Supplementary Material (ESI) for Chemical Communications

Supporting Information

Gold-catalyzed Oxidative Cleavage of Aryl-Substituted Alkynyl Ethers using Molecular Oxygen. Simultaneous Degradation of C-H and Single and Triple Carbon-Carbon bonds under Ambient Conditions

Arindam Das, Rupsha Chaudhuri and Rai-Shung Liu*

Department of Chemistry, National Tsing-Hua University, Hsinchu, Taiwan, ROC

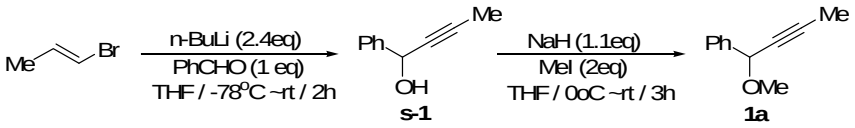
----------*e-mail: rsliu@mx.nthu.edu.tw*

Contents

(I) General Procedures

Unless otherwise stated, all commercial reagents were used without additional purification. Solvents were dried using standard methods and distilled before use. All reactions were carried out in oven-dried glassware using standard syringe, cannula, septa and other apparatus. NMR spectra were recorded at 400/600 MHz for ^{1}H NMR and 100/150 MHz for ^{13}C NMR in $CDCl_3$ or CD_2Cl_2 with tetramethylsilane (TMS) as internal standard. The chemical shifts are expressed in ppm and coupling constants are given in Hz. Data for ^{1}H NMR are recorded as follows: chemical shift (ppm), multiplicity (s, singlet; d, doublet; t, triplet; q, quarter; m, multiplet), coupling constant (Hz), integration. Data for ^{13}C NMR are reported in terms of chemical shift (δ, ppm). IR spectra data are given in reciprocal centimeters (cm^{-1}) and only selected absorbance is reported.

(I) Experimental Procedures for the Synthesis of Substrate (1a):



A THF solution (15 mL) of 1-bromo-1-propene (1.0 ml, 11.3 m.mol) was cooled to –78 °C and to this solution was added n-BuLi (2.5 M in THF, 9.0 ml, 22.5 m.mol) slowly. The mixture was warmed to –20 °C, and stirred for 0.5 h. The solution was again cooled to –78 °C before addition of benzaldehyde (1.0 g, 9.4 m.mol), and stirring was continued for another 0.5 h and warmed to room temperature. The reaction was quenched with water, extracted with ethyl acetate (25 ml). The extract was washed with brine solution, dried over $MgSO_4$, and concentrated under reduced pressure. The residue was eluted through a silica column to afford compound **s-1** (1.2 g, 8.2 m.mol, 87%) as yellow liquid.

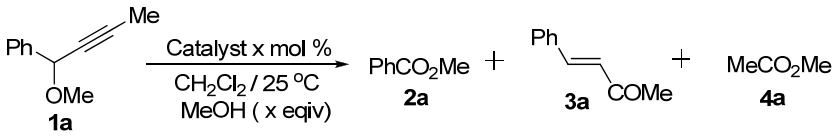
In a flask containing NaH (60% in oil, 360 mg, 9.0 m.mol) was washed with hexane to remove oil, and to the washed NaH solid was added dry THF (20 ml).The suspension was cool to 0 °C, and added compound **s-1** (1.20 g, 8.2 mmol). After stirring for 30 min, the mixture was added MeI (1.0 ml, 16.4 m.mol) with stirring for 1.5 h at room temperature. The reaction was quenched with water, extracted with ethyl acetate (20 ml). The extract was washed with brine solution, dried over $MgSO_4$, and concentrated under reduced pressure. The residue was eluted through a silica column to afford compound **1a** (1.1 g, 6.9 m.mol, 84%) as yellow liquid.

(II) Standard procedure for Gold-Catalyzed Oxidative Cleavage of Aryl-Substituted Alkynyl Ethers (1a) using Molecular Oxygen:

To a reaction vessel (ca. 25 mL), covered with aluminum foil, was added PPh_3AuCl (9.0 mg, 0.019 m.mol) and $AgNTf_2$ (7.0 mg, 0.019 m.mol), and the vessel was evacuated before it was charged with N_2 (140 mL) and O_2 (15 mL); the generated gas pressure was balanced with a balloon. To this mixture was added dry dichloromethane (1.0 ml), and the mixture was stirred for 10 min. To this solution was added dichloromethane (1.5 ml) of alkynyl ether **1a** (100 mg, 0.624 mmol), MeOH (0.08 ml, 1.87 mmol), and the resulting suspension was stirred for 15 h. The solution was concentrated, and eluted through a silica column (hexane/ethyl acetate = 50:1) to afford compound **2a** (69 mg, 0.51 m.mol, 81%) as a colourless liquid.

(III) Table S-1: Screning of Metal–Catalyst for Oxidative Cleavage of Aryl Substituted Alkynyl Ethers (1a) using Molecular Oxygen.

Table S-1



entry	catalyst (x mol %)	MeOH (x eqiv)	time (h)	yield[a] (%)
1.	PPh_3AuCl / $AgSbF_6$ (2 %)	–	24	**2a** (15%), **3a** (48%)
2.	PPh_3AuCl / $AgNTf_2$ (3 %)	–	24	**2a** (19%), **3a** (43%)
3.	PPh_3AuCl / $AgNTf_2$ (3 %)	2	15	**2a** (46%), **3a** (39%)
4.	**PPh_3AuCl / $AgNTf_2$ (3 %)**	**3**	**15**	**2a (81%), 3a (4%)**
5.	PPh_3AuCl / $AgSbF_6$(2 %)	3	12	**2a** (75%), **3a** (10%)
6.	AuCl / $AgSbF_6$ (4 %)	3	26	**2a** (68%), **3a** (9%)
7.	$PtCl_2$ / AgOTf (5 %)	3	24	**1a** (98%)[b]
8.	AuCl (5 %)	3	24	**1a** (99%)[b]
9.	$AgSbF_6$ (5%)	3	24	**1a** (99%)[b]
10.	AgOTf (5 %)	3	24	**1a** (99%)[b]
11.	AuCl / AgOTf (4 %)	3	26	**2a** (73%), **3a** (15%)
12.	AuCl / $AgBF_4$ (4 %)	3	26	**2a** (43%), **3a** (38%)

[a]Yields of 2a & 3a are reported after purification from silica column.
[b]Yields determined by ^1H NMR Spectroscopy..

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PROCNO                      1

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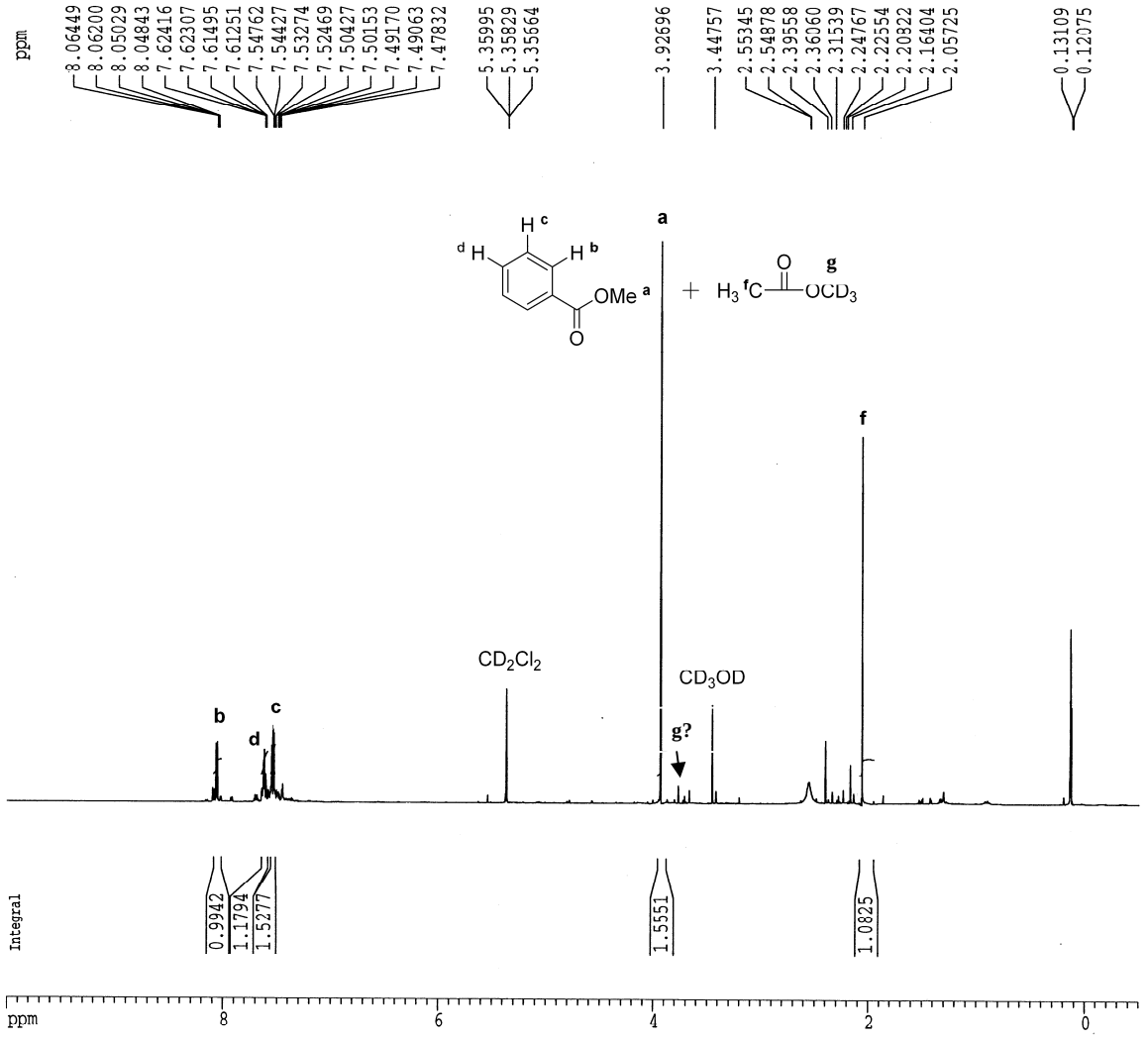

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PL12               11.30 dB
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SFO2          599.2029960 MHz

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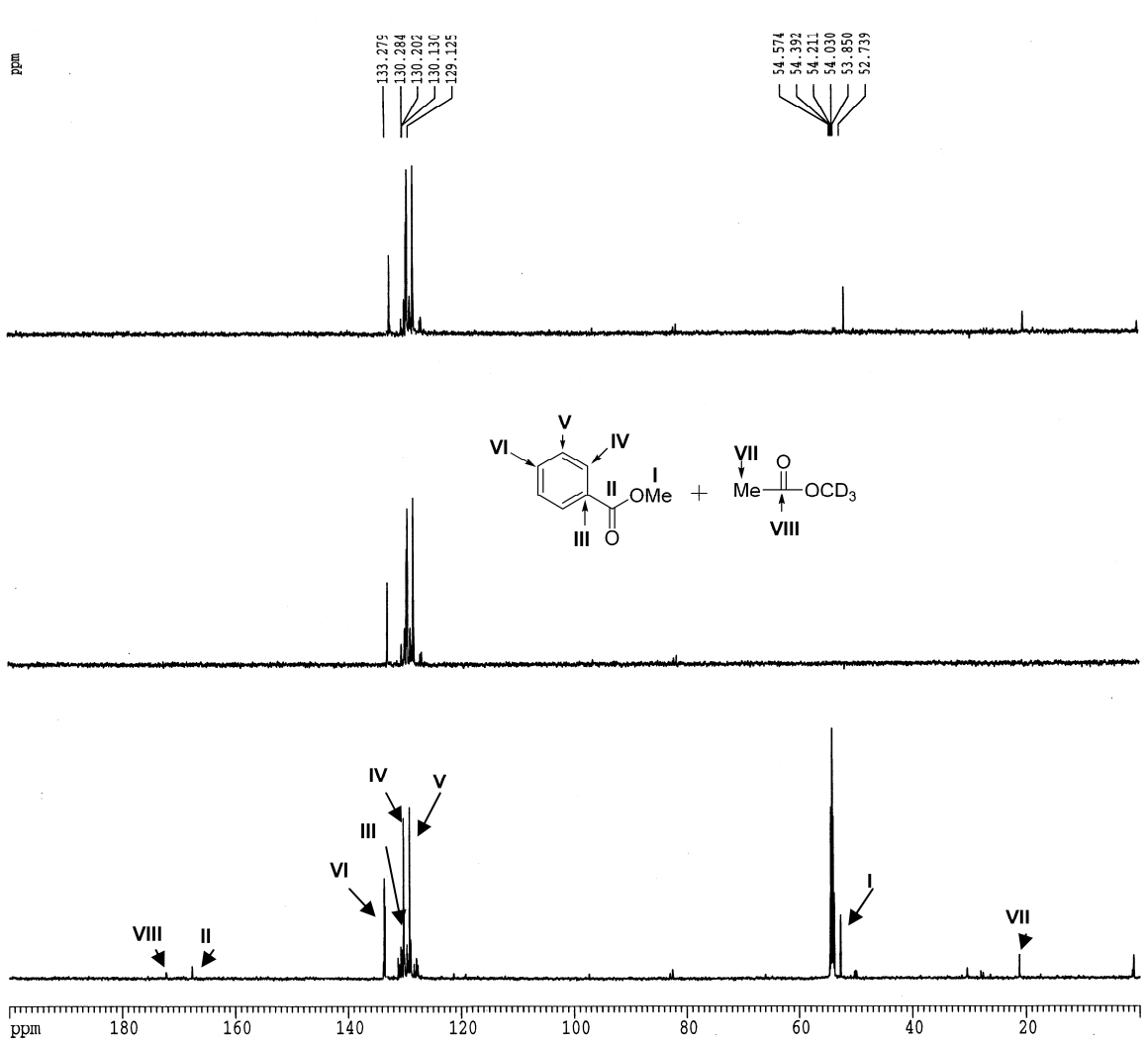

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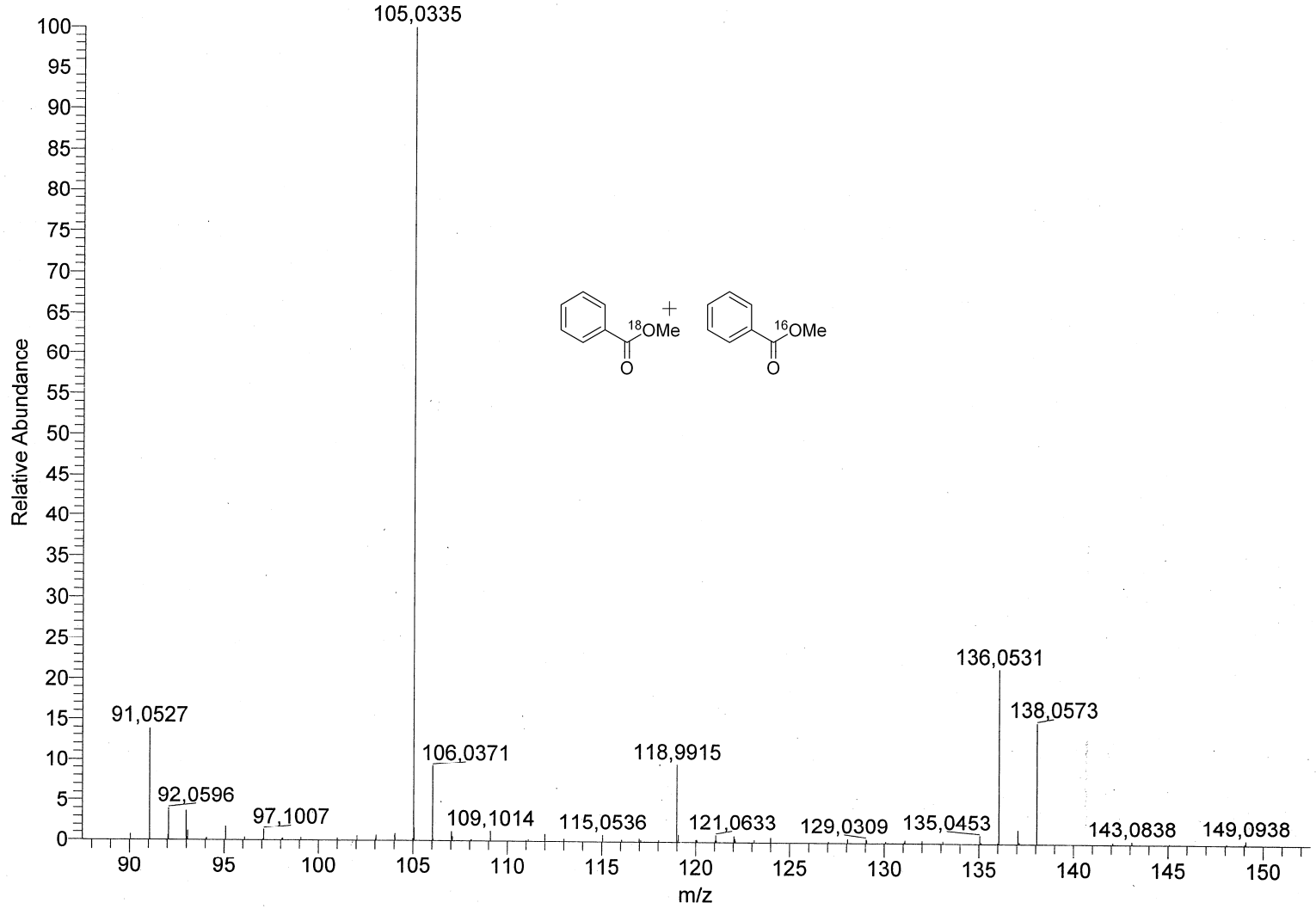
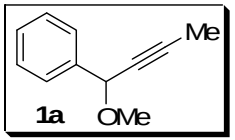


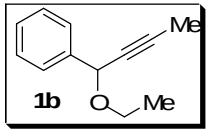
(IV) Spectral data for compound 1a-8b.

Spectra data for 1-(1-methoxybut-2-ynyl)benzene (1a):



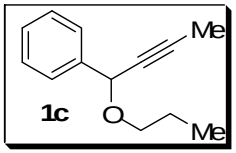
^{1}H NMR (400 MHz, $CDCl_3$) : δ 7.47 (d, *J*=7.2 Hz, 2 H), 7.37 ~ 7.30 (m, 3 H), 5.01 (d, *J* = 2.0 Hz, 1 H), 3.38 (s, 3 H), 1.91 (d, *J* = 2.0 Hz, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$) : δ 138.9, 128.3(2×CH), 128.1, 127.2(2×CH), 83.8, 76.7, 73.1, 55.6, 3.6; IR (nujol, cm^{-1}) : 3070 ~ 3020 (w), 2968 (s), 2135 (w) 1390 (s), 1130 (s); HRMS calcd for $C_{11}H_{12}O$: 160.0888, found: 160.0893.

Spectral data for 1-(1-ethoxybut-2-ynyl)benzene (1b):



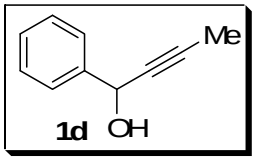
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.51 (d, *J* = 7.8 Hz, 2 H), 7.37 ~ 7.29 (m, 3 H), 5.11 (q, *J* = 2.0 Hz, 1 H), 3.73 ~ 3.65 (m, 1 H), 3.55 ~ 3.47 (m, 1 H), 1.89 (d, *J* = 2.0 Hz, 3 H), 1.24 (t, *J* = 7.0 Hz, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 139.5, 128.3 (2 x CH), 128.0, 127.2 (2 x CH), 83.3, 77.4, 71.5, 63.5, 15.0, 3.6; IR (nujol, cm^{-1}): 3066 ~ 3021 (w), 2969 (s), 2137 (w) 1388 (s), 1128 (s); HRMS calcd for $C_{12}H_{14}O$: 174.1045, found: 174.1048.

Spectral data for 1-(1-propoxybut-2-ynyl)benzene (1c):



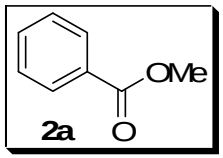
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.48 (d, *J* = 7.2 Hz, 2 H), 7.36 ~ 7.28 (m, 3 H), 5.08 (q, *J* = 2.1 Hz, 1 H), 3.58 ~ 3.52 (m, 1 H), 3.41 ~ 3.35 (m, 1 H), 1.89 (d, *J* = 2.1 Hz, 3 H), 1.65 ~ 1.59 (m, 2 H), 0.91 (t, *J* = 7.0 Hz, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 139.5, 128.2 (2 x CH), 127.9, 127.1 (2 x CH), 83.3, 77.4, 71.6, 69.8, 22.7, 10.5, 3.6; IR (nujol, cm^{-1}): 3069 ~ 3020 (w), 2966 (s), 2138 (w) 1392 (s), 1127 (s); HRMS calcd for $C_{13}H_{16}O$: 188.1201, found: 188.1204.

Spectra data for 1-phenylbut-2-yn-1-ol (1d):



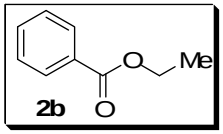
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.50 (d, *J* = 7.4 Hz, 2 H), 7.36 ~ 7.26 (m, 3 H), 5.38 (d, *J* = 1.1 Hz, 1 H), 2.91 (s, br, OH), 1.86 (d, *J* = 2.2 Hz, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 141.2, 128.3 (2 x CH), 127.9, 126.4 (2 x CH), 82.7, 79.2, 64.4, 3.5; IR (nujol, cm^{-1}): 3387 (w), 3070 ~ 3022 (w), 2968 (s), 2135 (w), 1130 (s), 1056 (s); HRMS calcd for $C_{10}H_{10}O$: 146.0732, found: 146.0730.

Spectra data for Methyl benzoate (2a):



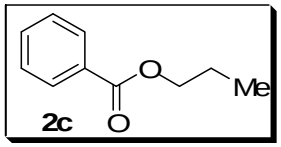
^{1}H NMR (400 MHz, $CDCl_3$) : δ 8.02 (d, *J*=7.2 Hz, 2 H), 7.54 (t, *J*=7.2 Hz, 1 H), 7.42 (t, *J*=7.2 Hz, 2 H), 3.9 (s, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$) : δ167.1, 132.9, 130.1, 129.5(2×CH), 128.3(2×CH), 52.1; IR (nujol, cm^{-1}) : 3070~3020 (w), 2958 (s), 1728 (s), 1584 (m), 1290 (s);HRMS calcd for $C_8H_8O_2$: 136.0524, found: 136.0529.

Spectra data for Ethyl benzoate (2b):



^{1}H NMR (400 MHz, $CDCl_3$): δ 8.03 (dd, *J* = 7.7, 1.2 Hz, 2 H), 7.55 ~ 7.51 (m, 1 H), 7.43 ~ 7.39 (m, 2 H), 4.36 (q, *J* = 7.1 Hz, 2 H), 1.38 (t, *J* = 7.1 Hz, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 166.6, 132.8, 130.5, 129.5(2×CH), 128.3(2×CH), 60.9, 14.3; IR (nujol, cm^{-1}): 3070~3025 (w), 2986 (s), 1726 (s), 1587 (m), 1286 (s), 1117 (s); HRMS calcd for $C_9H_{10}O_2$: 150.0681, found: 150.0685.

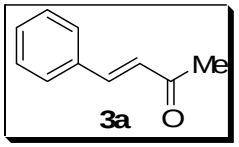
Spectra data for Propyl benzoate (2c):



^{1}H NMR (400 MHz, $CDCl_3$): δ 8.03 (dd, *J* = 8.2, 1.2 Hz, 2 H), 7.56 ~ 7.51 (m, 1 H), 7.44 ~ 7.39 (m, 2 H), 4.27 (t, *J* = 6.6 Hz, 2 H), 1.82 ~ 1.73 (m, 2 H), 1.02 (t, *J* = 7.4 Hz, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 166.7, 132.8, 130.5, 129.5(2×CH), 128.3(2×CH), 66.5, 22.1, 10.5; IR (nujol, cm^{-1}): 3070~3029 (w), 2955 (s), 1728 (s), 1588 (m), 1291 (s), 1120 (s); HRMS calcd for $C_{10}H_{12}O_2$: 164.0837,

found: 164.0841.

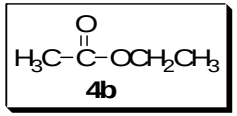
Spectra data for (*E*)-4-phenylbut-3-en-2-one (3a):



3a

^{1}H NMR (400 MHz, $CDCl_3$) : δ 7.54 ~ 7.48 (m, 3 H), 7.39 ~ 7.37 (m, 3 H), 6.70 (d, *J*=16 Hz, 1 H), 2.37 (s, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$) : δ198.5, 143.5, 134.3, 130.5, 128.9(2×CH), 128.2(2×CH), 127.1, 27.5; IR (nujol, cm^{-1}) : 2827 (w), 2834 (w), 1715 (s), 1620 (s); HRMS calcd for $C_{10}H_{10}O$: 146.0732, found: 146.0738.

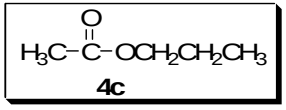
Spectra data for Ethyl acetate (4b):



4b

^{1}H NMR (400 MHz, $CDCl_3$): δ 4.07 (q, *J* = 7.1 Hz, 2 H), 2.0 (s, 3 H), 1.20 (t, *J* = 7.1 Hz, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 170.8, 60.1, 20.7, 13.9; IR (nujol, cm^{-1}): 2981 (w), 1752 (s), 1250 (s), 1055 (s); HRMS calcd for $C_4H_8O_2$: 88.0524, found: 88.0526.

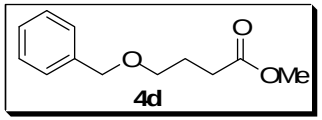
Spectra data for Propyl acetate (4c):



4c

^{1}H NMR (400 MHz, $CDCl_3$): δ 3.94 (t, *J* = 6.8 Hz, 2 H), 1.97 (s, 3 H), 1.59 ~ 1.54 (m, 2 H), 0.86 (t, *J* = 7.4 Hz, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 170.7, 65.6, 21.6, 20.4, 9.9; IR (nujol, cm^{-1}): 3105 (w), 1742 (s), 1255 (s), 1111 (s); HRMS calcd for $C_5H_{10}O_2$: 102.0681, found: 102.0683.

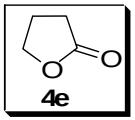
Spectra data for methyl 4-(benzyloxy)butanoate (4d):



4d

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.35 ~ 7.24 (m, 5 H), 4.48 (s, 2 H), 3.64 (s, 3 H), 3.49 (t, *J* = 6.2 Hz, 2 H), 2.42 (t, *J* = 7.4 Hz, 2 H), 1.96 ~ 1.91 (m, 2 H),; ^{13}C NMR (100 MHz, $CDCl_3$): δ 173.7, 138.3, 128.2 (2 x CH), 127.4, 127.3 (2 x CH), 72.7, 68.9, 51.3, 30.7, 24.9; IR (nujol, cm^{-1}): 3101 (w), 2988 (w), 1754 (s), 1259 (s), 1118 (s); HRMS calcd for $C_{12}H_{16}O_3$: 208.1099, found: 208.1097.

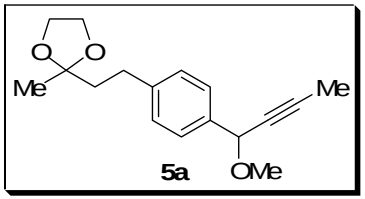
Spectra data for dihydrofuran-2(3*H*)-one (4e):



4e

^{1}H NMR (400 MHz, $CDCl_3$): δ 4.30 (t, *J* = 7.1 Hz, 2 H), 2.45 (t, *J* = 7.9 Hz, 2 H), 2.26 ~ 2.18 (m, 2 H),; ^{13}C NMR (100 MHz, $CDCl_3$): δ 177.8, 68.5, 27.7, 22.1; IR (nujol, cm^{-1}): 2987 (w), 1771 (s), 1249 (s), 1115 (s); HRMS calcd for $C_4H_6O_2$: 86.0368, found: 86.0371.

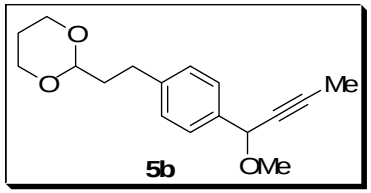
Spectra data for 2-(4-(1-methoxybut-2-ynyl)phenethyl)-2-methyl-1,3-dioxolane (5a):



5a

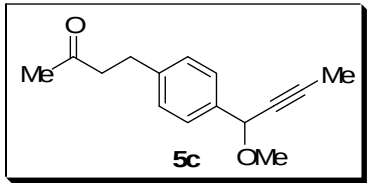
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.37 (d, *J* = 8.1 Hz, 2 H), 7.17 (d, *J* = 8.1 Hz, 2 H), 4.97 (q, *J* = 2.1 Hz, 1 H), 3.97 ~ 3.93 (m, 4 H), 3.36 (s, 3 H), 2.71 ~ 2.67 (m, 2 H), 1.95 ~ 1.91 (m, 2 H), 1.89 (d, *J* = 2.1 Hz, 3 H), 1.35 (s, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 142.3, 136.5, 128.3 (2 x CH), 127.4 (2 x CH), 109.6, 83.7, 77.0, 73.0, 64.7 (2 x CH), 55.6, 40.9, 29.9, 23.9, 3.7; IR (nujol, cm^{-1}): 3058 ~ 3022 (w), 2970 (s), 2135 (w) 1391 (s), 1165 (s), 1131 (s), 1035 (s); HRMS calcd for $C_{17}H_{22}O_3$: 274.1569, found: 274.1572.

Spectra data for 2-(4-(1-methoxybut-2-ynyl)phenethyl)-1,3-dioxane (5b):



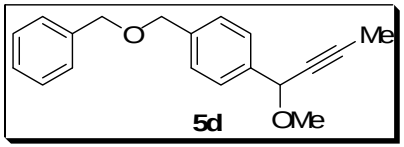
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.37 (d, *J* = 8.1 Hz, 2 H), 7.17 (d, *J* = 8.1 Hz, 2 H), 4.97 (q, *J* = 2.1 Hz, 1 H), 4.48 (t, *J* = 5.2 Hz, 1 H), 4.11 ~ 4.07 (m, 4 H), 3.76 ~ 3.69 (m, 2 H), 3.37 (s, 3 H), 2.71 ~ 2.67 (m, 2 H), 1.90 ~ 1.85 (m, 5 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 141.8, 136.7, 128.5 (2 x CH), 127.4 (2 x CH), 101.3, 83.7, 77.1, 73.0, 66.8 (2 x CH), 55.6, 36.6, 29.8, 25.8, 3.6; IR (nujol, cm^{-1}): 3065 ~ 3024 (w), 2972 (s), 2139 (w) 1389 (s), 1133 (s), 1114 (s); HRMS calcd for $C_{17}H_{22}O_3$: 274.1569, found: 274.1568.

Spectra data for 4-(4-(1-methoxybut-2-ynyl)phenyl)butan-2-one (5c):



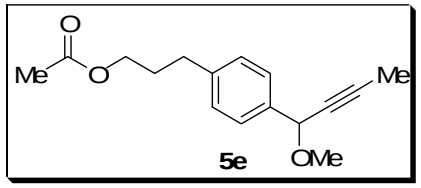
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.37 (d, *J* = 8.0 Hz, 2 H), 7.15 (d, *J* = 8.0 Hz, 2 H), 4.96 (q, *J* = 2.2 Hz, 1 H), 3.36 (s, 3 H), 2.86 (t, *J* = 7.8 Hz, 2 H), 2.72 (t, *J* = 7.8 Hz, 2 H), 2.11 (s, 3 H), 1.88 (d, *J* = 2.2 Hz, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 207.6, 140.9, 136.7, 128.1 (2 x CH), 127.3 (2 x CH), 83.6, 76.8, 72.8, 55.4, 44.8, 29.8, 29.2, 3.4; IR (nujol, cm^{-1}): 3069 ~ 3023 (w), 2972 (s), 2138 (w), 1715 (s), 1395 (s), 1128 (s); HRMS calcd for $C_{15}H_{18}O_2$: 230.1307, found: 230.1309.

Spectra data for 1-((benzyloxy)methyl)-4-(1-methoxybut-2-ynyl)benzene (5d):



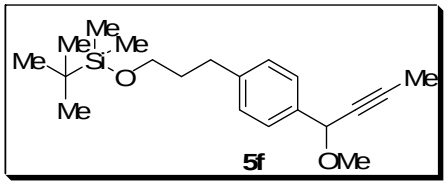
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.47 (d, *J* = 8.0 Hz, 2 H), 7.37 ~ 7.33 (m, 7 H), 5.01 (q, *J* = 2.0 Hz, 1 H), 4.55 (s, 2 H), 4.53 (s, 2 H), 3.38 (s, 3 H), 1.91 (d, *J* = 2.0 Hz, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 138.3, 138.2, 138.0, 128.3 (2 x CH), 127.7 (2 x CH), 127.6 (2 x CH), 127.5, 127.3 (2 x CH), 83.9, 76.8, 72.9, 71.9, 71.6, 55.1, 3.6; IR (nujol, cm^{-1}): 3070 ~ 3022 (w), 2971 (s), 2136 (w), 1398 (s), 1132 (s); HRMS calcd for $C_{19}H_{20}O_2$: 280.1463, found: 280.1468.

Spectra data for 3-(4-(1-methoxybut-2-ynyl)phenyl)propyl acetate (5e):



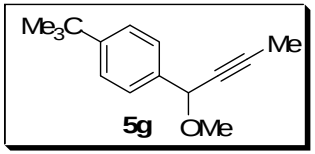
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.38 (d, *J* = 8.1 Hz, 2 H), 7.15 (d, *J* = 8.1 Hz, 2 H), 4.97 (q, *J* = 2.0 Hz, 1 H), 4.05 (t, *J* = 6.6 Hz, 2 H), 3.37 (s, 3 H), 2.66 (t, *J* = 7.4 Hz, 2 H), 2.03 (s, 3 H), 1.93 ~ 1.89 (m, 5 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 171.1, 141.3, 136.9, 128.4 (2 x CH), 127.5 (2 x CH), 83.8, 77.0, 73.0, 63.8, 55.7, 31.9, 30.1, 20.9, 3.7; IR (nujol, cm^{-1}): 3070 ~ 3021 (w), 2987 (s), 2971 (s), 2136 (w), 1755 (s), 1398 (s), 1260 (s), 1132 (s), 1120 (s); HRMS calcd for $C_{16}H_{20}O_3$: 260.1412, found: 260.1416.

Spectra data for (3-(4-(1-methoxybut-2-ynyl)phenyl)propoxy)(tert-butyl)dimethylsilane (5f):



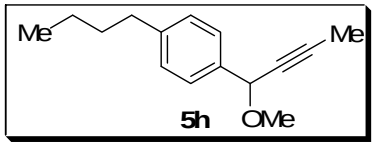
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.37 (d, *J* = 7.9 Hz, 2 H), 7.16 (d, *J* = 7.9 Hz, 2 H), 4.98 (q, *J* = 2.0 Hz, 1 H), 3.63 ~ 3.59 (m, 2 H), 3.36 (s, 3 H), 2.65 (t, *J* = 7.6 Hz, 2 H), 1.90 (d, *J* = 2.0 Hz, 3 H), 1.84 ~ 1.78 (m, 2 H), 0.89 (s, 9 H), 0.03 (s, 6 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 142.3, 136.4, 128.5 (2 x CH), 127.3 (2 x CH), 83.6, 77.0, 73.0, 62.2, 55.5, 34.3, 31.7, 25.9 (5 x CH_3), 18.3, 3.6; IR (nujol, cm^{-1}): 3070 ~ 3021 (w), 2971 (s), 2136 (w), 1398 (s), 1262 (s), 1132 (s), 1002 (s), 888 (s); HRMS calcd for $C_{20}H_{32}O_2Si$: 332.2172, found: 332.2174.

Spectra data for compound (5g):



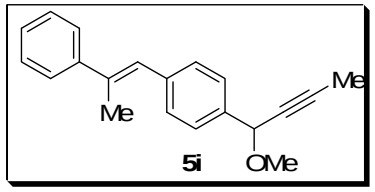
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.41 ~ 7.26 (m, 4 H), 4.98 (q, *J* = 2.1 Hz, 1 H), 3.38 (s, 3 H), 1.90 (d, *J* = 2.1 Hz, 3 H), 1.29 (s, 9 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 151.0, 136.0, 126.9 (2 x CH), 125.2 (2 x CH), 83.5, 77.0, 72.9, 55.5, 34.4, 31.2 (3 x CH_3), 3.5; IR (nujol, cm^{-1}): 3068 ~ 3021 (w), 2977 (s), 2139 (w), 1395 (s), 1136 (s); HRMS calcd for $C_{15}H_{20}O$: 216.1514, found: 216.1517.

Spectra data for 1-butyl-4-(1-methoxybut-2-ynyl)benzene (5h):



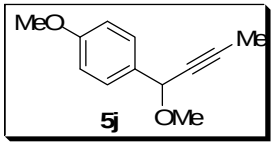
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.40 (d, *J* = 8.0 Hz, 2 H), 7.18 (d, *J* = 8.0 Hz, 2 H), 5.01 (q, *J* = 1.6 Hz, 1 H), 3.39 (s, 3 H), 2.61 (t, *J* = 7.6 Hz, 2 H), 1.91 (d, *J* = 1.6 Hz, 3 H), 1.62 ~ 1.58 (m, 2 H), 1.39 ~ 1.33 (m, 2 H), 0.93 (t, *J* = 7.2 Hz, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 142.9, 136.2, 128.3 (2 x CH), 127.2 (2 x CH), 83.5, 76.7, 73.0, 55.5, 35.2, 33.4, 22.2, 13.8, 3.5; IR (nujol, cm^{-1}): 3069 ~ 3020 (w), 2982 (s), 2148 (w), 1393 (s), 1129 (s); HRMS calcd for $C_{15}H_{20}O$:216.1514, found: 216.1519.

Spectra data for 1-(1-methoxybut-2-ynyl)-4-((E)-2-phenylprop-1-enyl)benzene (5i):



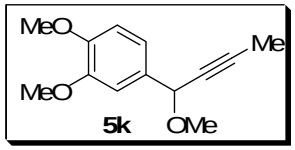
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.52 ~ 7.48 (m, 4 H), 7.38 ~ 7.34 (m, 5 H), 6.82 (s, 1 H), 5.04 (q, *J* = 2.1 Hz, 1 H), 3.41 (s, 3 H), 2.27 (s, 3 H), 1.93 (d, *J* = 2.1 Hz, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 143.8, 138.3, 137.7, 137.1, 129.2 (2 x CH), 128.3 (2 x CH), 127.3, 127.2 (3 x CH), 125.9 (2 x CH), 83.9, 76.9, 73.0, 55.7, 17.4, 3.7; IR (nujol, cm^{-1}): 3070 ~ 3018 (w), 2981 (s), 2139 (w), 1655 (s), 1396 (s), 1129 (s), 999 (s); HRMS calcd for $C_{20}H_{20}O$: 276.1514, found: 276.1518.

Spectra data for 1-methoxy-4-(1-methoxybut-2-ynyl) benzene (5j):



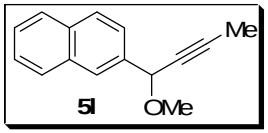
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.39 (d, *J* = 8.8 Hz, 2 H), 6.87 (d, *J* = 8.8 Hz, 2 H), 4.97 (q, *J* = 2.4 Hz, 1 H), 3.79 (s, 3 H), 3.35 (s, 3 H), 1.90 (d, *J* = 2.4 Hz, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 159.5, 131.2, 128.6 (2 x CH), 113.6 (2 x CH), 83.5, 77.0, 72.6, 55.3, 55.1, 3.5; IR (nujol, cm^{-1}): 3070 ~ 3019 (w), 2987 (s), 2141 (w), 1389 (s), 1142 (s); HRMS calcd for $C_{12}H_{14}O_2$: 190.0994, found: 190.0997.

Spectra data for 1,2-dimethoxy-4-(1-methoxybut-2-ynyl)benzene (5k):



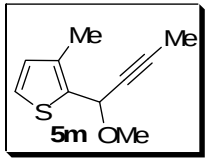
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.01 (s, 1 H), 6.99 (d, *J* = 8.6 Hz, 1 H), 6.82 (d, *J* = 8.6 Hz, 1 H), 4.95 (q, *J* = 2.0 Hz, 1 H), 3.88 (s, 3 H), 3.85 (s, 3 H), 3.36 (s, 3 H), 1.90 (d, *J* = 2.0 Hz, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 148.4 (2 x 4^{o}C), 131.2, 119.2, 110.2, 109.8, 83.0, 76.6, 72.3, 55.1 (2 x OMe), 54.8, 2.9; IR (nujol, cm^{-1}): 3067 ~ 3020 (w), 2979 (s), 2138 (w), 1386 (s), 1144 (s); HRMS calcd for $C_{13}H_{16}O_3$: 220.1099, found: 220.1095.

Spectra data for 2-(1-methoxybut-2-ynyl)naphthalene (5l):



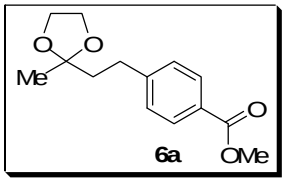
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.92 (d, *J* = 0.9 Hz, 1 H), 7.85 ~ 7.81 (m, 3 H), 7.6 (dd, *J* = 8.6 , 1.8 Hz, 1 H), 7.48 ~ 7.46 (m, 2 H), 5.18 (q, *J* = 2.2 Hz, 1 H), 3.42 (s, 3 H), 1.94 (d, *J* = 2.2 Hz, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 136.2, 132.9, 132.8, 128.0, 127.8, 127.3, 125.9, 125.8 (2 x CH), 124.8, 83.9, 76.8, 73.0, 55.3, 3.3; IR (nujol, cm^{-1}): 3069 ~ 3021 (w), 2975 (s), 2136 (w), 1391 (s), 1136 (s); HRMS calcd for $C_{15}H_{14}O$: 210.1045, found: 210.1049.

Spectra data for 2-(1-methoxybut-2-ynyl)-3-methylthiophene (5m):



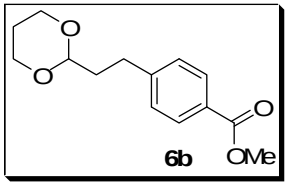
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.14 (d, *J* = 4.8 Hz, 1 H), 6.80 (d, *J* = 4.8 Hz, 1 H), 5.24 (q, *J* = 2.4 Hz, 1 H), 3.38 (s, 3 H), 2.23 (s, 3 H), 1.89 (d, *J* = 2.4 Hz, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 135.4, 134.9, 129.8, 123.7, 82.9, 76.4, 66.4, 54.9, 13.4, 3.3; IR (nujol, cm^{-1}): 3065 ~ 3020 (w), 2979 (s), 2137 (w), 1388 (s), 1139 (s), 591 (s); HRMS calcd for $C_{10}H_{12}OS$: 180.0609, found: 180.0612.

Spectra data for methyl 4-(2-(2-methyl-1,3-dioxolan-2-yl)ethyl)benzoate(6a):



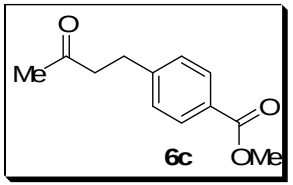
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.92 (d, *J* = 8.3 Hz, 2 H), 7.25 (d, *J* = 8.3 Hz, 2 H), 3.98 ~ 3.93 (m, 4 H), 3.87 (s, 3 H), 2.77 ~ 2.73 (m, 2 H), 1.97 ~ 1.93 (m, 2 H), 1.35 (s, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 167.1, 147.8, 129.7 (2 x CH), 128.3 (2 x CH), 127.7, 109.5, 64.8 (2 x CH_2), 52.0, 40.5, 30.3, 24.0; IR (nujol, cm^{-1}): 3058 ~ 3022 (w), 2974 (s), 1731 (s), 1391 (s), 1287 (s), 1165 (s), 1131 (s), 1118 (s), 1035 (s); HRMS calcd for $C_{14}H_{18}O_4$: 250.1205, found: 250.1208.

