

Electronic Supplementary Information

Benzyl radical addition reaction through the homolytic cleavage of a benzylic C–H bond

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General. NMR spectra were recorded at 300 MHz/75 MHz (^1H NMR/ ^{13}C NMR) or 500 MHz/125 MHz (^1H NMR/ ^{13}C NMR) using Varian Gemini-300 (300 MHz), Varian MERCURY plus 300 (300 MHz), or Varian NMR system AS 500 (500 MHz) spectrometers. Chemical shifts (δ) are reported as follows: chemical shift, multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, sext = sextet, m = multiplet, br = broad), coupling constants, and integration. IR spectra were obtained on a Perkin Elmer SpectrumOne A spectrometer. Mass spectra were obtained by EI, CI or ESI methods on a Hitachi M-4100 and Thermo Fisher Scientific Exactive. Melting points were recorded on Yanaco NP-S3 and are uncorrected. Preparative TLC separations (PTLC) were carried out on precoated silica gel plates (E. Merck 60F₂₅₄). Unless otherwise stated, all the reagents and solvents were used as received from the manufacturer.

Typical reaction procedure for radical addition to olefin **4**, **6** and **8**.

A mixture of olefin (0.35 mmol) and toluene derivatives (15 mL) was stirred at 180 °C for 0.5–3 h. The reaction mixture was concentrated in vacuo and purified by column chromatography on SiO₂ (AcOEt/hexane = 1/5) to afford the desired compounds.

2-(Phenyl)methylsuccinic Acid Dimethyl Ester (**5a**)

a colorless oil

IR ν_{max} (neat) cm^{-1} : 1736 (CO), 1437 (Ar).

^1H -NMR (300 MHz) δ : 2.41 (1H, dd, $J=16.5, 4.5$ Hz, $\text{CH}_2\text{CO}_2\text{Me}$), 2.68 (1H, dd, $J=16.5, 9.0$ Hz, $\text{CH}_2\text{CO}_2\text{Me}$), 2.76 (1H, dd, $J=13.5, 8.0$ Hz, CH_2Ar), 3.06 (1H, dd, $J=13.5, 7.0$ Hz, CH_2Ar), 3.11-3.18 (1H, m, 2-H), 3.64 (3H, s, CO_2CH_3), 3.67 (3H, s, CO_2CH_3), 7.14-7.32 (5H, m, Ar-H).

^{13}C -NMR (75 MHz) δ : 174.4, 172.0, 137.9, 128.8, 128.3, 126.5, 51.6, 51.4, 42.8, 37.5, 34.6.

HRMS m/z : Calcd for $\text{C}_{13}\text{H}_{17}\text{O}_4$ (M+H)⁺ 237.1121. Found : 237.11201.

2-[(4-Methoxyphenyl)methylsuccinic Acid Dimethyl Ester (**5b**)

a colorless oil

IR ν_{max} (neat) cm^{-1} : 1736 (CO), 1437 (Ar).

$^1\text{H-NMR}$ (300 MHz) δ : 2.40 (1H, dd, $J=17.0, 5.0$ Hz, $\text{CH}_2\text{CO}_2\text{Me}$), 2.67 (1H, dd, $J=17.0, 9.0$ Hz, $\text{CH}_2\text{CO}_2\text{Me}$), 2.70 (1H, dd, $J=13.5, 9.0$ Hz, CH_2Ar), 2.99 (1H, dd, $J=13.5, 6.0$ Hz, CH_2Ar), 3.04-3.14 (1H, m, 2-H), 3.64 (3H, s, CO_2CH_3), 3.67 (3H, s, CO_2CH_3), 3.79 (3H, s, Ar-OCH_3), 6.83 (2H, br d, $J=8.5$ Hz, Ar-H), 7.06 (2H, br d, $J=8.5$ Hz, Ar-H).

$^{13}\text{C-NMR}$ (75 MHz) δ : 174.7, 172.3, 158.4, 130.1, 130.0, 113.9, 55.2, 51.9, 51.7, 43.2, 36.9, 34.8.

HRMS m/z : Calcd for $\text{C}_{14}\text{H}_{19}\text{O}_5$ ($\text{M}+\text{H}$) $^+$ 267.1227. Found : 267.1227.

2-[(3-Methoxyphenyl)methylsuccinic Acid Dimethyl Ester (5c)]

a colorless oil

IR ν_{max} (neat) cm^{-1} : 1736 (CO), 1437 (Ar).

$^1\text{H-NMR}$ (300 MHz) δ : 2.41 (1H, dd, $J=17.0, 5.0$ Hz, $\text{CH}_2\text{CO}_2\text{Me}$), 2.67 (1H, dd, $J=17.0, 9.0$ Hz, $\text{CH}_2\text{CO}_2\text{Me}$), 2.72 (1H, dd, $J=13.5, 8.0$ Hz, CH_2Ar), 3.04 (1H, dd, $J=13.5, 6.0$ Hz, CH_2Ar), 3.08-3.16 (1H, m, 2-H), 3.64 (3H, s, CO_2CH_3), 3.68 (3H, s, CO_2CH_3), 3.79 (3H, s, Ar-OCH_3), 6.70-6.79 (3H, m, Ar-H), 7.20 (1H, t, $J=8.0$ Hz, Ar-H).

$^{13}\text{C-NMR}$ (75 MHz) δ : 174.7, 172.3, 159.8, 139.7, 129.6, 121.3, 114.7, 112.1, 55.2, 52.0, 51.8, 42.9, 37.7, 34.9.

HRMS m/z : Calcd for $\text{C}_{14}\text{H}_{18}\text{O}_5$ (M^+) 266.1153. Found : 266.1138.

2-[(2-Methoxyphenyl)methylsuccinic Acid Dimethyl Ester (5d)]

a colorless oil

IR ν_{max} (neat) cm^{-1} : 1736 (CO), 1438 (Ar).

$^1\text{H-NMR}$ (300 MHz) δ : 2.40 (1H, dd, $J=17.0, 5.0$ Hz, $\text{CH}_2\text{CO}_2\text{Me}$), 2.67 (1H, dd, $J=17.0, 9.0$ Hz, $\text{CH}_2\text{CO}_2\text{Me}$), 2.79 (1H, dd, $J=13.0, 8.0$ Hz, CH_2Ar), 3.04 (1H, dd, $J=13.0, 6.0$ Hz, CH_2Ar), 3.16-3.26 (1H, m, 2-H), 3.62 (3H, s, CO_2CH_3), 3.66 (3H, s, CO_2CH_3), 3.82 (3H, s, Ar-OCH_3), 6.82-6.89 (2H, m, Ar-H), 7.07 (1H, dd, $J=7.0, 1.0$ Hz, Ar-H), 7.21 (1H, dt, $J=7.5, 1.0$ Hz, Ar-H).

$^{13}\text{C-NMR}$ (75 MHz) δ : 175.3, 172.6, 157.7, 130.9, 128.9, 128.2, 126.5, 120.4, 110.3, 55.2, 51.8, 51.7, 41.3, 35.1, 32.6.

HRMS m/z : Calcd for $\text{C}_{14}\text{H}_{19}\text{O}_5$ ($\text{M}+\text{H}$) $^+$ 267.12269. Found : 267.1224.

2-[(3,4-Dimethoxyphenyl)methylsuccinic Acid Dimethyl Ester (5e)]

a colorless oil

IR ν_{max} (neat) cm^{-1} : 1736 (CO), 1439 (Ar).

$^1\text{H-NMR}$ (300 MHz) δ : 2.42 (1H, dd, $J=17.0, 5.0$ Hz, $\text{CH}_2\text{CO}_2\text{Me}$), 2.68 (1H, dd, $J=17.0, 9.0$ Hz, $\text{CH}_2\text{CO}_2\text{Me}$), 2.71 (1H, dd, $J=13.5, 8.0$ Hz, CH_2Ar), 3.00 (1H, dd, $J=13.5, 6.0$ Hz, CH_2Ar), 3.06-3.14 (1H, m, 2-H), 3.65 (3H, s, CO_2CH_3), 3.68 (3H, s, CO_2CH_3), 3.86 (3H, s, Ar-OCH_3), 3.87 (3H, s, Ar-OCH_3), 6.67 (1H, s, Ar-H), 6.69 (2H, d, $J=8.0$ Hz, Ar-H), 6.79 (2H, d, $J=8.0$ Hz, Ar-H).

$^{13}\text{C-NMR}$ (75 MHz) δ : 174.8, 172.4, 149.0, 147.9, 130.7, 122.2, 112.1, 111.3, 55.93, 55.88, 52.0, 51.8, 43.3, 37.4, 35.0.

HRMS m/z : Calcd for $\text{C}_{15}\text{H}_{20}\text{O}_6$ (M^+) 296.1259. Found : 296.1270.

2-[(4-Methylphenyl)methyl]succinic Acid Dimethyl Ester (5f)

a colorless oil

IR ν_{\max} (neat) cm^{-1} : 1737 (CO), 1437 (Ar).

$^1\text{H-NMR}$ (300 MHz) δ : 2.31 (3H, s, Ar- CH_3), 2.40 (1H, dd, $J=16.5, 4.5$ Hz, $\text{CH}_2\text{CO}_2\text{Me}$), 2.66 (1H, dd, $J=16.5, 9.0$ Hz, $\text{CH}_2\text{CO}_2\text{Me}$), 2.71 (1H, dd, $J=13.0, 8.0$ Hz, CH_2Ar), 3.01 (1H, dd, $J=13.0, 6.0$ Hz, CH_2Ar), 3.06-3.15 (1H, m, 2-H), 3.64 (3H, s, CO_2CH_3), 3.68 (3H, s, CO_2CH_3), 7.03 (2H, d, $J=8.0$ Hz, Ar- H), 7.10 (2H, d, $J=8.0$ Hz, Ar- H).

$^{13}\text{C-NMR}$ (75 MHz) δ : 174.8, 172.3, 136.2, 135.0, 129.3, 128.9, 51.9, 51.7, 43.1, 37.3, 34.8, 21.0.

HRMS m/z : Calcd for $\text{C}_{11}\text{H}_{19}\text{O}_4$ ($\text{M}+\text{H}^+$) 251.1278. Found : 251.1274.

2-[(3-Methylphenyl)methyl]succinic Acid Dimethyl Ester (5g)

a colorless oil

IR ν_{\max} (neat) cm^{-1} : 1736 (CO), 1436 (Ar).

$^1\text{H-NMR}$ (300 MHz) δ : 2.32 (3H, s, Ar- CH_3), 2.40 (1H, dd, $J=16.5, 5.0$ Hz, $\text{CH}_2\text{CO}_2\text{Me}$), 2.67 (1H, dd, $J=16.5, 10.0$ Hz, $\text{CH}_2\text{CO}_2\text{Me}$), 2.69 (1H, dd, $J=13.5, 9.0$ Hz, CH_2Ar), 3.02 (1H, dd, $J=13.5, 6.0$ Hz, CH_2Ar), 3.07-3.16 (1H, m, 2-H), 3.64 (3H, s, CO_2CH_3), 3.68 (3H, s, CO_2CH_3), 6.95 (1H, d, $J=8.0$ Hz, Ar- H), 6.96 (1H, s, Ar- H), 7.03 (1H, d, $J=8.0$ Hz, Ar- H), 7.17 (1H, t, $J=8.0$ Hz, Ar- H).

$^{13}\text{C-NMR}$ (75 MHz) δ : 174.8, 172.4, 138.2, 138.1, 129.8, 128.5, 127.5, 126.0, 51.9, 51.7, 43.1, 37.7, 34.9, 21.4.

HRMS m/z : Calcd for $\text{C}_{14}\text{H}_{19}\text{O}_4$ ($\text{M}+\text{H}^+$) 251.1305. Found : 251.1275.

2-[(2-Methylphenyl)methyl]succinic Acid Dimethyl Ester (5h)

a colorless oil

IR ν_{\max} (neat) cm^{-1} : 1736 (CO), 1436 (Ar).

$^1\text{H-NMR}$ (300 MHz) δ : 2.34 (3H, s, Ar- CH_3), 2.43 (1H, dd, $J=17.0, 5.0$ Hz, $\text{CH}_2\text{CO}_2\text{Me}$), 2.68-2.77 (2H, m, $\text{CHCH}_2\text{CO}_2\text{Me}$), 3.03-3.14 (2H, m, CH_2Ar), 3.63 (3H, s, CO_2CH_3), 3.67 (3H, s, CO_2CH_3), 7.06-7.16 (4H, m, Ar- H).

$^{13}\text{C-NMR}$ (75 MHz) δ : 175.1, 172.4, 136.5, 130.6, 129.9, 127.0, 126.0, 52.0, 51.8, 41.8, 35.3, 35.1, 19.4.

HRMS m/z : Calcd for $\text{C}_{14}\text{H}_{19}\text{O}_4$ ($\text{M}+\text{H}^+$) 251.1289. Found : 251.1277.

2-[(4-Chlorophenyl)methyl]succinic Acid Dimethyl Ester (5i)

a colorless oil

IR ν_{\max} (neat) cm^{-1} : 1739 (CO), 1493 (Ar).

$^1\text{H-NMR}$ (300 MHz) δ : 2.40 (1H, dd, $J=17.0, 5.0$ Hz, $\text{CH}_2\text{CO}_2\text{Me}$), 2.67 (1H, dd, $J=17.0, 9.0$ Hz, $\text{CH}_2\text{CO}_2\text{Me}$), 2.76 (1H, dd, $J=14.0, 8.0$ Hz, CH_2Ar), 3.00 (1H, dd, $J=14.0, 7.0$ Hz, CH_2Ar), 3.06-3.11 (1H, m, 2-H), 3.65 (3H, s, CO_2CH_3), 3.66 (3H, s, CO_2CH_3), 7.09 (2H, d, $J=8.0$ Hz, Ar- H), 7.26 (2H, d, $J=8.0$ Hz, Ar- H).

$^{13}\text{C-NMR}$ (75 MHz) δ : 174.4, 172.1, 136.7, 132.6, 130.4, 128.7, 52.0, 51.8, 42.9, 37.0, 34.9.

HRMS m/z : Calcd for $C_{13}H_{15}ClO_4$ (M^+) 270.0672. Found : 270.0665.

2-[(4-Bromophenyl)methyl]succinic Acid Dimethyl Ester (5j)

a colorless oil

IR ν_{\max} (neat) cm^{-1} : 1736 (CO), 1437 (Ar).

1H -NMR (300 MHz) δ : 2.40 (1H, dd, $J=16.5, 6.0$ Hz, CH_2CO_2Me), 2.67 (1H, dd, $J=16.5, 8.5$ Hz, CH_2CO_2Me), 2.74 (1H, dd, $J=13.0, 8.0$ Hz, CH_2Ar), 2.99 (1H, dd, $J=13.0, 7.0$ Hz, CH_2Ar), 3.05-3.13 (1H, m, 2-H), 3.65 (3H, s, CO_2CH_3), 3.66 (3H, s, CO_2CH_3), 7.03 (2H, d, $J=8.5$ Hz, Ar-H), 7.41 (2H, d, $J=8.5$ Hz, Ar-H).

^{13}C -NMR (75 MHz) δ : 174.3, 172.0, 137.2, 131.6, 130.7, 120.6, 52.0, 51.8, 42.8, 37.0, 34.9.

HRMS m/z : Calcd for $C_{13}H_{15}^{79}BrO_4$ (M^+) 314.0153. Found : 314.0167.

2-[(4-Fluorophenyl)methyl]succinic Acid Dimethyl Ester (5k)

a colorless oil

IR ν_{\max} (neat) cm^{-1} : 1738 (CO), 1438 (Ar).

1H -NMR (300 MHz) δ : 2.41 (1H, dd, $J=16.5, 5.0$ Hz, CH_2CO_2Me), 2.68 (1H, dd, $J=16.5, 9.0$ Hz, CH_2CO_2Me), 2.76 (1H, dd, $J=13.5, 8.5$ Hz, CH_2Ar), 3.00 (1H, dd, $J=13.5, 6.0$ Hz, CH_2Ar), 3.06-3.15 (1H, m, 2-H), 3.65 (3H, s, CO_2CH_3), 3.66 (3H, s, CO_2CH_3), 6.95-7.00 (2H, m, Ar-H), 7.09-7.14 (2H, m Ar-H).

^{13}C -NMR (75 MHz) δ : 172.2, 130.6, 130.5, 115.6, 115.3, 52.0, 51.9, 43.2, 37.0, 35.0.

HRMS m/z : Calcd for $C_{13}H_{15}FO_4$ (M^+) 254.0953. Found : 254.0967.

2-[(4-Iodophenyl)methyl]succinic Acid Dimethyl Ester (5l)

a colorless oil

IR ν_{\max} (neat) cm^{-1} : 1736 (CO), 1436 (Ar).

1H -NMR (300 MHz) δ : 2.40 (1H, dd, $J=17.0, 5.0$ Hz, CH_2CO_2Me), 2.67 (1H, dd, $J=17.0, 9.0$ Hz, CH_2CO_2Me), 2.72 (1H, dd, $J=14.0, 8.0$ Hz, CH_2Ar), 2.98 (1H, dd, $J=14.0, 7.0$ Hz, CH_2Ar), 3.05-3.12 (1H, m, 2-H), 3.65 (3H, s, CO_2CH_3), 3.66 (3H, s, CO_2CH_3), 6.91 (2H, d, $J=8.0$ Hz, Ar-H), 7.61 (2H, d, $J=8.0$ Hz, Ar-H).

^{13}C -NMR (75 MHz) δ : 174.3, 172.0, 137.8, 137.6, 131.0, 92.1, 52.0, 51.8, 42.8, 37.1, 34.9.

HRMS m/z : Calcd for $C_{13}H_{15}IO_4$ (M^+) 363.00881. Found : 363.0083.

2-[(3-Chlorophenyl)methyl]succinic Acid Dimethyl Ester (5m)

a colorless oil

IR ν_{\max} (neat) cm^{-1} : 1736 (CO), 1437 (Ar).

1H -NMR (300 MHz) δ : 2.41 (1H, dd, $J=17.0, 5.0$ Hz, CH_2CO_2Me), 2.68 (1H, dd, $J=17.0, 9.0$ Hz, CH_2CO_2Me), 2.75 (1H, dd, $J=13.5, 7.5$ Hz, CH_2Ar), 3.02 (1H, dd, $J=13.5, 7.0$ Hz, CH_2Ar), 3.07-3.14 (1H, m, 2-H), 3.66 (3H, s, CO_2CH_3), 3.67 (3H, s, CO_2CH_3), 7.02-7.06 (1H, m, Ar-H), 7.16 (1H, s, Ar-H), 7.21-7.23 (2H, m, Ar-H).

^{13}C -NMR (75 MHz) δ : 174.3, 172.1, 140.3, 134.3, 129.8, 129.1, 127.2, 127.0, 52.0, 51.9, 42.8, 37.3, 35.0.

HRMS m/z : Calcd for $\text{C}_{13}\text{H}_{15}^{35}\text{ClO}_4$ (M^+) 270.0658. Found : 270.0688.

2-[(2-Chlorophenyl)methyl]succinic Acid Dimethyl Ester (5n)

a colorless oil

IR ν_{max} (neat) cm^{-1} : 1737(CO), 1438 (Ar).

^1H -NMR (300 MHz) δ : 2.46 (1H, dd, $J=17.0$, 5.0 Hz, $\text{CH}_2\text{CO}_2\text{Me}$), 2.73 (1H, dd, $J=17.0$, 9.0 Hz, $\text{CH}_2\text{CO}_2\text{Me}$), 2.92 (1H, dd, $J=13.5$, 8.5 Hz, CH_2Ar), 3.16 (1H, dd, $J=13.5$, 7.0 Hz, CH_2Ar), 3.20-3.30 (1H, m, 2-H), 3.64 (3H, s, CO_2CH_3), 3.66 (3H, s, CO_2CH_3), 7.17-7.20 (3H, m, Ar-H), 7.34-7.37 (1H, m, Ar-H).

^{13}C -NMR (75 MHz) δ : 174.6, 172.1, 136.0, 134.3, 131.3, 129.8, 128.3, 126.9, 52.0, 51.8, 41.3, 35.4, 35.1.

HRMS m/z : Calcd for $\text{C}_{13}\text{H}_{15}^{35}\text{ClO}_4$ (M^+) 270.0658. Found : 270.0647.

2-[[4-(Methoxycarbonyl)phenyl]methyl]succinic Acid Dimethyl Ester (5o)

a colorless oil

IR ν_{max} (neat) cm^{-1} : 1736 (CO), 1437 (Ar).

^1H -NMR (300 MHz) δ : 2.41 (1H, dd, $J=16.5$, 5.0 Hz, $\text{CH}_2\text{CO}_2\text{Me}$), 2.69 (1H, dd, $J=16.5$, 8.5 Hz, $\text{CH}_2\text{CO}_2\text{Me}$), 2.88 (1H, dd, $J=12.5$, 7.5 Hz, CH_2Ar), 3.08 (1H, dd, $J=12.5$, 6.0 Hz, CH_2Ar), 3.13-3.21 (1H, m, 2-H), 3.65 (3H, s, CO_2CH_3), 3.66 (3H, s, CO_2CH_3), 3.91 (3H, d, $J=0.5$ Hz, Ar- CO_2CH_3), 7.23 (2H, d, $J=8.5$ Hz, Ar-H), 7.97 (2H, d, $J=8.5$ Hz, Ar-H).

^{13}C -NMR (75 MHz) δ : 174.3, 172.0, 166.9, 143.6, 129.9, 129.2, 129.1, 128.7, 127.1, 52.1, 52.0, 51.8, 42.7, 37.6, 35.0.

HRMS m/z : Calcd for $\text{C}_{15}\text{H}_{18}\text{O}_6$ (M^+) 294.1102. Found : 294.1132.

2-(1,2-Diphenylethyl)succinic Acid Dimethyl Ester (5p)

a colorless oil

6:1 mixture of diastereomers

IR ν_{max} (neat) cm^{-1} : 1737(CO), 1437 (Ar).

^1H -NMR (300 MHz) δ : 2.24 (1/7H, dd, $J=16.5$, 3.0 Hz, $\text{CH}_2\text{CO}_2\text{Me}$), 2.40 (6/7H, dd, $J=16.5$, 5.0 Hz, $\text{CH}_2\text{CO}_2\text{Me}$), 2.54 (1/7H, dd, $J=16.5$, 6.0 Hz, $\text{CH}_2\text{CO}_2\text{Me}$), 2.67 (6/7H, dd, $J=16.5$, 9.0 Hz, $\text{CH}_2\text{CO}_2\text{Me}$), 2.75 (6/7H, dd, $J=13.0$, 10.0 Hz, CH_2Ar), 2.85 (1/7H, dd, $J=13.0$, 4.0 Hz, CH_2Ar), 2.98-3.16 (1/7H+6/7H+1H+1H, m, CH_2Ar +2-H+ CHPh), 3.52 (3/7H, s, CO_2CH_3), 3.57 (3/7H, s, CO_2CH_3), 3.63 (18/7H, s, CO_2CH_3), 3.66 (18/7H, s, CO_2CH_3), 6.88-7.36 (10H, m, Ar-H).

^{13}C -NMR (75 MHz) δ : 174.8, 172.4, 138.2, 138.1, 129.6, 129.1, 128.63, 128.58, 128.5, 128.4, 127.7, 126.8, 126.7, 126.0, 52.0, 51.8, 46.2, 43.1, 37.8, 35.0.

HRMS m/z : Calcd for $\text{C}_{20}\text{H}_{23}\text{O}_4$ ($\text{M}+\text{H}$) $^+$ 327.2140. Found : 327.1585.

2-(1-Phenylpropyl)succinic Acid Dimethyl Ester (5q)

a colorless oil

1:1 mixture of diastereomers

IR ν_{\max} (neat) cm^{-1} : 1738(CO), 1437 (Ar).

