Supporting Information for

Direct Access to Anthranilic Acid Derivatives via CO_2 Incorporation Reaction Using Arynes

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Contents

General remarks.

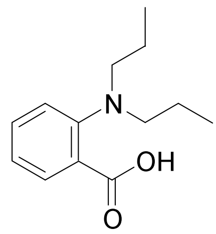
All manipulations of oxygen- and moisture-sensitive materials were conducted with a standard Schlenk technique under a purified argon atmosphere. Nuclear magnetic resonance spectra were taken on a JEOL EX-270 (^{1}H, 270 MHz; ^{13}C, 67.8 MHz) spectrometer or a JEOL Lambda-400 (^{1}H, 400 MHz; ^{13}C, 99.5 MHz) spectrometer using residual chloroform (^{1}H, $\delta = 7.26$) or $CDCl_3$ (^{13}C, $\delta = 77.0$) as an internal standard. ^{1}H NMR data are reported as follows: chemical shift, multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, quint = quintet, sept = septet, br = broad, m = multiplet), coupling constants (Hz), integration. High-resolution mass spectra were obtained with a JEOL JMS-SX102A spectrometer. Melting points were measured with Yanaco Micro Melting Point apparatus and uncorrected. Column chromatography was carried out using Merck Kieselgel 60. Unless otherwise noted, commercially available reagents were used without purification. 18-Crown-6 was recrystallized from distilled MeCN. KF (spray-dried) was vacuum dried at 100 °C for 12 h. THF was distilled from sodium/benzophenone ketyl. MeCN was distilled from phosphorus pentoxide.

Aryne precursors.

2-(Trimethylsilyl)phenyl triflate (**1a**),[1] 3-(trimethylsilyl)-5,6,7,8-tetrahydro-2-naphthyl triflate (**1b**),[2] 6-(trimethylsilyl)-5-indanyl triflate (**1c**),[3] 4,5-dimethyl-2-(trimethylsilyl)phenyl triflate (**1d**),[3] 3-(trimethylsilyl)-2-naphthyl triflate (**1e**),[2] 3,6-dimethyl-2-(trimethylsilyl)phenyl triflate (**1f**),[2] 4-fluoro-2-(trimethylsilyl)phenyl triflate (**1g**),[2] 4-methoxy-2-(trimethylsilyl)phenyl triflate (**1h**)[4] and 4-methyl-2-(trimethylsilyl)phenyl triflate (**1i**)[5] were prepared according to literature procedures.

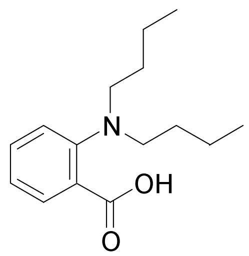
Three-component coupling of arynes, amines and CO_2: a general procedure.
A THF solution (1 mL) of an amine (0.165 mmol), 18-Crown-6 (0.079 g, 0.30 mmol) and KF (0.017 g, 0.30 mmol) was degassed through two freeze-thaw cycles, and the reaction flask was filled with CO_2 by connecting to a balloon (1 L). To this solution was added an aryne precursor (0.15 mmol), and the resulting mixture was stirred at 0 °C for the period as specified in Table 1 and Scheme 1. The mixture was diluted with ethyl acetate, filtered through a Celite plug, and concentrated. Silica-gel column chromatography (ethyl acetate as an eluent) gave the corresponding product.
In cases that an anthranilic acid was difficult to be isolated (Table 1, entries 3–5, 7, 9, 10, 12 or 13), a crude product was treated with 2 M solution of (trimethylsilyl)diazomethane in hexane (0.083 mL, 0.165 mmol), methanol (0.15 mL) and dichloromethane (3 mL) at room temperature for 12 h before the resulting mixture was quenched with acetic acid.[6] Evaporation of the solvent followed by silica-gel column chromatography (ethyl acetate as an eluent) gave the respective methyl ester.

***N*,*N*-Di-*n*-propylanthranilic acid (3aa)**



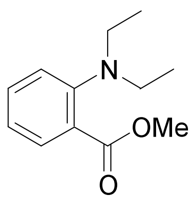
Isolated in 84% yield as a white solid: mp 107–115 °C; ^{1}H NMR ($CDCl_3$) δ 0.85 (d, J = 7.4 Hz, 6 H), 1.15–1.66 (m, 4 H), 3.00 (brs, 4 H), 7.34–7.43 (m, 2 H), 7.59 (td, J =7.7, 1.7 Hz, 1 H), 7.36 (dd, J = 7.6, 1.6 Hz, 1 H); ^{13}C NMR ($CDCl_3$) δ 11.4, 19.8, 58.9, 122.2, 127.4, 127.6, 131.7, 133.7 ,148.3, 167.8; HRMS Calcd for $C_{13}H_{19}NO_2$: M^+, 221.1416. Found: *m*/*z* 221.1412.

***N*,*N*-Di-*n*-butylanthranilic acid (3ab)**



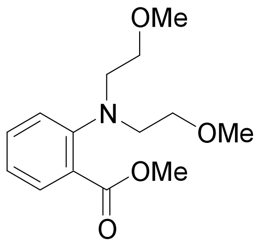
Isolated in 74% yield as a pale yellow solid: mp 54–58 °C; ^{1}H NMR ($CDCl_3$) δ 0.82 (d, J = 7.1 Hz, 6 H), 1.23 (brs, 6 H), 1.55 (brs, 2 H), 2.82-3.30 (m, 4 H), 7.30-7.48 (m, 2 H), 7.60 (td, J = 7.7, 1.7 Hz, 1 H), 8.31 (dd, J = 7.8, 1.6 Hz, 1 H); ^{13}C NMR ($CDCl_3$) δ 13.7, 20.3, 28.4, 57.1, 122.1, 127.4, 127.7, 131.7, 133.7, 148.0, 167.9; HRMS Calcd for $C_{15}H_{23}NO_2$: M^+, 249.1728. Found: m/z 249.1719.

***N*,*N*-Diethylanthranilic acid methyl ester (3ac)**



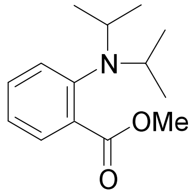
Isolated in 54% yield as a pale yellow oil: ^{1}H NMR ($CDCl_3$) δ 1.05 (t, J = 7.1 Hz, 3 H), 3.14 (q, J = 7.1 Hz, 4 H), 3.87 (s, 3 H), 6.92 (td, J = 7.5, 1.0 Hz, 1 H), 7.05 (d, J = 8.2 Hz, 1 H), 7.34 (ddd, J = 8.4, 7.2, 1.7 Hz, 1 H), 7.56 (dd, J = 7.5, 1.7 Hz, 1 H); ^{13}C NMR ($CDCl_3$) δ 12.3, 46.9, 51.97, 51.99, 120.5, 120.9, 126.1, 130.5, 131.5, 150.3, 169.3; HRMS Calcd for $C_{12}H_{17}NO_2$: M^+, 207.1253. Found: m/z 207.1252.

***N*,*N*-Bis(2-methoxyethyl)anthranilic acid methyl ester (3ad)**



Isolated in 64% yield as a pale yellow oil: ^{1}H NMR ($CDCl_3$) δ 3.28 (s, 3 H), 3.63 (t, J = 6.0 Hz, 4 H), 3.46 (t, J = 6.0 Hz, 4 H), 3.87 (s, 3 H), 6.97 (td, J = 7.4, 0.7 Hz, 1 H), 7.18 (d, J = 7.6 Hz, 1 H), 7.36 (td, J = 7.8, 1.8 Hz, 1 H), 7.58 (dd, J = 7.6, 1.8 Hz, 1 H),; ^{13}C NMR ($CDCl_3$) δ 52.02, 52.03, 53.5, 58.7, 70.7, 121.4, 122.4, 126.2, 130.63. 130.68, 131.8, 150.3, 168.8; HRMS Calcd for $C_{14}H_{21}NO_4$: M^+, 267.1471. Found: m/z 267.1462.

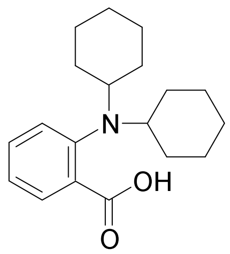
***N*,*N*-Diisopropylanthranilic acid methyl ester (3ae)**



Isolated in 79% yield as a pale yellow oil: ^{1}H NMR ($CDCl_3$) δ 0.98 (d, J = 6.5 Hz, 12 H), 3.48 (sept, J = 6.5 Hz, 2 H), 3.84 (s, 3 H), 7.17 (td, J = 7.3, 1.2 Hz, 1 H), 7.27 (dd, J = 8.2, 1.0 Hz, 1 H), 7.36 (ddd, J = 8.0, 7.2, 1.7 Hz, 1 H), 7.45 (dd, J = 7.5, 1.7 Hz, 1 H); ^{13}C

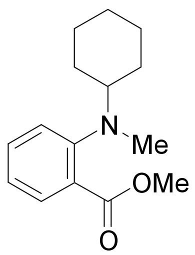
NMR ($CDCl_3$) δ 21.4, 29.7, 49.7, 51.7, 124.8, 128.2, 129.9, 130.0, 146.6, 170.0; HRMS Calcd for $C_{14}H_{21}NO_2$: M^+, 235.1572. Found: *m*/*z* 235.1580.

***N*,*N*-Dicyclohexylanthranilic acid (3af)**



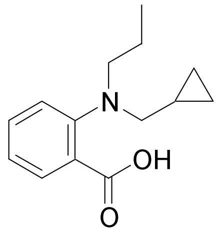
Isolated in 68% yield as a white solid: mp 155–157 °C; ^{1}H NMR ($CDCl_3$) δ 0.92–1.38 (m, 10 H), 1.60 (d, *J* = 12.8 Hz, 2 H), 1.67–1.85 (m, 6 H), 2.13 (d, *J* = 12.2 Hz, 2 H), 3.28–3.42 (m, 2 H), 7.25–7.32 (m, 1 H), 2.13 (td, *J* = 7.2, 1.3 Hz, 1 H), 7.52 (td, *J* = 7.9, 1.6 Hz, 1 H), 8.35 (dd, *J* = 7.9, 1.6 Hz, 1 H); ^{13}C NMR ($CDCl_3$) δ 25.30, 25.38, 25.39, 28.1, 30.2, 58.4, 125.1, 127.8, 129.8, 131.6, 132.2, 143.0, 168.5; HRMS Calcd for $C_{19}H_{27}NO_2$: M^+, 301.2042. Found: *m*/*z* 301.2040.

***N*-Cyclohexyl-*N*-methylanthranilic acid methyl ester (3ag)**



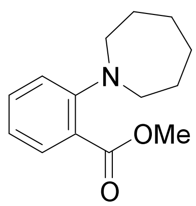
Isolated in 72% yield as a pale yellow oil: ^{1}H NMR ($CDCl_3$) δ 1.10 (tt, *J* = 12.7, 3.1 Hz, 1 H), 1.15–1.32 (m, 2 H), 1.38–1.54 (m, 2 H), 1.55–1.71 (m, 1 H), 1.72–1.87 (m, 4 H), 2.72 (s, 3 H), 3.07 (tt, *J* = 11.6, 3.1 Hz, 1 H), 3.88 (s, 3 H), 6.84 (td, *J* = 7.5, 1.0 Hz, 1 H), 6.97 (d, *J* = 8.2 Hz, 1 H), 7.32 (ddd, *J* = 8.7, 7.2, 1.7 Hz, 1 H), 7.58 (dd, *J* = 7.7, 1.9 Hz, 1 H),; ^{13}C NMR ($CDCl_3$) δ 25.9, 26.1, 29.5, 33.5, 51.96, 51.99, 64.1, 118.83, 118.86, 122.7, 131.1, 131.7, 151.8, 169.6; HRMS Calcd for $C_{15}H_{21}NO_2$: M^+, 247.1572. Found: *m*/*z* 247.1567.

***N*-(Cyclopropylmethyl)-*N*-propylanthranilic acid (3ah)**



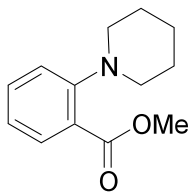
Isolated in 77% yield as a white solid: mp 107–111 °C; ^{1}H NMR ($CDCl_3$; 50 °C) δ -0.06–0.18 (m, 2 H), 0.39–0.55 (m, 2 H), 0.75–0.95 (m, 4 H), 1.31–1.75 (m, 2 H), 2.78–3.20 (m, 4 H), 7.34–7.44 (m, 2 H), 7.60 (td, J = 8.0, 1.7 Hz, 1 H), 8.31 (dd, J = 7.6, 1.4 Hz, 1 H); ^{13}C NMR ($CDCl_3$) δ 4.2, 8.0, 11.5, 19.8, 58.0, 62.4, 122.5, 127.5, 131.7, 133.5, 148.6, 167.9; HRMS Calcd for $C_{14}H_{19}NO_2$: M^+, 233.1416. Found: m/z 233.1413.

2-(Azepan-1-yl)benzoic acid methyl ester (3ai)



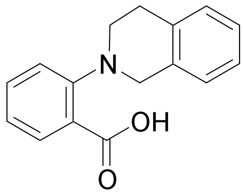
Isolated in 62% yield as a pale yellow oil: ^{1}H NMR ($CDCl_3$) δ 1.53–1.65 (m, 4 H), 1.71–1.83 (m, 4 H), 3.32 (t, J = 5.6 Hz, 4 H), 3.87 (s, 3 H), 6.74 (td, J = 1.0, 7.4 Hz, 1 H), 6.95 (dd, J = 8.4, 0.7 Hz, 1 H), 7.29 (td, J = 7.7, 1.7 Hz, 1 H), 7.52 (dd, J = 7.7, 1.7 Hz, 1 H),; ^{13}C NMR ($CDCl_3$) δ 28.1, 28.4, 30.0, 52.0, 52.7, 116.8, 116.9, 119.6, 131.1, 131.6, 151.2, 170.0; HRMS Calcd for $C_{14}H_{19}NO_2$: M^+, 233.1416. Found: m/z 233.1414.

2-(Piperidin-1-yl)benzoic acid methyl ester (3aj)



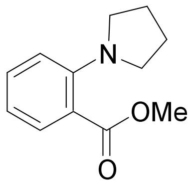
Isolated in 54% yield as a yellow oil: ^{1}H NMR ($CDCl_3$) δ 1.52–1.62 (m, 2 H), 1.67–1.78 (m, 4 H), 2.99 (d, J = 5.3 Hz, 4 H), 6.93 (td, J = 7.5, 1.0 Hz, 1 H), 7.01 (dd, J = 8.2, 1.0 Hz, 1 H), 7.37 (ddd, J = 8.2, 7.2, 1.7 Hz, 1 H), 7.68 (dd, J = 7.7, 1.7 Hz, 1 H),; ^{13}C NMR ($CDCl_3$) δ 24.2, 26.2, 52.0, 53.8, 118.6, 120.6, 123.9, 131.4, 132.4, 153.2, 168.9; HRMS Calcd for $C_{12}H_{15}NO_2$: M^+, 205.1103. Found: m/z 205.1108.

2-(3,4-Dihydro-(1*H*)-isoquinolin-2-yl)benzoic acid (3ak)



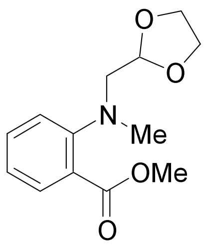
Isolated in 38% yield as a white solid: mp 176–179 °C; ^{1}H NMR ($CDCl_3$) δ 2.90–3.60 (m, 4 H), 4.24 (brs, 2 H), 7.03–7.13 (m, 1 H), 7.18–7.35 (m, 3 H), 7.43–7.58 (m, 2 H), 7.64 (ddd, J = 8.2, 7.5, 1.7 Hz, 1 H), 8.34 (dd, J = 7.7, 1.7 Hz, 1 H); ^{13}C NMR ($CDCl_3$) δ 29.0, 51.5, 55.8, 122.5, 125.2, 126.4, 126.5, 127.3, 127.7, 129.0, 132.0, 132.3, 132.4, 134.0, 150.7, 167.0; HRMS Calcd for $C_{16}H_{15}NO_2$: M^+, 253.1103. Found: *m*/*z* 253.1103.

2-(Pyrrolidin-1-yl)benzoic acid methyl ester (3al)



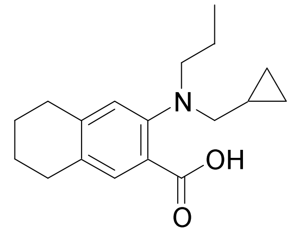
Isolated in 7% yield as a pale brown oil: ^{1}H NMR ($CDCl_3$) δ 1.89–1.99 (m, 4 H), 3.18–3.30 (m, 4 H), 3.88 (s, 3 H), 6.71 (td, J = 7.4, 1.0 Hz, 1 H), 6.78 (d, J = 8.5 Hz, 1 H), 7.29 (ddd, J = 8.7, 7.0, 1.7 Hz, 1 H), 7.57 (dd, J = 7.7, 1.7 Hz, 1 H); ^{13}C NMR ($CDCl_3$) δ 25.9, 50.8, 52.0, 113.9, 115.6, 117.1, 131.1, 131.8, 148.0, 169.6; HRMS Calcd for $C_{13}H_{17}NO_2$: M^+, 219.1259. Found: *m*/*z* 219.1261.

***N*-[(1,3-Dioxolan-2-yl)methyl]-*N*-methylanthranilic acid methyl ester (3am)**



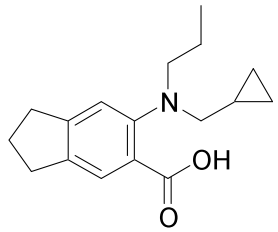
Isolated in 29% yield as a colorless oil: ^{1}H NMR ($CDCl_3$) δ 2.96 (s, 3 H), 3.30 (d, J = 4.1 Hz, 2 H), 3.82-3.88 (m, 2 H), 3.89 (s, 3 H), 3.93-3.98 (m, 2 H), 5.09 (t, J = 4.1 Hz, 1 H), 6.88 (t, J = 7.5 Hz, 1 H), 7.06 (d, J = 8.2 Hz, 1 H), 7.35 (td, J = 7.8, 1.7 Hz, 1 H), 7.62 (dd, J = 7.7, 1.7 Hz, 1 H); ^{13}C NMR ($CDCl_3$) δ 29.7, 41.2, 52.1, 59.2, 64.8, 103.7, 118.7, 119.6, 122.1, 131.3, 132.1, 151.8, 168.9; HRMS Calcd for $C_{13}H_{17}NO_4$: M^+, 251.1158. Found: *m*/*z* 251.1155.

***N*-(Cyclopropylmethyl)-*N*-propyl-4,5-tetramethyleneanthranilic acid (3bh)**



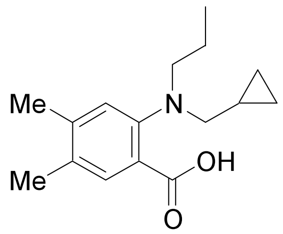
Isolated in 90% yield as a white solid: mp 142–147 °C; ^{1}H NMR ($CDCl_3$) δ -0.02–0.09 (m, 1 H), 0.16–0.27 (m, 1 H), 0.36–0.60 (m, 2 H), 0.73–0.92 (m, 4 H), 1.18–1.41 (m, 1 H), 1.48–1.68 (m, 1 H), 1.72–1.85 (m, 4 H), 2.72–2.97 (m, 7 H), 3.08–3.21 (m, 1 H), 7.01 (s, 1 H), 7.97 (s, 1 H); ^{13}C NMR ($CDCl_3$) δ 3.9, 4.6, 8.1, 11.5, 19.8, 22.5, 22.7, 28.8, 29.6, 58.1, 62.5, 122.5, 124.3, 132.1, 136.8, 143.5, 145.4, 168.5; HRMS Calcd for $C_{18}H_{25}NO_2$: M^+, 287.1885. Found: *m/z* 287.1881.

***N*-(Cyclopropylmethyl)-*N*-propyl-4,5-trimethyleneanthranilic acid (3ch)**



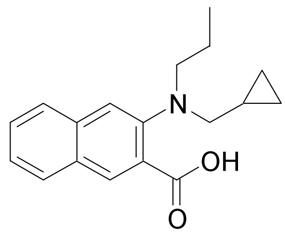
Isolated in 55% yield as a white solid: mp 119–123 °C; ^{1}H NMR ($CDCl_3$) δ -0.02–0.11 (m, 1 H), 0.15–0.27 (m, 1 H), 0.37–0.62 (m, 2 H), 0.72–1.01 (m, 4 H), 1.19–1.42 (m, 1 H), 1.50–1.70 (m, 1 H), 2.11 (quint, J = 7.5 Hz, 2 H), 2.75–2.14 (m, 7 H), 3.15 (td, J = 5.1, 11.6 Hz, 1 H), 7.19 (s, 1 H), 8.31 (s, 1 H); ^{13}C NMR ($CDCl_3$) δ 3.9, 4.6, 8.0, 11.5, 19.7, 25.4, 32.2, 33.1, 58.1, 62.5, 117.9, 125.3, 127.0, 143.9, 146.8, 150.6, 168.5; HRMS Calcd for $C_{17}H_{23}NO_2$: M^+, 273.1729. Found: *m/z* 273.1735.

***N*-(Cyclopropylmethyl)-*N*-propyl-4,5-dimethylanthranilic acid (3dh)**



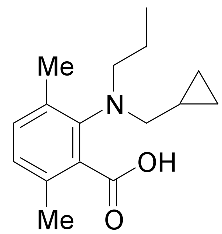
Isolated in 75% yield as a pale yellow solid: mp 117–121 °C; ^{1}H NMR ($CDCl_3$) δ -0.06–0.07 (m, 1 H), 0.14–0.25 (m, 1 H), 0.33–0.60 (m, 2 H), 0.73–0.92 (m, 4 H), 1.18–1.39 (m, 1 H), 1.48–1.68 (m, 1 H), 2.28 (s, 3 H), 2.30 (s, 3 H), 2.79–2.98 (m, 3 H), 3.08–3.21 (m, 1 H), 7.09 (s, 1 H), 8.04 (s, 1 H); ^{13}C NMR ($CDCl_3$) δ 3.9, 4.5, 8.1, 11.5, 19.3, 19.8, 20.2, 58.1, 62.4, 123.1, 124.7, 132.3, 136.3, 143.1, 145.9, 168.5; HRMS Calcd for $C_{16}H_{23}NO_2$: M^+, 261.1729. Found: *m*/*z* 261.1728.

3-[*N*-(Cyclopropylmethyl)-*N*-propylamino]-2-naphthoic acid (3eh)



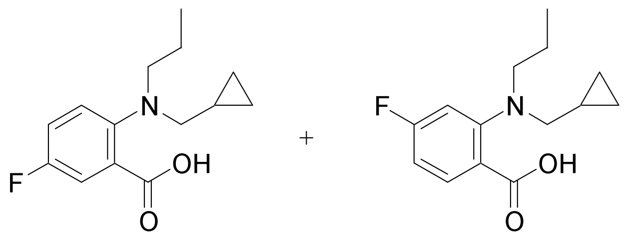
Isolated in 42% yield as a yellow solid: mp 142–146 °C; ^{1}H NMR ($CDCl_3$) δ -0.04–0.10 (m, 1 H), 0.15–0.30 (m, 1 H), 0.34–0.62 (m, 2 H), 0.76–0.99 (m, 4 H), 1.25–1.43 (m, 1 H), 1.55–1.78 (m, 1 H), 2.92–3.20 (m, 3 H), 3.20–3.39 (m, 1 H), 7.56 (ddd, *J* = 8.2, 7.0, 1.4 Hz, 1 H), 7.61 (ddd, *J* = 8.0, 6.8, 1.2 Hz, 1 H), 7.80 (s, 1 H), 7.83 (d, *J* = 7.7 Hz, 1 H), 7.99 (d, *J* = 8.0 Hz, 1 H), 8.91 (s, 1 H); ^{13}C NMR ($CDCl_3$) δ 4.1, 4.6, 8.1, 11.5, 19.8, 58.7, 63.0, 121.57, 121.59, 124.8, 127.1, 128.7, 129.5, 131.6, 133.4, 135.3, 145.0, 168.2; HRMS Calcd for $C_{18}H_{21}NO_2$: M^+, 283.1572. Found: *m*/*z* 283.1572.

***N*-(Cyclopropylmethyl)-*N*-propyl-3,6-dimethylanthranilic acid (3fh)**



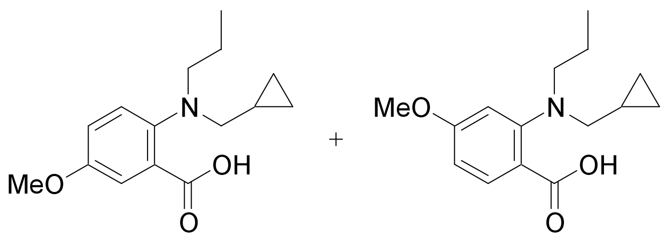
Isolated in 71% yield as a yellow oil: ^{1}H NMR ($CDCl_3$) δ 0.02–0.07 (m, 1 H), 0.21–0.27 (m, 1 H), 0.40–0.47 (m, 1 H), 0.53–0.61 (m, 1 H), 0.83–0.96 (m, 4 H), 1.32–1.46 (m, 1 H), 1.59–1.75 (m, 1 H), 2.38 (s, 3 H), 2.71 (s, 3 H), 3.06 (dd, *J* = 7.0, 2.8 Hz, 2 H), 3.11 (td, *J* = 12.1, 5.1 Hz, 1 H), 3.27 (td, *J* = 12.1, 5.1 Hz, 1 H), 7.13 (s, 2 H); ^{13}C NMR ($CDCl_3$) δ 3.8, 4.7, 8.5, 11.5, 19.7, 20.6, 24.4, 55.9, 59.6, 127.6, 132.08, 132.14, 134.8, 141.9, 145.0, 168.6; HRMS Calcd for $C_{16}H_{23}NO_2$: M^+, 261.1729. Found: *m*/*z* 261.1727.

A mixture of *N*-(cyclopropylmethyl)-*N*-propyl-5-fluoroanthranilic acid (3gh) and *N*-(cyclopropylmethyl)-*N*-propyl-4-fluoroanthranilic acid (3’gh)
(**3gh**:**3’gh** = 94:6)



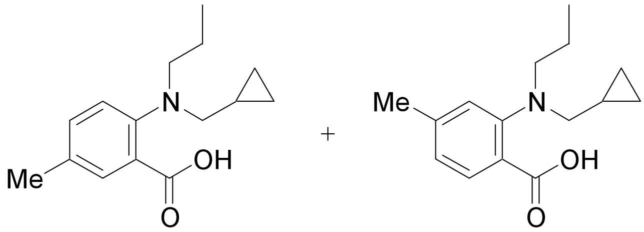
Isolated in 32% yield as a pale yellow solid: mp 106–109 °C; ^{1}H NMR ($CDCl_3$) δ -0.06–0.03 (m), 0.13–0.25 (m), 0.37–0.61 (m), 0.76–0.94 (m), 1.20–1.40 (m), 1.50–1.68 (m), 2.76–3.05 (m), 3.10–3.24 (m), 7.06–7.18 (m, *minor*), 7.23-7.32 (m), 7.37 (dd, J = 8.6, 4.6 Hz), 7.99 (dd, J = 8.9, 3.1 Hz, *major*), 8.33 (dd, J = 8.7, 6.5 Hz, *minor*); ^{13}C NMR ($CDCl_3$) δ 4.2, 4.4, 7.9 (*major*),8.1 (*minor*), 11.44, 11.47, 19.8 (*major*), 20.0 (*minor*), 58.21 (*minor*), 58.29 (*major*), 62.6, 110.3 ($J_{C\text{-}F}$ = 22.1 Hz, *minor*), 115.1 ($J_{C\text{-}F}$ = 21.3 Hz, *minor*), 119.1 ($J_{C\text{-}F}$ = 23.8 Hz, *major*), 120.6 ($J_{C\text{-}F}$ = 23.8 Hz, *major*), 124.4 ($J_{C\text{-}F}$ = 8.2 Hz, *major*), 129.8 ($J_{C\text{-}F}$ = 7.4 Hz), 134.0 ($J_{C\text{-}F}$ = 9.8 Hz), 144.1 ($J_{C\text{-}F}$ = 3.3 Hz), 151.3, 161.0 ($J_{C\text{-}F}$ = 249.4 Hz), 166.61, 166.62; HRMS Calcd for $C_{14}H_{18}FNO_2$: M^+, 251.1322. Found: *m*/*z* 251.1323.