Spectra data for methyl 4-(2-(1,3-dioxan-2-yl)ethyl)benzoate(6b):



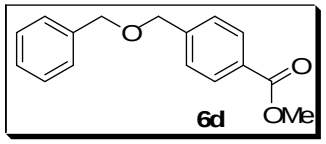
^{1}H NMR (600 MHz, $CDCl_3$): δ 7.91 (d, *J* = 7.9 Hz, 2 H), 7.23 (d, *J* = 7.9 Hz, 2 H), 4.45 (t, *J* = 5.1 Hz, 1 H), 4.08 ~ 4.06 (m, 2 H), 3.85 (s, 3 H), 3.72 ~ 3.68 (m, 2 H), 2.73 (t, *J* = 7.8 Hz, 2 H), 1.89 ~ 1.86 (m, 2 H), 1.35 ~ 1.30 (m, 2 H); ^{13}C NMR (150 MHz, $CDCl_3$): δ 167.0, 147.2, 129.6 (2 x CH), 128.4 (2 x CH), 127.8, 101.0, 66.8 (2 x CH_2), 51.9, 36.1, 30.0, 25.7; IR (nujol, cm^{-1}): 3059 ~ 3022 (w), 2977 (s), 1729 (s), 1394 (s), 1282 (s), 1130 (s), 1115 (s);HRMS calcd for $C_{14}H_{18}O_4$: 250.1205, found: 250.1209.

Spectra data for methyl 4-(3-oxobutyl)benzoate(6c):



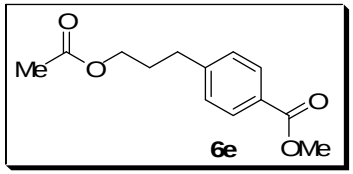
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.91 (d, *J* = 8.1 Hz, 2 H), 7.22 (d, *J* = 8.1 Hz, 2 H), 3.87 (s, 3 H), 2.91 (t, *J* = 7.5 Hz, 2 H), 2.75 (t, *J* = 7.5 Hz, 2 H), 2.11 (s, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 207.3, 166.9, 146.5, 129.8 (2 x CH), 128.3 (2 x CH), 128.1, 51.9, 44.5, 30.0, 29.6; IR (nujol, cm^{-1}): 3068 ~ 3023 (w), 2972 (s), 1730 (s), 1715 (s), 1395 (s), 1285(s), 1128 (s); HRMS calcd for $C_{12}H_{14}O_3$: 206.0943, found: 206.0947.

Spectra data for methyl 4-((benzyloxy)methyl)benzoate(6d):



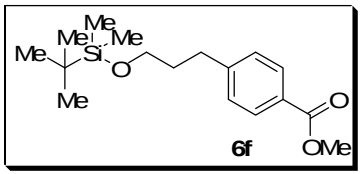
^{1}H NMR (400 MHz, $CDCl_3$): δ 8.02 (d, *J* = 8.3 Hz, 2 H), 7.42 (d, *J* = 8.3 Hz, 2 H), 7.37 ~ 7.28 (m, 5 H), 4.60 (s, 2 H), 4.57 (s, 2 H), 3.90 (s, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 166.9, 143.6, 137.9, 129.7 (2 x CH), 129.3, 128.4 (2 x CH), 127.7 (3 x CH_2), 127.2 (2 x CH), 72.4, 71.4, 52.0; IR (nujol, cm^{-1}): 3072 ~ 3022 (w), 2974 (s), 1728 (s), 1395 (s), 1288 (s), 1132 (s); HRMS calcd for $C_{16}H_{16}O_3$: 256.1099, found: 256.1103.

Spectra data for methyl 4-(3-acetoxypropyl)benzoate(6e):



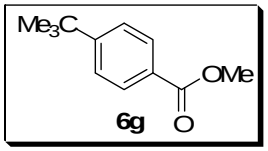
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.91 (d, *J* = 8.2 Hz, 2 H), 7.21 (d, *J* = 8.2 Hz, 2 H), 4.04 (t, *J* = 6.5 Hz, 2 H), 3.86 (s, 3 H), 2.70 (t, *J* = 7.4 Hz, 2 H), 2.00 (s, 3 H), 1.97 ~ 1.89 (m, 2 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 170.9, 166.9, 146.7, 129.7 (2 x CH), 128.3 (2 x CH), 127.9, 63.5, 51.9, 32.2, 29.7, 20.8; IR (nujol, cm^{-1}): 3070 ~ 3023 (w), 2986 (s), 2972 (s), 1752 (s), 1733 (s), 1398 (s), 1287 (s), 1260 (s), 1132 (s), 1120 (s); HRMS calcd for $C_{13}H_{16}O_4$: 236.1049, found: 236.1052.

Spectra data for compound (6f):



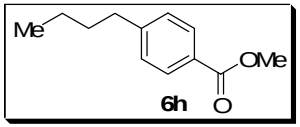
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.92 (d, *J* = 8.2 Hz, 2 H), 7.23 (d, *J* = 8.2 Hz, 2 H), 3.88 (s, 3 H), 3.60 (t, *J* = 6.2 Hz, 2 H), 2.71 (t, *J* = 7.5 Hz, 2 H), 1.86 ~ 1.79 (m, 2 H), 0.89 (s, 9 H), 0.03 (s, 6 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 167.2, 147.9, 129.6 (2 x CH), 128.5 (2 x CH), 127.7, 62.0, 51.9, 34.0, 32.1, 25.9 (5 x CH_3), 18.3; IR (nujol, cm^{-1}): 3068 ~ 3021 (w), 2973 (s), 1732 (s), 1398 (s), 1281 (s), 1262 (s), 1132 (s), 1004 (s), 890 (s); HRMS calcd for $C_{17}H_{28}O_3Si$: 308.1808, found: 308.1810.

Spectra data for methyl 4-*tert*-butylbenzoate (6g):



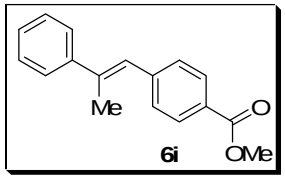
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.94 (d, *J* = 8.6 Hz, 2 H), 7.43 (d, *J* = 8.6 Hz, 2 H), 3.89 (s, 3 H), 1.32 (s, 9 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 166.8, 156.2, 129.2 (2 x CH), 127.2, 125.1 (2 x CH), 51.6, 34.8, 30.9 (3 x Me); IR (nujol, cm^{-1}): 3069 ~ 3021 (w), 2975 (s), 1725 (s), 1395 (s), 1279 (s), 1134 (s); HRMS calcd for $C_{12}H_{16}O_2$: 192.1150, found: 192.1153.

Spectra data for methyl 4-butylbenzoate (6h):



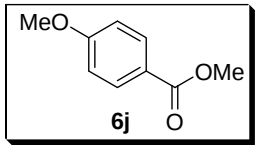
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.92 (d, *J* = 8.4 Hz, 2 H), 7.22 (d, *J* = 8.4 Hz, 2 H), 3.88 (s, 3 H), 2.64 (t, *J* = 7.6 Hz, 2 H), 1.63 ~ 1.56 (m, 2 H), 1.36 ~ 1.31 (m, 2 H), 0.91 (t, *J* = 7.2 Hz, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 167.1, 148.4, 129.6 (2 x CH), 128.4 (2 x CH), 127.6, 51.8, 35.6, 33.2, 22.3, 13.8; IR (nujol, cm^{-1}): 3069 ~ 3024 (w), 2986 (s), 1728 (s), 1393 (s), 1277 (s), 1129 (s); HRMS calcd for $C_{12}H_{16}O_2$: 192.1150, found: 192.1154.

Spectra data for methyl 4-((E)-2-phenylprop-1-enyl)benzoate (6i):



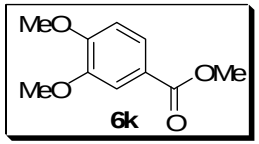
^{1}H NMR (400 MHz, $CDCl_3$): δ 8.03 (d, *J* = 8.2 Hz, 2 H), 7.52 (d, *J* = 7.7 Hz, 2 H), 7.43 ~ 7.30 (m, 5 H), 6.83 (s, 1 H), 3.92 (s, 3 H), 2.29 (s, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 166.9, 143.5, 143.0, 139.6, 129.5 (2 x CH), 129.0 (2 x CH), 128.4 (2 x CH), 127.9, 127.5, 126.8, 126.0 (2 x CH), 52.0, 17.7; IR (nujol, cm^{-1}): 3070 ~ 3022 (w), 2985 (s), 1732 (s), 1655 (s), 1398 (s), 1280 (s), 1131 (s), 996 (s); HRMS calcd for $C_{17}H_{16}O_2$: 252.1150, found: 252.1156.

Spectra data for methyl 4-methoxybenzoate (6j):



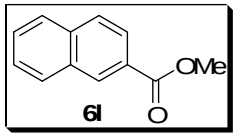
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.97 (d, *J* = 8.9 Hz, 2 H), 6.89 (d, *J* = 8.9 Hz, 2 H), 3.87 (s, 3 H), 3.84 (s, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 166.7, 163.2, 131.4 (2 x CH), 122.5, 113.5 (2 x CH), 55.2, 51.7; IR (nujol, cm^{-1}): 3073 ~ 3019 (w), 2988 (s), 1729 (s), 1391 (s), 1288 (s), 1143 (s); HRMS calcd for $C_9H_{10}O_3$: 166.0630, found: 166.0636.

Spectra data for methyl 3,4-dimethoxybenzoate (6k):



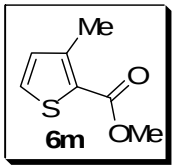
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.65 (d, *J* = 8.5 Hz, 1 H), 7.52 (s, 1 H), 6.68 (d, *J* = 8.5 Hz, 1 H), 3.91 (s, 6 H), 3.87 (s, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 166.9, 152.9, 148.6, 123.6, 122.7, 112.0, 110.3, 55.9 (2 x OMe), 51.9; IR (nujol, cm^{-1}): 3073 ~ 3017 (w), 2992 (s), 1732 (s), 1393 (s), 1286 (s), 1144 (s); HRMS calcd for $C_{10}H_{12}O_4$: 196.0736, found: 196.0741.

Spectra data for methyl 2-naphthoate (6l):



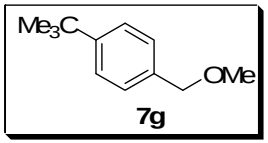
^{1}H NMR (400 MHz, $CDCl_3$): δ 8.60 (d, *J* = 0.6 Hz, 1 H), 8.05 (dd, *J* = 8.6, 1.7 Hz, 1 H), 7.93 (d, *J* = 8.0 Hz, 1 H), 7.86 (d, *J* = 8.6 Hz, 2 H), 7.59 ~ 7.52 (m, 2 H), 3.97 (s, 3 H); ^{13}C NMR (150 MHz, $CDCl_3$): δ 167.3, 135.5, 132.5, 131.0, 129.3, 128.2, 128.1, 127.7, 127.3, 126.6, 125.2, 52.2; IR (nujol, cm^{-1}): 3071 ~ 3021 (w), 2977 (s), 1735 (s), 1391 (s), 1282 (s), 1136 (s); HRMS calcd for $C_{12}H_{10}O_2$: 186.0681, found: 186.0685.

Spectra data for methyl 3-methylthiophene-2-carboxylate (6m):



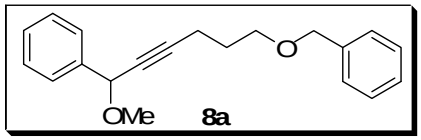
^{1}H NMR (600 MHz, $CDCl_3$): δ 7.36 (d, *J* = 5.0 Hz, 1 H), 6.89 (d, *J* = 5.0 Hz, 1 H), 3.84 (s, 3 H), 2.54 (s, 3 H); ^{13}C NMR (150 MHz, $CDCl_3$): δ 163.3, 146.3, 131.7, 130.0, 127.0, 51.7, 15.9; IR (nujol, cm^{-1}): 3069 ~ 3019 (w), 2976 (s), 1785 (s), 1389 (s), 1286 (s), 1139 (s), 591 (s); HRMS calcd for $C_7H_8O_2S$: 156.0245, found: 156.0249.

Spectra data for 1-*tert*-butyl-4-(methoxymethyl)benzene (7g):



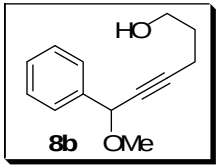
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.40 (d, *J* = 8.2 Hz, 2 H), 7.29 (d, *J* = 8.2 Hz, 2 H), 4.45 (s, 2 H), 3.40 (s, 3 H), 1.34 (s, 9 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 150.4, 135.1, 127.5 (2 x CH), 125.2 (2 x CH), 74.4, 57.9, 34.4, 31.3 (3 x Me); IR (nujol, cm^{-1}): 3068 ~ 3024 (w), 2975 (s), 1392 (s), 1138 (s); HRMS calcd for $C_{12}H_{18}O$: 178.1358, found: 178.1363.

Spectra data for 1-((6-methoxy-6-phenylhex-4-ynyloxy)methyl)benzene (8a):



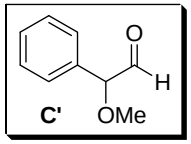
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.48 (dd, *J* = 7.1, 1.6 Hz, 2 H), 7.37 ~ 7.27 (m, 8 H), 5.05 (t, *J* = 1.8 Hz, 1 H), 4.49 (s, 2 H), 3.57 (t, *J* = 6.2 Hz, 2 H), 3.38 (s, 3 H), 2.44 ~ 2.40 (m, 2 H), 1.89 ~ 1.82 (m, 2 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 138.9, 138.3, 128.2 (3 x CH), 128.1, 127.5 (2 x CH), 127.4 (2 x CH), 127.3 (2 x CH), 87.7, 77.9, 73.1, 72.8, 68.6, 55.5, 28.7, 15.6; IR (nujol, cm^{-1}): 3068 ~ 3024 (w), 2143 (w), 1387 (s), 1135 (s); HRMS calcd for $C_{20}H_{22}O_2$: 294.1620, found: 294.1623.

Spectra data for 6-methoxy-6-phenylhex-4-yn-1-ol (8b):



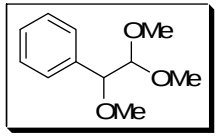
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.47 (d, *J* = 8.2 Hz, 2 H), 7.36 ~ 7.27 (m, 3 H), 5.03 (s, 1 H), 3.69 (t, *J* = 5.8 Hz, 2 H), 3.37 (s, 3 H), 2.39 ~ 2.36 (m, 2 H), 1.76 (t, *J* = 6.6 Hz, 2 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 138.8, 128.3 (2 x CH), 128.2, 127.3 (2 x CH), 87.7, 78.1, 73.1, 61.4, 55.6, 31.2, 15.3; IR (nujol, cm^{-1}): 3648 (s), 3073 ~ 3024 (w), 2144 (w), 1384 (s), 1135 (s), 1066 (s); HRMS calcd for $C_{13}H_{16}O_2$: 204.1150, found: 204.1152.

Spectra data for 2-methoxy-2-phenylacetaldehyde (C'):

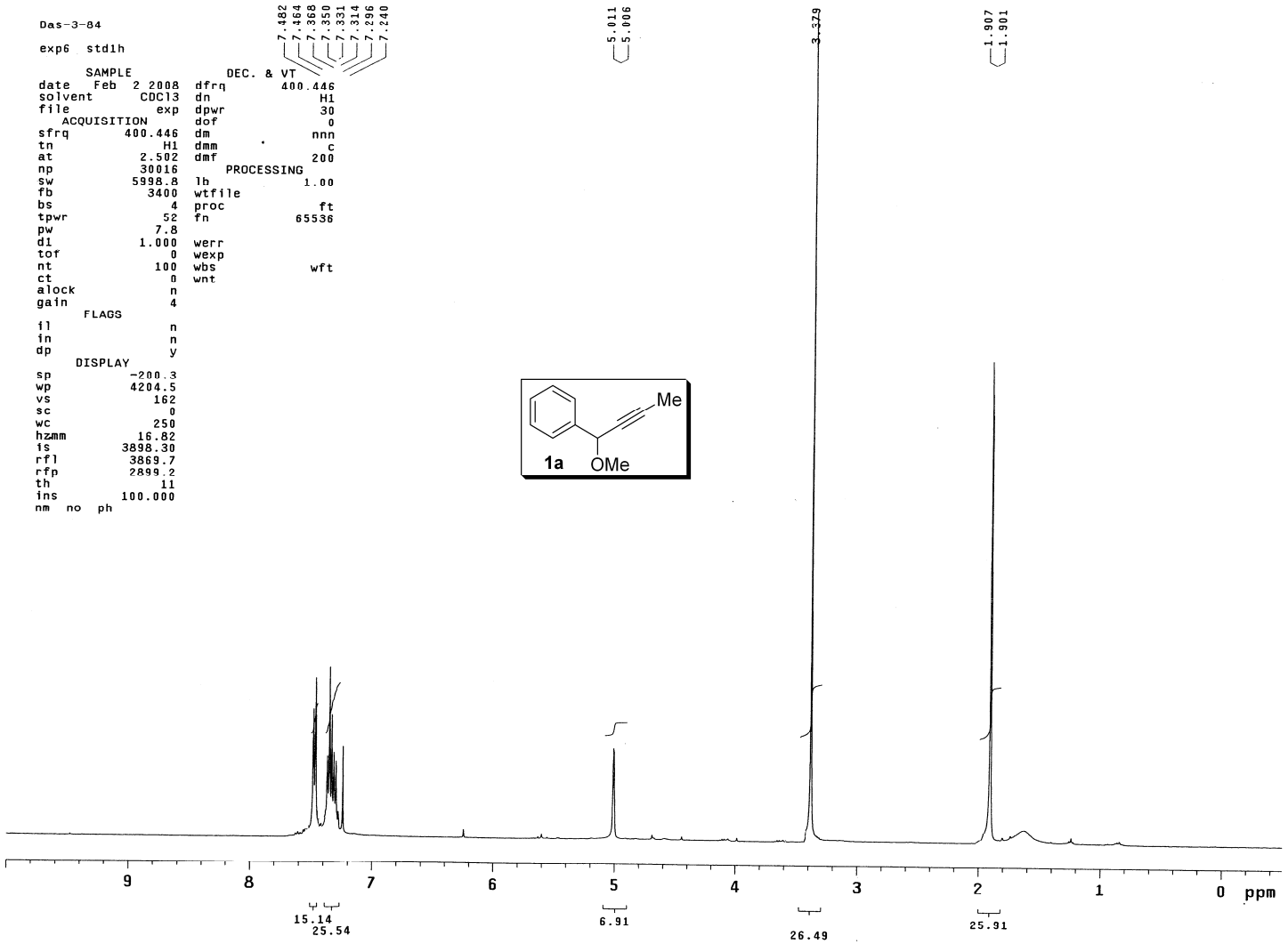


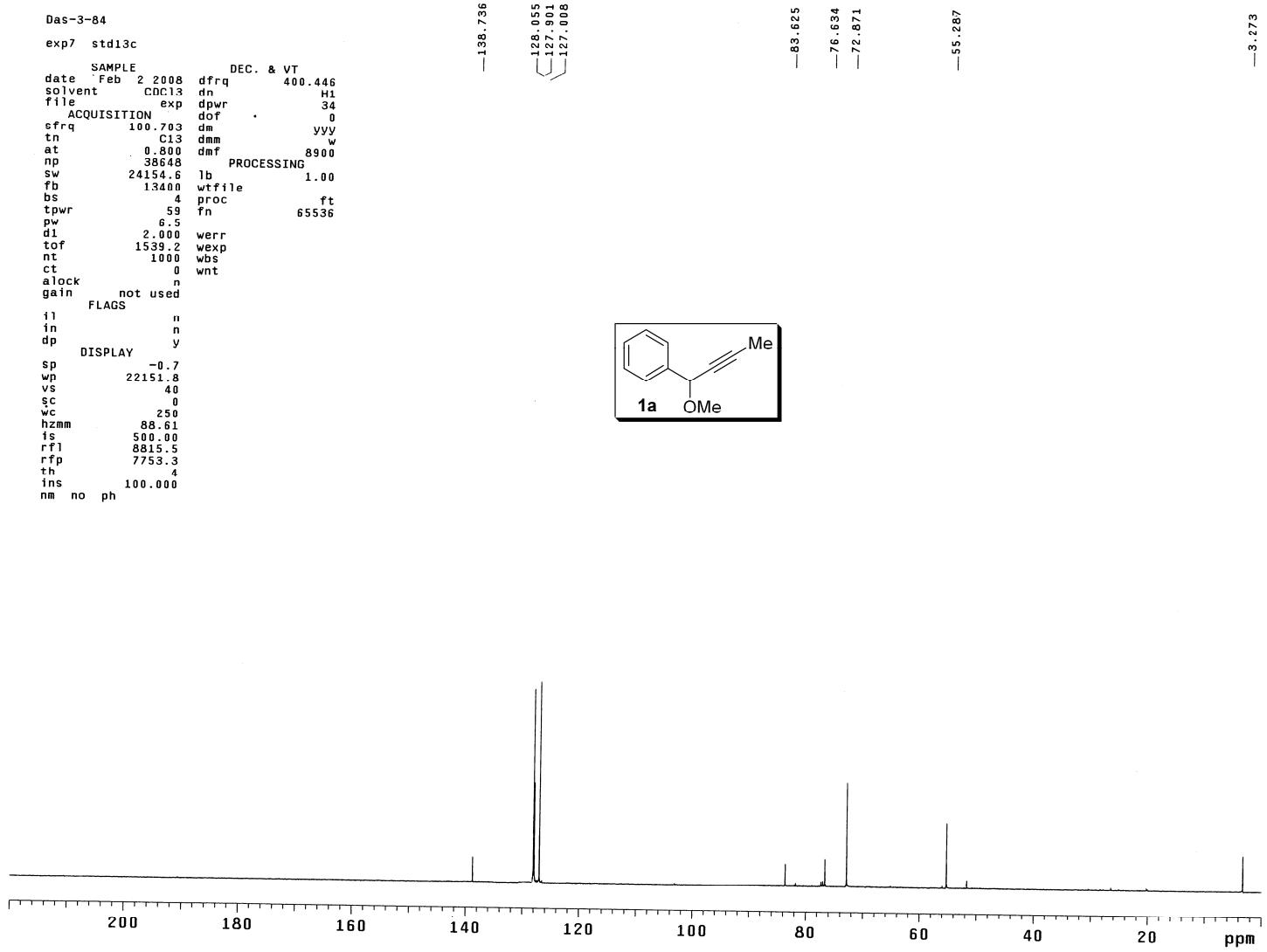
^{1}H NMR (400 MHz, CD_2Cl_2): δ 9.60 (d, *J* = 1.5 Hz, 1 H), 7.43 ~ 7.36 (m, 5 H), 4.35 (d, *J* = 1.5 Hz, 1 H), 3.43 (s, 3 H); ^{13}C NMR (100 MHz, CD_2Cl_2): δ 198.9, 134.5, 129.2 (2 x CH), 129.1, 127.8 (2 x CH), 88.4, 57.4; IR (nujol, cm^{-1}): 3070 ~ 3021 (w), 1726 (s), 1386 (s), 1133 (s); HRMS calcd for $C_9H_{10}O_2$: 150.0681, found: 150.0684.

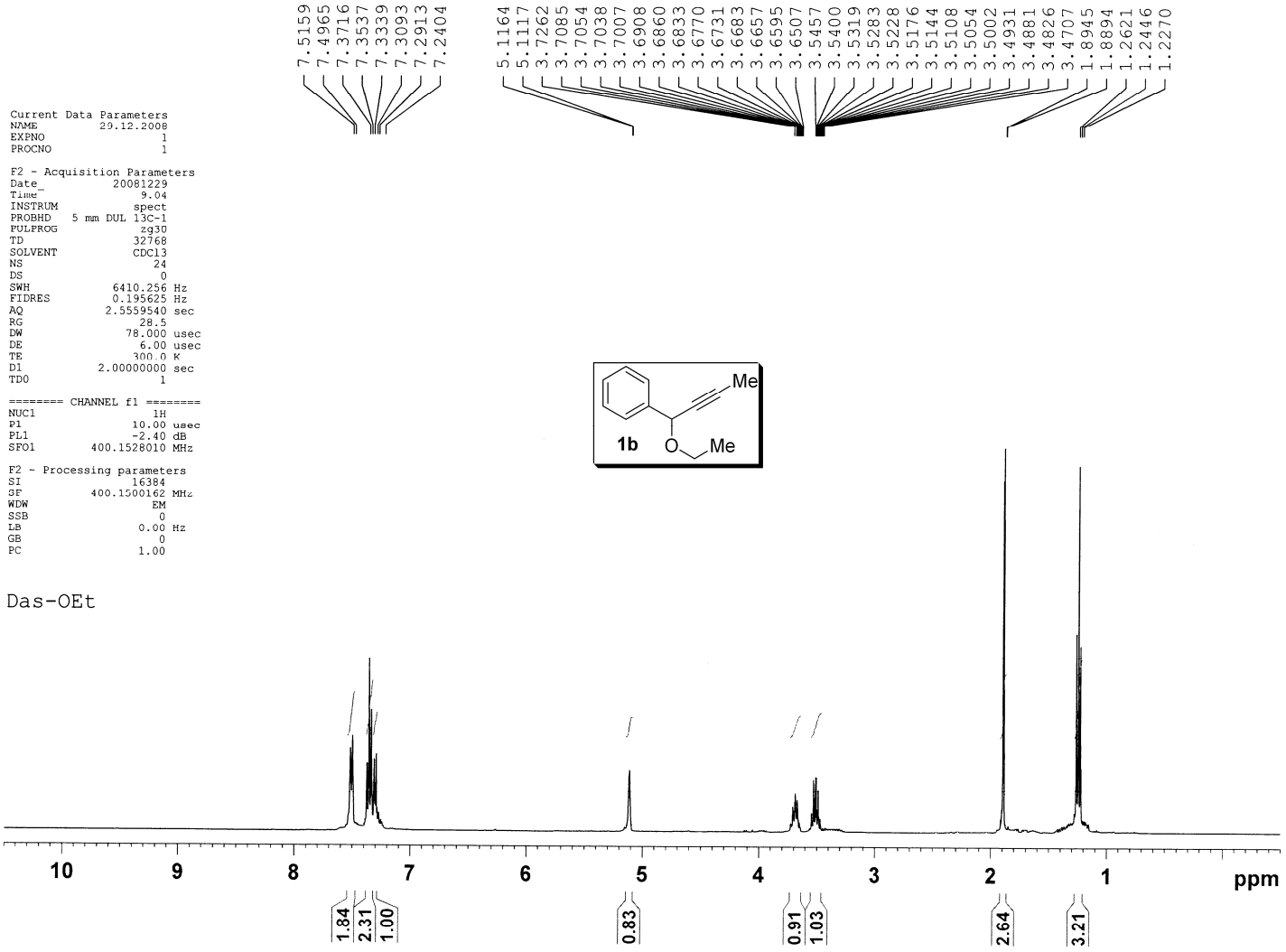
Spectra data for 1-(1,2,2-trimethoxyethyl)benzene :

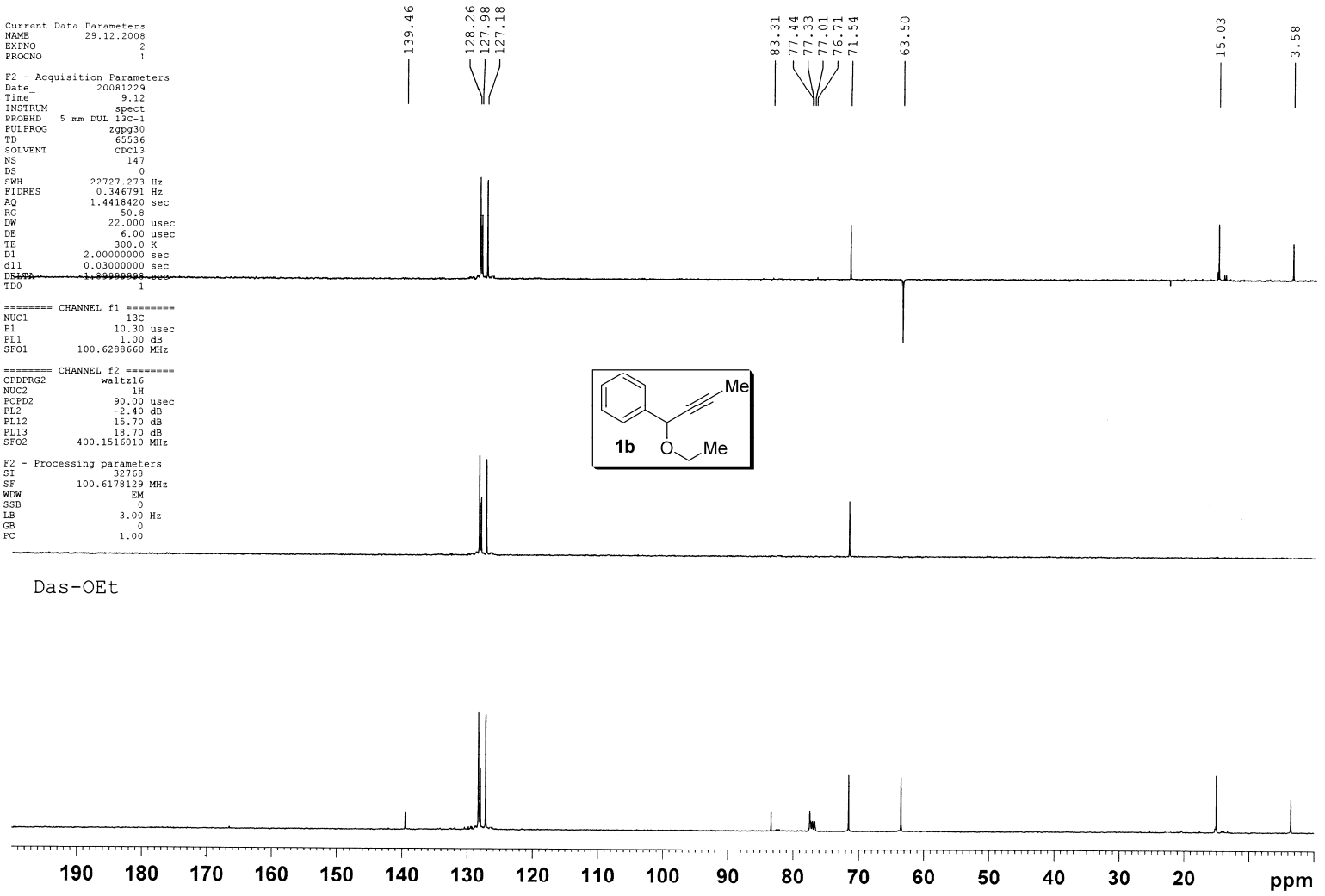


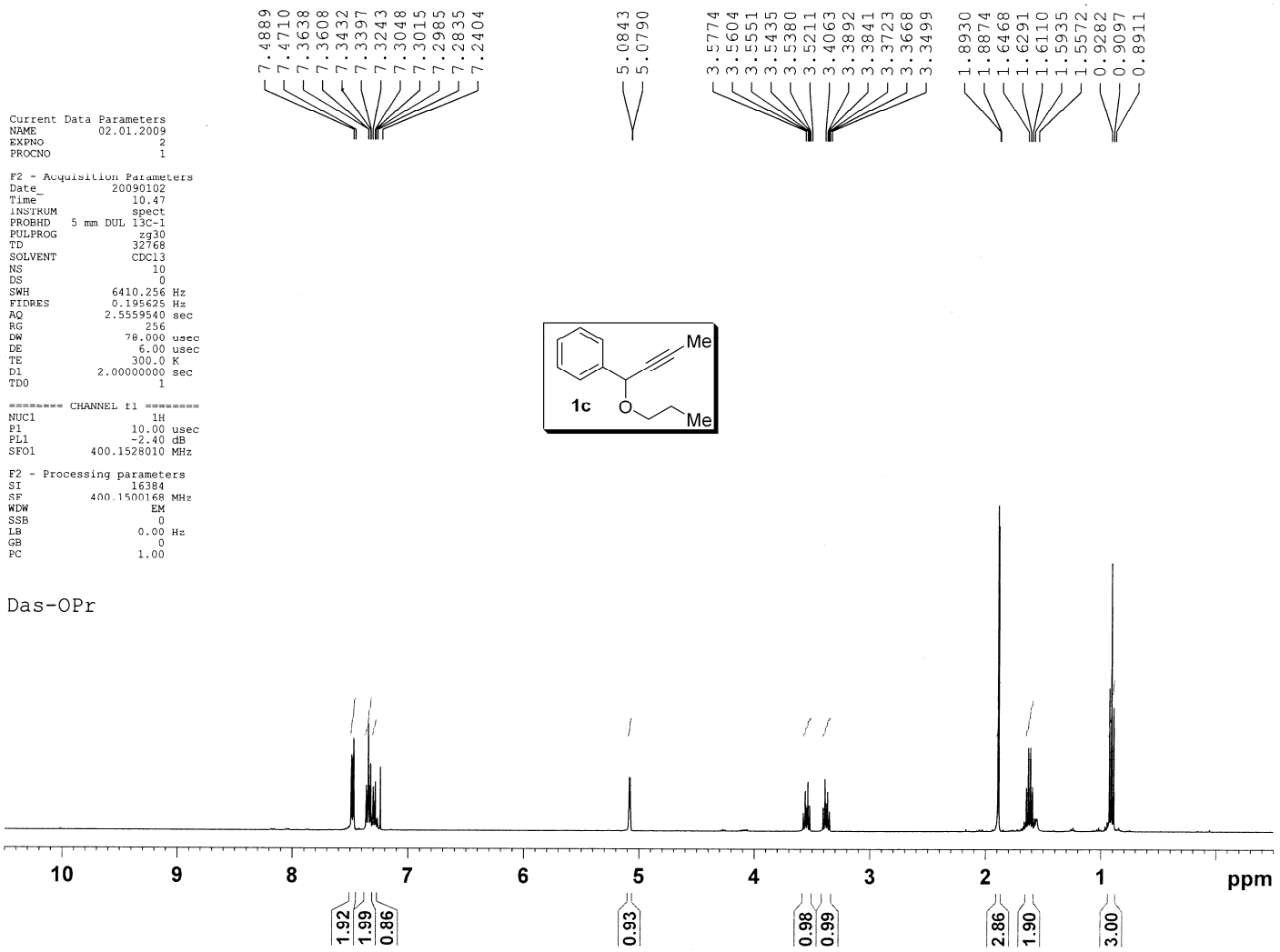
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.35 ~ 7.33 (m, 5 H), 4.36 (d, *J* = 6.2 Hz, 1 H), 4.16 (d, *J* = 6.2 Hz, 1 H), 3.44 (s, 3 H), 3.24 (s, 3 H), 3.19 (s, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 137.9, 128.2 (2 x CH), 128.0, 127.9 (2 x CH), 106.4, 83.8, 56.9, 55.7, 54.3; IR (nujol, cm^{-1}): 3071 ~ 3016 (w), 1383 (s), 1305 (s), 1136 (s); HRMS calcd for $C_{11}H_{16}O_3$: 196.1099, found: 196.1103.

