

CuFe₂O₄ nanoparticles as a highly efficient and magnetically recoverable catalyst for the synthesis of medicinally privileged spiropyrimidine scaffolds

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Supplementary data

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Experimental Procedures

1 General

All the chemicals used were of research grade and were used without further purification. The melting points of all compounds were determined on a Toshniwal apparatus. The purity of compounds was checked on thin layers of silica Gel-G coated glass plates and n-hexane: ethyl acetate (8:2) as eluent. IR spectra were recorded on a Shimadzu FT IR-8400S spectrophotometer using KBr pellets. ^1H and ^{13}C NMR spectra were recorded in CDCl_3 using TMS as an internal standard on a Bruker spectrophotometer at 300 and 75 MHz respectively. Mass spectrums of representative compounds were recorded on JEOL-SX-102 Mass spectrometer at 70 eV. Elemental microanalyses were carried out on a Carlo-Erba 1108 CHN analyzer. Single crystal X-ray diffraction was performed on a Bruker Kappa Apex II instrument.

2 Preparation CuFe_2O_4 nanoparticles

CuFe_2O_4 nanoparticles were prepared by thermal decomposition of $\text{Cu}(\text{NO}_3)_2$ and $\text{Fe}(\text{NO}_3)_3$ in water in the presence of sodium hydroxide. Briefly, to a solution of $\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$ (3.34 g, 8.2 mmol) and $\text{Cu}(\text{NO}_3)_2 \cdot 3\text{H}_2\text{O}$ (1 g, 4.1 mmol) in 75 ml of distilled water, 3 g (75 mmol) of NaOH dissolved in 15 ml of water was added at room temperature over a period of 10 min. during which reddish-black precipitate was formed. Then the reaction mixture was warmed to 90°C and stirred under ultrasonic irradiation for two hours. After 2 h, it was cooled to room temperature and the magnetic particles so formed were separated by a magnetic separator. It was then washed with water (3 X 30 ml) and catalyst was kept in air oven for overnight at 80°C . Then the catalyst was ground in a mortar-pestle and kept in a furnace at 700°C for 5 h (step up temperature $20^\circ\text{C}/\text{min}$) and then cooled to room temperature. 930 mg of magnetic CuFe_2O_4 particles of size 35-50 nm were obtained.

3 Catalyst characterizations

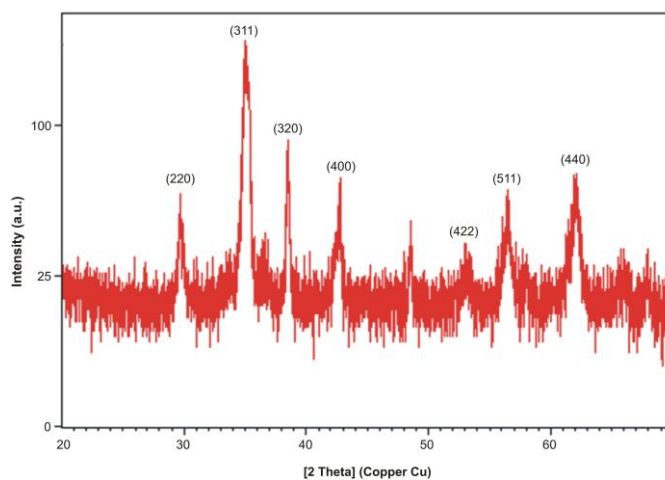
The wide angle X-ray diffraction pattern of the sample was obtained using Bragg-Brentano geometry on PANalytical X'pert pro diffractometer in 2Θ range of $20\text{-}70^\circ$ with Cu-K α radiation source ($\lambda = 1.5406 \text{ \AA}$). The X-ray tube was operated at 45 kV and 40 mA. TEM measurements of the sample were carried out using a JEOL transmission electron microscope. Sample for the TEM was prepared by making a clear dispersion of nanoparticles in dimethyl formaldehyde and putting a drop of it on a carbon-coated copper grid. Formation of copper ferrite nanoparticles was first ascertained by electron dispersive X-ray (EDX) analysis combined with scanning electron microscope (SEM). SEM was done 'JEOL JSM-6610LV' Scanning Electron Microscope combined with EDX system (INCA Analyzer). For SEM analysis, the sample was dispersed on the aluminium stub used for sample mounting. The sample was scanned at an accelerating voltage of 20 kV at a working distance of 15mm. The particle size was measured at a magnification of 10kX.

4 General procedure for the synthesis of spiropyrimidine derivatives 4 (a-o)

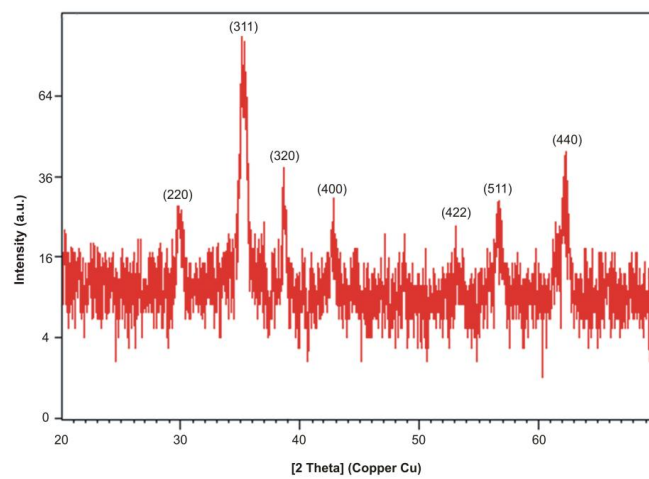
To a solution of cyclic ketones (1mmol), aromatic amines (2 mmol), formaldehyde (3.3 mmol, 36% aqueous solution), and a catalytic amount of CuFe_2O_4 (10 mol%) in ethanol (5 mL) was stirred at room temperature for the stipulated times. After completion of the reaction monitored by TLC, 10 mL ethanol was added to the reaction mixture and the catalyst CuFe_2O_4 was separated magnetically. The reaction mixture was allowed to stand overnight. The solid material was filtered off, washed with water (2X10 mL), dried and recrystallized from ethanol to furnish pure spiropyrimidine derivatives.

5 Reusability of the catalyst

After completion of the reaction, 10 mL ethanol was added to the reaction mixture and the catalyst CuFe_2O_4 was separated magnetically, washed with ethanol and then air dried. The recovered catalyst was used directly in the next runs and no substantial loss of activity was observed up to four cycles.

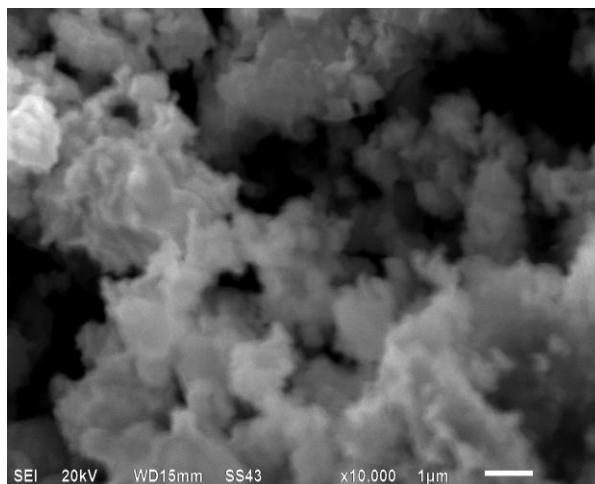


(a)

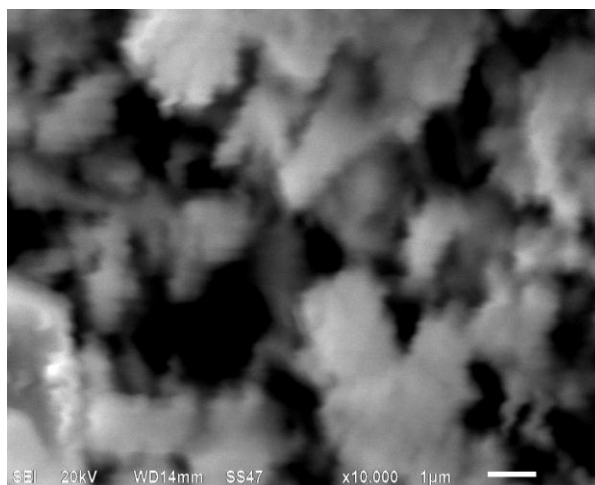


(b)

Fig. 1. (a) XRD spectrum of native CuFe_2O_4 catalyst. (b) XRD spectrum of reused CuFe_2O_4 catalyst after 4th cycle

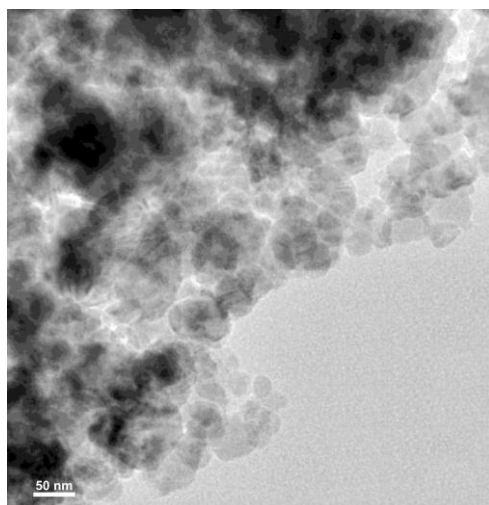


(a)

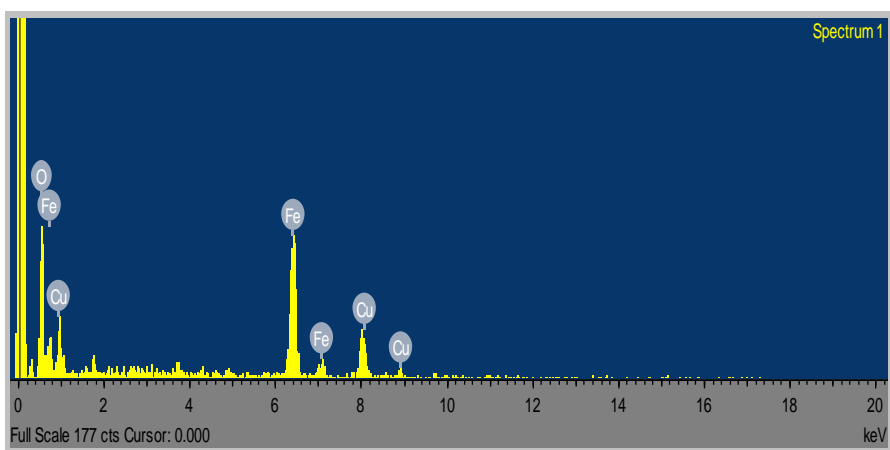


(b)

Fig. 2. (a) SEM image of native CuFe_2O_4 catalyst. (b) SEM image of reused CuFe_2O_4 catalyst after 4th cycle.

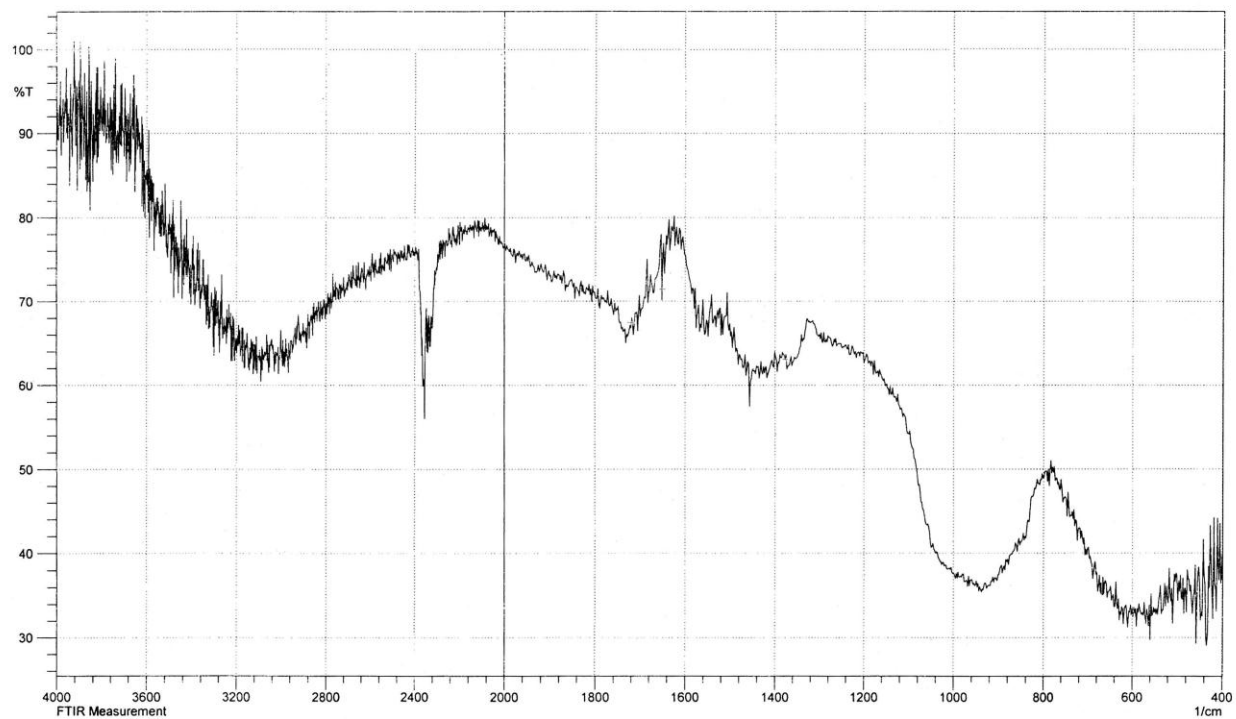


(a)

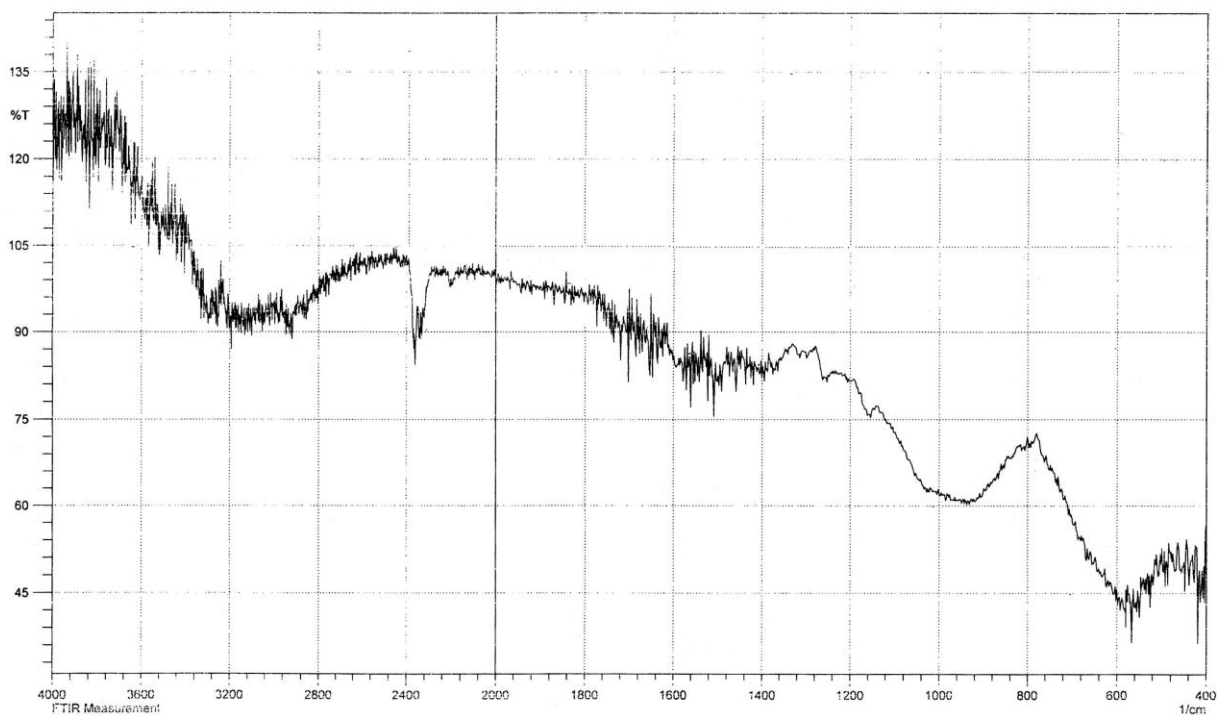


(b)

Fig. 3. (a) TEM image of native CuFe₂O₄ catalyst. (b) The EDX spectrum of native CuFe₂O₄ catalyst.



(a)



(b)

Fig. 4. (a) FT-IR spectrum of native CuFe_2O_4 catalyst. (b) FT-IR spectrum of reused CuFe_2O_4 catalyst after 4th cycle.

