Supporting Information

One Pot Preparation of Trifluoromethylated Homoallylic *N*-Acylhydrazines or *α*-Methylene-*γ*-lactams from acylhydrazine, trifluoroacetaldehyde methyl hemiacetal, allylic bromide and tin

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1. General methods

Flash chromatography was performed using silica gel 60 (230-400 mesh). Analytical thin layer chromatography (TLC) was done using Qingdao silica Gel (silica gel GF254). TLC plates were analyzed by an exposure to ultraviolet (UV) light and/or submersion in phosphomolybdic acid solution or in I_2. High-resolution mass spectra were recorded on a Bruker APEX II Fourier transform ion cyclotron resonance mass spectrometer. ^{1}H NMR and ^{13}C NMR spectra were recorded on Varian Mercury 400 plus or Agilent DD_2 600 instrument in $CDCl_3$ solution using TMS as an internal standard at room temperature. The following abbreviations are used for the multiplicities: s = singlet, d = doublet, dd = doublet of doublet, t = triplet, q = quadruplet, m = multiplet, br = broad signal for proton spectra; coupling constants (*J*) are reported in Hertz (Hz). Melting points were determined on a Beijing Taike X-4 apparatus and were uncorrected. The solvents were distilled by standard methods. Reagents were obtained from commercial suppliers and used without further purification unless otherwise noted.

2. Syntheses of Compounds 4 and 6

2.1 General procedures for synthesis of trifluoromethylated homoallylic *N*-acylhydrazines 4

A solution of acylhydrazine **1** (0.36 mmol), trifluoroacetaldehyde methyl hemiacetal **2** (0.54 mmol), allylic bromide **3** (0.72 mmol) and tin powder (0.9 mmol) in THF (5 mL) was stirred at reflux for 12-18 h (monitored by TLC). After completion, the reaction mixture was cooled to room temperature, saturated NH_4Cl solution (10 mL) was added into the mixture and stirred for 10 min, then the mixture was extracted with CH_2Cl_2 (3×10 mL). The combined organic extracts were dried over anhydrous $MgSO_4$ and concentrated in vacuum. Purification of the residue by silica gel column chromatography using petroleum ether : acetone (6:1-4:1) as eluent furnished the products **4**.

***N′*-(1,1,1-Trifluoropent-4-en-2-yl)benzohydrazide (4a).** White solid; 79.1 mg, 85% yield; mp 96-97 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.74-7.72 (m, 3H), 7.55 (s, 1H), 7.46 (d, J = 6.8 Hz, 2H), 6.02-6.00 (m, 1H), 5.33 (d, J = 14.8 Hz, 2H), 4.94 (s, 1H), 3.50 (s, 1H), 2.64-2.61 (m, 1H), 2.39-2.31 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 167.3, 132.1, 132.0, 131.91, 128.63, 126.79, 125.9 (q, $J_{C\text{-}F}$ = 279.4 Hz),119.9, 60.5 (q, $J_{C\text{-}F}$ = 26.8 Hz), 31.4 (d, $J_{C\text{-}F}$ = 2.1 Hz); ^{19}F NMR (376 MHz, $CDCl_3$) δ -75.5 (d, $J_{F\text{-}H}$ = 6.8 Hz); HRMS (ESI) m/z $C_{12}H_{14}F_3N_2O$ $[M + H]^+$ calcd. 259.1053, found: 259.1051.

4-Methoxy-*N′*-(1,1,1-trifluoropent-4-en-2-yl)benzohydrazide (4b). White solid; 85.4 mg, 82% yield; mp 99-100 °C; ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.73 (s, 1H), 7.69 (d, J = 8.4 Hz, 2H), 6.91 (d, J = 9.0 Hz, 2H), 6.02-5.95 (m, 1H), 5.31-5.28 (m, 2H), 4.92 (d, J = 6.0 Hz, 1H), 3.84 (s, 3H), 3.49-3.44 (m, 1H), 2.61-2.58 (m, 1H), 2.36-2.30 (m,1H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 167.0, 162.7, 132.1, 128.7, 126.1 (q, $J_{C\text{-}F}$ = 279.6 Hz), 124.4, 119.9, 113.9, 60.7 (q, $J_{C\text{-}F}$ = 26.7 Hz), 55.4, 31.5 (d, $J_{C\text{-}F}$ = 2.1 Hz); ^{19}F NMR (376 MHz, $CDCl_3$) δ -75.5 (d, $J_{F\text{-}H}$ = 6.8 Hz); HRMS (ESI) m/z $C_{13}H_{16}F_3N_2O_2$ $[M + H]^+$ calcd 289.1158, found: 289.1157.

2-Methyl-*N′*-(1,1,1-trifluoropent-4-en-2-yl)benzohydrazide (4c). White solid; 42.1 mg, 43% yield; mp 79-80 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.42 (s, 1H), 7.36-7.31 (m, 2H), 7.23-7.18 (m, 2H), 6.06-5.95 (m, 1H), 5.35-5.30 (m, 2H), 4.91 (d, J = 6.4 Hz, 1H), 3.57-3.48 (m, 1H), 2.65-2.59 (m, 1H), 2.43 (s, 3H), 2.39-2.31 (m, 1H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 169.4, 136.8, 133.3, 131.9, 131.2, 130.6, 127.0, 126.0 (q, $J_{C\text{-}F}$ = 279.6 Hz), 125.8, 120.1, 60.6 (q, J = 26.7 Hz), 31.5 (d, $J_{C\text{-}F}$ = 2.1 Hz), 19.6; ^{19}F NMR (376 MHz, $CDCl_3$) δ -75.3 (d, $J_{F\text{-}H}$ = 6.8 Hz,); HRMS (ESI) m/z $C_{13}H_{16}F_3N_2O$ $[M + H]^+$ calcd 273.1209, found: 273.1216.

3-Methyl-*N′*-(1,1,1-trifluoropent-4-en-2-yl)benzohydrazide (4d). White solid; 71.5 mg, 73% yield; mp 75-77 °C; ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.82 (d, J = 6.0 Hz, 1H), 7.54 (s, 1H), 7.49 (d, J = 7.2 Hz, 1H), 7.33 (d, J = 7.2 Hz, 1H), 7.32-7.30 (m, 1H), 6.02-5.95 (m, 1H), 5.32-5.28 (m, 2H), 4.92 (d, J = 6.0 Hz, 1H), 3.51-3.45 (m, 1H), 2.60 (d, J = 14.4 Hz, 1H), 2.38 (s, 3H), 2.36-2.31 (m,1H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 167.6, 138.6, 132.9, 132.1, 132.1, 128.6, 127.6, 126.1 (q, $J_{C\text{-}F}$ = 279.6 Hz), 123.8, 119.9, 60.6 (q, $J_{C\text{-}F}$ = 26.7 Hz), 31.5 (d, $J_{C\text{-}F}$ = 2.1 Hz),

21.3; ^{19}F NMR (376 MHz, $CDCl_3$) δ -75.5 (d, J_{F-H} = 6.8 Hz); HRMS (ESI) m/z $C_{13}H_{16}F_3N_2O$ $[M + H]^+$ calcd 273.1209, found: 273.1215.

4-Methyl-*N′*-(1,1,1-trifluoropent-4-en-2-yl)benzohydrazide (4e). White solid; 81.5 mg, 84% yield; mp 100-101 °C; ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.81 (br, 1H), 7.62 (d, *J* = 7.8 Hz, 2H), 7.23 (d, *J* = 7.8 Hz, 2H), 6.02-5.95 (m, 1H), 5.31-5.28 (m, 2H), 4.92 (d, *J* = 5.4 Hz, 1H), 3.47 (s, 1H), 2.59 (d, *J* = 15.0 Hz, 1H), 2.39 (s, 3H), 2.36-2.30 (m, 1H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 167.4, 142.8, 132.0, 129.4, 129.3, 126.9, 126.0 (q, J_{C-F} = 279.4 Hz), 119.9, 60.6 (q, J_{C-F} = 26.7 Hz), 31.5 (d, J_{C-F} = 2.1 Hz), 21.5; ^{19}F NMR (376 MHz, $CDCl_3$) δ -75.5 (d, J_{F-H} = 6.8 Hz); HRMS (ESI) m/z $C_{13}H_{16}F_3N_2O$ $[M + H]^+$ calcd 273.1209, found: 273.1214.

4-Fluoro-*N′*-(1,1,1-trifluoropent-4-en-2-yl)benzohydrazide (4f). White solid; 80.0 mg, 81% yield; mp 81-82 °C; ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.75 (s, 1H), 7.74-7.73 (m, 2H), 7.12 (t, *J* = 8.4 Hz, 2H), 6.01-5.96 (m, 1H), 5.32-5.30 (m, 2H), 4.91 (d, *J* = 6.0 Hz, 1H), 3.50-3.45 (m, 1H), 2.61 (d, *J* = 14.4 Hz, 1H), 2.36-2.30 (m, 1H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 166.4, 165.1 (d, J_{C-F} = 251.9 Hz), 131.9, 129.3 (d, J_{C-F} = 9.0 Hz), 128.3 (d, J_{C-F} = 3.2 Hz), 126.0 (q, J_{C-F} = 279.6 Hz), 120.1, 115.9 (d, J_{C-F} = 22.0 Hz), 60.6 (q, J_{C-F} = 26.5 Hz), 31.5 (d, J_{C-F} = 2.1 Hz); ^{19}F NMR (376 MHz, $CDCl_3$) δ -75.5 (d, J_{F-H} = 6.8 Hz), -107.1 (m); HRMS (ESI) m/z $C_{12}H_{12}NaF_4N_2O$ $[M + Na]^+$ calcd 299.0778, found: 299.0783.

4-Chloro-*N′*-(1,1,1-trifluoropent-4-en-2-yl)benzohydrazide (4g). White solid; 90.1 mg, 86% yield; mp 114-115 °C; ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.81 (s, 1H), 7.67 (d, *J* = 12.0 Hz, 2H), 7.42 (d, *J* = 12.0 Hz, 2H), 6.01-5.95 (m, 1H), 5.33-5.30 (m, 2H), 4.92 (d, *J* = 6.0 Hz, 1H), 3.51-3.46 (m, 1H), 2.63-2.59 (m, 1H), 2.36-2.31 (m, 1H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 166.4, 138.6, 131.9, 130.5, 129.1, 128.3, 126.0 (q, J_{C-F} = 279.5 Hz), 120.1, 60.6 (q, J_{C-F} = 26.5 Hz), 31.5 (d, J_{C-F} = 2.1 Hz);^{19}F NMR (376 MHz, $CDCl_3$) δ -75.5 (d, J_{F-H} = 6.8 Hz); HRMS (ESI) m/z $C_{12}H_{13}ClF_3N_2O$ $[M + H]^+$ calcd 293.0663, found: 293.0662.

3-Bromo-*N′*-(1,1,1-trifluoropent-4-en-2-yl)benzohydrazide (4h). White solid; 90.1 mg, 75% yield; mp 80-82 °C; ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.88-7.85 (m, 2H), 7.67-7.63 (m, 2H), 7.33-7.31 (m, 1H), 6.01-5.95 (m, 1H), 5.33-5.30 (m, 2H), 4.91 (d, *J* = 6.0 Hz, 1H), 3.51-3.46 (m, 1H), 2.64-2.60 (m, 1H), 2.37-2.31 (m, 1H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 166.0, 135.2, 134.1, 131.9, 130.3, 130.2, 126.0 (q, J_{C-F} = 279.6 Hz), 125.4, 122.9, 120.2, 60.6 (q, J_{C-F} = 27.0 Hz), 31.5 (d, J_{C-F} = 2.1 Hz); ^{19}F NMR (376 MHz, $CDCl_3$) δ -75.4 (d, J_{F-H} = 6.8 Hz); HRMS (ESI) m/z $C_{12}H_{13}BrF_3N_2O$ $[M + H]^+$ calcd 337.0158, found: 337.0159.

4-(Trifluoromethyl)-*N′*-(1,1,1-trifluoropent-4-en-2-yl)benzohydrazide (4i). White solid; 75.8 mg, 65% yield; mp 87-89 °C; ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.95 (s, 1H), 7.83 (d, *J* = 7.8 Hz, 2H), 7.70 (d, *J* = 8.4 Hz, 2H), 6.01-5.94 (m, 1H), 5.33-5.29 (m, 2H), 4.93 (d, *J* = 6.0 Hz, 1H), 3.53-3.47 (m, 1H), 2.63-2.60 (m, 1H), 2.36-2.28 (m, 1H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 166.2, 135.4, 133.9 (q, J_{C-F} = 32.5 Hz), 131.8, 127.4, 126.0 (q, J_{C-F} = 279.6 Hz), 125.8 (d, J_{C-F} = 3.5 Hz), 123.4 (q, J_{C-F} = 271.2 Hz), 120.3, 60.5 (q, J_{C-F} = 27.1 Hz), 31.4 (d, J_{C-F} = 2.1 Hz); ^{19}F NMR (376 MHz, $CDCl_3$) δ -63.6 (d, J_{F-H} = 5.3 Hz), -75.5 (d, J_{F-H} = 6.8 Hz); HRMS (ESI) m/z $C_{13}H_{13}F_6N_2O$ $[M + H]^+$ calcd 327.0927, found: 327.0926.

***N′*-(1,1,1-Trifluoropent-4-en-2-yl)furan-2-carbohydrazide (4j)**. White solid; 76.1 mg, 86% yield; mp 65-67 °C; ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.92 (s, 1H), 7.46 (s, 1H), 7.15 (d, *J* = 3.6 Hz, 1H), 6.52 (t, *J* = 1.8 Hz, 1H), 6.02-5.96 (m, 1H), 5.33-5.29 (m, 2H), 4.82 (d, *J* = 6.0 Hz, 1H), 3.49-3.47 (m, 1H), 2.62-2.59 (m, 1H), 2.36-2.31 (m, 1H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 158.1, 146.2, 144.6, 132.0, 126.0 (q, J_{C-F} = 279.6 Hz), 120.1, 115.3, 112.1, 60.8 (q, J_{C-F} = 27.0 Hz), 31.4 (d, J_{C-F} = 2.1 Hz); ^{19}F NMR (376 MHz, $CDCl_3$) δ -75.7 (d, J_{F-H} = 6.8 Hz); HRMS (ESI) m/z $C_{10}H_{12}F_3N_2O_2$ $[M + H]^+$ calcd 249.0845, found: 249.0842.

***N′*-(1,1,1-Trifluoro-3,3-dimethylpent-4-en-2-yl)benzohydrazide (4k)**. Colourless oil; 61.8 mg, 60% yield; ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.71-7.70 (m, 2H), 7.69 (d, *J* = 1.2 Hz, 1H), 7.52-7.50 (m, 1H), 7.43-7.40 (m, 2H), 6.08 (dd, *J* = 17.4, 10.8 Hz, 1H), 5.22-5.18 (m, 2H), 5.00 (d, *J* = 6.0 Hz, 1H), 3.21 (q, *J* = 8.4 Hz, 1H), 1.31 (s, 3H), 1.24 (s, 3H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 167.0, 143.7, 132.3, 132.1, 128.7, 126.8, 126.7 (q, J_{C-F} = 282.4 Hz), 114.0, 68.3 (q, J_{C-F} = 24.2 Hz), 39.0, 25.8, 22.4; ^{19}F NMR (376 MHz, $CDCl_3$) δ -66.9 (d, J_{F-H} = 7.8 Hz); HRMS (ESI) m/z $C_{14}H_{18}F_3N_2O$ $[M + H]^+$ calcd 287.1366, found: 287.1363.

4-Fluoro-*N′*-(1,1,1-trifluoro-3,3-dimethylpent-4-en-2-yl)benzohydrazide (4l). White solid; 73.8 mg, 68% yield; mp 85-86 °C; ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.74-7.73(m, 2H), 7.72 (s, 1H), 7.13-7.10 (m, 2H), 6.08 (dd, *J* = 17.4, 10.8 Hz, 1H), 5.23-5.19 (m, 2H), 5.00 (d, *J* = 6.0 Hz, 1H), 3.22 (q, *J* = 7.8 Hz, 1H), 1.32 (s, 3H), 1.25 (s, 3H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 166.0, 165.1 (d, J_{C-F} = 236.7 Hz), 143.6, 129.2 (d, J_{C-F} = 9.0 Hz), 128.5 (d, J_{C-F} = 3.0 Hz), 126.7 (q, J_{C-F} = 282.6 Hz), 115.9 (d, J_{C-F} = 21.9 Hz), 114.0, 68.3 (q, J_{C-F} = 24.4 Hz), 39.0, 25.8, 22.4; ^{19}F NMR (376 MHz, $CDCl_3$) δ -66.9 (d, J_{F-H} = 7.8 Hz), -107.2 (m); HRMS (ESI) m/z $C_{14}H_{16}NaF_4N_2O$ $[M + Na]^+$ calcd 327.1091, found: 327.1096.

4-Chloro-*N′*-(1,1,1-trifluoro-3,3-dimethylpent-4-en-2-yl)benzohydrazide (4m). White solid; 86.1 mg, 75% yield; mp 103-105 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.74 (d, *J* = 6.0 Hz, 1H), 7.65 (d, *J* = 8.4 Hz, 2H), 7.41 (d, *J* = 8.0 Hz, 2H), 6.07 (dd, *J* =17.2, 10.8 Hz, 1H), 5.24-5.19 (m, 2H), 4.99 (d, *J* = 6.0 Hz, 1H), 3.21 (q, *J* = 8.0 Hz, 1H), 1.32 (s, 3H), 1.25 (s, 3H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 166.0, 143.6, 138.4, 130.7, 129.0, 128.3, 126.7 (q, J_{C-F} = 282.6 Hz), 114.1, 68.2 (q, J_{C-F} = 24.3 Hz), 39.0, 25.8, 22.3; ^{19}F NMR (376 MHz, $CDCl_3$) δ -66.9 (d, J_{F-H} = 7.8 Hz); HRMS (ESI) m/z $C_{14}H_{17}ClF_3N_2O$ $[M + H]^+$ calcd 321.0976, found: 321.0975.

3-Bromo-*N′*-(1,1,1-trifluoro-3,3-dimethylpent-4-en-2-yl)benzohydrazide (4n). White solid; 109.8 mg, 84% yield; mp 70-72 °C; ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.85 (s, 1H), 7.72 (d, *J* = 6.0 Hz, 1H), 7.64 (d, *J* = 7.8 Hz, 1H), 7.60 (d, *J* = 7.8 Hz, 1H), 7.30 (t, *J* = 7.8 Hz, 1H), 6.07 (dd, *J* = 17.4, 10.8 Hz, 1H), 5.23-5.19 (m, 2H), 4.98 (d, *J* = 6.0 Hz, 1H), 3.20 (q, *J* = 8.4 Hz, 1H), 1.31 (s, 3H), 1.24 (s, 3H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 165.6, 143.6, 135.0, 134.3, 130.3, 130.2, 126.6 (q, J_{C-F} = 282.6 Hz), 125.4, 122.9, 114.1, 68.2 (q, J_{C-F} = 24.3 Hz), 39.0, 25.8, 22.4; ^{19}F NMR (376 MHz, $CDCl_3$) δ -66.9 (d, J_{F-H} = 7.6 Hz); HRMS (ESI) m/z $C_{14}H_{17}BrF_3N_2O$ $[M + H]^+$ calcd 365.0471, found: 365.0470.

4-Methyl-*N′*-(1,1,1-trifluoro-3,3-dimethylpent-4-en-2-yl)benzohydrazide (4o). White solid; 83.1 mg, 77% yield; mp 129-130 °C; ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.64 (d, *J* = 6.0 Hz, 1H), 7.61 (d, *J* = 8.4 Hz, 2H), 7.23 (d, *J* = 8.4 Hz, 2H), 6.10 (dd, *J* = 17.4, 10.8 Hz, 1H), 5.24-5.20 (m, 2H), 5.01 (d, *J* = 6.0 Hz, 1H), 3.21 (q, *J* = 8.4 Hz, 1H), 2.39 (s, 3H), 1.33 (s, 3H), 1.26 (s, 3H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 166.9, 143.7, 142.6, 129.5, 129.4, 126.8, 126.7 (q, J_{C-F} = 282.6 Hz), 113.9, 68.3 (q, J_{C-F} = 24.0 Hz), 39.0, 25.8 (d, J_{C-F} = 1.7 Hz), 22.5, 21.4; ^{19}F NMR (376 MHz, $CDCl_3$) δ -66.9 (d, J_{F-H} = 7.8 Hz); HRMS (ESI) m/z $C_{15}H_{20}F_3N_2O$ $[M + H]^+$ calcd 301.1522, found: 301.1521.

4-Methoxy-*N′*-(1,1,1-trifluoro-3,3-dimethylpent-4-en-2-yl)benzohydrazide (4p). White solid; 79.4 mg, 70% yield; mp 80-82 °C; ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.67 (d, *J* = 8.4 Hz, 2H), 7.55 (d, *J* = 6.0 Hz, 1H), 6.92 (d, *J* = 8.4 Hz, 2H), 6.09 (dd, *J* = 17.4, 10.8 Hz, 1H), 5.23-5.19 (m, 2H), 5.01 (d, *J* = 6.0 Hz, 1H), 3.84 (s, 3H), 3.20 (q, *J* = 7.8 Hz, 1H), 1.32 (s, 3H), 1.25 (s, 3H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 166.6, 162.6, 143.7, 128.7, 126.8 (q, J_{C-F} = 282.7 Hz), 124.5, 114.0, 113.9, 68.4 (q, J_{C-F} = 24.0 Hz), 55.4, 39.0, 25.8, 22.5; ^{19}F NMR (376 MHz, $CDCl_3$) δ -66.9 (d, J_{F-H} = 7.9 Hz); HRMS (ESI) m/z $C_{15}H_{20}F_3N_2O_2$ $[M + H]^+$ calcd 317.1471, found: 317.1479.

***N′*-(1,1,1-Trifluoro-3,3-dimethylpent-4-en-2-yl)furan-2-carbohydrazide (4q)**. Colourless oil; 50.3 mg, 51% yield; mp 99-100 °C; ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.81 (d, *J* = 5.4 Hz, 1H), 7.45 (s, 1H), 7.26 (s, 1H), 7.13 (d, *J* = 1.8 Hz, 1H), 6.50 (d, *J* = 1.2 Hz, 1H), 6.08 (dd, *J* = 17.4, 10.8 Hz, 1H), 5.23-5.19 (m, 1H), 4.87 (d, *J* = 6.6 Hz, 1H), 3.19 (q, *J* = 7.8 Hz, 1H), 1.32 (s, 1H), 1.24 (s, 1H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 157.7, 146.3, 144.5, 143.6, 126.7 (q, J_{C-F} = 282.6 Hz), 115.1, 114.1, 112.1, 68.4 (q, J_{C-F} = 24.4 Hz), 39.0, 25.8, 22.4; ^{19}F NMR (376 MHz, $CDCl_3$) δ -71.1 (d, J_{F-H} = 7.9 Hz); HRMS (ESI) m/z $C_{12}H_{15}NaF_3N_2O_2$ $[M + Na]^+$ calcd 299.0978, found: 299.0993.

2.2 General procedures for synthesis of trifluoromethylated *α*-methylene-*γ*-lactams 6

A solution of acylhydrazine **1** (0.36 mmol), trifluoroacetaldehyde methyl hemiacetal **2** (0.54 mmol), ethyl 2-(bromomethyl)acrylate **5** (1.08 mmol) and tin powder (1.26 mmol) in EtOH (5 mL) was stirred at reflux for 15-18 h (monitored by TLC). After completion, the reaction mixture was cooled to room temperature, saturated NH_4Cl solution (10 mL) was added into the mixture and stirred for 10 min, then the mixture was extracted with CH_2Cl_2 (3×10 mL). The

combined organic extracts were dried over anhydrous $MgSO_4$ and concentrated in vacuum. Purification of the residue by silica gel column chromatography using petroleum ether : acetone (5:1-4:1) as eluent furnished the products **6**.

***N*-(3-Methylene-2-oxo-5-(trifluoromethyl)pyrrolidin-1-yl)benzamide (6a)**. White solid; 86.0 mg, 84% yield; mp 177-178 °C; ^{1}H NMR (600 MHz, $CDCl_3$) δ 10.01 (s, 1H), 7.78 (d, J = 8.4 Hz, 2H), 7.42 (s, 1H), 7.29 (t, J = 7.8 Hz, 2H), 6.23 (s, 1H), 5.59 (s, 1H), 4.50-4.48 (m, 1H), 3.25-3.20 (m, 1H), 2.89 (d, J = 18.0 Hz, 1H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 168.0, 165.5, 132.9, 132.4, 130.5, 128.5, 127.5, 124.6 (q, $J_{C\text{-}F}$ = 280.0 Hz), 120.6, 56.7 (q, $J_{C\text{-}F}$ = 31.5 Hz), 24.8; ^{19}F NMR (376 MHz, $CDCl_3$) δ -76.9 (d, $J_{F\text{-}H}$ = 6.4 Hz); HRMS (ESI) m/z $C_{13}H_{12}F_3N_2O_2$ $[M + H]^+$ calcd 285.0845, found: 285.0842.

4-Fluoro-*N*-(3-methylene-2-oxo-5-(trifluoromethyl)pyrrolidin-1-yl)benzamide (6b). White solid; 71.2 mg, 66 % yield; mp 154-156 °C; ^{1}H NMR (600 MHz, $CDCl_3$) δ 10.19 (s, 1H), 7.82-7.79 (m, 2H), 6.96 (t, J = 8.4 Hz, 2H), 6.24 (s, 1H), 5.61 (s, 1H), 4.46 (s, 1H), 3.25-3.21 (m, 1H), 2.90 (d, J = 18.0 Hz, 1H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 168.3, 165.2 (d, J = 252.5 Hz), 164.2, 132.8, 130.0 (d, J = 9.2 Hz), 126.6 (d, J = 2.8 Hz), 124.5 (q, $J_{C\text{-}F}$ = 280.0 Hz), 120.8, 115.6 (d, J = 22.0 Hz), 56.8 (q, $J_{C\text{-}F}$ = 32.0 Hz), 24.8; ^{19}F NMR (376 MHz, $CDCl_3$) δ -77.0 (d, $J_{F\text{-}H}$ = 6.4 Hz), 106.5 (m); HRMS (ESI) m/z $C_{13}H_{10}NaF_4N_2O_2$ $[M + Na]^+$ calcd 325.0571, found: 325.0577.

4-Chloro-*N*-(3-methylene-2-oxo-5-(trifluoromethyl)pyrrolidin-1-yl)benzamide (6c). White solid; 79.9 mg, 70% yield; mp 160-162 °C; ^{1}H NMR (600 MHz, $CDCl_3$) δ 10.25 (s, 1H), 7.68 (d, J = 8.4 Hz, 2H), 7.23 (d, J = 8.4 Hz, 2H), 6.22 (s, 1H), 5.60 (s, 1H), 4.45-4.42 (m, 1H), 3.23-3.19 (m, 1H), 2.90-2.86 (m, 1H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 168.3, 164.3, 138.9, 132.7, 128.8, 128.7, 128.6, 124.4 (q, $J_{C\text{-}F}$ = 280.0 Hz), 120.9, 56.8 (q, $J_{C\text{-}F}$ = 32.0 Hz), 24.8; ^{19}F NMR (376 MHz, $CDCl_3$) δ -77.0 (d, $J_{F\text{-}H}$ = 6.4 Hz); HRMS (ESI) m/z $C_{13}H_{14}ClF_3N_3O_2$ $[M + NH_4]^+$ calcd 336.0721, found: 336.0723.

3-Bromo-*N*-(3-methylene-2-oxo-5-(trifluoromethyl)pyrrolidin-1-yl)benzamide (6d). White solid; 90.9 mg, 69% yield; mp 122-124 °C; ^{1}H NMR (600 MHz, $CDCl_3$) δ 10.03 (s, 1H), 7.89 (s, 1H), 7.71 (d, J = 7.8 Hz, 1H), 7.58 (d, J = 7.8 Hz, 1H), 7.19-7.16 (m, 1H), 6.23 (t, J = 2.4 Hz, 1H), 5.60 (s, 1H), 4.47-4.44 (m, 1H), 3.25-3.20 (m, 1H), 2.90 (dd, J = 17.4, 2.4 Hz, 1H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 168.0, 163.9, 135.5, 132.7, 132.2, 130.9, 130.1, 125.6, 124.4 (q, $J_{C\text{-}F}$ = 280.2 Hz), 122.8, 120.9, 56.8 (q, $J_{C\text{-}F}$ = 31.6 Hz), 24.8; ^{19}F NMR (376 MHz, $CDCl_3$) δ -76.9 (d, $J_{F\text{-}H}$ = 6.1 Hz); HRMS (ESI) m/z $C_{13}H_{10}NaBrF_3N_2O_2$ $[M + Na]^+$ calcd 384.9770, found: 384.9777.

***N*-(3-Methylene-2-oxo-5-(trifluoromethyl)pyrrolidin-1-yl)-4-(trifluoromethyl)benzamide (6e)**. White solid; 81.1 mg, 64% yield; mp 137-138 °C; ^{1}H NMR (600 MHz, $CDCl_3$) δ 10.16 (s, 1H), 7.84 (d, J = 8.4 Hz, 2H), 7.53 (d, J = 8.4 Hz, 2H), 6.27 (t, J = 2.4 Hz, 1H), 5.64 (s, 1H), 4.49-4.46 (m, 1H), 3.28-3.23 (m, 1H), 2.93 (dd, J = 17.4, 2.4 Hz, 1H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 168.1, 163.9, 134.0 (q, $J_{C\text{-}F}$ = 32.4 Hz), 133.5, 132.5, 127.8, 125.6 (q, J = 3.6 Hz), 124.4 (q, $J_{C\text{-}F}$ = 280.0 Hz), 123.4 (q, $J_{C\text{-}F}$ = 270.7 Hz), 121.1, 56.9 (q, $J_{C\text{-}F}$ = 32.0 Hz), 24.9; ^{19}F NMR (376 MHz, $CDCl_3$) δ -67.8 (s), -81.0 (d, $J_{F\text{-}H}$ = 6.0 Hz); HRMS (ESI) m/z $C_{14}H_{10}NaF_6N_2O_2$ $[M + Na]^+$ calcd 375.0539, found: 375.0544.

4-Methoxy-*N*-(3-methylene-2-oxo-5-(trifluoromethyl)pyrrolidin-1-yl)benzamide (6f). White solid; 91.9 mg, 81% yield; mp 158-160 °C; ^{1}H NMR (600 MHz, $CDCl_3$) δ 9.94 (br, 1H), 7.77 (d, J = 8.4 Hz, 2H), 6.76-6.75 (m, 2H), 6.22 (s, 1H), 5.58 (s, 1H), 4.47 (s, 1H), 3.79 (s, 3H), 3.24-3.20 (m, 1H), 2.88 (d, J = 17.4 Hz, 1H), ^{13}C NMR (150 MHz, $CDCl_3$) δ 168.1, 165.1, 162.9, 133.0, 129.5, 124.6 (q, $J_{C\text{-}F}$ = 279.9 Hz), 122.9, 120.4, 113.7, 56.9 (q, $J_{C\text{-}F}$ = 31.6 Hz), 55.3, 24.8. ^{19}F NMR (376 MHz, $CDCl_3$) δ -76.9 (d, $J_{F\text{-}H}$ = 6.2 Hz); HRMS (ESI) m/z $C_{14}H_{13}NaF_3N_2O_3$ $[M + Na]^+$ calcd 337.0770, found: 337.0778.

3-Methyl-*N*-(3-methylene-2-oxo-5-(trifluoromethyl)pyrrolidin-1-yl)benzamide (6h). White solid; 45.0 mg, 42% yield; mp 141-143 °C; ^{1}H NMR (600 MHz, $CDCl_3$) δ 9.80 (s, 1H), 7.61 (d, J = 6.0 Hz, 2H), 7.23 (d, J = 7.8 Hz, 1H), 7.18 (t, J = 7.8 Hz, 1H), 6.22 (t, J = 2.4 Hz, 1H), 5.58 (t, J = 2.4 Hz, 1H), 4.52-4.48 (m, 1H), 3.25-3.20 (m, 1H), 2.91-2.87 (m, 1H), 2.27 (s, 3H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 167.9, 165.6, 138.3, 133.1, 133.0, 130.5, 128.4, 128.12, 124.5, 124.6 (q, $J_{C\text{-}F}$ = 279.9 Hz), 120.4, 56.7 (q, $J_{C\text{-}F}$ = 31.6 Hz), 24.8, 21.1; ^{19}F NMR (376

MHz, $CDCl_3$) δ -76.9 (d, J_{F-H} = 6.2 Hz); HRMS (ESI) m/z $C_{14}H_{13}NaF_3N_2O_2$ $[M + Na]^+$ calcd 321.0821, found: 321.0826.

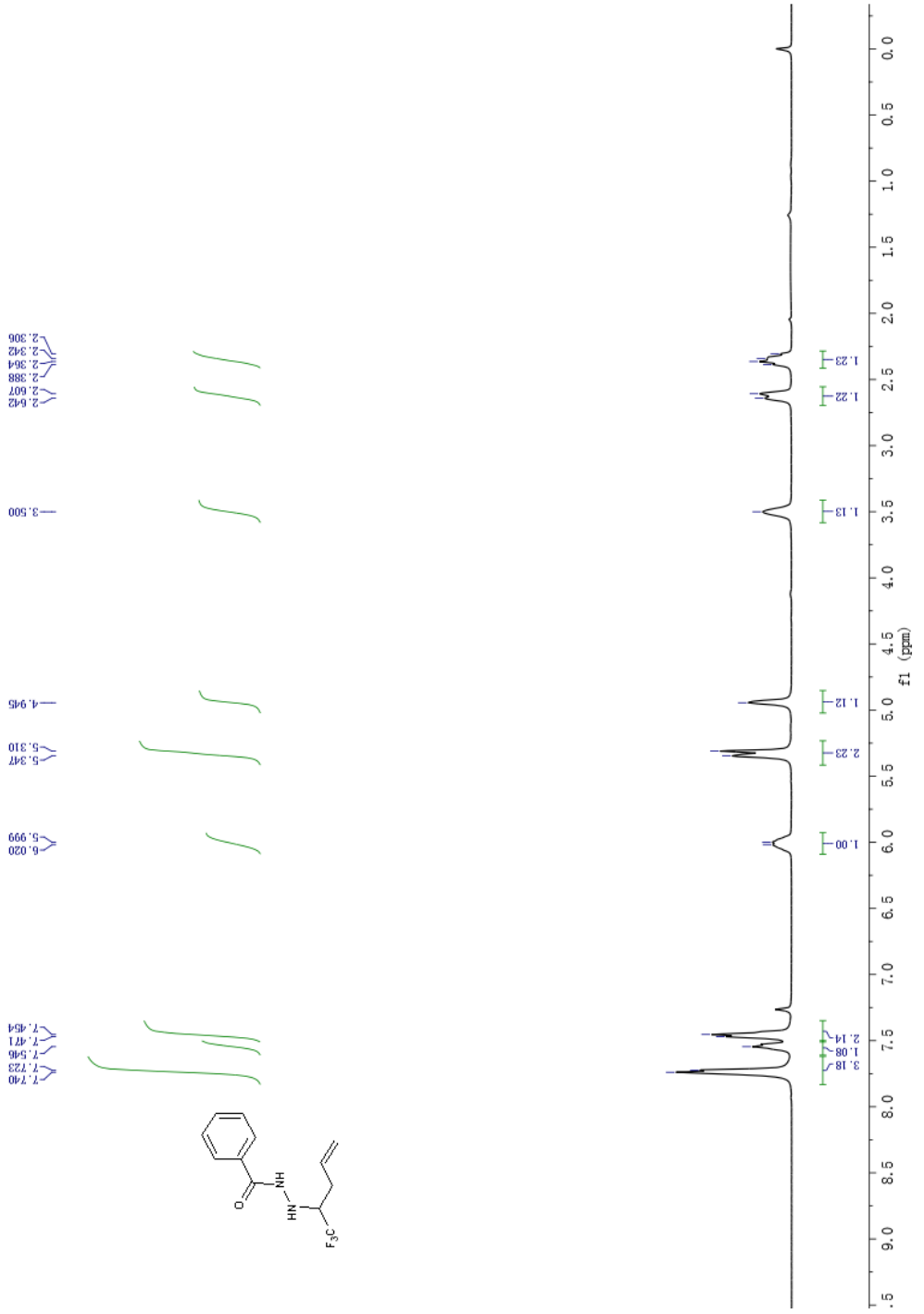
4-Methyl-*N*-(3-methylene-2-oxo-5-(trifluoromethyl)pyrrolidin-1-yl)benzamide (6i). White solid; 91.2mg, 85% yield; mp 140-142 °C; ^1H NMR (600 MHz, $CDCl_3$) δ 9.92 (s, 1H), 7.67 (d, *J* = 7.8 Hz, 2H), 7.06 (d, *J* = 7.8 Hz, 2H), 6.20 (s, 1H), 5.56 (s, 1H), 4.47 (s, 1H), 3.22-3.18 (m, 1H), 2.87 (d, *J* = 17.4 Hz, 1H), 2.31 (s, 3H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 168.0, 165.5, 143.0, 133.0, 129.1, 127.8, 127.5, 124.6 (q, J_{C-F} = 280.1 Hz), 120.4, 56.8 (q, J_{C-F} = 31.6 Hz), 24.8, 21.4; ^{19}F NMR (376 MHz, $CDCl_3$) δ -76.9 (d, J_{F-H} = 6.2 Hz); HRMS (ESI) m/z $C_{14}H_{13}NaF_3N_2O_2$ $[M + Na]^+$ calcd 321.0821, found: 321.0828.

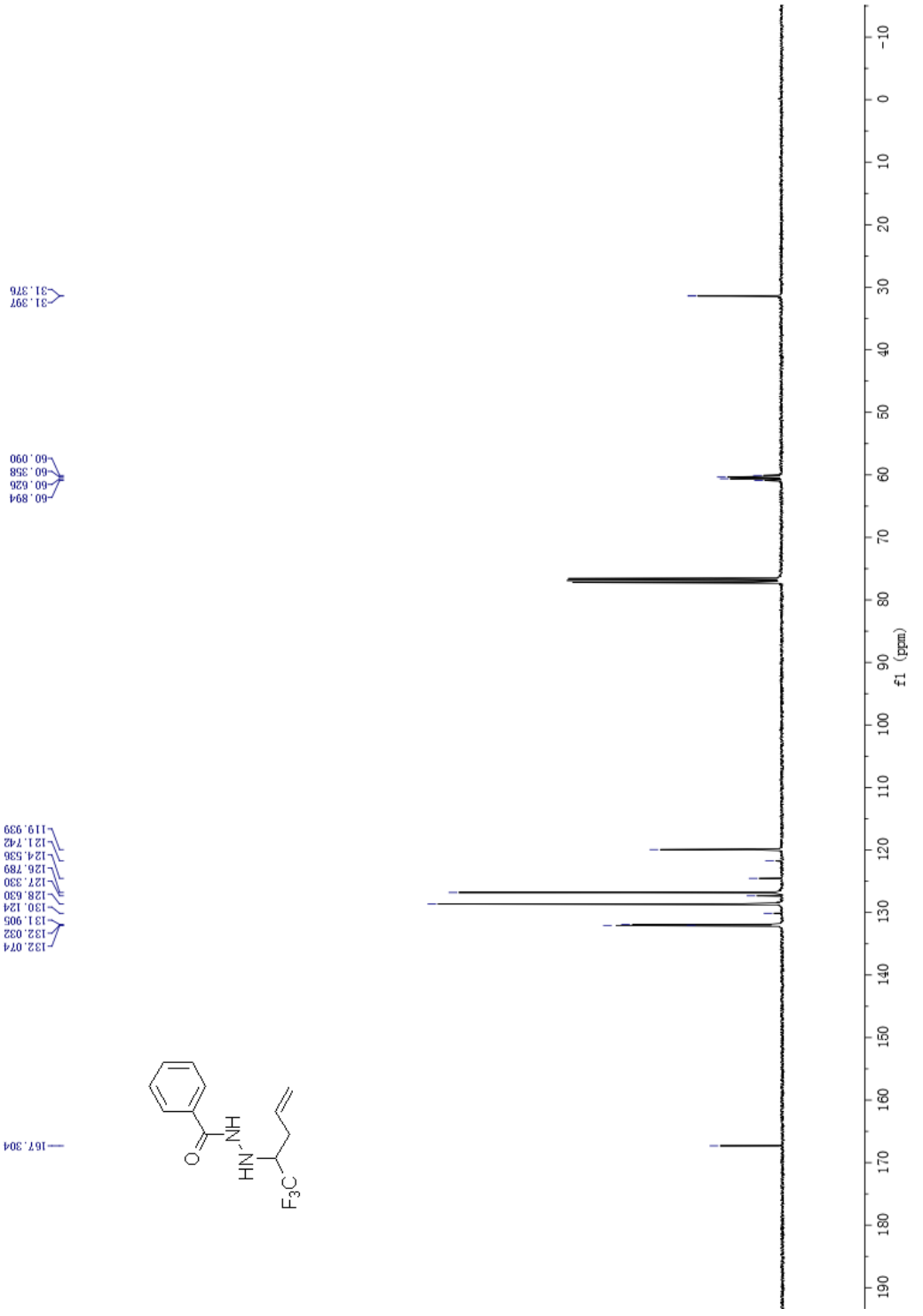
***N*-(3-Methylene-2-oxo-5-(trifluoromethyl)pyrrolidin-1-yl)furan-2-carboxamide (6j)**. White solid; 80.2 mg, 82% yield; mp 154-155 °C; ^1H NMR (400 MHz, $CDCl_3$) δ 9.59 (br, 1H), 7.42 (d, *J* = 1.6 Hz, 1H), 7.14 (d, *J* = 3.2 Hz, 1H), 6.42 (dd, *J* = 4.0, 2.0 Hz, 1H), 6.23 (t, *J* = 2.4 Hz, 1H), 5.59 (t, *J* = 2.4 Hz, 1H), 4.50-4.44 (m, 1H), 3.23 (dd, *J* = 17.6, 9.2 Hz, 1H), 2.93-2.88 (m, 1H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 167.3, 156.5, 145.4, 145.2, 132.8, 124.6 (q, J_{C-F} = 280.0 Hz), 120.5, 116.4, 112.0, 56.9 (q, J_{C-F} = 31.6 Hz), 24.8 (d, *J* = 1.8 Hz); ^{19}F NMR (376 MHz, $CDCl_3$) δ -76.9 (d, J_{F-H} = 6.4 Hz); HRMS (ESI) m/z $C_{11}H_9NaF_3N_2O_3$ $[M + Na]^+$ calcd 297.0457, found: 297.0461.

***N*-(3-Methylene-2-oxo-5-(trifluoromethyl)pyrrolidin-1-yl)-2-naphthamide (6k)**. White solid; 90.2 mg, 75% yield; mp 187-189 °C; ^1H NMR (600 MHz, $CDCl_3$) δ 8.60 (s, 1H), 8.35 (d, *J* = 7.8 Hz, 1H), 7.90 (d, *J* = 8.4 Hz, 1H), 7.83 (d, *J* = 7.8 Hz, 1H), 7.68 (d, *J* = 6.6 Hz, 1H), 7.55-7.50 (m, 2H), 7.36-7.34 (m, 1H), 6.18 (d, *J* = 1.8 Hz, 1H), 5.55 (d, *J* = 1.2 Hz, 1H), 4.59 (s, 1H), 3.21 (dd, *J* = 16.2, 9.6 Hz, 1H), 2.88 (d, *J* = 17.4 Hz, 1H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 167.8, 167.2, 133.5, 132.7, 131.9, 130.2, 129.9, 128.3, 127.6, 126.6, 126.1, 125.1, 124.5, 124.7 (q, J_{C-F} = 279.6 Hz), 120.6, 56.7 (q, J_{C-F} = 31.6 Hz), 24.7; ^{19}F NMR (376 MHz, $CDCl_3$) δ -76.7 (d, J_{F-H} = 6.0 Hz); HRMS (ESI) m/z $C_{17}H_{13}NaF_3N_2O_2$ $[M + Na]^+$ calcd 357.0821, found: 357.0826.

NMR and HRMS Spectra of compounds 4

N'-(1,1,1-trifluoropent-4-en-2-yl)benzohydrazide(4a)

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60.626
60.358
60.090
31.397
31.376
f1 (ppm)


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-75.484
O
NH
HN
F3C
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-15
-20
-25
-30
-35
-40
-45
-50
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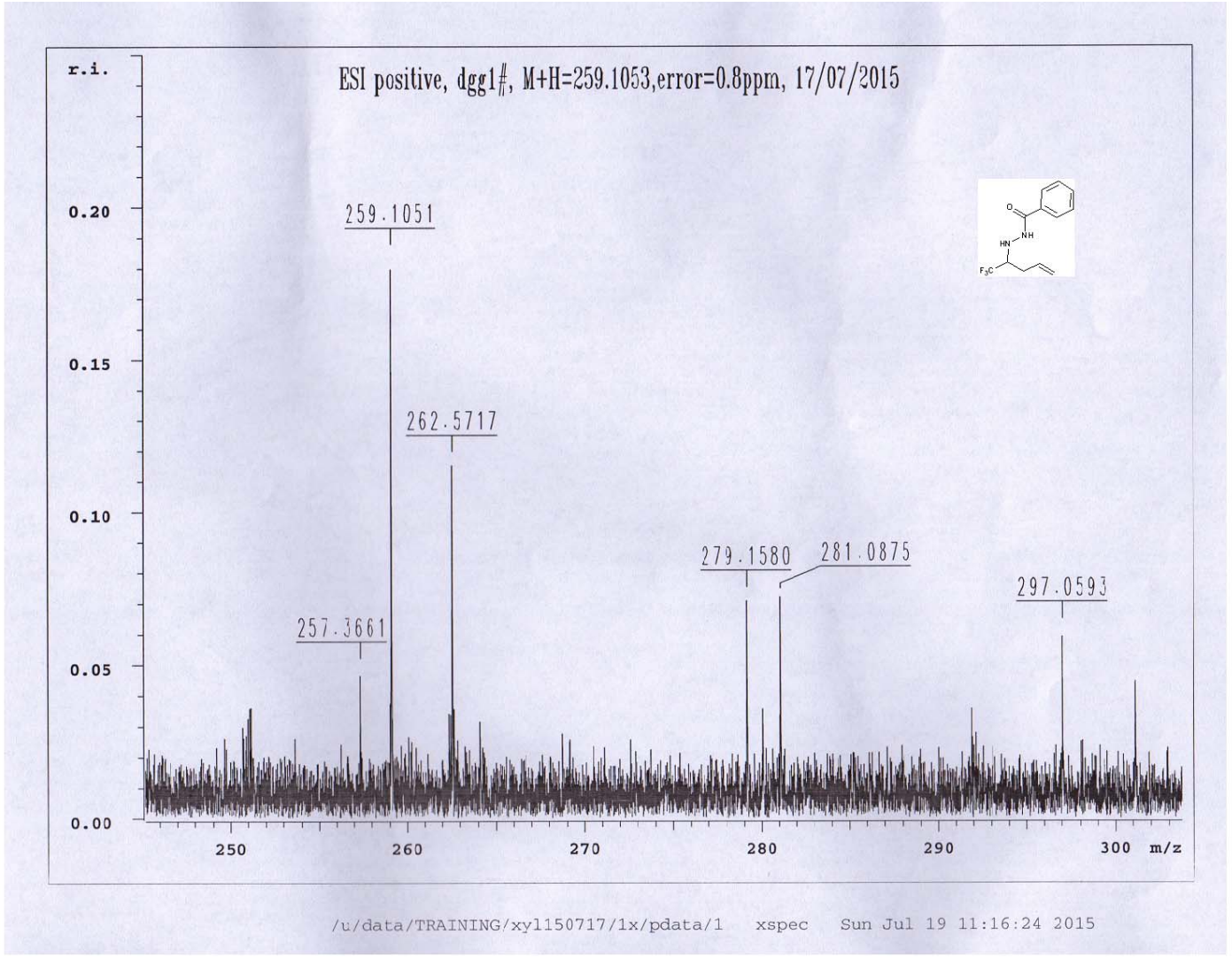
f1 (ppm)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250
260
270
280
290
300
m/z
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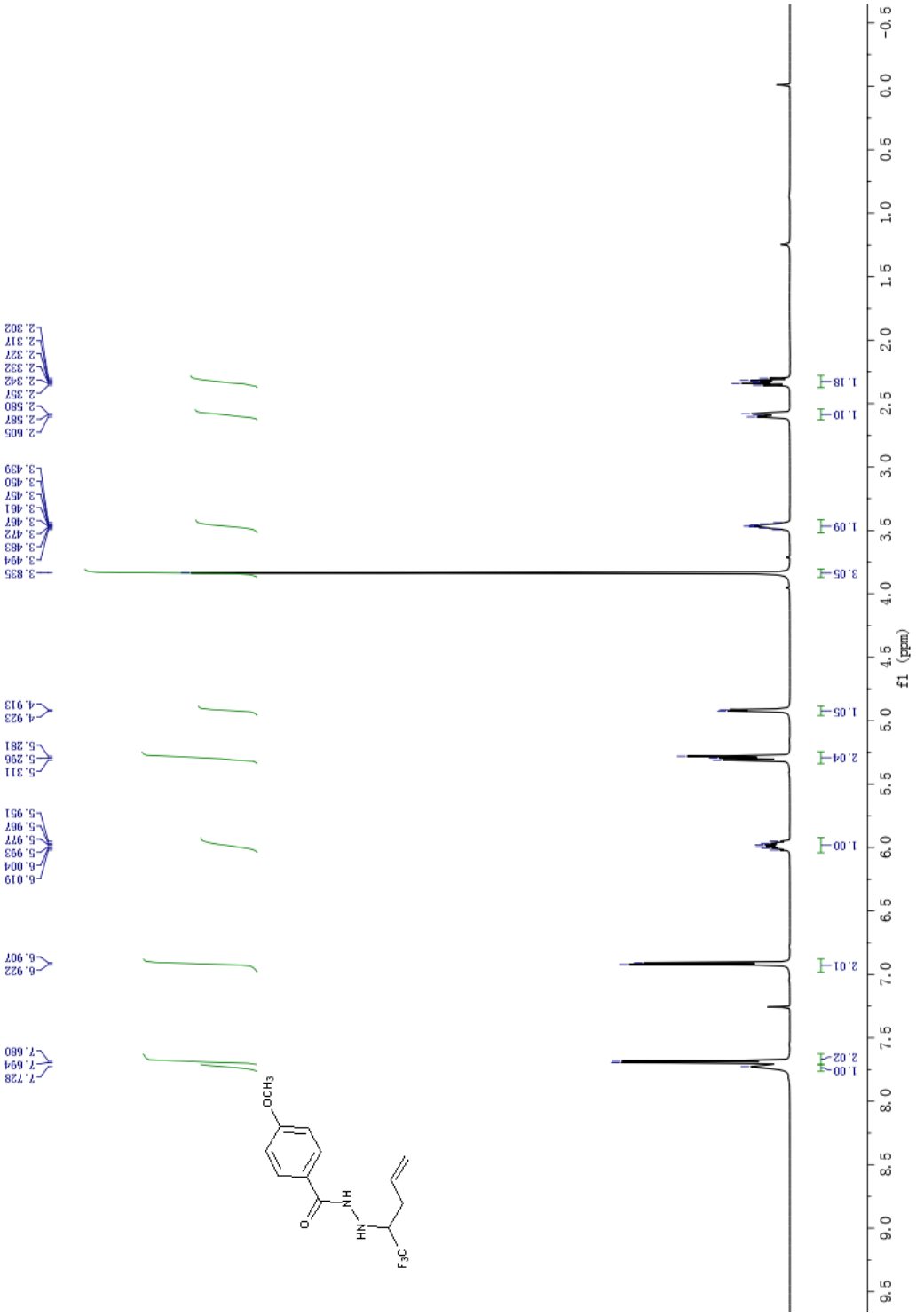
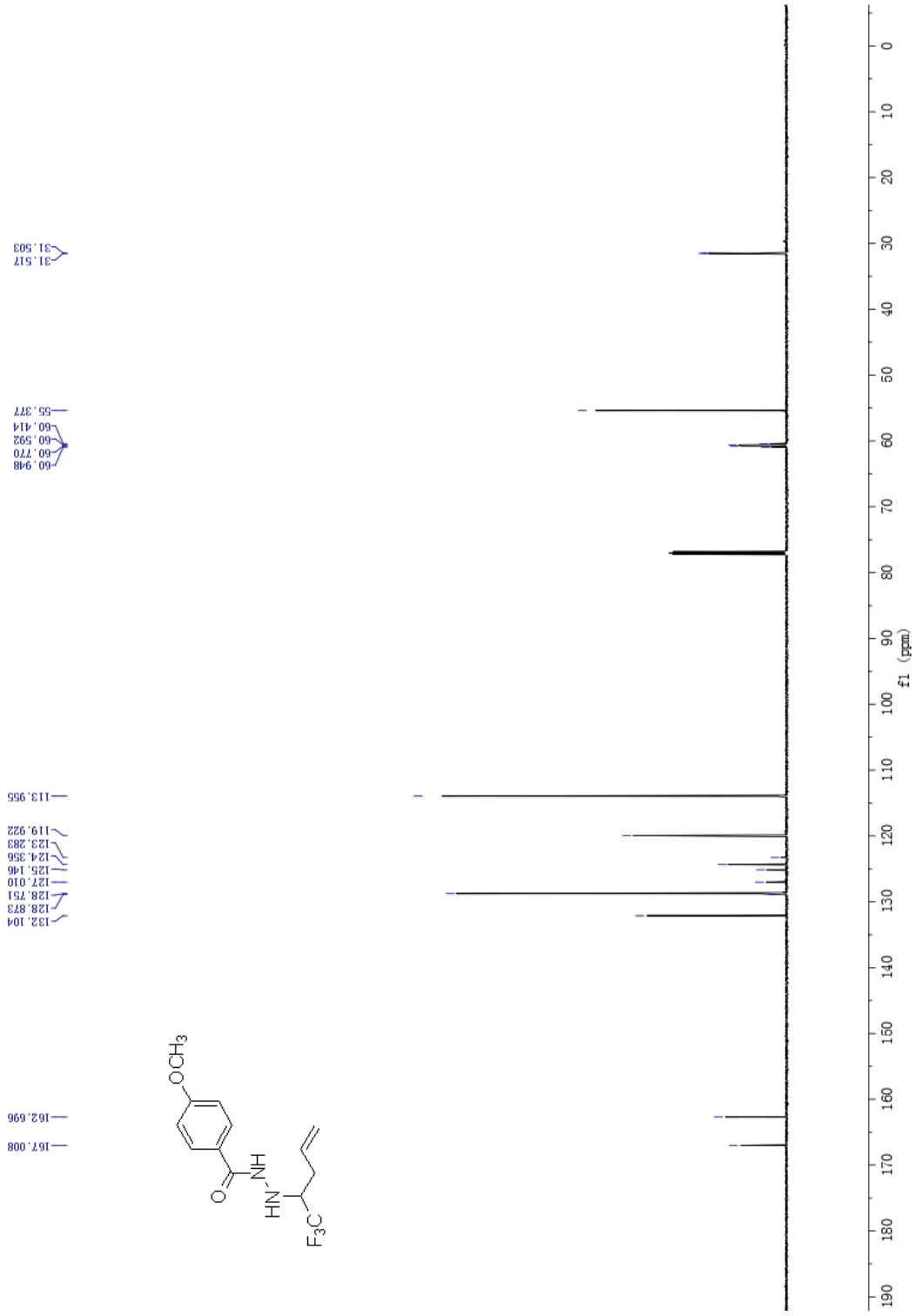
4-methoxy-N'-(1,1,1-trifluoropent-4-en-2-yl)benzohydrazide(4b)

