

Supporting Information

Mild Oxidation of Tosylmethylisocyanide to Tosylmethylisocyanate: Utility in Synthetic and Medicinal Chemistry

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General Procedures:

^1H NMR and ^{13}C NMR were taken on a Varian Mercury-300, Varian Inova-400, or Varian Inova-500 spectrometer using CDCl_3 with 0.05% v/v TMS or acetone- d_6 or $\text{DMSO-}d_6$ as solvents, recorded in δ (ppm) and referenced to TMS (0.00 ppm for ^1H NMR and 77.16 ppm for ^{13}C NMR) or acetone- d_6 (2.05 ppm for ^1H NMR and 29.84 ppm for ^{13}C NMR) or $\text{DMSO-}d_6$ (2.50 ppm for ^1H NMR and 39.52 ppm for ^{13}C NMR). IR spectra were obtained using a Thermo Nicolet Avatar 370DTGS FT-IR spectrometer or a Thermo Nicolet iS10 FT-IR spectrometer with a diamond ATR attachment and recorded in wavenumbers (cm^{-1}). Melting points were measured using a Thomas Hoover Uni-melt Capillary Melting Point Apparatus. Mass spectra were measured at Cornell's Life Sciences Core Laboratories Center using an ABI/MDS Sciex 4000 Q Trap instrument. Chemicals were obtained from Aldrich, Acros, Pharmco-AAPER, Macron Chemicals, J.T. Baker, EMD, Matheson Coleman & Bell, or Alfa Aesar, and used as received unless specified.

Preparation of Tosylmethylisocyanate 2

A solution of tosylmethylisocyanide (1.95 g, 10 mmol) in dry CH_2Cl_2 (30 mL) and dry DMSO (710 μL , 10 mmol) under N_2 in a 100 mL RBF was cooled to $-78\text{ }^\circ\text{C}$, then TFAA (840 μL , 6.0 mmol) was added. The resulting solution was stirred vigorously at $-78\text{ }^\circ\text{C}$ for 5 min, then warmed to rt and stirred for 5 min. One drop of the reaction solution was removed via Pasteur

pipet and submitted to IR analysis to confirm that the reaction was complete. Solvent was then removed using a rotary evaporator and the residue was concentrated *in vacuo* (0.2 Torr, 5 min) to afford tosylmethylisocyanate (2.11 g, 100%) as a yellow oil. The crude oil was dissolved in diethyl ether (14 mL), and the resulting clear solution was transferred to a clean 20 mL glass vial. Hexanes (6 mL) was added, and the vial was capped and shaken. The resulting suspension was transferred to another clean 20 mL glass vial, which was then left untouched in a fridge to crystallize (-20 °C, 2 h, see Note). The supernatant was decanted and the newly formed crystals were concentrated *in vacuo* (0.2 Torr, 30 min) to remove traces of solvent, affording tosylmethylisocyanate **2** (1.12 g, 53%) as pale yellow crystals: mp (69-70 °C), ¹H NMR, and IR matched literature values.¹ ¹H NMR (300 MHz, CDCl₃) δ 7.84 (d, *J*=8.2 Hz, 2H), 7.42 (d, *J*=8.2 Hz, 2H), 4.39 (s, 2H), 2.49 (s, 3H). IR (neat) 2999 (m), 2934 (m), 2246 (s), 1595 (m).

Note: Seed crystals of **2** may be obtained by cooling the initial diethyl ether/hexanes suspension to -78 °C, and improve both yield and crystal quality.

General Preparation of Tosylmethylcarbamates 3a-3b

A solution of tosylmethylisocyanate **2** (211 mg, 1.0 mmol) in neat alcohol (2 mL) in a 25 mL RBF was stirred at rt for 16 h. Solvent was then removed using a rotary evaporator and the residue was concentrated *in vacuo* to afford a yellow paste, which was purified by flash column chromatography (1:1 ethyl acetate:hexanes) to afford the desired tosylmethylcarbamates **3a-3b** as white solids.

Tosylmethylcarbamate 3a: 191 mg (71% yield); mp (108-109 °C) and ¹H NMR matched literature values.² ¹H NMR (400 MHz, CDCl₃) δ 7.79 (d, *J*=8.2 Hz, 2H), 7.36 (d, *J*=8.2 Hz, 2H), 5.40 (br, 1H), 4.54 (d, *J*=7.0 Hz, 2H), 3.98 (q, *J*=7.1 Hz, 2H), 2.45 (s, 3H), 1.14 (t, *J*=7.1 Hz, 3H).

Tosylmethylcarbamate 3b: 162 mg, 60% yield; mp 99.5-101 °C; ¹H NMR (400 MHz, CDCl₃) δ 7.79 (d, *J*=8.2 Hz, 2H), 7.35 (d, *J*=8.2 Hz, 2H), 5.53 (br t, *J*=7.0 Hz, 1H), 4.67 (sept, *J*=6.3 Hz, 1H), 4.55 (d, *J*=7.0 Hz, 2H), 2.44 (s, 3H), 1.11 (d, *J*=6.3 Hz, 6H). ¹³C NMR (100 MHz, CDCl₃) δ 154.79, 145.43, 133.88, 129.97, 129.10, 69.70, 62.40, 21.91, 21.82. IR (CH₂Cl₂) 3341(br s), 3062 (m), 2983 (s), 2935 (m), 1723 (s), 1597 (m), 1526 (s). ESI-MS (CH₃OH) 272.1 (M+H), 289.1 (M+NH₄), 294.1 (M+Na), 310.0 (M+K).

General Preparation of Tosylmethylcarbamates 3c-3e

A solution of tosylmethylisocyanate **2** (317 mg, 1.5 mmol) and cyclohexanol or cholesterol or butanethiol (0.5 mmol) in chloroform (0.5 mL) in a 25 mL RBF was stirred at rt for 16 h. Solvent was then removed using a rotary evaporator and the residue was concentrated *in vacuo* to afford a yellow paste, which was purified by flash column chromatography (1:2 ethyl acetate:hexanes) to afford the desired tosylmethylcarbamates **3c-3e**, respectively, as white solids.

Tosylmethylcarbamate 3c (47 mg, 30% yield); mp 98–100 °C; ¹H NMR (400 MHz, CDCl₃) δ 7.79 (d, *J*=8.2 Hz, 2H), 7.34 (d, *J*=8.2 Hz, 2H), 5.61 (br t, *J*=7.0 Hz, 1H), 4.56 (d, *J*=7.0 Hz, 2H), 4.41 (br, 1H), 2.44 (s, 3H), 1.67–1.26 (m, 10H). ¹³C NMR (125 MHz, CDCl₃) δ 154.7, 145.5, 133.9, 130.0, 129.1, 74.5, 62.4, 31.7, 25.4, 23.6, 21.9.

Tosylmethylcarbamate 3d (95 mg, 32% yield); mp 153–155 °C; ¹H NMR (400 MHz, CDCl₃) δ 7.79 (d, *J*=8.2 Hz, 2H), 7.35 (d, *J*=8.2 Hz, 2H), 5.37 (br t, *J*=7.0 Hz, 1H), 5.29 (br m, 1H), 4.53 (m, 2H), 4.26 (m, 1H), 2.45 (s, 3H), 2.19–0.67 (m, 43H). ¹³C NMR (100 MHz, CDCl₃) δ 168.9, 154.5, 145.3, 139.3, 133.7, 129.9, 129.0, 122.8, 75.6, 62.2, 56.6, 56.1, 49.9, 42.3, 39.7, 39.5, 38.2, 36.8, 36.5, 36.1, 35.8, 31.8, 28.2, 28.0, 27.7, 24.2, 23.8, 22.8, 22.5, 21.7, 21.0, 19.3, 18.7, 11.8. IR (CH₂Cl₂) 3421 (m), 3054 (m), 2949 (s), 2868 (m), 1710 (s), 1597 (m), 1509 (m). ESI-MS (CH₃OH) 615.7 (M+NH₄), 620.7 (M+Na), 636.6 (M+K).

Tosylmethylcarbamate 3e (18 mg, 12% yield): ^1H NMR (300 MHz, CDCl_3) δ 7.79 (d, $J=8.2$ Hz, 2H), 7.35 (d, $J=8.2$ Hz, 2H), 6.32 (br t, $J=6.8$ Hz, 1H), 4.67 (d, $J=6.8$ Hz, 2H), 2.74 (t, $J=7.3$ Hz, 2H), 2.45 (s, 3H), 1.47–1.36 (m, 2H), 1.36–1.24 (m, 2H), 0.88 (t, $J=7.3$ Hz, 3H). ^{13}C NMR (75 MHz, CDCl_3) δ 168.1, 145.6, 133.8, 130.1, 129.1, 61.4, 32.3, 29.9, 21.9, 21.8, 13.7. IR (CH_2Cl_2) 3312 (br s), 2958 (s), 2931 (s), 2872 (m), 1735 (m), 1681 (s), 1597 (m), 1518 (s). ESI-MS (CH_3OH) 302.1 (M+H), 319.1 (M+ NH_4), 324.0 (M+Na), 340.1 (M+K).

General Preparation of Tosylmethylureas 4a-4e

A solution of amine (0.25 mmol) in dry THF (2 mL) in a 50 mL RBF was cooled to -78 $^\circ\text{C}$, then a solution of tosylmethylisocyanate **2** (53 mg, 0.25 mmol) in dry THF (0.5 mL) was added over a few minutes. The resulting solution was stirred vigorously at -78 $^\circ\text{C}$ for 30 min, then warmed to rt and stirred for 1 h. Solvent was then removed using a rotary evaporator, and the residue was concentrated *in vacuo* to afford a yellow solid, which was purified by trituration or flash column chromatography to afford the desired tosylmethylureas **4a-4e** as white solids.

Tosylmethylurea 4a (43 mg, 61% yield); mp 100–102 $^\circ\text{C}$; ^1H NMR (300 MHz, CDCl_3) δ 7.79 (d, $J=8.2$ Hz, 2H), 7.34 (d, $J=8.2$ Hz, 2H), 6.21 (br t, $J=6.7$ Hz, 1H), 5.37 (br t, $J=6.0$ Hz, 1H), 4.68 (d, $J=6.7$ Hz, 2H), 3.03 (dd, $J_1=6.0$ Hz, $J_2=6.7$ Hz, 2H), 2.44 (s, 3H), 1.28 (m, 4H), 0.86 (t, $J=7.1$ Hz, 3H). ^{13}C NMR (75 MHz, CDCl_3) δ 156.5, 145.4, 134.0, 130.0, 128.9, 62.7, 40.2, 32.2, 21.9, 20.0, 13.9. IR (CH_2Cl_2) 3359 (br s), 2958 (s), 2931 (s), 2872 (m), 1650 (s), 1565 (s). ESI-MS (CH_3OH) 285.3 (M+H), 302.3 (M+ NH_4), 307.2 (M+Na), 323.2 (M+K). [Purified by flash column chromatography (3:1 ethyl acetate:hexanes)]

Tosylmethylurea 4b (39 mg, 55% yield); mp 136–138 $^\circ\text{C}$; ^1H NMR (300 MHz, CDCl_3) δ 7.80 (d, $J=8.2$ Hz, 2H), 7.35 (d, $J=8.2$ Hz, 2H), 6.09 (br t, $J=6.7$ Hz, 1H), 5.20 (s, 1H), 4.65 (d, $J=6.7$ Hz, 2H), 2.44 (s, 3H), 1.14 (s, 9H). ^{13}C NMR (75 MHz, CDCl_3) δ 155.0, 145.4, 133.9,

130.0, 129.0, 62.4, 50.6, 29.1, 21.8. IR (CH₂Cl₂) 3377 (br s), 2968 (s), 2928 (m), 1658 (s), 1555 (s). ESI-MS (CH₃OH) 285.4 (M+H), 302.3 (M+NH₄), 307.2 (M+Na), 323.3 (M+K). [Purified by flash column chromatography (2:1 ethyl acetate:hexanes)]

Tosylmethylurea 4c (15 mg, 21% yield); mp 169–170 °C; ¹H NMR (300 MHz, CDCl₃) δ 7.78 (d, *J*=8.2 Hz, 2H), 7.33 (d, *J*=8.2 Hz, 2H), 5.24 (br t, *J*=6.7 Hz, 1H), 4.71 (d, *J*=6.7 Hz, 2H), 3.26 (m, 4H), 2.44 (s, 3H), 1.88 (m, 4H). ¹³C NMR (75 MHz, CDCl₃) δ 154.2, 145.2, 134.4, 129.9, 128.9, 62.4, 45.8, 25.6, 21.9. IR (CH₂Cl₂) 3279 (br s), 2974 (m), 2875 (m), 1649 (s), 1531 (s). ESI-MS (CH₃OH) 283.2 (M+H), 305.2 (M+Na), 321.2 (M+K). [Purified by flash column chromatography (ethyl acetate)]

Tosylmethylurea 4d (55 mg, 72% yield); mp 155–156.5 °C; ¹H NMR (300 MHz, acetone-*d*₆) δ 8.18 (br s, 1H), 7.79 (d, *J*=8.4 Hz, 2H), 7.40 (d, *J*=8.2 Hz, 2H), 7.33 (d, *J*=8.4 Hz, 2H), 7.21 (m, 2H), 6.94 (m, 1H), 6.66 (br t, *J*=6.7 Hz, 1H), 4.71 (d, *J*=6.7 Hz, 2H), 2.40 (s, 3H). ¹³C NMR (75 MHz, acetone-*d*₆) δ 154.3, 145.6, 140.6, 136.3, 130.5, 129.7, 129.5, 123.0, 119.3, 62.3, 21.5. IR (neat) 3344 (m), 3057 (w), 2979 (w), 2922 (w), 1655 (s), 1601 (m), 1556 (s), 1500 (m). ESI-MS (CH₃OH) 305.2 (M+H), 322.2 (M+NH₄), 327.2 (M+Na), 343.1 (M+K). [Purified by trituration with chloroform (3 x 1 mL)]

Tosylmethylurea 4e (43 mg, 54% yield); mp 135–137 °C. ¹H NMR (300 MHz, DMSO-*d*₆) δ 7.80 (s, 1H), 7.75 (d, *J*=8.2 Hz, 2H), 7.43 (d, *J*=8.2 Hz, 2H), 7.11 (m, 1H), 6.99 (d, *J*=7.8 Hz, 1H), 6.81 (m, 1H), 6.67 (d, *J*=6.8 Hz, 1H), 6.49 (m, 1H), 4.69 (d, *J*=6.8 Hz, 2H), 2.41 (s, 3H). ¹³C NMR (75 MHz, DMSO-*d*₆) δ 154.2, 144.5, 140.9, 134.8, 129.7, 128.5, 124.7, 124.2, 124.0, 116.7, 115.8, 61.6, 21.1. IR (neat) 3296 (m), 3063 (w), 2979 (w), 2920 (w), 1644 (m), 1625 (m), 1591 (m), 1562 (s). ESI-MS (CH₃OH) 319.8 (M+H), 342.1 (M+Na). [Purified by trituration with chloroform (3 x 1 mL)]

General Preparation of Tosylmethyamides 5a-5e

A solution of tosylmethylisocyanate **2** (53 mg, 0.25 mmol) in dry DMF (0.5 mL) in a 50 mL RBF was cooled to -40 °C, then a pre-mixed solution of carboxylic acid (0.375 mmol) and N,N-diisopropylethylamine (52 μ L, 0.3 mmol) in dry DMF (2 mL) was added along the flask wall over a few minutes. The resulting solution was stirred vigorously at -40 °C for 30 min, then warmed to rt and stirred for 2 h. Water (5 mL) was added and the aqueous phase was extracted with ethyl acetate (4 x 10 mL). The combined organic extracts were washed with water (2 x 10 mL) and brine (2 mL), dried (MgSO₄), filtered and concentrated *in vacuo* to afford a yellow oil, which was purified by flash column chromatography (7:3 chloroform:ethyl acetate) to afford the desired tosylmethyamides **5a-5e** as white solids.

Tosylmethyamide 5a (40 mg, 56% yield); mp 155–156 °C. ¹H NMR (300 MHz, CDCl₃) δ 7.78 (d, *J*=8.2 Hz, 2H), 7.68 (d, *J*=7.2 Hz, 2H), 7.51 (m, 1H), 7.40 (m, 2H), 7.33 (br t, *J*=6.7 Hz, 1H), 7.29 (d, *J*=8.2 Hz, 2H), 4.91 (d, *J*=6.7 Hz, 2H), 2.40 (s, 3H). ¹³C NMR (75 MHz, CDCl₃) δ 166.6, 145.6, 134.0, 132.8, 132.5, 130.1, 128.9, 128.8, 127.2, 61.0, 21.9. IR (neat) 3299 (m), 3061 (w), 2949 (w), 1657 (s), 1540 (s). ESI-MS (CH₃OH) 290.2 (M+H), 307.2 (M+NH₄), 312.2 (M+Na), 328.2 (M+K). ¹H NMR and IR matched literature values.³

Tosylmethyamide 5b (32 mg, 40% yield): mp 154.5–156 °C. ¹H NMR (300 MHz, CDCl₃) δ 7.78 (d, *J*=8.2 Hz, 2H), 7.66 (d, *J*=8.6 Hz, 2H), 7.28 (d, *J*=8.2 Hz, 2H), 7.20 (br t, *J*=6.7 Hz, 1H), 6.88 (d, *J*=8.6 Hz, 2H), 4.90 (d, *J*=6.7 Hz, 2H), 3.83 (s, 3H), 2.40 (s, 3H). ¹³C NMR (75 MHz, CDCl₃) δ 166.0, 162.9, 145.5, 134.1, 130.0, 129.2, 128.9, 125.0, 114.0, 61.1, 55.6, 21.9. IR (neat) 3358 (m), 2986 (w), 2924 (w), 1659 (s), 1608 (m), 1533 (m), 1499 (s). ESI-MS (CH₃OH) 320.3 (M+H), 337.4 (M+NH₄), 342.3 (M+Na), 358.2 (M+K).

Tosylmethylamide 5c (18 mg, 23% yield): mp 110–111 °C. ¹H NMR (300 MHz, CDCl₃) δ 7.83 (m, 1H), 7.80 (d, *J*=8.2 Hz, 2H), 7.51 (m, 1H), 7.38 (br t, *J*=6.7 Hz, 1H), 7.31 (d, *J*=8.2 Hz, 2H), 7.24 (m, 1H), 7.15 (m, 1H), 4.90 (dd, *J*₁=6.7 Hz, *J*₂=1.5 Hz, 2H), 2.42 (s, 3H). ¹³C NMR (75 MHz, CDCl₃) δ 162.3 (d, *J*=36.6 Hz), 159.0, 145.6, 134.3 (d, *J*=9.6 Hz), 133.9, 132.1 (d, *J*=2.3 Hz), 130.1, 129.0, 125.1 (d, *J*=3.2 Hz), 119.6 (d, *J*=11.5 Hz), 116.2 (d, *J*=24.3 Hz), 60.7, 21.9. IR (neat) 3302 (m), 2995 (m), 2928 (w), 1657 (s), 1612 (m), 1522 (s). ESI-MS (CH₃OH) 308.4 (M+H), 325.4 (M+NH₄), 330.4 (M+Na), 346.4 (M+K).

Tosylmethylamide 5d (45 mg, 53% yield): ¹H NMR (300 MHz, CDCl₃) δ 8.23 (s, 1H), 7.85 (m, 3H), 7.81 (d, *J*=8.2 Hz, 2H), 7.71 (m, 1H), 7.55 (m, 2H), 7.48 (br t, *J*=6.7 Hz, 1H), 7.26 (d, *J*=8.2 Hz, 2H), 4.98 (d, *J*=6.7 Hz, 2H), 2.38 (s, 3H). ¹³C NMR (75 MHz, CDCl₃) δ 166.7, 145.6, 135.2, 134.0, 132.5, 130.1, 129.9, 129.2, 128.9, 128.8, 128.3, 128.1, 127.8, 127.1, 123.5, 61.1, 21.9. IR (neat) 3330 (m), 3057 (w), 2927 (w), 1732 (m), 1655 (s), 1596 (m), 1524 (s), 1503 (s). ESI-MS (CH₃OH) 340.4 (M+H), 357.5 (M+NH₄), 362.4 (M+Na), 378.4 (M+K).