Spectral data of compounds 4 (a-o)

(4a) 2,4-bis-(4-fluorophenyl)-2,4-diazaspiro[5.5]undecan-7-one. White Solid; (Yield: 82%); mp 122-124 °C; IR (KBr): 2944, 2785, 1712, 1576, 1486, 1233, 1208, 827 cm⁻¹; ¹H NMR (300 MHz, CDCl₃): δ 1.70-1.64 (m, 2H), 1.87-1.84 (m, 4H), 2.36 (t, *J* = 6.3 Hz, 2H), 3.50-3.37 (q, *J* = 12.6 Hz, 4H), 4.15 (d, *J* = 11.4 Hz, 1H), 4.61 (d, *J* = 11.1 Hz, 1H), 7.00-6.97 (m, 8H, ArH), ¹³C NMR (75 MHz, CDCl₃): 20.8, 27.8, 35.0, 39.1, 49.9, 56.2, 70.1, 115.6, 115.9, 119.2, 146.2, 156.0, 159.2, 212.8; MS (ESI) *m/z*: 356 [M]⁺. Anal. Calcd for C₂₁H₂₂F₂N₂O: C, 70.77; H, 6.22; N, 7.86. Found: C, 70.65; H, 6.17; N, 8.05.

(4b) 2,4-bis-(4-trifluoromethylphenyl)-2,4-diazaspiro[5.5]undecan-7-one. White Solid; (Yield: 73%); mp 178-180 °C; IR (KBr): 2958, 2802, 1716, 1562, 1498, 1236, 1222, 816 cm⁻¹; ¹H NMR (300 MHz, CDCl₃): δ 1.76-1.69 (m, 2H), 1.93-1.88 (m, 4H), 2.42 (t, *J* = 6.6 Hz, 2H), 3.63 (m, 4H), 4.19 (d, *J* = 11.1 Hz, 1H), 4.60 (d, *J* = 11.7 Hz, 1H), 7.32-6.95 (m, 8H, ArH), ¹³C NMR (75 MHz, CDCl₃): 21.2, 27.5, 34.3, 41.8, 49.5, 55.4, 69.9, 112.7, 113.2, 117.6, 145.9, 153.5, 159.8, 211.3; MS (ESI) *m/z*: 456 [M]⁺. Anal. Calcd for C₂₃H₂₂F₆N₂O: C, 60.52; H, 4.86; N, 6.14. Found: C, 60.58; H, 4.72; N, 6.04.

(4c) 2,4-bis-(4-chlorophenyl)-2,4-diazaspiro[5.5]undecan-7-one. White Solid; (Yield: 79%); mp 160-162 °C; IR (KBr): 2948, 2782, 1708, 1592, 1488, 1232, 1216, 828 cm⁻¹; ¹H NMR (300 MHz, CDCl₃): δ 1.71-1.67 (m, 2H), 1.90-1.86 (m, 4H), 2.37 (t, *J* = 6.3 Hz, 2H), 3.51 (l, 4H), 4.22 (d, *J* = 11.4 Hz, 1H), 4.74 (d, *J* = 11.4 Hz, 1H), 6.98 (d, *J* = 7.2 Hz, 4H), 7.24 (d, *J* = 10.2 Hz, 4H), ¹³C NMR (75 MHz, CDCl₃): 20.8, 27.6, 34.7, 39.1, 49.9, 55.2, 67.9, 118.2, 125.2, 129.1, 148.3, 212.5; MS (ESI) *m/z*: 389 [M]⁺. Anal. Calcd for C₂₁H₂₂Cl₂N₂O: C, 64.79; H, 5.70; N, 7.20. Found: C, 64.88; H, 5.77; N, 7.08.

(4d) 2,4-di-p-tolyl-2,4-diazaspiro[5.5]undecan-7-one. White Solid; (Yield: 74%); mp 124-126 °C; IR (KBr): 2932, 1708, 1546, 1460, 1234, 1210, 816 cm⁻¹; ¹H NMR (300 MHz, CDCl₃): δ 1.68-1.66 (m, 2H), 1.88-1.86 (m, 4H), 2.29 (s, 6H), 2.39 (t, *J* = 6.0 Hz, 2H), 3.40 (d, *J* = 12.3 Hz, 2H), 3.52 (d, *J* = 12.6 Hz, 2H), 4.10 (d, *J* = 11.4 Hz, 1H), 4.79 (d, *J* = 11.1 Hz, 1H), 6.96 (d, *J* = 8.1 Hz, 4H), 7.10 (d, *J* = 8.1 Hz, 4H), ¹³C NMR (75 MHz, CDCl₃): 20.4, 20.8, 27.7, 29.6, 34.6, 39.1, 50.0, 55.4, 69.4, 117.4, 129.7, 147.8, 213.3; MS (ESI) *m/z*: 348 [M]⁺. Anal. Calcd for C₂₃H₂₈N₂O: C, 79.27; H, 8.10; N, 8.04. Found: C, 79.41; H, 8.21; N, 7.95.

(4e) 2,4-bis-(3-chloro-4-fluorophenyl)-2,4-diazaspiro[5.5]undecan-7-one. White Solid; (Yield: 75%); mp 248-250 °C; IR (KBr): 2948, 2782, 1708, 1592, 1488, 1232, 1216, 828 cm⁻¹; ¹H NMR (300 MHz, CDCl₃): δ 1.72-1.69 (m, 2H), 1.99-1.81 (m, 4H), 2.46 (t, *J* = 6.9 Hz, 2H), 3.35 (d, *J* = 12.6 Hz, 2H), 3.59 (d, *J* = 12.6 Hz, 2H), 4.28 (d, *J* = 11.4 Hz, 1H), 4.71 (d, *J* = 11.4 Hz, 1H), 6.87 (d, *J* = 8.4 Hz, 2H), 6.92 (s, 2H), 7.24 (d, *J* = 8.7 Hz, 2H), ¹³C NMR (75 MHz, CDCl₃): 20.3, 27.9, 34.8, 39.2, 50.8, 55.4, 69.3, 114.4, 119.9, 129.2, 131.6, 135.5, 148.7, 213.3; MS (ESI) *m/z*: 425 [M]⁺. Anal. Calcd for C₂₁H₂₀Cl₂F₂N₂O: C, 59.31; H, 4.74; N, 6.59. Found: C, 59.19; H, 4.82; N, 6.69.

(4f) 2,4-bis-(4-fluorophenyl)-10-methyl-2,4-diazaspiro[5.5]undecan-7-one. White Solid, (Yield: 76%); mp 130-132 °C; IR (KBr): 2978, 2922, 2863, 1712, 1609, 1498, 1474, 1256, 1126, 1026, 912, 806, 744 cm⁻¹; ¹H NMR (300 MHz, CDCl₃): δ 0.83 (d, *J* = 6.0 Hz, 3H), 1.06 (t, *J* = 12.6 Hz, 1H), 1.41-1.31 (m, 1H), 2.00 (br s, 2H), 2.37-2.22 (m, 2H), 2.57-2.45 (m, 1H), 3.03 (d, *J* = 12.3 Hz, 1H), 3.30 (d, *J* = 12.6 Hz, 1H), 3.61 (d, *J* = 12.6 Hz, 1H), 3.75 (d, *J* = 12.6 Hz, 1H), 4.14 (d, *J* = 11.4 Hz, 1H), 4.62 (d, *J* = 11.1 Hz, 1H), 7.00-6.98 (m, 8H), ¹³C NMR (75 MHz, CDCl₃): 21.2, 27.3, 35.7, 38.6, 43.1, 49.3, 56.0, 57.1, 70.2, 115.6, 115.9, 118.9, 119.0, 119.6, 146.2, 156.1, 159.1, 213.0; MS (ESI) *m/z*: 370 [M]⁺. Anal. Calcd for C₂₂H₂₄F₂N₂O: C, 71.33; H, 6.53; N, 7.56. Found: C, 71.60; H, 6.58; N, 7.35.

(4g) 2,4-bis-(4-trifluoromethylphenyl)-10-methyl-2,4-diazaspiro[5.5]undecan-7-one. White Solid, (Yield: 70%); mp 204-206 °C; IR (KBr): 2972, 2934, 2855, 1710, 1617, 1532, 1463, 1222, 1138, 1018, 922, 806, 738 cm⁻¹; ¹H NMR (300 MHz, CDCl₃): δ 0.92 (d, *J* = 6.6 Hz, 3H), 1.05 (t, *J* = 12.0 Hz, 1H), 1.63-1.58 (m, 1H), 2.13-2.01 (m, 2H), 2.58-2.52 (m, 3H), 3.24 (d, *J* = 12.3 Hz, 1H), 3.48 (d, *J* = 12.9 Hz, 1H), 3.68 (d, *J* = 11.7 Hz, 1H), 3.82 (d, *J* = 12.3 Hz, 1H), 4.21 (d, *J* = 11.7 Hz, 1H), 4.69 (d, *J* = 11.7 Hz, 1H), 7.11 (d, *J* = 8.1 Hz, 4H), 7.29 (d, *J* = 8.4 Hz, 4H), ¹³C NMR (75 MHz, CDCl₃): 21.8, 27.1, 37.7, 39.4, 42.1, 50.5, 55.9, 57.6, 69.4, 115.3, 118.1, 123.5, 127.1, 130.2, 135.6, 142.4, 150.9, 212.5; MS (ESI) *m/z*: 470 [M]⁺. Anal. Calcd for C₂₄H₂₄F₆N₂O: C, 61.27; H, 5.14; N, 5.95. Found: C, 61.50; H, 5.18; N, 5.78.

(4h) 2,4-bis-(4-chlorophenyl)-10-methyl-2,4-diazaspiro[5.5]undecan-7-one. White Solid, (Yield: 75%); mp 166-168 °C; IR (KBr): 2960, 2928, 2860, 1704, 1618, 1502, 1462, 1240, 1126, 1014, 910, 814, 736 cm⁻¹; ¹H NMR (300 MHz, CDCl₃): δ 0.85 (d, *J* = 6.3 Hz, 3H), 1.09 (t, *J* = 12.6 Hz, 1H), 2.04-1.98 (m, 3H), 2.35-2.28 (m, 2H), 2.51-2.49 (m, 1H), 3.14 (d, *J* = 12.9 Hz, 1H), 3.39 (d, *J* = 12.6 Hz, 1H), 3.71 (d, *J* = 12.6 Hz, 1H), 3.84 (d, *J* = 12.9 Hz, 1H), 4.25 (d, *J* = 11.4 Hz, 1H), 4.75 (d, *J* = 11.7 Hz, 1H), 7.06-6.98 (m, 4H), 7.27-7.23 (m, 4H), ¹³C NMR (75 MHz, CDCl₃): 21.2, 27.4, 35.6, 38.6, 42.9, 49.4, 55.0, 56.1, 68.1, 118.0, 118.6, 125.0, 125.6, 129.1, 129.2, 148.3, 212.6; MS (ESI) *m/z*: 403 [M]⁺. Anal. Calcd for C₂₂H₂₄Cl₂N₂O: C, 65.51; H, 6.00; N, 6.95. Found: C, 65.42; H, 6.07; N, 6.82.

(4i) 10-Methyl-2,4-di-p-tolyl-2,4-diazaspiro[5.5]undecan-7-one. White Solid, (Yield: 72%); mp 104-106 °C; IR (KBr): 2962, 2926, 1710, 1614, 1520, 1456, 1388, 1224, 1136, 918, 816, 732, 524 cm⁻¹; ¹H NMR (300 MHz, CDCl₃): δ 0.79 (d, *J* = 5.6 Hz, 3H), 1.03 (t, *J* = 12.8 Hz, 1H), 1.42-1.25 (m, 1H), 1.99-1.94 (m, 2H), 2.28-2.22 (l, 7H), 2.42-2.38 (m, 1H), 2.60-2.49 (m, 1H), 3.16 (d, *J* = 12.6 Hz, 1H), 3.21 (d, *J* = 12.6 Hz, 1H), 3.56 (d, *J* = 12.6 Hz, 1H), 3.82 (d, *J* = 12.6 Hz, 1H), 4.10 (d, *J* = 11.4 Hz, 1H), 4.78 (d, *J* = 11.4 Hz, 1H), 6.88-6.83 (m, 4H), 7.11 (d, *J* = 9.2 Hz, 4H), ¹³C NMR (75 MHz, CDCl₃): 20.5, 21.0, 27.3, 35.4, 37.1, 43.1, 49.8, 55.0, 56.9, 70.3, 116.7, 117.6, 129.5, 130.3, 147.9, 212.8; MS (ESI) *m/z*: 362 [M]⁺. Anal. Calcd for C₂₄H₃₀N₂O: C, 79.52; H, 8.34; N, 7.73. Found: C, 79.63; H, 8.26; N, 7.62.

(4j) 2,4-bis-(3-chloro-4-fluorophenyl)-10-methyl-2,4-diazaspiro[5.5]undecan-7-one. White Solid, (Yield: 64%); mp 236-238 °C; IR (KBr): 2960, 2928, 2860, 1704, 1618,

1502, 1462, 1240, 1126, 1014, 910, 814, 736 cm^{-1} ; ^1H NMR (300 MHz, CDCl_3): δ 0.91 (d, $J = 6.9$ Hz, 3H), 1.03 (t, $J = 12.6$ Hz, 1H), 1.68-1.61 (m, 1H), 2.04-1.98 (m, 2H), 2.51-2.45 (m, 3H), 3.29 (d, $J = 12.3$ Hz, 2H), 3.54 (d, $J = 12.3$ Hz, 1H), 3.83 (d, $J = 12.3$ Hz, 1H), 4.21 (d, $J = 11.1$ Hz, 1H), 4.73 (d, $J = 11.1$ Hz, 1H), 6.81-6.74 (m, 2H), 6.91 (s, 2H), 7.19 (d, $J = 8.4$ Hz, 2H), ^{13}C NMR (75 MHz, CDCl_3): 21.9, 27.8, 36.2, 39.7, 42.9, 50.3, 55.1, 56.8, 67.4, 115.3, 118.8, 120.5, 124.1, 130.5, 136.7, 151.3, 212.6; MS (ESI) m/z : 439 $[\text{M}]^+$. Anal. Calcd for $\text{C}_{22}\text{H}_{22}\text{Cl}_2\text{F}_2\text{N}_2\text{O}$: C, 60.15; H, 5.05; N, 6.38. Found: C, 60.03; H, 5.14; N, 6.27.

(4k)2,4-bis(4-fluorophenyl)-10-(1,1-dioxa-2,2-dimethylene)-2,4-diazaspiro[5.5]undecan-7-one. White solid, (Yield: 58%); mp 130-132 $^\circ\text{C}$; IR (KBr): 2988, 1704, 1528, 1445, 1256, 1047, 932, 830, 718 cm^{-1} ; ^1H NMR (300 MHz, CDCl_3): δ 2.02 (t, $J = 6.9$ Hz, 2H), 2.14 (s, 2H), 2.57 (t, $J = 6.6$ Hz, 2H), 3.30 (d, $J = 12.3$ Hz, 2H), 3.92-3.66 (m, 6H), 4.00 (d, $J = 10.8$ Hz, 1H), 4.72 (d, $J = 11.1$ Hz, 1H), 6.99-6.97 (m, 8H), ^{13}C NMR (75 MHz, CDCl_3): 35.0, 36.3, 40.5, 48.7, 57.0, 64.4, 69.8, 107.1, 115.5, 115.8, 119.2, 146.4, 155.9, 159.1, 211.4; MS (ESI) m/z : 414 $[\text{M}]^+$. Anal. Calcd for $\text{C}_{23}\text{H}_{24}\text{F}_2\text{N}_2\text{O}_3$: C, 66.65; H, 5.84; N, 6.76. Found: C, 66.82; H, 5.77; N, 6.48.

(4l)2,4-bis(4-trifluoromethylphenyl)-10-(1,1-dioxa-2,2-dimethylene)-2,4-diazaspiro[5.5]undecan-7-one. White solid, (Yield: 54%); mp 144-146 $^\circ\text{C}$; IR (KBr): 2954, 1708, 1512, 1456, 1244, 1042, 918, 826, 726 cm^{-1} ; ^1H NMR (300 MHz, CDCl_3): δ 2.13-2.01 (m, 4H), 2.43-2.37 (m, 1H), 2.97-2.65 (m, 4H), 3.22-3.16 (m, 1H) 3.44-3.37 (m, 2H), 4.05-4.02 (m, 4H), 6.57 (d, $J = 8.7$ Hz, 4H), 7.37 (d, $J = 8.7$ Hz, 4H), ^{13}C NMR (75 MHz, CDCl_3): 34.4, 38.3, 38.7, 43.0, 46.0, 48.9, 64.7, 64.8, 107.0, 111.9, 126.6, 150.2, 211.5; MS (ESI) m/z : 514 $[\text{M}]^+$. Anal. Calcd for $\text{C}_{25}\text{H}_{24}\text{F}_6\text{N}_2\text{O}_3$: C, 58.37; H, 4.70; N, 5.45. Found: C, 58.48; H, 4.47; N, 5.52.

(4m)2,4-bis(4-chlorophenyl)-10-(1,1-dioxa-2,2-dimethylene)-2,4-diazaspiro[5.5]undecan-7-one. White solid, (Yield: 61%); mp 132-134 $^\circ\text{C}$; IR (KBr): 2954, 1708, 1512, 1456, 1244, 1042, 918, 826, 726 cm^{-1} ; ^1H NMR (300 MHz, CDCl_3): δ 2.02 (t, $J = 6.9$ Hz, 2H), 2.11 (s, 2H), 2.58 (t, $J = 6.9$ Hz, 2H), 3.37 (d, $J = 12.6$ Hz, 2H), 3.85-3.74 (m, 4H), 3.93-3.88 (m, 2H), 4.08 (d, $J = 11.1$ Hz, 1H), 4.84 (d, $J = 11.4$ Hz, 1H), 6.98 (d, $J = 9.0$ Hz, 4H), 7.23 (d, $J = 9.0$ Hz, 4H), ^{13}C NMR (75 MHz, CDCl_3): 34.9, 36.3, 40.4, 48.7, 56.0, 64.4, 67.7, 106.9, 118.3, 125.2, 129.1, 148.4, 211.2; MS (ESI) m/z : 447 $[\text{M}]^+$. Anal. Calcd for $\text{C}_{23}\text{H}_{24}\text{Cl}_2\text{N}_2\text{O}_3$: C, 61.75; H, 5.41; N, 6.26. Found: C, 61.84; H, 5.47; N, 6.18.

