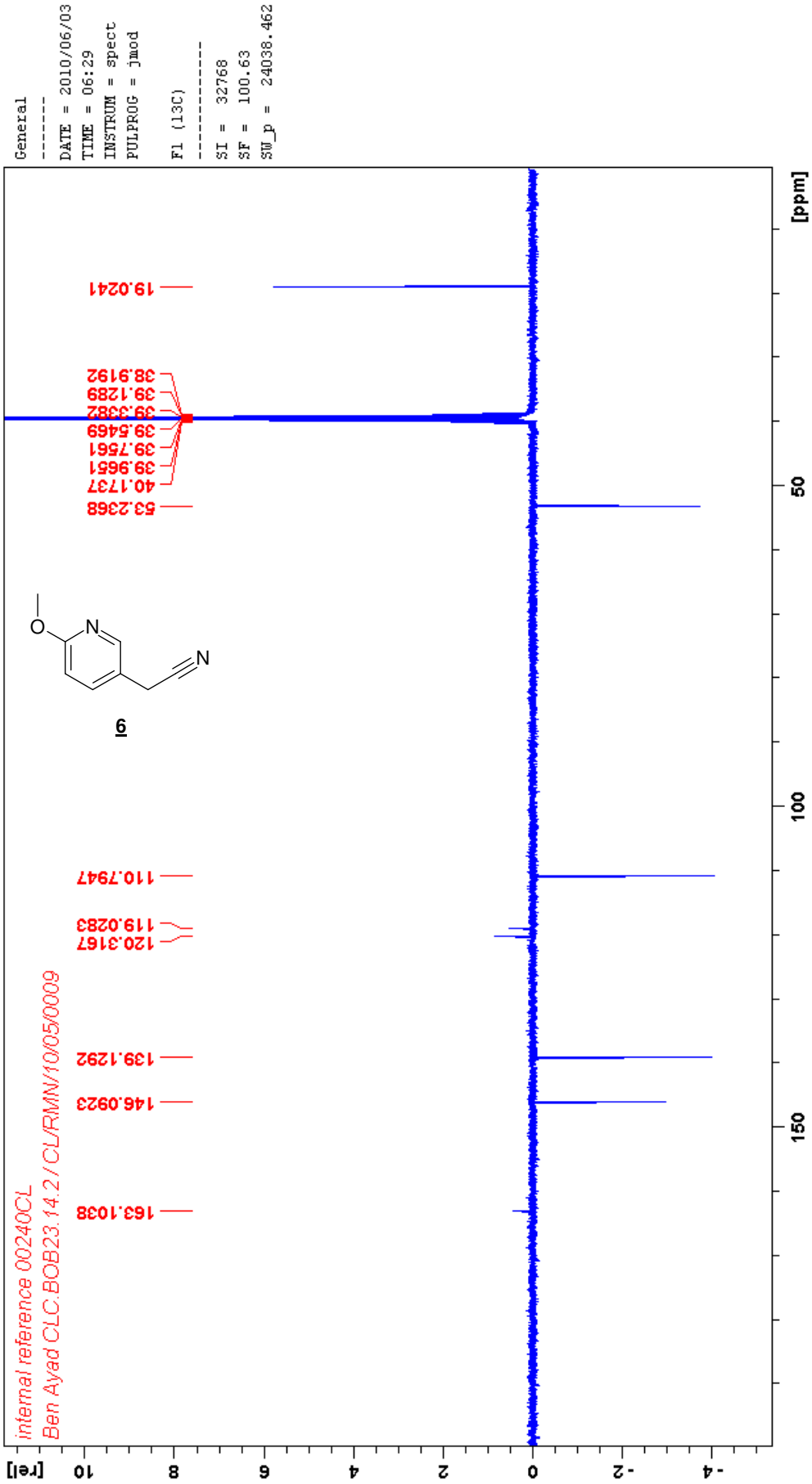
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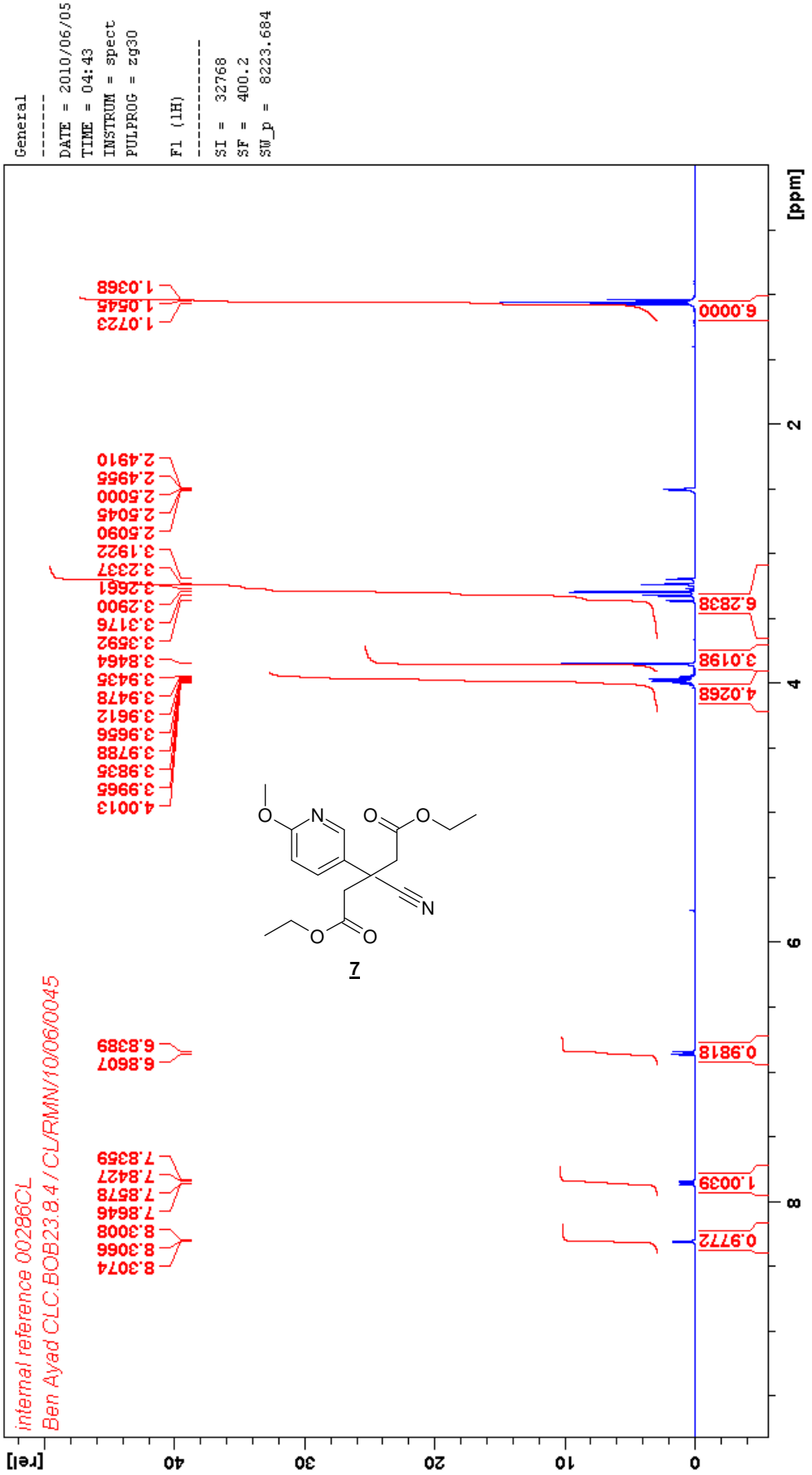
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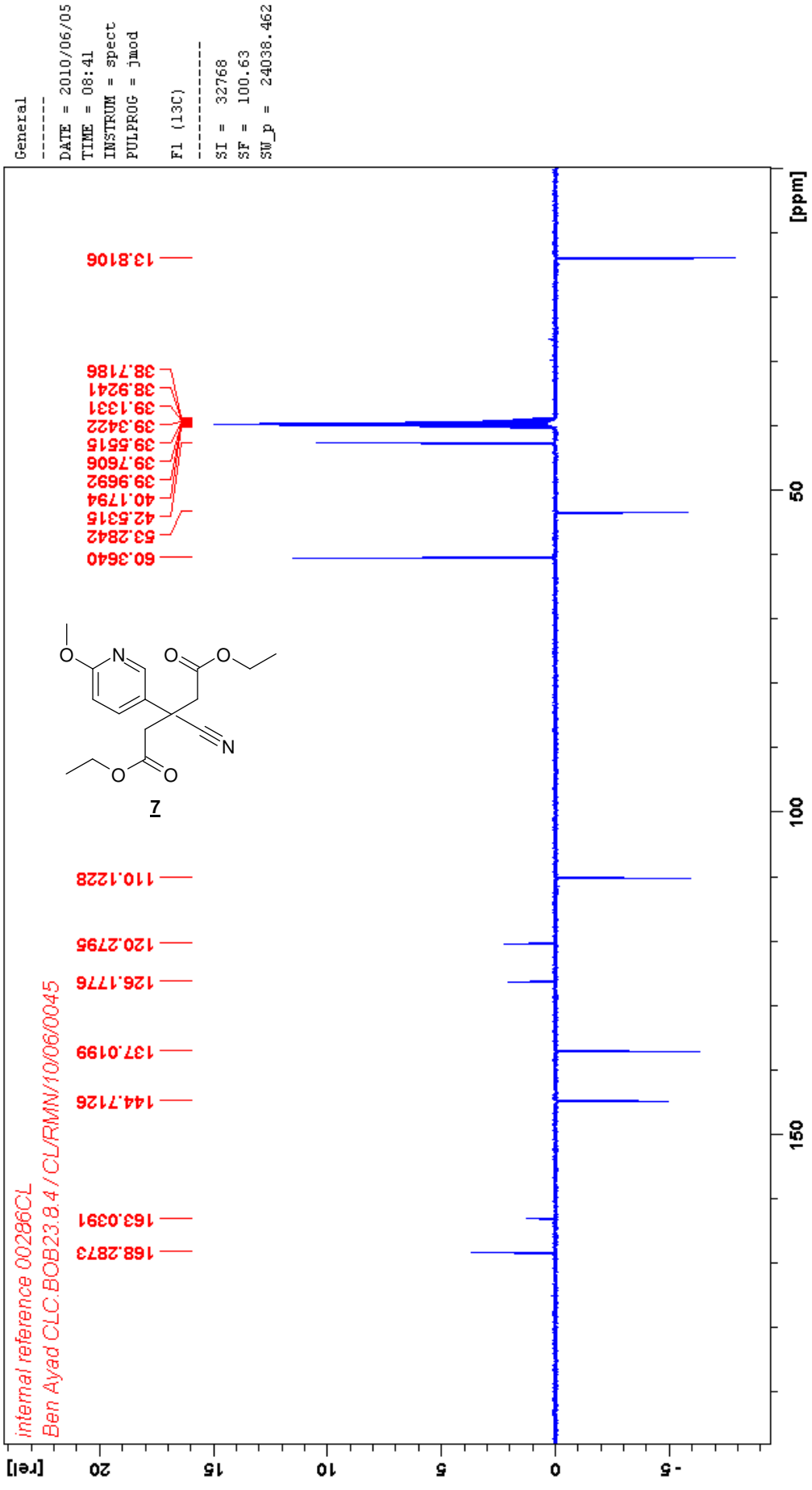
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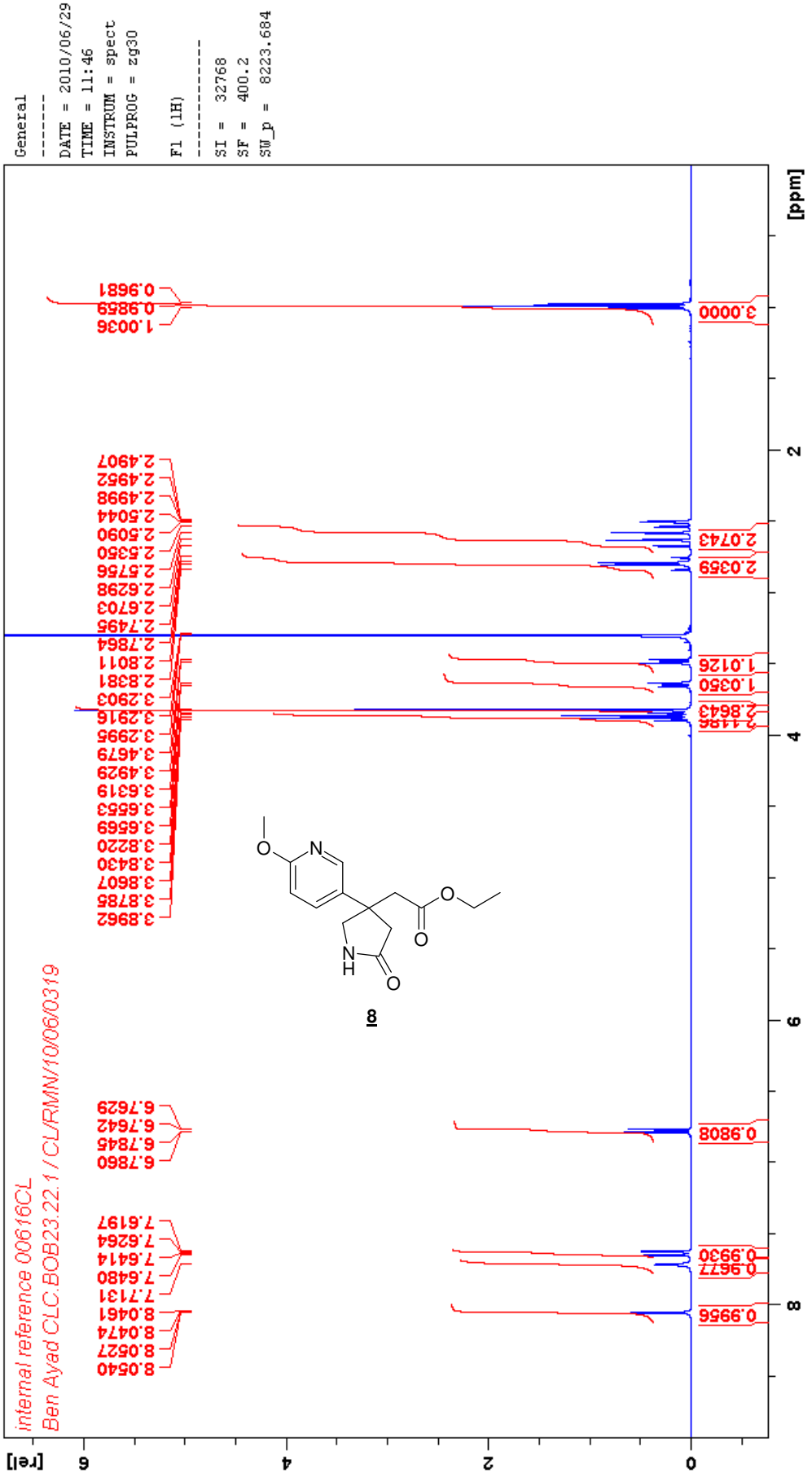
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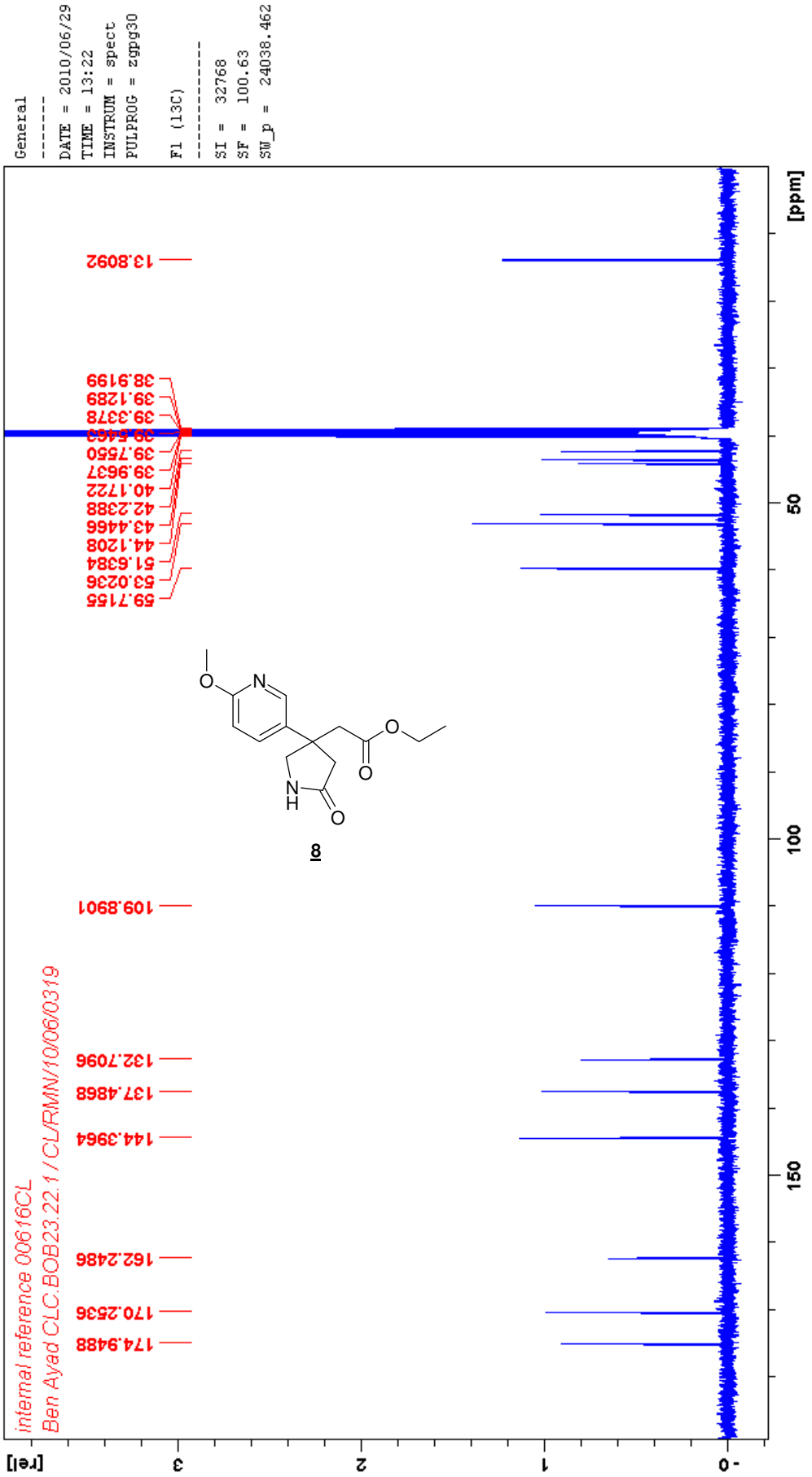
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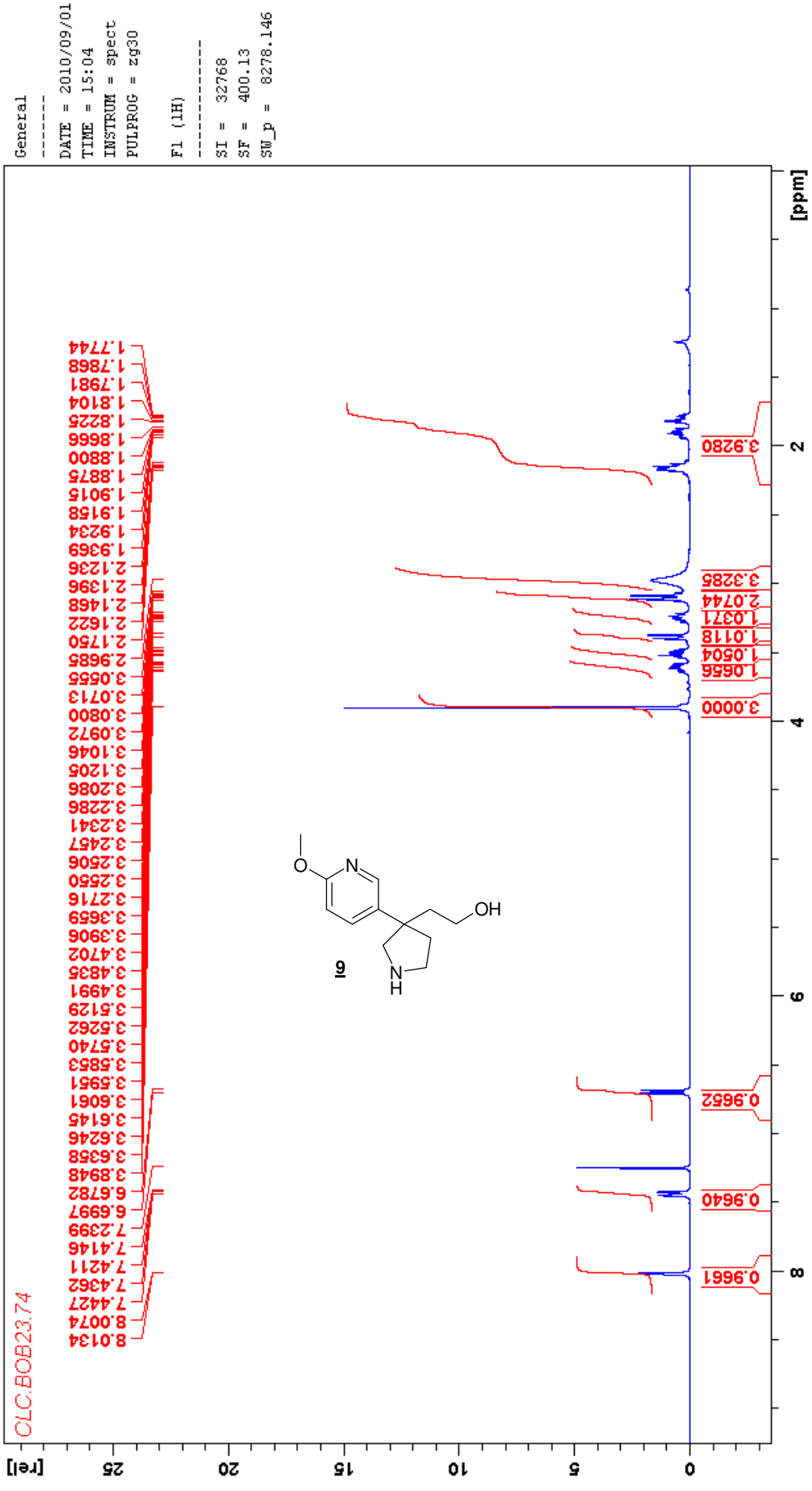
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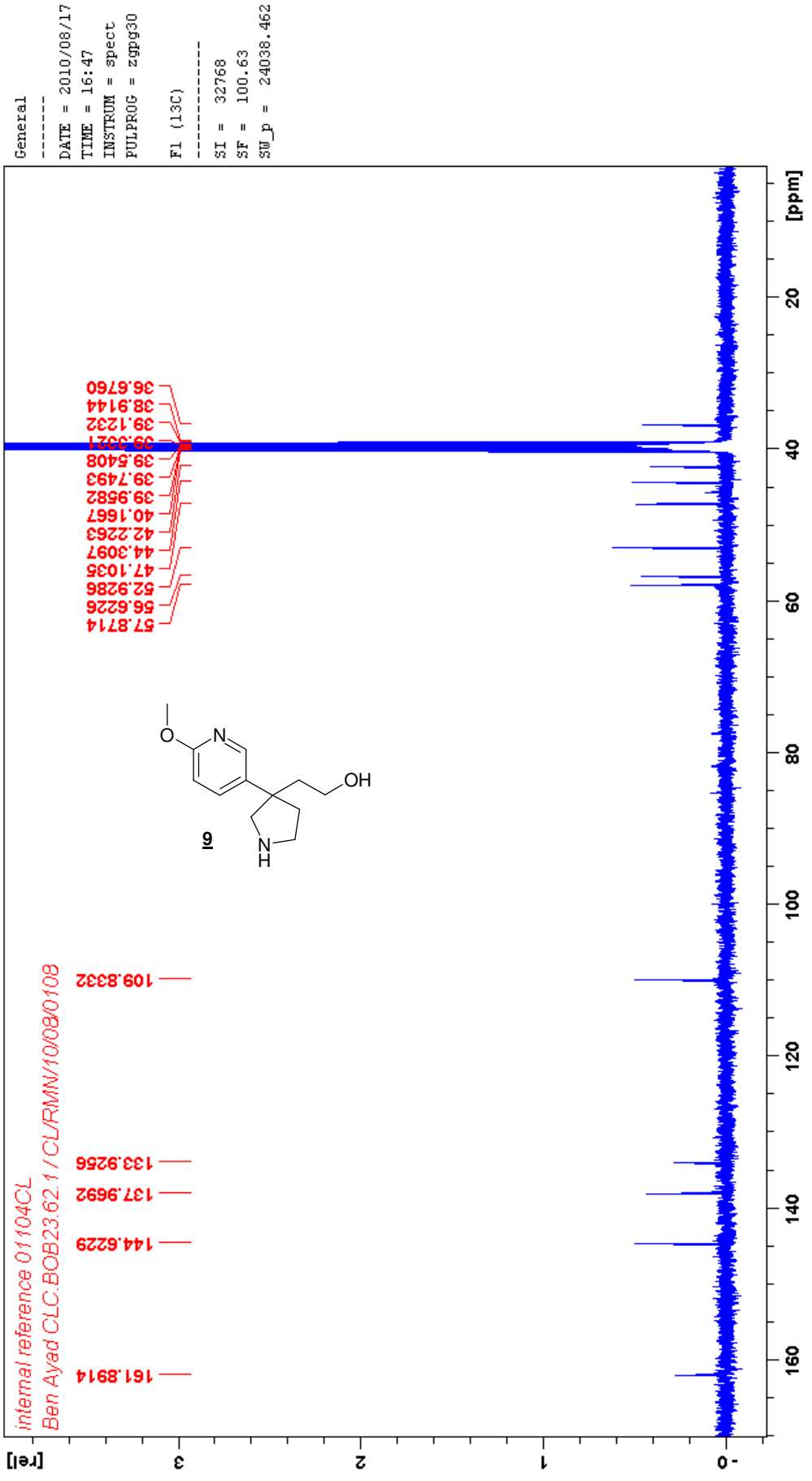
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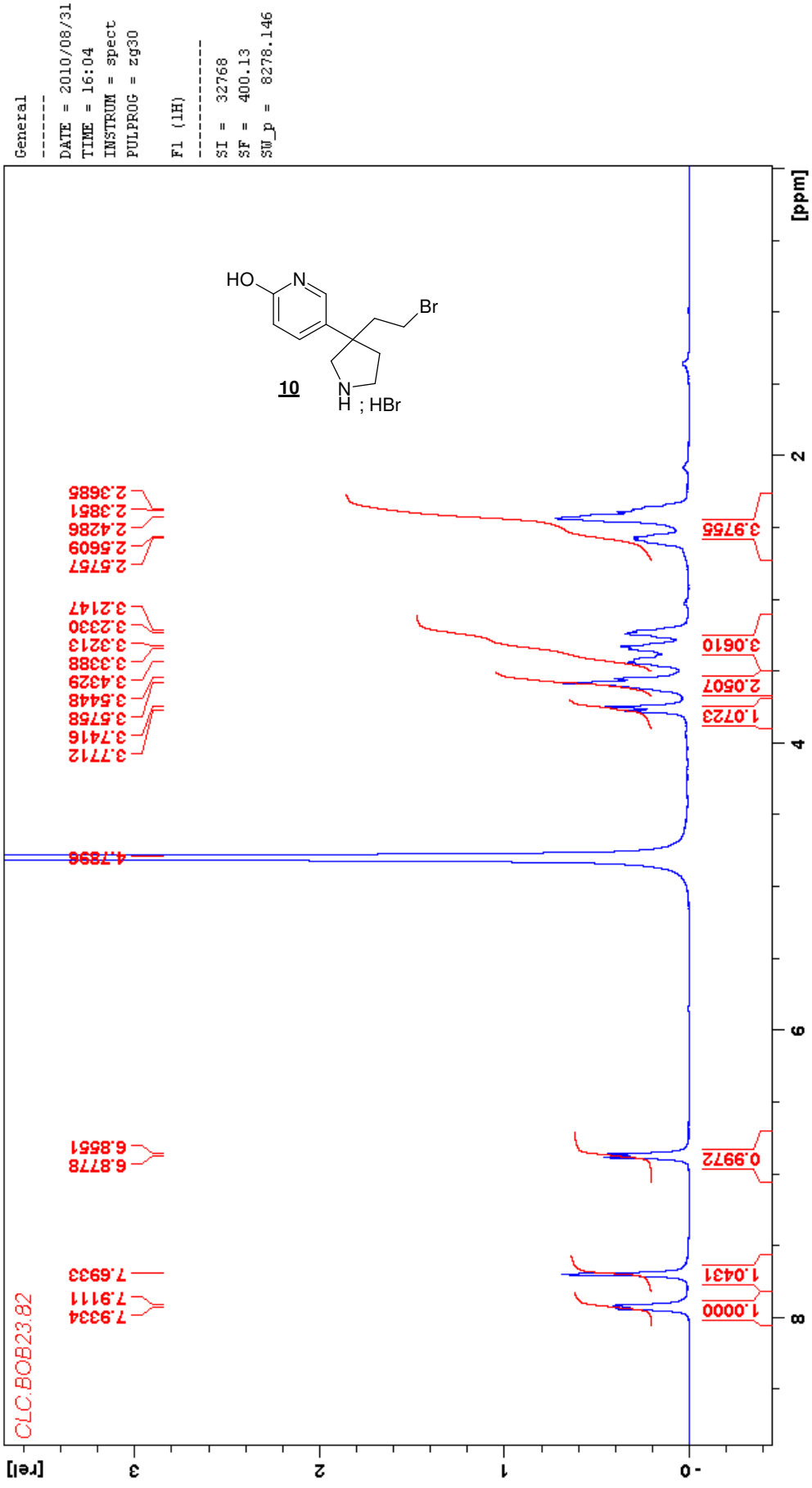