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HN
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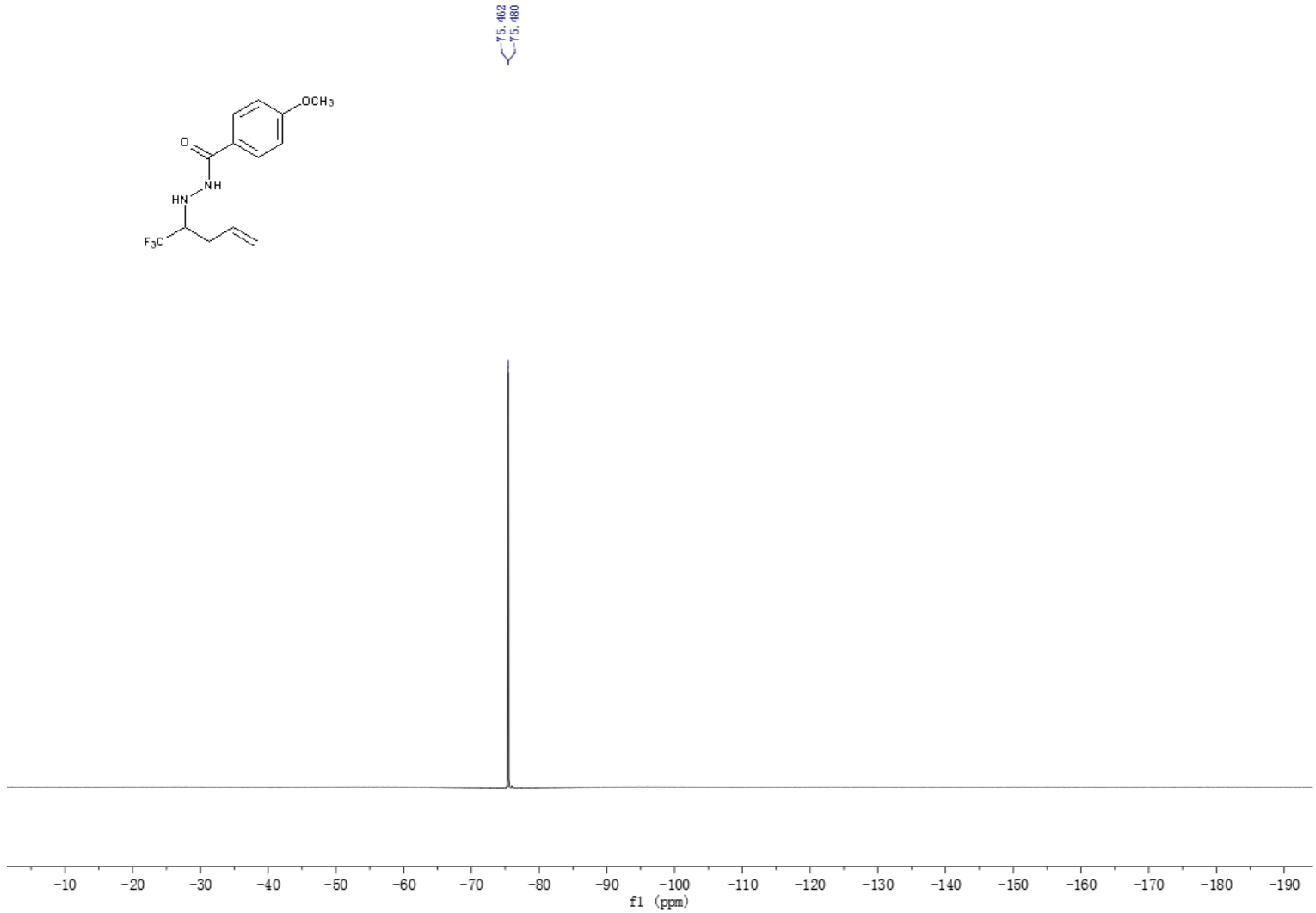
f1 (ppm)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HN
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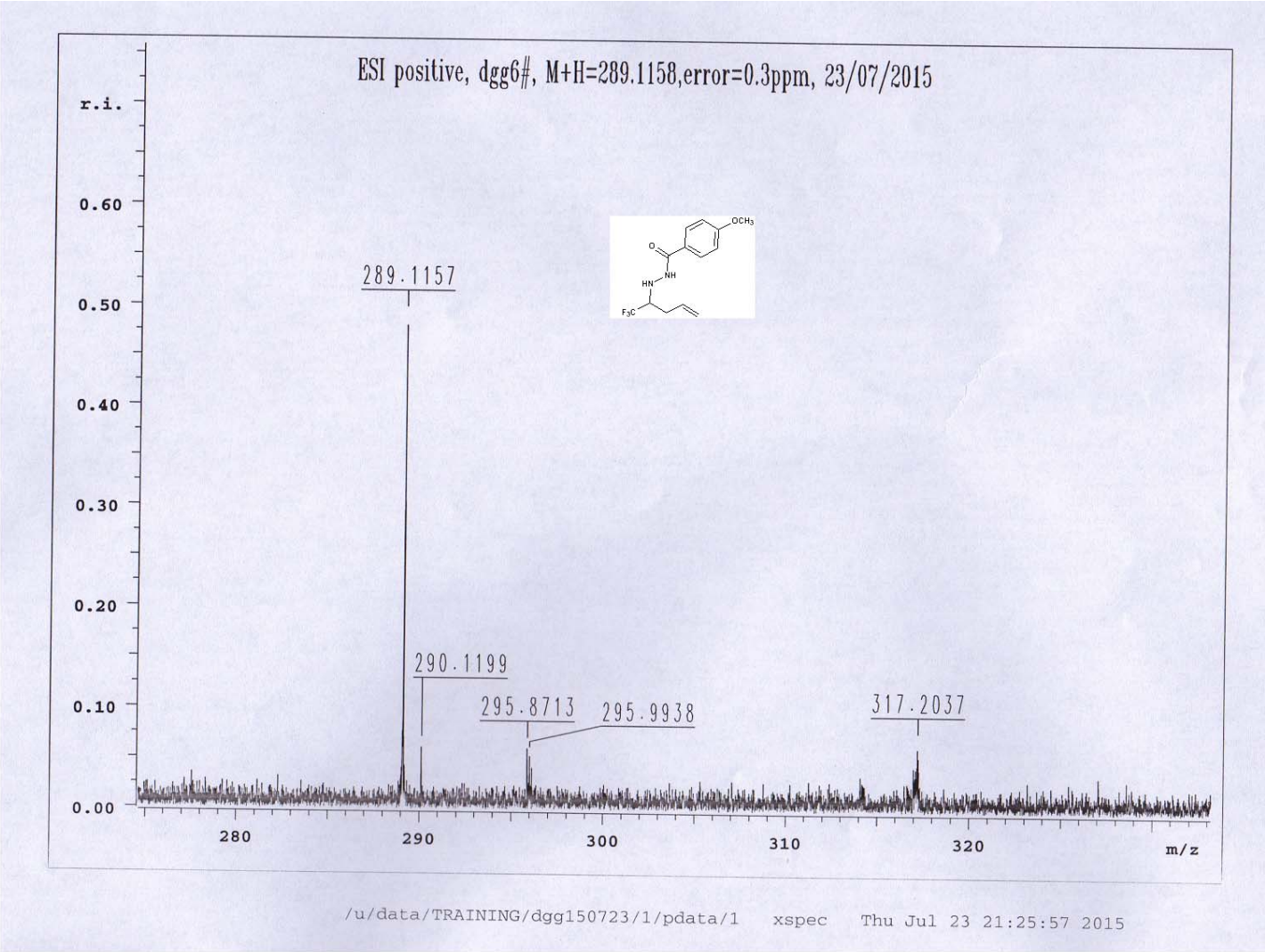
-150
-160
-170
-180
-190
f1 (ppm)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m/z
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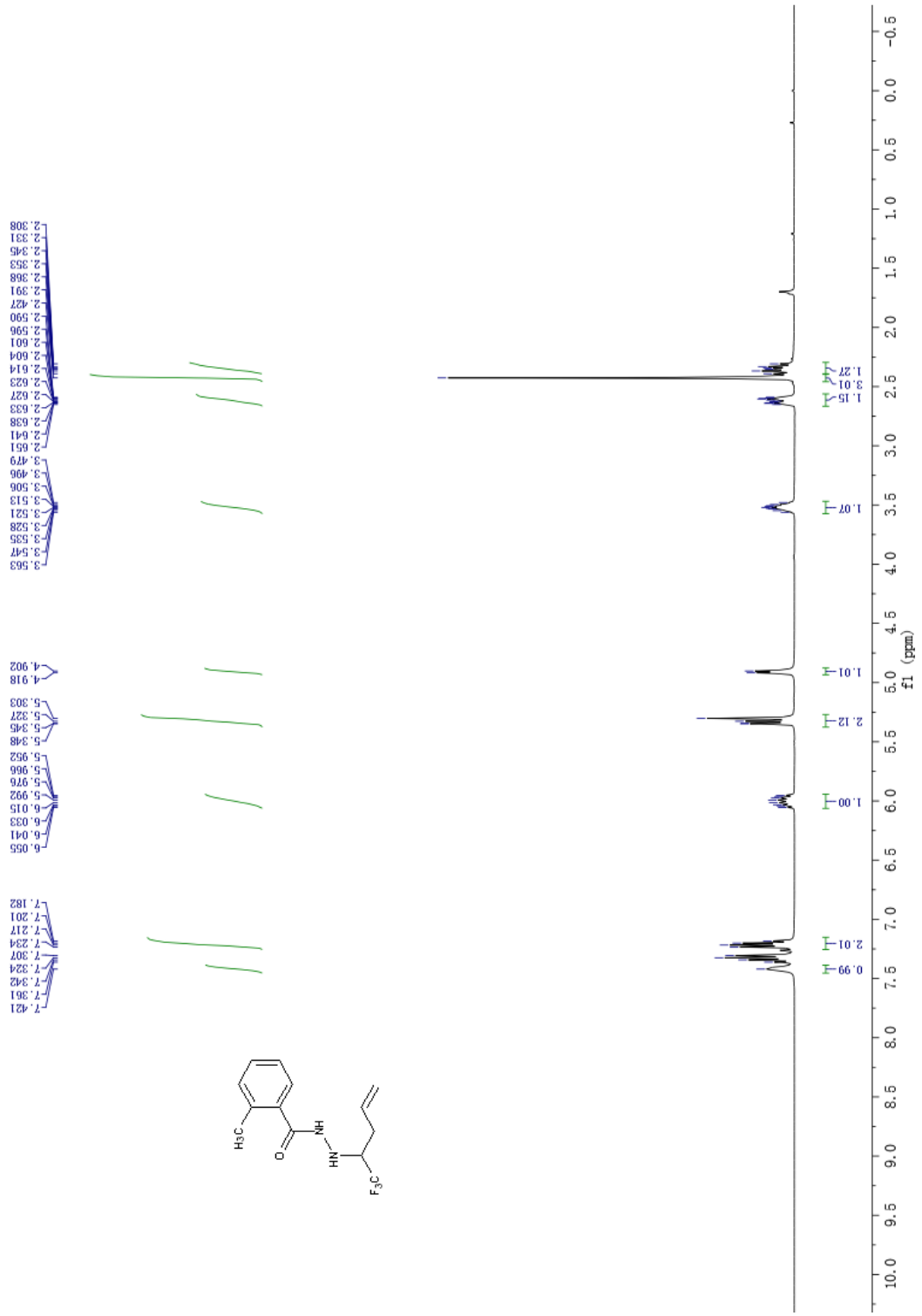
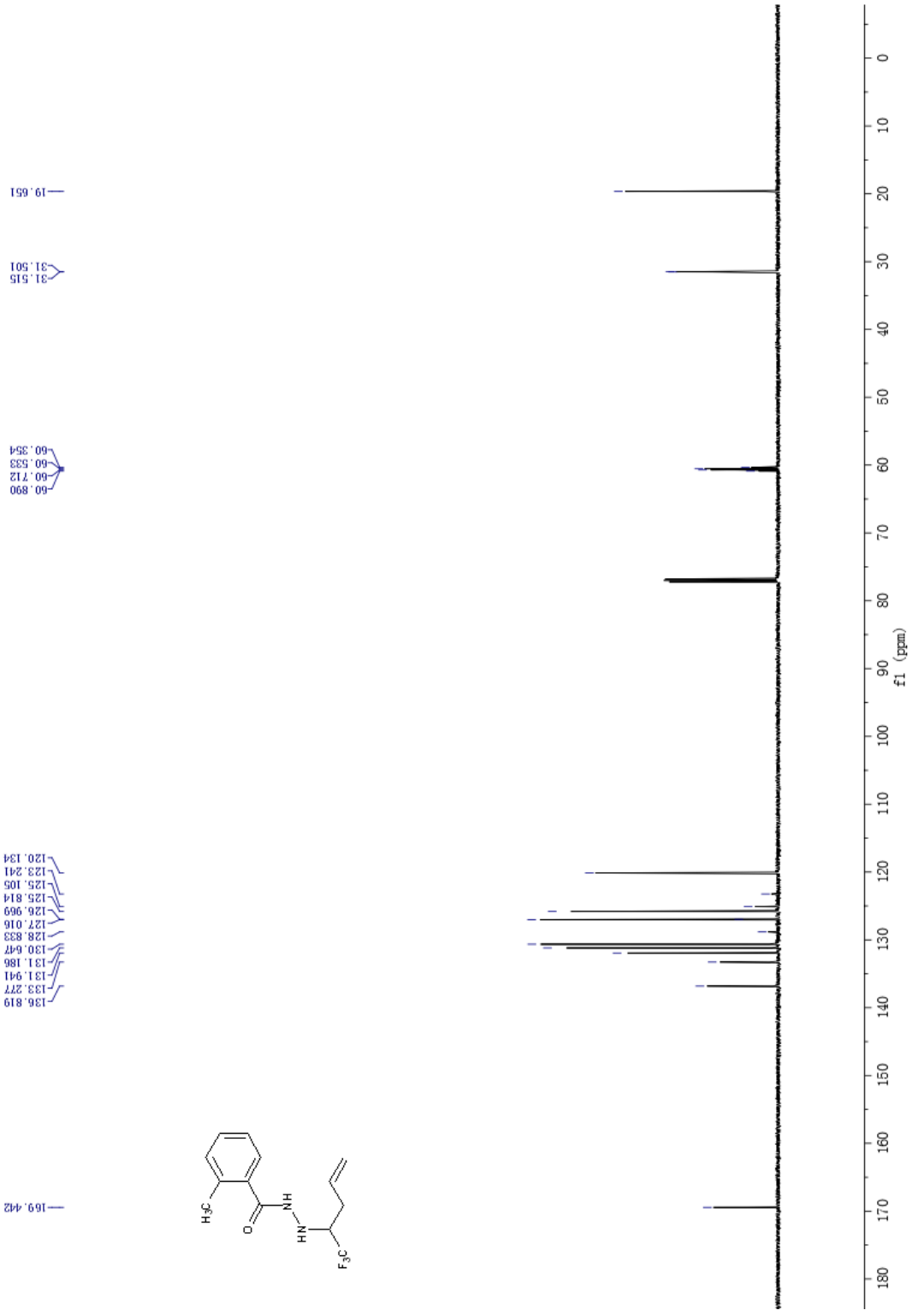
2-methyl-N'-(1,1,1-trifluoropent-4-en-2-yl)benzohydrazide(4c)

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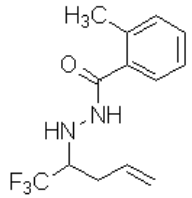
31.501
19.651
f1 (ppm)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HN
F3C

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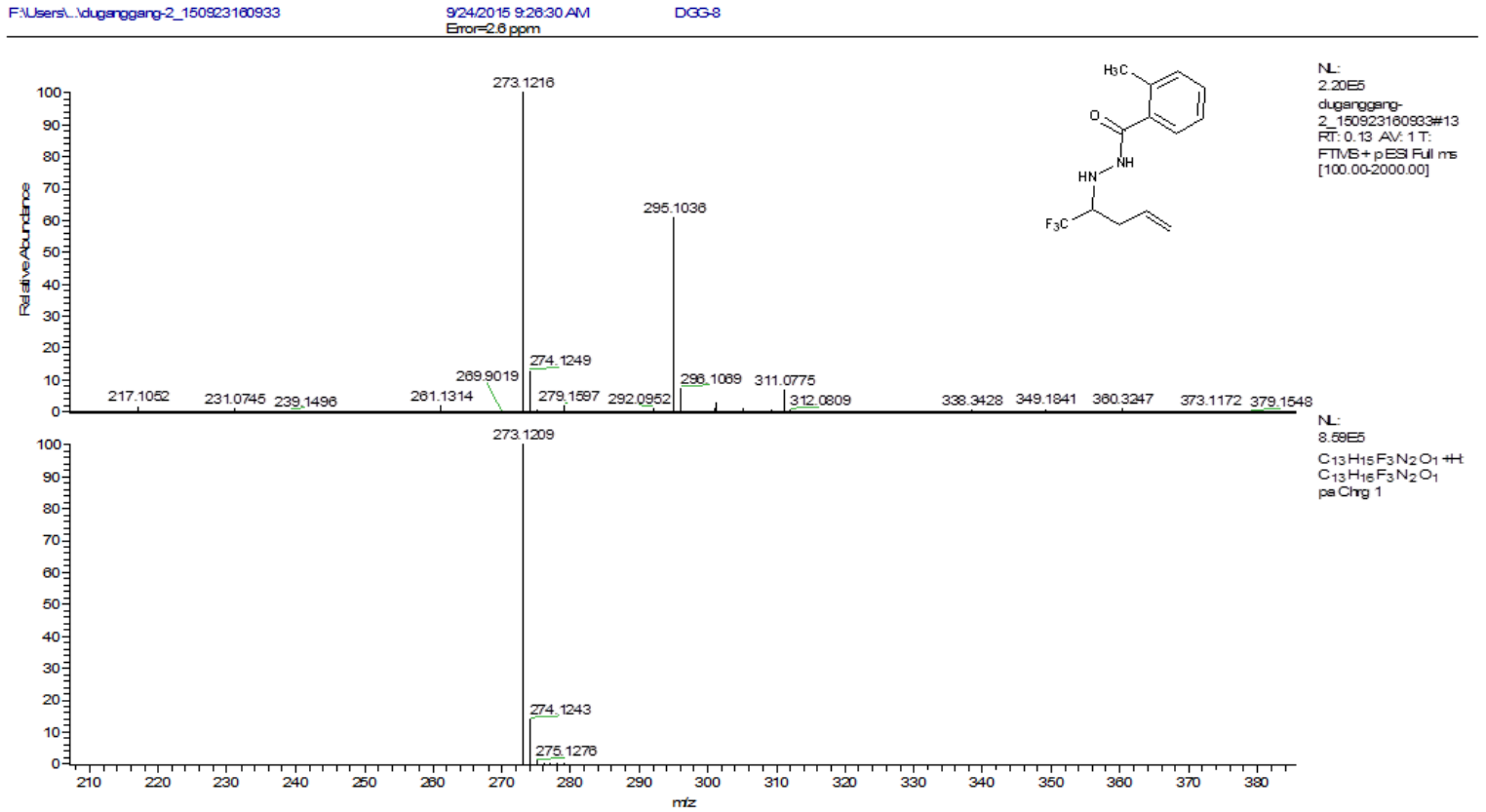
f1 (ppm)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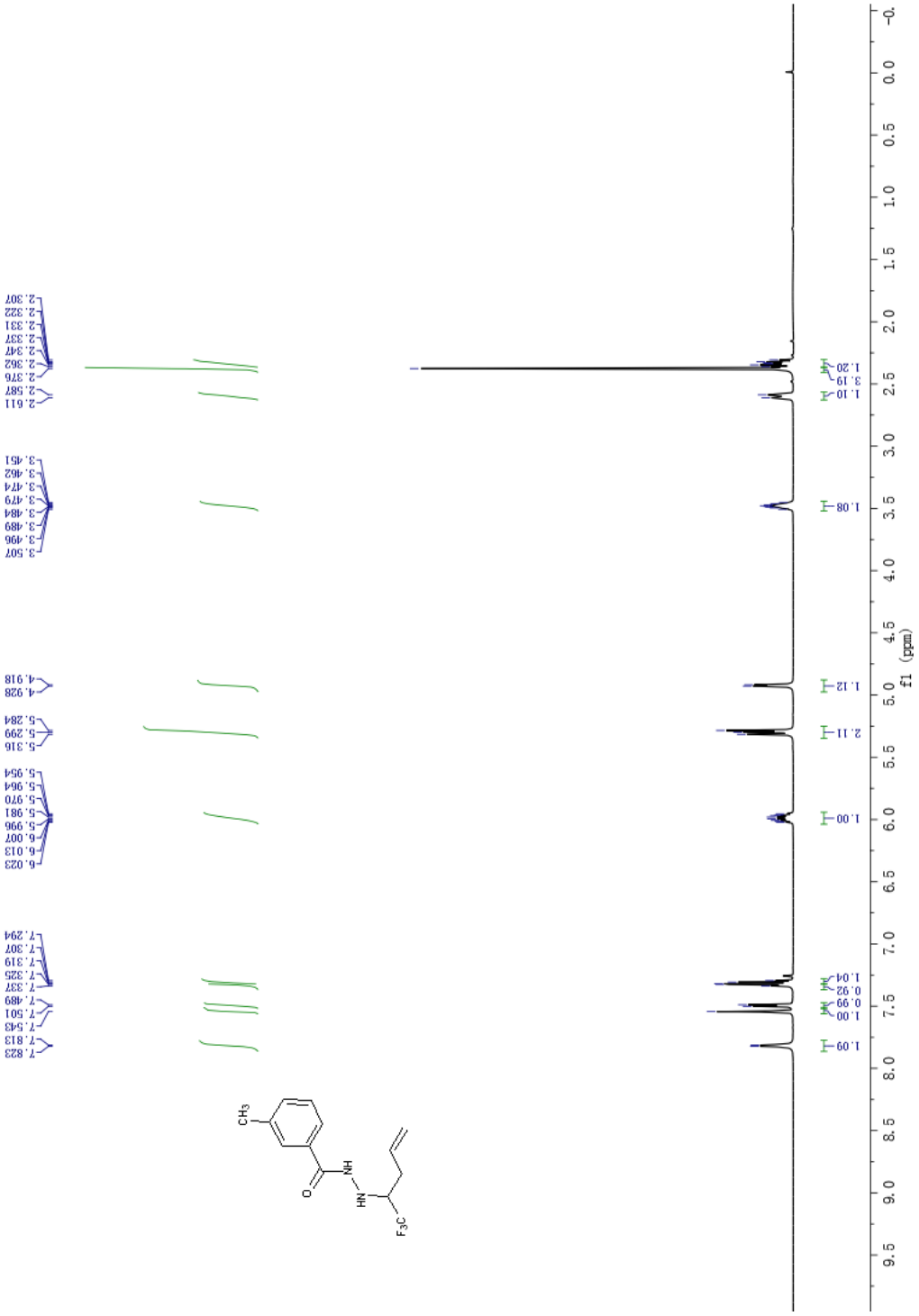
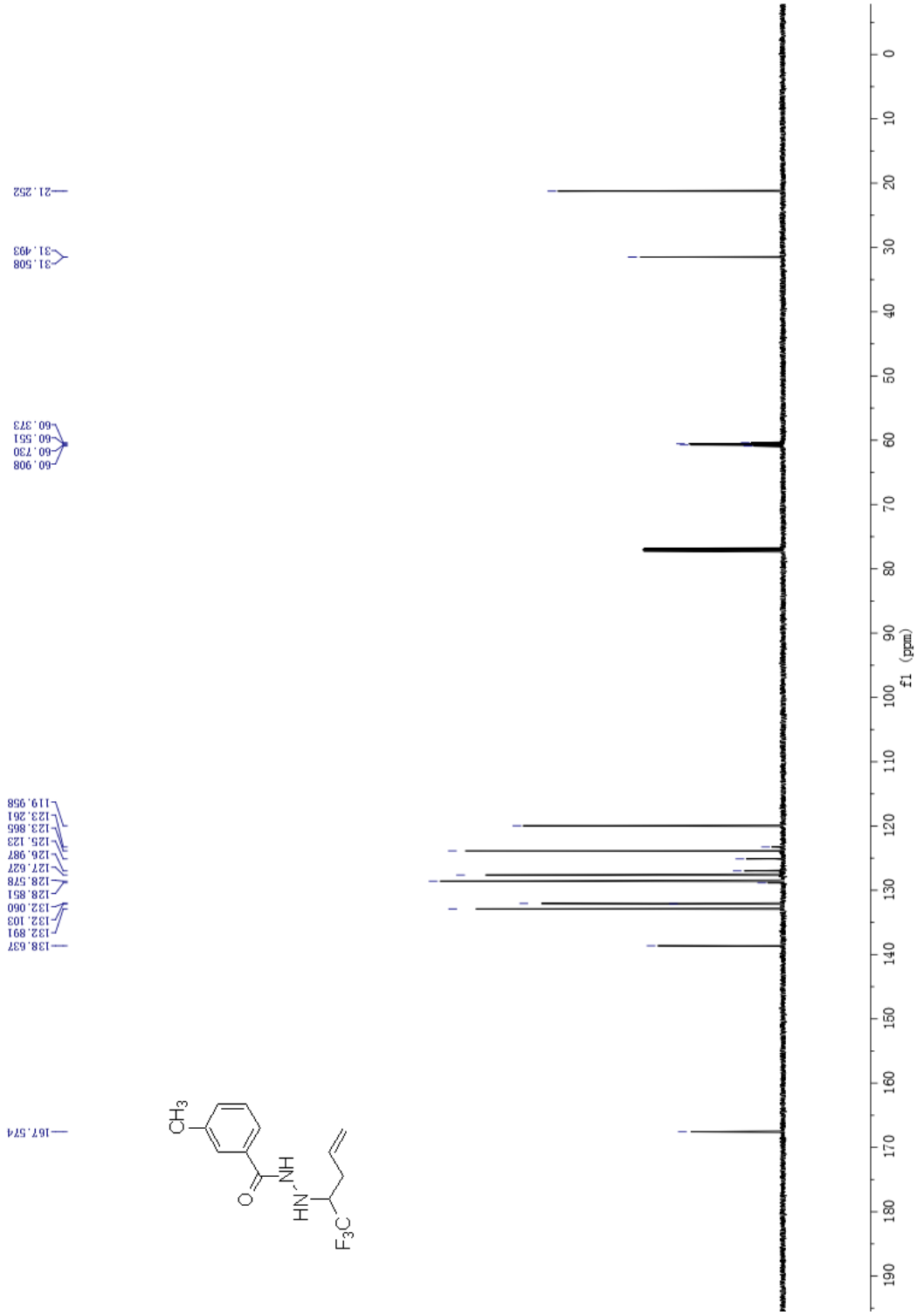
m/z

3-methyl-N'-(1,1,1-trifluoropent-4-en-2-yl)benzohydrazide(4d)

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CH3
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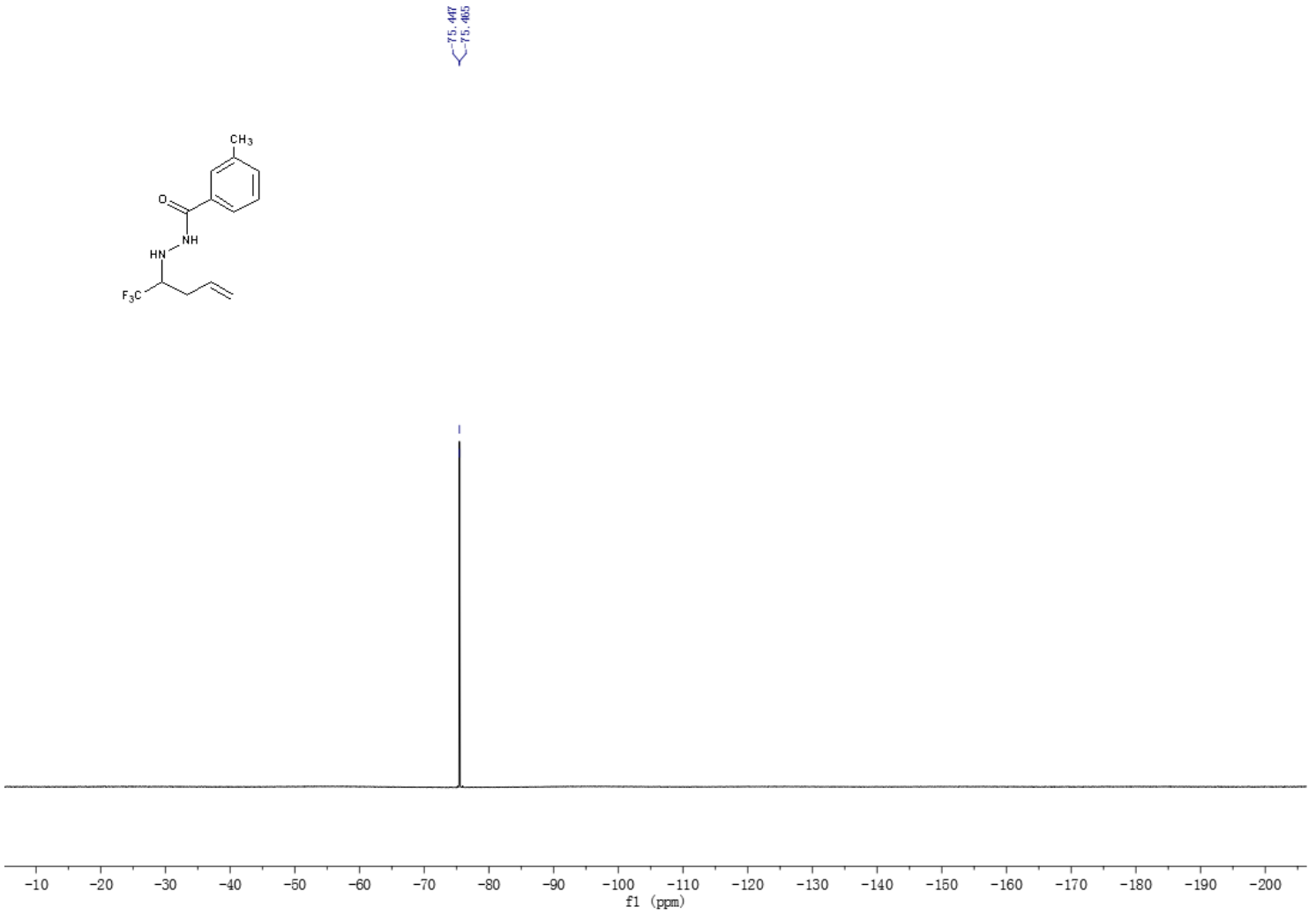
NH
HN
F3C
f1 (ppm)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HN
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-170
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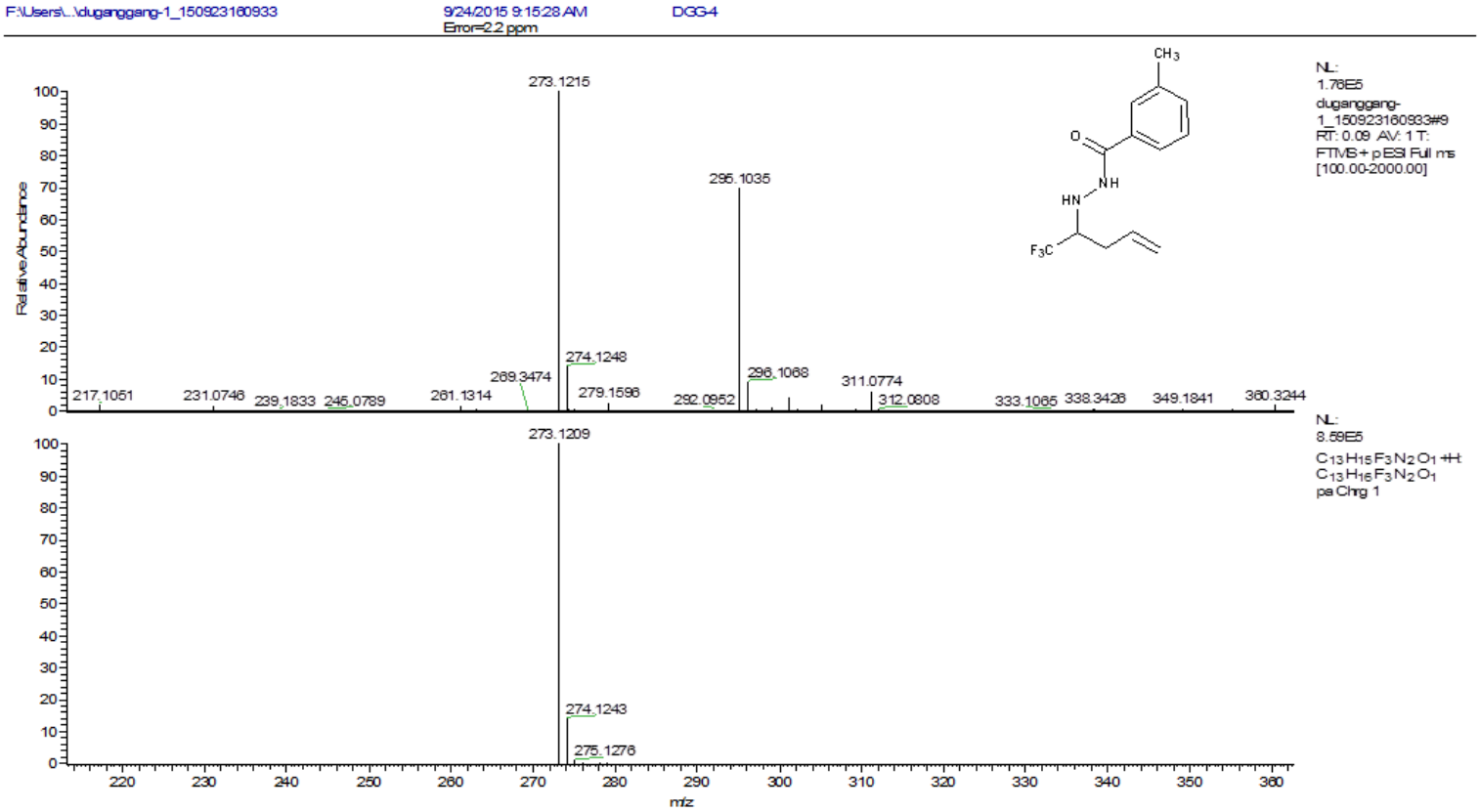
f1 (ppm)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Relative Abundance
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FTMS + p ESI Full ms
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NL:
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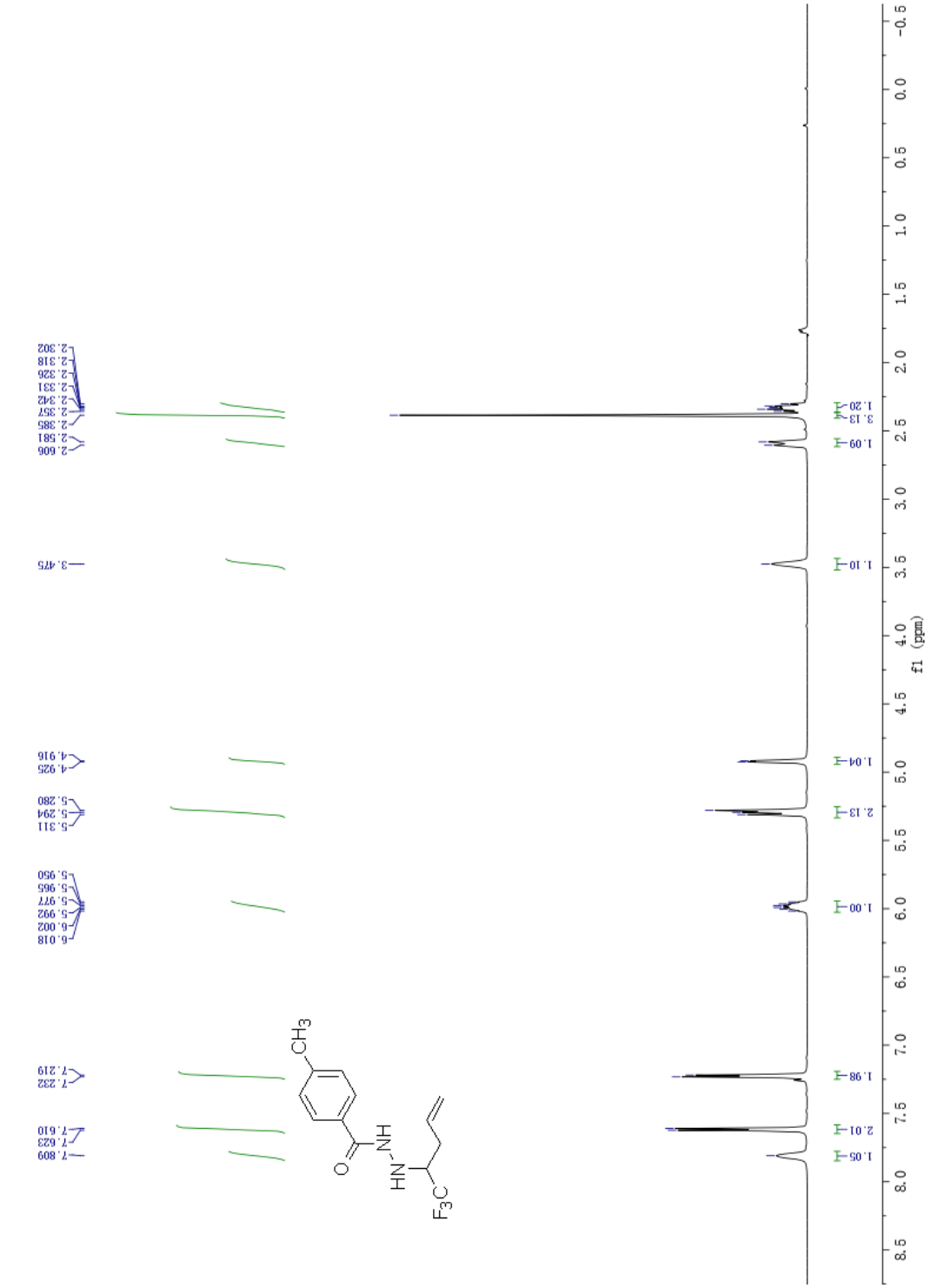
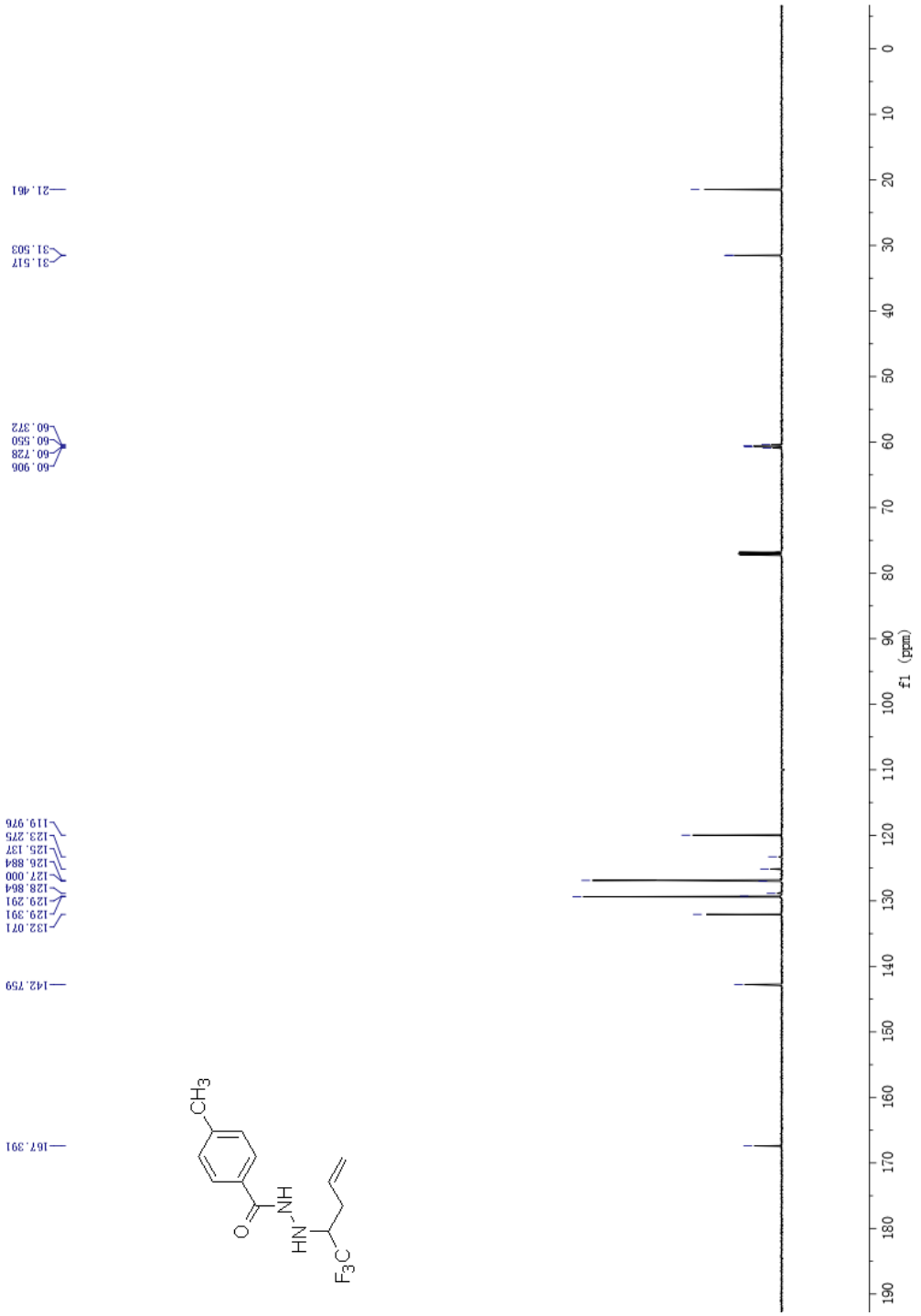
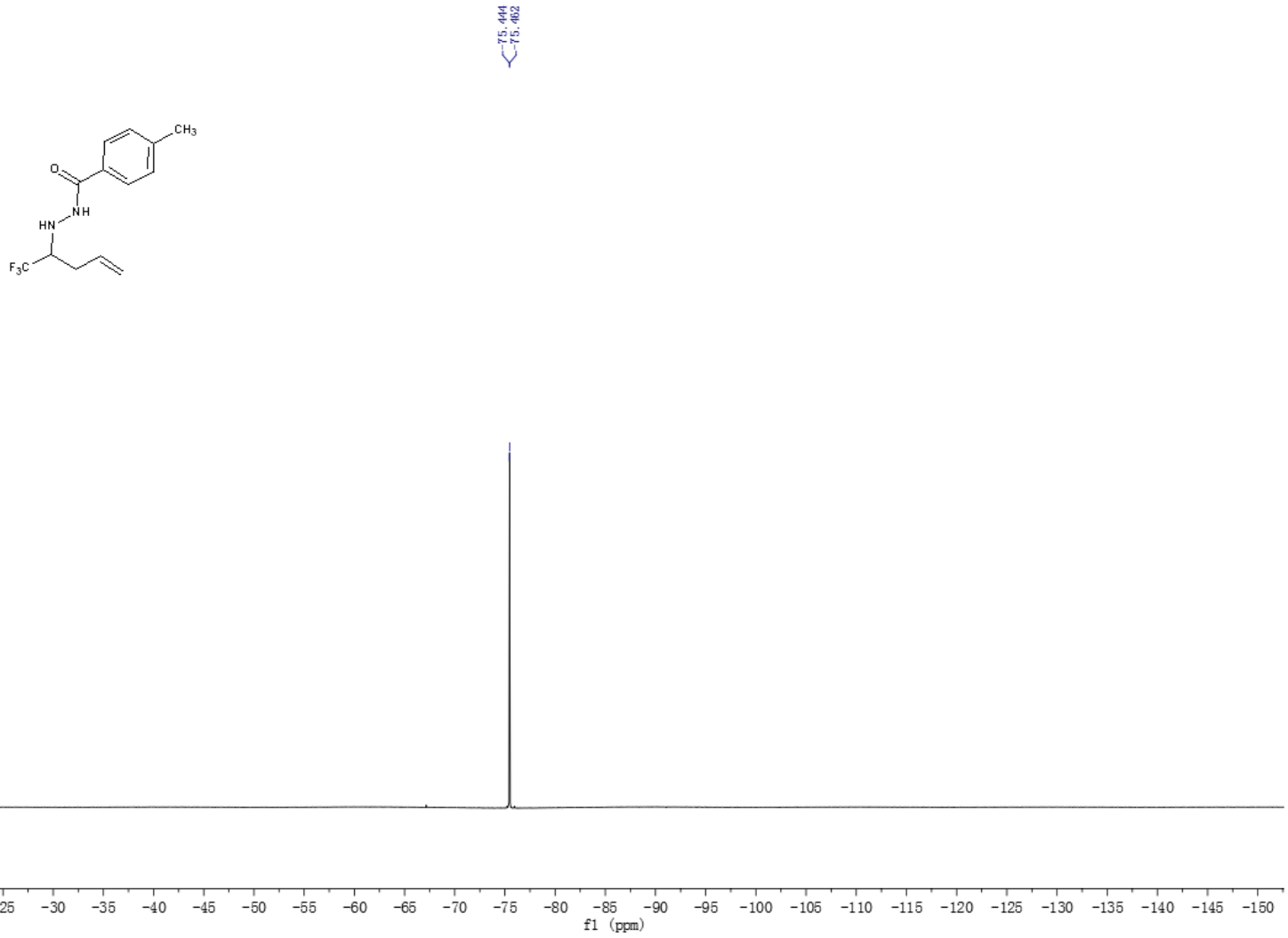
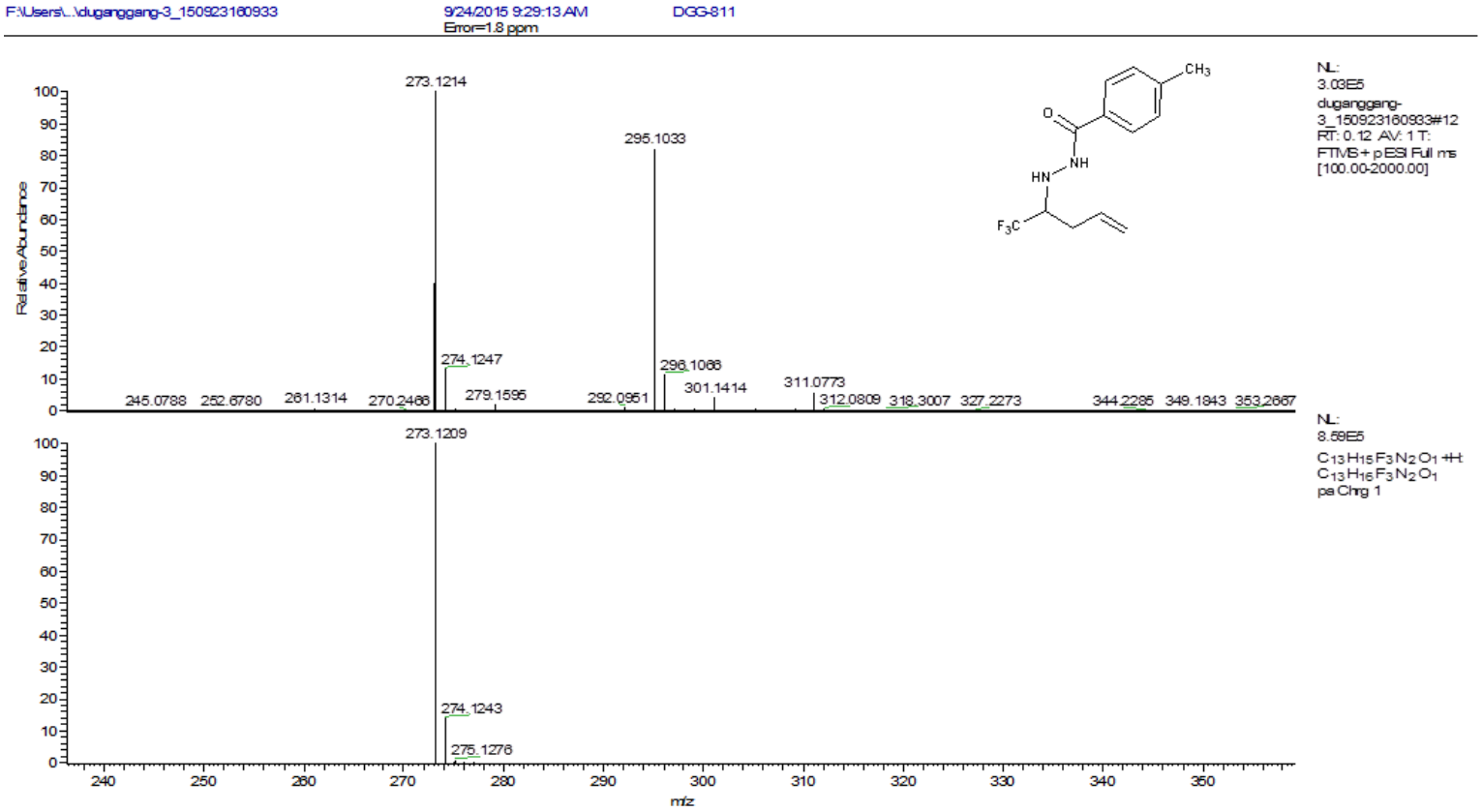
C13H16F3N2O1
pa Chrg 1
273.1209
274.1243
275.1276
m/z

4-methyl-N'-(1,1,1-trifluoropent-4-en-2-yl)benzohydrazide(4e)

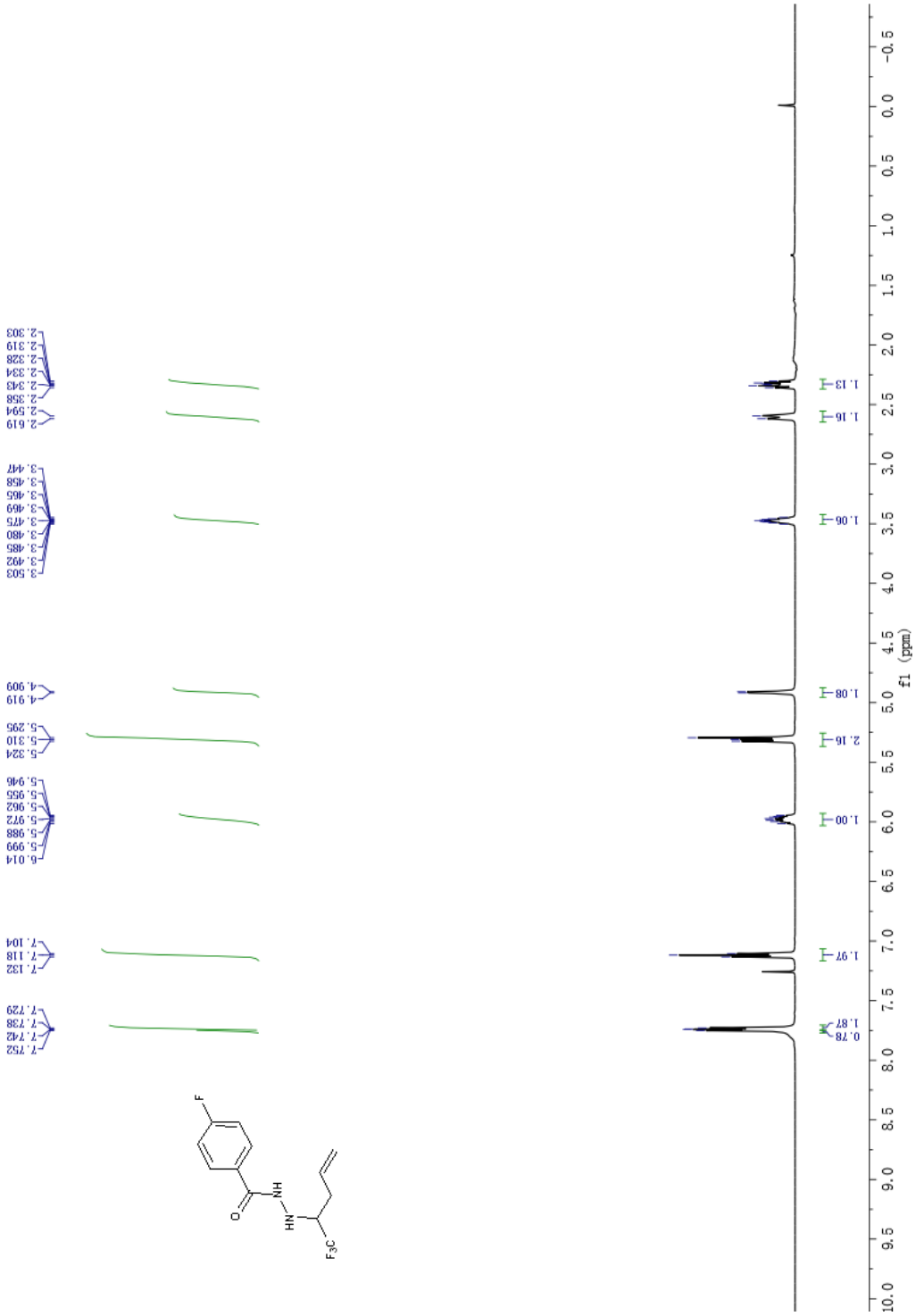


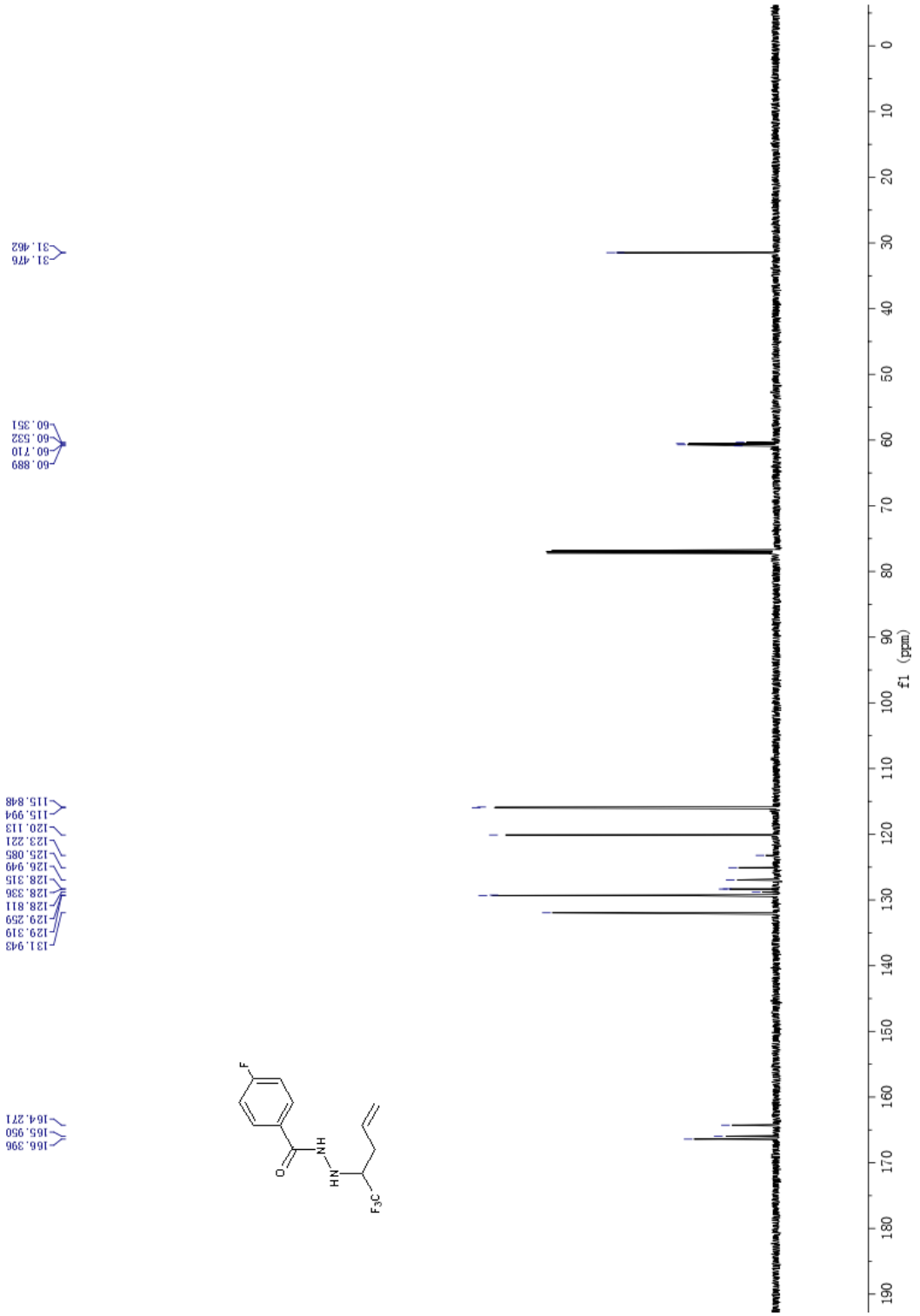

21.461
31.503
31.517
60.372
60.550
60.728
60.906
119.976
123.275
125.137
126.884
127.000
128.864
129.291
129.391
132.071
142.759
167.391
CH3
O
NH
HN
F3C
f1 (ppm)

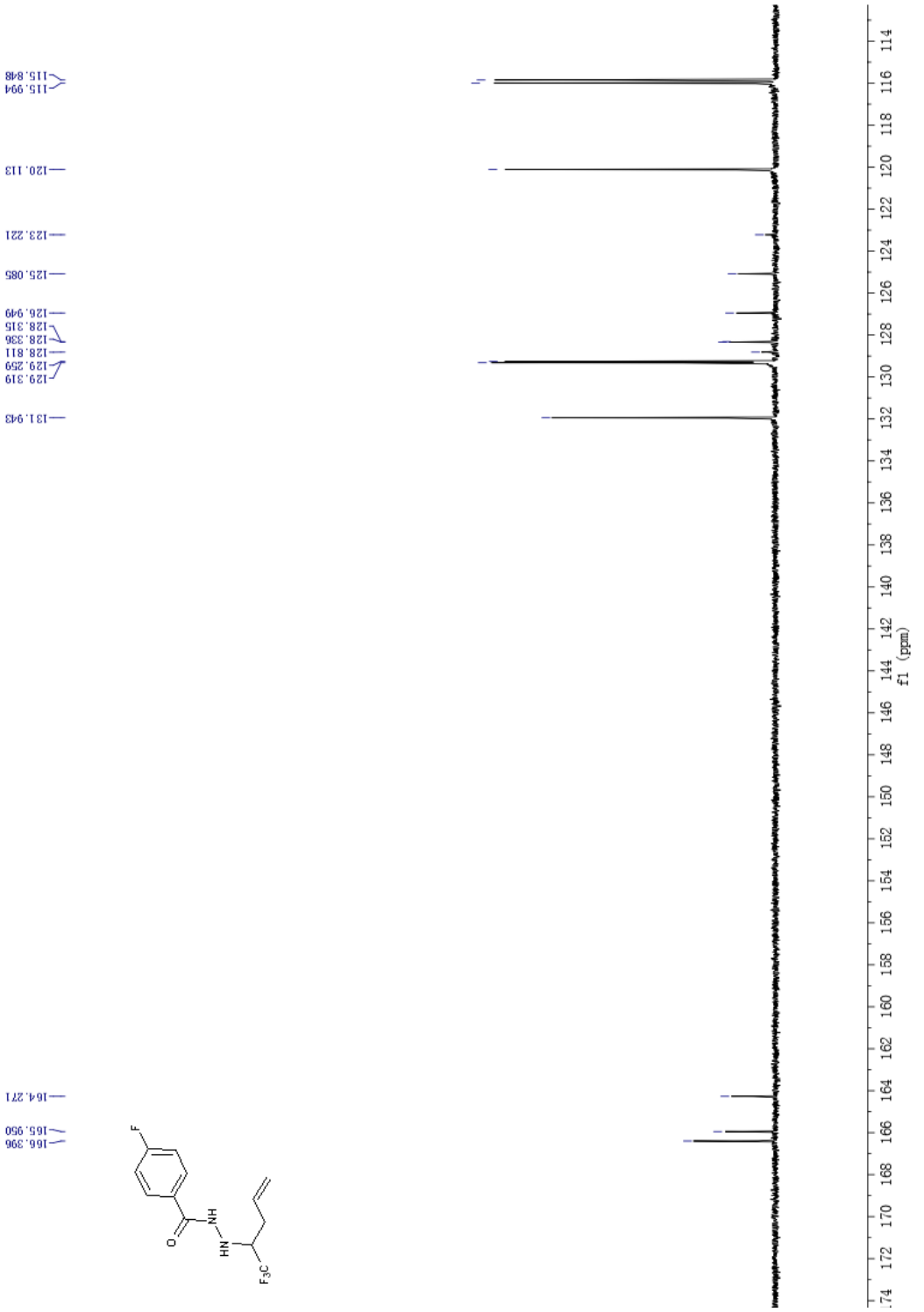

-75.444
-75.462
CH3
O
NH
HN
F3C
-20 -25 -30 -35 -40 -45 -50 -55 -60 -65 -70 -75 -80 -85 -90 -95 -100 -105 -110 -115 -120 -125 -130 -135 -140 -145 -150
f1 (ppm)