Tosylmethylamide 5e (29 mg, 37% yield): mp 64–65 °C. ¹H NMR (300 MHz, CDCl₃) δ 7.82 (d, *J*=8.2 Hz, 2H), 7.51 (d, *J*=15.7 Hz, 1H), 7.43 (m, 2H), 7.34 (m, 3H), 7.31 (d, *J*=8.2 Hz, 2H), 7.11 (br t, *J*=6.7 Hz, 1H), 6.44 (d, *J*=15.7 Hz, 1H), 4.87 (d, *J*=6.7 Hz, 2H), 2.40 (s, 3H). ¹³C NMR (75 MHz, CDCl₃) δ 165.3, 145.6, 143.1, 134.3, 134.1, 130.3, 130.1, 129.0, 128.8, 128.1, 118.9, 60.9, 21.9. IR (CH₂Cl₂) 3351 (m), 3054 (m), 2991 (m), 2934 (w), 1676 (s), 1634 (s), 1597 (m), 1513 (s). ESI-MS (CH₃OH) 316.3 (M+H), 333.3 (M+NH₄), 338.3 (M+Na), 354.2 (M+K).

Preparation of N-(n-butoxymethyl)benzamide 6

CuI (59 mg, 0.31 mmol) in an oven-dried 50 mL RBF was heated *in vacuo* with a heat gun for 10 min then let cool to rt under N₂. Dry THF (1.7 mL) was added and the resulting suspension was cooled to 0 °C. *n*-Butyllithium (350 μL, 0.56 mmol) was added by syringe along

the flask wall over a few minutes, and the resulting mixture was stirred at 0 °C for 2 min, then cooled to -78 °C. A solution of tosylmethylisocyanate **2** (40 mg, 0.14 mmol) in dry THF (0.2 mL) was added along the flask wall over a few minutes, and the resulting mixture was stirred at -78 °C for 1 h, then warmed to 0 °C. Saturated NH₄Cl solution (5 mL) was added, and the resulting mixture was swirled for a few minutes. Ammonium hydroxide solution (28% NH₃) was added dropwise until the mixture became a clear solution, which was then extracted with diethyl ether (4 x 15 mL). The combined organic extracts were washed with brine (3 mL), dried (MgSO₄), filtered and concentrated *in vacuo* to afford a white paste, which was purified by flash column chromatography (17:3 chloroform:ethyl acetate) to afford the desired N-(*n*-butoxymethyl)benzamide **6** as a clear oil (21 mg, 72% yield): ¹H NMR (300 MHz, CDCl₃) δ 7.81 (d, *J*=7.0 Hz, 2H), 7.48 (m, 3H), 6.84 (br t, *J*=6.7 Hz, 1H), 4.95 (d, *J*=6.7 Hz, 2H), 3.57 (t, *J*=6.6 Hz, 2H), 1.57 (m, 2H), 1.36 (m, 2H), 0.91 (t, *J*=7.3 Hz, 3H). ¹³C NMR (75 MHz, CDCl₃) δ 167.9, 134.1, 132.0, 128.8, 127.2, 70.7, 68.7, 31.9, 19.4, 14.0. IR (neat) 3333 (br s), 2958 (s), 2932 (s), 2871 (m), 1652 (s), 1533 (s). ESI-MS (CH₃OH) 208.4 (M+H), 225.4 (M+NH₄), 230.4 (M+Na), 246.4 (M+K).

References

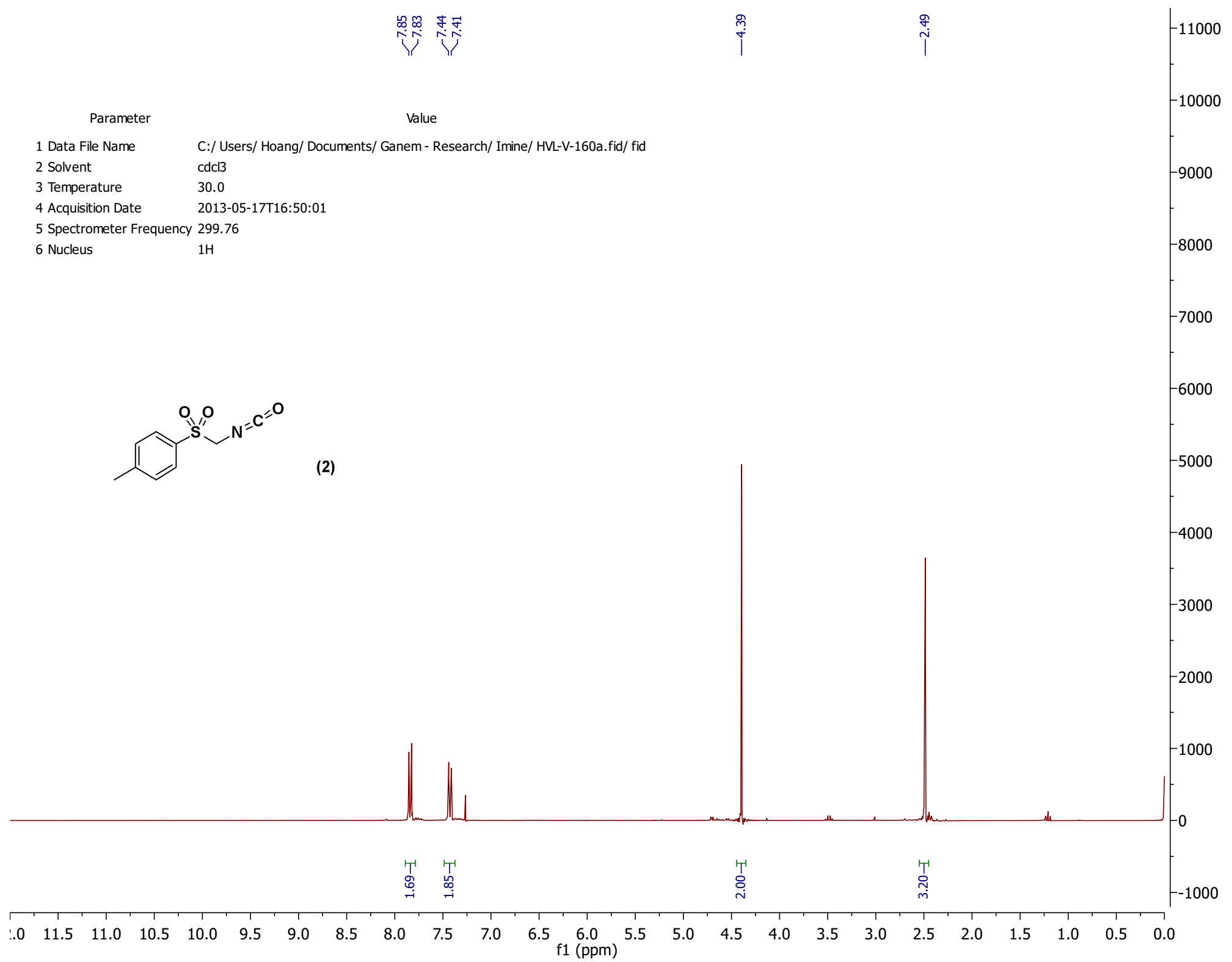
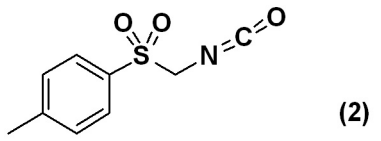
1. Olijnsma, T.; Engberts, J. B. F. N.; Strating, J. *Rec. Trav. Chim. Pays-Bas* **1970**, *89*, 897–906.
2. van Leusen, A. M.; Strating, J. *Org. Synth.* **1977**, *57*, 95–101.
3. Olijnsma, T.; Engberts, J. B. F. N.; Strating, J. *Rec. Trav. Chim. Pays-Bas* **1967**, *86*, 463–473.

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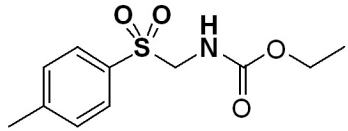
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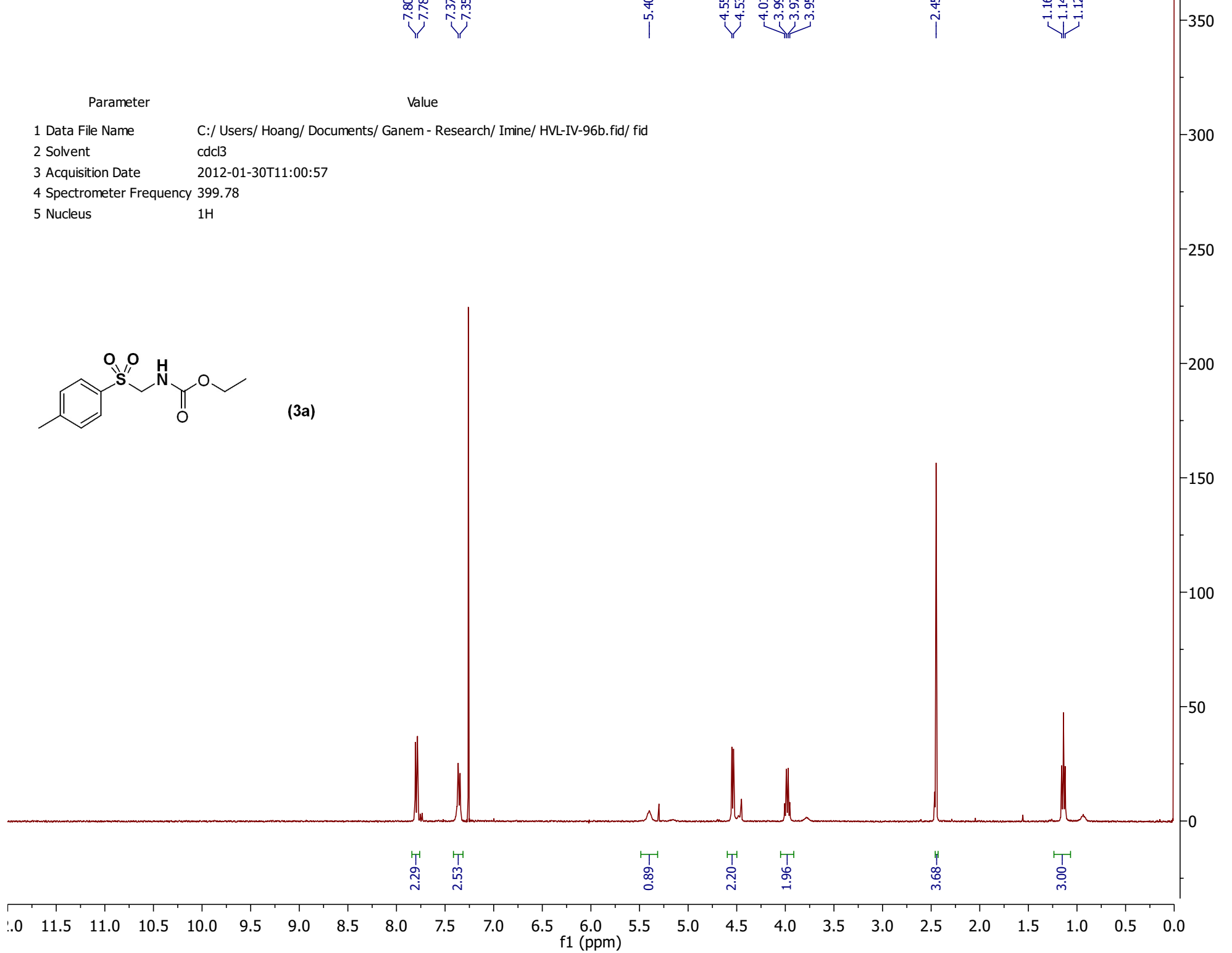


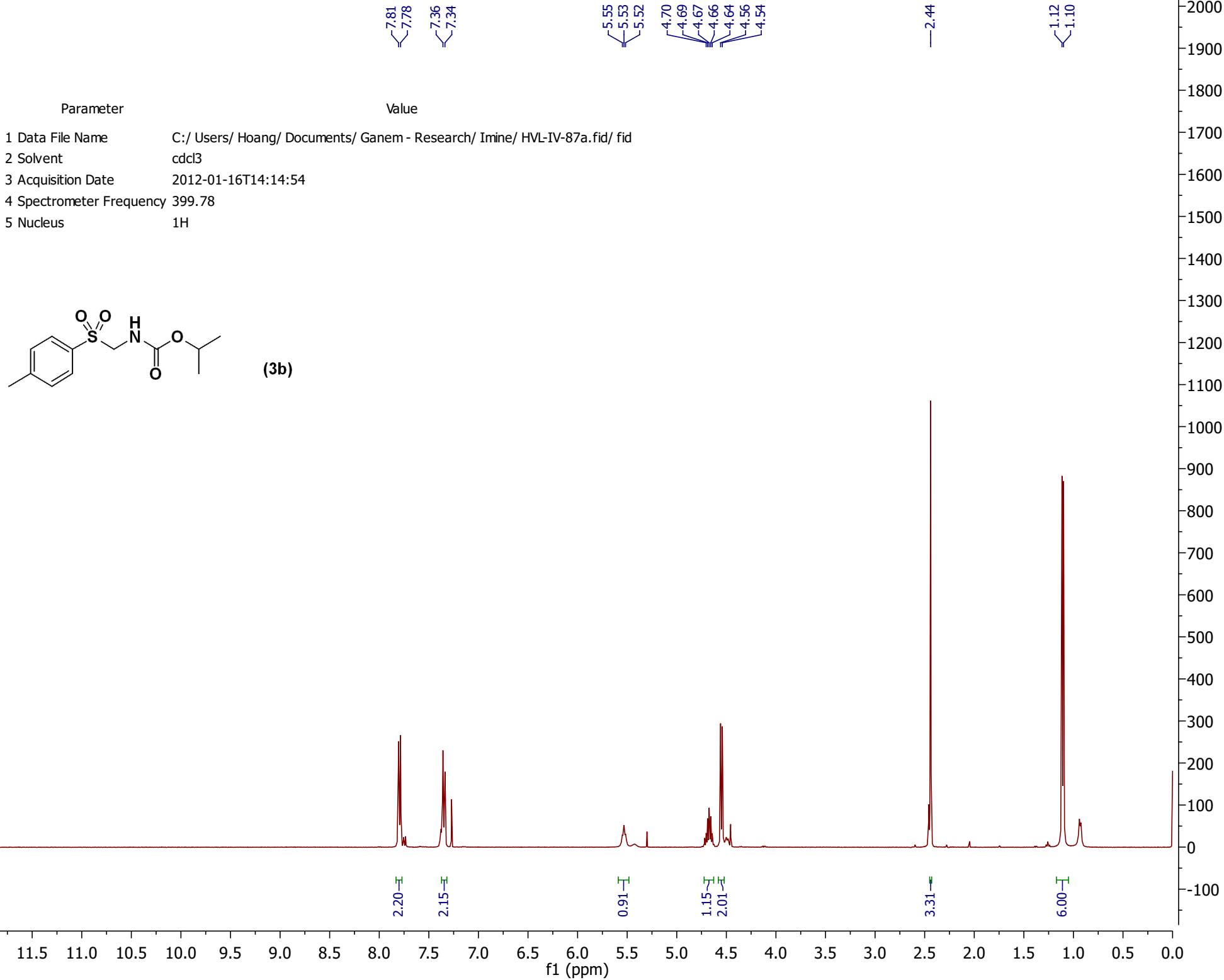
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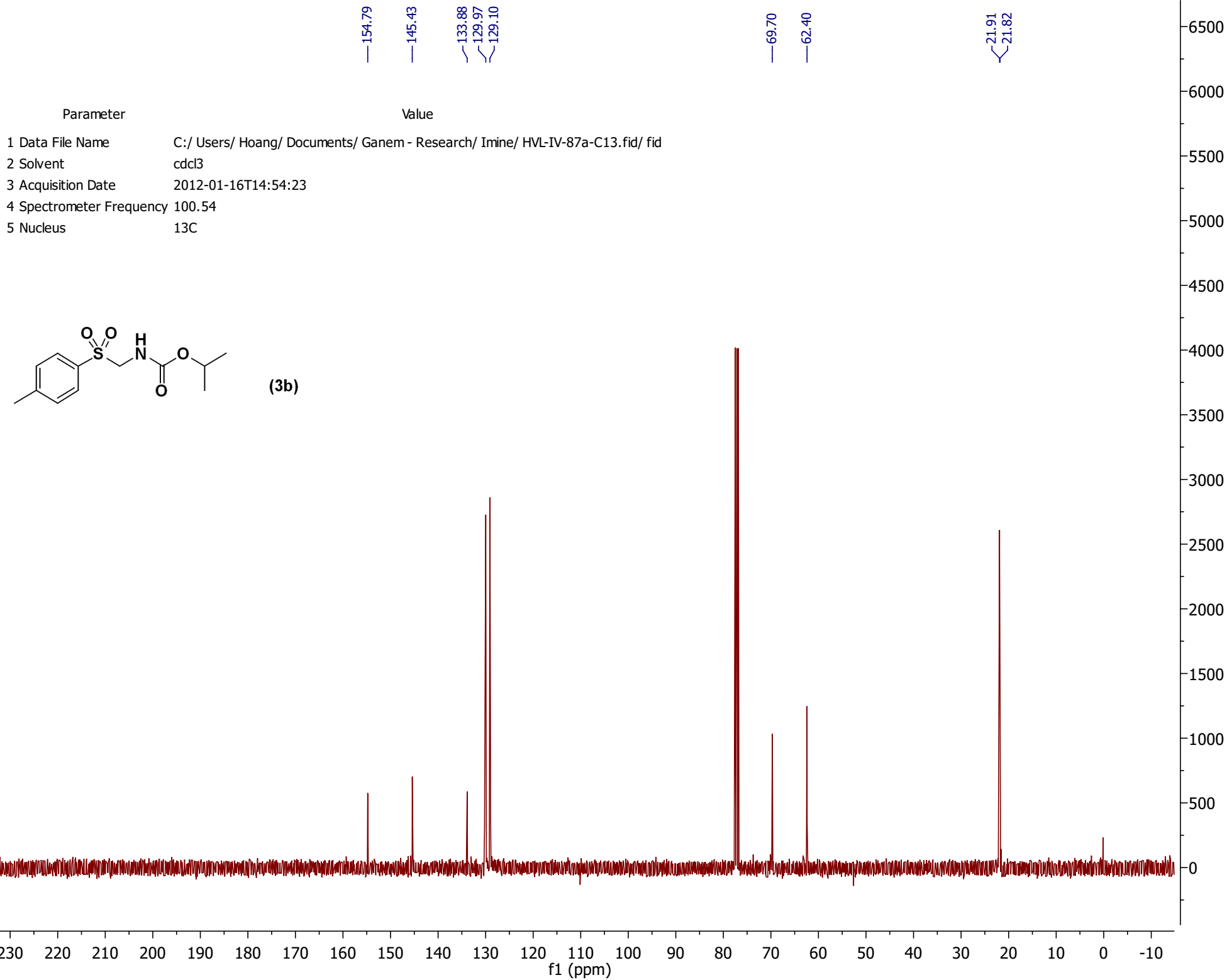
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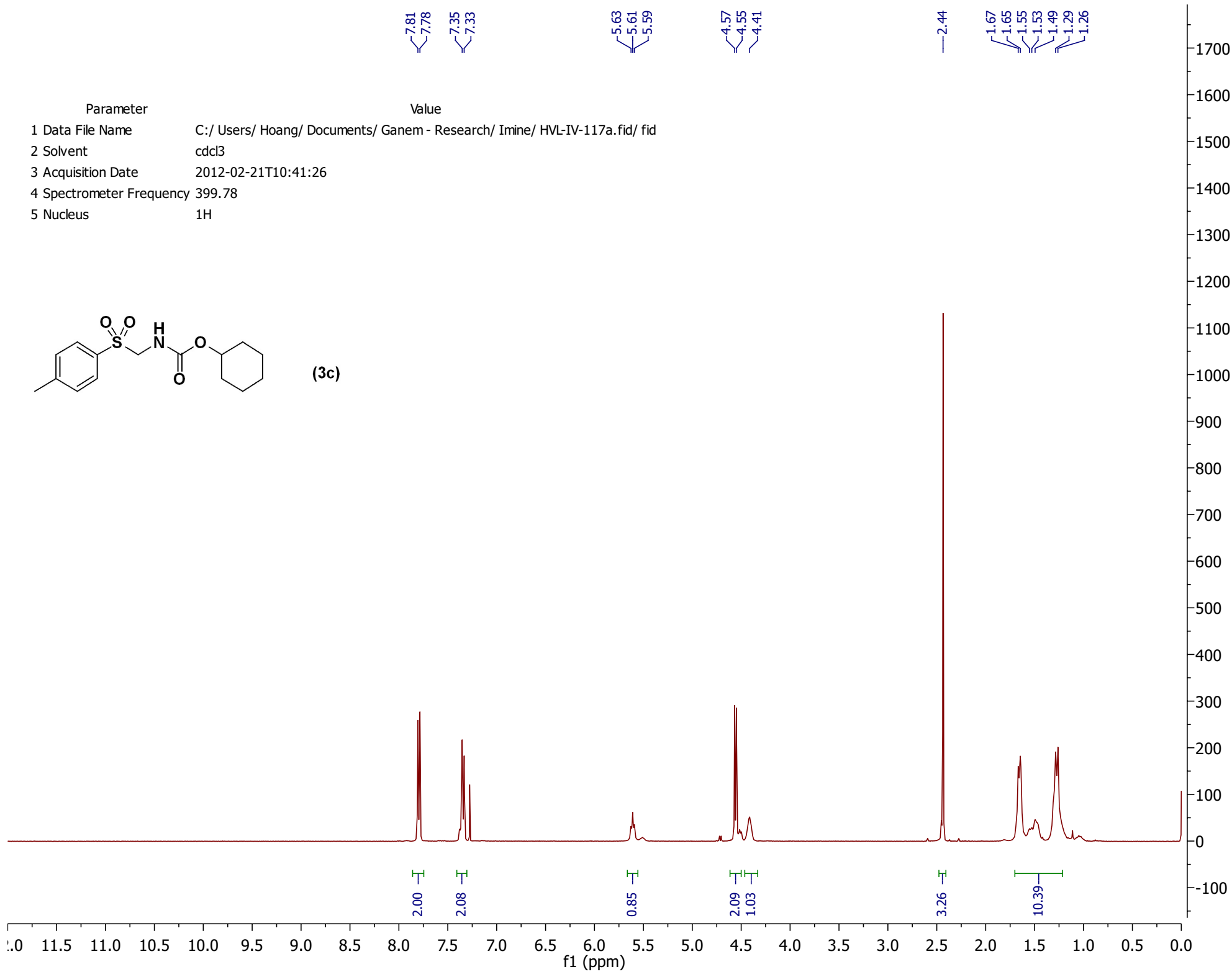
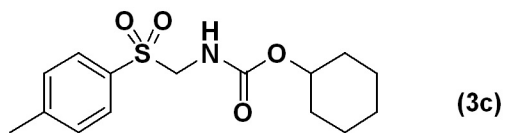
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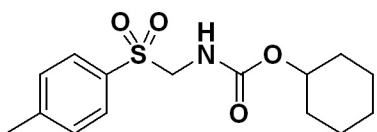