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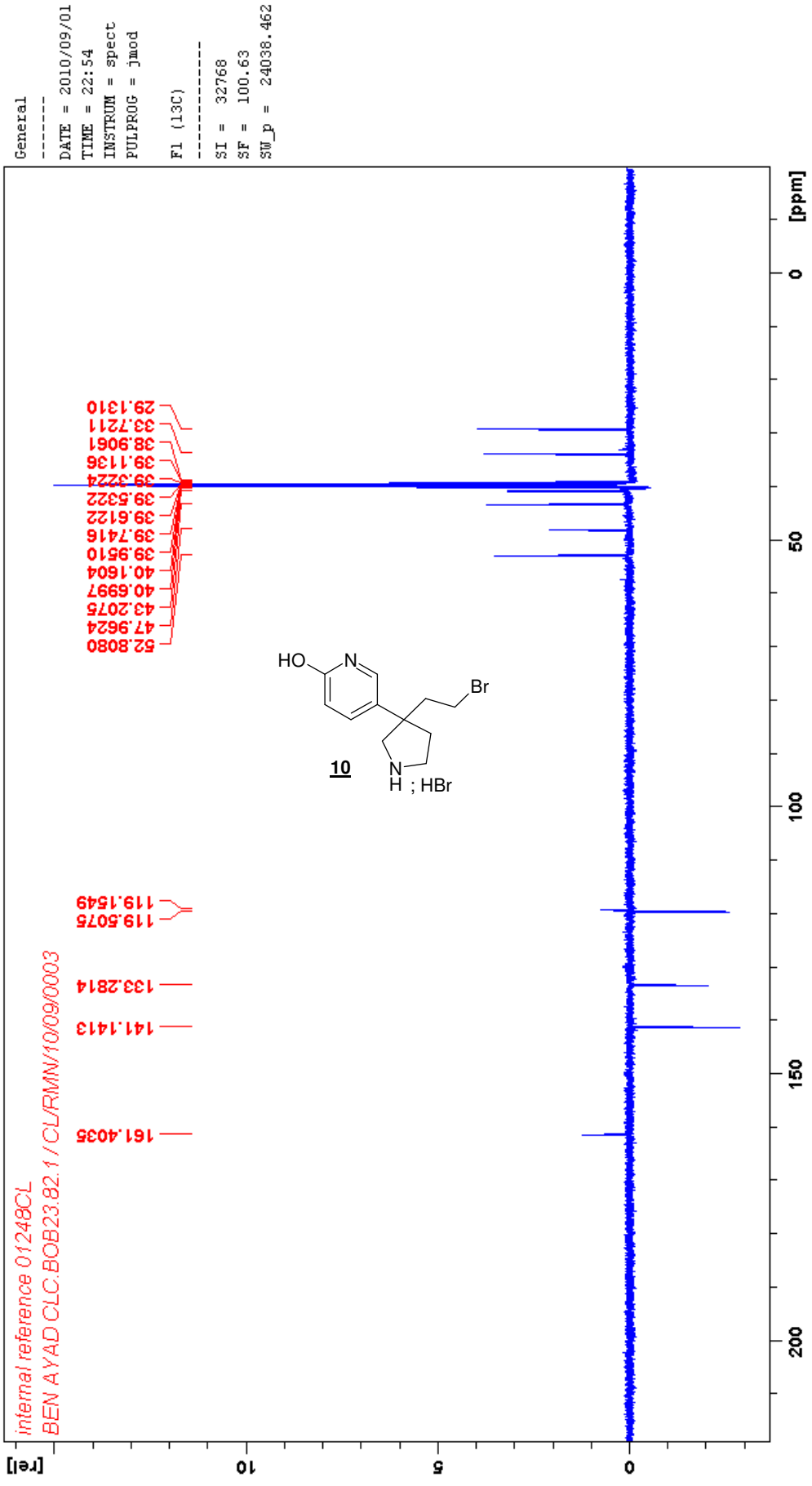
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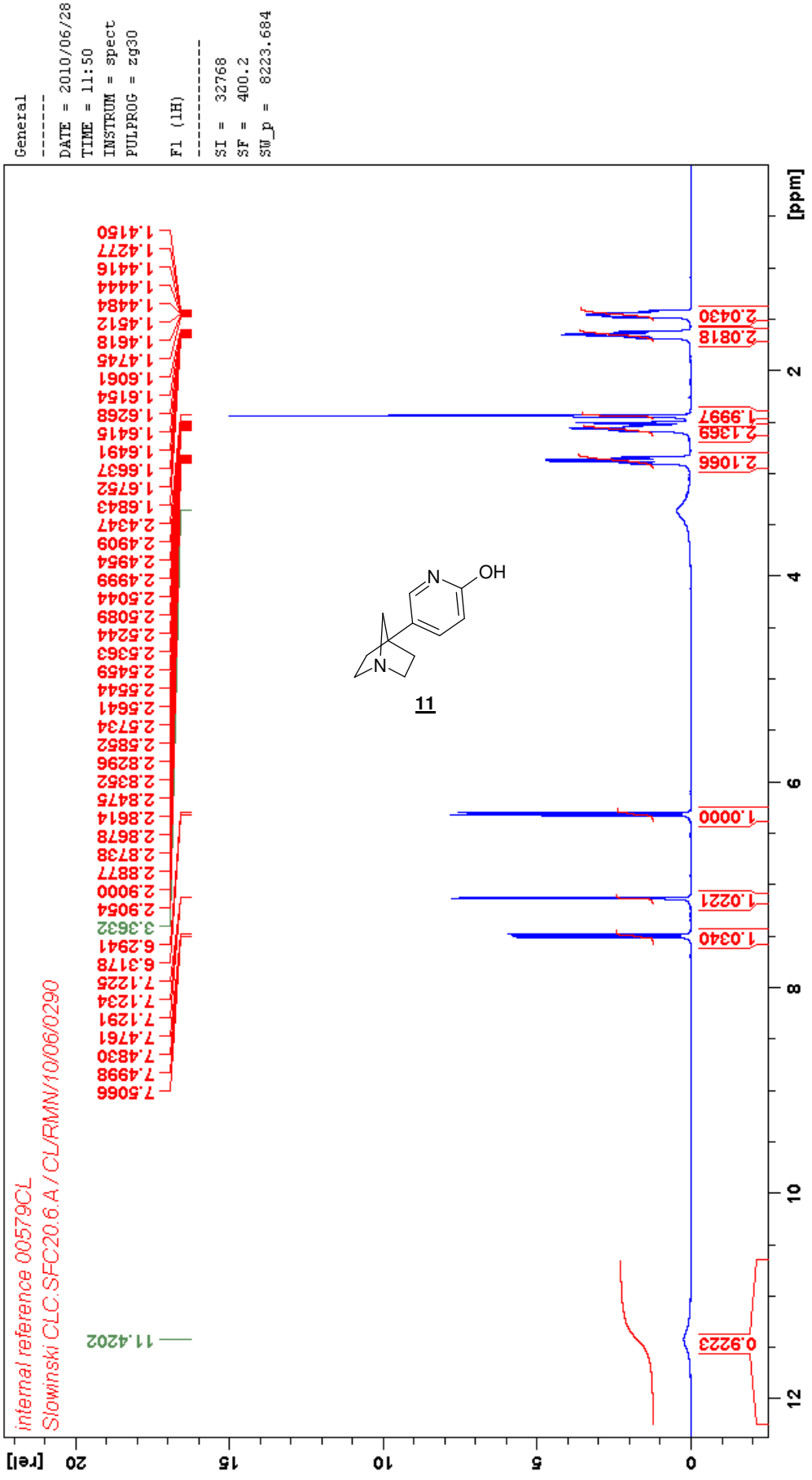
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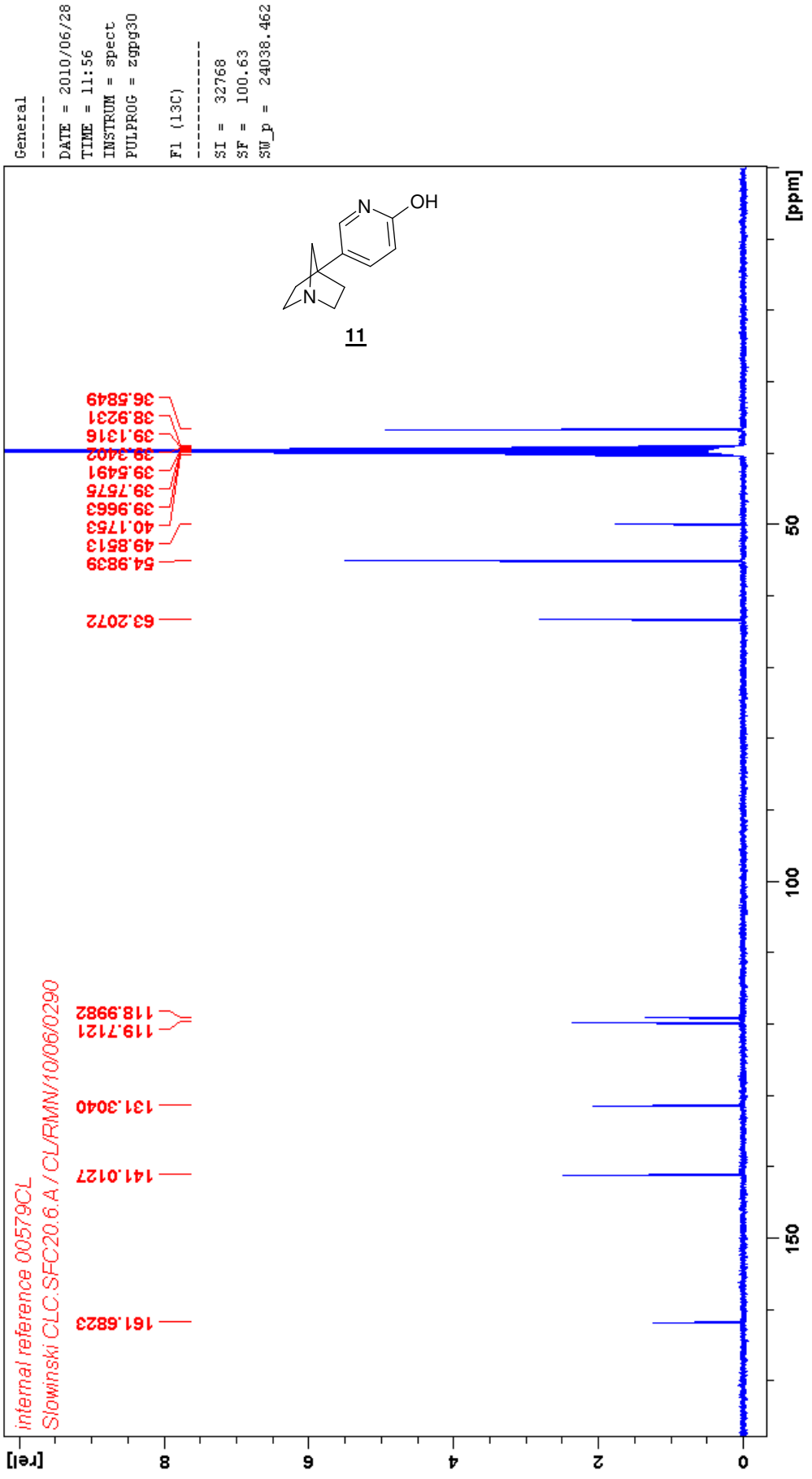
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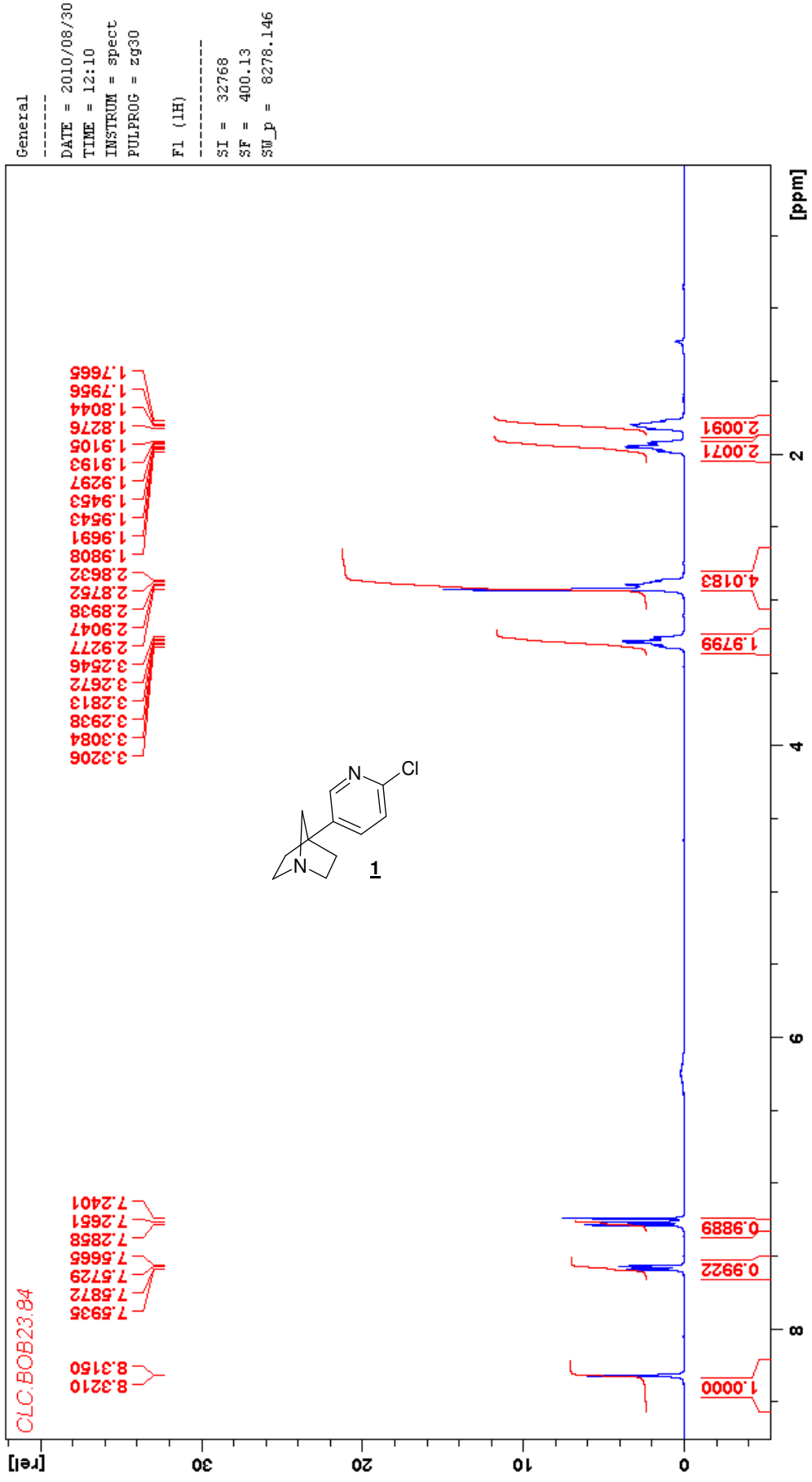
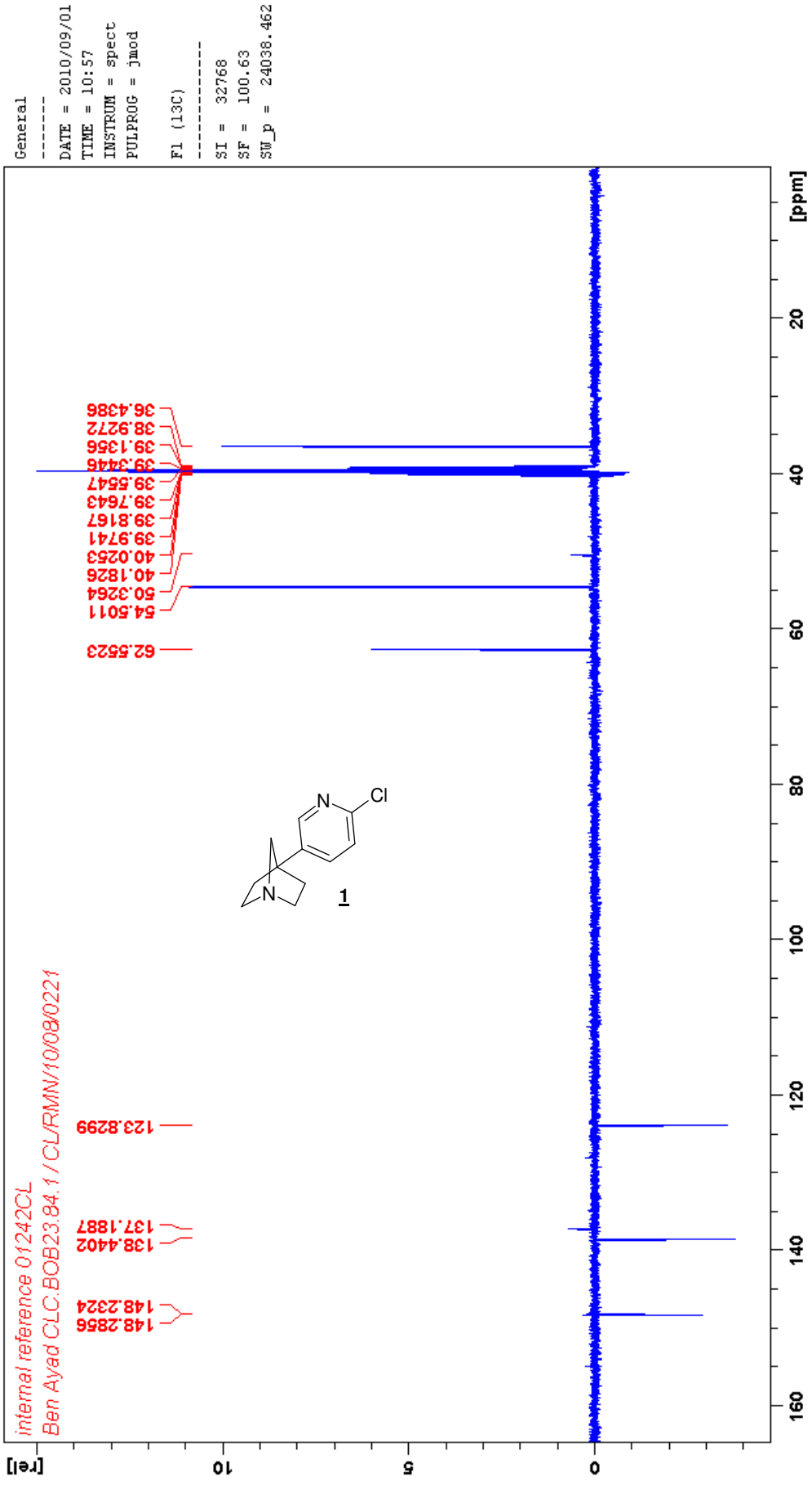
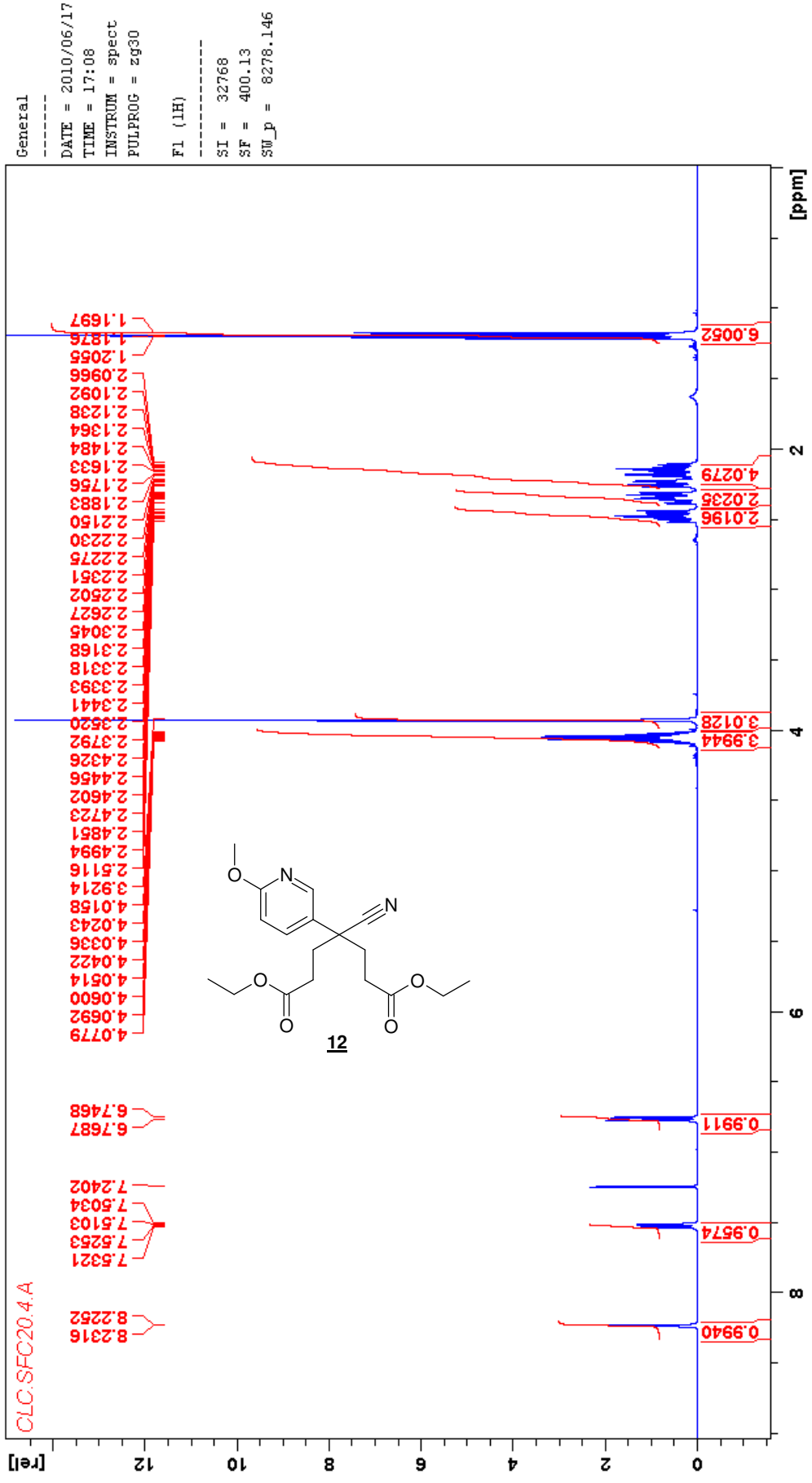
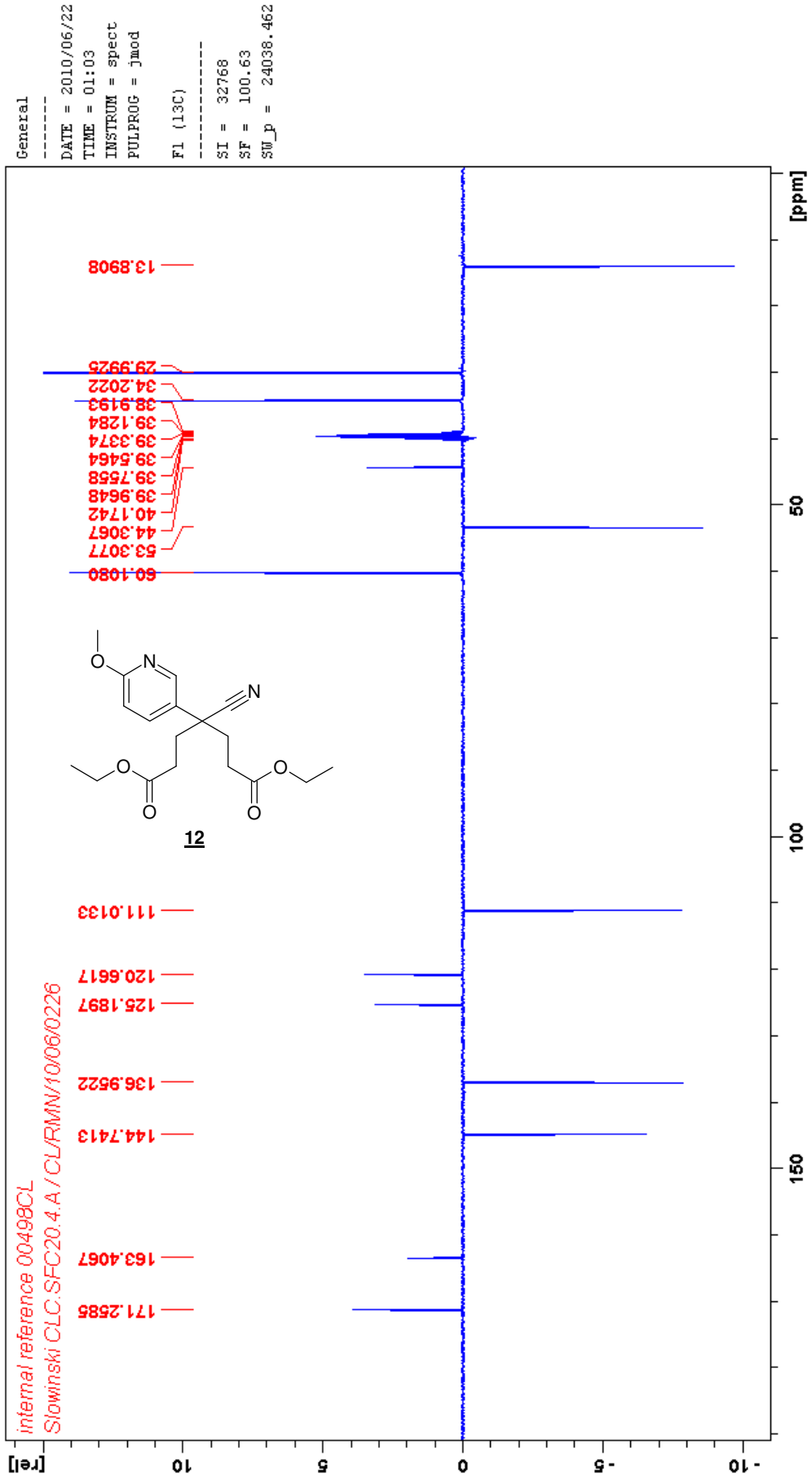
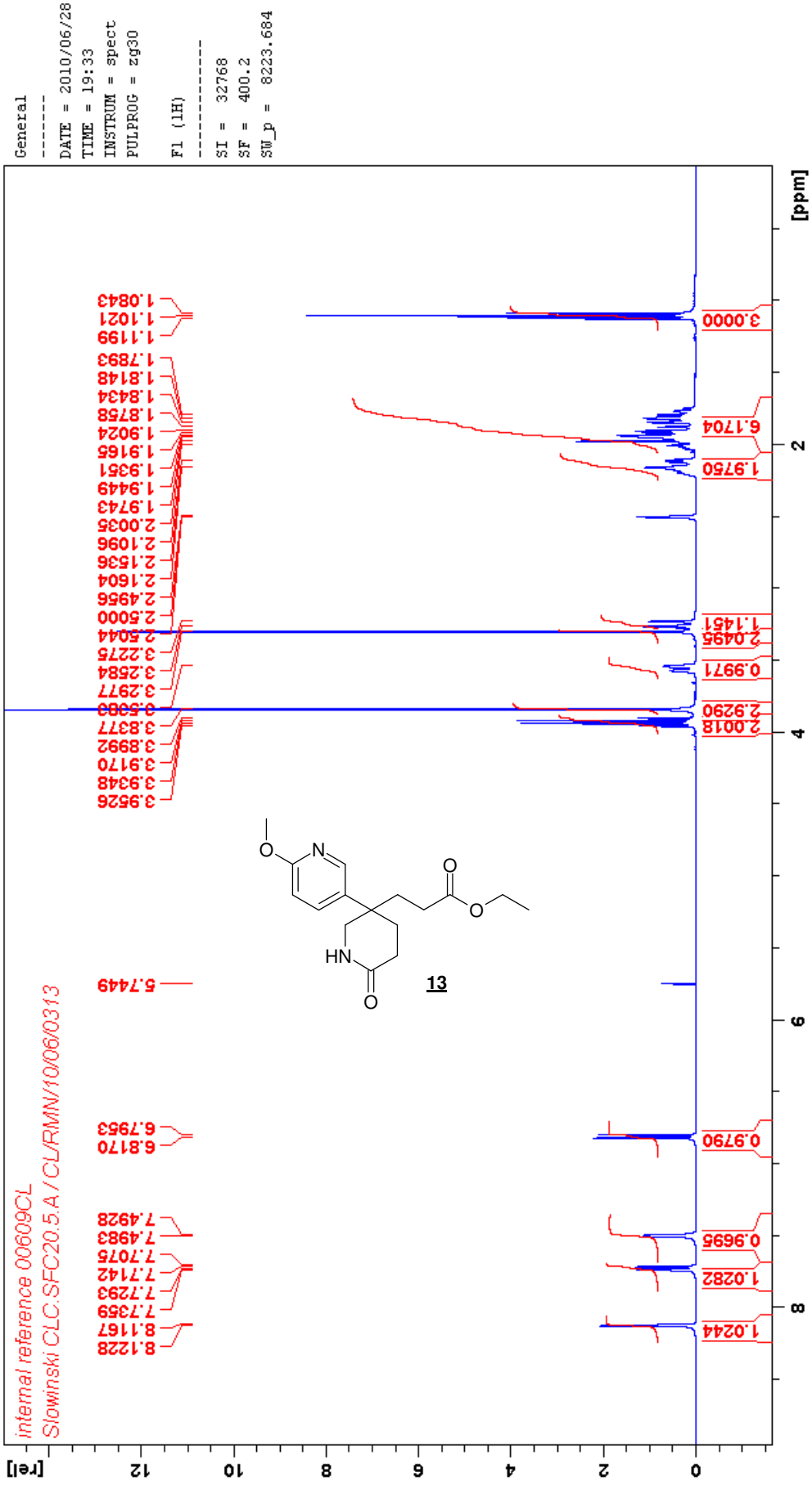
54.9839
63.2072
118.9982
119.7121
131.3040
141.0127
161.6823
internal reference 00579CL
Slowinski CLC.SFC20.6.A / CL/RMN/10/06/0290
[rel]
[ppm]