(4n)10-(1,1-dioxa-2,2-dimethylene)-2,4-di-p-tolyl-2,4-diazaspiro[5.5]undecan-7-one. White Solid, (Yield: 52%); mp 126-128 $^\circ\text{C}$; IR (KBr): 2928, 1710, 1522, 1108, 914, 734, 652 cm^{-1} ; ^1H NMR (300 MHz, CDCl_3): δ 2.02 (t, $J = 7.6$ Hz, 2H), 2.16 (s, 2H), 2.27 (s, 6H), 2.62 (t, $J = 6.6$ Hz, 2H), 3.24 (d, $J = 12.8$ Hz, 2H), 3.79-3.72 (l, 4H), 3.88-3.82 (m, 2H), 3.93 (d, $J = 11.1$ Hz, 1H), 4.92 (d, $J = 10.8$ Hz, 1H), 6.98 (d, $J = 8.6$ Hz, 4H), 7.08 (d, $J = 8.6$ Hz, 4H), ^{13}C NMR (75 MHz, CDCl_3): 20.7, 35.3, 36.4, 40.1, 49.6, 56.4, 64.3, 69.7, 107.2, 117.4, 129.3, 149.7, 212.4; MS (ESI) m/z : 406 $[\text{M}]^+$. Anal. Calcd for $\text{C}_{25}\text{H}_{30}\text{N}_2\text{O}_3$: C, 73.86; H, 7.44; N, 6.89. Found: C, 73.75; H, 7.54; N, 6.74.

(4o)2,4-bis(3-chloro-4-fluorophenyl)-10-(1,1-dioxo-2,2-dimethylene)-2,4-diazaspiro[5.5]undecan-7-one. White solid, (Yield: 55%); mp 206-208 °C; IR (KBr): 2954, 1708, 1512, 1456, 1244, 1042, 918, 826, 726 cm⁻¹; ¹H NMR (300 MHz, CDCl₃): δ 2.09 (t, *J* = 7.5 Hz, 2H), 2.23 (s, 2H), 2.64 (t, *J* = 6.9 Hz, 2H), 3.38 (d, *J* = 12.3 Hz, 2H), 3.84-3.72 (m, 4H), 3.92-3.88 (m, 2H), 4.21 (d, *J* = 11.7 Hz, 1H), 4.85 (d, *J* = 11.7 Hz, 1H), 6.91 (s, 2H), 6.99 (d, *J* = 9.0 Hz, 2H), 7.29 (d, *J* = 9.0 Hz, 2H), ¹³C NMR (75 MHz, CDCl₃): 24.6, 30.1, 37.1, 45.7, 54.1, 62.9, 64.8, 105.9, 118.2, 122.5, 128.1, 134.5, 138.5, 148.3, 211.3; MS (ESI) *m/z*: 483 [M]⁺. Anal. Calcd for C₂₃H₂₂Cl₂F₂N₂O₃: C, 57.15; H, 4.59; N, 5.80. Found: C, 57.26; H, 4.70; N, 5.63.



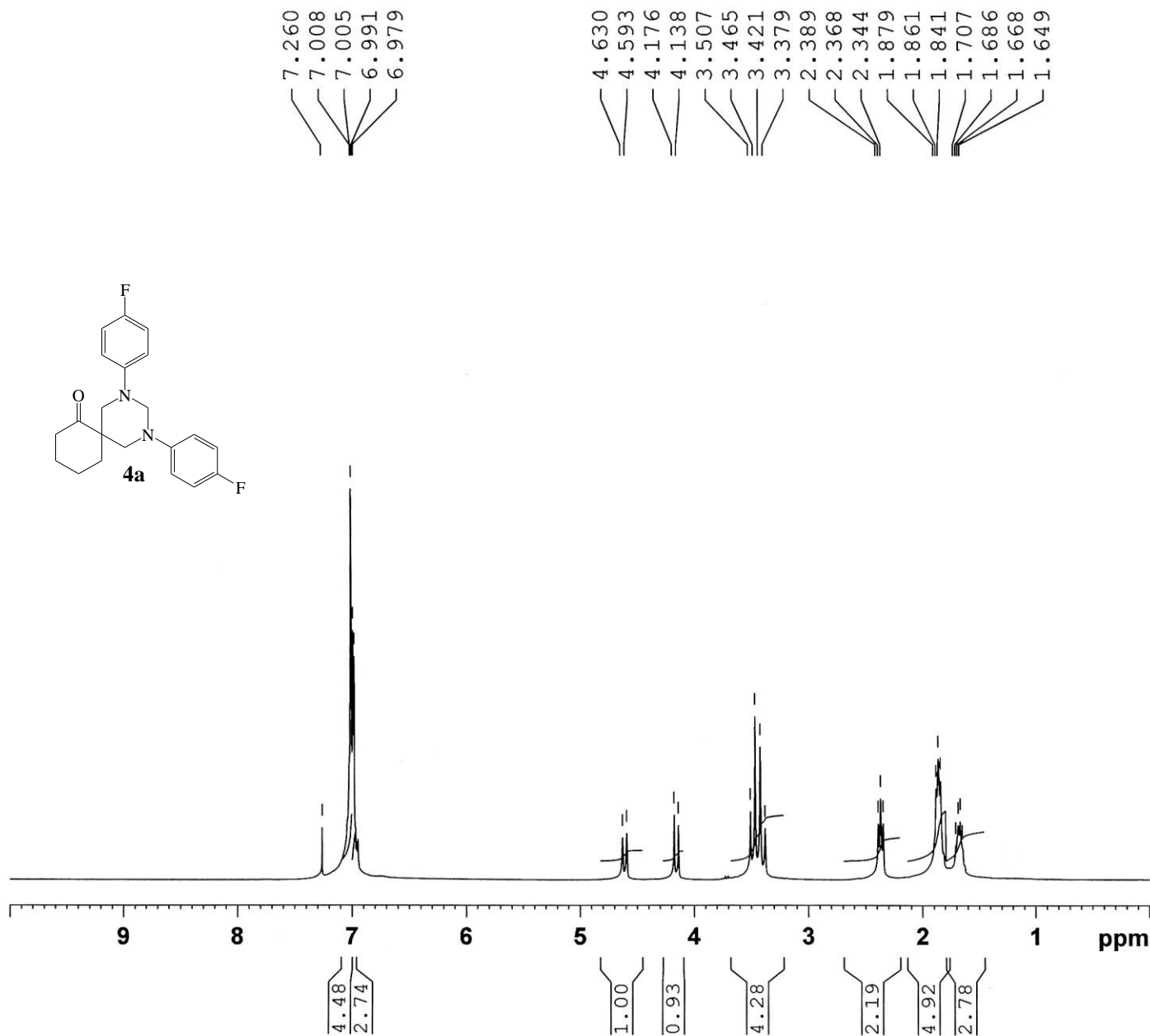
Current Data Parameters
NAME proton
EXPNO 200
PROCNO 1

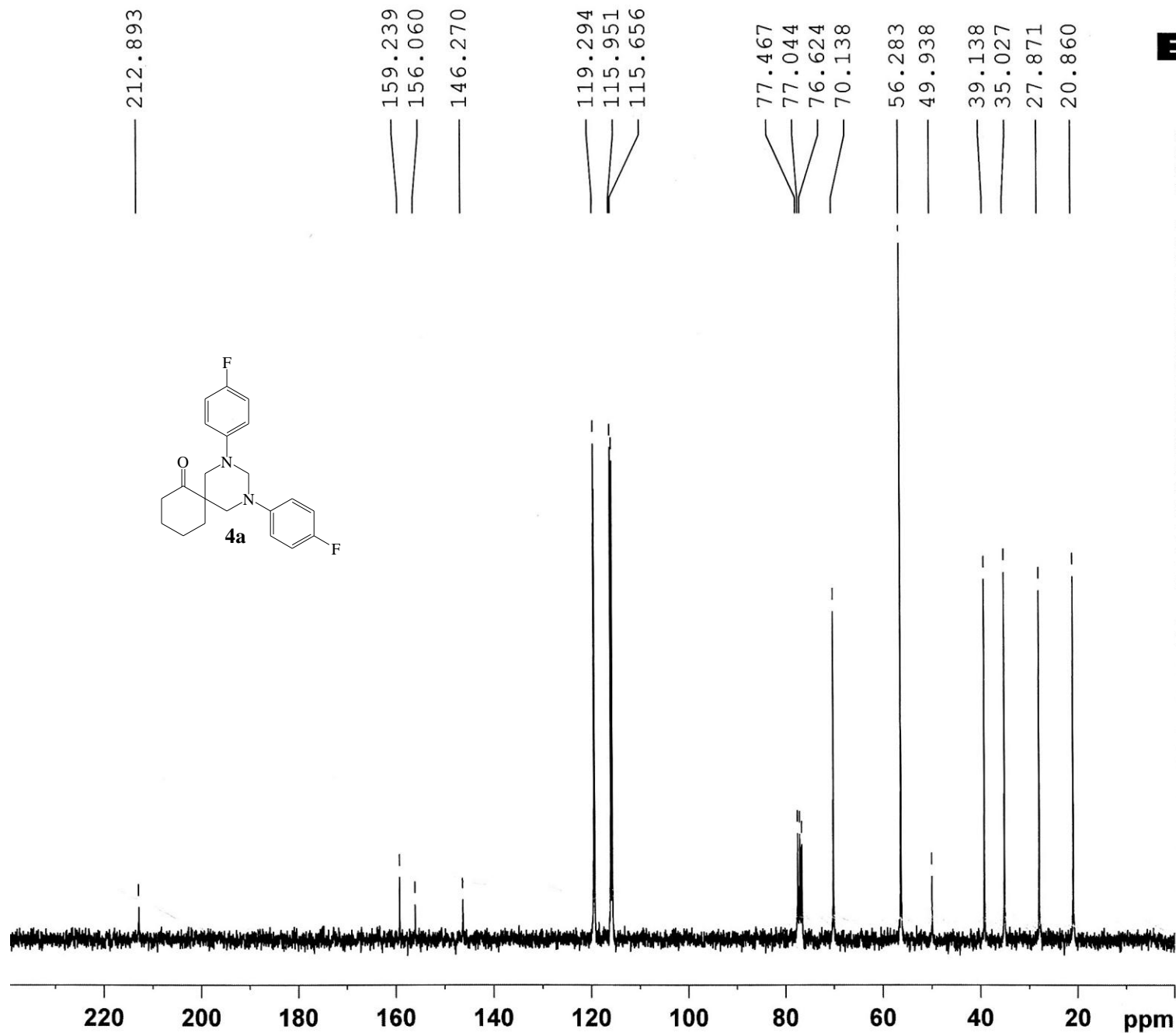
F2 - Acquisition Parameters

Date_ 20120814
Time 13.58
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 26
DS 0
SWH 4807.692 Hz
FIDRES 0.146719 Hz
AQ 3.4079220 sec
RG 80.6
DW 104.000 usec
DE 6.00 usec
TE 291.8 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 11.00 usec
PL1 0.00 dB
SFO1 300.1320008 MHz

F2 - Processing parameters
SI 16384
SF 300.1300022 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00





Current Data Parameters
NAME carbon
EXPNO 8
PROCNO 1

F2 - Acquisition Parameters
Date_ 20120814
Time_ 14.08
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zgpg
TD 32768
SOLVENT CDCl3
NS 488
DS 4
SWH 19531.250 Hz
FIDRES 0.596046 Hz
AQ 0.8389108 sec
RG 71.8
DW 25.600 usec
DE 6.00 usec
TE 292.4 K
D1 1.00000000 sec
d11 0.03000000 sec
DELTA 0.89999998 sec
TDO 1

===== CHANNEL f1 =====
NUC1 13C
P1 9.20 usec
PL1 -1.00 dB
SFO1 75.4772005 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL12 17.20 dB
PL13 20.20 dB
PL2 0.00 dB
SFO2 300.1315007 MHz

F2 - Processing parameters
SI 131072
SF 75.4677490 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.40