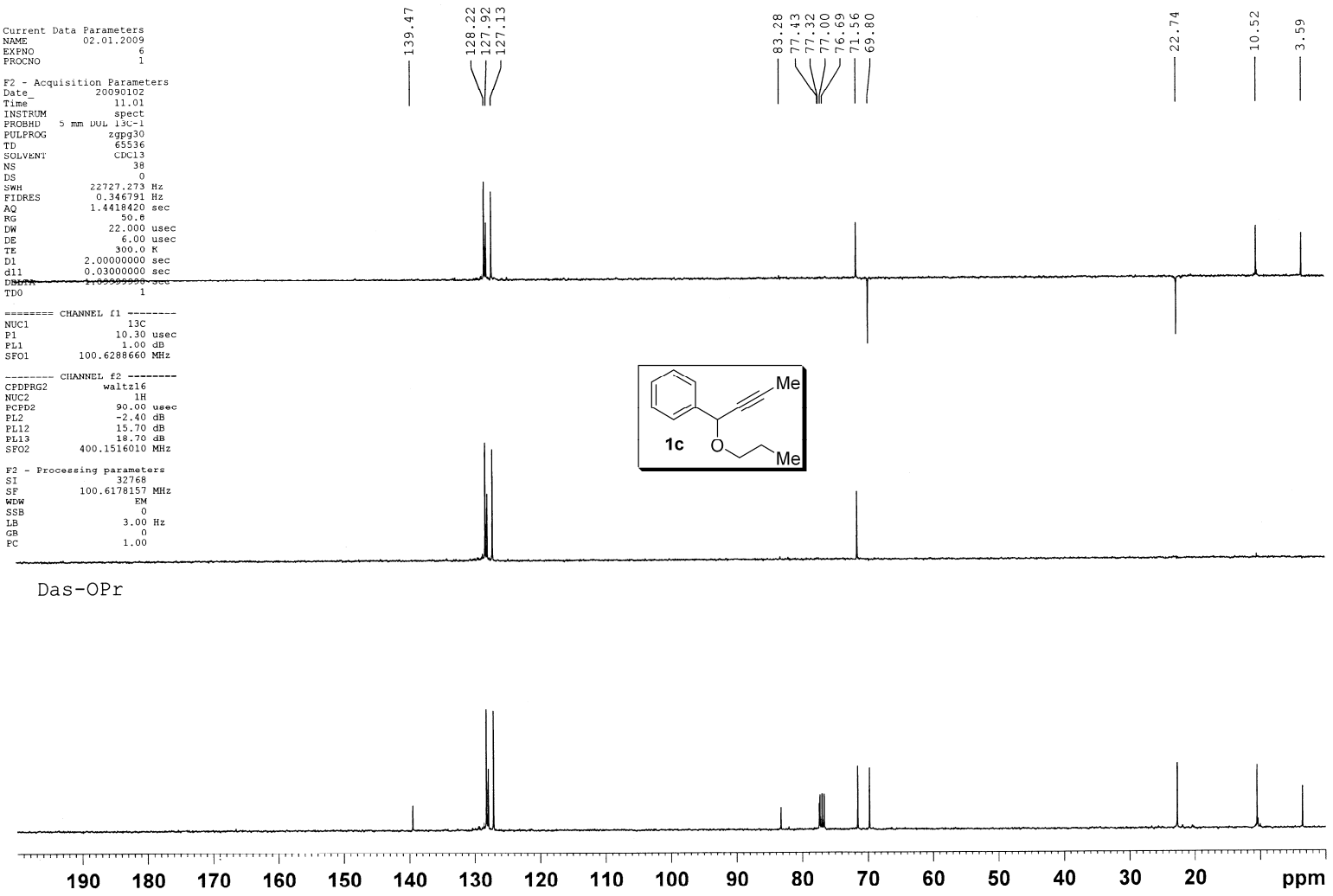


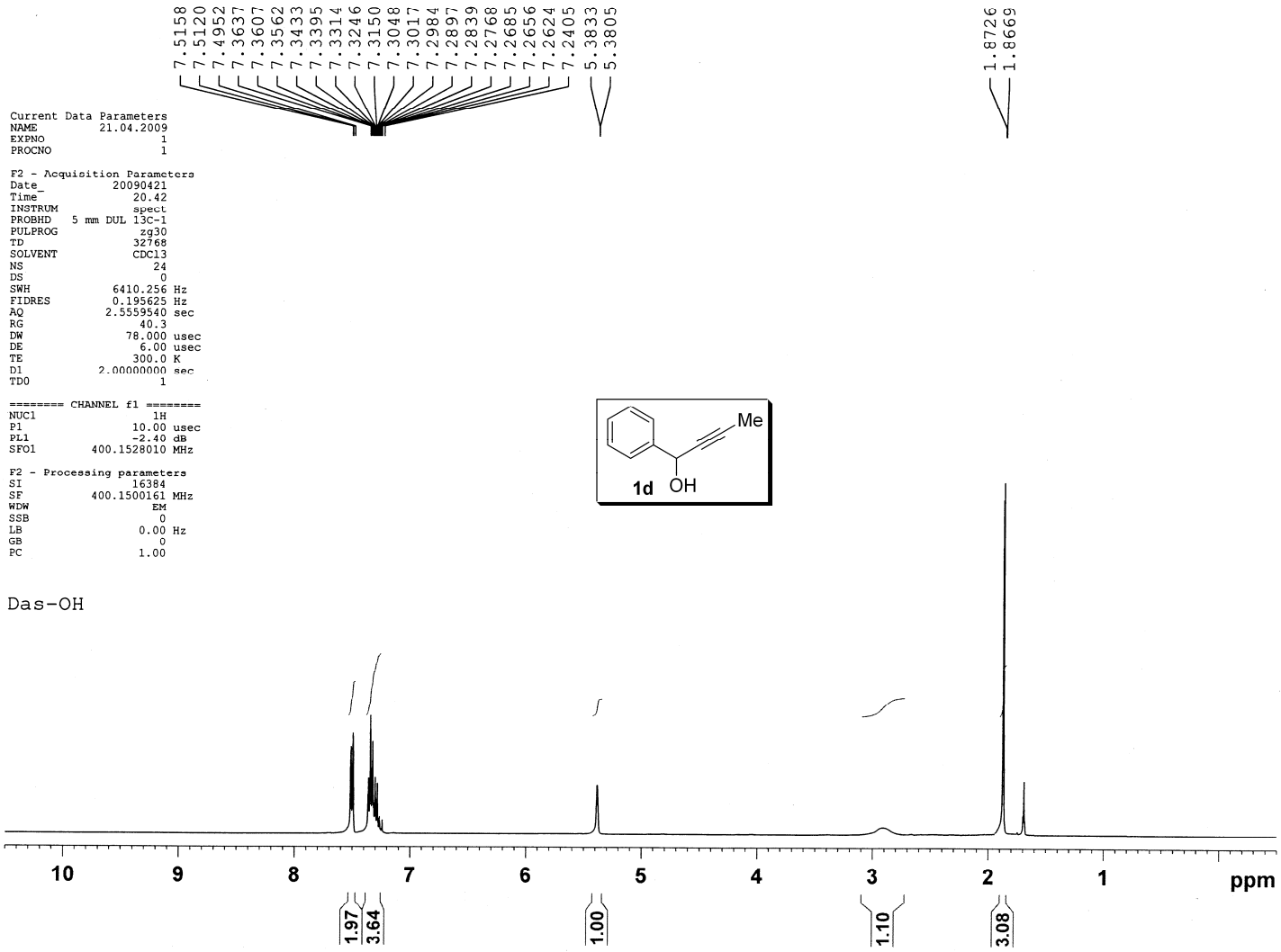


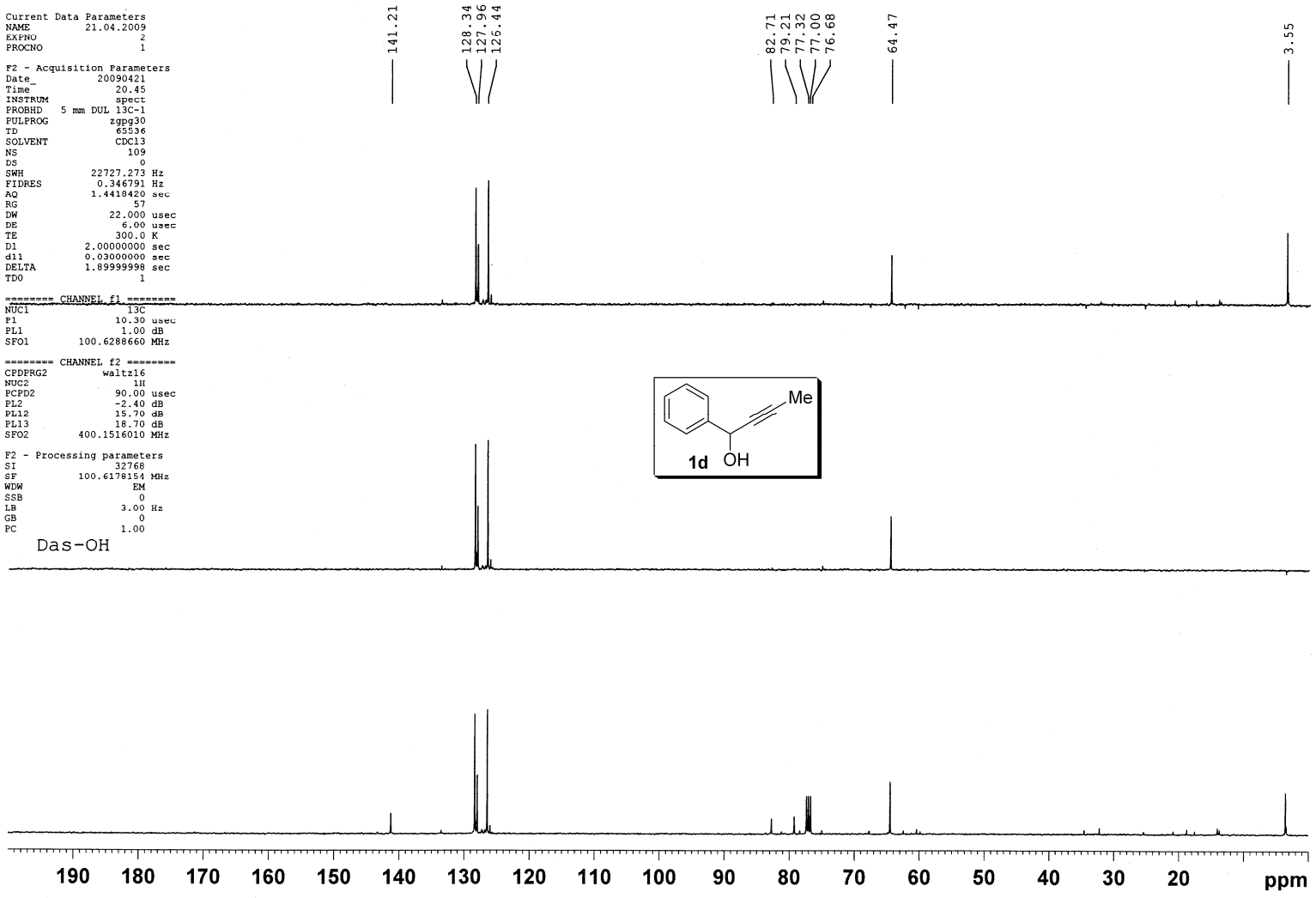


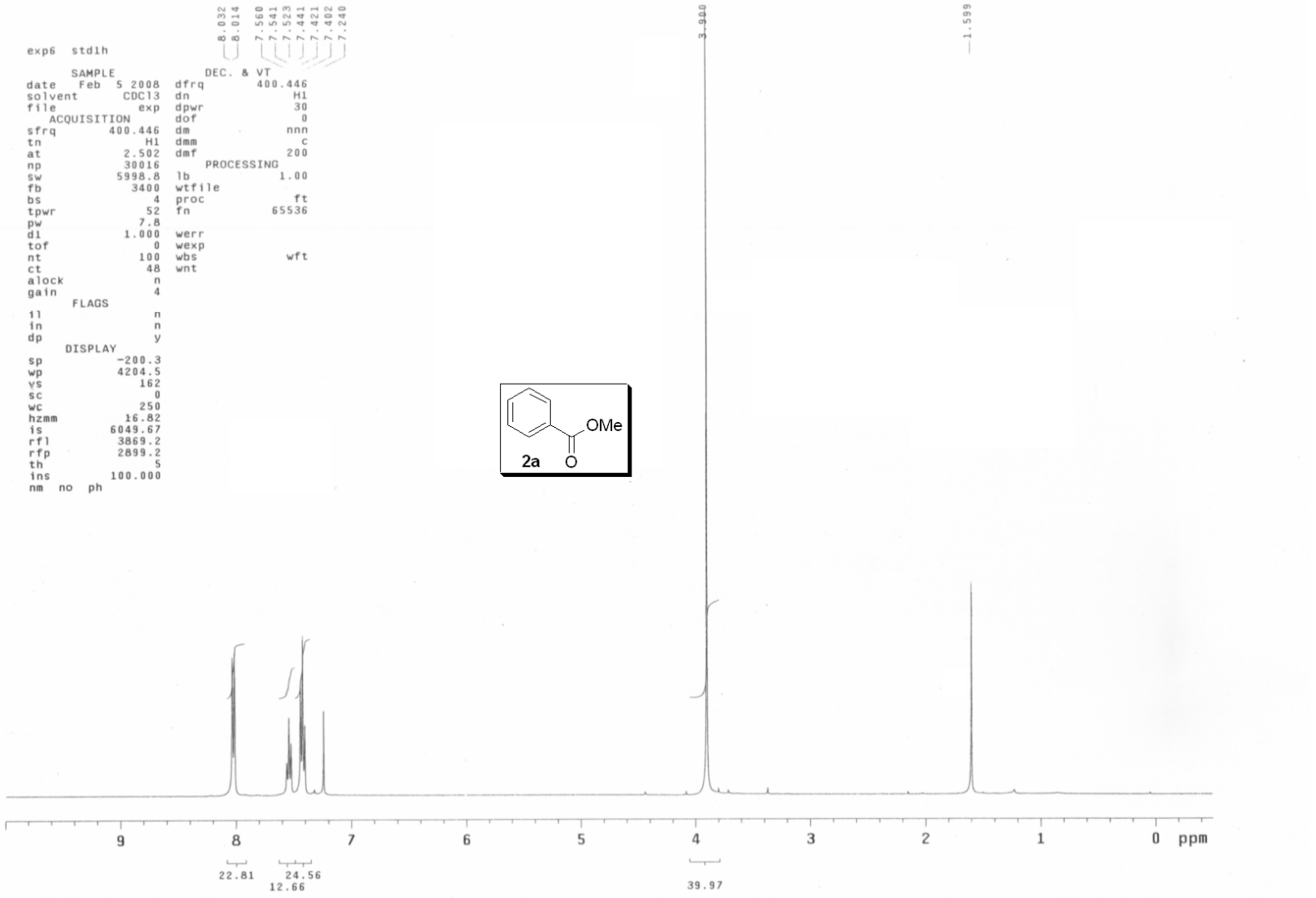


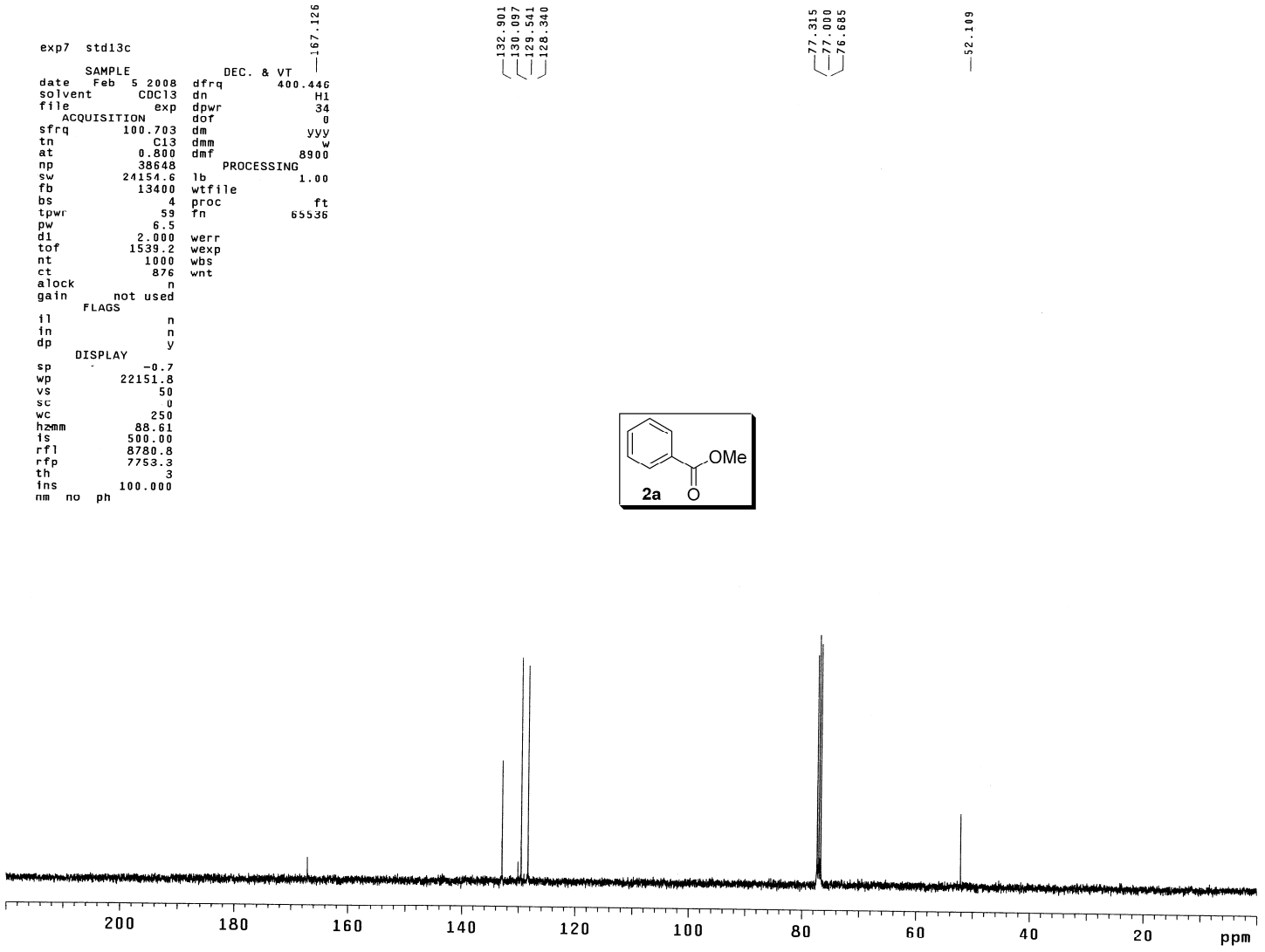








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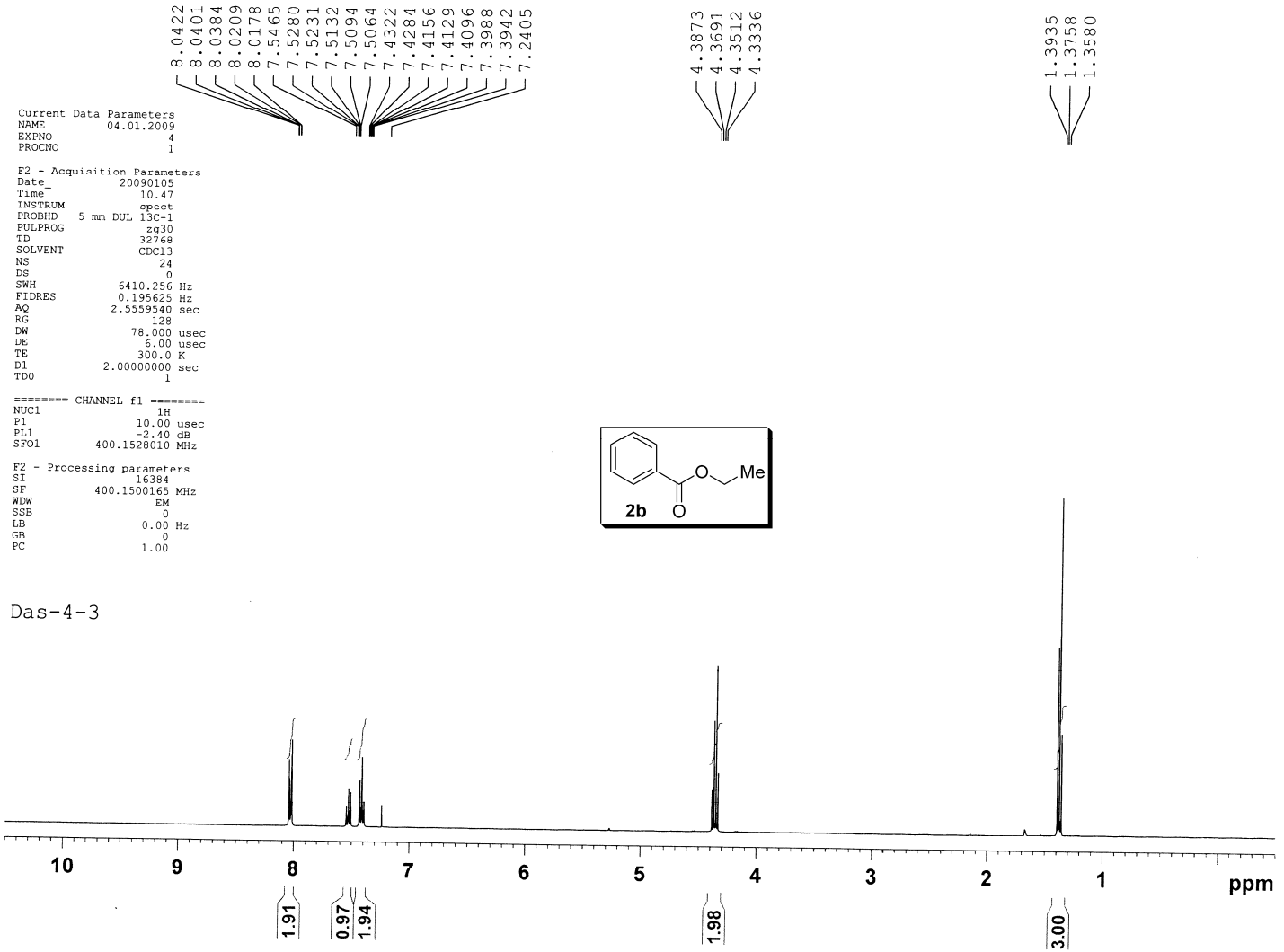

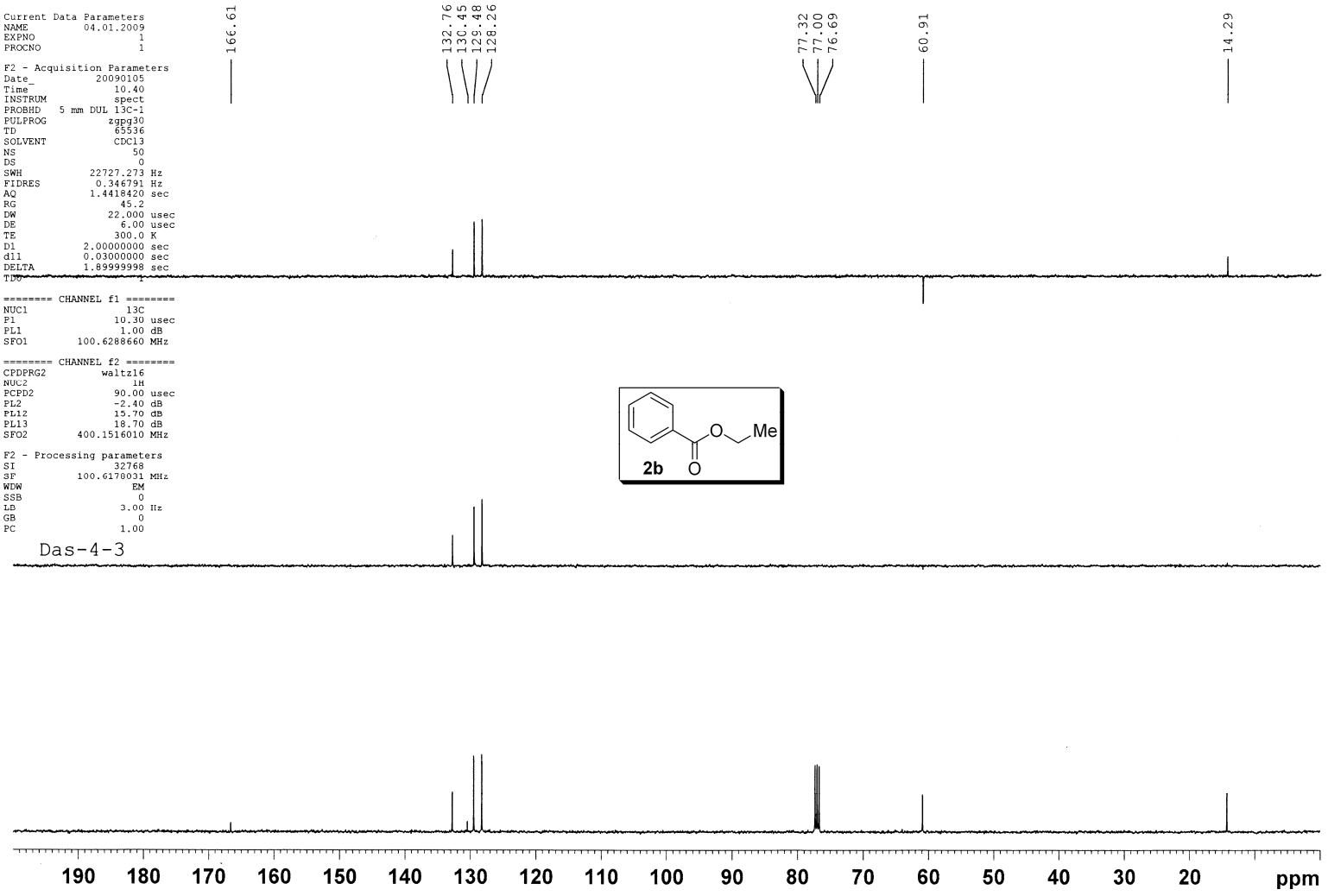

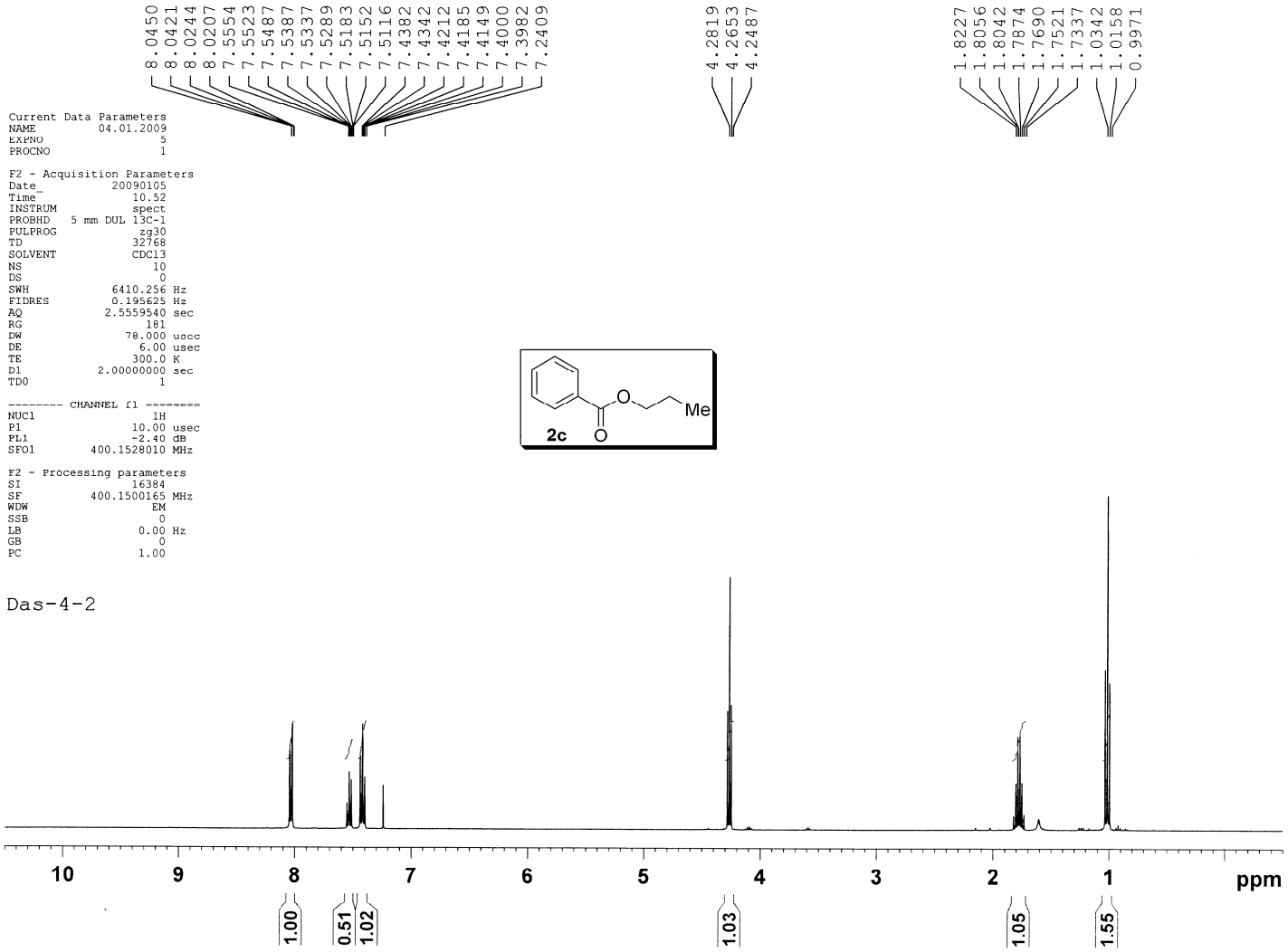

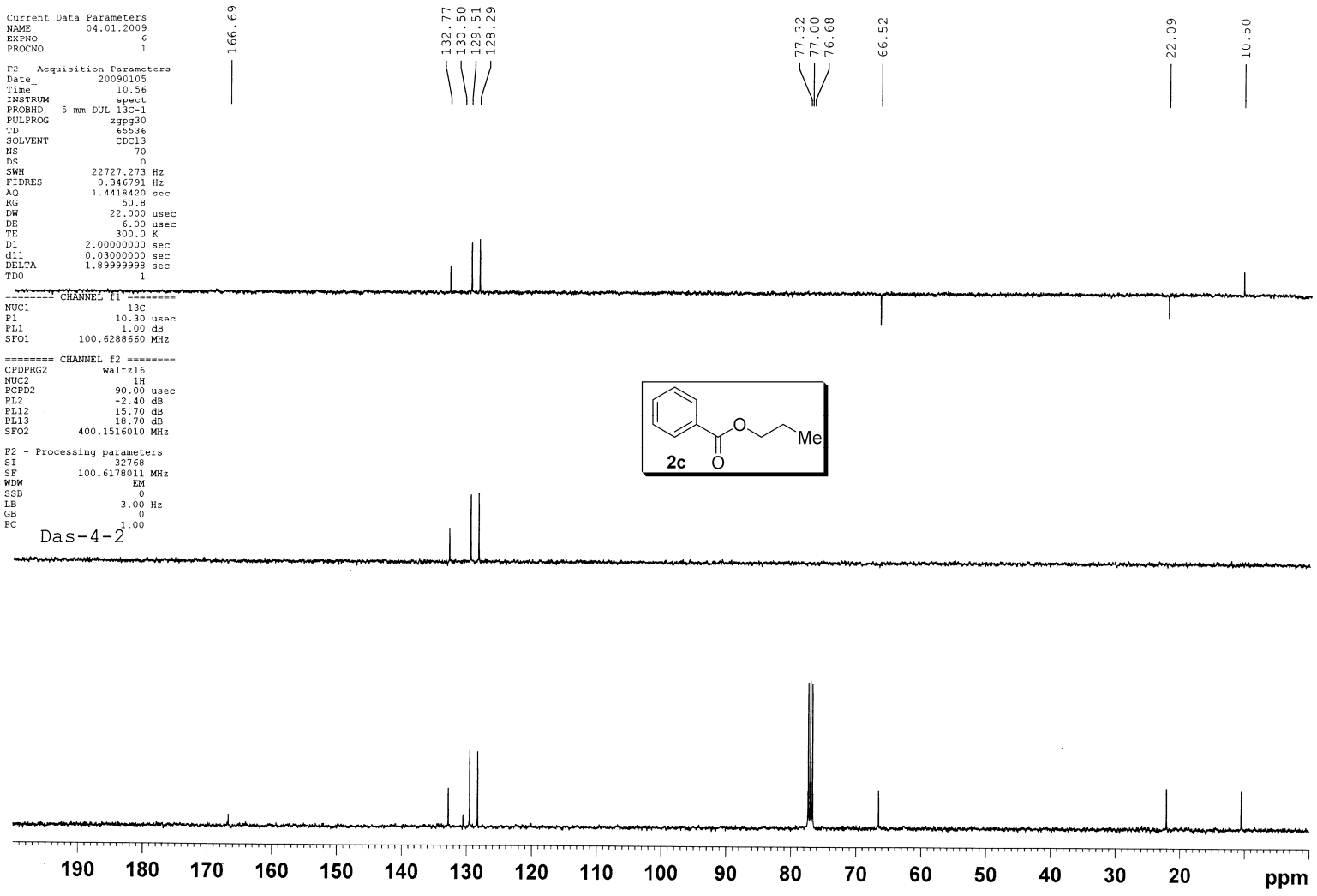

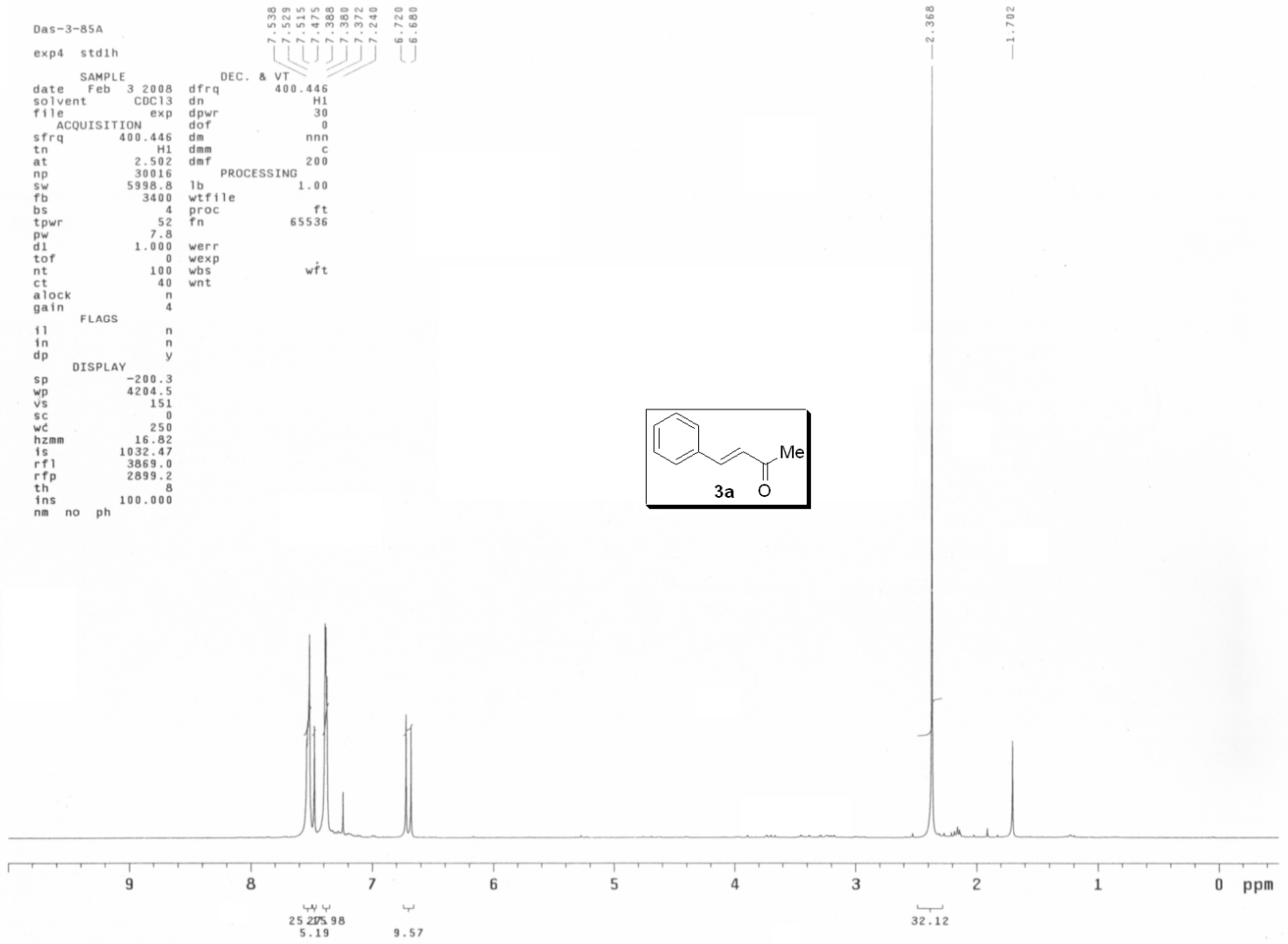

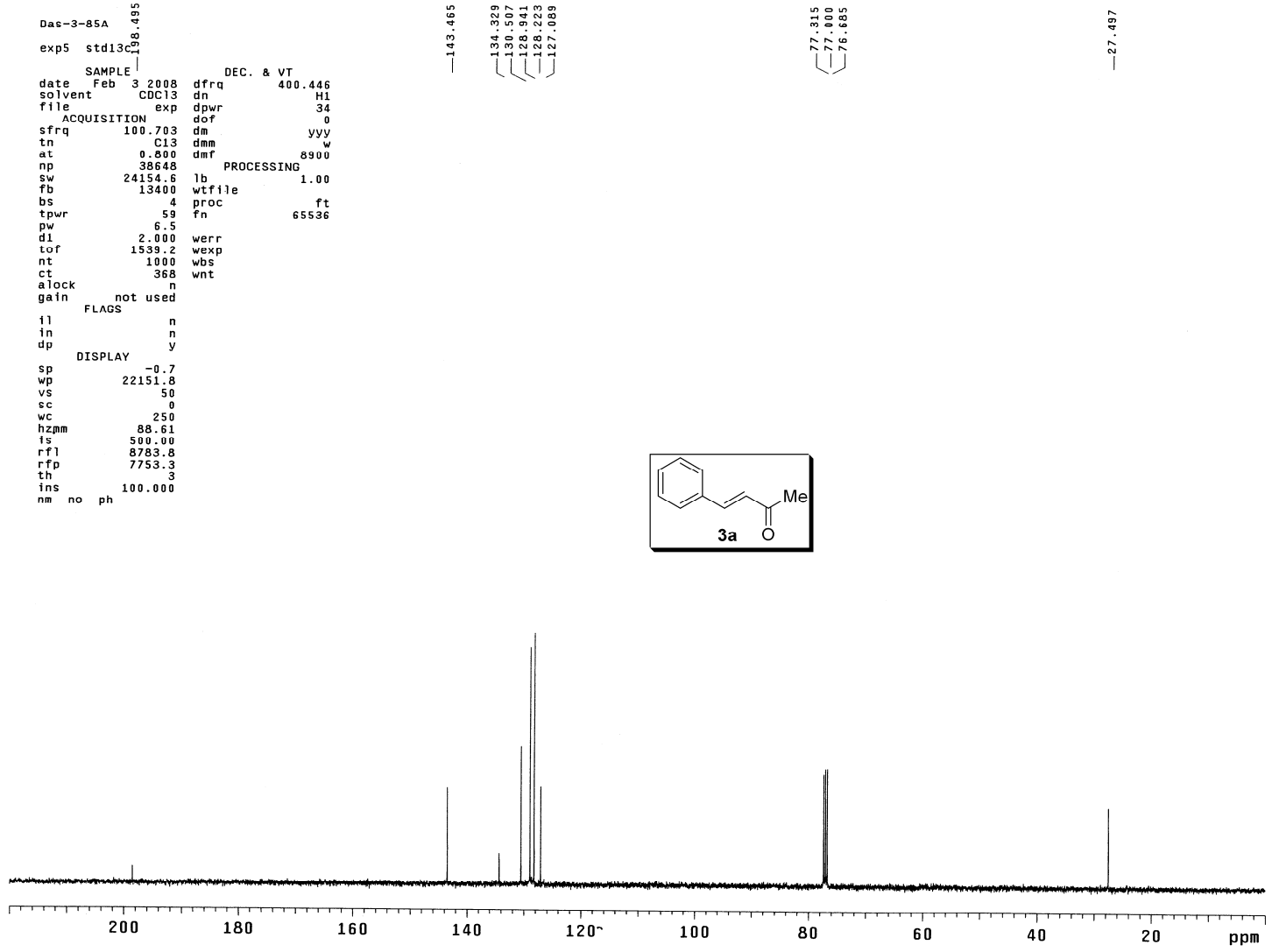
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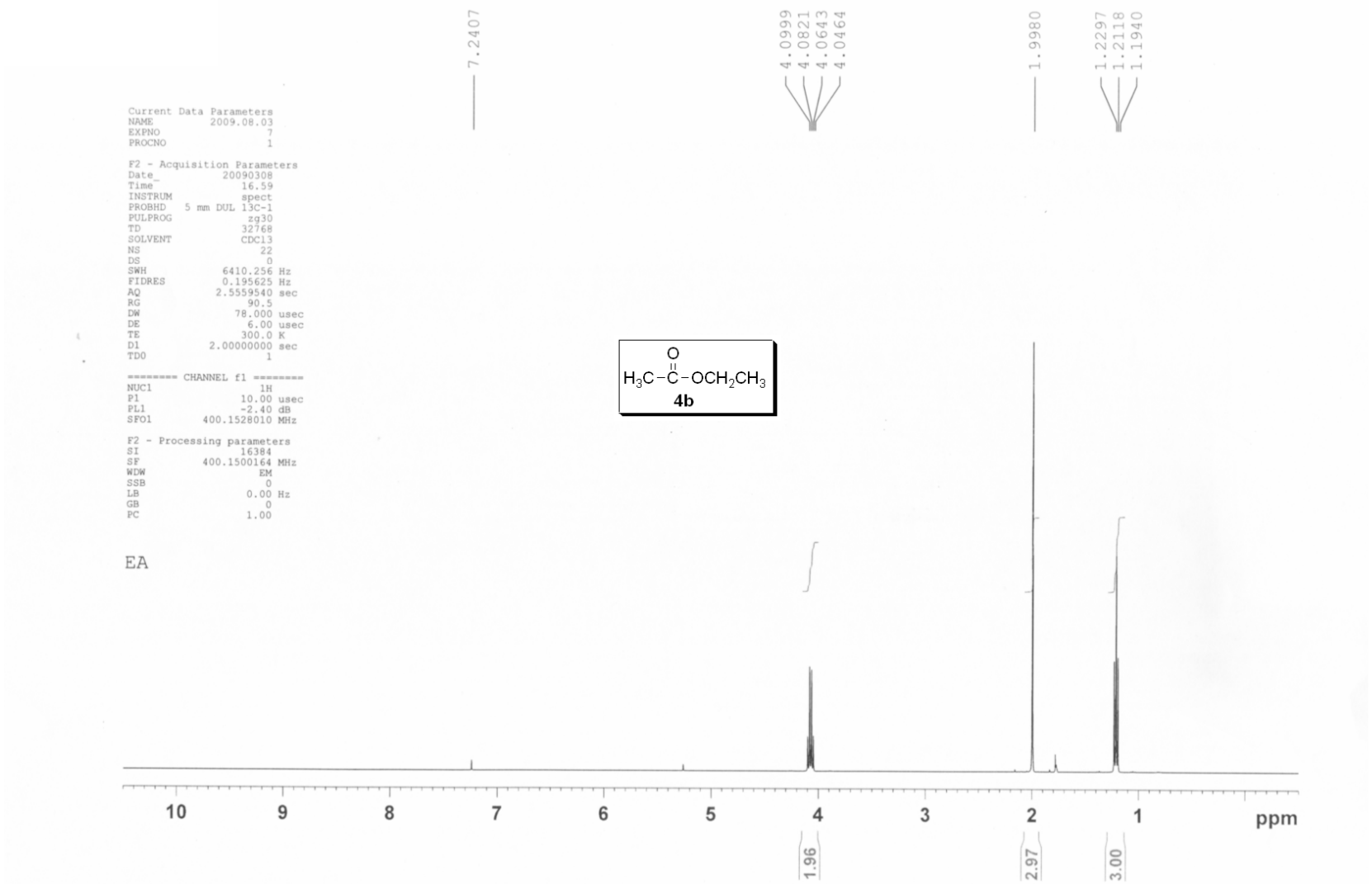
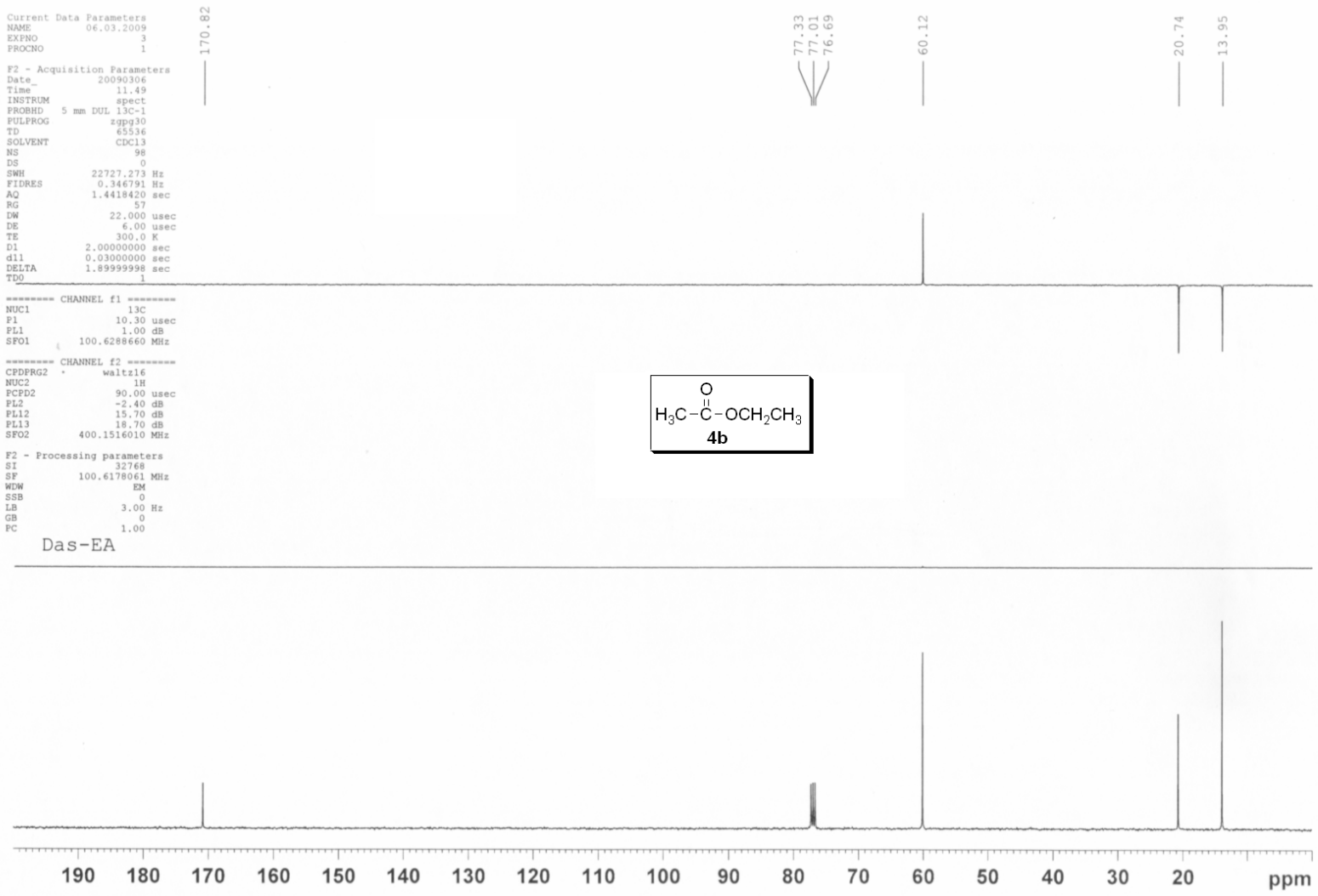
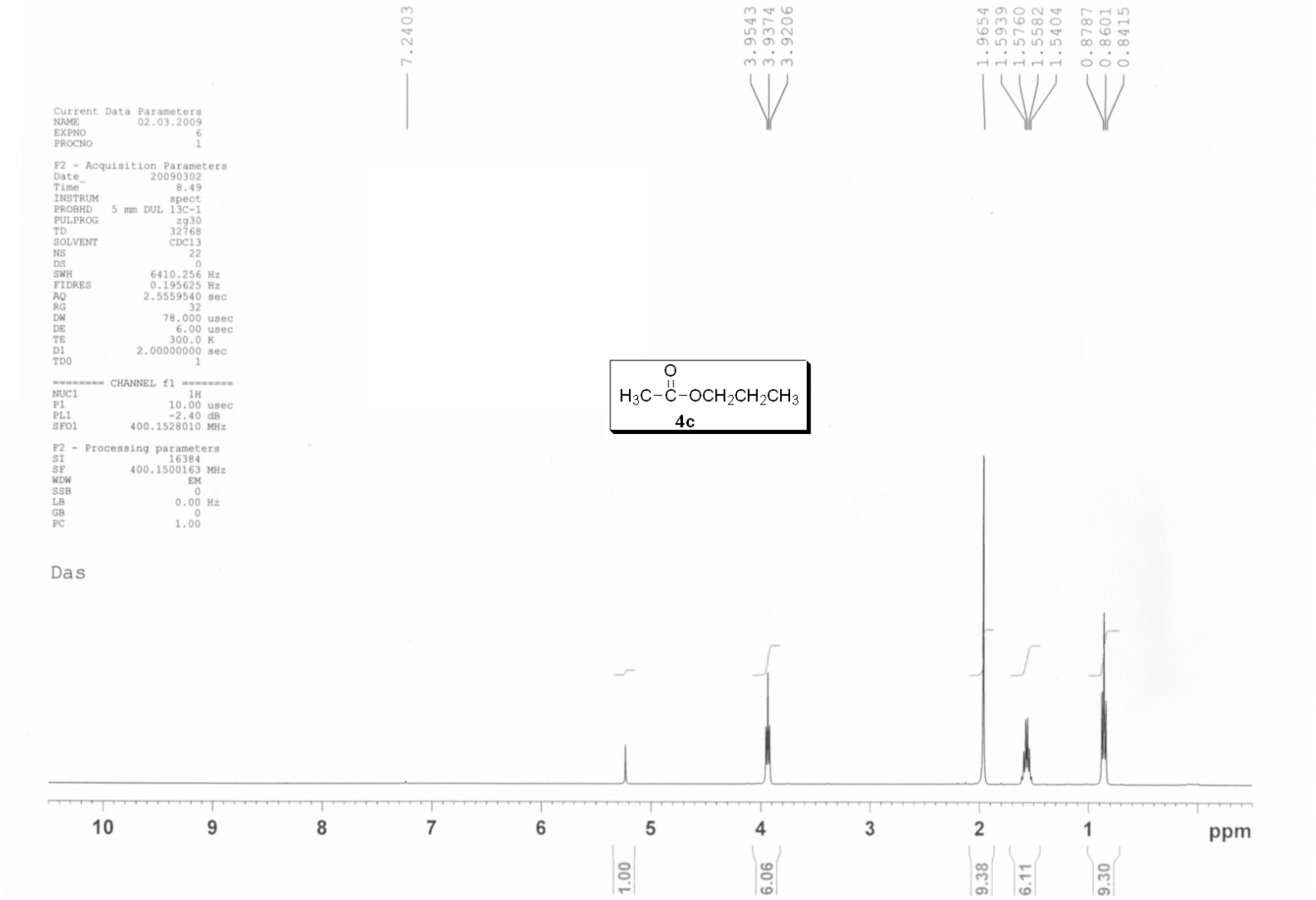
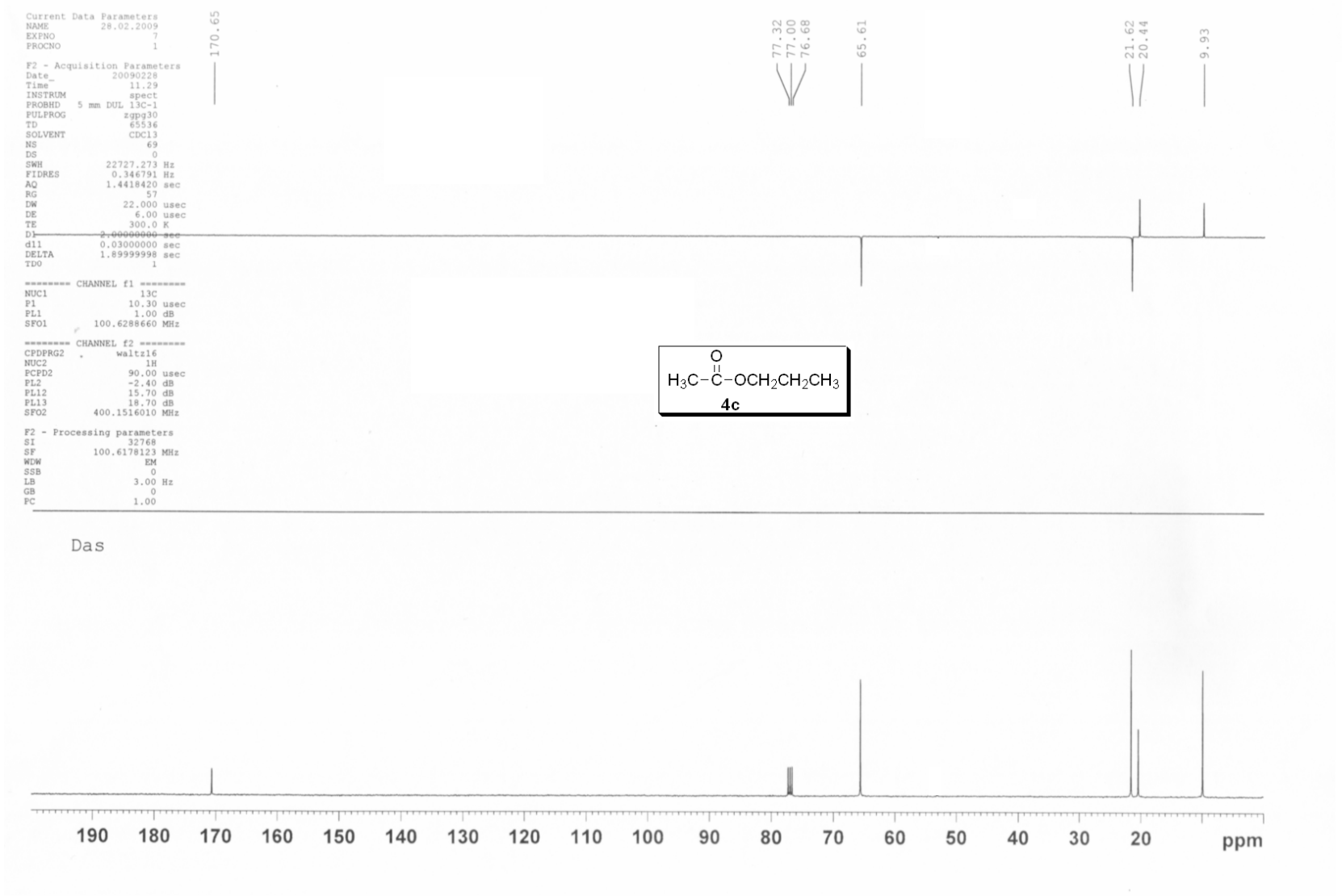
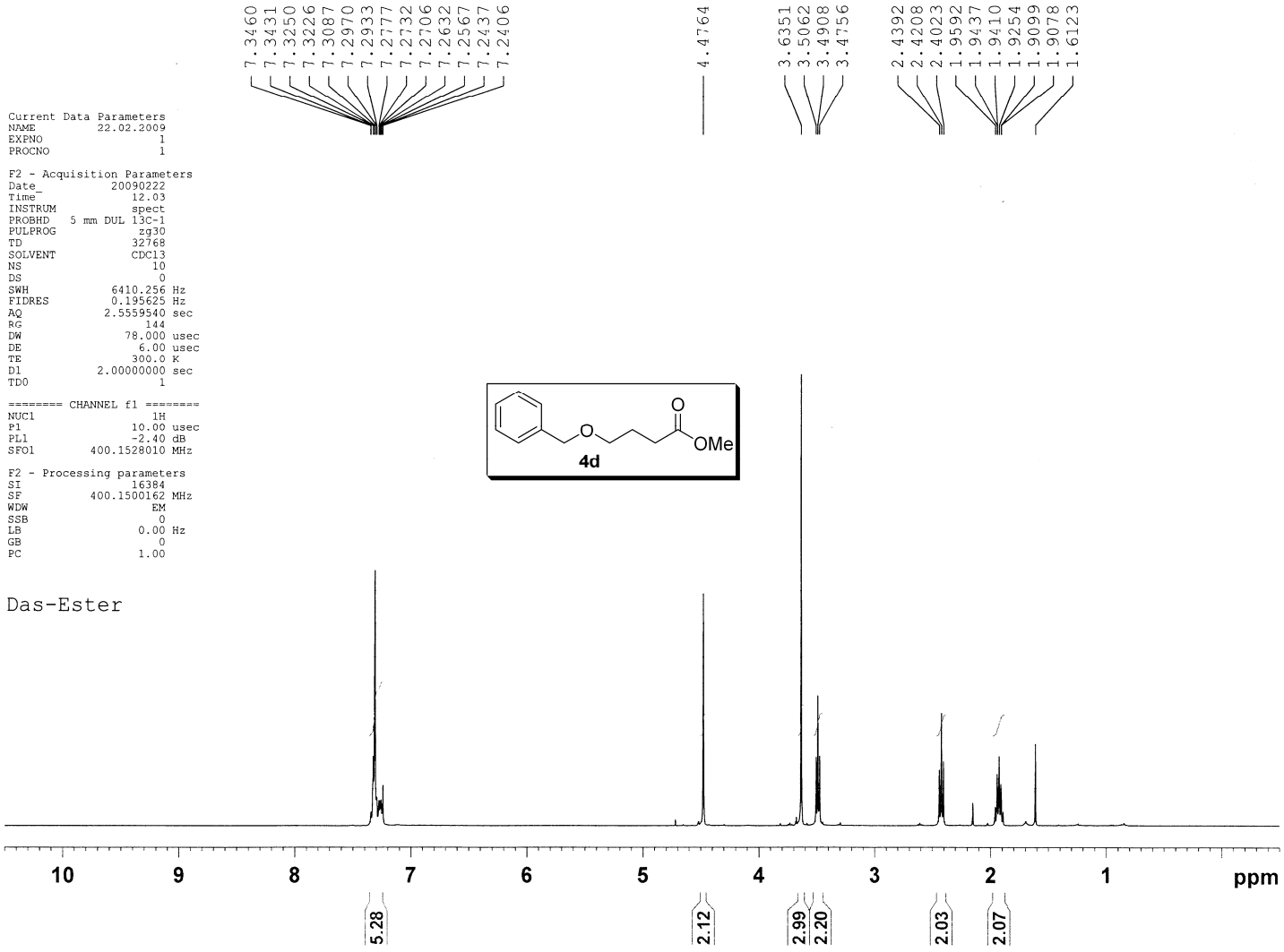
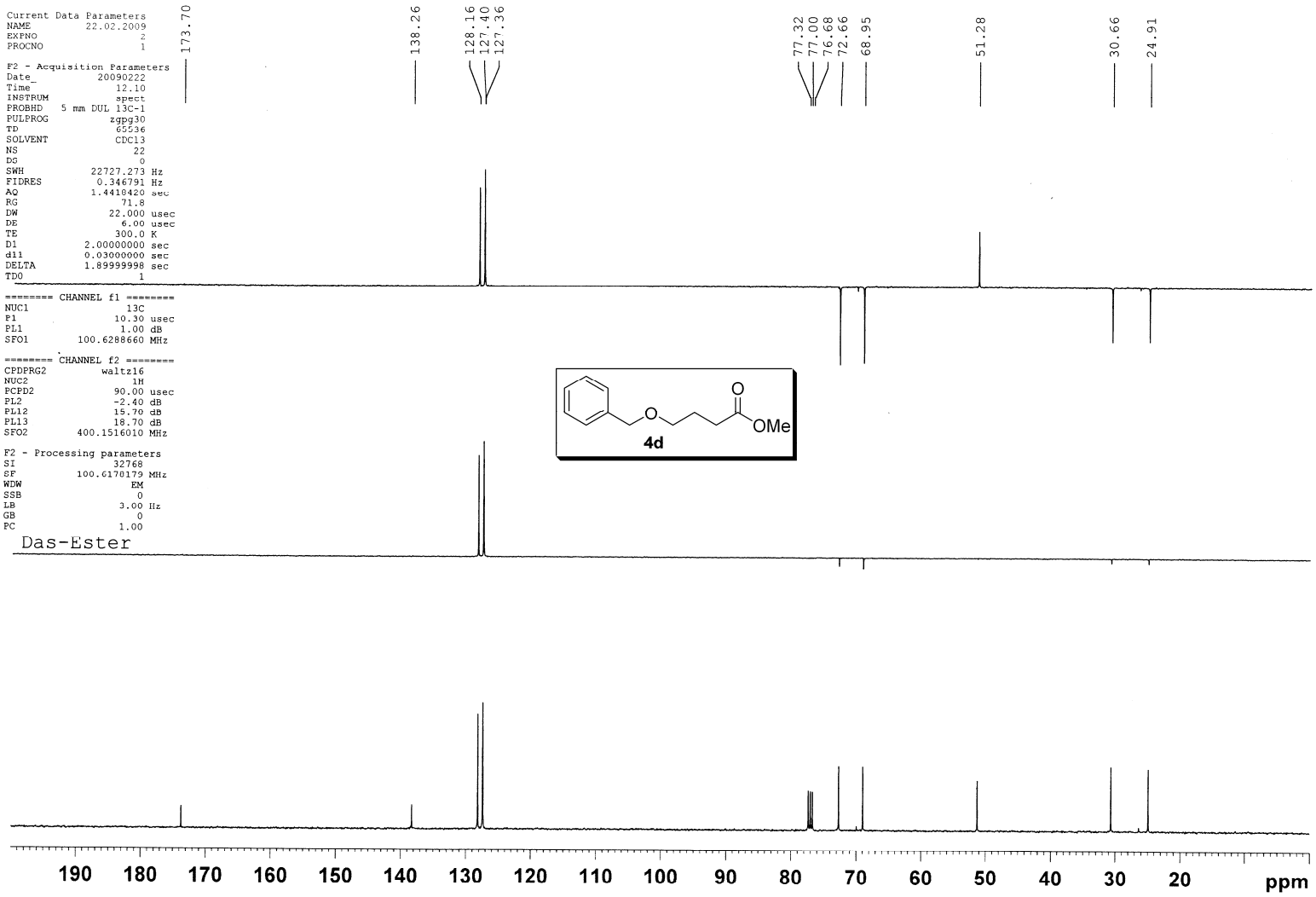