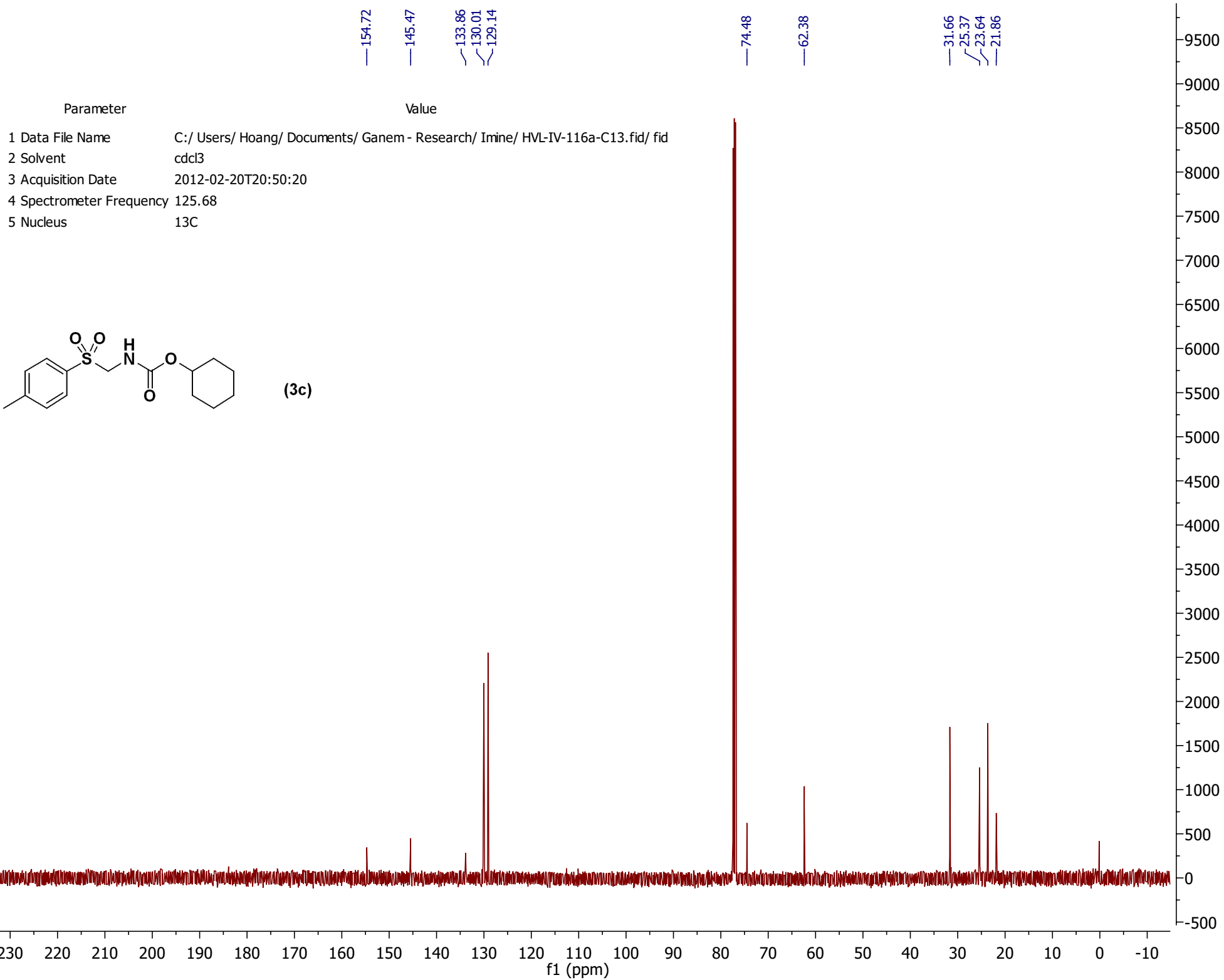
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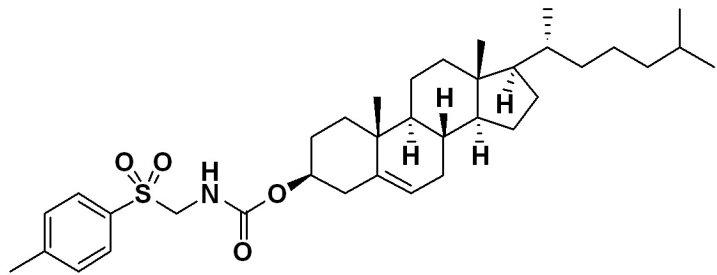




(3c)

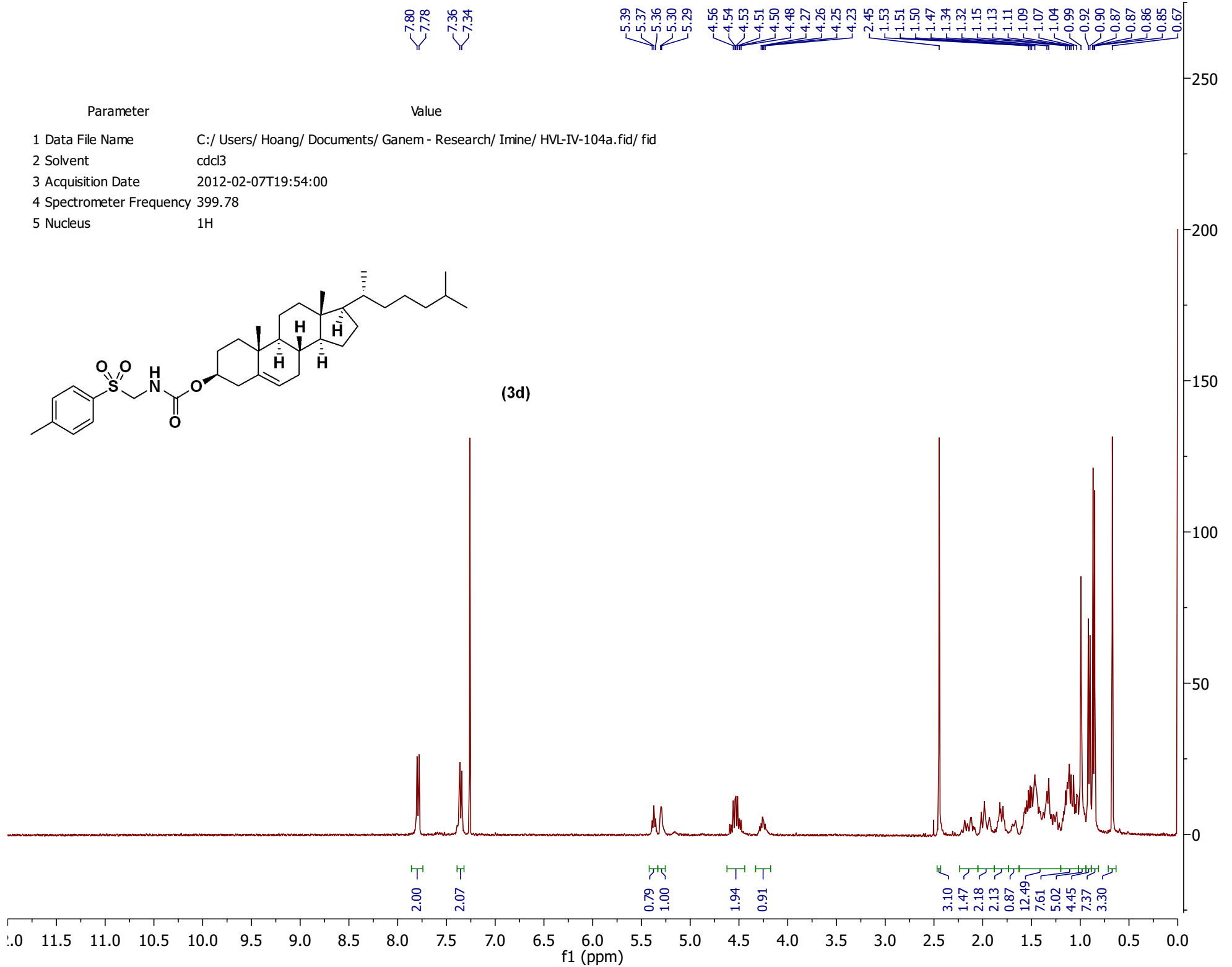
Parameter	Value
1 Data File Name	C:/Users/Hoang/Documents/Ganem - Research/Imine/HVL-IV-116a-C13.fid/ fid
2 Solvent	cdcl3
3 Acquisition Date	2012-02-20T20:50:20
4 Spectrometer Frequency	125.68
5 Nucleus	13C

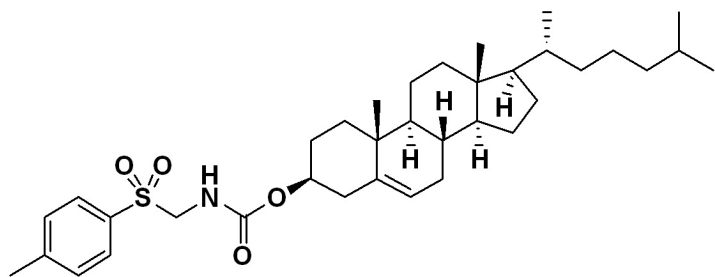




(3d)

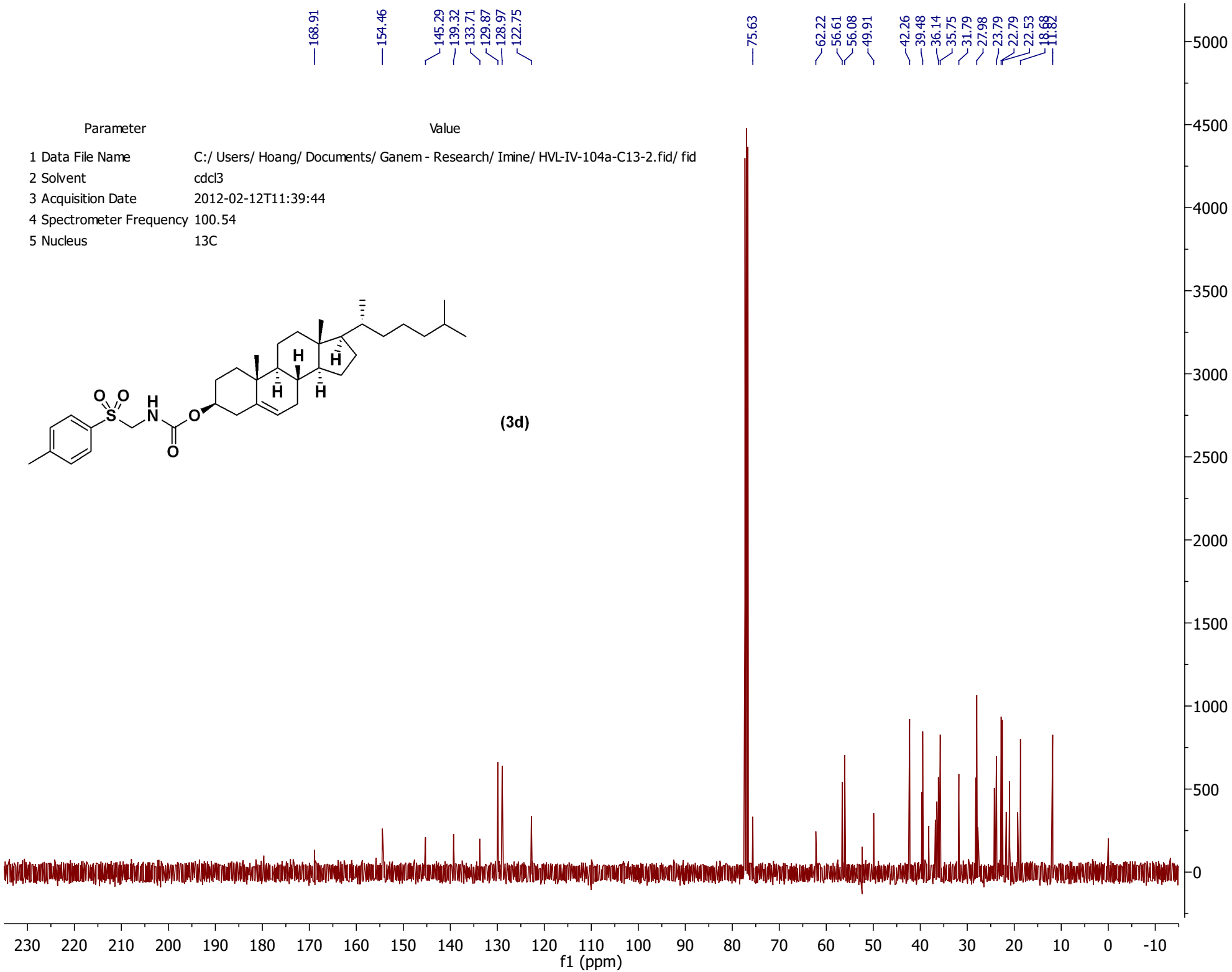
Parameter	Value
1 Data File Name	C:/ Users/ Hoang/ Documents/ Ganem - Research/ Imine/ HVL-IV-104a.fid/ fid
2 Solvent	cdcl3
3 Acquisition Date	2012-02-07T19:54:00
4 Spectrometer Frequency	399.78
5 Nucleus	1H



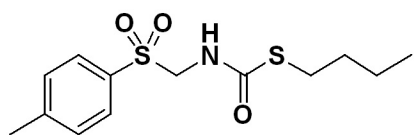


(3d)

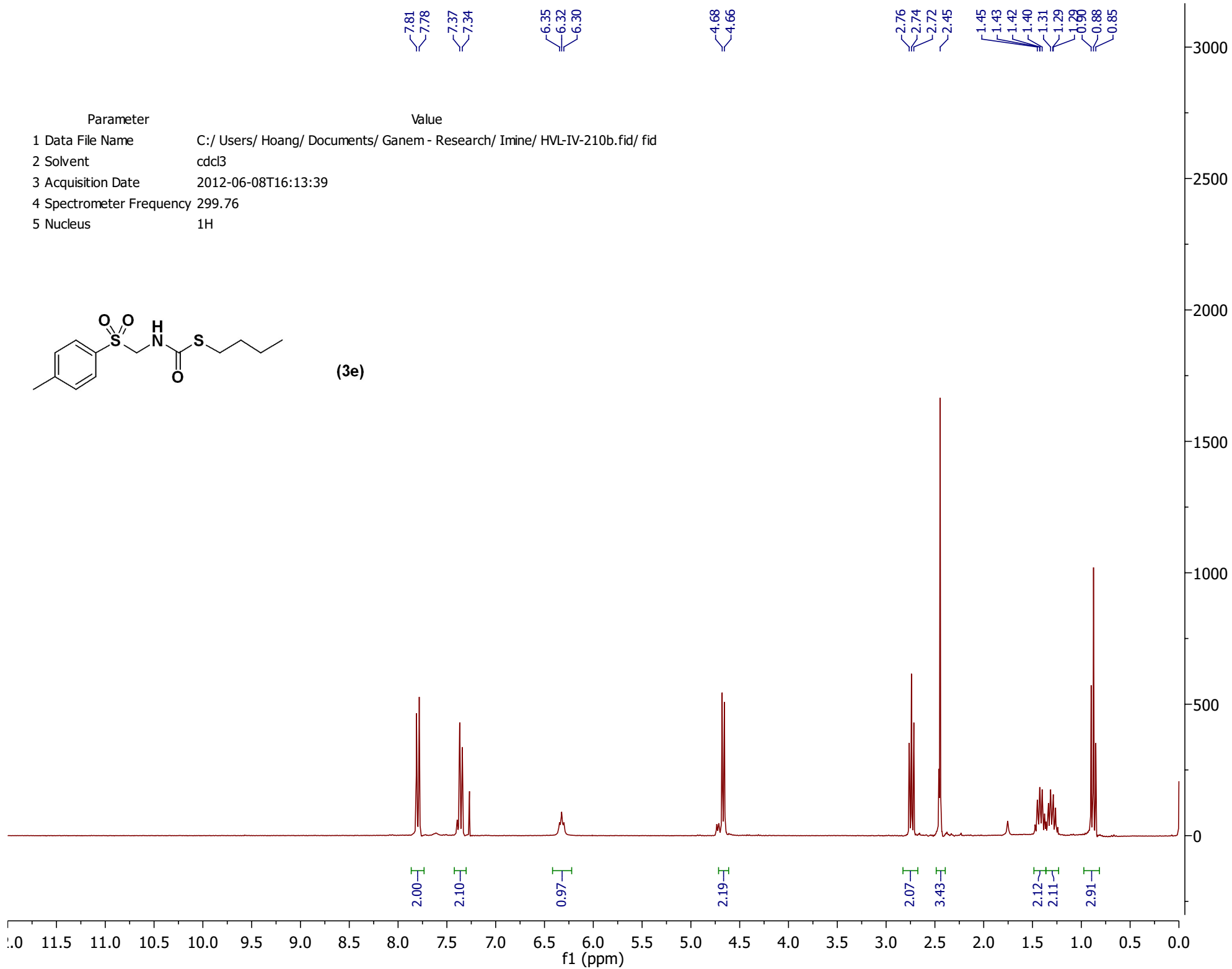
Parameter	Value
1 Data File Name	C:/ Users/ Hoang/ Documents/ Ganem - Research/ Imine/ HVL-IV-104a-C13-2.fid/ fid
2 Solvent	cdcl3
3 Acquisition Date	2012-02-12T11:39:44
4 Spectrometer Frequency	100.54
5 Nucleus	13C