A mixture of *N*-(cyclopropylmethyl)-*N*-propyl-5-methoxyanthranilic acid (3hh) and *N*-(cyclopropylmethyl)-*N*-propyl-4-methoxyanthranilic acid (3'hh)
(**3hh**:**3'hh** = 45:55)



Isolated in 64% yield as a brown oil: ^{1}H NMR ($CDCl_3$) δ -0.05–0.08 (m), 0.13–0.23 (m), 0.34–0.59 (m), 0.76–0.93 (m), 1.17–1.40 (m), 1.50–1.65 (m), 2.77–3.00 (m), 3.03–3.21 (m), 3.85 (s), 3.86 (s), 6.84 (d, J = 2.4 Hz, *major*), 6.90 (dd, J = 8.7, 2.4 Hz, *major*), 7.10 (dd, J = 8.9, 3.0 Hz, *minor*), 7.25 (d, J = 8.7 Hz, *minor*), 7.79 (d, J = 8.7 Hz, *minor*), 8.24 (d, J = 8.7 Hz, *major*); ^{13}C NMR ($CDCl_3$) δ 4.0, 4.4, 7.9, 8.1, 11.4, 11.5, 19.7, 19.9, 55.6, 55.7, 58.1, 58.2, 62.47, 62.49, 109.0, 112.1, 113.9, 120.1, 121.0, 123.3, 128.7, 133.4, 140.6, 150.6, 158.3, 163.6, 167.7, 167.9; HRMS Calcd for $C_{15}H_{23}NO_3$: M^+, 263.1521. Found: *m*/*z* 263.1521.

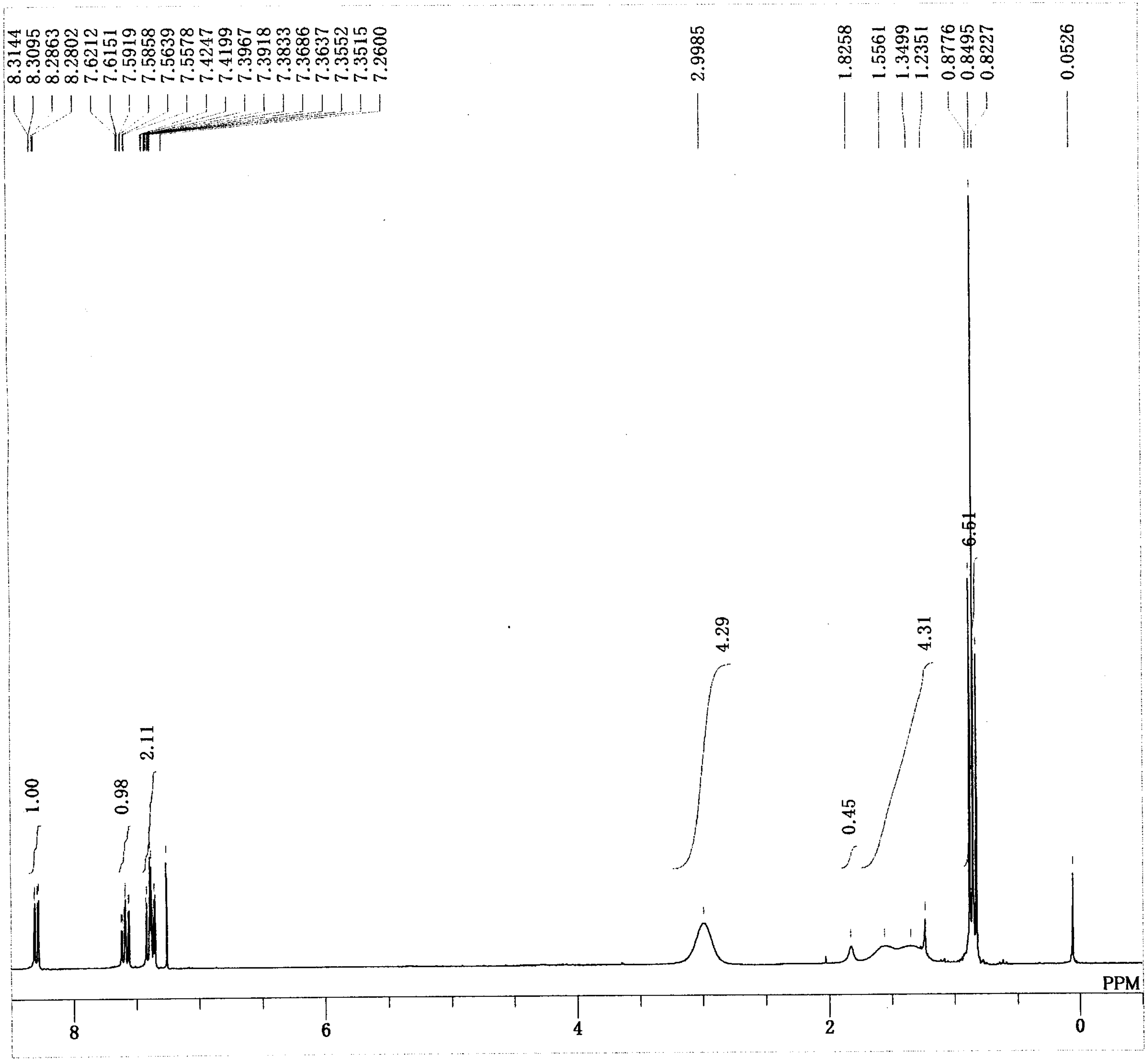
A mixture of *N*-(cyclopropylmethyl)-*N*-propyl-5-methylanthranilic acid (3ih) and *N*-(cyclopropylmethyl)-*N*-propyl-4-methylanthranilic acid (3'ih)
(**3ih**:**3'ih** = 42:58)



Isolated in 55% yield as a pale yellow solid: mp 57–61 °C; ^{1}H NMR ($CDCl_3$) δ -0.06–0.08 (m,), 0.11–0.25 (m), 0.34–0.62 (m), 0.76–0.95 (m), 1.20–1.40 (m), 1.50–1.68 (m), 2.38 (s, *minor*), 2.41 (s, *major*), 2.79-3.02 (m), 3.07-3.23 (m), 7.14 (s, *major*), 7.19 (dd, J = 8.0, 1.0 Hz, *major*), 7.24 (d, J = 8.2 Hz, *minor*), 7.37 (ddd, J = 7.5, 2.2, 0.7 Hz, *minor*), 8.11 (d, J = 1.7 Hz, *minor*), 8.17 (d, J = 8.0 Hz, *major*); ^{13}C NMR ($CDCl_3$) δ 4.0, 4.4, 8.0, 8.1, 11.47, 11.49, 19.78, 19.88, 20.9, 21.6, 58.0, 58.1, 62.40, 62.43, 122.2, 122.9, 124.7, 127.0, 128.4, 131.6, 131.9, 134.2, 137.6, 144.4, 145.8, 148.7, 168.0, 168.2; HRMS Calcd for $C_{15}H_{21}NO_2$: M^+, 247.1572. Found: *m/z* 247.1577.

References

1. Himeshima, Y.; Sonoda, T.; Kobayashi, H. *Chem. Lett.* **1983**, 1211–1214.
2. Yoshida, H.; Terayama, T.; Ohshita, J.; Kunai, A. *Chem. Commun.* **2004**, 1980–1981.
3. Yoshida, H.; Sugiura, S.; Kunai, A. *Org. Lett.* **2002**, *4*, 2767–2769.
4. Yoshida, H.; Ikadai, J.; Shudo, M.; Ohshita, J.; Kunai, A. *J. Am. Chem. Soc.* **2003**, *125*, 6638–6639.
5. Yoshikawa, E.; Radhakrishnan, K. V.; Yamamoto, Y. *J. Am. Chem. Soc.* **2000**, *122*, 7280–7286.
6. Yamagishi, T.; Okumura, Y.; Nukui, S.; Nakao, K. (PFIZER INC., PFIZER JAPAN INC.) WO 2005021508, **2005**.



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PD	3.9670 sec
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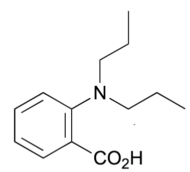
RGAIN	19



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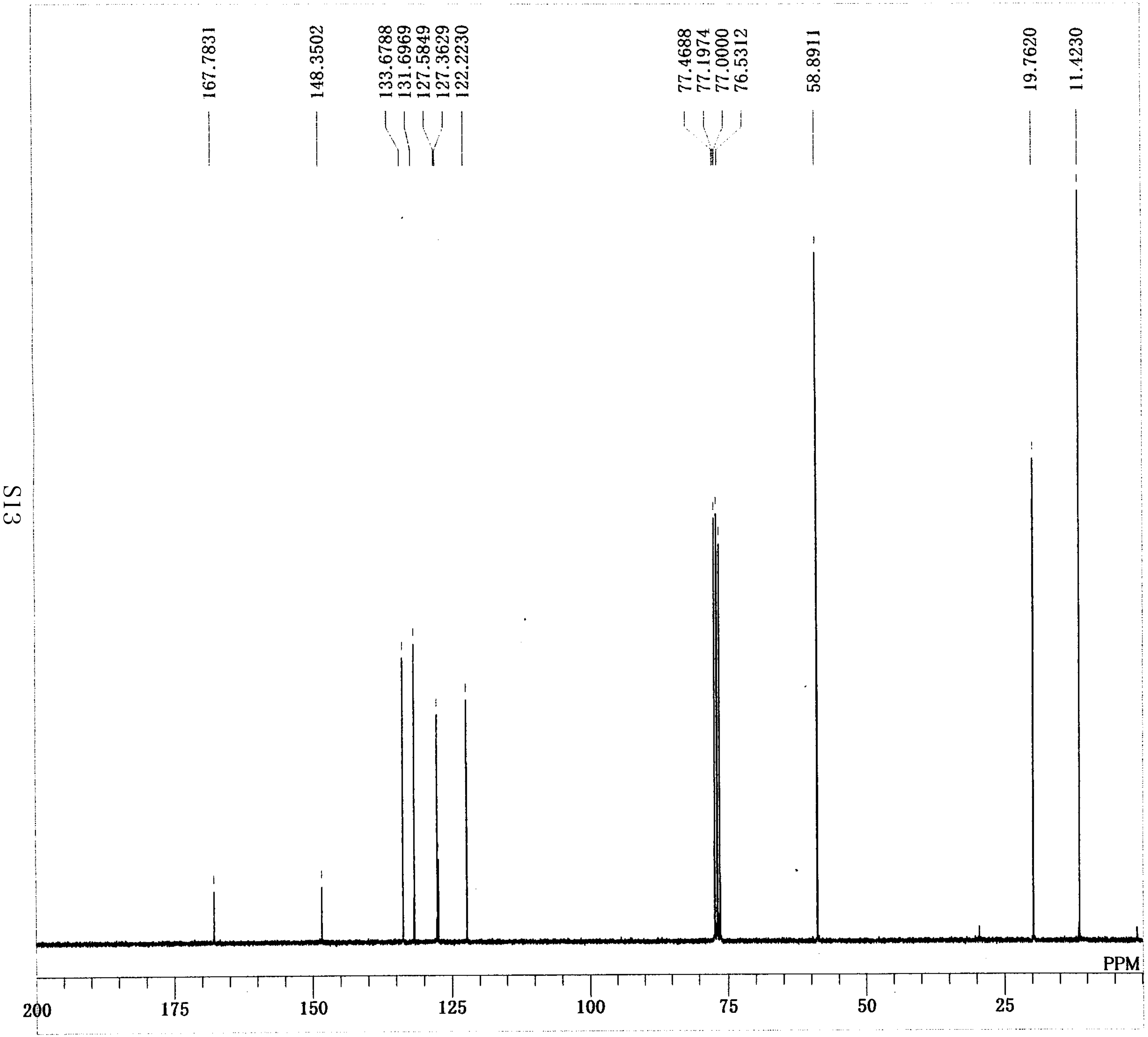

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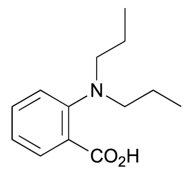
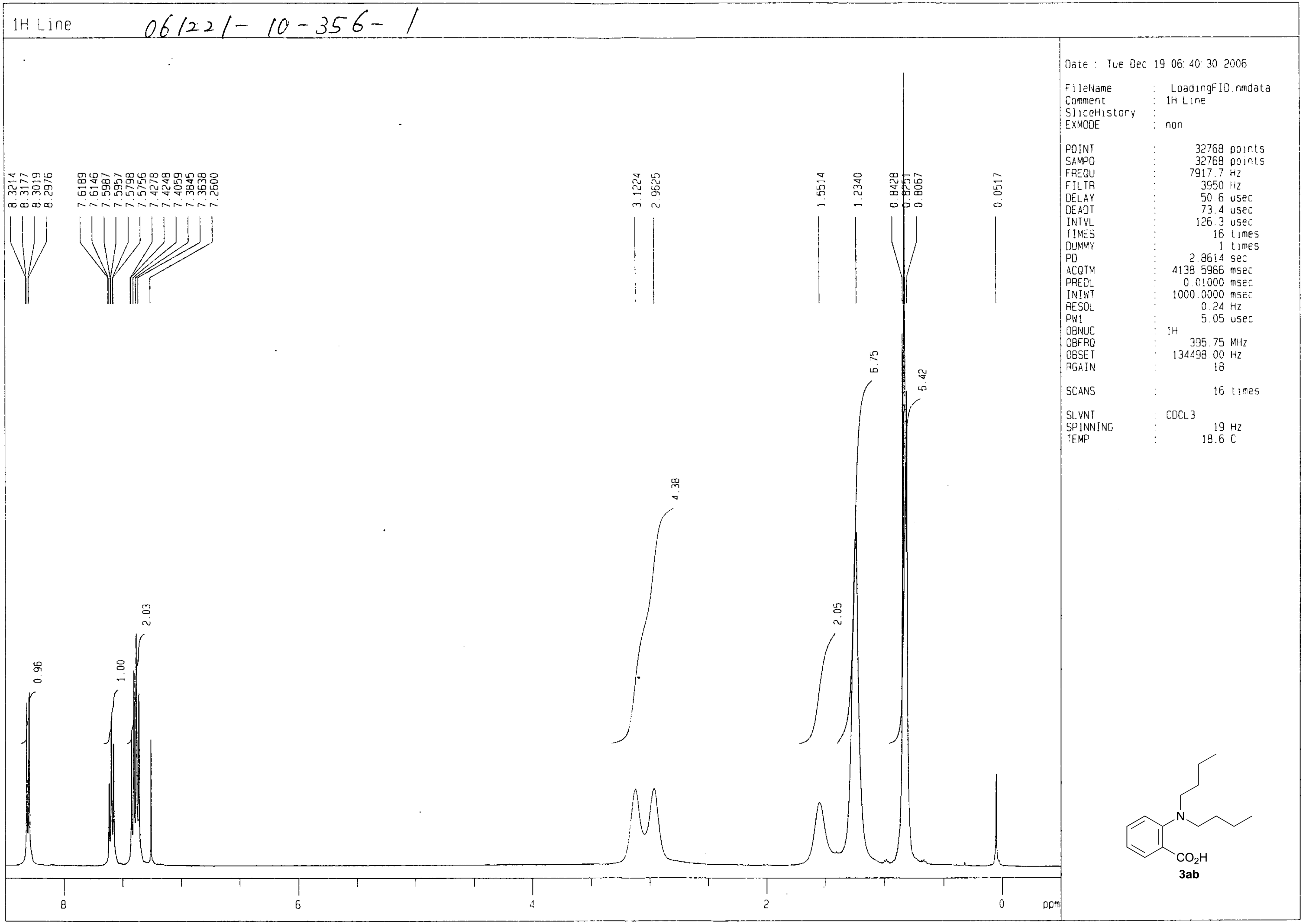


3aa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DUMMY : 1 times
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TEMP : 18.6 C
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7.4248
7.4059
7.3845
7.3638
7.2600
3.1224
2.9625
1.5514
1.2340
0.8428
0.8251
0.8067
0.0517
0.96
1.00
2.03
4.38
2.05
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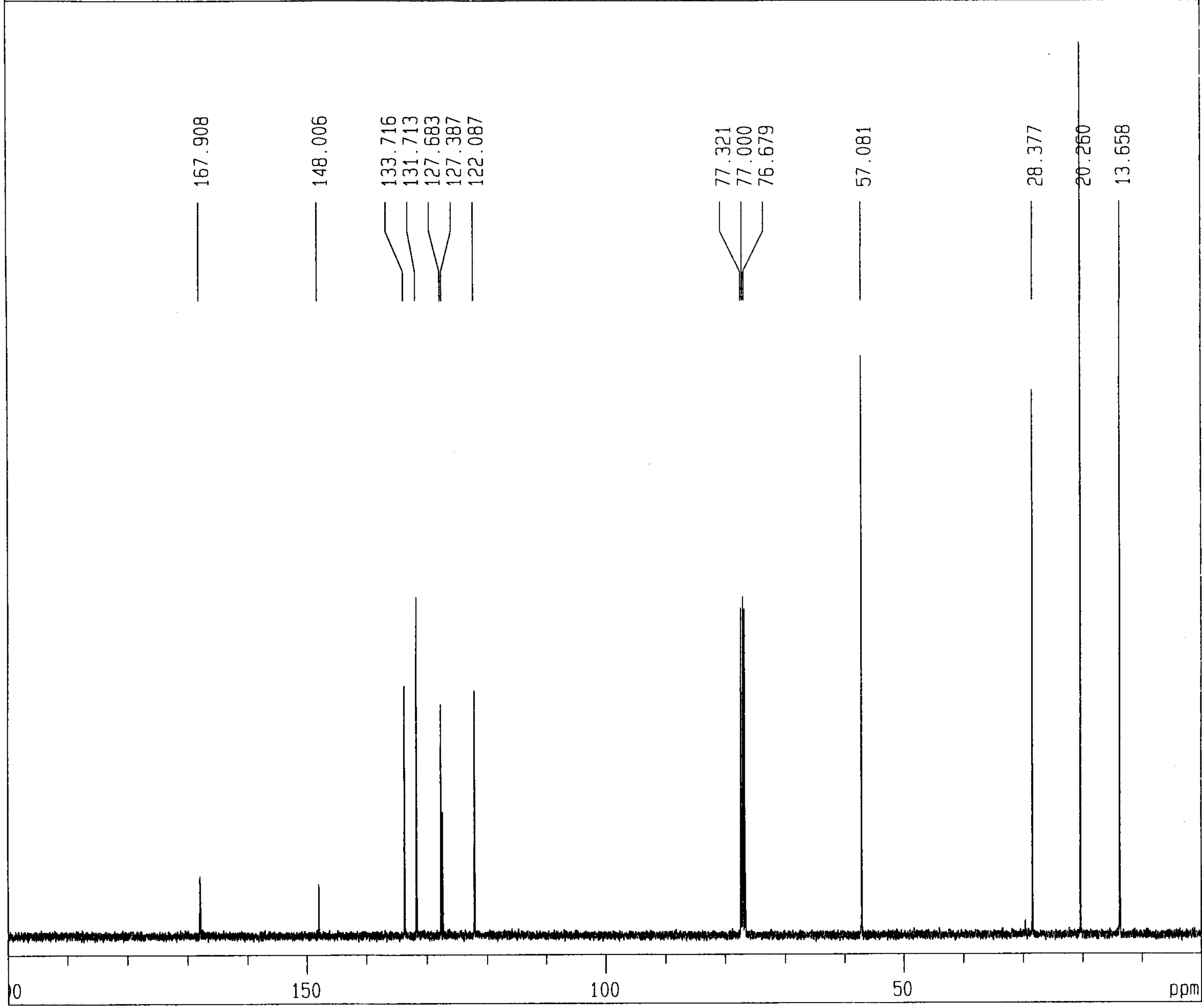
6.42
8
6
4
2
0
ppm
N
CO_2H
3ab

1H Line 06/22 3-10-356-1-^{13}C



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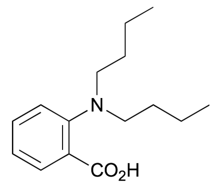
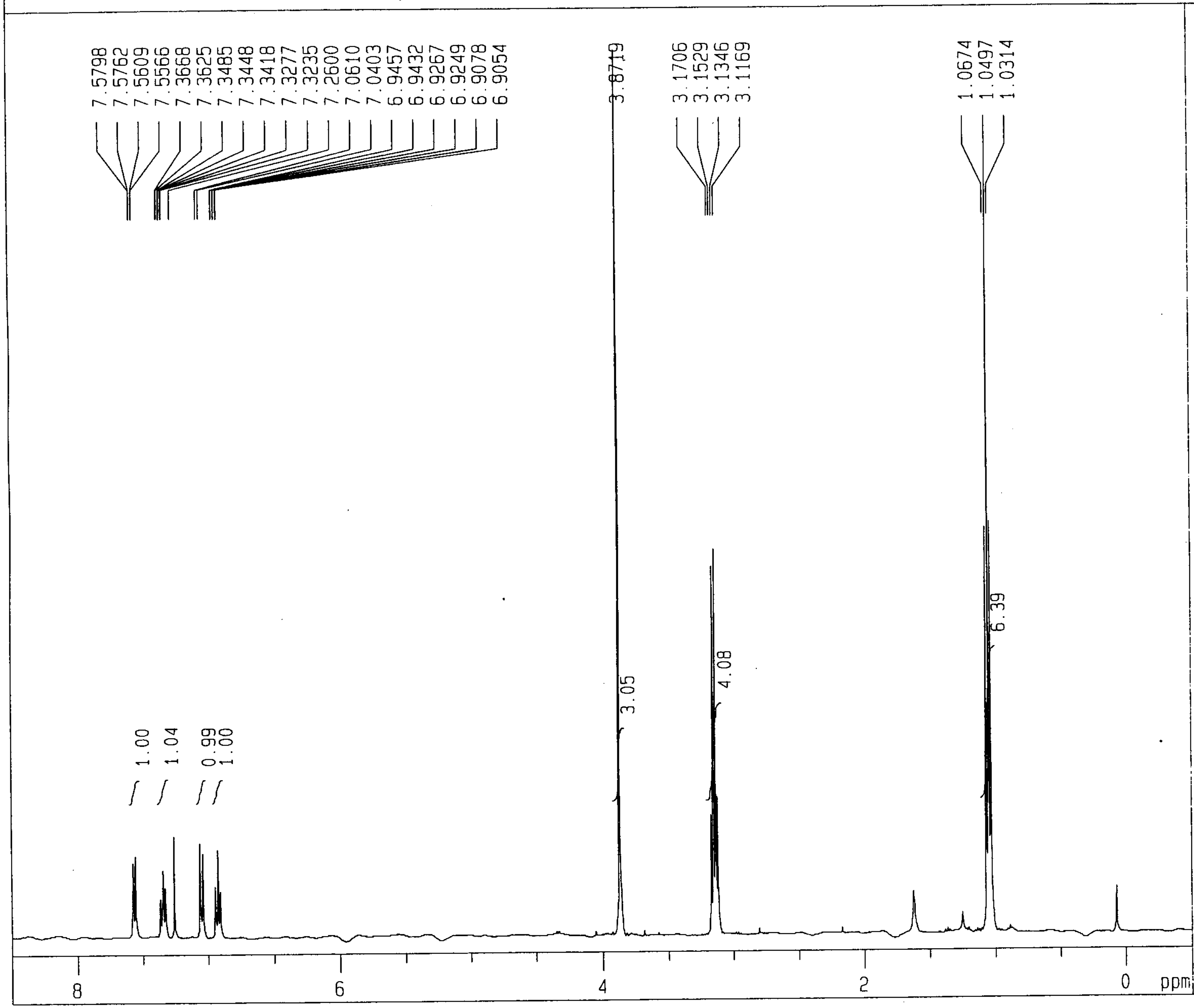


3ab

1H Line 070510-12-427-3



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RESOL	:	0.24 Hz
PW1	:	5.05 usec
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OBFRQ	:	395.75 MHz
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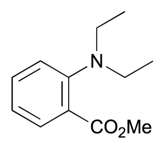

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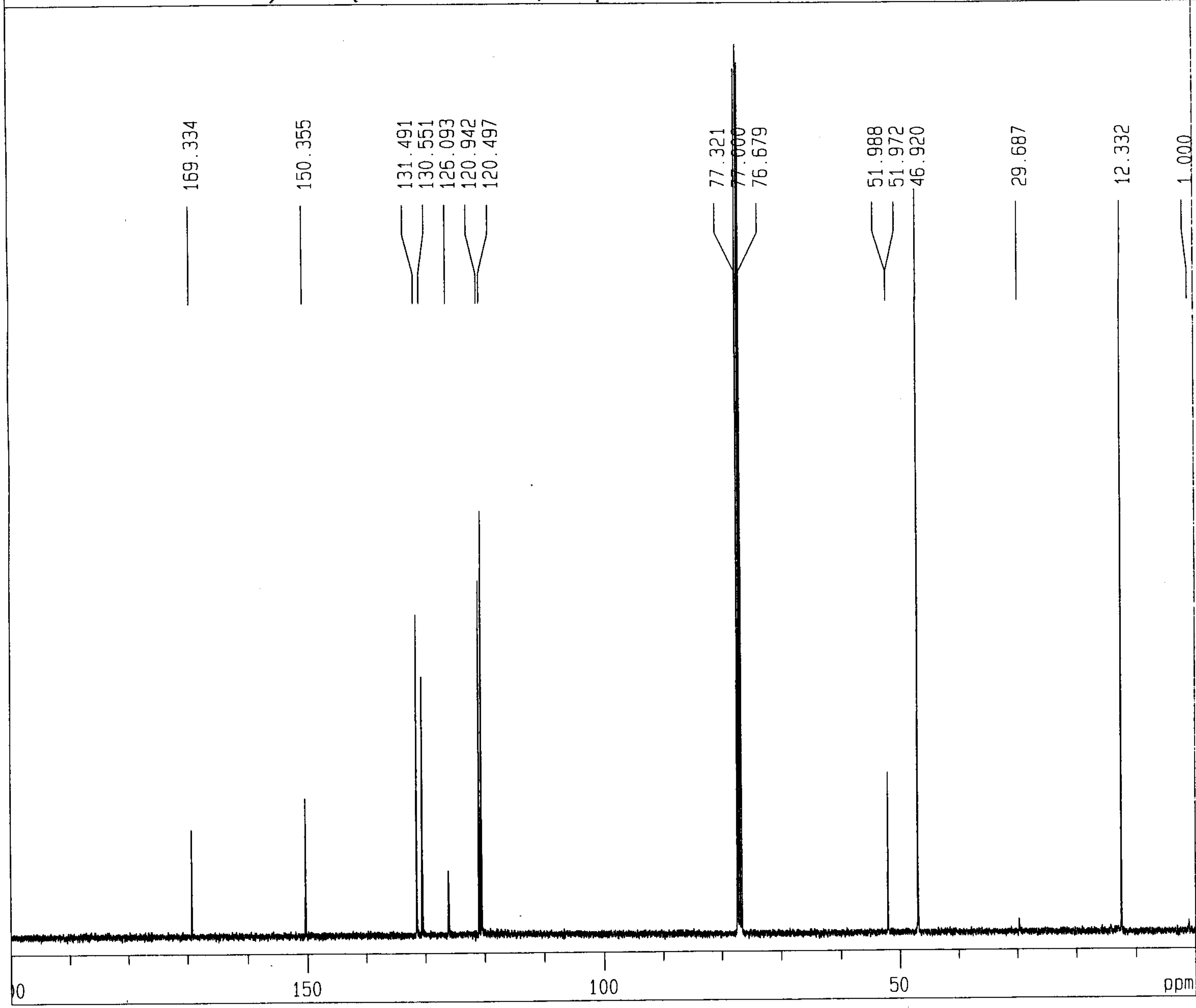

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RESOL	:	0.82 Hz
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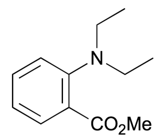