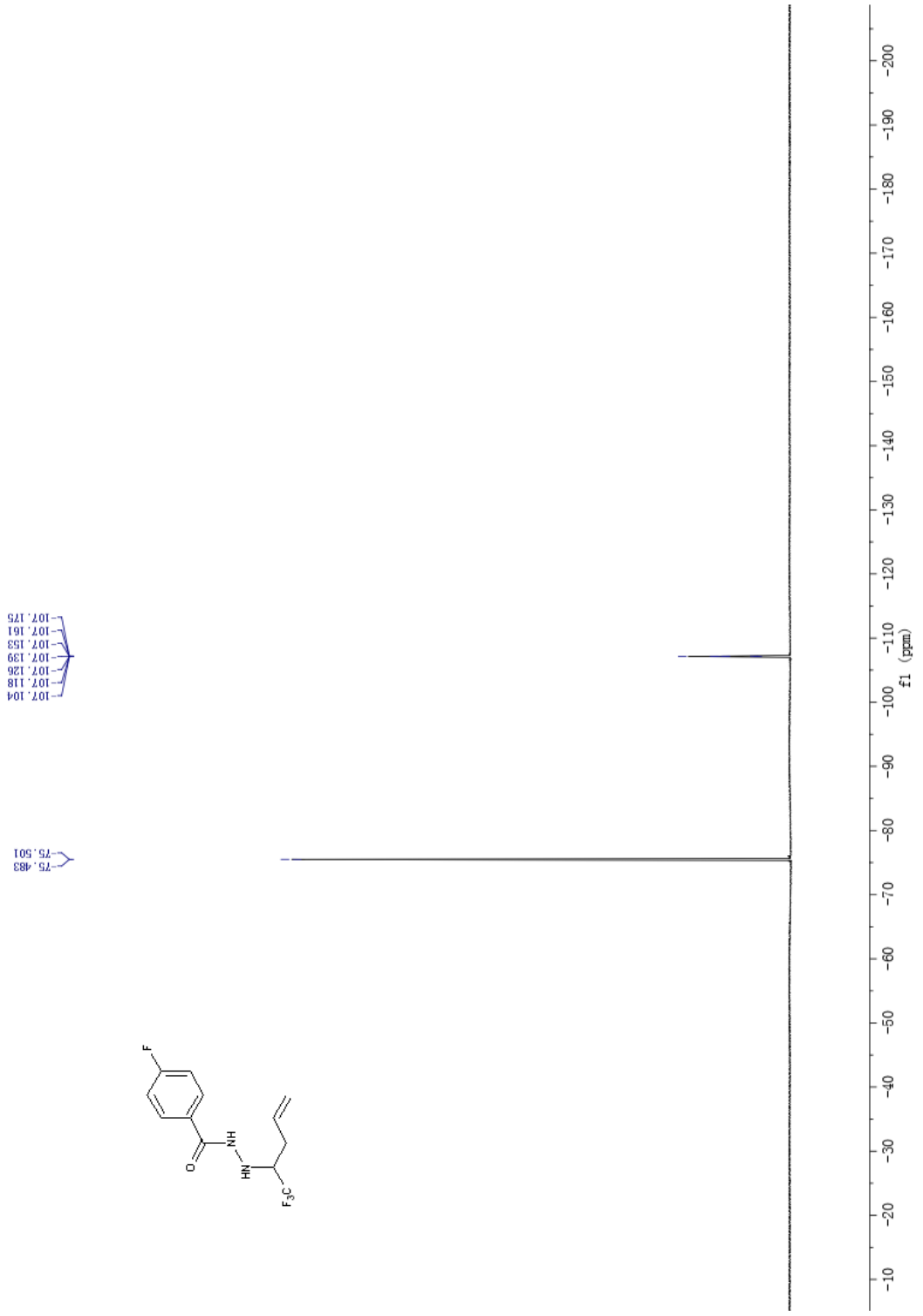

F:\Users\...\duganggang-3_150923160933
9/24/2015 9:29:13 AM
Error=1.8 ppm
DGG-811
273.1214
295.1033
274.1247
296.1066
301.1414
311.0773
245.0788
252.6780
261.1314
270.2466
279.1595
292.0951
312.0809
318.3007
327.2273
344.2285
349.1843
353.2667
Relative Abundance
NL:
3.03E5
duganggang-3_150923160933#12 RT: 0.12 AV: 1 T: FTMS + p ESI Full ms [100.00-2000.00]
273.1209
274.1243
275.1276
NL:
8.59E5
C13H15F3N2O1 +H
C13H16F3N2O1
pa Chrg 1
m/z

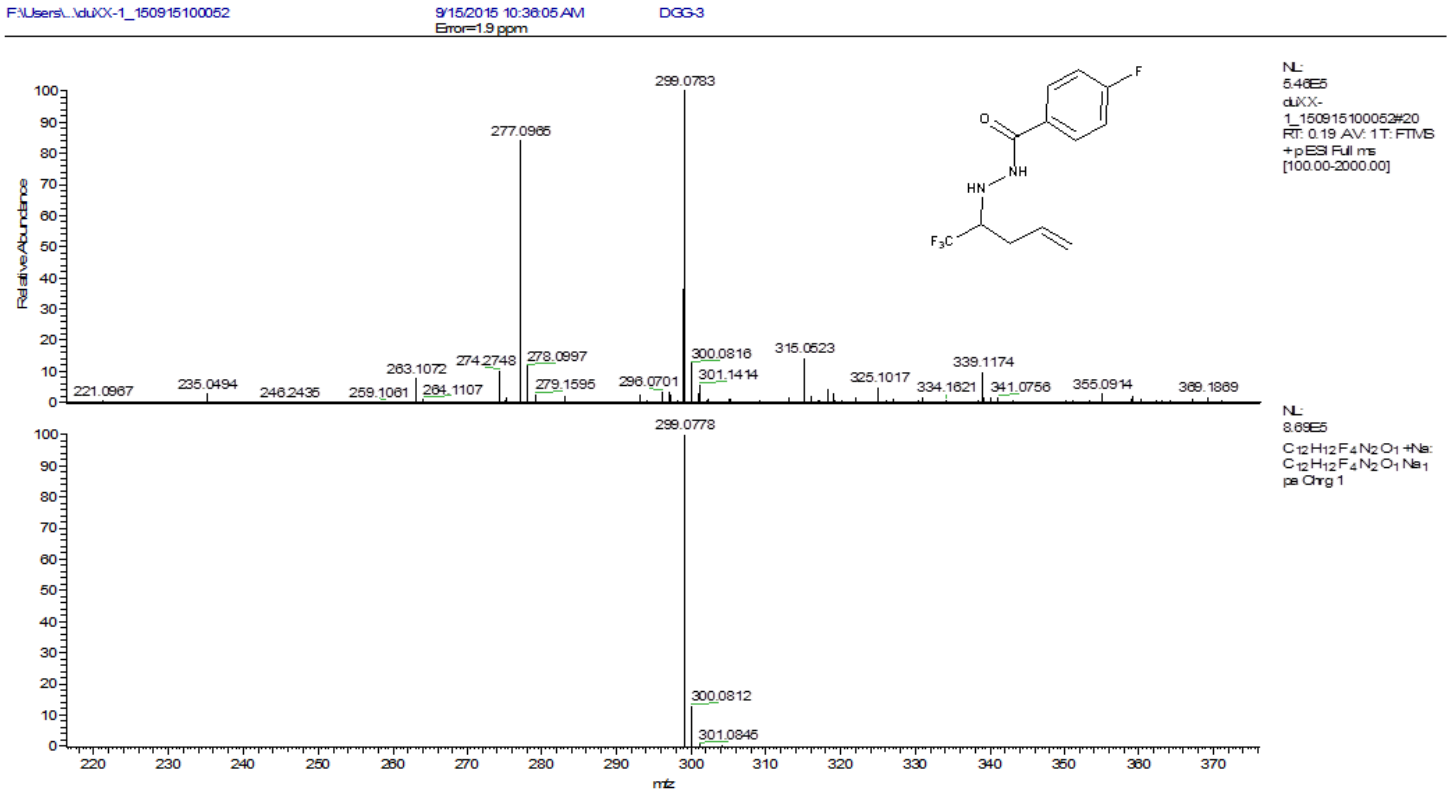
4-fluoro-N'-(1,1,1-trifluoropent-4-en-2-yl)benzohydrazide(4f)



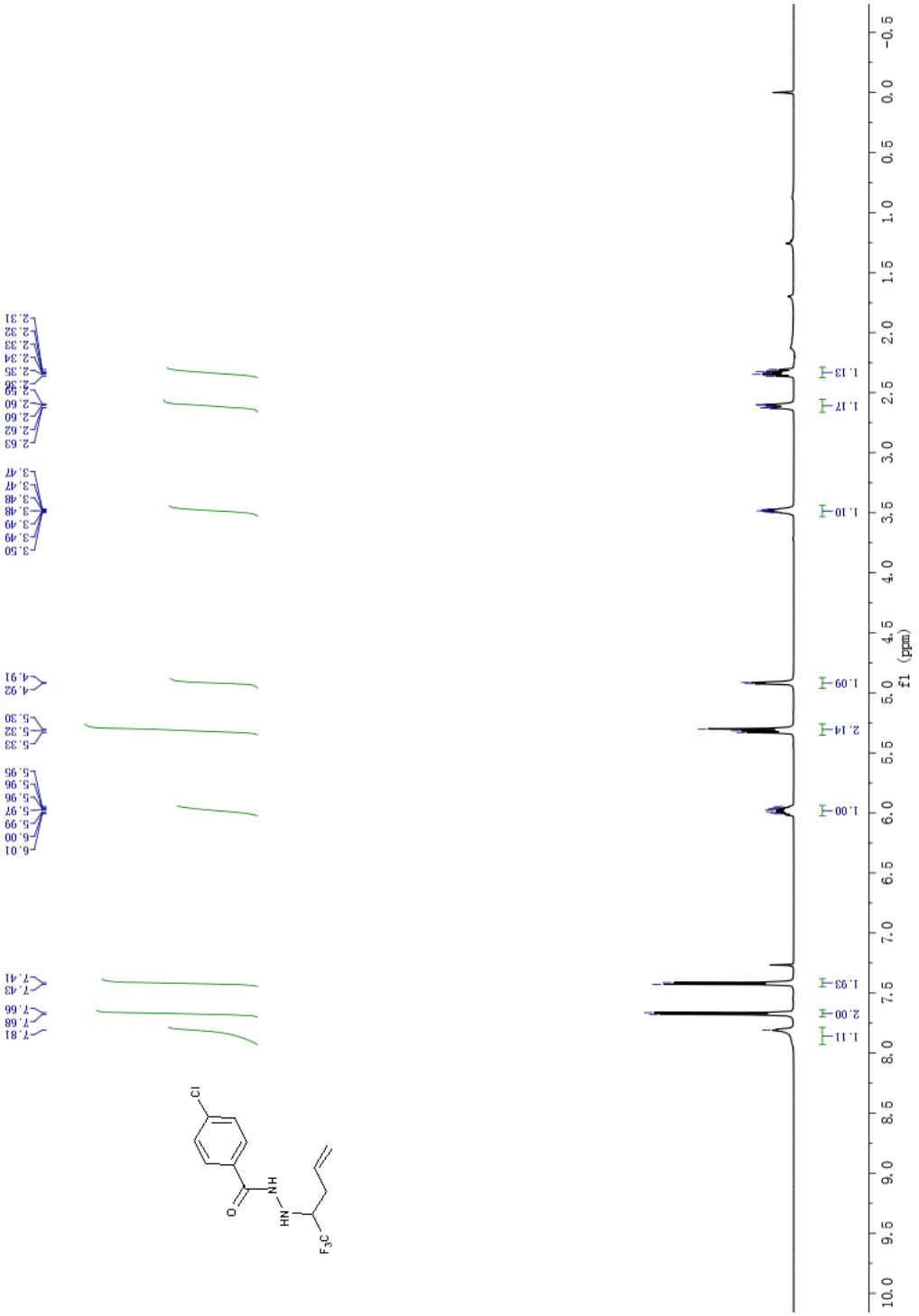

31.476
31.462
60.889
60.710
60.532
60.351
115.848
115.994
120.118
123.221
125.085
126.949
128.315
128.336
128.811
129.259
129.319
131.948
164.271
165.950
166.396
F
O
NH
HN
F3C
f1 (ppm)

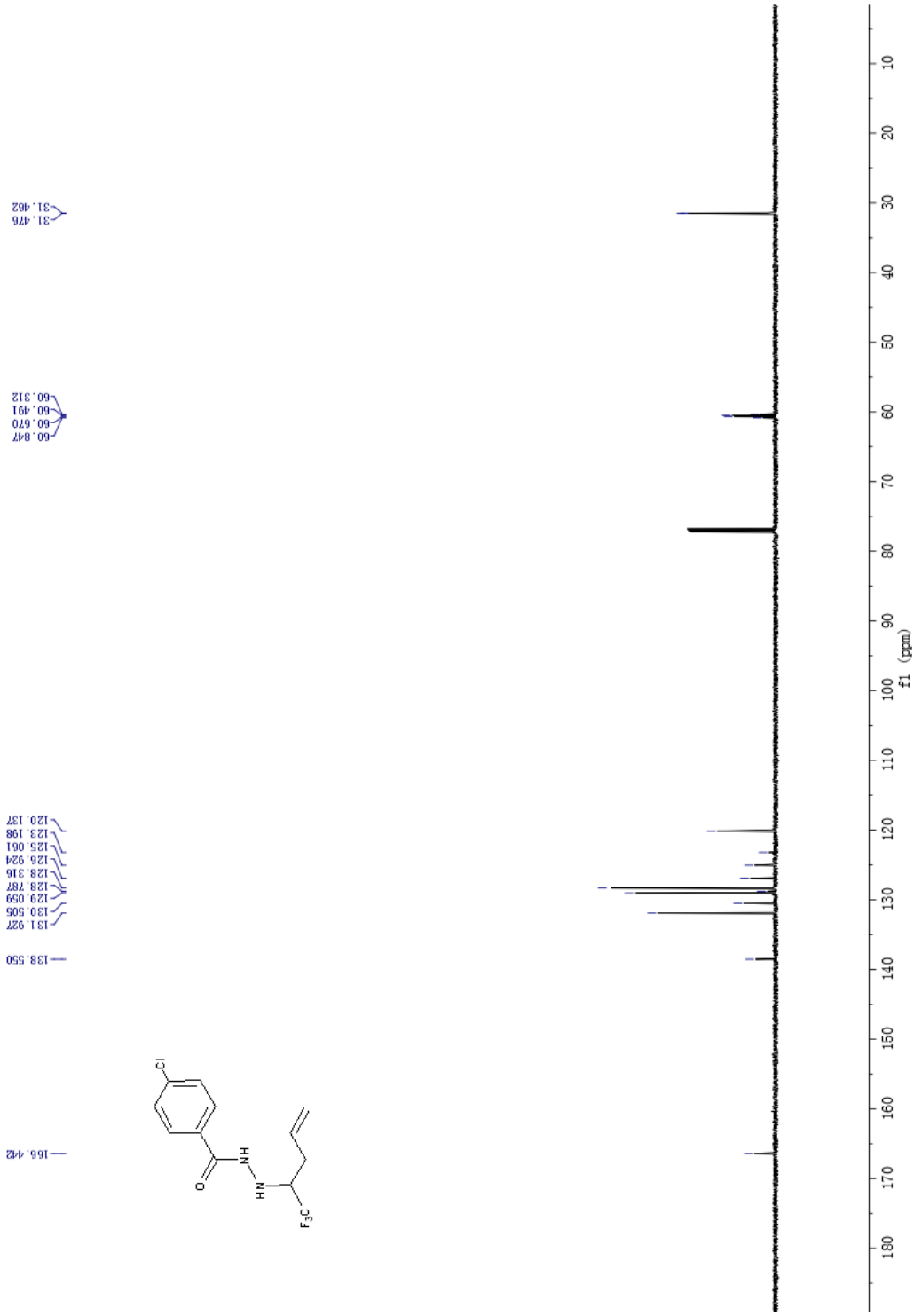

115.848
115.994
120.113
123.221
125.085
126.949
128.315
128.336
128.811
129.259
129.319
131.943
164.271
165.950
166.396
F
O
NH
HN
F3C
f1 (ppm)

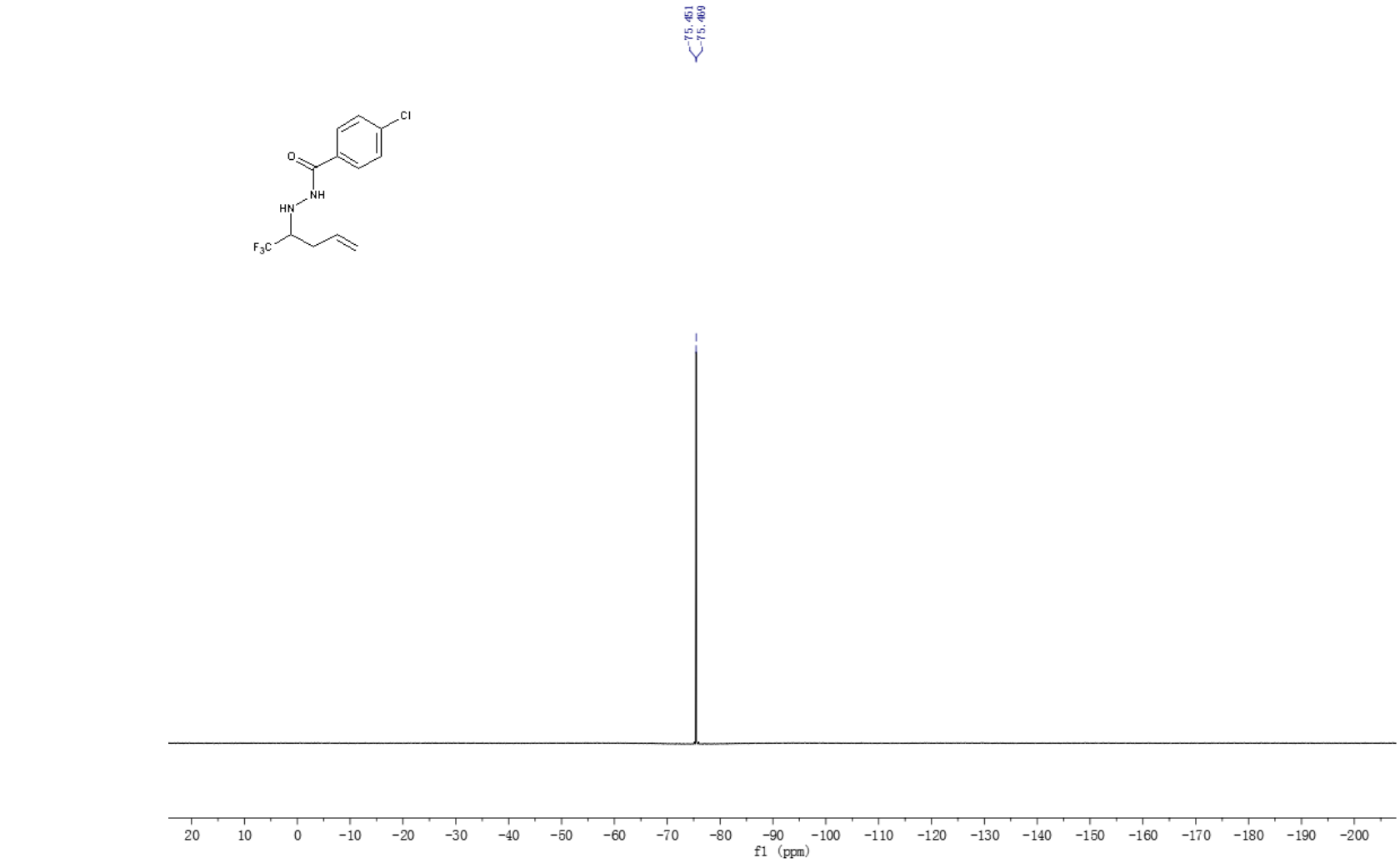

-107.175
-107.161
-107.153
-107.139
-107.126
-107.118
-107.104
-75.483
-75.501
F
O
NH
HN
F3C
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190
-200
f1 (ppm)

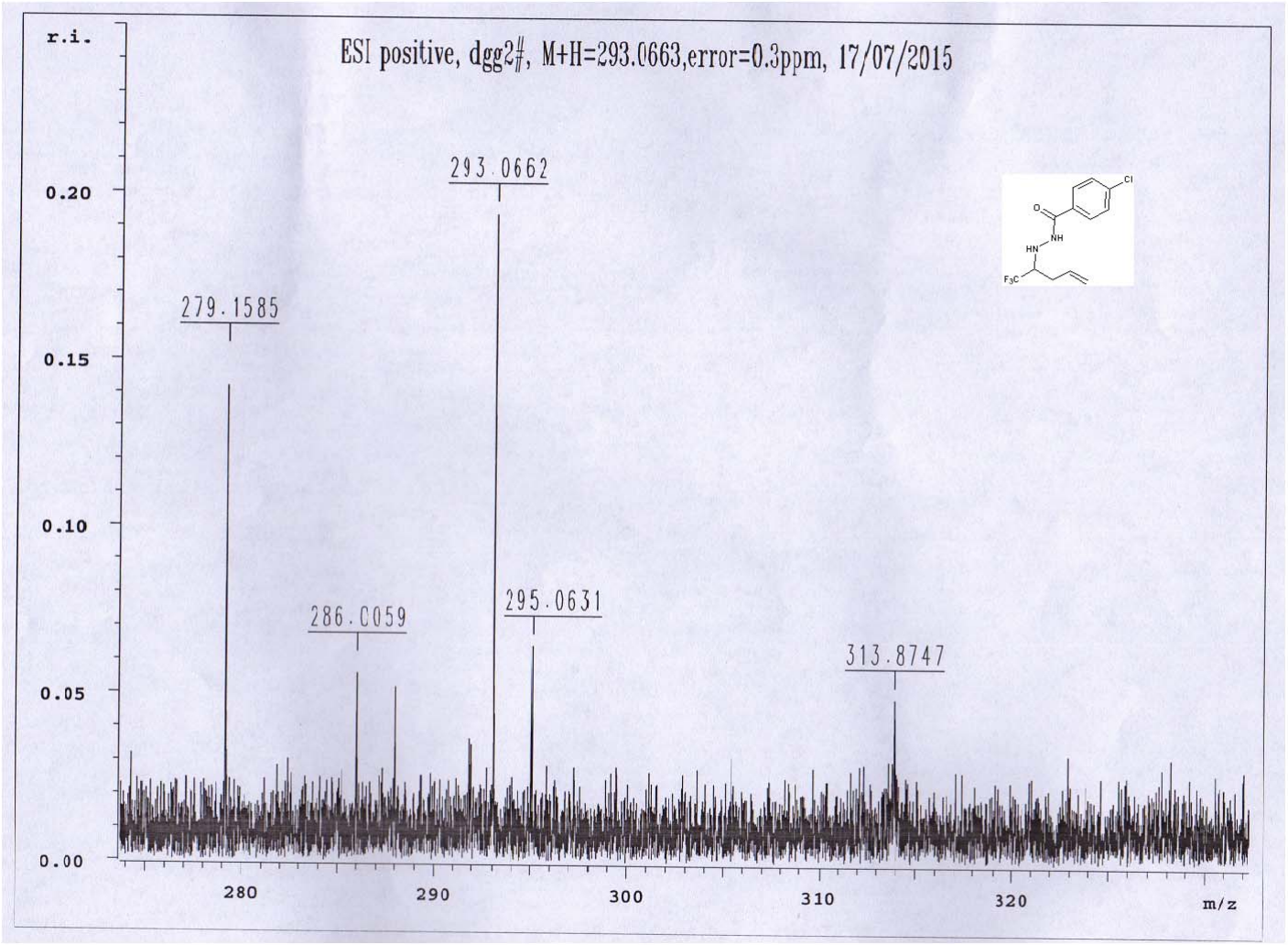

F:\Users\...\duXX-1_150915100052
9/15/2015 10:36:05 AM
Error=1.9 ppm
DGG-3
Relative Abundance
m/z
299.0783
277.0965
300.0816
315.0523
339.1174
263.1072
274.2748
278.0997
301.1414
325.1017
334.1621
341.0756
355.0914
369.1869
221.0967
235.0494
246.2435
259.1061
264.1107
279.1595
296.0701
299.0778
300.0812
301.0845
NL:
5.46E5
duXX-
1_150915100052#20
RT: 0.19 AV: 1 T: FTMS
+ p ESI Full ms
[100.00-2000.00]
NL:
8.69E5
C12H12F4N2O1+Na:
C12H12F4N2O1Na1
pa Chrg 1

4-chloro-N'-(1,1,1-trifluoropent-4-en-2-yl)benzohydrazide(4g)

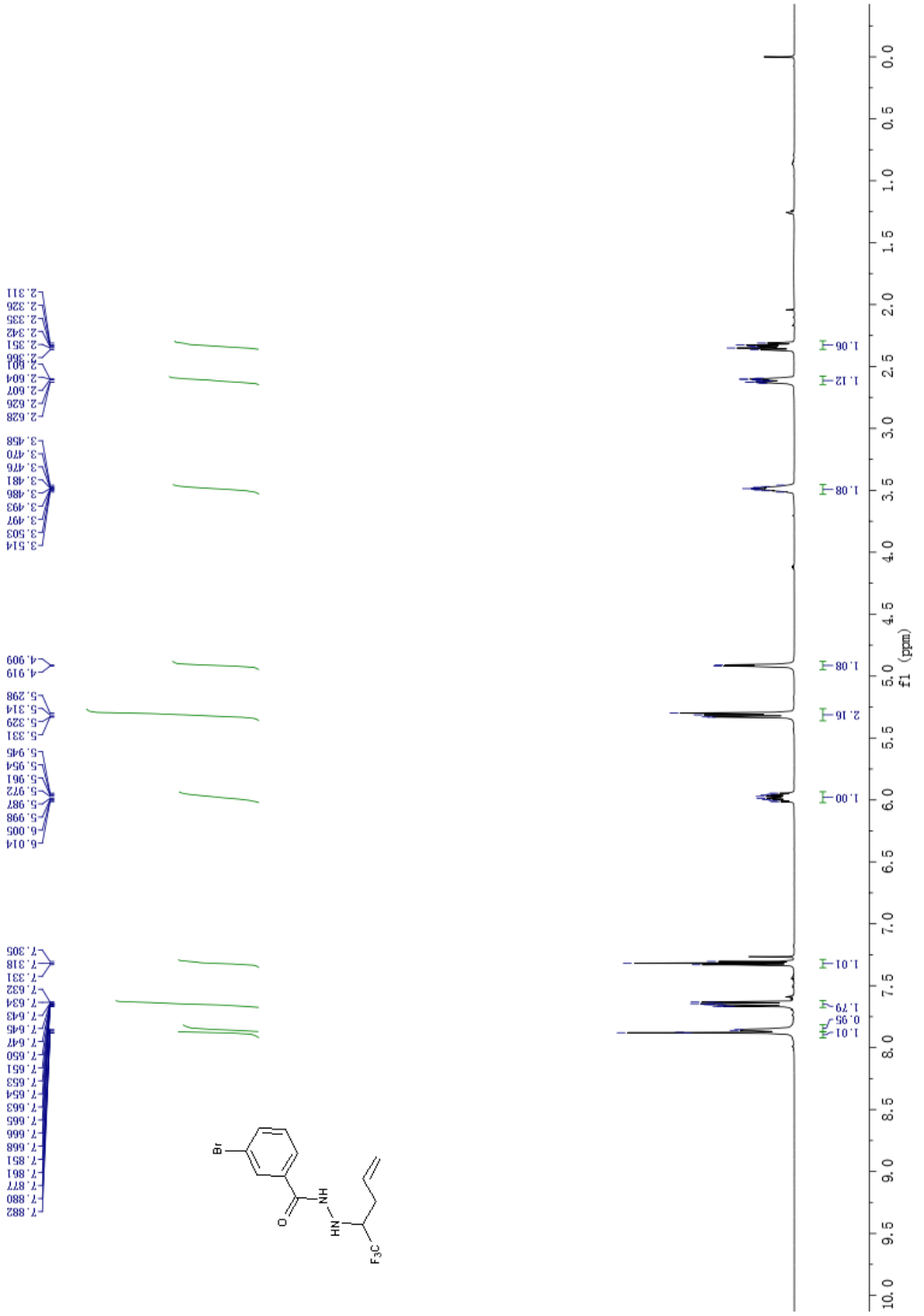


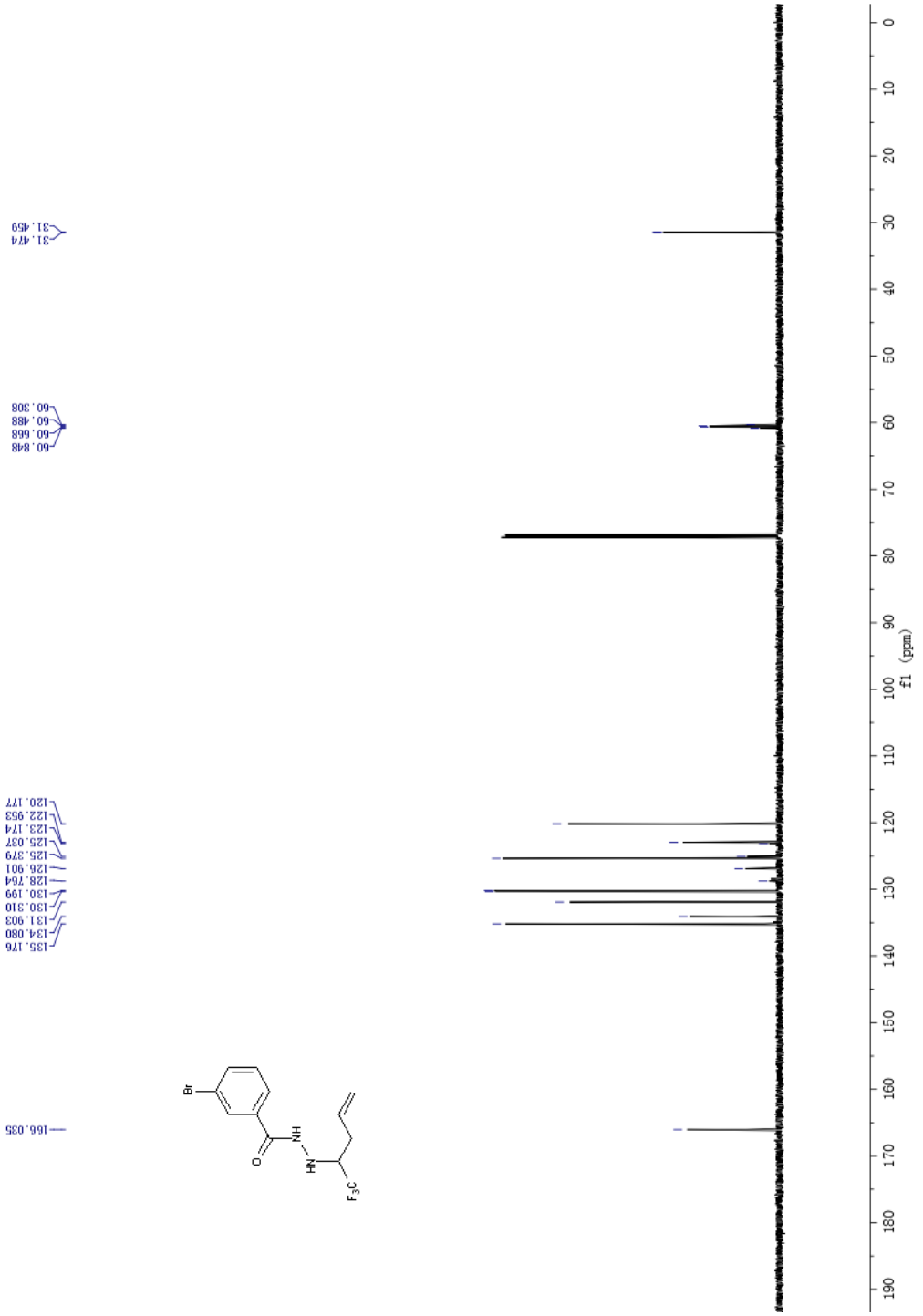

31.476
31.462
60.847
60.670
60.491
60.312
166.442
138.550
131.927
130.505
129.059
128.787
128.316
126.924
125.061
123.198
120.137
Cl
O
NH
HN
F3C
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
160
170
180
f1 (ppm)

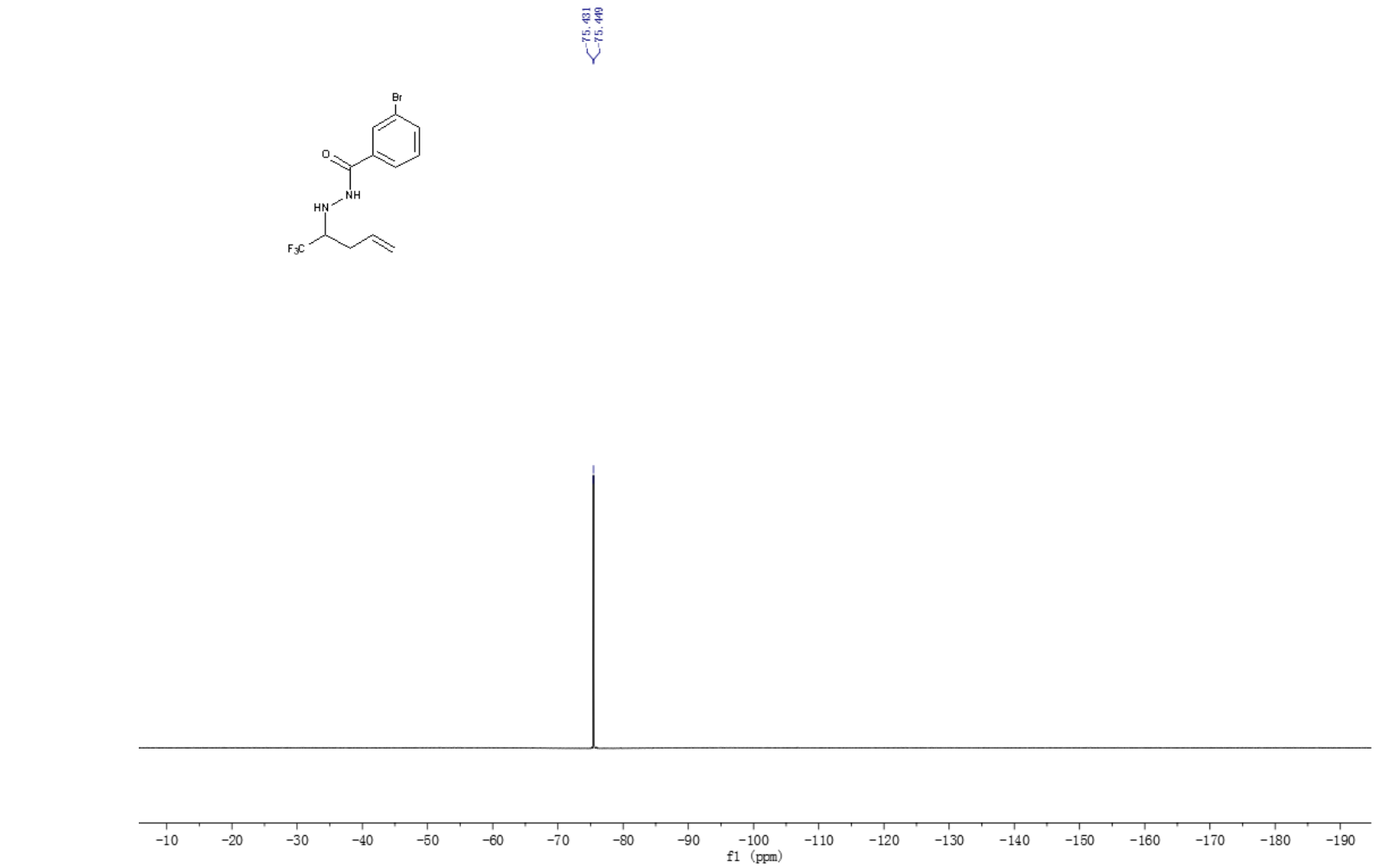

-75.451
-75.469
Cl
O
NH
HN
F3C
20
10
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190
-200
f1 (ppm)

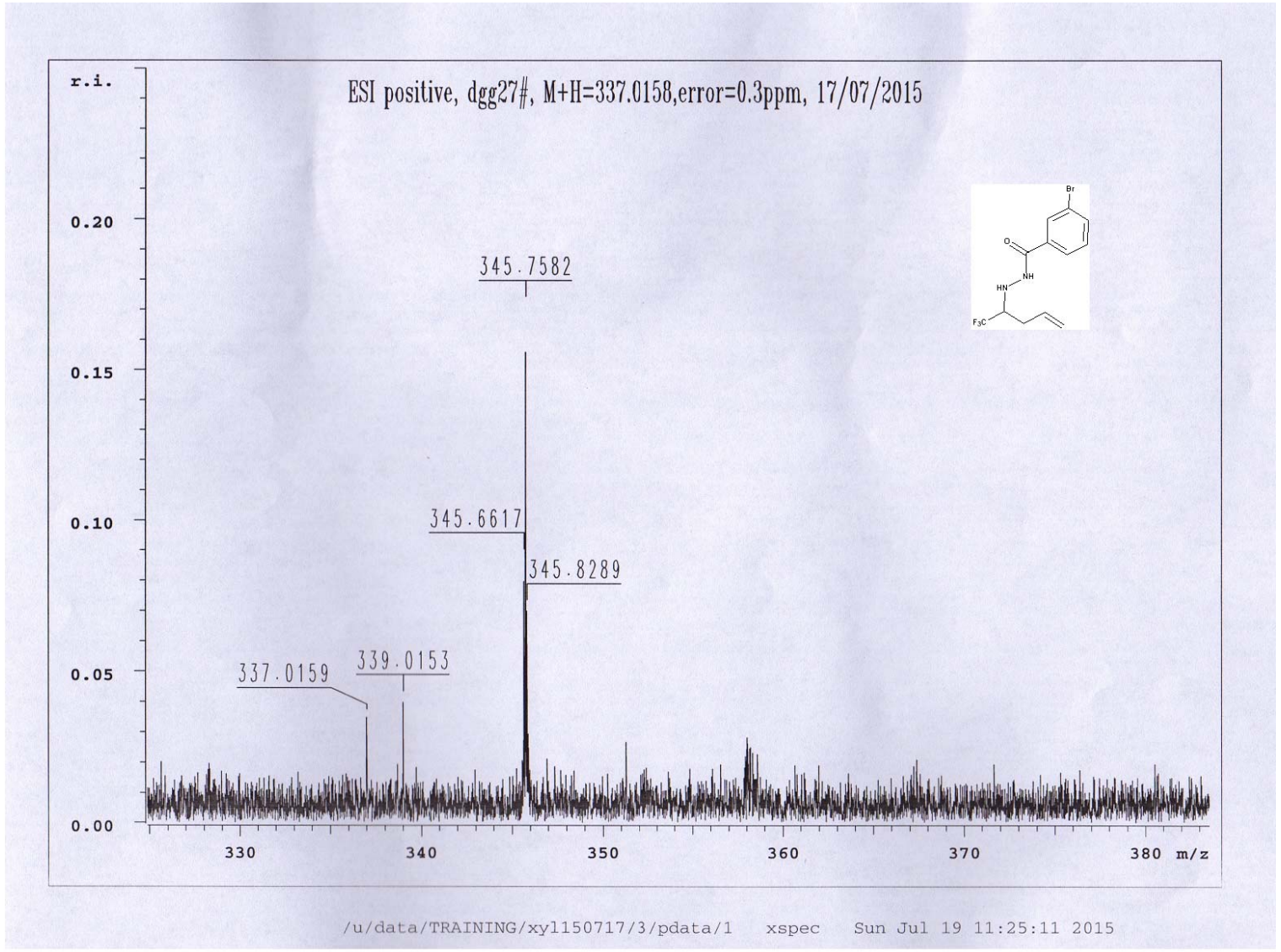

ESI positive, dgg2#, M+H=293.0663,error=0.3ppm, 17/07/2015
r.i.
0.20
0.15
0.10
0.05
0.00
293.0662
279.1585
286.0059
295.0631
313.8747
280
290
300
310
320
m/z
Cl
O
NH
HN
F3C

3-bromo-N'-(1,1,1-trifluoropent-4-en-2-yl)benzohydrazide(4h)

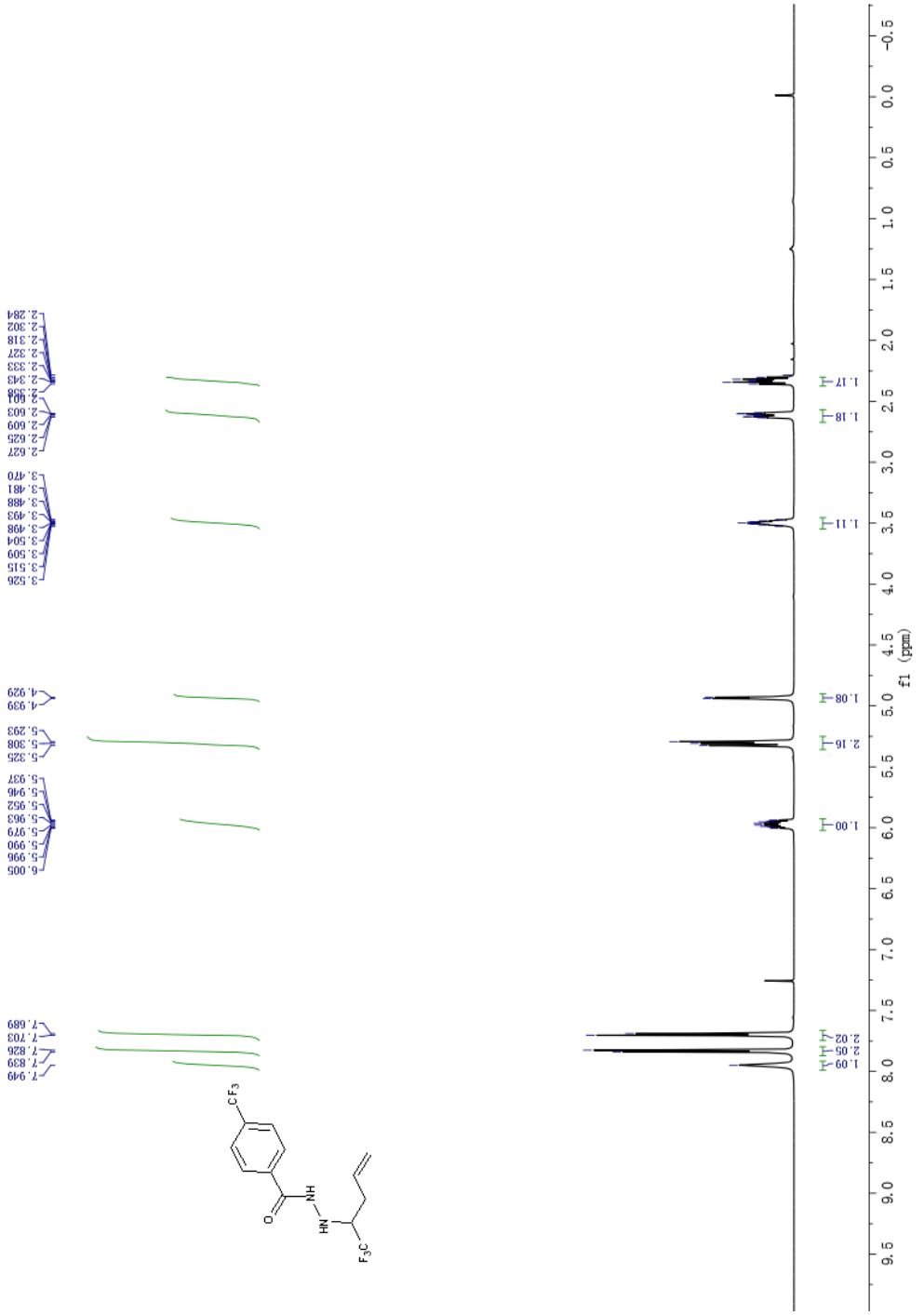


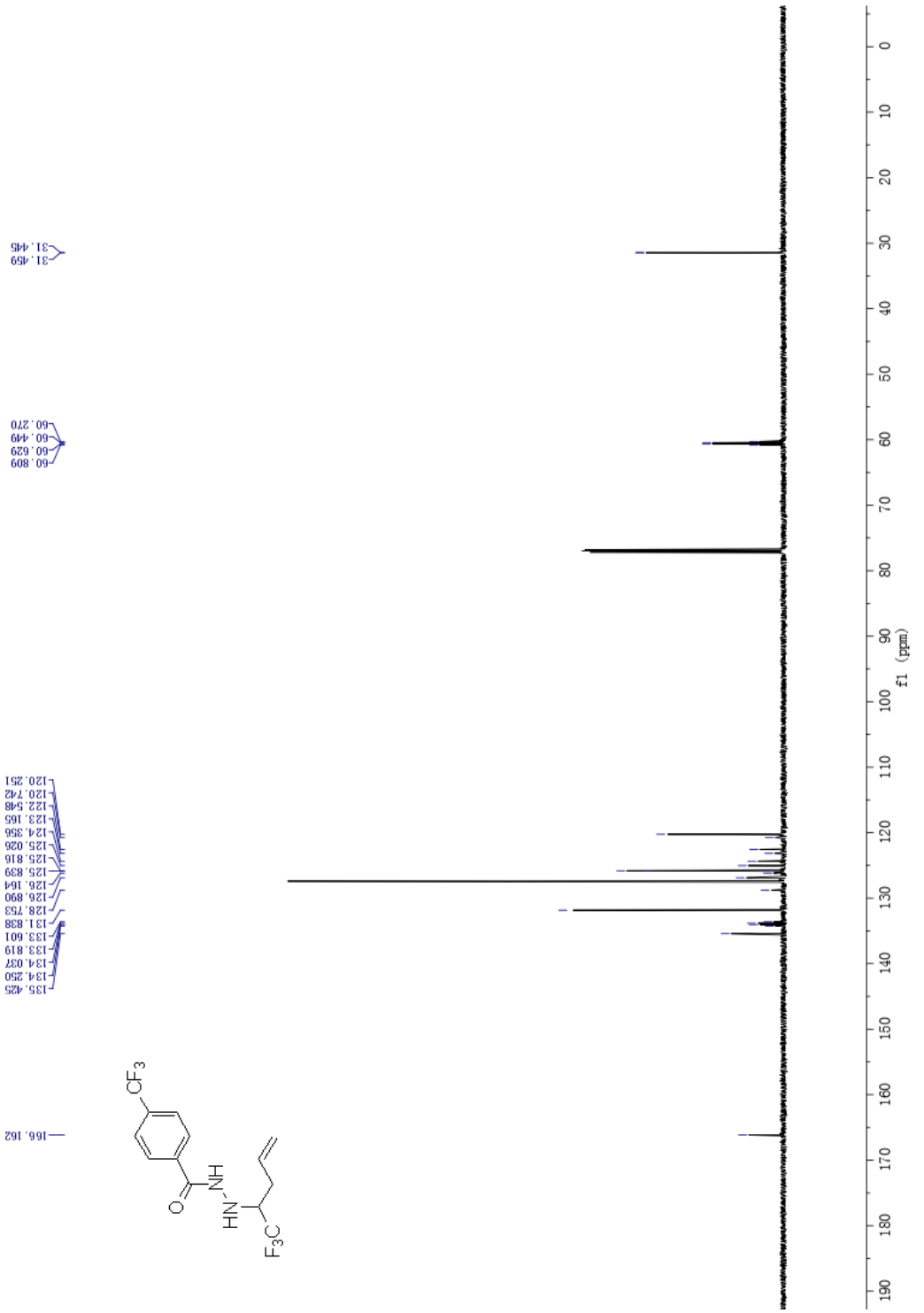

31.474
31.459
60.848
60.668
60.488
60.308
166.035
135.176
134.080
131.903
130.310
130.199
128.764
126.901
125.379
125.037
123.174
122.953
120.177
Br
O
NH
HN
F3C
0
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
160
170
180
190
f1 (ppm)

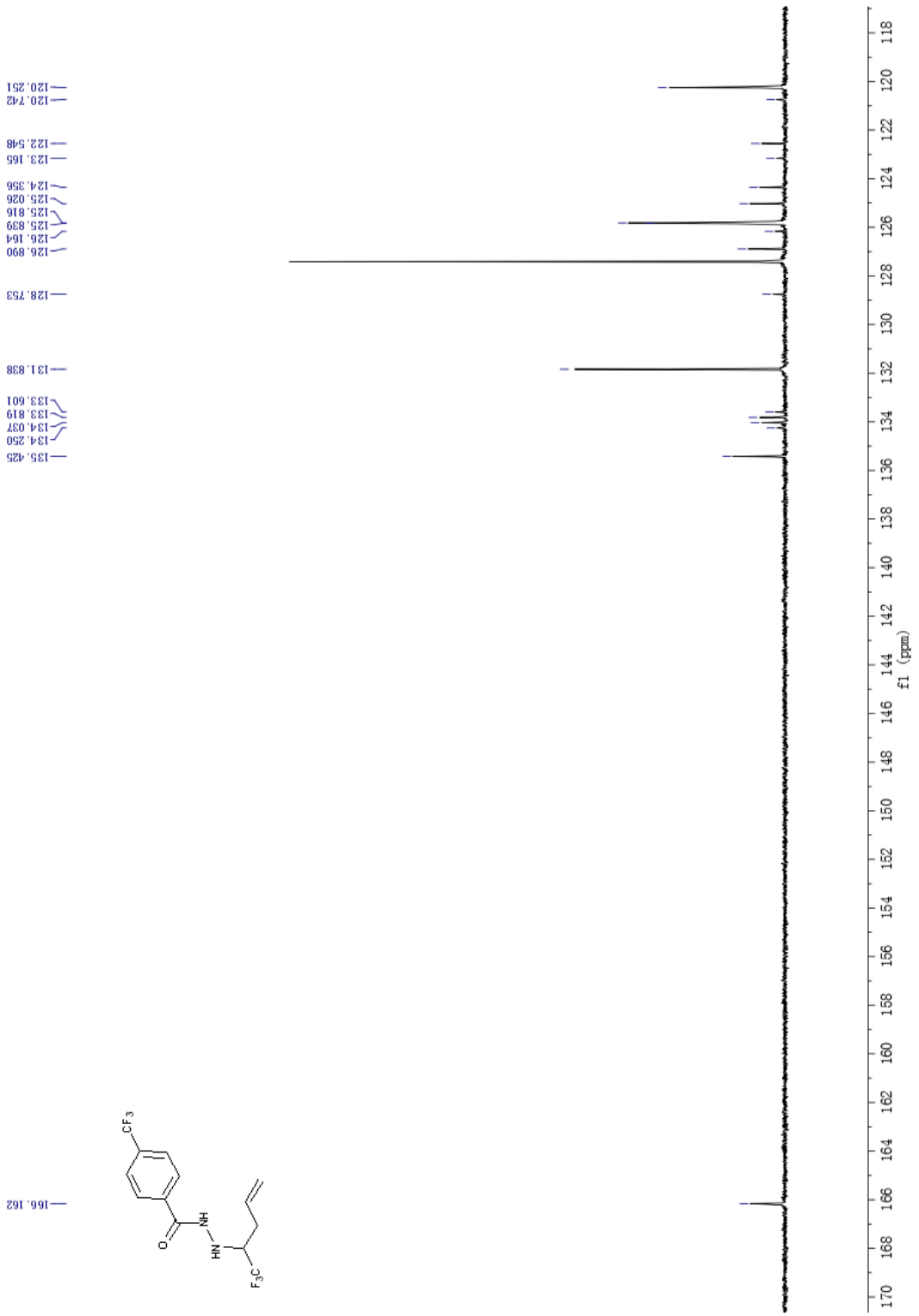

-75.431
-75.449
Br
O
NH
HN
F3C
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190
f1 (ppm)

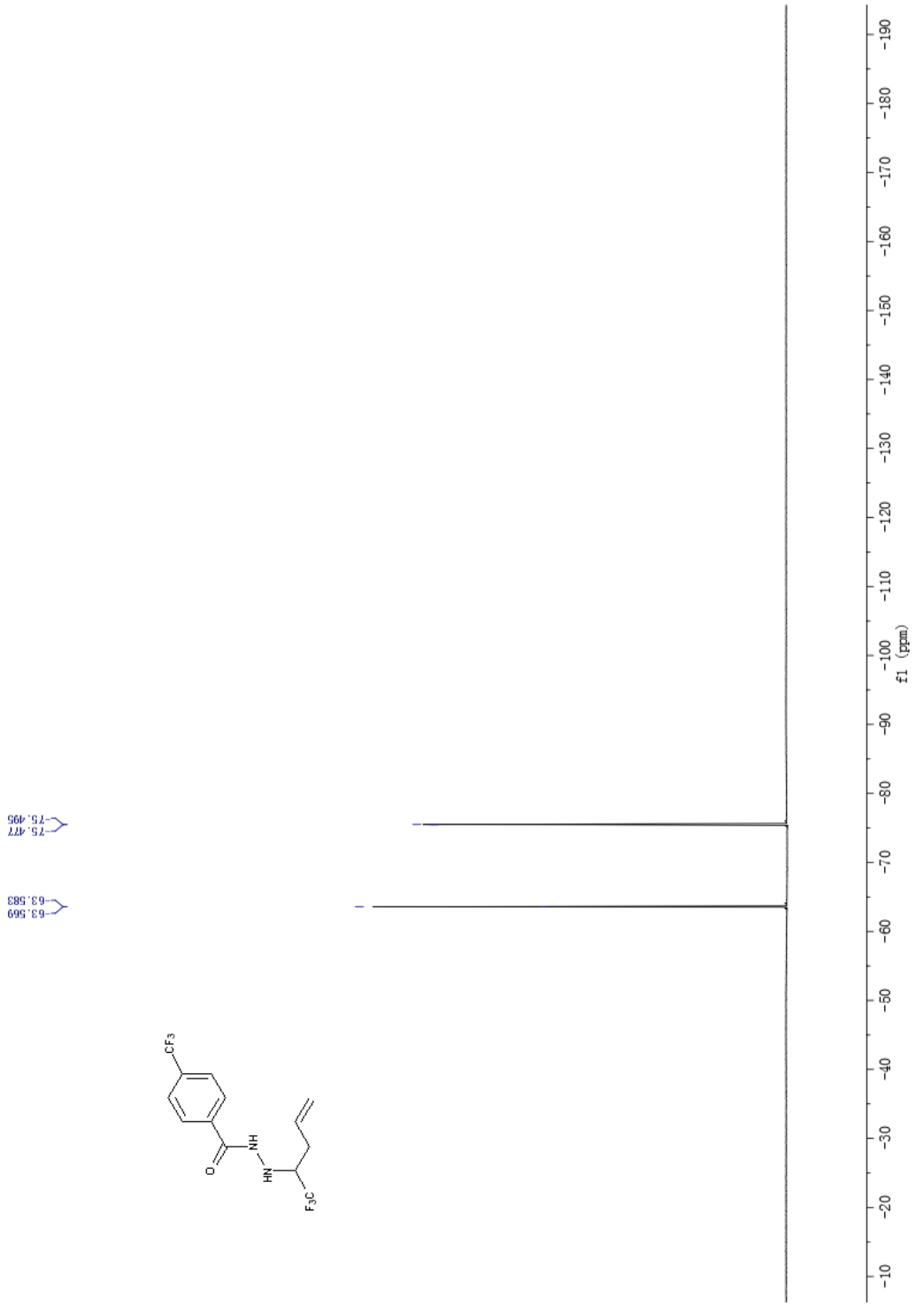

ESI positive, dgg27#, M+H=337.0158,error=0.3ppm, 17/07/2015
r.i.
0.20
0.15
0.10
0.05
0.00
345.7582
345.6617
345.8289
337.0159
339.0153
330
340
350
360
370
380 m/z
Br
O
NH
HN
F3C
/u/data/TRAINING/xyl150717/3/pdata/1 xspec Sun Jul 19 11:25:11 2015

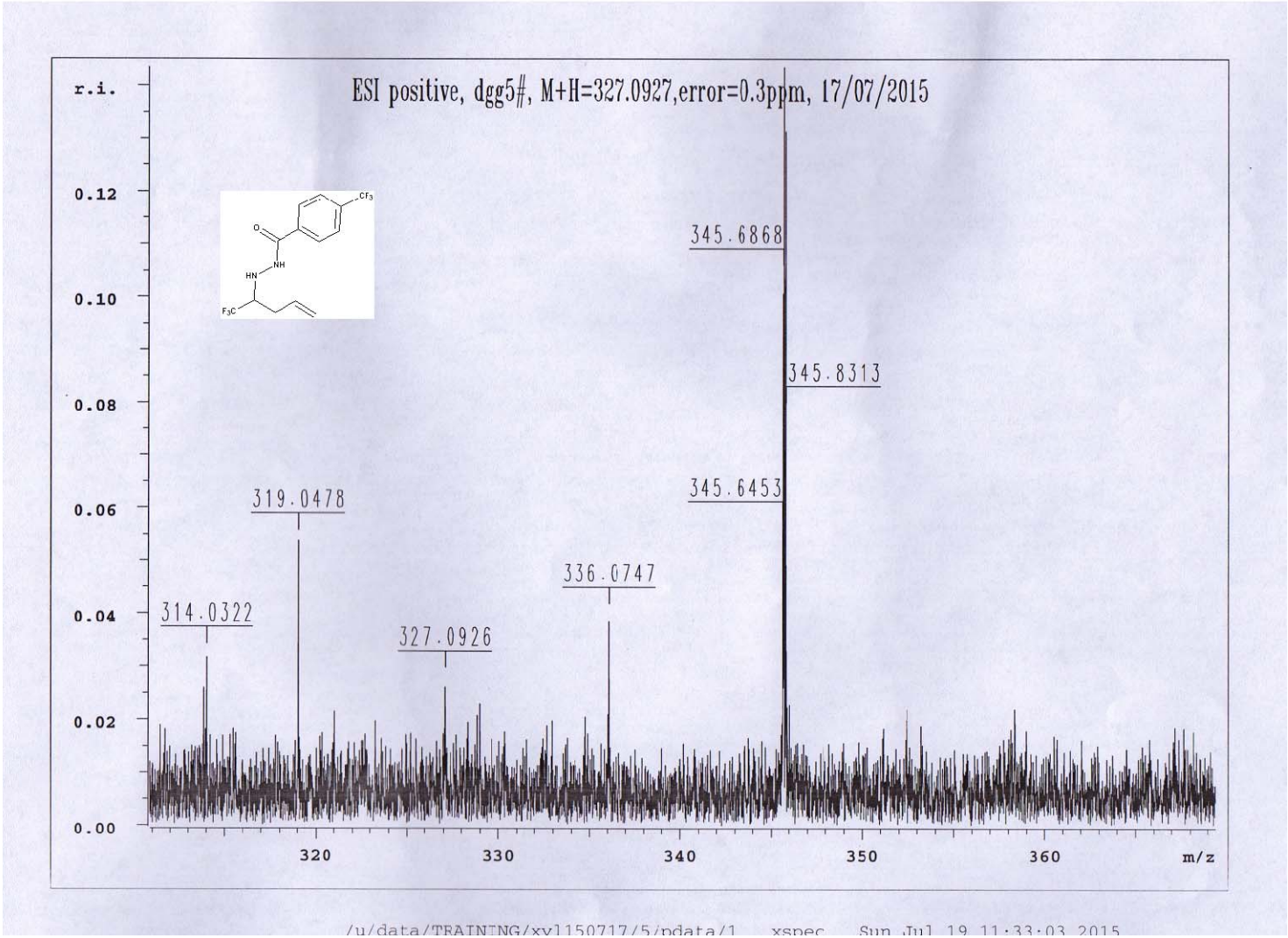
4-(trifluoromethyl)-N'-(1,1,1-trifluoropent-4-en-2-yl)benzohydrazide(4i)