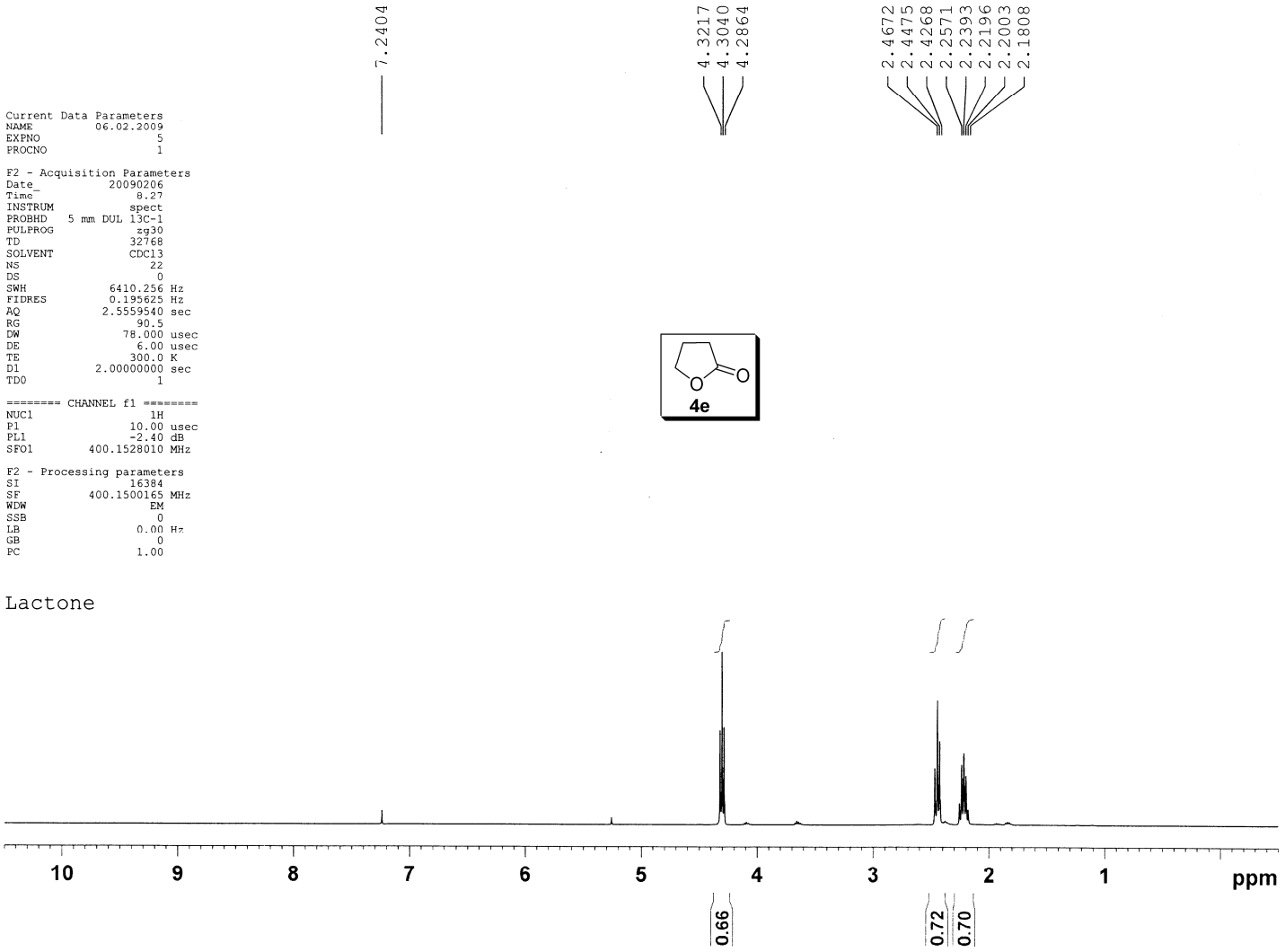


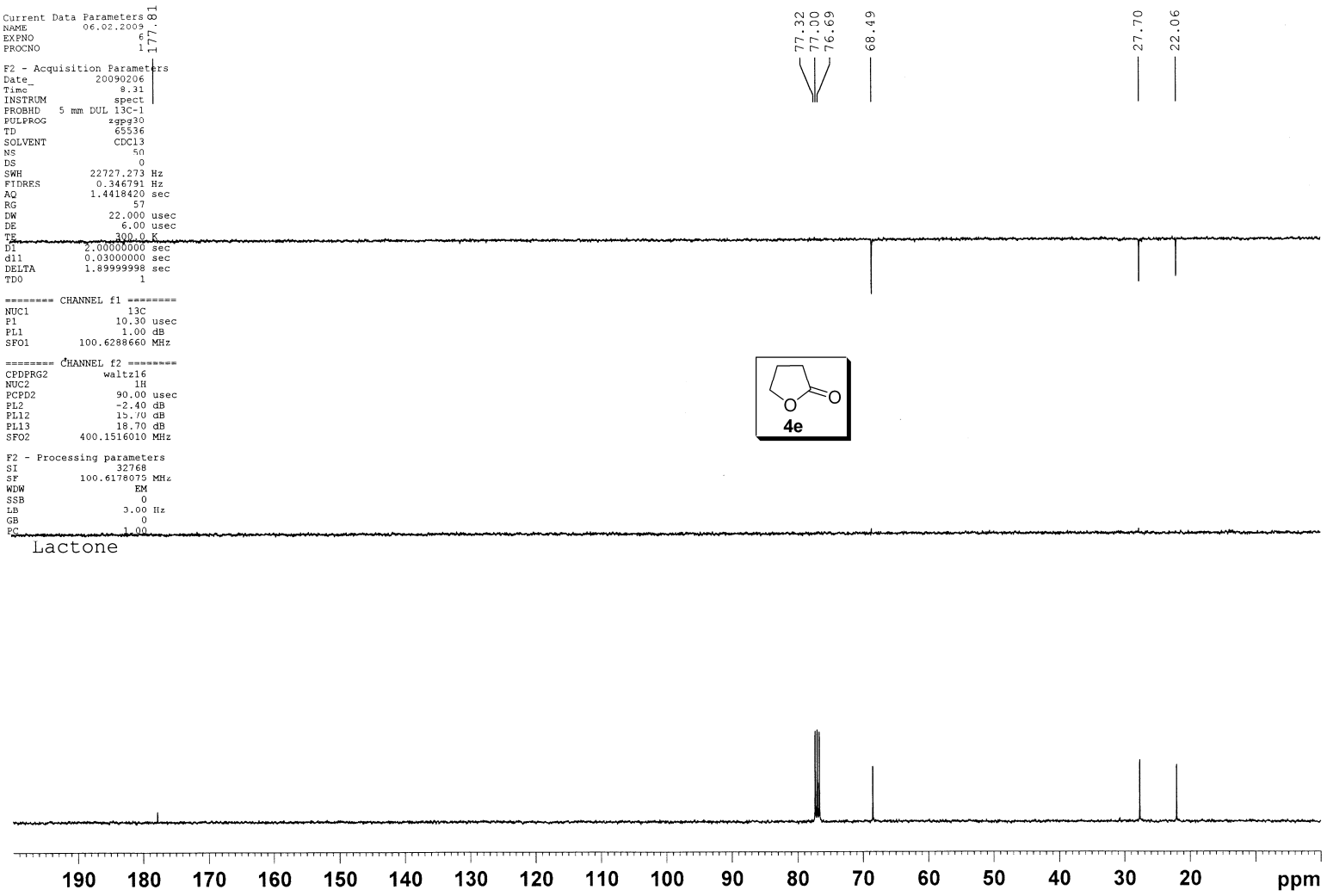


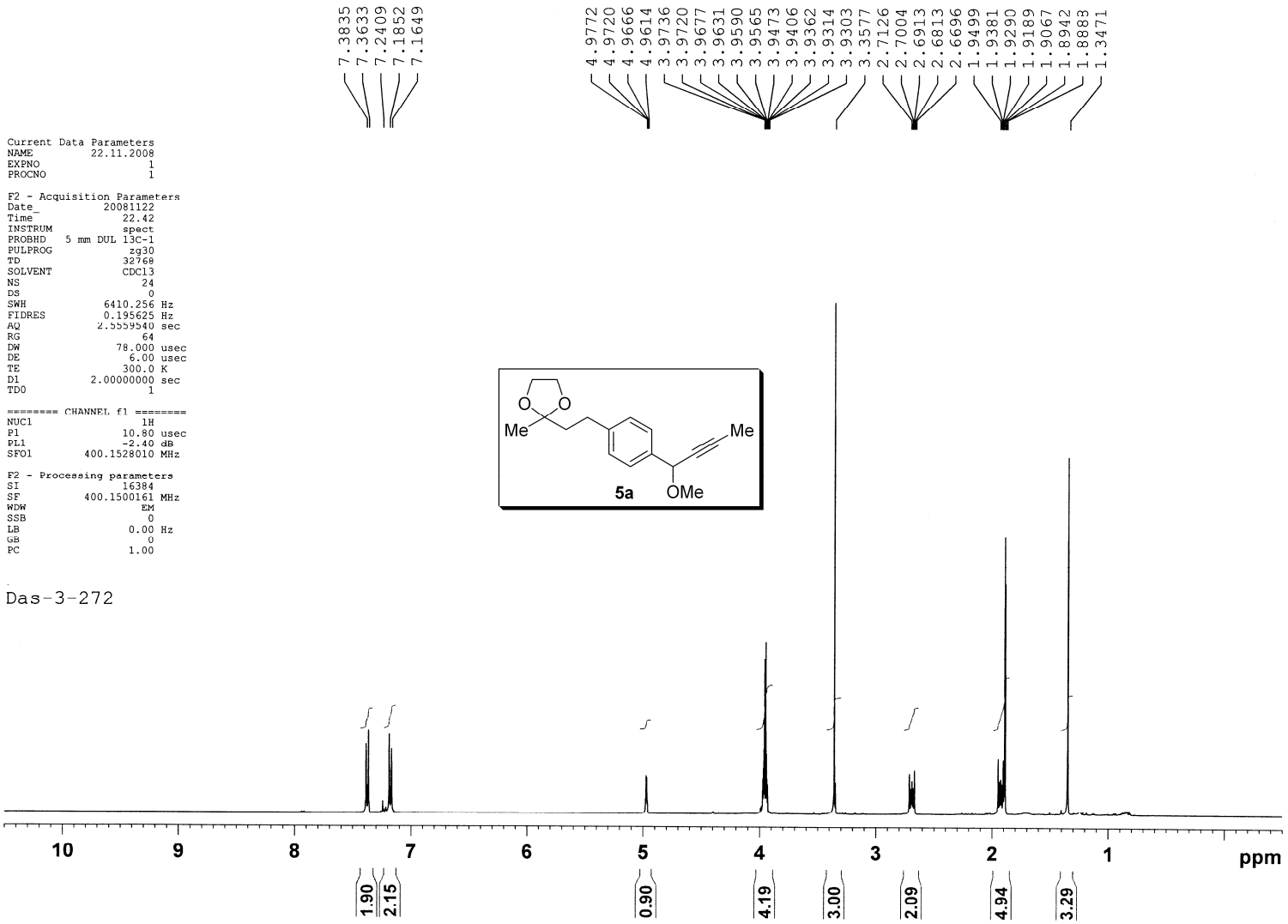


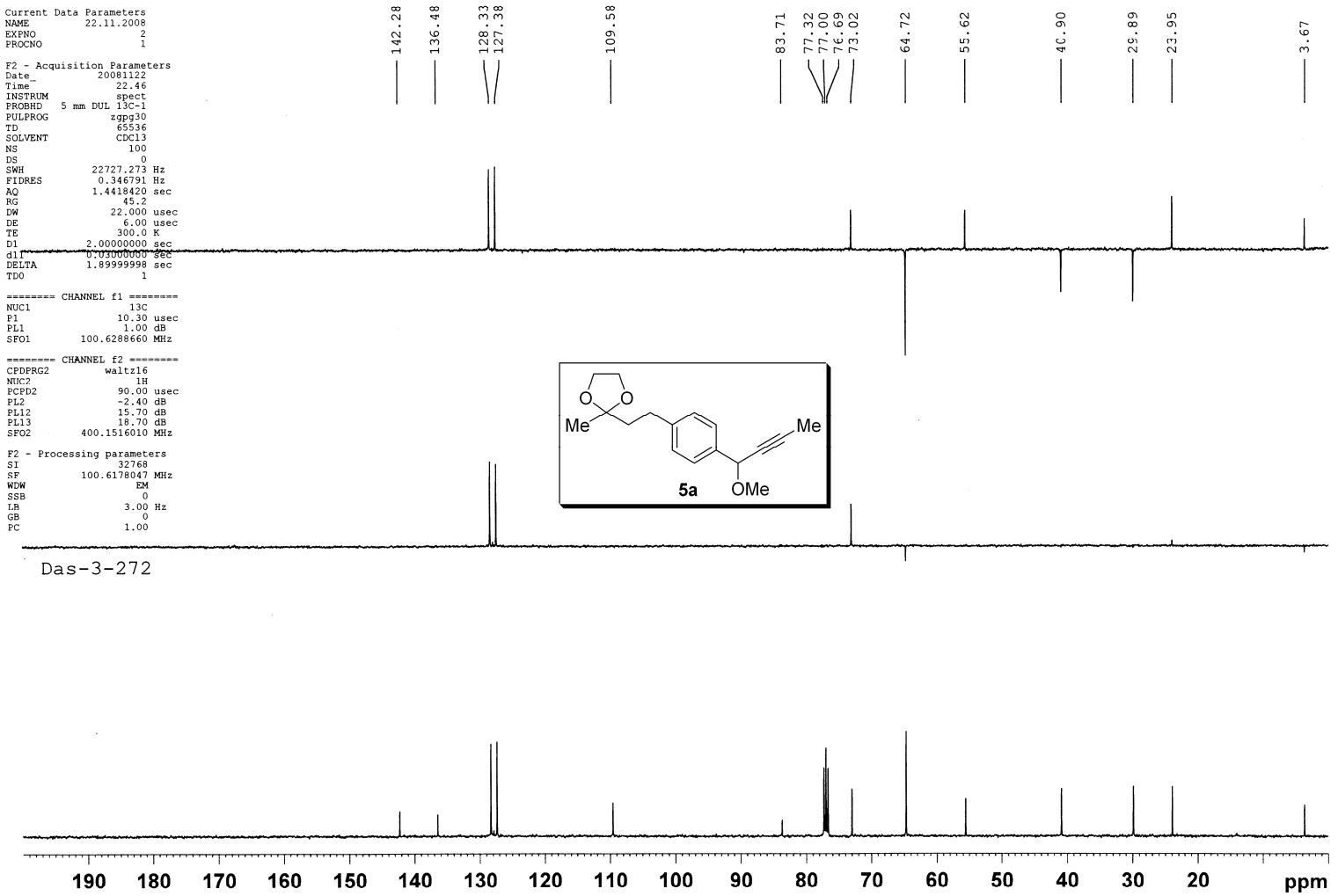


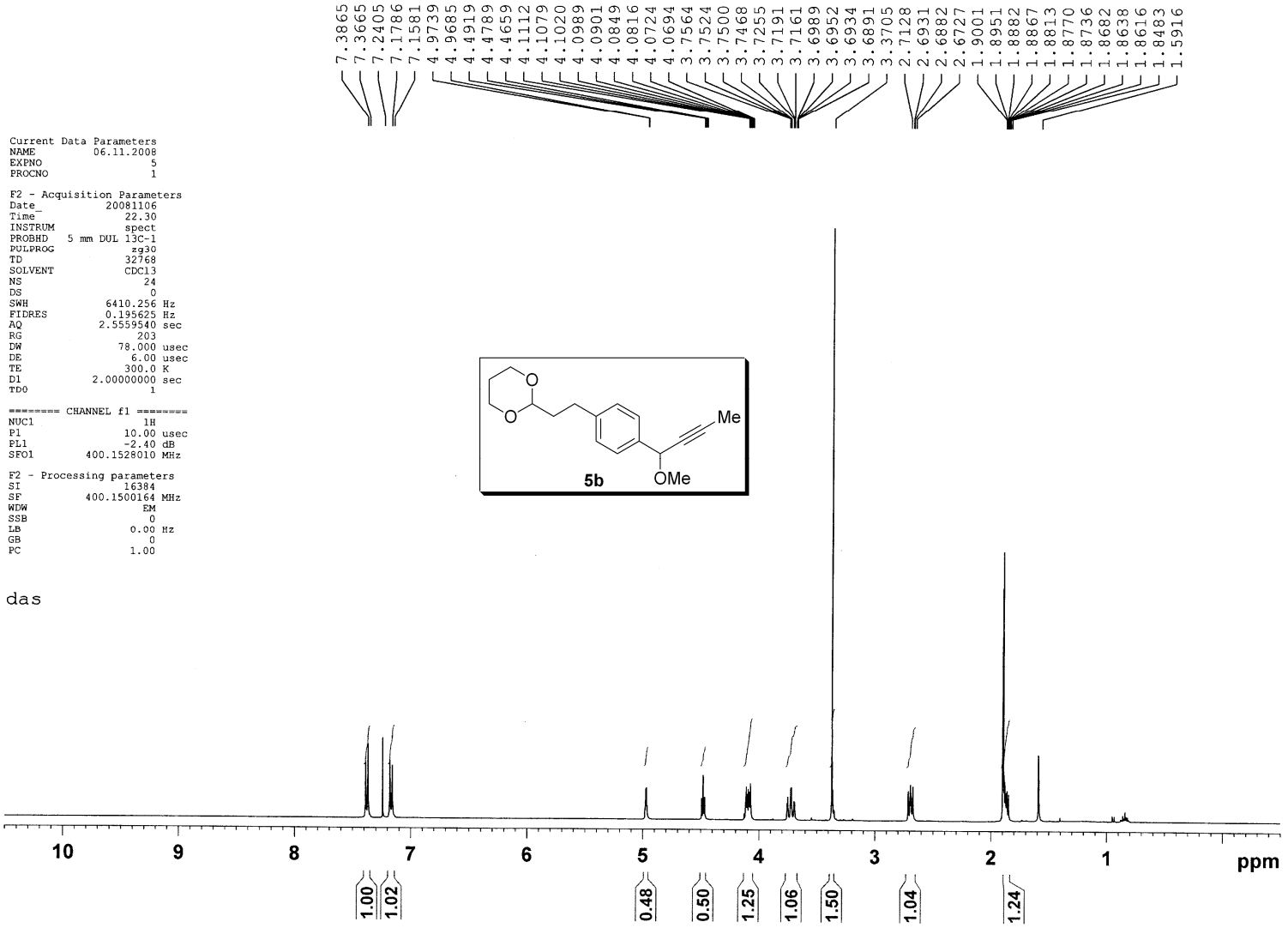

Current Data Parameters
NAME 22.02.2009
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090222
Time 12.10
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 22
DS 0
SWH 22727.273 Hz
FIDRES 0.346791 Hz
AQ 1.4418420 sec
RG 71.8
DW 22.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 10.30 usec
PL1 1.00 dB
SFO1 100.6288660 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -2.40 dB
PL12 15.70 dB
PL13 18.70 dB
SFO2 400.1516010 MHz
F2 - Processing parameters
SI 32768
SF 100.6170179 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.00
Das-Ester
173.70
138.26
128.16
127.40
127.36
77.32
77.00
76.68
72.66
68.95
51.28
30.66
24.91
O
O
OMe
4d
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

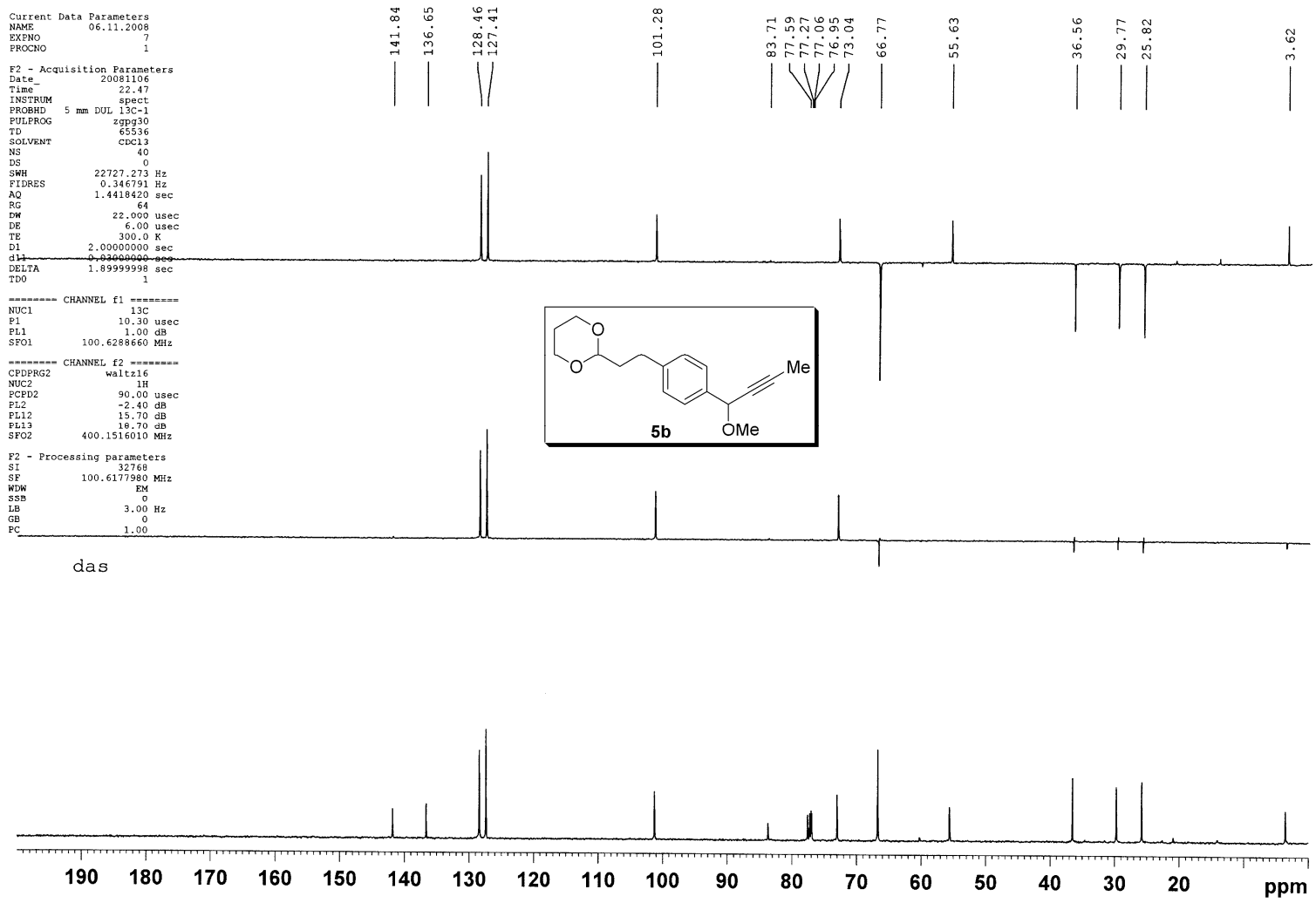


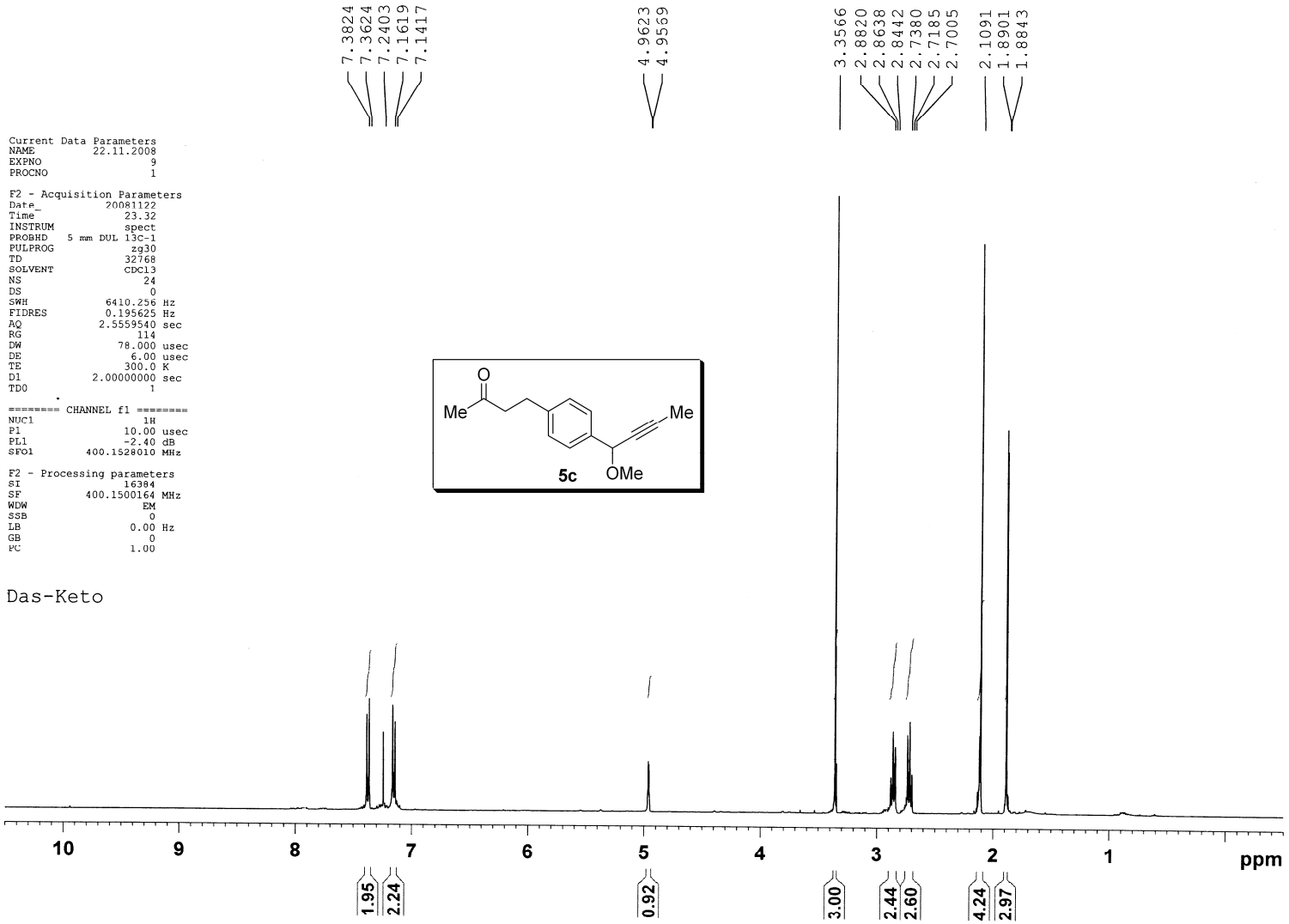

Current Data Parameters
NAME 06.02.2009
EXPNO 6
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090206
Time 8.31
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 50
DS 0
SWH 22727.273 Hz
FIDRES 0.346791 Hz
AQ 1.4418420 sec
RG 57
DW 22.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 10.30 usec
PL1 1.00 dB
SFO1 100.6288660 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -2.40 dB
PL12 15.70 dB
PL13 18.70 dB
SFO2 400.1516010 MHz
F2 - Processing parameters
SI 32768
SF 100.6178075 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.00
177.81
77.32
77.00
76.69
68.49
27.70
22.06
4e
Lactone
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