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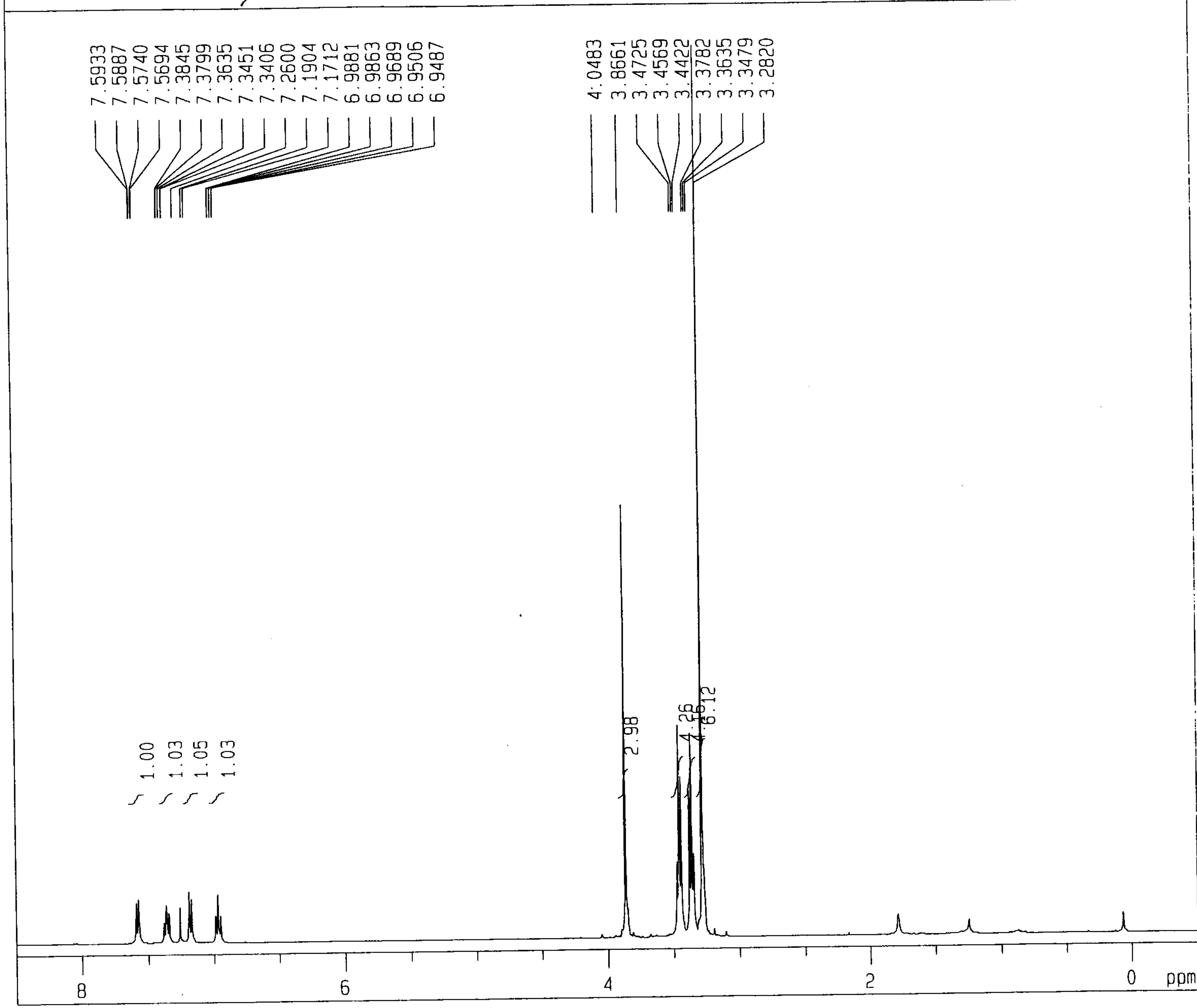
1H Line 070515 - 12 - 441 - 3



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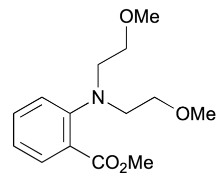
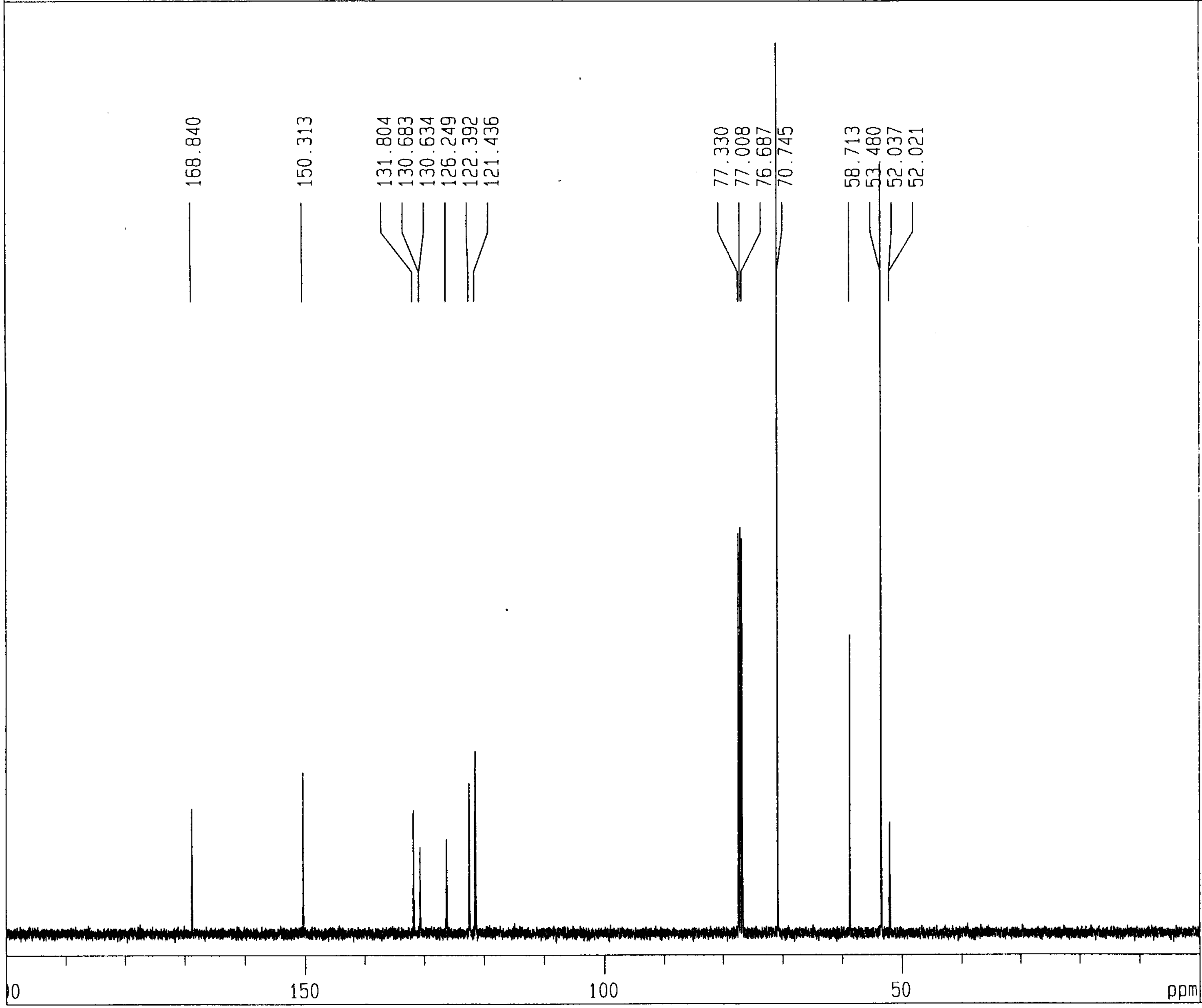


3ad

1H Line 070515 - 12 - 441 - 3 - 13C



Date : Mon Feb 12 01:43:45 2007

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INTVL	:	37.2 usec
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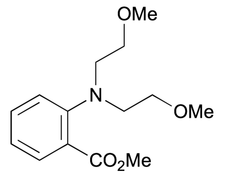

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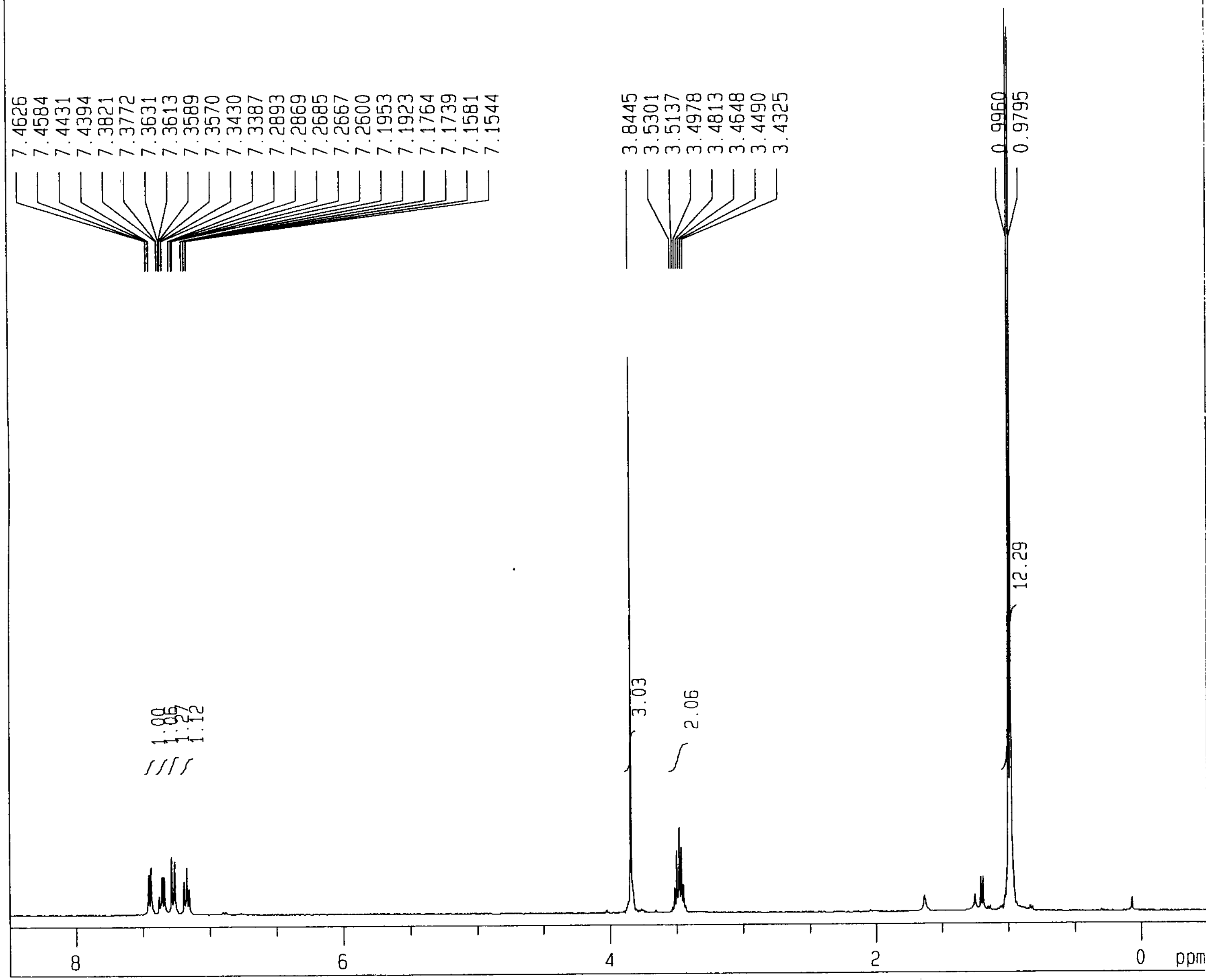
1H Line 070522-13-487-3



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DUMMY	:	1 times
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OBFRQ	:	395.75 MHz
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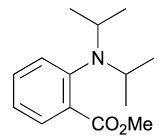
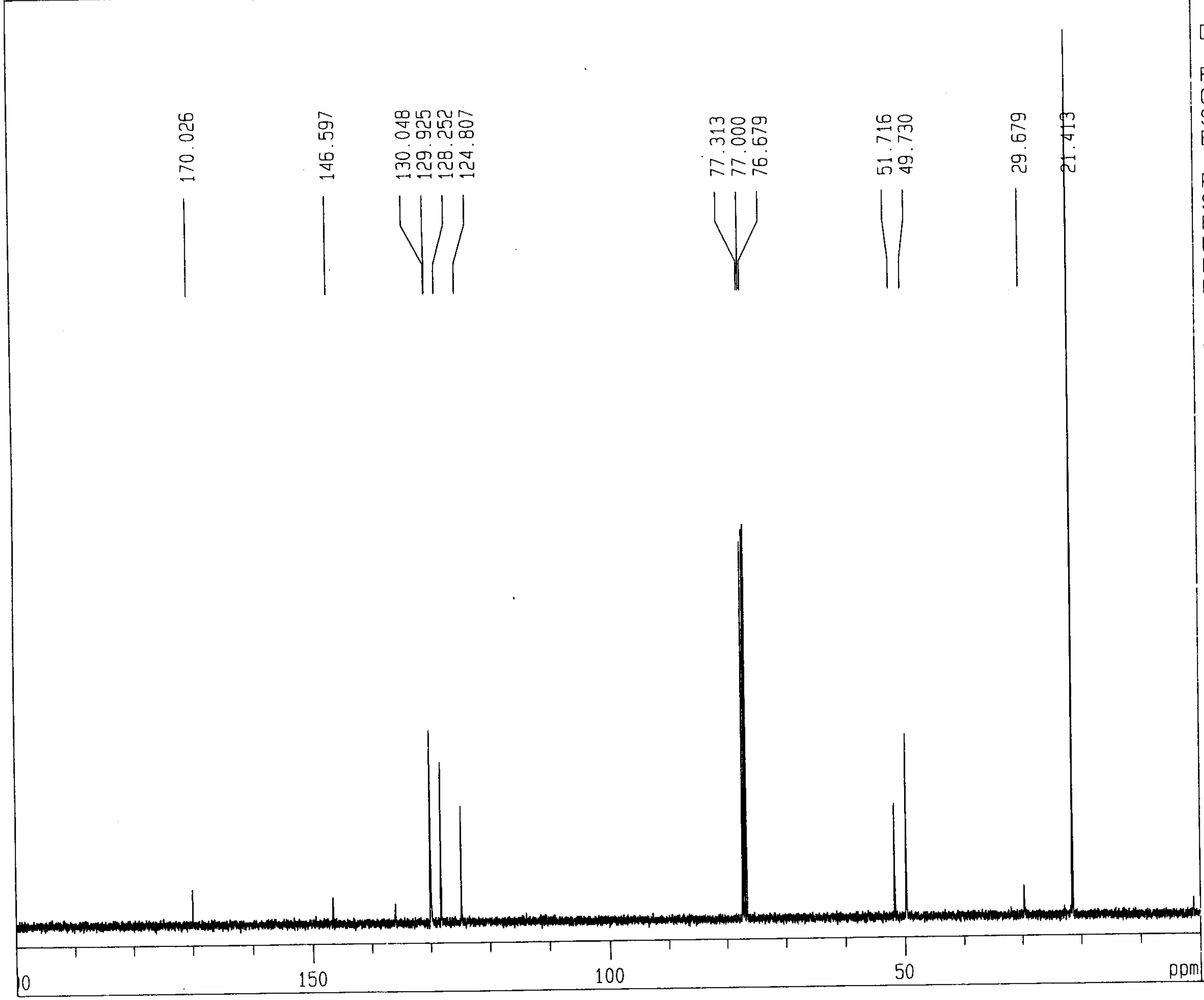


3ae

1H Line 070522-13-445-3-13C



Date : Mon Feb 12 01:43:45 2007

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DEADT	:	19.1 usec
INTVL	:	37.2 usec
TIMES	:	20000 times
DUMMY	:	4 times
PD	:	1.7810 sec
ACQTM	:	1218.9696 msec
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INIWT	:	1000.0000 msec
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OBFRQ	:	99.45 MHz
OBSET	:	104750.00 Hz
RGAIN	:	28
IRNUC	:	1H
IRFRQ	:	395.75 MHz
IRSET	:	134498.00 Hz
IRRPW	:	45.0 usec
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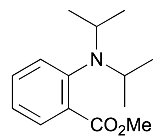
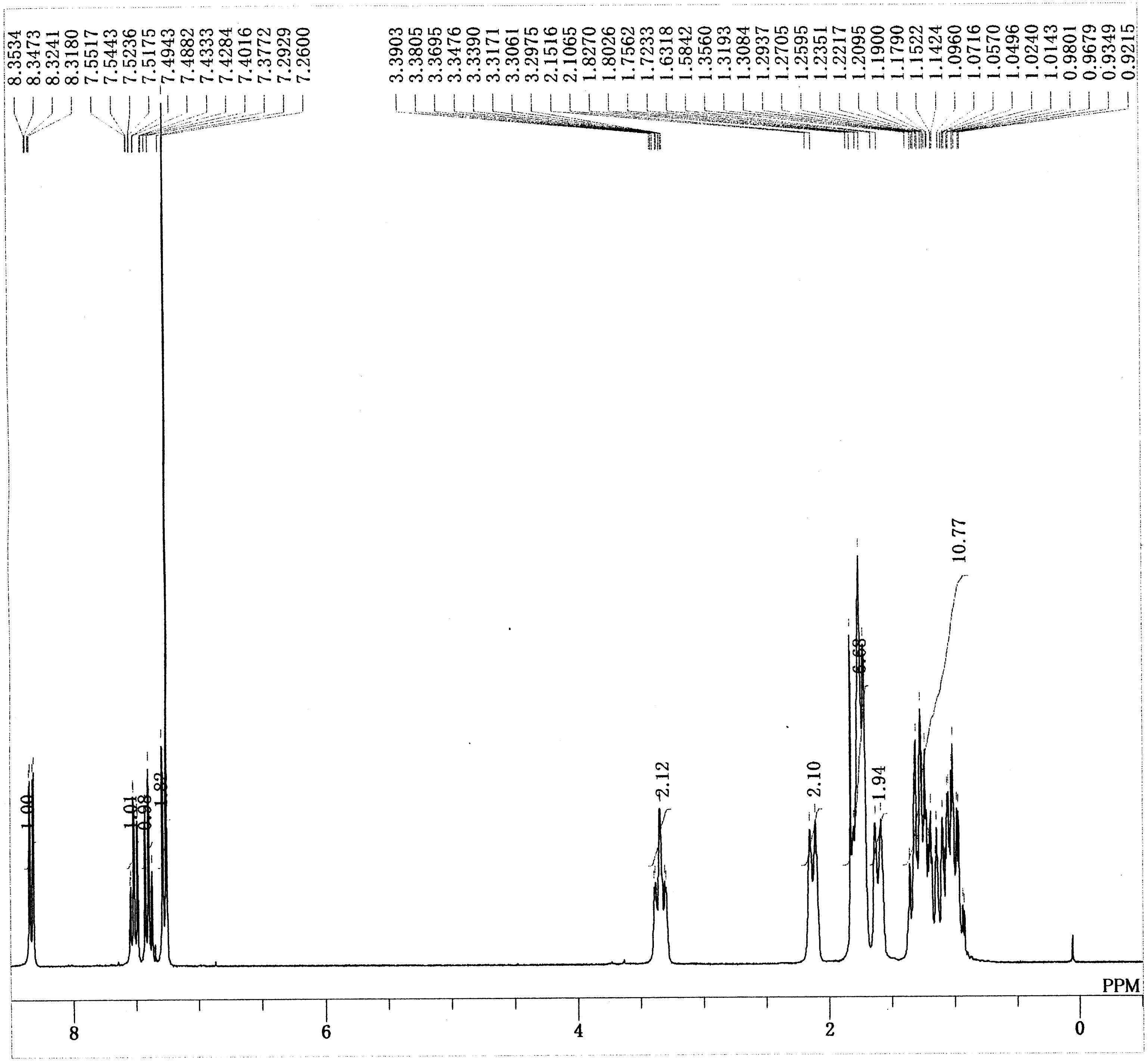


3ae



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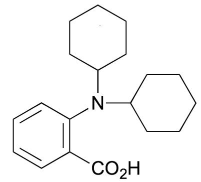
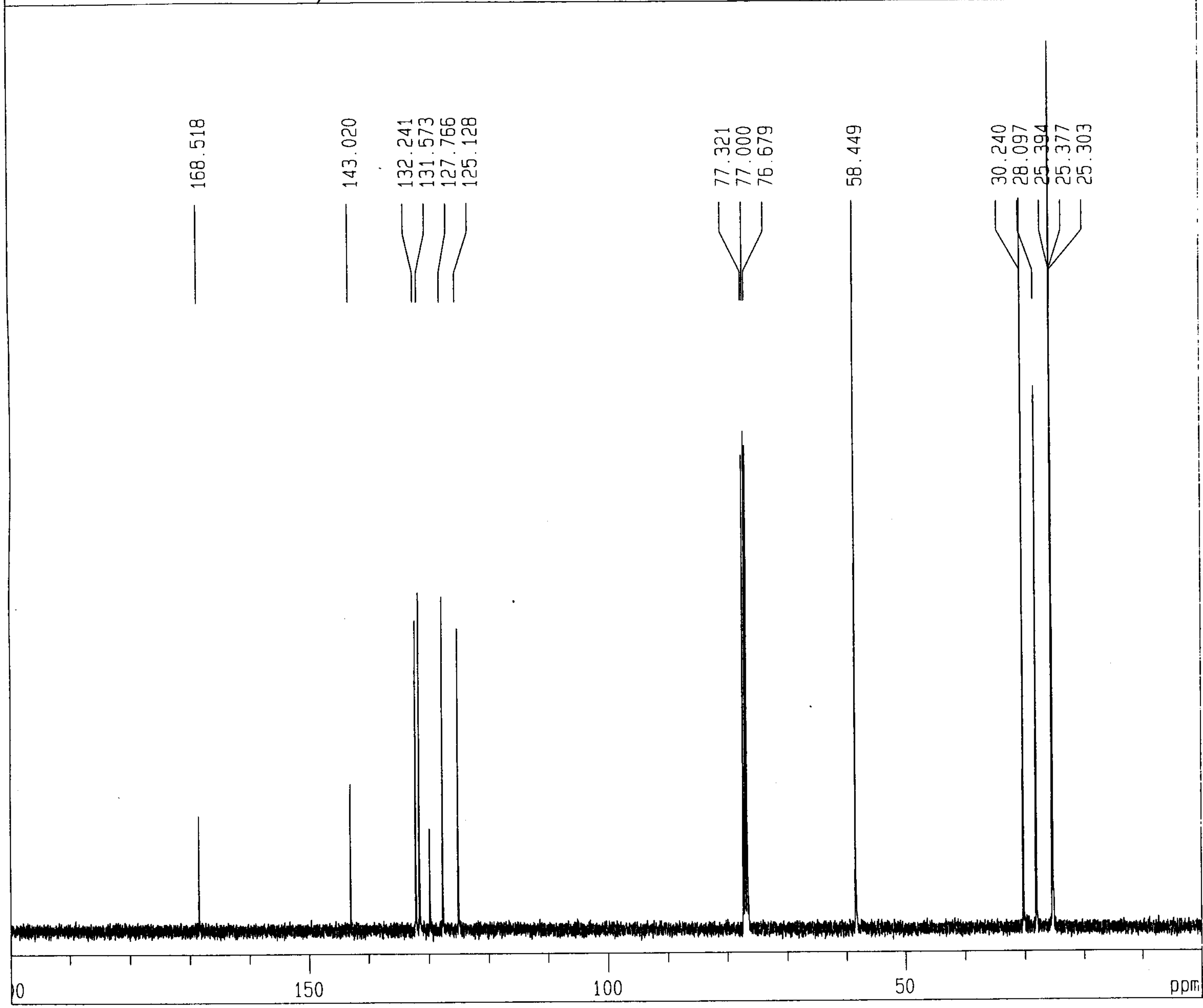
PW1	5.40 usec
IRNUC	1H
CTEMP	19.5 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.12 Hz
RGAIN	16



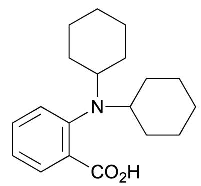
3af

1H Line 070830-15 - 522-3-13C



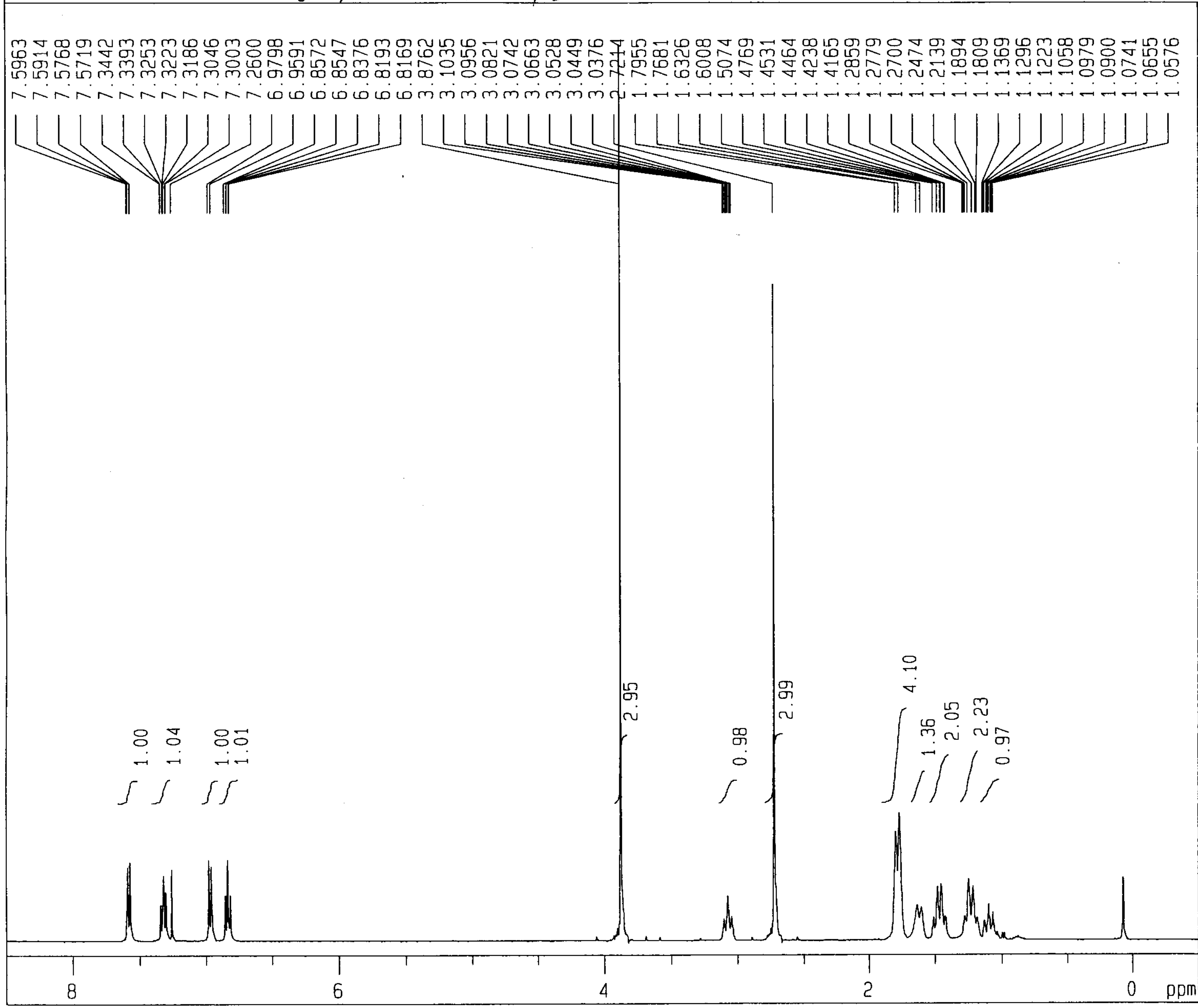
Date : Mon Feb 12 01:43:45 2007

FileName	:	.LoadingFID.nmdata
Comment	:	1H Line
SliceHistory	:	
EXMODE	:	bcm
POINT	:	32768 points
SAMPO	:	32768 points
FREQU	:	26881.7 Hz
FILTR	:	13450 Hz
DELAY	:	14.9 usec
DEADT	:	19.1 usec
INTVL	:	37.2 usec
TIMES	:	20000 times
DUMMY	:	4 times
PD	:	1.7810 sec
ACQTM	:	1218.9696 msec
PREDL	:	0.01000 msec
INIWT	:	1000.0000 msec
RESOL	:	0.82 Hz
PW1	:	6.50 usec
OBNUC	:	13C
OBFRQ	:	99.45 MHz
OBSET	:	104750.00 Hz
RGAIN	:	29
IRNUC	:	1H
IRFRQ	:	395.75 MHz
IRSET	:	134498.00 Hz
IRRPW	:	45.0 usec
IRRNS	:	0
SCANS	:	1658 times
SLVNT	:	CDCL3
SPINNING	:	17 Hz
TEMP	:	21.0 C