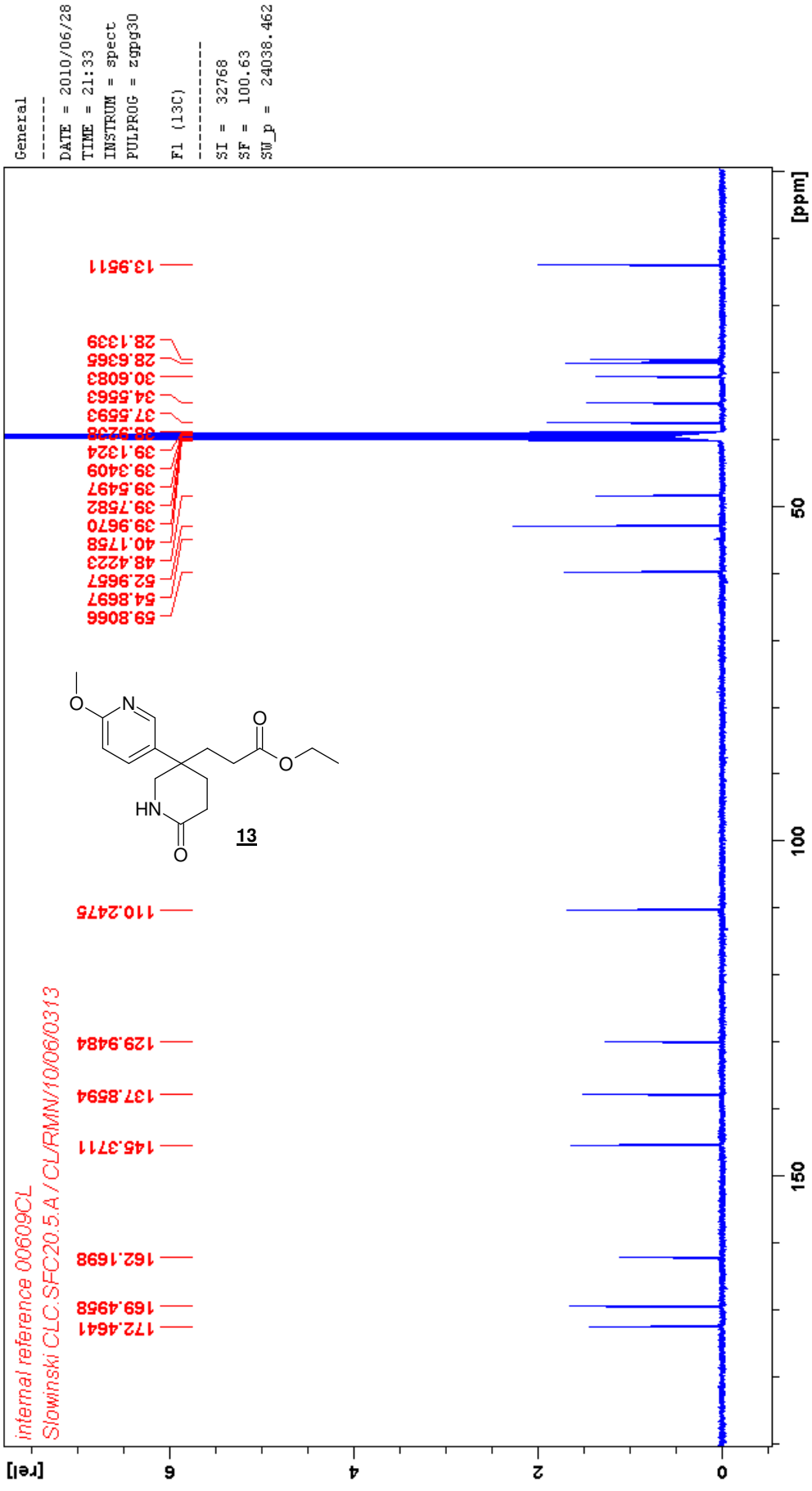

General
DATE = 2010/08/30
TIME = 12:10
INSTRUM = spect
PULPROG = zg30
F1 (1H)
SI = 32768
SF = 400.13
SW_p = 8278.146
CLC.BOB23.84
[rel]
[ppm]
1.0000
0.9922
0.9889
2.0071
2.0091
4.0183
1.9799
8.3210
8.3150
7.5935
7.5872
7.5729
7.5665
7.2858
7.2651
7.2401
3.3206
3.3084
3.2938
3.2813
3.2672
3.2546
2.9277
2.9047
2.8938
2.8752
2.8632
1.9808
1.9691
1.9543
1.9453
1.9297
1.9193
1.9105
1.8276
1.8044
1.7956
1.7665
N
Cl
N
1

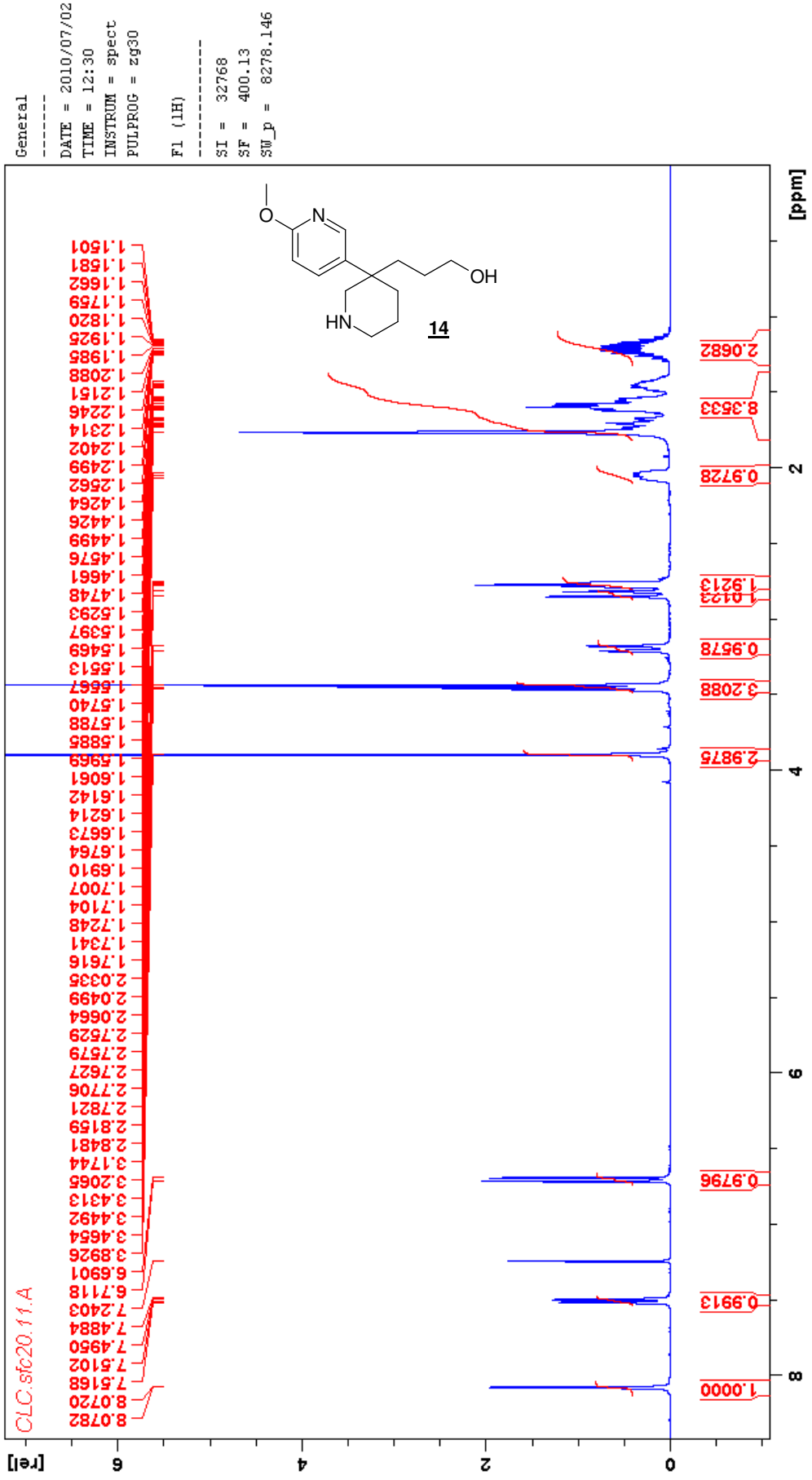

General
DATE = 2010/09/01
TIME = 10:57
INSTRUM = spect
PULPROG = jmod
F1 (13C)
SI = 32768
SF = 100.63
SW_p = 24038.462
internal reference 01242CL
Ben Ayad CLC.BOB23.84.1 / CL/RMN/10/08/0221
148.2856
148.2324
138.4402
137.1887
123.8299
62.5523
54.5011
50.3264
40.1826
40.0253
39.9741
39.8167
39.7643
39.5547
39.3446
39.1356
38.9272
36.4386
Cl
N
N
1
[rel]
[ppm]

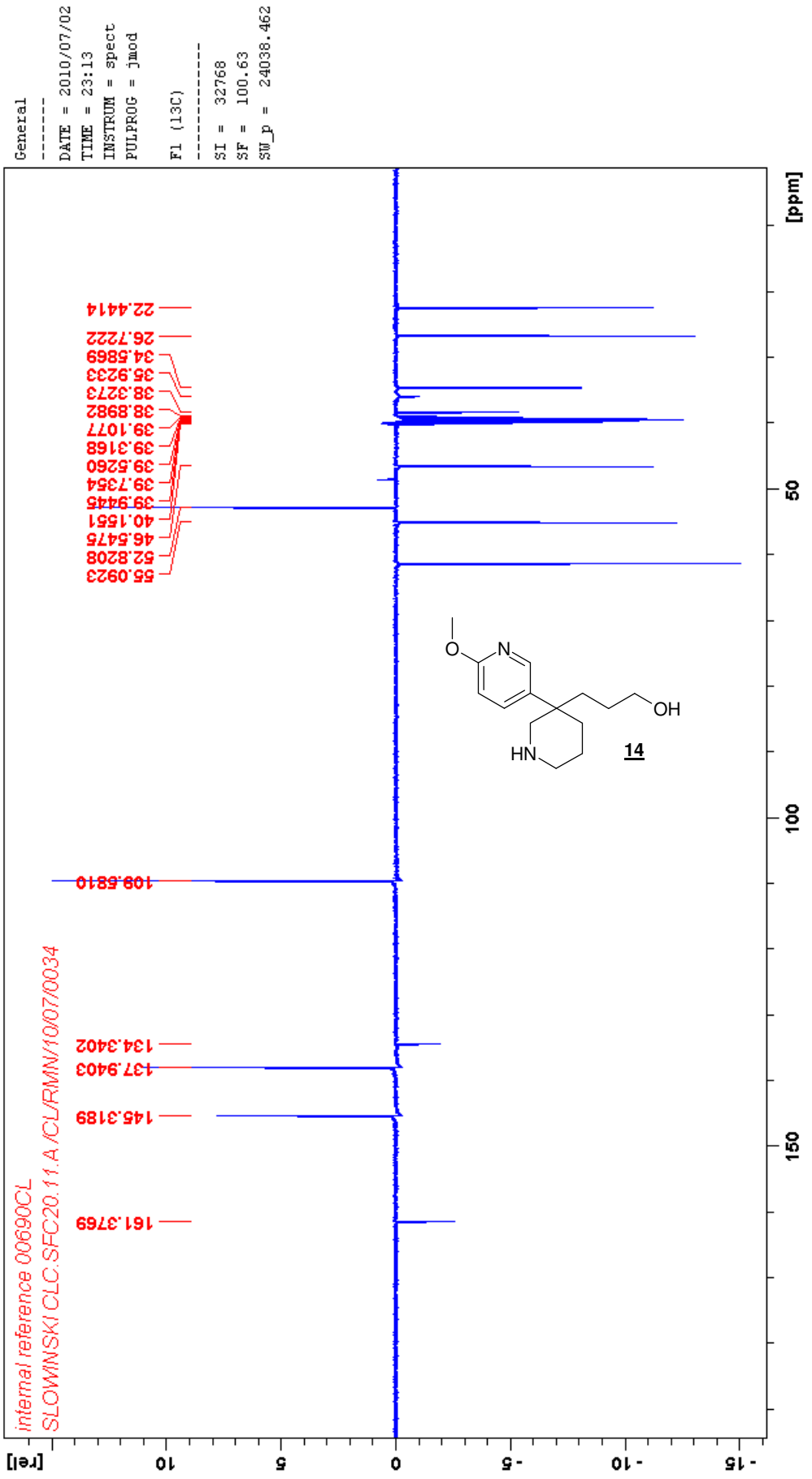

General
DATE = 2010/06/17
TIME = 17:08
INSTRUM = spect
PULPROG = zg30
F1 (1H)
SI = 32768
SF = 400.13
SW_p = 8278.146
CLC.SFC20.4.A
[rel]
[ppm]
12
0.9940
0.9574
0.9911
3.9944
3.0128
2.0196
2.0235
4.0279
6.0052