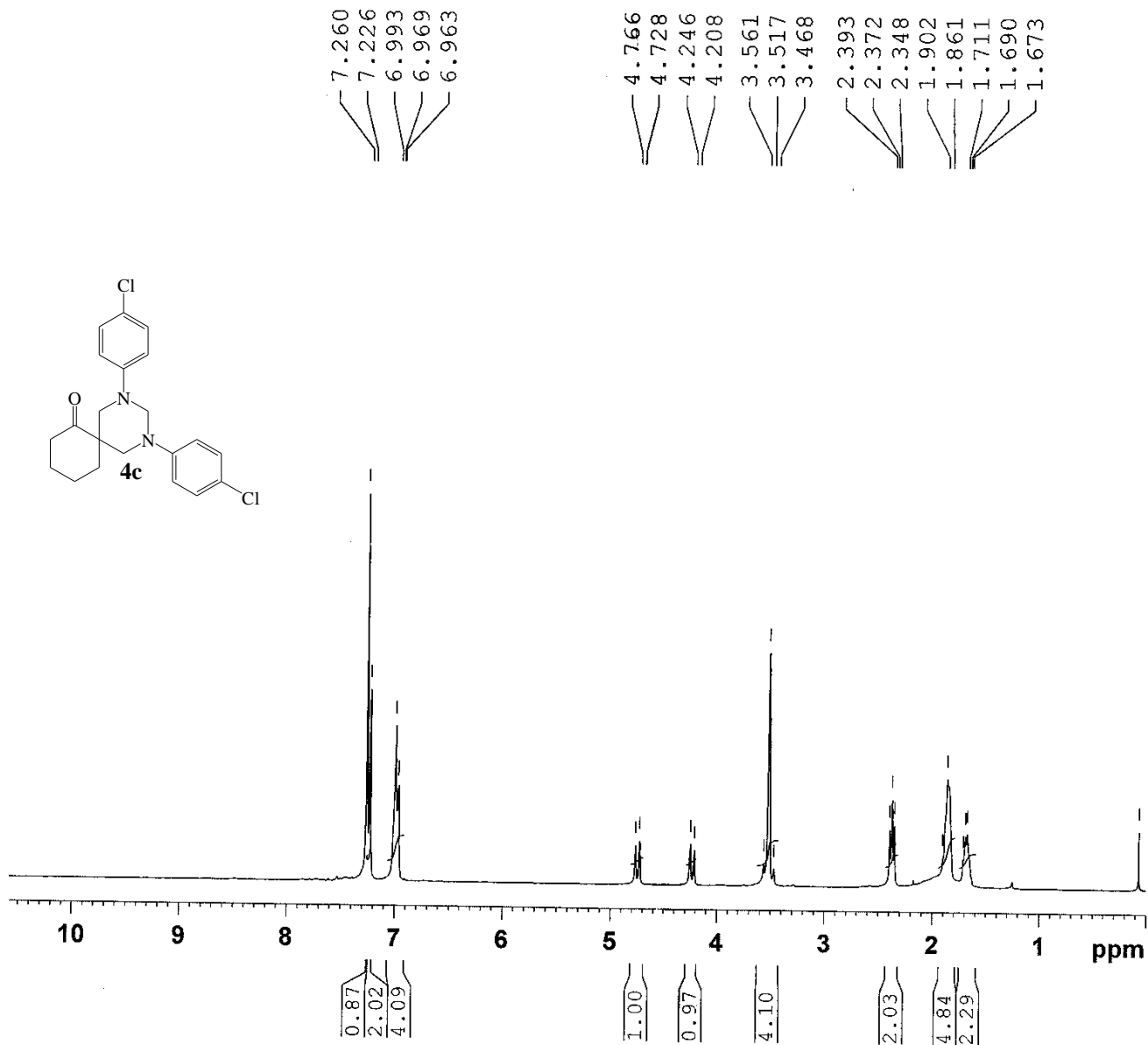


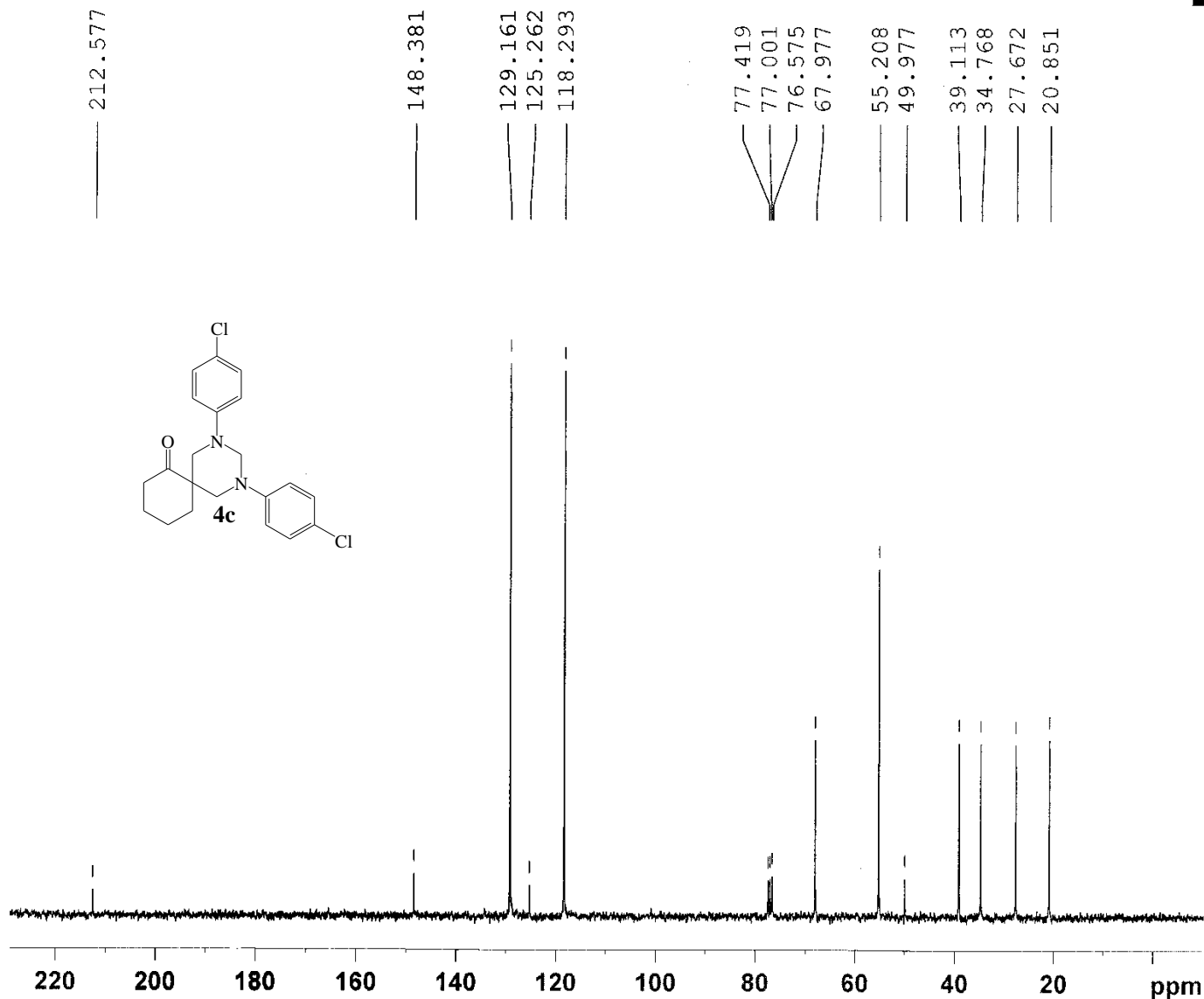
Current Data Parameters
NAME prasadH
EXPNO 1939
PROCNO 1

F2 - Acquisition Parameters
Date_ 20110808
Time_ 15.04
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 32
DS 0
SWH 4807.692 Hz
FIDRES 0.146719 Hz
AQ 3.4079220 sec
RG 406
DW 104.000 usec
DE 6.00 usec
TE 292.6 K
D1 1.00000000 sec
TD0 1

==== CHANNEL f1 =====
NUC1 1H
P1 11.00 usec
PL1 0.00 dB
SF01 300.132008 MHz

F2 - Processing parameters
SI 16384
SF 300.130040 MHz
WDW EM
SSB 0
LB 0.50 Hz
GB 0
PC 1.00





Current Data Parameters
NAME prasad C
EXPNO 462
PROCNO 1

F2 - Acquisition Parameters
Date 20110923
Time 11.10
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zgpg
TD 32768
SOLVENT CDC13
NS 201
DS 4
SWH 18115.941 Hz
FIDRES 0.552855 Hz
AQ 0.9044468 sec
RG 144
DW 27.600 usec
DE 6.00 usec
TE 292.3 K
D1 1.00000000 sec
d11 0.03000000 sec
DELTA 0.89999998 sec
TD0 1

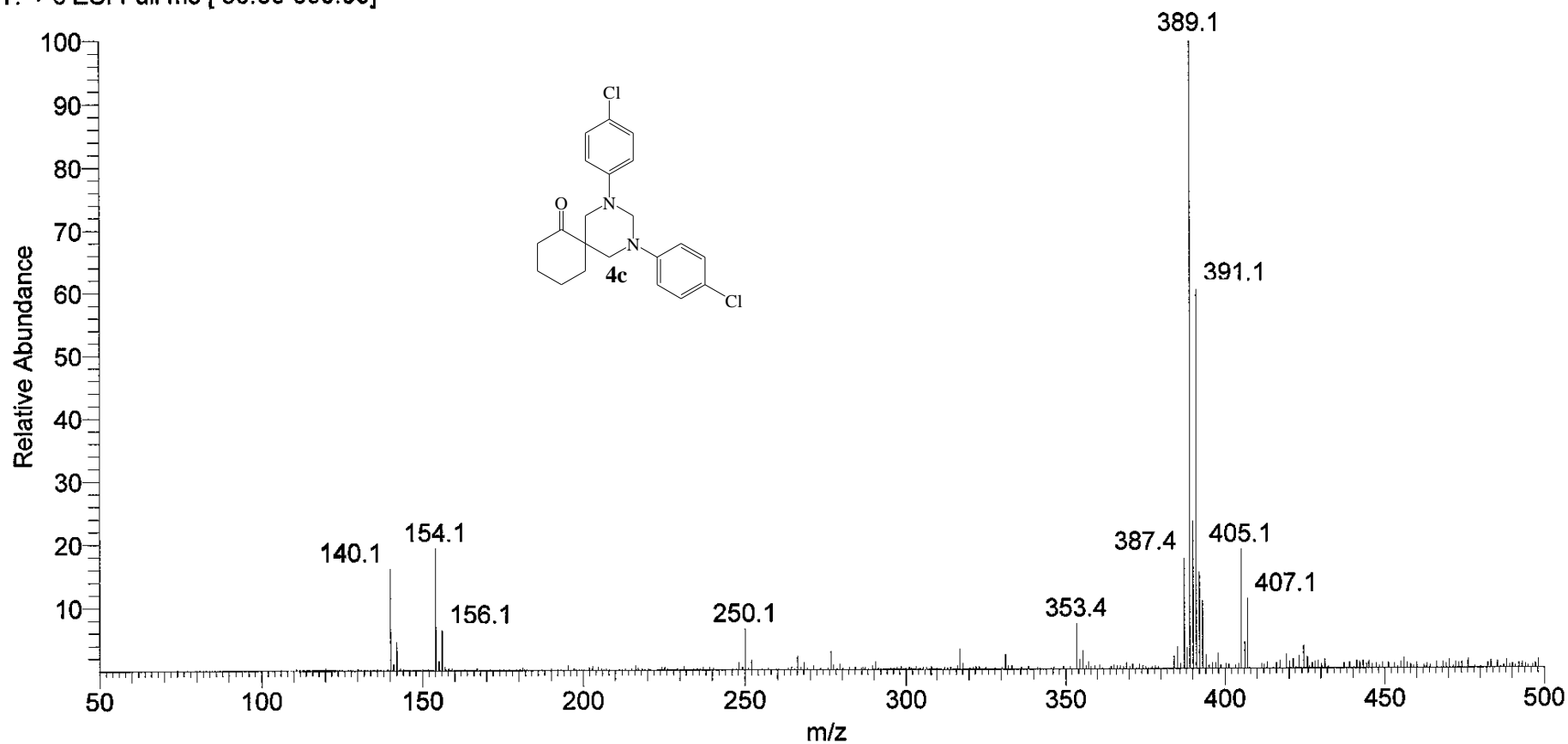
==== CHANNEL f1 =====
NUC1 13C
P1 9.20 usec
PL1 -1.00 dB
SFO1 75.4760505 MHz

==== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL12 17.20 dB
PL13 20.20 dB
PL2 0.00 dB
SFO2 300.1315007 MHz

F2 - Processing parameters
SI 131072
SF 75.4677554 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40

MSAIF, CDRI LUCKNOW

Original Data Path: 11SEP6161.RAW
Current Data Path: C:\Data2011\SEP11\
Sample ID: AVJ-82 #6411
Acquisition Date: 9/6/2011 2:17:40 PM
Vial: B:21
11SEP6161 #60-146 RT: 0.79-1.80 AV: 87 SB: 1 0.00 NL: 1.57E6
T: + c ESI Full ms [50.00-500.00]



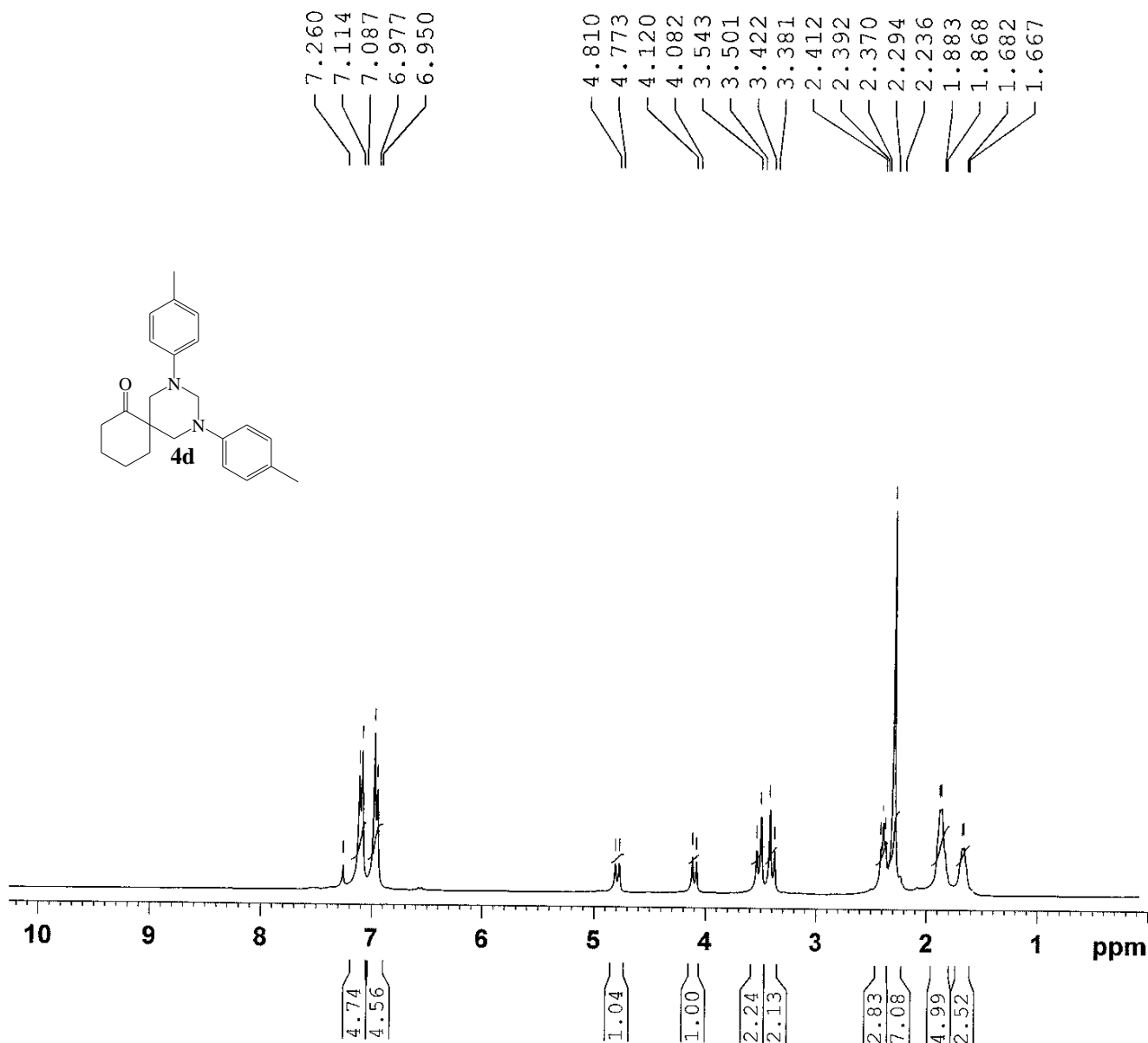
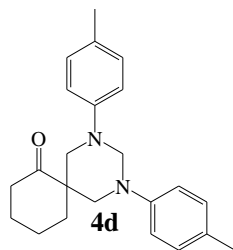


Current Data Parameters
NAME prasadH
EXPNO 1519
PROCNO 1

F2 - Acquisition Parameters
Date_ 20101116
Time 11.02
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 32
DS 0
SWH 4807.692 Hz
FIDRES 0.146719 Hz
AQ 3.4079220 sec
RG 64
DW 104.000 usec
DE 6.00 usec
TE 291.9 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 11.00 usec
PL1 0.00 dB
SFO1 300.1320008 MHz

F2 - Processing parameters
SI 16384
SF 300.1300045 MHz
WDW EM
SSB 0
LB 0.50 Hz
GB 0
PC 1.00





Current Data Parameters
NAME prasad C
EXPNO 372
PROCNO 1

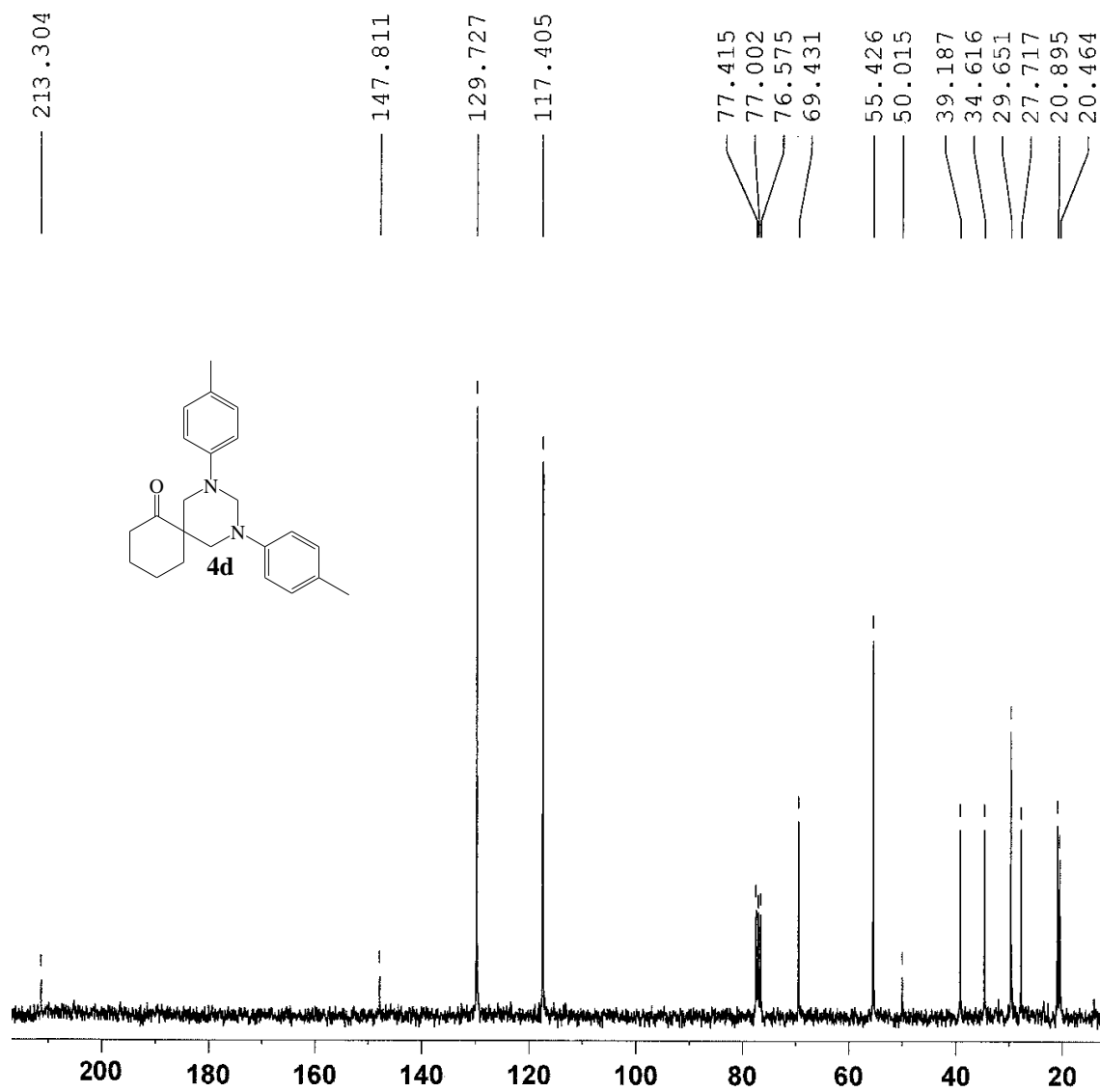
F2 - Acquisition Parameters

Date_ 20101116
Time_ 11.05
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zgpg
TD 32768
SOLVENT CDCl3
NS 261
DS 4
SWH 18115.941 Hz
FIDRES 0.552855 Hz
AQ 0.9044468 sec
RG 80.6
DW 27.600 usec
DE 6.00 usec
TE 292.1 K
D1 1.00000000 sec
d11 0.03000000 sec
DELTA 0.89999998 sec
TDO 1