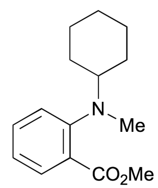


3af

1H Line 070521-13-446-3

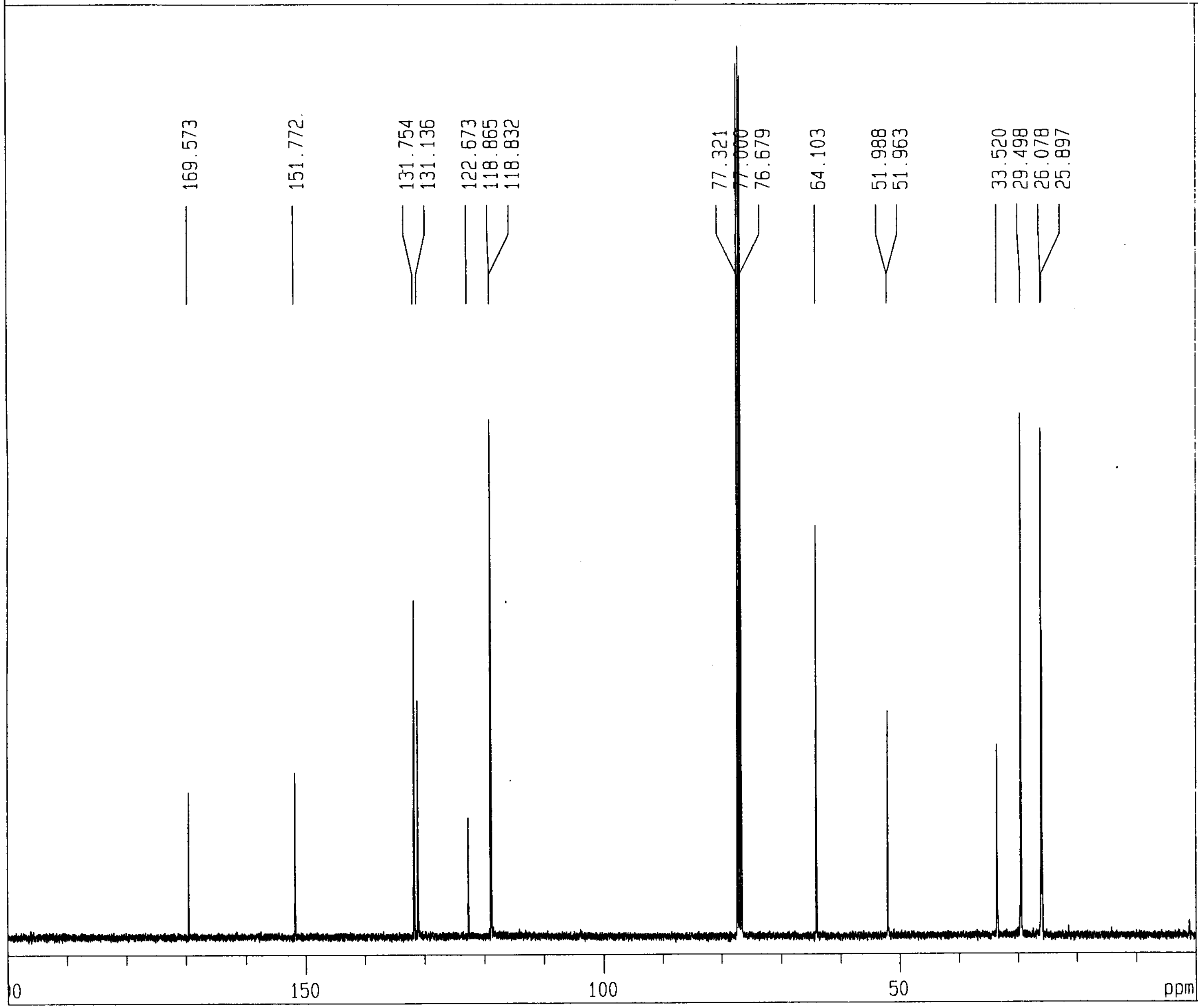


Date : Fri May 18 14:23:39 2007		
FileName	:	.LoadingFID.nmdata
Comment	:	1H Line
SliceHistory	:	
EXMODE	:	non
POINT	:	32768 points
SAMPO	:	32768 points
FREQU	:	7917.7 Hz
FILTR	:	3950 Hz
DELAY	:	50.6 usec
DEADT	:	73.4 usec
INTVL	:	126.3 usec
TIMES	:	16 times
DUMMY	:	1 times
PD	:	2.8614 sec
ACQTM	:	4138.5986 msec
PREDL	:	0.01000 msec
INIWT	:	1000.0000 msec
RESOL	:	0.24 Hz
PW1	:	5.05 usec
OBNUC	:	1H
OBFRQ	:	395.75 MHz
OBSET	:	134498.00 Hz
RGAIN	:	20
SCANS	:	16 times
SLVNT	:	CDCL3
SPINNING	:	9 Hz
TEMP	:	19.1 C

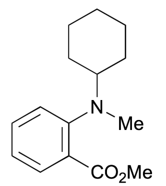


3ag

1H Line   070521-13-446-3-13C

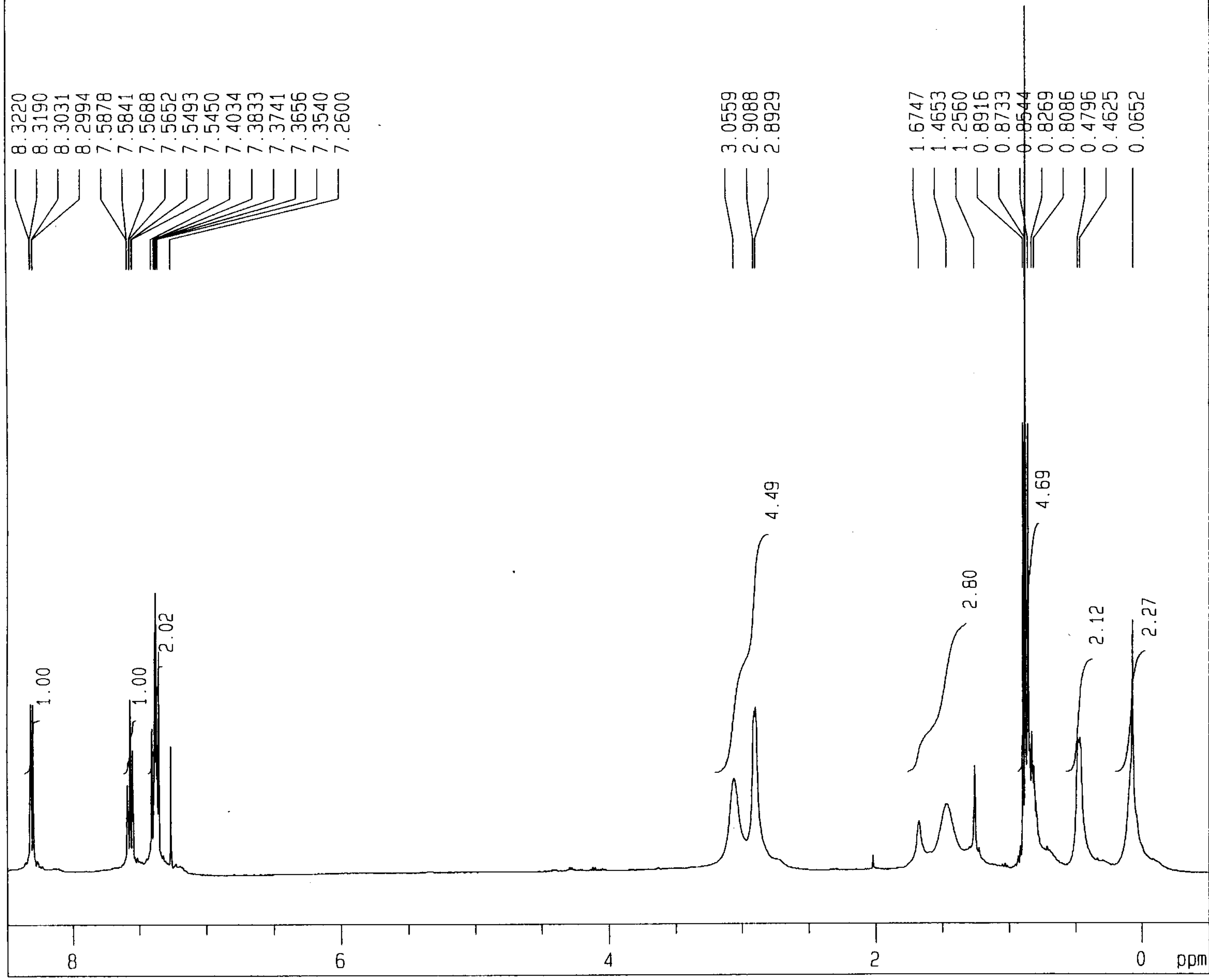


Date : Mon Feb 12 01:43:45 2007		
FileName	:	.LoadingFID.nmdata
Comment	:	1H Line
SliceHistory	:	
EXMODE	:	bcm
POINT	:	32768 points
SAMPO	:	32768 points
FREQU	:	26881.7 Hz
FILTR	:	13450 Hz
DELAY	:	14.9 usec
DEADT	:	19.1 usec
INTVL	:	37.2 usec
TIMES	:	20000 times
DUMMY	:	4 times
PD	:	1.7810 sec
ACQTM	:	1218.9696 msec
PREDL	:	0.01000 msec
INIWT	:	1000.0000 msec
RESOL	:	0.82 Hz
PW1	:	6.50 usec
OBNUC	:	13C
OBFRQ	:	99.45 MHz
OBSET	:	104750.00 Hz
RGAIN	:	28
IRNUC	:	1H
IRFRQ	:	395.75 MHz
IRSET	:	134498.00 Hz
IRRPW	:	45.0 usec
IRRNS	:	0
SCANS	:	8903 times
SLVNT	:	CDCL3
SPINNING	:	3 Hz
TEMP	:	19.9 C



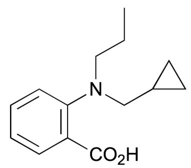
3ag

1H Line 070903-15-527-3

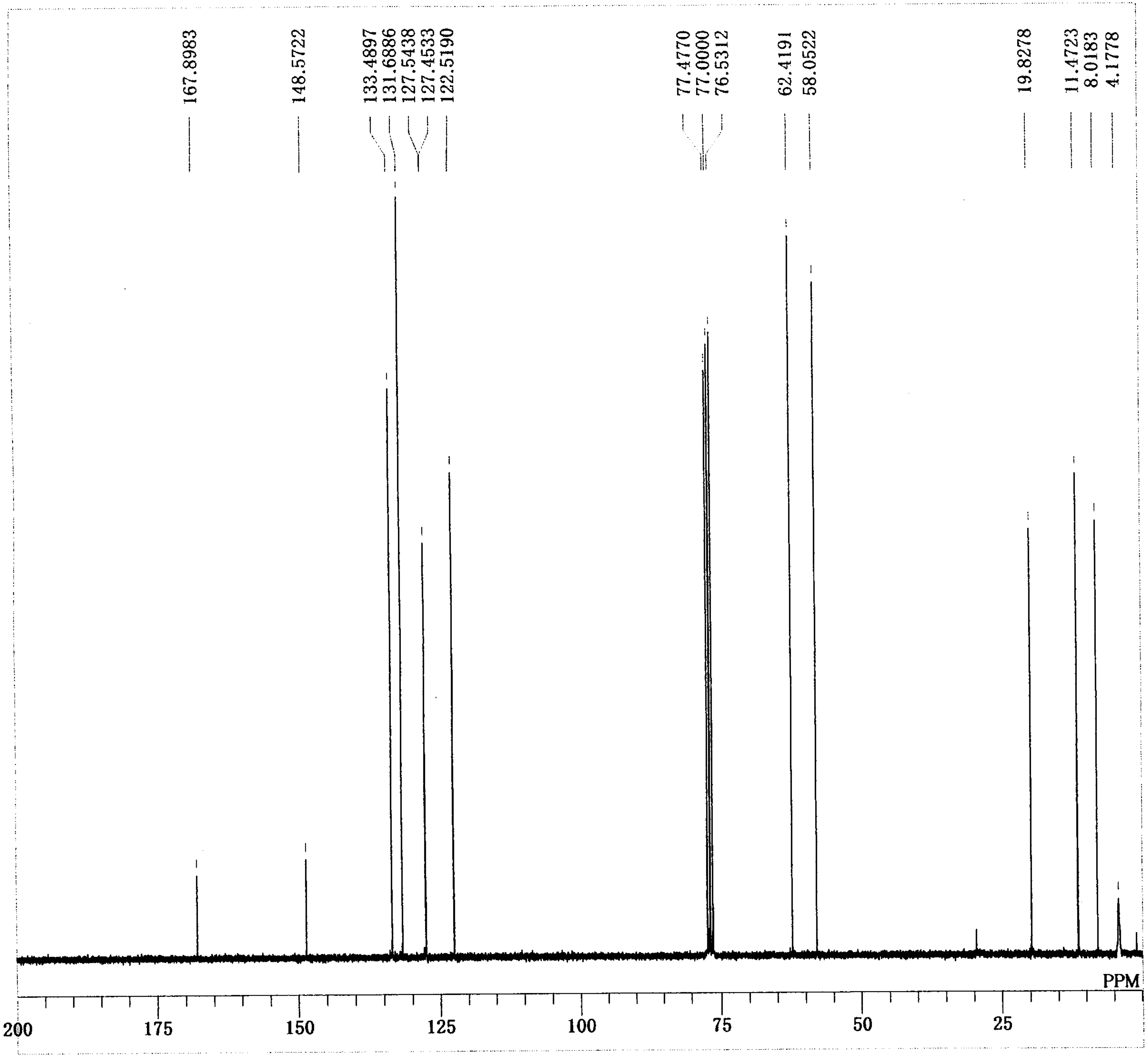


Date : Thu Aug 30 22:11:55 2007

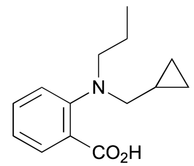
FileName	:	.LoadingFID.nmdata
Comment	:	1H Line
SliceHistory	:	
EXMODE	:	non
POINT	:	32768 points
SAMPO	:	32768 points
FREQU	:	7917.7 Hz
FILTR	:	3950 Hz
DELAY	:	50.6 usec
DEADT	:	73.4 usec
INTVL	:	126.3 usec
TIMES	:	32 times
DUMMY	:	1 times
PD	:	2.8614 sec
ACQTM	:	4138.5986 msec
PREDL	:	0.01000 msec
INIWT	:	1000.0000 msec
RESOL	:	0.24 Hz
PW1	:	5.05 usec
OBNUC	:	1H
OBFRQ	:	395.75 MHz
OBSET	:	134498.00 Hz
RGAIN	:	19
SCANS	:	32 times
SLVNT	:	CDCL3
SPINNING	:	13 Hz
TEMP	:	50.0 C



3ah

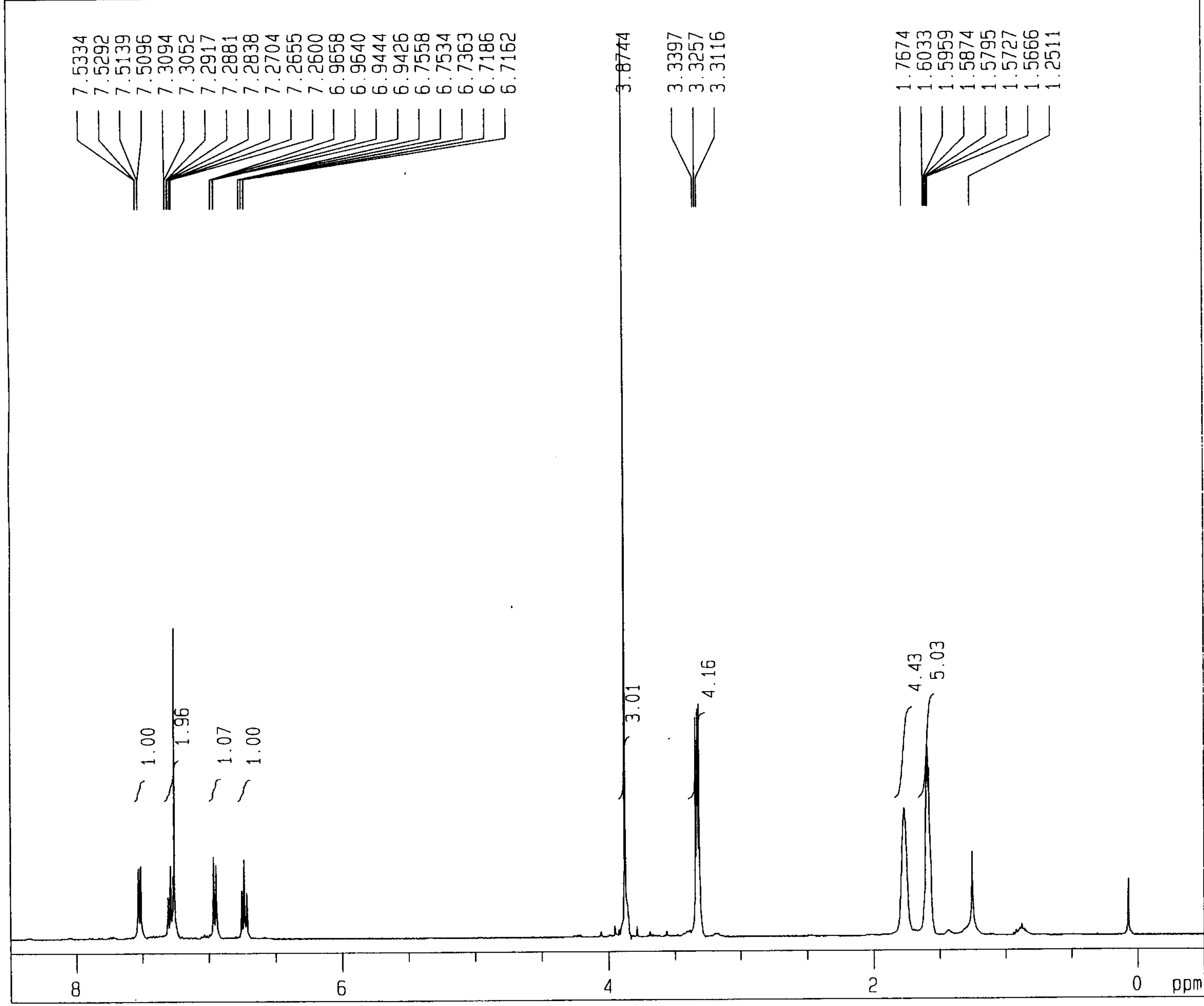


DFILE	_DEFAULT.ALS
COMNT	
DATIM	Sat Sep 01 08:43:59 2007
OBNUC	13C
EXMOD	BCM
OBFRQ	67.80 MHz
OBSET	135.00 KHz
OBFIN	5200.00 Hz
POINT	32768
FREQU	18306.64 Hz
SCANS	10644
ACQTM	1.7900 sec
PD	1.2100 sec
PW1	3.50 usec
IRNUC	1H
CTEMP	19.6 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	28



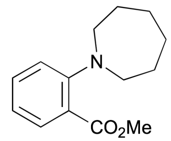
3ah

1H Line 07d524-13-448-3



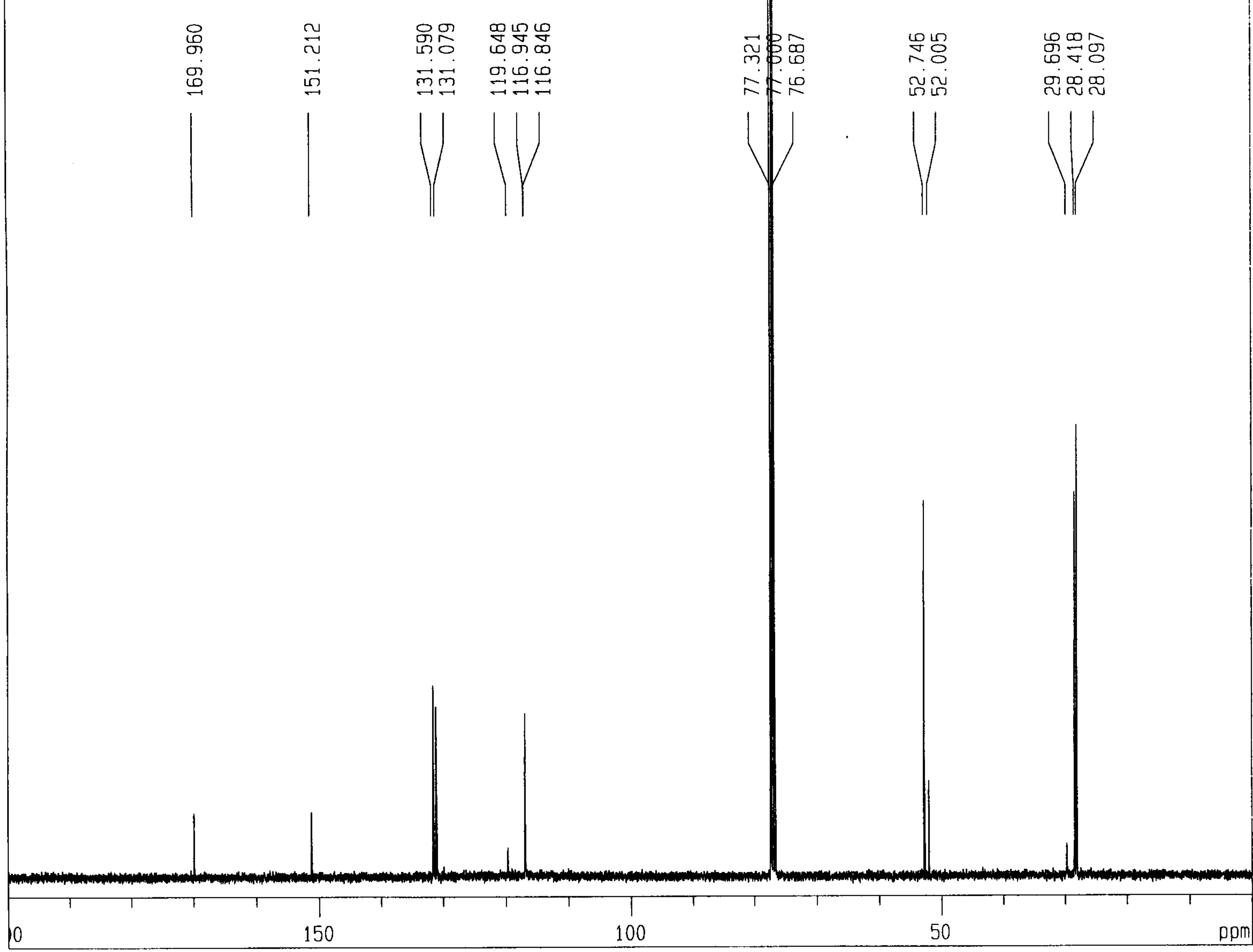
Date : Mon May 21 12:08:05 2007

FileName	:	.LoadingFID.nmdata
Comment	:	1H Line
SliceHistory	:	
EXMODE	:	non
POINT	:	32768 points
SAMPO	:	32768 points
FREQU	:	7917.7 Hz
FILTR	:	3950 Hz
DELAY	:	50.6 usec
DEADT	:	73.4 usec
INTVL	:	126.3 usec
TIMES	:	16 times
DUMMY	:	1 times
PD	:	2.8614 sec
ACQTM	:	4138.5986 msec
PREDL	:	0.01000 msec
INIWT	:	1000.0000 msec
RESOL	:	0.24 Hz
PW1	:	5.05 usec
OBNUC	:	1H
OBFRQ	:	395.75 MHz
OBSET	:	134498.00 Hz
RGAIN	:	23
SCANS	:	16 times
SLVNT	:	CDCL3
SPINNING	:	17 Hz
TEMP	:	19.7 C

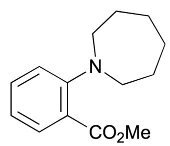


3ai

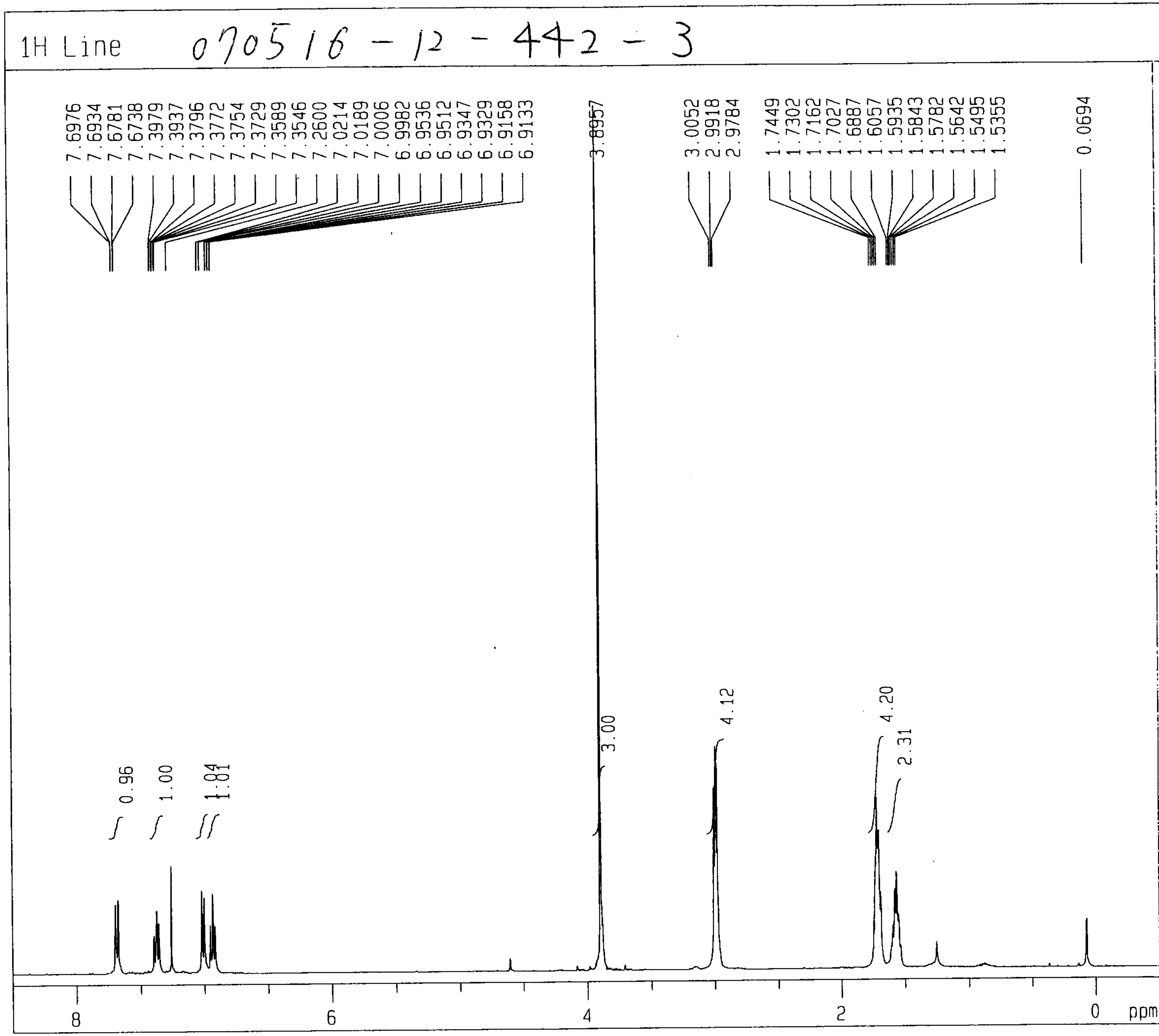
1H Line 070524-13-448-3-13C



Date : Mon Feb 12 01:43:45 2007		
FileName	:	.LoadingFID.nmdata
Comment	:	1H Line
SliceHistory	:	
EXMODE	:	bcm
POINT	:	32768 points
SAMPO	:	32768 points
FREQU	:	26881.7 Hz
FILTR	:	13450 Hz
DELAY	:	14.9 usec
DEADT	:	19.1 usec
INTVL	:	37.2 usec
TIMES	:	20000 times
DUMMY	:	4 times
PD	:	1.7810 sec
ACQTM	:	1218.9696 msec
PREDL	:	0.01000 msec
INIWT	:	1000.0000 msec
RESOL	:	0.82 Hz
PW1	:	6.50 usec
OBNUC	:	13C
OBFRQ	:	99.45 MHz
OBSET	:	104750.00 Hz
RGAIN	:	28
IRNUC	:	1H
IRFRQ	:	395.75 MHz
IRSET	:	134498.00 Hz
IRRPW	:	45.0 usec
IRRNS	:	0
SCANS	:	11370 times
SLVNT	:	CDCL3
SPINNING	:	3 Hz
TEMP	:	20.5 C