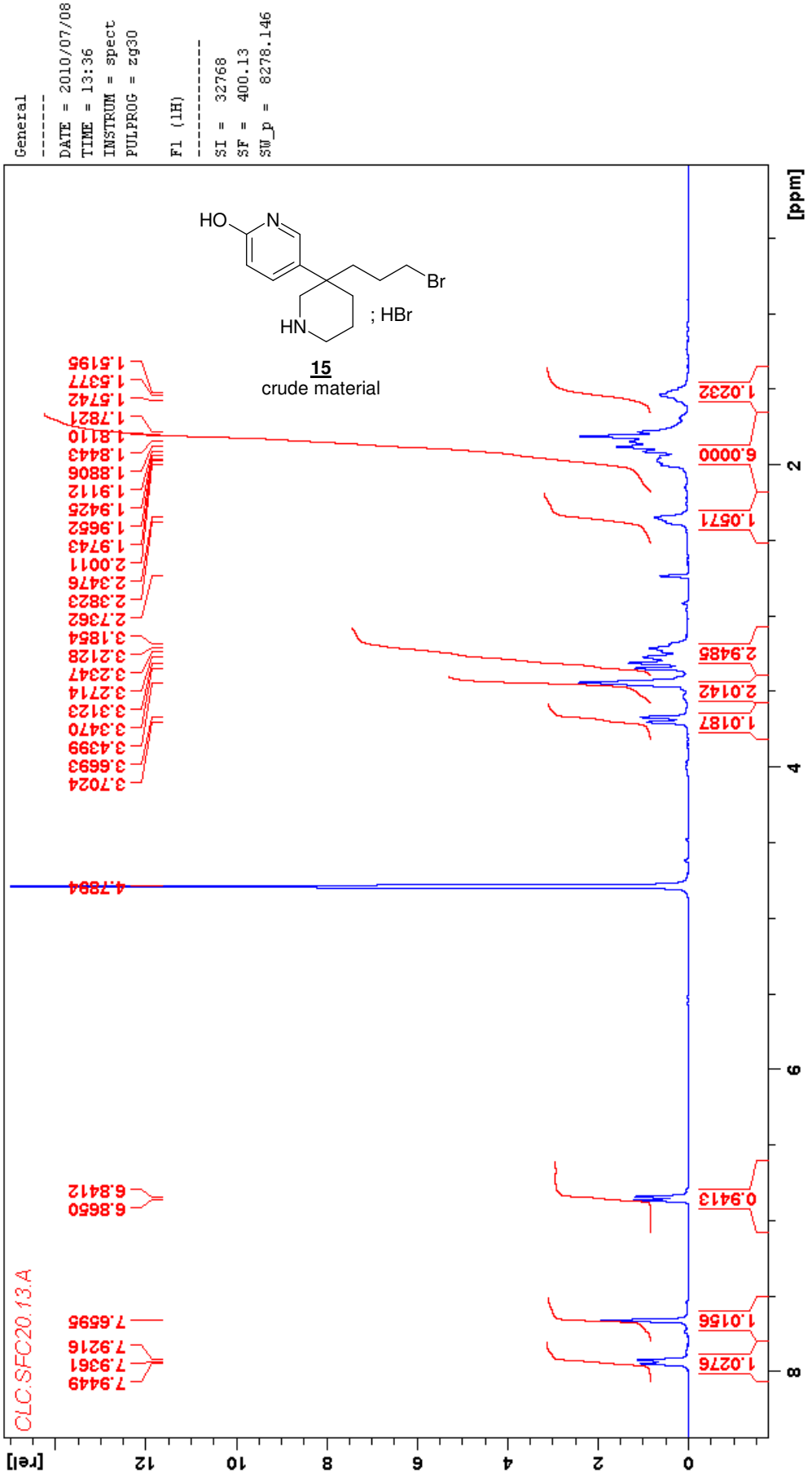

General
DATE = 2010/06/22
TIME = 01:03
INSTRUM = spect
PULPROG = jmod
F1 (13C)
SI = 32768
SF = 100.63
SW_p = 24038.462
13.8908
29.9925
34.2022
38.9193
39.1284
39.3374
39.5464
39.7558
39.9648
40.1742
44.3067
53.3077
60.1080
111.0133
120.6617
125.1897
136.9522
144.7413
163.4067
171.2585
internal reference 00498CL
Slowinski CLC.SFC20.4.A / CL/RMN/10/06/0226
[rel]
[ppm]
12

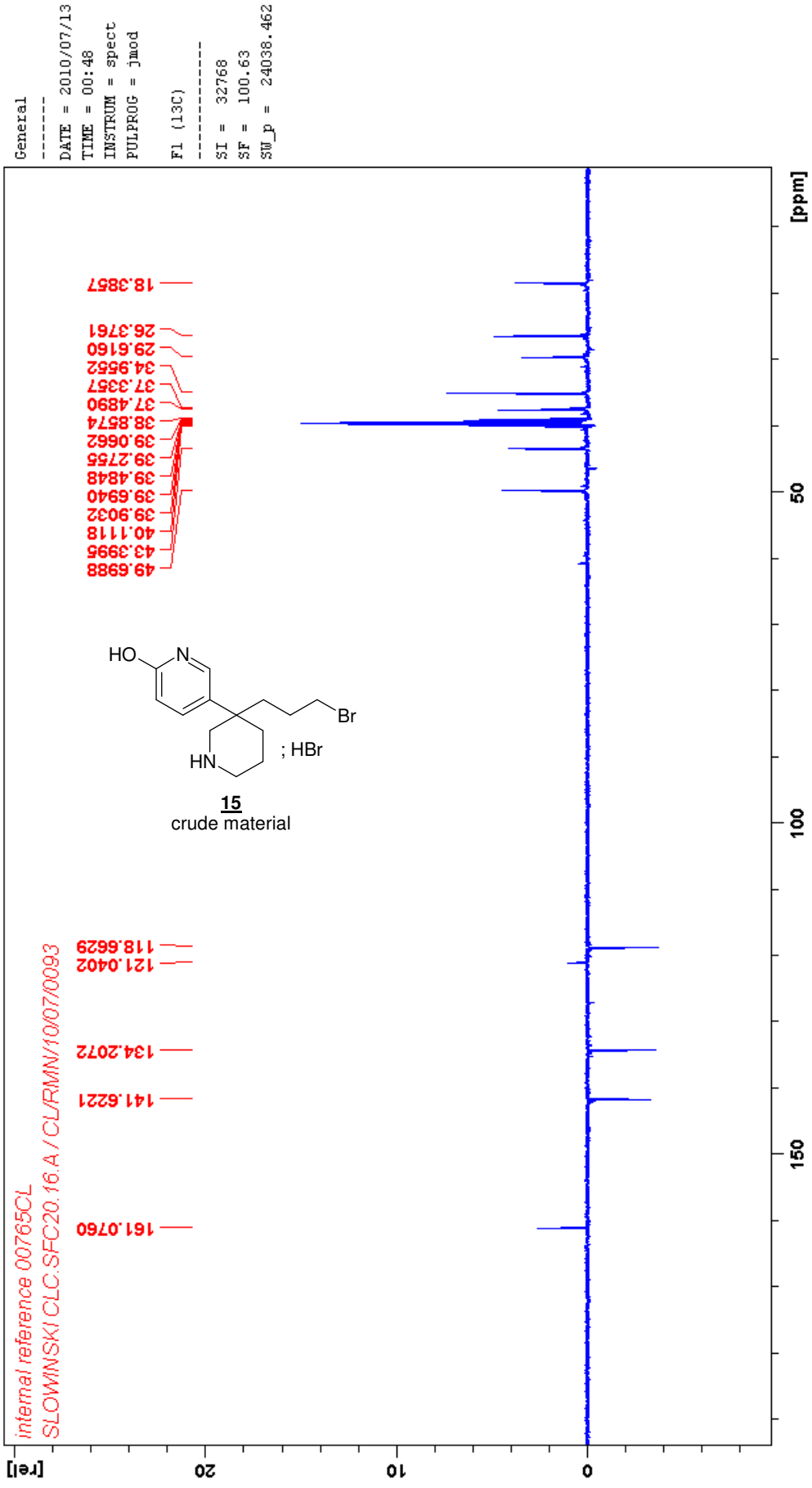

General
DATE = 2010/06/28
TIME = 19:33
INSTRUM = spect
PULPROG = zg30
F1 (1H)
SI = 32768
SF = 400.2
SW_p = 8223.684
internal reference 00609CL
Slowinski CLC.SFC20.5.A / CL/RMN/10/06/0313
[rel]
[ppm]
13

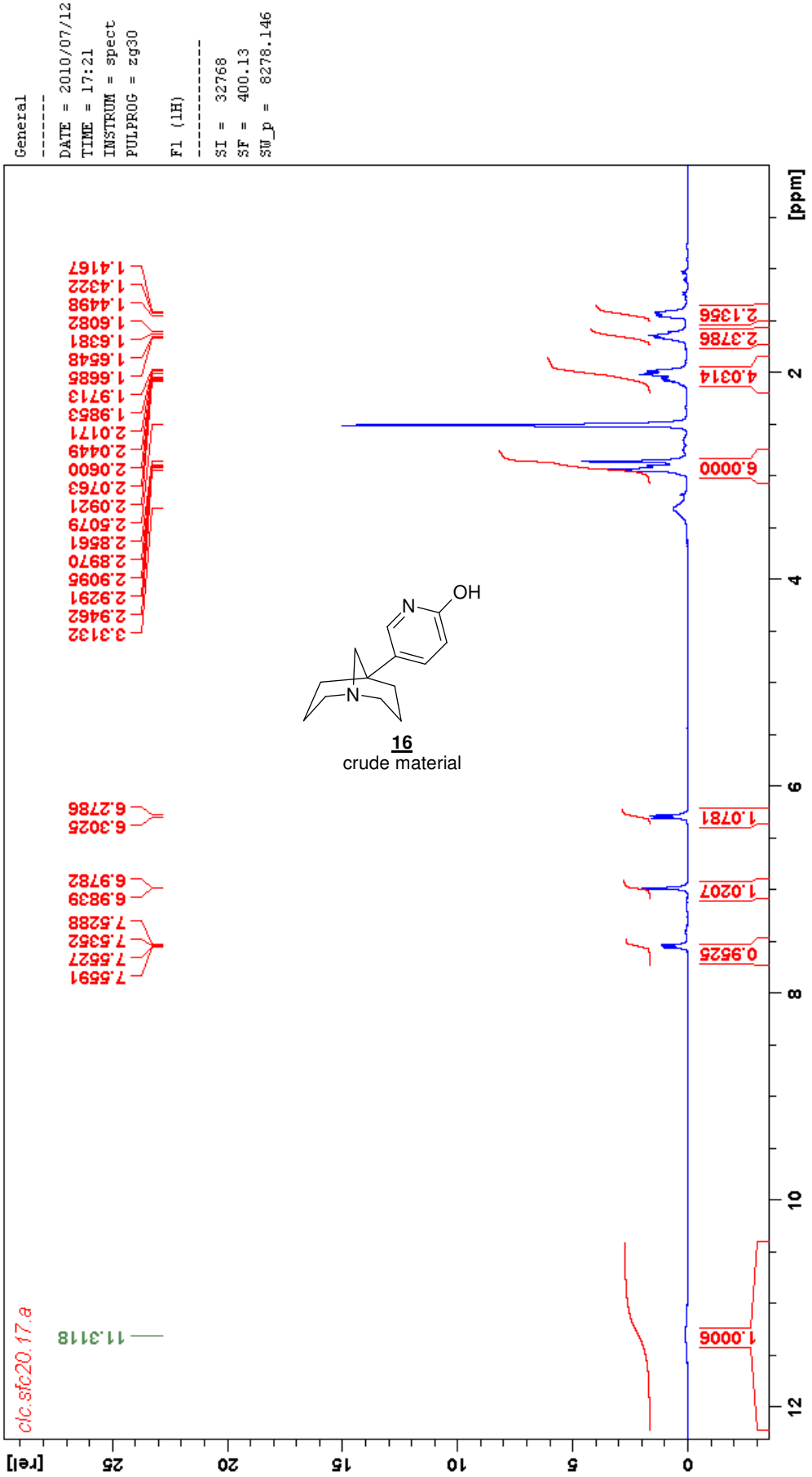

General
DATE = 2010/06/28
TIME = 21:33
INSTRUM = spect
PULPROG = zgpg30
F1 (13C)
SI = 32768
SF = 100.63
SW_p = 24038.462
13.9511
28.1339
28.6365
30.6083
34.5563
37.5593
39.1324
39.3409
39.5497
39.7582
39.9670
40.1758
48.4223
52.9657
54.8697
59.8066
110.2475
129.9484
137.8594
145.3711
162.1698
169.4958
172.4641
13
internal reference 00609CL
Slowinski CLC.SFC20.5.A / CL/RMN/10/06/0313
[rel]
[ppm]


General
DATE = 2010/07/02
TIME = 12:30
INSTRUM = spect
PULPROG = zg30
F1 (1H)
SI = 32768
SF = 400.13
SW_p = 8278.146
O
N
OH
HN
14
[ppm]
[rel]
CLC sfc20 11.A
1.0000
0.9913
0.9796
3.2065
2.9875
3.2088
0.9578
1.9213
1.0123
0.9728
8.3533
2.0682

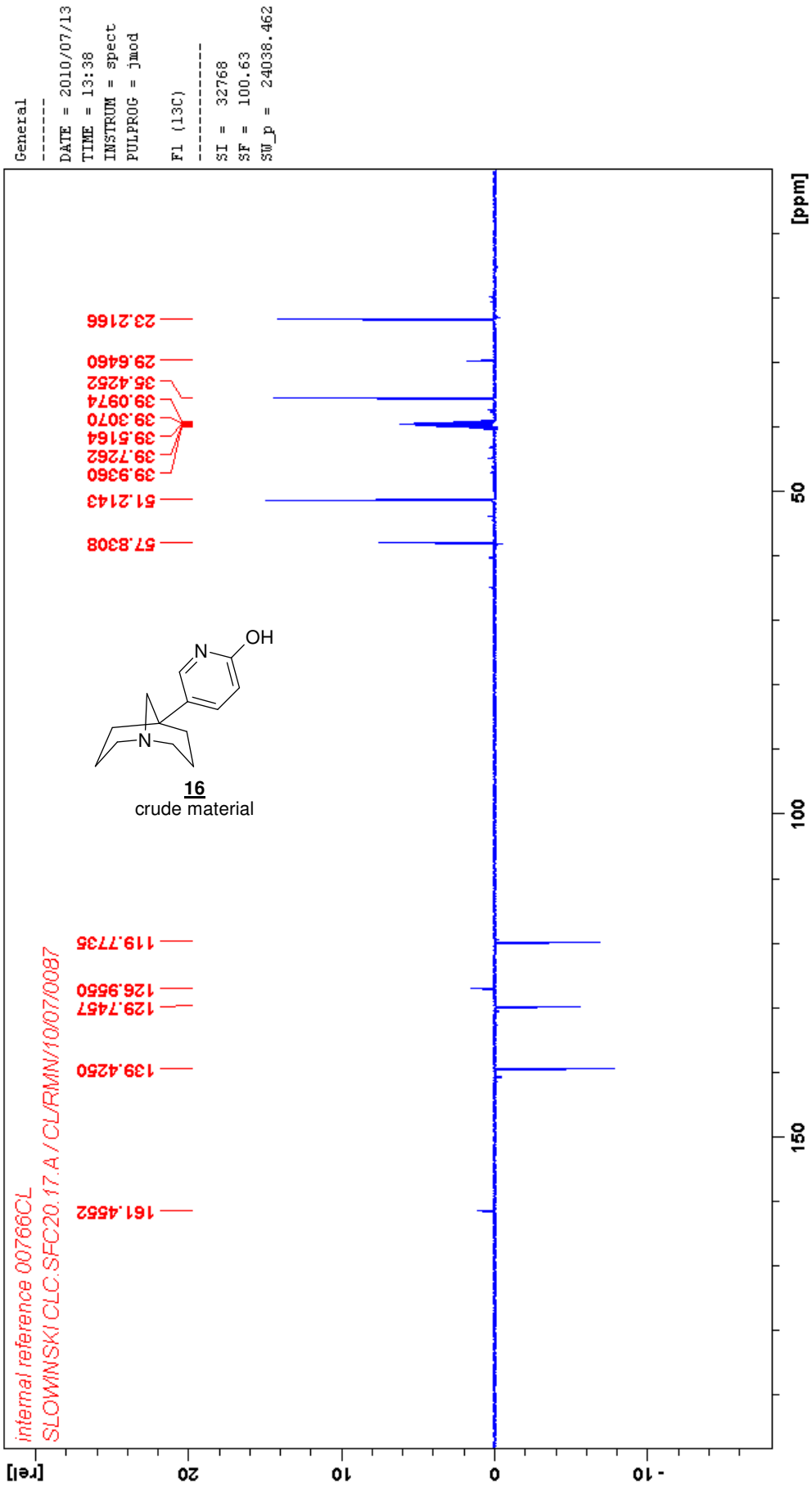

General
DATE = 2010/07/02
TIME = 23:13
INSTRUM = spect
PULPROG = jmod
F1 (13C)
SI = 32768
SF = 100.63
SW_p = 24038.462
22.4414
26.7222
34.5869
35.9233
38.3273
38.8982
39.1077
39.3168
39.5260
39.7354
39.9445
40.1551
46.5475
52.8208
55.0923
109.5810
134.3402
137.9403
145.3189
161.3769
internal reference 00690CL
SLOWINSKI CLC.SFC20.11.A /CL/RMN/10/07/0034
rel
[ppm]
O
N
OH
HN
14

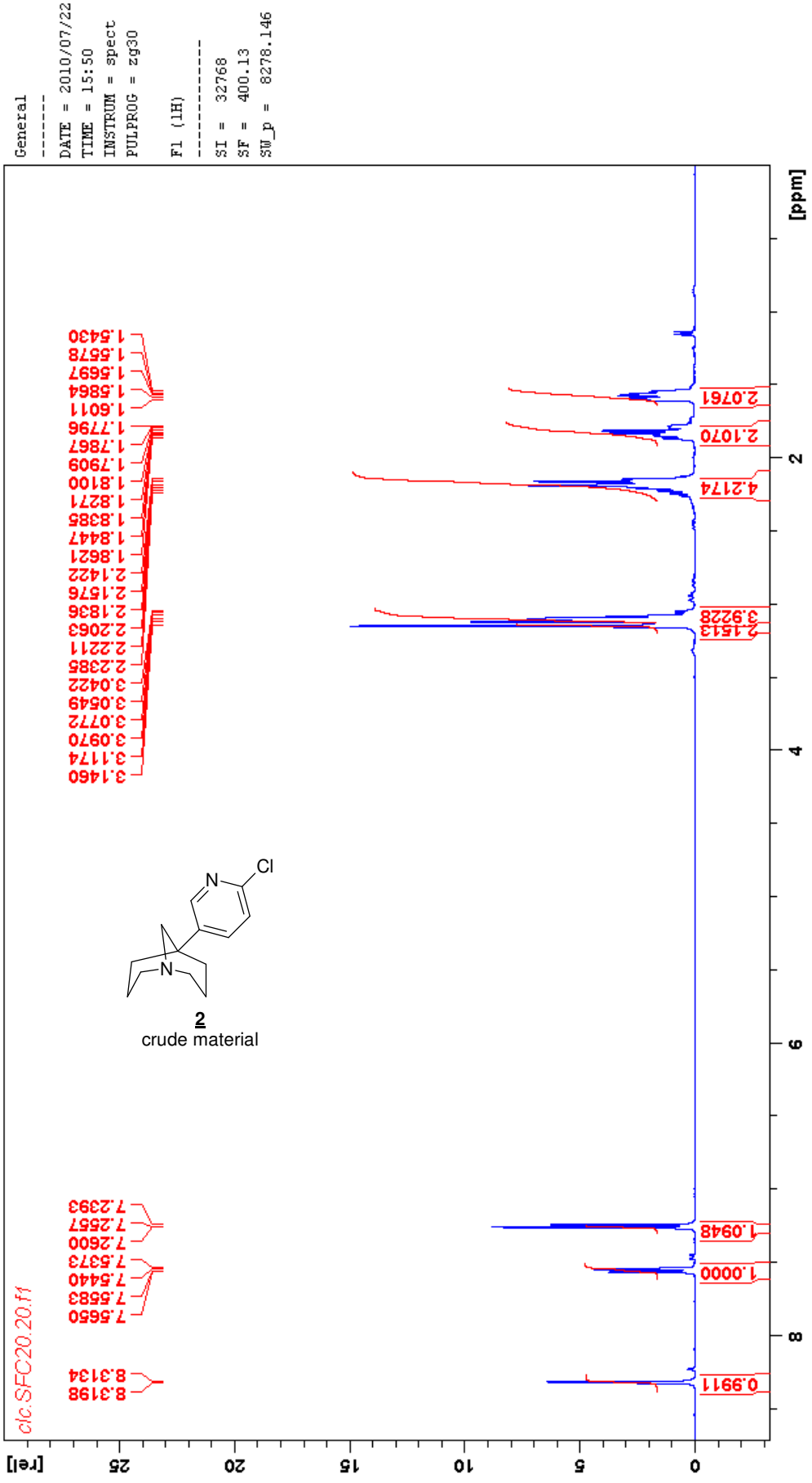

General
DATE = 2010/07/08
TIME = 13:36
INSTRUM = spect
PULPROG = zg30
F1 (1H)
SI = 32768
SF = 400.13
SW_p = 8278.146
HO
N
Br
HN
; HBr
15
crude material
CLC.SFC20.13.A
[rel]
[ppm]


General
DATE = 2010/07/13
TIME = 00:48
INSTRUM = spect
PULPROG = jmod
F1 (13C)
SI = 32768
SF = 100.63
SW_p = 24038.462
18.3857
26.3761
29.6160
34.9562
37.3357
37.4890
38.8574
39.0662
39.2755
39.4848
39.6940
39.9032
40.1118
43.3995
49.6988
HO
N
Br
HN
; HBr
15
crude material
118.6629
121.0402
134.2072
141.6221
161.0760
internal reference 00765CL
SLOWINSKI CLC.SFC20.16.A / CL/RMN/10/07/0093
[rel]
20
10
0
[ppm]
50
100
150