Parameter	Value
1 Data File Name	C:/Users/Hoang/Documents/Ganem - Research/Imine/HVL-IV-210b.fid/ fid
2 Solvent	cdcl3
3 Acquisition Date	2012-06-08T16:13:39
4 Spectrometer Frequency	299.76
5 Nucleus	1H

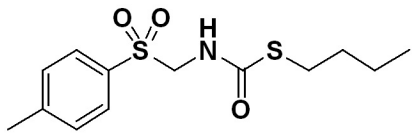


(3e)

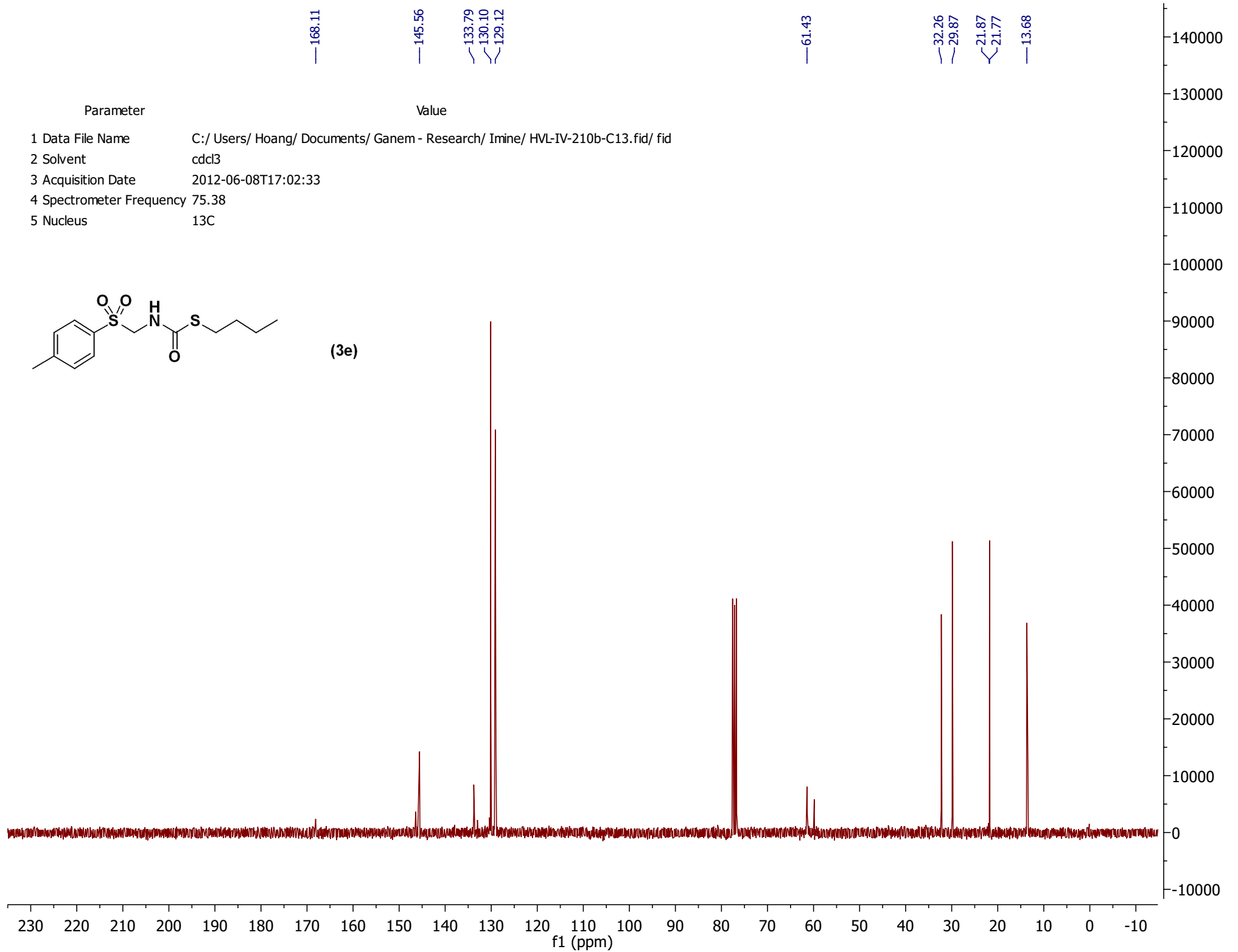


—168.11 —145.56 —133.79 —130.10 —129.12 —61.43 —32.26 —29.87 —21.87 —21.77 —13.68

Parameter	Value
1 Data File Name	C:/Users/Hoang/Documents/Ganem - Research/Imine/HVL-IV-210b-C13.fid/ fid
2 Solvent	cdcl3
3 Acquisition Date	2012-06-08T17:02:33
4 Spectrometer Frequency	75.38
5 Nucleus	13C

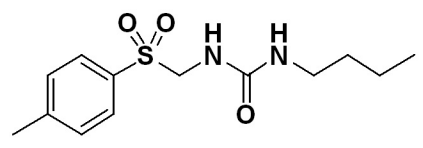


(3e)

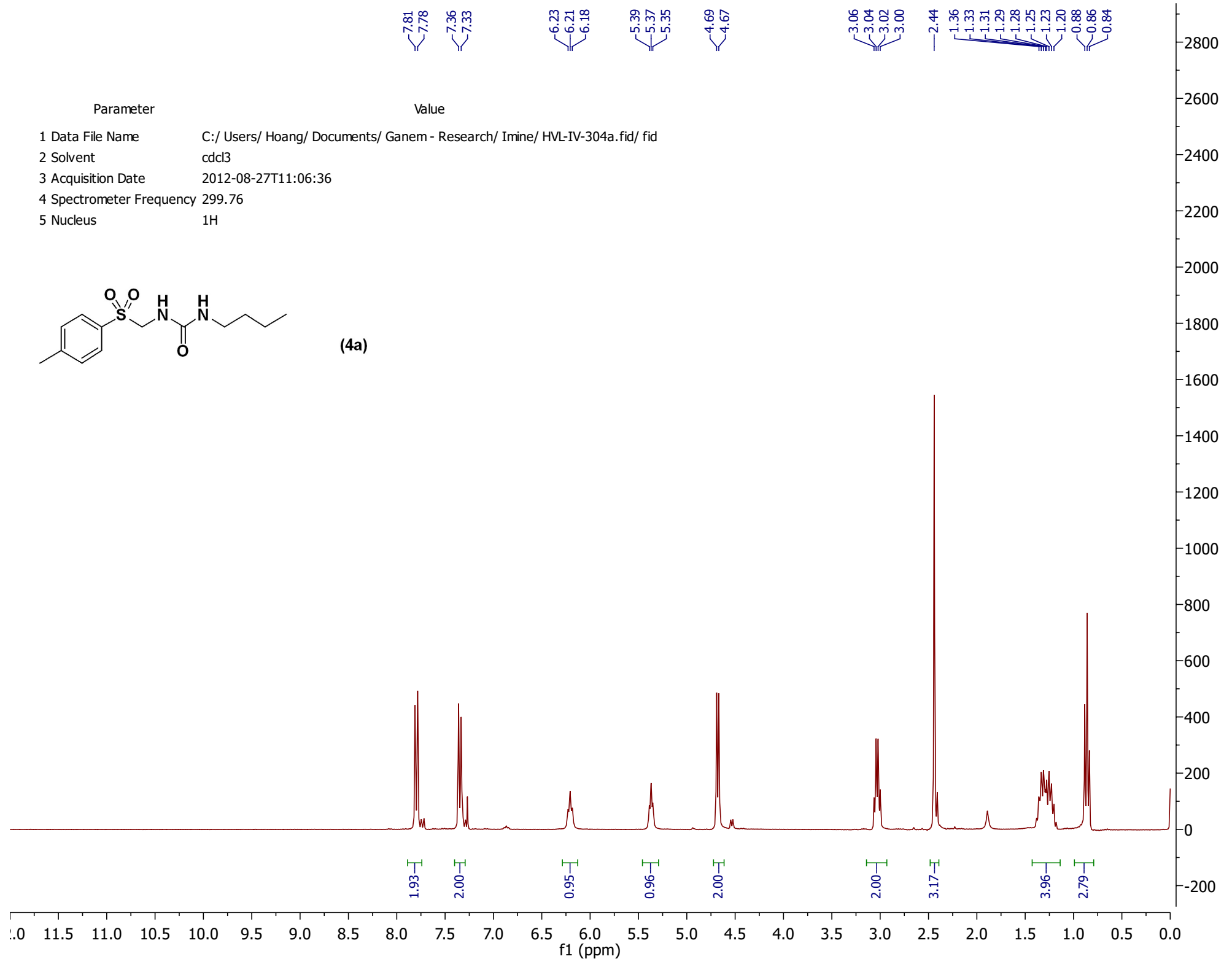


7.81 7.78 7.36 7.33 6.23 6.21 6.18 5.39 5.37 5.35 4.69 4.67 3.06 3.04 3.02 3.00 2.44 1.36 1.33 1.31 1.29 1.28 1.25 1.23 1.20 0.88 0.86 0.84

Parameter	Value
1 Data File Name	C:/Users/Hoang/Documents/Ganem - Research/Imine/HVL-IV-304a.fid/ fid
2 Solvent	cdcl3
3 Acquisition Date	2012-08-27T11:06:36
4 Spectrometer Frequency	299.76
5 Nucleus	1H

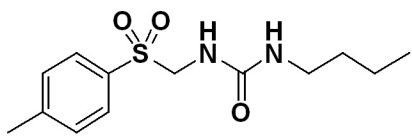


(4a)

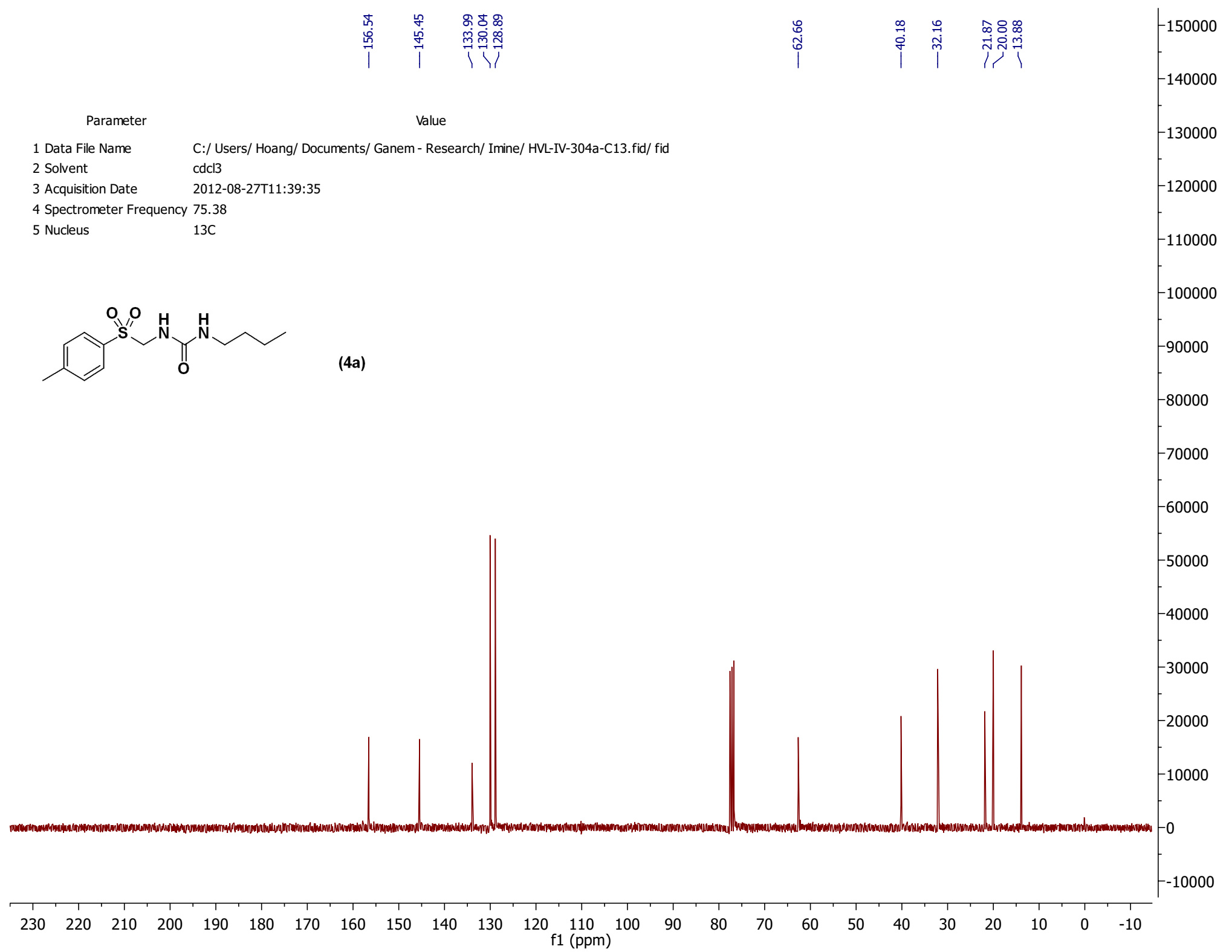


156.54
145.45
133.99
130.04
128.89
62.66
40.18
32.16
21.87
20.00
13.88

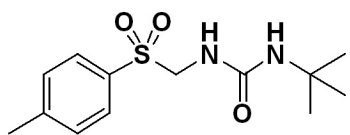
Parameter	Value
1 Data File Name	C:/Users/Hoang/Documents/Ganem - Research/Imine/HVL-IV-304a-C13.fid/ fid
2 Solvent	cdcl3
3 Acquisition Date	2012-08-27T11:39:35
4 Spectrometer Frequency	75.38
5 Nucleus	13C



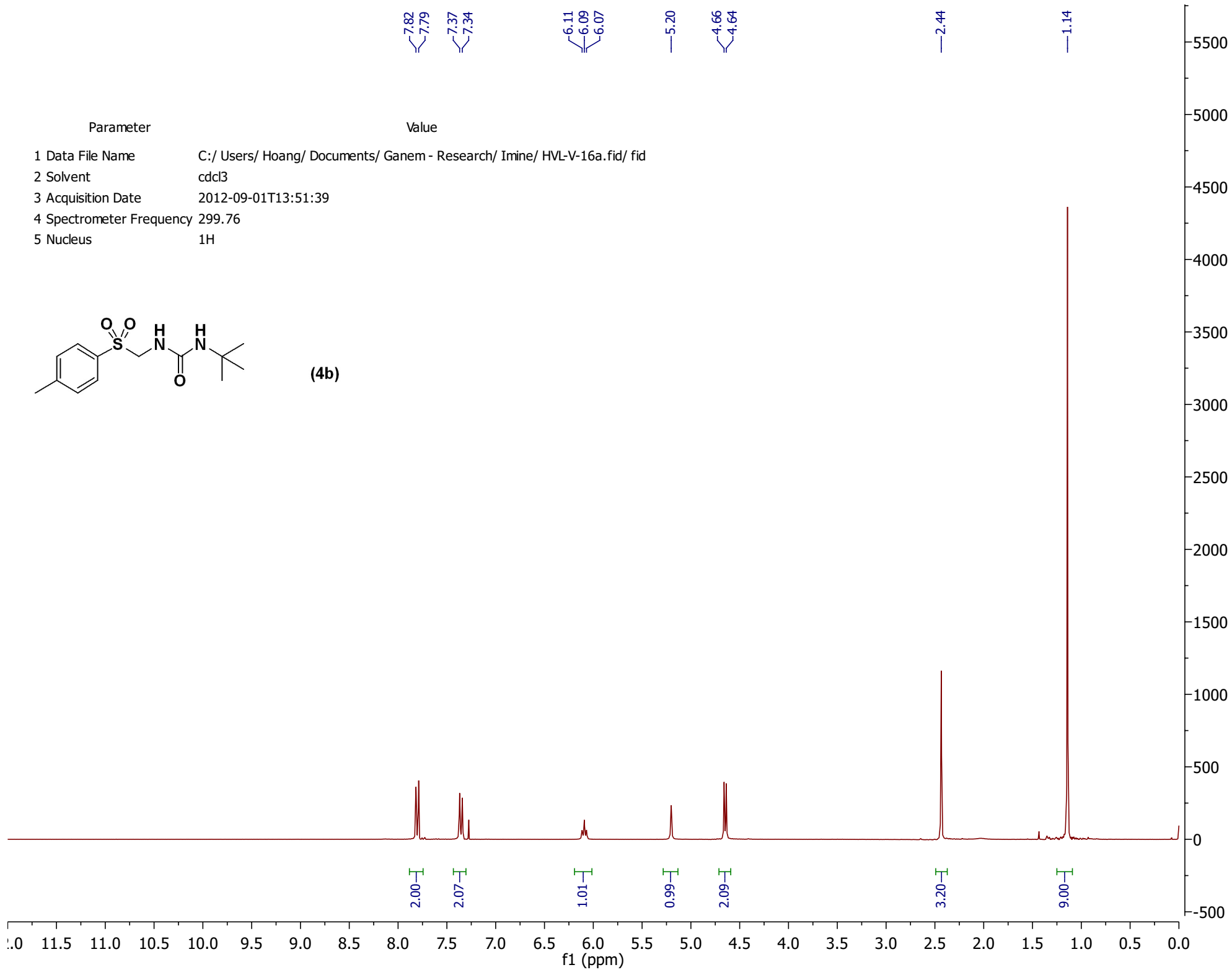
(4a)



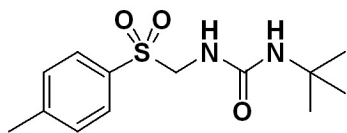
Parameter	Value
1 Data File Name	C:/Users/Hoang/Documents/Ganem - Research/Imine/HVL-V-16a.fid/fid
2 Solvent	cdcl3
3 Acquisition Date	2012-09-01T13:51:39
4 Spectrometer Frequency	299.76
5 Nucleus	1H