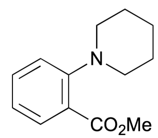


3ai



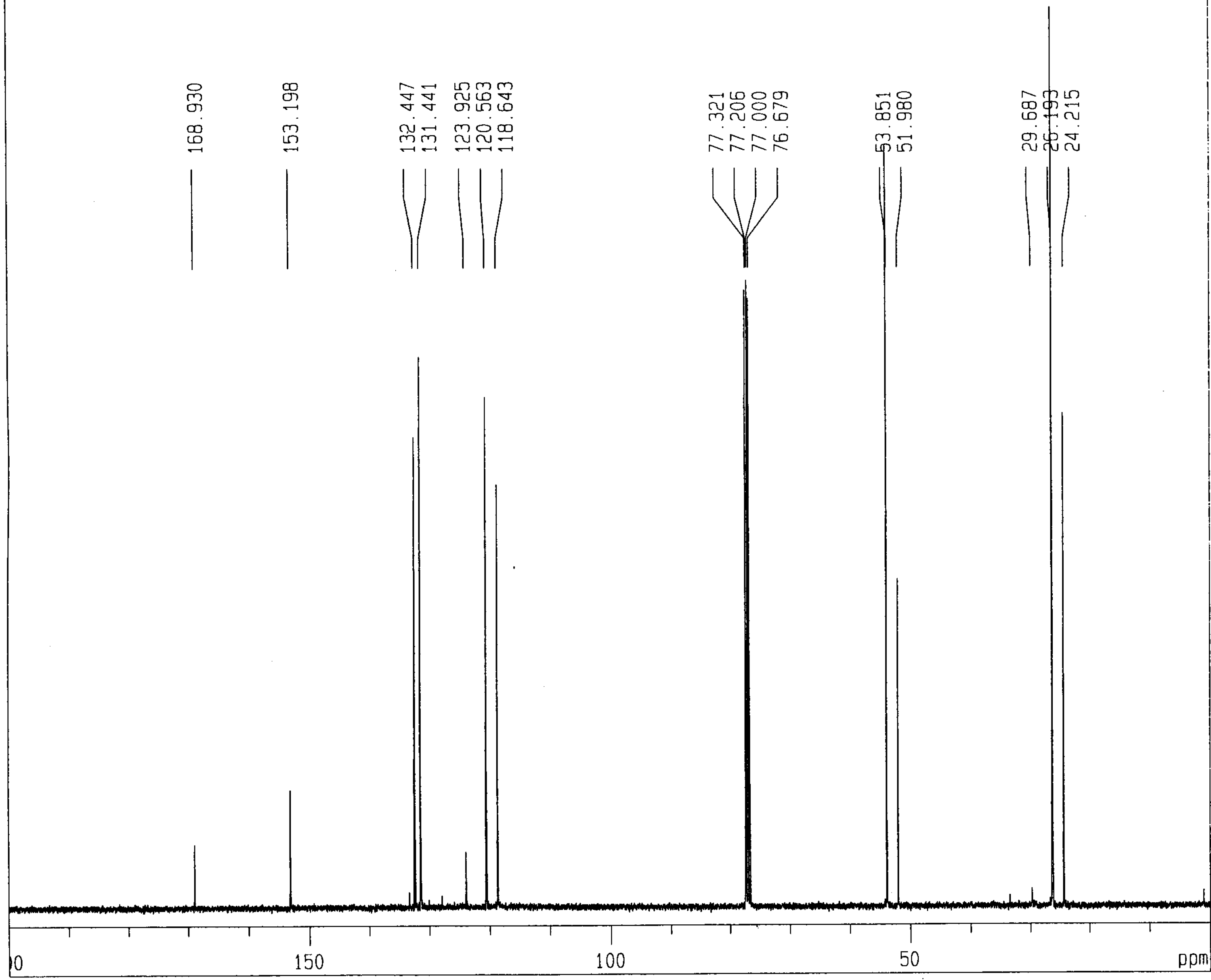
Date : Sun May 13 12:07:48 2007

FileName	:	.LoadingFID.nmdata
Comment	:	1H Line
SliceHistory	:	
EXMODE	:	non
POINT	:	32768 points
SAMPO	:	32768 points
FREQU	:	7917.7 Hz
FILTR	:	3950 Hz
DELAY	:	50.6 usec
DEADT	:	73.4 usec
INTVL	:	126.3 usec
TIMES	:	16 times
DUMMY	:	1 times
PD	:	2.8614 sec
ACQTM	:	4138.5986 msec
PREDL	:	0.01000 msec
INIWT	:	1000.0000 msec
RESOL	:	0.24 Hz
PW1	:	5.05 usec
OBNUC	:	1H
OBFRQ	:	395.75 MHz
OBSET	:	134498.00 Hz
RGAIN	:	21
SCANS	:	16 times
SLVNT	:	CDCL3
SPINNING	:	16 Hz
TEMP	:	19.9 C



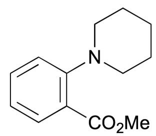
3aj

1H Line 070516-12-442-3-^{13}C



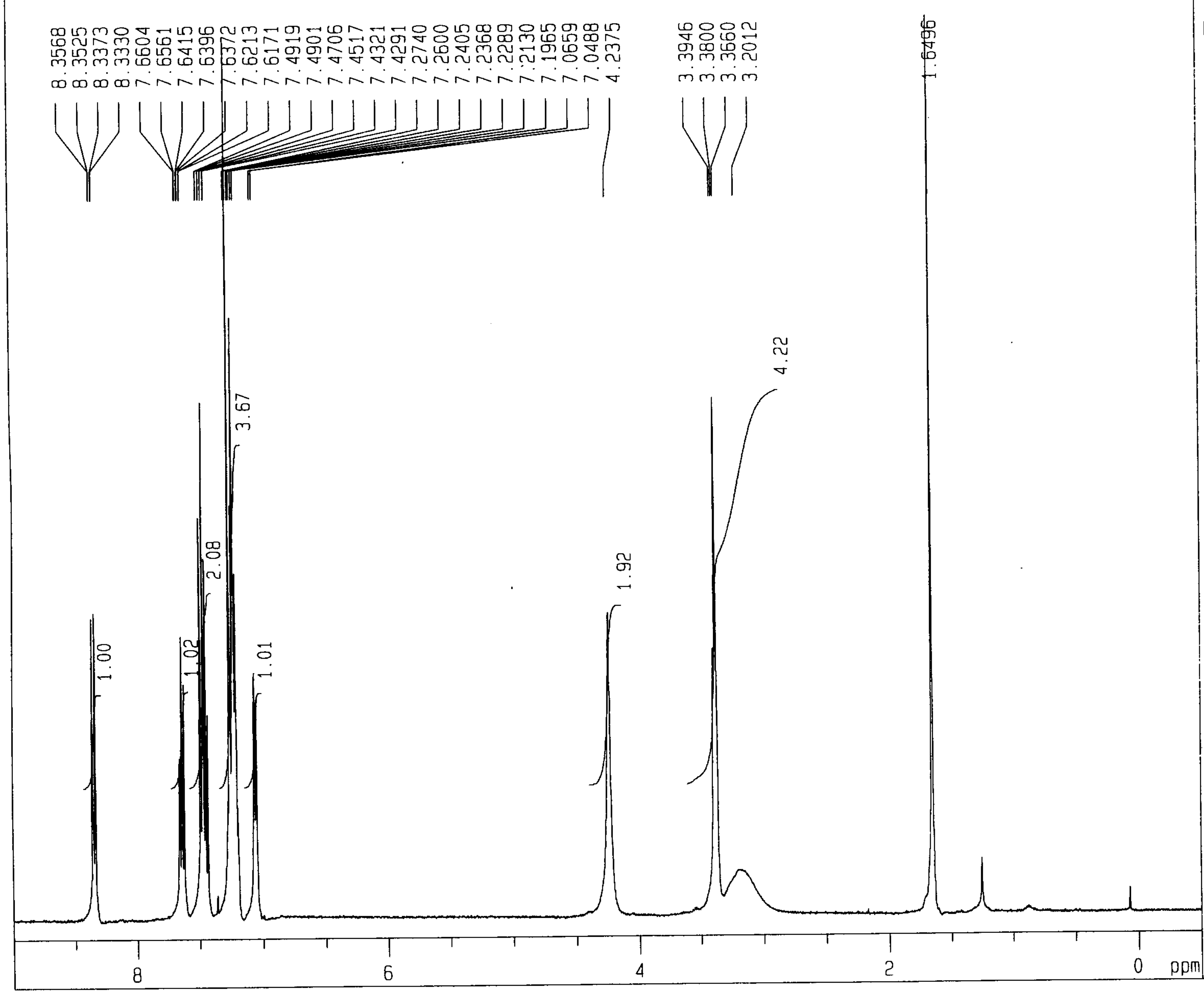
Date : Mon Feb 12 01:43:45 2007

FileName	:	.LoadingFID.nmdata
Comment	:	1H Line
SliceHistory	:	
EXMODE	:	bcm
POINT	:	32768 points
SAMPO	:	32768 points
FREQU	:	26881.7 Hz
FILTR	:	13450 Hz
DELAY	:	14.9 usec
DEADT	:	19.1 usec
INTVL	:	37.2 usec
TIMES	:	20000 times
DUMMY	:	4 times
PD	:	1.7810 sec
ACQTM	:	1218.9696 msec
PREDL	:	0.01000 msec
INIWT	:	1000.0000 msec
RESOL	:	0.82 Hz
PW1	:	6.50 usec
OBNUC	:	13C
OBFRQ	:	99.45 MHz
OBSET	:	104750.00 Hz
RGAIN	:	28
IRNUC	:	1H
IRFRQ	:	395.75 MHz
IRSET	:	134498.00 Hz
IRRPW	:	45.0 usec
IRRNS	:	0
SCANS	:	11584 times
SLVNT	:	CDCL3
SPINNING	:	17 Hz
TEMP	:	21.1 C



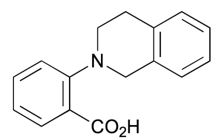
3aj

1H Line 070615-13-462-2



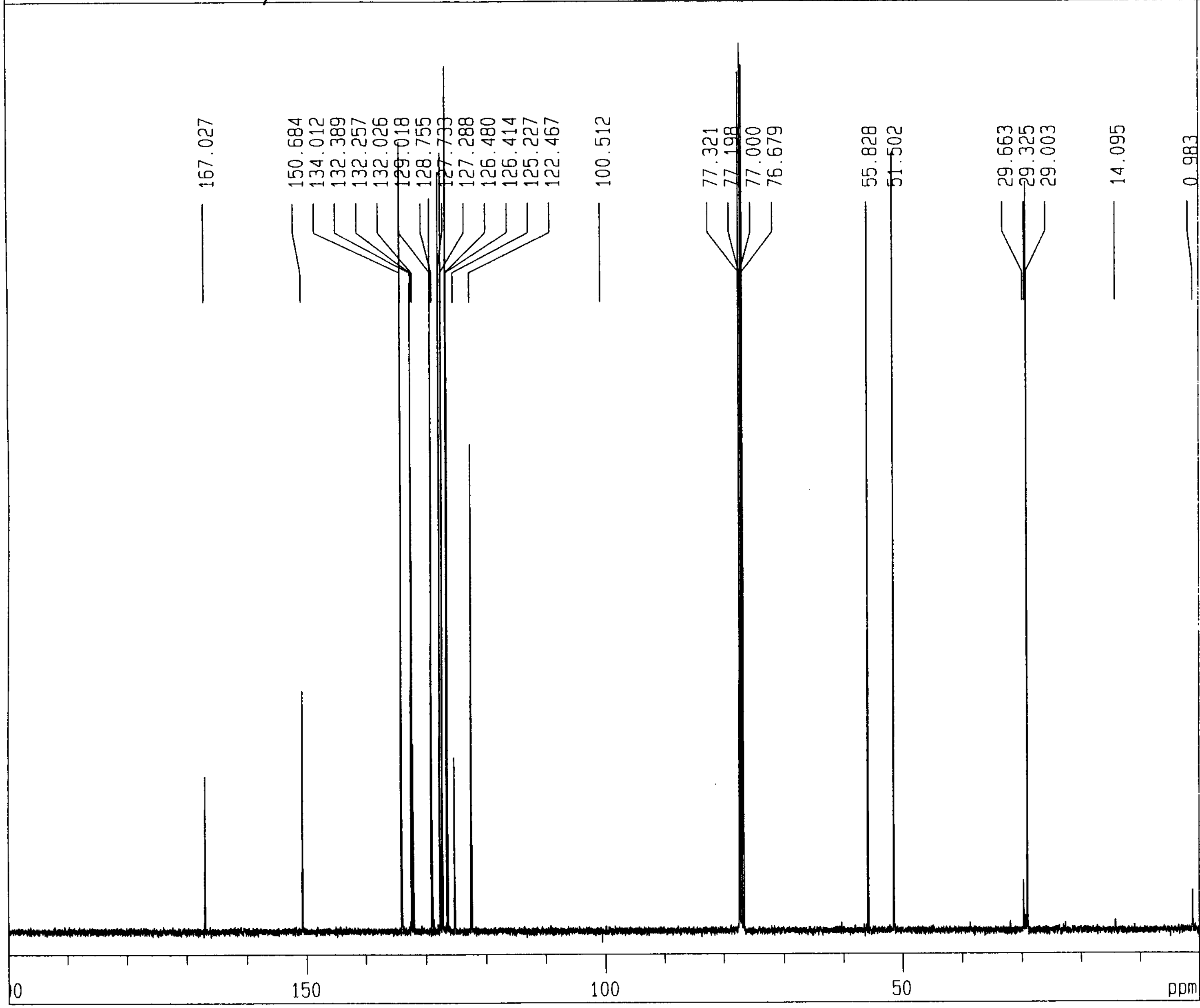
Date : Tue Jun 12 08:36:34 2007

FileName	:	.LoadingFID.nmdata
Comment	:	1H Line
SliceHistory	:	
EXMODE	:	non
POINT	:	32768 points
SAMPO	:	32768 points
FREQU	:	7917.7 Hz
FILTR	:	3950 Hz
DELAY	:	50.6 usec
DEADT	:	73.4 usec
INTVL	:	126.3 usec
TIMES	:	16 times
DUMMY	:	1 times
PD	:	2.8614 sec
ACQTM	:	4138.5986 msec
PREDL	:	0.01000 msec
INIWT	:	1000.0000 msec
RESOL	:	0.24 Hz
PW1	:	5.05 usec
OBNUC	:	1H
OBFRQ	:	395.75 MHz
OBSET	:	134498.00 Hz
RGAIN	:	24
SCANS	:	16 times
SLVNT	:	CDCL3
SPINNING	:	21 Hz
TEMP	:	20.4 C



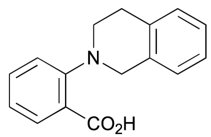
3ak

1H Line ↄ70612 - 13- 462 - 1- ^{13}C



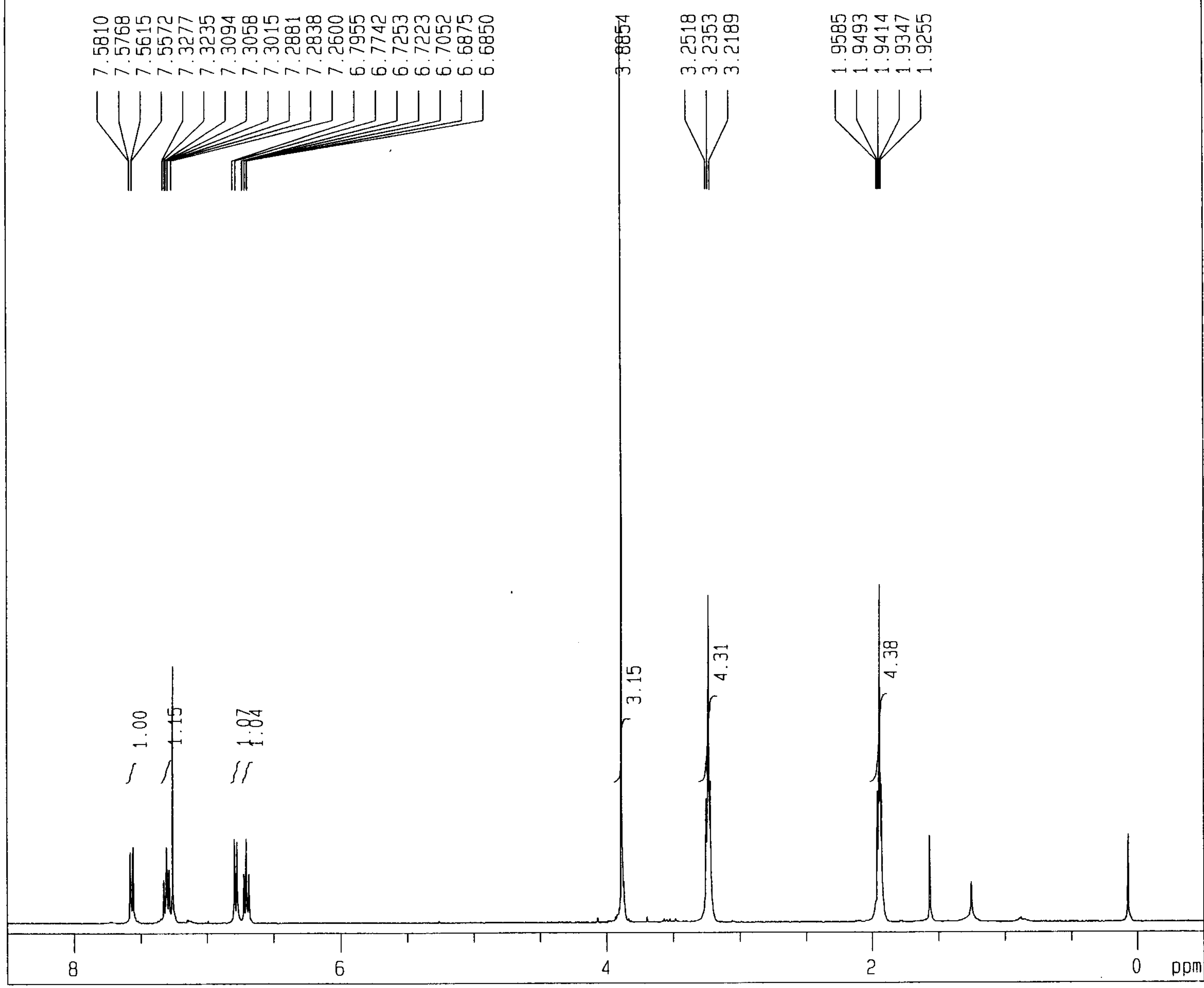
Date : Sat Jun 9 21:51:08 2007

FileName	:	.LoadingFID.nmdata
Comment	:	1H Line
SliceHistory	:	
EXMODE	:	bcm
POINT	:	32768 points
SAMPO	:	32768 points
FREQU	:	26881.7 Hz
FILTR	:	13450 Hz
DELAY	:	14.9 usec
DEADT	:	19.1 usec
INTVL	:	37.2 usec
TIMES	:	20000 times
DUMMY	:	4 times
PD	:	1.7810 sec
ACQTM	:	1218.9696 msec
PREDL	:	0.01000 msec
INIWT	:	1000.0000 msec
RESOL	:	0.82 Hz
PW1	:	6.50 usec
OBNUC	:	13C
OBFRQ	:	99.45 MHz
OBSET	:	104750.00 Hz
RGAIN	:	28
IRNUC	:	1H
IRFRQ	:	395.75 MHz
IRSET	:	134498.00 Hz
IRRPW	:	45.0 usec
IRRNS	:	0
SCANS	:	12228 times
SLVNT	:	CDCL3
SPINNING	:	16 Hz
TEMP	:	21.0 C



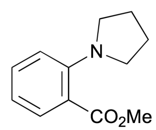
3ak

1H Line 070619-13-465-1



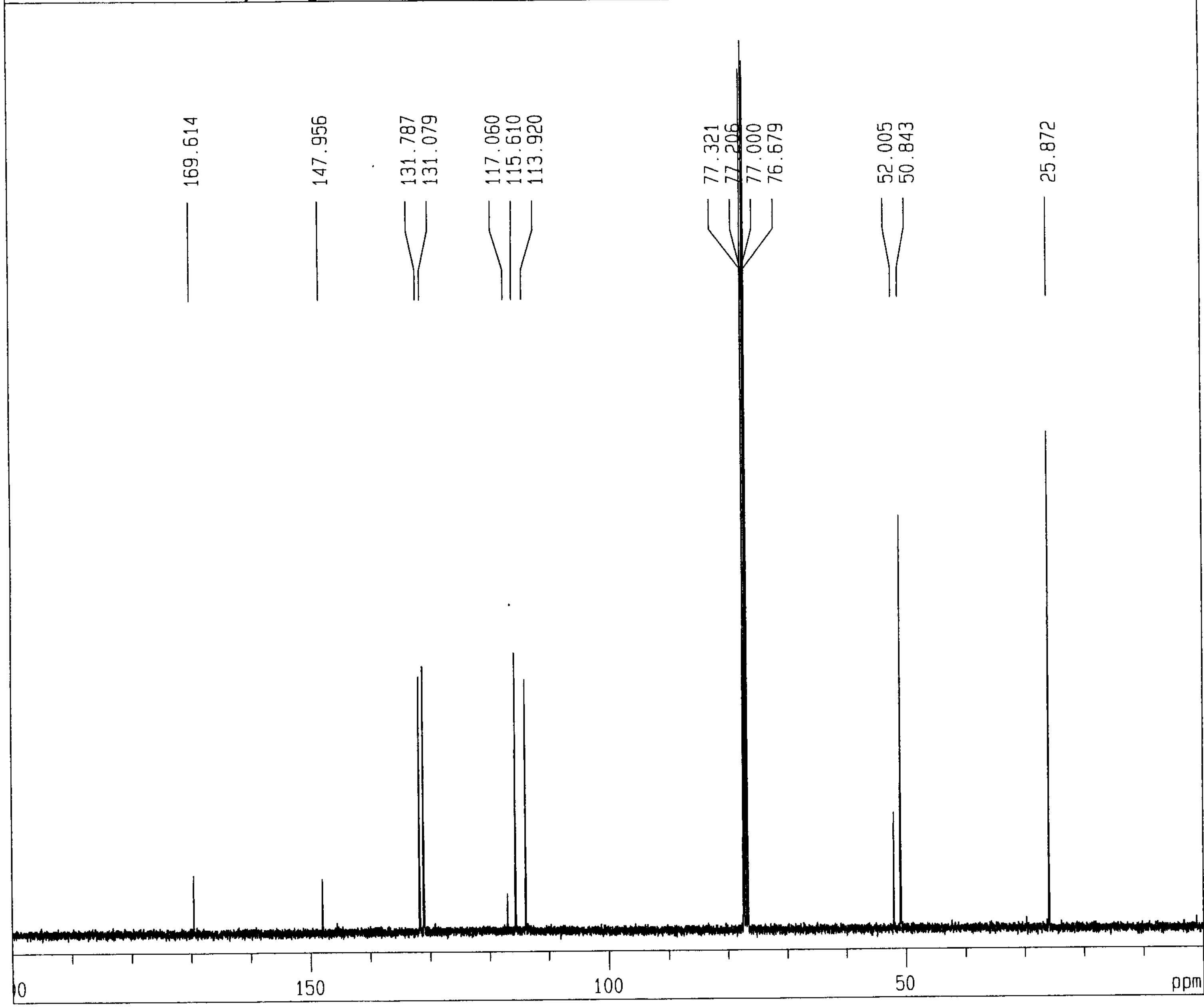
Date : Sat Jun 16 11:41:16 2007

FileName	:	.LoadingFID.nmdata
Comment	:	1H Line
SliceHistory	:	
EXMODE	:	non
POINT	:	32768 points
SAMPO	:	32768 points
FREQU	:	7917.7 Hz
FILTR	:	3950 Hz
DELAY	:	50.6 usec
DEADT	:	73.4 usec
INTVL	:	126.3 usec
TIMES	:	32 times
DUMMY	:	1 times
PD	:	2.8614 sec
ACQTM	:	4138.5986 msec
PREDL	:	0.01000 msec
INIWT	:	1000.0000 msec
RESOL	:	0.24 Hz
PW1	:	5.05 usec
OBNUC	:	1H
OBFRQ	:	395.75 MHz
OBSET	:	134498.00 Hz
RGAIN	:	25
SCANS	:	32 times
SLVNT	:	CDCL3
SPINNING	:	19 Hz
TEMP	:	19.9 C



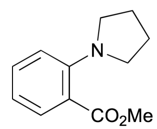
3al

1H Line 070619-13-465-1-13C



Date : Mon Feb 12 01:43:45 2007

FileName	:	.LoadingFID.nmdata
Comment	:	1H Line
SliceHistory	:	
EXMODE	:	bcm
POINT	:	32768 points
SAMPO	:	32768 points
FREQU	:	26881.7 Hz
FILTR	:	13450 Hz
DELAY	:	14.9 usec
DEADT	:	19.1 usec
INTVL	:	37.2 usec
TIMES	:	20000 times
DUMMY	:	4 times
PD	:	1.7810 sec
ACQTM	:	1218.9696 msec
PREDL	:	0.01000 msec
INIWT	:	1000.0000 msec
RESOL	:	0.82 Hz
PW1	:	6.50 usec
OBNUC	:	13C
OBFRQ	:	99.45 MHz
OBSET	:	104750.00 Hz
RGAIN	:	31
IRNUC	:	1H
IRFRQ	:	395.75 MHz
IRSET	:	134498.00 Hz
IRRPW	:	45.0 usec
IRRNS	:	0
SCANS	:	12046 times
SLVNT	:	CDCL3
SPINNING	:	19 Hz
TEMP	:	20.9 C