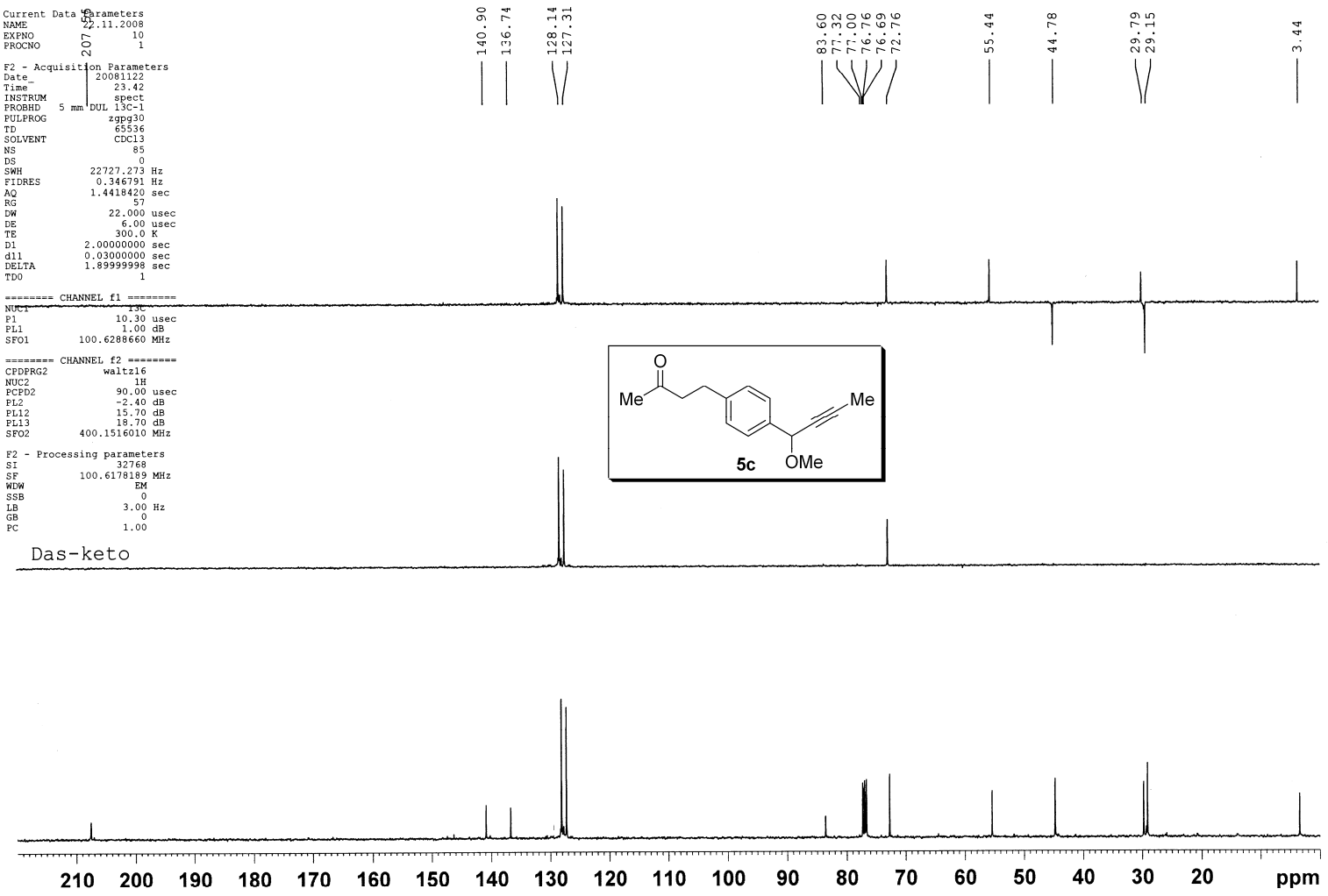


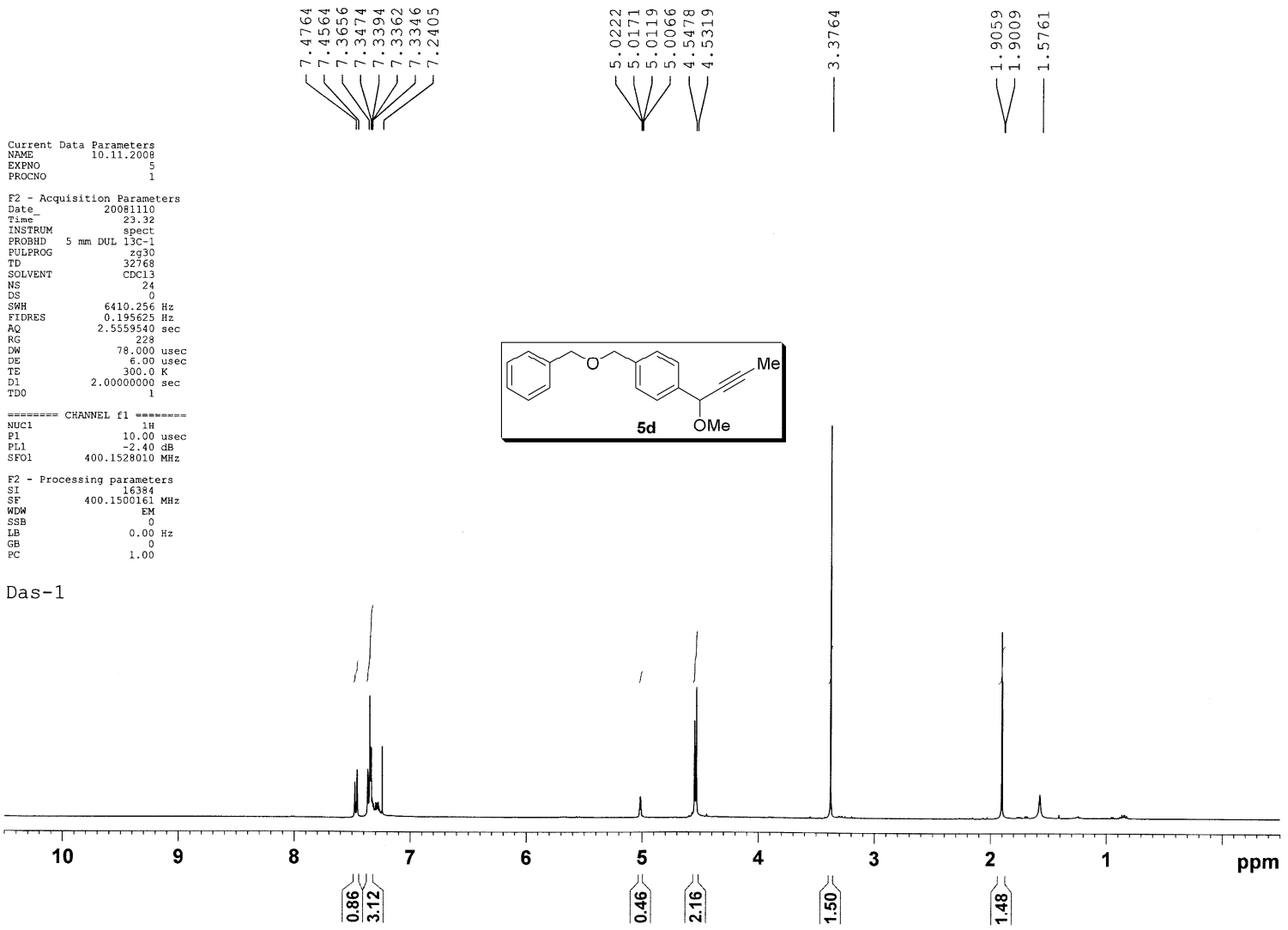


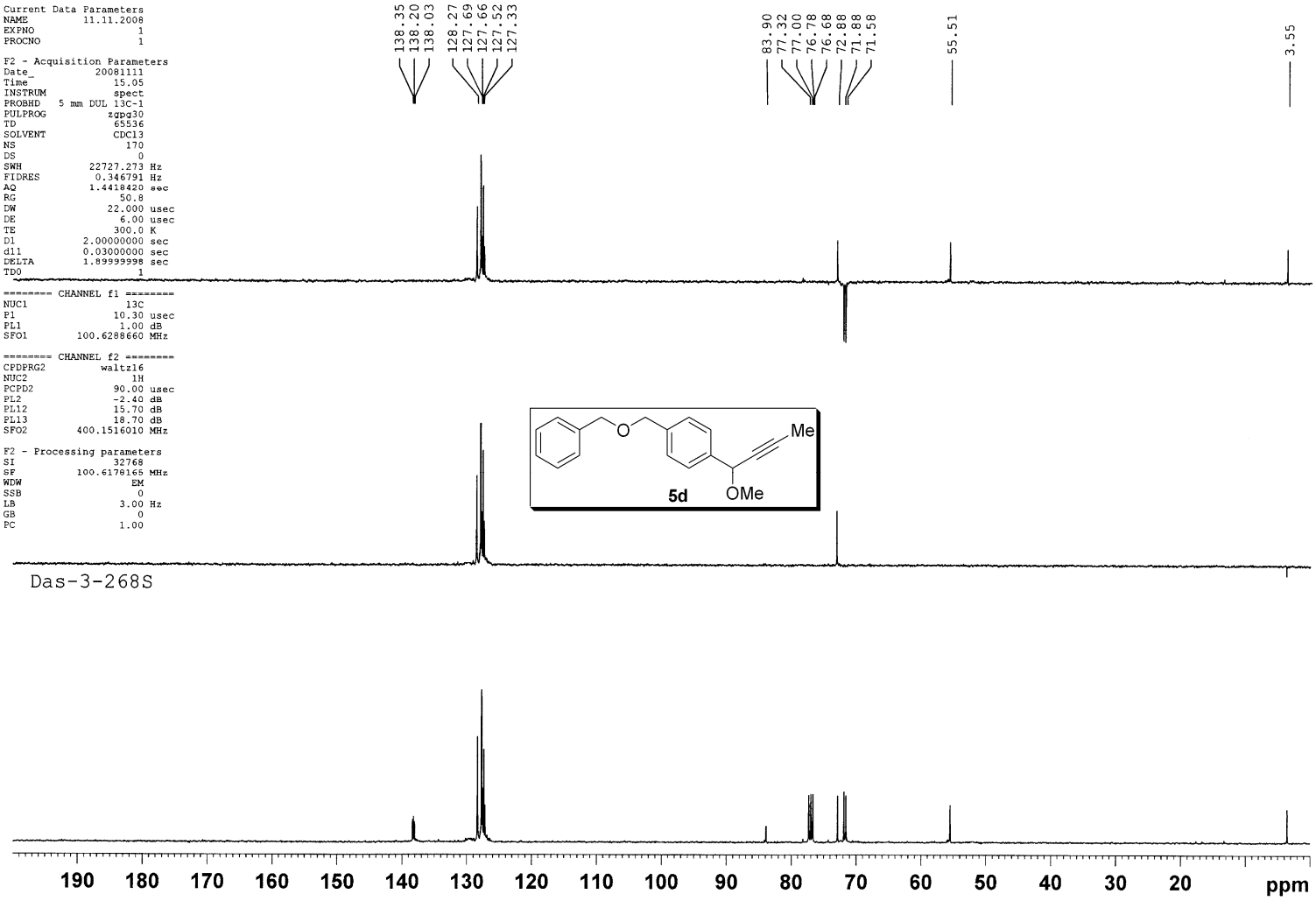


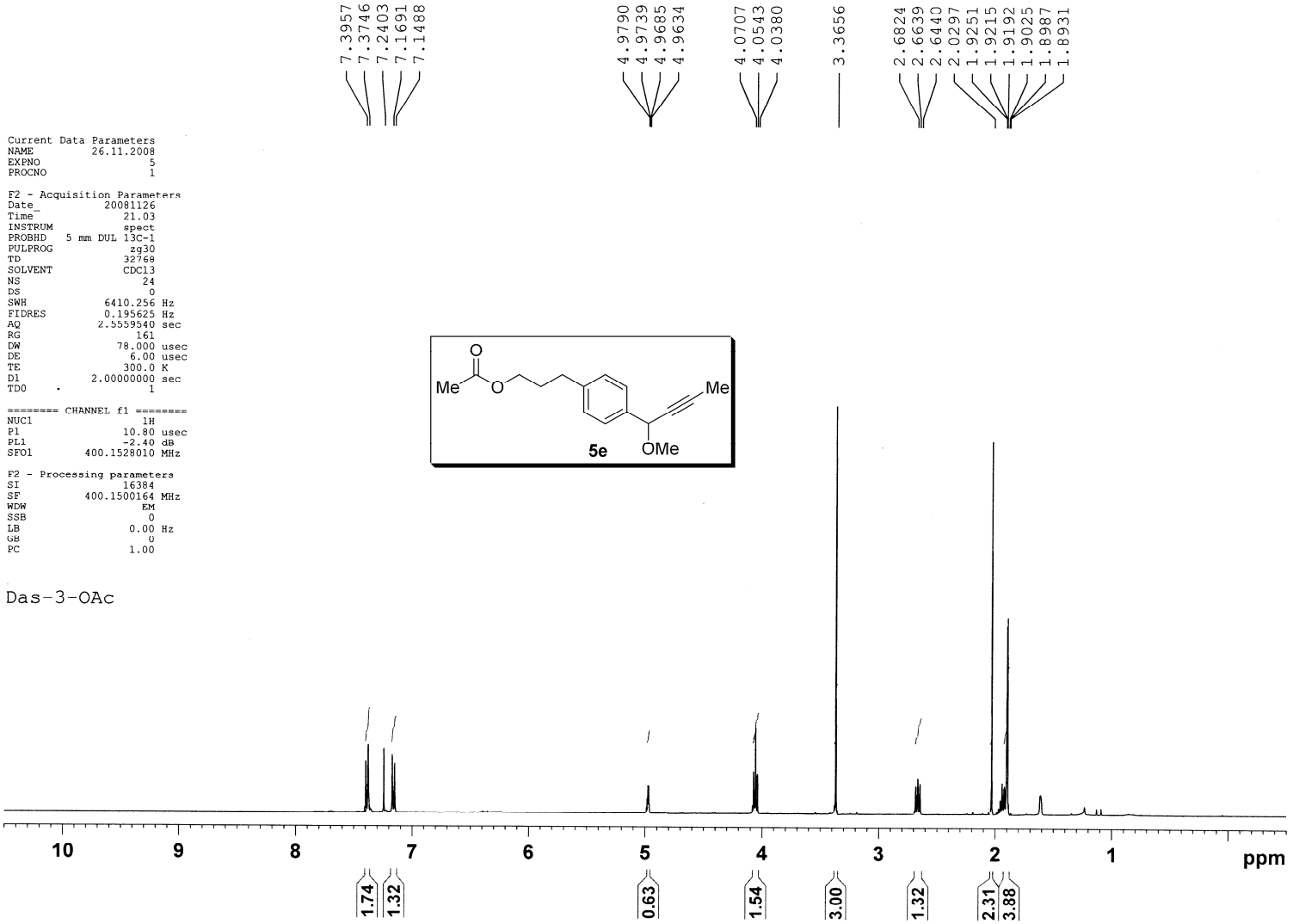


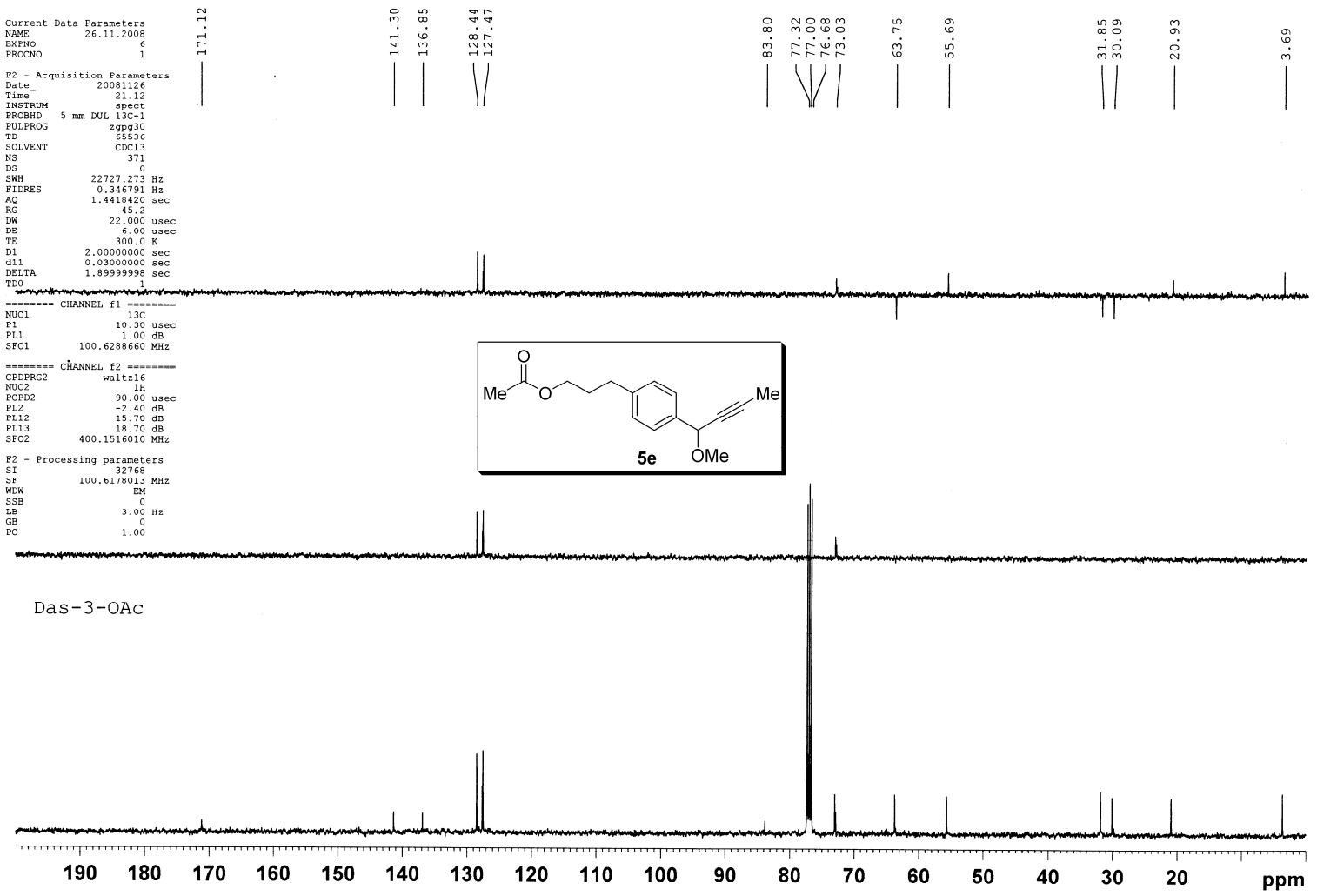


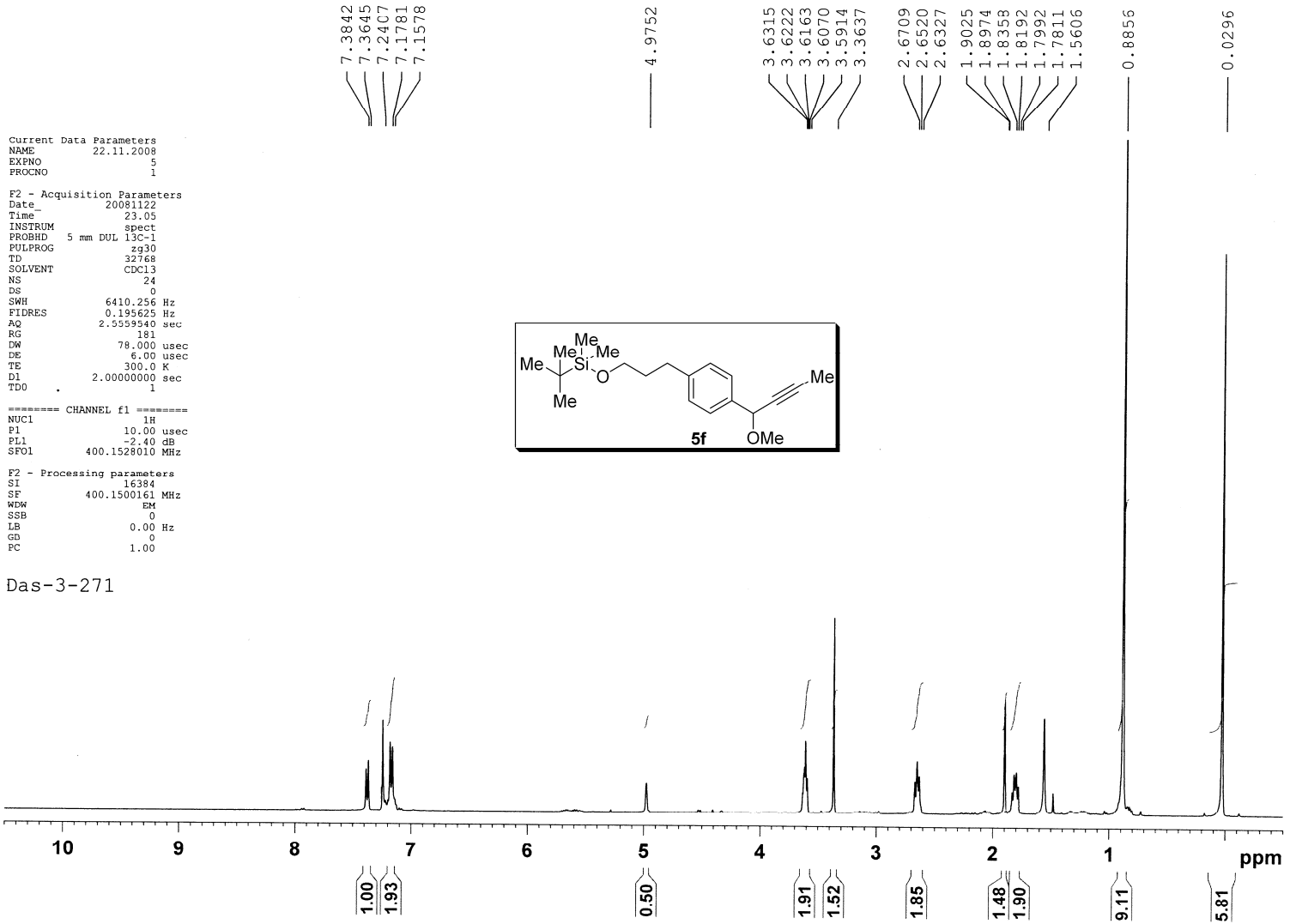


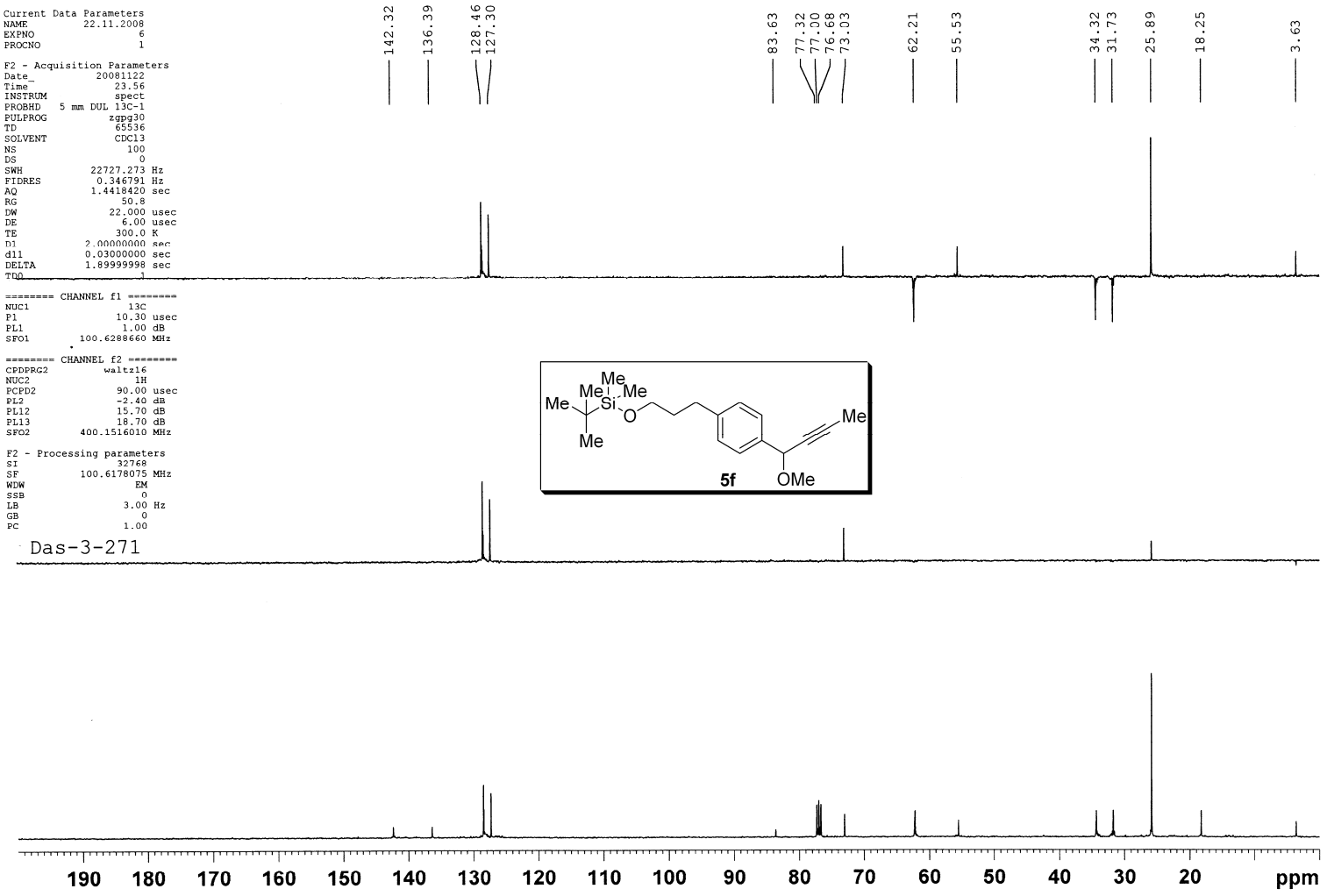


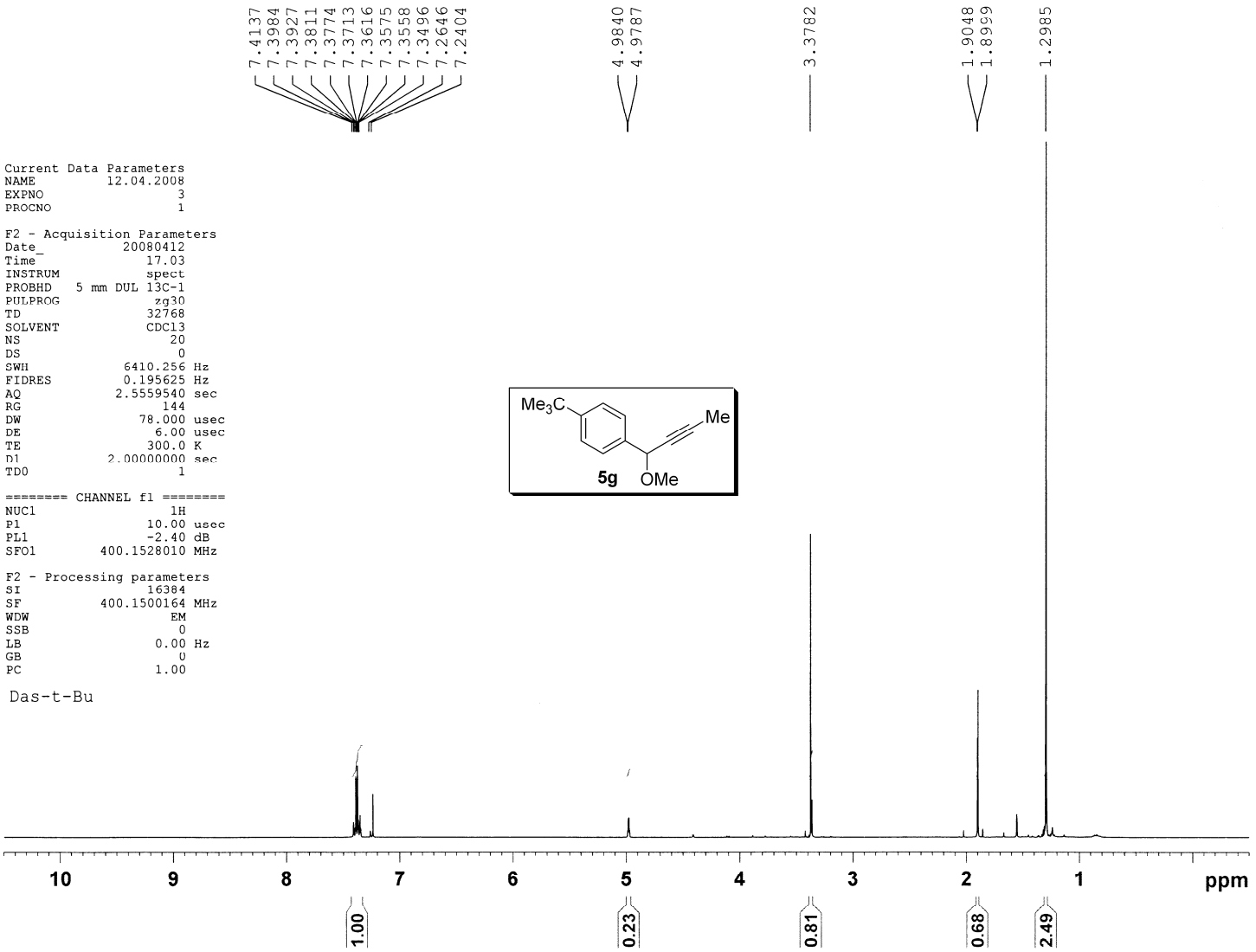


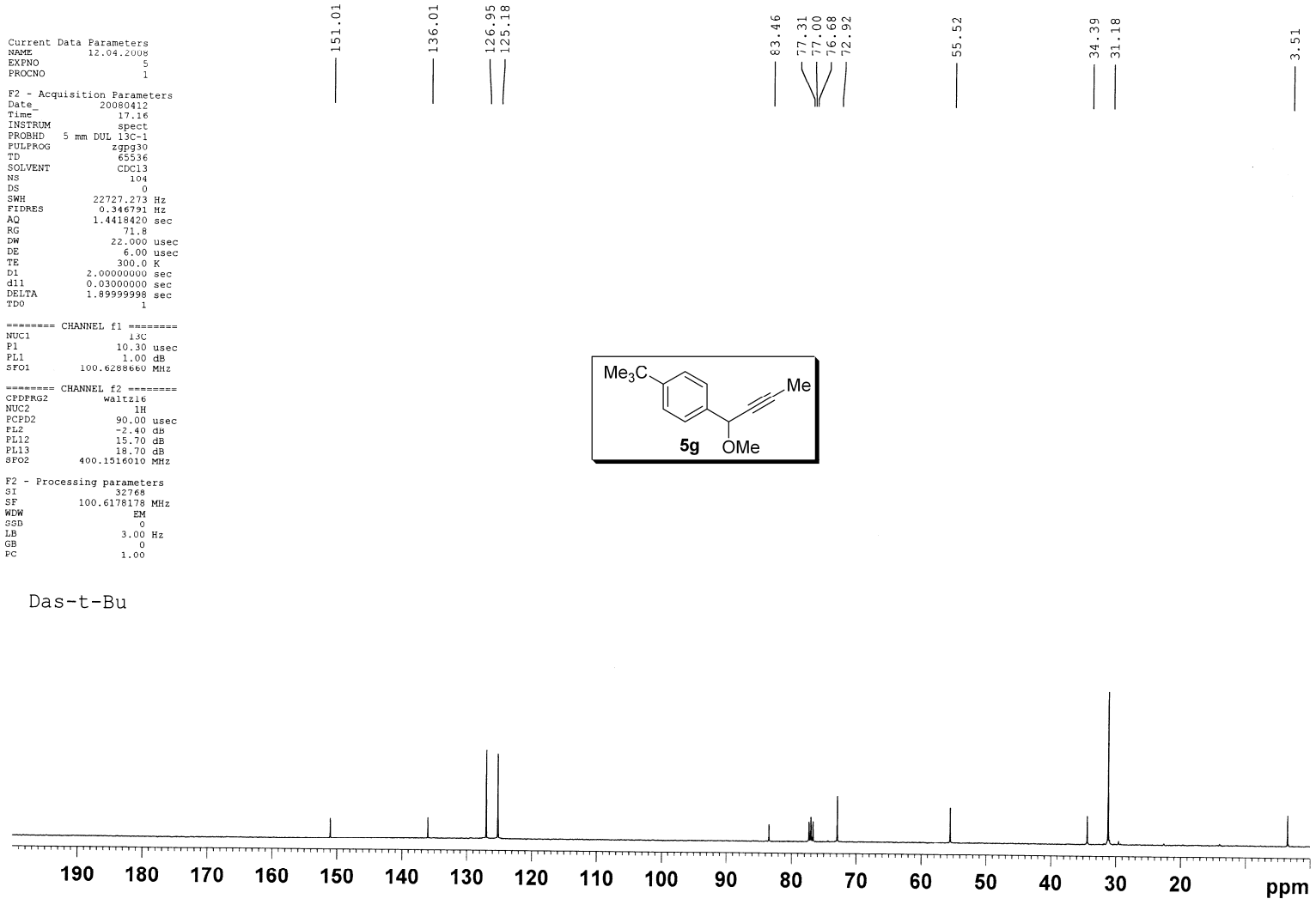


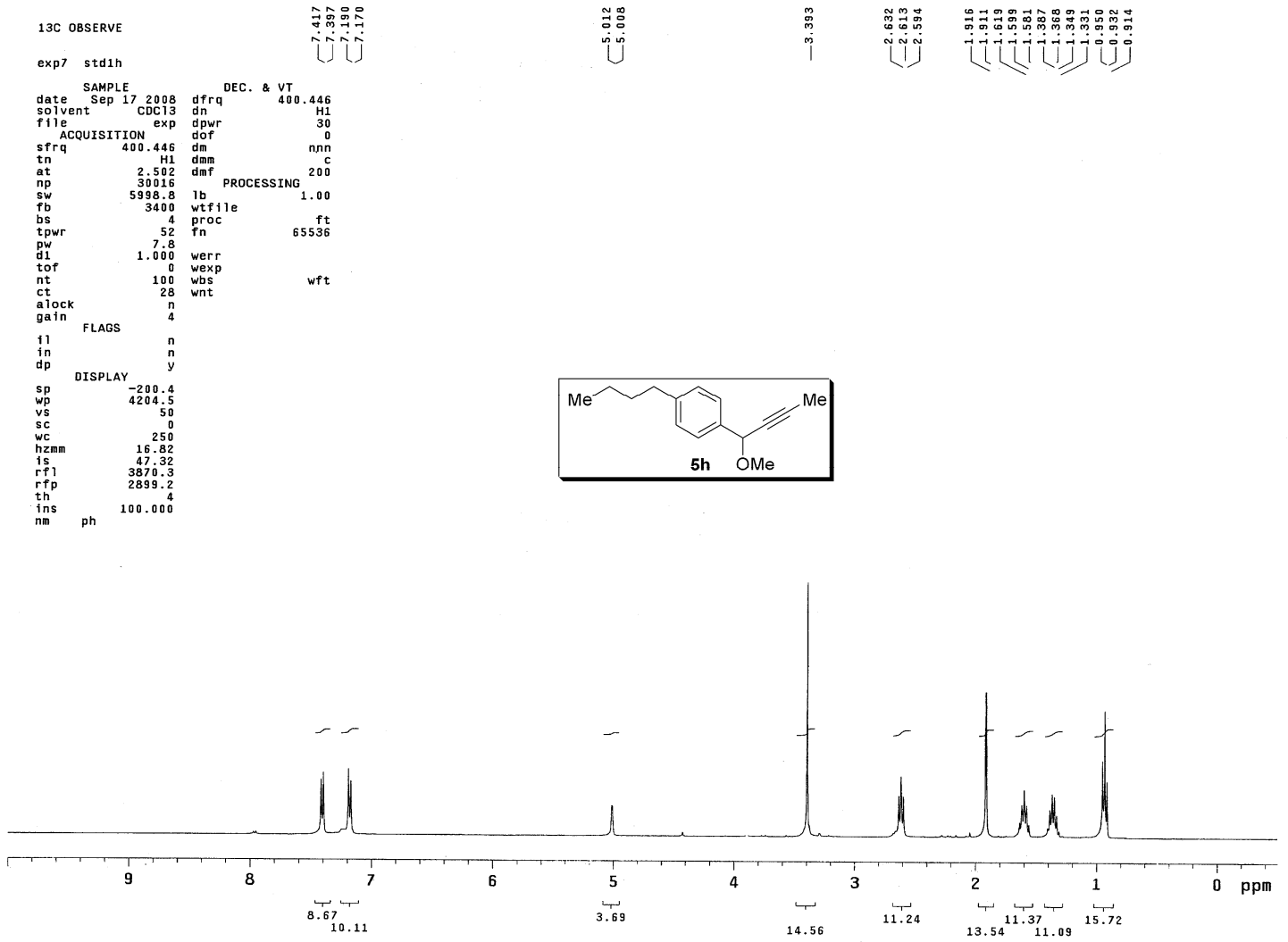


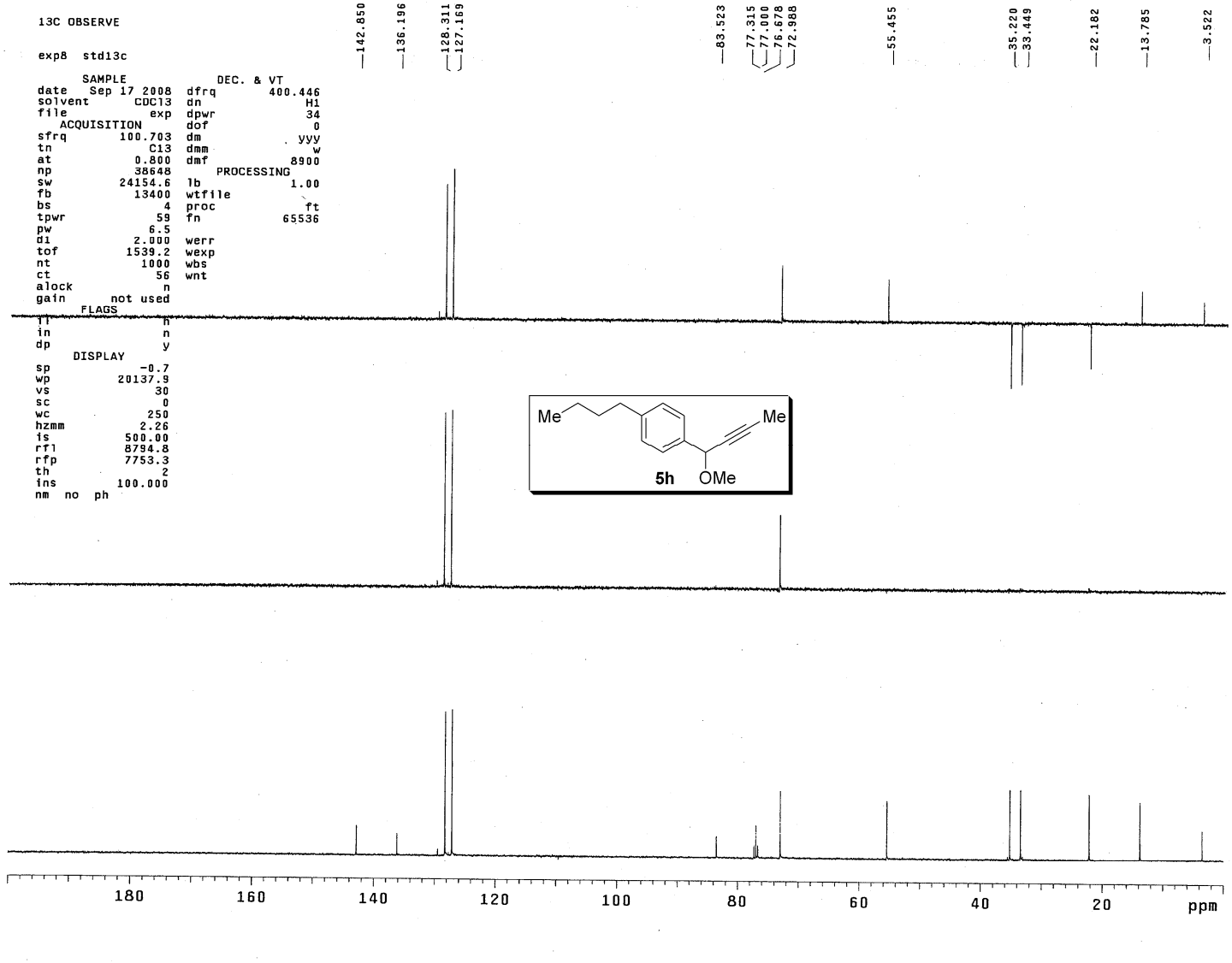


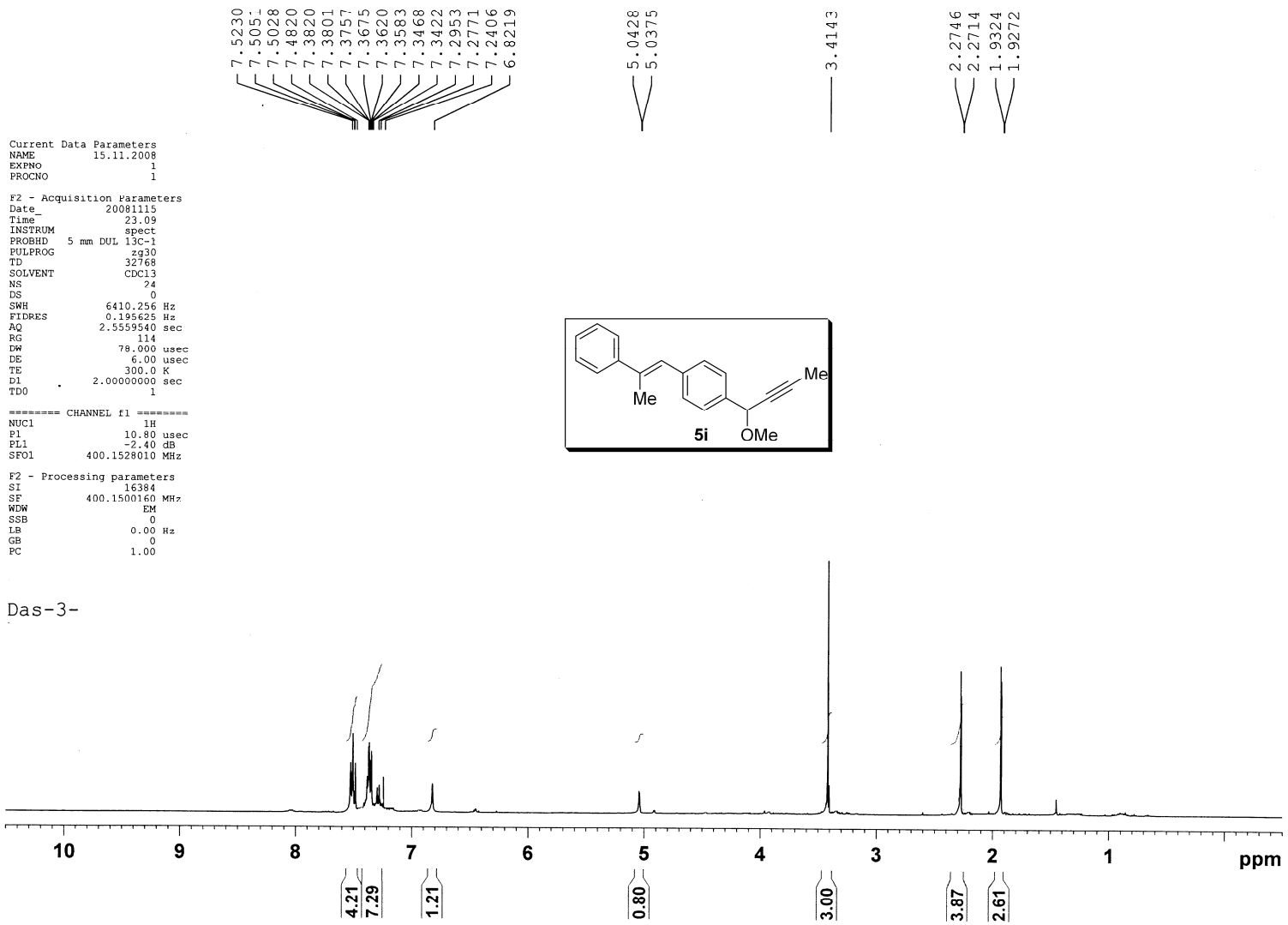


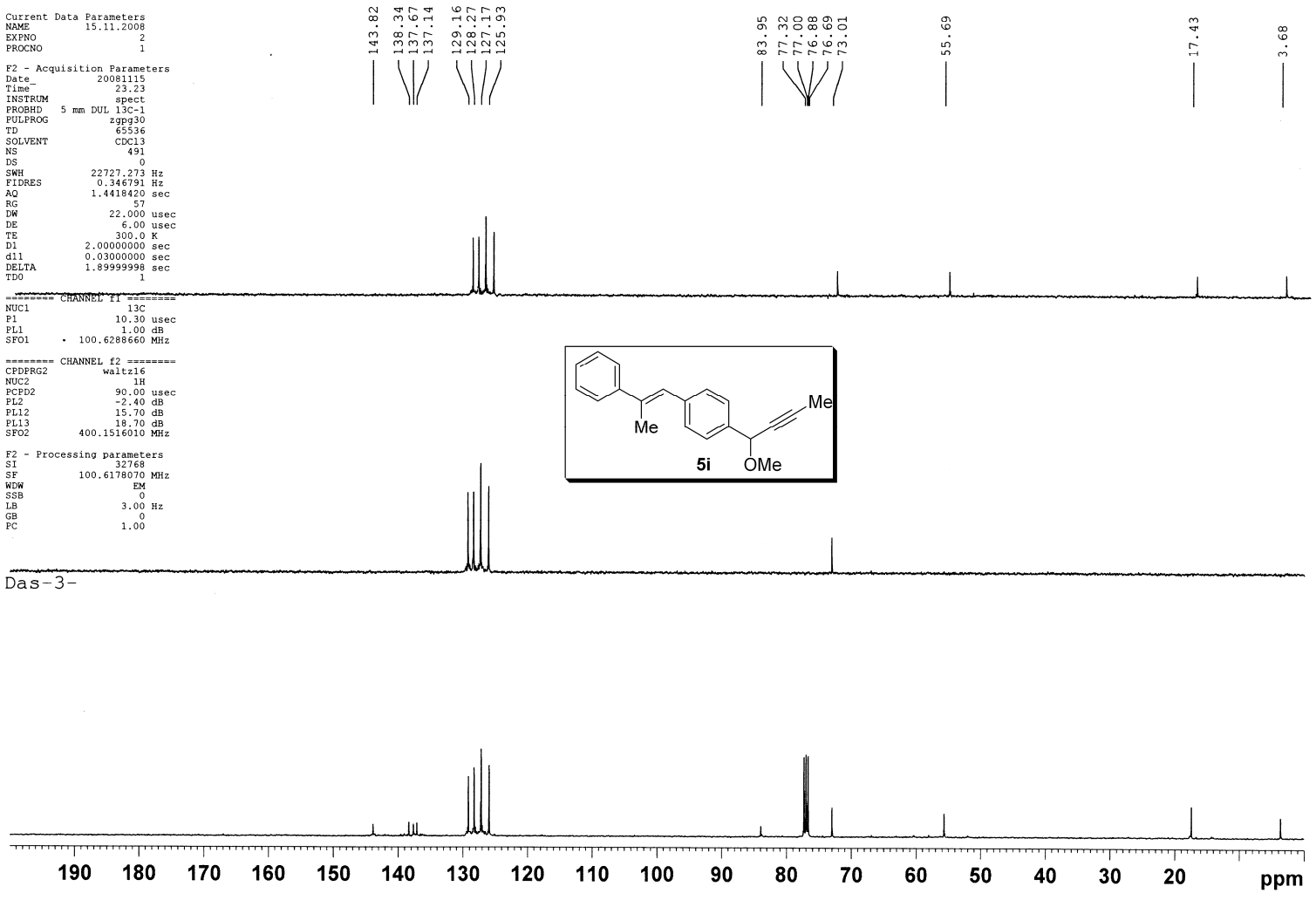


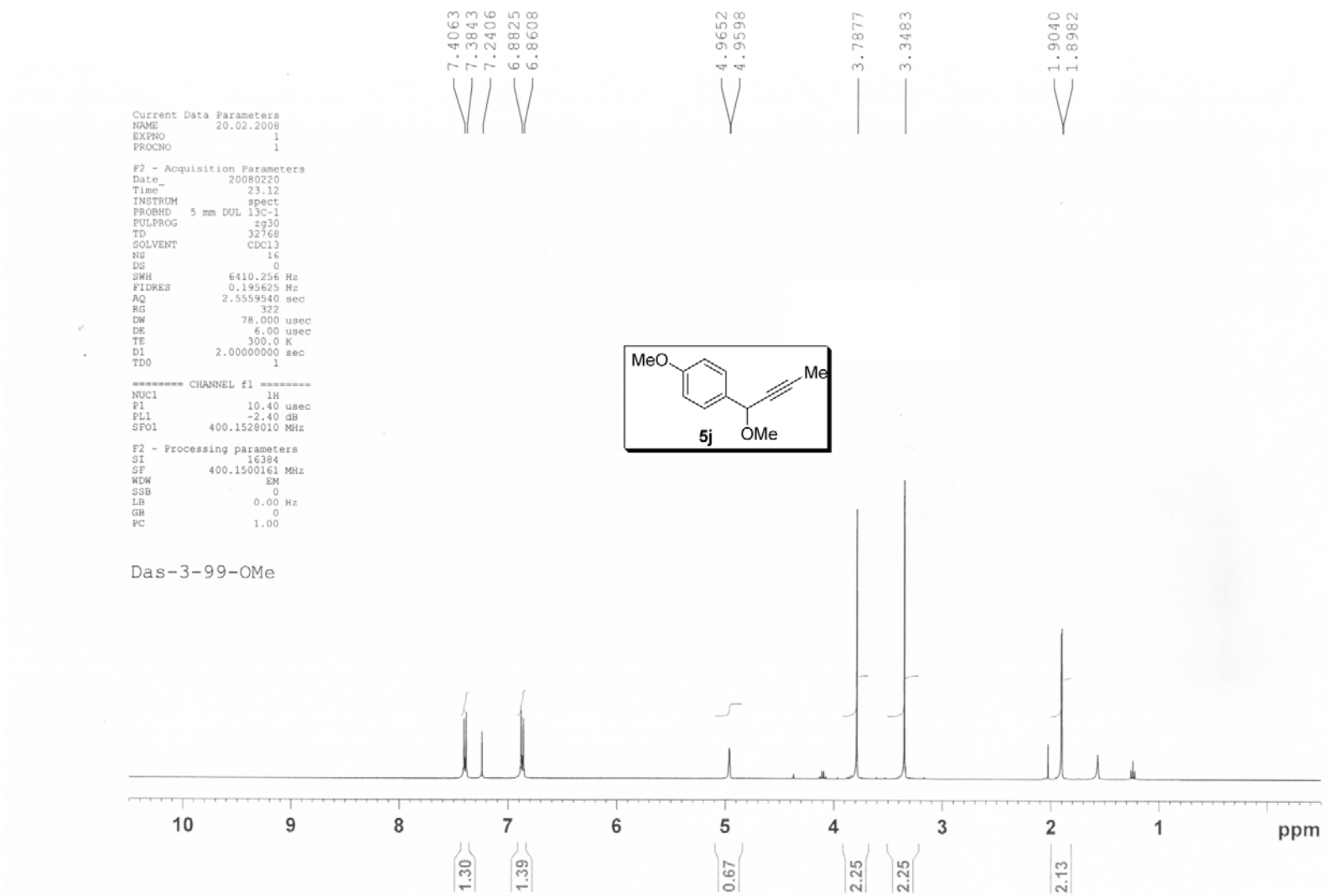


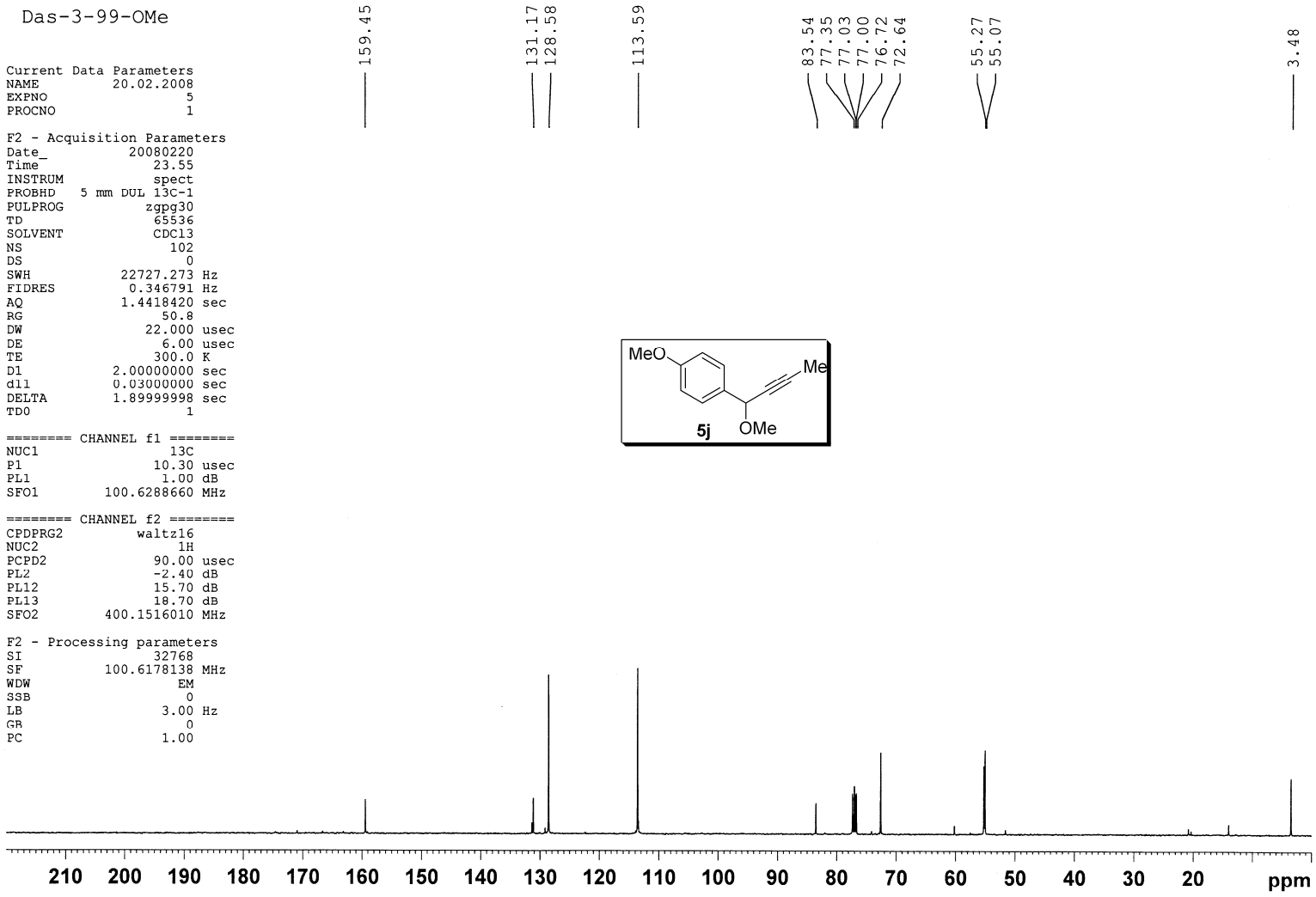

13C OBSERVE
exp7 std1h
SAMPLE
date Sep 17 2008
solvent CDCl3
file exp
ACQUISITION
sfrq 400.446
tn H1
at 2.502
np 30016
sw 5998.8
fb 3400
bs 4
tpwr 52
pw 7.8
d1 1.000
tof 0
nt 100
ct 28
alock n
gain 4
FLAGS
il n
in n
dp y
DISPLAY
sp -200.4
wp 4204.5
vs 50
sc 0
wc 250
hzmm 16.82
is 47.32
rfl 3870.3
rfp 2899.2
th 4
ins 100.000
nm ph
DEC. & VT
dfrq 400.446
dn H1
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
PROCESSING
lb 1.00
wtfile
proc ft
fn 65536
werr
wexp
wbs wft
wnt
7.417
7.397
7.190
7.170
5.012
5.008
3.393
2.632
2.613
2.594
1.916
1.911
1.619
1.599
1.581
1.387
1.368
1.349
1.331
0.950
0.932
0.914
Me
Me
OMe
5h
9
8
7
6
5
4
3
2
1
0 ppm
8.67
10.11
3.69
14.56
11.24
13.54
11.37
11.09
15.72

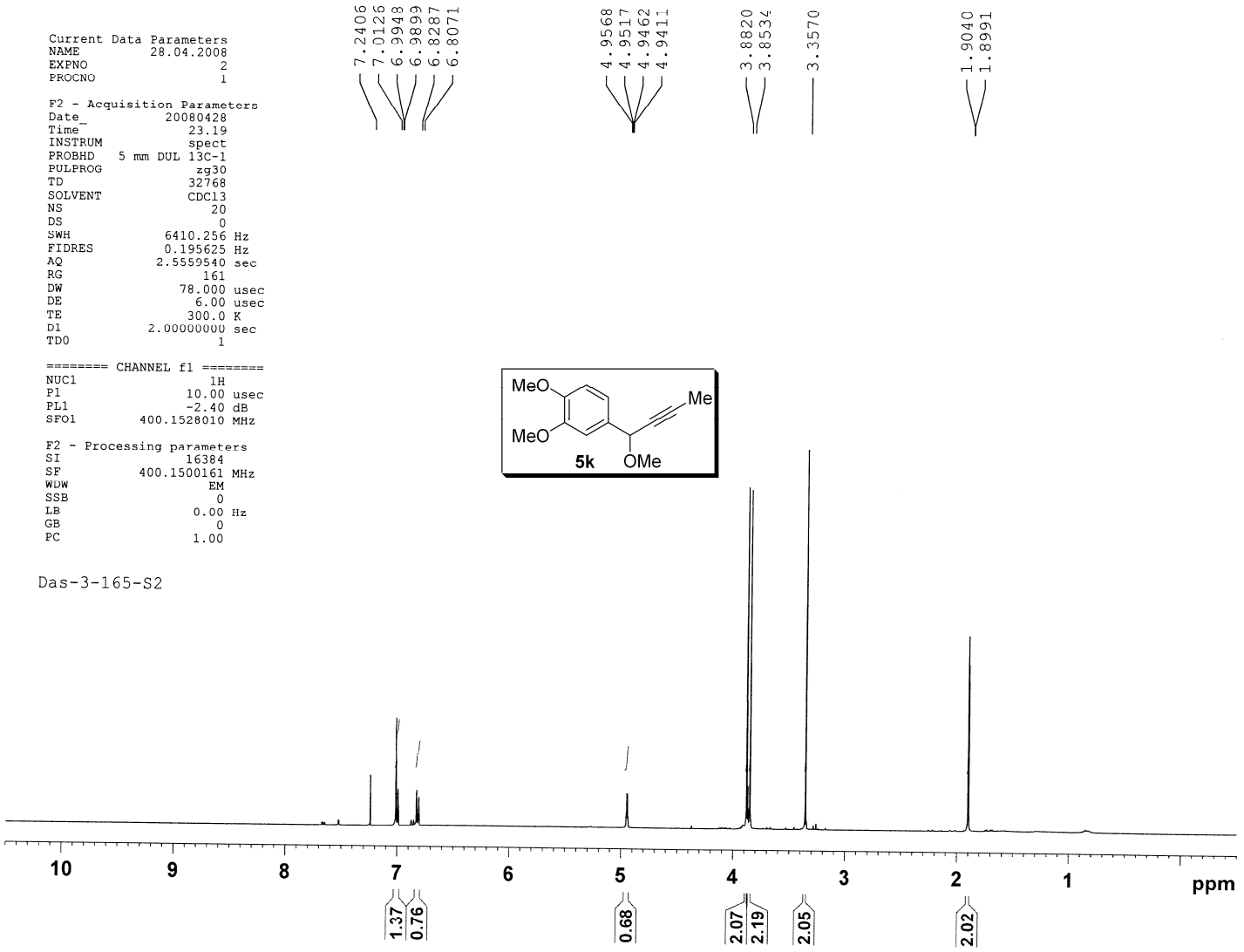


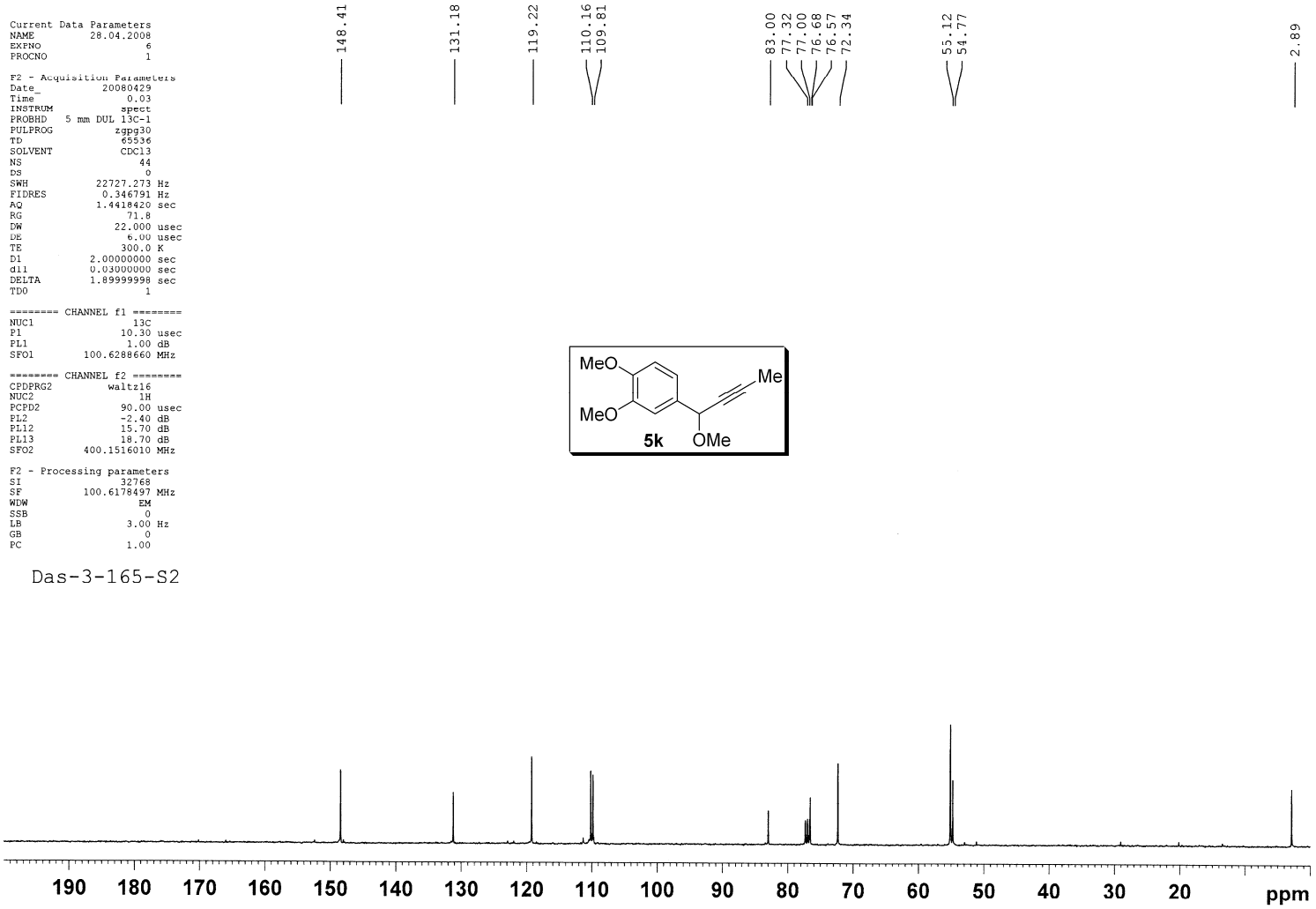


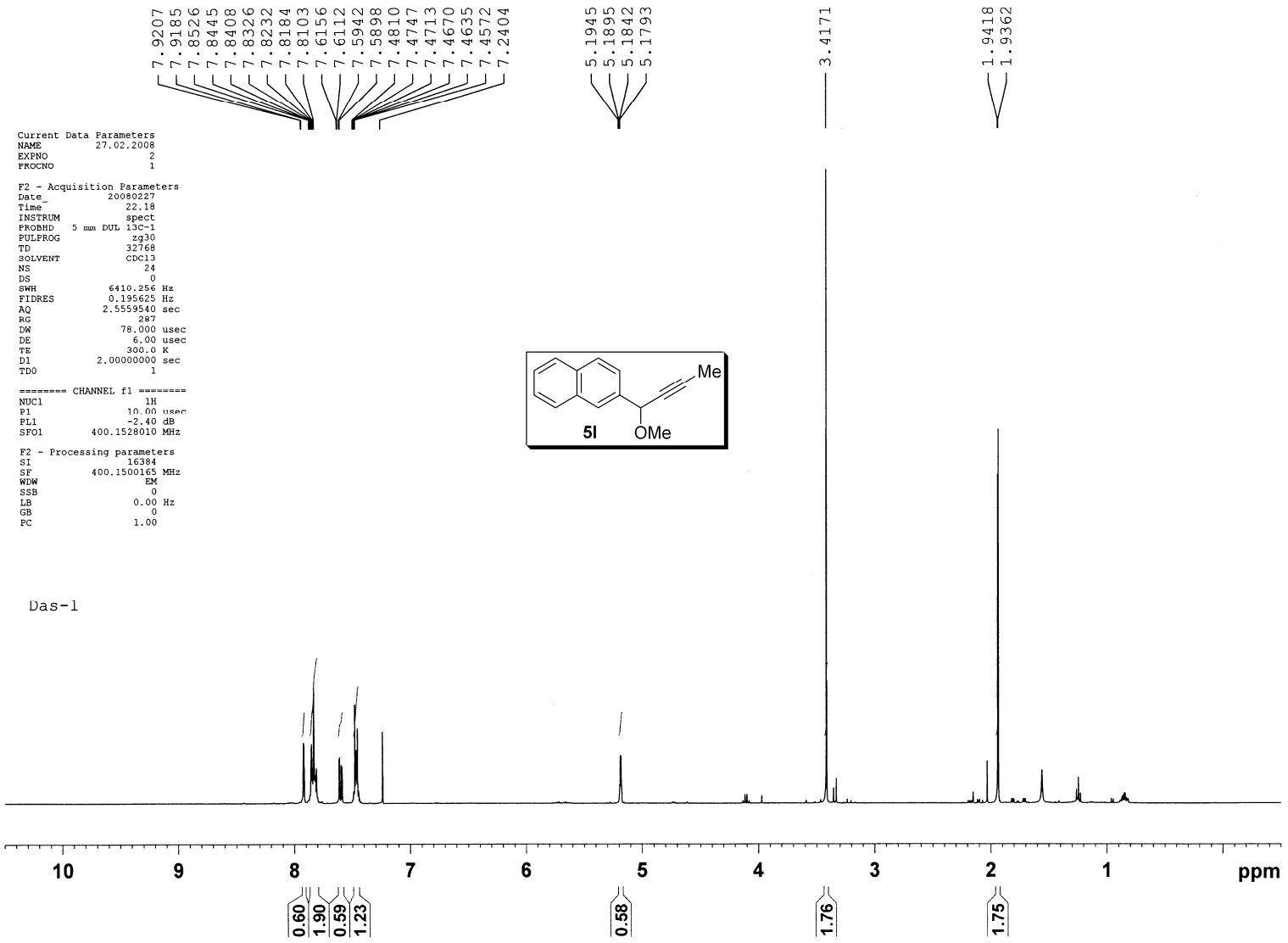