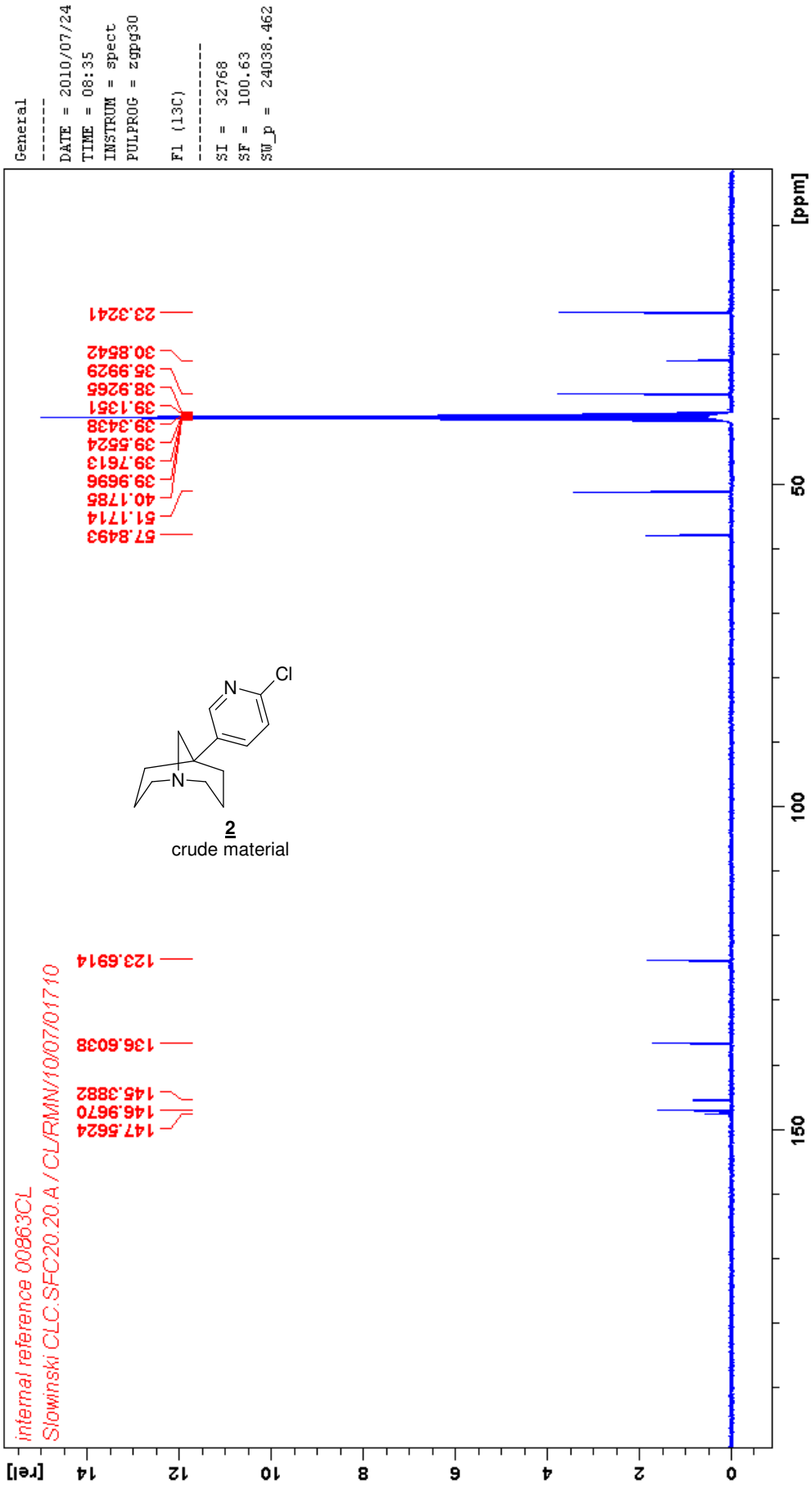


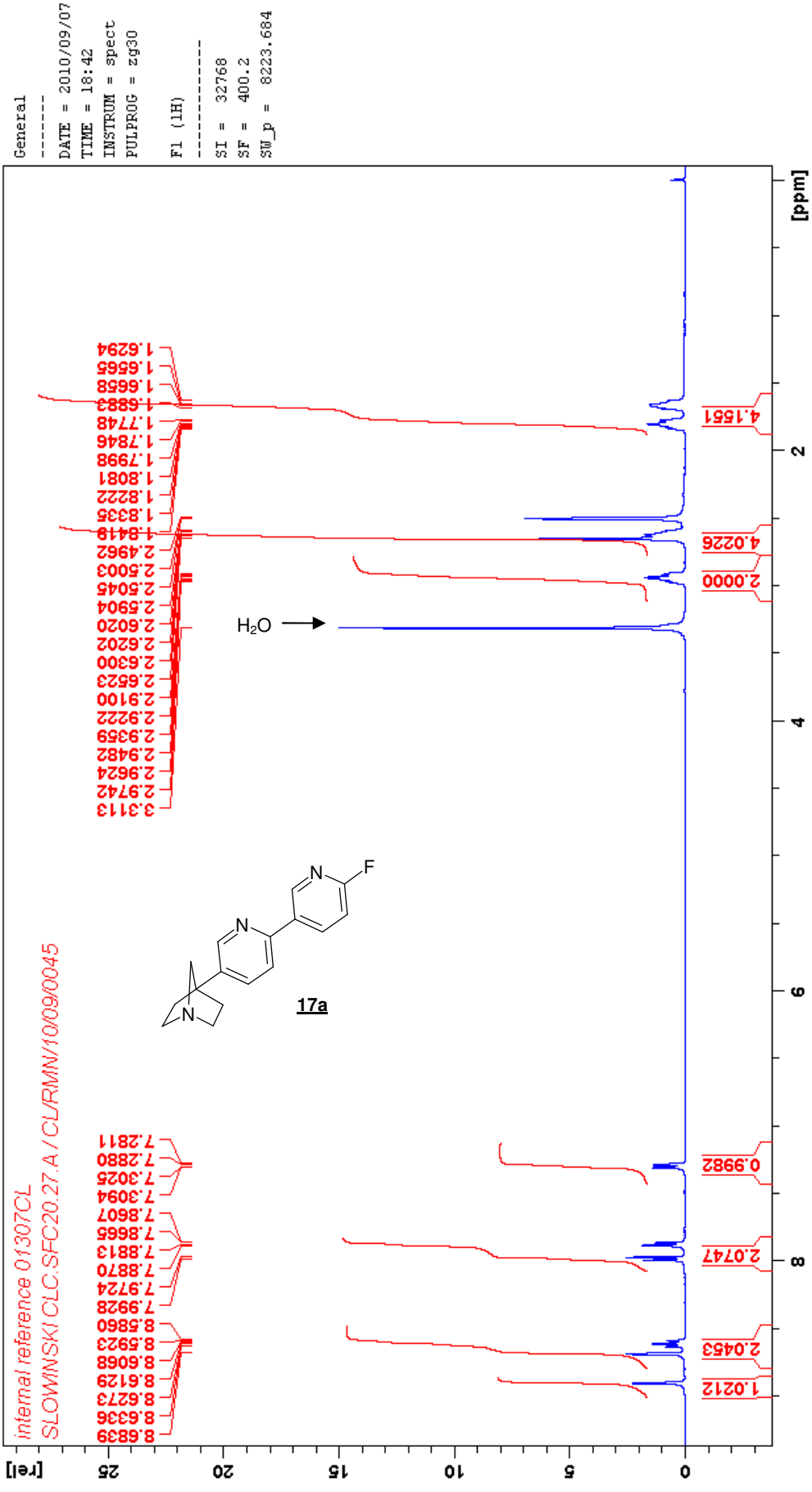
16
crude material

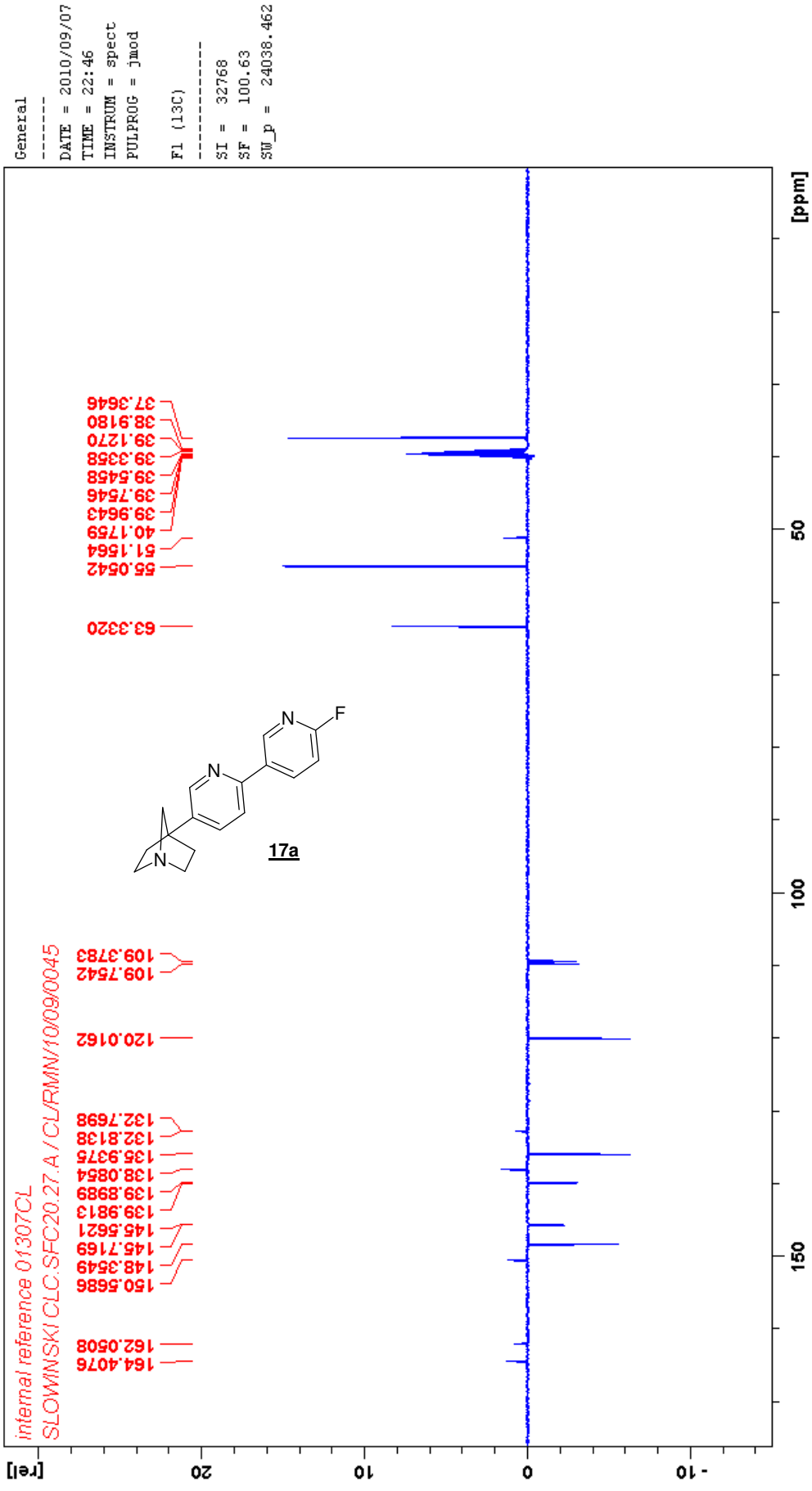

General
DATE = 2010/07/13
TIME = 13:38
INSTRUM = spect
PULPROG = jmod
F1 (13C)
SI = 32768
SF = 100.63
SW_p = 24038.462
23.2166
29.6460
35.4252
39.0974
39.3070
39.5164
39.7262
39.9360
51.2143
57.8308
119.7735
126.9550
129.7457
139.4250
161.4552
OH
N
N
16
crude material
internal reference 00766CL
SLOWINSKI CLC.SFC20.17.A / CL/RMN/10/07/0087
[rel]
20
10
0
-10
50
100
150
[ppm]

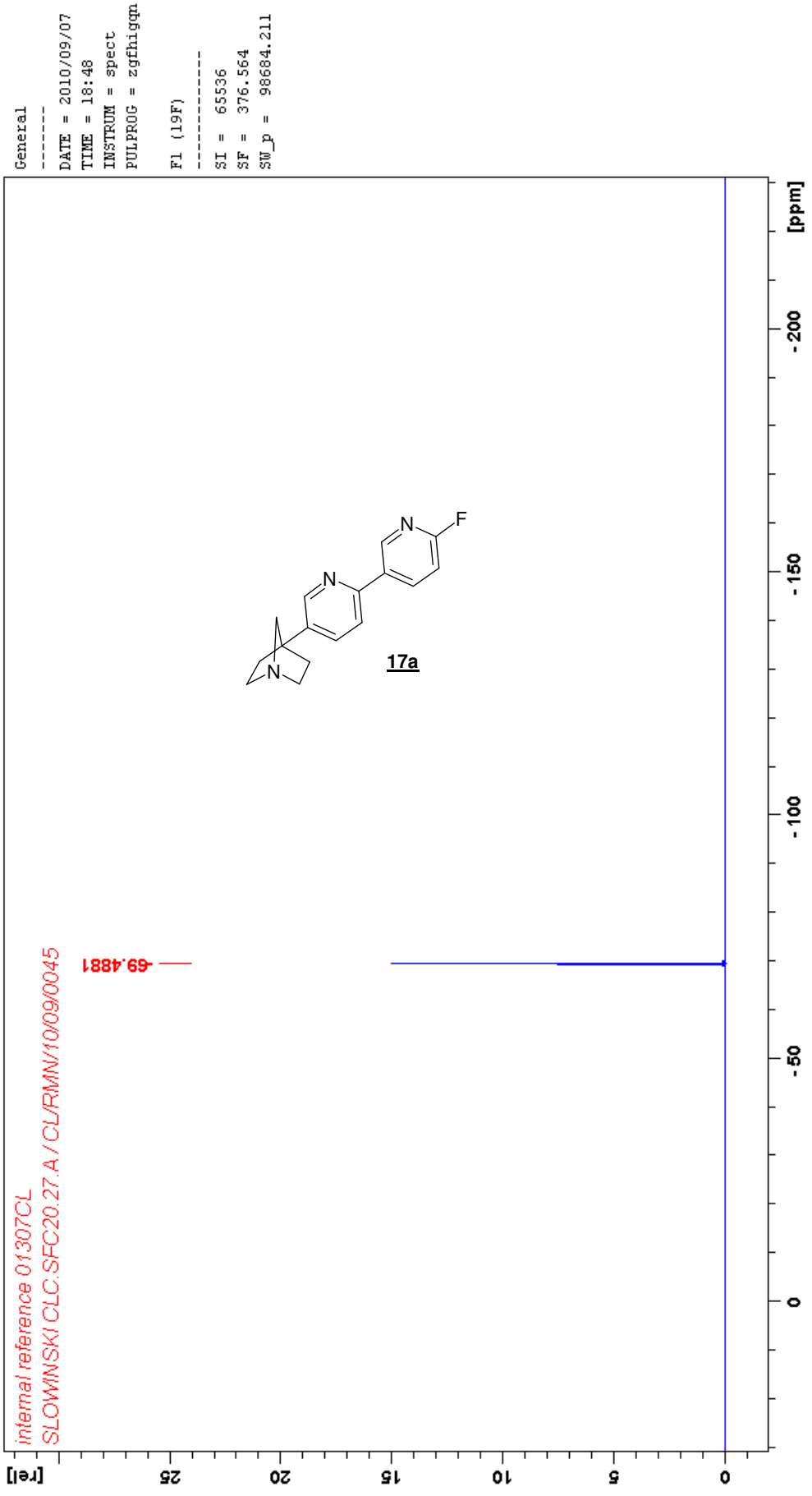


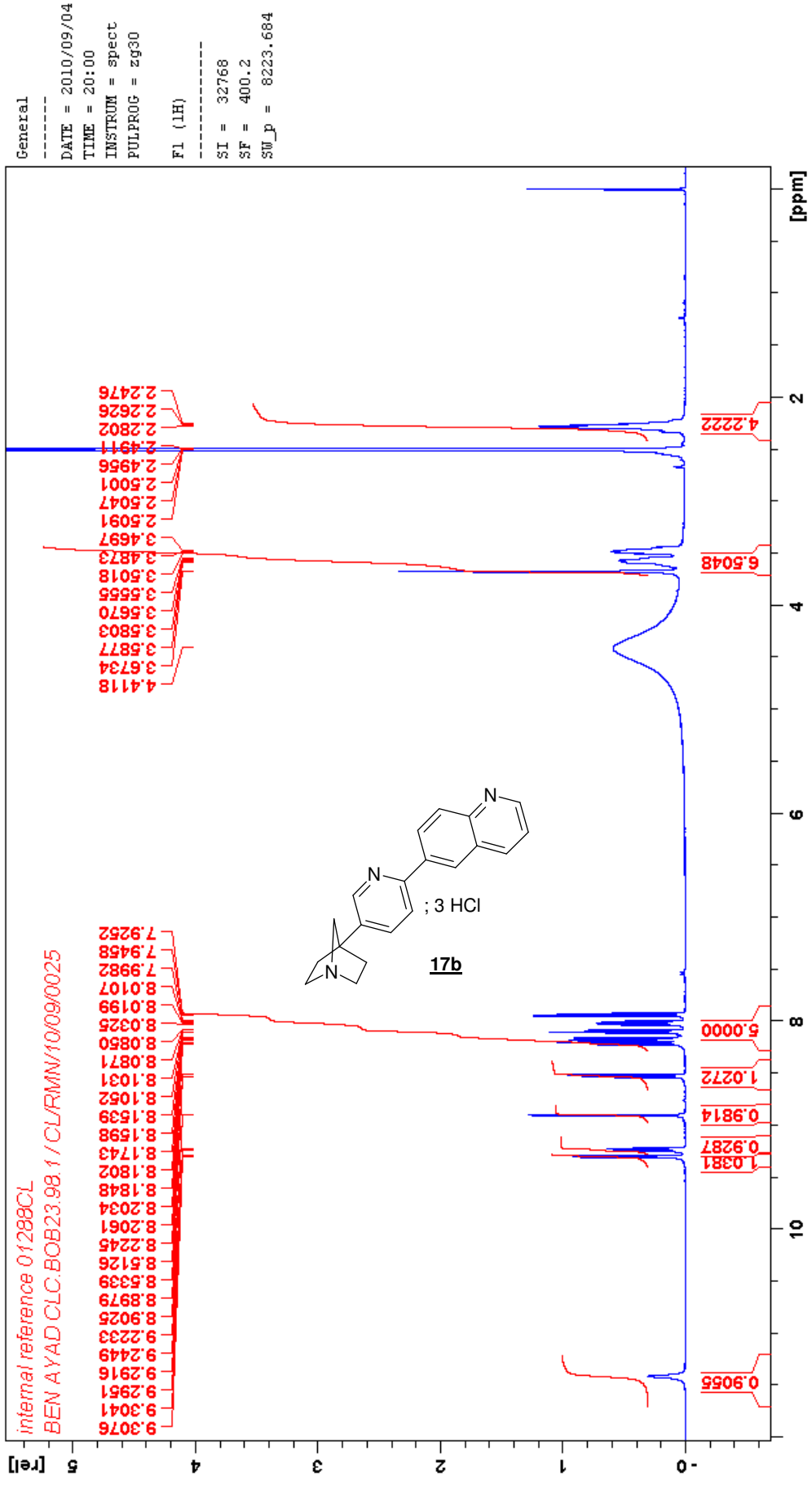
2
crude material

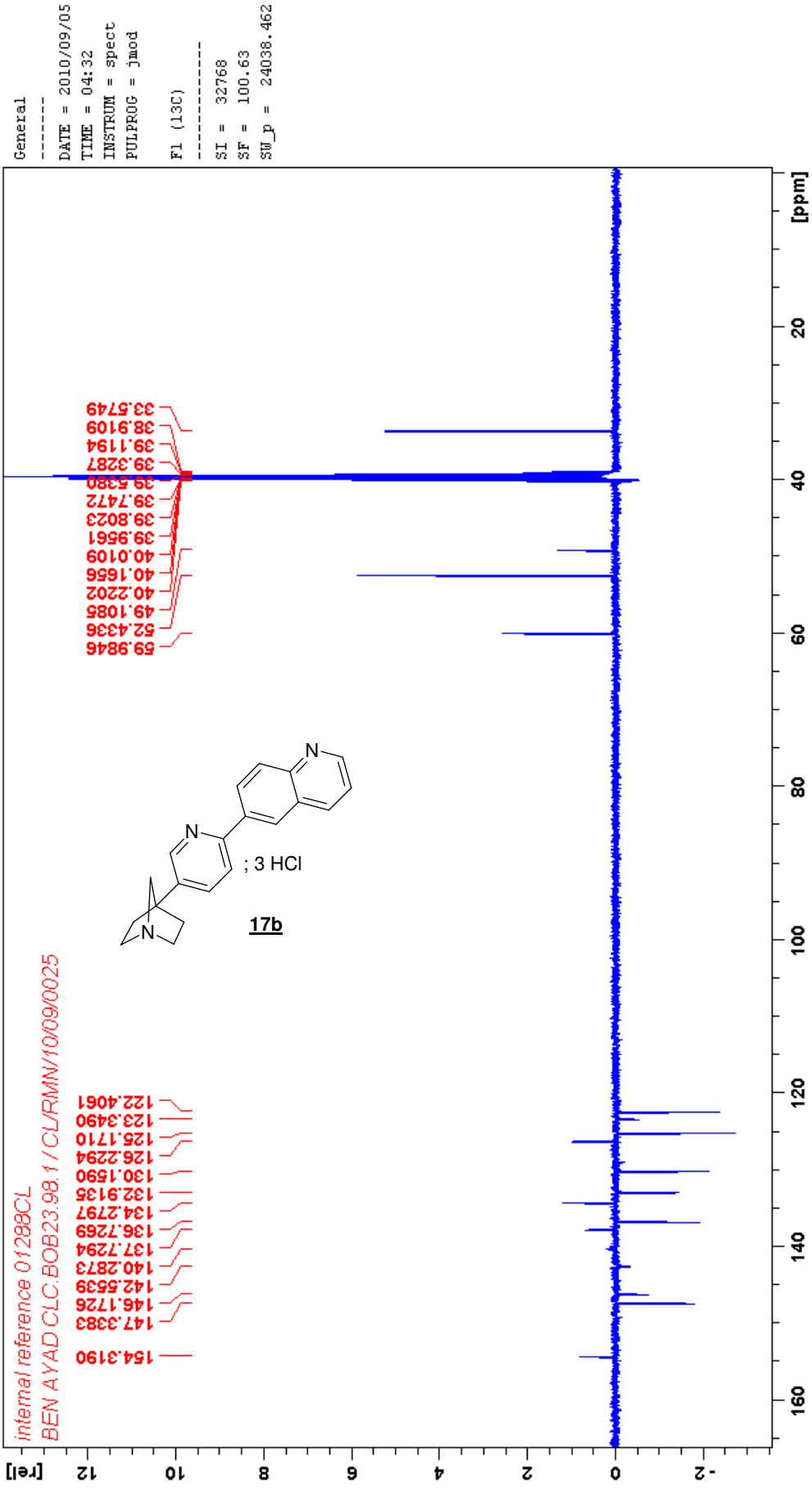

General
DATE = 2010/07/24
TIME = 08:35
INSTRUM = spect
PULPROG = zgpg30
F1 (13C)
SI = 32768
SF = 100.63
SW_p = 24038.462
23.3241
30.8542
35.9929
38.9265
39.1351
39.3438
39.5524
39.7613
39.9696
40.1785
51.1714
57.8493
123.6914
136.6038
145.3882
146.9670
147.5624
Cl
N
N
2
crude material
internal reference 00863CL
Slowinski CLC.SFC20.20.A / CL/RMN/10/07/01710
[rel]
[ppm]