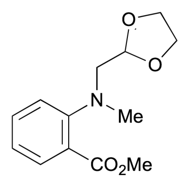


3al

1H Line 070709-14-483-3

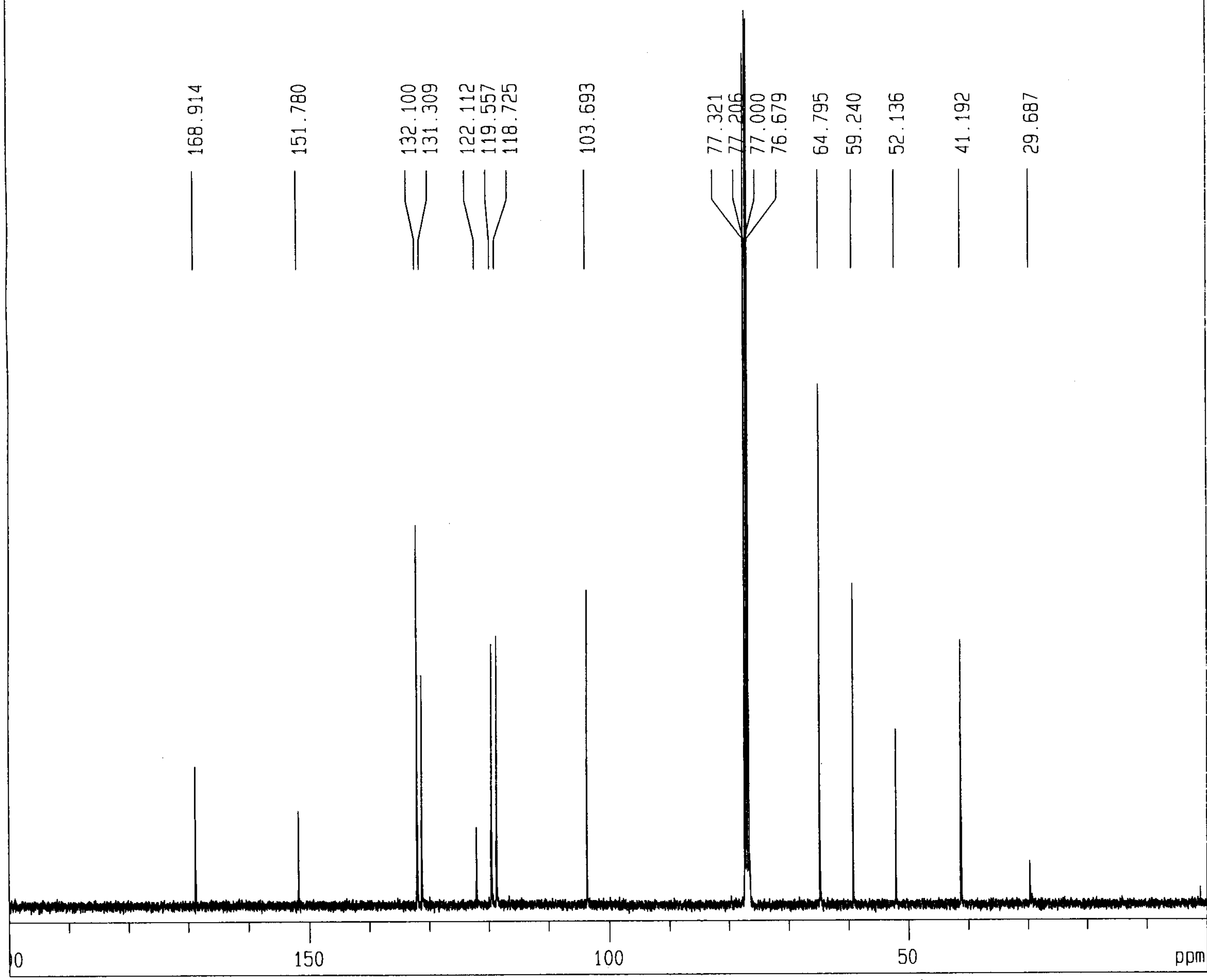
Date : Fri Jul 6 11:23:38 2007

FileName	:	.LoadingFID.nmdata
Comment	:	1H Line
SliceHistory	:	
EXMODE	:	non
POINT	:	32768 points
SAMPO	:	32768 points
FREQU	:	7917.7 Hz
FILTR	:	3950 Hz
DELAY	:	50.6 usec
DEADT	:	73.4 usec
INTVL	:	126.3 usec
TIMES	:	16 times
DUMMY	:	1 times
PD	:	2.8614 sec
ACQTM	:	4138.5986 msec
PREDL	:	0.01000 msec
INIWT	:	1000.0000 msec
RESOL	:	0.24 Hz
PW1	:	5.05 usec
OBNUC	:	1H
OBFRQ	:	395.75 MHz
OBSET	:	134498.00 Hz
RGAIN	:	28
SCANS	:	16 times
SLVNT	:	CDCL3
SPINNING	:	19 Hz
TEMP	:	20.1 C

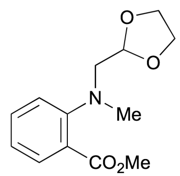


3am

1H Line 070710-14-483-3-13C

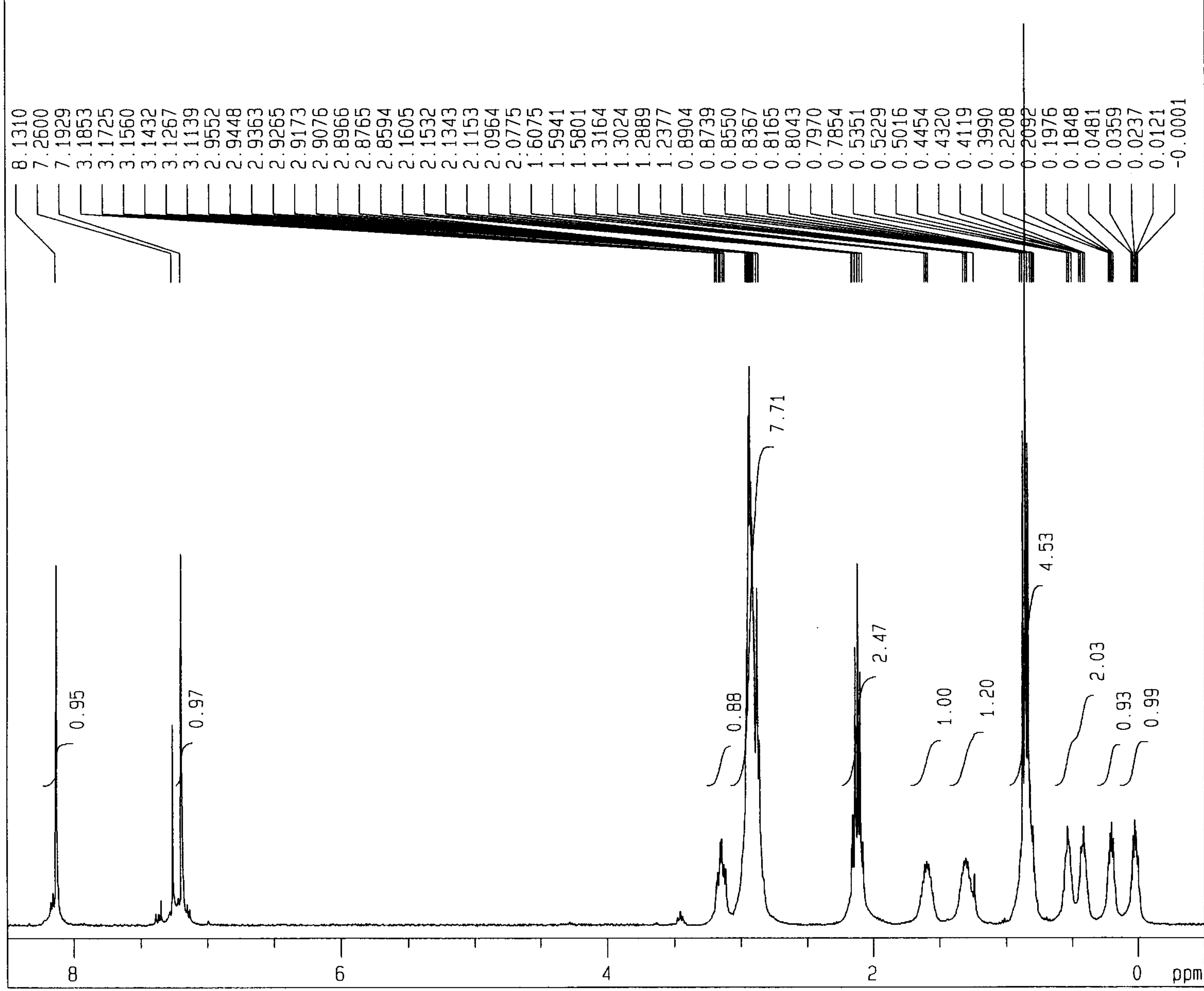


Date	:	Mon Feb 12 01:43:45 2007
FileName	:	.LoadingFID.nmdata
Comment	:	1H Line
SliceHistory	:	
EXMODE	:	bcm
POINT	:	32768 points
SAMPO	:	32768 points
FREQU	:	26881.7 Hz
FILTR	:	13450 Hz
DELAY	:	14.9 usec
DEADT	:	19.1 usec
INTVL	:	37.2 usec
TIMES	:	20000 times
DUMMY	:	4 times
PD	:	1.7810 sec
ACQTM	:	1218.9696 msec
PREDL	:	0.01000 msec
INIWT	:	1000.0000 msec
RESOL	:	0.82 Hz
PW1	:	6.50 usec
OBNUC	:	13C
OBFRQ	:	99.45 MHz
OBSET	:	104750.00 Hz
RGAIN	:	28
IRNUC	:	1H
IRFRQ	:	395.75 MHz
IRSET	:	134498.00 Hz
IRRPW	:	45.0 usec
IRRNS	:	0
SCANS	:	9659 times
SLVNT	:	CDCL3
SPINNING	:	19 Hz
TEMP	:	20.9 C



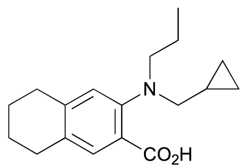
3am

1H Line 070929-15-549-2



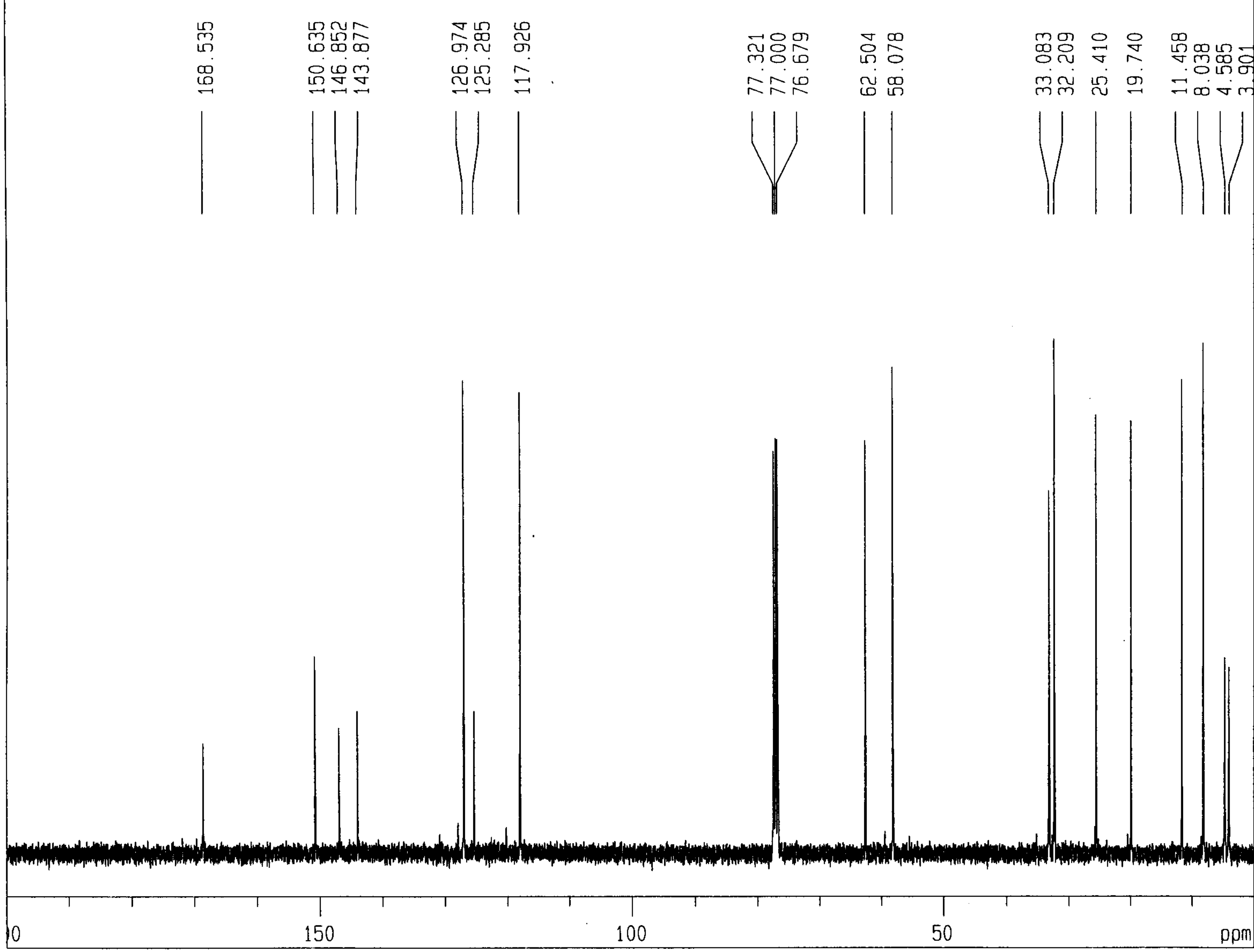
Date : Wed Sep 26 06:27:51 2007

FileName	:	.LoadingFID.nmdata
Comment	:	1H Line
SliceHistory	:	
EXMODE	:	non
POINT	:	32768 points
SAMPO	:	32768 points
FREQU	:	7917.7 Hz
FILTR	:	3950 Hz
DELAY	:	50.6 usec
DEADT	:	73.4 usec
INTVL	:	126.3 usec
TIMES	:	16 times
DUMMY	:	1 times
PD	:	2.8614 sec
ACQTM	:	4138.5986 msec
PREDL	:	0.01000 msec
INIWT	:	1000.0000 msec
RESOL	:	0.24 Hz
PW1	:	5.05 usec
OBNUC	:	1H
OBFRQ	:	395.75 MHz
OBSET	:	134498.00 Hz
RGAIN	:	23
SCANS	:	16 times
SLVNT	:	CDCL3
SPINNING	:	16 Hz
TEMP	:	20.3 C



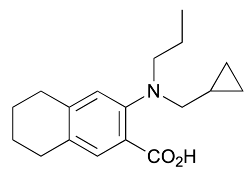
3bh

1H Line 09/002-15-549-2-13C

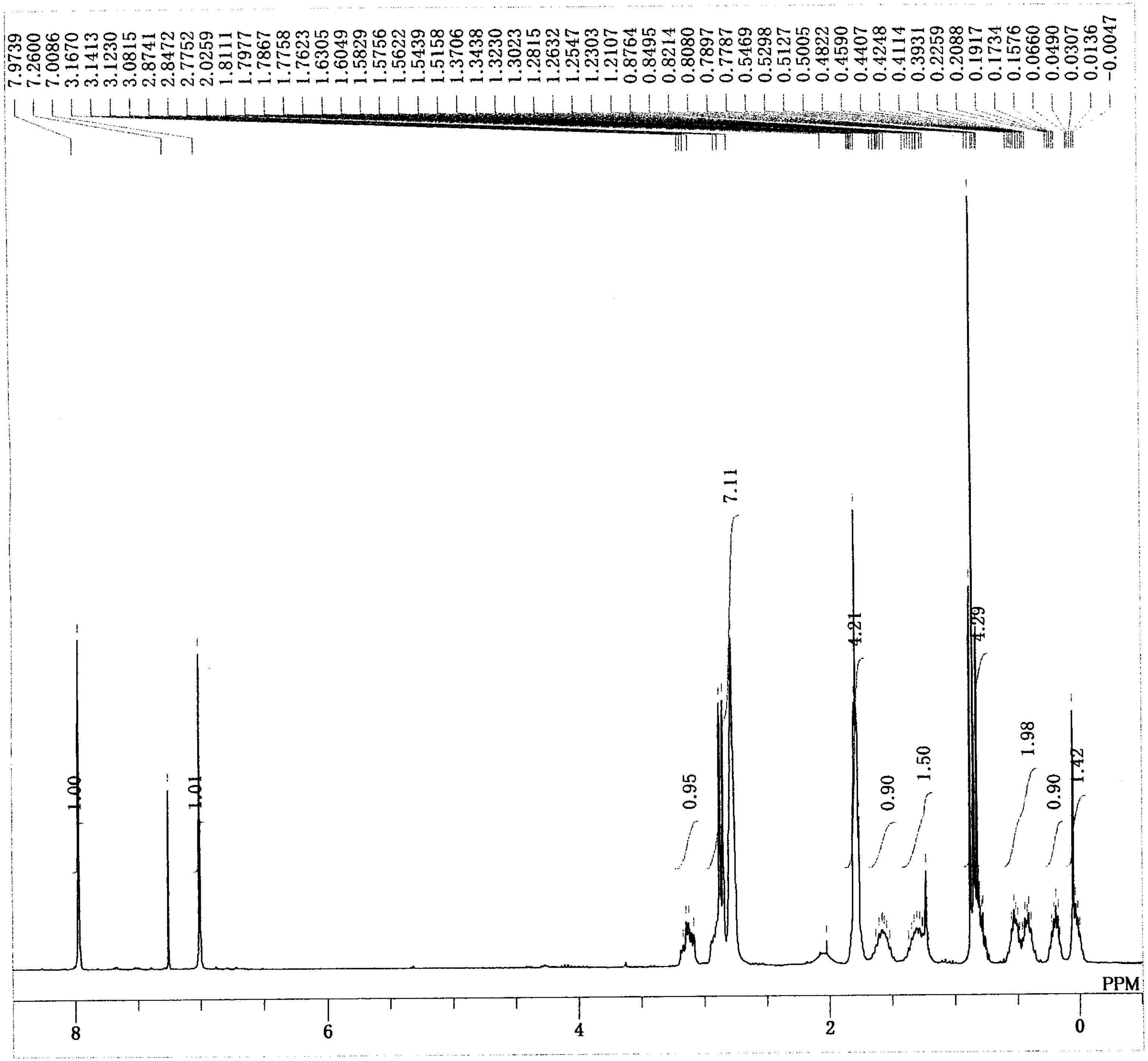


Date : Mon Feb 12 01:43:45 2007

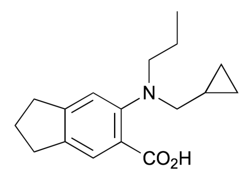
FileName	:	.LoadingFID.nmdata
Comment	:	1H Line
SliceHistory	:	
EXMODE	:	bcm
POINT	:	32768 points
SAMPO	:	32768 points
FREQU	:	26881.7 Hz
FILTR	:	13450 Hz
DELAY	:	14.9 usec
DEADT	:	19.1 usec
INTVL	:	37.2 usec
TIMES	:	20000 times
DUMMY	:	4 times
PD	:	1.7810 sec
ACQTM	:	1218.9696 msec
PREDL	:	0.01000 msec
INIWT	:	1000.0000 msec
RESOL	:	0.82 Hz
PW1	:	6.50 usec
OBNUC	:	13C
OBFRQ	:	99.45 MHz
OBSET	:	104750.00 Hz
RGAIN	:	29
IRNUC	:	1H
IRFRQ	:	395.75 MHz
IRSET	:	134498.00 Hz
IRRPW	:	45.0 usec
IRRNS	:	0
SCANS	:	1815 times
SLVNT	:	CDCL3
SPINNING	:	17 Hz
TEMP	:	21.6 C



3bh

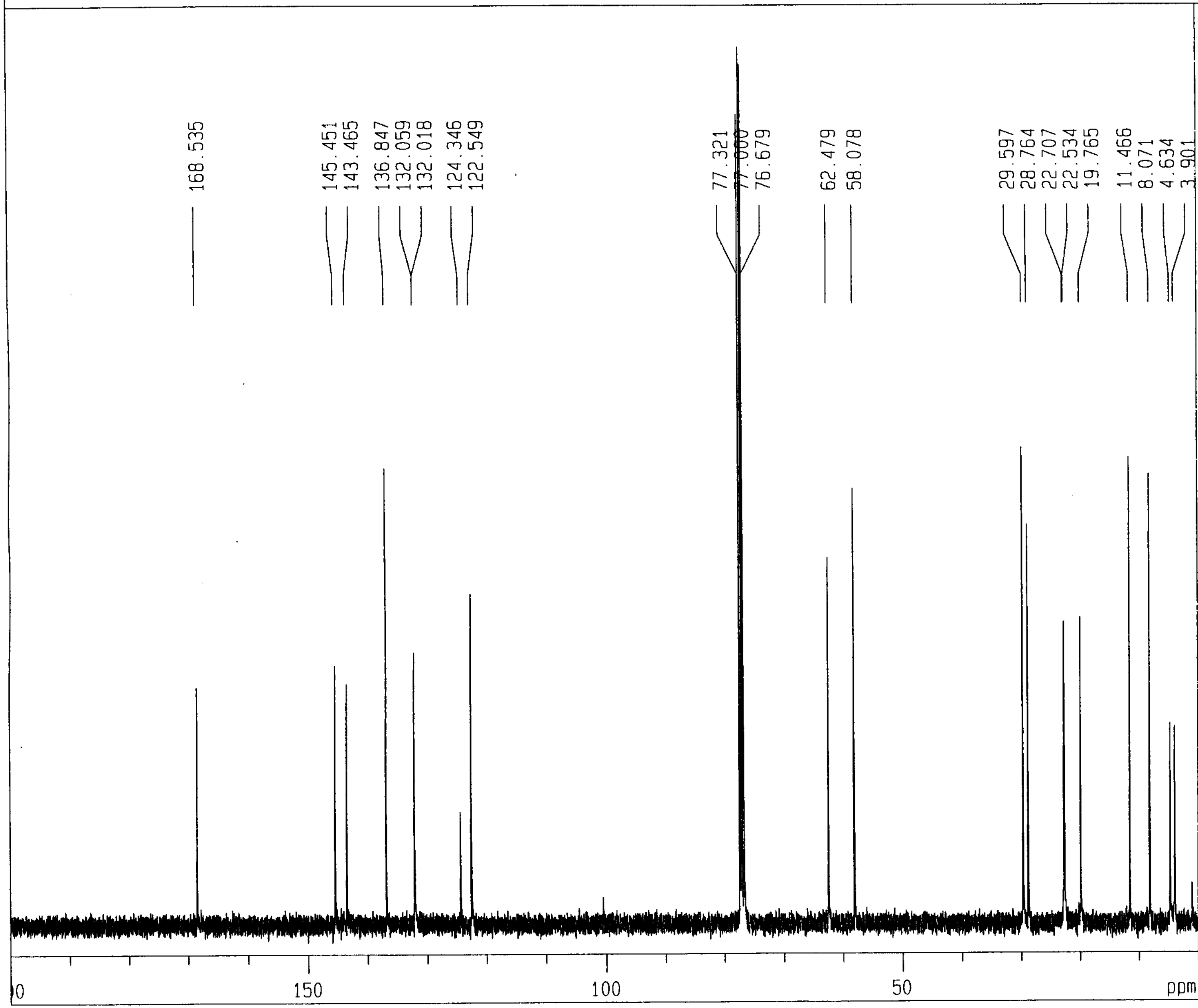


DFILE	_DEFAULT.ALS
COMNT	
DATIM	Fri Sep 14 09:32:45 2007
OBNUC	1H
EXMOD	NON
OBFRQ	270.05 MHz
OBSET	112.00 KHz
OBFIN	5800.00 Hz
POINT	16384
FREQU	5401.76 Hz
SCANS	16
ACQTM	3.0331 sec
PD	3.9670 sec
PW1	5.40 usec
IRNUC	1H
CTEMP	17.7 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.12 Hz
RGAIN	15

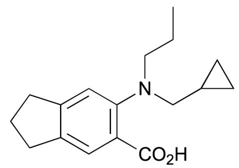


3ch

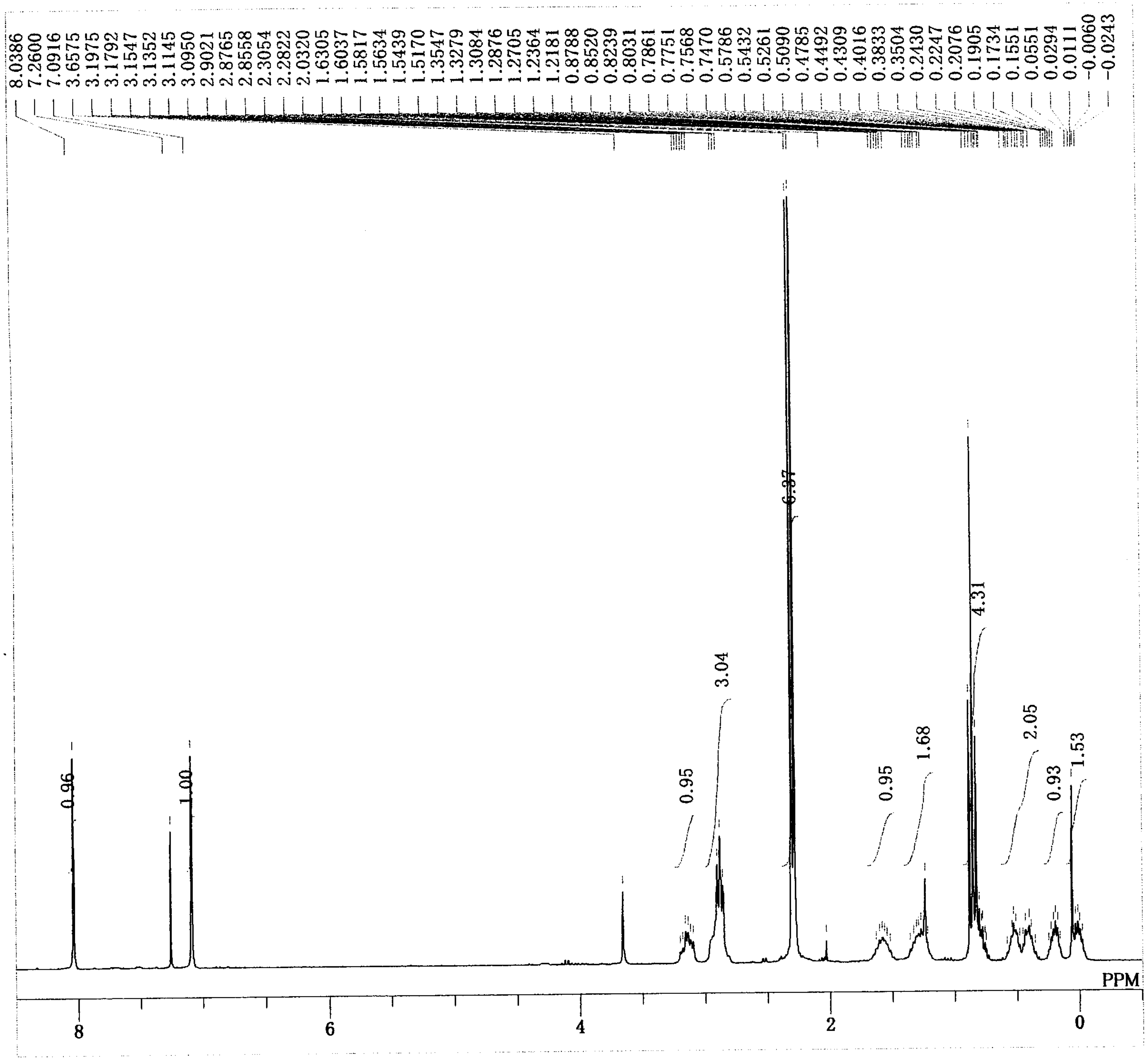
1H Line 070911-15-529-2-13C



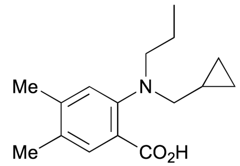
Date	:	Sat Sep 8 22:21:41 2007
FileName	:	.LoadingFID.nmdata
Comment	:	1H Line
SliceHistory	:	
EXMODE	:	bcm
POINT	:	32768 points
SAMPO	:	32768 points
FREQU	:	26881.7 Hz
FILTR	:	13450 Hz
DELAY	:	14.9 usec
DEADT	:	19.1 usec
INTVL	:	37.2 usec
TIMES	:	20000 times
DUMMY	:	4 times
PD	:	1.7810 sec
ACQTM	:	1218.9696 msec
PREDL	:	0.01000 msec
INIWT	:	1000.0000 msec
RESOL	:	0.82 Hz
PW1	:	6.50 usec
OBNUC	:	13C
OBFRQ	:	99.45 MHz
OBSET	:	104750.00 Hz
RGAIN	:	28
IRNUC	:	1H
IRFRQ	:	395.75 MHz
IRSET	:	134498.00 Hz
IRRPW	:	45.0 usec
IRRNS	:	0
SCANS	:	2352 times
SLVNT	:	CDCL3
SPINNING	:	18 Hz
TEMP	:	21.0 C