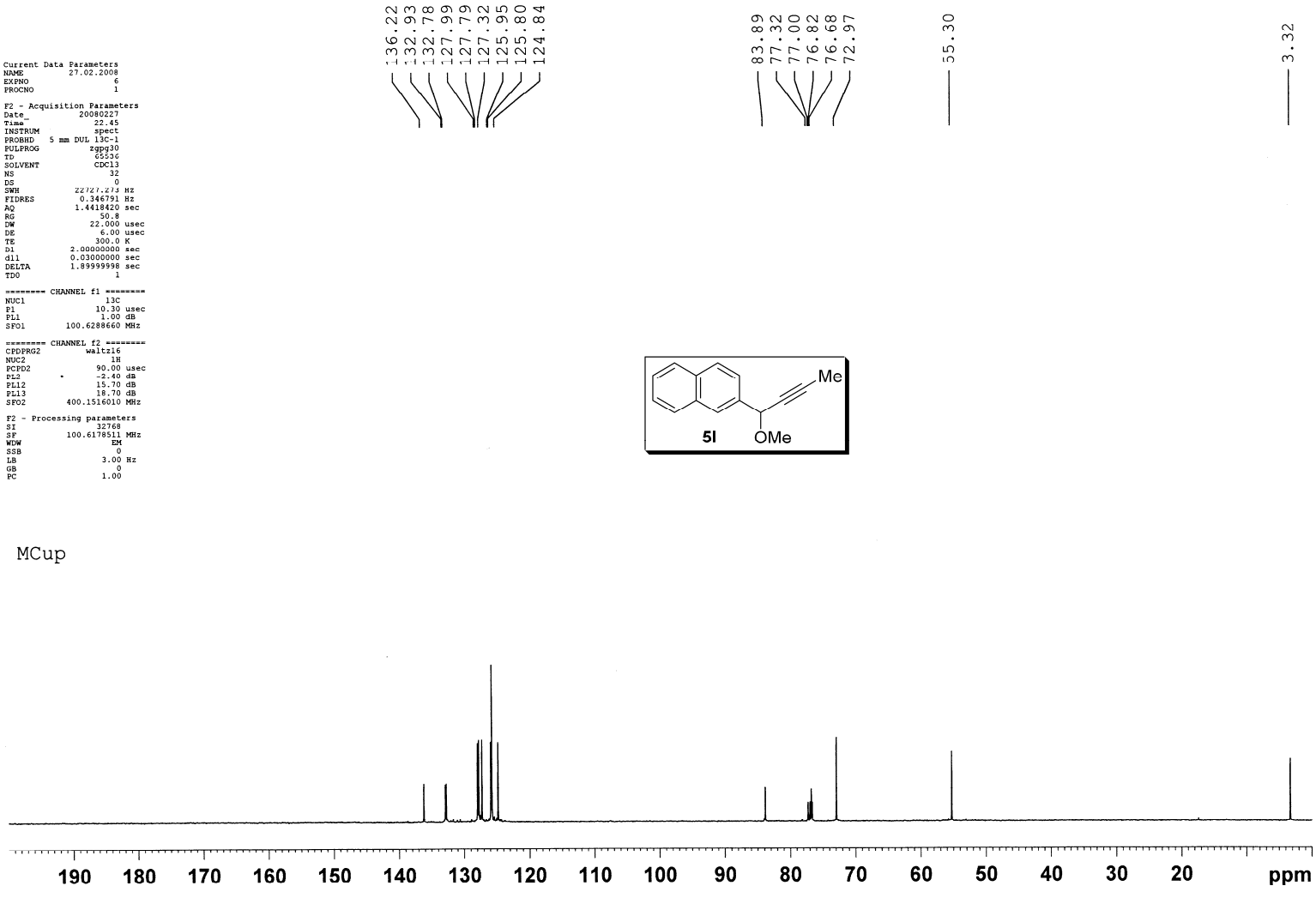

7.4063
7.3843
7.2406
6.8825
6.8608
4.9652
4.9598
3.7877
3.3483
1.9040
1.8982
Current Data Parameters
NAME 20.02.2008
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20080220
Time 23.12
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 6410.256 Hz
FIDRES 0.195625 Hz
AQ 2.5559540 sec
RG 322
DW 78.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 10.40 usec
PL1 -2.40 dB
SFO1 400.1528010 MHz
F2 - Processing parameters
SI 16384
SF 400.1500161 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
Das-3-99-OMe
MeO
Me
OMe
5j
10
9
8
7
6
5
4
3
2
1
ppm
1.30
1.39
0.67
2.25
2.25
2.13

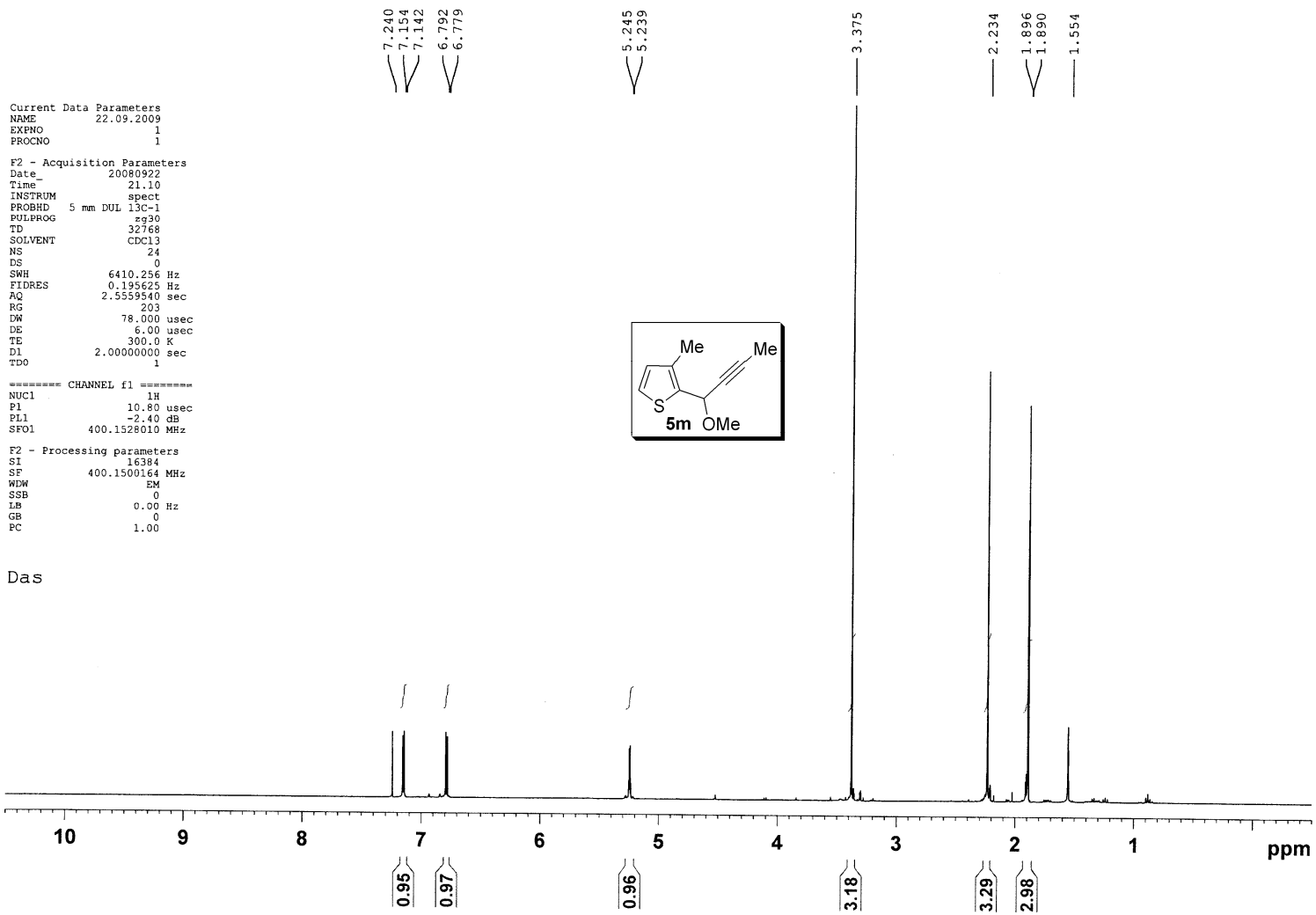


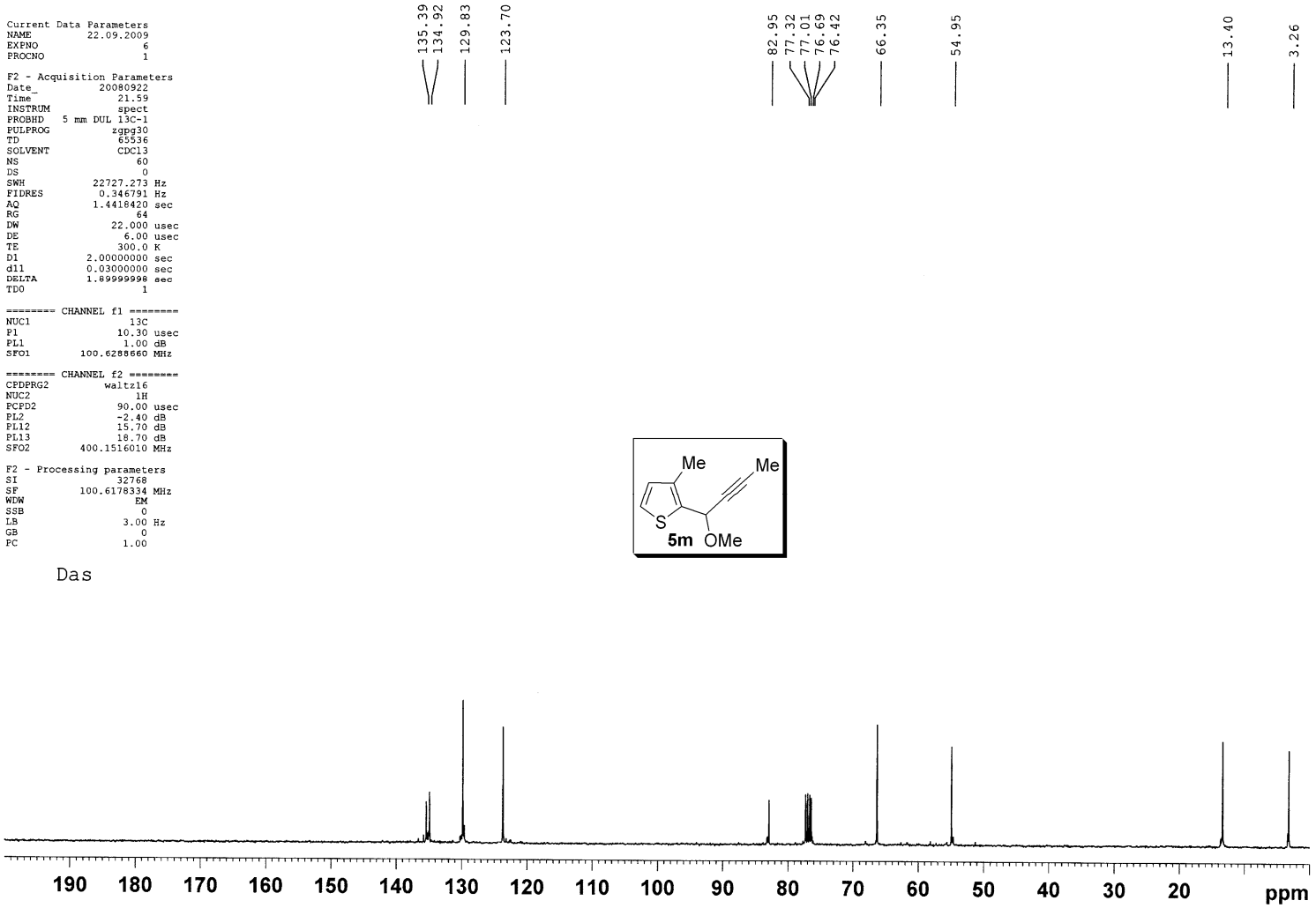


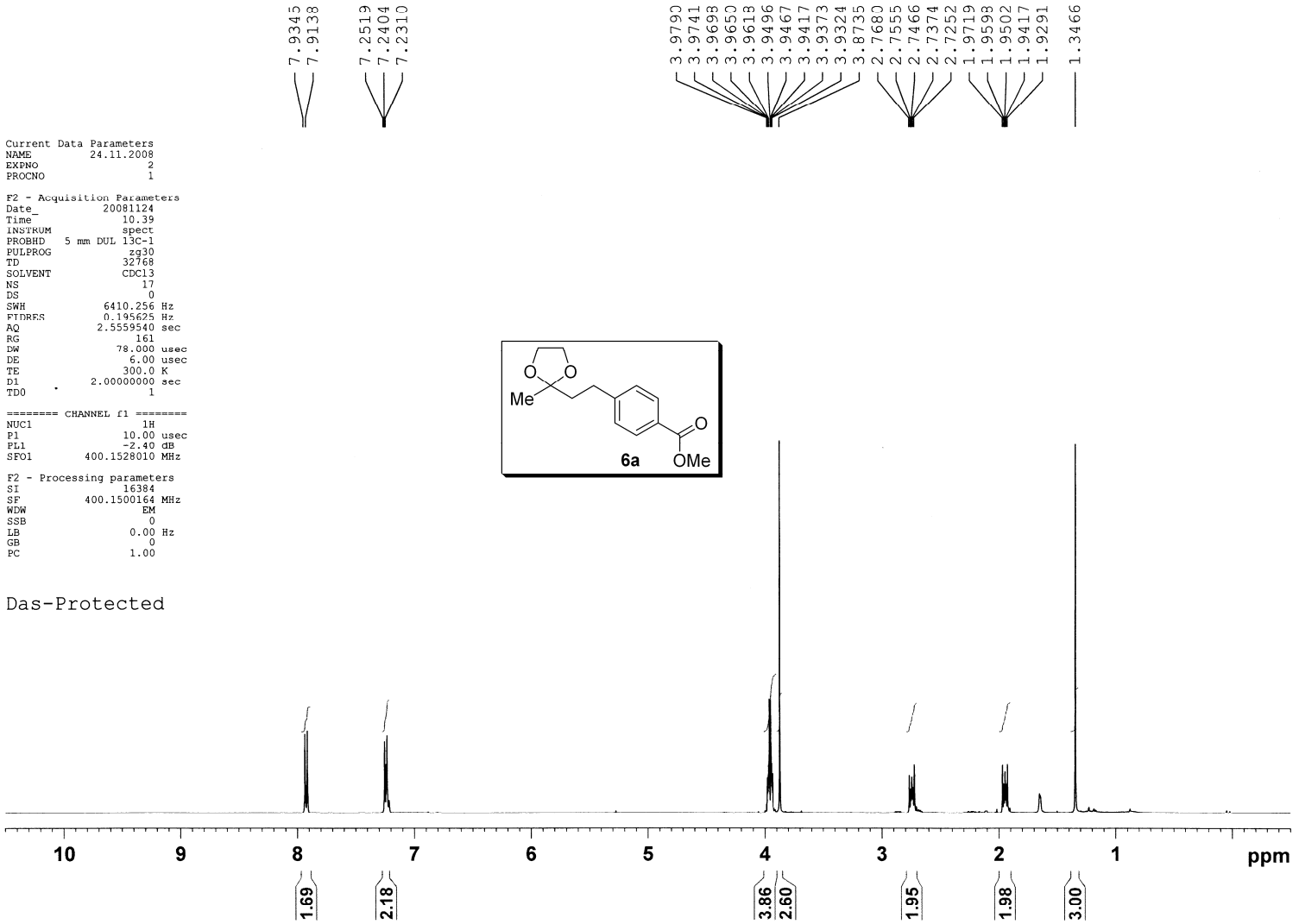

Current Data Parameters
NAME 28.04.2008
EXPNO 6
PROCNO 1
F2 - Acquisition Parameters
Date_ 20080429
Time 0.03
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 44
DS 0
SWH 22727.273 Hz
FIDRES 0.346791 Hz
AQ 1.4418420 sec
RG 71.8
DW 22.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 10.30 usec
PL1 1.00 dB
SFO1 100.6288660 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -2.40 dB
PL12 15.70 dB
PL13 18.70 dB
SFO2 400.1516010 MHz
F2 - Processing parameters
SI 32768
SF 100.6178497 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.00
Das-3-165-S2
148.41
131.18
119.22
110.16
109.81
83.00
77.32
77.00
76.68
76.57
72.34
55.12
54.77
2.89
MeO
MeO
Me
OMe
5k
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm