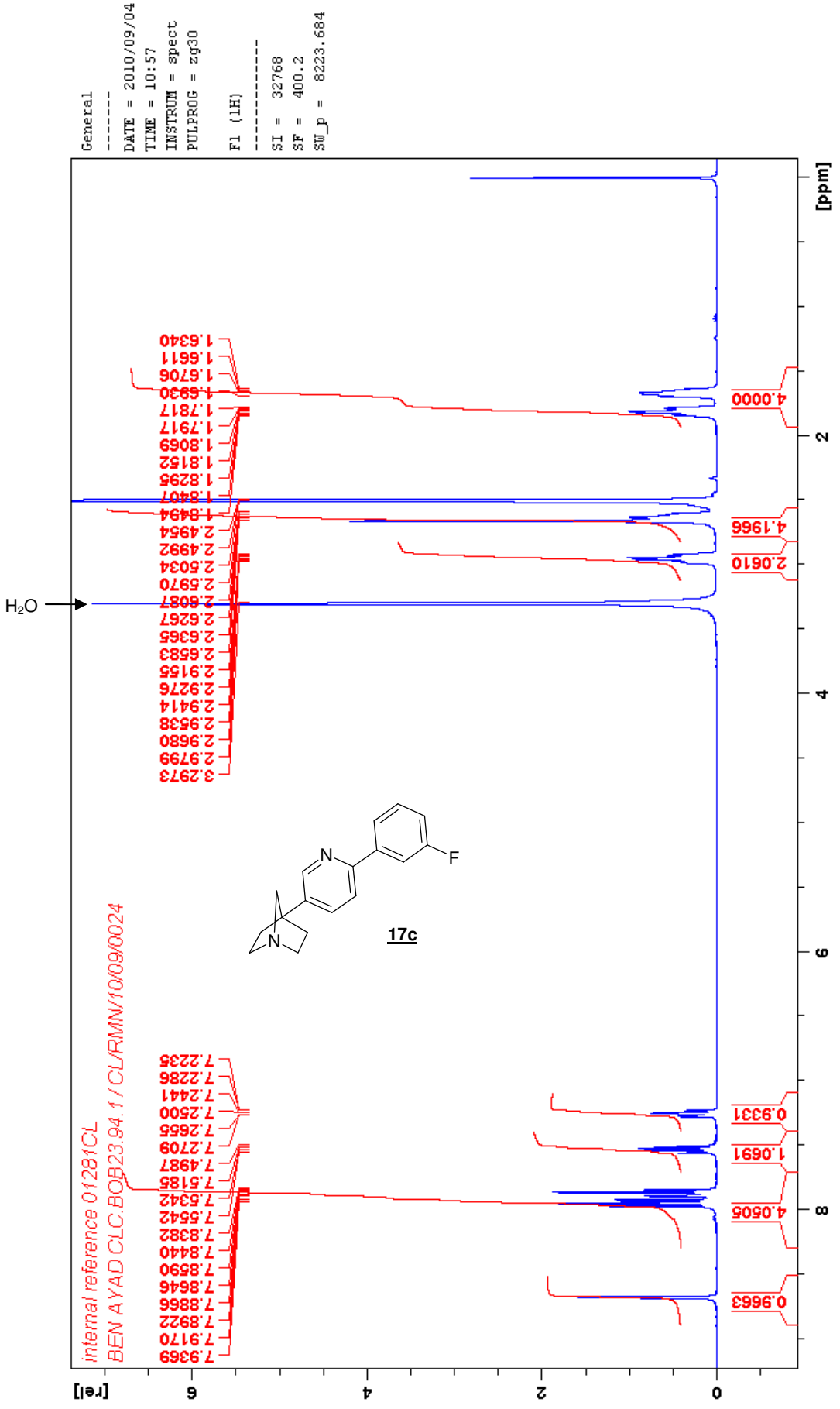

General
DATE = 2010/09/07
TIME = 18:42
INSTRUM = spect
PULPROG = zg30
F1 (1H)
SI = 32768
SF = 400.2
SW_p = 8223.684
internal reference 01307CL
SLOWINSKI CLC SFC20.27.A / CL/RMN/10/09/0045
H2O
17a
[rel]
[ppm]

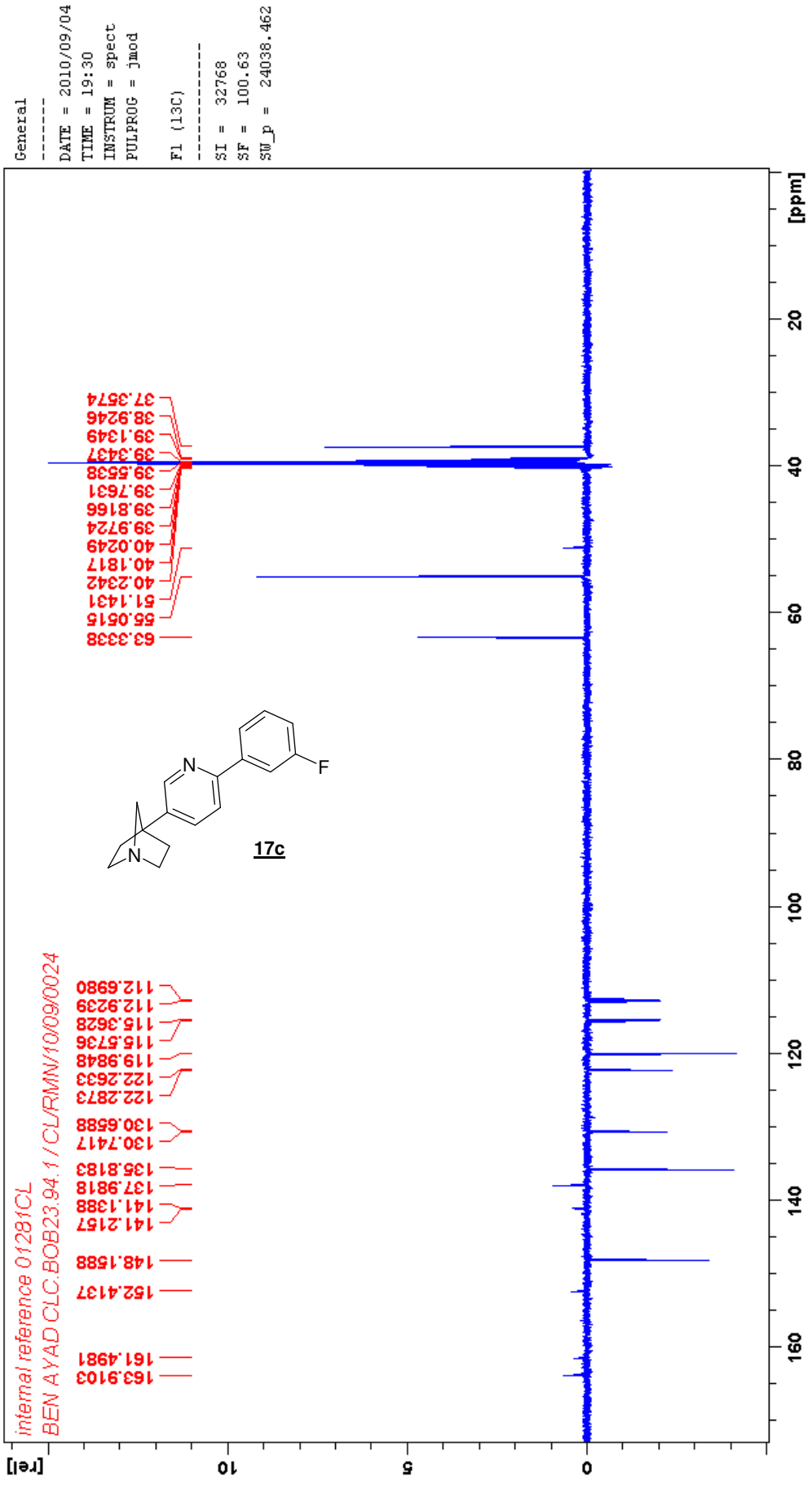

General
DATE = 2010/09/07
TIME = 22:46
INSTRUM = spect
PULPROG = jmod
F1 (13C)
SI = 32768
SF = 100.63
SW_p = 24038.462
internal reference 01307CL
SLOWINSKI CLC.SFC20.27.A / CL/RMN/10/09/0045
164.4076
162.0508
150.5686
148.3549
145.7169
145.5621
139.9813
139.8989
138.0854
135.9375
132.8138
132.7698
120.0162
109.7542
109.3783
63.3320
55.0542
51.1564
40.1759
39.9643
39.7546
39.5458
39.3358
39.1270
38.9180
37.3646
17a
[rel]
20
10
0
-10
[ppm]
50
100
150

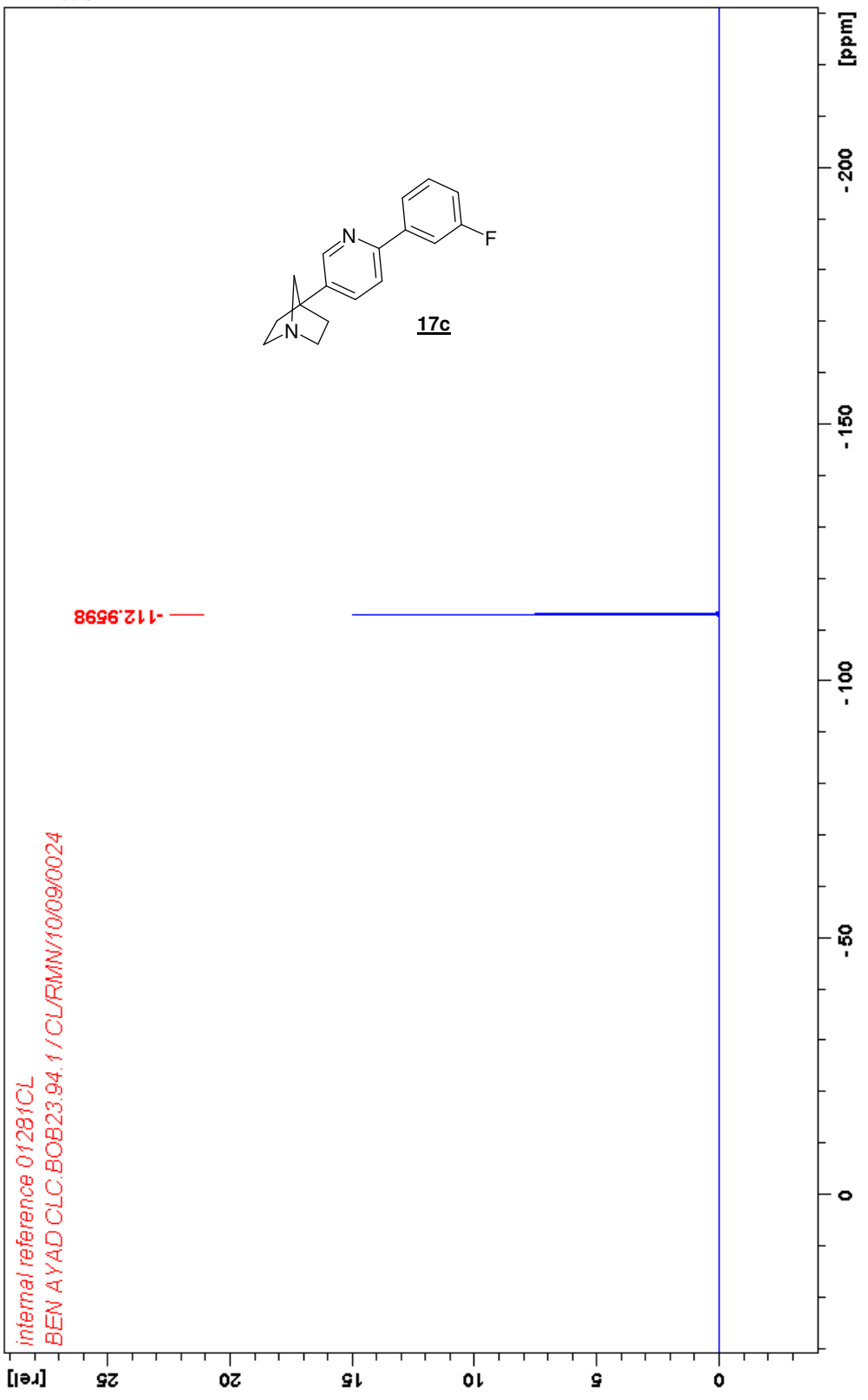

General
DATE = 2010/09/07
TIME = 18:48
INSTRUM = spect
PULPROG = zgfhigqn
F1 (19F)
SI = 65536
SF = 376.564
SW_p = 98684.211
-69.4881
internal reference 01307CL
SLOWINSKI CLC.SFC20.27.A / CL/RMN/10/09/0045
[rel]
[ppm]
17a

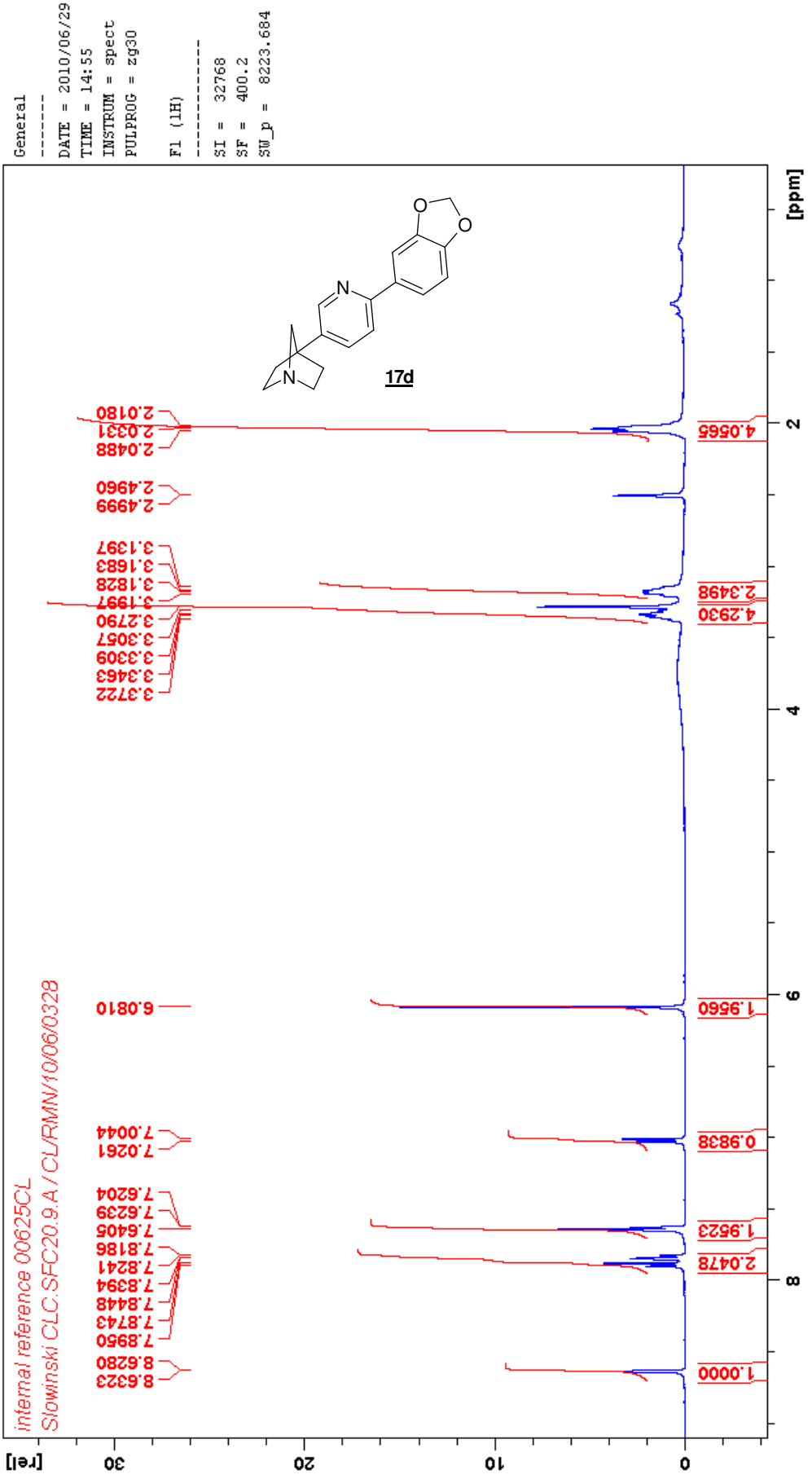

General
DATE = 2010/09/04
TIME = 20:00
INSTRUM = spect
PULPROG = zg30
F1 (1H)
SI = 32768
SF = 400.2
SW_p = 8223.684
internal reference 01288CL
BEN AYAD CLC.BOB23.98.1 / CL/RMN/10/09/0025
; 3 HCl
17b
[rel]
[ppm]

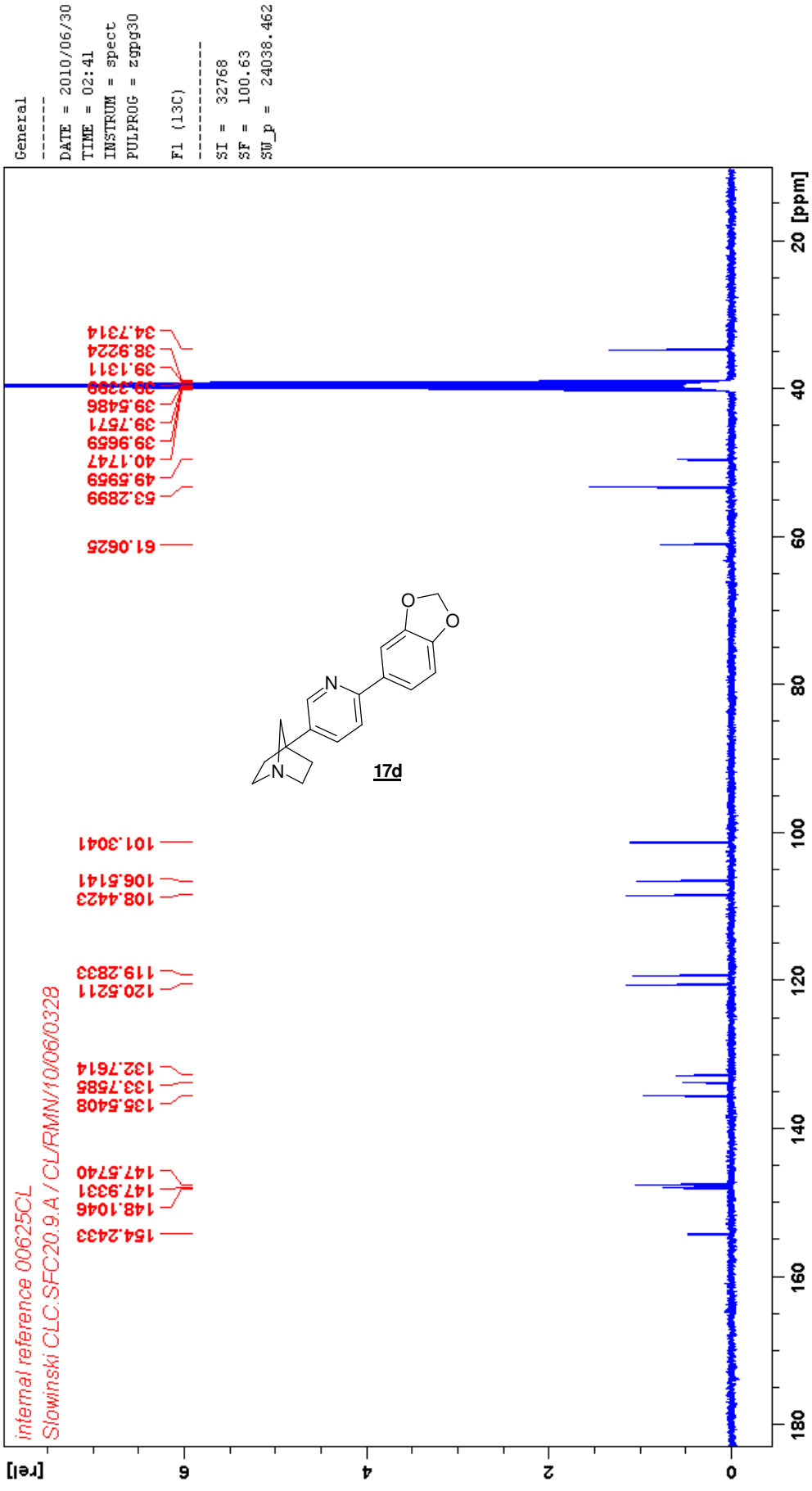

General
DATE = 2010/09/05
TIME = 04:32
INSTRUM = spect
PULPROG = jmod
F1 (13C)
SI = 32768
SF = 100.63
SW_p = 24038.462
59.9846
52.4336
49.1085
40.2202
40.1656
40.0109
39.9561
39.8023
39.7472
39.5380
39.3287
39.1194
38.9109
33.5749
154.3190
147.3383
146.1726
142.5539
140.2873
137.7294
136.7269
134.2797
132.9135
130.1590
126.2294
125.1710
123.3490
122.4061
; 3 HCl
17b
internal reference 01288CL
BEN AYAD CLC.BOB23.98.1 / CL/RMN/10/09/0025
[rel]
[ppm]

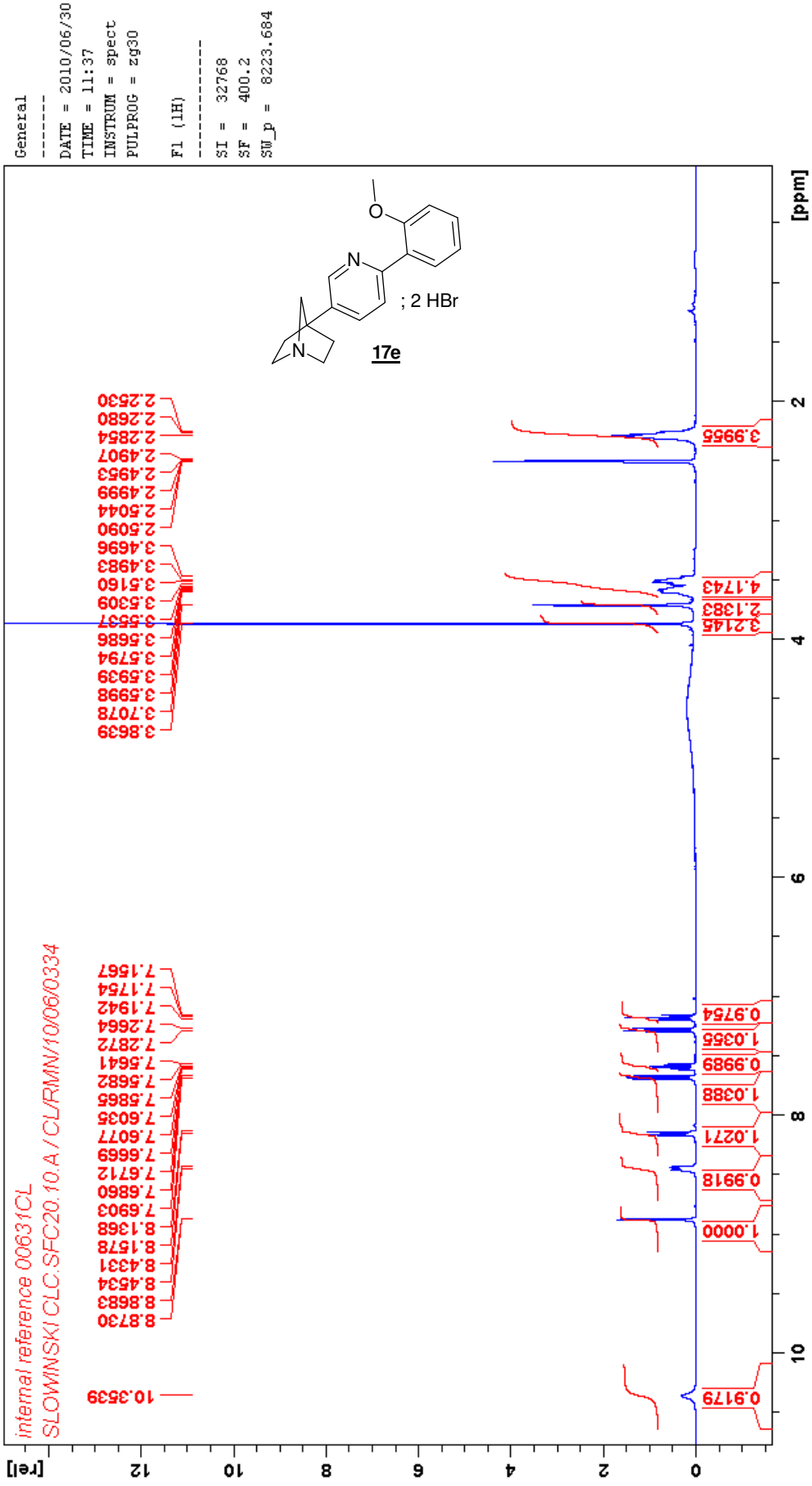

General
DATE = 2010/09/04
TIME = 10:57
INSTRUM = spect
PULPROG = zg30
F1 (1H)
SI = 32768
SF = 400.2
SW_p = 8223.684
internal reference 01281CL
BEN AYAD CLC.BOB23.94.1 / CL/RMN/10/09/0024
H2O
N
F
17c
[rel]
[ppm]
0.9663
4.0505
1.0691
0.9331
2.0610
4.1966
4.0000