3ch

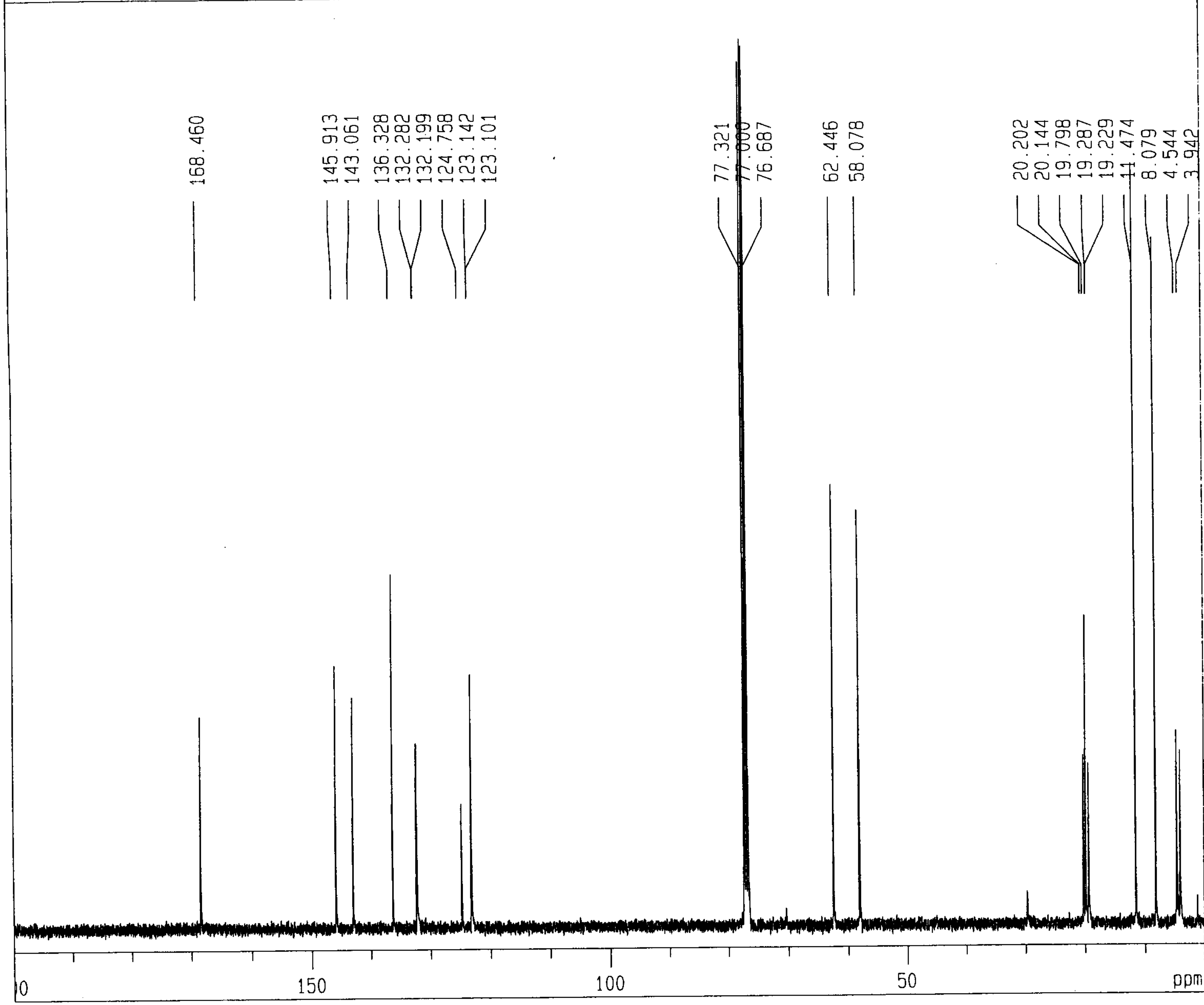


DFILE	_DEFAULT.ALS
COMNT	
DATIM	Mon Sep 10 14:07:43 2007
OBNUC	1H
EXMOD	NON
OBFRQ	270.05 MHz
OBSET	112.00 KHz
OBFIN	5800.00 Hz
POINT	16384
FREQU	5401.76 Hz
SCANS	16
ACQTM	3.0331 sec
PD	3.9670 sec
PW1	5.40 usec
IRNUC	1H
CTEMP	17.7 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.12 Hz
RGAIN	17



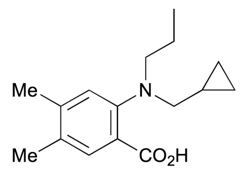
3dh

1H Line 070911-15-528-2-13C



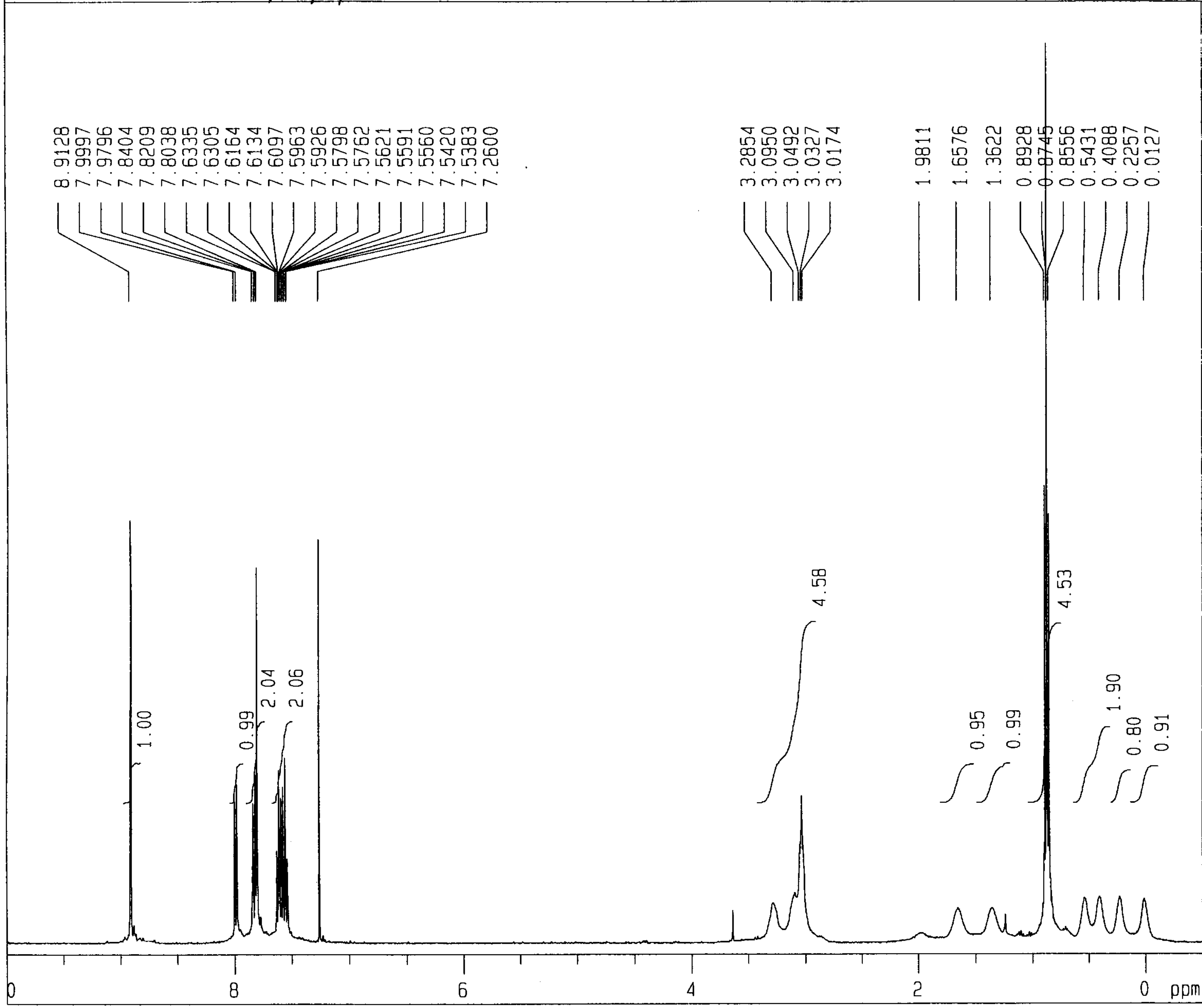
Date : Sat Sep 8 20:19:43 2007

FileName	:	.LoadingFID.nmdata
Comment	:	1H Line
SliceHistory	:	
EXMODE	:	bcm
POINT	:	32768 points
SAMPO	:	32768 points
FREQU	:	26881.7 Hz
FILTR	:	13450 Hz
DELAY	:	14.9 usec
DEADT	:	19.1 usec
INTVL	:	37.2 usec
TIMES	:	20000 times
DUMMY	:	4 times
PD	:	1.7810 sec
ACQTM	:	1218.9696 msec
PREDL	:	0.01000 msec
INIWT	:	1000.0000 msec
RESOL	:	0.82 Hz
PW1	:	6.50 usec
OBNUC	:	13C
OBFRQ	:	99.45 MHz
OBSET	:	104750.00 Hz
RGAIN	:	28
IRNUC	:	1H
IRFRQ	:	395.75 MHz
IRSET	:	134498.00 Hz
IRRPW	:	45.0 usec
IRRNS	:	0
SCANS	:	10329 times
SLVNT	:	CDCL3
SPINNING	:	17 Hz
TEMP	:	20.8 C



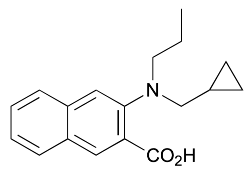
3dh

1H Line 070915-15-540-2



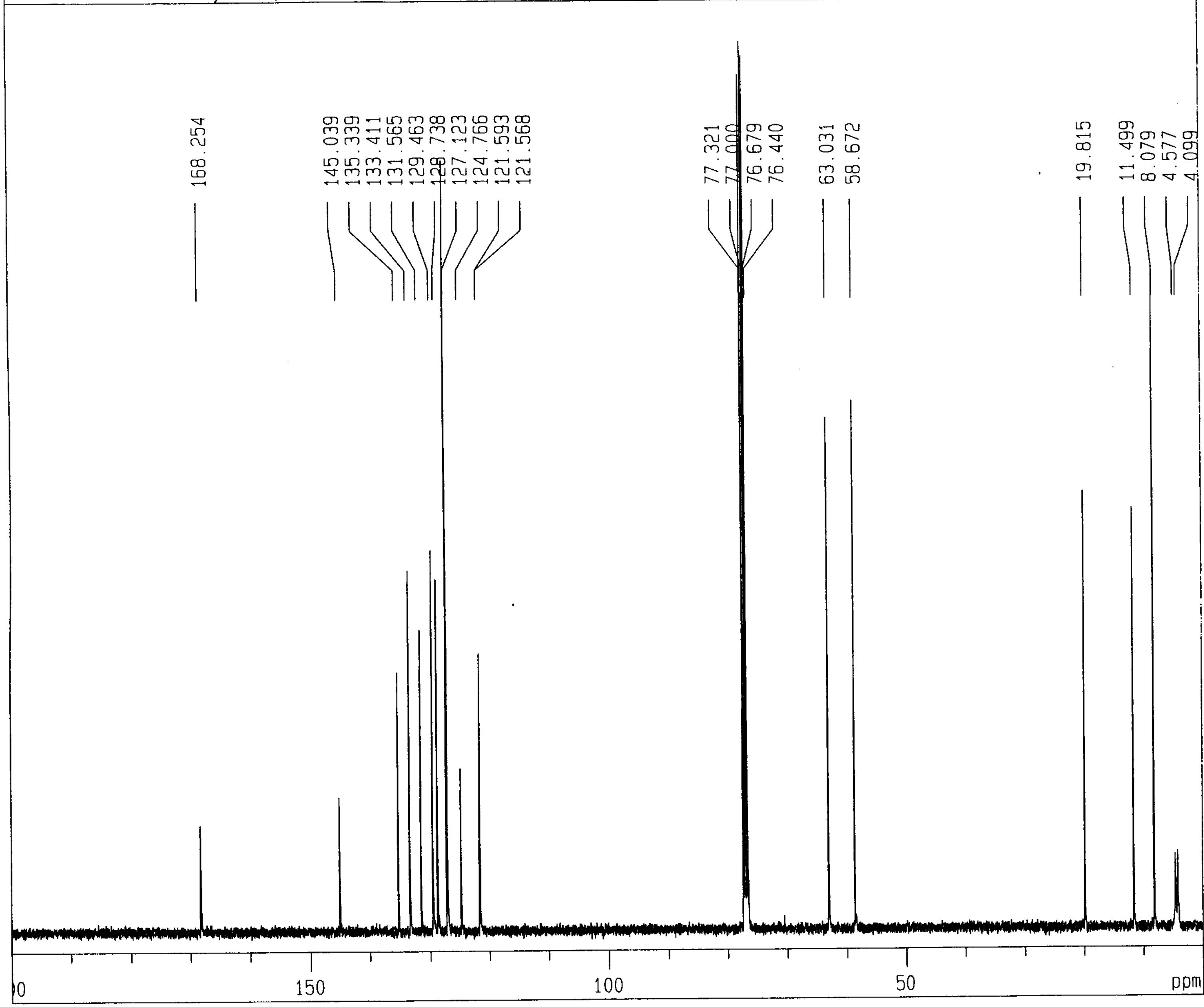
Date : Sat Sep 15 12:02:52 2007

FileName	:	.LoadingFID.nmdata
Comment	:	1H Line
SliceHistory	:	
EXMODE	:	non
POINT	:	32768 points
SAMPO	:	32768 points
FREQU	:	7917.7 Hz
FILTR	:	3950 Hz
DELAY	:	50.6 usec
DEADT	:	73.4 usec
INTVL	:	126.3 usec
TIMES	:	16 times
DUMMY	:	1 times
PD	:	2.8614 sec
ACQTM	:	4138.5986 msec
PREDL	:	0.01000 msec
INIWT	:	1000.0000 msec
RESOL	:	0.24 Hz
PW1	:	5.05 usec
OBNUC	:	1H
OBFRQ	:	395.75 MHz
OBSET	:	134498.00 Hz
RGAIN	:	22
SCANS	:	16 times
SLVNT	:	CDCL3
SPINNING	:	17 Hz
TEMP	:	20.5 C



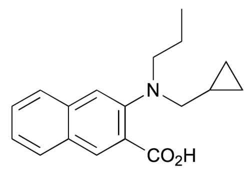
3eh

1H Line 070918-15-540-2-13C



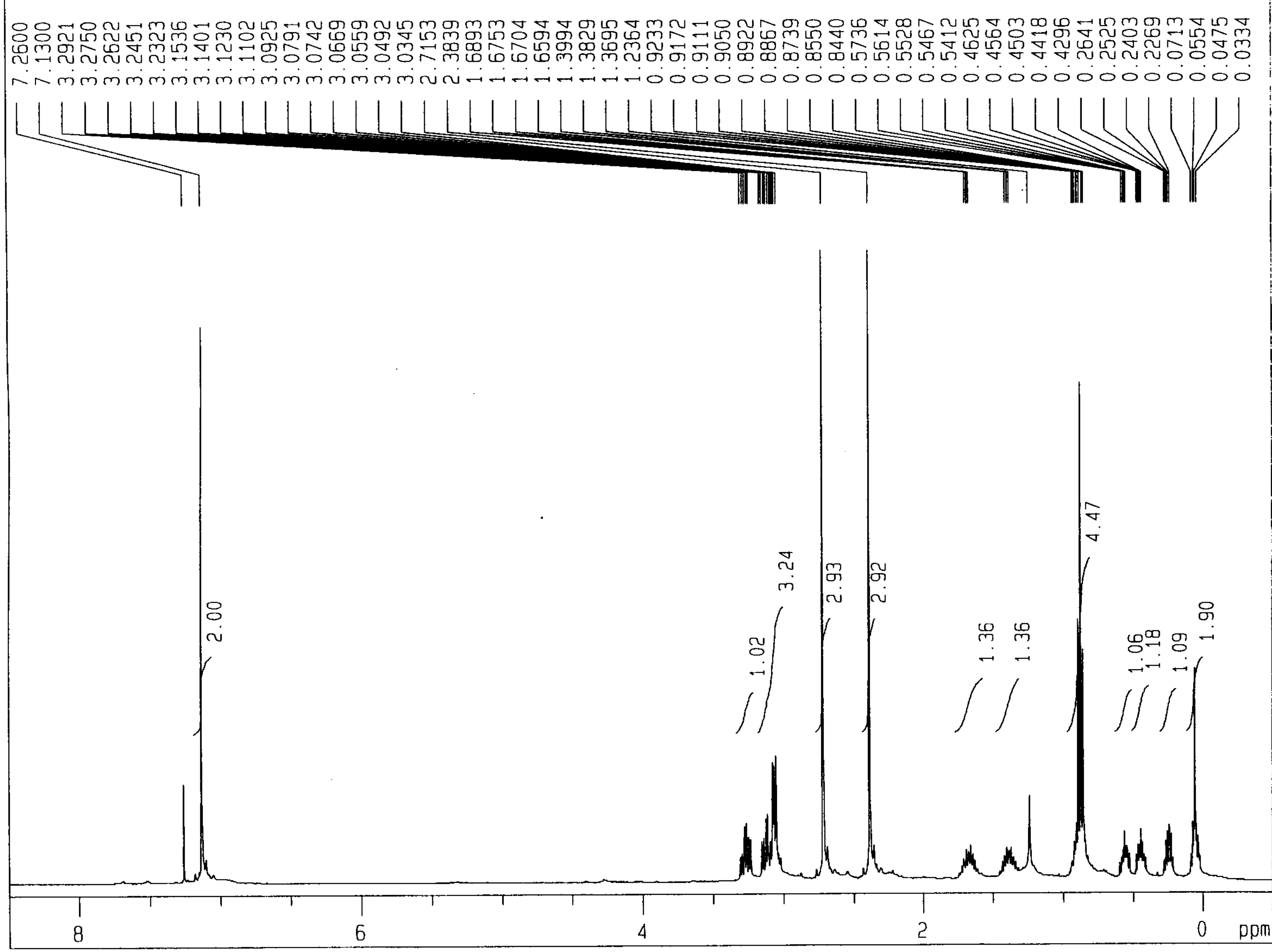
Date : Sat Sep 15 21:53:47 2007

FileName	:	.LoadingFID.nmdata
Comment	:	1H Line
SliceHistory	:	
EXMODE	:	bcm
POINT	:	32768 points
SAMPO	:	32768 points
FREQU	:	26881.7 Hz
FILTR	:	13450 Hz
DELAY	:	14.9 usec
DEADT	:	19.1 usec
INTVL	:	37.2 usec
TIMES	:	20000 times
DUMMY	:	4 times
PD	:	1.7810 sec
ACQTM	:	1218.9696 msec
PREDL	:	0.01000 msec
INIWT	:	1000.0000 msec
RESOL	:	0.82 Hz
PW1	:	6.50 usec
OBNUC	:	13C
OBFRQ	:	99.45 MHz
OBSET	:	104750.00 Hz
RGAIN	:	28
IRNUC	:	1H
IRFRQ	:	395.75 MHz
IRSET	:	134498.00 Hz
IRRPW	:	45.0 usec
IRRNS	:	0
SCANS	:	11755 times
SLVNT	:	CDCL3
SPINNING	:	20 Hz
TEMP	:	20.8 C



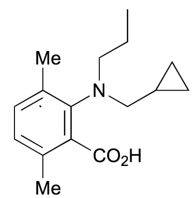
3eh

1H Line 070920-15-545-1



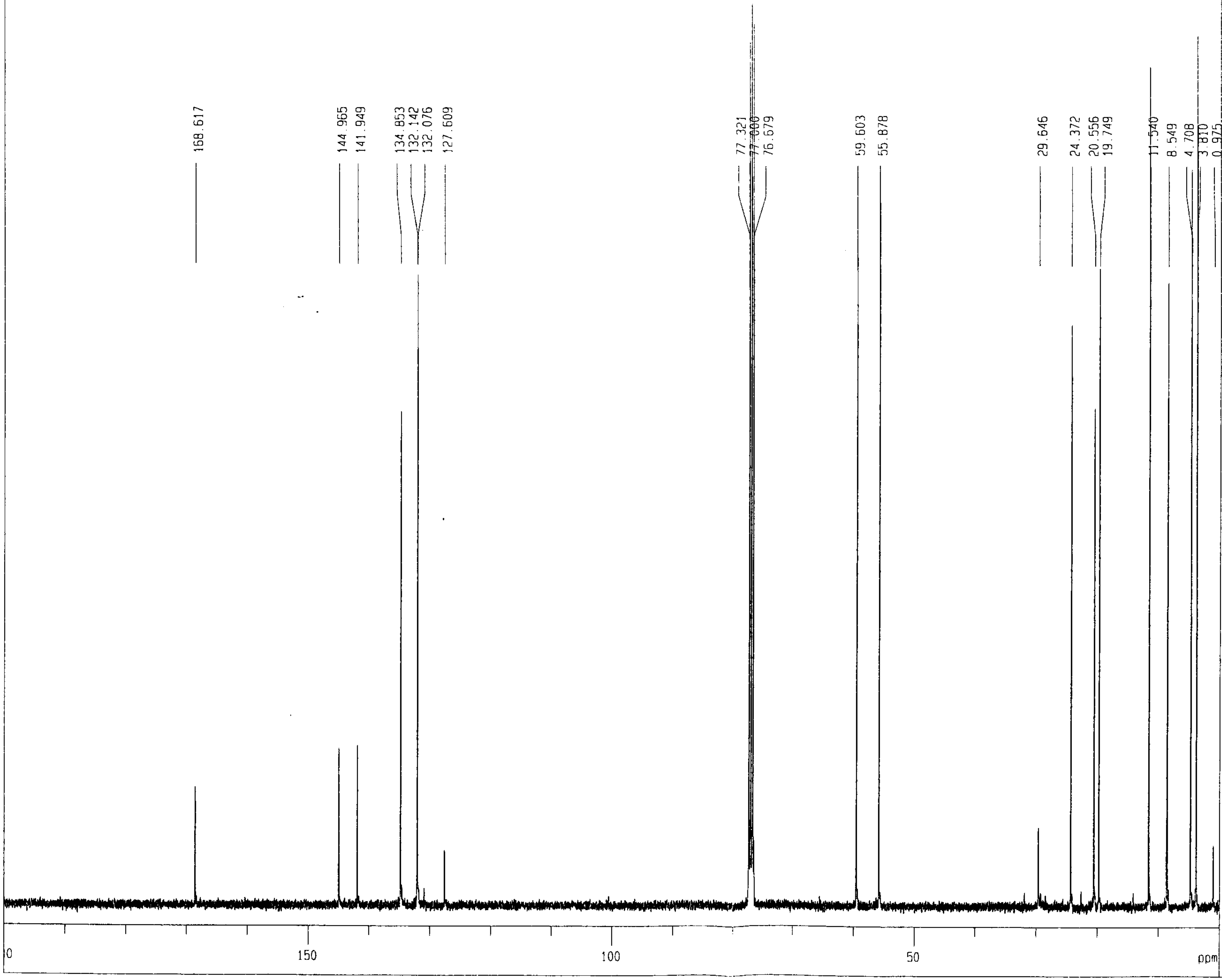
Date : Sun Sep 16 22:21:02 2007

FileName	:	.LoadingFID.nmdata
Comment	:	1H Line
SliceHistory	:	
EXMODE	:	non
POINT	:	32768 points
SAMPO	:	32768 points
FREQU	:	7917.7 Hz
FILTR	:	3950 Hz
DELAY	:	50.6 usec
DEADT	:	73.4 usec
INTVL	:	126.3 usec
TIMES	:	16 times
DUMMY	:	1 times
PD	:	2.8614 sec
ACQTM	:	4138.5986 msec
PREDL	:	0.01000 msec
INIWT	:	1000.0000 msec
RESOL	:	0.24 Hz
PW1	:	5.05 usec
OBNUC	:	1H
OBFRQ	:	395.75 MHz
OBSET	:	134498.00 Hz
RGAIN	:	18
SCANS	:	16 times
SLVNT	:	CDCL3
SPINNING	:	18 Hz
TEMP	:	20.3 C

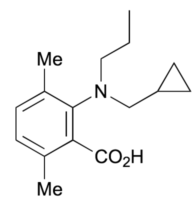


3fh

1H Line 070920-15-545-1-13C

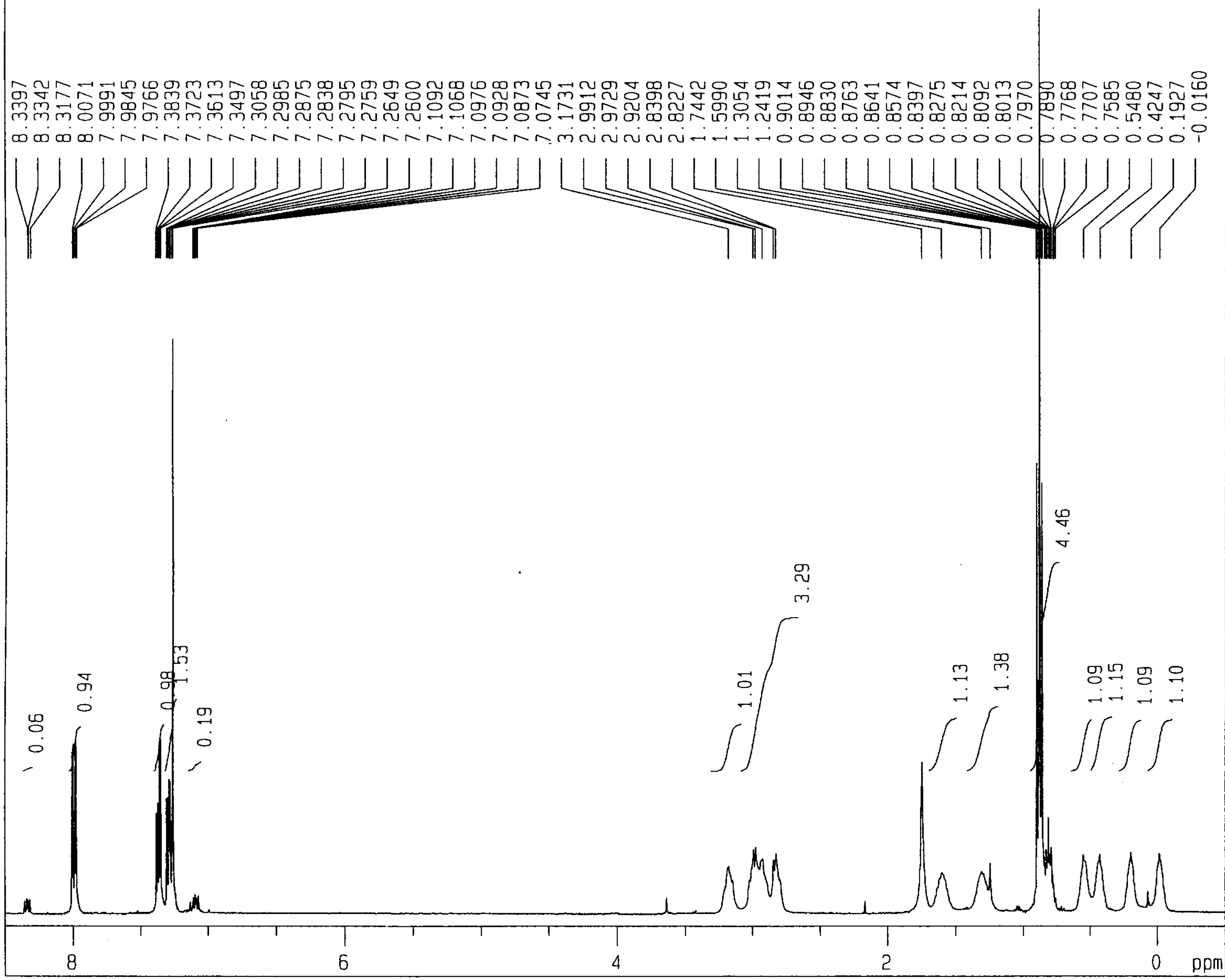


Date	:	Mon Sep 17 21:57:08 2007
FileName	:	.LoadingFID.nmdata
Comment	:	1H Line
SliceHistory	:	
EXMODE	:	bcm
POINT	:	32768 points
SAMPO	:	32768 points
FREQU	:	26881.7 Hz
FILTR	:	13450 Hz
DELAY	:	14.9 usec
DEADT	:	19.1 usec
INTVL	:	37.2 usec
TIMES	:	20000 times
DUMMY	:	4 times
PD	:	1.7810 sec
ACQTM	:	1218.9696 msec
PREDL	:	0.01000 msec
INIWT	:	1000.0000 msec
RESOL	:	0.82 Hz
PW1	:	6.50 usec
OBNUC	:	13C
OBFRQ	:	99.45 MHz
OBSET	:	104750.00 Hz
RGAIN	:	28
IRNUC	:	1H
IRFRQ	:	395.75 MHz
IRSET	:	134498.00 Hz
IRRPW	:	45.0 usec
IRRNS	:	0
SCANS	:	12438 times
SLVNT	:	CDCL3
SPINNING	:	18 Hz
TEMP	:	21.8 C