=====
CHANNEL f1
NUC1 13C
P1 9.20 usec
PL1 -1.00 dB
SFO1 75.4740505 MHz

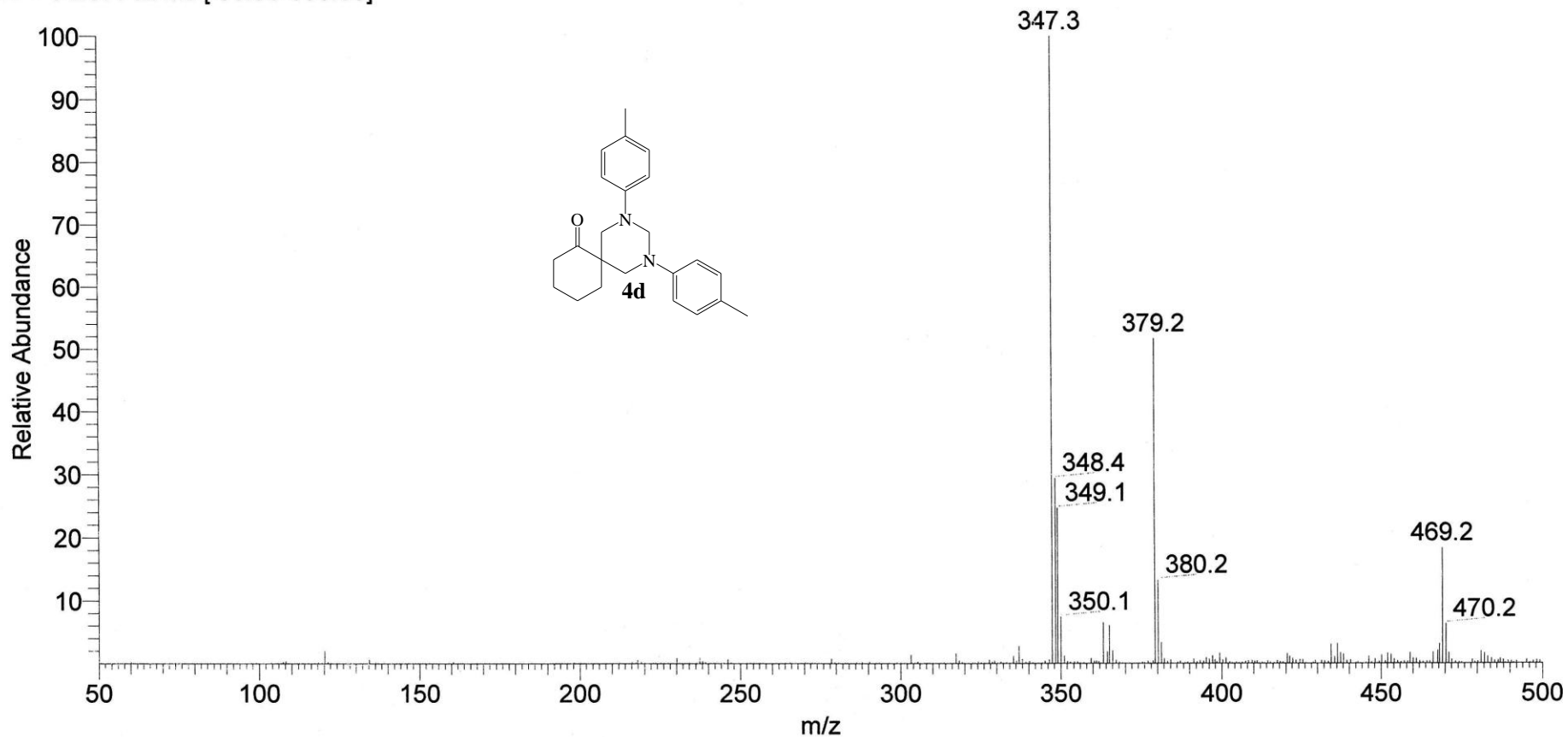
=====
CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PLI2 17.20 dB
PLI3 20.20 dB
PL2 0.00 dB
SFO2 300.1315007 MHz

F2 - Processing parameters
SI 131072
SF 75.4677548 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40



MSAIF, CDRI LUCKNOW

Original Data Path: 10E15NOV15111150151_114809.RAW
Current Data Path: C:\GAP0032\TCW03A\NOV10\
Sample ID: AVJ-62 #5694
Acquisition Date: 11/15/2010 2:28:14 PM
Vial: B:19
10E15NOV15111150151_114809 #11-64 RT: 0.10-0.60 AV: 54 SB: 1 0.00 NL: 1.10E8
T: + c ESI Full ms [50.00-500.00]





Current Data Parameters
NAME prasad C
EXPNO 466
PROCNO 1

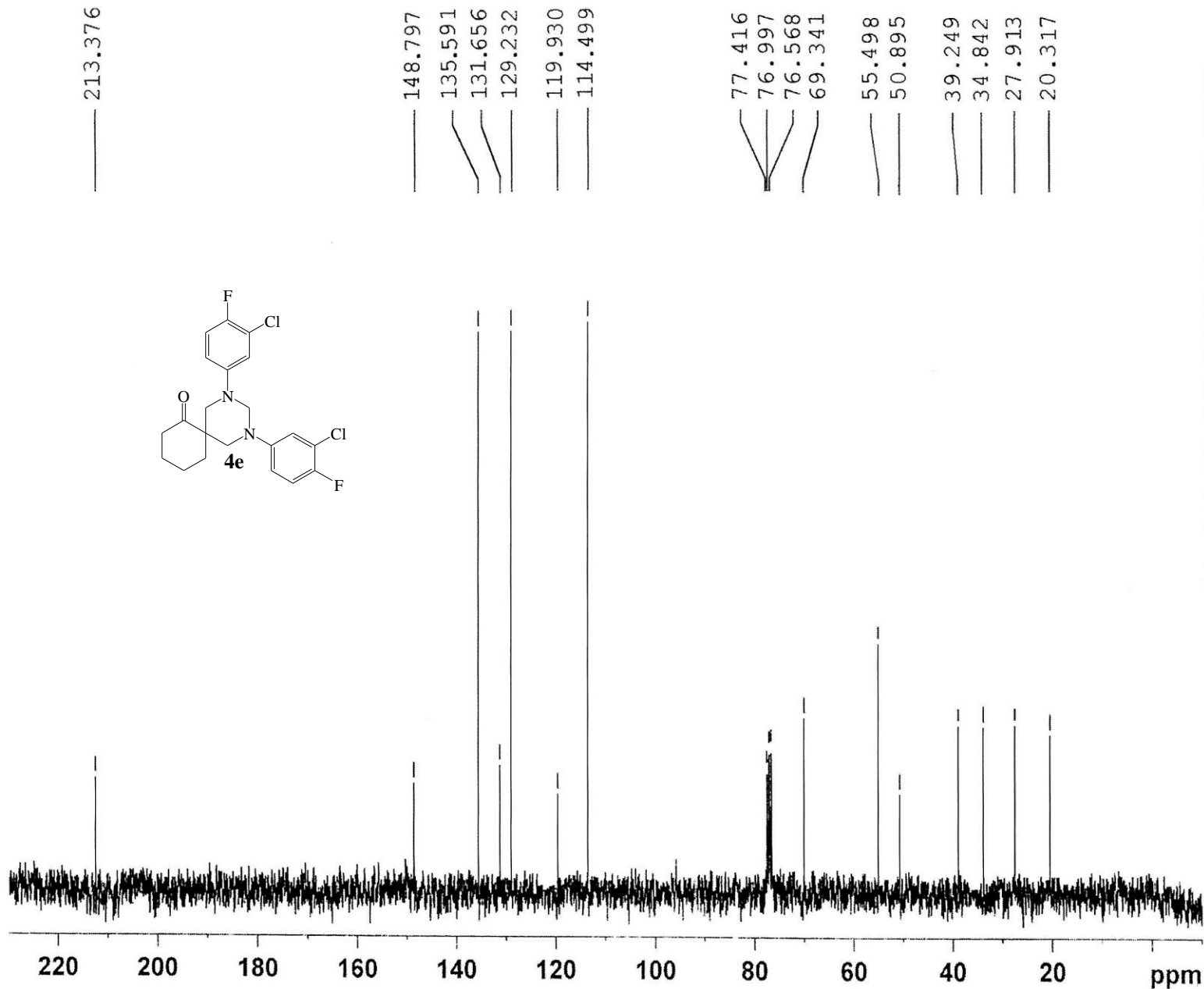
F2 - Acquisition Parameters

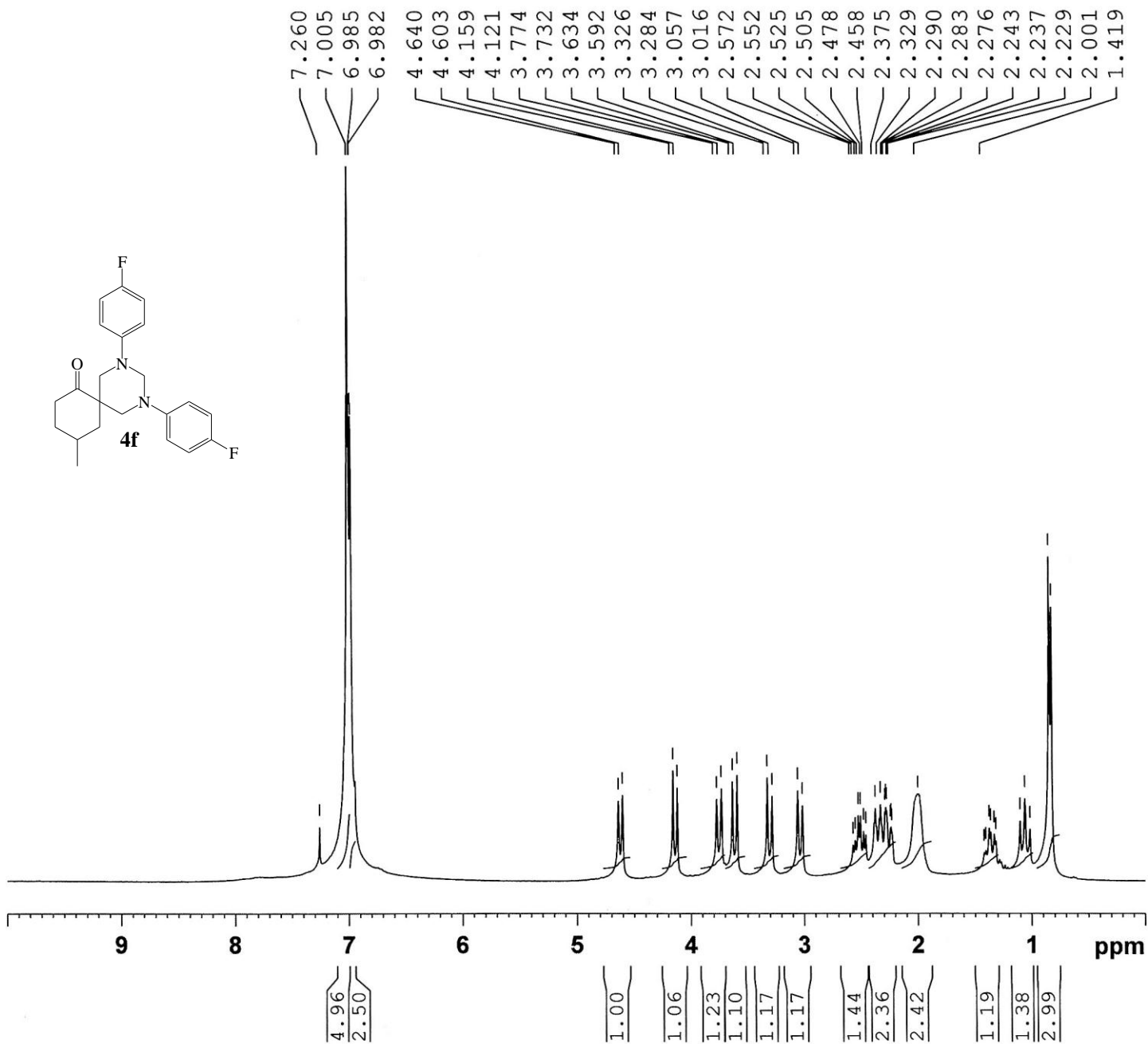
Date 20110923
Time 12.33
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zgpg
TD 32768
SOLVENT CDCl3
NS 97
DS 4
SWH 18115.941 Hz
FIDRES 0.552855 Hz
AQ 0.9044468 sec
RG 144
DW 27.600 usec
DE 6.00 usec
TE 293.1 K
D1 1.00000000 sec
d11 0.03000000 sec
DELTA 0.89999998 sec
TDO 1

==== CHANNEL f1 =====
NUC1 13C
P1 9.20 usec
PL1 -1.00 dB
SFO1 75.4760505 MHz

==== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL12 17.20 dB
PL13 20.20 dB
PL2 0.00 dB
SFO2 300.1315007 MHz

F2 - Processing parameters
SI 131072
SF 75.4677534 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40



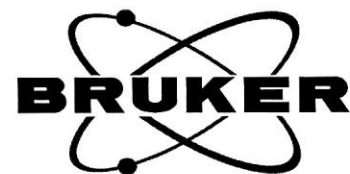


Current Data Parameters
NAME proton
EXPNO 201
PROCNO 1

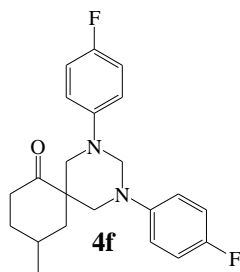
F2 - Acquisition Parameters
Date_ 20120814
Time_ 14.21
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 28
DS 0
SWH 4807.692 Hz
FIDRES 0.146719 Hz
AQ 3.4079220 sec
RG 80.6
DW 104.000 usec
DE 6.00 usec
TE 292.0 K
D1 1.00000000 sec
TD0 1

==== CHANNEL f1 =====
NUC1 1H
P1 11.00 usec
PL1 0.00 dB
SFO1 300.1320008 MHz

F2 - Processing parameters
SI 16384
SF 300.1300021 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00



4.640
4.603
4.159
4.121
3.774
3.732
3.634
3.592
3.326
3.284
3.057
3.016
2.572
2.552
2.525
2.505
2.478
2.458
2.375
2.329
2.290
2.283
2.276
2.243
2.237
2.229
2.001
1.419
1.404
1.374
1.361
1.330
1.316
1.103
1.061
1.017
0.844
0.824

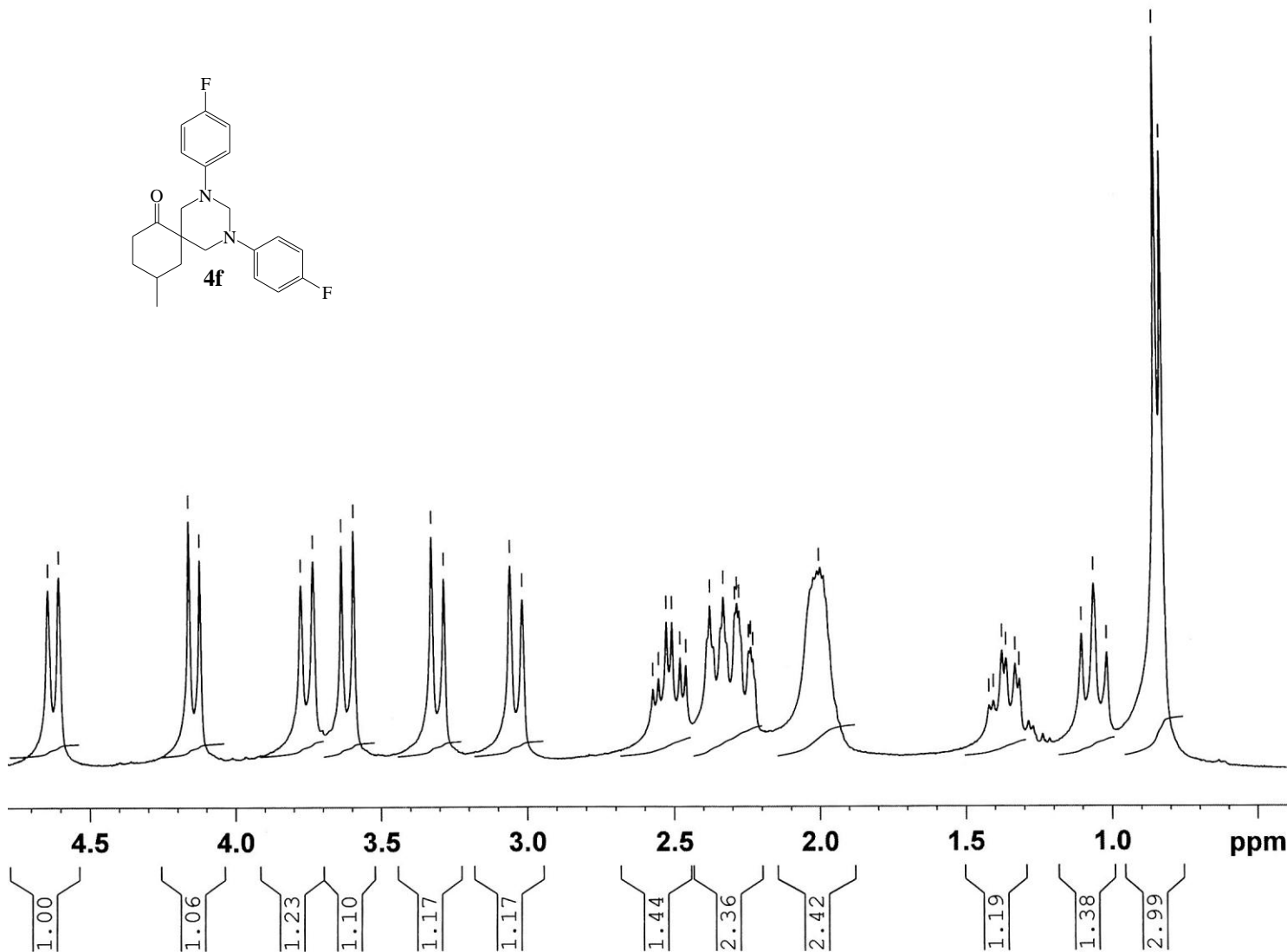


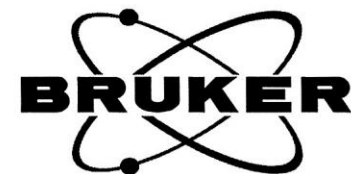
Current Data Parameters
NAME proton
EXPNO 201
PROCNO 1