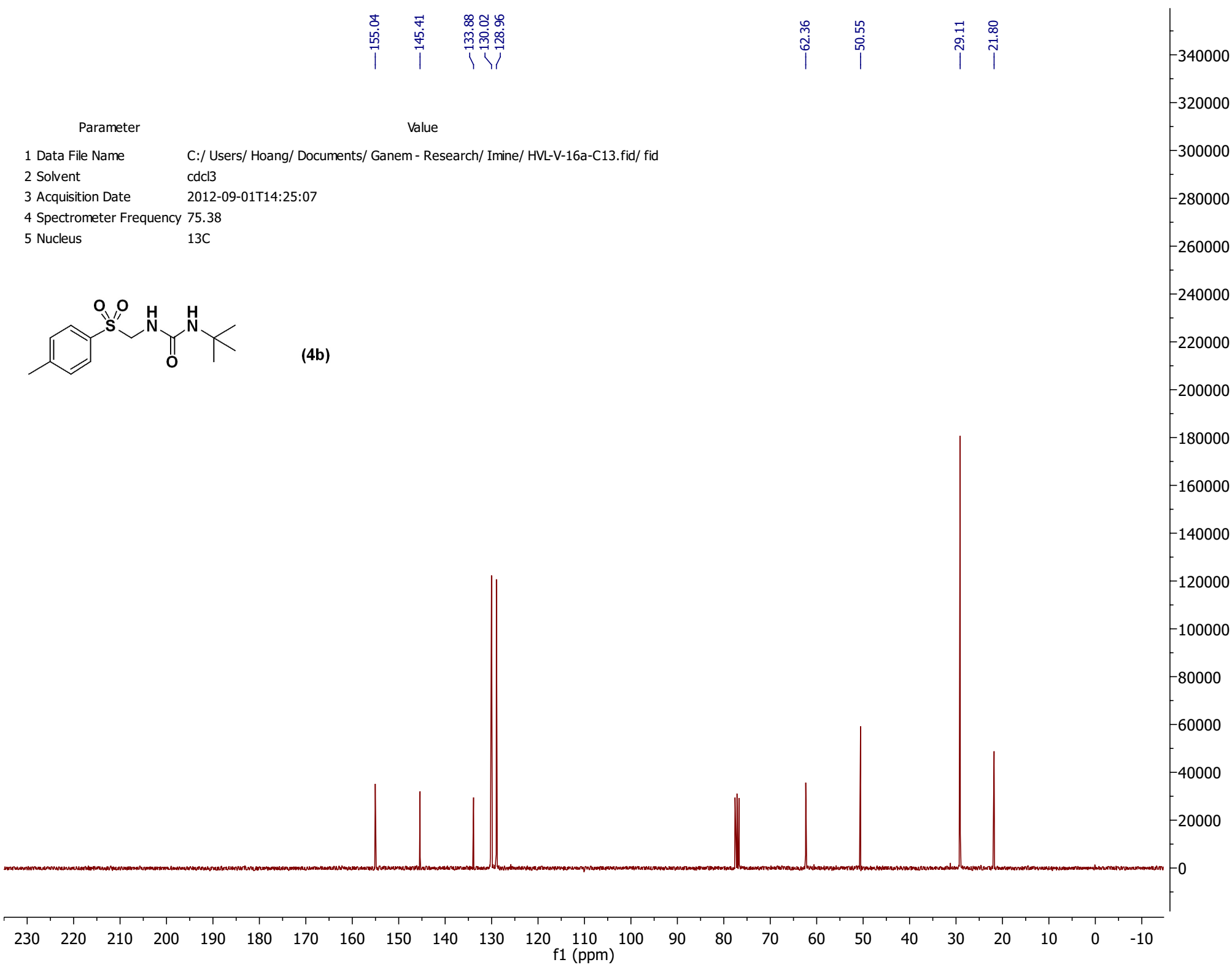
(4b)

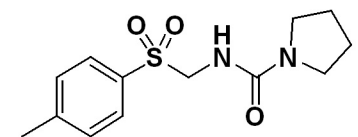


Parameter	Value
1 Data File Name	C:/Users/Hoang/Documents/Ganem - Research/Imine/HVL-V-16a-C13.fid/ fid
2 Solvent	cdcl3
3 Acquisition Date	2012-09-01T14:25:07
4 Spectrometer Frequency	75.38
5 Nucleus	¹³ C



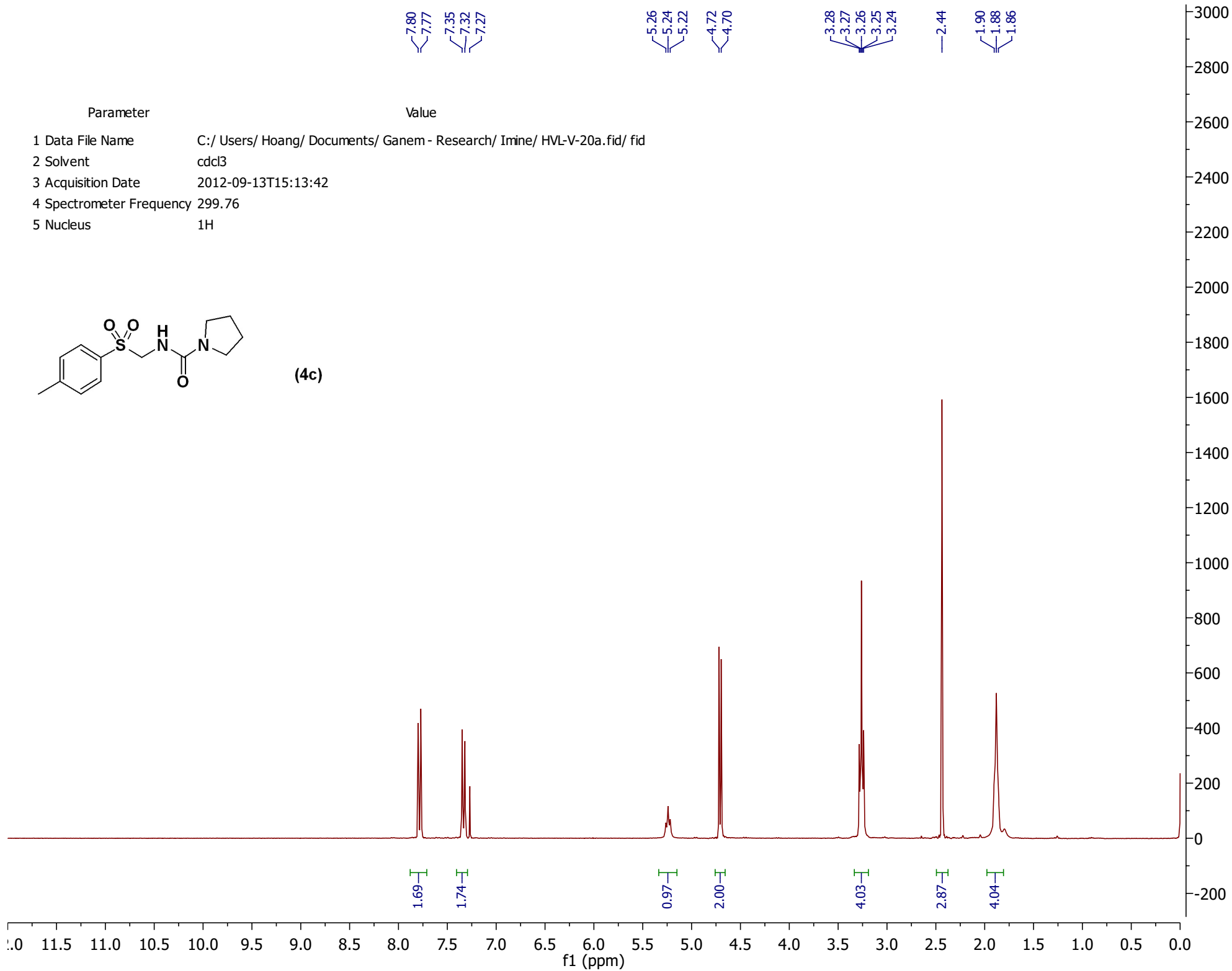
(4b)





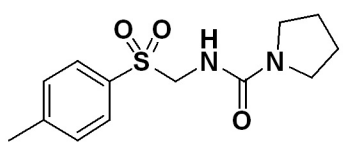
(4c)

Parameter	Value
1 Data File Name	C:/Users/Hoang/Documents/Ganem - Research/Imine/HVL-V-20a.fid/fid
2 Solvent	cdcl3
3 Acquisition Date	2012-09-13T15:13:42
4 Spectrometer Frequency	299.76
5 Nucleus	1H

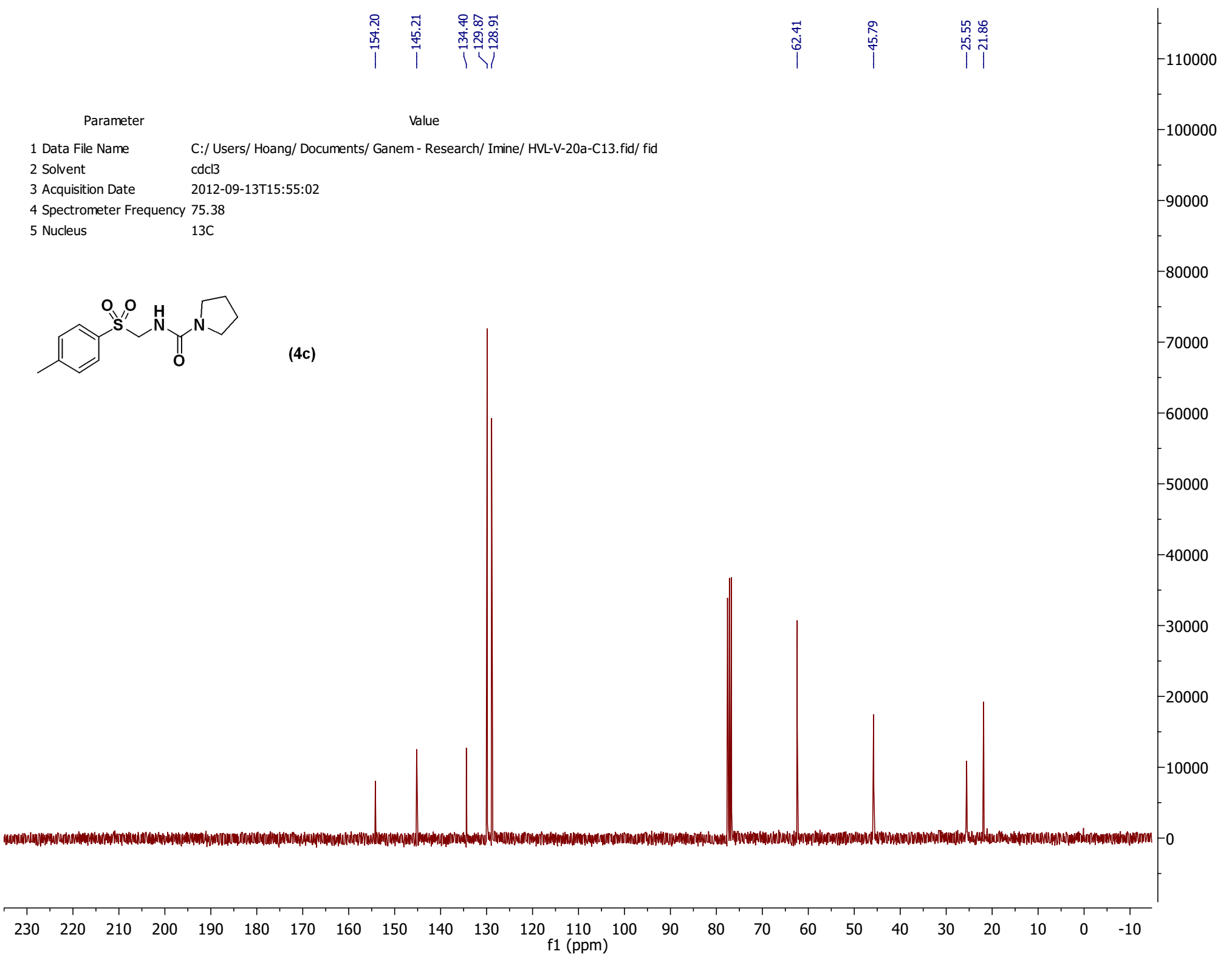


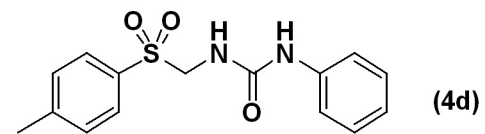
— 154.20 — 145.21 — 134.40 — 129.87 — 128.91 — 62.41 — 45.79 — 25.55 — 21.86

Parameter	Value
1 Data File Name	C:/Users/Hoang/Documents/Ganem - Research/Imine/HVL-V-20a-C13.fid/ fid
2 Solvent	cdcl3
3 Acquisition Date	2012-09-13T15:55:02
4 Spectrometer Frequency	75.38
5 Nucleus	13C

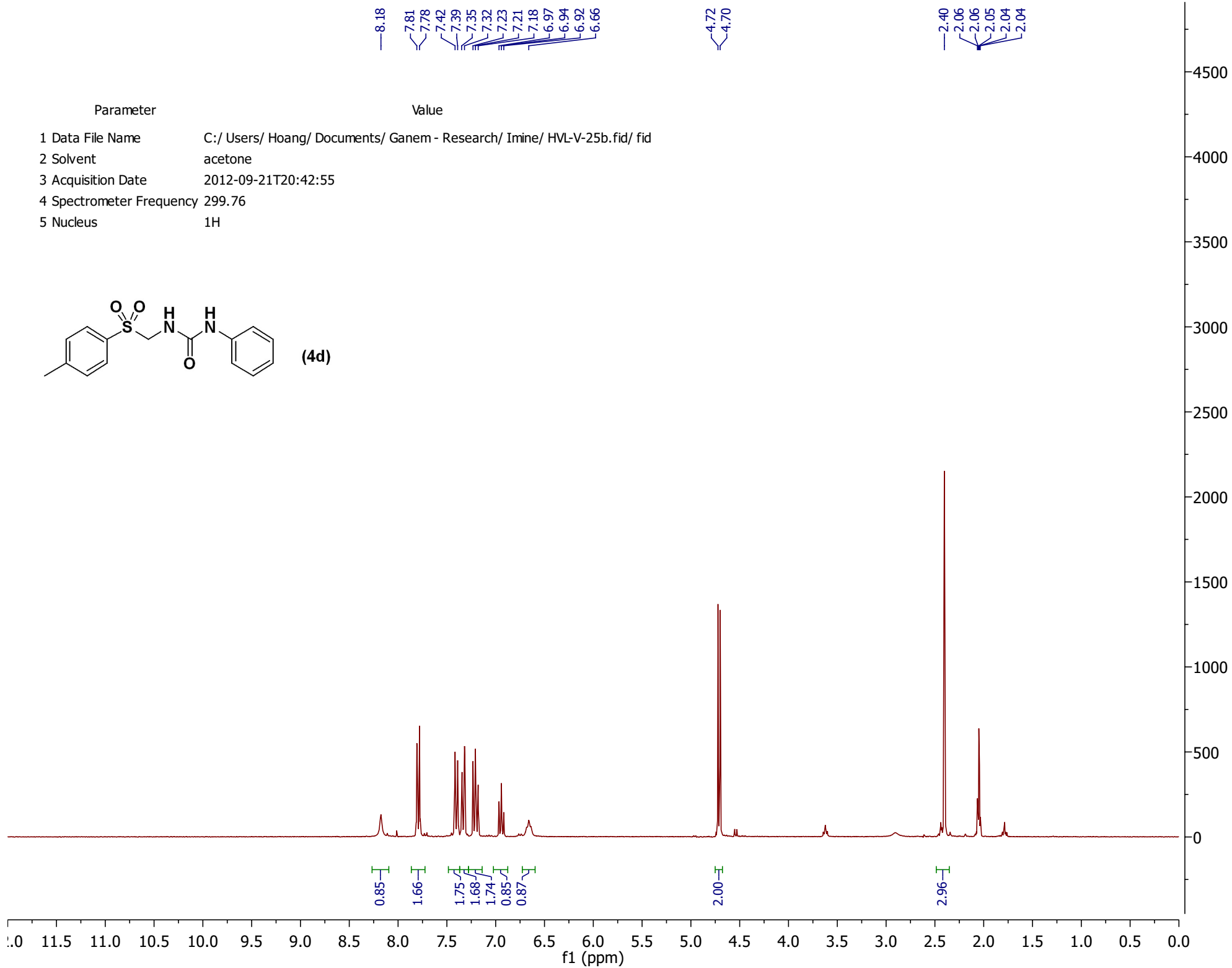


(4c)

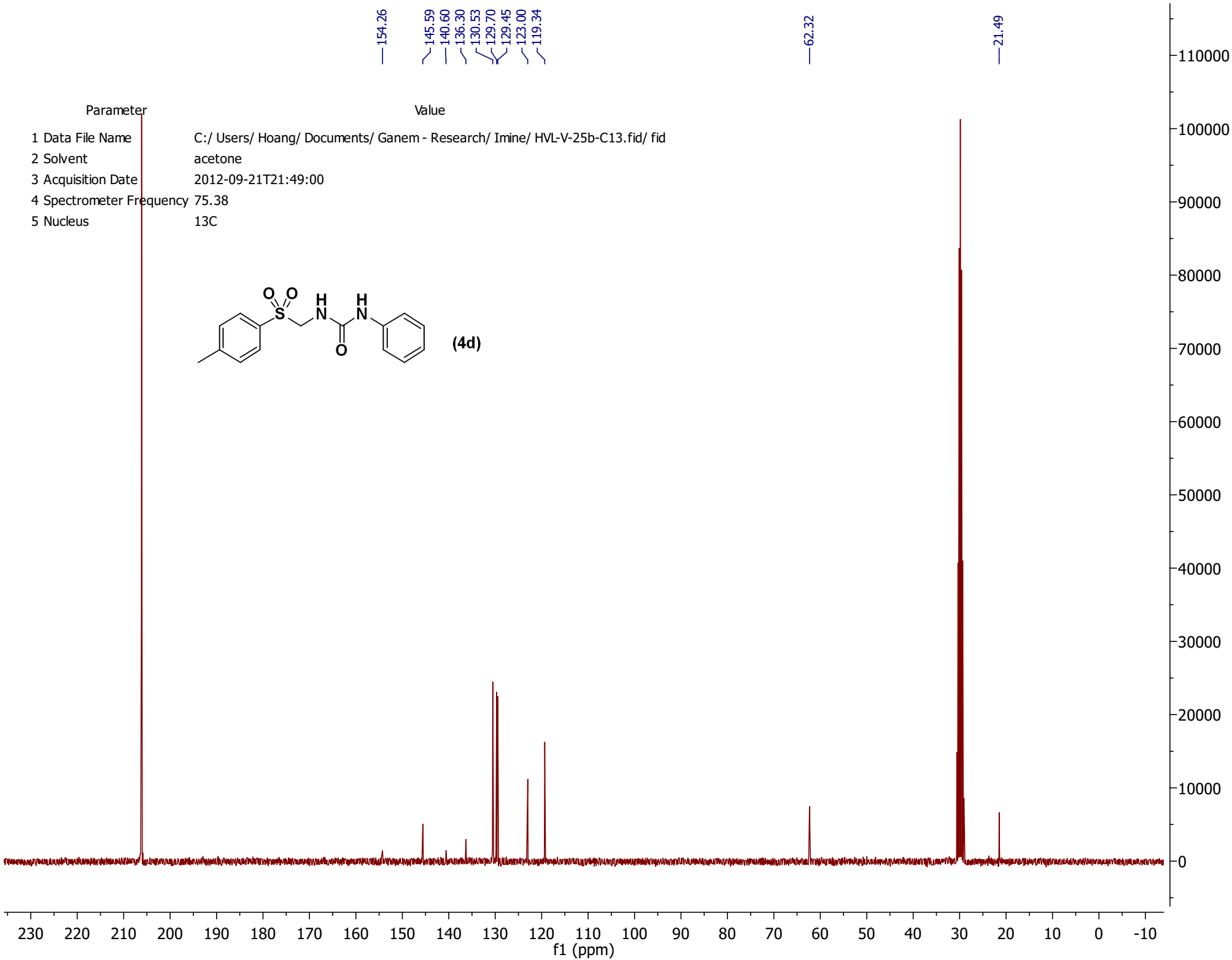
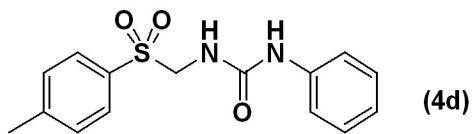