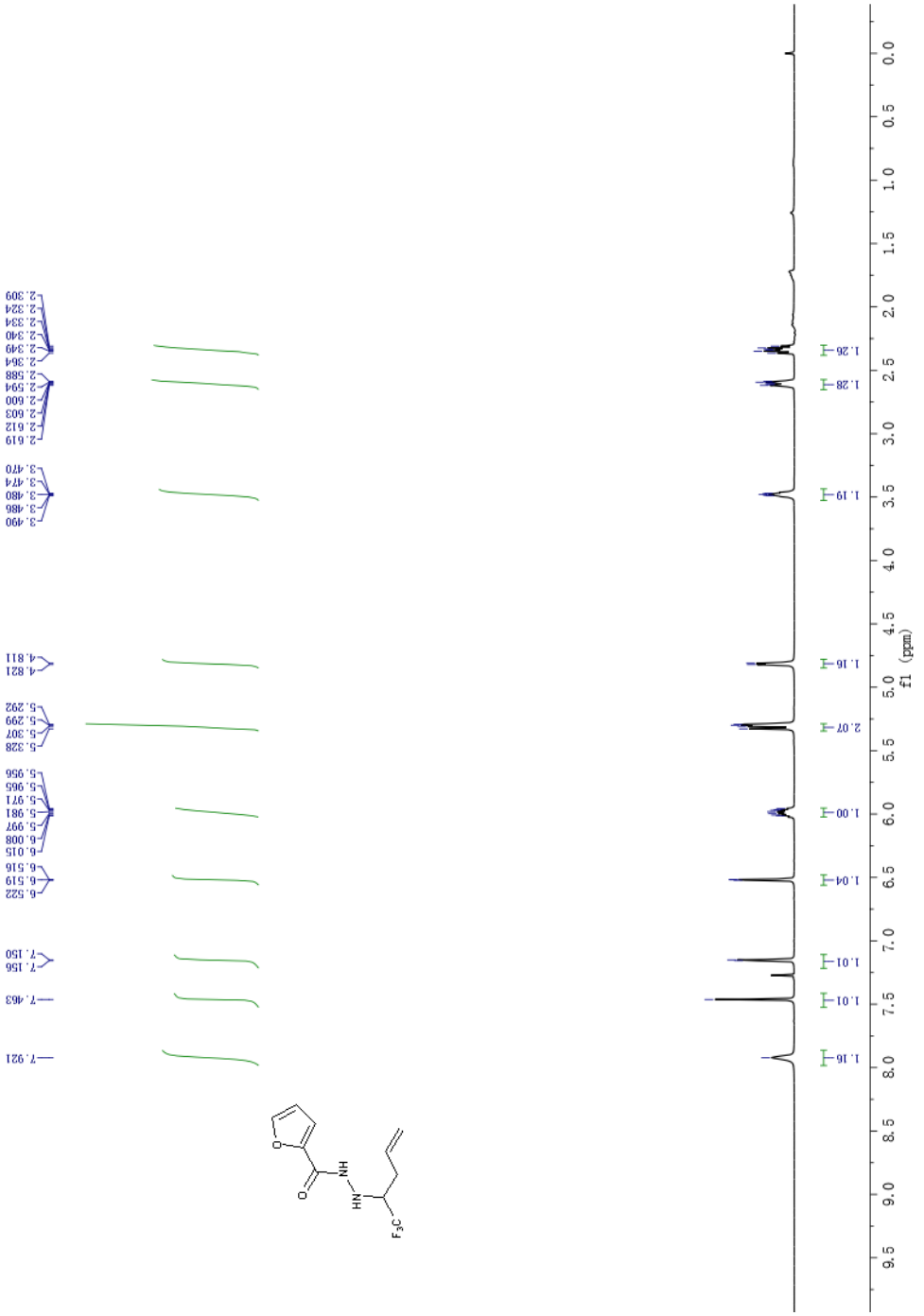

166.162
135.425
134.250
134.037
133.819
133.601
131.838
128.753
126.890
126.164
125.839
125.816
125.026
124.356
123.165
122.548
120.742
120.251
60.809
60.629
60.449
60.270
31.459
31.445
CF3
O
HN
NH
F3C
f1 (ppm)

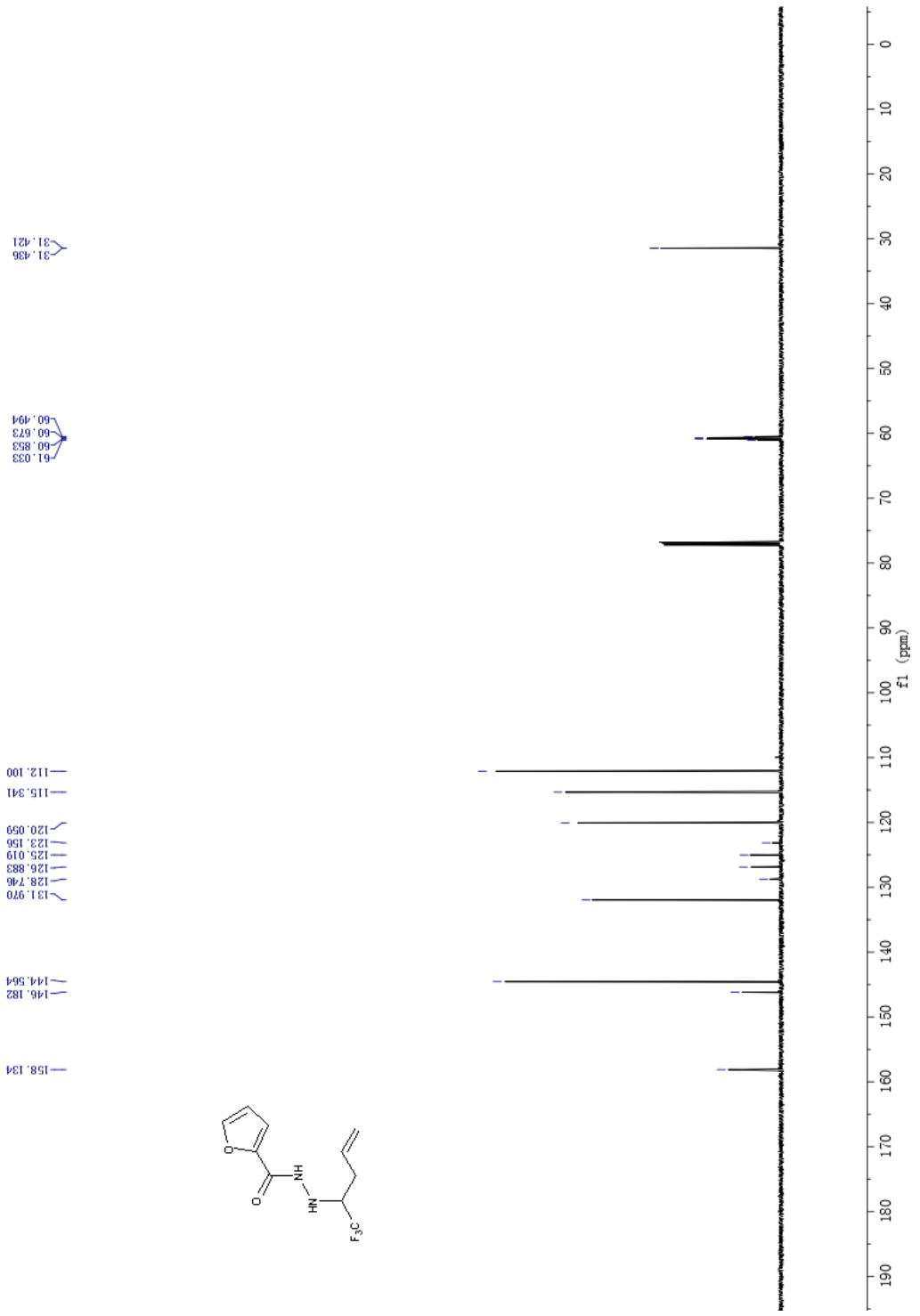

120.251
120.742
122.548
123.165
124.356
125.026
125.816
125.839
126.164
126.890
128.753
131.838
133.601
133.819
134.037
134.250
135.425
166.162
CF3
O
NH
HN
F3C
118
120
122
124
126
128
130
132
134
136
138
140
142
144
146
148
150
152
154
156
158
160
162
164
166
168
170
f1 (ppm)

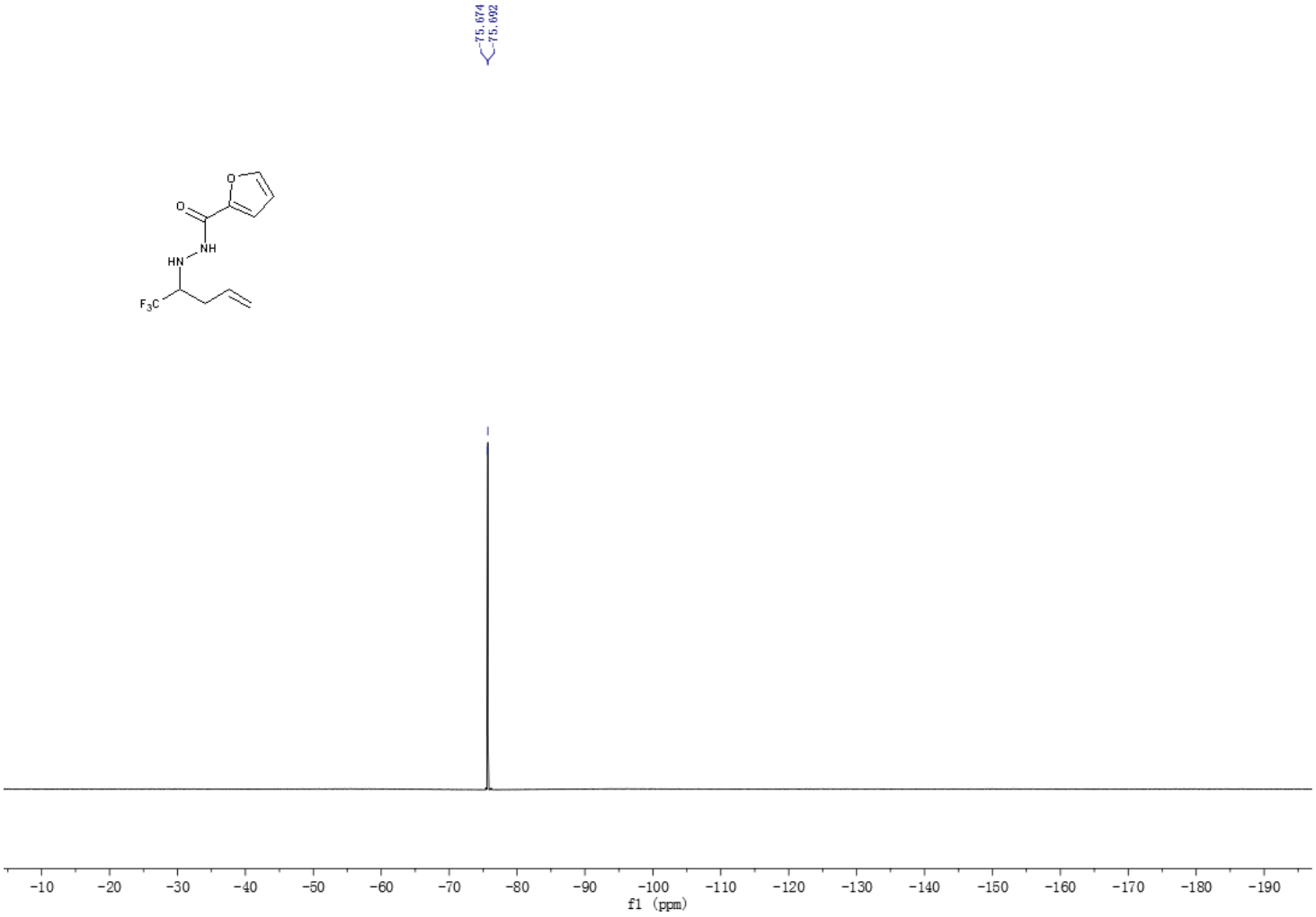

-63.569
-63.583
-75.477
-75.495
O
NH
HN
F3C
CF3
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190
f1 (ppm)

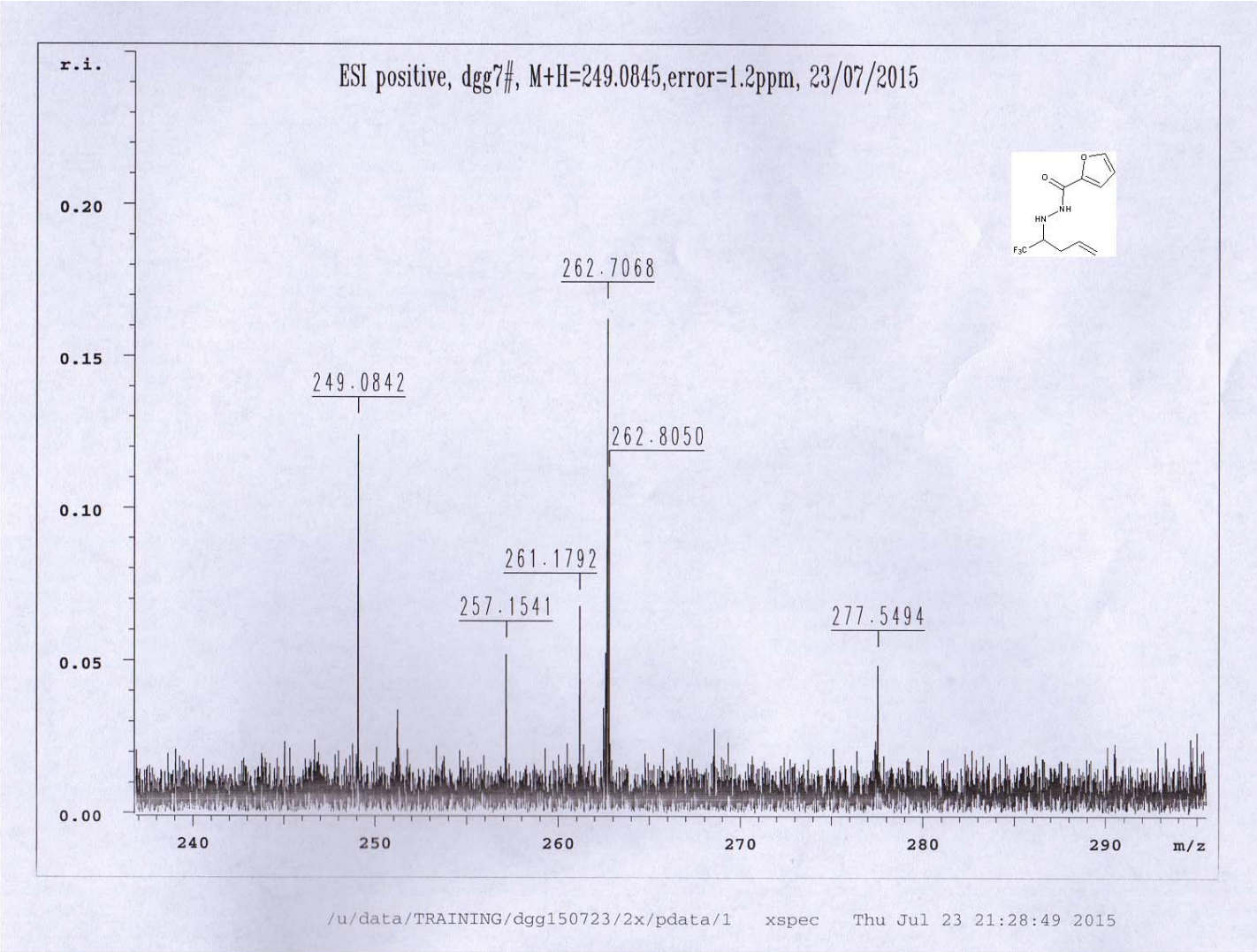

ESI positive, dgg5#, M+H=327.0927,error=0.3ppm, 17/07/2015
r.i.
0.12
0.10
0.08
0.06
0.04
0.02
0.00
345.6868
345.8313
345.6453
319.0478
336.0747
314.0322
327.0926
320
330
340
350
360
m/z
/u/data/TRAINING/xyl150717/5/pdata/1 xspec Sun Jul 19 11:33:03 2015

N'-(1,1,1-trifluoropent-4-en-2-yl)furan-2-carbohydrazide(4j)

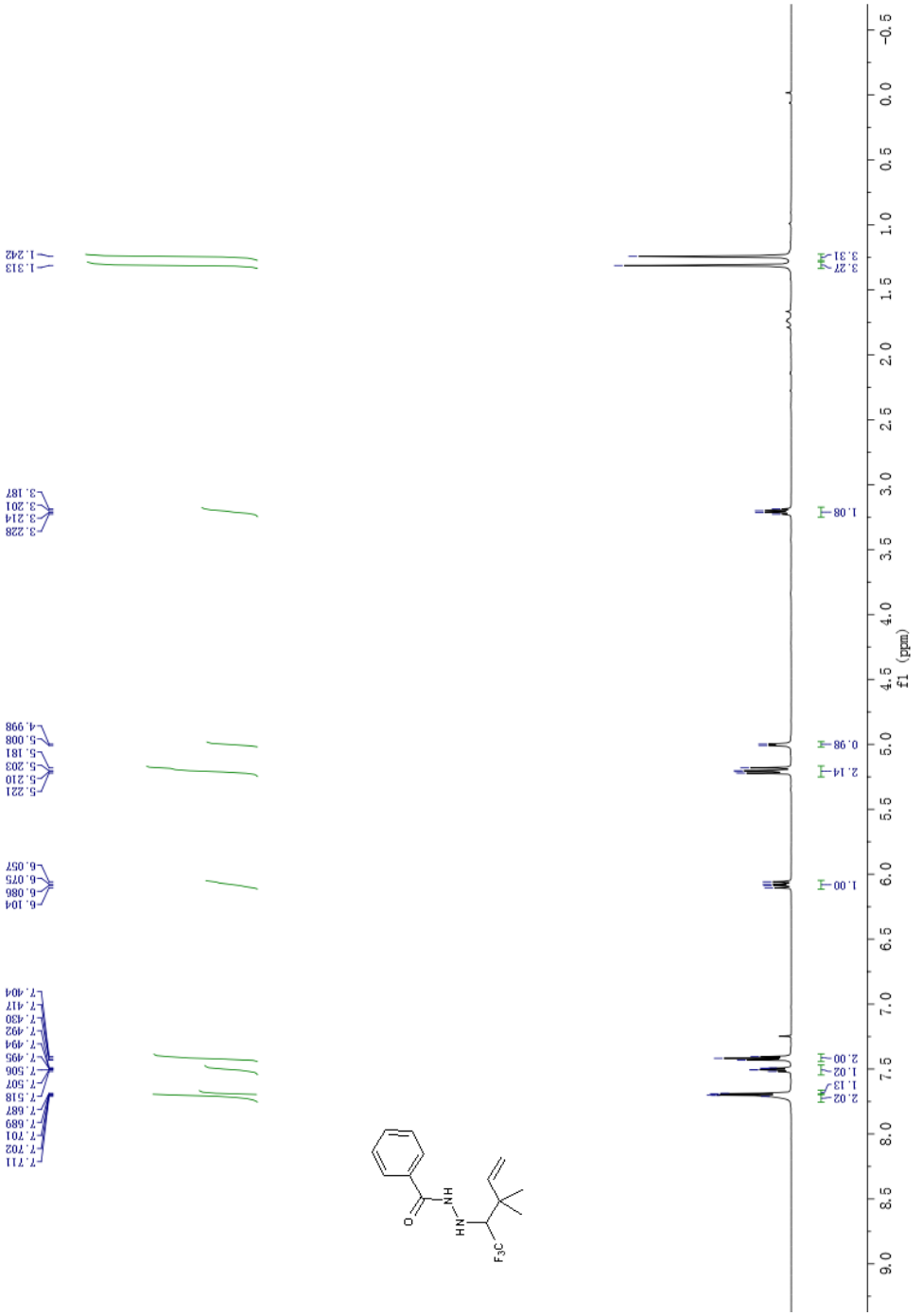


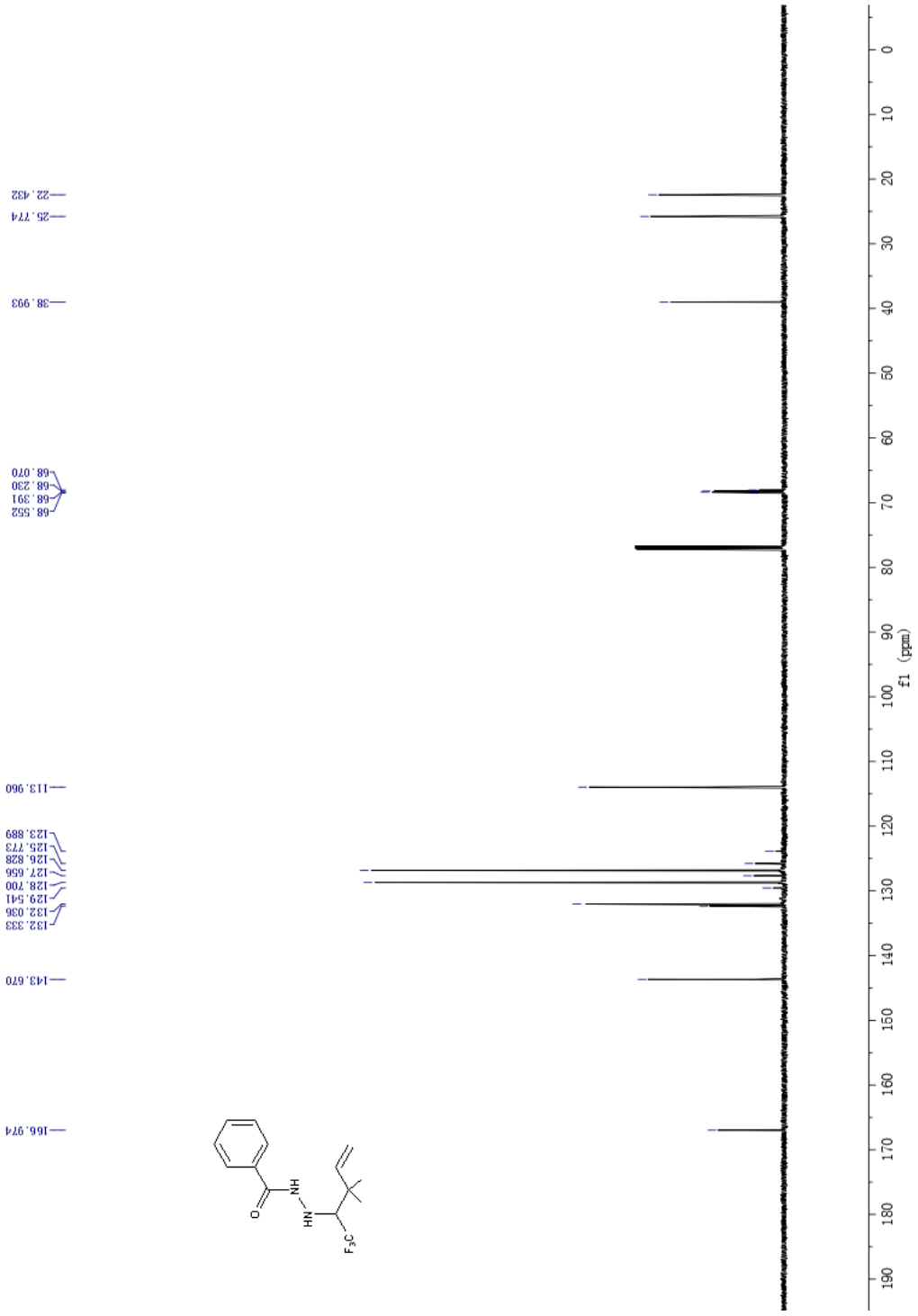

158.134
146.182
144.564
131.970
128.746
126.883
125.019
123.156
120.059
115.341
112.100
61.033
60.853
60.673
60.494
31.436
31.421
O
NH
HN
F3C
f1 (ppm)

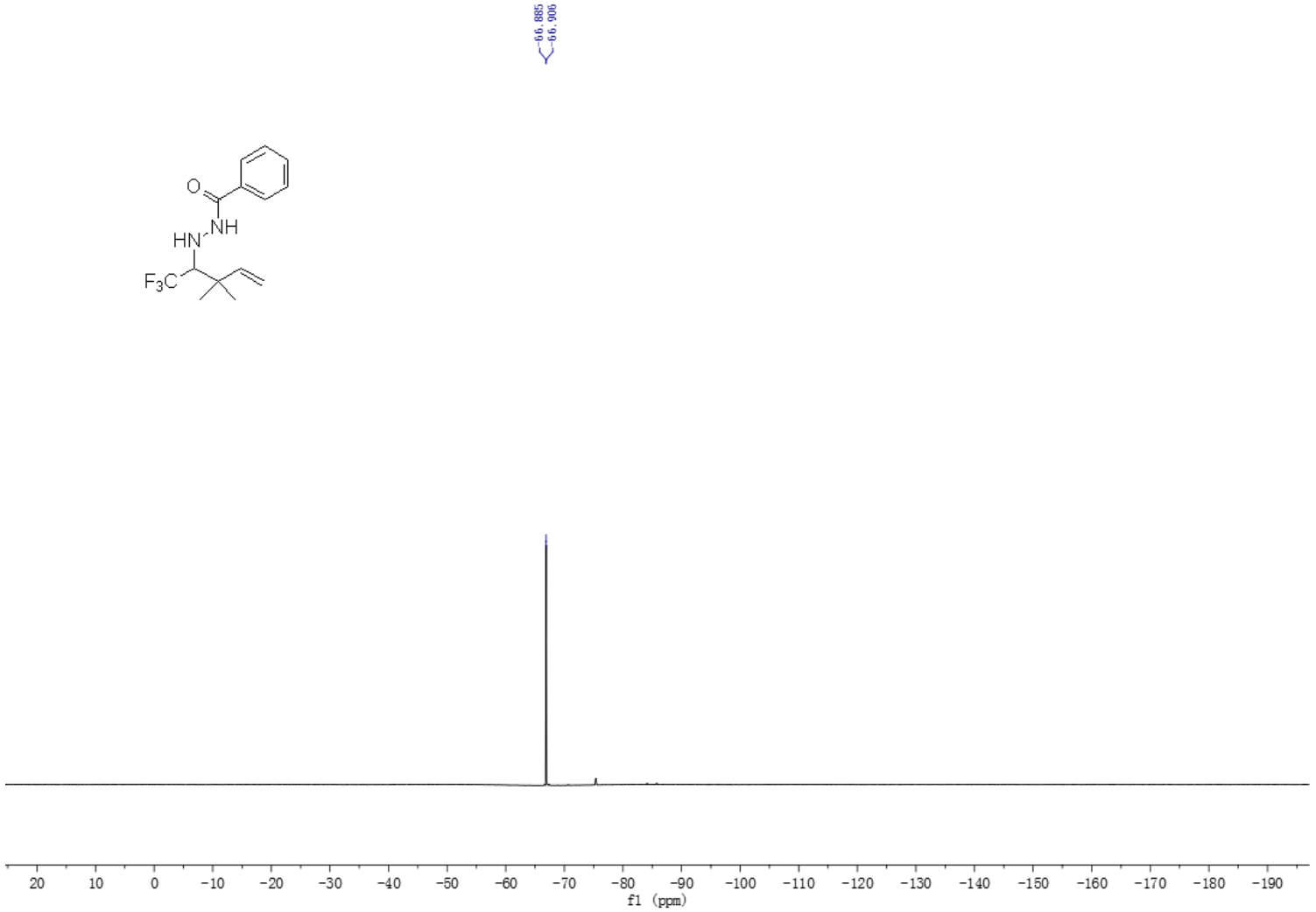

-75.674
-75.692
O
O
NH
HN
F3C
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190
f1 (ppm)

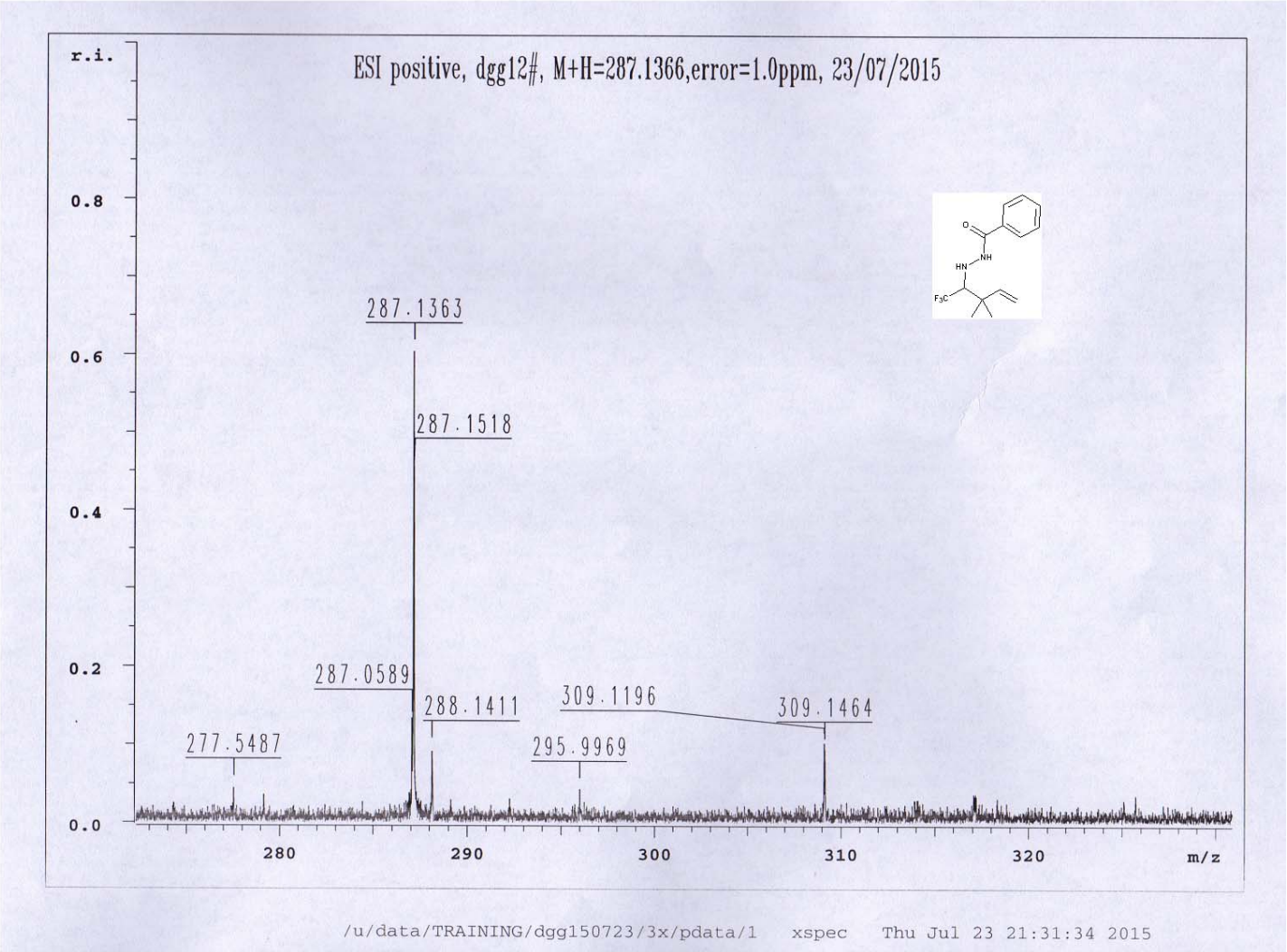

ESI positive, dgg7#, M+H=249.0845,error=1.2ppm, 23/07/2015
r.i.
0.20
0.15
0.10
0.05
0.00
262.7068
249.0842
262.8050
261.1792
257.1541
277.5494
240
250
260
270
280
290
m/z
/u/data/TRAINING/dgg150723/2x/pdata/1 xspec Thu Jul 23 21:28:49 2015

N'-(1,1,1-trifluoro-3,3-dimethylpent-4-en-2-yl)benzohydrazide(4k)

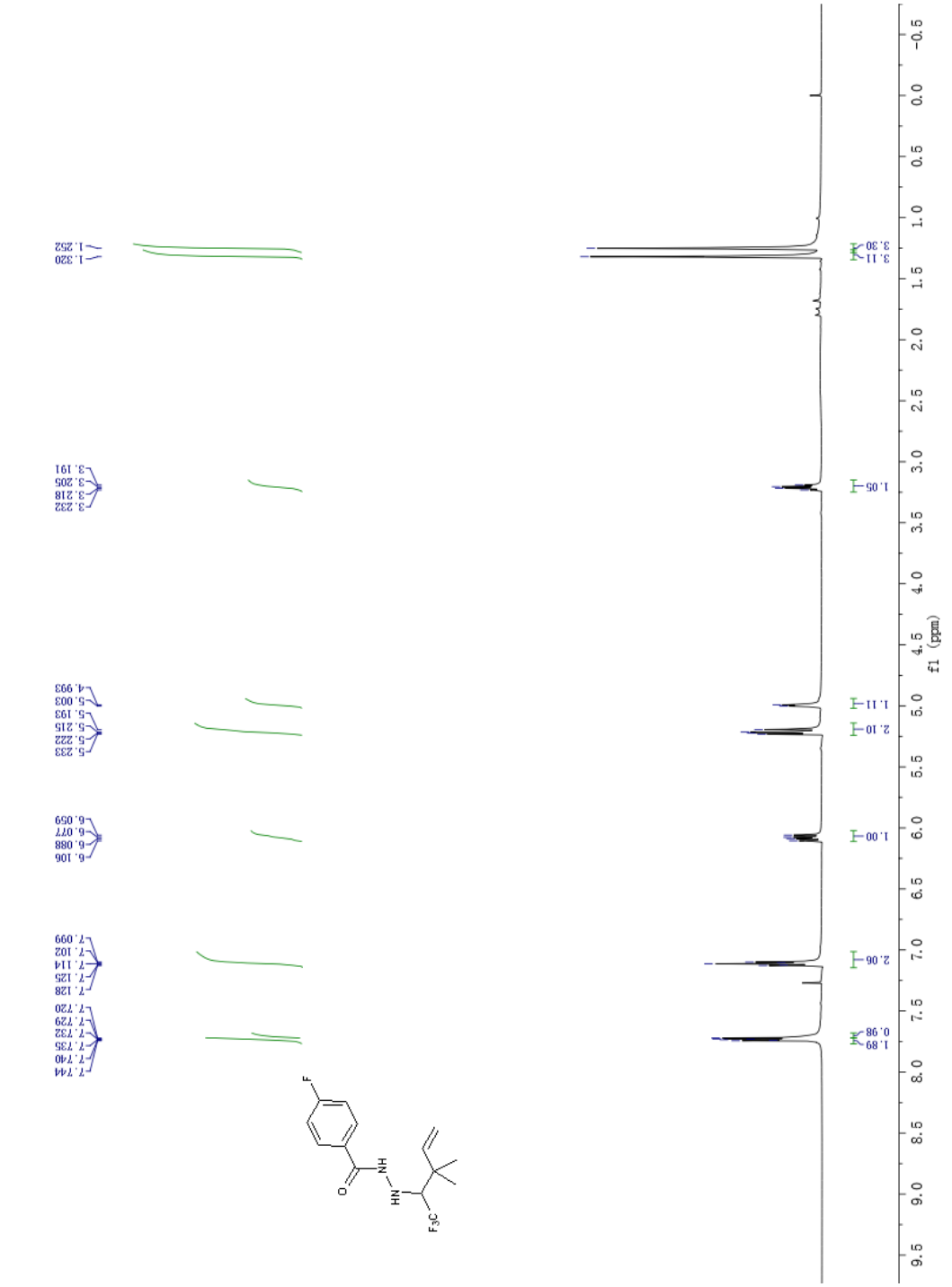


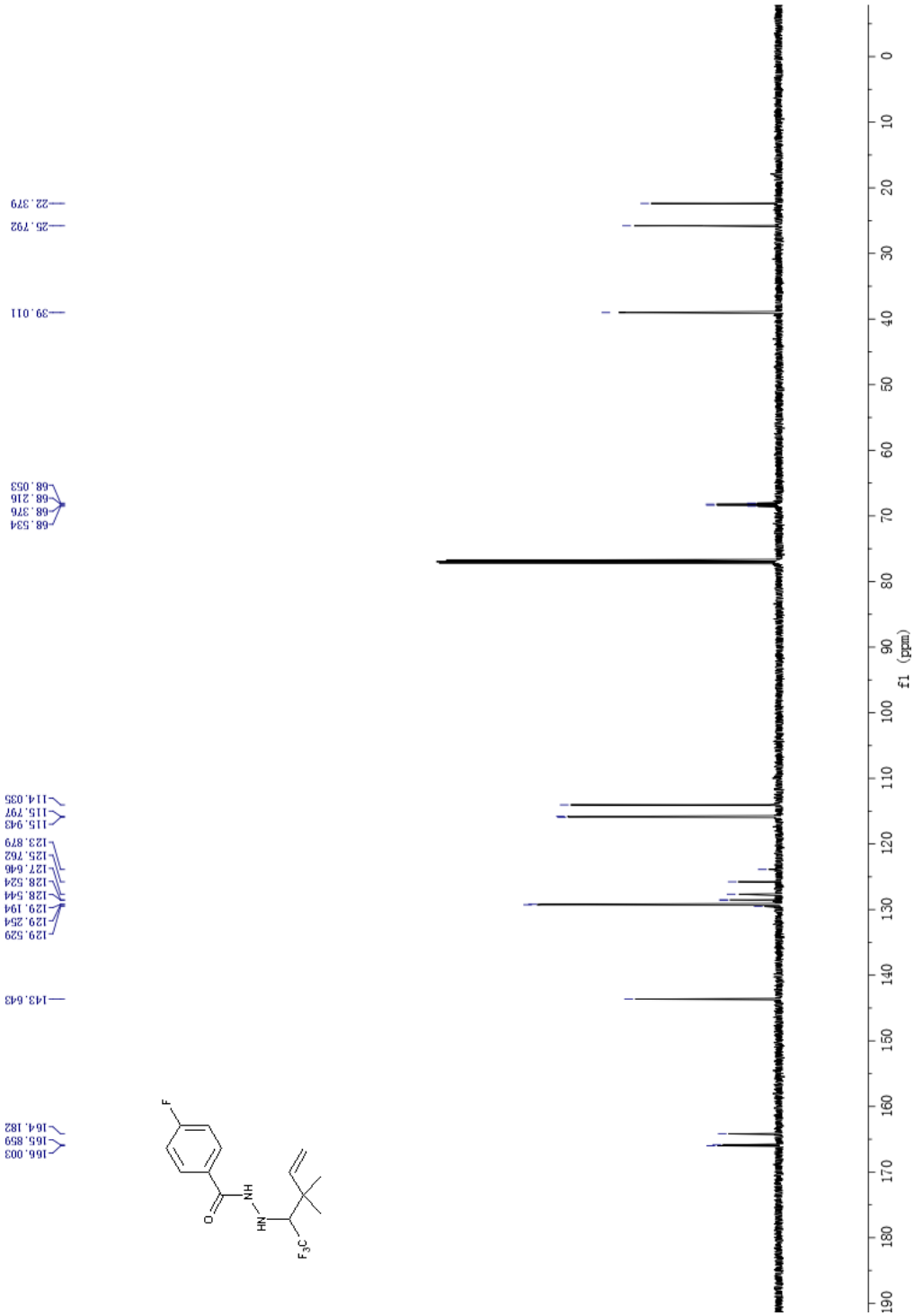

166.974
143.670
132.333
132.036
129.541
128.700
127.656
126.828
125.773
123.889
113.960
68.552
68.391
68.230
68.070
38.993
25.774
22.432
f1 (ppm)
0
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
160
170
180
190
O
NH
HN
F3C

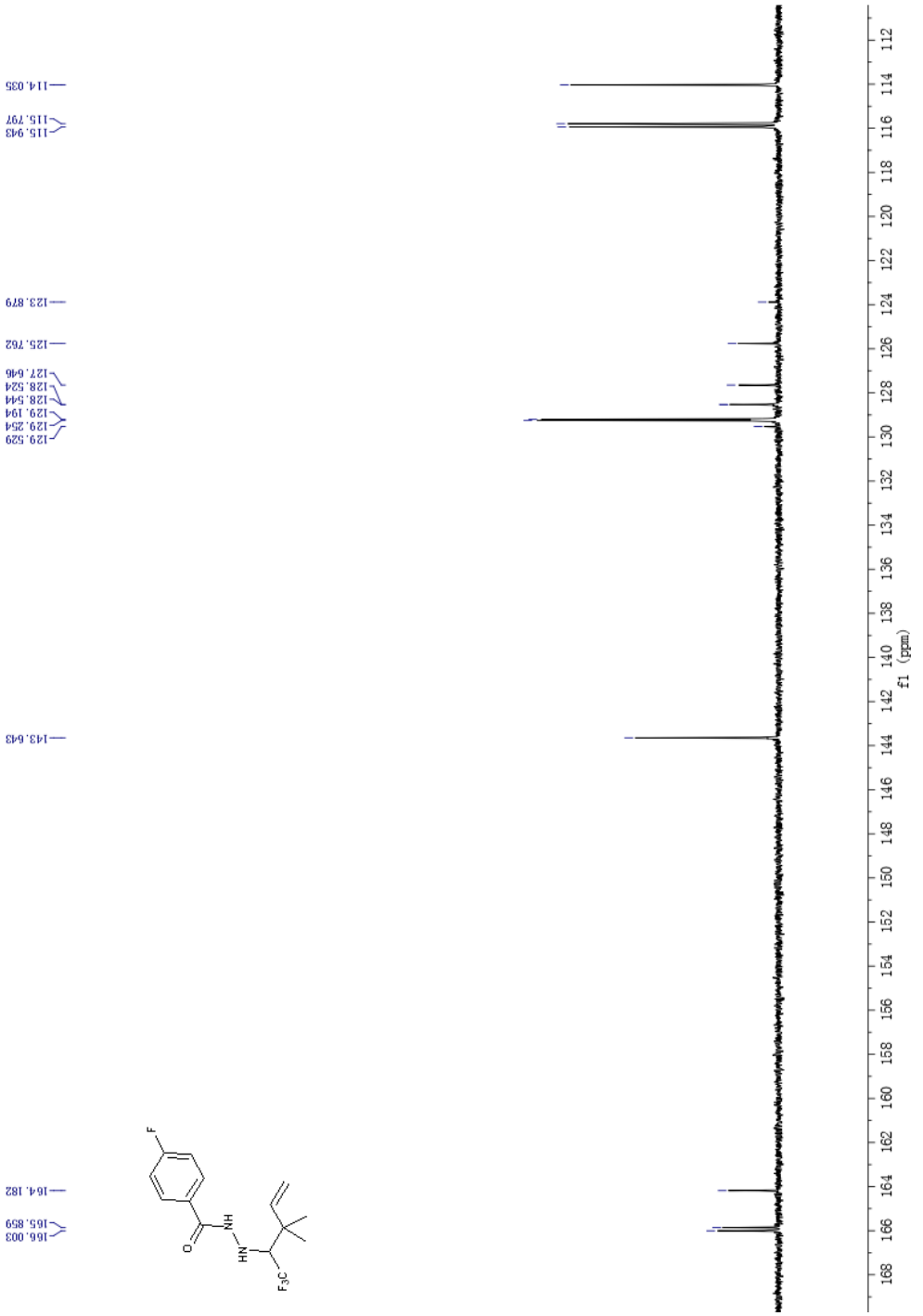

-66.885
-66.906
O
NH
HN
F3C
20
10
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190
f1 (ppm)

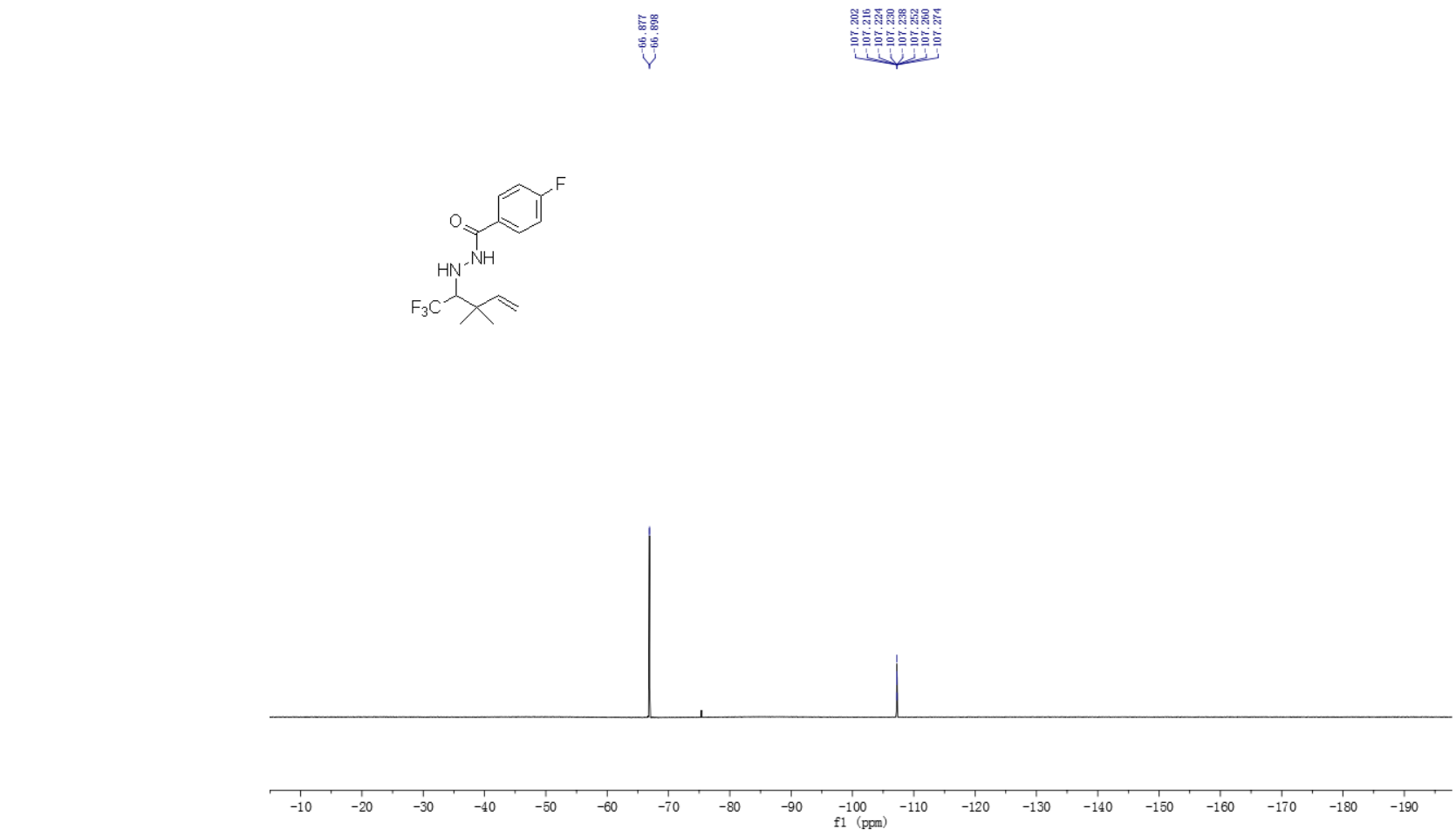

ESI positive, dgg12#, M+H=287.1366,error=1.0ppm, 23/07/2015
r.i.
0.8
0.6
0.4
0.2
0.0
287.1363
287.1518
287.0589
288.1411
309.1196
309.1464
277.5487
295.9969
280
290
300
310
320
m/z
/u/data/TRAINING/dgg150723/3x/pdata/1 xspec Thu Jul 23 21:31:34 2015

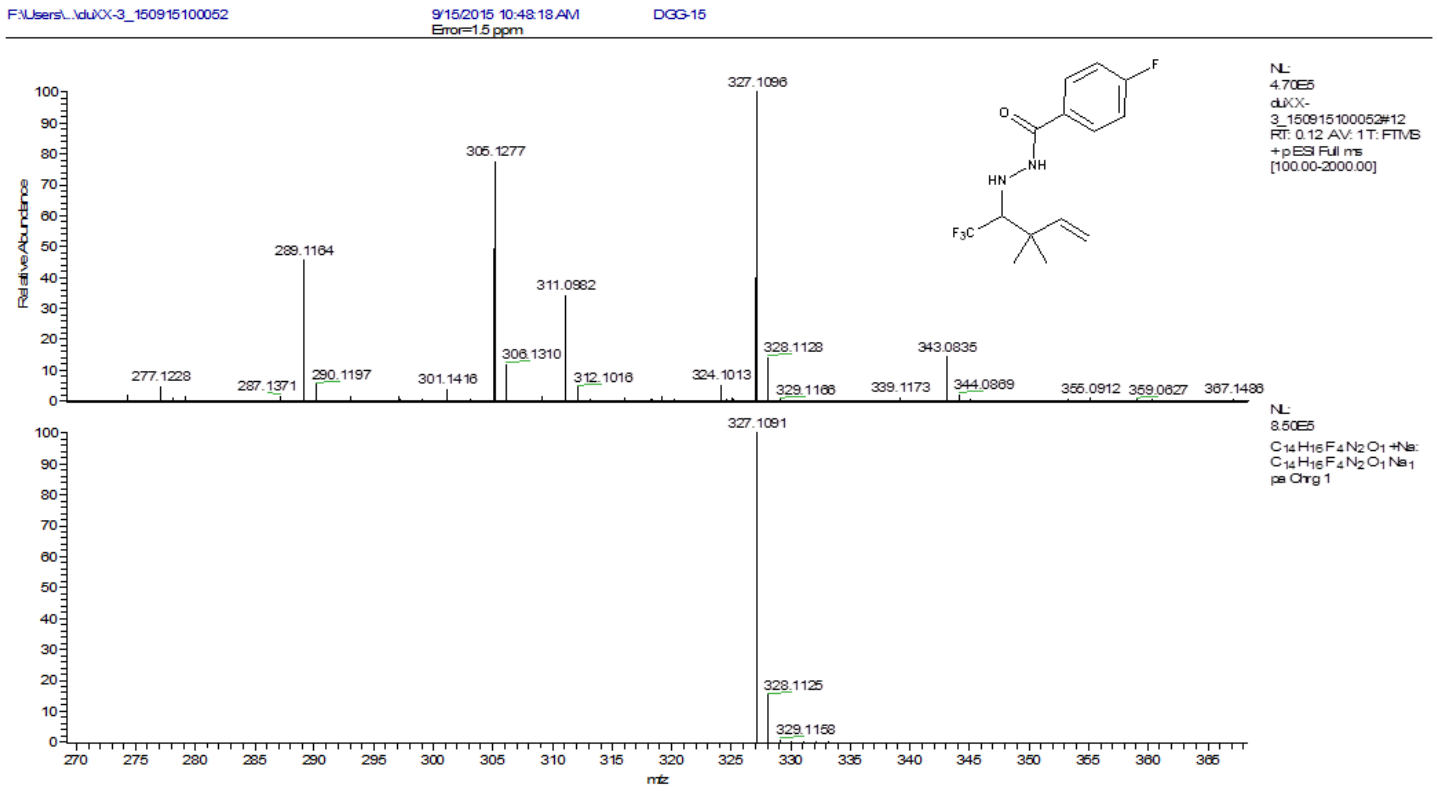
4-fluoro-N'-(1,1,1-trifluoro-3,3-dimethylpent-4-en-2-yl)benzohydrazide(4l)



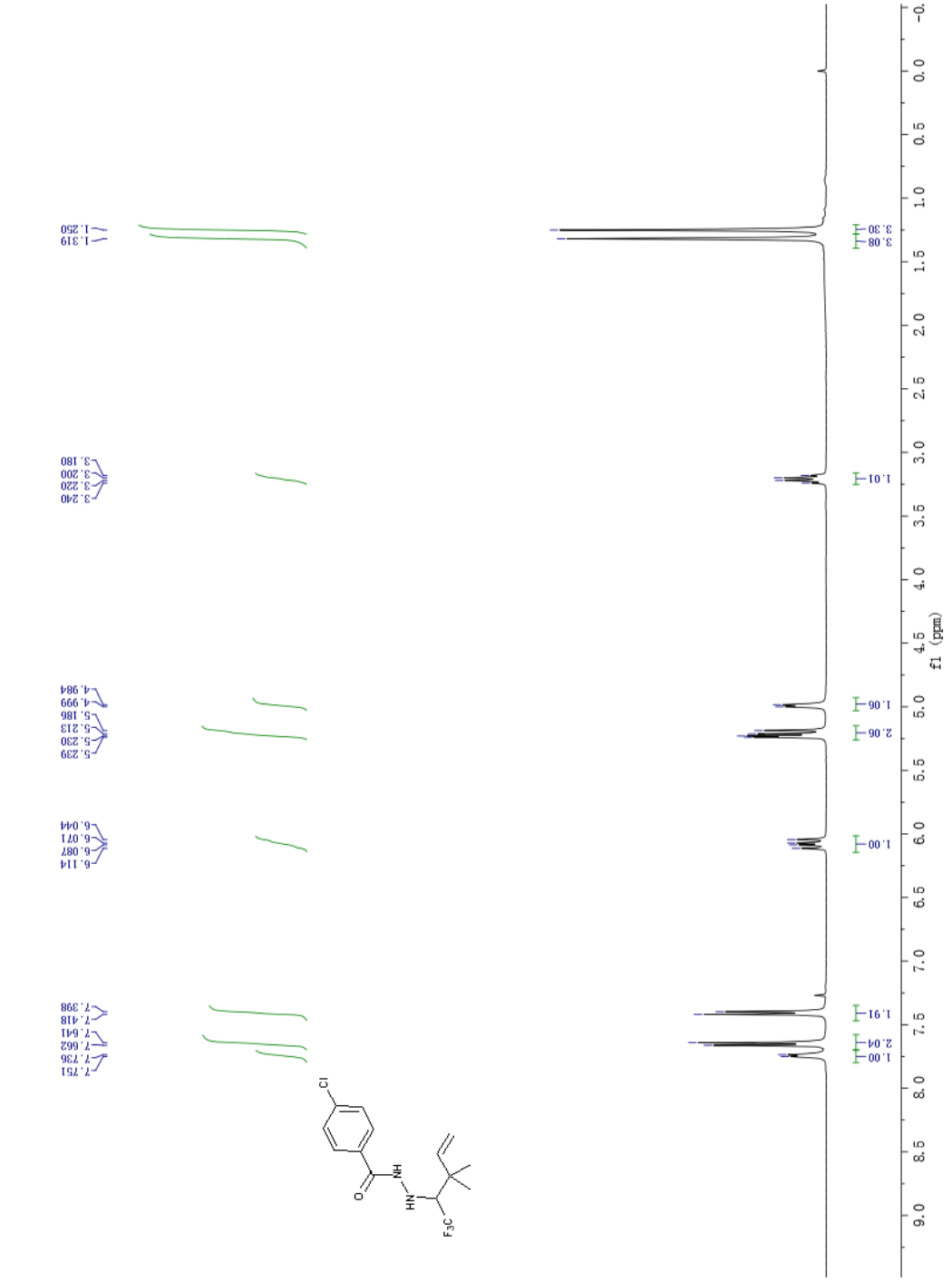

166.003
165.859
164.182
143.643
129.529
129.254
129.194
128.544
128.524
127.646
125.762
123.879
115.943
115.797
114.035
68.534
68.376
68.216
68.053
39.011
25.792
22.379
F
O
NH
HN
F3C
f1 (ppm)

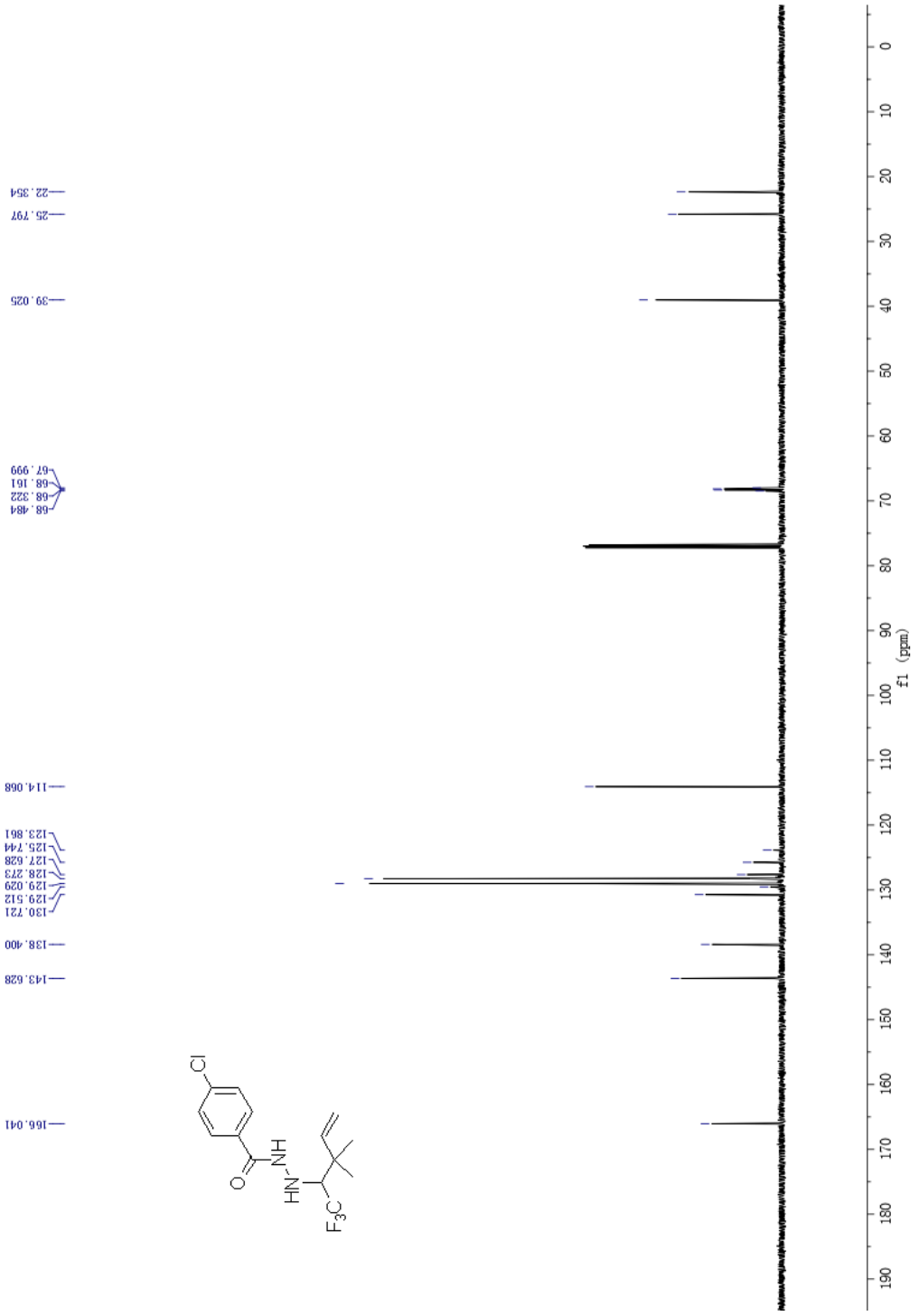

114.035
115.943
115.797
123.879
125.762
127.646
128.524
128.544
129.194
129.254
129.529
143.643
164.182
165.859
166.003
F
O
NH
HN
F3C
f1 (ppm)