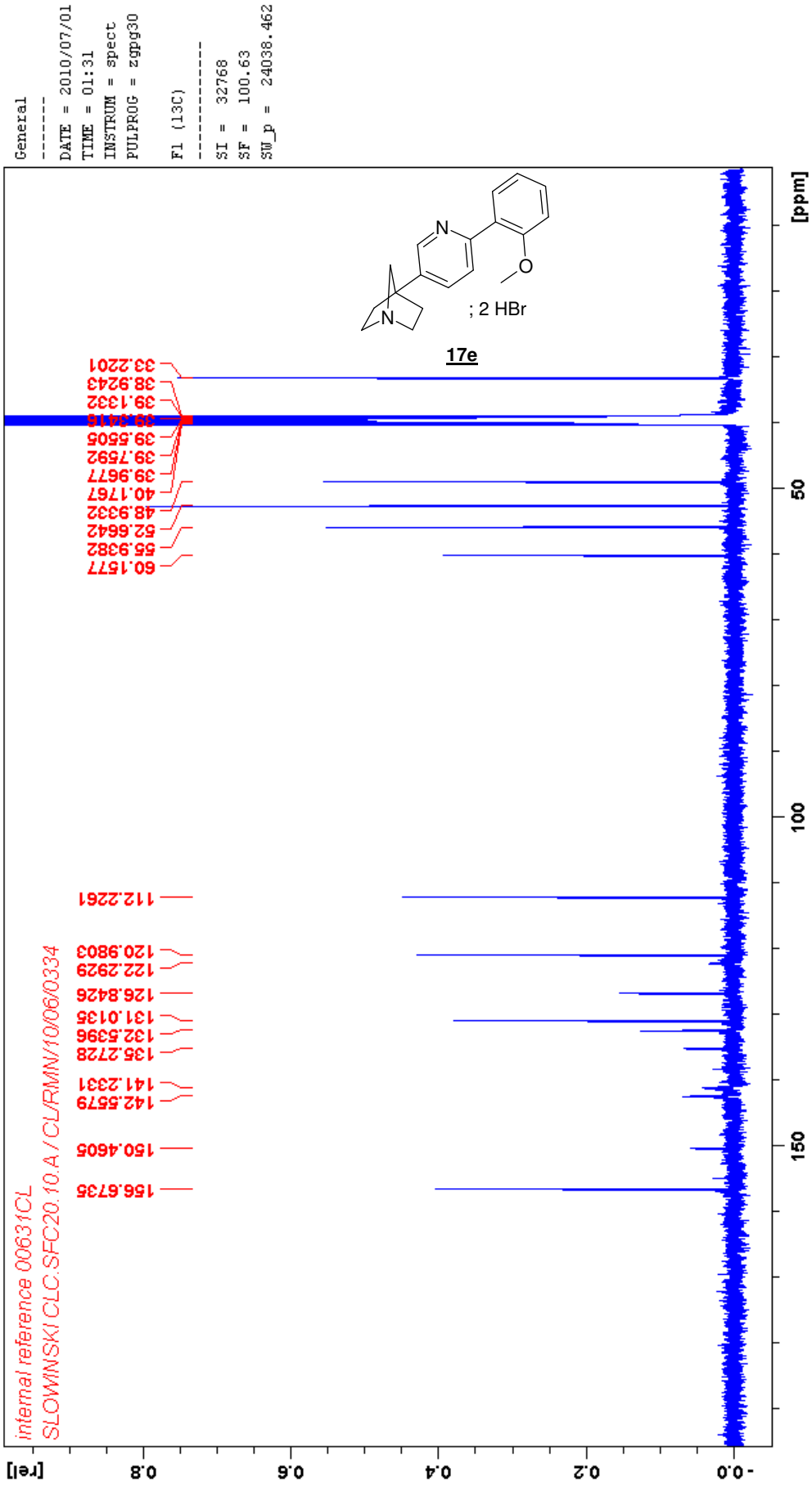

General
DATE = 2010/09/04
TIME = 19:30
INSTRUM = spect
PULPROG = jmod
F1 (13C)
SI = 32768
SF = 100.63
SW_p = 24038.462
internal reference 01281CL
BEN AYAD CLC.BOB23.94.1 / CL/RMN/10/09/0024
63.3338
55.0515
51.1431
40.2342
40.1817
40.0249
39.9724
39.8166
39.7631
39.5538
39.3437
39.1349
38.9246
37.3574
163.9103
161.4981
152.4137
148.1588
141.2157
141.1388
137.9818
135.8183
130.7417
130.6588
122.2873
122.2633
119.9848
115.5736
115.3628
112.9239
112.6980
N
F
N
17c
[rel]
[ppm]

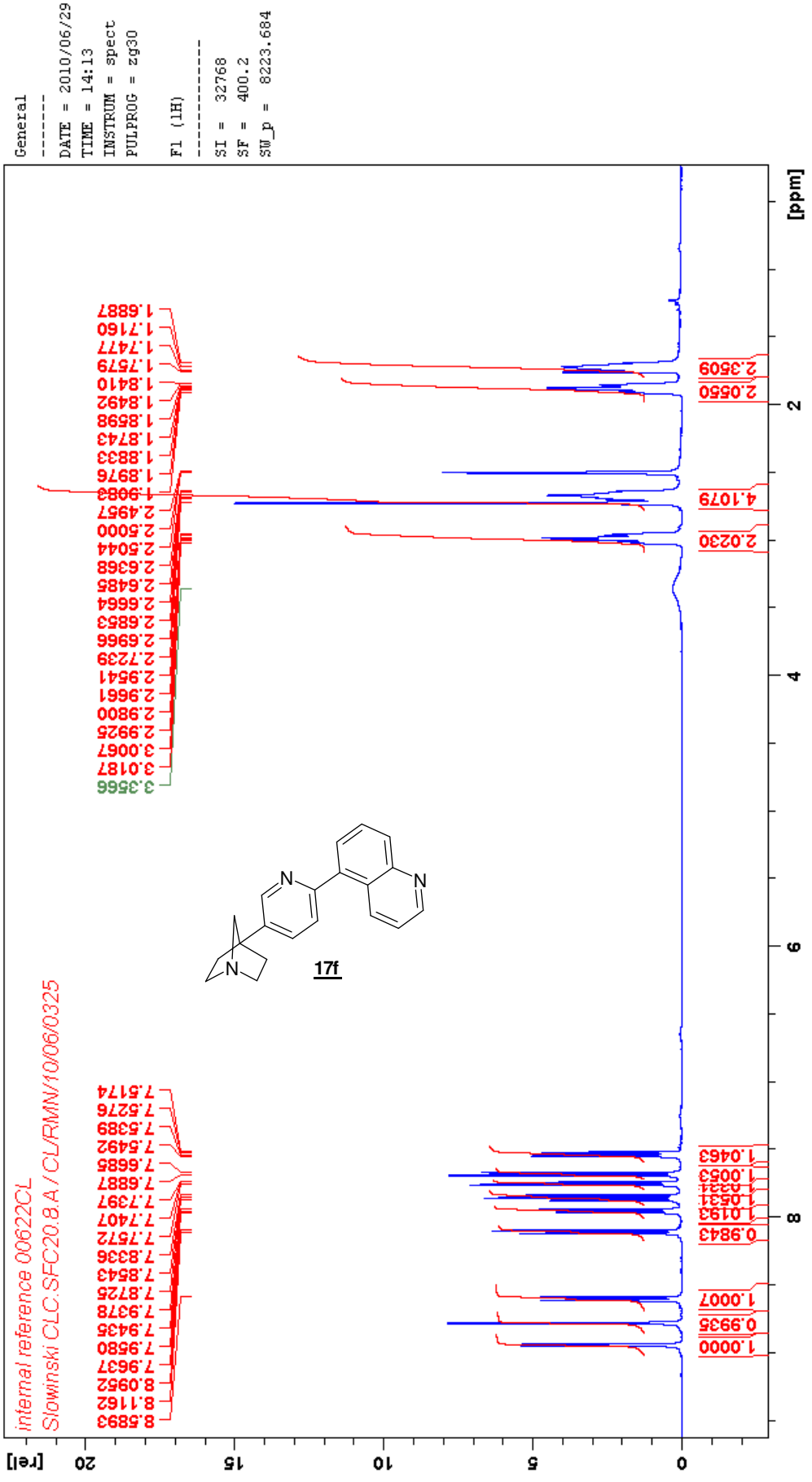

General
DATE = 2010/09/04
TIME = 19:46
INSTRUM = spect
PULPROG = zgfhigqn
F1 (19F)
SI = 65536
SF = 376.564
SW_p = 98684.211
-112.9598
17c
internal reference 01281CL
BEN AYAD CLC.BOB23.94.1 / CL/RMN/10/09/0024
[rel]
[ppm]

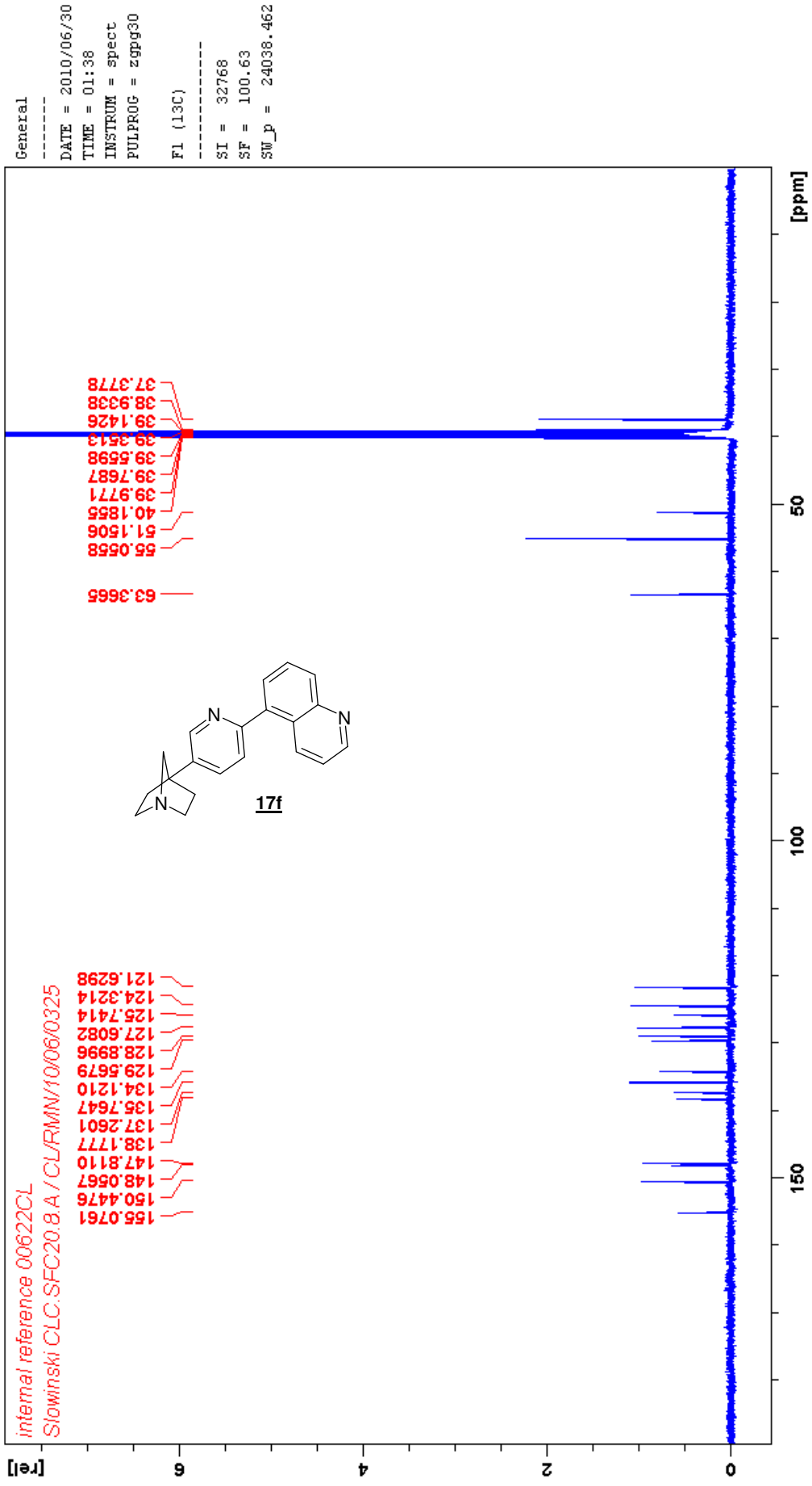

General
DATE = 2010/06/29
TIME = 14:55
INSTRUM = spect
PULPROG = zg30
F1 (1H)
SI = 32768
SF = 400.2
SW_p = 8223.684
17d
internal reference 00625CL
Slowinski CLC.SFC20.9.A / CL/RMN/10/06/0328
[rel]
[ppm]
8.6323
8.6280
7.8950
7.8743
7.8448
7.8394
7.8241
7.8186
7.6405
7.6239
7.6204
7.0261
7.0044
6.0810
3.3722
3.3463
3.3309
3.3057
3.2790
3.1997
3.1828
3.1683
3.1397
2.4999
2.4960
2.0488
2.0331
2.0180
1.0000
2.0478
1.9523
1.9560
0.9838
2.3498
4.2930
4.0565

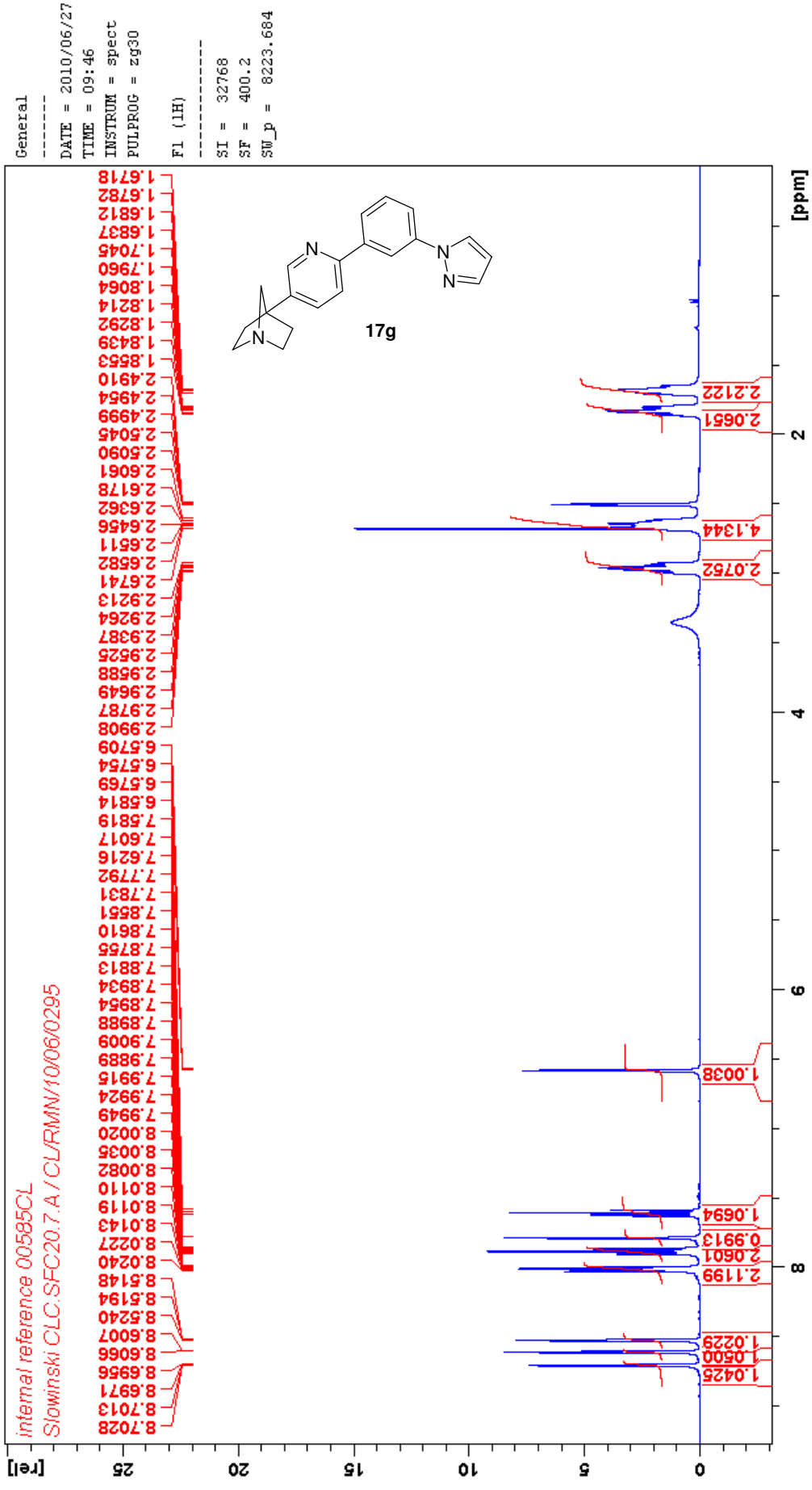

General
DATE = 2010/06/30
TIME = 02:41
INSTRUM = spect
PULPROG = zgpg30
F1 (13C)
SI = 32768
SF = 100.63
SW_p = 24038.462
34.7314
38.9224
39.1311
39.3389
39.5486
39.7571
39.9659
40.1747
49.5959
53.2899
61.0625
101.3041
106.5141
108.4423
119.2833
120.5211
132.7614
133.7585
135.5408
147.5740
147.9331
148.1046
154.2433
O
O
N
N
17d
internal reference 00625CL
Slowinski CLC.SFC20.9.A / CL/RMN/10/06/0328
[rel]
[ppm]

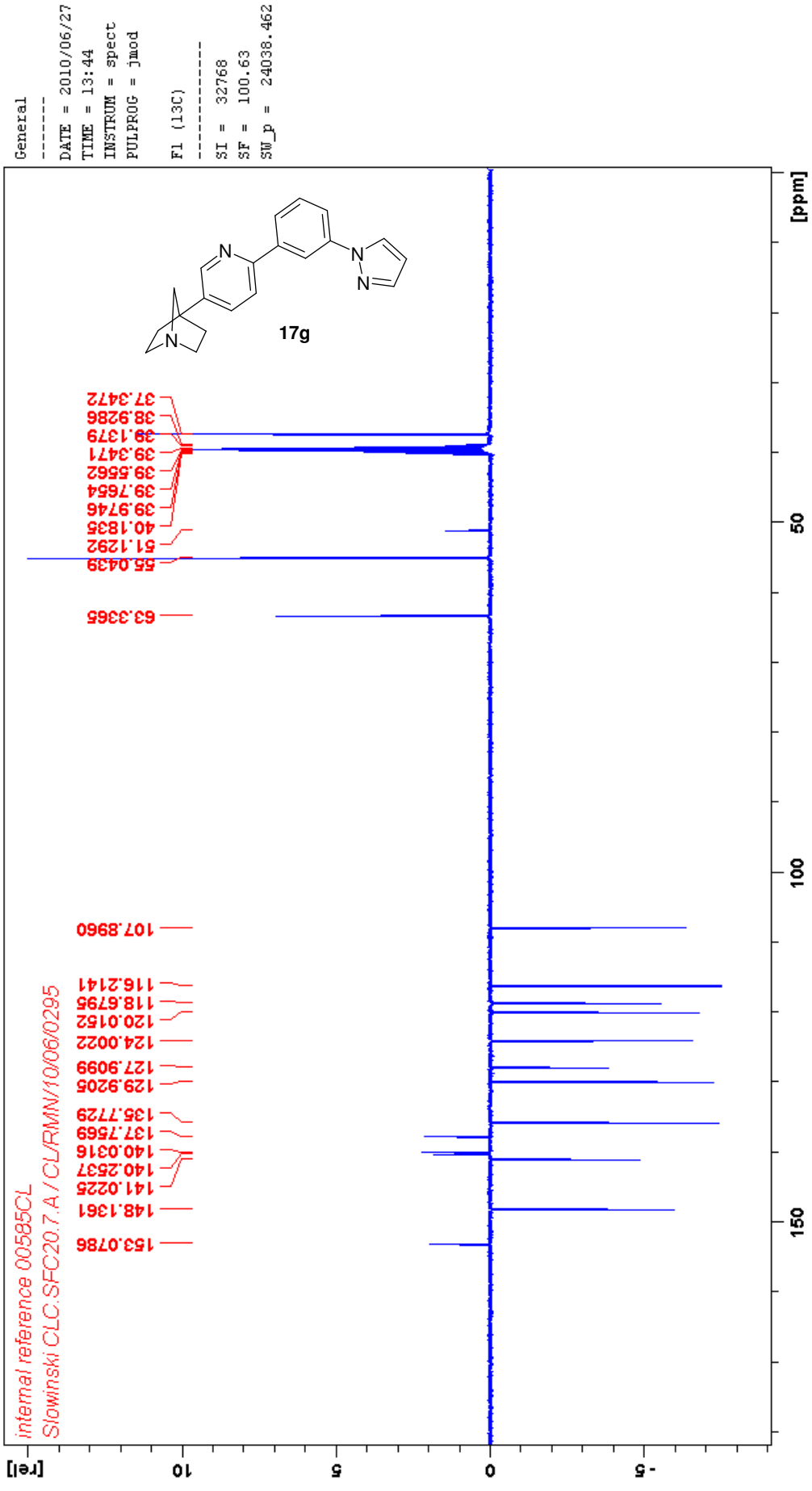

General
DATE = 2010/06/30
TIME = 11:37
INSTRUM = spect
PULPROG = zg30
F1 (1H)
SI = 32768
SF = 400.2
SW_p = 8223.684
; 2 HBr
17e
internal reference 00631CL
SLOWINSKI CLC.SFC20.10.A / CL/RMN/10/06/0334
[rel]
[ppm]
10.3539
8.8730
8.8683
8.4534
8.4331
8.1578
8.1368
7.6903
7.6860
7.6712
7.6669
7.6077
7.6035
7.5865
7.5682
7.5641
7.2872
7.2664
7.1942
7.1754
7.1567
3.8639
3.7078
3.5998
3.5939
3.5794
3.5686
3.5537
3.5309
3.5160
3.4983
3.4696
2.5090
2.5044
2.4999
2.4953
2.4907
2.2854
2.2680
2.2530
0.9179
1.0000
0.9918
1.0271
1.0388
0.9989
1.0355
0.9754
3.9955
4.1743
2.1383
3.2145