Parameter	Value
1 Data File Name	C:/Users/Hoang/Documents/Ganem - Research/Imine/HVL-V-25b.fid/ fid
2 Solvent	acetone
3 Acquisition Date	2012-09-21T20:42:55
4 Spectrometer Frequency	299.76
5 Nucleus	¹ H



Parameter Value
1 Data File Name C:/Users/Hoang/Documents/Ganem - Research/Imine/HVL-V-25b-C13.fid/ fid
2 Solvent acetone
3 Acquisition Date 2012-09-21T21:49:00
4 Spectrometer Frequency 75.38
5 Nucleus ¹³C



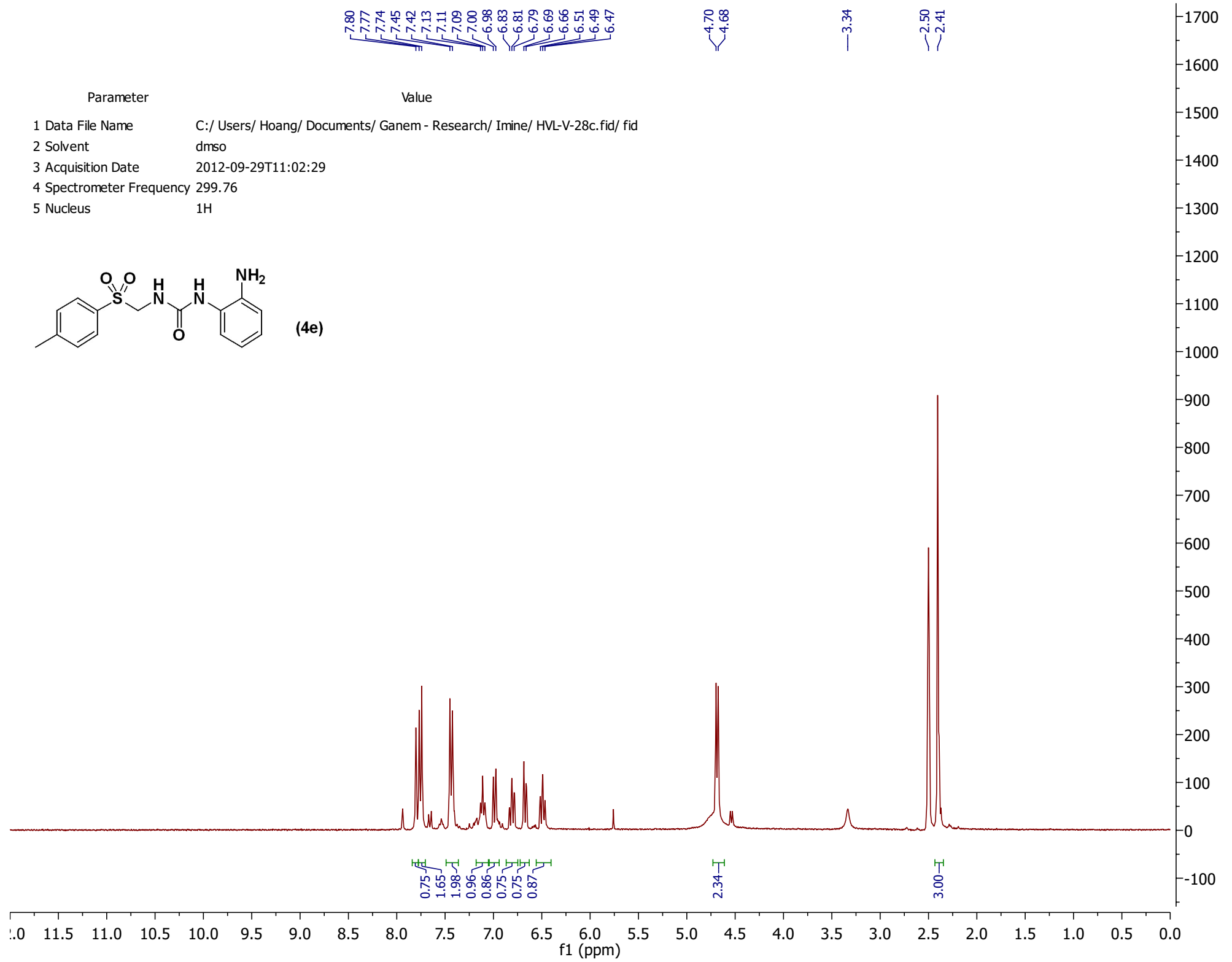
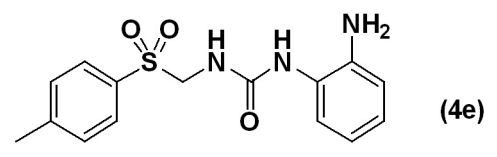
7.80
7.77
7.74
7.45
7.42
7.13
7.11
7.09
7.00
6.98
6.83
6.81
6.79
6.69
6.66
6.51
6.49
6.47

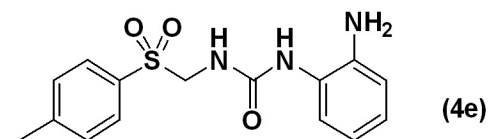
4.70
4.68

3.34

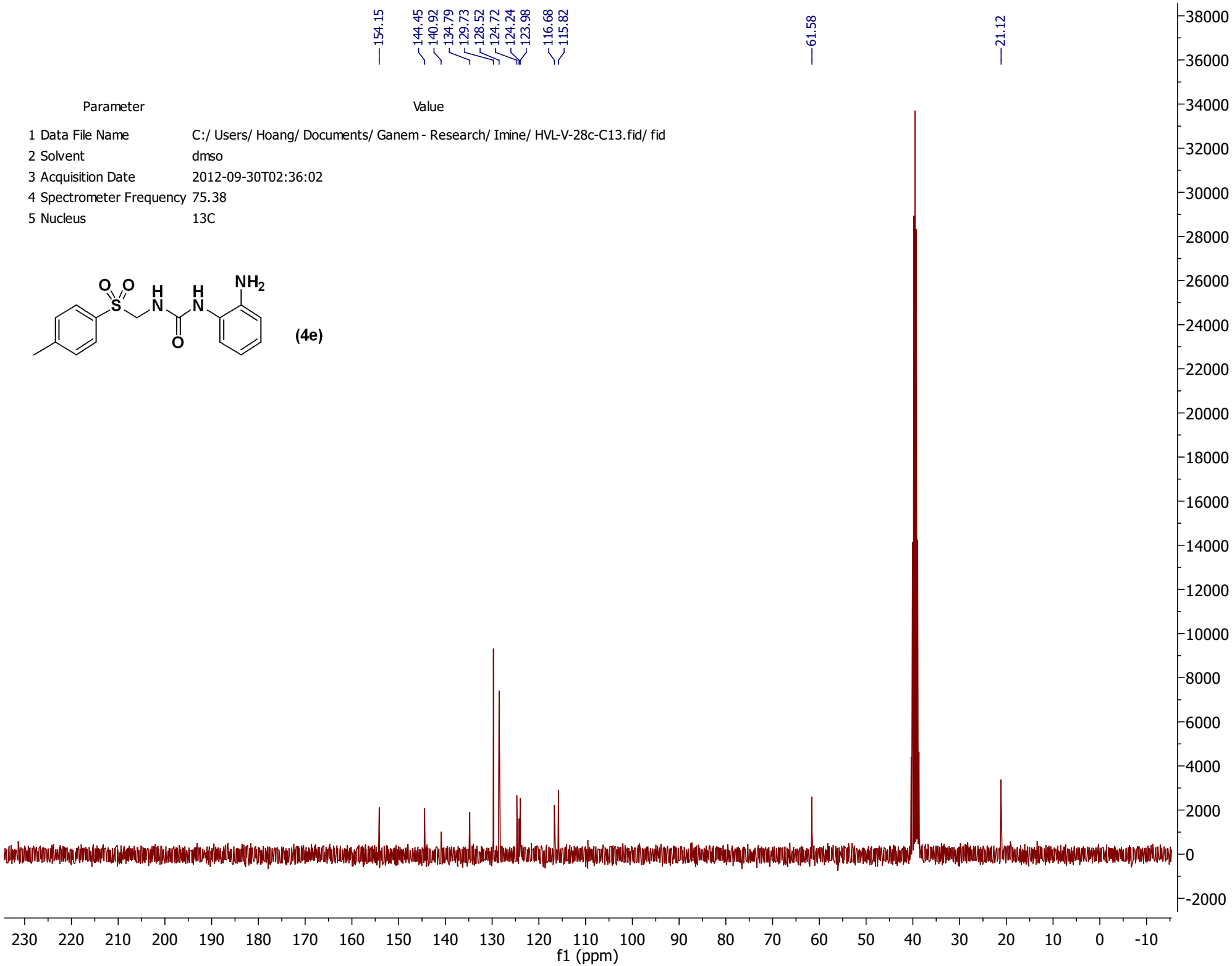
2.50
2.41

Parameter	Value
1 Data File Name	C:/ Users/ Hoang/ Documents/ Ganem - Research/ Imine/ HVL-V-28c.fid/ fid
2 Solvent	dms0
3 Acquisition Date	2012-09-29T11:02:29
4 Spectrometer Frequency	299.76
5 Nucleus	1H





Parameter	Value
1 Data File Name	C:/Users/Hoang/Documents/Ganem - Research/Imine/HVL-V-28c-C13.fid/ fid
2 Solvent	dmsO
3 Acquisition Date	2012-09-30T02:36:02
4 Spectrometer Frequency	75.38
5 Nucleus	¹³ C

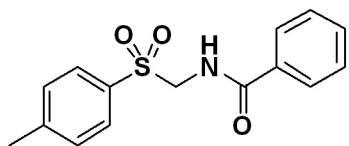


7.80
7.77
7.69
7.67
7.54
7.51
7.49
7.42
7.42
7.40
7.40
7.37
7.35
7.33
7.31
7.28

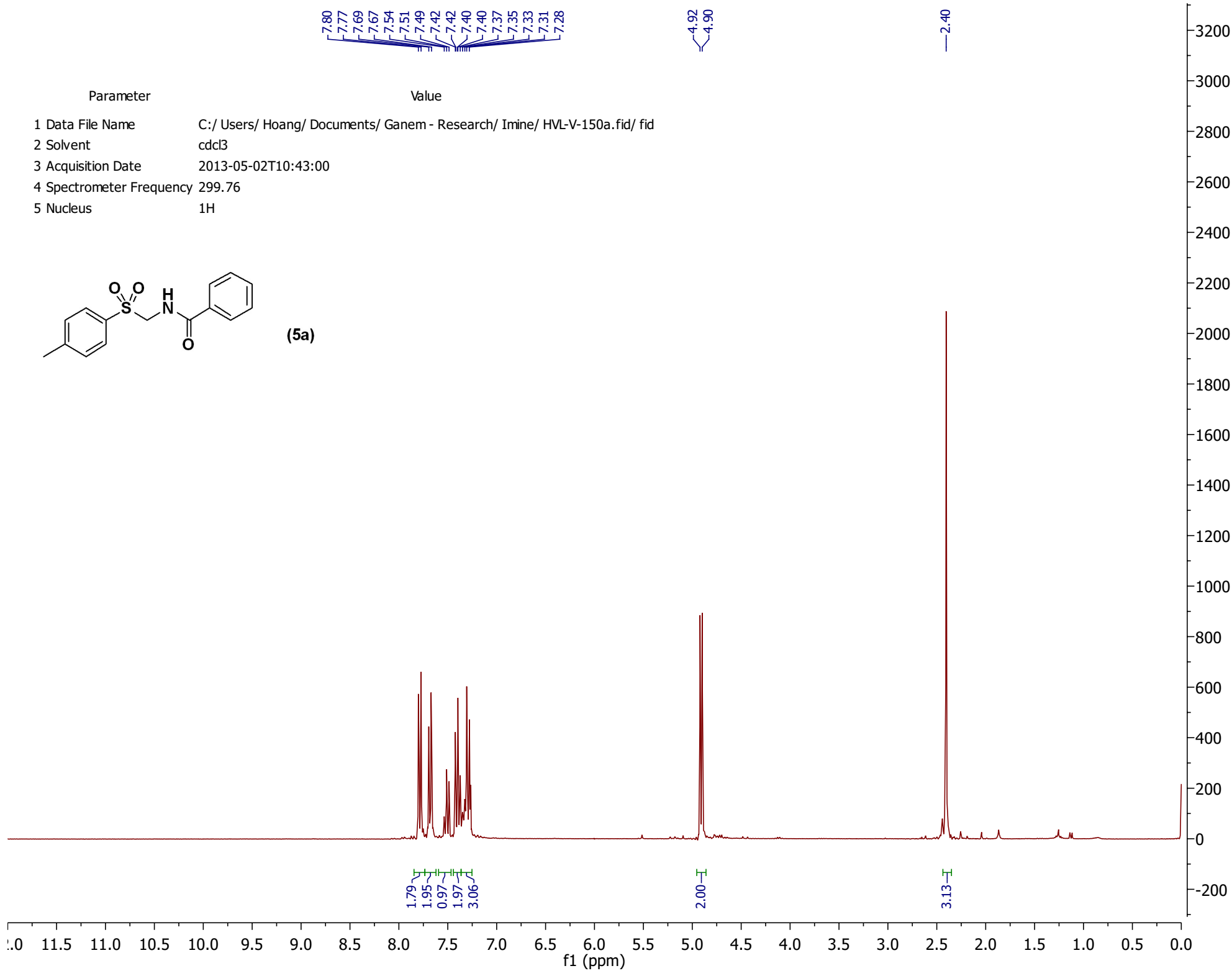
4.92
4.90

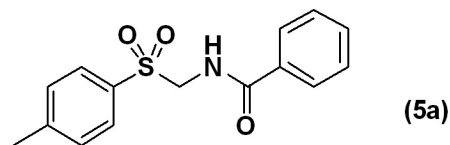
2.40

Parameter	Value
1 Data File Name	C:/ Users/ Hoang/ Documents/ Ganem - Research/ Imine/ HVL-V-150a.fid/ fid
2 Solvent	cdcl3
3 Acquisition Date	2013-05-02T10:43:00
4 Spectrometer Frequency	299.76
5 Nucleus	1H

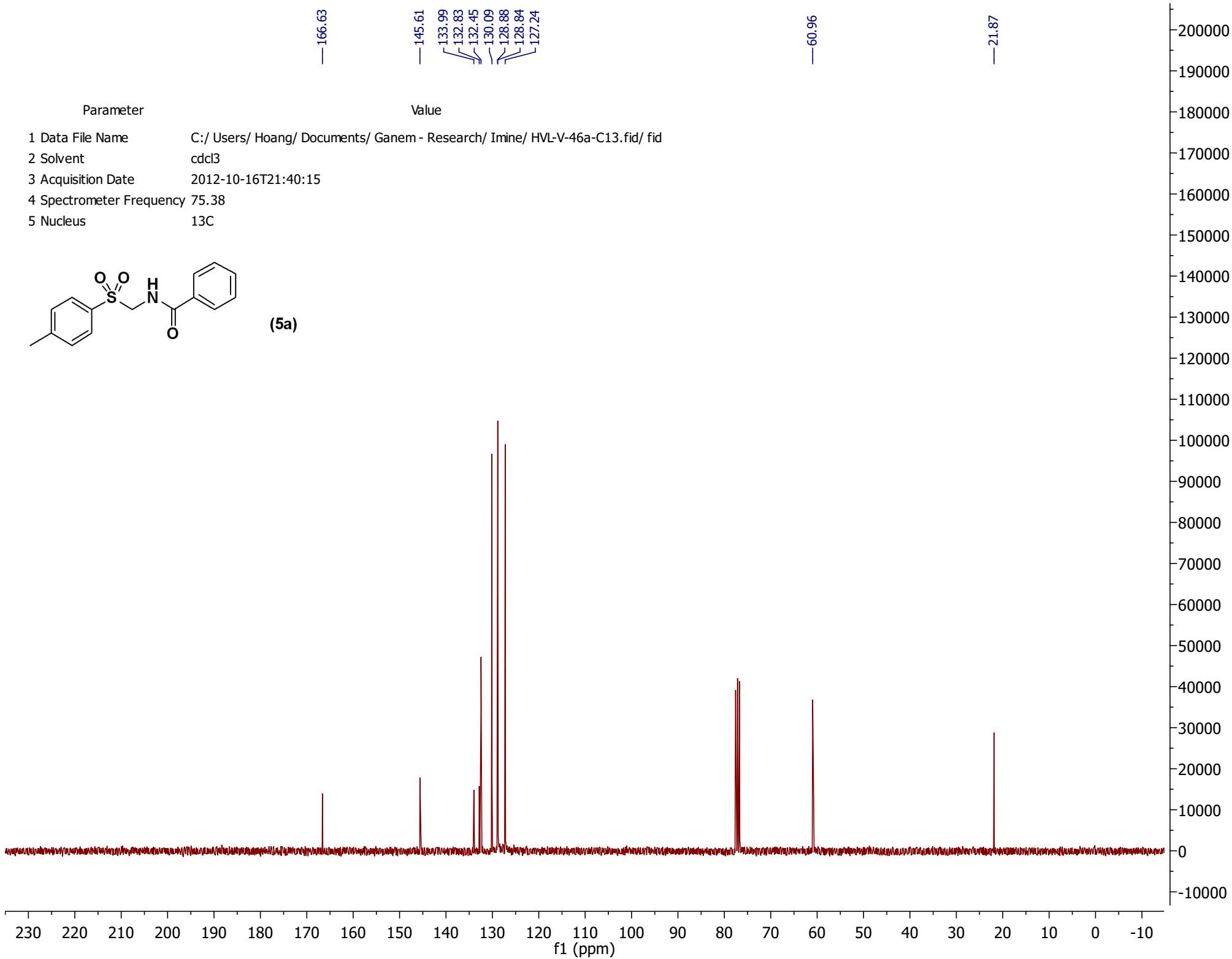


(5a)





Parameter	Value
1 Data File Name	C:/Users/Hoang/Documents/Ganem - Research/Imine/HVL-V-46a-C13.fid/ fid
2 Solvent	cdcl3
3 Acquisition Date	2012-10-16T21:40:15
4 Spectrometer Frequency	75.38
5 Nucleus	13C