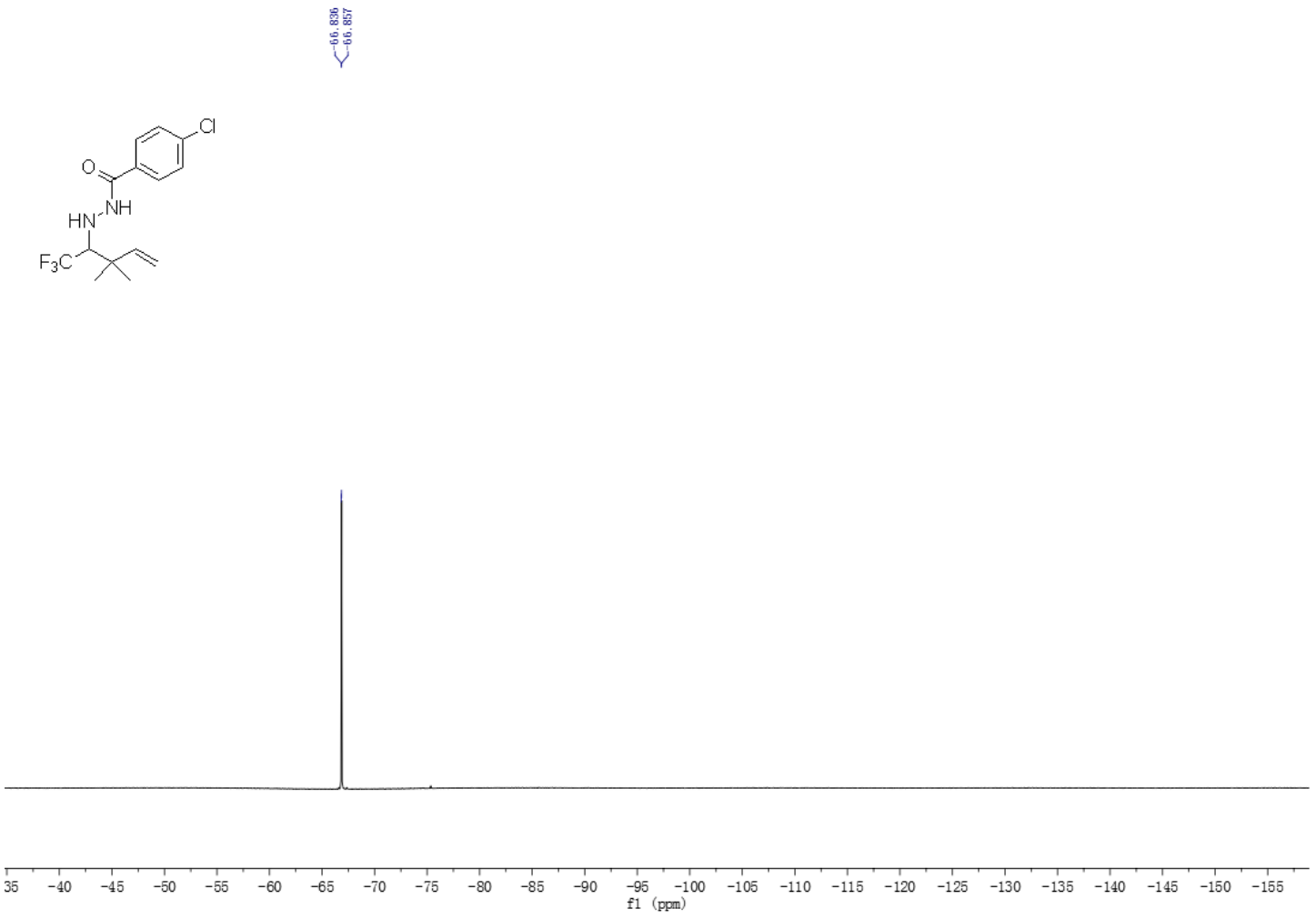

-66.877
-66.898
-107.202
-107.216
-107.224
-107.230
-107.238
-107.252
-107.260
-107.274
F
O
NH
HN
F3C
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190
f1 (ppm)

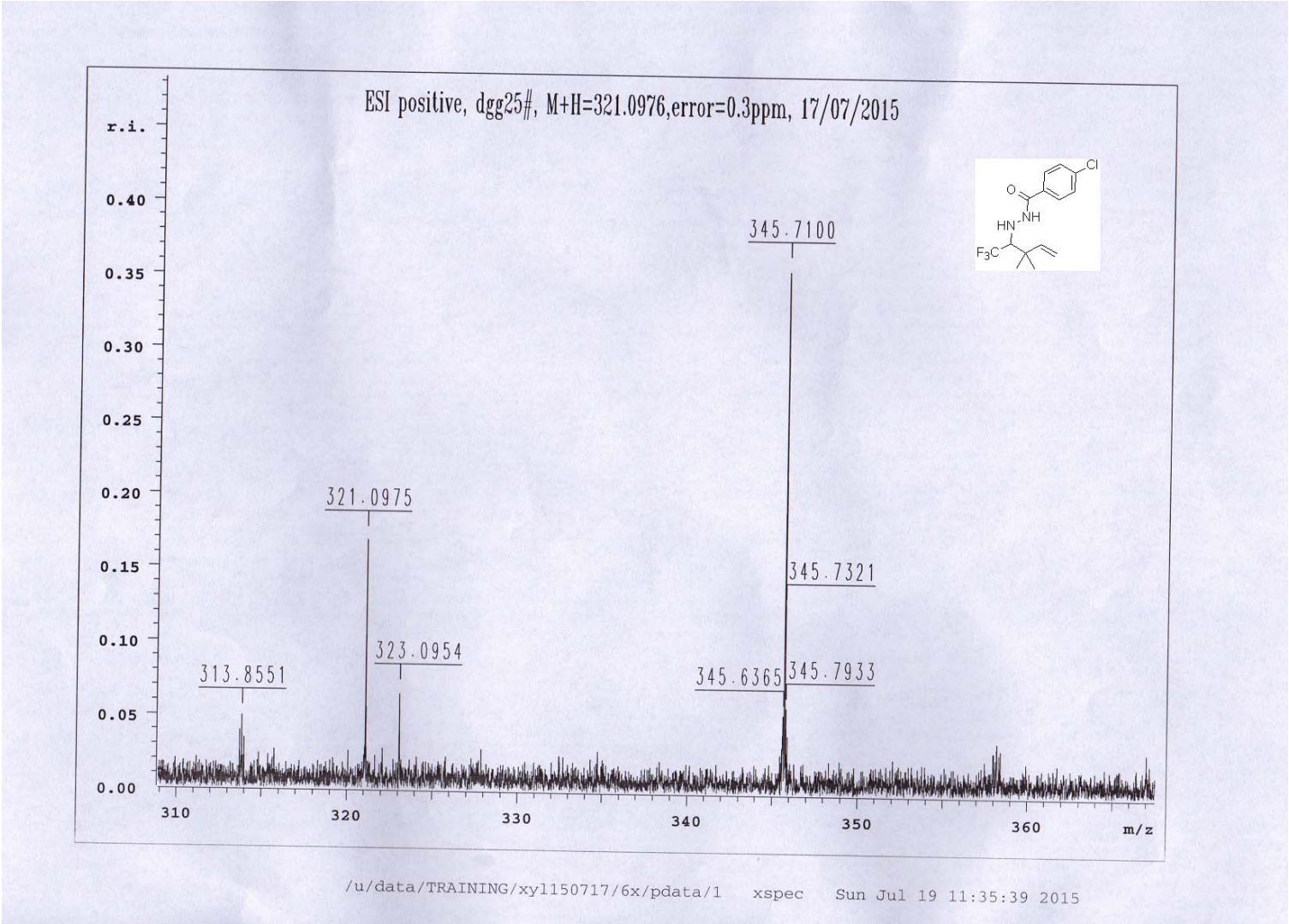

F:\Users\...\duXX-3_150915100052
9/15/2015 10:48:18 AM
Error=1.5 ppm
DGG-15
NL:
4.70E5
duXX-3_150915100052#12
RT: 0.12 AV: 1 T: FTMS + p ESI Full ms [100.00-2000.00]
327.1096
305.1277
289.1164
311.0982
306.1310
328.1128
343.0835
277.1228
287.1371
290.1197
301.1416
312.1016
324.1013
329.1166
339.1173
344.0869
355.0912
359.0627
367.1486
Relative Abundance
NL:
8.50E5
C14 H15 F4 N2 O1 +Na: C14 H15 F4 N2 O1 Na1 pa Chrg 1
327.1091
328.1125
329.1158
m/z

4-chloro-N'-(1,1,1-trifluoro-3,3-dimethylpent-4-en-2-yl)benzohydrazide(4m)

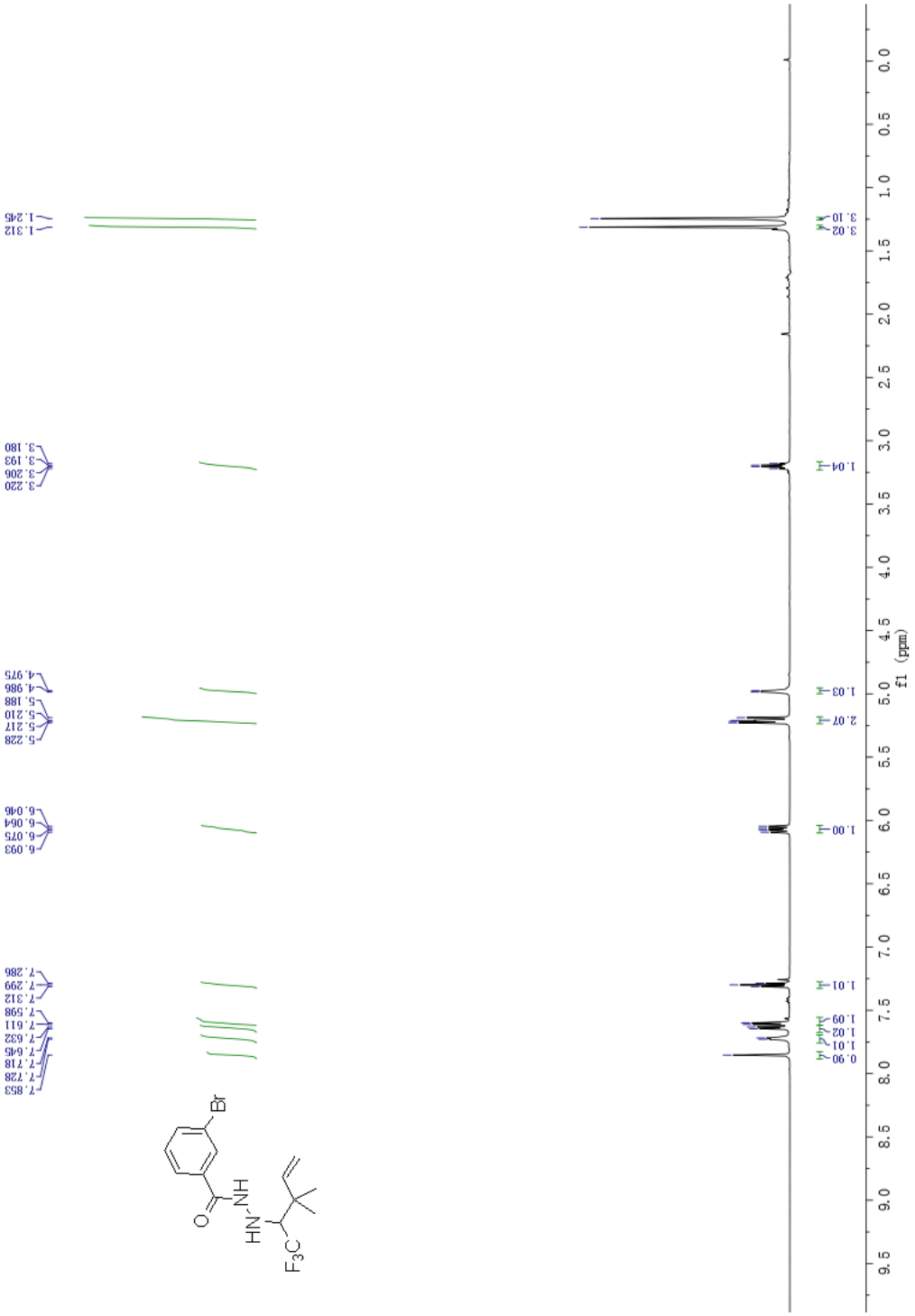


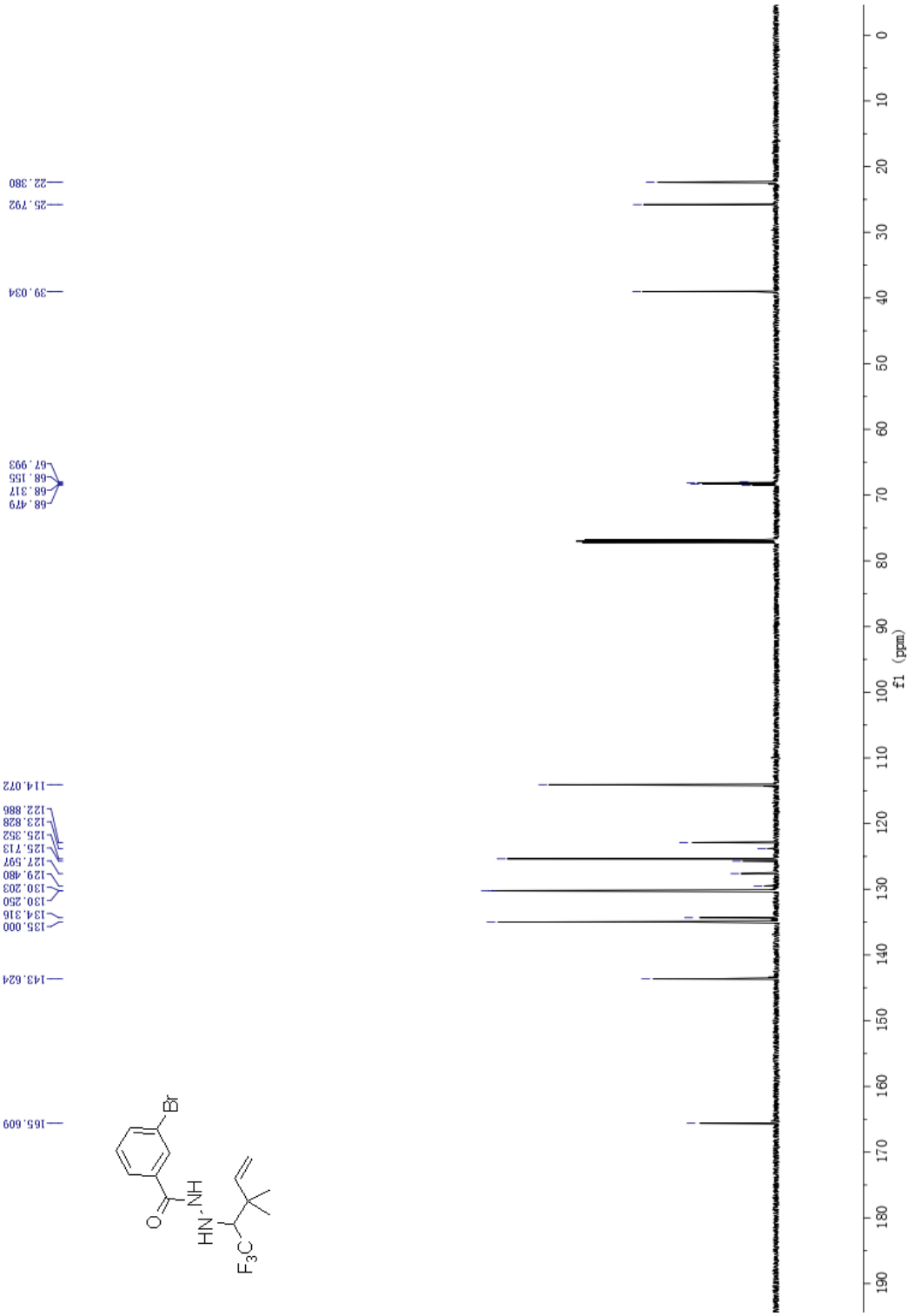

166.041
143.628
138.400
130.721
129.512
129.029
128.273
127.628
125.744
123.861
114.068
68.484
68.322
68.161
67.999
39.025
25.797
22.354
f1 (ppm)
Cl
O
HN
NH
F3C

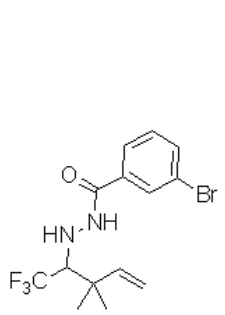

-66.836
-66.857
Cl
O
NH
HN
F3C
35 -40 -45 -50 -55 -60 -65 -70 -75 -80 -85 -90 -95 -100 -105 -110 -115 -120 -125 -130 -135 -140 -145 -150 -155
f1 (ppm)


ESI positive, dgg25#, M+H=321.0976,error=0.3ppm, 17/07/2015
r.i.
0.40
0.35
0.30
0.25
0.20
0.15
0.10
0.05
0.00
345.7100
321.0975
345.7321
323.0954
313.8551
345.6365
345.7933
310
320
330
340
350
360
m/z
Cl
O
NH
HN
F3C
/u/data/TRAINING/xyl150717/6x/pdata/1 xspec Sun Jul 19 11:35:39 2015

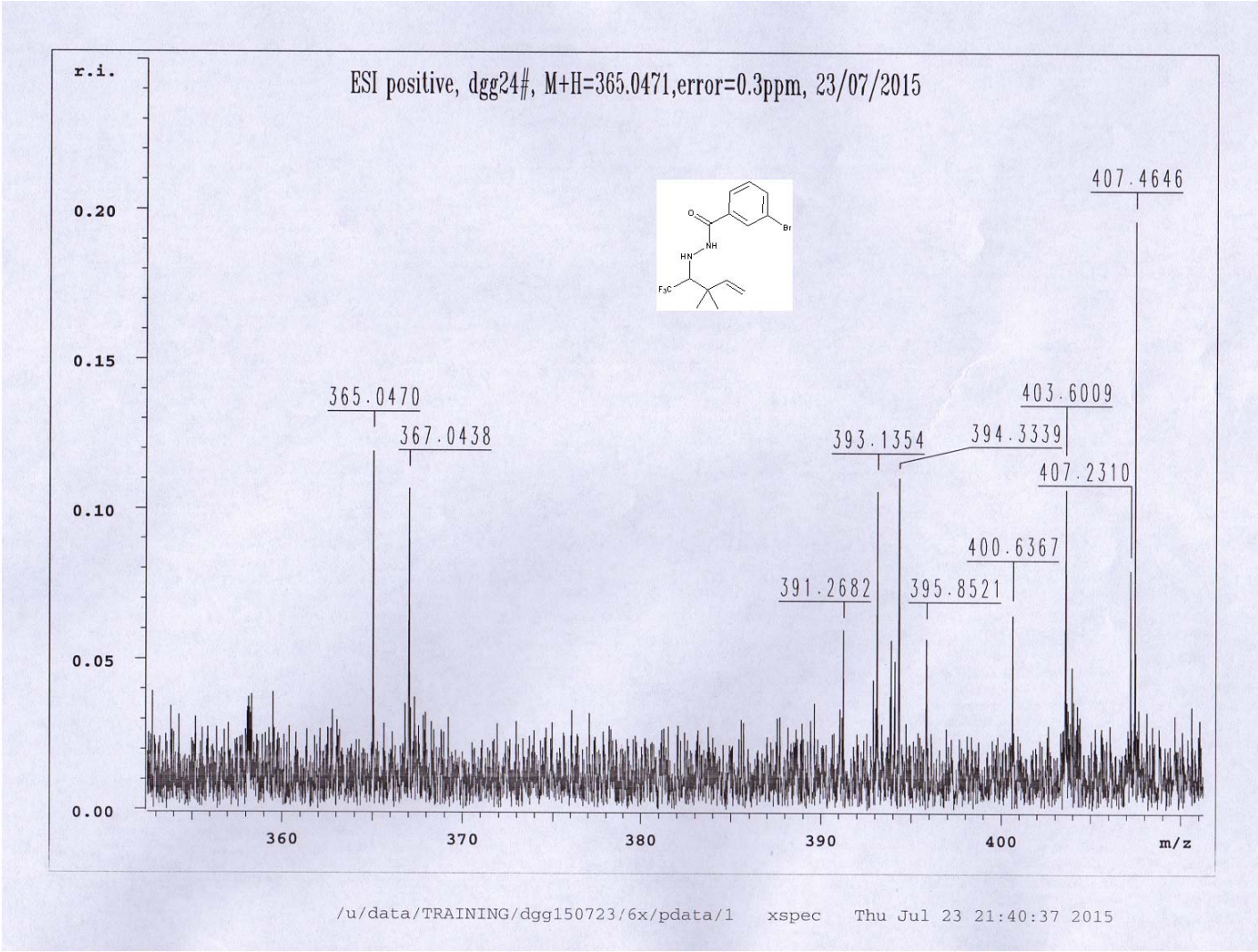
3-bromo-N'-(1,1,1-trifluoro-3,3-dimethylpent-4-en-2-yl)benzohydrazide(4n)



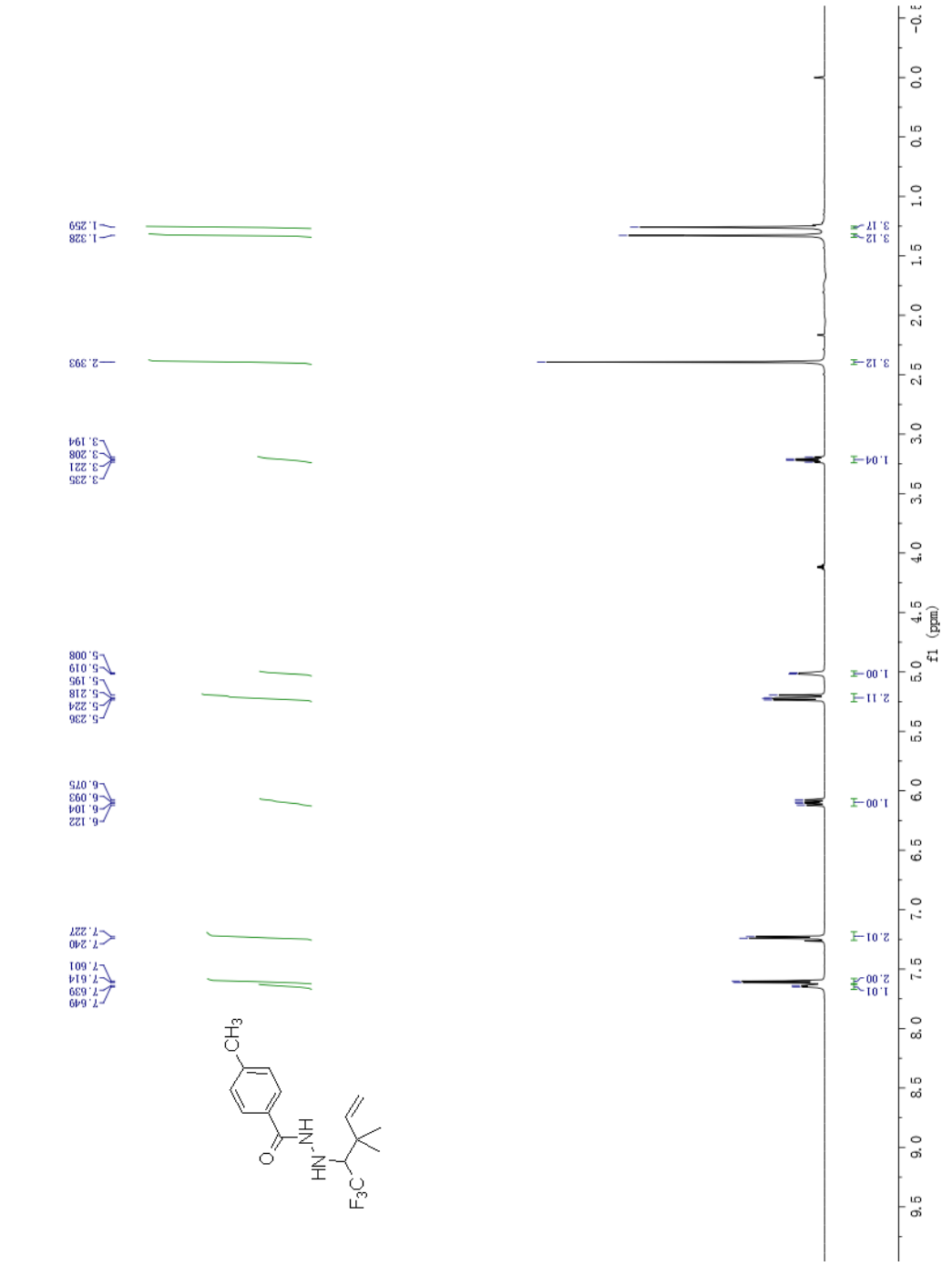

22.380
25.792
39.034
67.993
68.155
68.317
68.479
114.072
122.886
123.828
125.352
125.713
127.597
129.480
130.203
130.250
134.316
135.000
143.624
165.609
Br
O
HN
NH
F3C
f1 (ppm)

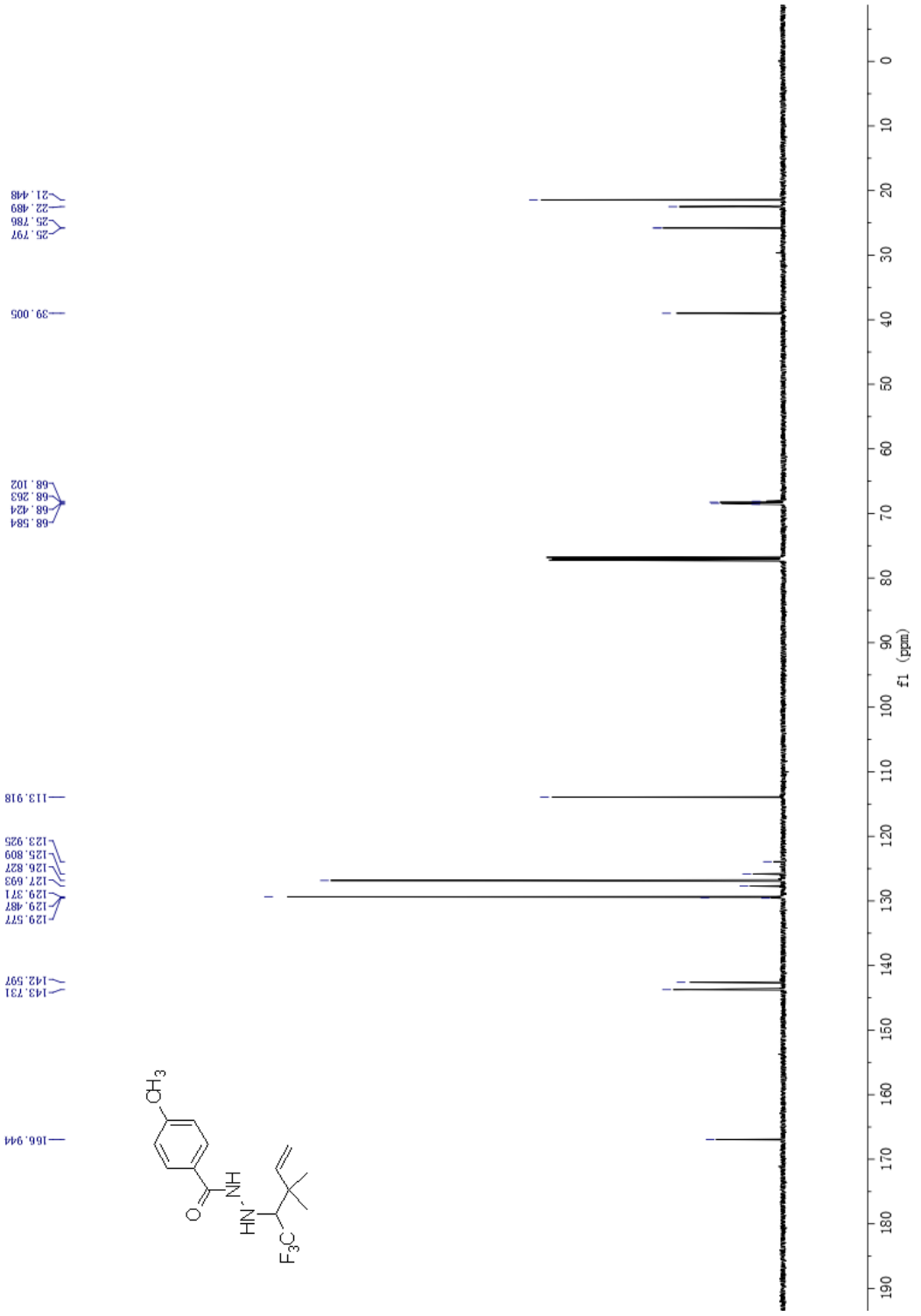

O
Br
NH
HN
F3C

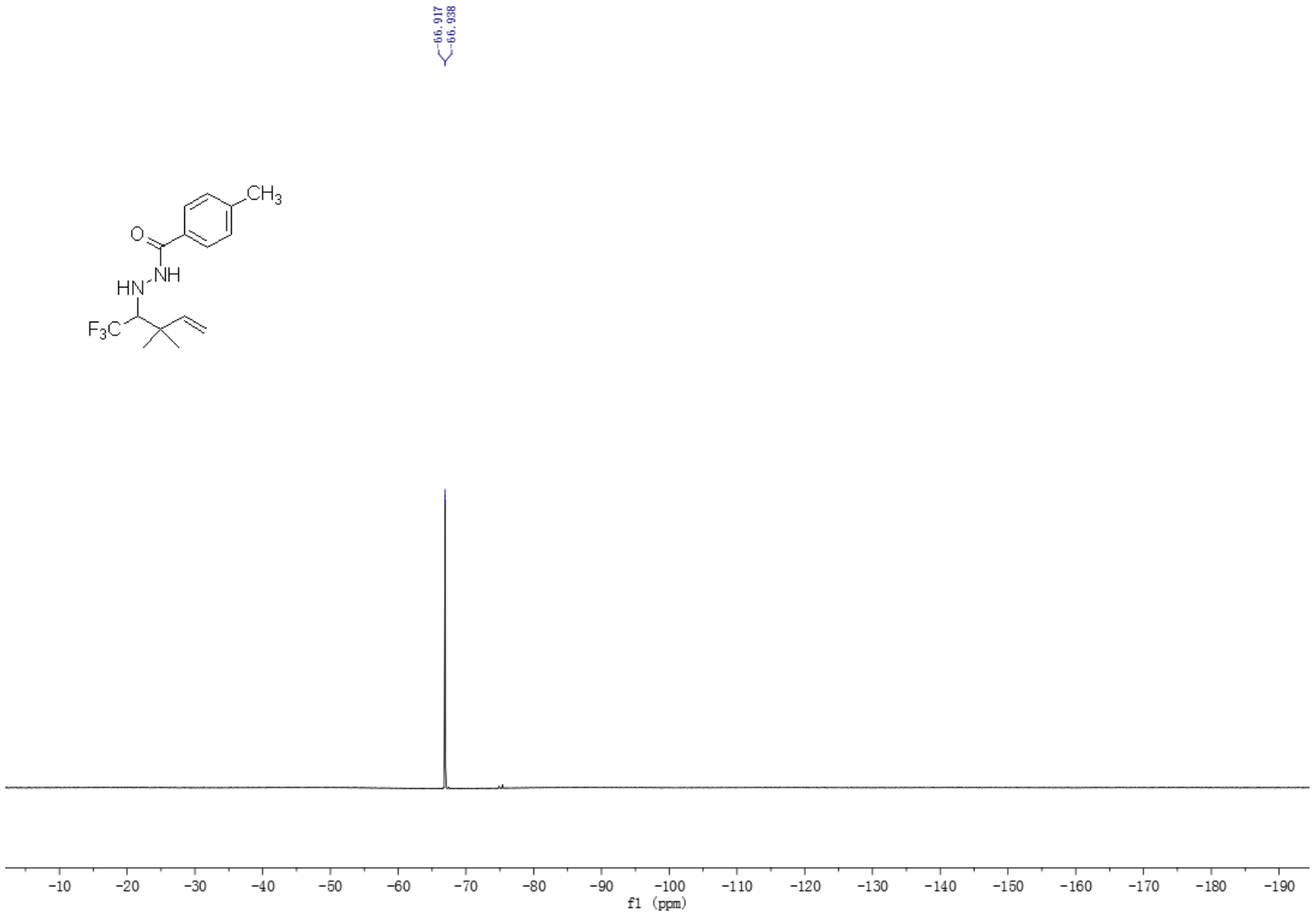
-66.865
-66.885
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190
f1 (ppm)

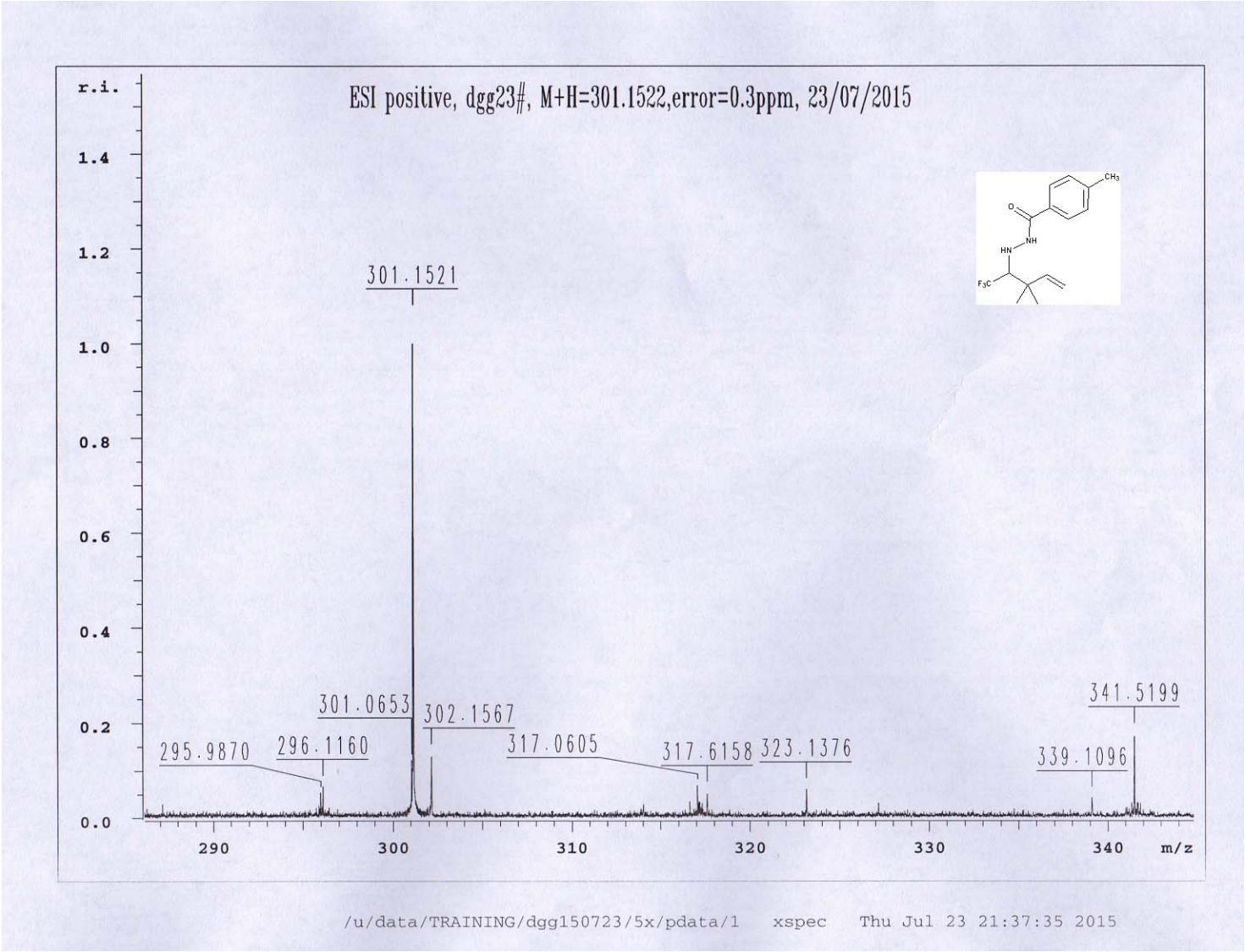

ESI positive, dgg24#, M+H=365.0471,error=0.3ppm, 23/07/2015
r.i.
0.20
0.15
0.10
0.05
0.00
365.0470
367.0438
391.2682
393.1354
394.3339
395.8521
400.6367
403.6009
407.2310
407.4646
360
370
380
390
400
m/z
/u/data/TRAINING/dgg150723/6x/pdata/1 xspec Thu Jul 23 21:40:37 2015

4-methyl-N'-(1,1,1-trifluoro-3,3-dimethylpent-4-en-2-yl)benzohydrazide(4o)

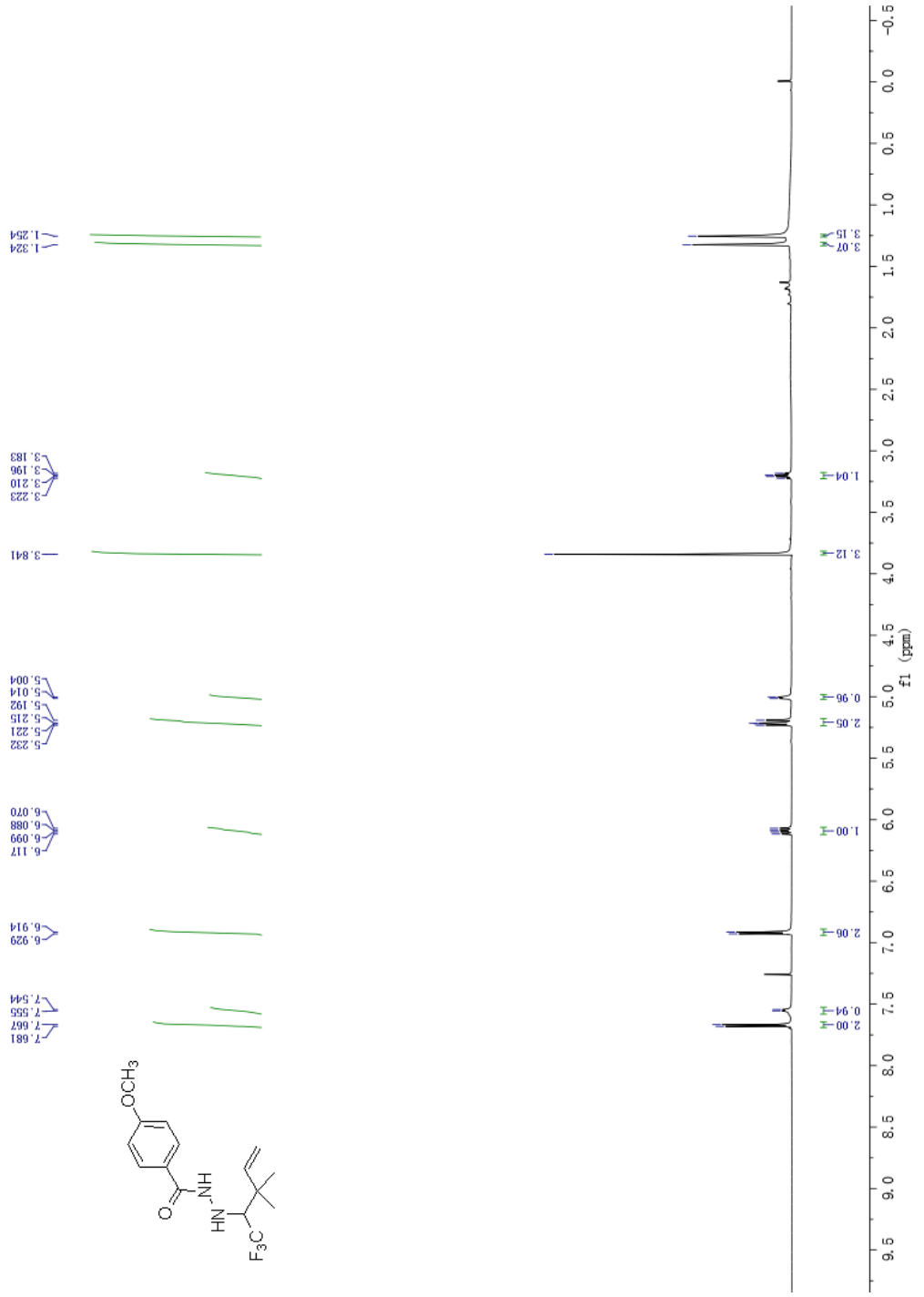


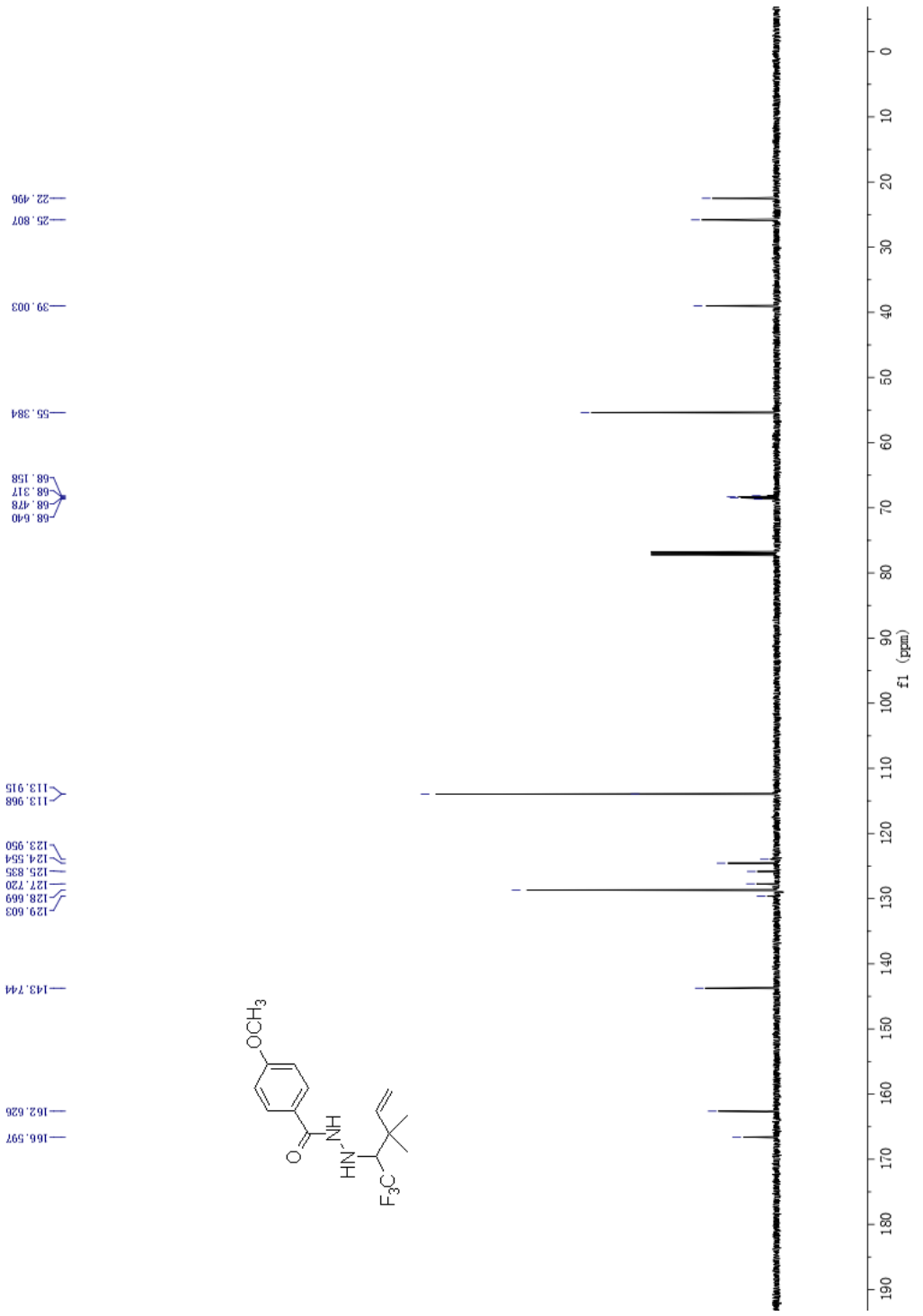

21.448
22.489
25.797
25.786
39.005
68.584
68.424
68.263
68.102
113.918
123.925
125.809
126.827
127.693
129.371
129.487
129.577
142.597
143.731
166.944
CH3
O
NH
HN
F3C
f1 (ppm)

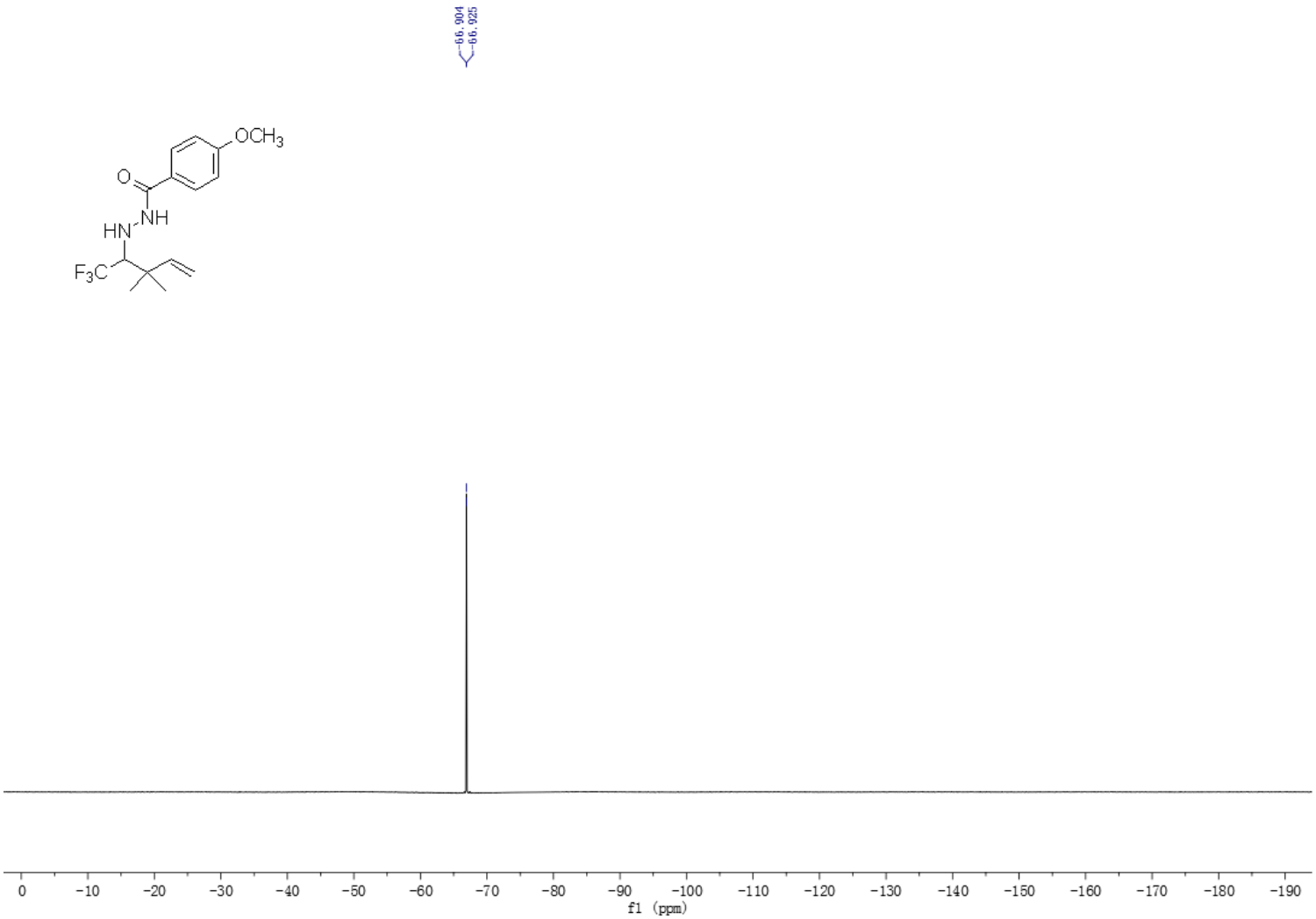

-66.917
-66.938
CH3
O
NH
HN
F3C
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190
f1 (ppm)

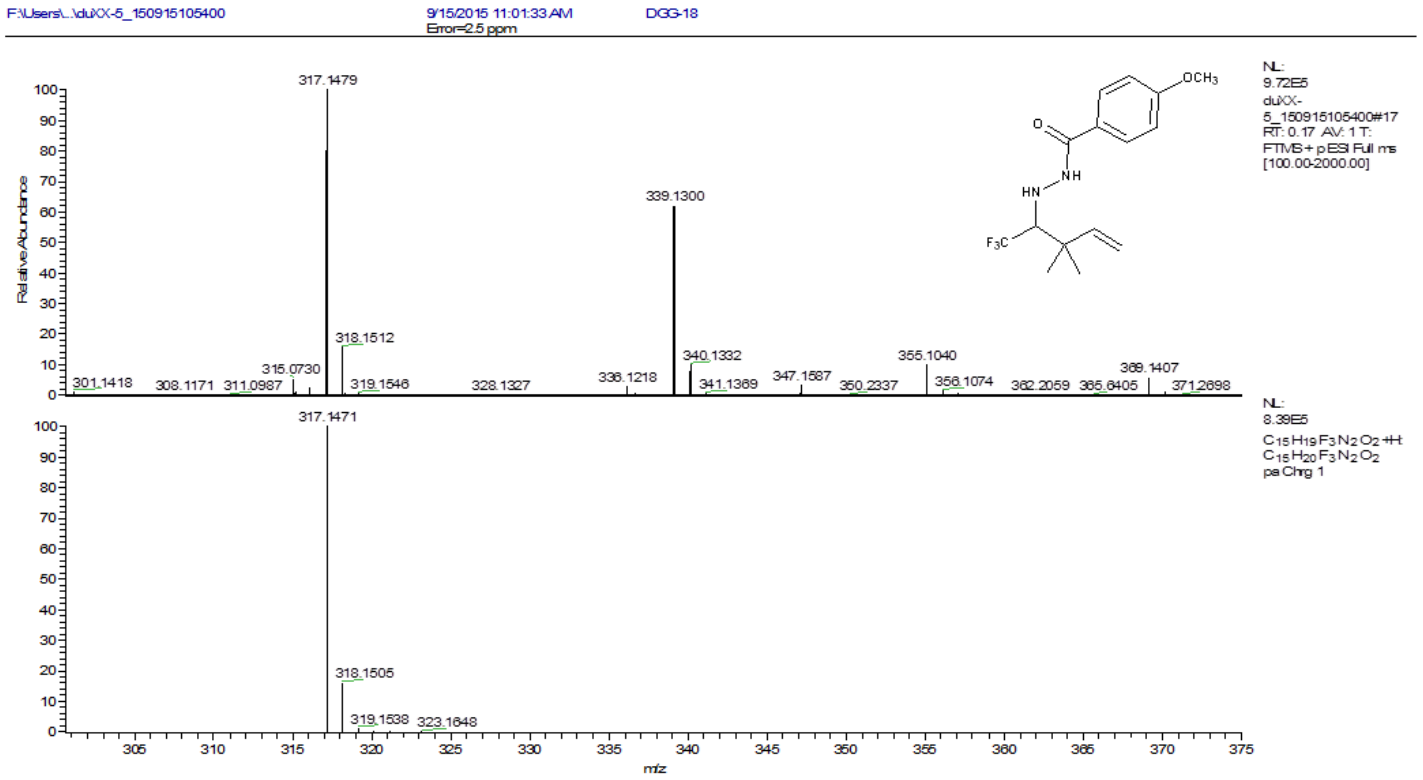

ESI positive, dgg23#, M+H=301.1522,error=0.3ppm, 23/07/2015
r.i.
1.4
1.2
1.0
0.8
0.6
0.4
0.2
0.0
301.1521
301.0653
302.1567
295.9870
296.1160
317.0605
317.6158
323.1376
339.1096
341.5199
290
300
310
320
330
340
m/z
/u/data/TRAINING/dgg150723/5x/pdata/1 xspec Thu Jul 23 21:37:35 2015

4-methoxy-N'-(1,1,1-trifluoro-3,3-dimethylpent-4-en-2-yl)benzohydrazide(4p)

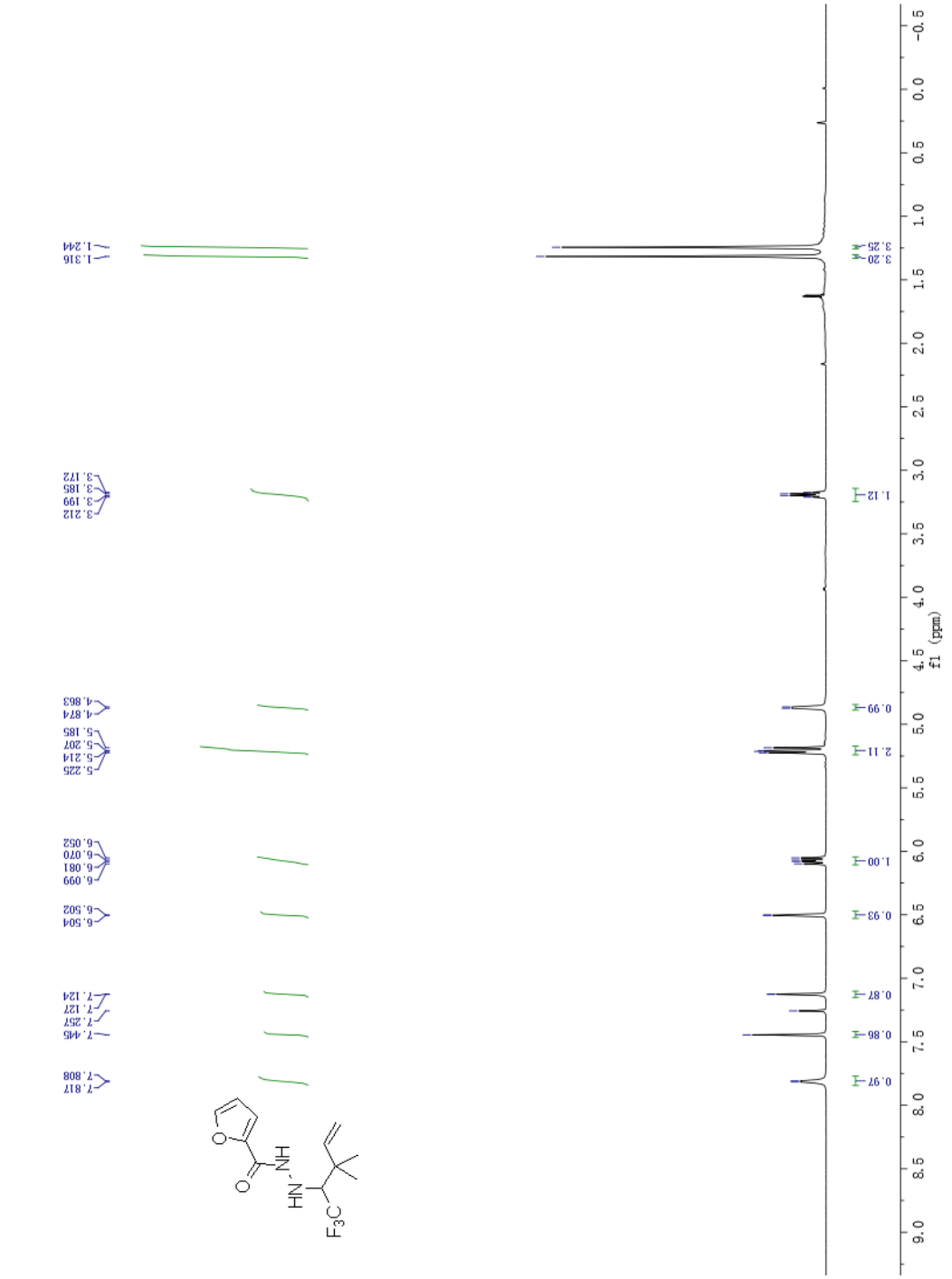


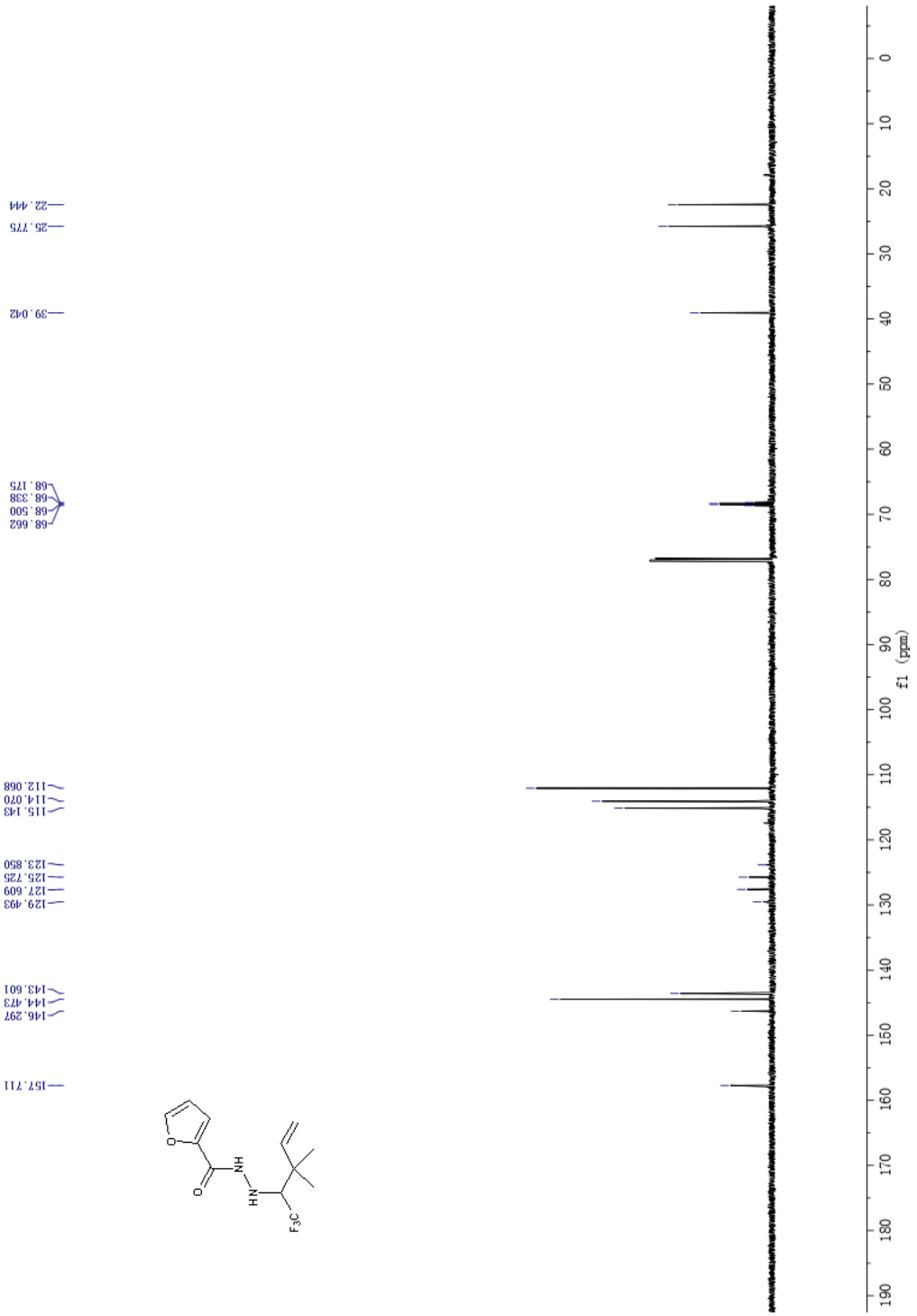

166.597
162.626
143.744
129.603
128.669
127.720
125.835
124.554
123.950
113.968
113.915
68.640
68.478
68.317
68.158
55.384
39.003
25.807
22.496
OCH3
O
NH
HN
F3C
f1 (ppm)