$^1\text{H-NMR}$ (300 MHz) δ : 1.277 (3/2H, d, $J=7.0$ Hz, CH_2CH_3), 1.282 (3/2H, d, $J=7.0$ Hz, CH_2CH_3), 2.19 (1/2H, dd, $J=17.0$, 3.5 Hz, $\text{CH}_2\text{CO}_2\text{Me}$), 2.34 (1/2H, dd, $J=17.0$, 3.5 Hz, $\text{CH}_2\text{CO}_2\text{Me}$), 2.59 (1/2H, dd, $J=17.0$, 11.0 Hz, $\text{CH}_2\text{CO}_2\text{Me}$), 2.75 (1/2H, dd, $J=17.0$, 11.0 Hz, $\text{CH}_2\text{CO}_2\text{Me}$), 2.85-3.03 (1H, m, 2-H), 3.09-3.16 (1/2H, m, CHPh), 3.19-3.28 (1/2H, m, CHPh), 3.57 (3H, s, CO_2CH_3), 3.62 (3/2H, s, CO_2CH_3), 3.75 (3/2H, s, CO_2CH_3), 7.15-7.34 (5H, m, Ar-H).

$^{13}\text{C-NMR}$ (75 MHz) δ : 175.2, 174.3, 172.7, 172.4, 143.5, 143.1, 128.8, 128.5, 127.5, 127.4, 127.0, 126.8, 51.9, 51.82, 51.79, 51.7, 48.4, 48.2, 42.5, 40.8, 35.4, 32.3, 20.3, 16.4.

HRMS m/z : Calcd for $\text{C}_{14}\text{H}_{19}\text{O}_4$ (M+H)⁺ 251.1518. Found : 251.1251.

2-[(4-Methoxyphenyl)methyl]butanedinitrile (7)

a colorless oil

IR ν_{\max} (neat) cm^{-1} : 2248 (CN), 1515 (Ar).

$^1\text{H-NMR}$ (300 MHz) δ : 2.63 (2H, d, $J=6.0$ Hz, CH_2CN), 2.96-3.15 (3H, m, 2-H+ CH_2Ar), 3.80 (3H, s, Ar-O CH_3), 6.90 (2H, br d, $J=8.5$ Hz, Ar-H), 7.18 (2H, br d, $J=8.5$ Hz, Ar-H).

$^{13}\text{C-NMR}$ (75 MHz) δ : 159.4, 130.3, 126.4, 118.7, 115.7, 114.6, 55.4, 36.2, 30.3, 20.0.

HRMS m/z : Calcd for $\text{C}_{12}\text{H}_{12}\text{N}_2\text{O}$ (M)⁺ 200.0949. Found : 200.0940.

2-[(4-Methoxyphenyl)methyl]-1,4-diphenyl-1,4-butanedione (9)

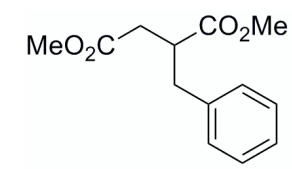
a colorless oil

IR ν_{\max} (neat) cm^{-1} : 1679 (CO), 1513 (Ar).

$^1\text{H-NMR}$ (300 MHz) δ : 2.67 (1H, dd, $J=14.0$, 9.0 Hz, CH_2Ar), 3.09 (1H, dd, $J=18.5$, 5.0 Hz, CH_2COPh), 3.09 (1H, dd, $J=14.0$, 5.0 Hz, CH_2Ar), 3.69 (1H, dd, $J=18.5$, 9.0 Hz, CH_2COPh), 3.76 (3H, s, Ar-O CH_3), 4.33-4.42 (1H, m, 2-H), 6.80 (2H, d, $J=8.5\text{Hz}$, Ar-H), 7.12 (2H, d, $J=8.5\text{Hz}$, Ar-H), 7.39-7.59 (6H, m, COC_6H_5), 7.90 (2H, d, $J=7.0$ Hz, COC_6H_5), 8.04 (2H, d, $J=7.0$ Hz, COC_6H_5)

$^{13}\text{C-NMR}$ (75 MHz) δ : 202.9, 198.6, 158.4, 136.7, 136.7, 133.2, 133.0, 130.8, 130.7, 129.0, 128.7, 128.6, 128.1, 114.1, 55.3, 43.7, 40.2, 37.4.

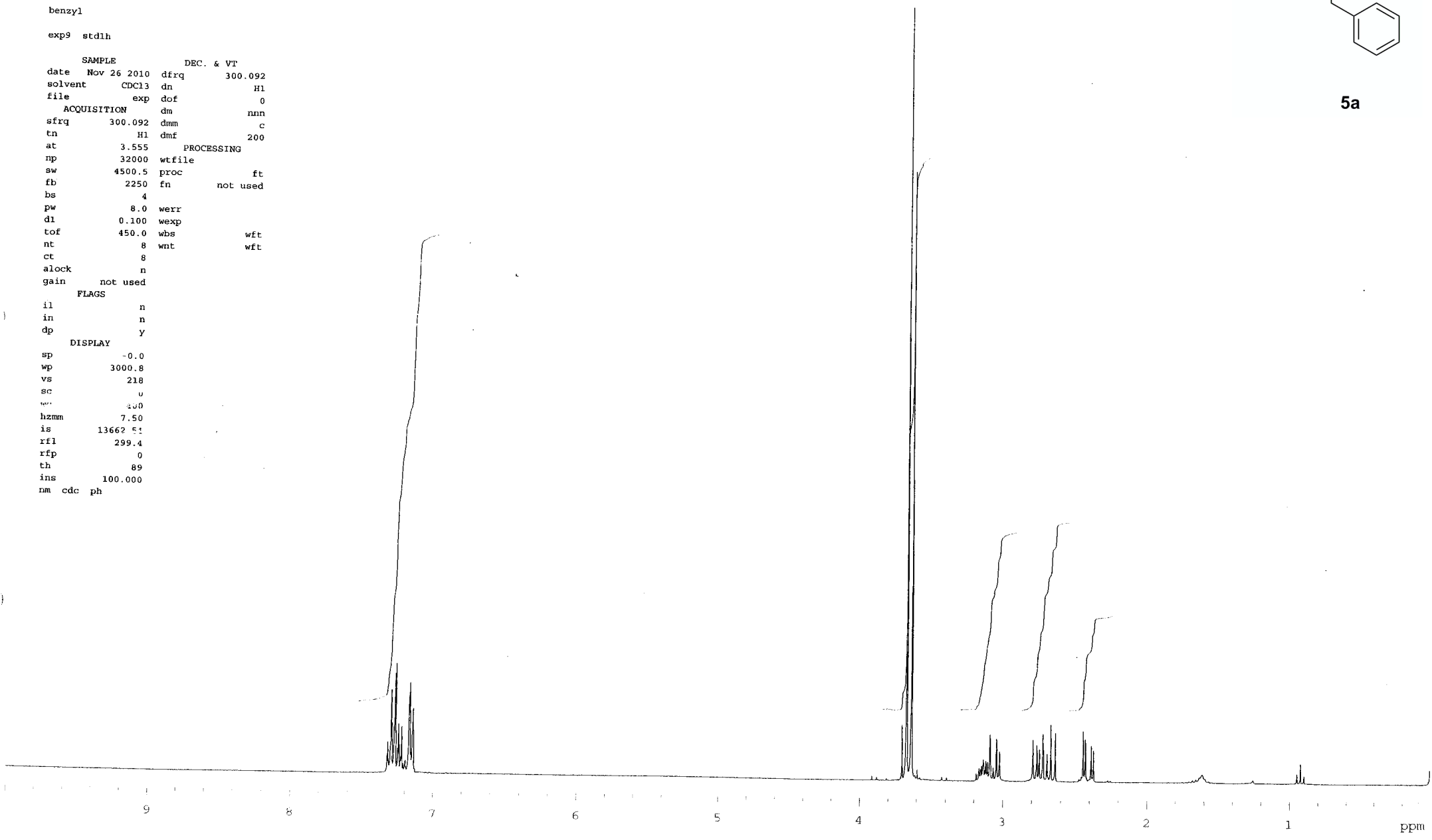
HRMS m/z : Calcd for $\text{C}_{24}\text{H}_{22}\text{O}_3$ (M)⁺ 358.1567. Found : 358.1577.

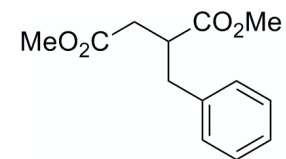


5a

```

benzyl
exp9 stdlh
SAMPLE          DEC. & VT
date Nov 26 2010 dfrq      300.092
solvent CDCl3  dn         H1
file      exp   dof         0
ACQUISITION    dm         nnn
sfrq      300.092 dmm        c
tn         H1   dmF        200
at         3.555  PROCESSING
np         32000 wtfile
sw         4500.5 proc      ft
fb         2250  fn        not used
bs         4
pw         8.0  werr
dl         0.100 wexp
tof        450.0 wbs      wft
nt         8   wnt      wft
ct         8
alock      n
gain      not used
FLAGS
il         n
in         n
dp         y
DISPLAY
sp         -0.0
wp         3000.8
vs         218
sc         v
h2mm      7.50
is        13662 51
rfl       299.4
rfp        0
th         89
ins       100.000
nm cdc ph
  
```

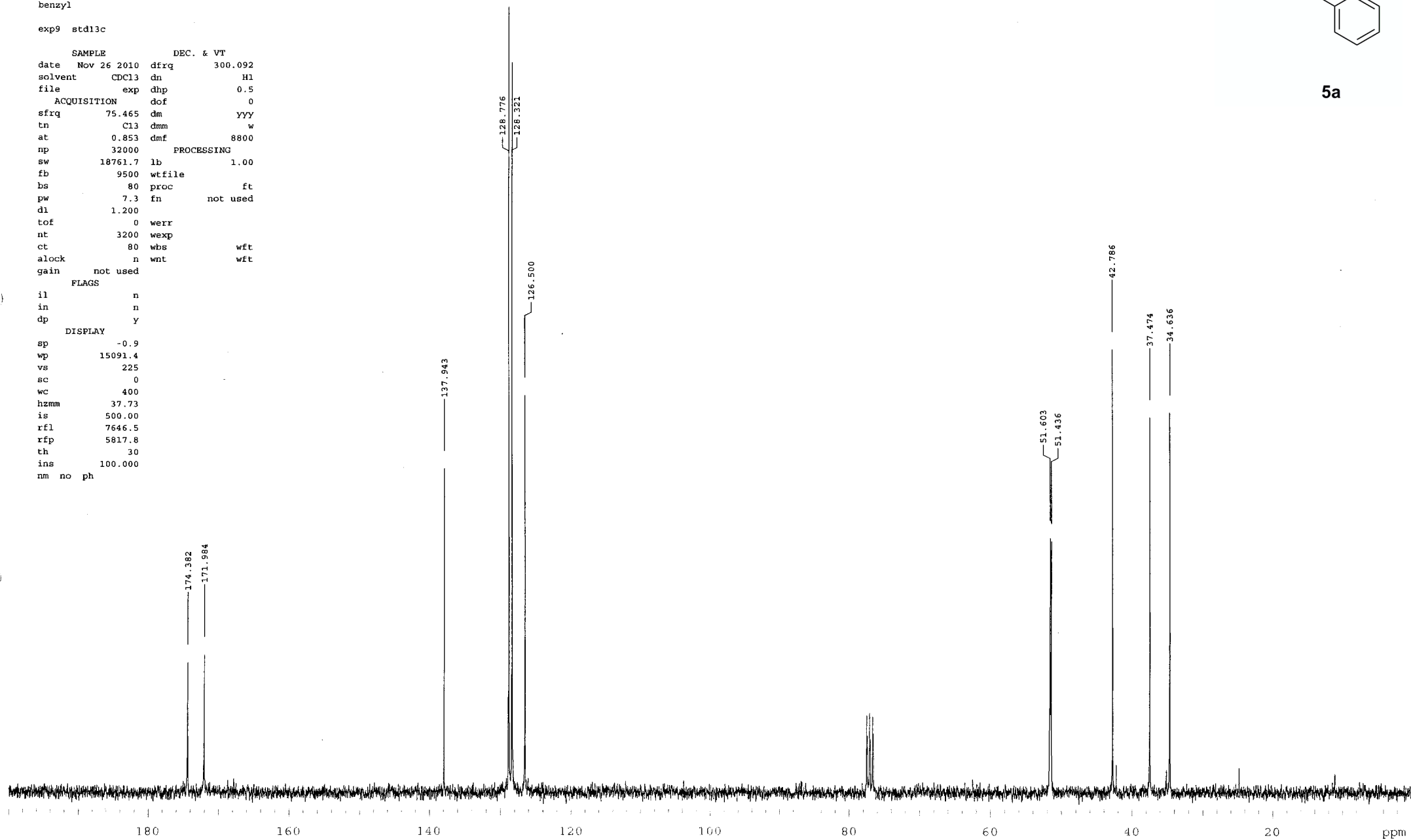


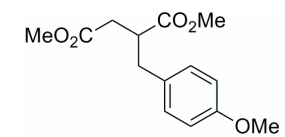


5a

```

benzyl
exp9 std13c
SAMPLE          DEC. & VT
date Nov 26 2010 dfrq      300.092
solvent CDCl3  dn         H1
file      exp  dhp        0.5
ACQUISITION    dof        0
sfrq      75.465 dm        YYY
tn         C13 dmm        w
at         0.853 dmf       8800
np         32000 PROCESSING
sw         18761.7 lb       1.00
fb         9500 wtfile
bs         80  proc       ft
pw         7.3  fn       not used
dl         1.200
tof         0  werr
nt         3200 wexp
ct         80  wbs       wft
alock      n  wnt       wft
gain      not used
FLAGS
il         n
in         n
dp         y
DISPLAY
sp         -0.9
wp         15091.4
vs         225
sc         0
wc         400
hzmm      37.73
is         500.00
rfl       7646.5
rfp       5817.8
th         30
ins       100.000
nm no ph
  
```



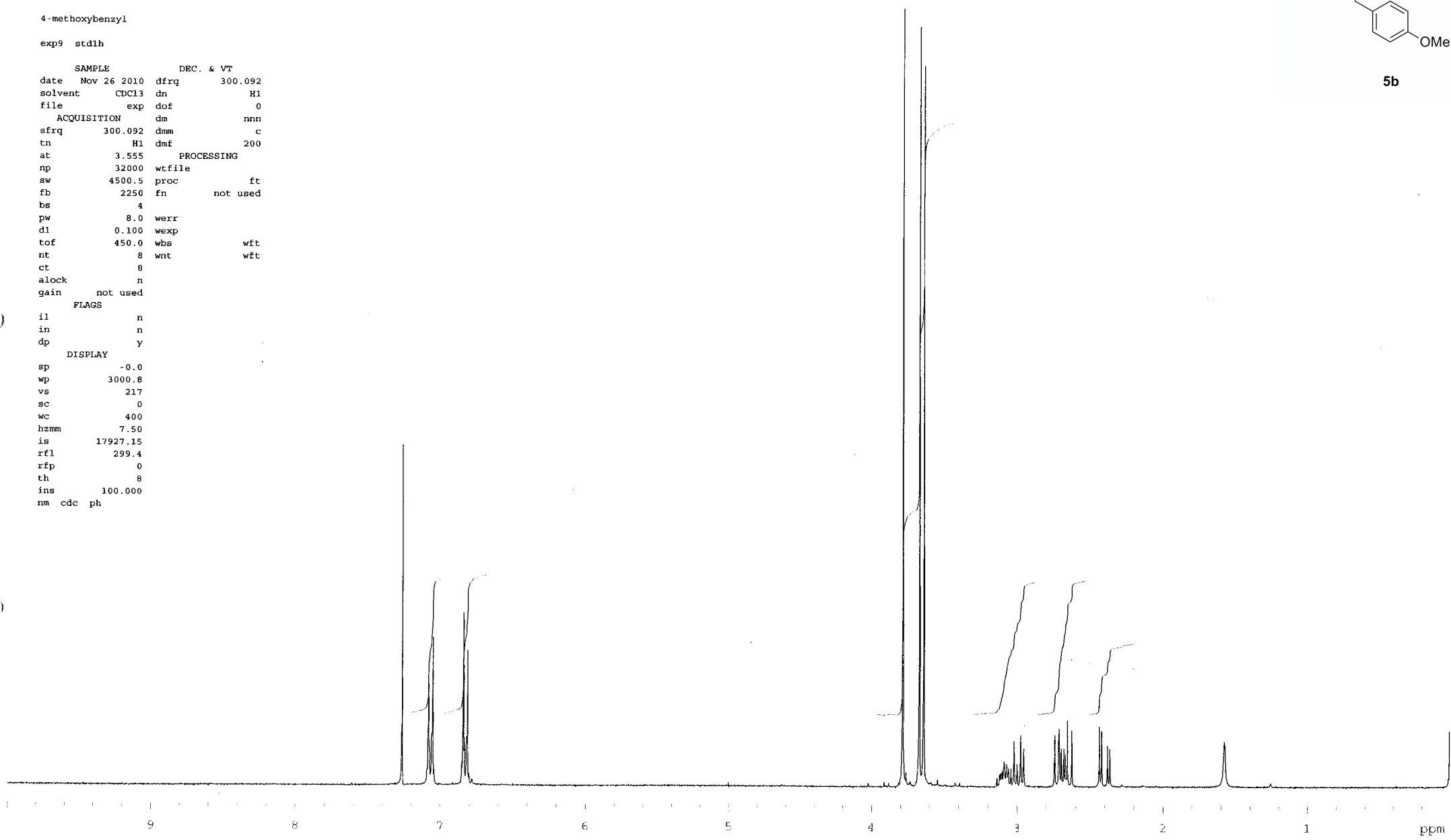


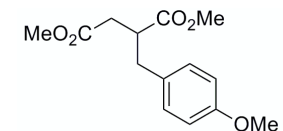
5b

4-methoxybenzyl

exp9 stdlh

SAMPLE		DEC. & VT	
date	Nov 26 2010	dfrq	300.092
solvent	CDCl3	dn	H1
file	exp	dof	0
ACQUISITION		dm	nnn
sfrq	300.092	dmm	c
tn	H1	dof	200
at	3.555	PROCESSING	
np	32000	wtfile	
sw	4500.5	proc	ft
fb	2250	fn	not used
bs	4		
pw	8.0	werr	
d1	0.100	wexp	
tof	450.0	wbs	wft
nt	8	wnt	wft
ct	8		
alock	n		
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
DISPLAY			
sp	-0.0		
wp	3000.8		
vs	217		
sc	0		
wc	400		
hzmm	7.50		
is	17927.15		
rfl	299.4		
rfp	0		
th	8		
ins	100.000		
nm	cdc ph		



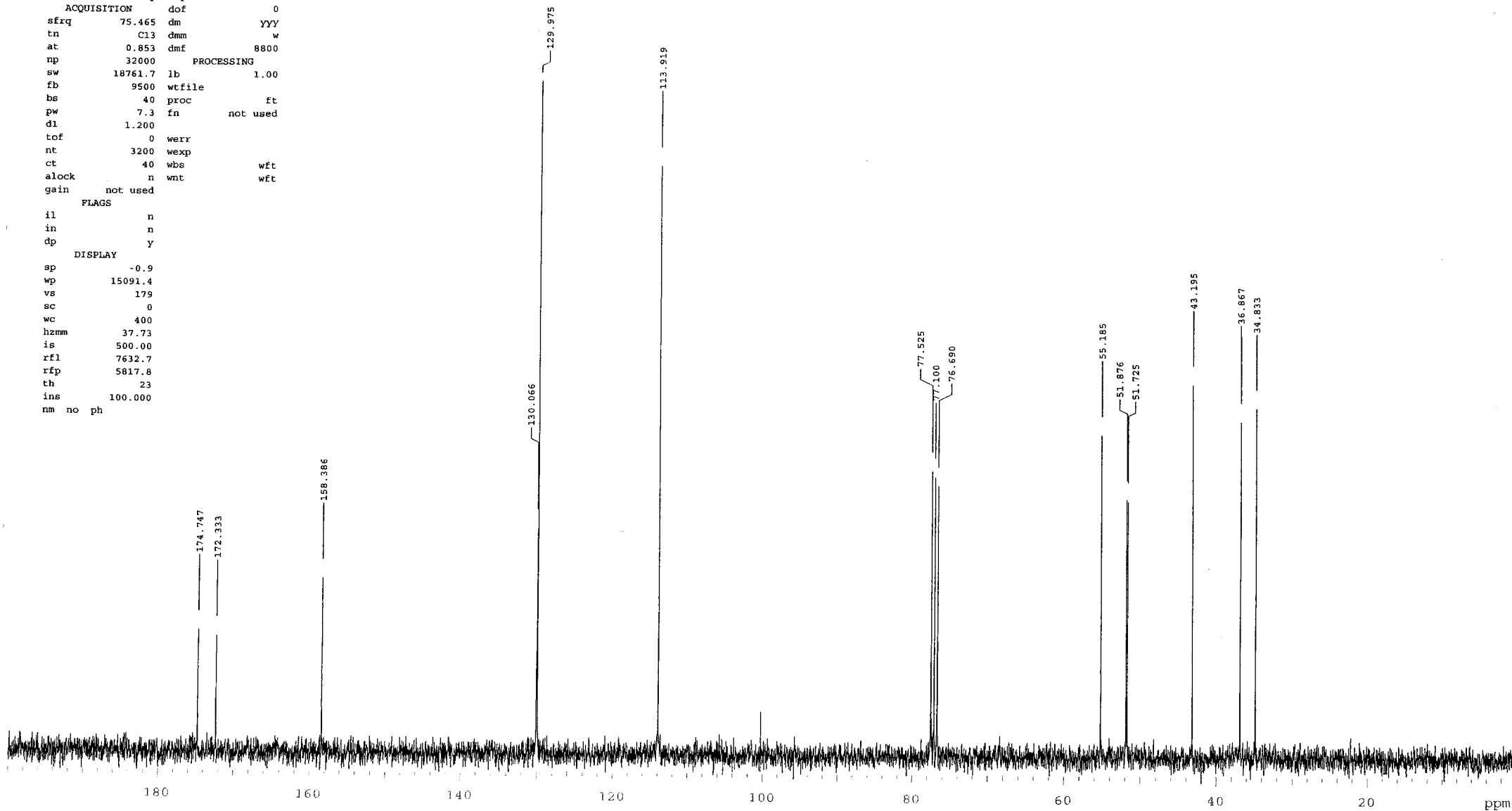


5b

methoxybenzyl

exp9 std13c

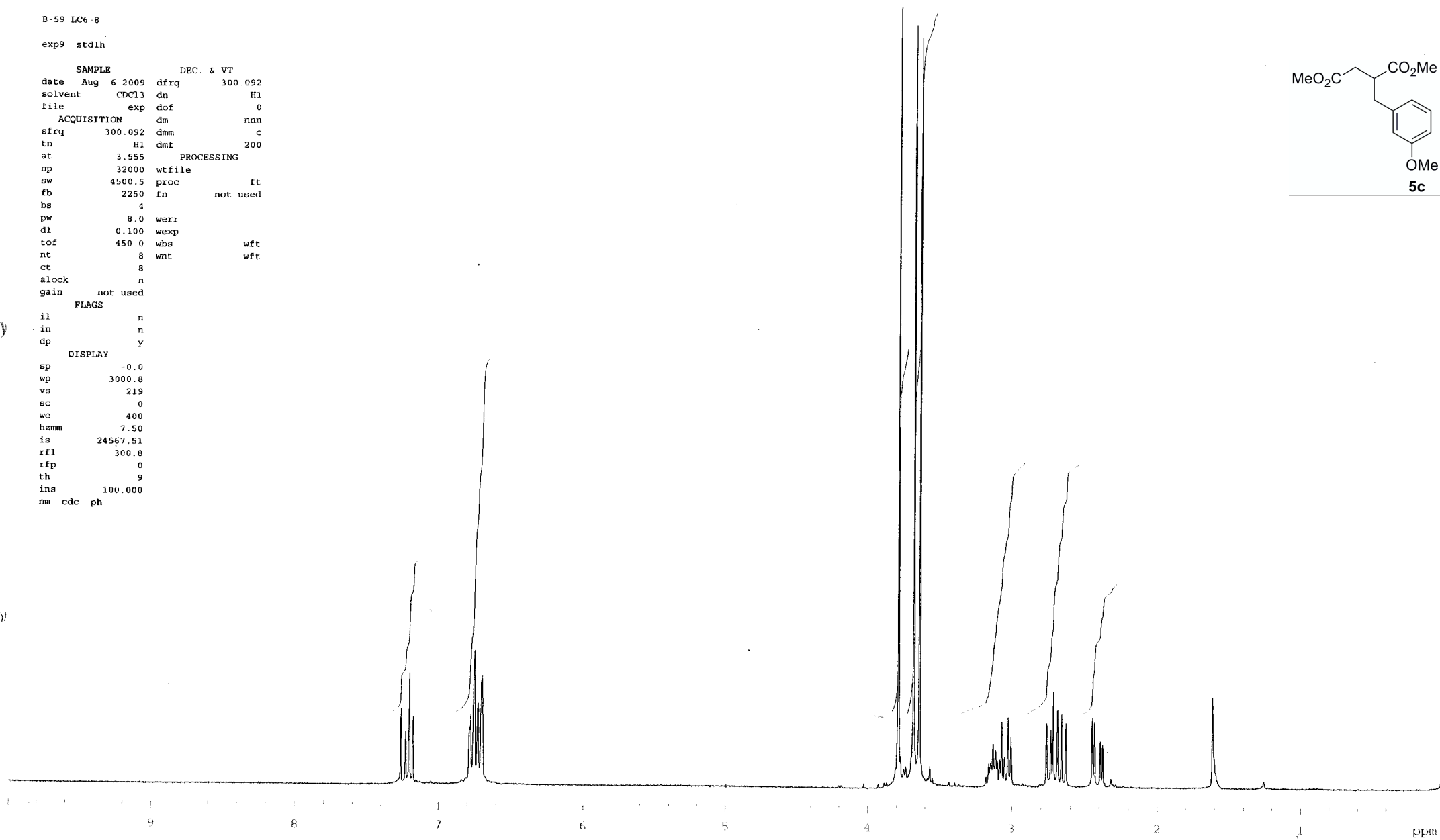
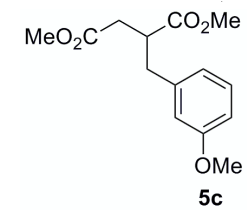
SAMPLE		DEC. & VT	
date	Nov 26 2010	dfrq	300.092
solvent	CDC13	dn	H1
file	exp	dhp	0.5
ACQUISITION		dof	0
sfrq	75.465	dm	YYY
tn	C13	dmm	w
at	0.853	dmf	8800
np	32000	PROCESSING	
sw	18761.7	lb	1.00
fb	9500	wtfile	
bs	40	proc	ft
pw	7.3	fn	not used
d1	1.200		
tof	0	werr	
nt	3200	wexp	
ct	40	wbs	wft
alock	n	wnt	wft
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
DISPLAY			
sp	-0.9		
wp	15091.4		
vs	179		
sc	0		
wc	400		
hzmm	37.73		
is	500.00		
rfl	7632.7		
rfp	5817.8		
th	23		
ins	100.000		
nm	no	ph	