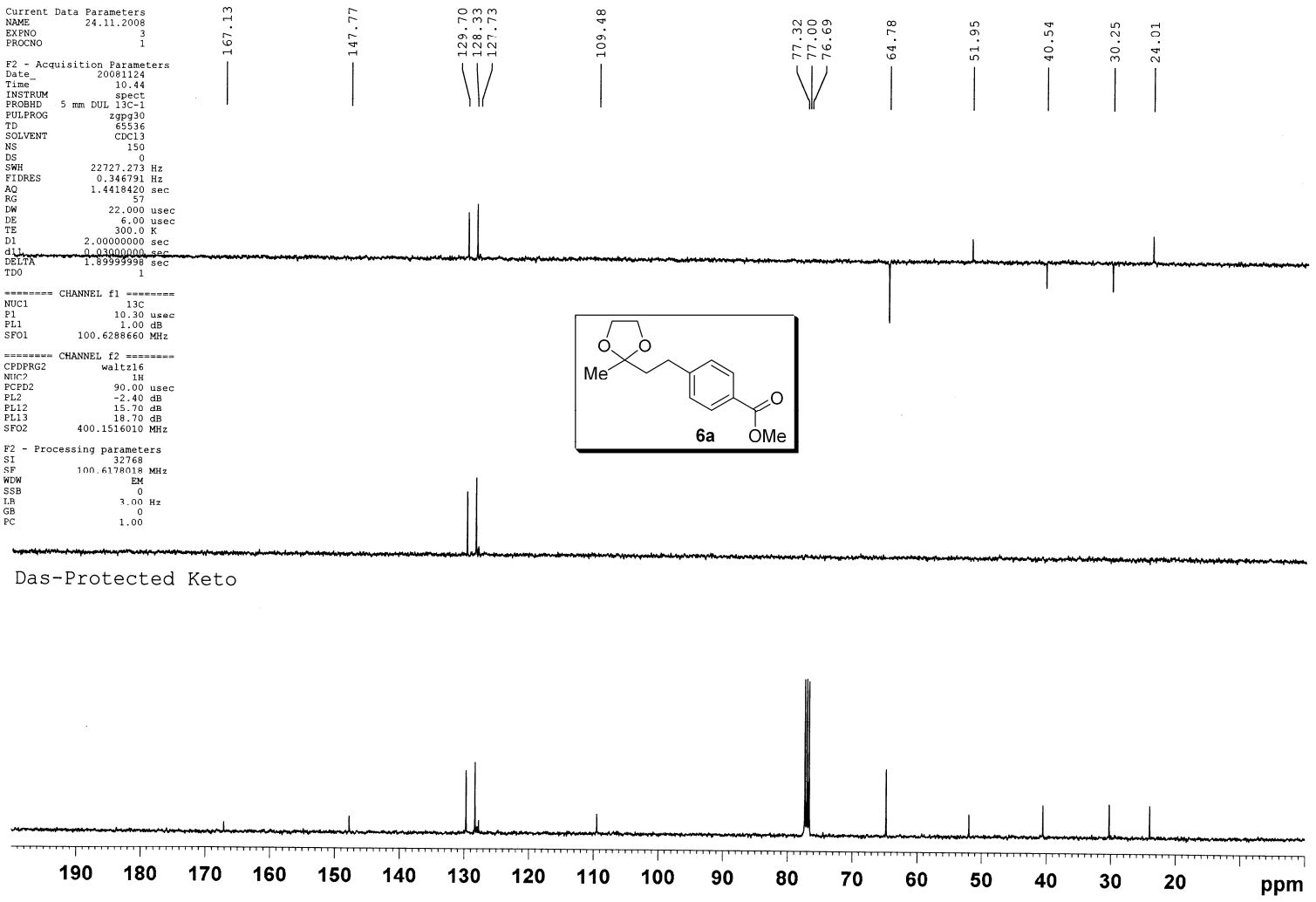












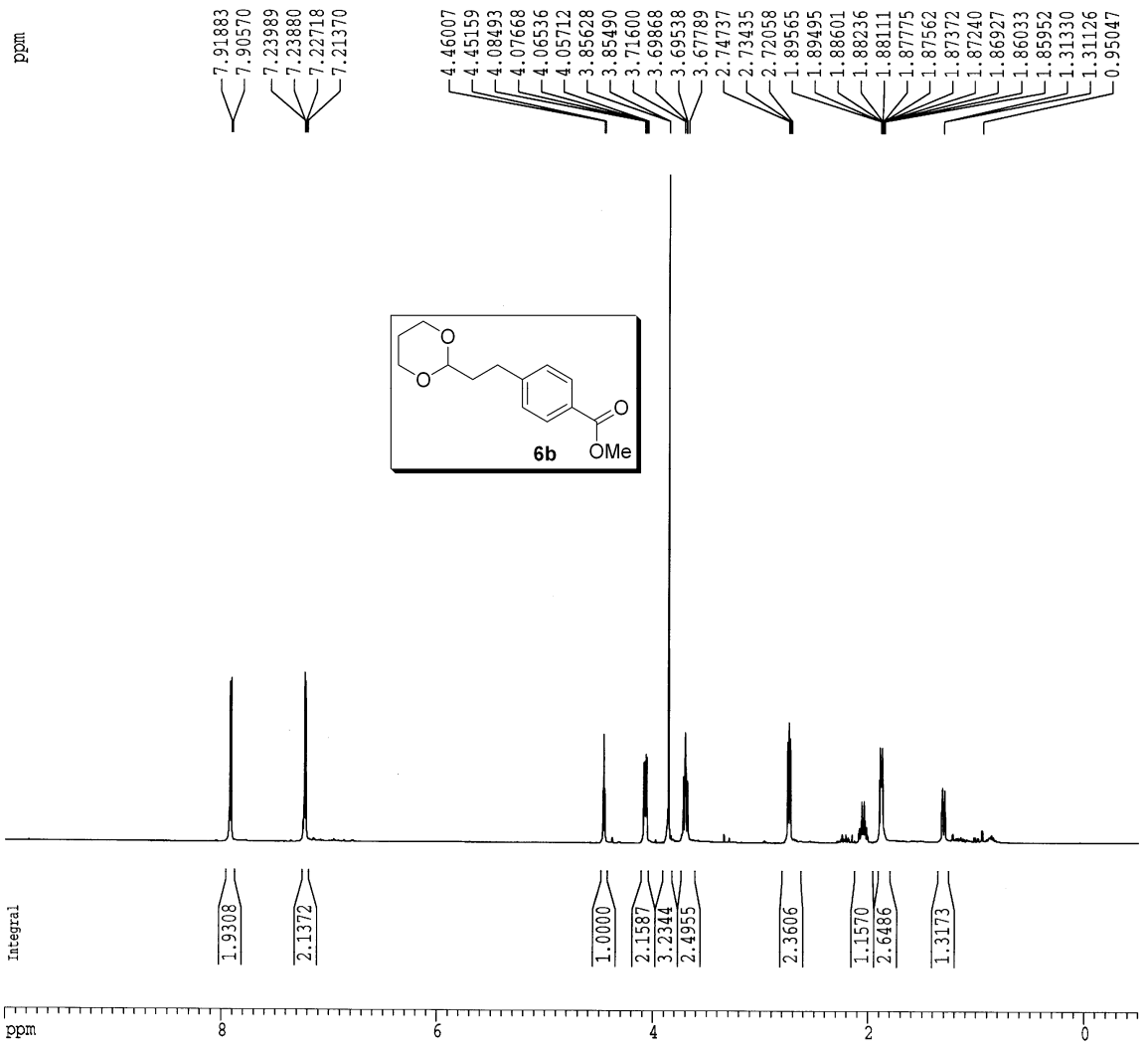
```
Current Data Parameters
NAME          Das-3-268
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20081109
Time              19.32
INSTRUM           spect
PROBHD   5 mm QNP  1H/1
PULPROG             zg
TD                32768
SOLVENT           CDCl3
NS                   16
DS                    0
SWH            8389.262 Hz
FIDRES         0.256020 Hz
AQ             1.9530228 sec
RG                  256
DW               59.600 usec
DE                6.50 usec
TE               298.4 K
D1            1.50000000 sec
MCREST        0.00000000 sec
MCWRK         0.01500000 sec

======== CHANNEL f1 ========
NUC1                 1H
P1                10.00 usec
PL1                3.00 dB
SFO1        599.2029960 MHz

F2 - Processing parameters
SI                32768
SF          599.2000247 MHz
WDW                  no
SSB                   0
LB                 0.00 Hz
GB                    0
PC                 0.50

1D NMR plot parameters
CX                20.00 cm
CY                12.00 cm
F1P               10.000 ppm
F1              5992.00 Hz
F2P               -0.500 ppm
F2              -299.60 Hz
PPMCM          0.52500 ppm/cm
HZCM         314.58002 Hz/cm
```



```
Current Data Parameters
NAME          Das-3-268
EXPNO                 2
PROCNO                1

F2 - Acquisition Parameters
Date_          20081109
Time              19.37
INSTRUM           spect
PROBHD   5 mm QNP  1H/1
PULPROG           zgpg
TD                32768
SOLVENT           CD2Cl2
NS                   77
DS                    0
SWH           45045.047 Hz
FIDRES         1.374666 Hz
AQ            0.3637748 sec
RG                 1024
DW               11.100 usec
DE                6.50 usec
TE               298.3 K
D1           3.00000000 sec
d11          0.03000000 sec
DELTA        2.90000010 sec
MCREST       0.00000000 sec
MCWRK        0.01500000 sec

======== CHANNEL f1 ========
NUC1                 13C
P1                  4.50 usec
PL1                 0.00 dB
SFO1         150.6855336 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2              92.00 usec
PL2               120.00 dB
PL12               11.30 dB
PL13               14.00 dB
SFO2         599.2029960 MHz

F2 - Processing parameters
SI                32768
SF          150.6689750 MHz
WDW                  EM
SSB                   0
LB                 3.00 Hz
GB                    0
PC                 1.00

1D NMR plot parameters
CX                20.00 cm
CY                 4.50 cm
F1P              200.000 ppm
F1             30133.79 Hz
F2P              0.000 ppm
F2               0.00 Hz
PPMCM          10.00000 ppm/cm
HZCM         1506.68970 Hz/cm
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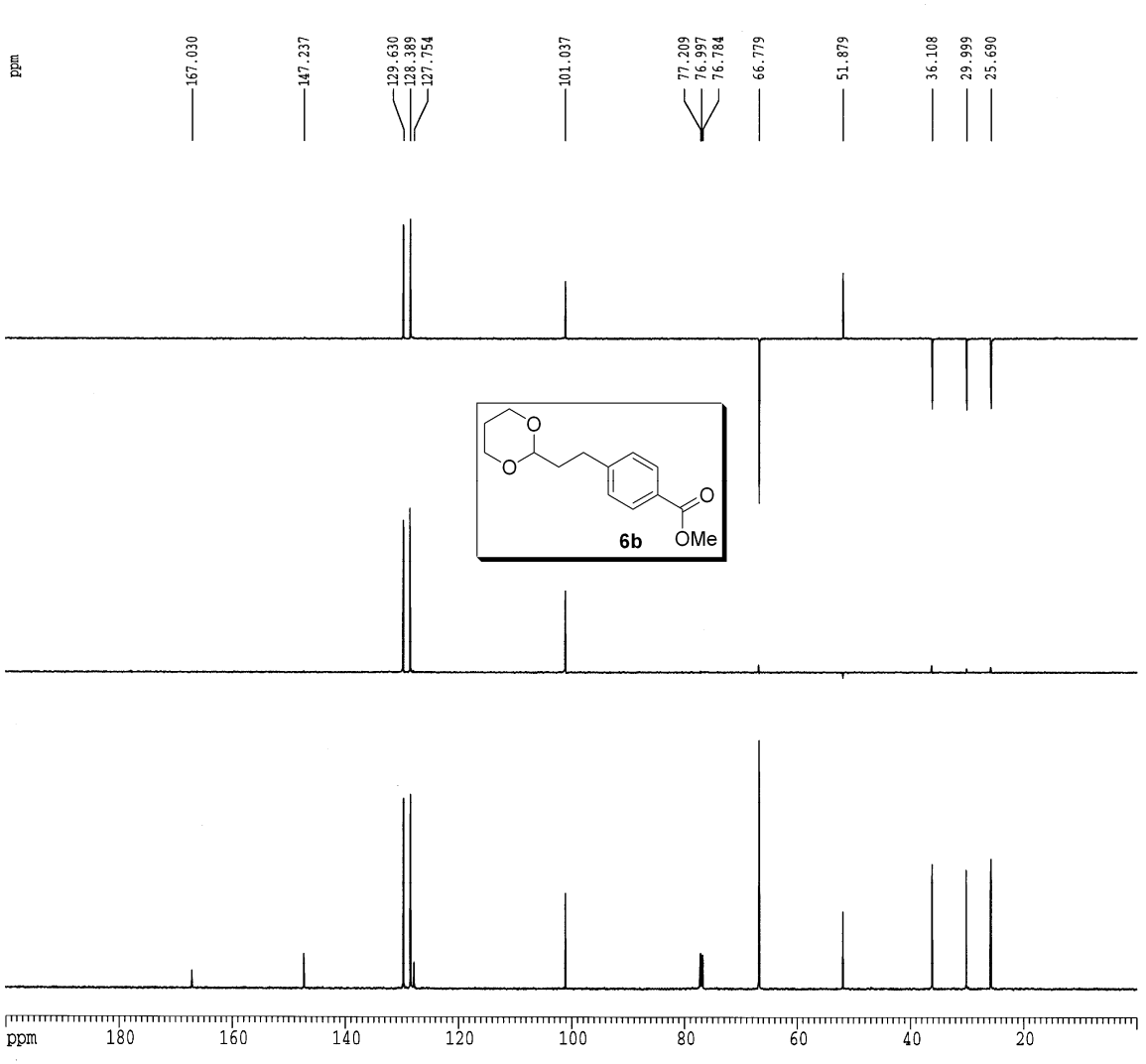
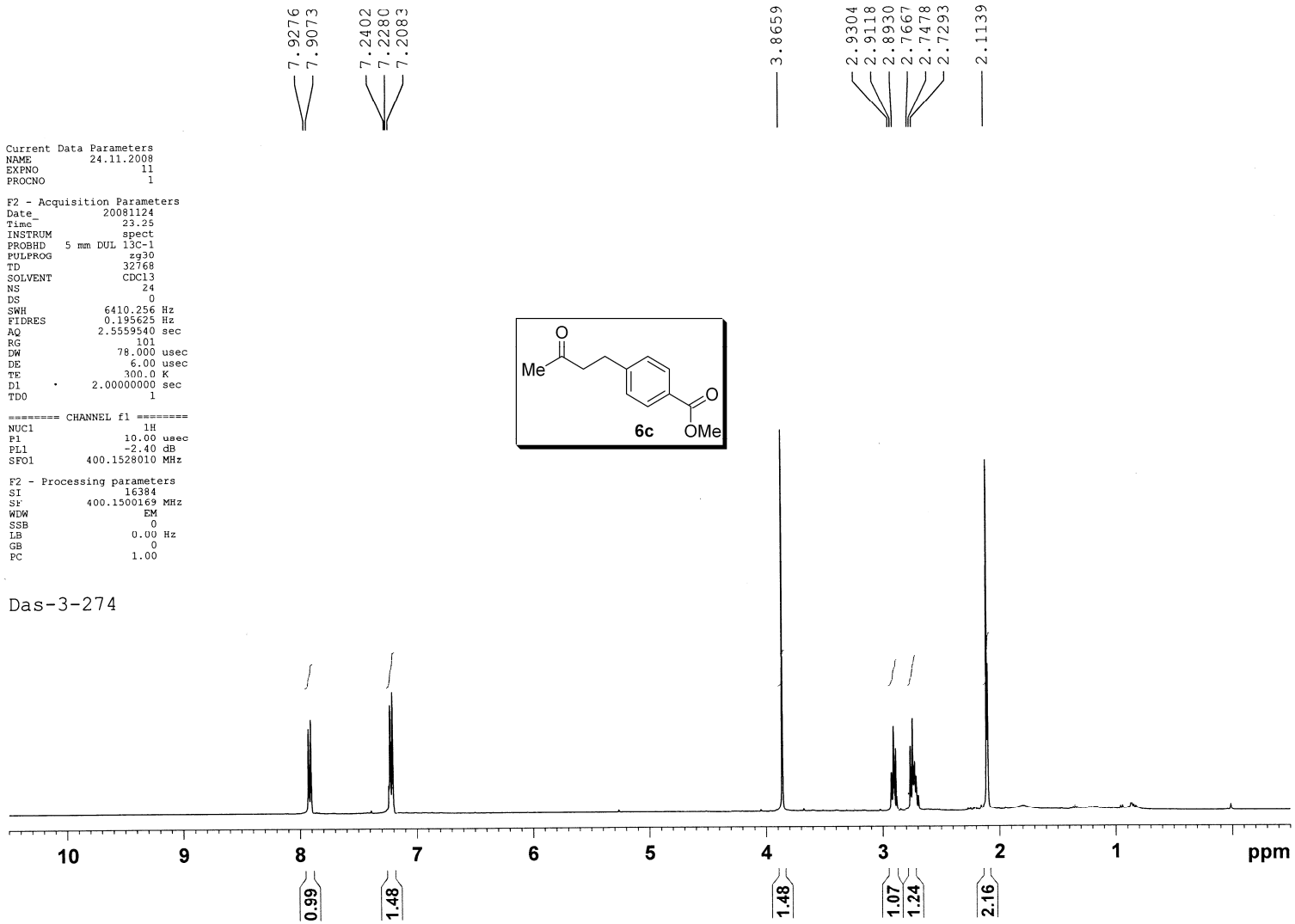
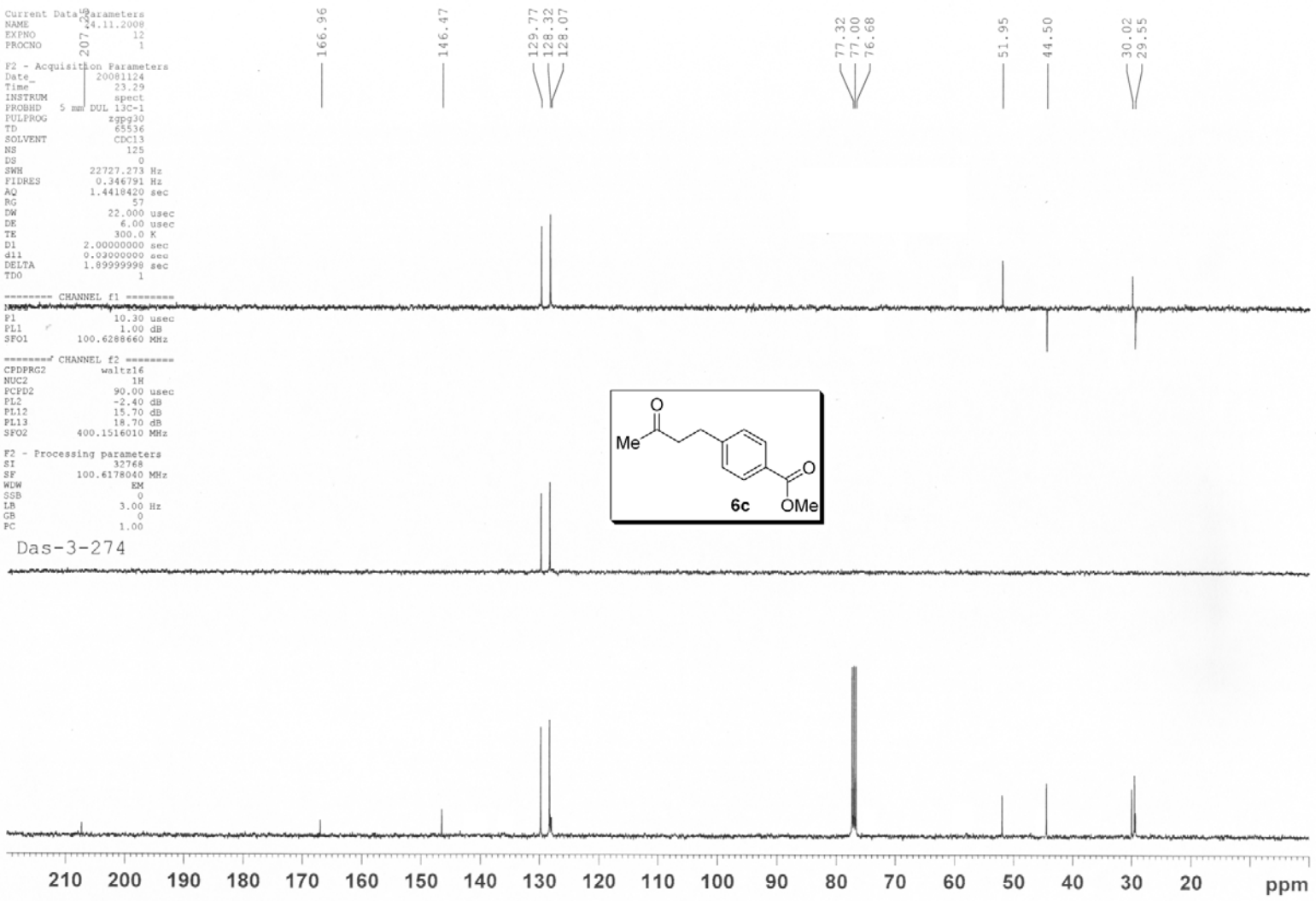
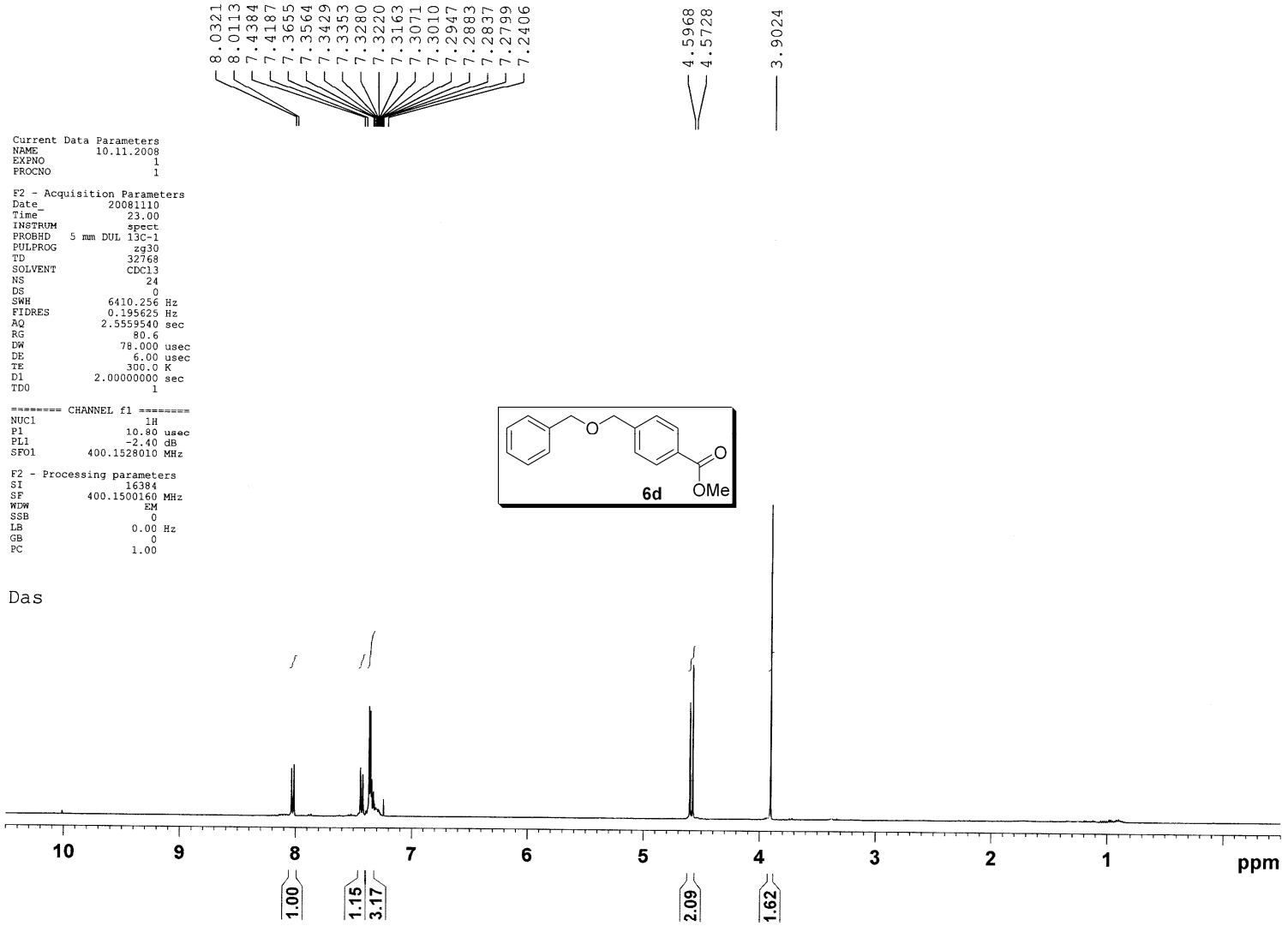
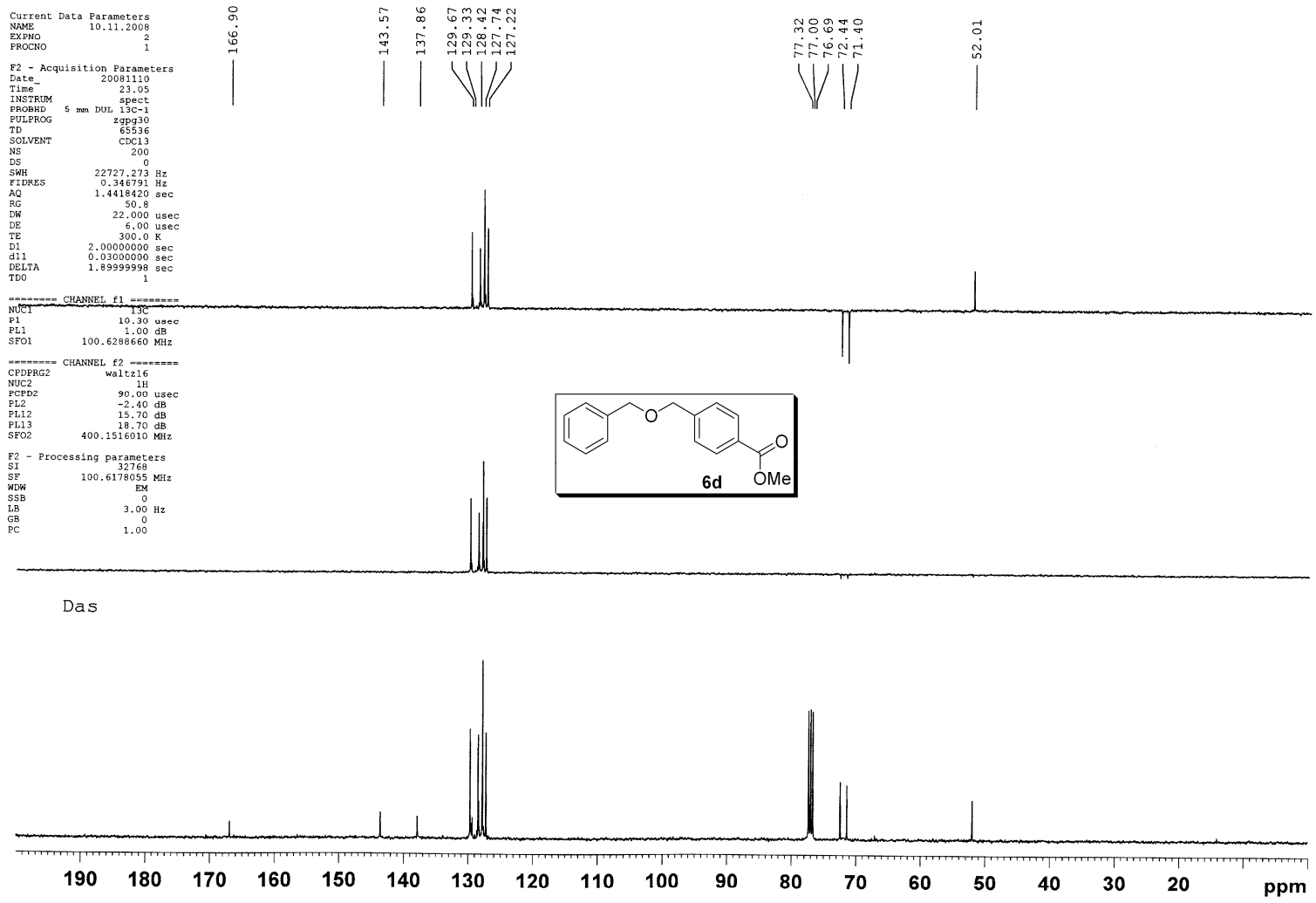
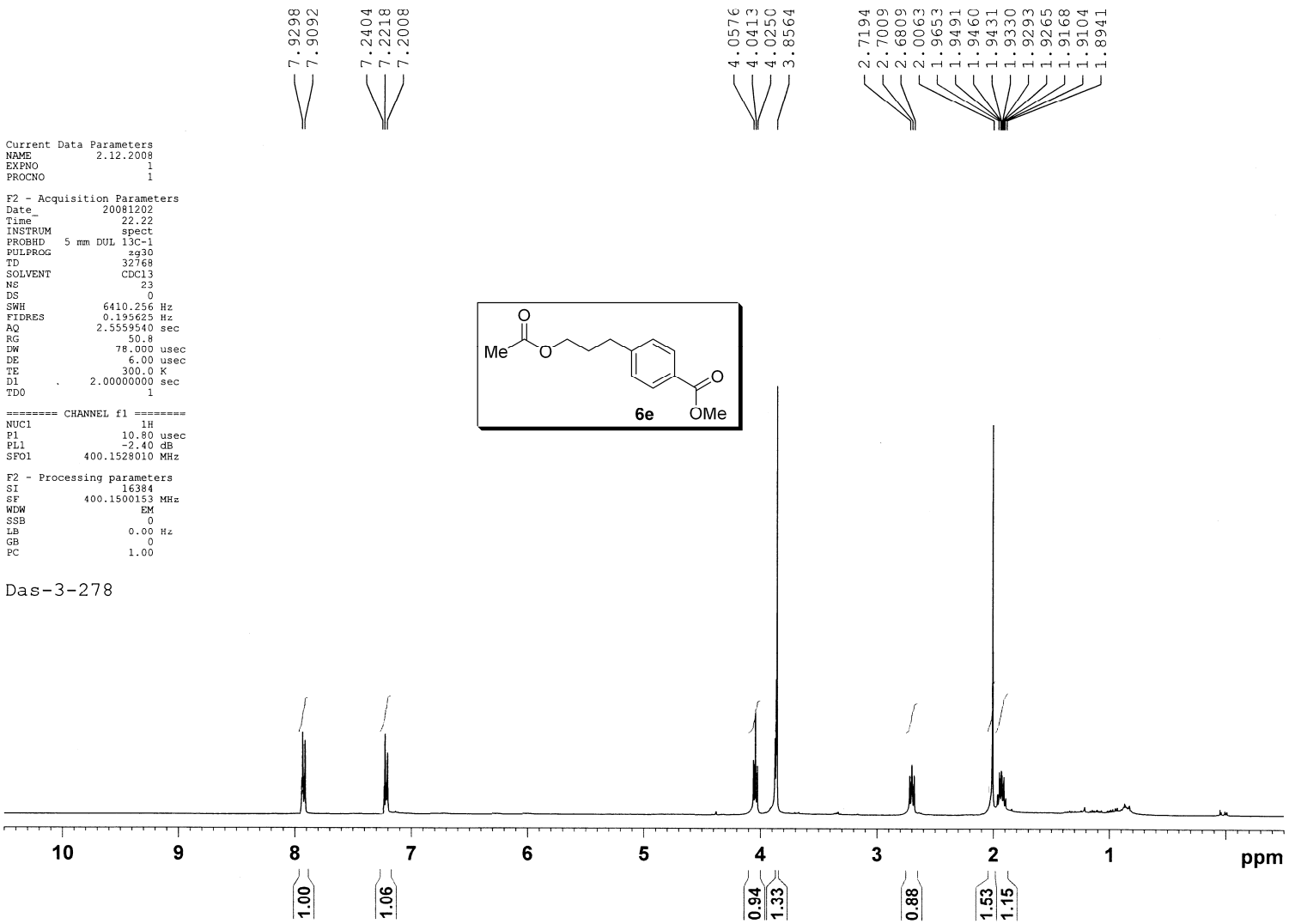


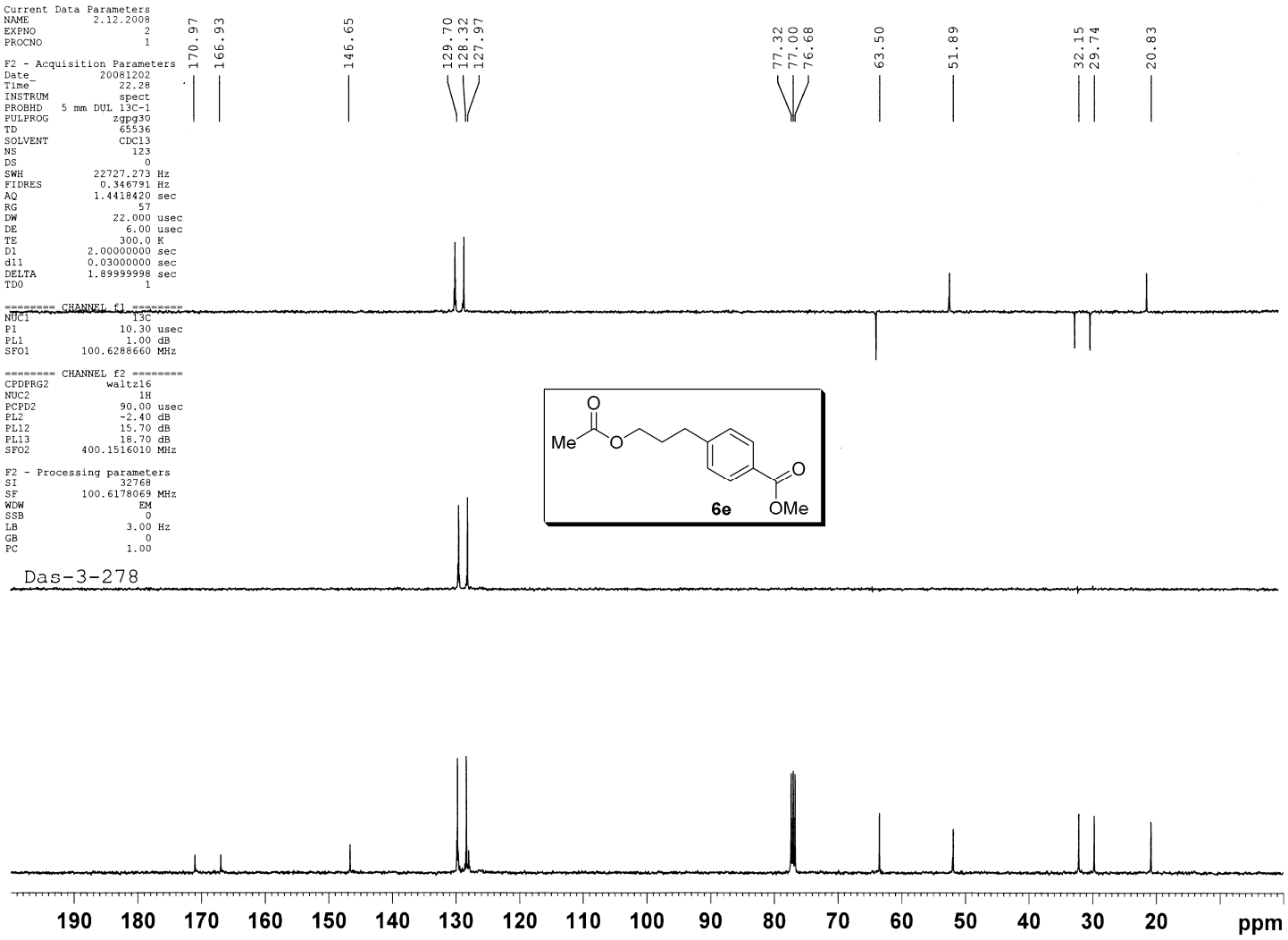


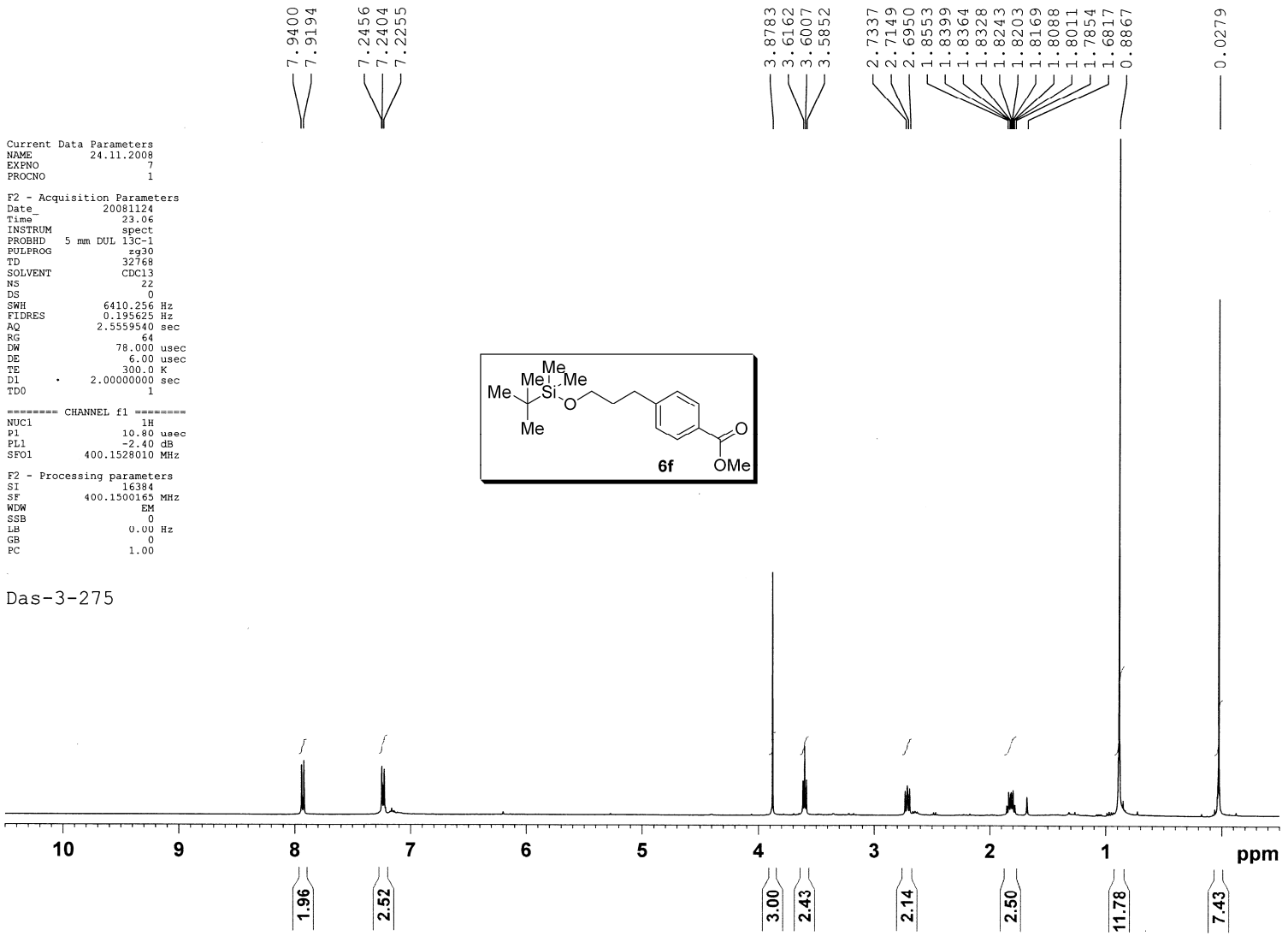


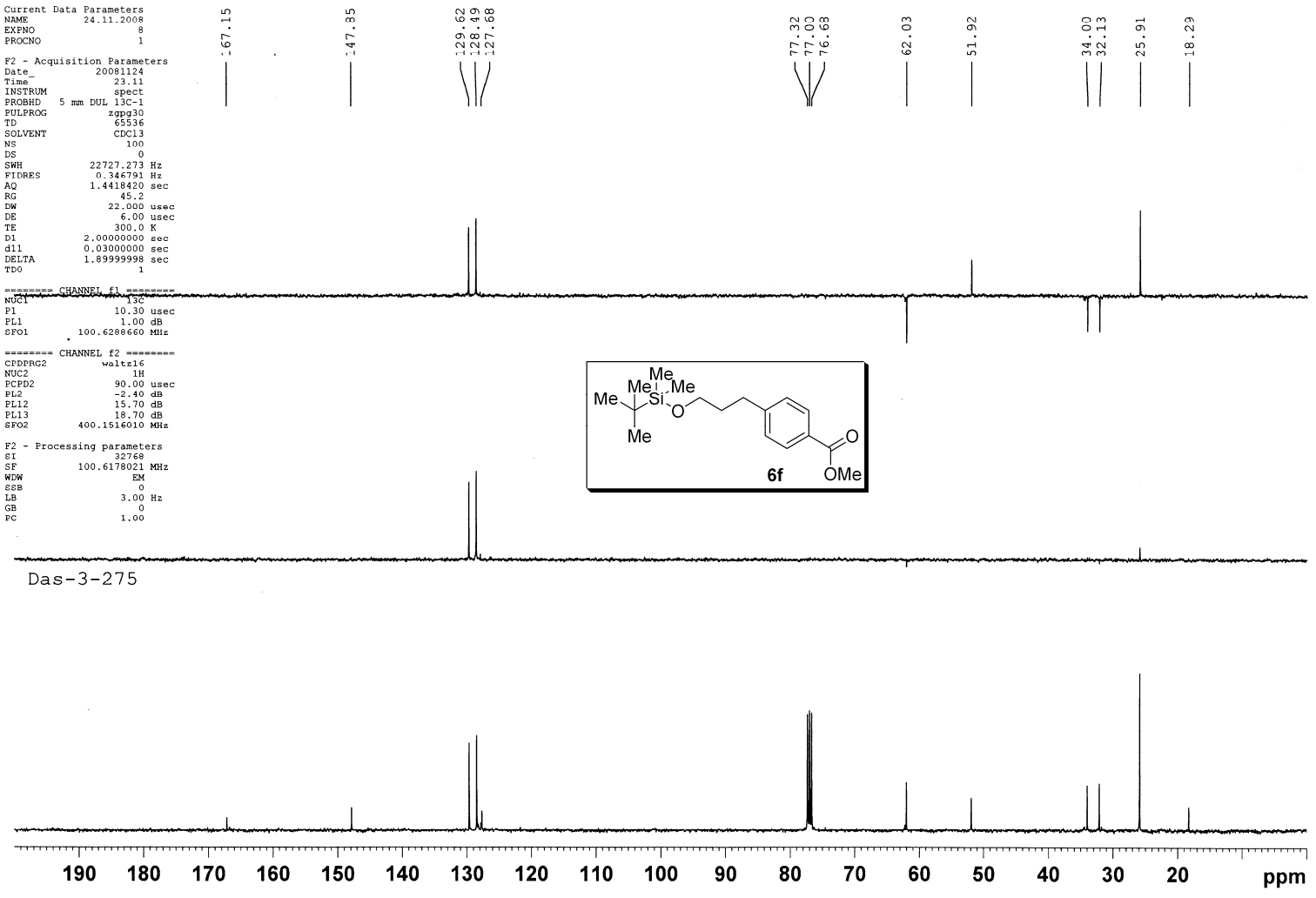


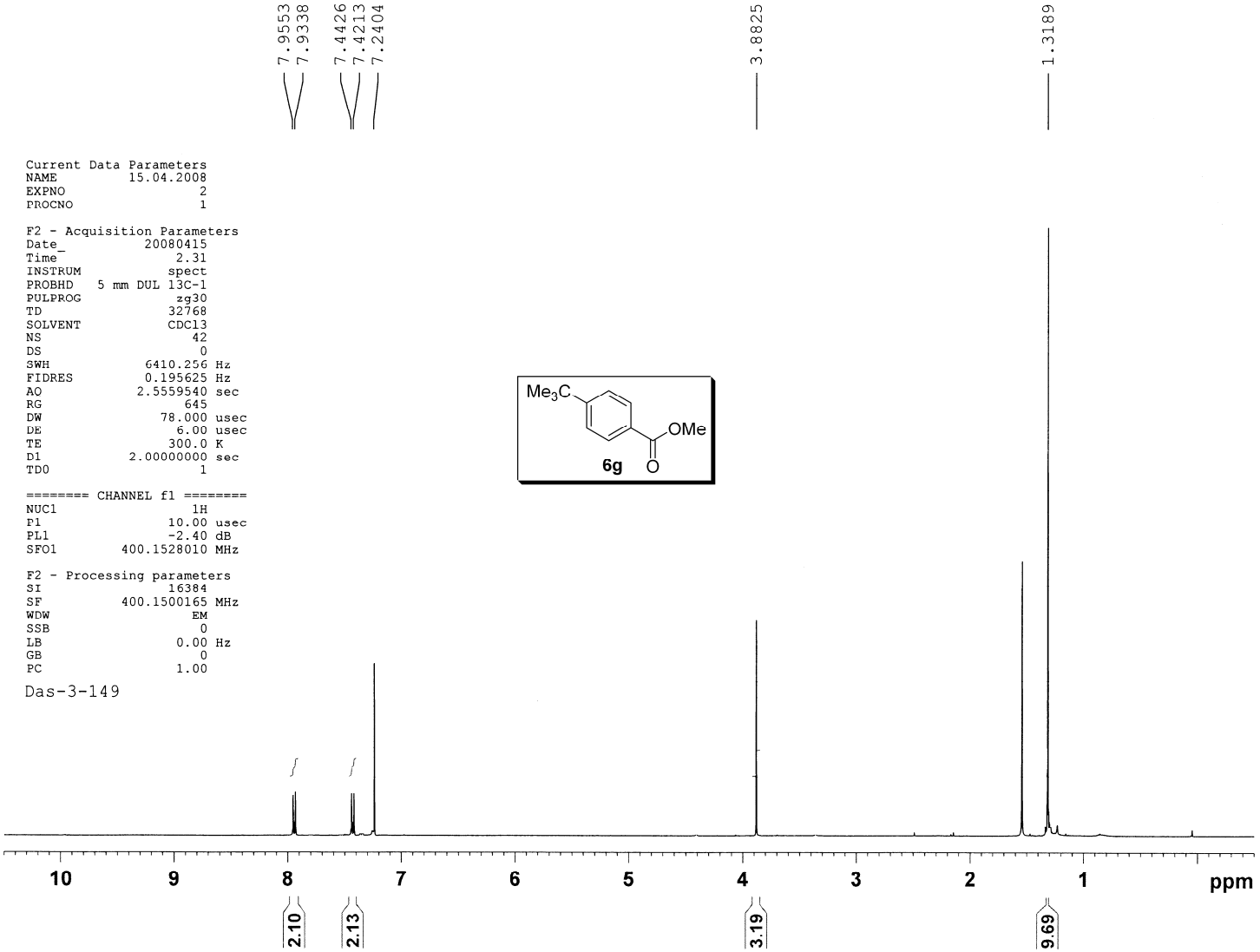


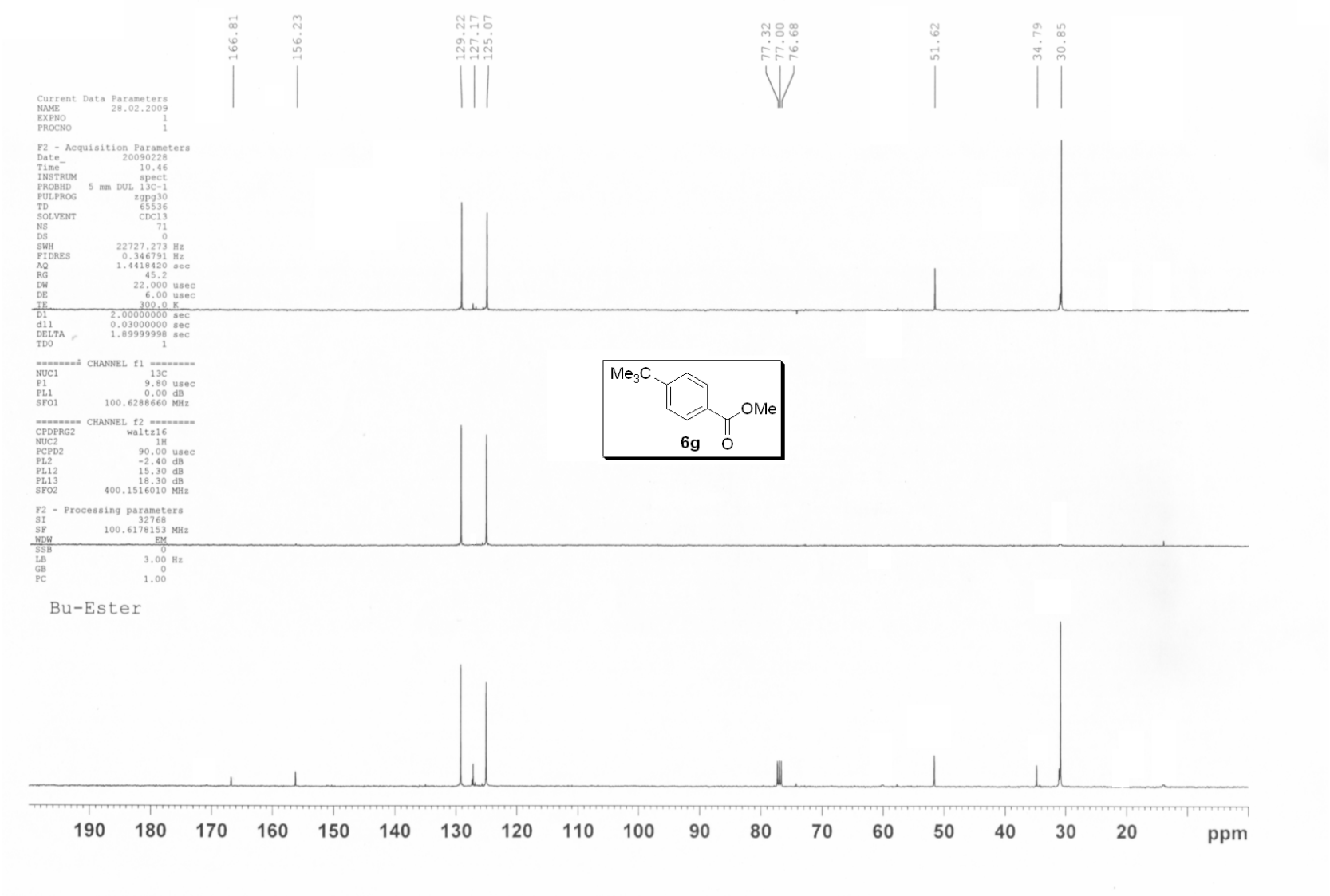


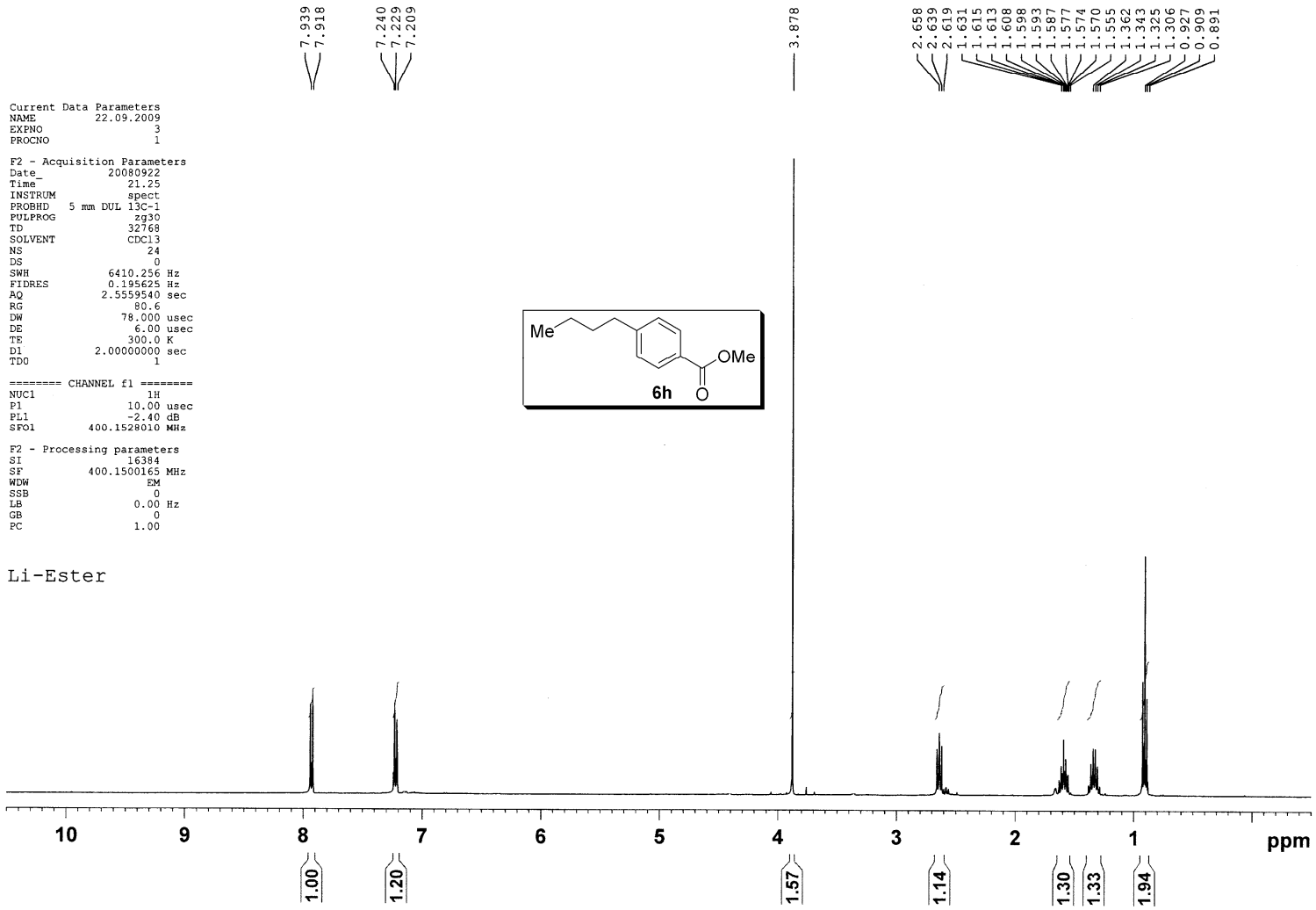

Current Data Parameters
NAME 2.12.2008
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20081202
Time 22.28
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 123
DS 0
SWH 22727.273 Hz
FIDRES 0.346791 Hz
AQ 1.4418420 sec
RG 57
DW 22.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 10.30 usec
PL1 1.00 dB
SFO1 100.6288660 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -2.40 dB
PL12 15.70 dB
PL13 18.70 dB
SFO2 400.1516010 MHz
F2 - Processing parameters
SI 32768
SF 100.6178069 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.00
170.97
166.93
146.65
129.70
128.32
127.97
77.32
77.00
76.68
63.50
51.89
32.15
29.74
20.83
Me
O
O
O
O
OMe
6e
Das-3-278
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm

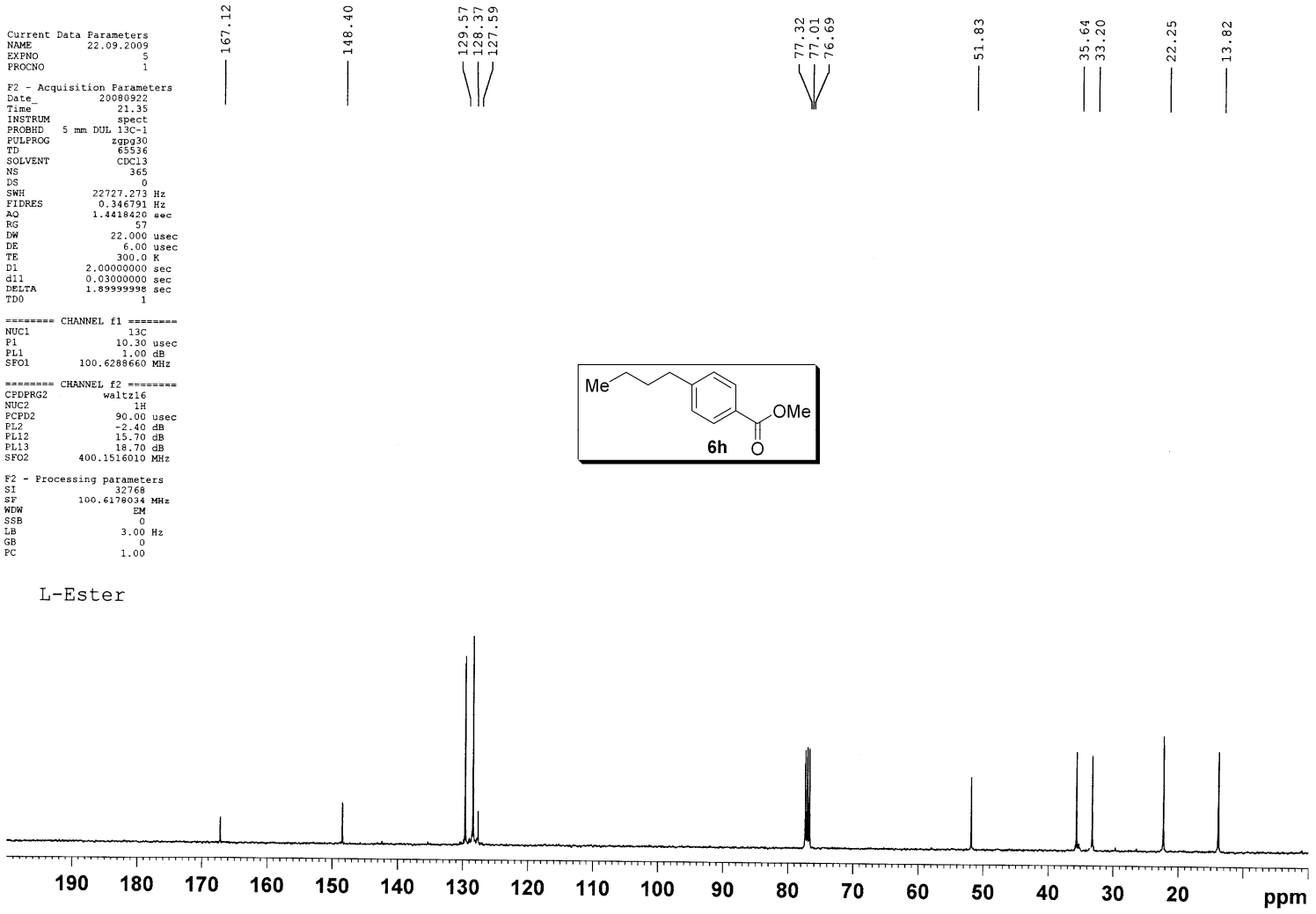


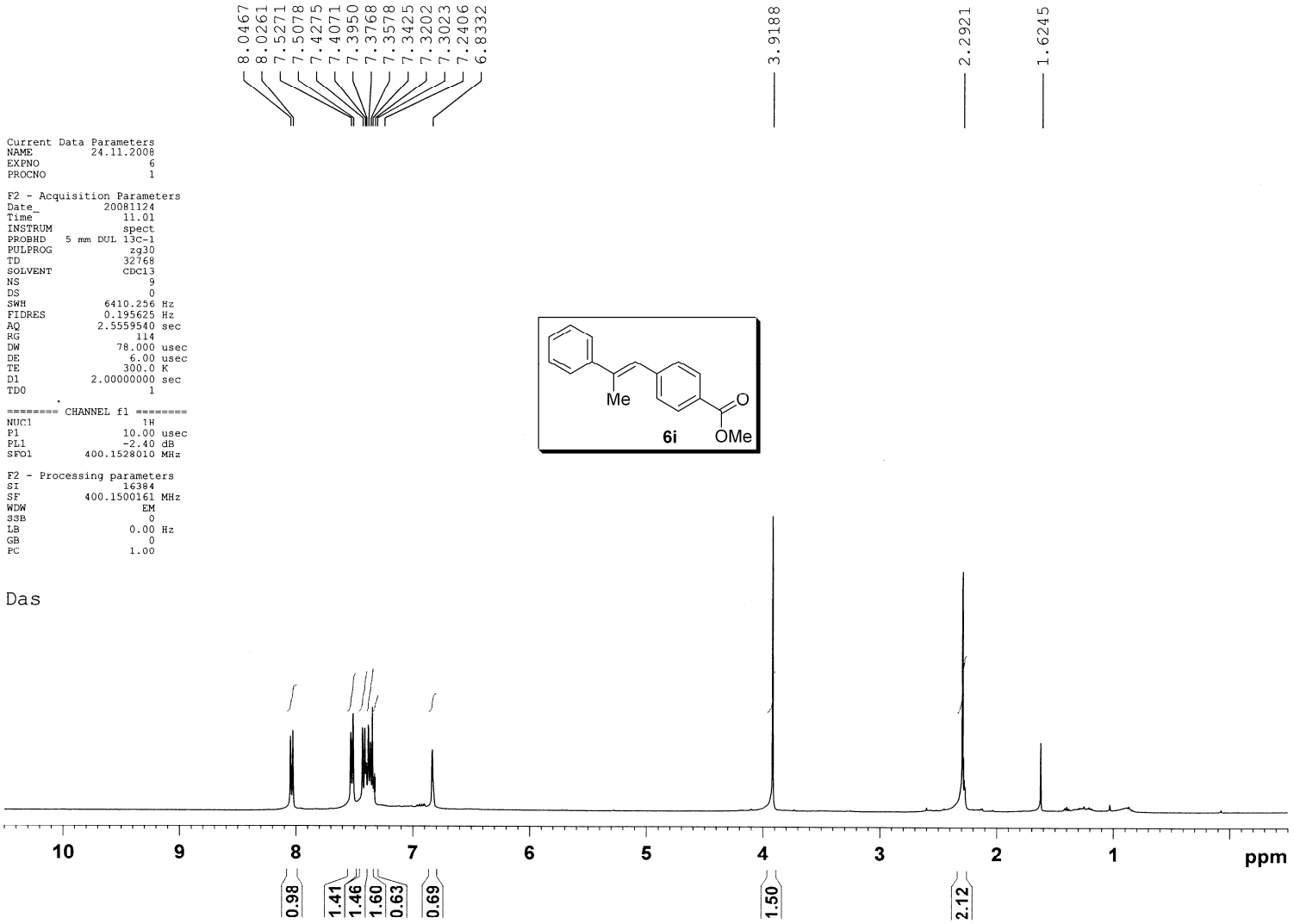


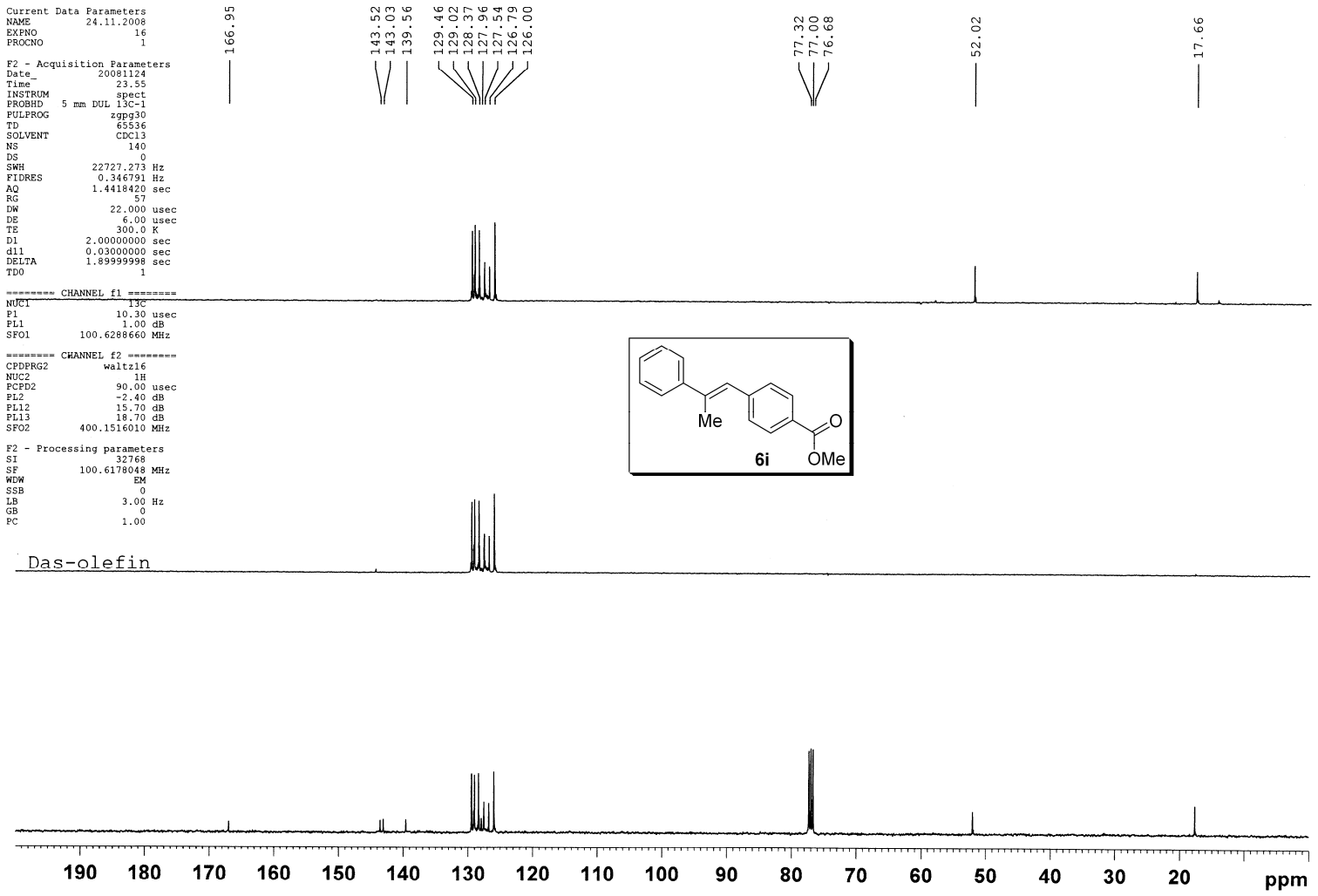


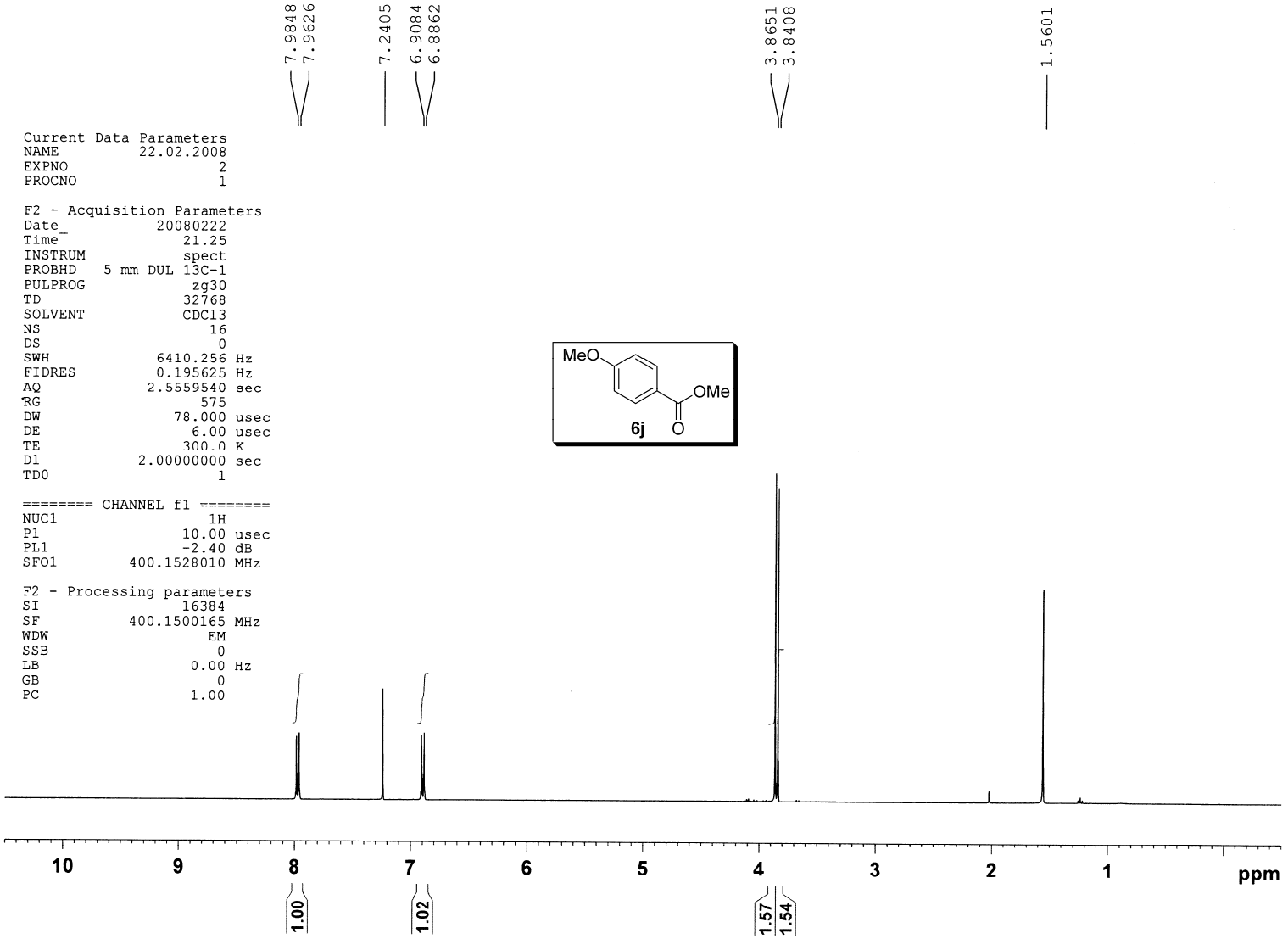


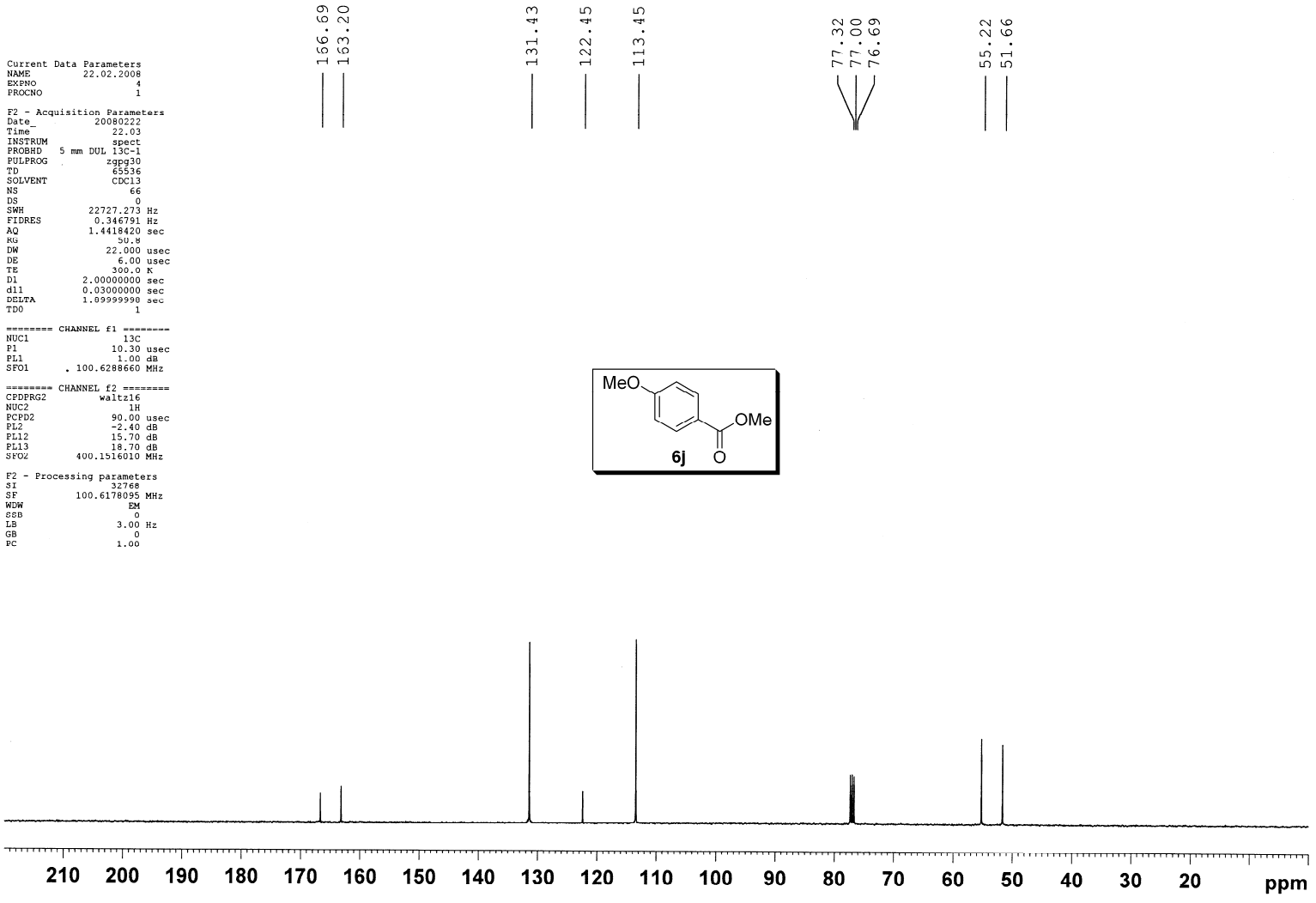


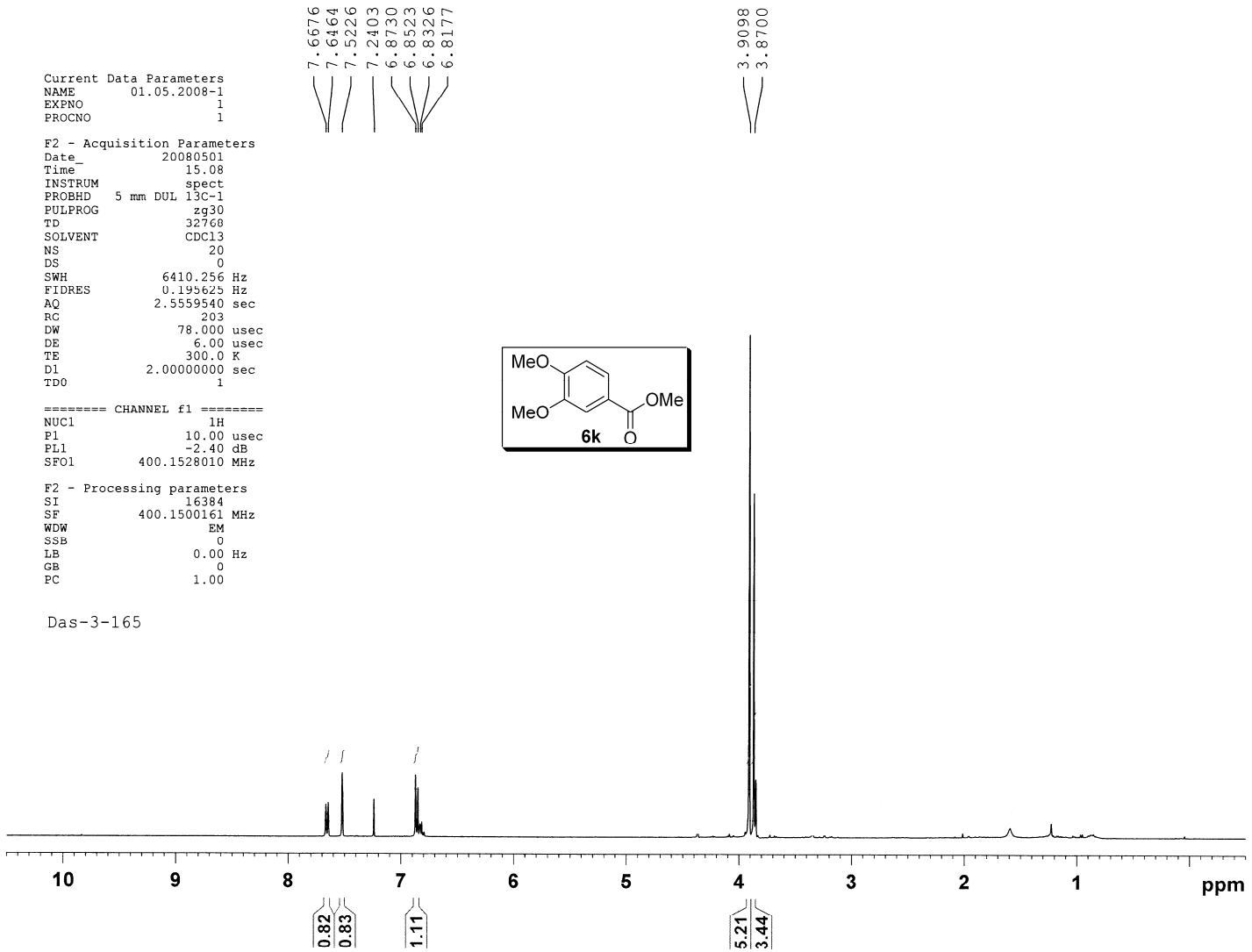