F2 - Acquisition Parameters
Date_ 20120814
Time_ 14.21
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 28
DS 0
SWH 4807.692 Hz
FIDRES 0.146719 Hz
AQ 3.4079220 sec
RG 80.6
DW 104.000 usec
DE 6.00 usec
TE 292.0 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 11.00 usec
PL1 0.00 dB
SFO1 300.1320008 MHz

F2 - Processing parameters
SI 16384
SF 300.1300021 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00





Current Data Parameters
 NAME carbon
 EXPNO 9
 PROCNO 1

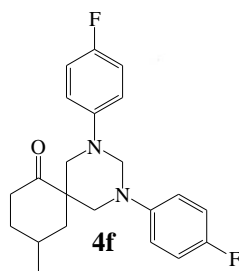
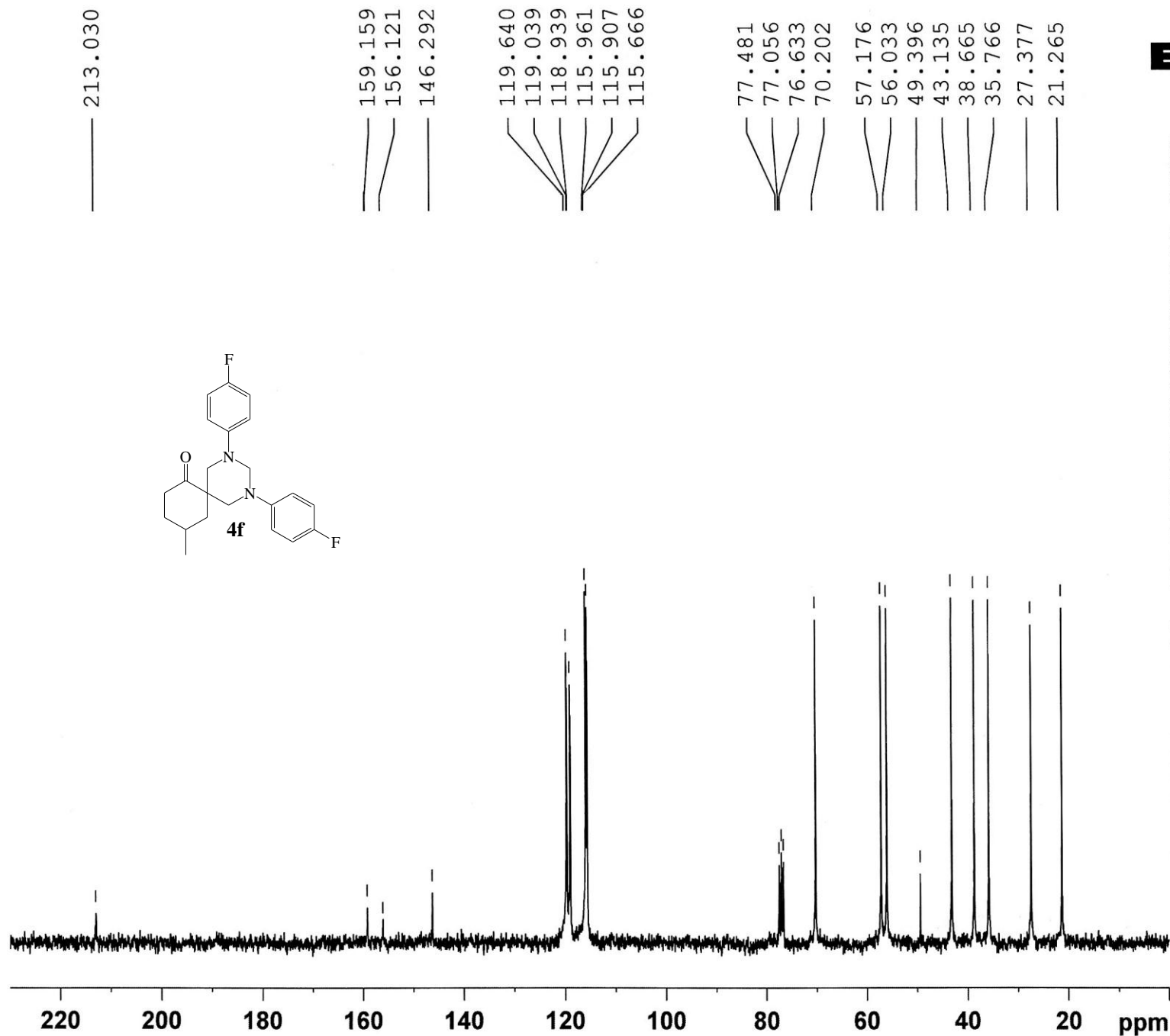
F2 - Acquisition Parameters

Date 20120814
 Time 14.29
 INSTRUM spect
 PROBHD 5 mm BBO BB-1H
 PULPROG zgpg
 TD 32768
 SOLVENT CDCl3
 NS 713
 DS 4
 SWH 19531.250 Hz
 FIDRES 0.596046 Hz
 AQ 0.8389108 sec
 RG 71.8
 DW 25.600 usec
 DE 6.00 usec
 TE 292.2 K
 D1 1.00000000 sec
 d11 0.03000000 sec
 DELTA 0.89999998 sec
 TDO 1

==== CHANNEL f1 =====
 NUC1 13C
 P1 9.20 usec
 PL1 -1.00 dB
 SFO1 75.4772005 MHz

==== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 80.00 usec
 PL12 17.20 dB
 PL13 20.20 dB
 PL2 0.00 dB
 SFO2 300.1315007 MHz

F2 - Processing parameters
 SI 131072
 SF 75.4677490 MHz
 WDW EM
 SSB 0
 LB 3.00 Hz
 GB 0
 PC 1.40





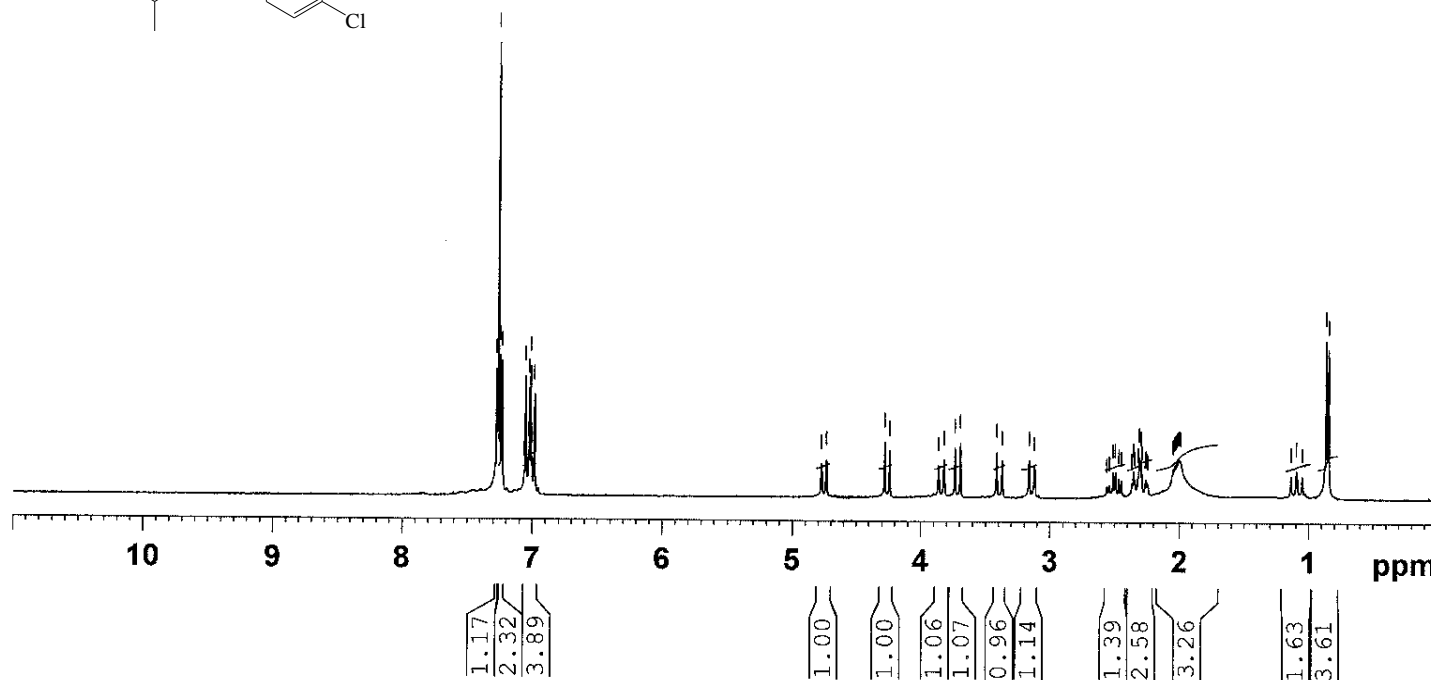
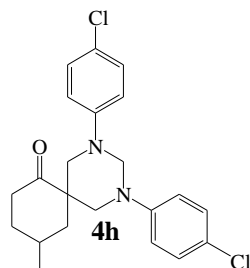
Current Data Parameters
NAME prasadh
EXPNO 1940
PROCNO 1

F2 - Acquisition Parameters
Date_ 20110808
Time_ 15.13
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 32768
SOLVENT CDC13
NS 32
DS 0
SWH 4807.692 Hz
FIDRES 0.146719 Hz
AQ 3.4079220 sec
RG 406
DW 104.000 usec
DE 6.00 usec
TE 292.6 K
D1 1.00000000 sec
TD0 1

==== CHANNEL f1 =====
NUC1 1H
P1 11.00 usec
PL1 0.00 dB
SFO1 300.1320008 MHz

F2 - Processing parameters
SI 16384
SF 300.1300039 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

7.274
7.260
7.245
7.230
7.063
7.052
7.045
7.029
7.023
7.012
7.005
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6.983
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4.735
4.277
4.239
3.861
3.818
3.735
3.693
3.412
3.370
3.162
3.119
2.511
2.491
2.353
2.307
2.295
2.288
2.045
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2.016
2.006
1.998
1.988
1.136
1.094
1.049
0.865
0.844





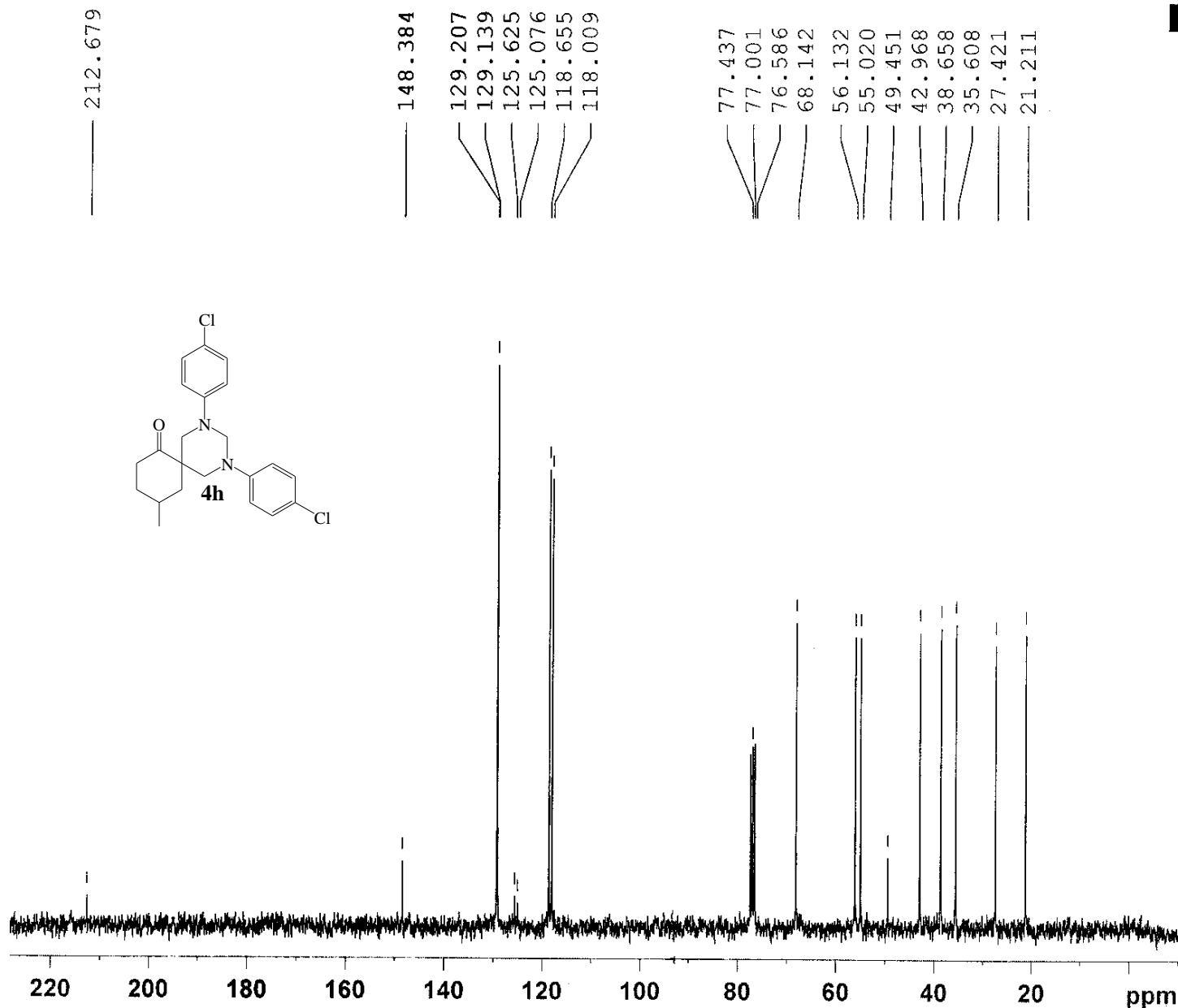
Current Data Parameters
 NAME prasad C
 EXPNO 463
 PROCNO 1

F2 - Acquisition Parameters
 Date 20110923
 Time 11.22
 INSTRUM spect
 PROBHD 5 mm BBO BB-1H
 PULPROG zgpg
 TD 32768
 SOLVENT CDC13
 NS 413
 DS 4
 SWH 18115.941 Hz
 FIDRES 0.552855 Hz
 AQ 0.9044468 sec
 RG 144
 DW 27.600 usec
 DE 6.00 usec
 TE 292.6 K
 D1 1.00000000 sec
 d11 0.03000000 sec
 DELTA 0.89999998 sec
 TDO 1

==== CHANNEL f1 =====
 NUC1 13C
 P1 9.20 usec
 PL1 -1.00 dB
 SFO1 75.4760505 MHz

==== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 80.00 usec
 PL12 17.20 dB
 PL13 20.20 dB
 PL2 0.00 dB
 SFO2 300.1315007 MHz

F2 - Processing parameters
 SI 131072
 SF 75.4677528 MHz
 WDW EM
 SSB 0
 LB 3.00 Hz
 GB 0
 PC 1.40