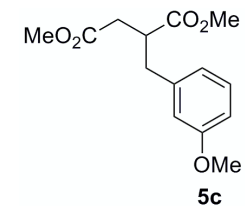


B-59 LC6-8

exp9 stdlh

SAMPLE		DEC. & VT	
date	Aug 6 2009	dfrq	300.092
solvent	CDCl3	dn	H1
file		exp	0
ACQUISITION			
sfrq	300.092	dmm	c
tn	H1	dmf	200
at	3.555	PROCESSING	
np	32000	wtfile	
sw	4500.5	proc	ft
fb	2250	fn	not used
bs	4		
pw	8.0	werr	
dl	0.100	wexp	
tof	450.0	wbs	wft
nt	8	wnt	wft
ct	8		
alock	n		
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
DISPLAY			
sp	-0.0		
wp	3000.8		
vs	219		
sc	0		
wc	400		
hzmm	7.50		
is	24567.51		
rfl	300.8		
rfp	0		
th	9		
ins	100.000		
nm	cdc ph		

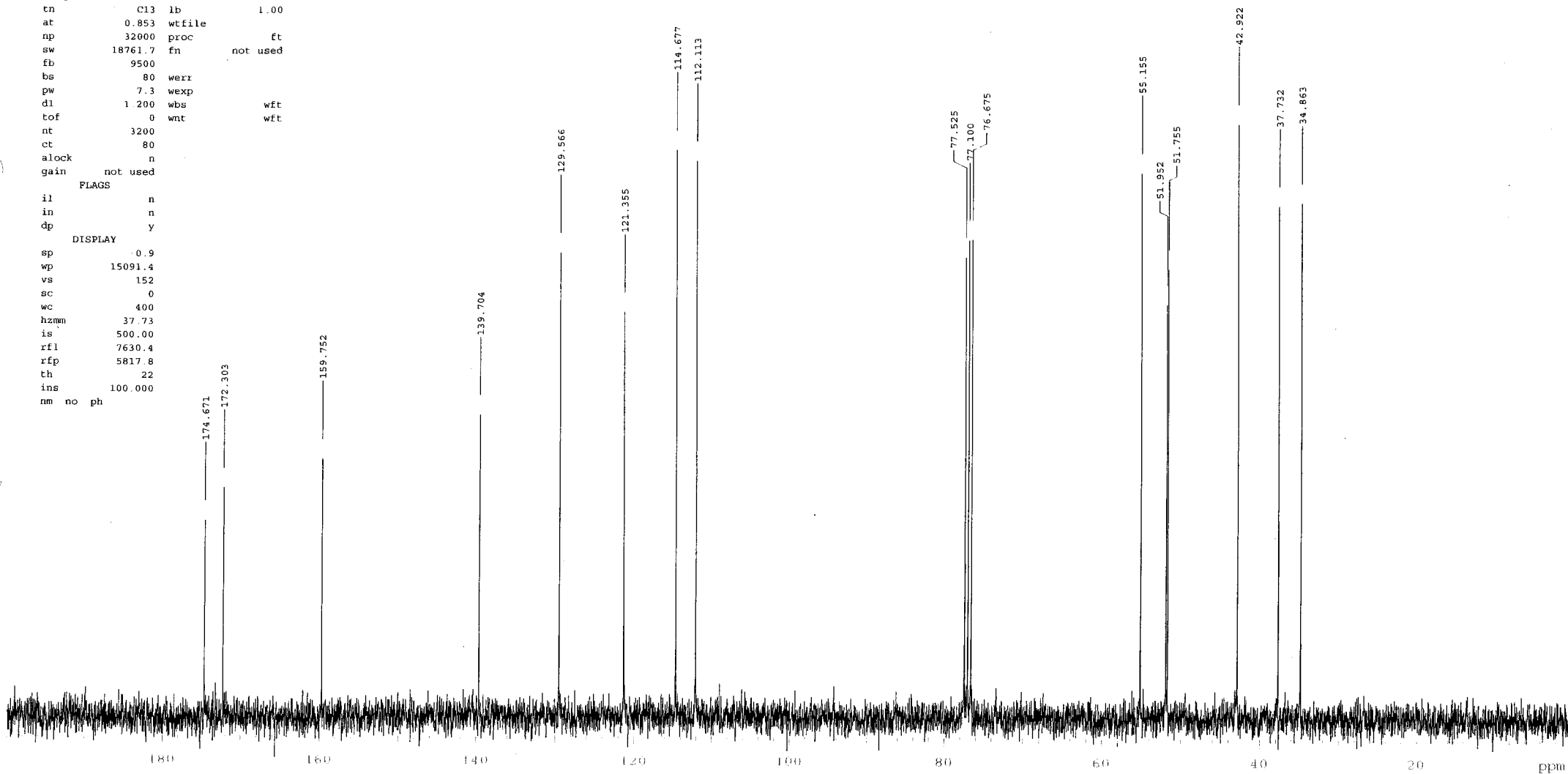


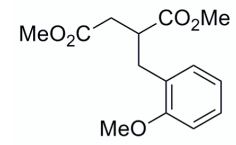


B-59 LC6-8

exp2 std13c

SAMPLE		DEC. & VT	
date	Nov 30 2009	dfrq	300.092
solvent	CDCl3	dn	H1
file	/net/kp010003-	dhp	0.5
/export/home/vnmr1-		dof	0
/gem300/yakuhin/no-		dm	yyy
v/kon_b_59 fid		dmm	w
ACQUISITION		dmf	8800
sfrq	75.465	PROCESSING	
tn	C13	lb	1.00
at	0.853	wtfile	
np	32000	proc	ft
sw	18761.7	fn	not used
fb	9500		
bs	80	werr	
pw	7.3	wexp	
di	1.200	wbs	wft
tof	0	wnt	wft
nt	3200		
ct	80		
alock	n		
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
DISPLAY			
sp	0.9		
wp	15091.4		
vs	152		
sc	0		
wc	400		
hzmh	37.73		
is	500.00		
rfl	7630.4		
rfp	5817.8		
th	22		
ins	100.000		
nm	no	ph	



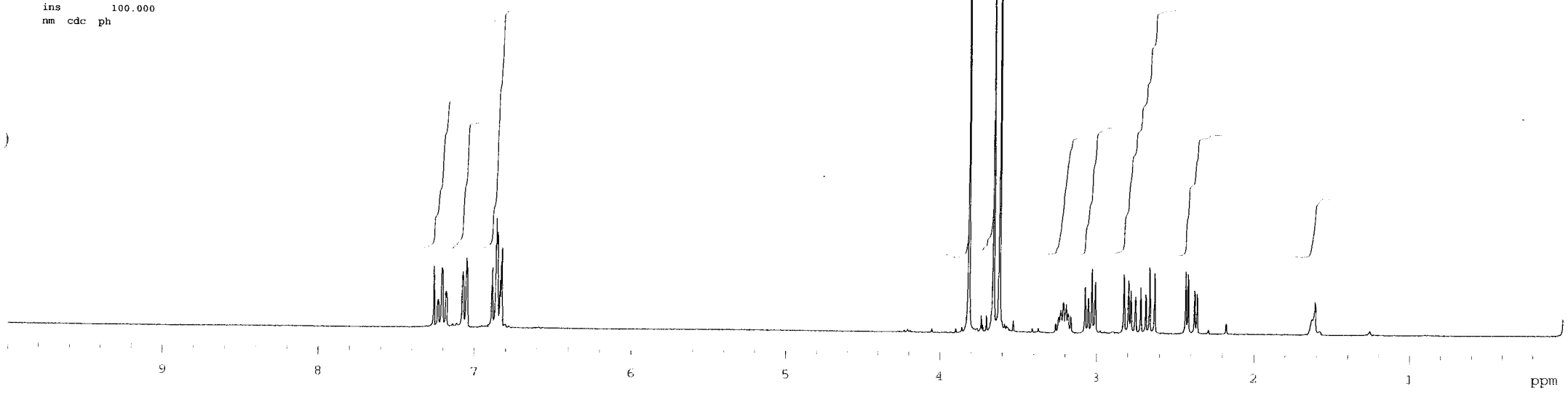


5d

```

B-57 LC7-8
exp9 std1h
SAMPLE          DEC. & VT
date   Aug 5 2009  dfrq   300 092
solvent CDCl3      dn      H1
file    exp      dof      0
ACQUISITION
sfrq   300.092  dmm      nnn
tn     H1      dmf      200
at     3.555   PROCESSING
np     32000   wtfile
sw     4500.5  proc      ft
fb     2250    fn      not used
bs     4
pw     8.0    werr
dl     0.100  wexp
tof    450.0  wbs      wft
nt     8      wnt      wft
ct     8
alock  n
gain   not used
FLAGS
il     n
in     n
dp     Y
DISPLAY
sp     0.0
wp     3000.8
vs     214
sc     0
wc     400
hzmm   7.50
is     21841.17
rf1    301.6
rfp    0
th     24
ins    100.000
nm cdc ph

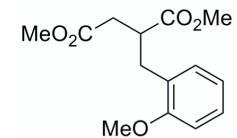
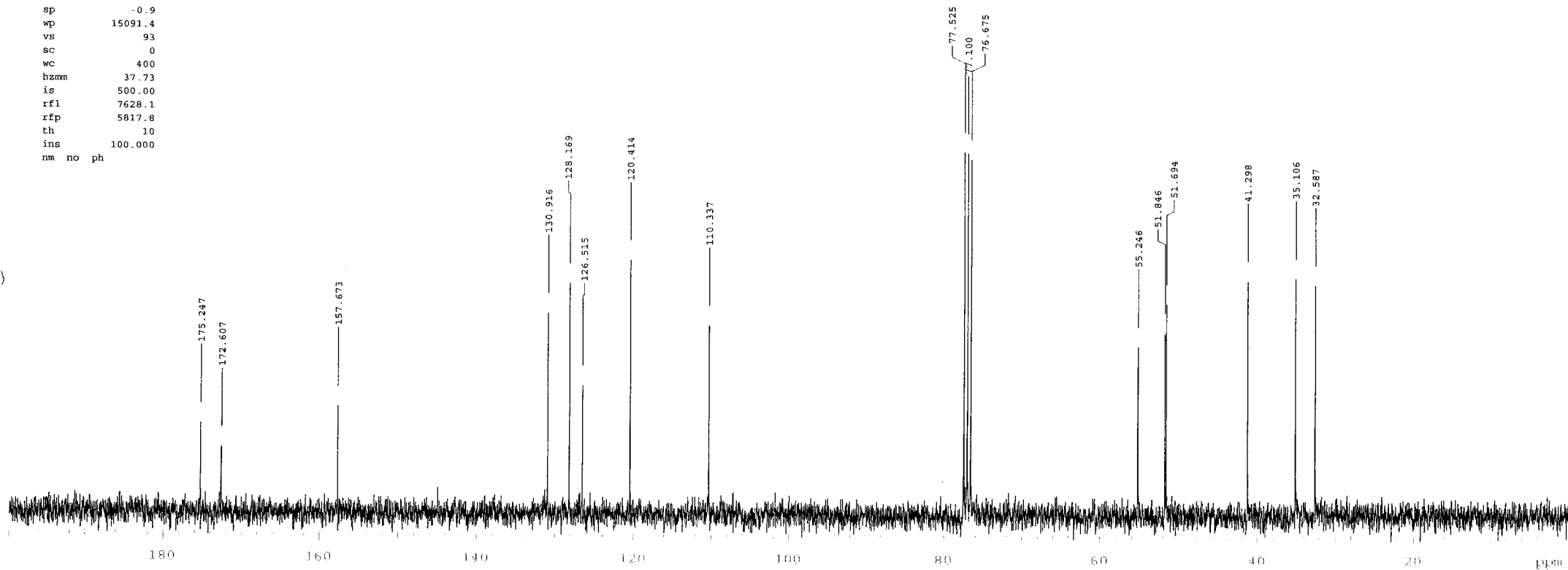
```



B-57 LC7-8

exp9 std13c

```
SAMPLE          DEC. & VT
date  Dec 28 2009  dfrq    300.092
solvent  CDCl3    dn      H1
file     exp     dhp      0.5
ACQUISITION      dof      0
sfrq     75.465  dm       yyy
tn       C13    dmm      w
at       0.853  dmf      8800
np       32000  PROCESSING
sw       18761.7 lb       1.00
fb       9500   wtfile
bs       80    proc      ft
pw       7.3   fn       not used
dl       1.200
tof      0     werr
nt       3200  wexp
ct       80   wbs      wft
alock    n    wnt      wft
gain     not used
FLAGS
il       n
in       n
dp       y
DISPLAY
sp       -0.9
wp       15091.4
vs       93
sc       0
wc       400
hzmm     37.73
is       500.00
rfl      7628.1
rfp      5817.8
th       10
ins      100.000
nm no ph
```

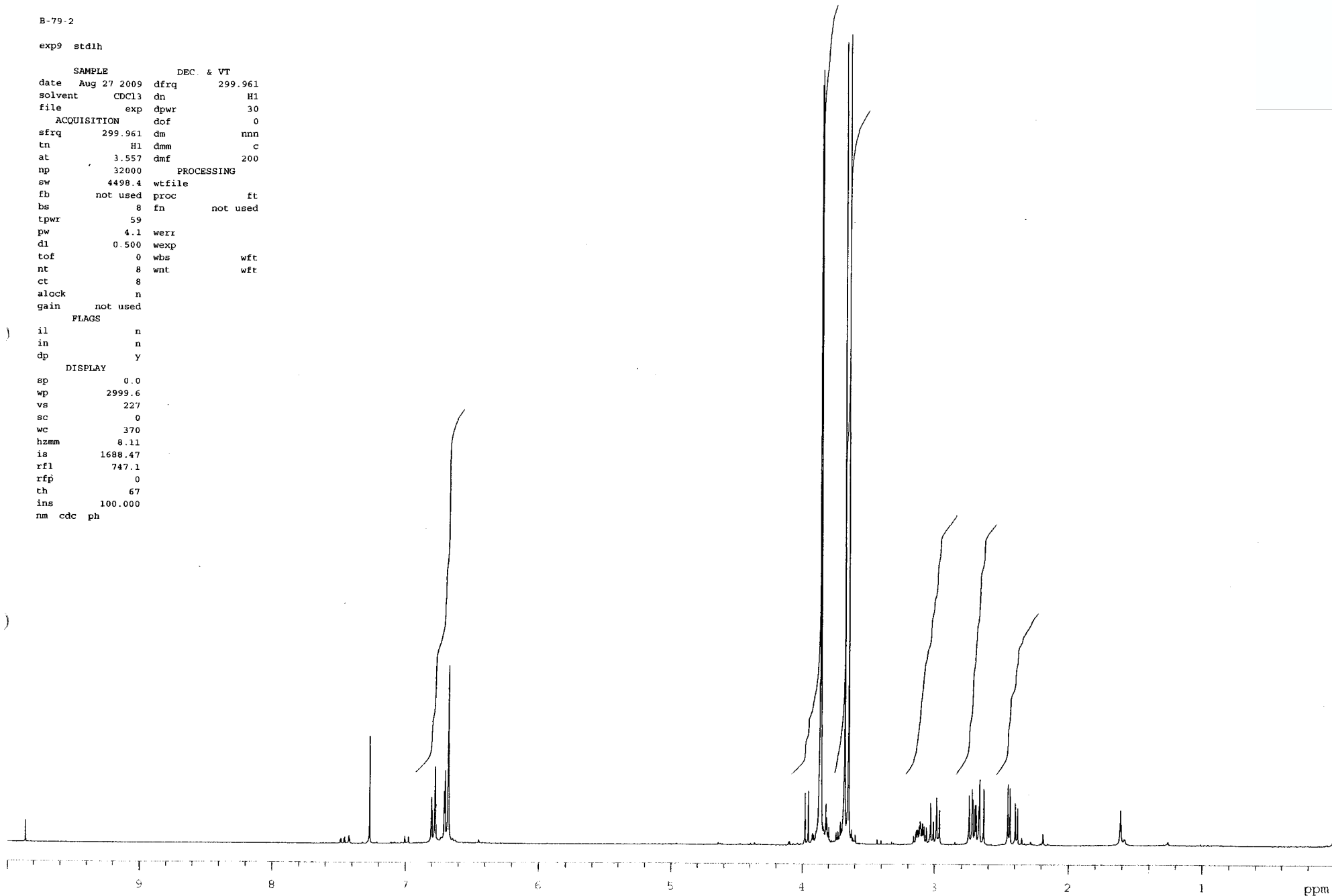
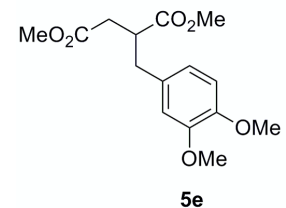


5d

B-79-2

exp9 std1h

```
      SAMPLE          DEC. & VT
date  Aug 27 2009    dfrq    299.961
solvent CDCl3      dn      H1
file    exp      dpwr     30
      ACQUISITION
sfrq    299.961    dm      nnn
tn      H1      dmm      c
at      3.557    dmf     200
np      32000
sw      4498.4    wtfile
fb      not used  proc      ft
bs      8        fn      not used
tpwr    59
pw      4.1      werr
dl      0.500    wexp
tof     0        wbs      wft
nt      8        wnt     wft
ct      8
alock   n
gain    not used
      FLAGS
il      n
in      n
dp      y
      DISPLAY
sp      0.0
wp      2999.6
vs      227
sc      0
wc      370
hzmm    8.11
is      1688.47
rf1     747.1
rfp     0
th      67
ins     100.000
nm cdc ph
```



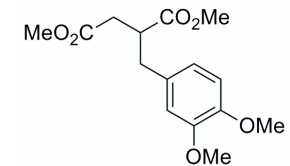
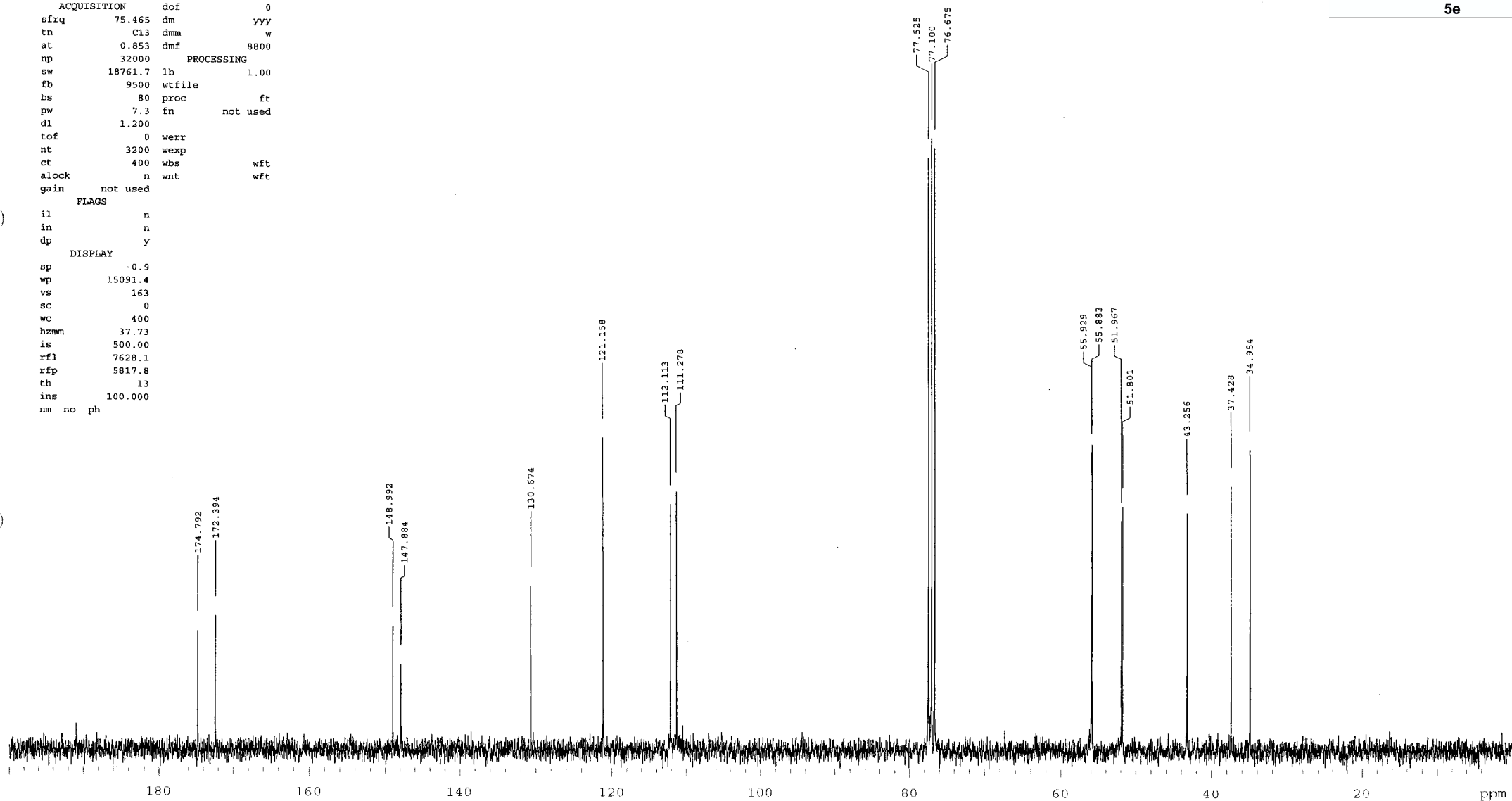
B-79-2

exp9 std13c

```
SAMPLE          DEC. & VT
date   Jun 13 2010 dfrq      300.092
solvent CDC13  dn          H1
file    exp    dhp         0.5
ACQUISITION  dof          0
sfrq     75.465 dm         yyy
tn       C13  dmm         w
at       0.853 dmf        8800
np       32000
sw      18761.7 lb         1.00
fb       9500 wtfile
bs       80  proc         ft
pw       7.3  fn         not used
di       1.200
tof      0   werr
nt       3200 wexp
ct       400  wbs         wft
alock    n   wnt         wft
gain    not used

FLAGS
il      n
in      n
dp      Y

DISPLAY
sp      -0.9
wp      15091.4
vs      163
sc      0
wc      400
hzmm    37.73
is      500.00
rfl     7628.1
rfp     5817.8
th      13
ins     100.000
nm no ph
```