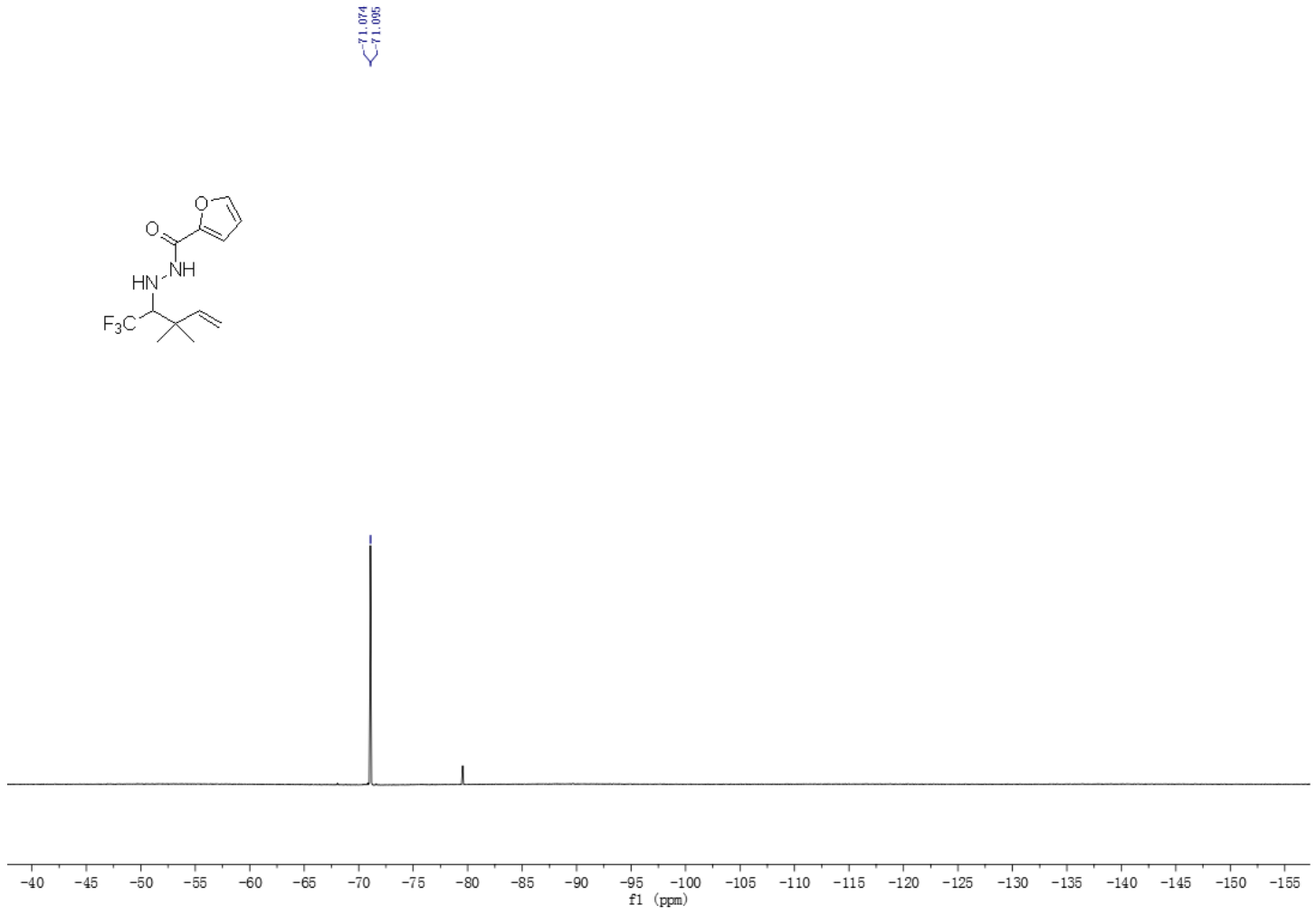

-66.904
-66.925
OCH3
O
NH
HN
F3C
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190
f1 (ppm)

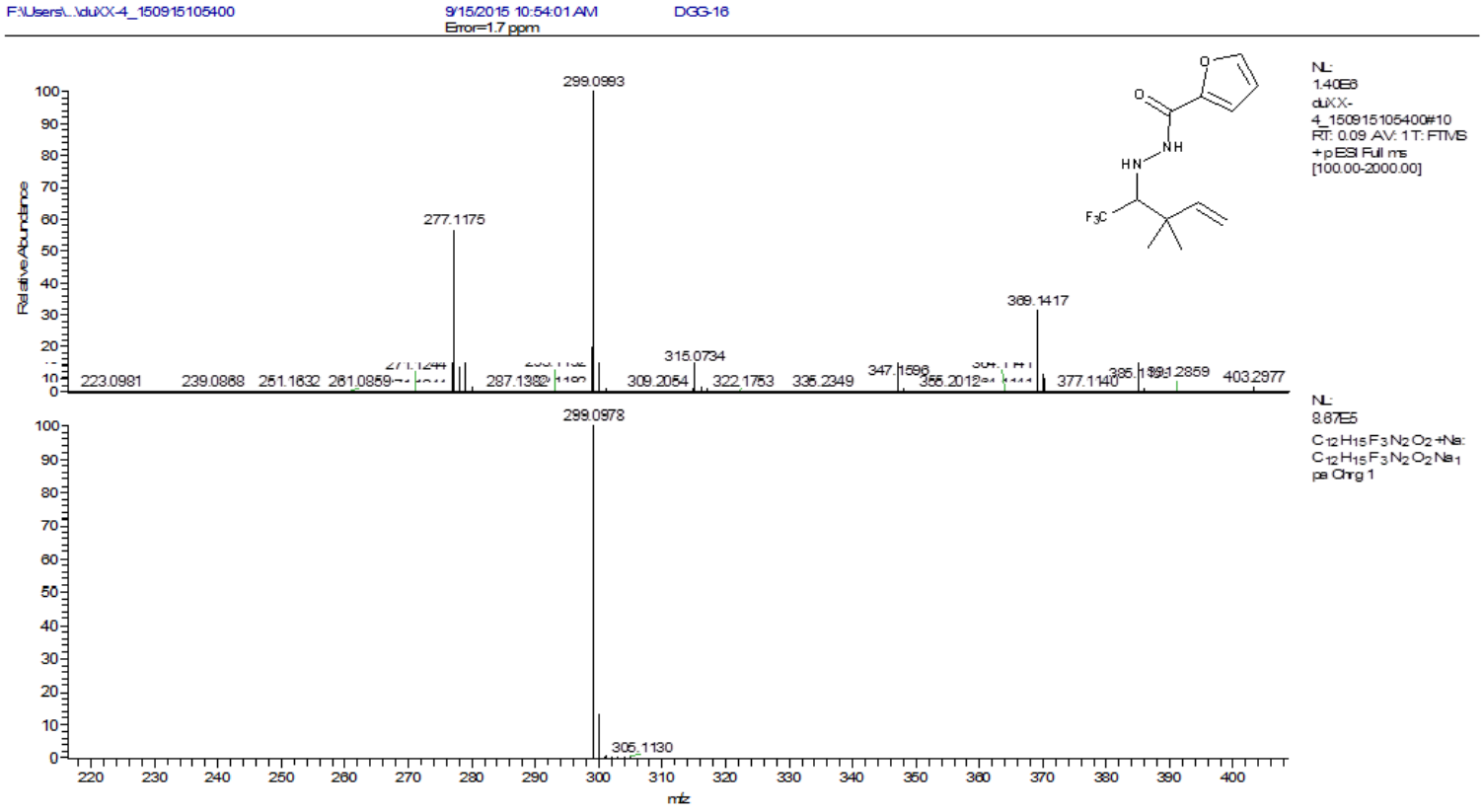

F:\Users\...\duXX-5_150915105400
9/15/2015 11:01:33 AM
Error=2.5 ppm
DGG-18
317.1479
339.1300
318.1512
340.1332
355.1040
369.1407
315.0730
301.1418
308.1171
311.0987
319.1546
328.1327
336.1218
341.1369
347.1587
350.2337
356.1074
362.2059
365.6405
371.2698
OCH3
O
NH
HN
F3C
NL:
9.72E5
duXX-
5_150915105400#17
RT: 0.17 AV: 1 T:
FTMS + p ESI Full ms
[100.00-2000.00]
Relative Abundance
317.1471
318.1505
319.1538
323.1648
NL:
8.39E5
C15H19F3N2O2+H
C15H20F3N2O2
pa Chrg 1
m/z

N'-(1,1,1-trifluoro-3,3-dimethylpent-4-en-2-yl)furan-2-carbohydrazide(4q)



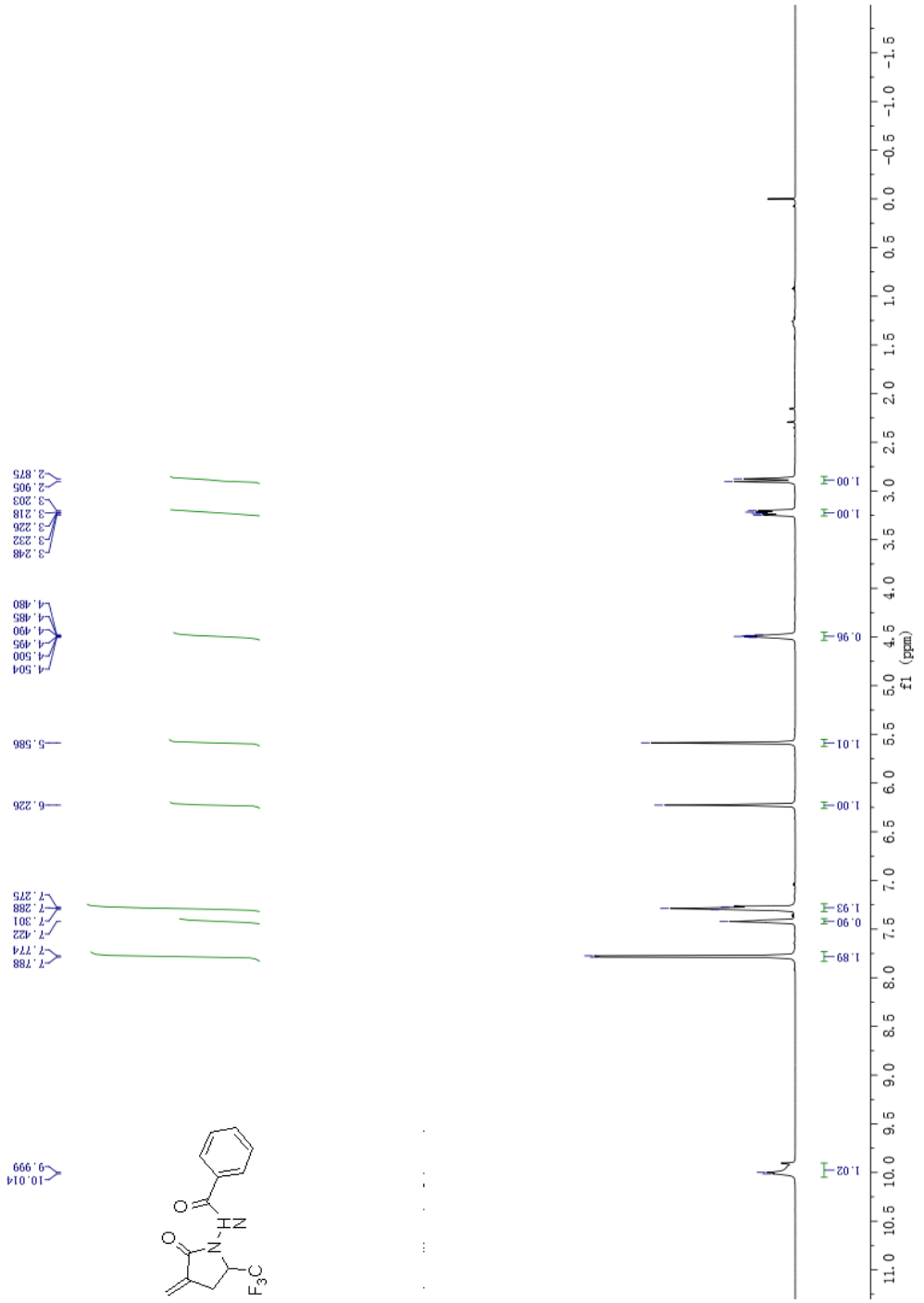

22.444
25.775
39.042
68.175
68.338
68.500
68.662
112.068
114.070
115.143
123.850
125.725
127.609
129.493
143.601
144.473
146.297
157.711
O
NH
HN
F3C
f1 (ppm)

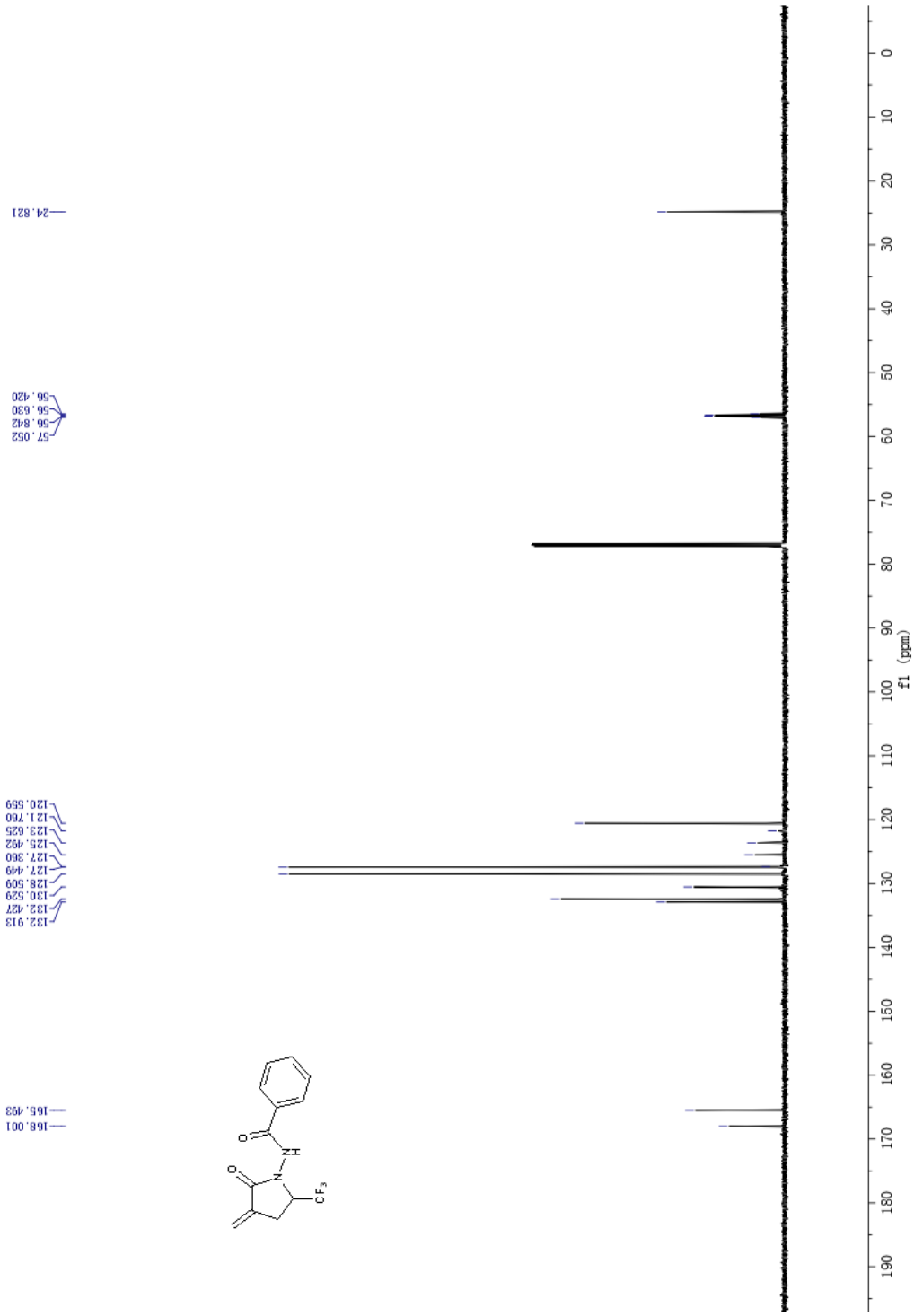

-71.074
-71.085
O
O
NH
HN
F3C
-40
-45
-50
-55
-60
-65
-70
-75
-80
-85
-90
-95
-100
-105
-110
-115
-120
-125
-130
-135
-140
-145
-150
-155
f1 (ppm)

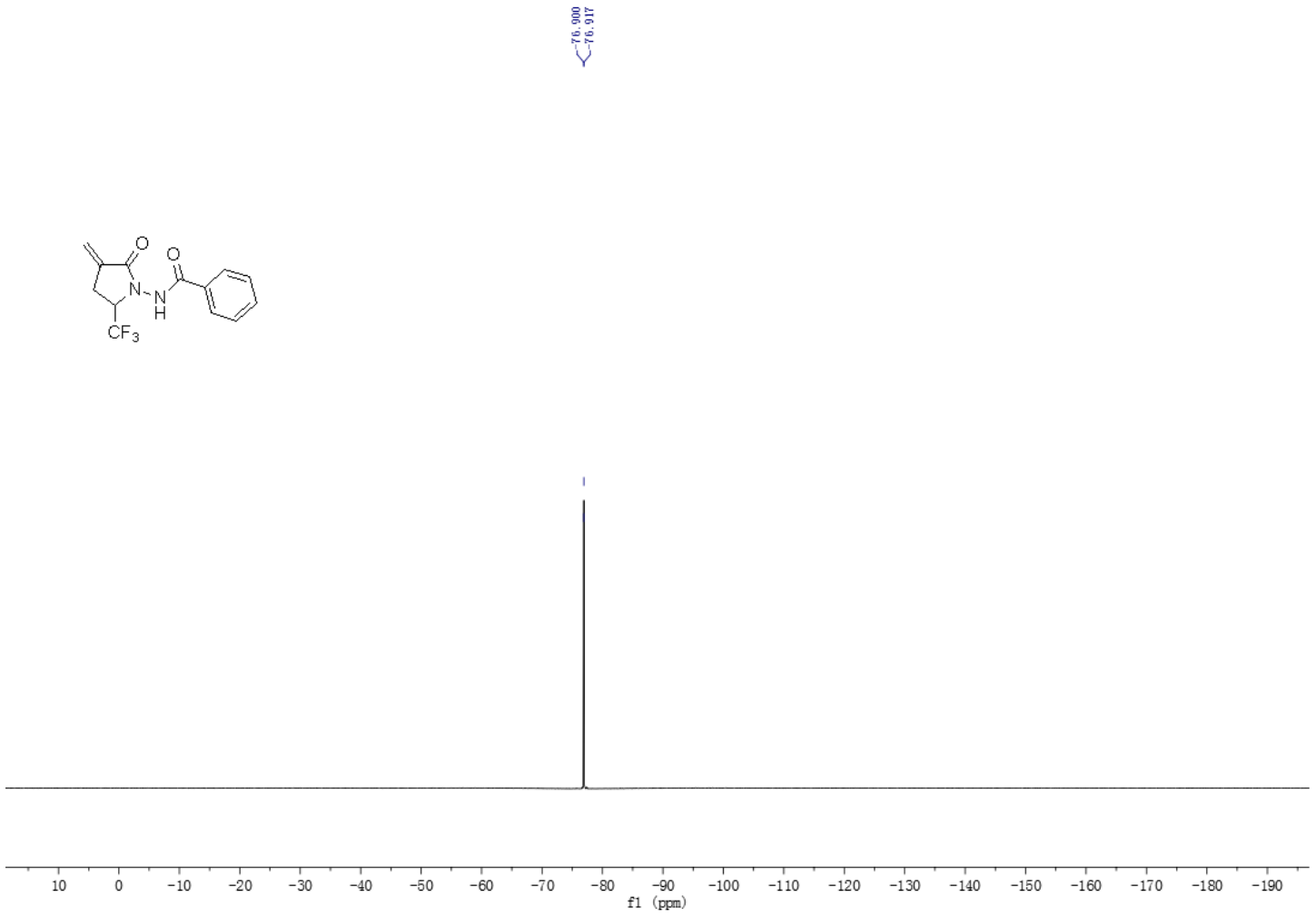

F:\Users\...\duXX-4_150915105400
9/15/2015 10:54:01 AM
DGG-16
Error=1.7 ppm
299.0993
277.1175
389.1417
315.0734
223.0981
239.0868
251.1632
261.0859
287.1382
309.2054
322.1753
335.2349
347.1596
355.2012
377.1140
391.2859
403.2977
Relative Abundance
NL:
1.40E8
duXX-
4_150915105400#10
RT: 0.09 AV: 1 T: FTMS
+ p ESI Full ms
[100.00-2000.00]
299.0978
305.1130
NL:
8.87E5
C12H15F3N2O2+Na:
C12H15F3N2O2Na1
pa Chrg 1
m/z

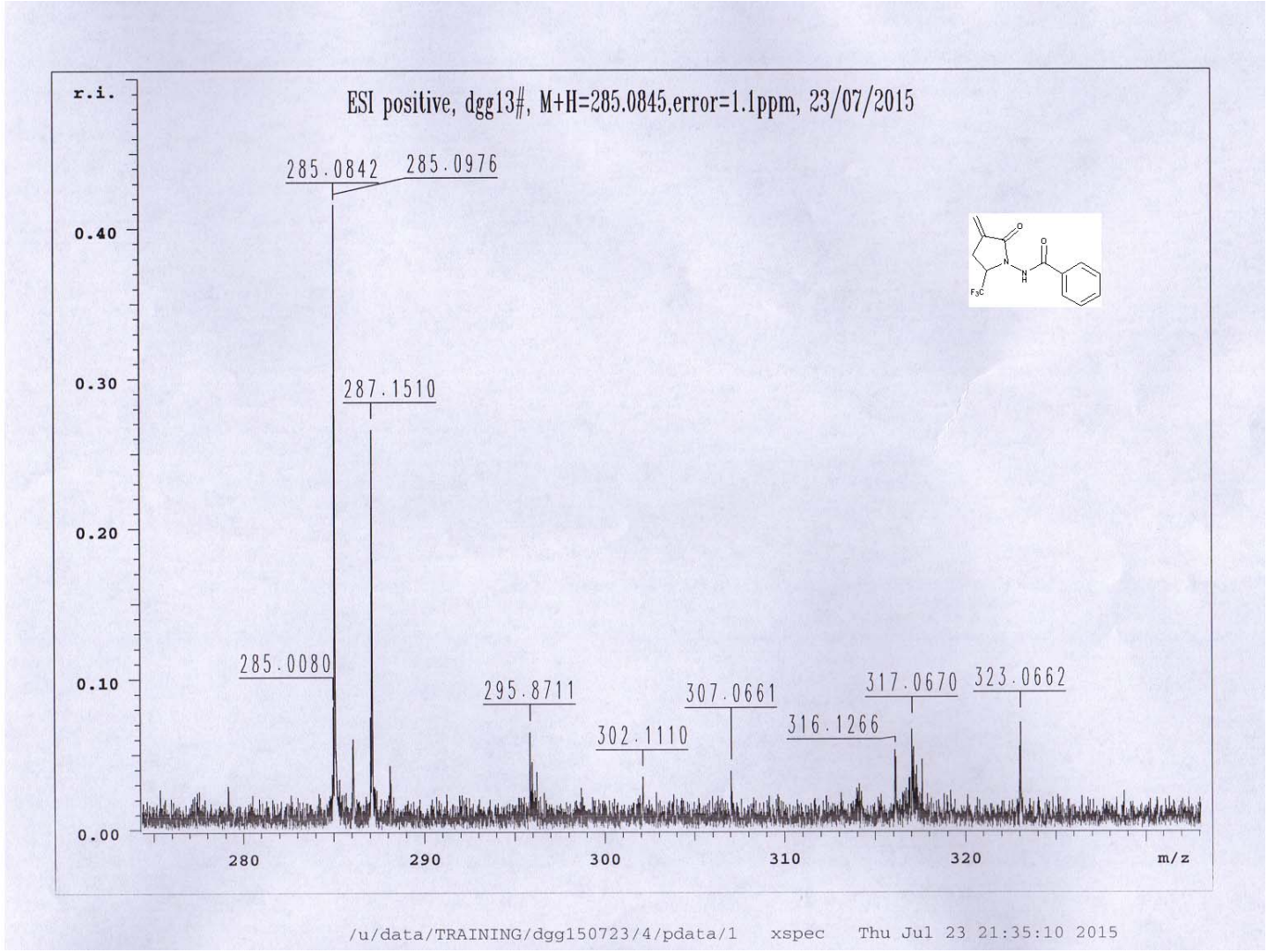
NMR Spectra of compounds 6

N-(3-methylene-2-oxo-5-(trifluoromethyl)pyrrolidin-1-yl)benzamide(6a)

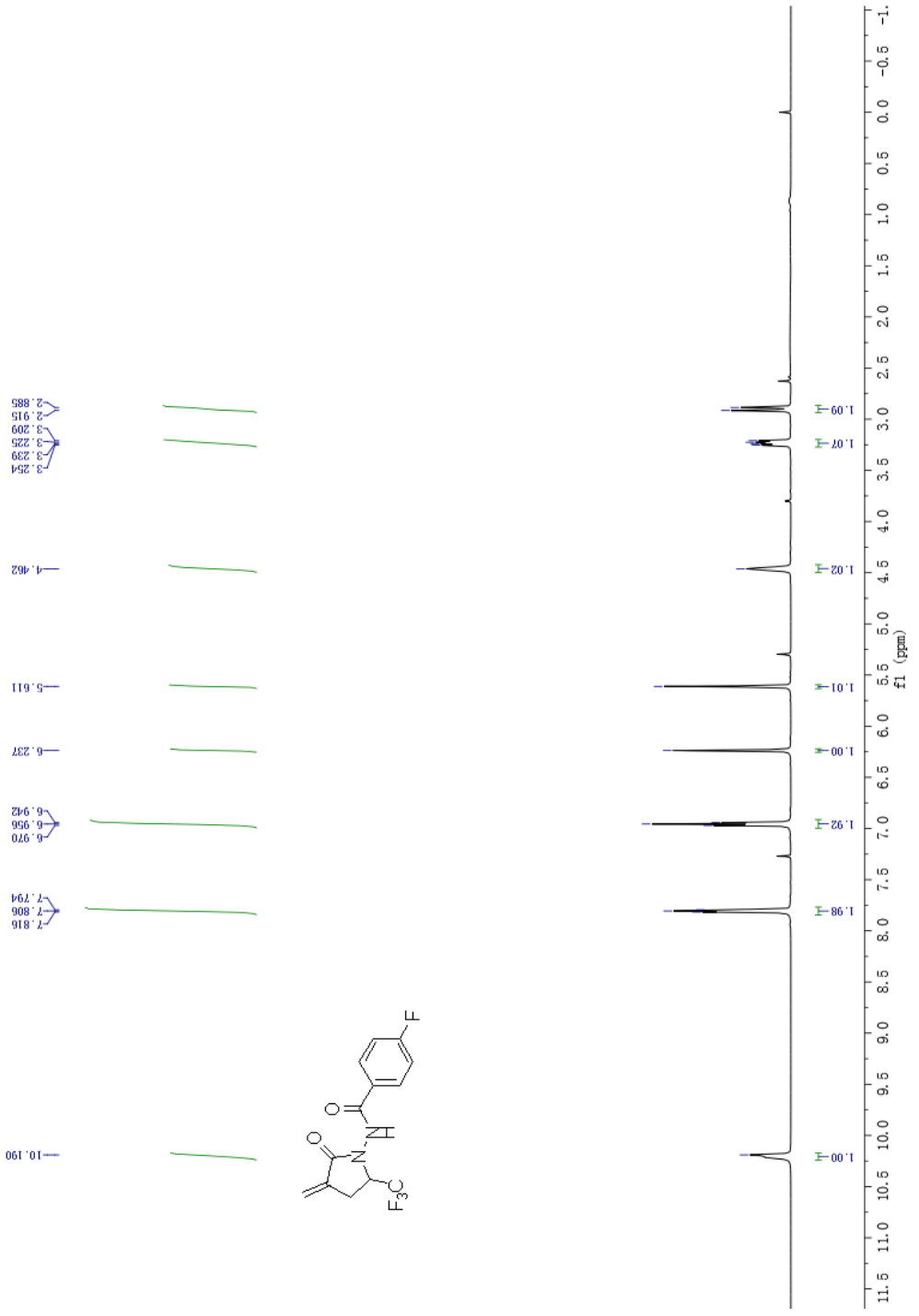


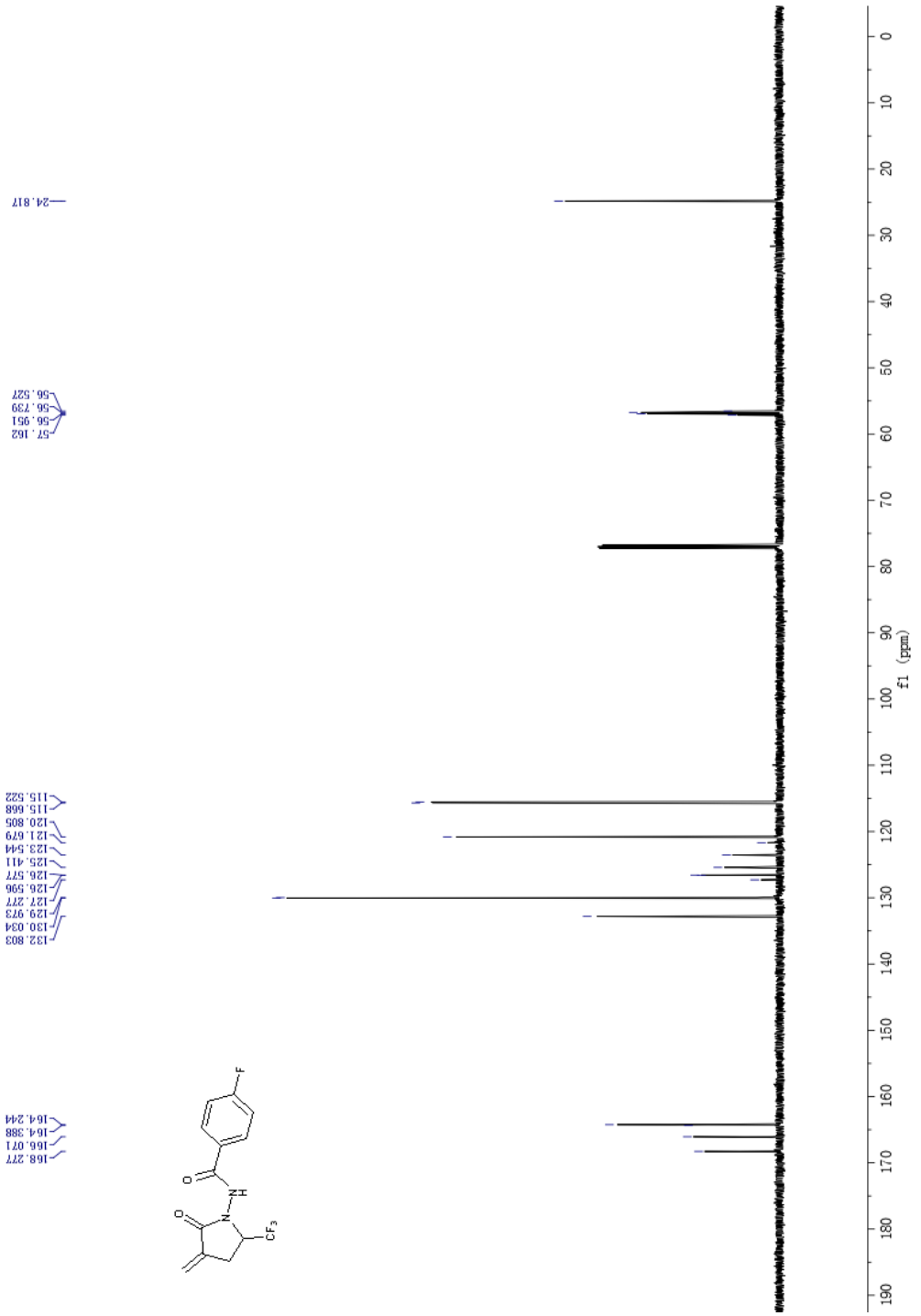

24.821
57.052
56.842
56.630
56.420
132.913
132.427
130.529
128.509
127.449
127.360
125.492
123.625
121.760
120.559
165.493
168.001
f1 (ppm)

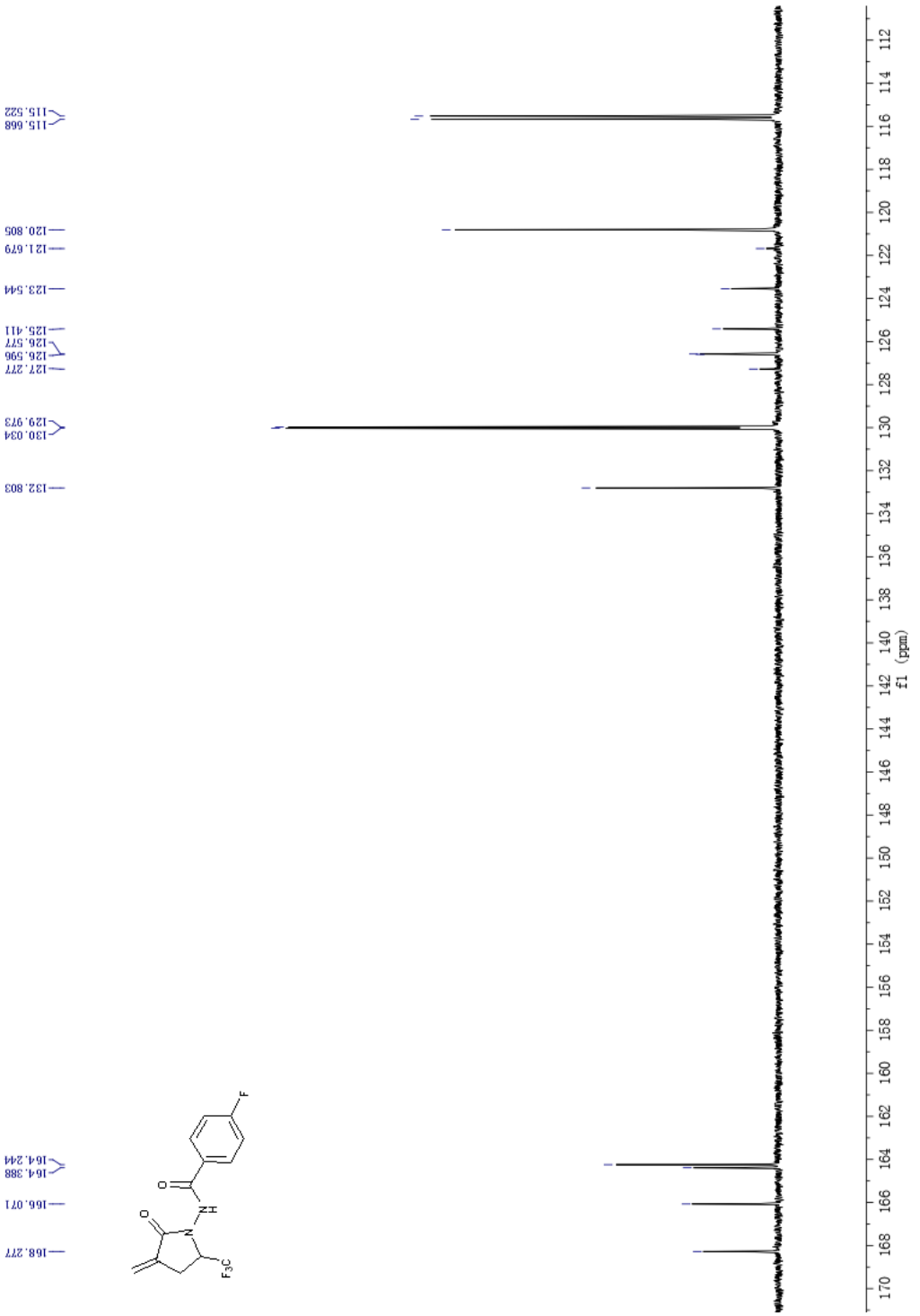

-76.900
-76.917
O
O
N
N
H
CF3
10
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190
f1 (ppm)

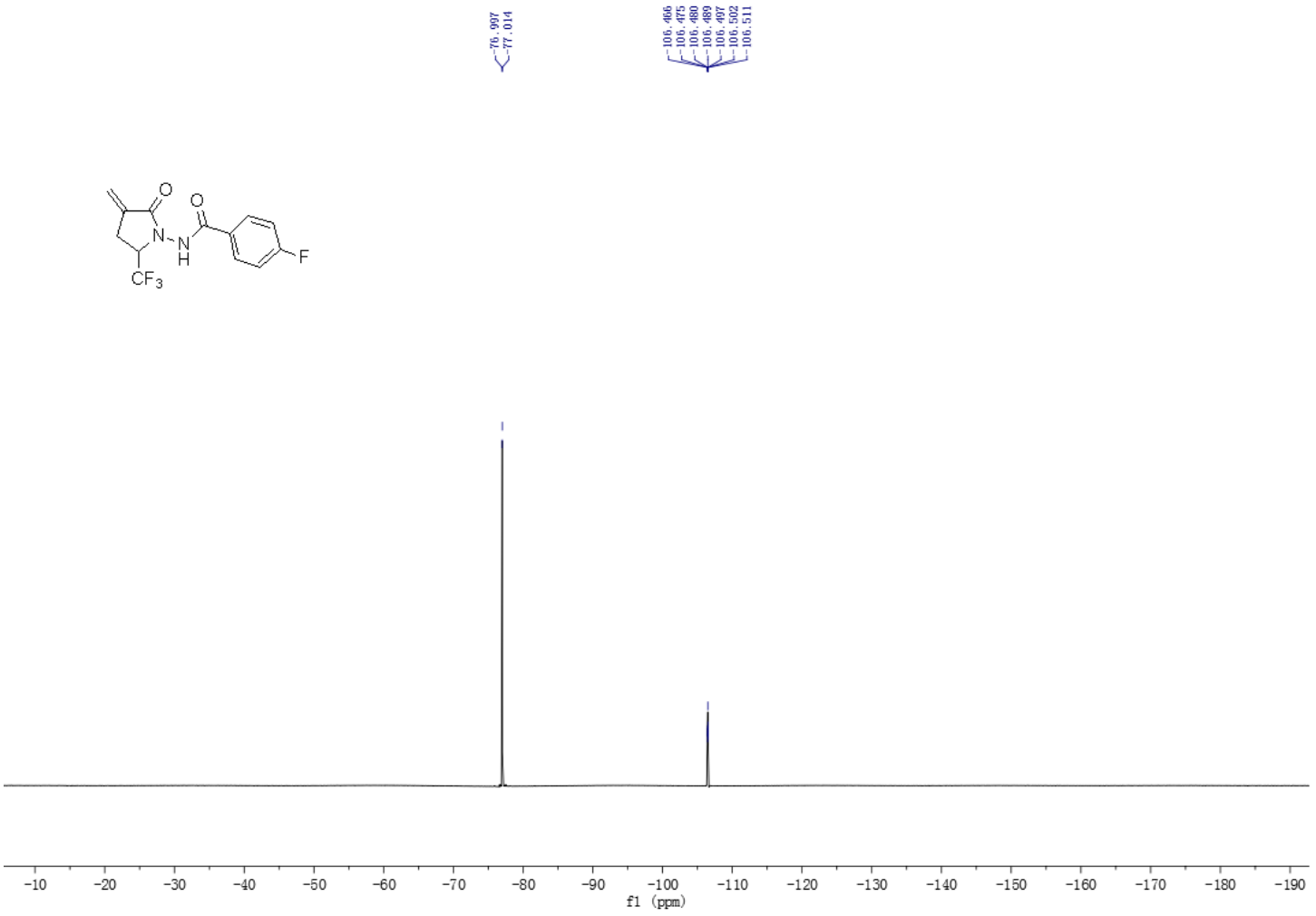

ESI positive, dgg13#, M+H=285.0845,error=1.1ppm, 23/07/2015
r.i.
0.40
0.30
0.20
0.10
0.00
285.0842
285.0976
287.1510
285.0080
295.8711
302.1110
307.0661
316.1266
317.0670
323.0662
280
290
300
310
320
m/z
/u/data/TRAINING/dgg150723/4/pdata/1 xspec Thu Jul 23 21:35:10 2015

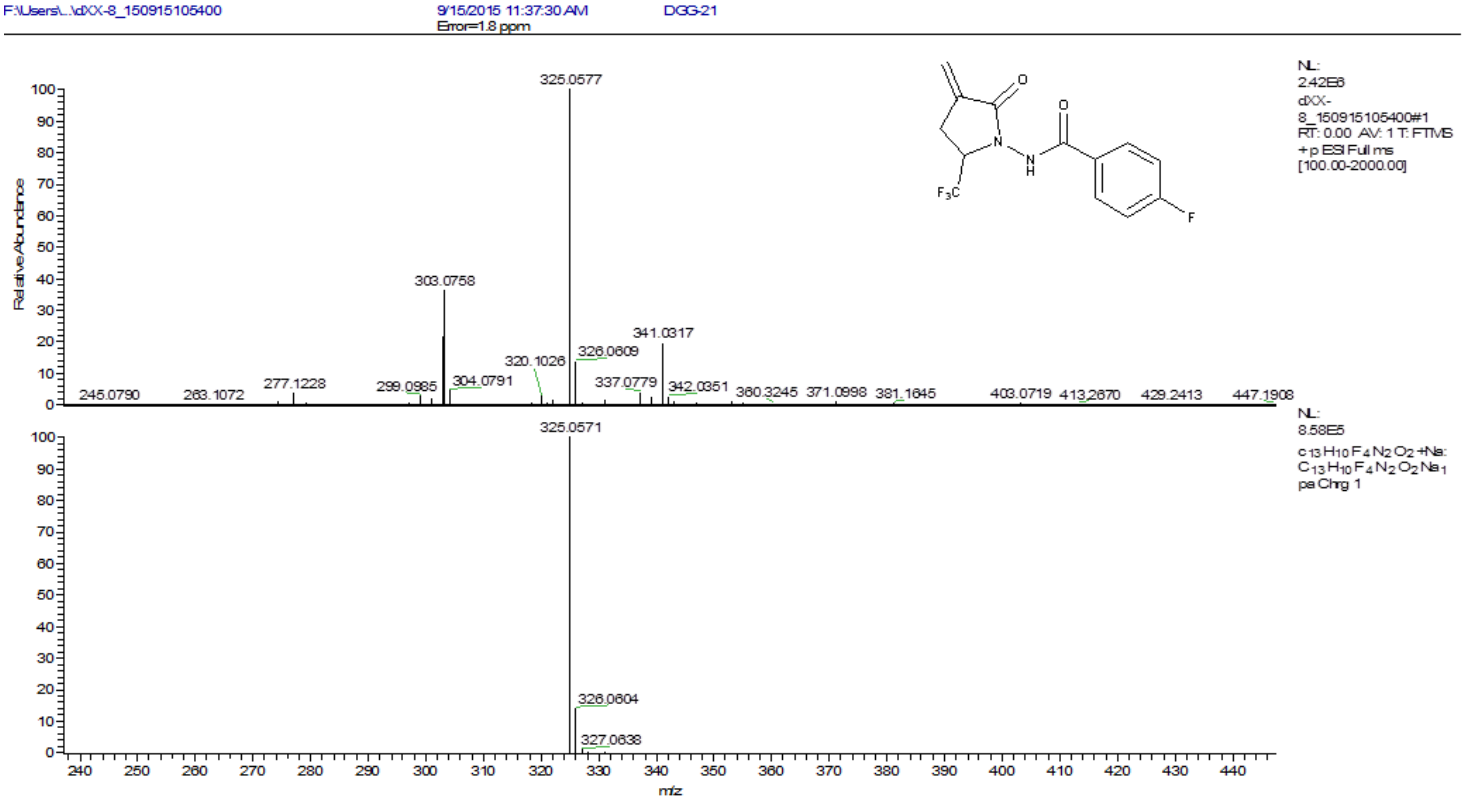
4-fluoro-N-(3-methylene-2-oxo-5-(trifluoromethyl)pyrrolidin-1-yl)benzamide(6b)



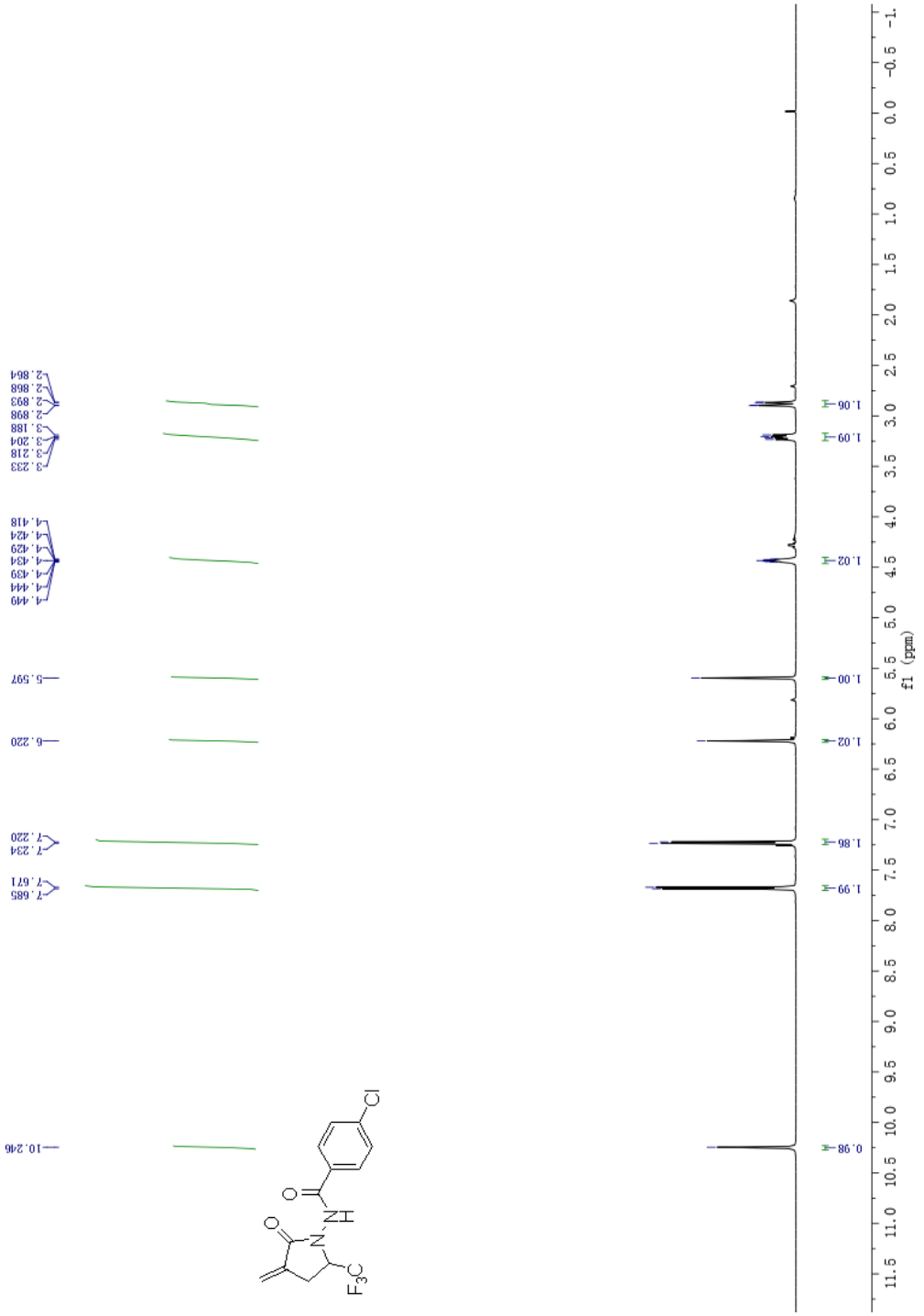

24.817
57.162
56.951
56.739
56.527
115.522
115.608
120.805
121.679
123.544
125.411
126.577
126.596
127.277
129.973
130.034
132.803
164.244
164.388
166.071
168.277
F
O
N
H
O
N
CF3
0
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
160
170
180
190
f1 (ppm)

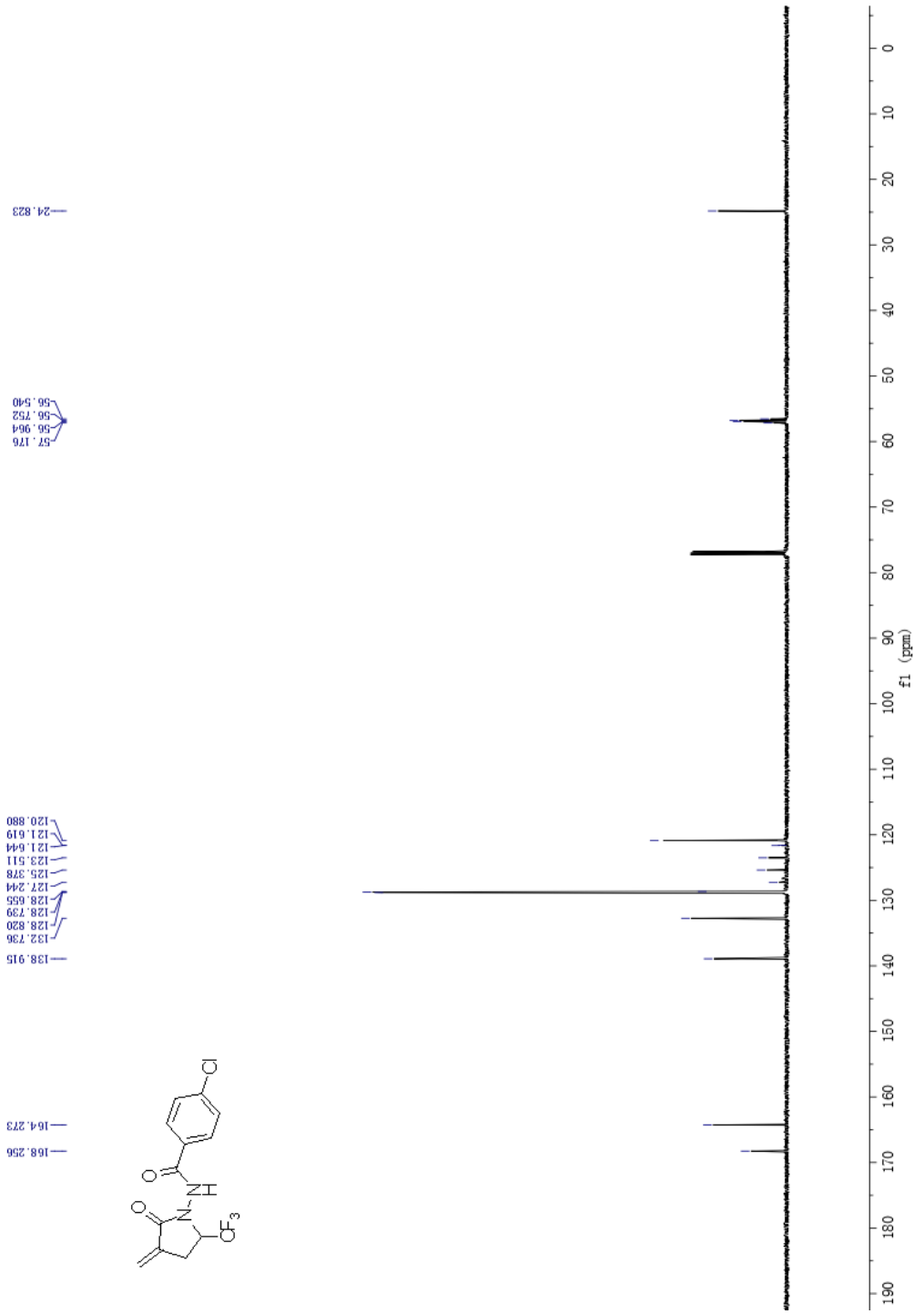

115.522
115.668
120.805
121.679
123.544
125.411
126.577
126.596
127.277
129.973
130.034
132.803
164.244
164.388
166.071
168.277
F
O
N
H
O
N
F3C
170
168
166
164
162
160
158
156
154
152
150
148
146
144
142
140
138
136
134
132
130
128
126
124
122
120
118
116
114
112
f1 (ppm)

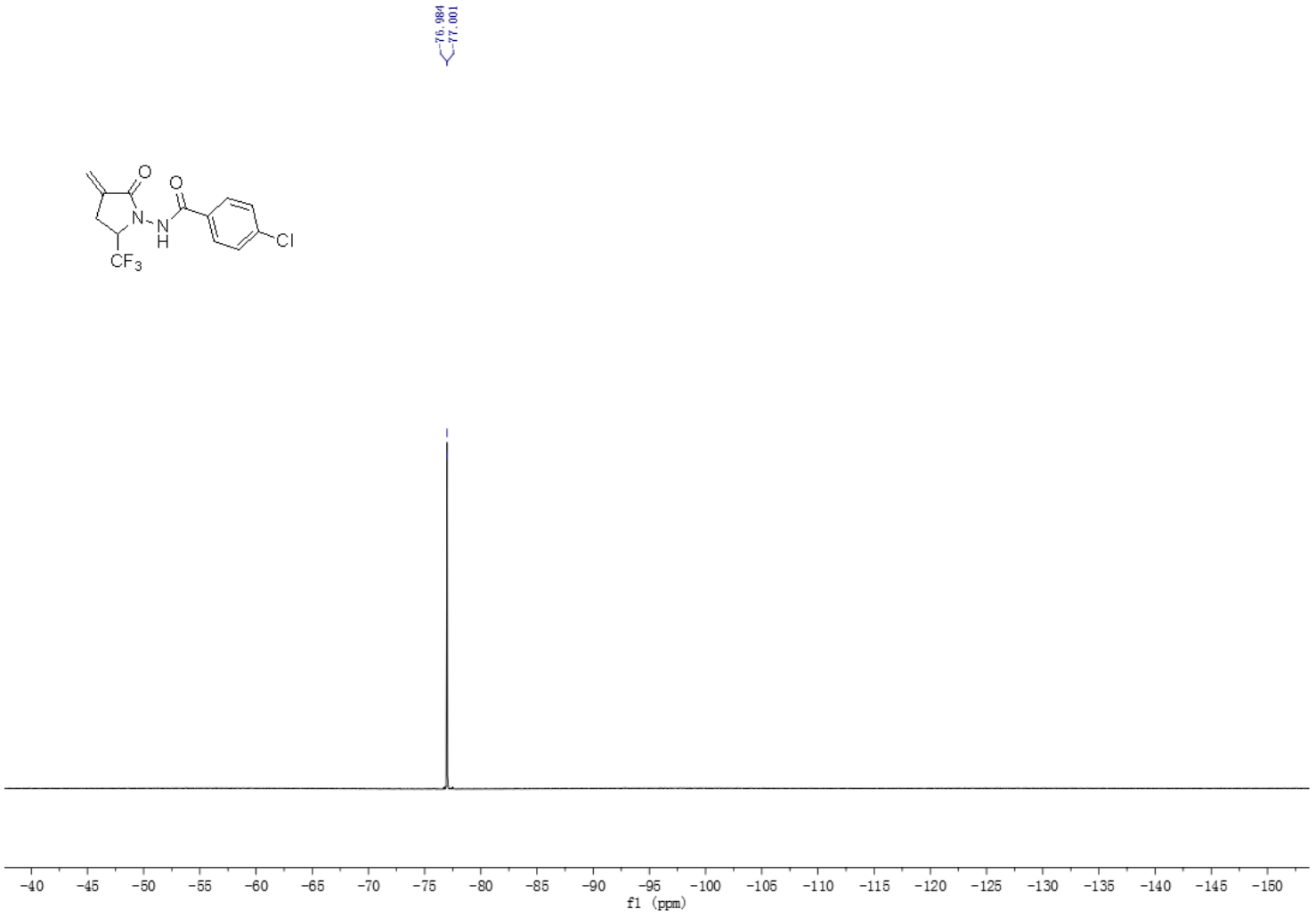

-76.997
-77.014
-106.466
-106.475
-106.480
-106.489
-106.497
-106.502
-106.511
O
O
N
N
H
CF3
F
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190
f1 (ppm)