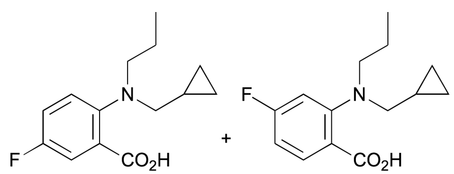
3fh

1H Line 07002-15-551-#2



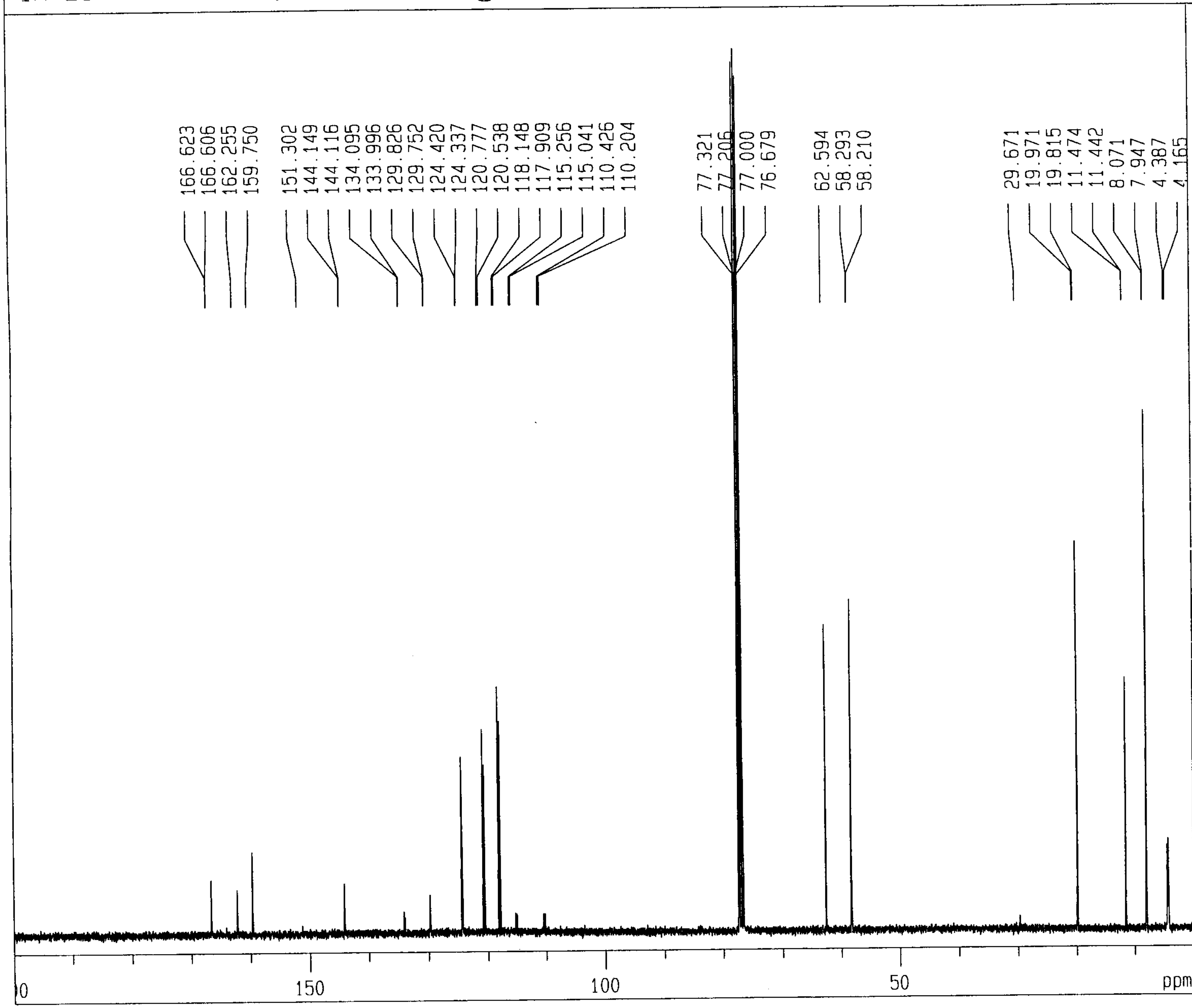
Date : Fri Sep 28 22:38:50 2007

FileName	:	.LoadingFID.nmdata
Comment	:	1H Line
SliceHistory	:	
EXMODE	:	non
POINT	:	32768 points
SAMPO	:	32768 points
FREQU	:	7917.7 Hz
FILTR	:	3950 Hz
DELAY	:	50.6 usec
DEADT	:	73.4 usec
INTVL	:	126.3 usec
TIMES	:	16 times
DUMMY	:	1 times
PD	:	2.8614 sec
ACQTM	:	4138.5986 msec
PREDL	:	0.01000 msec
INIWT	:	1000.0000 msec
RESOL	:	0.24 Hz
PW1	:	5.05 usec
OBNUC	:	1H
OBFRQ	:	395.75 MHz
OBSET	:	134498.00 Hz
RGAIN	:	22
SCANS	:	16 times
SLVNT	:	CDCL3
SPINNING	:	13 Hz
TEMP	:	19.9 C



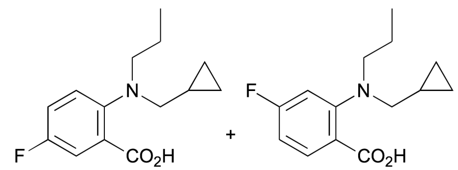
3gh 3'gh

1H Line 071003-15-551-2-^{13}C



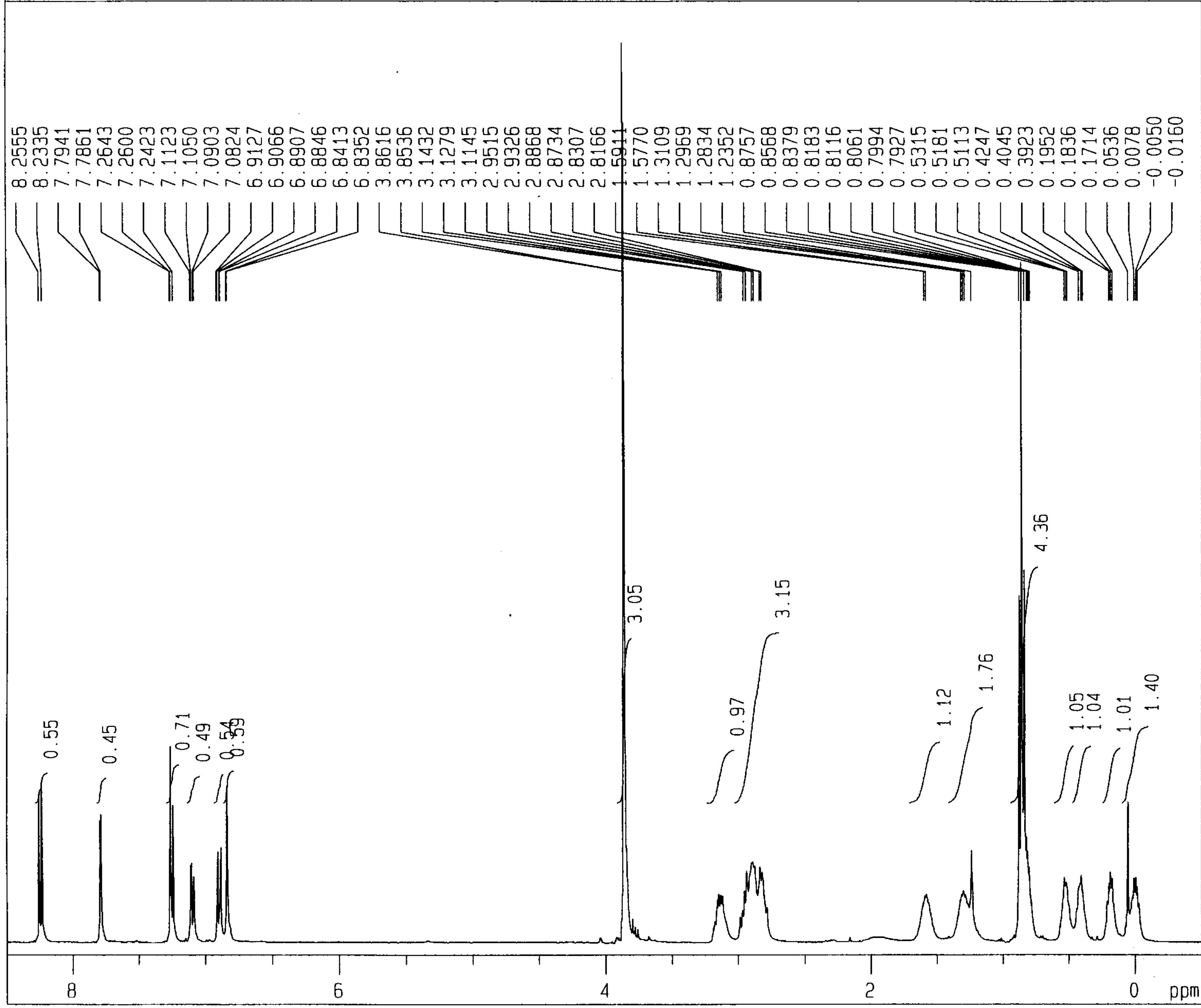
Date : Mon Feb 12 01:43:45 2007

FileName	:	.LoadingFID.nmdata
Comment	:	1H Line
SliceHistory	:	
EXMODE	:	bcm
POINT	:	32768 points
SAMPO	:	32768 points
FREQU	:	26881.7 Hz
FILTR	:	13450 Hz
DELAY	:	14.9 usec
DEADT	:	19.1 usec
INTVL	:	37.2 usec
TIMES	:	20000 times
DUMMY	:	4 times
PD	:	1.7810 sec
ACQTM	:	1218.9696 msec
PREDL	:	0.01000 msec
INIWT	:	1000.0000 msec
RESOL	:	0.82 Hz
PW1	:	6.50 usec
OBNUC	:	13C
OBFRQ	:	99.45 MHz
OBSET	:	104750.00 Hz
RGAIN	:	28
IRNUC	:	1H
IRFRQ	:	395.75 MHz
IRSET	:	134498.00 Hz
IRRPW	:	45.0 usec
IRRNS	:	0
SCANS	:	10533 times
SLVNT	:	CDCL3
SPINNING	:	19 Hz
TEMP	:	21.3 C



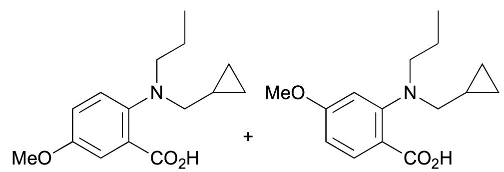
3gh 3'gh

1H Line 071002-15-555-1

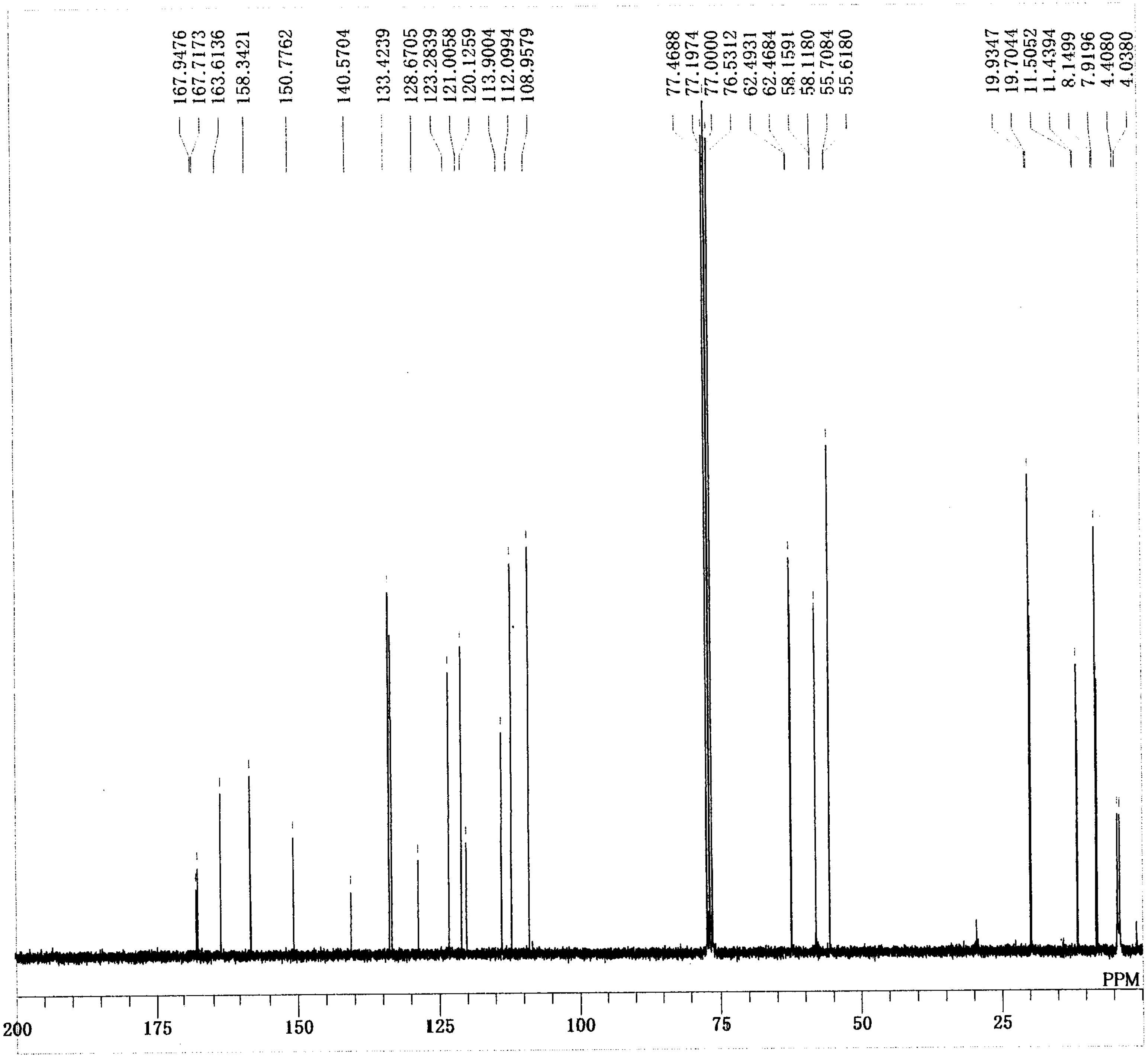


Date : Fri Sep 28 22:30:47 2007

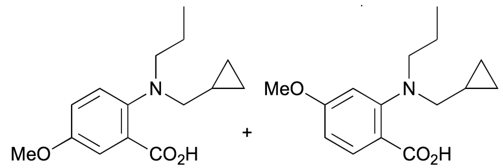
FileName	:	.LoadingFID.nmdata
Comment	:	1H Line
SliceHistory	:	
EXMODE	:	non
POINT	:	32768 points
SAMPO	:	32768 points
FREQU	:	7917.7 Hz
FILTR	:	3950 Hz
DELAY	:	50.6 usec
DEADT	:	73.4 usec
INTVL	:	126.3 usec
TIMES	:	16 times
DUMMY	:	1 times
PD	:	2.8614 sec
ACQTM	:	4138.5986 msec
PREDL	:	0.01000 msec
INIWT	:	1000.0000 msec
RESOL	:	0.24 Hz
PW1	:	5.05 usec
OBNUC	:	1H
OBFRQ	:	395.75 MHz
OBSET	:	134498.00 Hz
RGAIN	:	19
SCANS	:	16 times
SLVNT	:	CDCL3
SPINNING	:	14 Hz
TEMP	:	19.9 C



3hh 3'hh

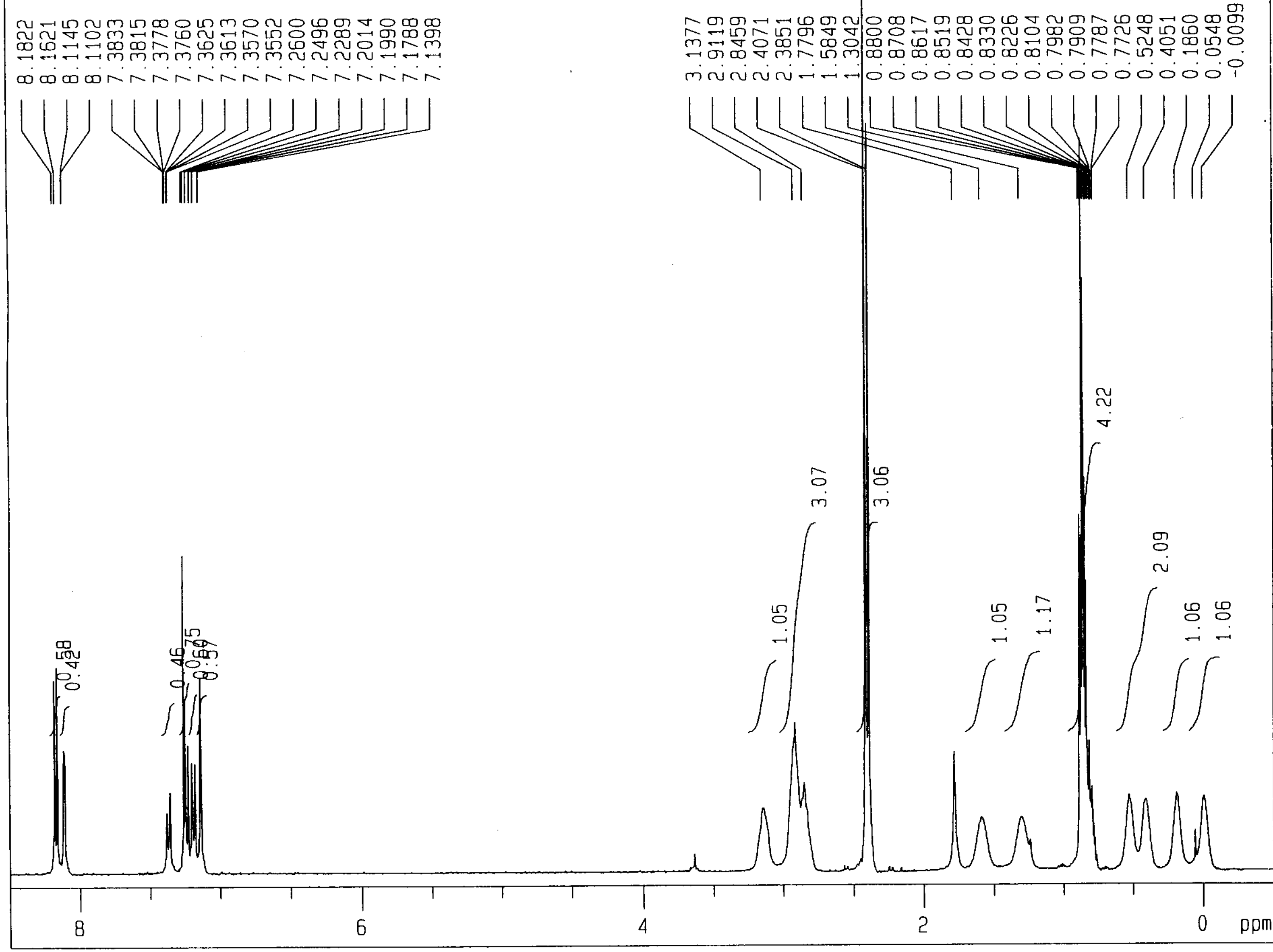


DFILE	_DEFAULT.ALS
COMNT	
DATIM	Thu Oct 04 08:48:16 2007
OBNUC	13C
EXMOD	BCM
OBFRQ	67.80 MHz
OBSET	135.00 KHz
OBFIN	5200.00 Hz
POINT	32768
FREQU	18306.64 Hz
SCANS	11332
ACQTM	1.7900 sec
PD	1.2100 sec
PW1	3.50 usec
IRNUC	1H
CTEMP	22.8 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	27



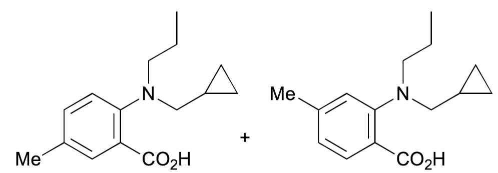
3hh 3'hh

1H Line 071005-15-548-2

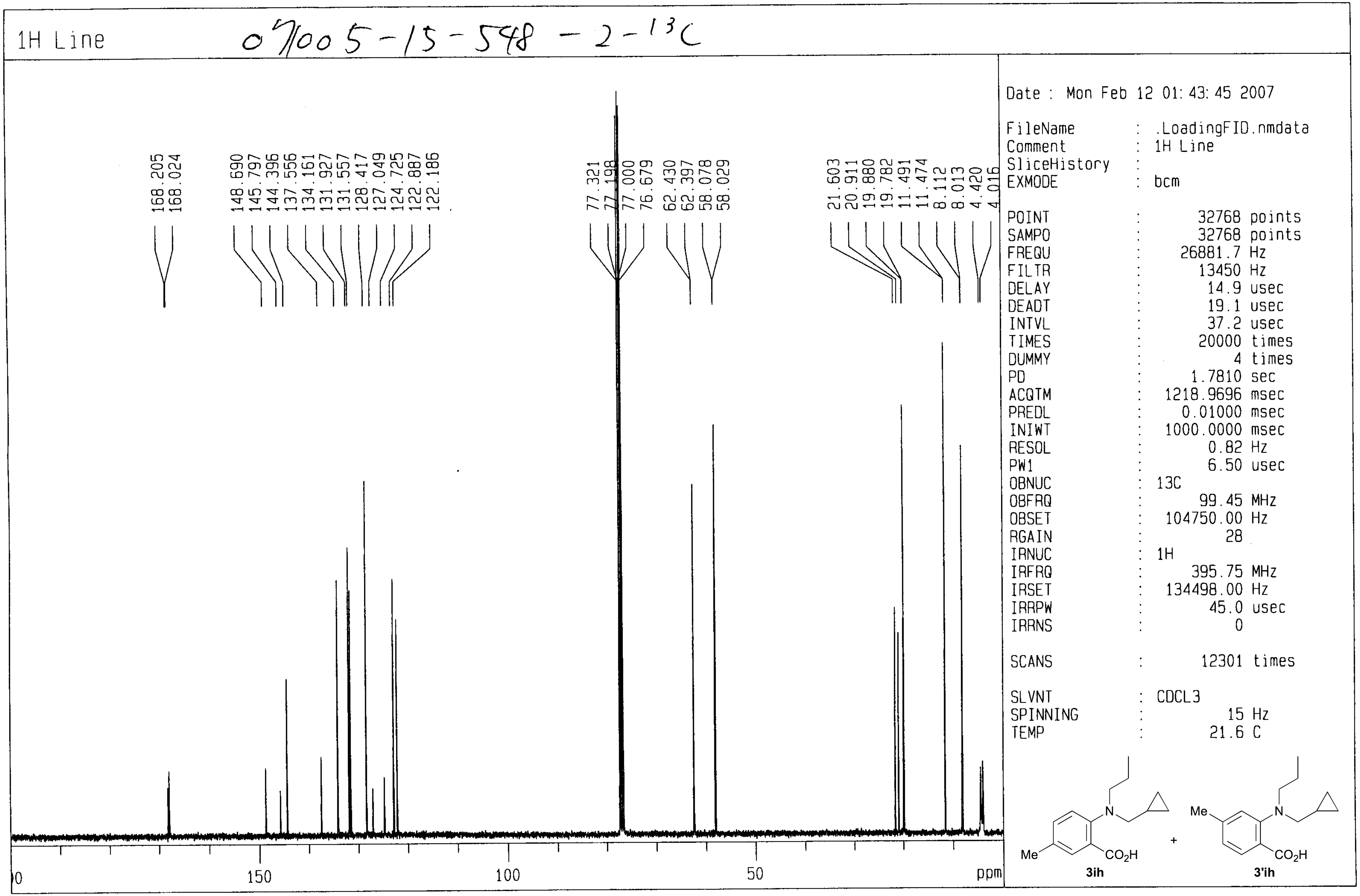


Date : Tue Oct 2 21:32:57 2007

FileName	:	.LoadingFID.nmdata
Comment	:	1H Line
SliceHistory	:	
EXMODE	:	non
POINT	:	32768 points
SAMPO	:	32768 points
FREQU	:	7917.7 Hz
FILTR	:	3950 Hz
DELAY	:	50.6 usec
DEADT	:	73.4 usec
INTVL	:	126.3 usec
TIMES	:	16 times
DUMMY	:	1 times
PD	:	2.8614 sec
ACQTM	:	4138.5986 msec
PREDL	:	0.01000 msec
INIWT	:	1000.0000 msec
RESOL	:	0.24 Hz
PW1	:	5.05 usec
OBNUC	:	1H
OBFRQ	:	395.75 MHz
OBSET	:	134498.00 Hz
RGAIN	:	20
SCANS	:	16 times
SLVNT	:	CDCL3
SPINNING	:	15 Hz
TEMP	:	20.9 C



3ih 3'ih


1H Line
07005-15-548-2-13C
168.205
168.024
148.690
145.797
144.396
137.556
134.161
131.927
131.557
128.417
127.049
124.725
122.887
122.186
77.321
77.198
77.000
76.679
62.430
62.397
58.078
58.029
21.603
20.911
19.880
19.782
11.491
11.474
8.112
8.013
4.420
4.016
150
100
50
ppm
Date : Mon Feb 12 01:43:45 2007
FileName : .LoadingFID.nmdata
Comment : 1H Line
SliceHistory :
EXMODE : bcm
POINT : 32768 points
SAMPO : 32768 points
FREQU : 26881.7 Hz
FILTR : 13450 Hz
DELAY : 14.9 usec
DEADT : 19.1 usec
INTVL : 37.2 usec
TIMES : 20000 times
DUMMY : 4 times
PD : 1.7810 sec
ACQTM : 1218.9696 msec
PREDL : 0.01000 msec
INIWT : 1000.0000 msec
RESOL : 0.82 Hz
PW1 : 6.50 usec
OBNUC : 13C
OBFRQ : 99.45 MHz
OBSET : 104750.00 Hz
RGAIN : 28
IRNUC : 1H
IRFRQ : 395.75 MHz
IRSET : 134498.00 Hz
IRRPW : 45.0 usec
IRRNS : 0
SCANS : 12301 times
SLVNT : CDCL3
SPINNING : 15 Hz
TEMP : 21.6 C
3ih
3'ih