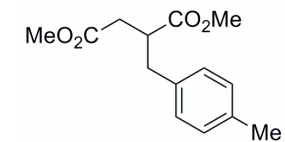


5e

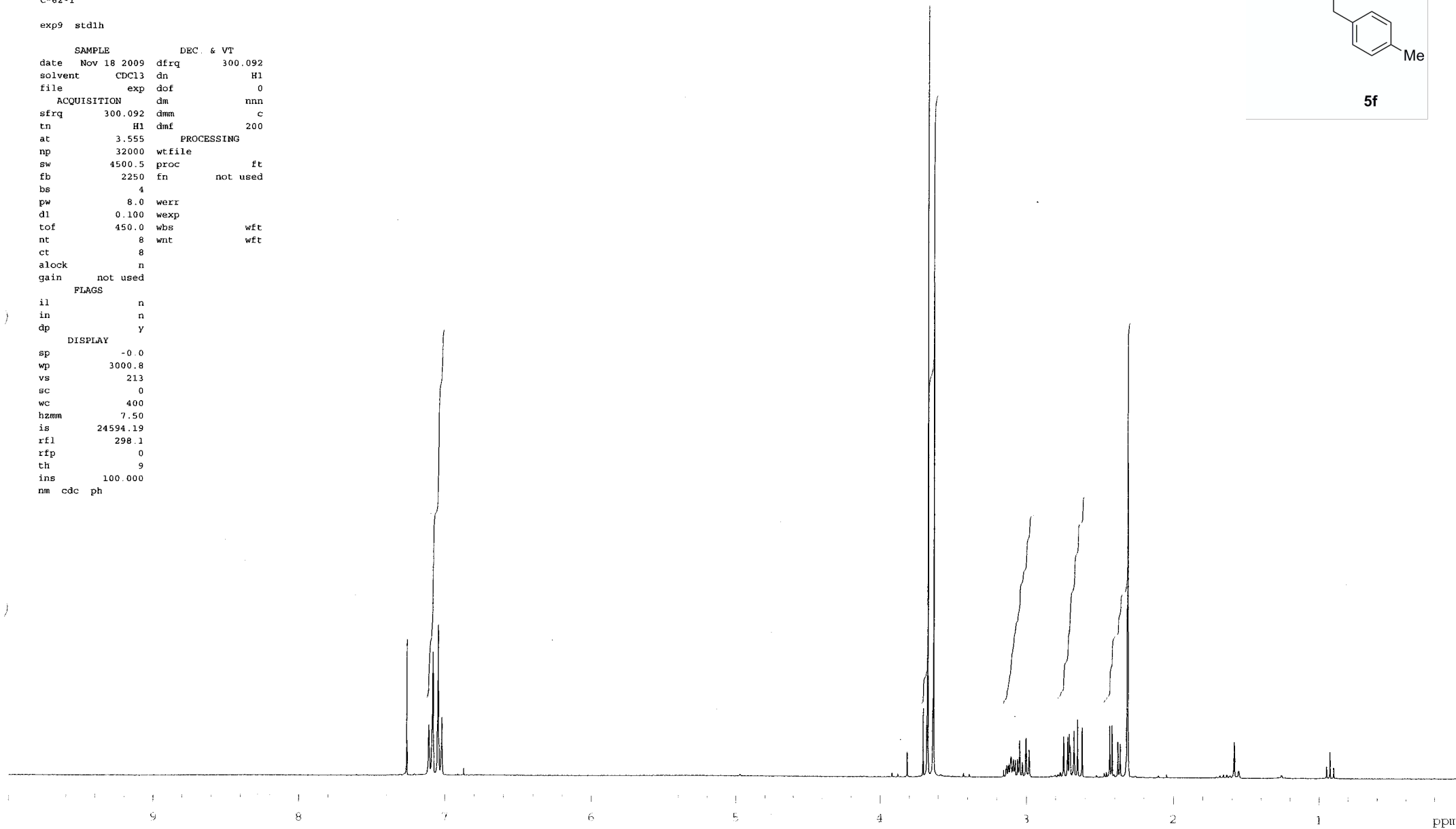
C-62-1

exp9 stdlh

```
SAMPLE          DEC. & VT
date  Nov 18 2009  dfrq    300.092
solvent  CDCl3  dn      H1
file     exp    dof      0
ACQUISITION      dm      nnn
sfrq    300.092 dmm      c
tn      H1    dmf      200
at      3.555  PROCESSING
np      32000  wtfile
sw      4500.5 proc      ft
fb      2250  fn      not used
bs      4
pw      8.0  werr
dl      0.100 wexp
tof     450.0 wbs      wft
nt      8    wnt      wft
ct      8
alock   n
gain   not used
FLAGS
il      n
in      n
dp      y
DISPLAY
sp      -0.0
wp      3000.8
vs      213
sc      0
wc      400
hzmm    7.50
is      24594.19
rf1     298.1
rfp     0
th      9
ins     100.000
nm  cdc  ph
```



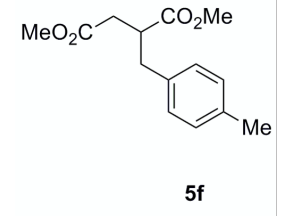
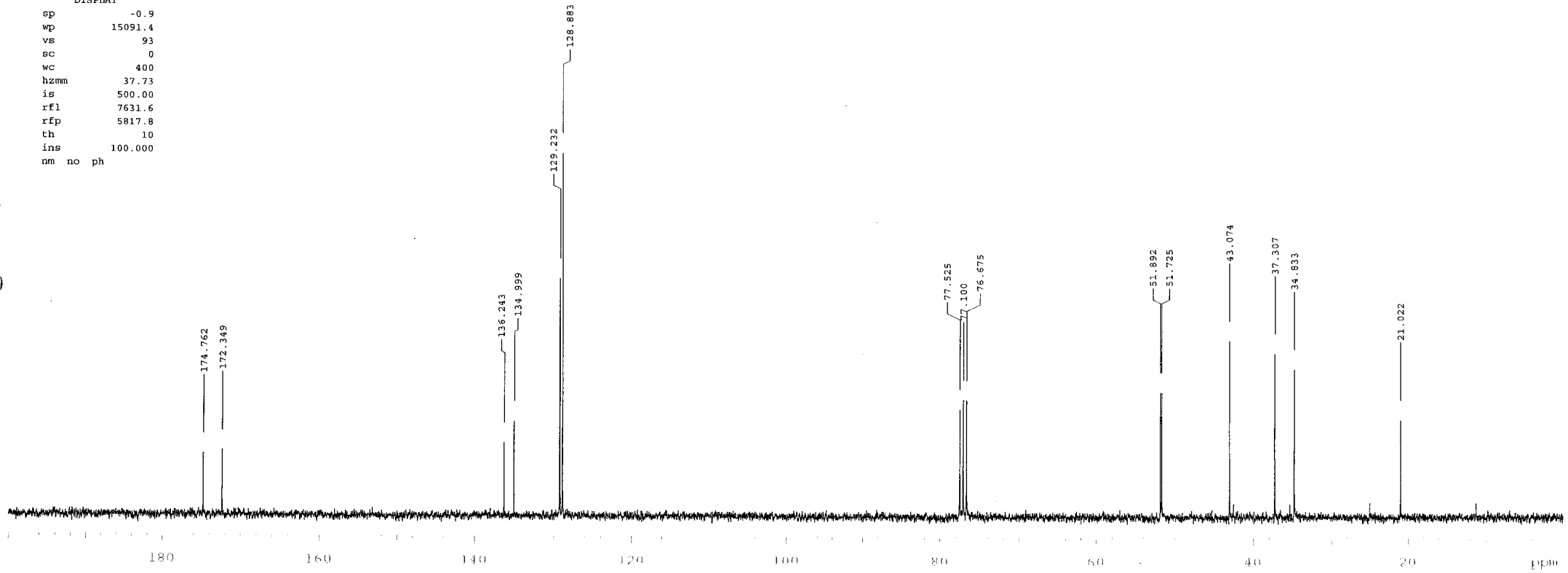
5f

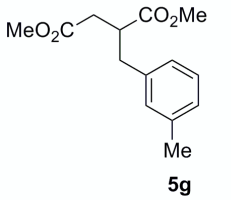


C-62-1

exp9 std13c

```
SAMPLE          DEC. & VT
date  Dec 28 2009  dfrq    300.092
solvent  CDCl3    dn      H1
file     exp     dhp      0.5
ACQUISITION  dof      0
sfrq     75.465  dm       yyy
tn       C13    dmm      w
at       0.853  dmf      8800
np       32000  PROCESSING
sw       18761.7 lb       1.00
fb       9500  wtfile
bs       80   proc      ft
pw       7.3  fn       not used
d1       1.200
tof      0    werr
nt       3200  wexp
ct       80   wbs      wft
alock    n    wnt      wft
gain     not used
FLAGS
il       n
in       n
dp       y
DISPLAY
sp       -0.9
wp       15091.4
vs       93
sc       0
wc       400
hzmm     37.73
is       500.00
rfl      7631.6
rfp      5817.8
th       10
ins      100.000
nm no ph
```

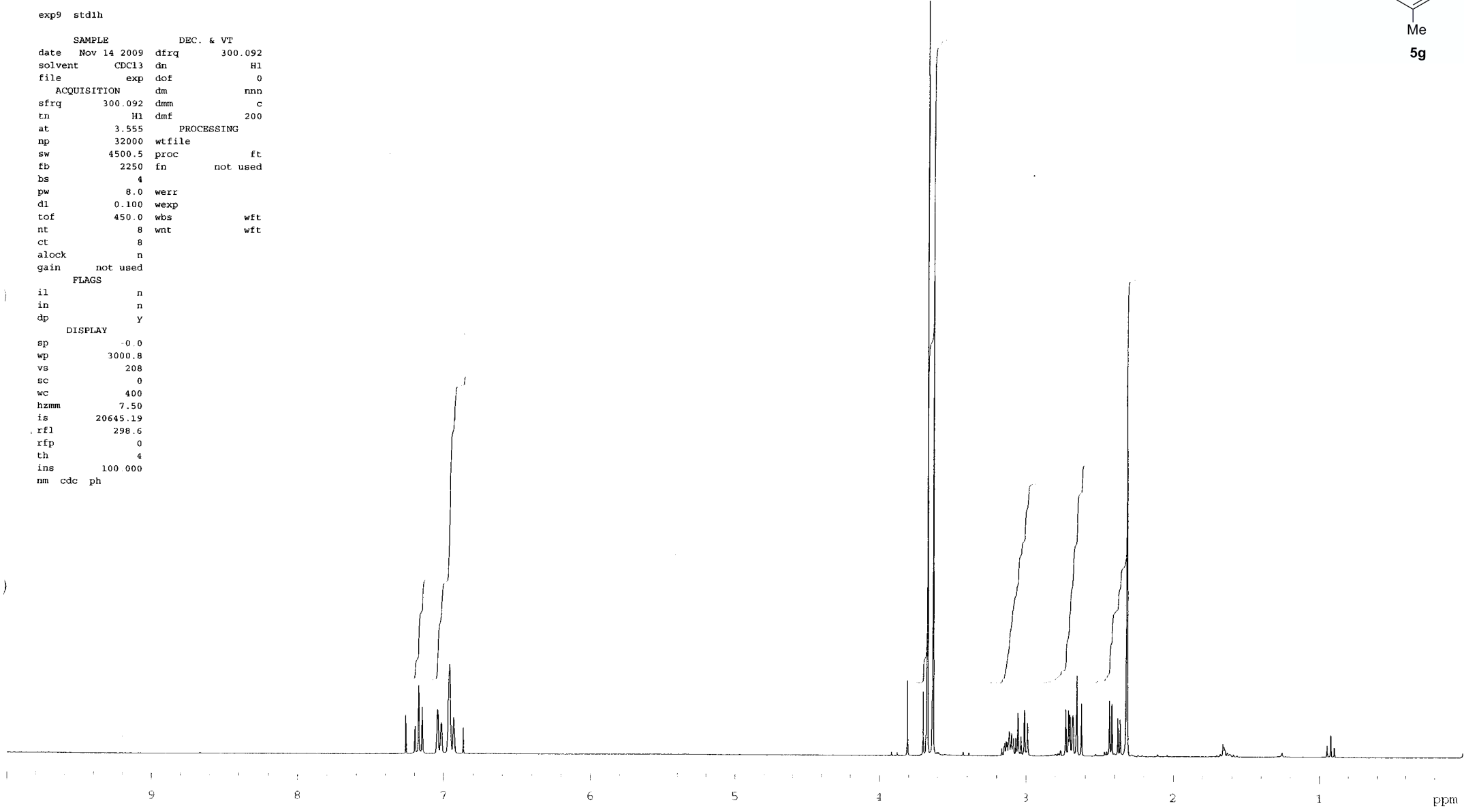


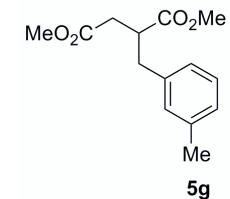


C-64
4

exp9 std1h

SAMPLE		DEC. & VT	
date	Nov 14 2009	dfrq	300.092
solvent	CDCl3	dn	H1
file	exp	dof	0
ACQUISITION		dm	nmm
sfrq	300.092	dmm	c
tn	H1	dmf	200
at	3.555	PROCESSING	
np	32000	wtfile	
sw	4500.5	proc	ft
fb	2250	fn	not used
bs	4		
pw	8.0	werr	
dl	0.100	wexp	
tof	450.0	wbs	wft
nt	8	wnt	wft
ct	8		
alock	n		
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
DISPLAY			
sp	-0.0		
wp	3000.8		
vs	208		
sc	0		
wc	400		
hzmm	7.50		
is	20645.19		
rfl	298.6		
rEp	0		
th	4		
ins	100 000		
nm	cdc ph		

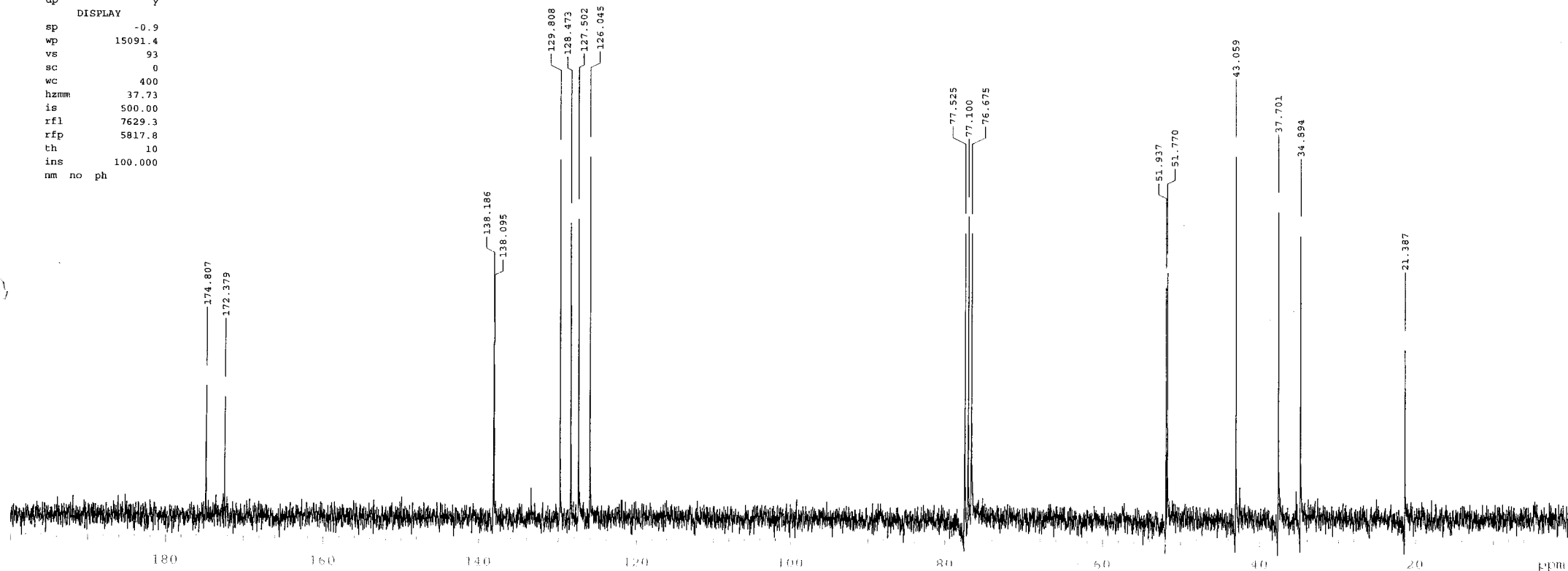




C-64
4

exp9 std13c

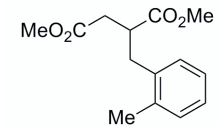
SAMPLE		DEC. & VT	
date	Dec 28 2009	dfrq	300.092
solvent	CDCl3	dn	H1
file	exp	dhp	0.5
ACQUISITION		dof	
sfrq	75.465	dm	YYY
tn	C13	dmm	w
at	0.853	dmf	8800
np	32000	PROCESSING	
sw	18761.7	lb	1.00
fb	9500	wtfile	
bs	80	proc	ft
pw	7.3	fn	not used
d1	1.200		
tof	0	werr	
nt	3200	wexp	
ct	80	wbs	wft
alock	n	wnt	wft
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
DISPLAY			
sp	-0.9		
wp	15091.4		
vs	93		
sc	0		
wc	400		
hzmm	37.73		
is	500.00		
rfl	7629.3		
rfp	5817.8		
th	10		
ins	100.000		
nm	no	ph	



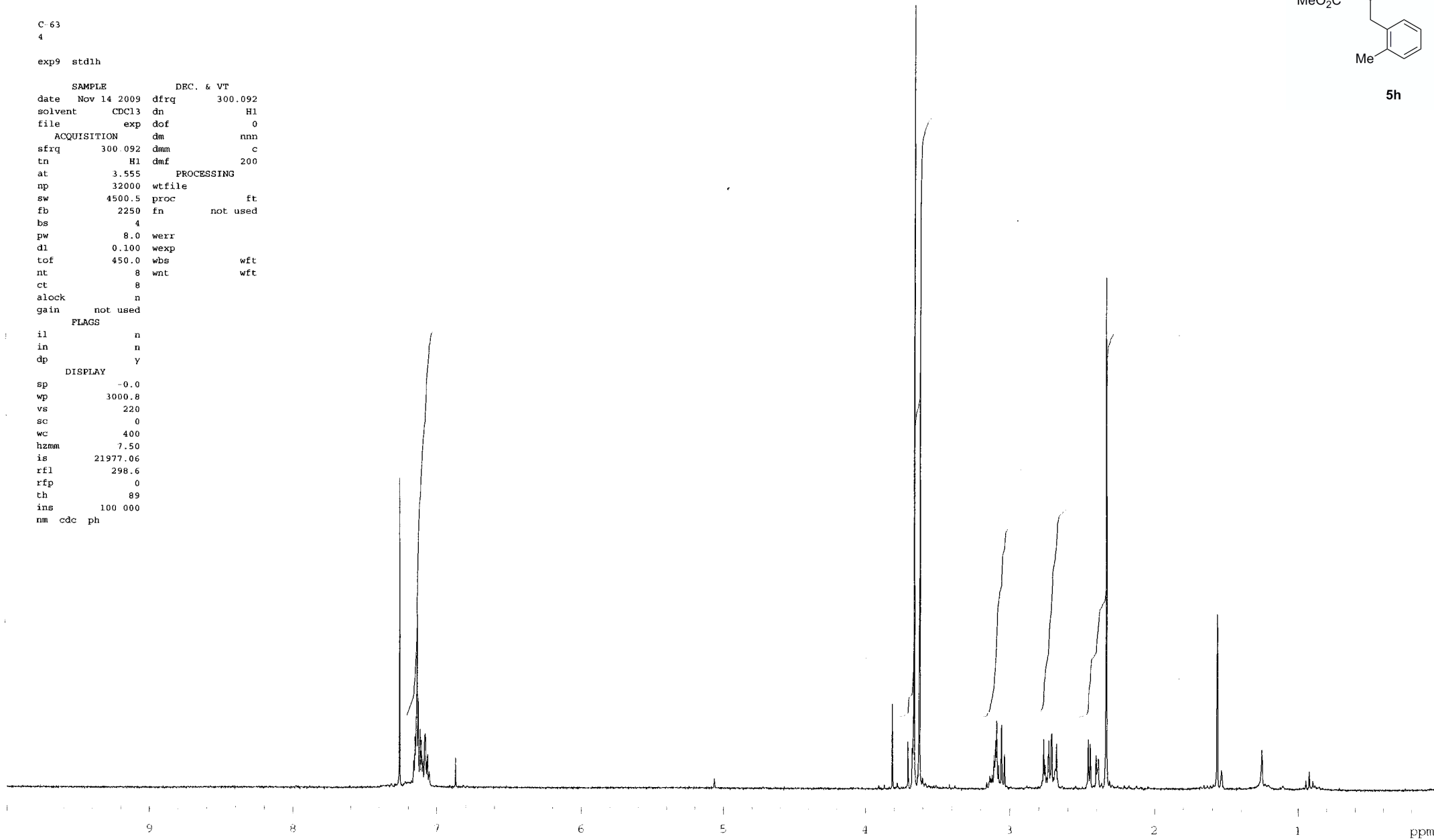
C 63
4

exp9 std1h

```
SAMPLE          DEC. & VT
date   Nov 14 2009  dfrq      300.092
solvent CDCl3      dn         H1
file    exp        dof         0
ACQUISITION      dm          nnn
sfrq    300 092    dmm         c
tn      H1        dmf         200
at      3.555     PROCESSING
np      32000     wfile
sw      4500.5    proc         ft
fb      2250     fn          not used
bs      4
pw      8.0      werr
dl      0.100    wexp
tof     450.0    wbs          wft
nt      8        wnt          wft
ct      8
alock   n
gain   not used
FLAGS
il      n
in      n
dp      y
DISPLAY
sp      -0.0
wp      3000.8
vs      220
sc      0
wc      400
hzmm    7.50
is      21977.06
rfl     298.6
rfp     0
th      89
ins     100 000
nm   cdc ph
```