7.79
7.77
7.68
7.65
7.30
7.27
7.22
7.20
7.18
6.90
6.87

4.91
4.89

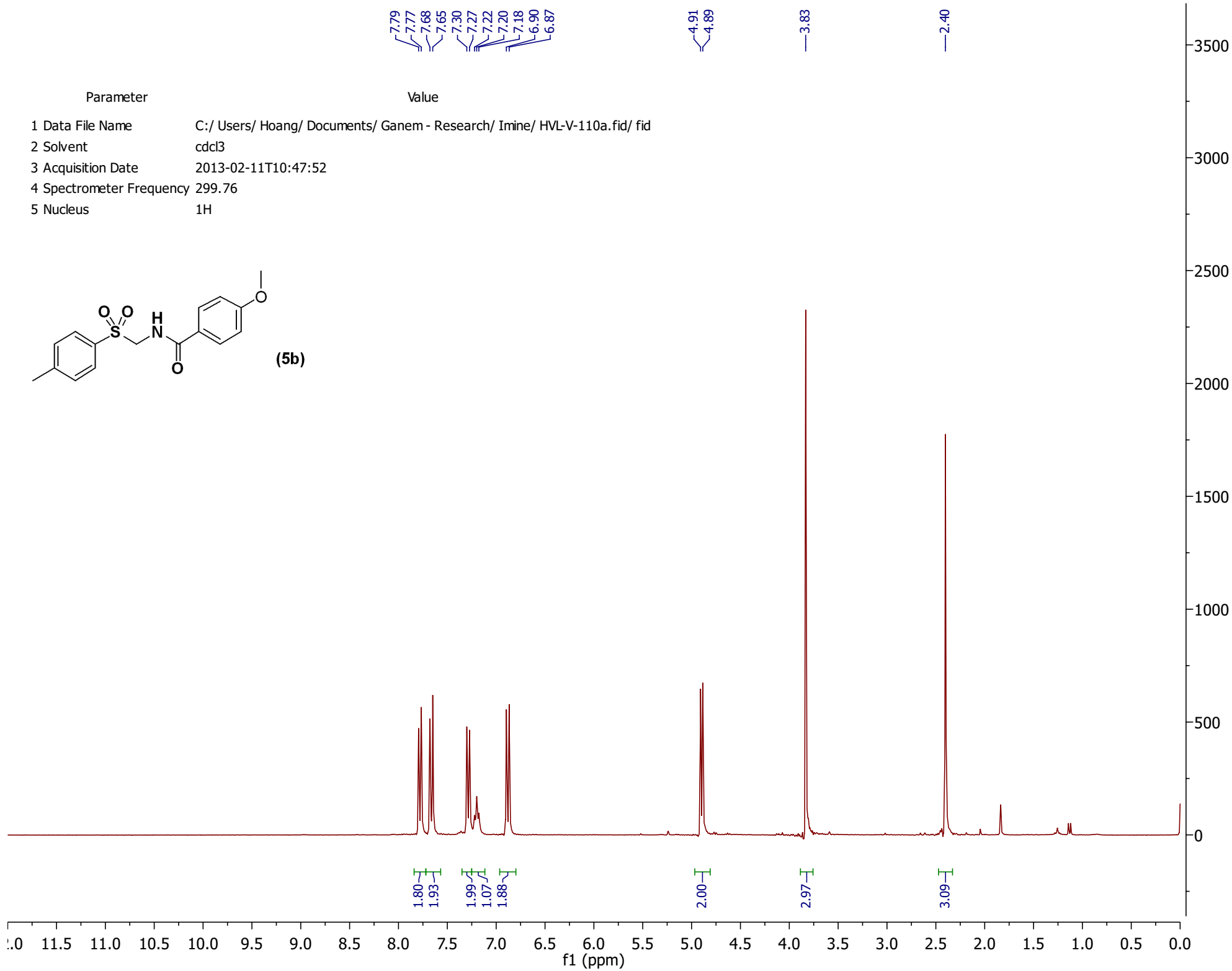
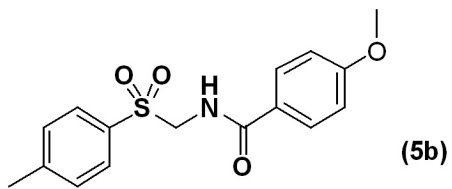
3.83

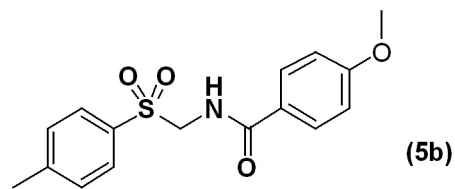
2.40

Parameter

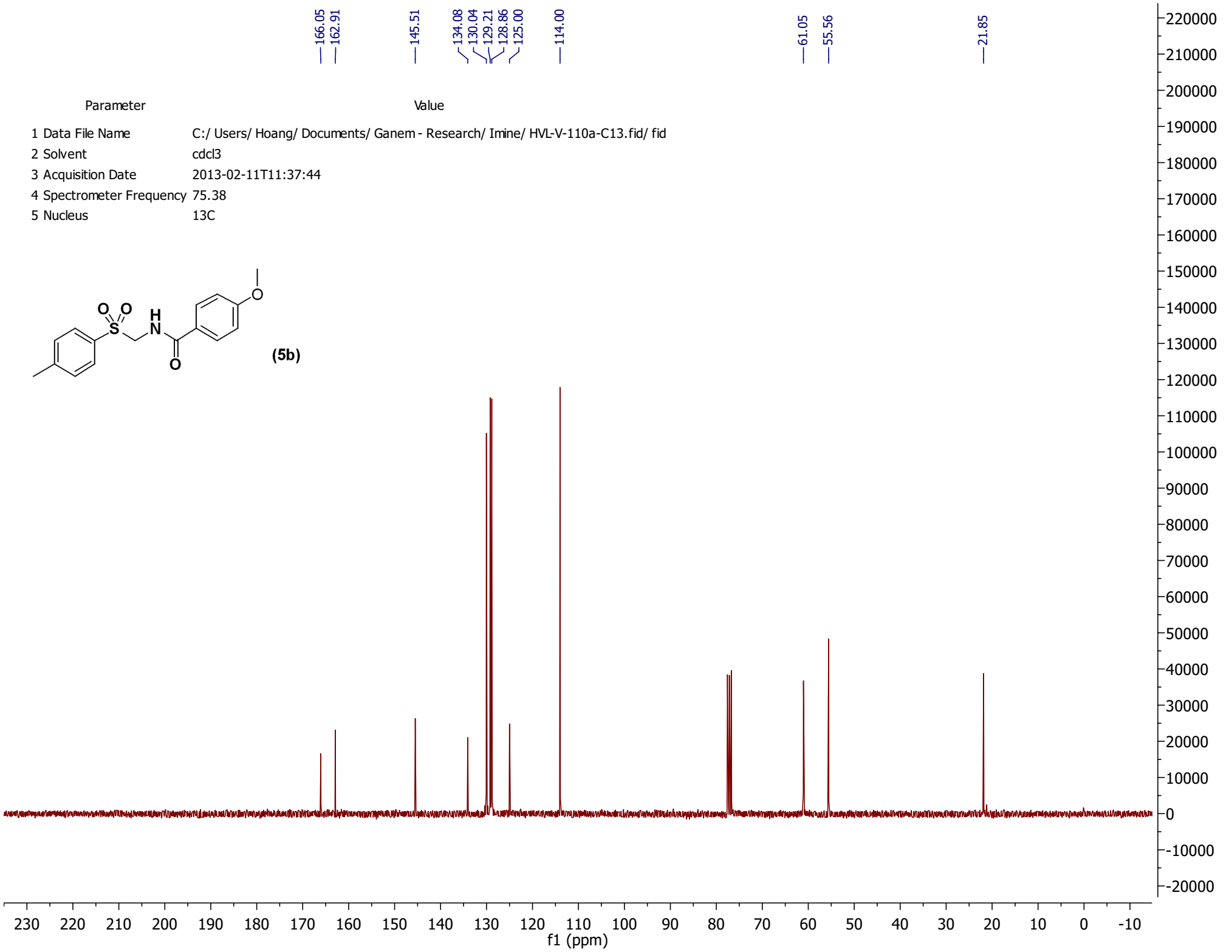
Value

1 Data File Name C:/Users/Hoang/Documents/Ganem - Research/Imine/HVL-V-110a.fid/ fid
2 Solvent cdcl3
3 Acquisition Date 2013-02-11T10:47:52
4 Spectrometer Frequency 299.76
5 Nucleus 1H





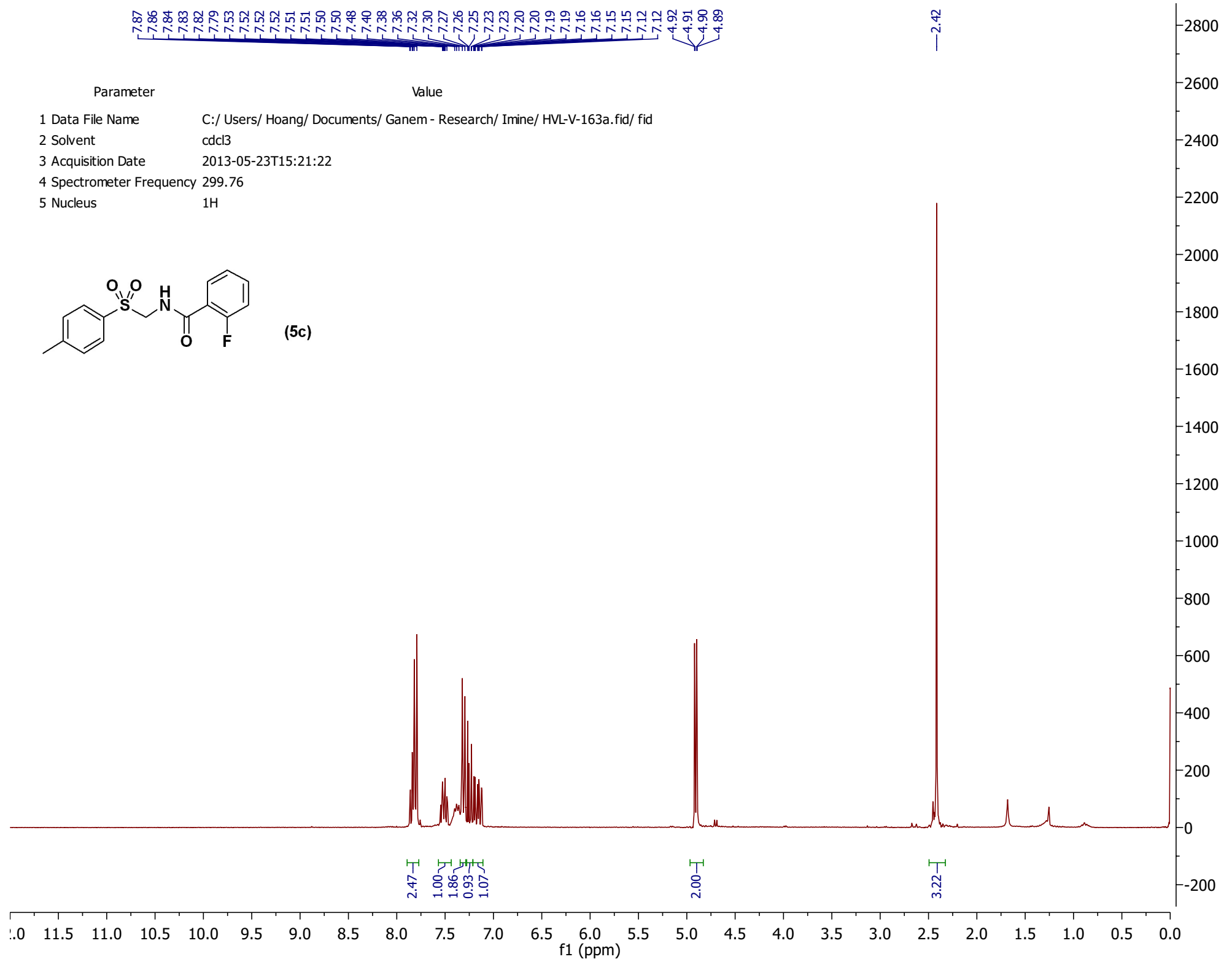
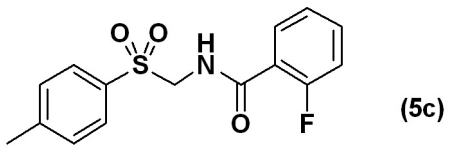
Parameter	Value
1 Data File Name	C:/Users/Hoang/Documents/Ganem - Research/Imine/HVL-V-110a-C13.fid/ fid
2 Solvent	cdcl3
3 Acquisition Date	2013-02-11T11:37:44
4 Spectrometer Frequency	75.38
5 Nucleus	13C



7.87
7.86
7.84
7.83
7.82
7.79
7.53
7.52
7.52
7.51
7.51
7.50
7.50
7.48
7.40
7.38
7.36
7.32
7.30
7.27
7.26
7.25
7.23
7.23
7.20
7.20
7.19
7.19
7.16
7.16
7.15
7.15
7.12
7.12
4.92
4.91
4.90
4.89

2.42

Parameter	Value
1 Data File Name	C:/Users/Hoang/Documents/Ganem - Research/Imine/HVL-V-163a.fid/fid
2 Solvent	cdcl3
3 Acquisition Date	2013-05-23T15:21:22
4 Spectrometer Frequency	299.76
5 Nucleus	1H

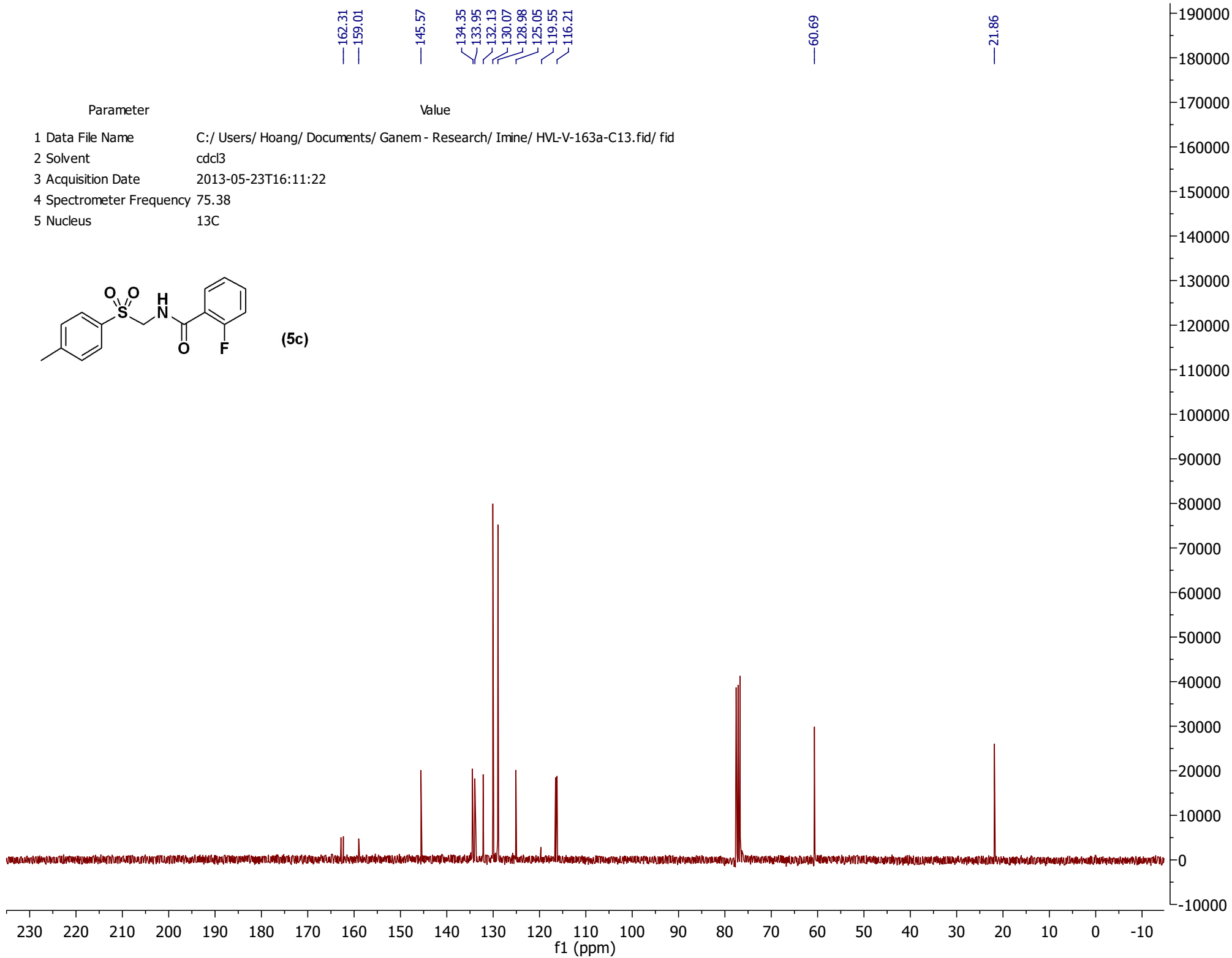
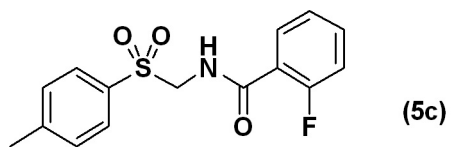


162.31
159.01
145.57
134.35
133.95
132.13
130.07
128.98
125.05
119.55
116.21

60.69

21.86

Parameter	Value
1 Data File Name	C:/Users/Hoang/Documents/Ganem - Research/Imine/HVL-V-163a-C13.fid/ fid
2 Solvent	cdcl3
3 Acquisition Date	2013-05-23T16:11:22
4 Spectrometer Frequency	75.38
5 Nucleus	13C

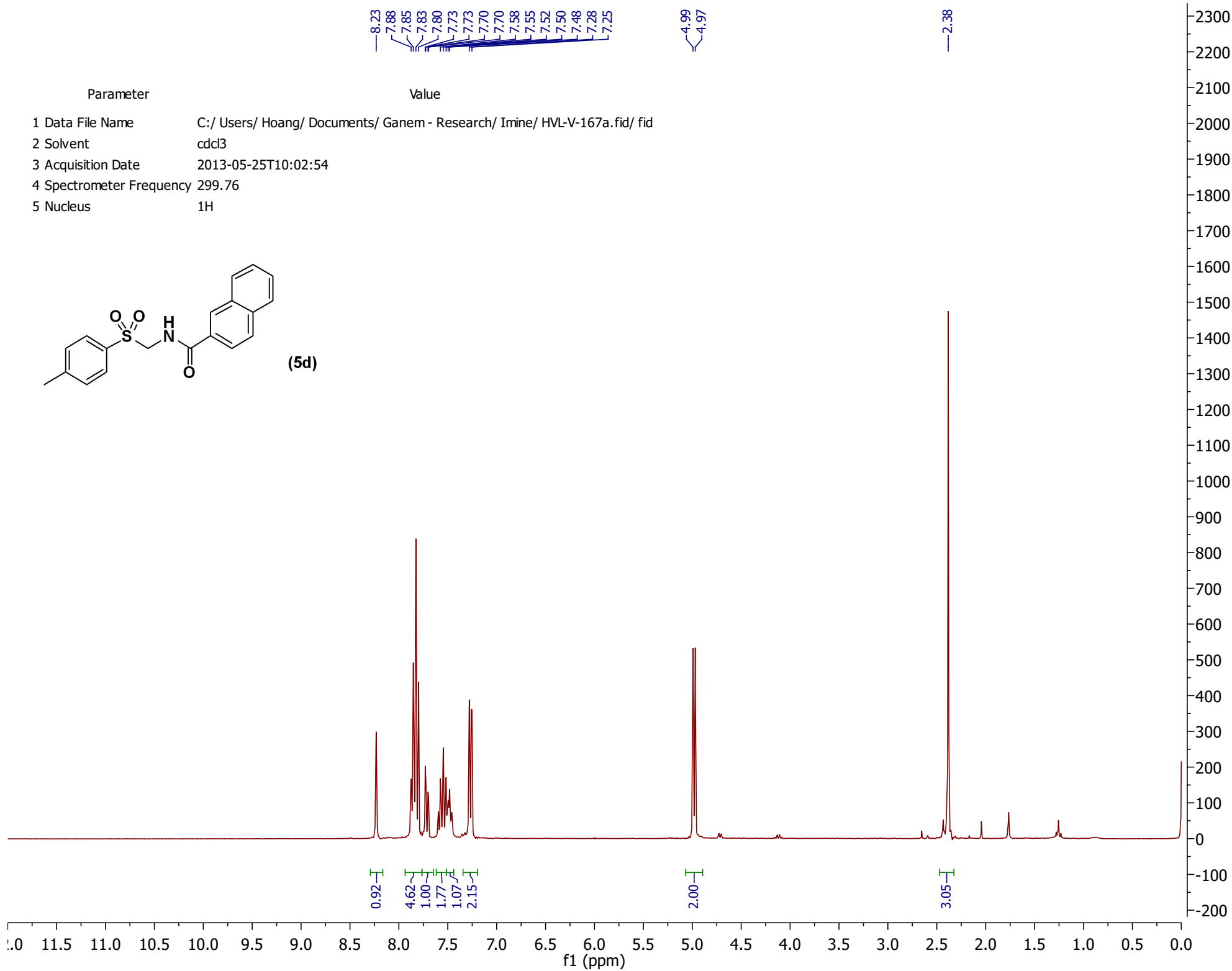
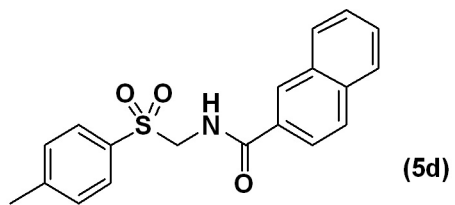