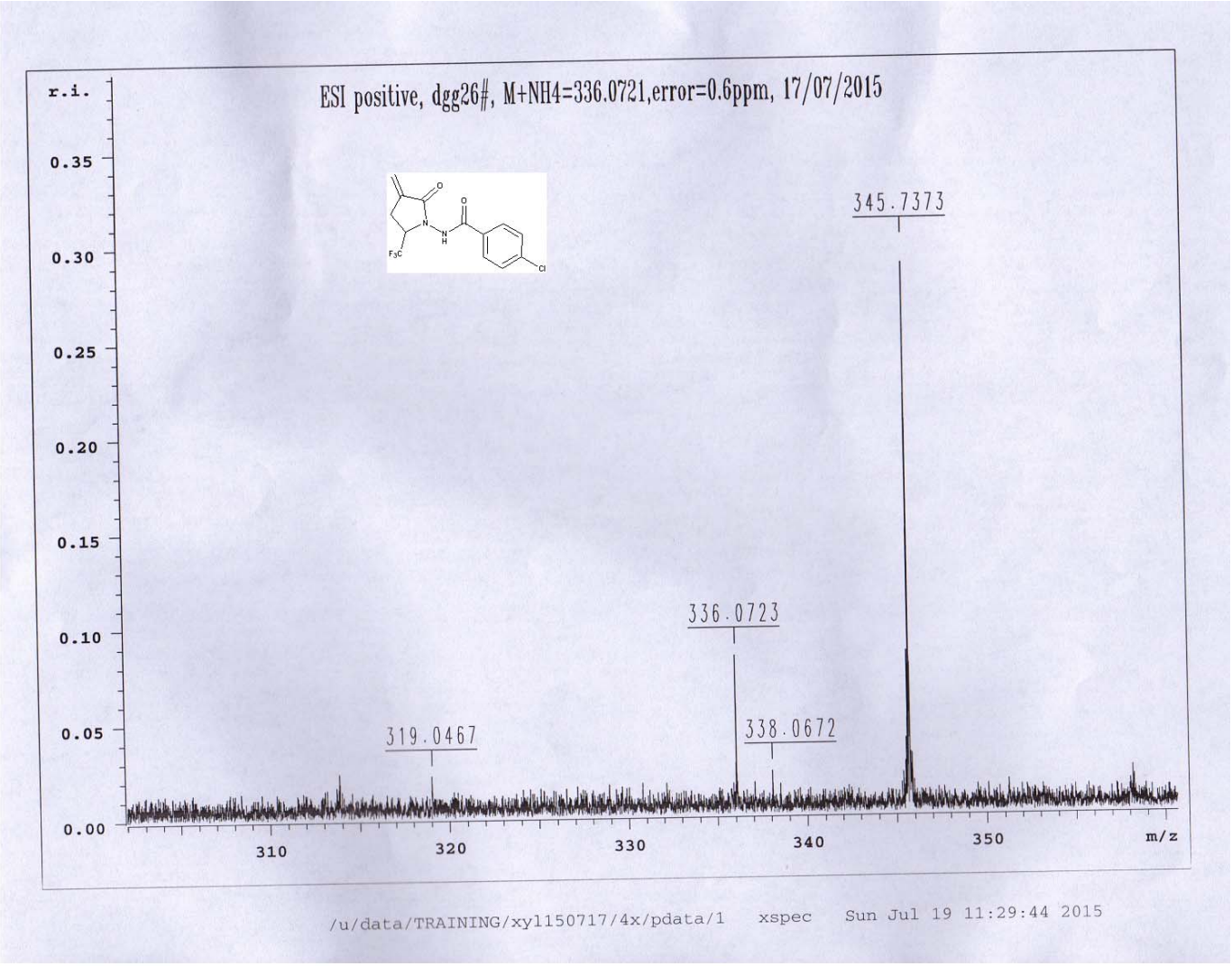

F:\Users\...\dXX-8_150915105400
9/15/2015 11:37:30 AM
Error=1.8 ppm
DGG-21
NL:
2.42E8
dXX-8_150915105400#1
RT: 0.00 AV: 1 T: FTMS + p ESI Full ms [100.00-2000.00]
325.0577
303.0758
341.0317
326.0609
320.1026
337.0779
342.0351
304.0791
299.0985
277.1228
245.0790
263.1072
360.3245
371.0998
381.1645
403.0719
413.2670
429.2413
447.1908
Relative Abundance
NL:
8.58E5
C13H10F4N2O2+Na:
C13H10F4N2O2Na1
pa Chrg 1
325.0571
326.0604
327.0638
m/z

4-chloro-N-(3-methylene-2-oxo-5-(trifluoromethyl)pyrrolidin-1-yl)benzamide(6c)

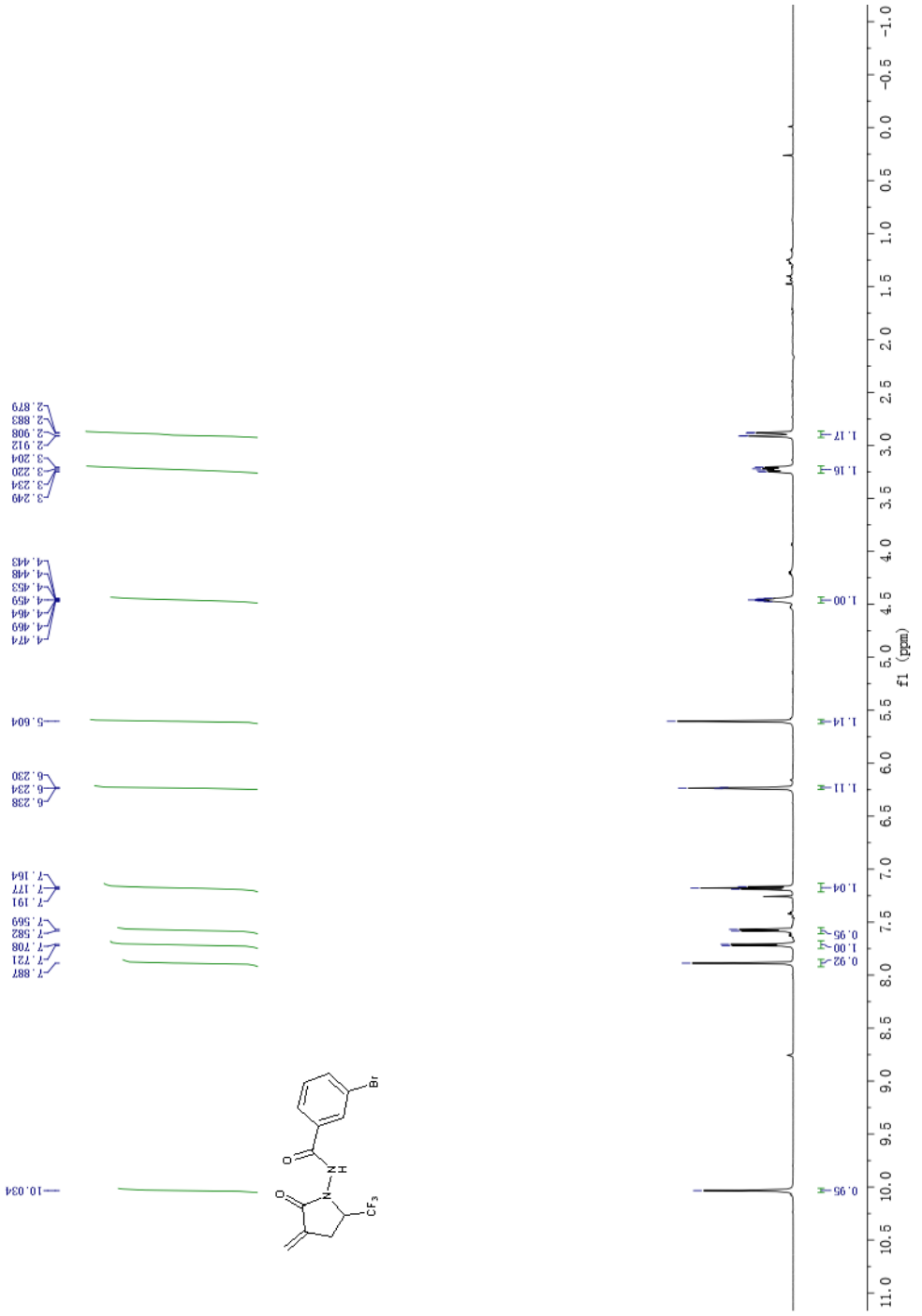


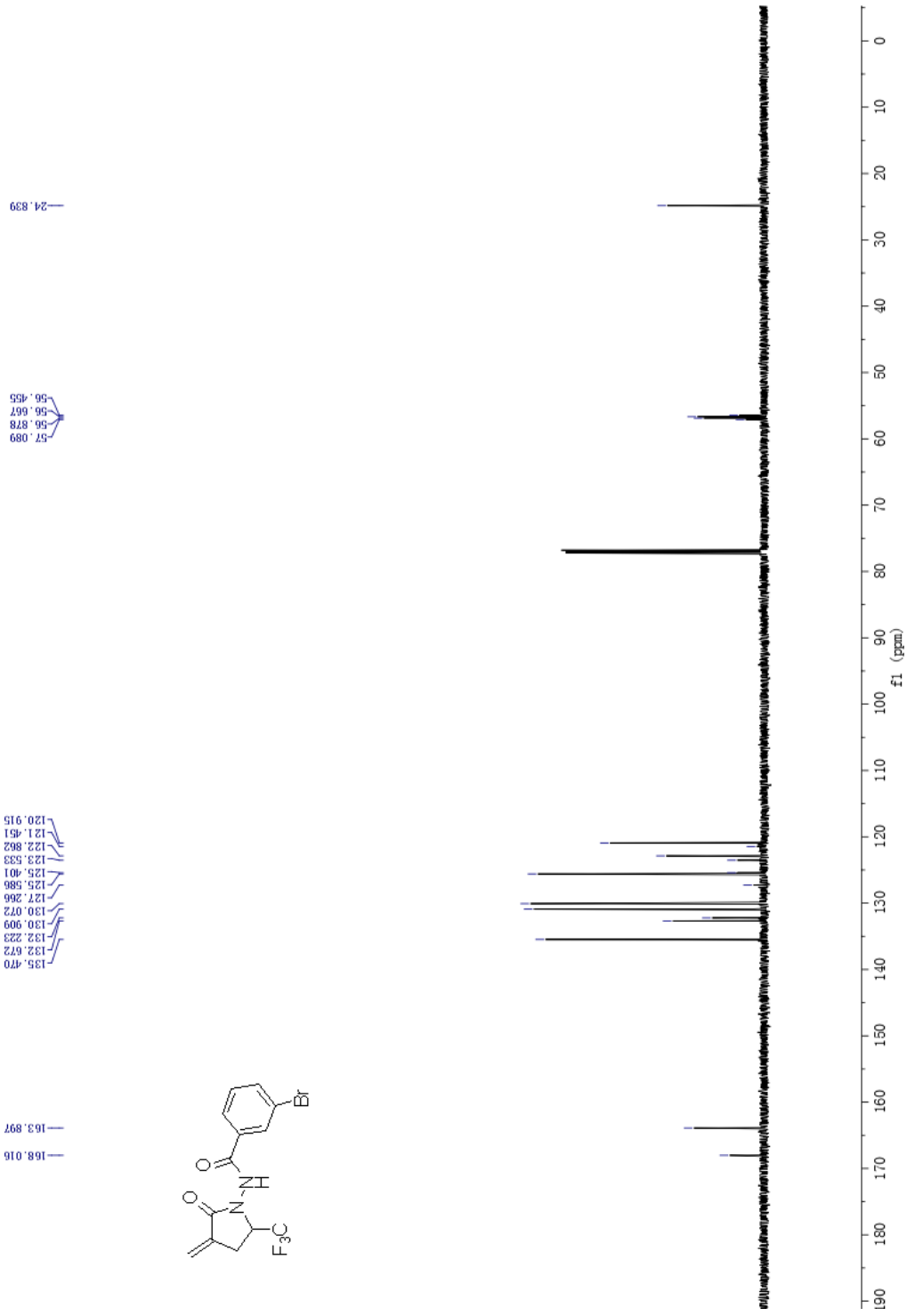

24.823
57.176
56.964
56.752
56.540
120.880
121.619
121.644
123.511
125.378
127.244
128.655
128.739
128.820
132.736
138.915
164.273
168.256
Cl
O
N
NH
O
CF3
0
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
160
170
180
190
f1 (ppm)

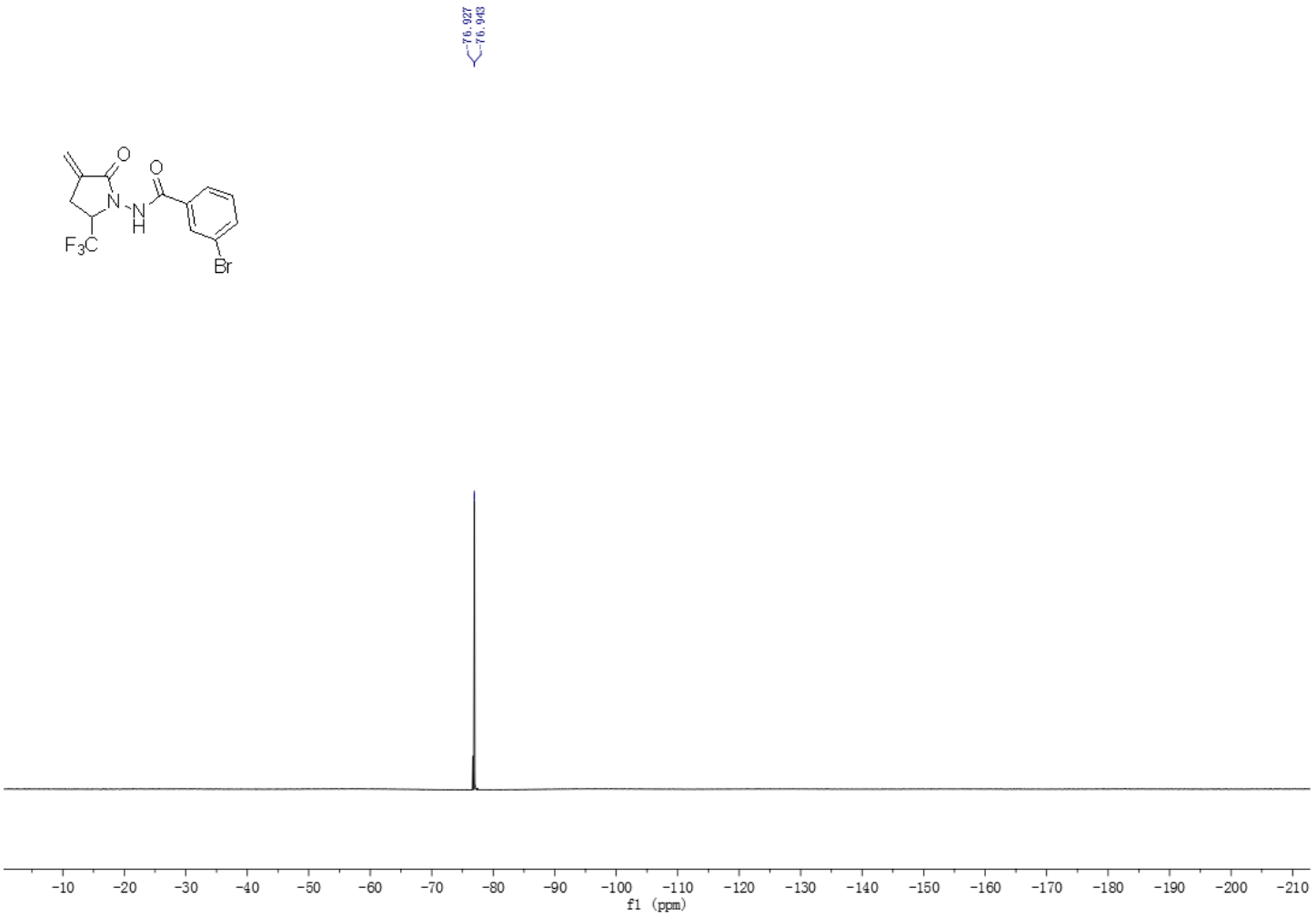

-76.984
-77.001
O
O
N
N
H
Cl
CF3
-40
-45
-50
-55
-60
-65
-70
-75
-80
-85
-90
-95
-100
-105
-110
-115
-120
-125
-130
-135
-140
-145
-150
f1 (ppm)

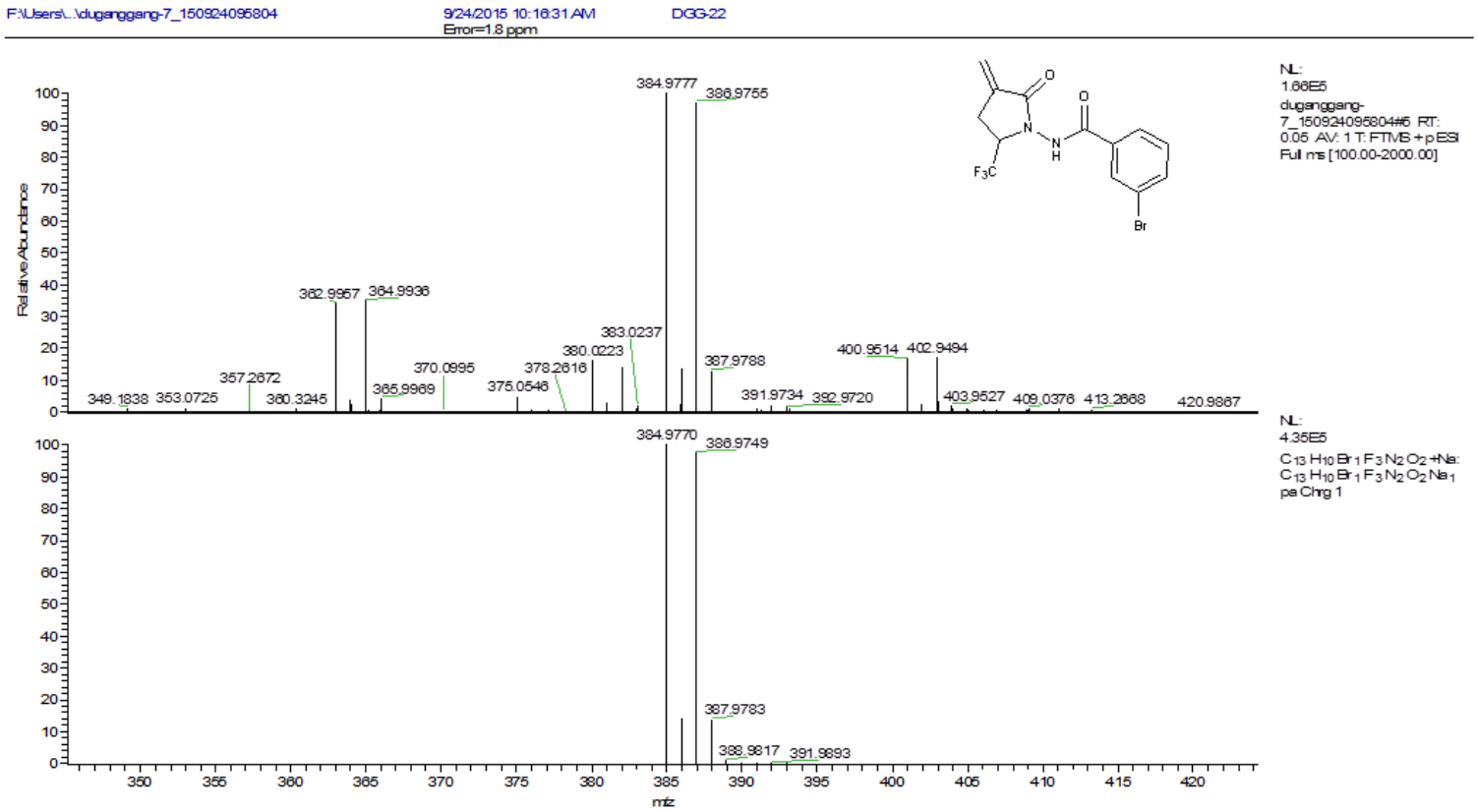

ESI positive, dgg26#, M+NH4=336.0721,error=0.6ppm, 17/07/2015
r.i.
0.35
0.30
0.25
0.20
0.15
0.10
0.05
0.00
345.7373
336.0723
338.0672
319.0467
310
320
330
340
350
m/z
/u/data/TRAINING/xy1150717/4x/pdata/1 xspec Sun Jul 19 11:29:44 2015

3-bromo-N-(3-methylene-2-oxo-5-(trifluoromethyl)pyrrolidin-1-yl)benzamide(6d)

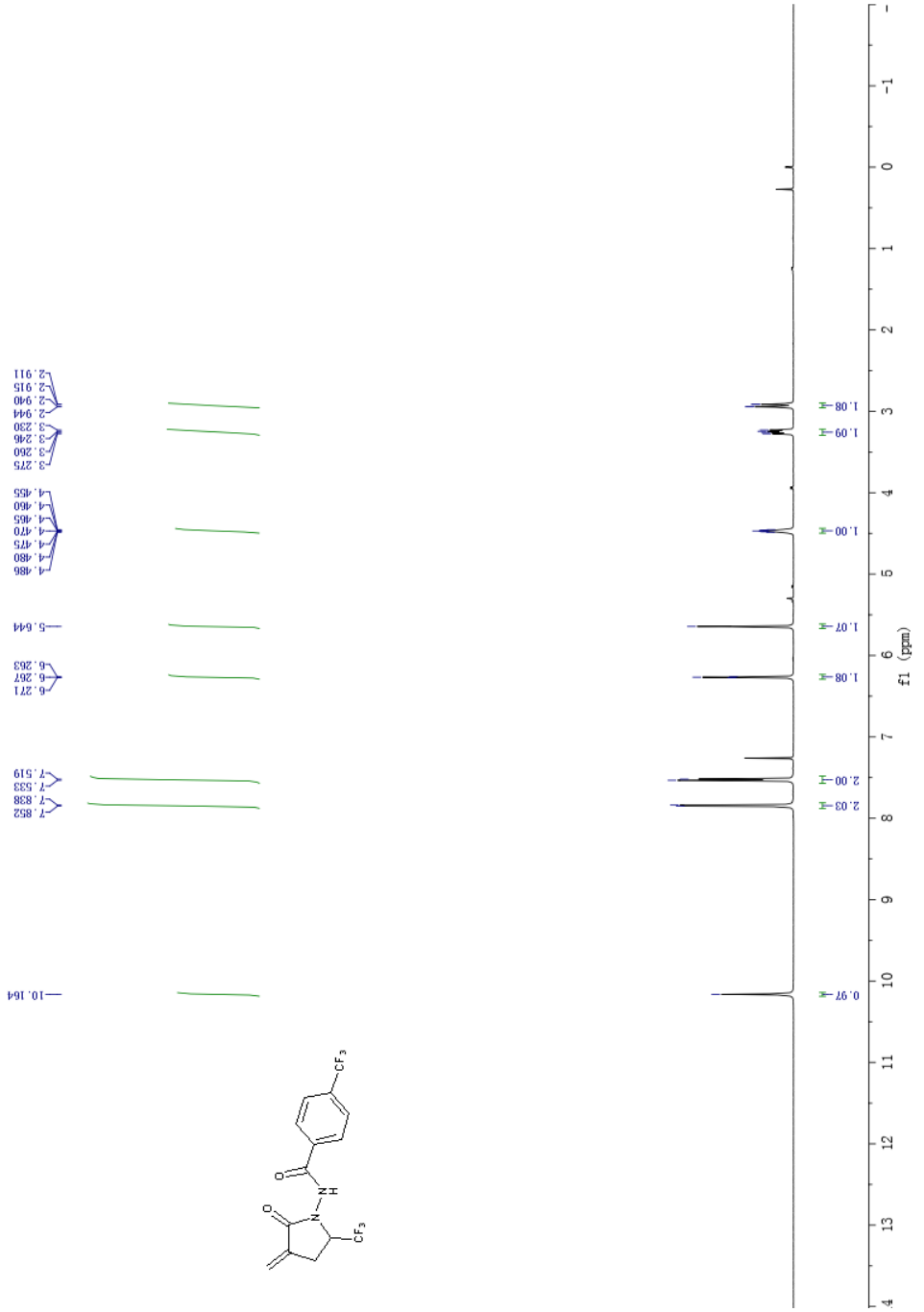


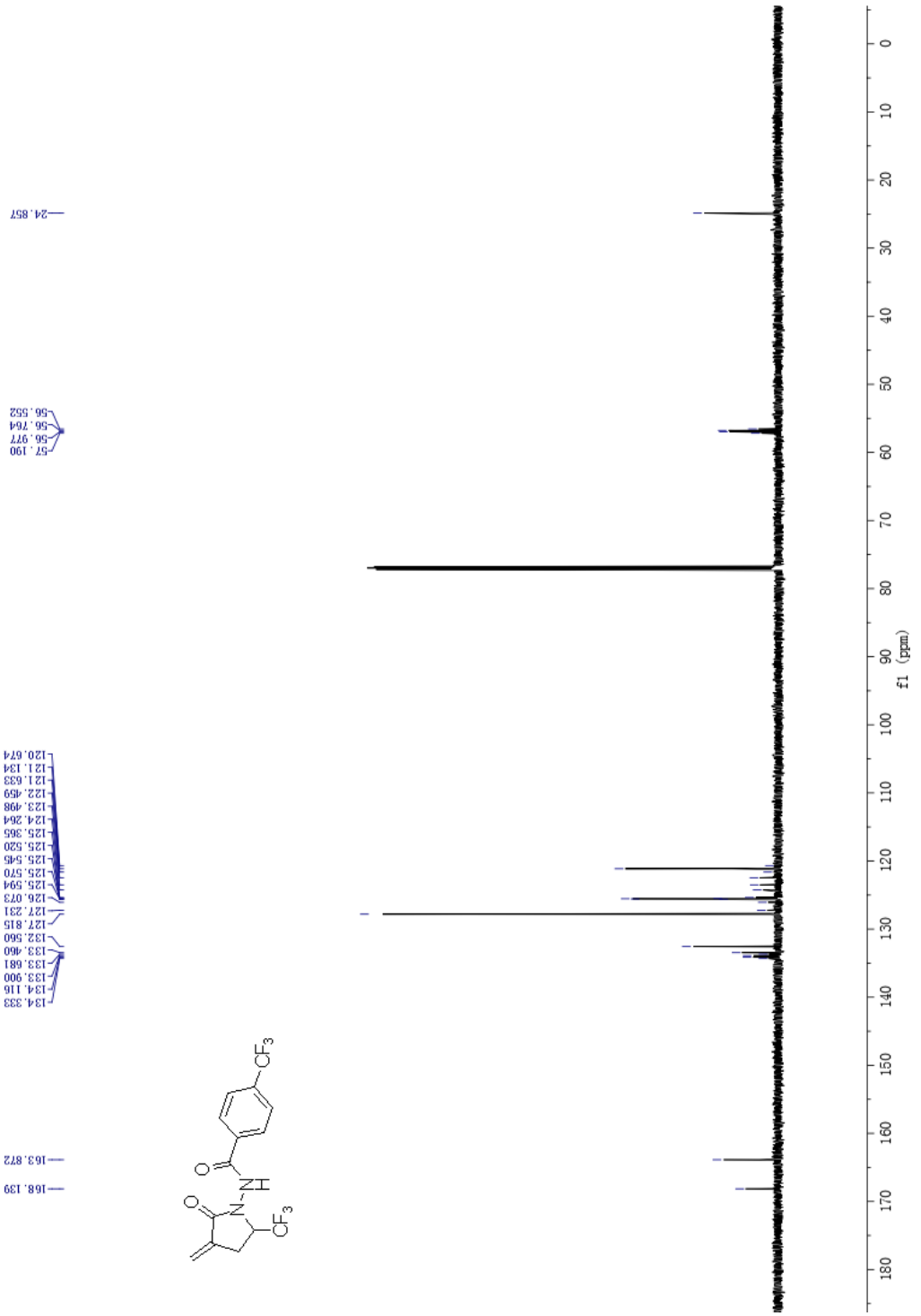

24.839
56.455
56.667
56.878
57.089
120.915
121.451
122.862
123.533
125.401
125.586
127.266
130.072
130.909
132.223
132.672
135.470
163.897
168.016
Br
O
N
H
O
F3C
f1 (ppm)

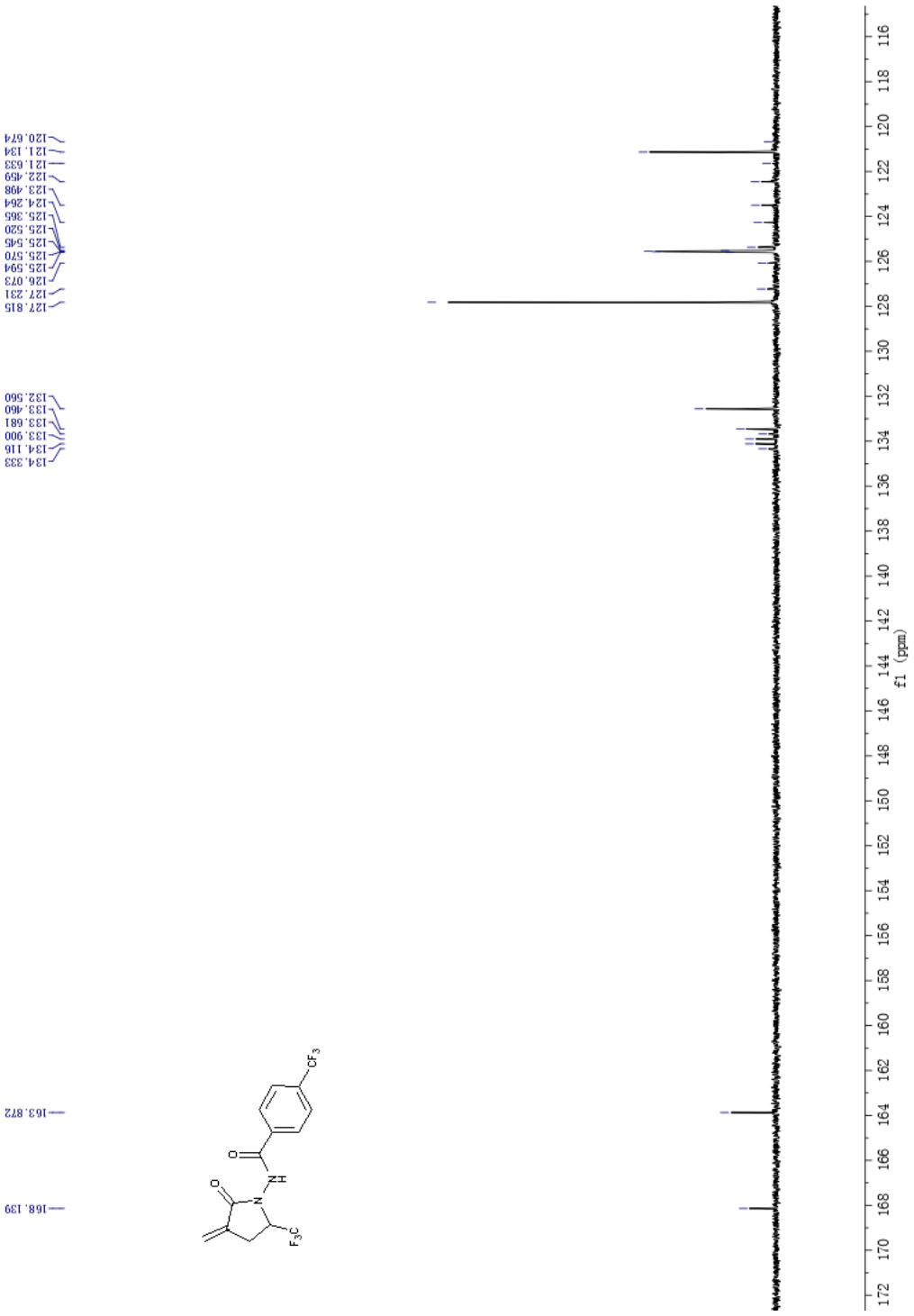

-76.927
-76.943
O
O
N
N
H
F3C
Br
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190
-200
-210
f1 (ppm)

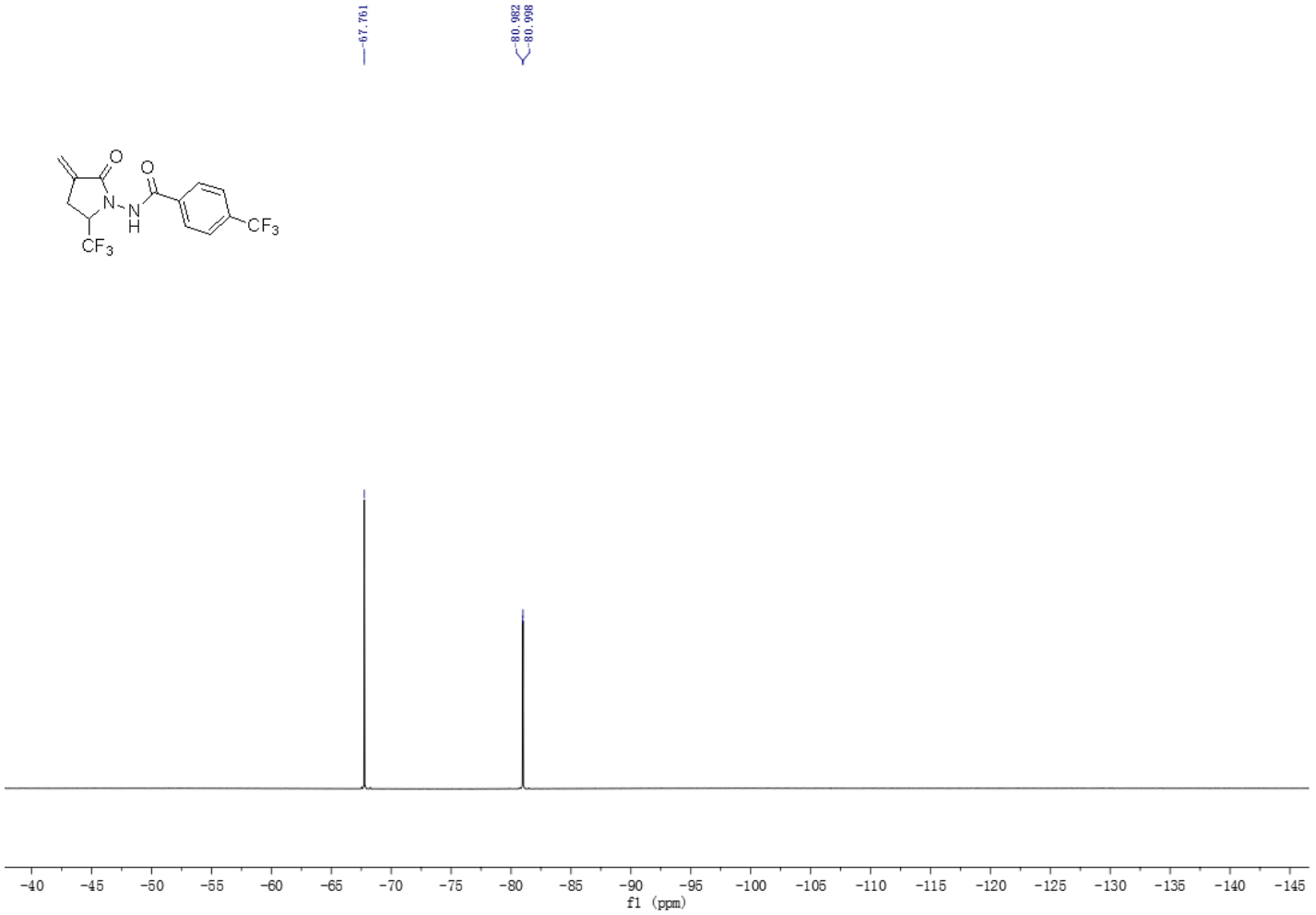

F:\Users\..\duganggang-7_150924095804
9/24/2015 10:16:31 AM
Error=1.8 ppm
DGG-22
NL:
1.66E5
duganggang-7_150924095804#6 RT: 0.05 AV: 1 T: FTMS + p ESI Full ms [100.00-2000.00]
Relative Abundance
384.9777
386.9755
382.9957
364.9936
383.0237
380.0223
387.9788
400.9514
402.9494
370.0995
378.2616
357.2672
375.0546
365.9969
349.1838
353.0725
360.3245
391.9734
392.9720
403.9527
409.0376
413.2668
420.9867
NL:
4.35E5
C13 H10 Br1 F3 N2 O2 +Na: C13 H10 Br1 F3 N2 O2 Na1 pa Chrg 1
384.9770
386.9749
387.9783
388.9817
391.9893
m/z

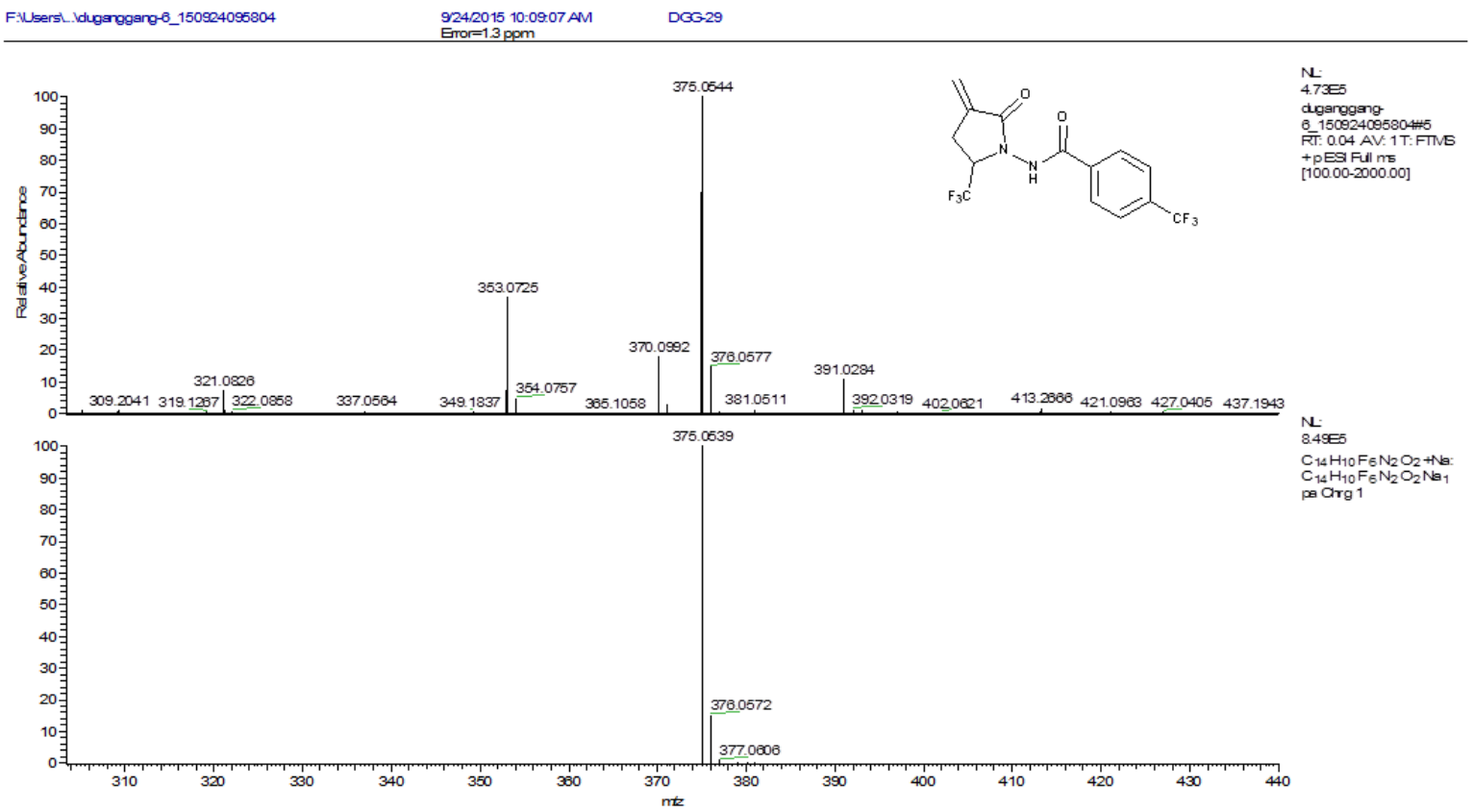
N-(3-methylene-2-oxo-5-(trifluoromethyl)pyrrolidin-1-yl)-4-(trifluoromethyl)benzamide(6e)



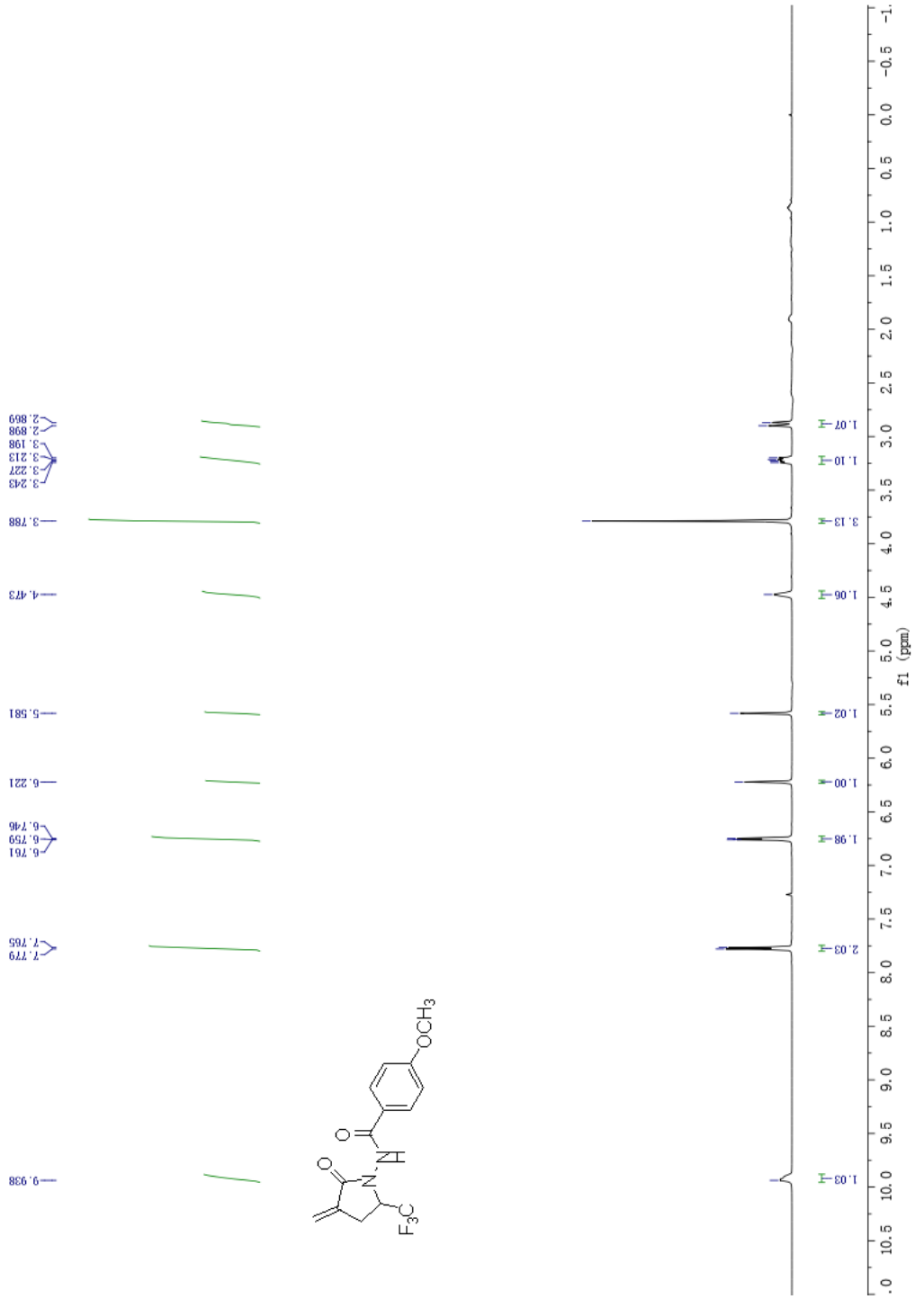

24.857
56.552
56.764
56.977
57.190
120.674
121.134
121.633
122.459
123.498
124.264
125.365
125.520
125.546
125.570
125.594
126.073
127.231
127.815
132.560
133.460
133.681
133.900
134.116
134.333
163.872
168.139
O
N
N
H
O
CF3
CF3
0
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
160
170
180
f1 (ppm)

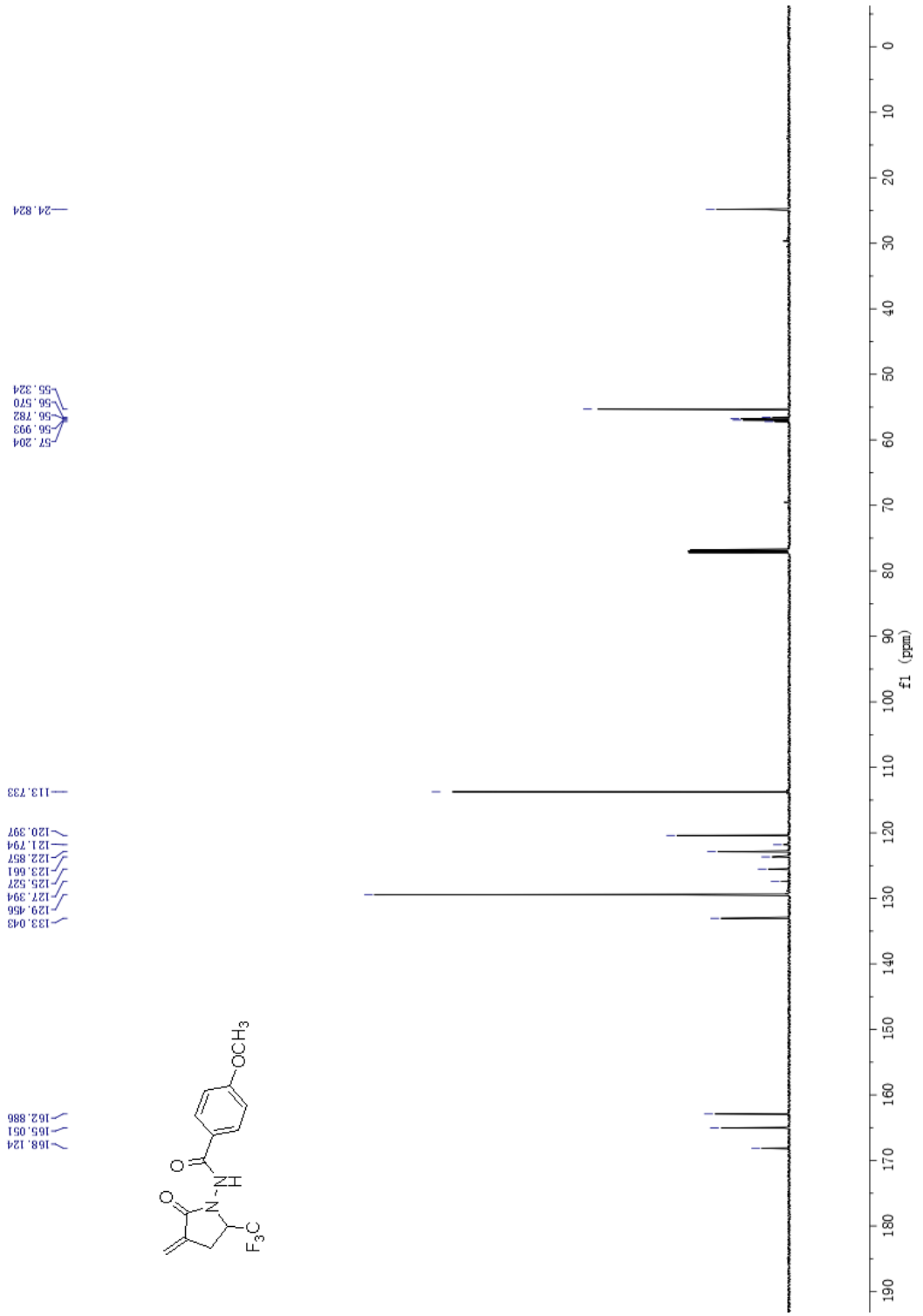

168.139
163.872
134.333
134.116
133.900
133.681
133.460
132.560
127.815
127.231
126.073
125.594
125.570
125.546
125.520
125.365
124.264
123.498
122.459
121.633
121.134
120.674
O
N
H
O
F3C
CF3
f1 (ppm)

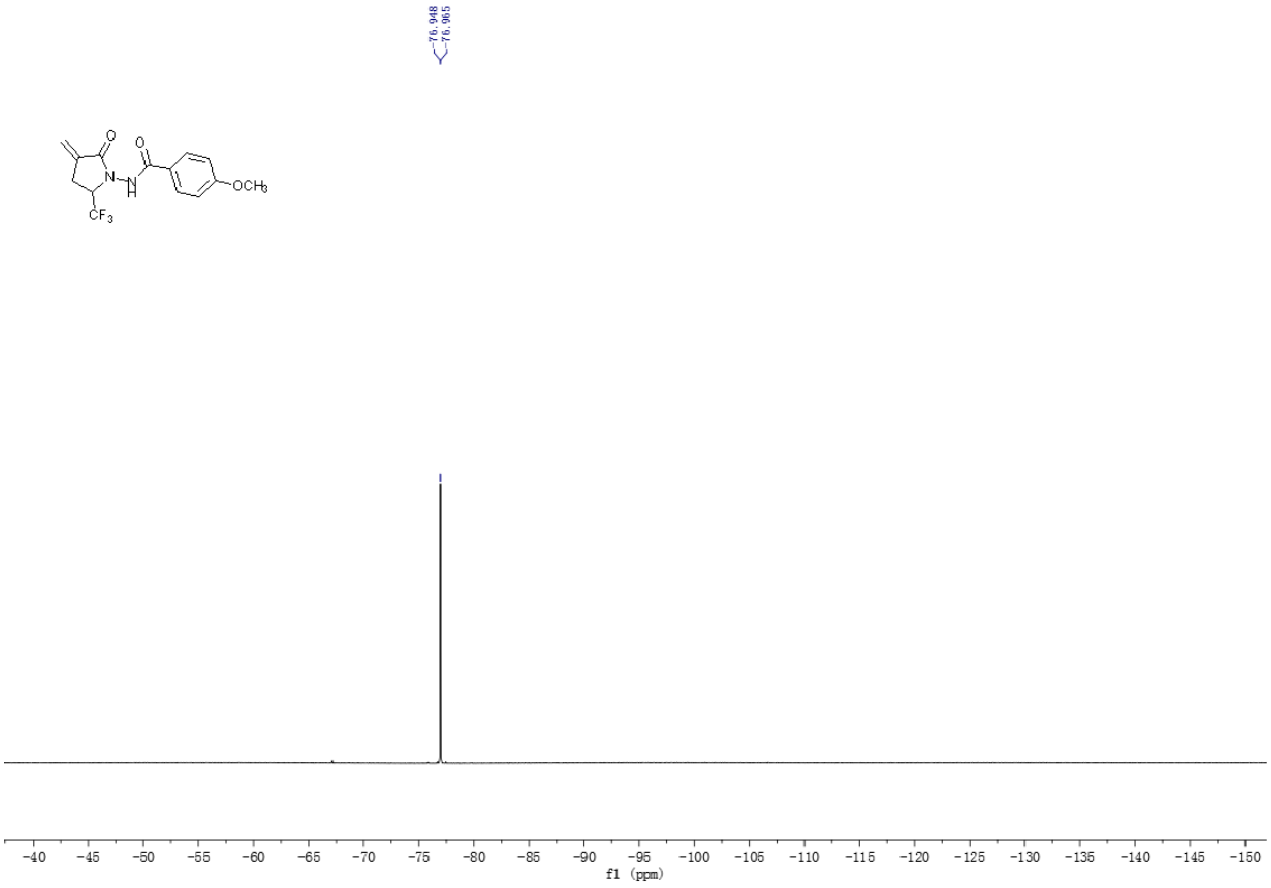

-67.761
-80.982
-80.998
O
O
N
N
H
CF3
CF3
-40
-45
-50
-55
-60
-65
-70
-75
-80
-85
-90
-95
-100
-105
-110
-115
-120
-125
-130
-135
-140
-145
f1 (ppm)

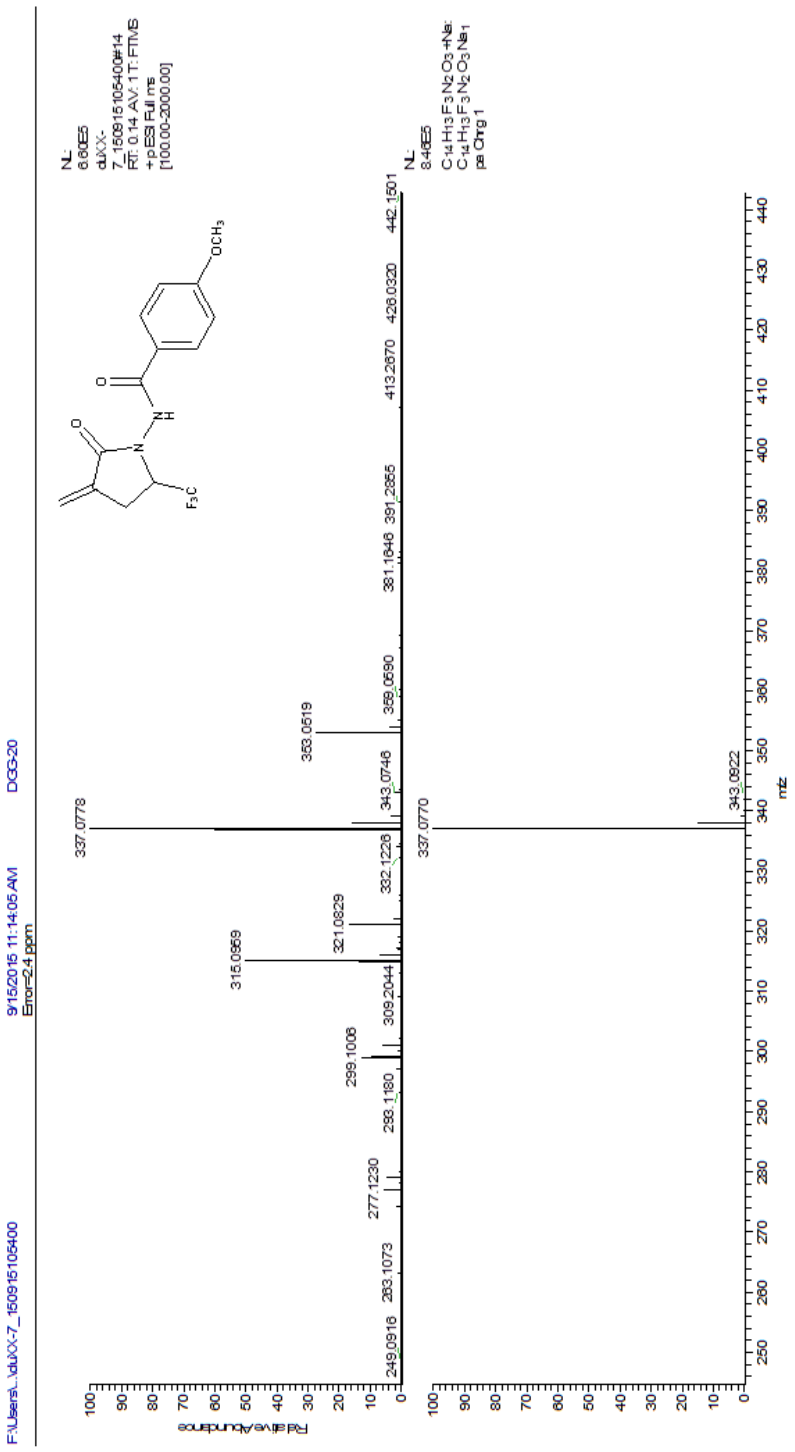

F:\Users\...\duganggang-6_150924095804
9/24/2015 10:09:07 AM
Error=1.3 ppm
DGG-29
NL:
4.73E5
duganggang-6_150924095804#5
RT: 0.04 AV: 1 T: FTMS + p ESI Full ms [100.00-2000.00]
NL:
8.49E5
C14 H10 F6 N2 O2 +Na: C14 H10 F6 N2 O2 Na1 pa Chrg 1
Relative Abundance
m/z
375.0544
353.0725
370.0992
376.0577
391.0284
321.0826
354.0757
309.2041
319.1287
322.0858
337.0564
349.1837
365.1058
381.0511
392.0319
402.0621
413.2866
421.0963
427.0405
437.1943
375.0539
376.0572
377.0606

4-methoxy-N-(3-methylene-2-oxo-5-(trifluoromethyl)pyrrolidin-1-yl)benzamide(6f)