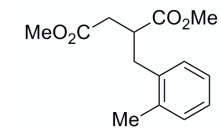
5h



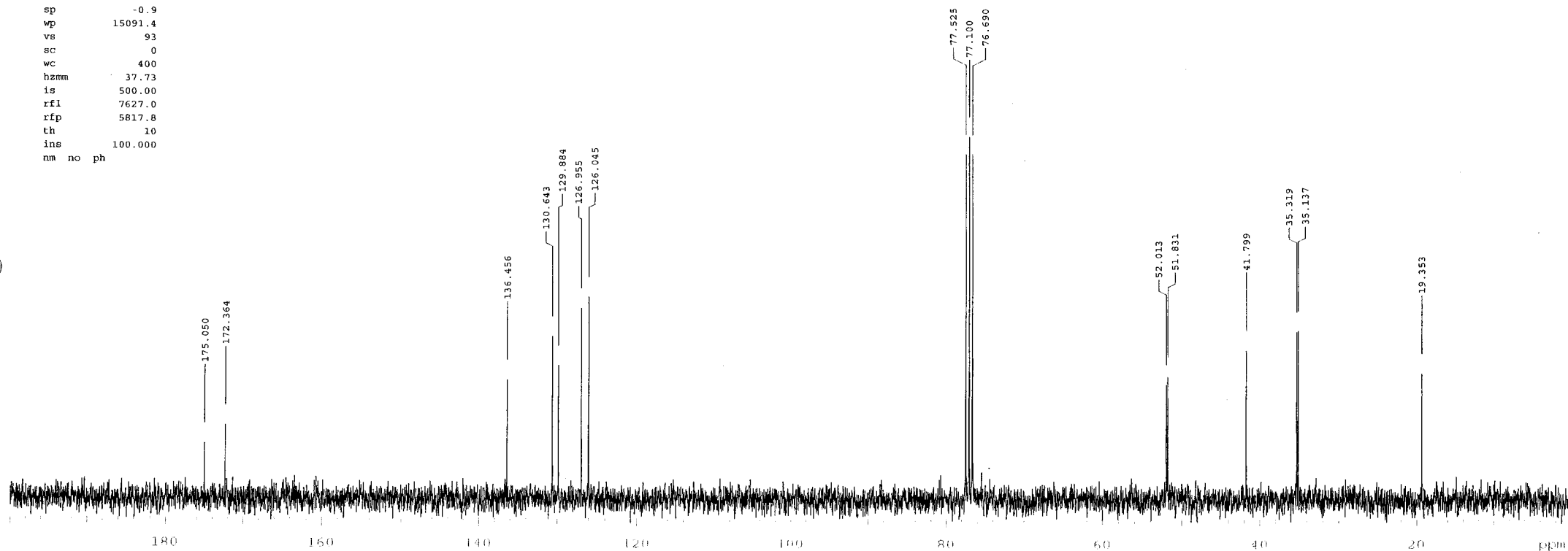
C-63
4

exp9 std13c

```
SAMPLE          DEC. & VT
date  Dec 28 2009  dfrq    300.092
solvent  CDCl3      dn      H1
file     exp       dhp      0.5
ACQUISITION    dof      0
sfrq     75.465    dm      yyy
tn       C13      dmm     w
at       0.853    dmf     8800
np       32000    PROCESSING
sw       18761.7   lb      1.00
fb       9500     wtfile
bs       80      proc     ft
pw       7.3     fn      not used
dl       1.200
tof      0       werr
nt       3200    wexp
ct       80     wbs      wft
alock    n      wnt      wft
gain     not used
FLAGS
il       n
in       n
dp       y
DISPLAY
sp       -0.9
wp       15091.4
vs       93
sc       0
wc       400
hzmm     37.73
is       500.00
rf1      7627.0
rfp      5817.8
th       10
ins      100.000
nm no ph
```



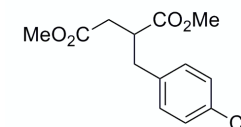
5h



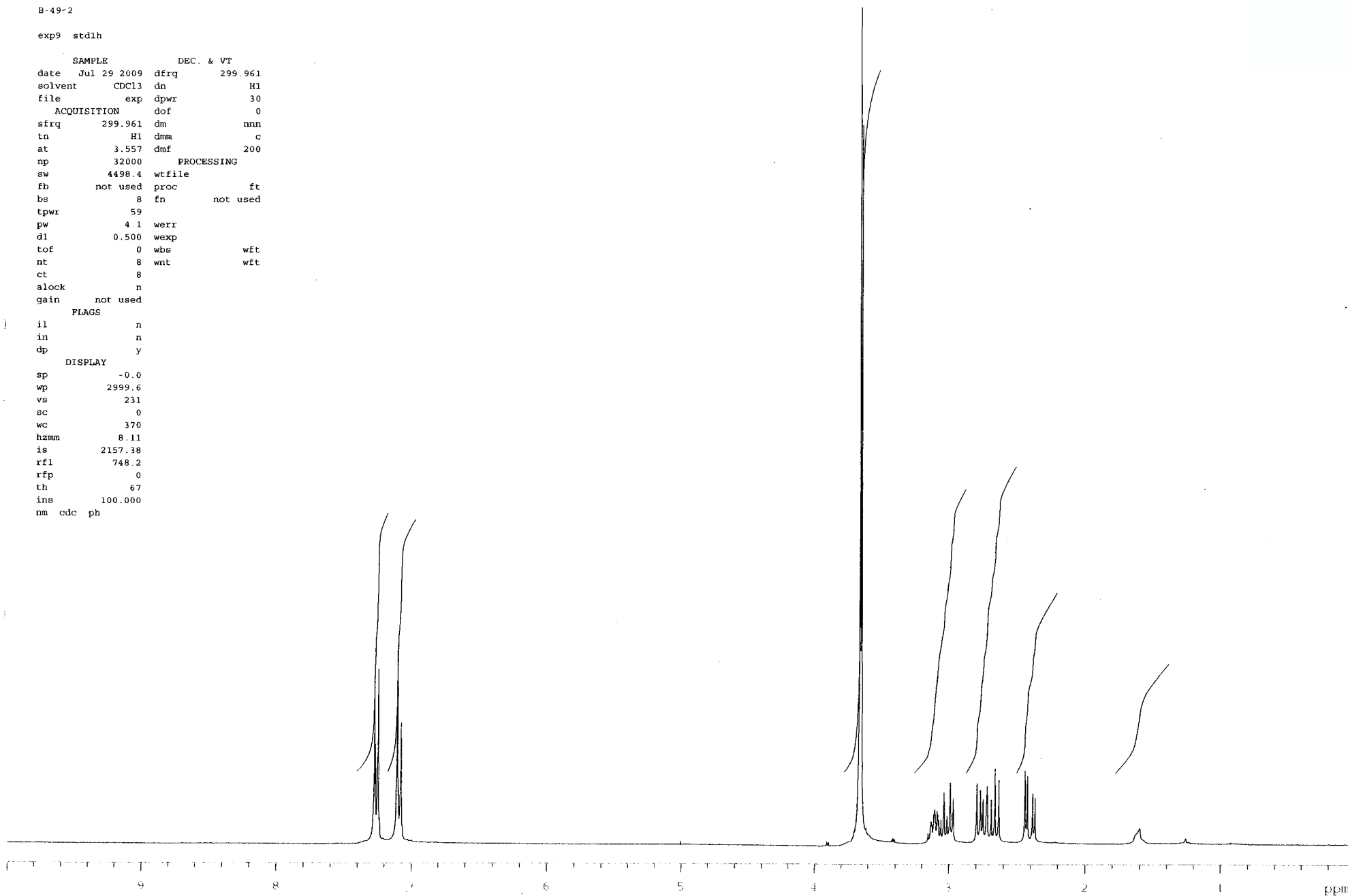
B-49-2

exp9 std1h

```
      SAMPLE          DEC. & VT
date   Jul 29 2009   dfrq      299.961
solvent CDCl3      dn          H1
file    exp         dpwr       30
ACQUISITION          dof       0
sfrq    299.961    dm          nnn
tn      H1         dmm         c
at      3.557     dmf         200
np      32000     PROCESSING
sw      4498.4    wtfile
fb      not used  proc          ft
bs      8         fn          not used
tpwr    59
pw      4.1      werr
dl      0.500    wexp
tof     0        wbs          wft
nt      8        wnt          wft
ct      8
alock   n
gain    not used
      FLAGS
il      n
in      n
dp      y
      DISPLAY
sp      -0.0
wp      2999.6
vs      231
sc      0
wc      370
hzmm    8.11
is      2157.38
rfl     748.2
rfp     0
th      67
ins     100.000
nm      cdc ph
```



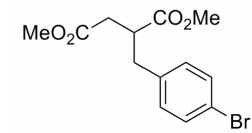
5i



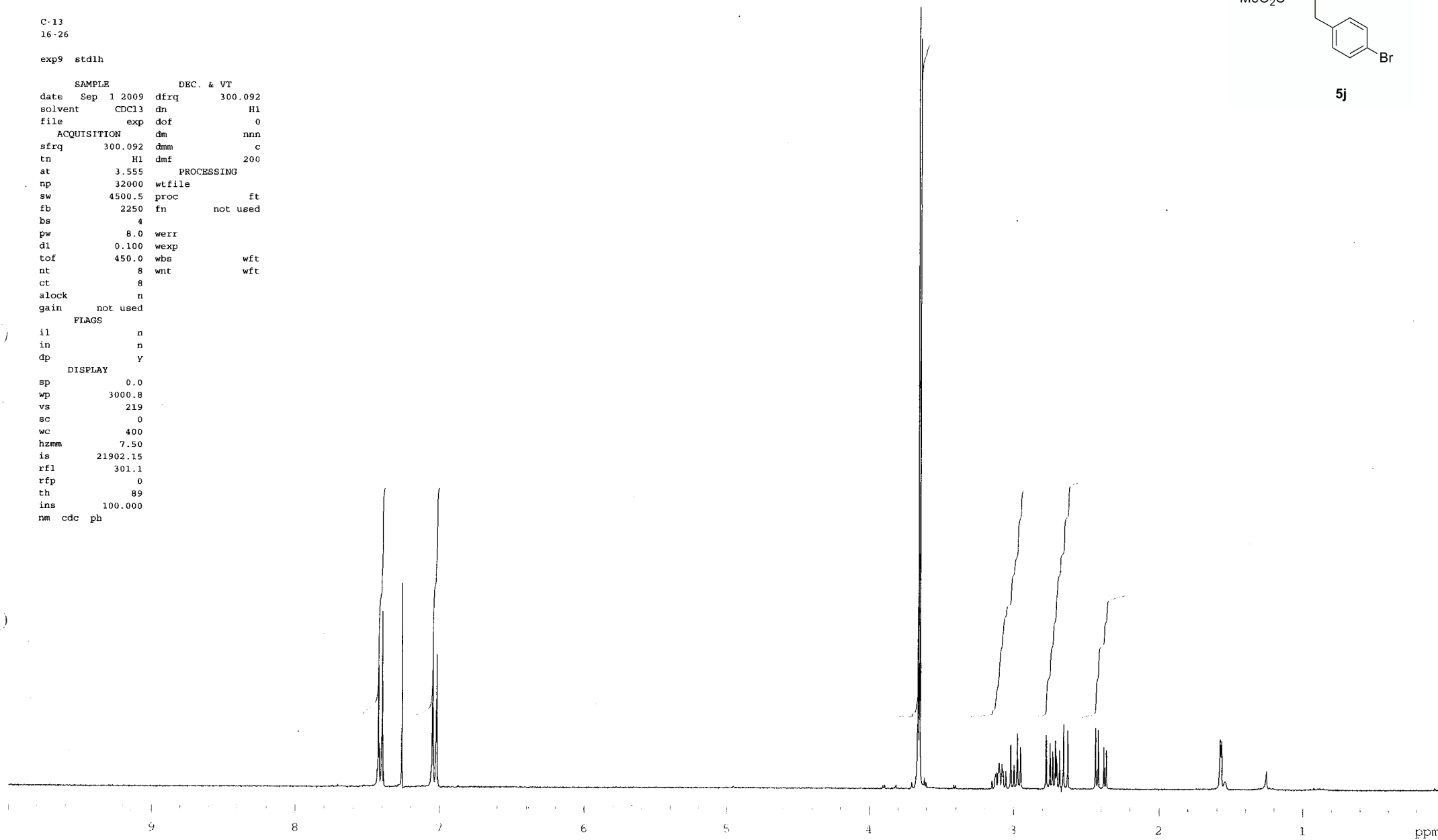
C-13
16-26

exp9 std1h

```
      SAMPLE      DEC. & VT
date  Sep 1 2009  dfrq    300.092
solvent  CDCl3  dn        H1
file      exp    dof        0
ACQUISITION      dm        nnn
sfrq    300.092  dmm        c
tn       H1     dmf        200
at       3.555   PROCESSING
np       32000  wtfile
sw       4500.5 proc        ft
fb       2250  fn        not used
bs        4
pw       8.0   werr
dl       0.100 wexp
tof      450.0 wbs        wft
nt        8   wnt        wft
ct        8
alock    n
gain    not used
      FLAGS
il       n
in       n
dp       y
      DISPLAY
sp        0.0
wp      3000.8
vs       219
sc        0
wc       400
hzmm     7.50
is     21902.15
rfl      301.1
rfp       0
th       89
ins     100.000
nm  cdc  ph
```



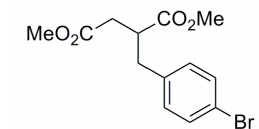
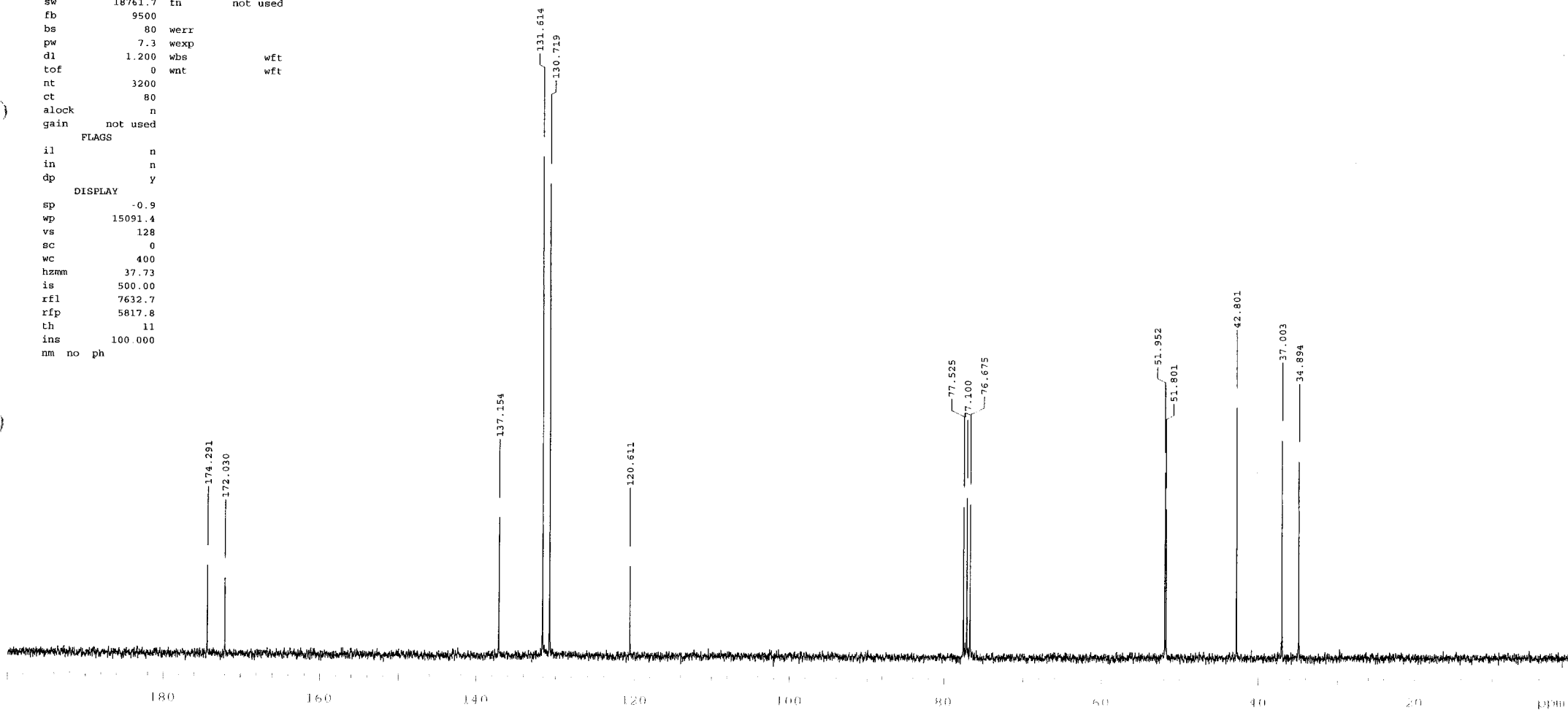
5j



C-13
16-26

exp2 std13c

```
SAMPLE          DEC. & VT
date  Nov 30 2009  dfrq      300.092
solvent  CDCl3      dn        H1
file  /net/kp010003  dhp      0.5
/export/home/vnmr1-  dof      0
/gem300/yakuhin/no-  dm      yyy
v/kon_c_13  fid      dmm      w
ACQUISITION      dmf      8800
sfrq      75.465      PROCESSING
tn         C13      lb        1.00
at         0.853      wtfile
np         32000      proc      ft
sw         18761.7    fn        not used
fb         9500
bs         80      werr
pw         7.3      wexp
dl         1.200      wbs      wft
tof        0      wnt      wft
nt         3200
ct         80
alock      n
gain      not used
FLAGS
il         n
in         n
dp         y
DISPLAY
sp         -0.9
wp         15091.4
vs         128
sc         0
wc         400
hzmh      37.73
is         500.00
rfl       7632.7
rfp       5817.8
th        11
ins       100.000
nm no ph
```

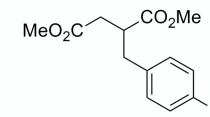


5j

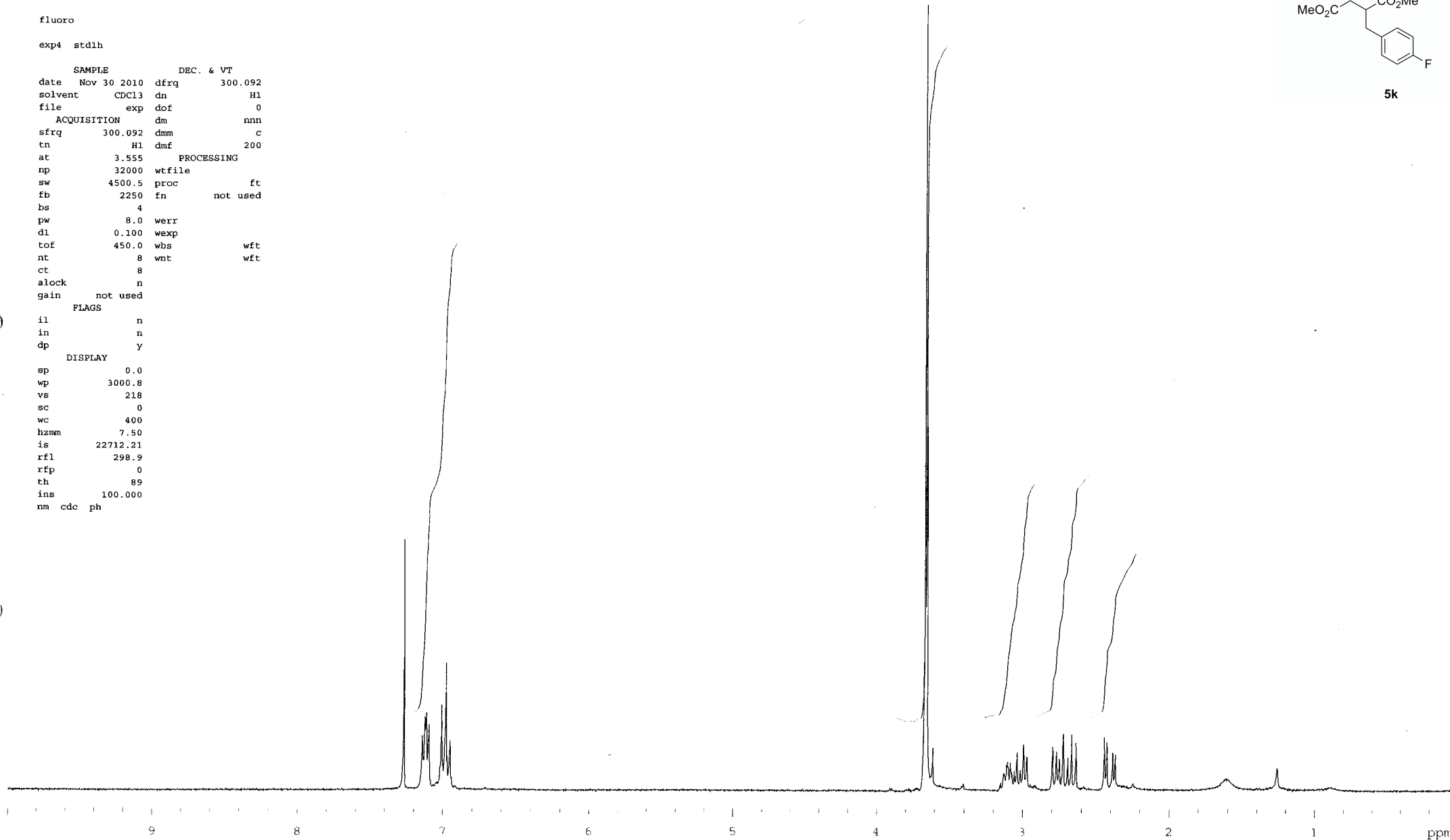
fluoro

exp4 stdlh

SAMPLE		DEC. & VT	
date	Nov 30 2010	dfrq	300.092
solvent	CDCl3	dn	H1
file	exp	dof	0
ACQUISITION		PROCESsing	
sfrq	300.092	dmm	nnn
tn	H1	dmf	c
at	3.555		200
np	32000	wtfile	
sw	4500.5	proc	ft
fb	2250	fn	not used
bs	4		
pw	8.0	werr	
dl	0.100	wexp	
tof	450.0	wbs	wft
nt	8	wnt	wft
ct	8		
alock	n		
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
DISPLAY			
sp	0.0		
wp	3000.8		
vs	218		
sc	0		
wc	400		
hzmn	7.50		
is	22712.21		
rfl	298.9		
rfp	0		
th	89		
ins	100.000		
nm	cdc ph		



5k



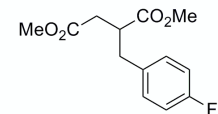
C-12-1

exp9 std13c

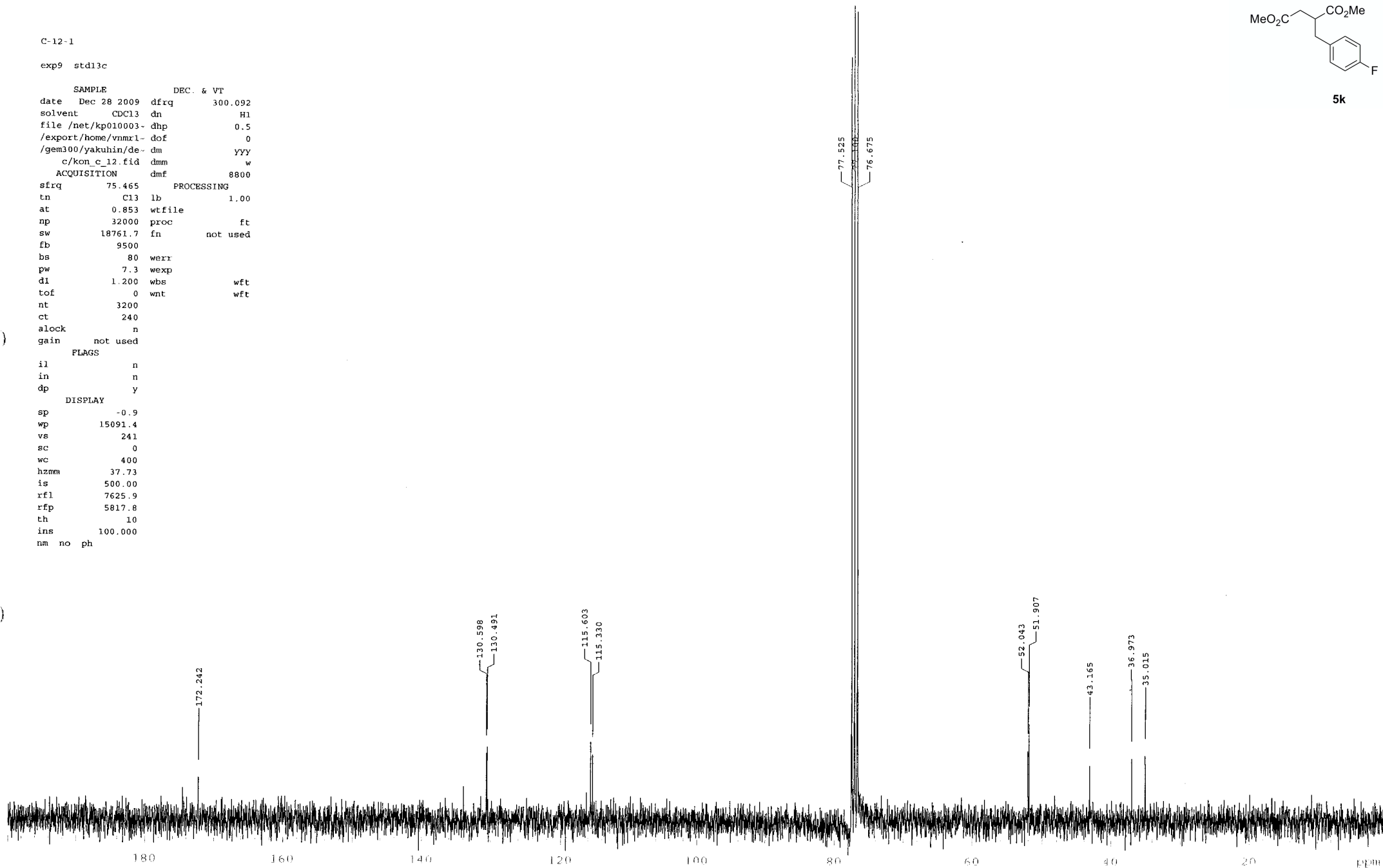
```
SAMPLE          DEC. & VT
date  Dec 28 2009  dfrq  300.092
solvent  CDC13  dn      H1
file  /net/kp010003- dhp  0.5
/export/home/vnmr1- dof  0
/gem300/yakuhin/de- dm   YYY
c/kon_c_12.fid  dmm   w
ACQUISITION  dmf     8800
sfrq  75.465  PROCESSING
tn     C13  lb   1.00
at     0.853  wtfile
np     32000  proc  ft
sw    18761.7  fn   not used
fb     9500
bs     80  werr
pw     7.3  wexp
dl     1.200  wbs   wft
tof     0  wnt   wft
nt     3200
ct     240
alock  n
gain  not used

FLAGS
il     n
in     n
dp     y

DISPLAY
sp     -0.9
wp    15091.4
vs     241
sc     0
wc     400
hzmm   37.73
is     500.00
rfl    7625.9
rfp    5817.8
th     10
ins    100.000
nm  no  ph
```