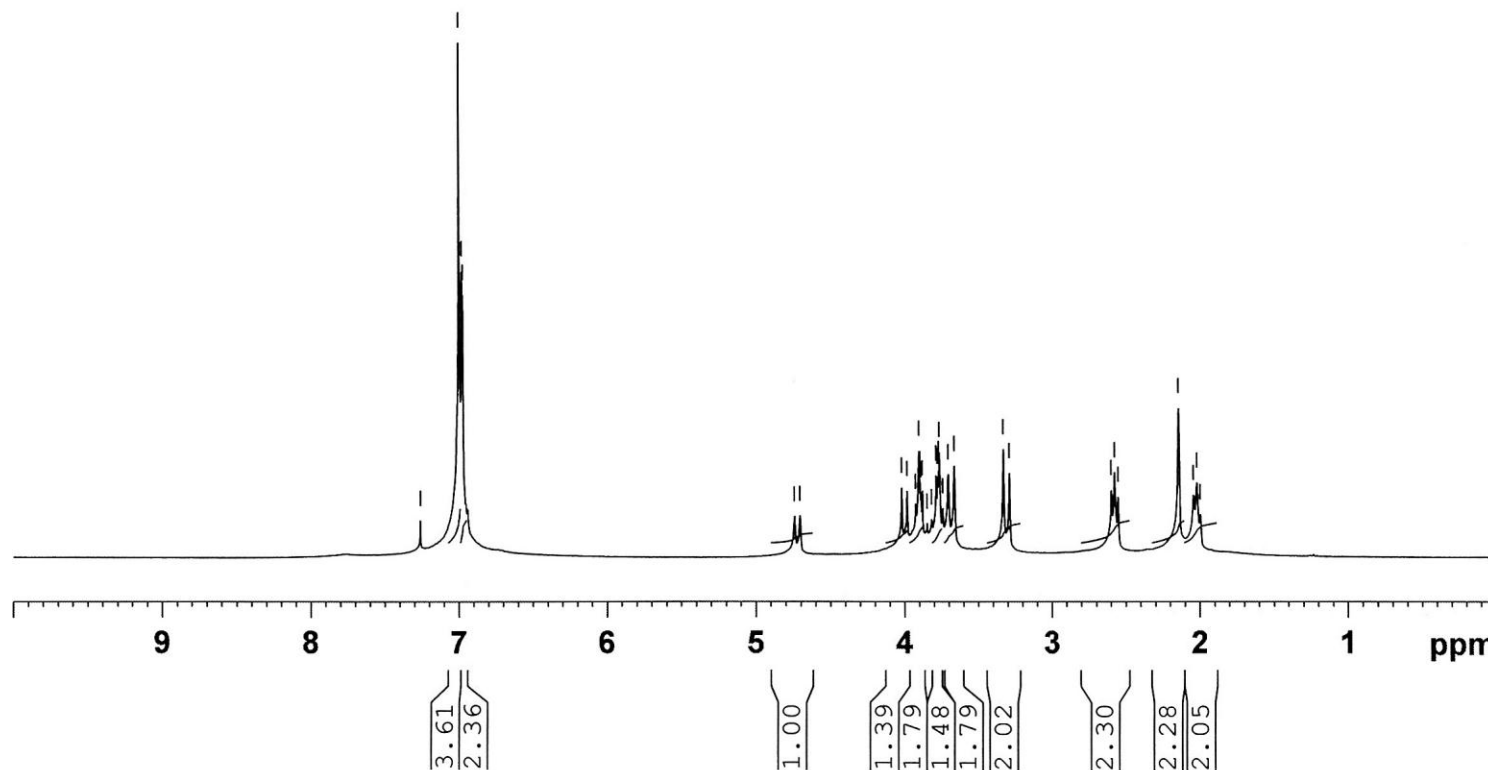
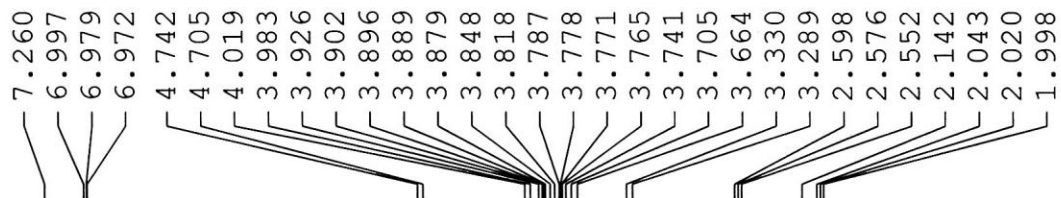
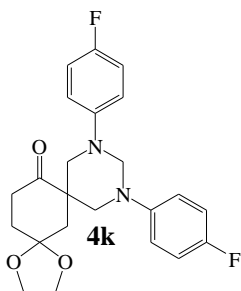


Current Data Parameters
NAME proton
EXPNO 202
PROCNO 1

F2 - Acquisition Parameters
Date_ 20120814
Time_ 14.51
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 32768
SOLVENT CDC13
NS 20
DS 0
SWH 4807.692 Hz
FIDRES 0.146719 Hz
AQ 3.4079220 sec
RG 80.6
DW 104.000 usec
DE 6.00 usec
TE 291.8 K
D1 1.0000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 11.00 usec
PL1 0.00 dB
SFO1 300.1320008 MHz

F2 - Processing parameters
SI 16384
SF 300.1300022 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00





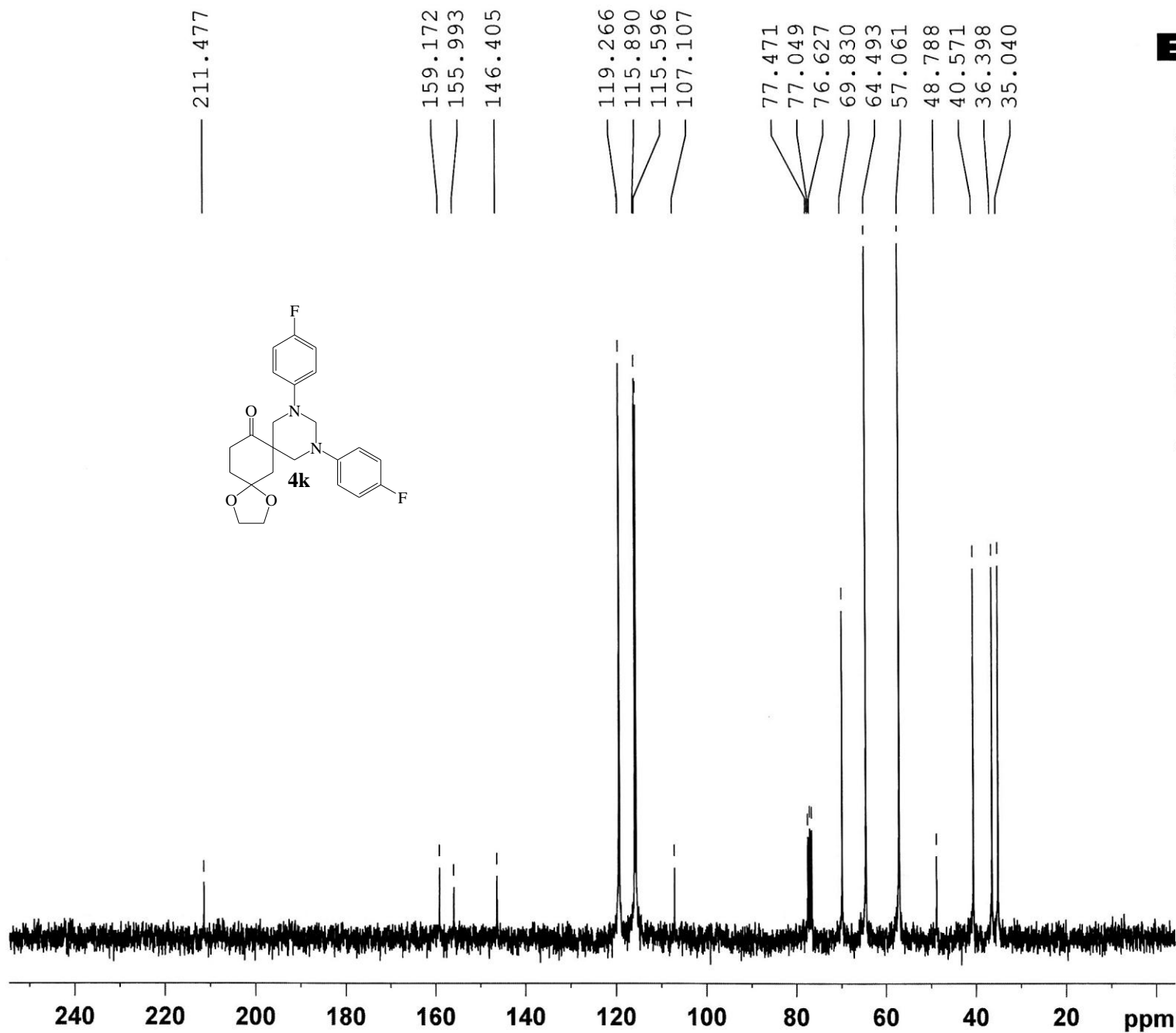
Current Data Parameters
NAME carbon
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20120814
Time_ 14.56
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zgpg
TD 32768
SOLVENT CDCl3
NS 416
DS 4
SWH 19531.250 Hz
FIDRES 0.596046 Hz
AQ 0.8389108 sec
RG 71.8
DW 25.600 usec
DE 6.00 usec
TE 292.3 K
D1 1.00000000 sec
d11 0.03000000 sec
DELTA 0.89999998 sec
TDO 1

===== CHANNEL f1 =====
NUC1 13C
P1 9.20 usec
PL1 -1.00 dB
SFO1 75.4772005 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL12 17.20 dB
PL13 20.20 dB
PL2 0.00 dB
SFO2 300.1315007 MHz

F2 - Processing parameters
SI 131072
SF 75.4677490 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.40





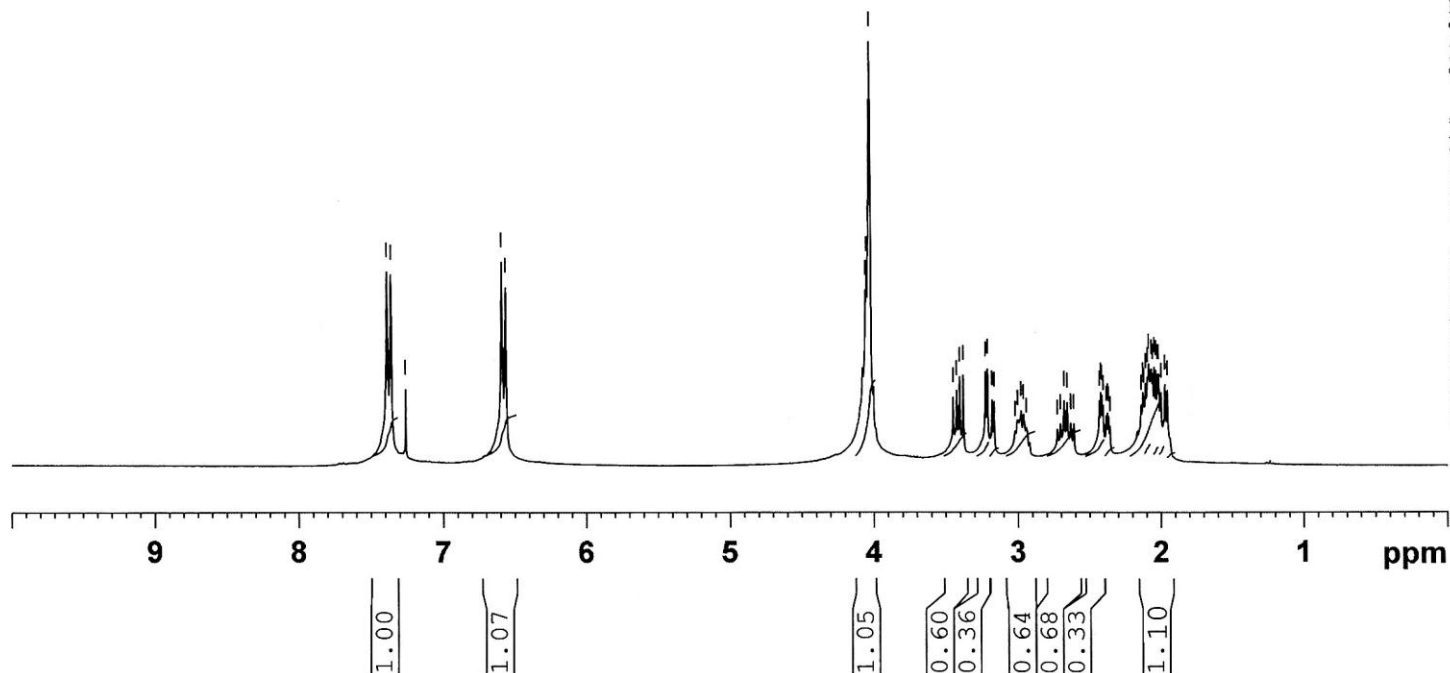
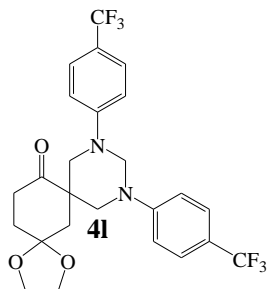
Current Data Parameters
NAME proton
EXPNO 203
PROCNO 1

F2 - Acquisition Parameters
Date_ 20120814
Time_ 15.11
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 33
DS 0
SWH 4807.692 Hz
FIDRES 0.146719 Hz
AQ 3.4079220 sec
RG 80.6
DW 104.000 usec
DE 6.00 usec
TE 292.0 K
D1 1.0000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 11.00 usec
PL1 0.00 dB
SFO1 300.1320008 MHz

F2 - Processing parameters
SI 16384
SF 300.1300022 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

7.391
7.362
7.260
6.592
6.563
4.056
4.049
4.039
4.029
4.022
3.449
3.425
3.403
3.379
3.224
3.210
3.178
3.164
2.978
2.677
2.655
2.431
2.421
2.414
2.404
2.373
2.138
2.126
2.118
2.107
2.094
2.083
2.066
2.057
2.046
2.035
2.024
2.015





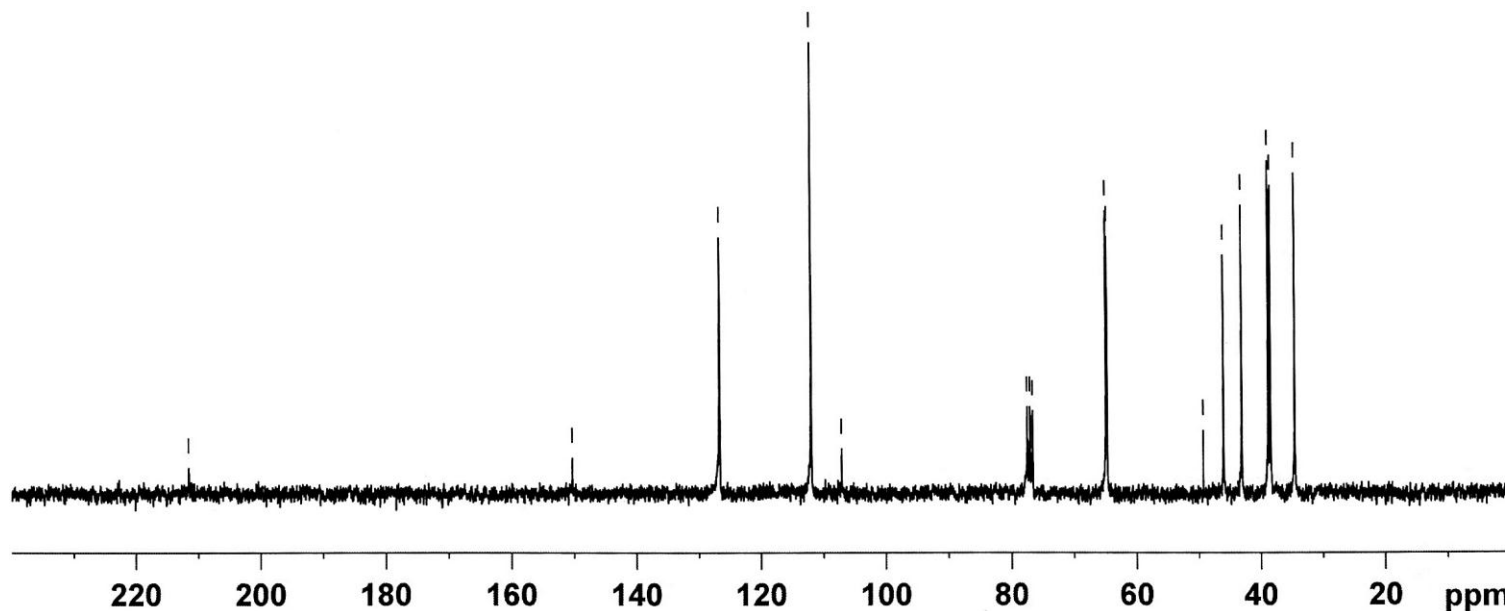
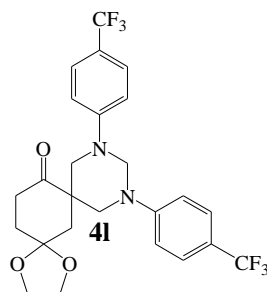
211.578

150.294

126.686
126.639

111.968
107.097

77.471
77.042
76.619
64.889
64.707
48.948
46.002
43.060
38.732
38.362
34.461



Current Data Parameters
NAME carbon
EXPNO 11
PROCNO 1

F2 - Acquisition Parameters

Date 20120814
Time 15.14
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zgpg
TD 32768
SOLVENT CDC13
NS 471
DS 4
SWH 19531.250 Hz
FIDRES 0.596046 Hz
AQ 0.8389108 sec
RG 71.8
DW 25.600 usec
DE 6.00 usec
TE 292.0 K
D1 1.00000000 sec
d11 0.03000000 sec
DELTA 0.89999998 sec
TDO 1

==== CHANNEL f1 =====
NUC1 13C
P1 9.20 usec
PL1 -1.00 dB
SFO1 75.4772005 MHz

==== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL12 17.20 dB
PL13 20.20 dB
PL2 0.00 dB
SFO2 300.1315007 MHz