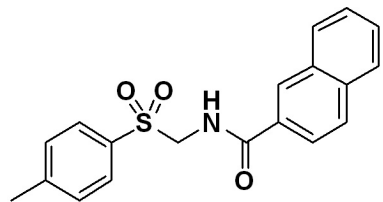
8.23
7.88
7.85
7.83
7.80
7.773
7.770
7.770
7.58
7.55
7.52
7.50
7.48
7.28
7.25

4.99
4.97

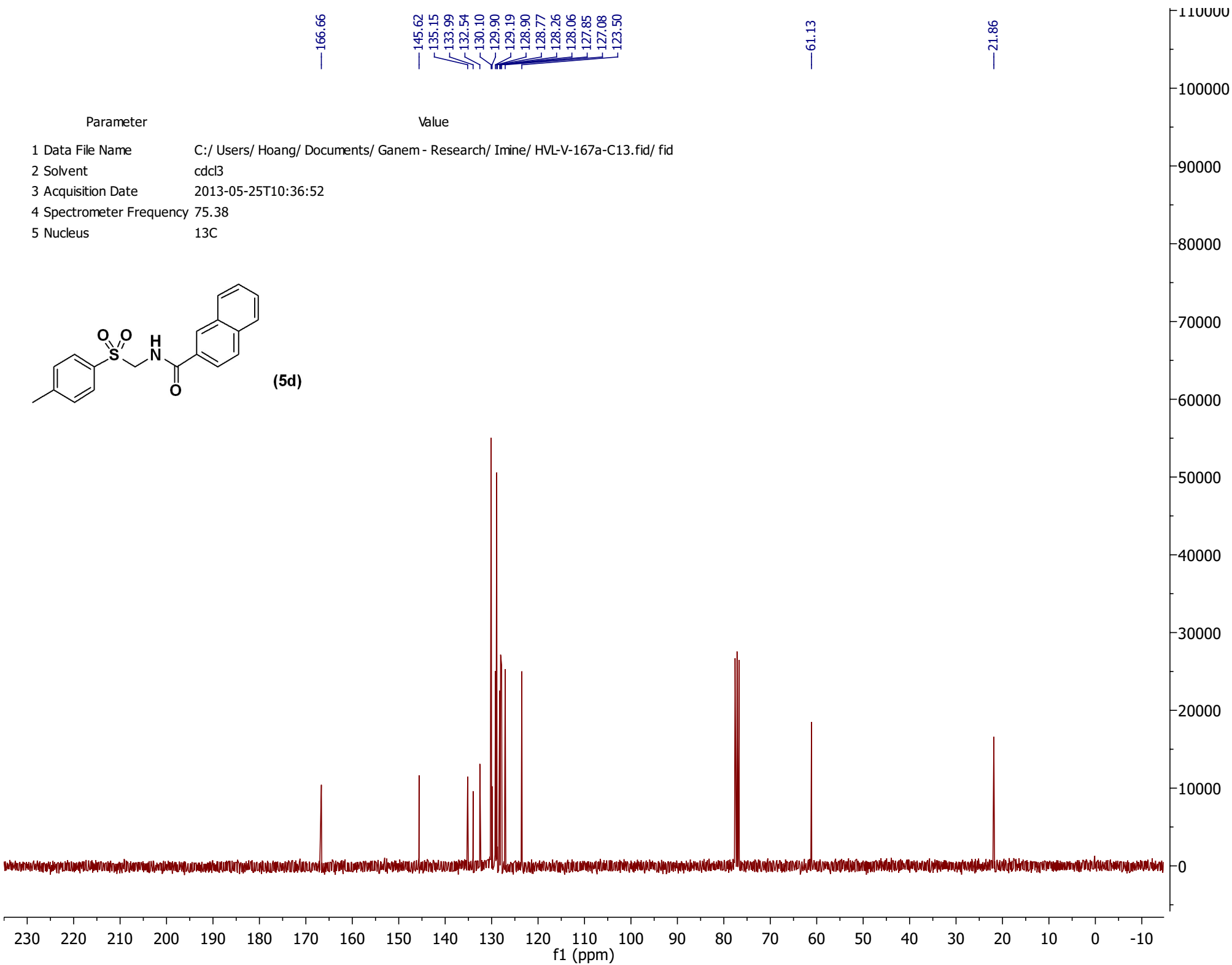
2.38

Parameter	Value
1 Data File Name	C:/ Users/ Hoang/ Documents/ Ganem - Research/ Imine/ HVL-V-167a.fid/ fid
2 Solvent	cdcl3
3 Acquisition Date	2013-05-25T10:02:54
4 Spectrometer Frequency	299.76
5 Nucleus	1H





Parameter	Value
1 Data File Name	C:/Users/Hoang/Documents/Ganem - Research/Imine/HVL-V-167a-C13.fid/ fid
2 Solvent	cdcl3
3 Acquisition Date	2013-05-25T10:36:52
4 Spectrometer Frequency	75.38
5 Nucleus	¹³ C

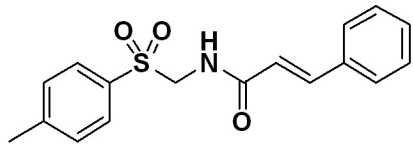


7.84
7.81
7.54
7.48
7.45
7.44
7.43
7.42
7.36
7.35
7.34
7.34
7.33
7.32
7.30
7.11
6.47
6.42

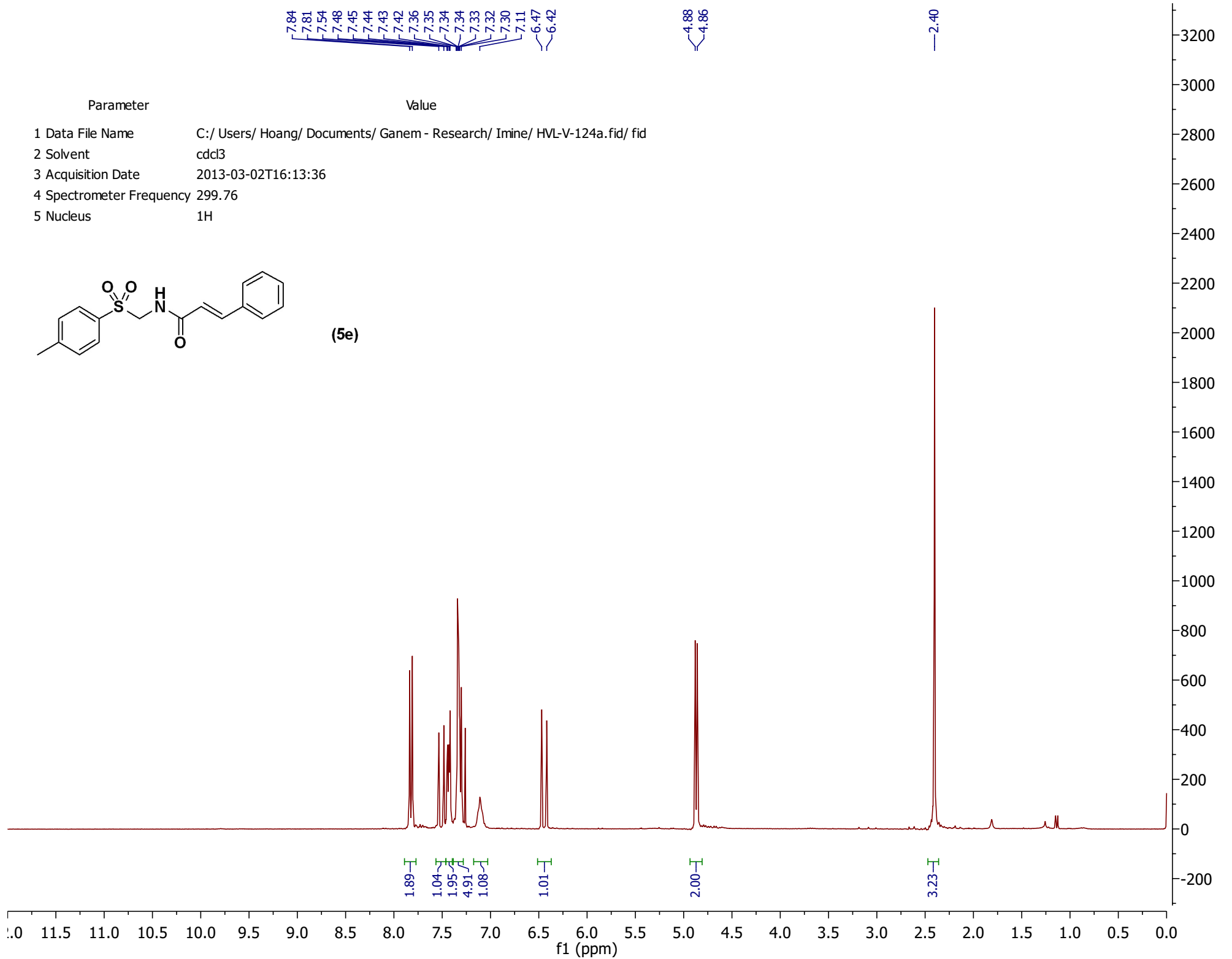
4.88
4.86

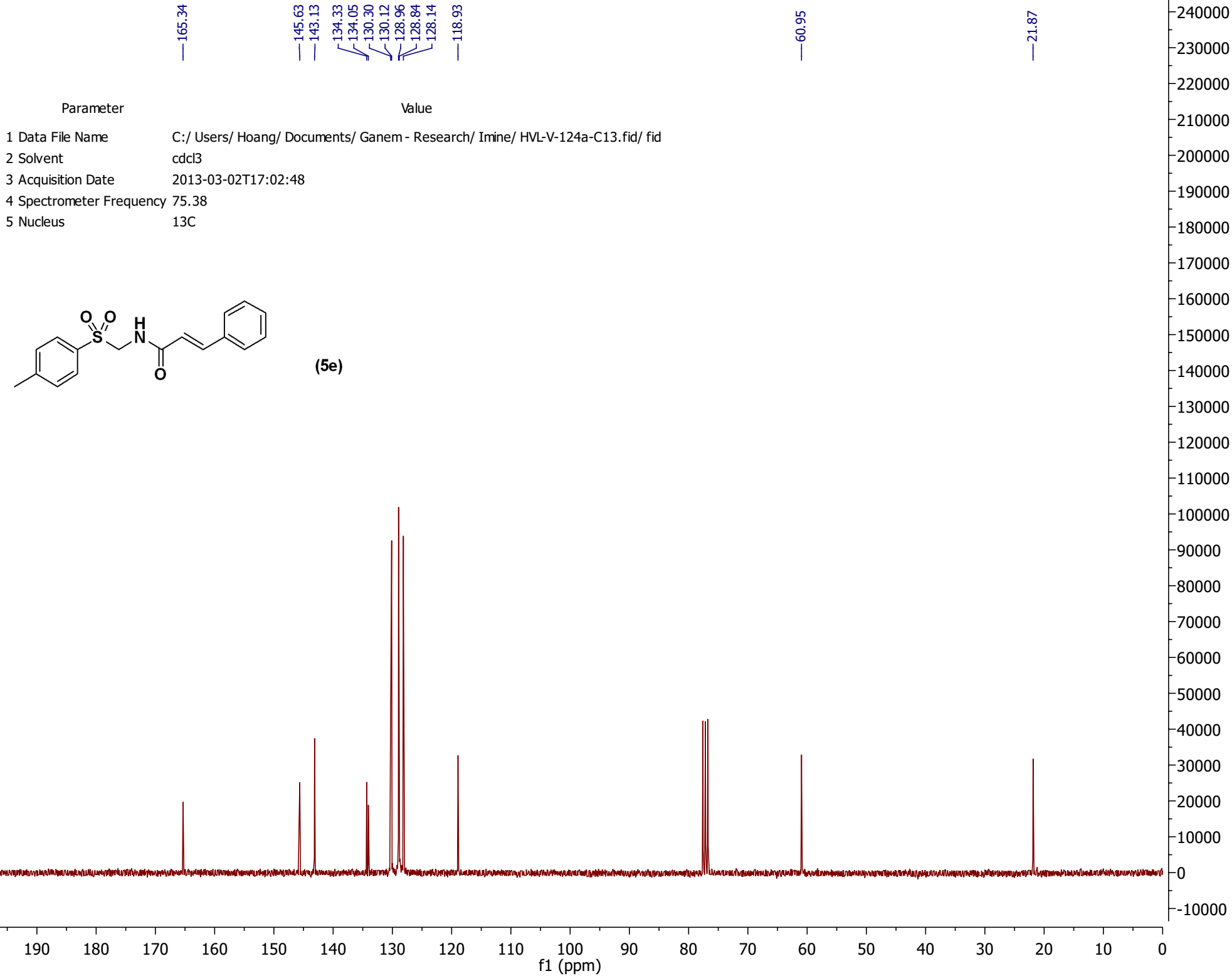
2.40

Parameter	Value
1 Data File Name	C:/Users/Hoang/Documents/Ganem - Research/Imine/HVL-V-124a.fid/fid
2 Solvent	cdcl3
3 Acquisition Date	2013-03-02T16:13:36
4 Spectrometer Frequency	299.76
5 Nucleus	1H

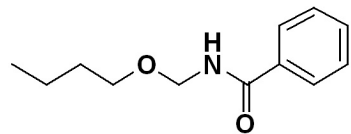


(5e)



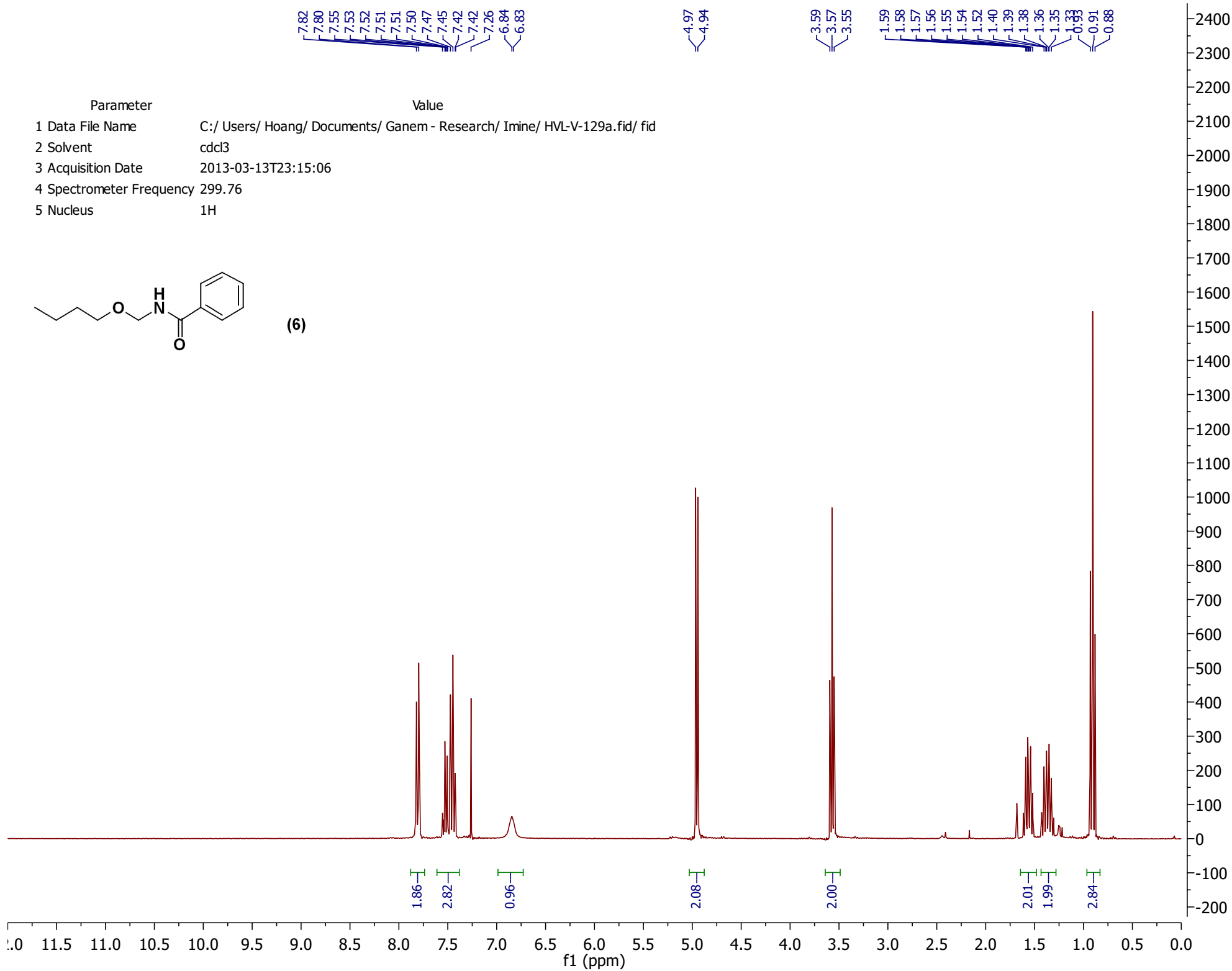


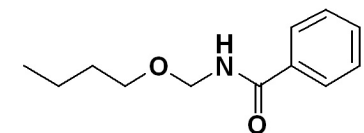
Parameter	Value
1 Data File Name	C:/Users/Hoang/Documents/Ganem - Research/Imine/HVL-V-124a-C13.fid/fid
2 Solvent	cdcl3
3 Acquisition Date	2013-03-02T17:02:48
4 Spectrometer Frequency	75.38
5 Nucleus	¹³ C



(6)

Parameter	Value
1 Data File Name	C:/ Users/ Hoang/ Documents/ Ganem - Research/ Imine/ HVL-V-129a.fid/ fid
2 Solvent	cdcl3
3 Acquisition Date	2013-03-13T23:15:06
4 Spectrometer Frequency	299.76
5 Nucleus	1H





(6)

Parameter	Value
1 Data File Name	C:/Users/Hoang/Documents/Ganem - Research/Imine/HVL-V-129a-C13.fid/ fid
2 Solvent	cdcl3
3 Acquisition Date	2013-03-13T23:49:19
4 Spectrometer Frequency	75.38
5 Nucleus	13C