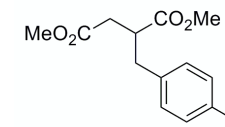
5k



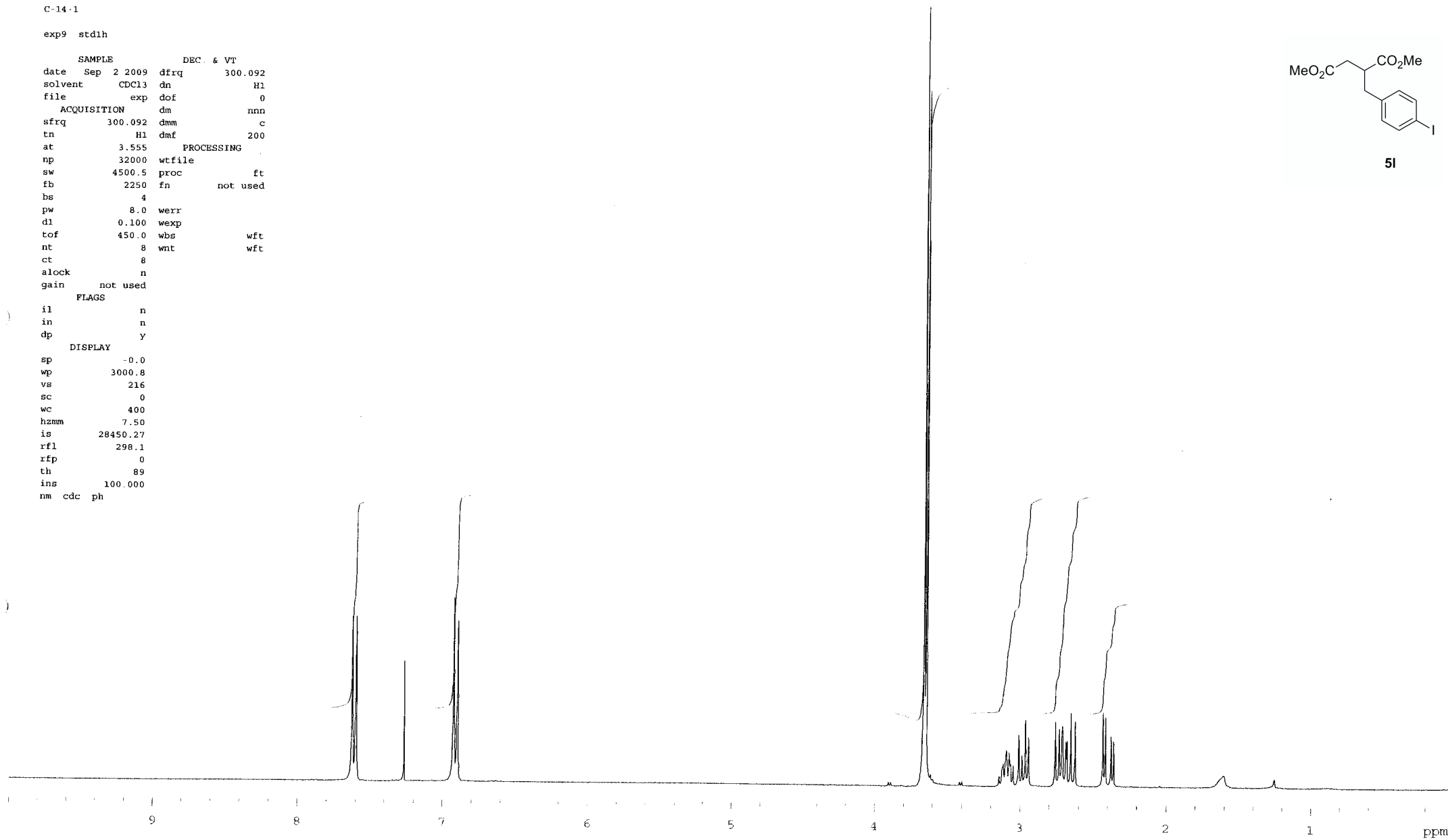
C-14-1

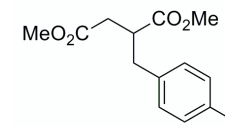
exp9 stdih

```
SAMPLE          DEC. & VT
date Sep 2 2009 dfrq          300.092
solvent CDC13   dn           H1
file      exp   dof           0
ACQUISITION    dm           nnn
sfrq      300.092 dmm         c
tn        H1   dmf           200
at        3.555   PROCESSING
np        32000  wtfile
sw        4500.5  proc           ft
fb        2250   fn           not used
bs         4
pw         8.0   werr
dl         0.100 wexp
tof        450.0  wbs           wft
nt         8     wnt           wft
ct         8
alock      n
gain      not used
FLAGS
il         n
in         n
dp         y
DISPLAY
sp         -0.0
wp        3000.8
vs         216
sc         0
wc         400
hzmm      7.50
is        28450.27
rfl       298.1
rfp        0
th         89
ins       100.000
nm cdc ph
```



5I





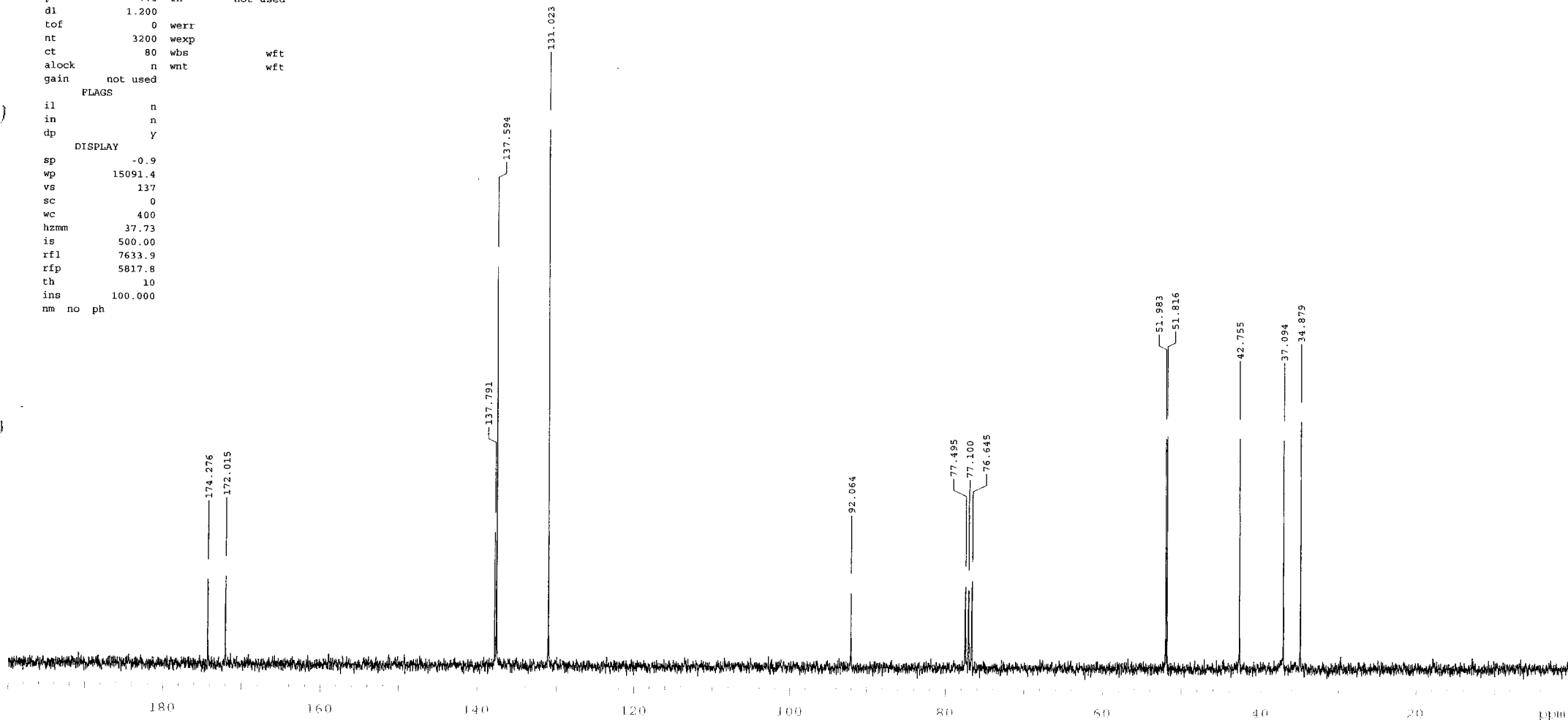
51

C-14-1

exp9 std13c

```

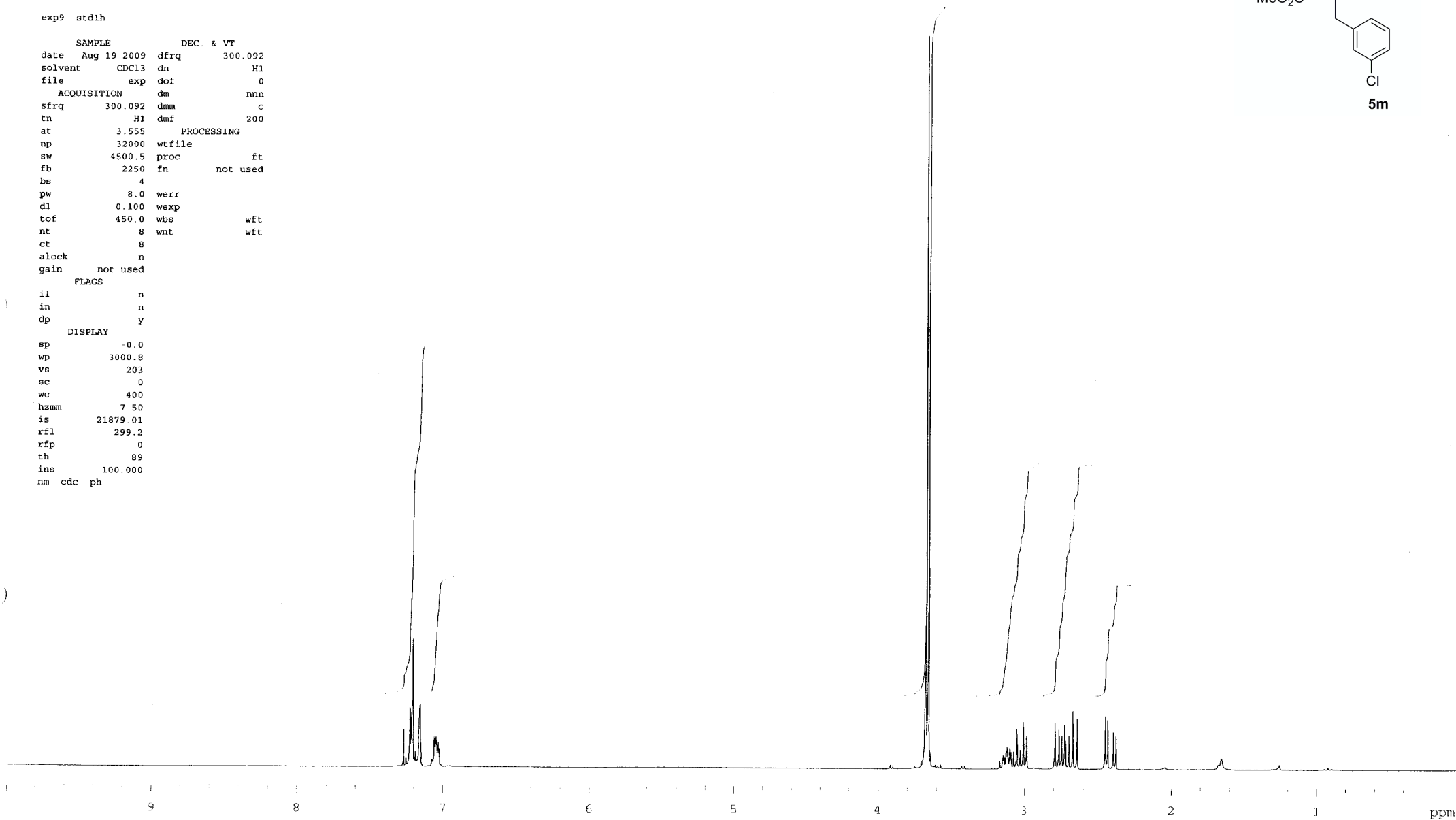
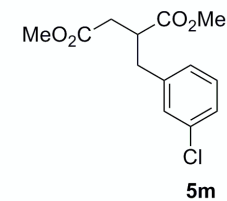
SAMPLE          DEC. & VT
date  Dec 28 2009  dfrq      300.092
solvent  CDCl3     dn        H1
file     exp      dhp        0.5
ACQUISITION
sfrq     75.465   dm         yyy
tn       C13     dmm        w
at       0.853   dmf        8800
np       32000
sw       18761.7 lb         1.00
fb       9500   wtfile
bs       80     proc        ft
pw       7.3   fn         not used
dl       1.200
tof      0     werr
nt       3200  wexp
ct       80   wbs         wft
alock    n     wnt         wft
gain     not used
FLAGS
il       n
in       n
dp       Y
DISPLAY
sp      -0.9
wp     15091.4
vs      137
sc       0
wc      400
hzmm    37.73
is      500.00
rf1     7633.9
rfp     5817.8
th      10
ins     100.000
nm no ph
  
```

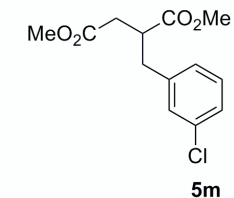


B-67-2

exp9 std1h

```
SAMPLE          DEC. & VT
date   Aug 19 2009   dfrq      300.092
solvent CDCl3      dn         H1
file           exp     dof         0
ACQUISITION
sfrq      300.092   dnm         c
tn         H1      dmf         200
at         3.555   PROCESSING
np         32000   wtfile
sw         4500.5   proc         ft
fb         2250    fn         not used
bs         4
pw         8.0    werr
dl         0.100   wexp
tof        450.0   wbs         wft
nt         8      wnt         wft
ct         8
alock      n
gain      not used
FLAGS
il         n
in         n
dp         Y
DISPLAY
sp         -0.0
wp         3000.8
vs         203
sc         0
wc         400
hzmm      7.50
is        21879.01
rfl       299.2
rfp        0
th         89
ins       100.000
nm cdc ph
```





B-67-2

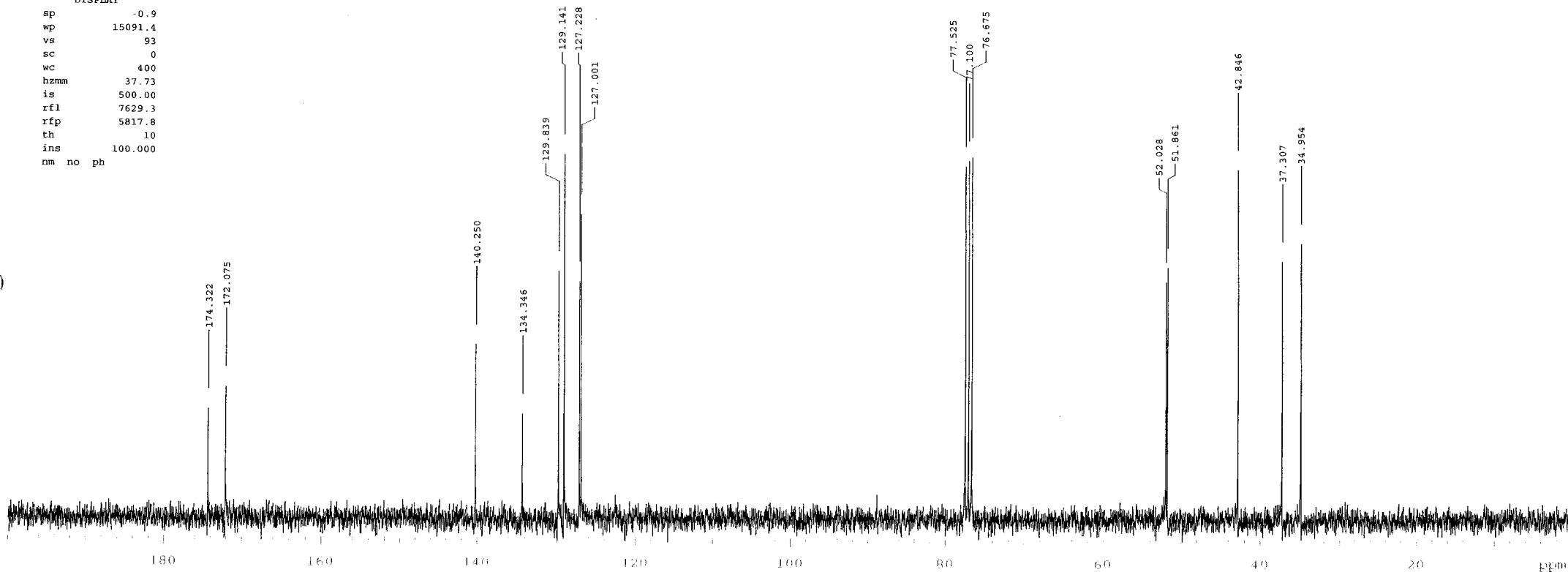
exp9 std13c

```

SAMPLE          DEC. & VT
date  Dec 28 2009  dfrq    300.092
solvent  CDCl3  dn      H1
file     exp   dhp      0.5
          ACQUISITION
sfrq     75.465  dm      yyy
tn       C13   dmm      w
at       0.853  dmf     8800
np       32000  PROCESSING
sw       18761.7 lb      1.00
fb       9500  wtfile
bs       80    proc     ft
pw       7.3  fn      not used
dl       1.200
tof      0    werr
nt       3200  wexp
ct       80   wbs      wft
alock    n    wnt      wft
gain     not used

FLAGS
il       n
in       n
dp       y

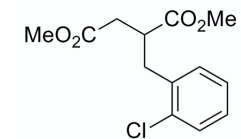
DISPLAY
sp       -0.9
wp       15091.4
vs       93
sc       0
wc       400
hzmh    37.73
is       500.00
rfl     7629.3
rfp     5817.8
th       10
ins     100.000
nm no ph
  
```



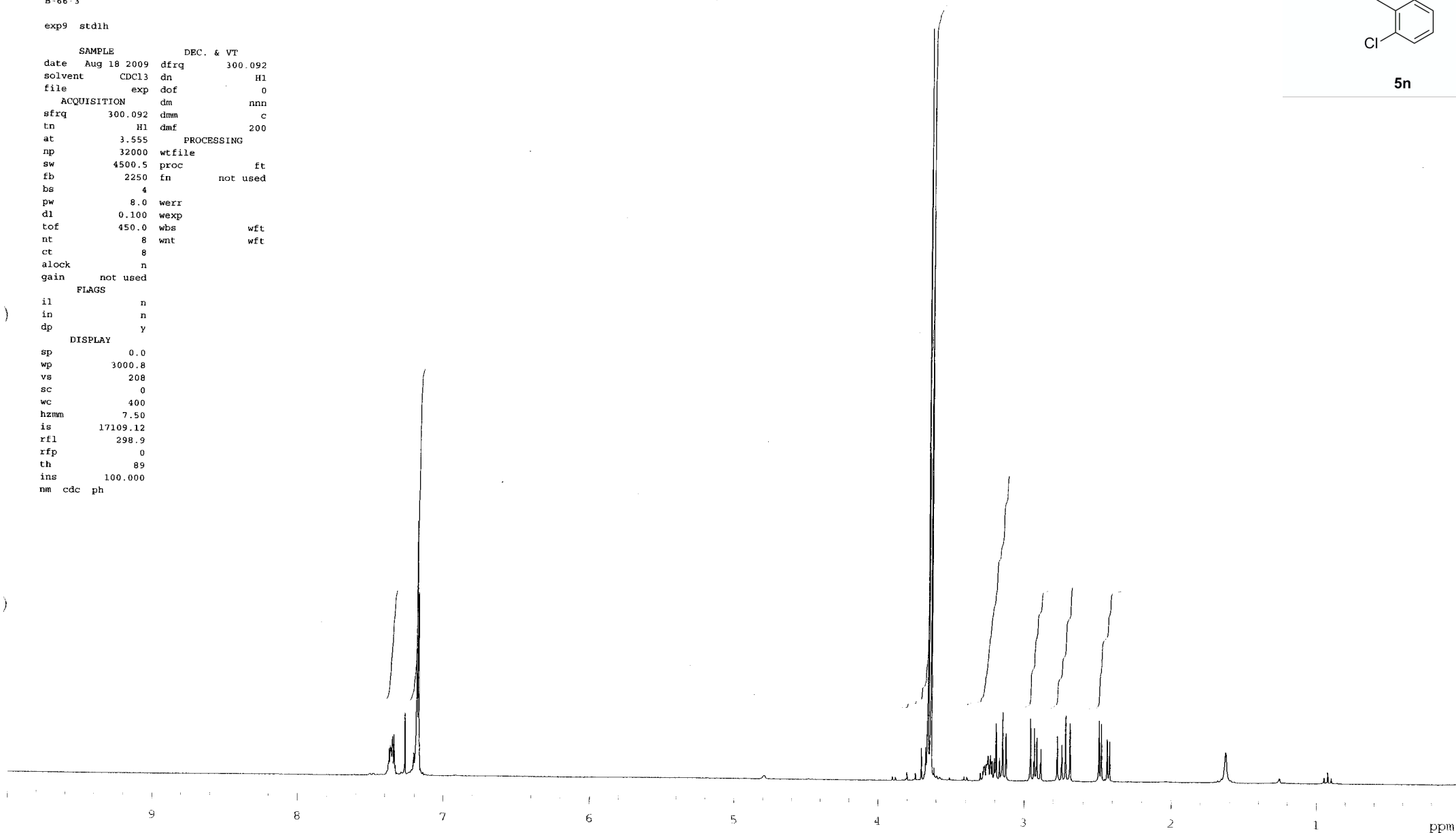
B-66-3

exp9 stdih

```
SAMPLE          DEC. & VT
date   Aug 18 2009   dfrq   300.092
solvent  CDCl3      dn      H1
file    exp         dof     0
ACQUISITION dm      nnn
sfrq    300.092   dmm     c
tn      H1        dmf     200
at      3.555     PROCESSING
np      32000    wtfile
sw      4500.5   proc     ft
fb      2250    fn      not used
bs      4
pw      8.0     werr
dl      0.100   wexp
tof     450.0   wbs      wft
nt      8       wnt      wft
ct      8
alock   n
gain    not used
FLAGS
il      n
in      n
dp      y
DISPLAY
sp      0.0
wp      3000.8
vs      208
sc      0
wc      400
hzmm    7.50
is      17109.12
rfl     298.9
rfp     0
th      89
ins     100.000
nm cdc ph
```