F2 - Processing parameters
SI 131072
SF 75.4677490 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.40

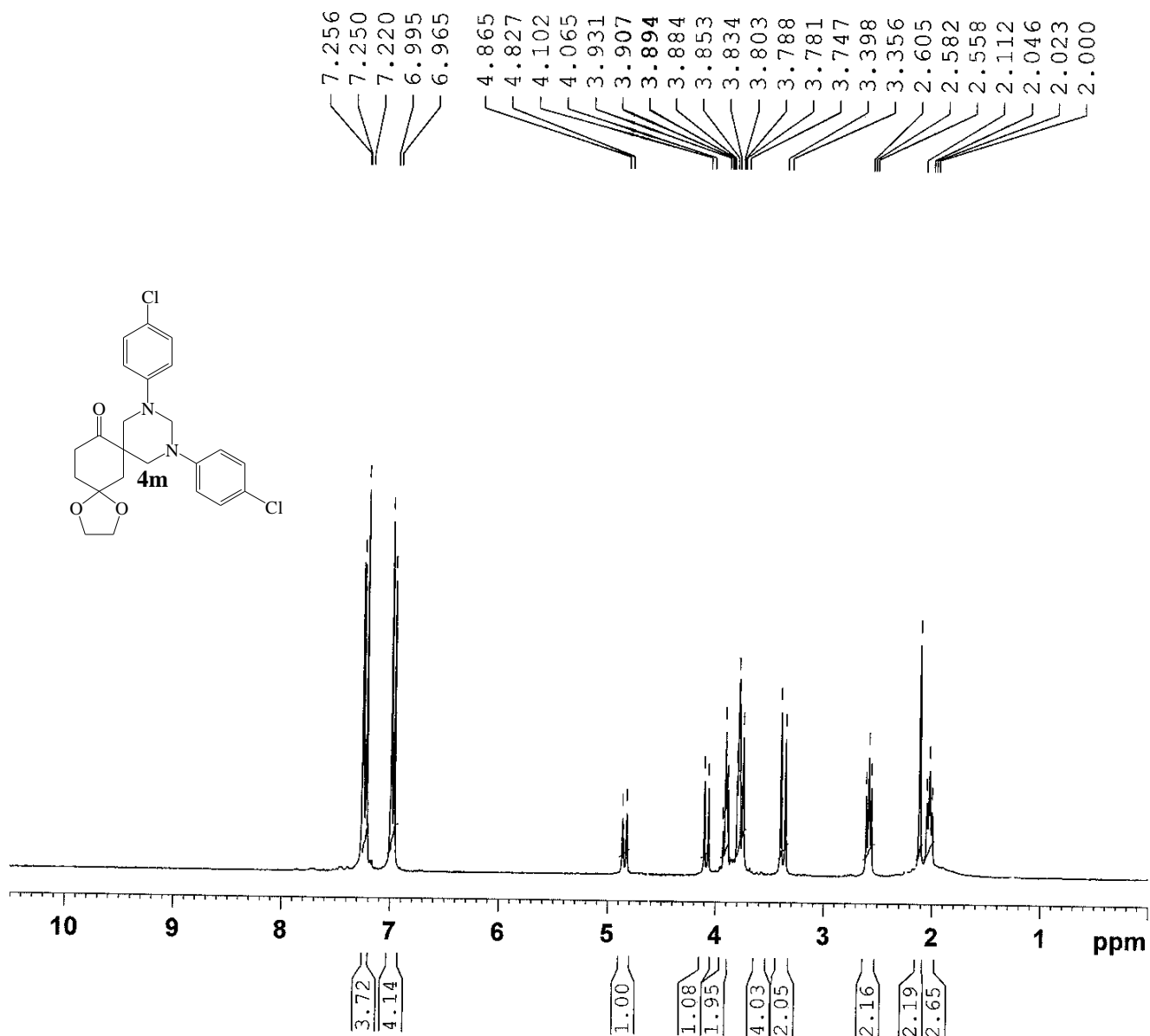


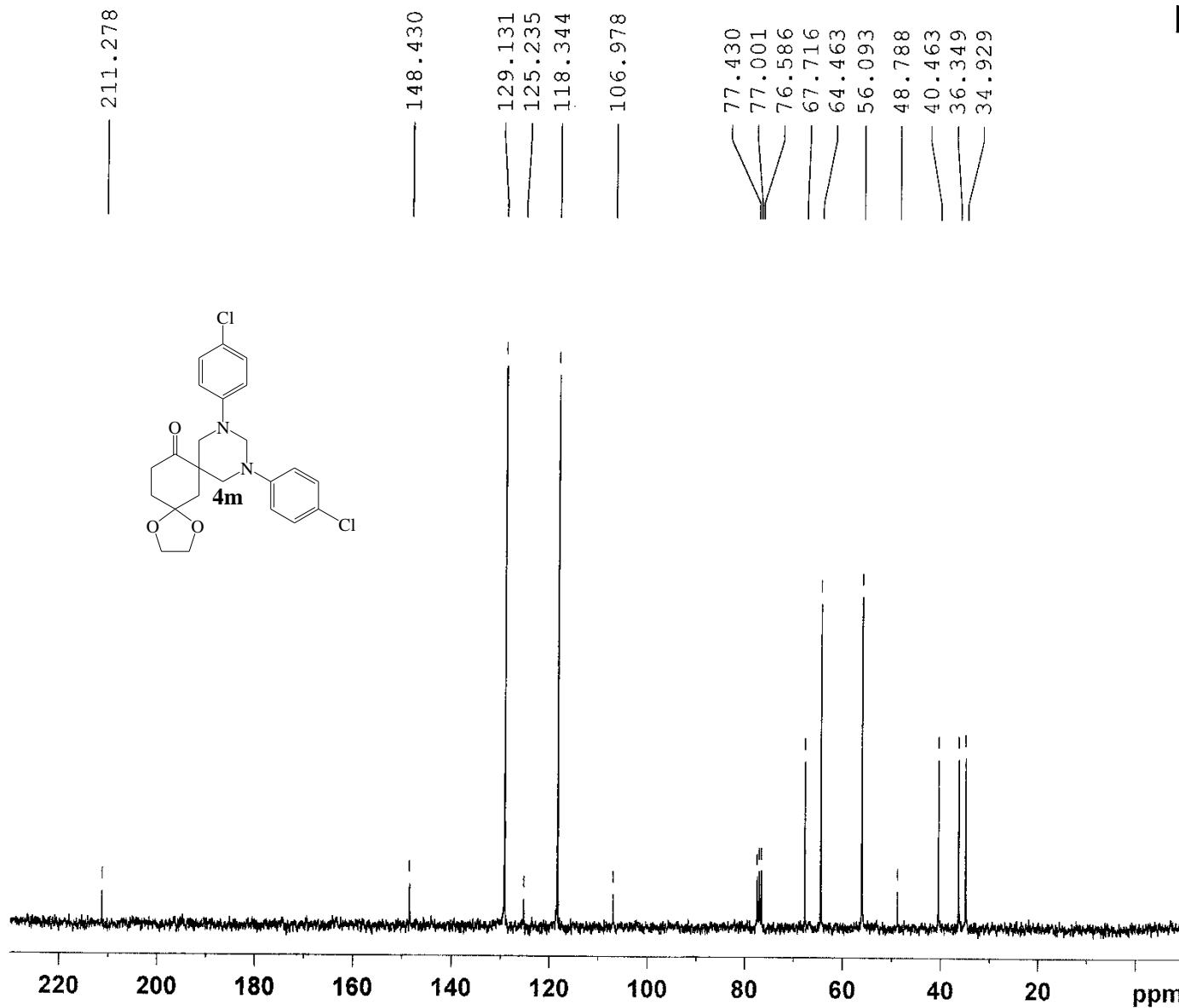
Current Data Parameters
NAME prasadh
EXPNO 1941
PROCNO 1

F2 - Acquisition Parameters
Date_ 20110808
Time_ 15.21
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 32
DS 0
SWH 4807.692 Hz
FIDRES 0.146719 Hz
AQ 3.4079220 sec
RG 406
DW 104.000 usec
DE 6.00 usec
TE 293.0 K
D1 1.00000000 sec
TD0 1

==== CHANNEL f1 =====
NUC1 1H
P1 11.00 usec
PL1 0.00 dB
SFO1 300.132008 MHz

F2 - Processing parameters
SI 16384
SF 300.1300051 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00





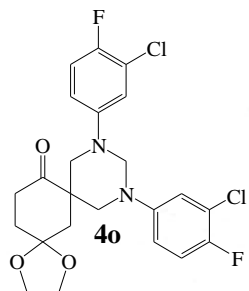
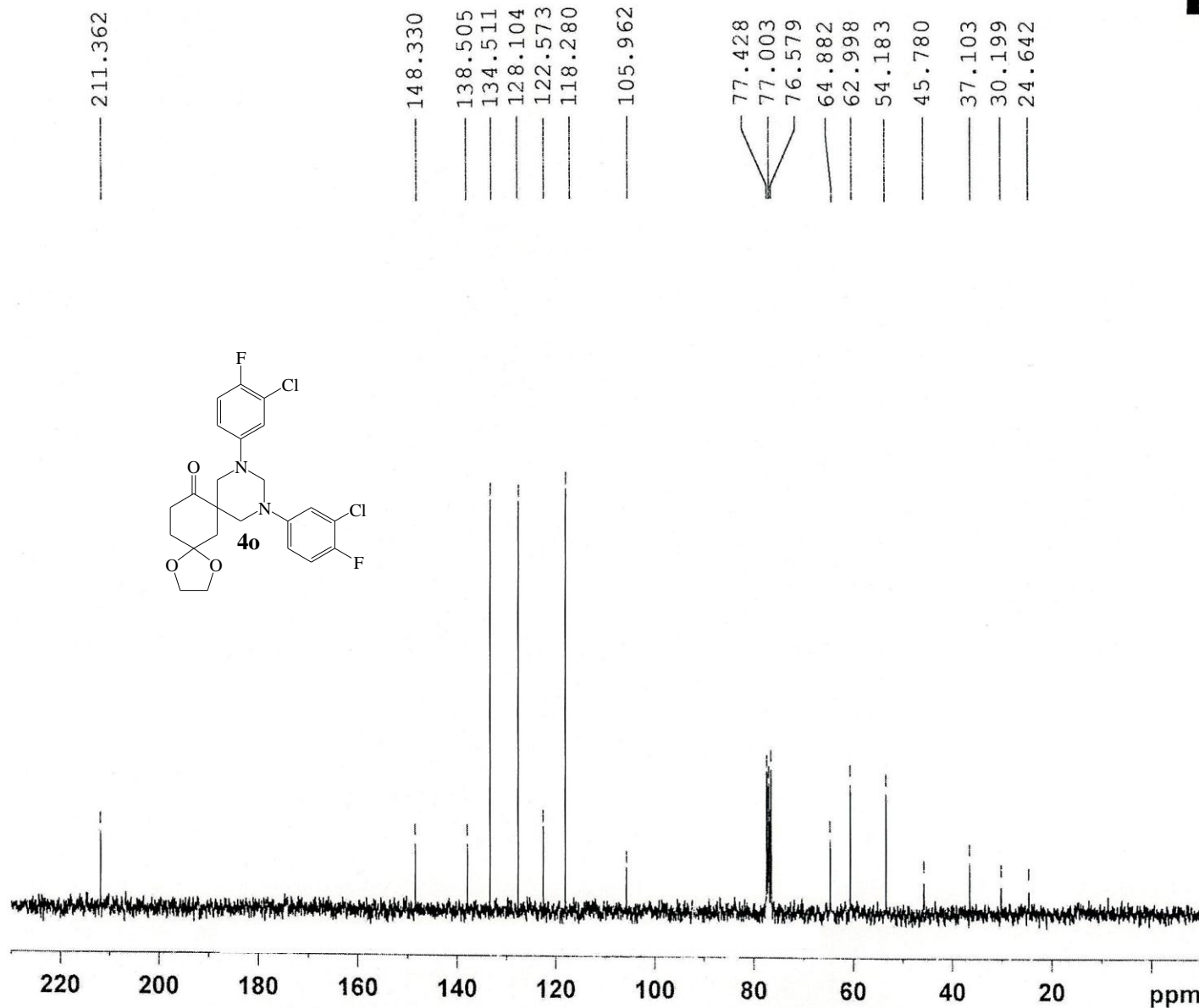
Current Data Parameters
NAME prasad C
EXPNO 464
PROCNO 1

F2 - Acquisition Parameters
Date_ 20110923
Time_ 11.39
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zgpg
TD 32768
SOLVENT CDCl3
NS 167
DS 4
SWH 18115.941 Hz
FIDRES 0.552855 Hz
AQ 0.9044468 sec
RG 144
DW 27.600 usec
DE 6.00 usec
TE 293.0 K
D1 1.0000000 sec
d11 0.0300000 sec
DELTA 0.89999998 sec
TDO 1

==== CHANNEL f1 =====
NUC1 13C
P1 9.20 usec
PL1 -1.00 dB
SFO1 75.4760505 MHz

==== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL12 17.20 dB
PL13 20.20 dB
PL2 0.00 dB
SFO2 300.1315007 MHz

F2 - Processing parameters
SI 131072
SF 75.4677532 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40



Current Data Parameters
NAME prasad C
EXPNO 467
PROCNO 1

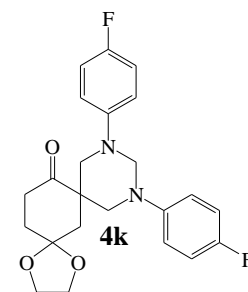
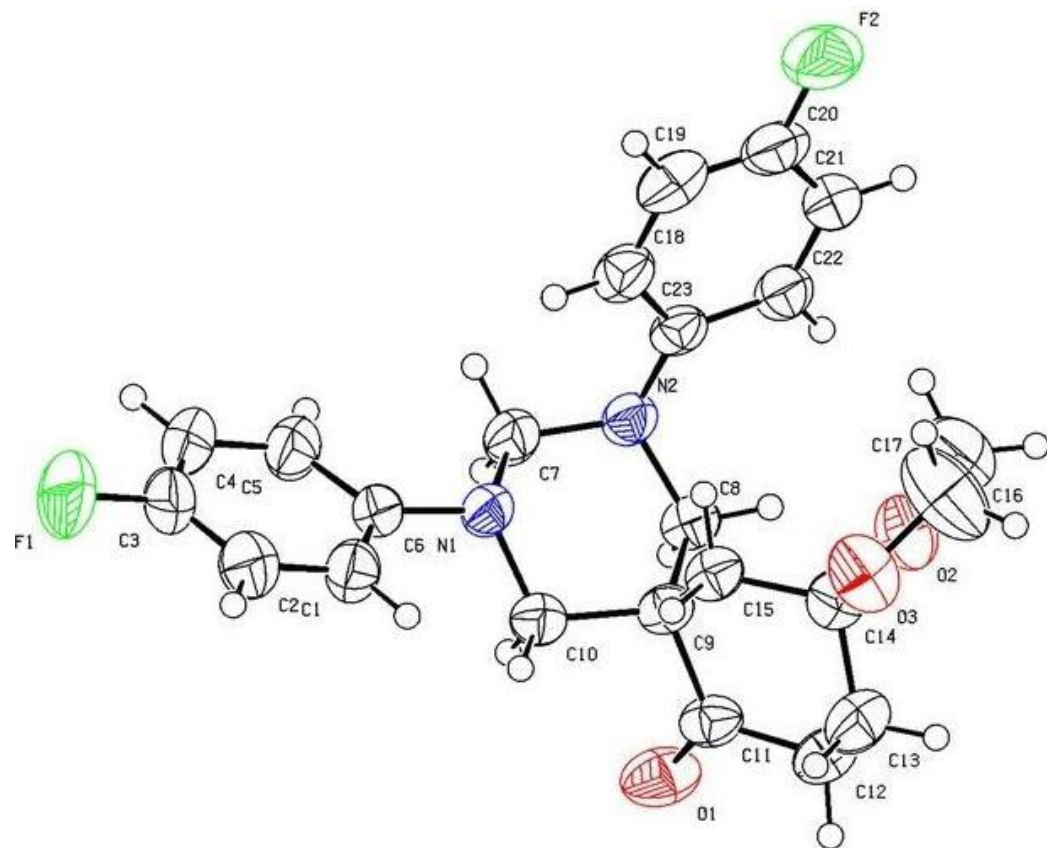
F2 - Acquisition Parameters

Date 20110923
Time 13.10
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zgpg
TD 32768
SOLVENT CDCl3
NS 512
DS 4
SWH 18115.941 Hz
FIDRES 0.552855 Hz
AQ 0.9044468 sec
RG 144
DW 27.600 usec
DE 6.00 usec
TE 293.0 K
D1 1.00000000 sec
d11 0.03000000 sec
DELTA 0.89999998 sec
TD0 1

==== CHANNEL f1 =====
NUC1 13C
P1 9.20 usec
PL1 -1.00 dB
SFO1 75.4760505 MHz

==== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL12 17.20 dB
PL13 20.20 dB
PL2 0.00 dB
SFO2 300.1315007 MHz

F2 - Processing parameters
SI 131072
SF 75.4677506 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40



(CCDC 897553)