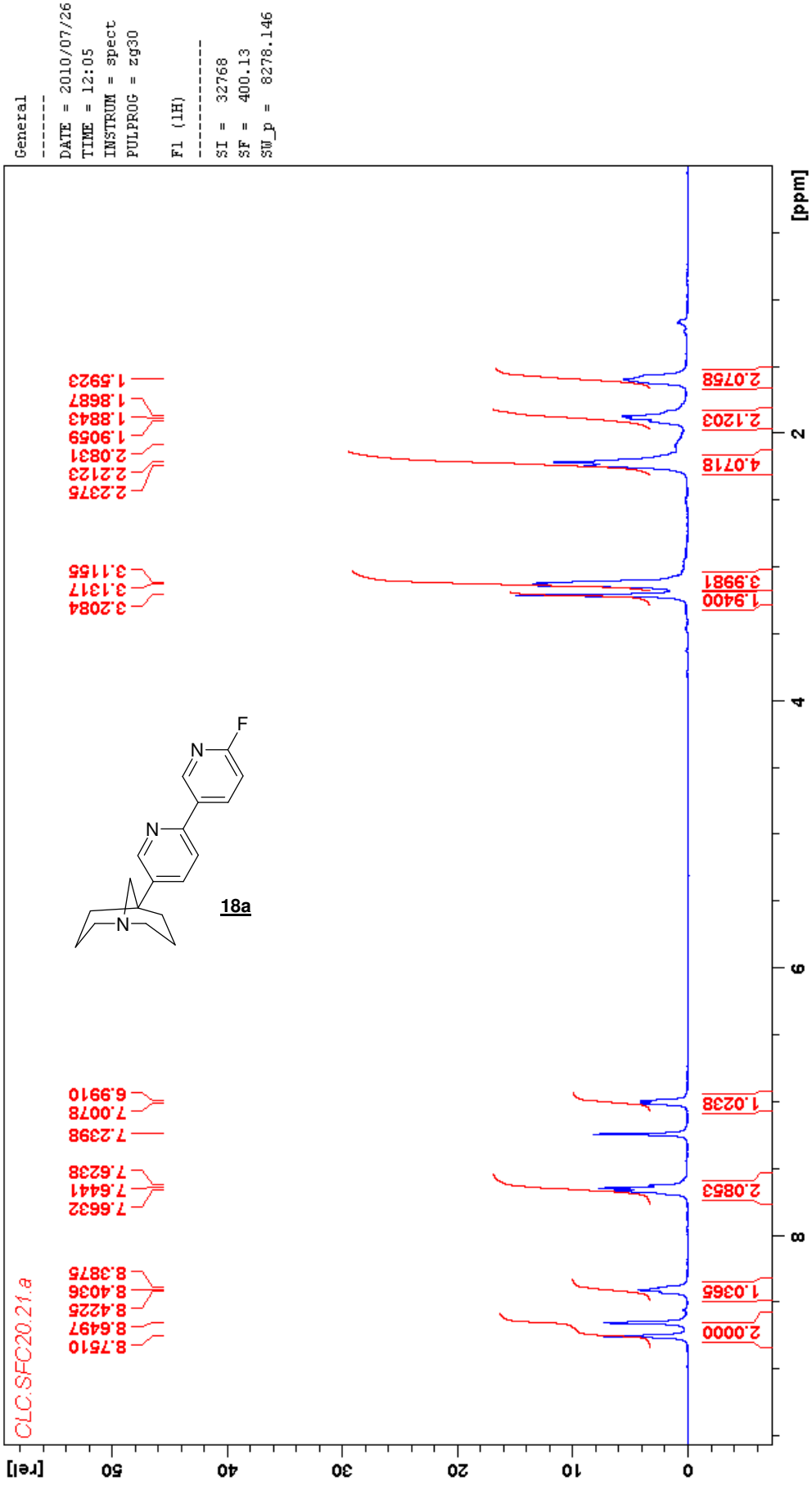

General
DATE = 2010/07/01
TIME = 01:31
INSTRUM = spect
PULPROG = zgpg30
F1 (13C)
SI = 32768
SF = 100.63
SW_p = 24038.462
; 2 HBr
17e
33.2201
38.9243
39.1332
39.3416
39.5505
39.7592
39.9677
40.1767
48.9332
52.6642
55.9382
60.1577
112.2261
120.9803
122.2929
126.8426
131.0135
132.5396
135.2728
141.2331
142.5579
150.4605
156.6735
internal reference 00631CL
SLOWINSKI CLC.SFC20.10.A / CL/RMN/10/06/0334
[rel]
[ppm]

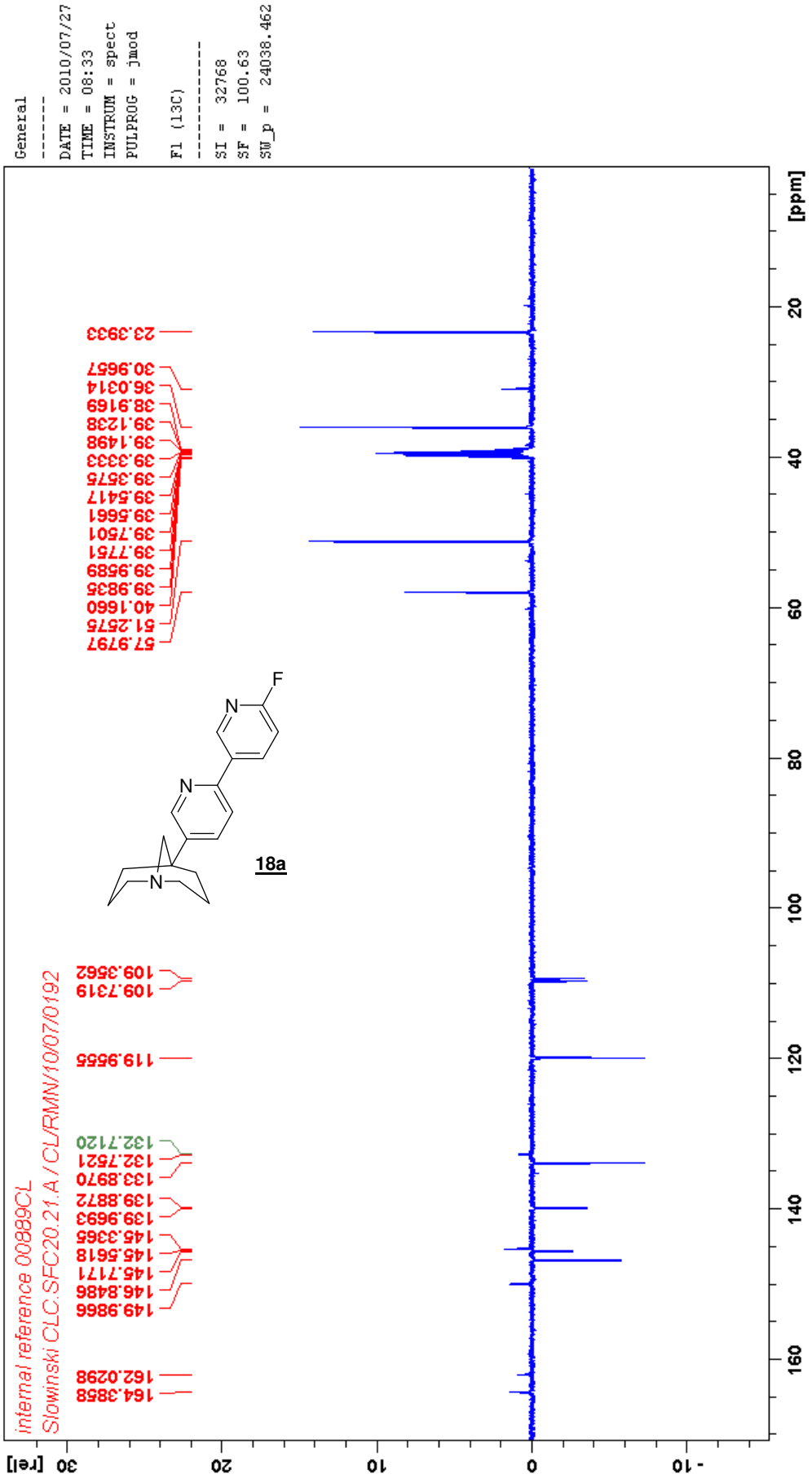

General
DATE = 2010/06/29
TIME = 14:13
INSTRUM = spect
PULPROG = zg30
F1 (1H)
SI = 32768
SF = 400.2
SW_p = 8223.684
internal reference 00622CL
Slowinski CLC.SFC20.8.A / CL/RMN/10/06/0325
[rel]
[ppm]
17f

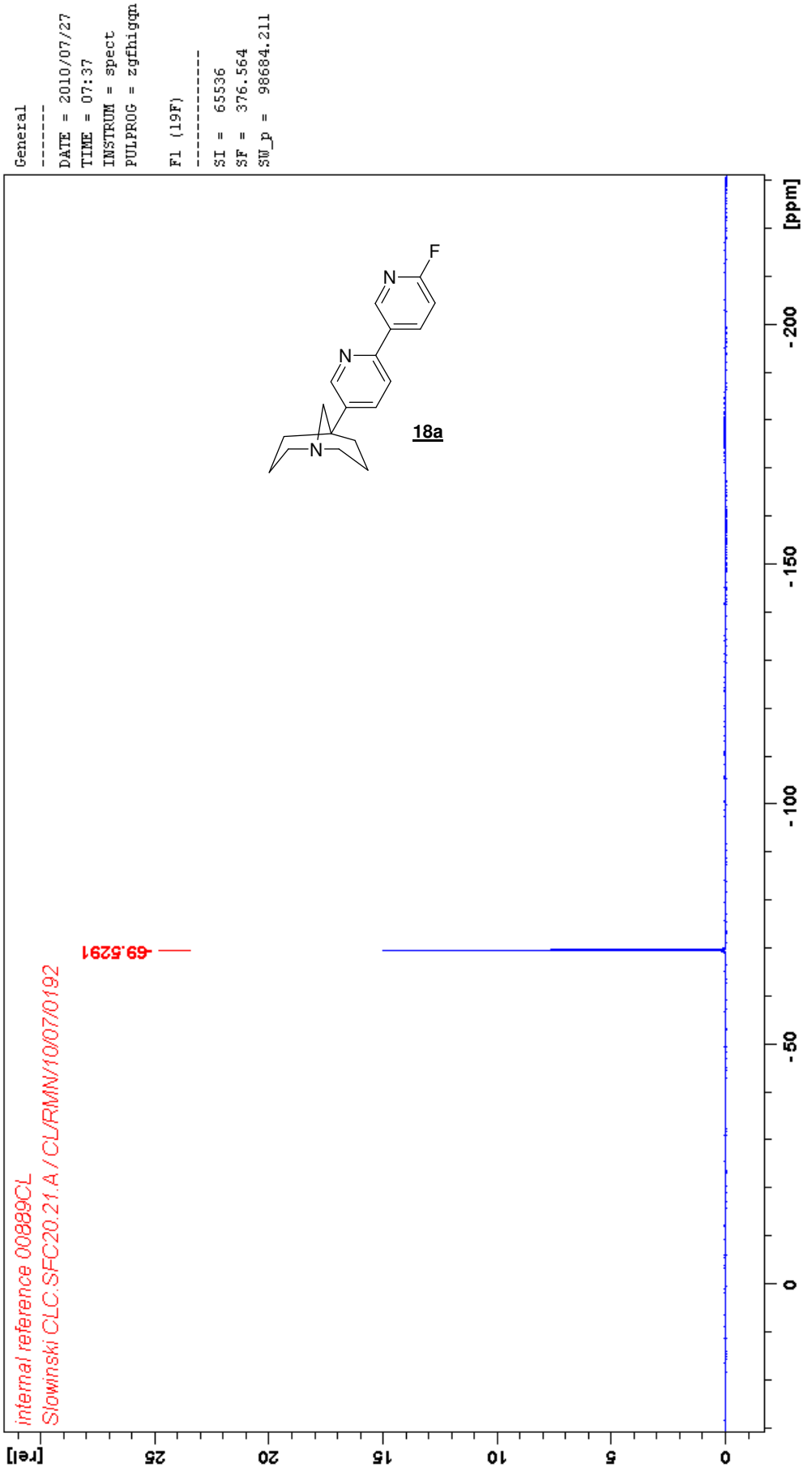

General
DATE = 2010/06/30
TIME = 01:38
INSTRUM = spect
PULPROG = zgpg30
F1 (13C)
SI = 32768
SF = 100.63
SW_p = 24038.462
37.3778
38.9338
39.1426
39.3513
39.5598
39.7687
39.9771
40.1855
51.1506
55.0558
63.3665
155.0761
150.4476
148.0567
147.8110
138.1777
137.2601
135.7647
134.1210
129.5679
128.8996
127.6082
125.7414
124.3214
121.6298
Internal reference 00622CL
Slowinski CLC.SFC20.8.A / CL/RMN/10/06/0325
[rel]
[ppm]
17f

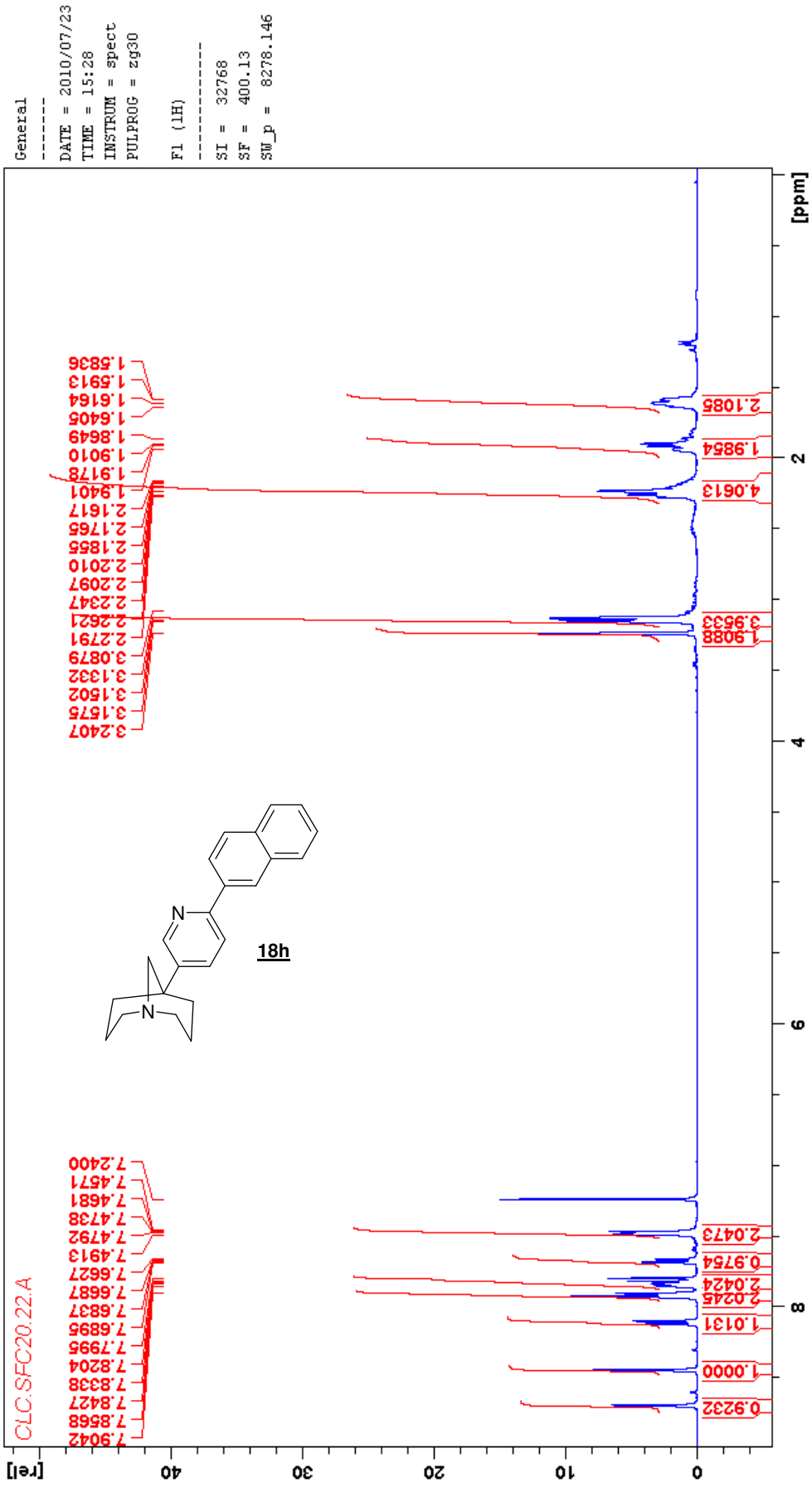

General
DATE = 2010/06/27
TIME = 09:46
INSTRUM = spect
PULPROG = zg30
F1 (1H)
SI = 32768
SF = 400.2
SW_p = 8223.684
internal reference 00585CL
Slowinski CLC.SFC20.7.A / CL/RMN/10/06/0295
17g
[rel]
[ppm]
1.0425
1.0500
1.0229
2.1199
2.0601
0.9913
1.0694
1.0038
2.0752
4.1344
2.0651
2.2122

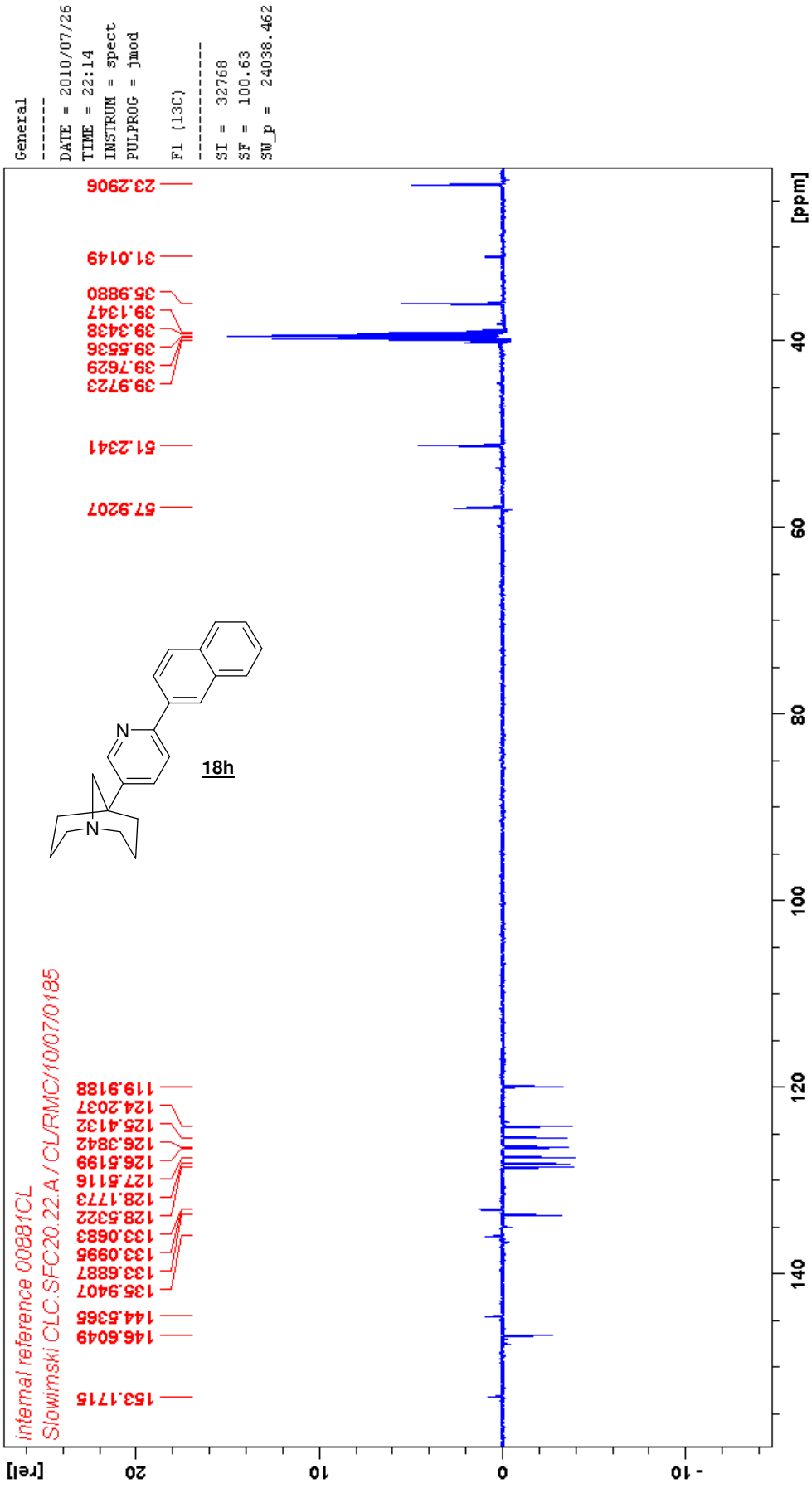

General
DATE = 2010/06/27
TIME = 13:44
INSTRUM = spect
PULPROG = jmod
F1 (13C)
SI = 32768
SF = 100.63
SW_p = 24038.462
17g
37.3472
38.9286
39.1379
39.3471
39.5562
39.7654
39.9746
40.1835
51.1292
55.0439
63.3365
107.8960
116.2141
118.6795
120.0152
124.0022
127.9099
129.9205
135.7729
137.7669
140.0316
140.2537
141.0225
148.1361
153.0786
internal reference 00585CL
Slowinski CLC.SFC20.7.A / CL/RMN/10/06/0295
[rel]
[ppm]


General
DATE = 2010/07/26
TIME = 12:05
INSTRUM = spect
PULPROG = zg30
F1 (1H)
SI = 32768
SF = 400.13
SW_p = 8278.146
CLC.SFC20.21.a
8.7510
8.6497
8.4225
8.4036
8.3875
7.6632
7.6441
7.6238
7.2398
7.0078
6.9910
3.2084
3.1317
3.1155
2.2375
2.2123
2.0831
1.9059
1.8843
1.8687
1.5923
2.0000
1.0365
2.0853
1.0238
1.9400
3.9981
4.0718
2.1203
2.0758
[rel]
[ppm]
18a


General
DATE = 2010/07/27
TIME = 08:33
INSTRUM = spect
PULPROG = jmod
F1 (13C)
SI = 32768
SF = 100.63
SW_p = 24038.462
internal reference 00889CL
Slowinski CLC.SFC20.21.A / CL/RMN/10/07/0192
23.3933
30.9657
36.0314
38.9169
39.1238
39.1498
39.3333
39.3575
39.5417
39.5661
39.7501
39.7751
39.9589
39.9835
40.1660
51.2575
57.9797
109.3562
109.7319
119.9555
132.7120
132.7521
133.8970
139.8872
139.9693
145.3365
145.5618
145.7171
146.8486
149.9866
162.0298
164.3858
F
N
N
N
18a
[rel]
30
20
10
0
-10
[ppm]
160
140
120
100
80
60
40
20


General
DATE = 2010/07/27
TIME = 07:37
INSTRUM = spect
PULPROG = zgfhigqn
F1 (19F)
SI = 65536
SF = 376.564
SW_p = 98684.211
-69.5291
internal reference 00889CL
Slowinski CLC.SFC20.21.A / CL/RMN/10/07/0192
[rel]
[ppm]
18a


General
DATE = 2010/07/23
TIME = 15:28
INSTRUM = spect
PULPROG = zg30
F1 (1H)
SI = 32768
SF = 400.13
SW_p = 8278.146
CLC SFC20 22 A
[rel]
[ppm]
18h
N
N
7.9042
7.8568
7.8427
7.8338
7.8204
7.7995
7.6895
7.6837
7.6687
7.6627
7.4913
7.4792
7.4738
7.4681
7.4571
7.2400
0.9232
1.0000
1.0131
2.0245
2.0424
0.9754
2.0473
2.1085
1.9854
4.0613
1.9401
1.9178
1.9010
1.8649
1.8405
1.6164
1.5913
1.5836
3.2407
3.1575
3.1502
3.1332
3.0879
2.2791
2.2621
2.2347
2.2097
2.2010
2.1855
2.1765
2.1617
3.9533
1.9088


General
DATE = 2010/07/26
TIME = 22:14
INSTRUM = spect
PULPROG = jmod
F1 (13C)
SI = 32768
SF = 100.63
SW_p = 24038.462
23.2906
31.0149
35.9880
39.1347
39.3438
39.5536
39.7629
39.9723
51.2341
57.9207
119.9188
124.2037
125.4132
126.3842
126.5199
127.5116
128.1773
128.5322
133.0683
133.0995
133.6887
135.9407
144.5365
146.6049
153.1715
18h
N
N
internal reference 00881CL
Slowinski CLC.SFC20.22.A / CL/RMC/10/07/0185
[rel]
20
10
0
-10
[ppm]
140
120
100
80
60
40