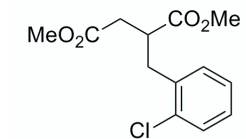
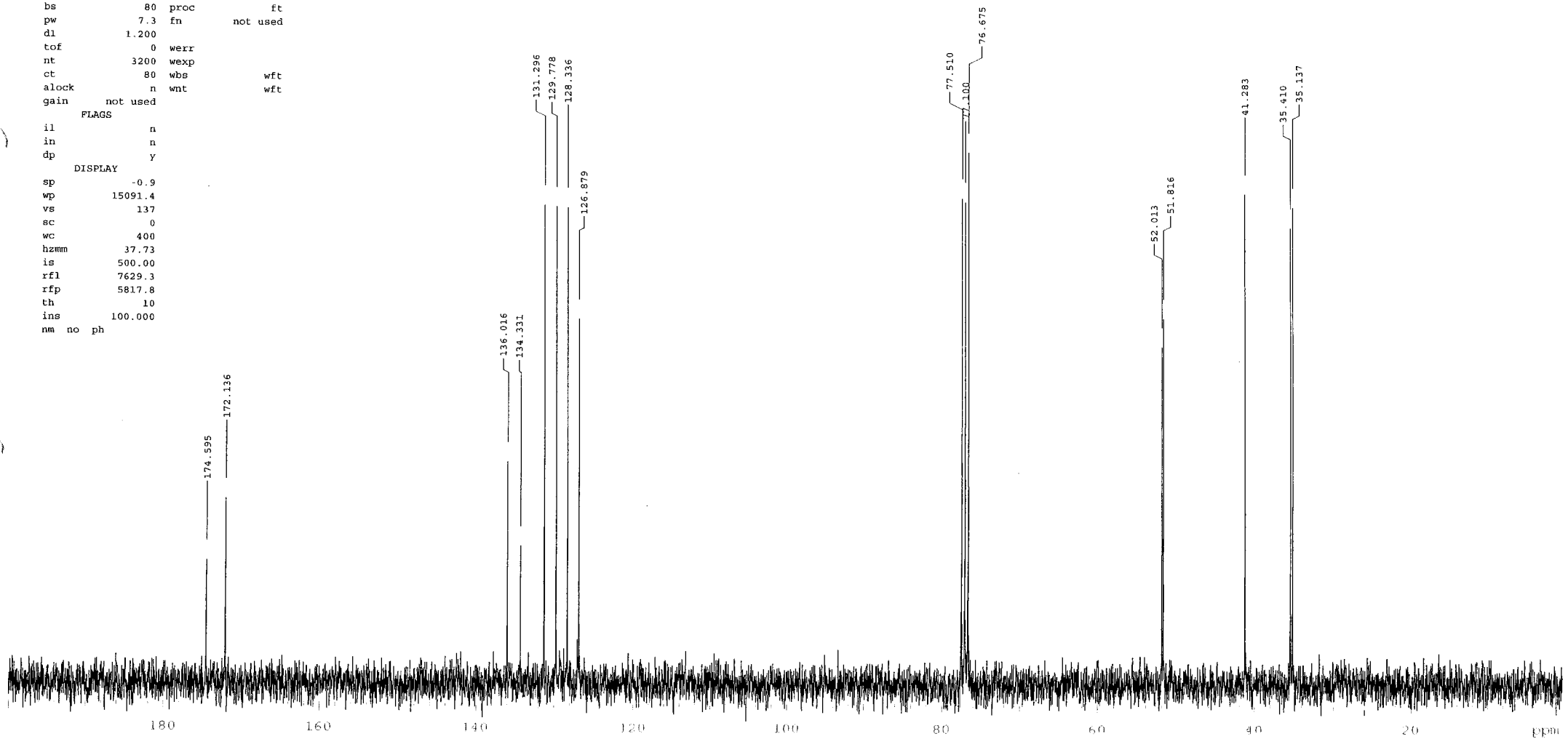
5n



B-66-3

exp9 std13c

```
SAMPLE          DEC. & VT
date   Dec 28 2009   dfrq      300.092
solvent  CDCl3      dn         H1
file    exp         dhp        0.5
          ACQUISITION
sfrq    75.465     dm         yyy
tn      C13        dmm        w
at      0.853     dmf        8800
np      32000      PROCESSING
sw      18761.7    lb         1.00
fb      9500      wtfile
bs      80        proc        ft
pw      7.3      fn         not used
dl      1.200
tof     0         werr
nt      3200     wexp
ct      80      wbs
alock   n        wnt         wft
gain    not used
          PLAYS
il      n
in      n
dp      y
          DISPLAY
sp      -0.9
wp      15091.4
vs      137
sc      0
wc      400
h2mm    37.73
is      500.00
rfl     7629.3
rfp     5817.8
th      10
ins     100.000
nm      no ph
```

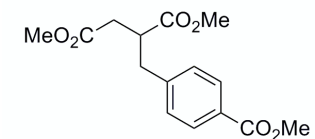


5n

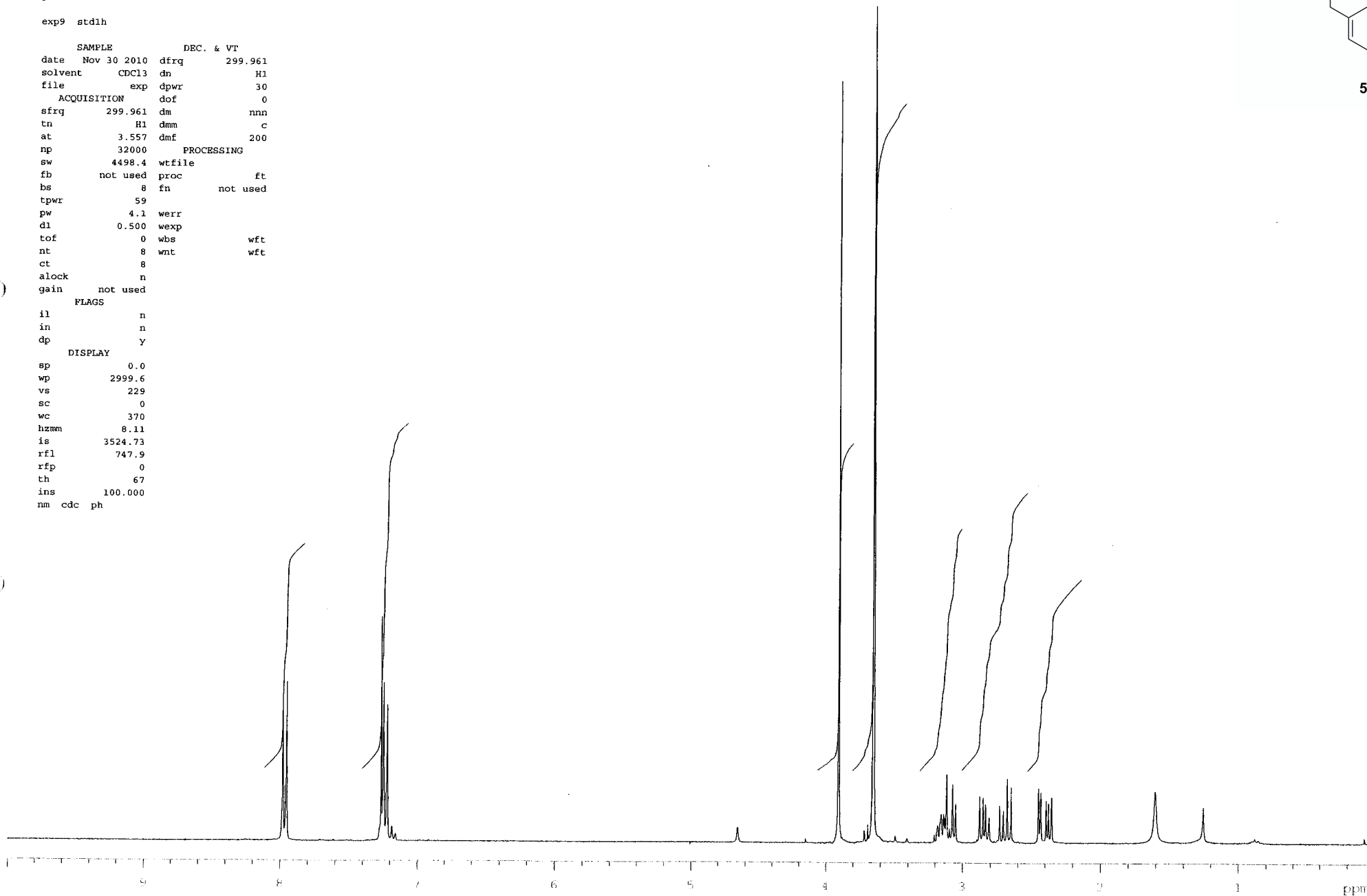
p-toluate

exp9 stdlh

```
      SAMPLE      DEC. & VT
date  Nov 30 2010  dfrq    299.961
solvent  CDCl3    dn      H1
file     exp      dpwr    30
ACQUISITION  dof      0
sfrq    299.961  dm      nnn
tn      H1      dmm      c
at      3.557   dmf      200
np      32000   PROCESSING
sw      4498.4  wtfile
fb      not used proc      ft
bs      8      fn      not used
tpwr    59
pw      4.1    werr
dl      0.500  wexp
tof     0      wbs      wft
nt      8      wnt      wft
ct      8
alock   n
gain    not used
      FLAGS
il      n
in      n
dp      Y
      DISPLAY
sp      0.0
wp      2999.6
vs      229
sc      0
wc      370
hzmm    8.11
is      3524.73
rfl     747.9
rfp     0
th      67
ins     100.000
nm      cdc   ph
```



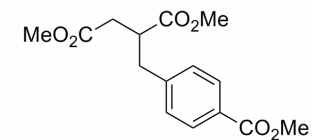
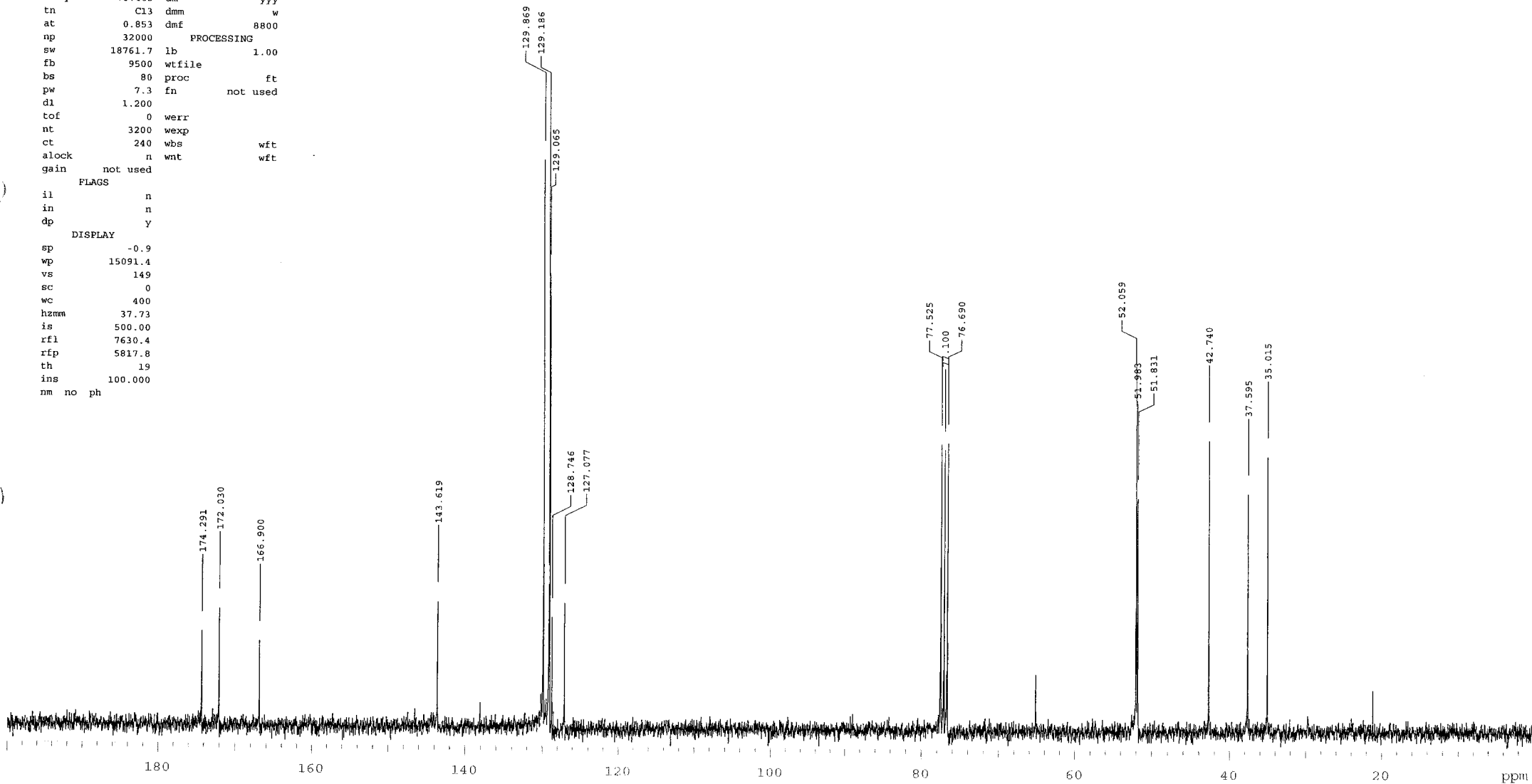
5o



C-1
26-43

exp9 std13c

```
SAMPLE          DEC. & VT
date Jun  9 2010 dfrq      300.092
solvent CDCl3  dn         H1
file      exp  dhp         0.5
ACQUISITION  dof         0
sfrq      75.465 dm        YYY
tn        C13  dmm         w
at        0.853 dmf        8800
np        32000
sw      18761.7 lb         1.00
fb        9500 wtfile
bs        80  proc         ft
pw        7.3  fn         not used
dl        1.200
tof        0  werr
nt        3200 wexp
ct        240  wbs         wft
alock     n  wnt         wft
gain     not used
FLAGS
il        n
in        n
dp        Y
DISPLAY
sp        -0.9
wp      15091.4
vs        149
sc         0
wc        400
hzmm     37.73
is        500.00
rfl      7630.4
rfp      5817.8
th        19
ins      100.000
nm no ph
```

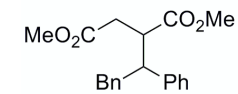


5o

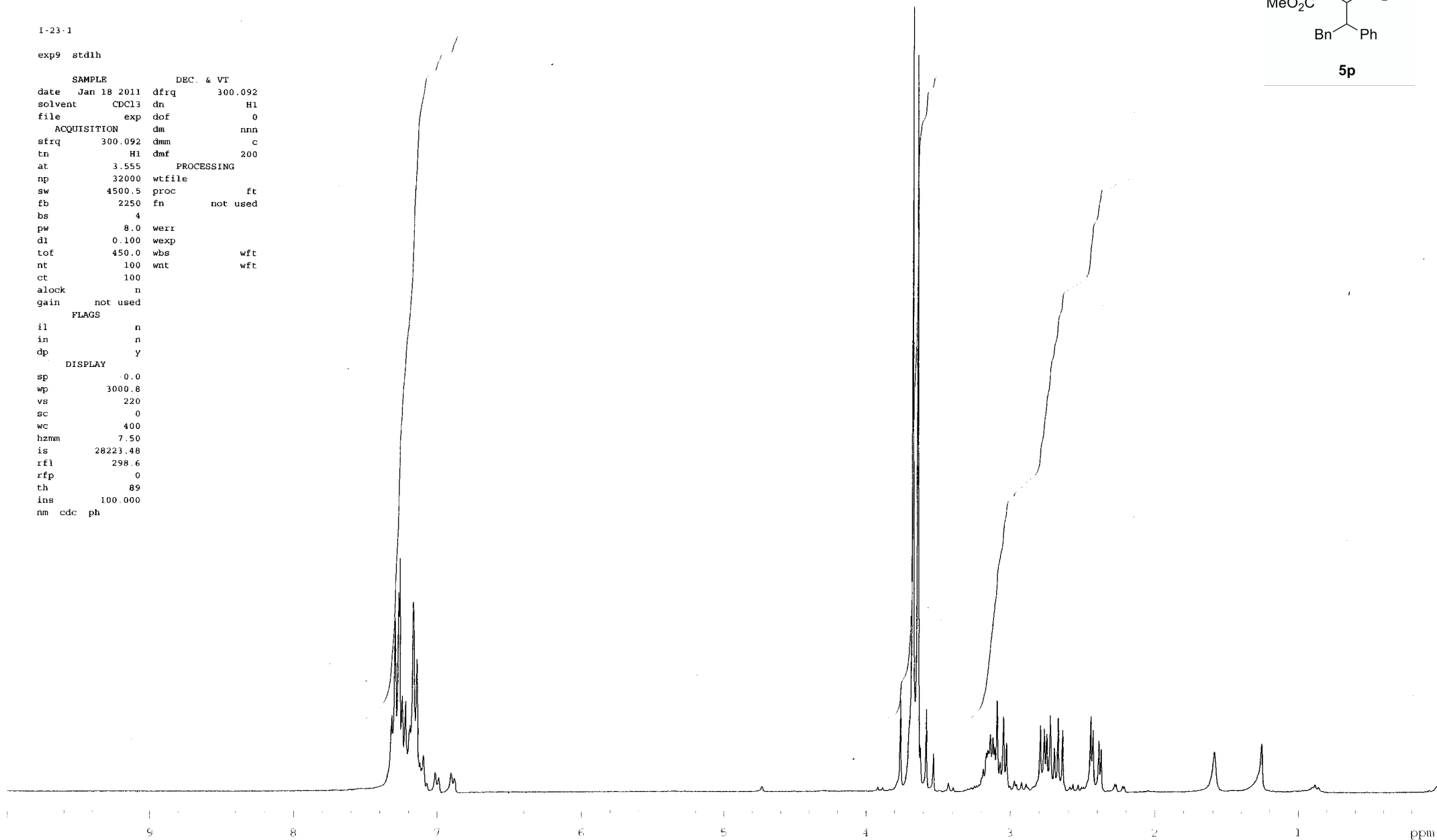
I-23-1

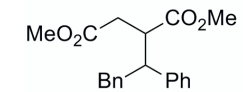
exp9 stdlh

```
SAMPLE          DEC. & VT
date  Jan 18 2011  dfrq    300.092
solvent  CDCl3    dn      H1
file     exp     dof      0
          ACQUISITION  dm      nnn
sfrq    300.092  dmm     c
tn      H1      dmf     200
at      3.555   PROCESSING
np      32000   wtfile
sw      4500.5  proc      ft
fb      2250   fn      not used
bs      4
pw      8.0    weirr
dl      0.100  wexp
tof     450.0  wbs      wft
nt      100   wnt      wft
ct      100
alock   n
gain    not used
          FLAGS
il      n
in      n
dp      y
          DISPLAY
sp      0.0
wp      3000.8
vs      220
sc      0
wc      400
hzmm    7.50
is      28223.48
rfl     298.6
rfp     0
th      89
ins     100.000
nm      cdc  ph
```



5p





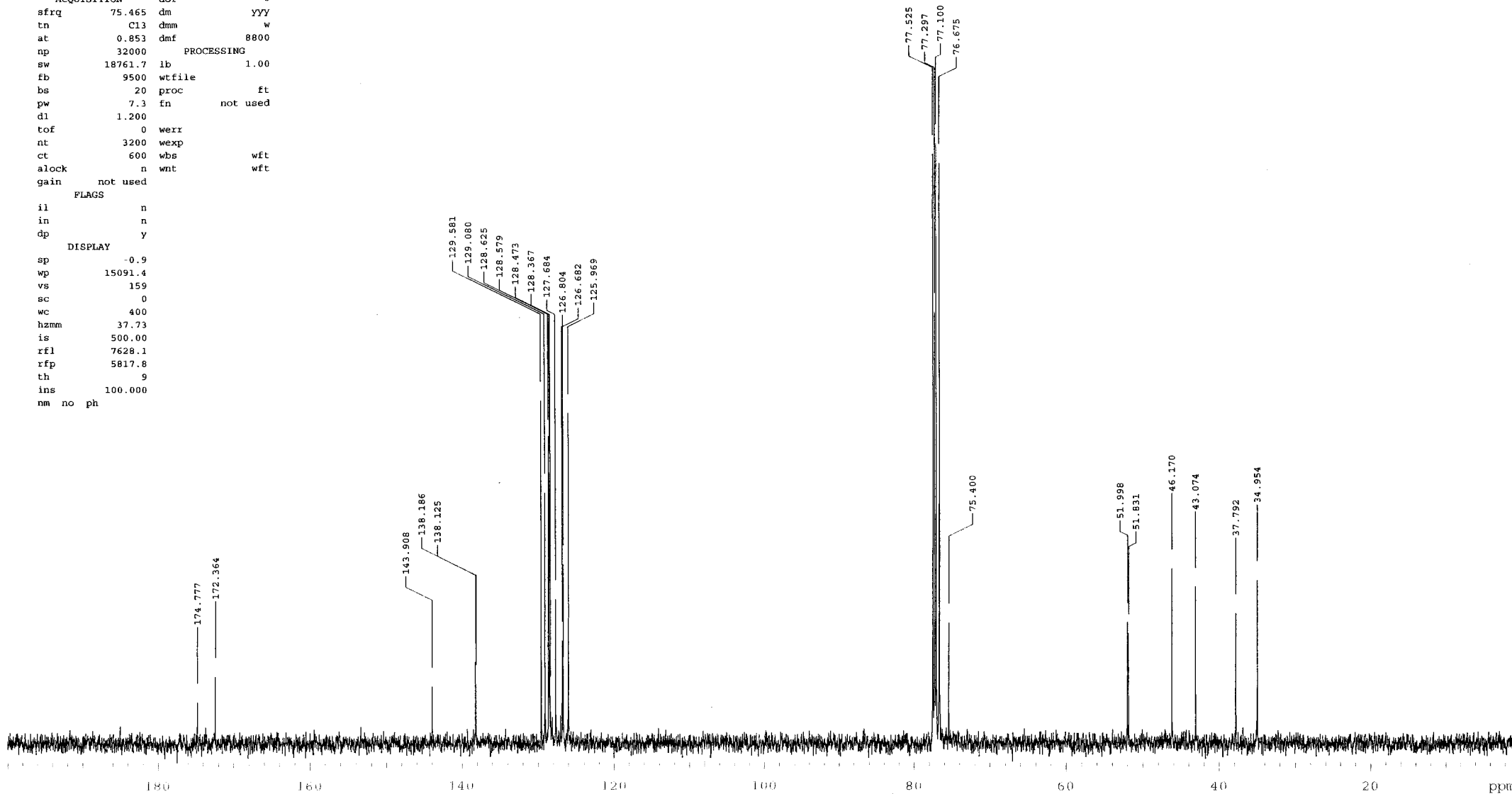
5p

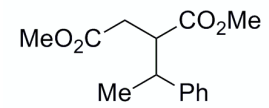
I-3 A-3

exp9 std13c

```

SAMPLE          DEC. & VT
date   Dec 4 2010   dfrq   300.092
solvent CDCl3      dn      H1
file    exp dhp      0.5
ACQUISITION
sfrq    75.465   dm      YYY
tn      C13     dmm      w
at      0.853   dmf      8800
np      32000   PROCESSING
sw      18761.7 lb      1.00
fb      9500   wtfile
bs      20     proc
pw      7.3   fn      not used
dl      1.200
tof      0     werr
nt      3200  wexp
ct      600   wbs
alock    n    wnt      wft
gain    not used
FLAGS
il      n
in      n
dp      y
DISPLAY
sp      -0.9
wp      15091.4
vs      159
sc      0
wc      400
hzmm    37.73
is      500.00
rfl     7628.1
rfp     5817.8
th      9
ins     100.000
nm no ph
  