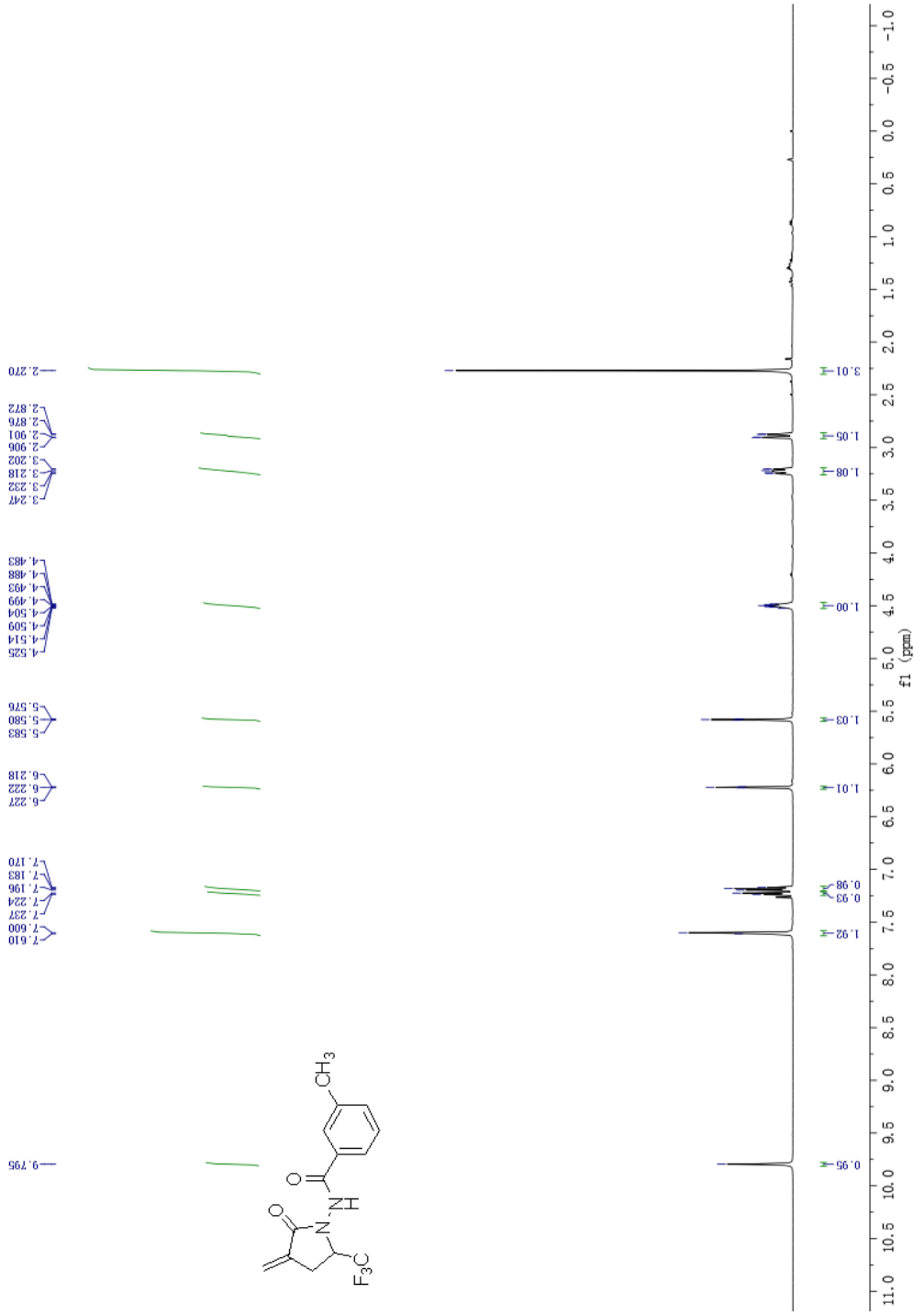


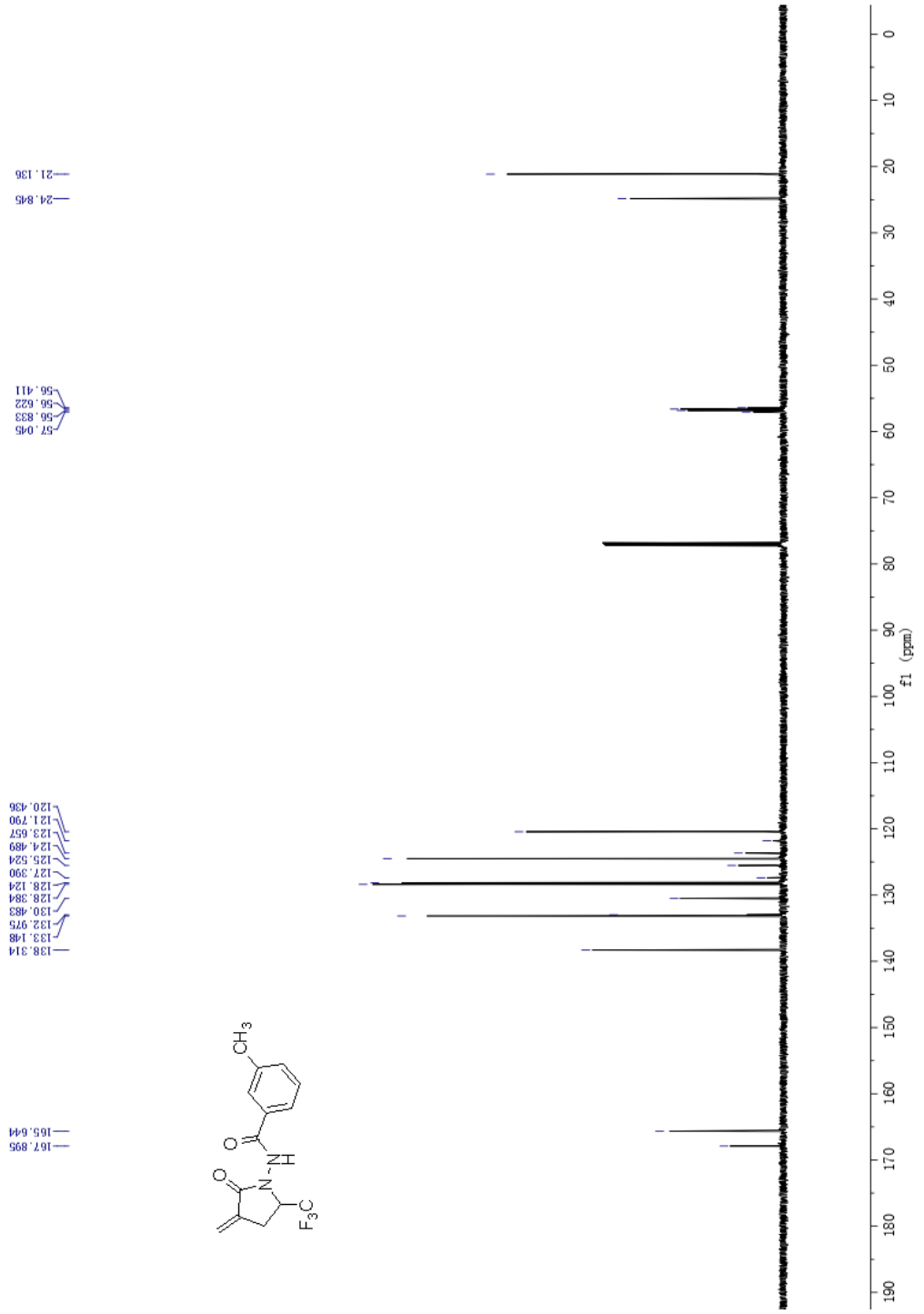

24.824
55.324
56.570
56.782
56.993
57.204
113.733
120.397
121.794
122.857
123.661
125.527
127.394
129.456
133.043
162.886
165.051
168.124
O
N
H
O
OCH3
F3C
f1 (ppm)

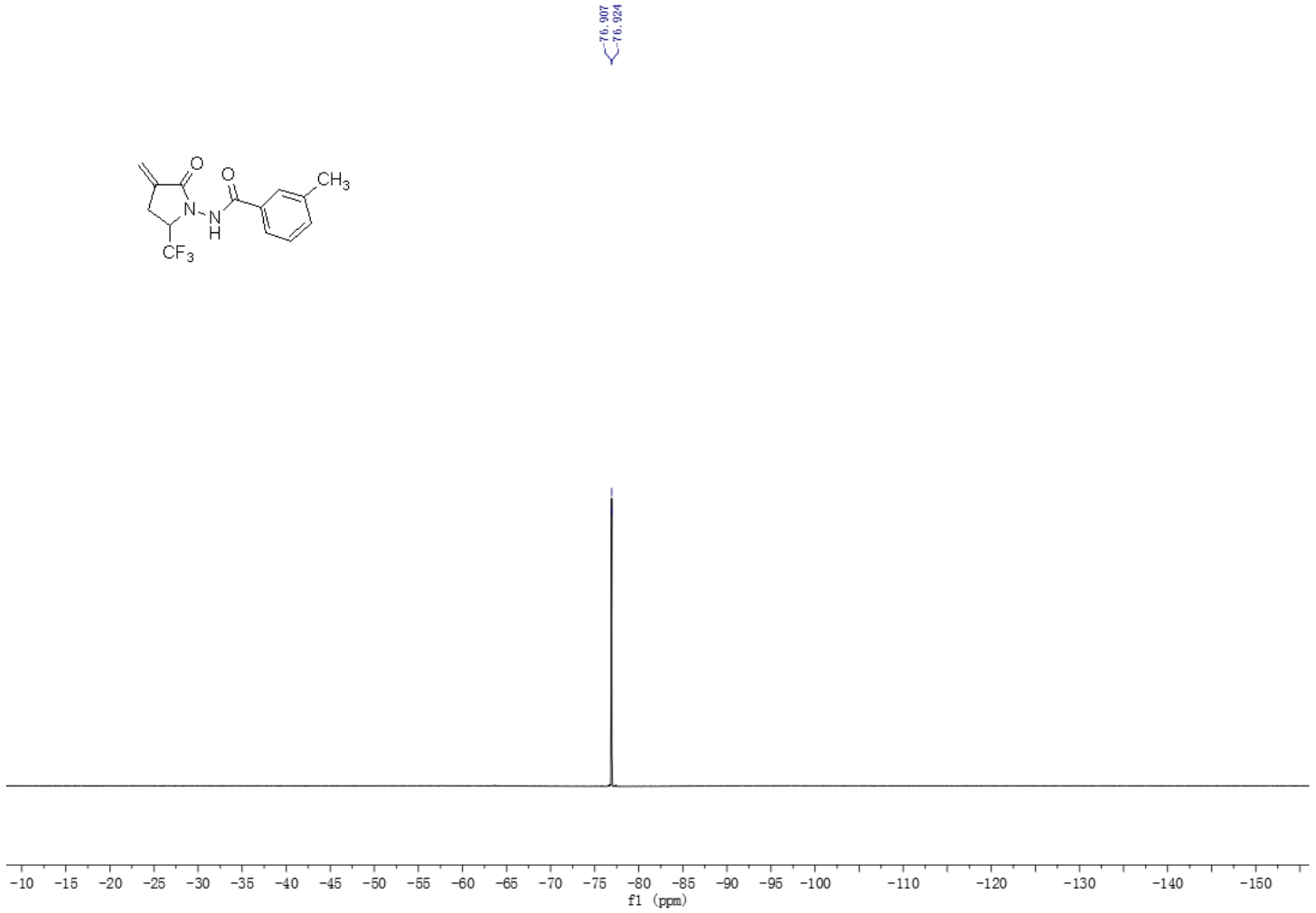

-76.948
-76.965
O
O
N
H
OCH3
CF3
-40 -45 -50 -55 -60 -65 -70 -75 -80 -85 -90 -95 -100 -105 -110 -115 -120 -125 -130 -135 -140 -145 -150
f1 (ppm)

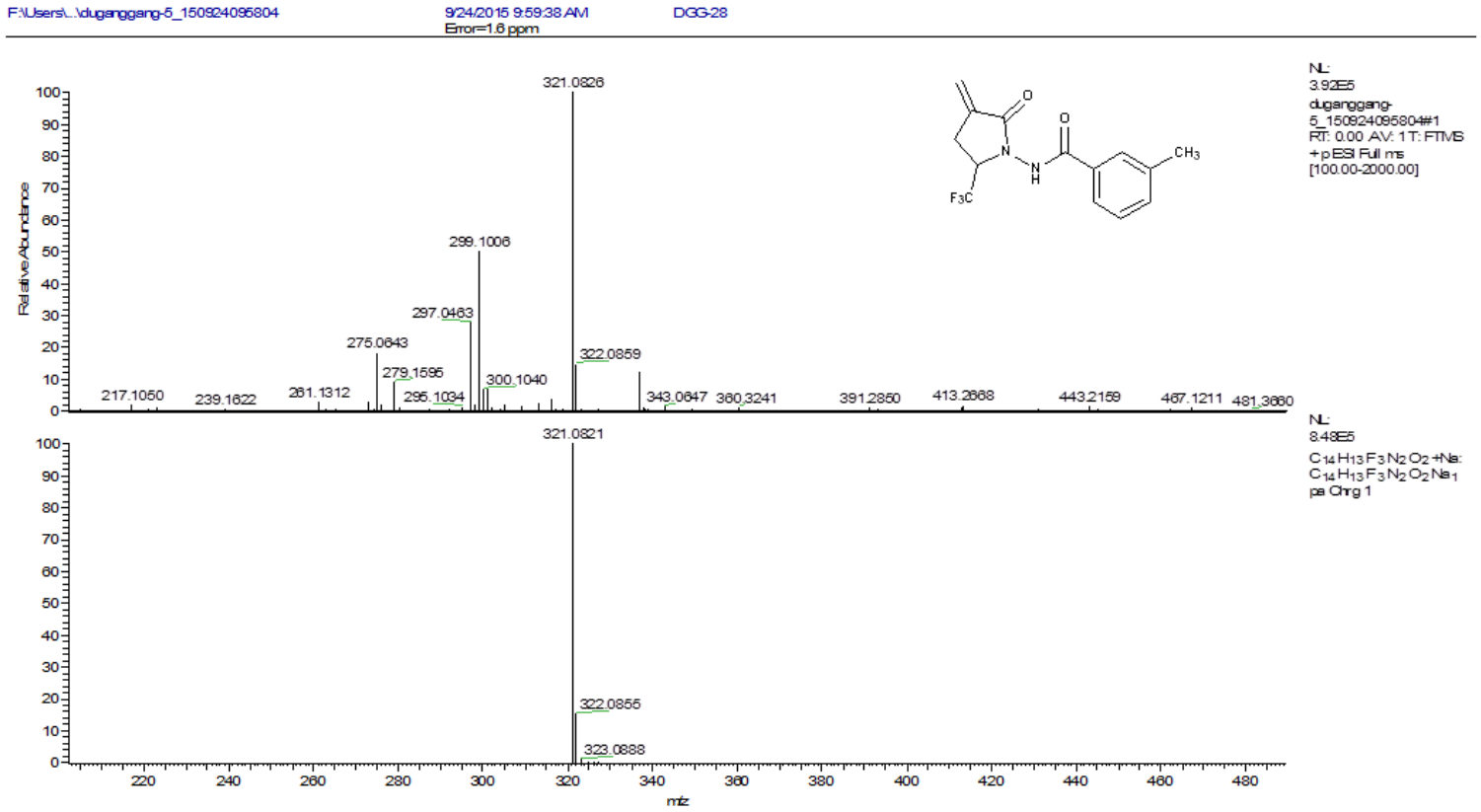

F:\Users\...\duX-7_150915105400
9/15/2015 11:14:05 AM
Error=2.4 ppm
DG3-20
NL:
8.60E5
duX-
7_150915105400#14
RT: 0.14 AV: 1 T: FTMS
+ p ESI Full ms
[100.00-2000.00]
NL:
8.46E5
C14H13F3N2O3+Na
C14H13F3N2O3Na1
pa Chrg 1
Relative Abundance
m/z
337.0778
353.0519
315.0959
321.0829
332.1228
343.0746
359.0690
381.1848
391.2855
413.2670
426.0320
442.1501
249.0918
283.1073
277.1230
288.1180
299.1006
309.2044
337.0770
343.0922

3-methyl-N-(3-methylene-2-oxo-5-(trifluoromethyl)pyrrolidin-1-yl)benzamide(6h)

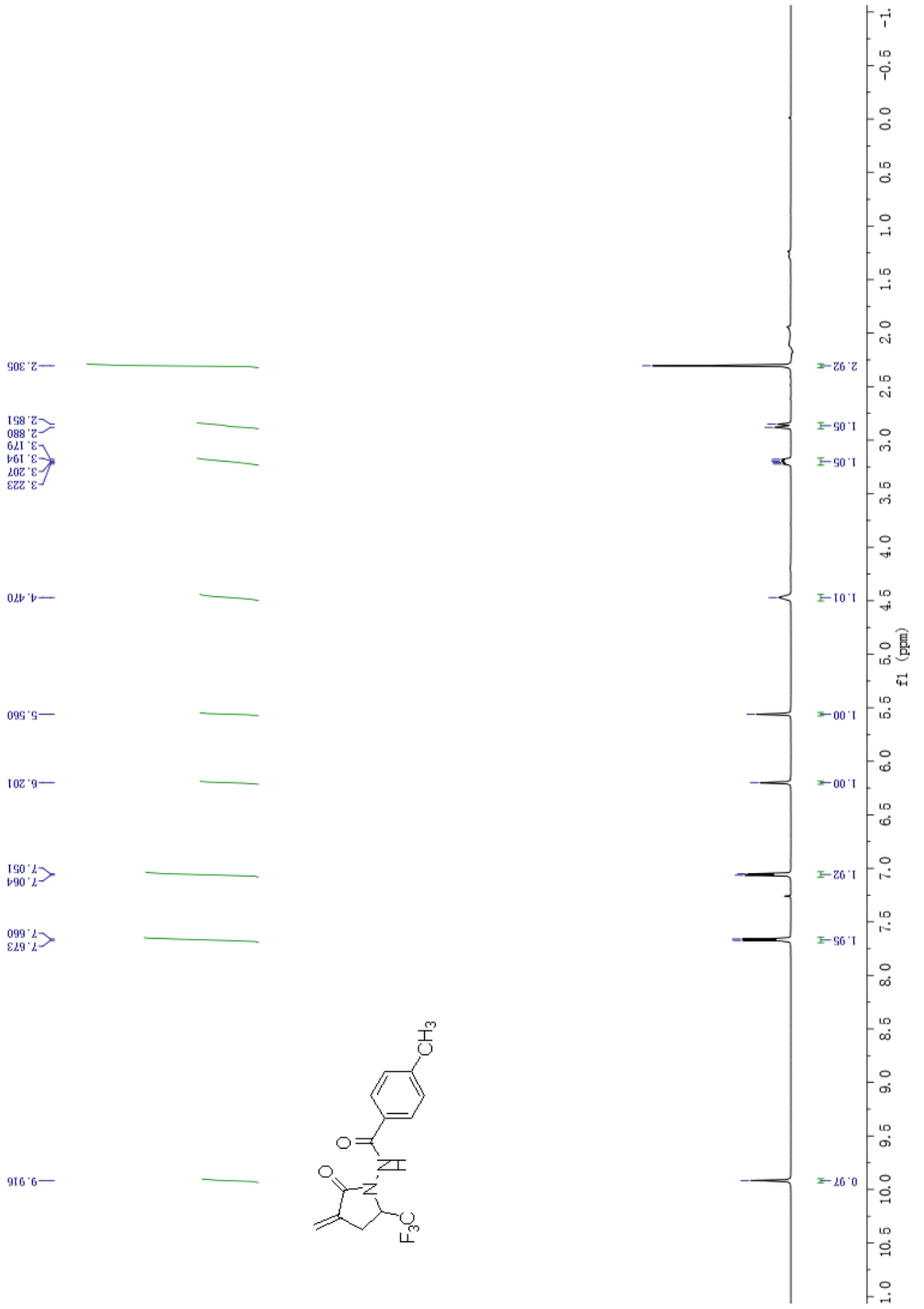


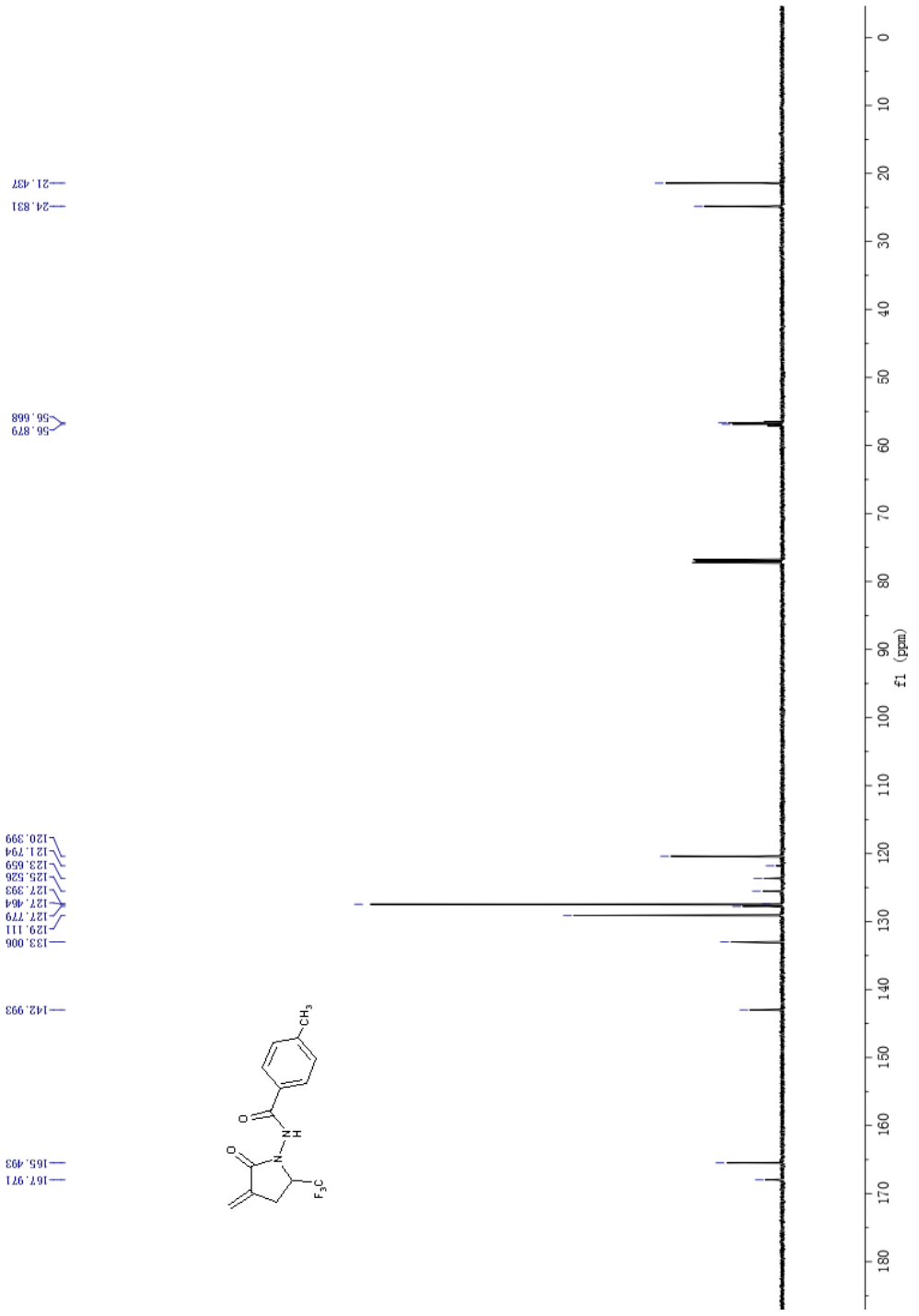

21.136
24.845
56.411
56.622
56.833
57.045
120.436
121.790
123.657
124.489
125.524
127.390
128.124
128.384
130.483
132.975
133.148
138.314
165.644
167.895
f1 (ppm)

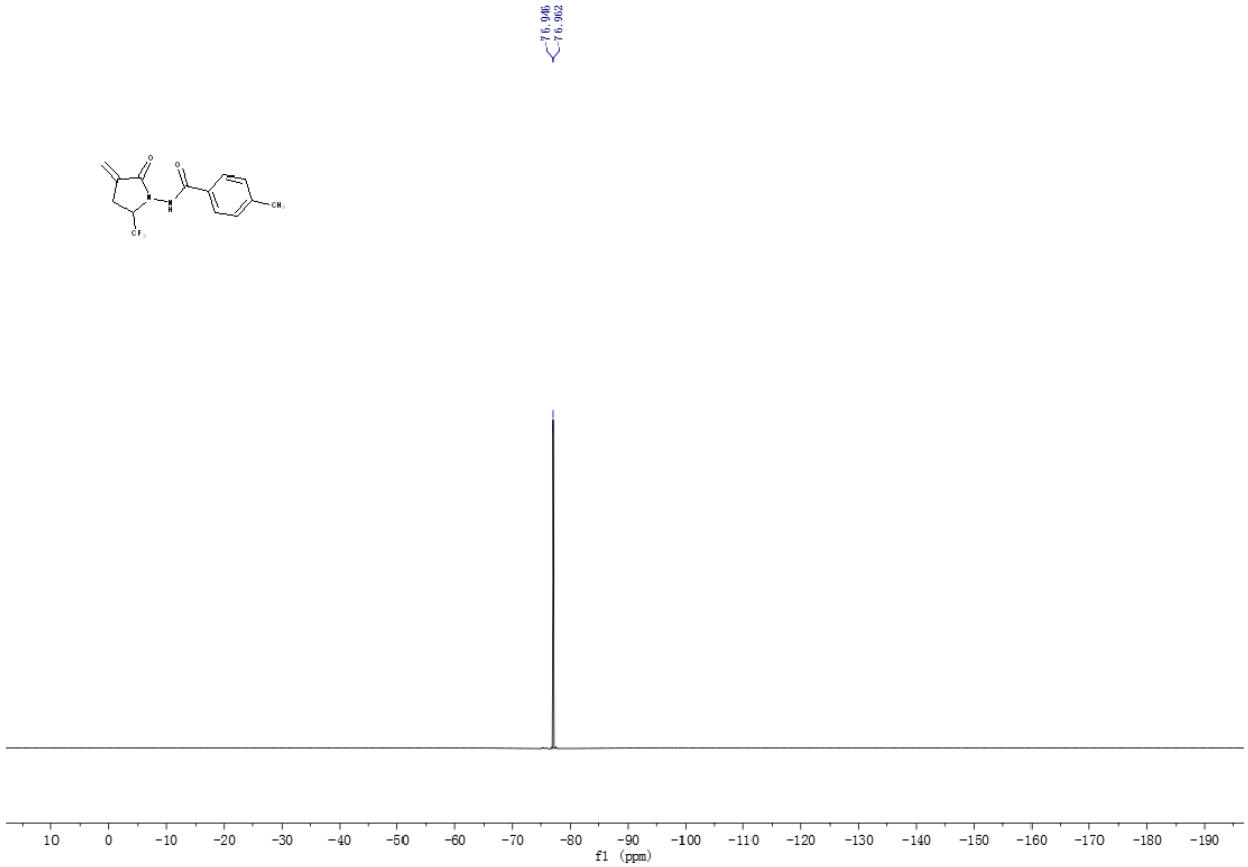

-76.907
-76.924
O
O
N
N
H
CH3
CF3
-10 -15 -20 -25 -30 -35 -40 -45 -50 -55 -60 -65 -70 -75 -80 -85 -90 -95 -100 -110 -120 -130 -140 -150
f1 (ppm)

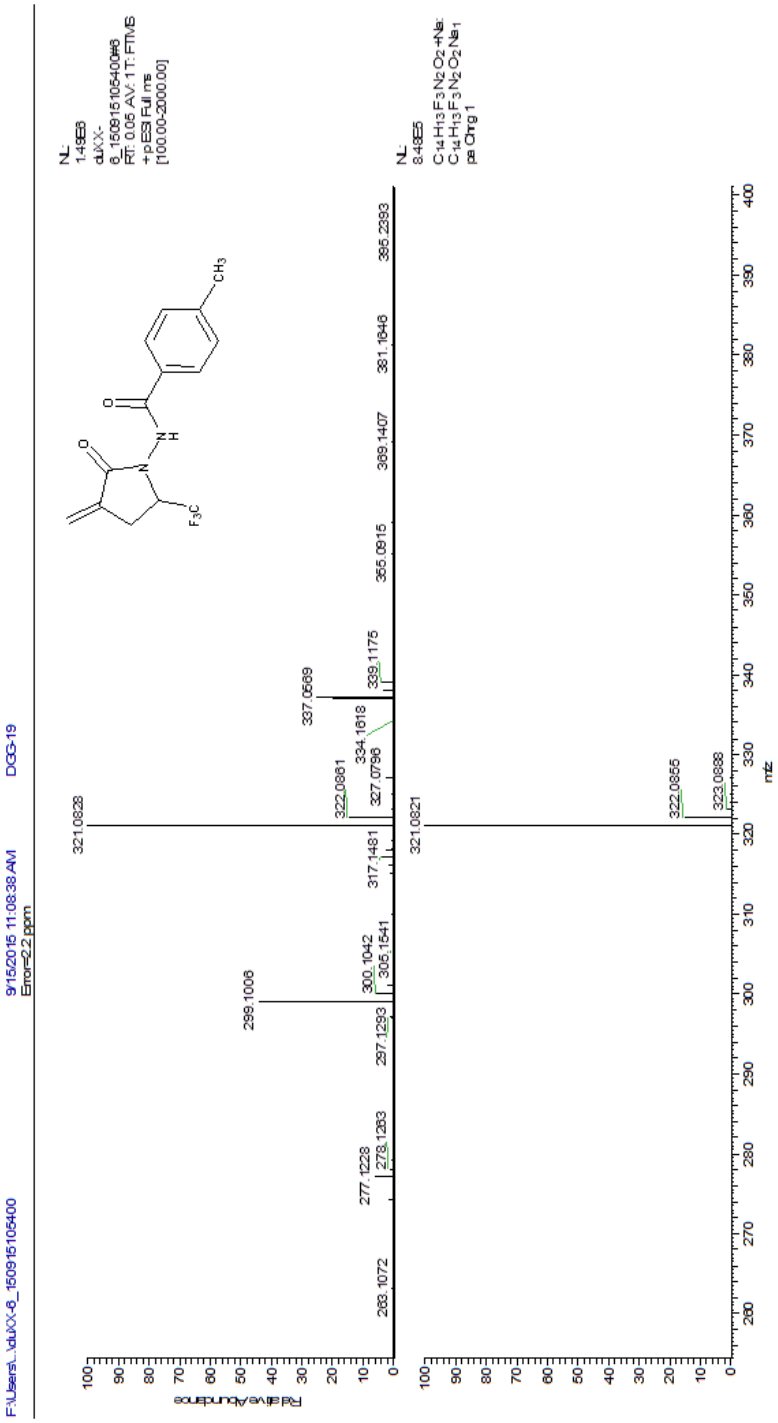

F:\Users\...\duganggang-5_150924095804
9/24/2015 9:59:38 AM
Error=1.6 ppm
DGG-28
321.0826
299.1006
297.0463
275.0643
279.1595
322.0859
300.1040
217.1050
239.1622
261.1312
295.1034
343.0647
360.3241
391.2850
413.2868
443.2159
467.1211
481.3660
Relative Abundance
NL:
3.92E5
duganggang-5_150924095804#1
RT: 0.00 AV: 1 T: FTMS + p ESI Full ms [100.00-2000.00]
321.0821
322.0855
323.0888
NL:
8.48E5
C14 H13 F3 N2 O2 +Na: C14 H13 F3 N2 O2 Na1 pa Chrg 1
m/z

4-methyl-N-(3-methylene-2-oxo-5-(trifluoromethyl)pyrrolidin-1-yl)benzamide(6i)

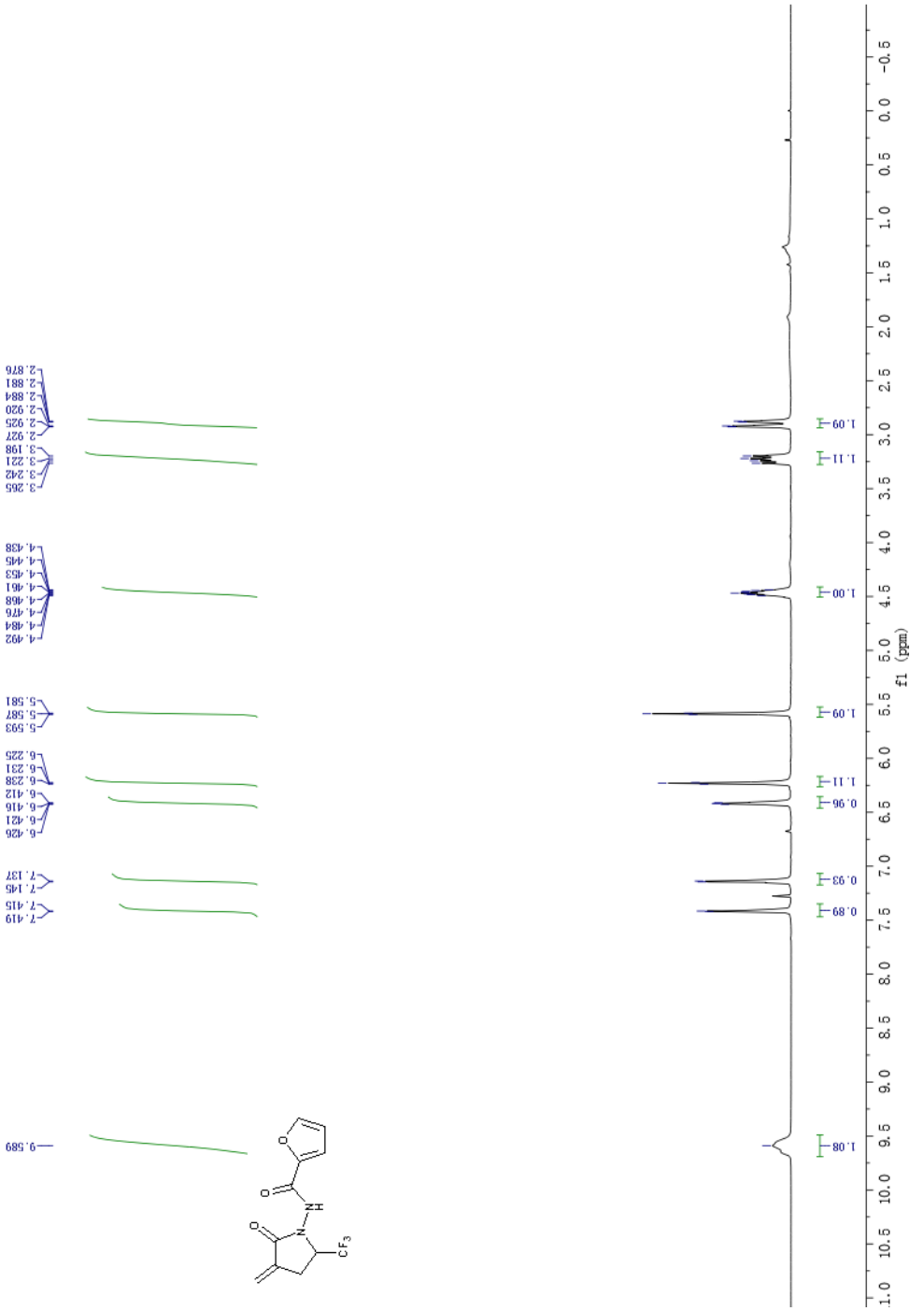


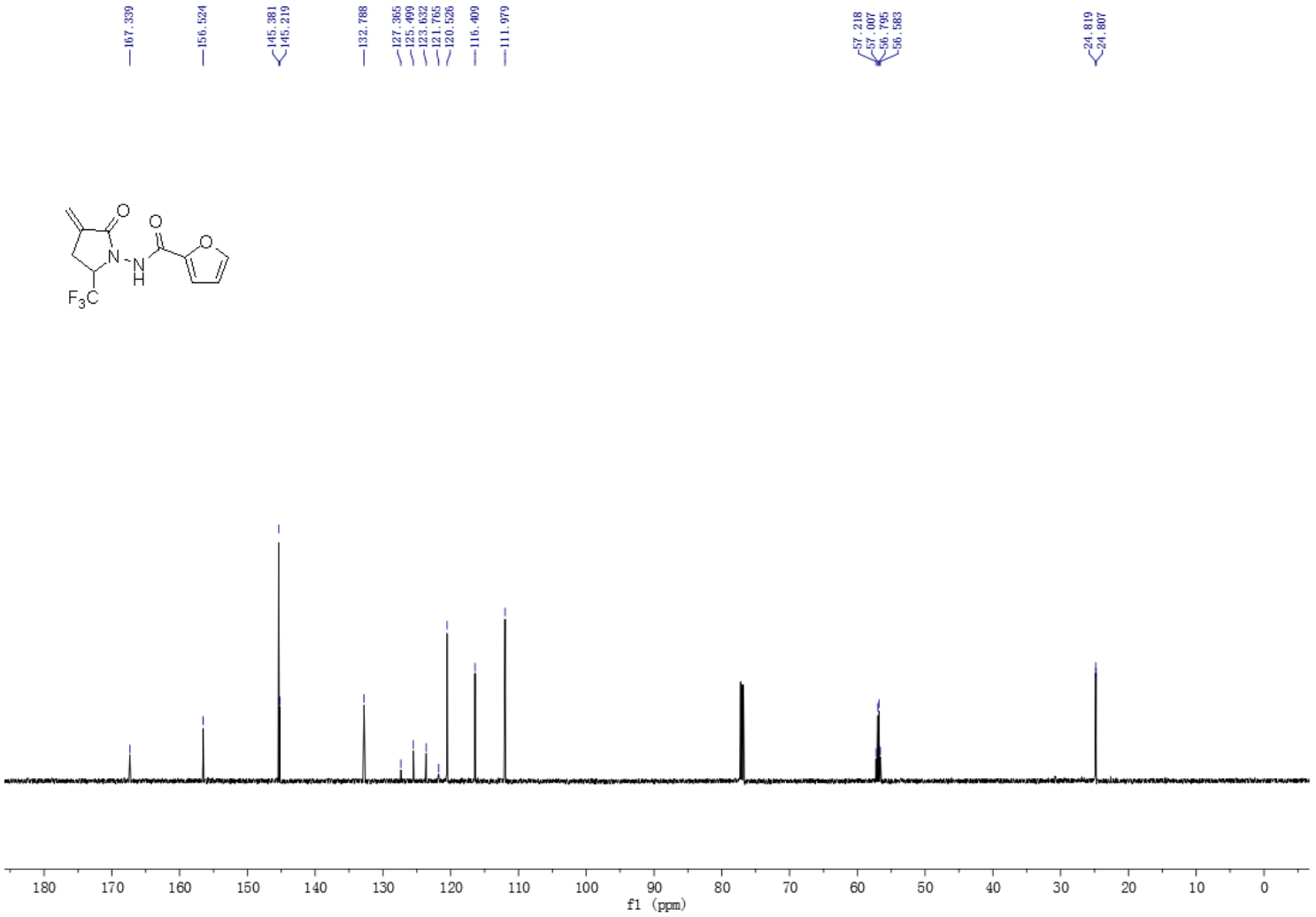

21.437
24.831
56.879
56.668
120.399
121.794
123.659
125.526
127.393
127.464
127.779
129.111
133.006
142.993
165.493
167.971
f1 (ppm)


-76.946
-76.962
10
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190
f1 (ppm)

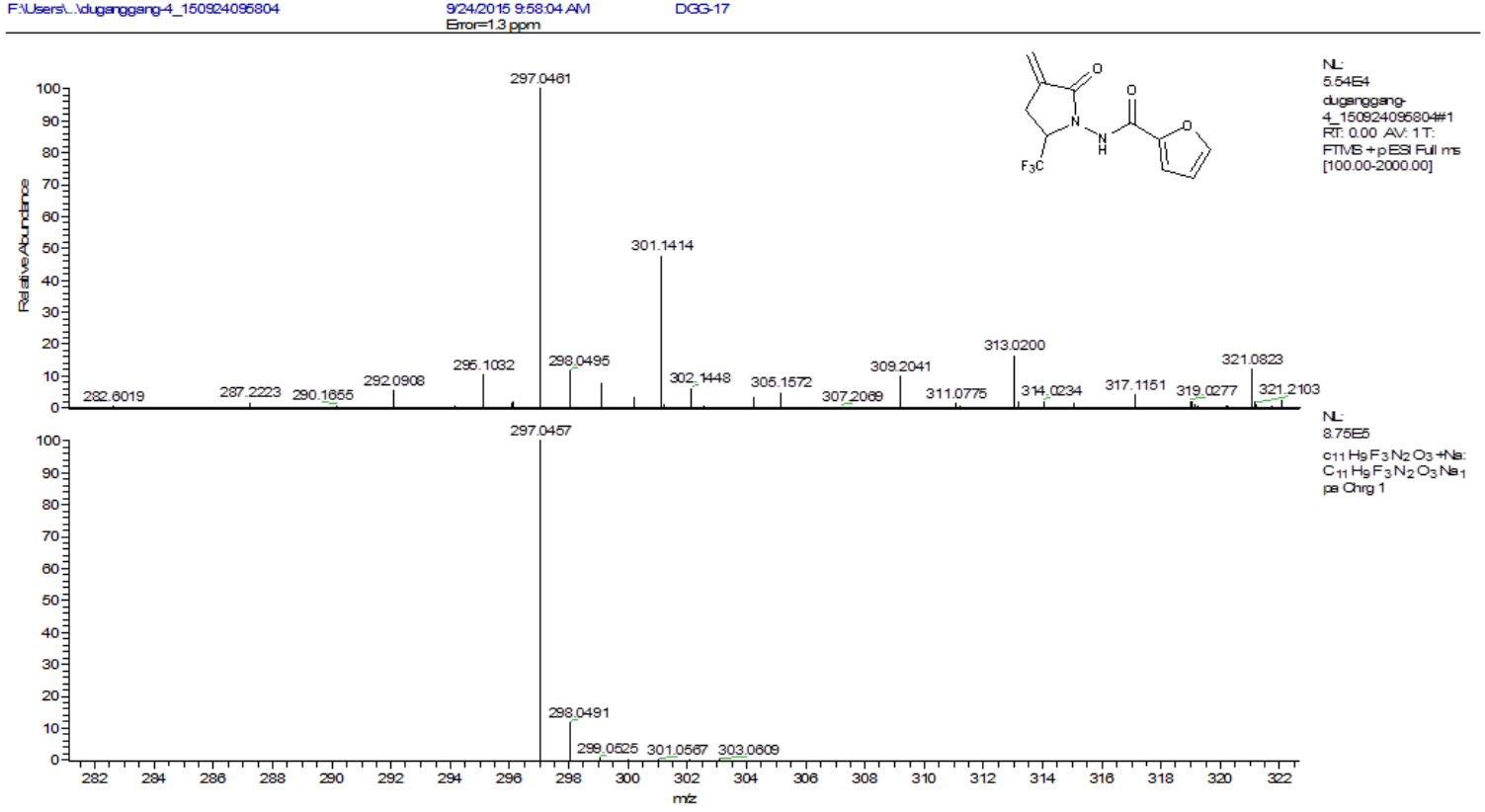

F:\Users\...\dukXX-6_150915105400
9/15/2015 11:08:38 AM
Error=2.2 ppm
DGG-19
NL:
1.49E8
dukXX-
6_150915105400#8
RT: 0.05 AV: 1 T: FTMS
+ pESI Full ms
[100.00-2000.00]
NL:
8.48E5
C14H13F3N2O2+Na
C14H13F3N2O2Na1
pa Chrg 1
Relative Abundance
m/z
321.0828
299.1006
337.0569
322.0881
334.1618
327.0796
339.1175
317.1481
263.1072
277.1228
278.1263
297.1293
300.1042
305.1541
355.0915
369.1407
381.1646
385.2393
321.0821
322.0856
323.0888

N-(3-methylene-2-oxo-5-(trifluoromethyl)pyrrolidin-1-yl)furan-2-carboxamide(6j)

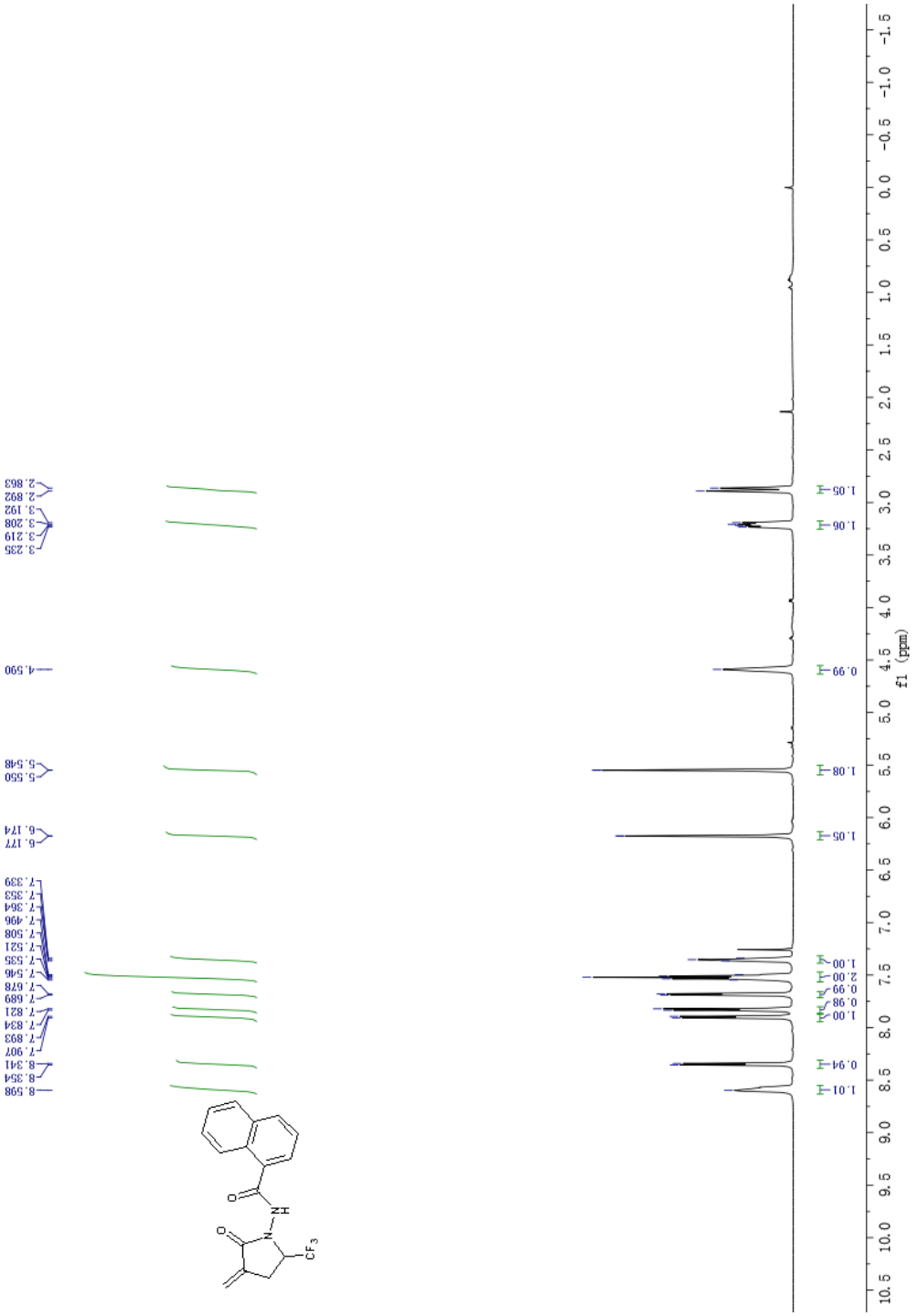


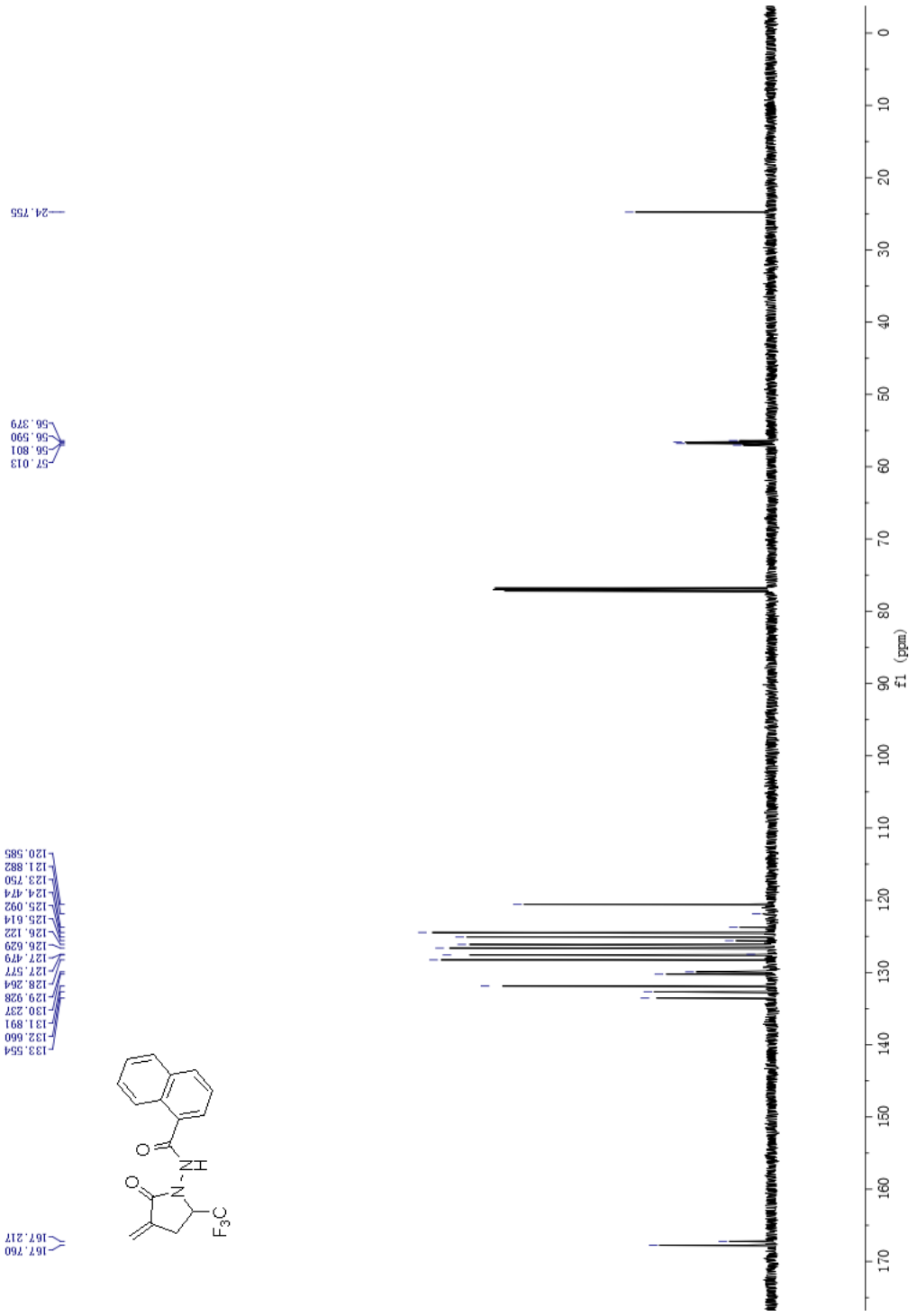

167.339
156.524
145.381
145.219
132.788
127.365
125.499
123.632
121.765
120.526
116.409
111.979
57.218
57.007
56.795
56.583
24.819
24.807
O
O
N
N
H
O
F3C
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

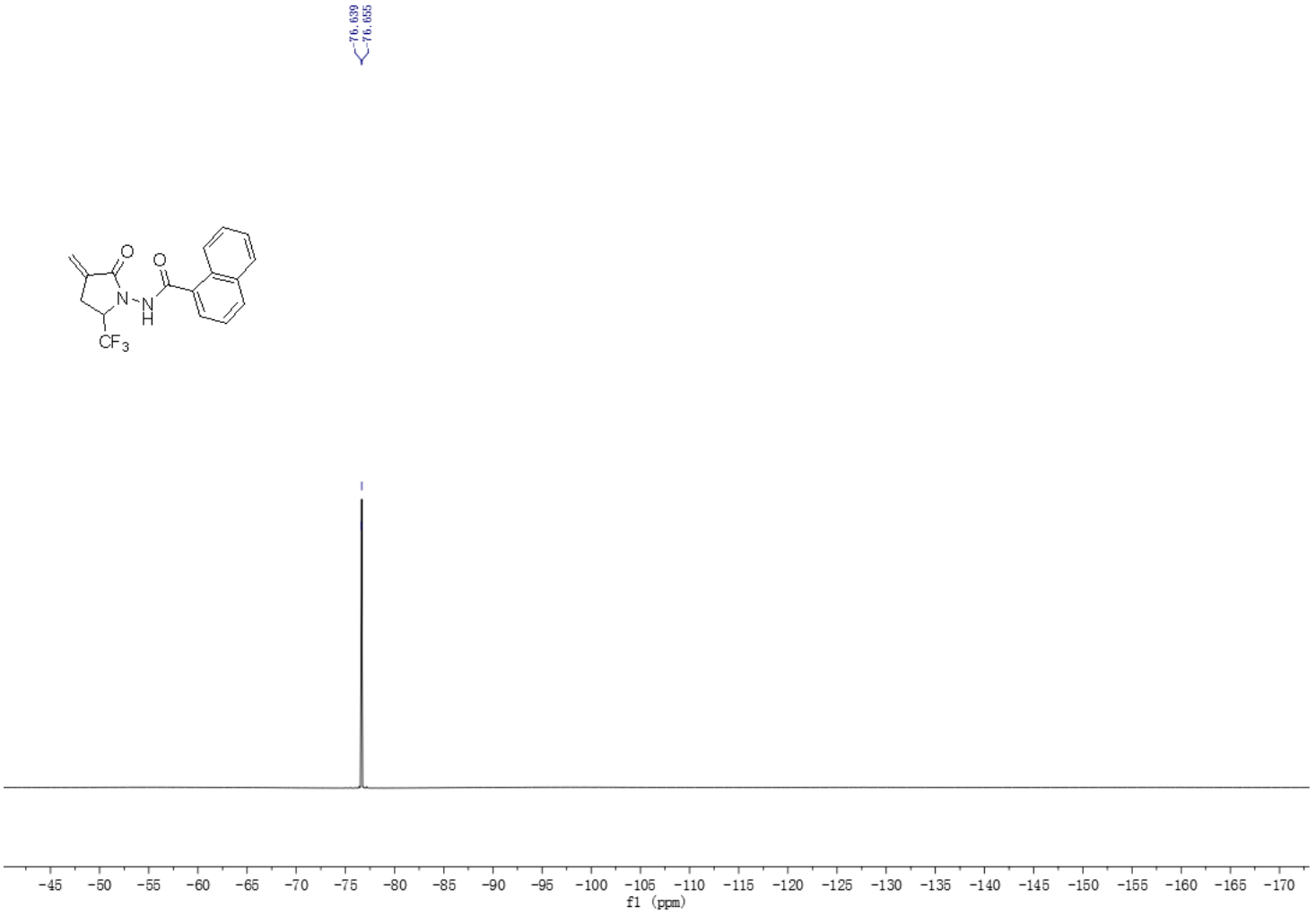
-76.851
-76.868
f1 (ppm)

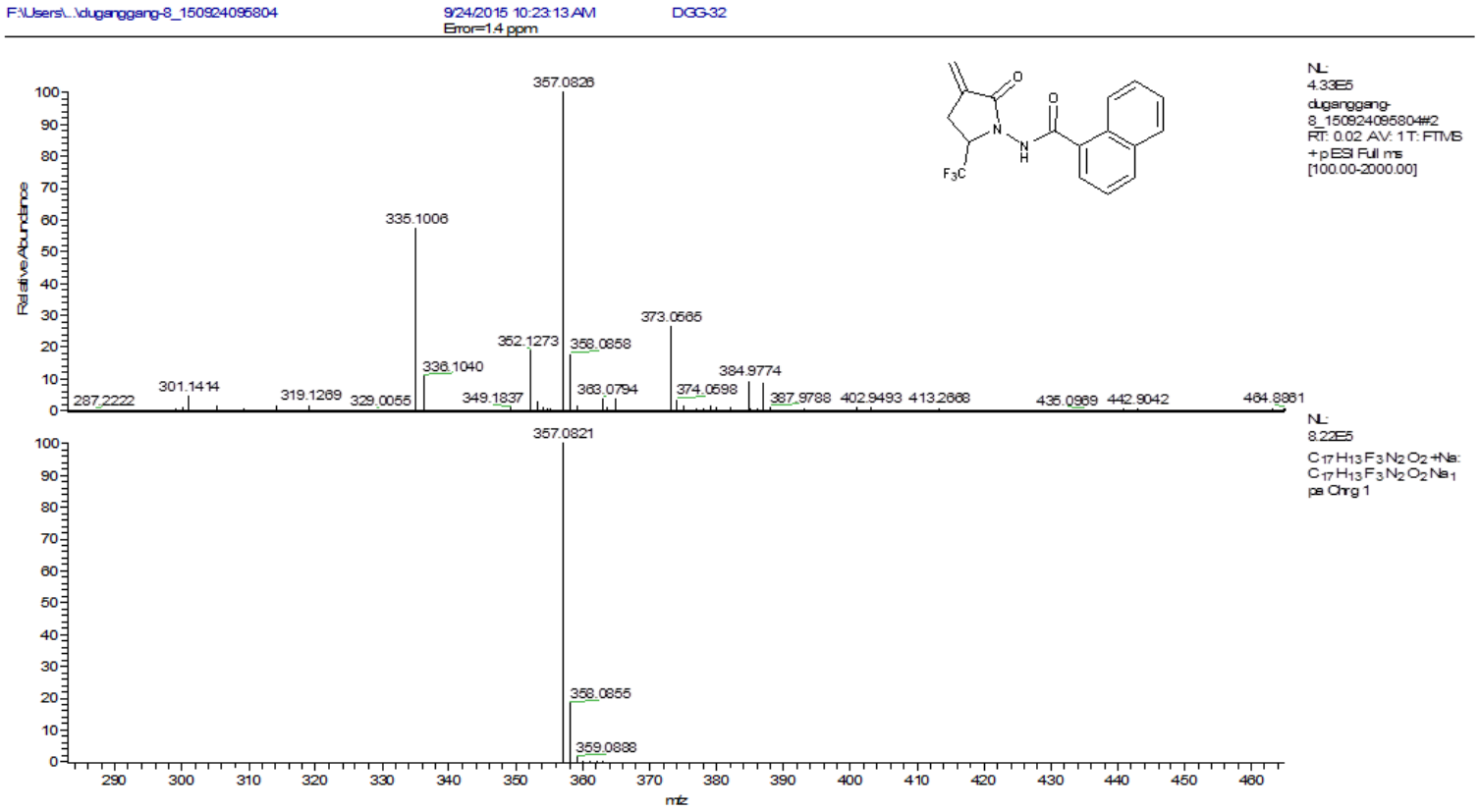

F:\Users\...\duganggang-4_150924095804
9/24/2015 9:58:04 AM
Error=1.3 ppm
DGG-17
NL:
5.54E4
duganggang-4_150924095804#1
RT: 0.00 AV: 1 T:
FTMS + p ESI Full ms
[100.00-2000.00]
297.0461
301.1414
313.0200
321.0823
298.0495
295.1032
309.2041
292.0908
282.6019
287.2223
290.1655
302.1448
305.1572
307.2069
311.0775
314.0234
317.1151
319.0277
321.2103
Relative Abundance
NL:
8.75E5
c11 H9 F3 N2 O3 +Na:
C11 H9 F3 N2 O3 Na1
pa Chrg 1
297.0457
298.0491
299.0525
301.0567
303.0609
m/z

N-(3-methylene-2-oxo-5-(trifluoromethyl)pyrrolidin-1-yl)-2-naphthamide(6k)




167.760
167.217
133.554
132.660
131.891
130.237
129.928
128.264
127.577
127.479
126.629
126.122
125.614
125.092
124.474
123.750
121.882
120.585
57.013
56.801
56.590
56.379
24.755
F3C
O
N
H
f1 (ppm)


-76.639
-76.655
O
O
N
N
H
CF3
-45 -50 -55 -60 -65 -70 -75 -80 -85 -90 -95 -100 -105 -110 -115 -120 -125 -130 -135 -140 -145 -150 -155 -160 -165 -170
f1 (ppm)


F:\Users\..\duganggang-8_150924095804
9/24/2015 10:23:13 AM
Error=1.4 ppm
DGG-32
NL:
4.33E5
duganggang-8_150924095804#2
RT: 0.02 AV: 1 T: FTMS + p ESI Full ms [100.00-2000.00]
Relative Abundance
357.0826
335.1006
373.0565
352.1273
358.0858
336.1040
384.9774
287.2222
301.1414
319.1269
329.0055
349.1837
363.0794
374.0598
387.9788
402.9493
413.2668
435.0989
442.9042
464.8861
NL:
8.22E5
C17H13F3N2O2+Na:
C17H13F3N2O2Na1
pa Chrg 1
357.0821
358.0855
359.0888
m/z