```



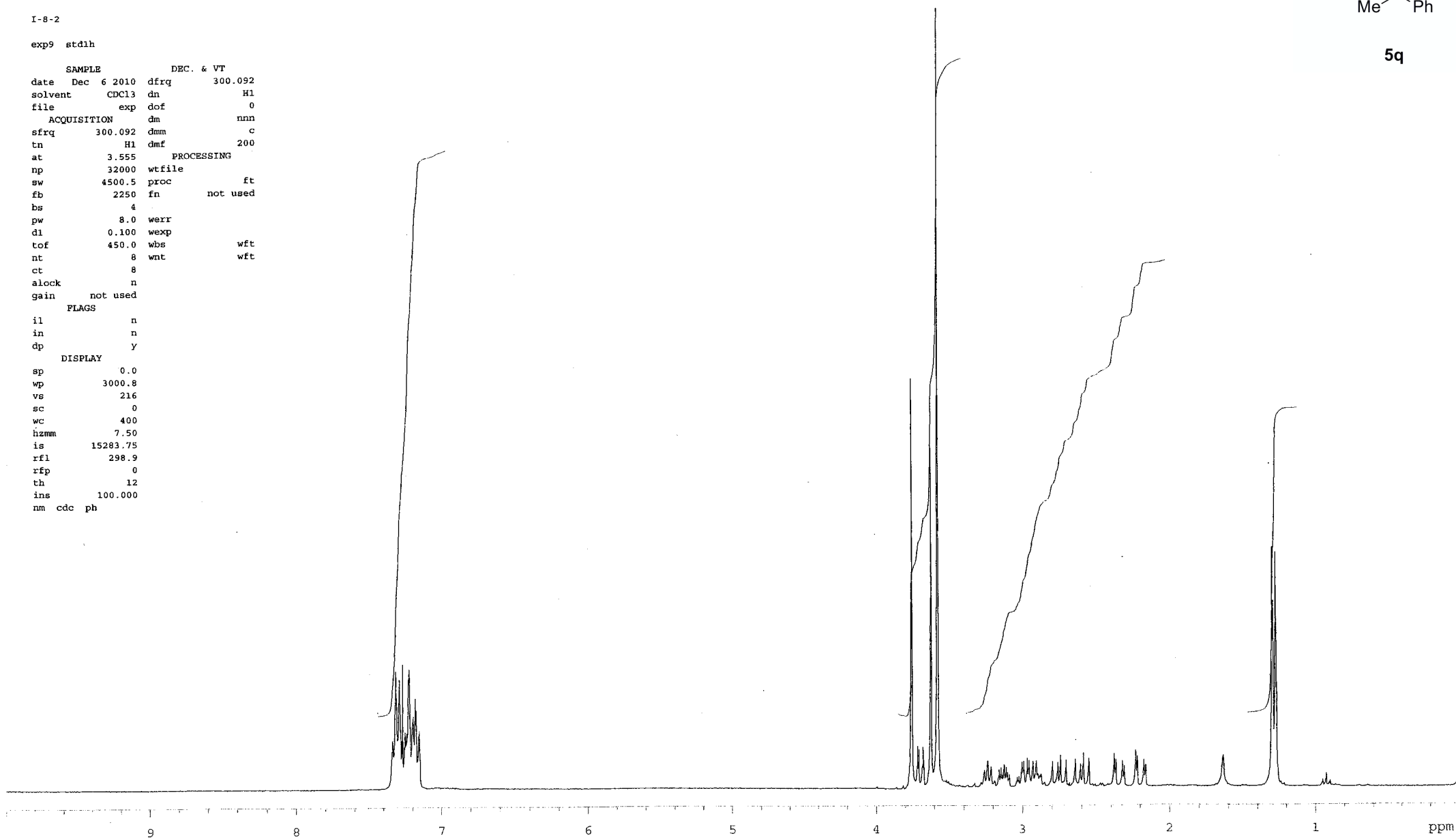


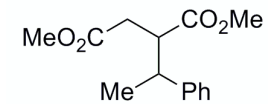
5q

I-8-2

exp9 stdlh

SAMPLE		DEC. & VT	
date	Dec 6 2010	dfrq	300.092
solvent	CDCl3	dn	H1
file	exp	dof	0
ACQUISITION		dm	nnn
sfrq	300.092	dmm	c
tn	H1	dmf	200
at	3.555	PROCESSING	
np	32000	wtfile	
sw	4500.5	proc	ft
fb	2250	fn	not used
bs	4		
pw	8.0	werrf	
dl	0.100	wexp	
tof	450.0	wbs	wft
nt	8	wnt	wft
ct	8		
alock	n		
gain	not used		
FLAGS			
il	n		
in	n		
dp	Y		
DISPLAY			
sp	0.0		
wp	3000.8		
vs	216		
sc	0		
wc	400		
hzmm	7.50		
is	15283.75		
rfl	298.9		
rfp	0		
th	12		
ins	100.000		
nm	cdc ph		





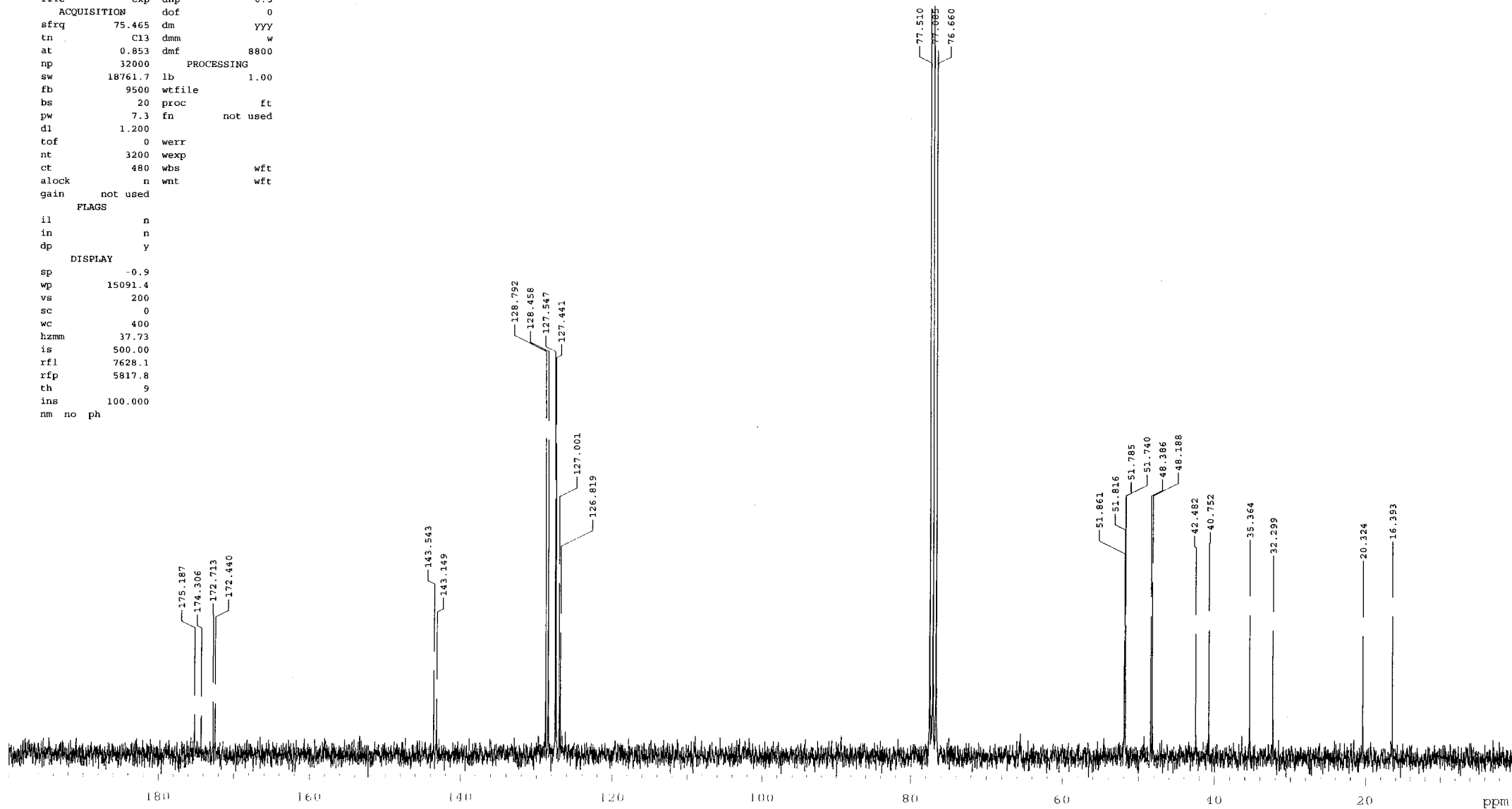
5q

ethylbenzene

exp9 std13c

```

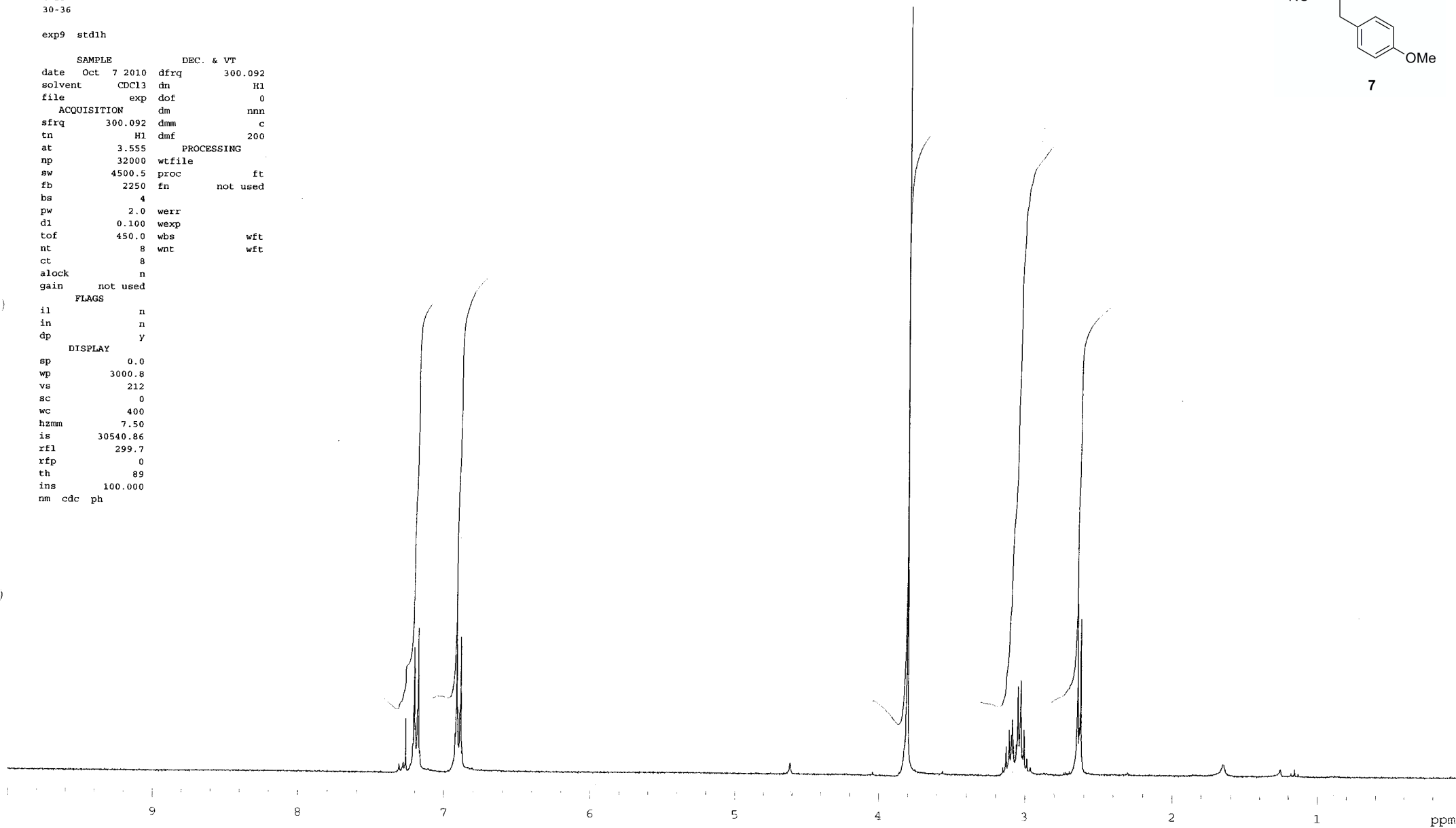
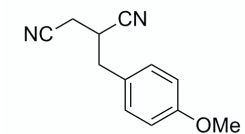
SAMPLE          DEC. & VT
date Dec 6 2010 dfrq      300.092
solvent CDCl3   dn        H1
file          exp dhp      0.5
ACQUISITION
sfrq      75.465 dm       YYY
tn         C13 dmm       w
at         0.853 dmf      8800
np         32000
sw         18761.7 lb     1.00
fb         9500 wtfile
bs         20  proc      ft
pw         7.3  fn      not used
dl         1.200
tof         0   werr
nt         3200 wexp
ct         480 wbs
alock      n   wnt      wft
gain       not used
PLAGS
il         n
in         n
dp         y
DISPLAY
sp         -0.9
wp         15091.4
vs         200
sc         0
wc         400
hzmm      37.73
is         500.00
rfl       7628.1
rfp       5817.8
th         9
ins       100.000
nm no ph
  
```

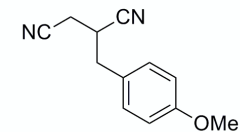


C-25
30-36

exp9 std1h

SAMPLE		DEC. & VT	
date	Oct 7 2010	dfrq	300.092
solvent	CDCl3	dn	H1
file	exp	dof	0
ACQUISITION		nmn	
sfrq	300.092	dmm	c
tn	H1	dmf	200
at	3.555	PROCESSING	
np	32000	wtfile	
sw	4500.5	proc	ft
fb	2250	fn	not used
bs	4		
pw	2.0	werr	
d1	0.100	wexp	
tof	450.0	wbs	wft
nt	8	wnt	wft
ct	8		
alock	n		
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
DISPLAY			
sp	0.0		
wp	3000.8		
vs	212		
sc	0		
wc	400		
hzmm	7.50		
is	30540.86		
rfl	299.7		
rfp	0		
th	89		
ins	100.000		
nm	cdc	ph	





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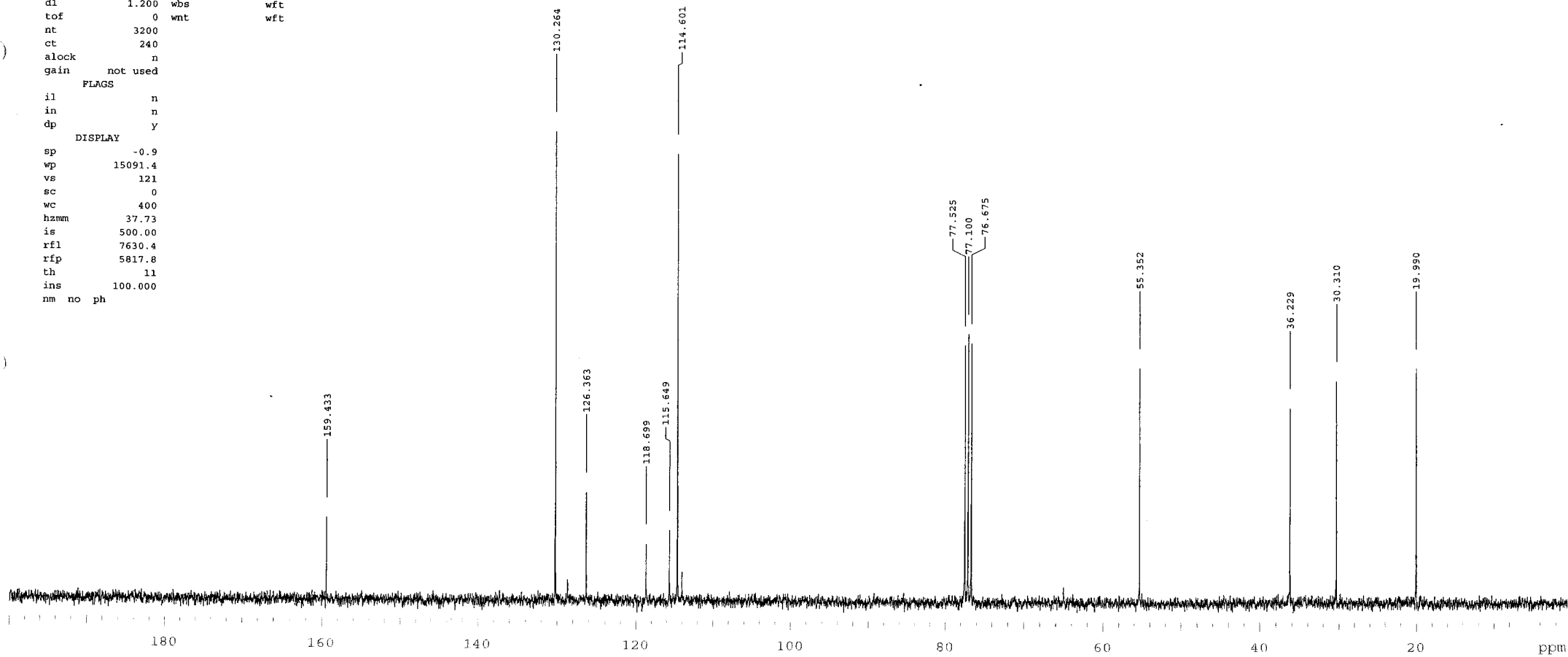
```

C-25
30-36

exp9 std13c

SAMPLE          DEC. & VT
date   Oct  7 2010  dfrq   300.092
solvent  CDCl3  dn      H1
file  /net/kp010003- dhp    0.5
/export/home/vnmr1- dof    0
/gem300/yakuhin/se- dm    yyy
p/kon_c_25.fid  dmm      w
ACQUISITION    dmf      8800
sfrq   75.465      PROCESSING
tn      C13  lb      1.00
at      0.853  wtfile
np      32000  proc    ft
sw      18761.7  fn    not used
fb      9500
bs      80  werr
pw      7.3  wexp
dl      1.200  wbs    wft
tof      0  wnt    wft
nt      3200
ct      240
alock   n
gain   not used
FLAGS
il      n
in      n
dp      y
DISPLAY
sp      -0.9
wp      15091.4
vs      121
sc      0
wc      400
hzmm    37.73
is      500.00
rf1     7630.4
rfp     5817.8
th      11
ins     100.000
nm  no  ph

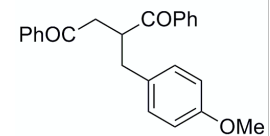
```



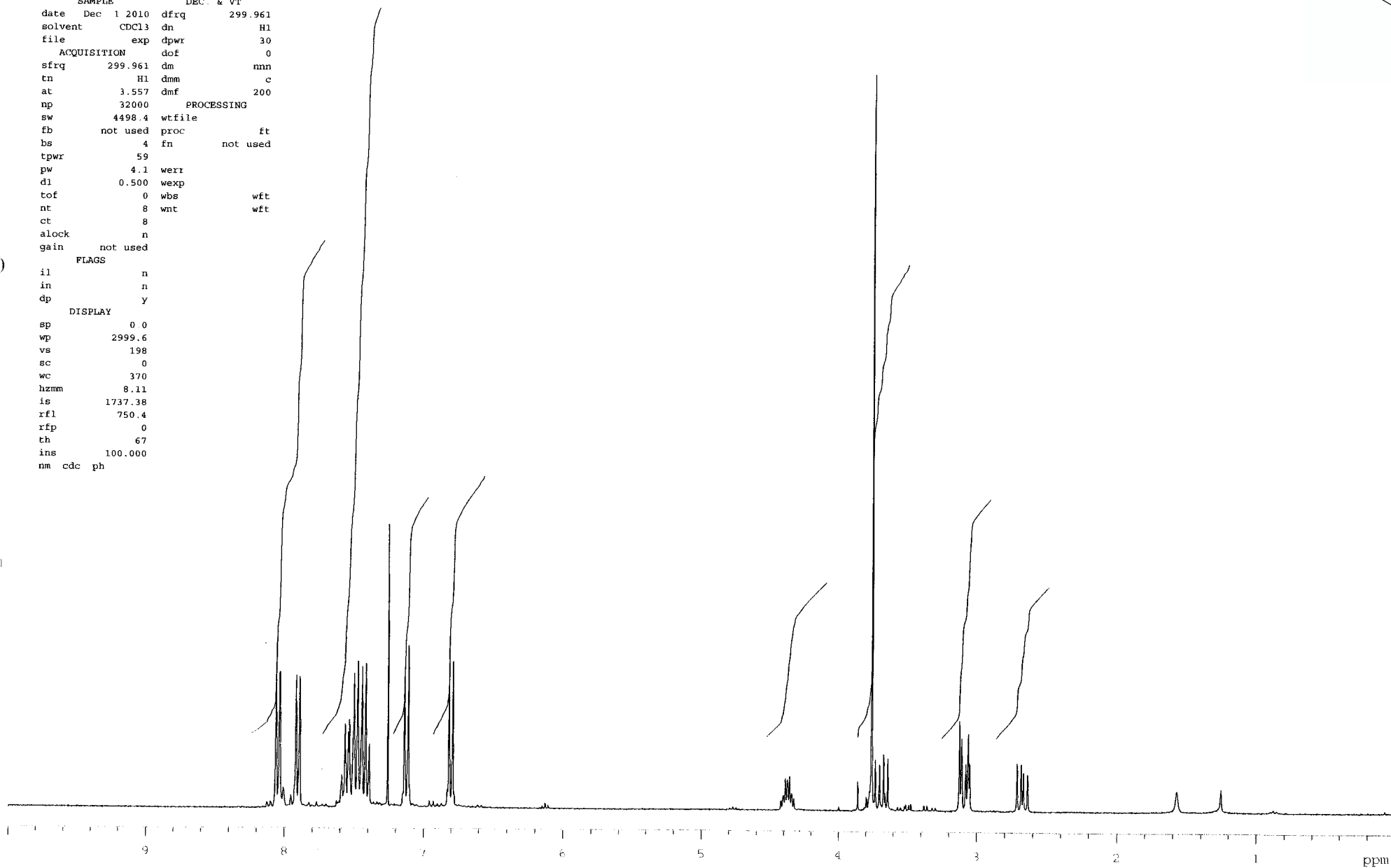
C-33

expl stdlh

```
SAMPLE      DEC & VT
date Dec 1 2010 dfrq      299.961
solvent CDCl3 dn          H1
file      exp dpwr       30
ACQUISITION dof          0
sfrq      299.961 dm      nnn
tn         H1 dmm        c
at         3.557 dmf      200
np         32000 PROCESSING
sw         4498.4 wtfile
fb not used proc        ft
bs         4 fn         not used
tpwr       59
pw         4.1 werr
dl         0.500 wexp
tof        0 wbs        wft
nt         8 wnt        wft
ct         8
alock      n
gain not used
FLAGS
il         n
in         n
dp         y
DISPLAY
sp         0 0
wp         2999.6
vs         198
sc         0
wc         370
hzmm       8.11
is         1737.38
rfl        750.4
rfp        0
th         67
ins        100.000
nm cdc ph
```



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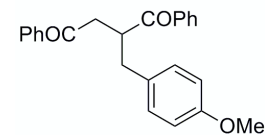
C-33-2

exp9 std13c

SAMPLE DEC. & VT
date Jun 13 2010 dfrq 300.092
solvent CDCl3 dn H1
file exp dhp 0.5
ACQUISITION dof 0
sfrq 75.465 dm yyy
tn C13 dmm w
at 0.853 dmf 8800
np 32000 PROCESSING
sw 18761.7 lb 1.00
fb 9500 wtfile
bs 80 proc ft
pw 7.3 fn not used
dl 1.200
tof 0 werr
nt 3200 wexp
ct 400 wbs wft
alock n wnt wft
gain not used

FLAGS
il n
in n
dp y

DISPLAY
sp -0.9
wp 16599.6
vs 145
sc 0
wc 400
hzmm 41.50
is 500.00
rf1 7628.1
rfp 5817.8
th 6
ins 100.000
na no ph



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