```
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NAME          Das-3-165
EXPNO                 2
PROCNO                1

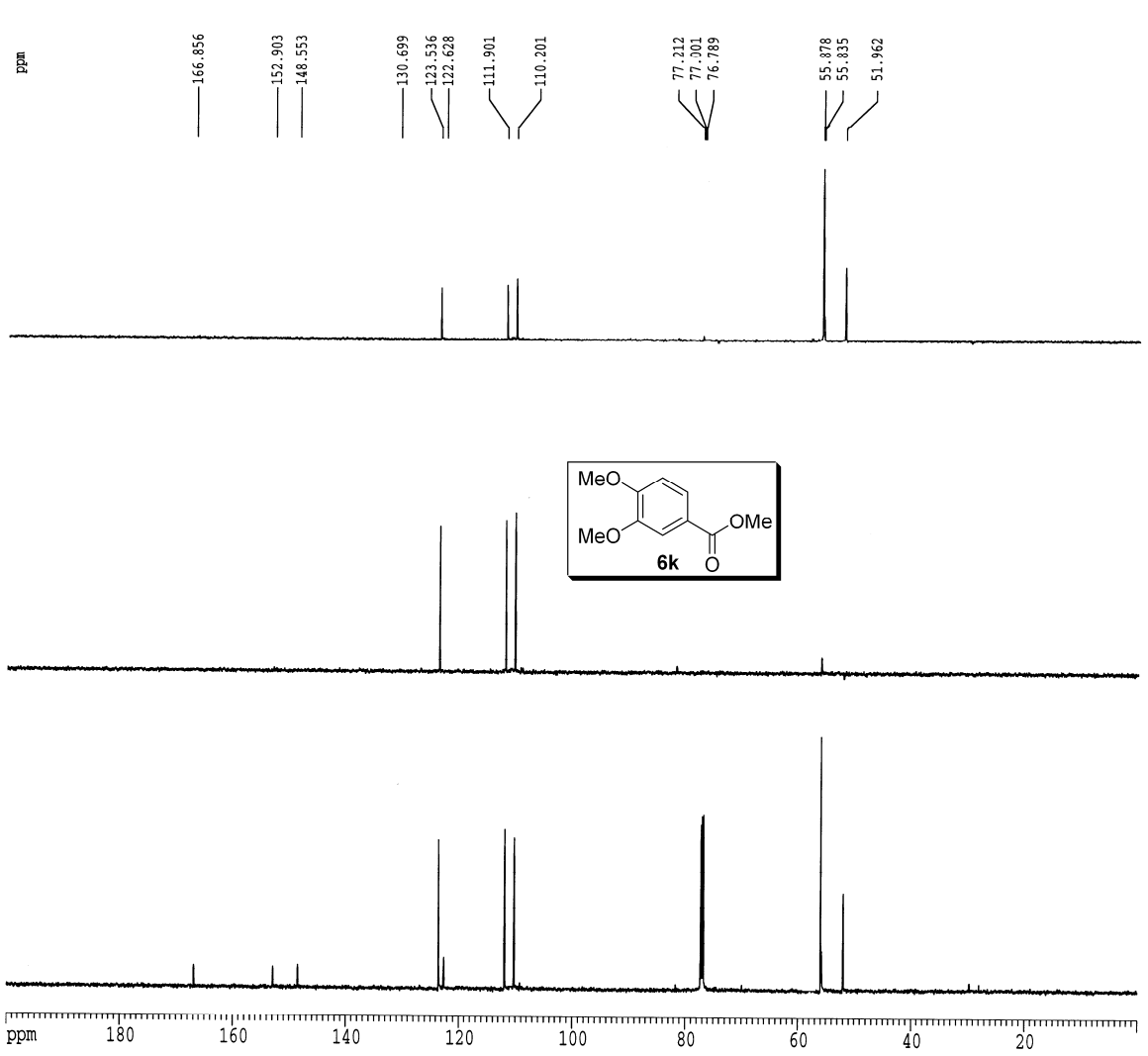
F2 - Acquisition Parameters
Date_          20080501
Time              14.13
INSTRUM           spect
PROBHD   5 mm QNP  1H/1
PULPROG           zgpg
TD               32768
SOLVENT           CDCl3
NS                  494
DS                    0
SWH           45045.047 Hz
FIDRES         1.374666 Hz
AQ            0.3637748 sec
RG                 1024
DW               11.100 usec
DE                6.50 usec
TE               299.6 K
D1           3.00000000 sec
d11          0.03000000 sec
DELTA        2.90000010 sec
MCREST       0.00000000 sec
MCWRK        0.01500000 sec

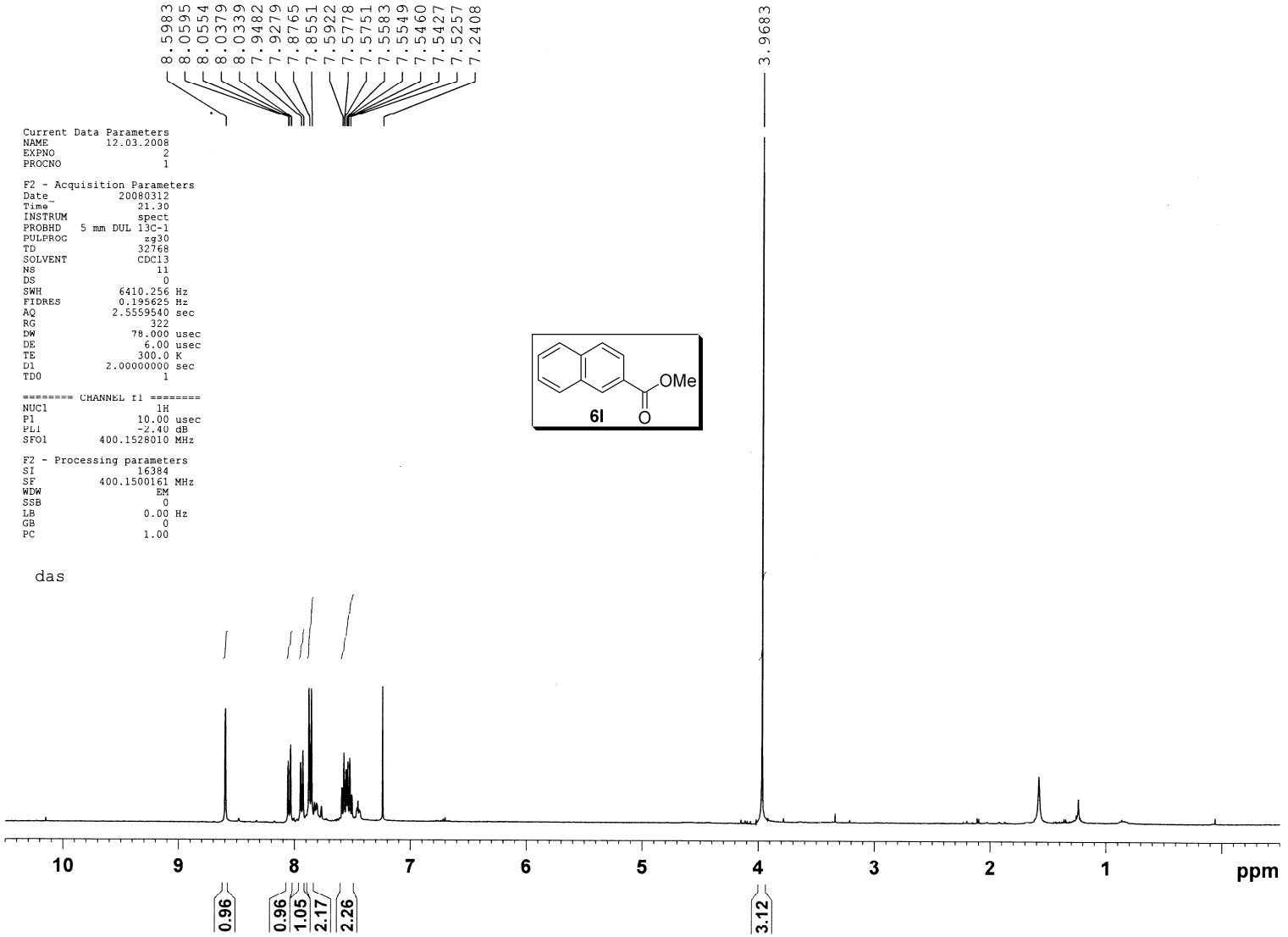
======== CHANNEL f1 ========
NUC1                13C
P1                 4.50 usec
PL1                0.00 dB
SFO1        150.7099812 MHz

======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                1H
PCPD2             92.00 usec
PL2              120.00 dB
PL12              11.30 dB
PL13              14.00 dB
SFO2        599.3034966 MHz

F2 - Processing parameters
SI                 32768
SF          150.6941122 MHz
WDW                   EM
SSB                    0
LB                  3.00 Hz
GB                     0
PC                  1.00

1D NMR plot parameters
CX                 20.00 cm
CY                  4.50 cm
F1P               200.000 ppm
F1              30138.82 Hz
F2P                0.000 ppm
F2                 0.00 Hz
PPMCM          10.00000 ppm/cm
HZCM         1506.94104 Hz/cm
```





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Current Data Parameters
NAME                  Das-3-121
EXPNO                         2
PROCNO                        1

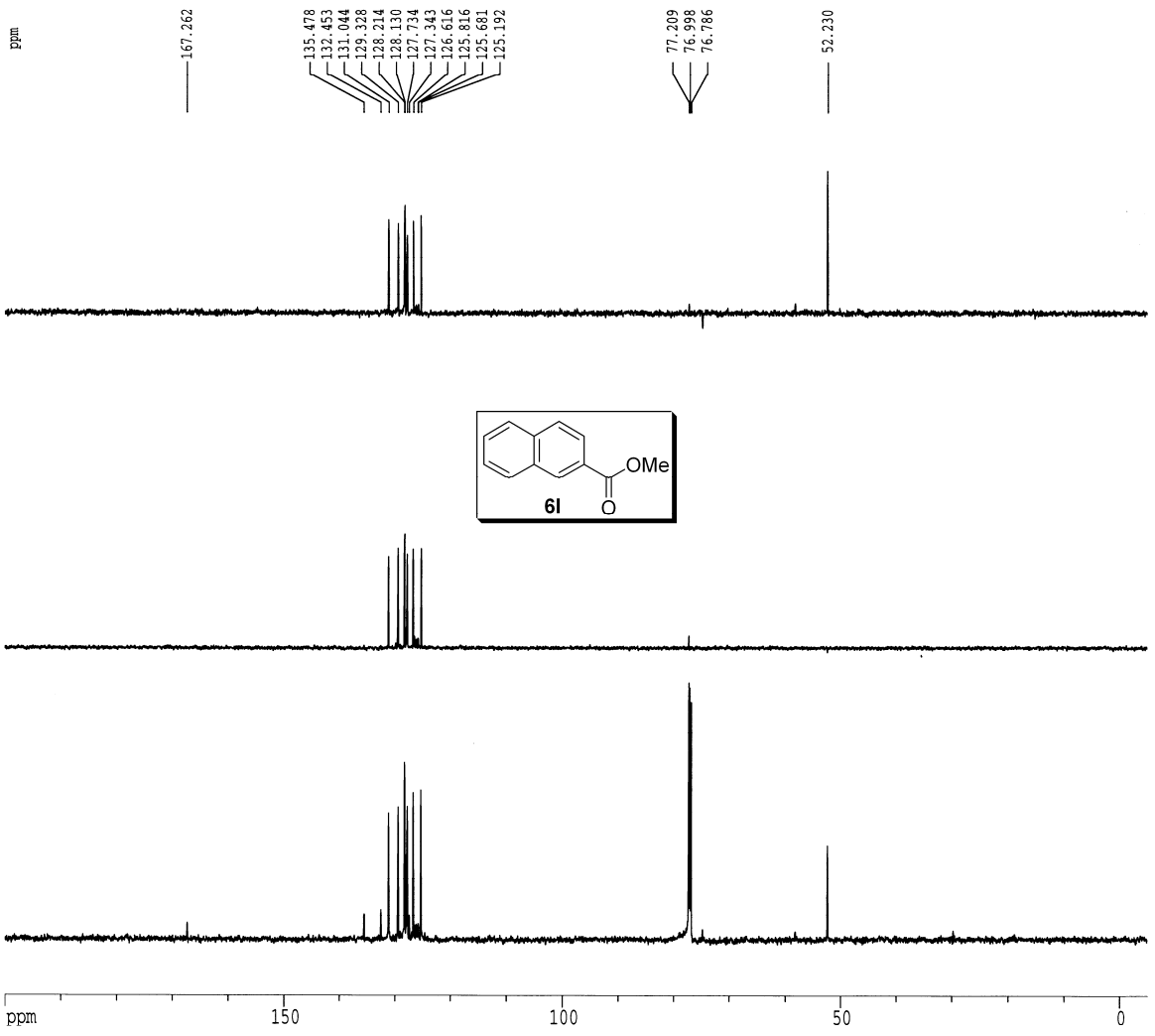
F2 - Acquisition Parameters
Date_               20080312
Time                    16.26
INSTRUM                 spect
PROBHD   5 mm QNP  1H/1
PULPROG                 zgpg
TD                      32768
SOLVENT                 CDCl3
NS                        400
DS                          0
SWH                 45045.047 Hz
FIDRES                1.374666 Hz
AQ                  0.3637748 sec
RG                       4096
DW                     11.100 usec
DE                      6.50 usec
TE                     297.0 K
D1                  3.00000000 sec
d11                 0.03000000 sec
DELTA               2.90000010 sec
MCREST              0.00000000 sec
MCWRK               0.01500000 sec

======== CHANNEL f1 ========
NUC1                      13C
P1                      4.50 usec
PL1                     0.00 dB
SFO1               150.7090011 MHz

======== CHANNEL f2 ========
CPDPRG2                 waltz16
NUC2                        1H
PCPD2                   92.00 usec
PL2                    120.00 dB
PL12                    11.30 dB
PL13                    14.00 dB
SFO2               599.3034966 MHz

F2 - Processing parameters
SI                        32768
SF                 150.6941136 MHz
WDW                        EM
SSB                         0
LB                      5.00 Hz
GB                          0
PC                      1.00

1D NMR plot parameters
CX                     20.00 cm
CY                      4.50 cm
F1P                   200.000 ppm
F1                  30138.82 Hz
F2P                    -5.000 ppm
F2                   -753.47 Hz
PPMCM                10.25000 ppm/cm
HZCM                1544.61475 Hz/cm
```



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Current Data Parameters
NAME          Das-3-259
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20080923
Time               1.29
INSTRUM           spect
PROBHD   5 mm QNP  1H/1
PULPROG              zg
TD                32768
SOLVENT           CDCl3
NS                   16
DS                    0
SWH            8389.262 Hz
FIDRES         0.256020 Hz
AQ            1.9530228 sec
RG                  128
DW               59.600 usec
DE                6.50 usec
TE               300.5 K
D1           1.50000000 sec
MCREST       0.00000000 sec
MCWRK        0.01500000 sec

======== CHANNEL f1 ========
NUC1                 1H
P1                10.00 usec
PL1                0.00 dB
SFO1       599.2029960 MHz

F2 - Processing parameters
SI                16384
SF          599.2000254 MHz
WDW                  no
SSB                   0
LB                 0.00 Hz
GB                    0
PC                 0.50

1D NMR plot parameters
CX                20.00 cm
CY                20.00 cm
F1P               9.000 ppm
F1              5392.80 Hz
F2P               0.500 ppm
F2              299.60 Hz
PPMCM          0.42500 ppm/cm
HZCM         254.66000 Hz/cm
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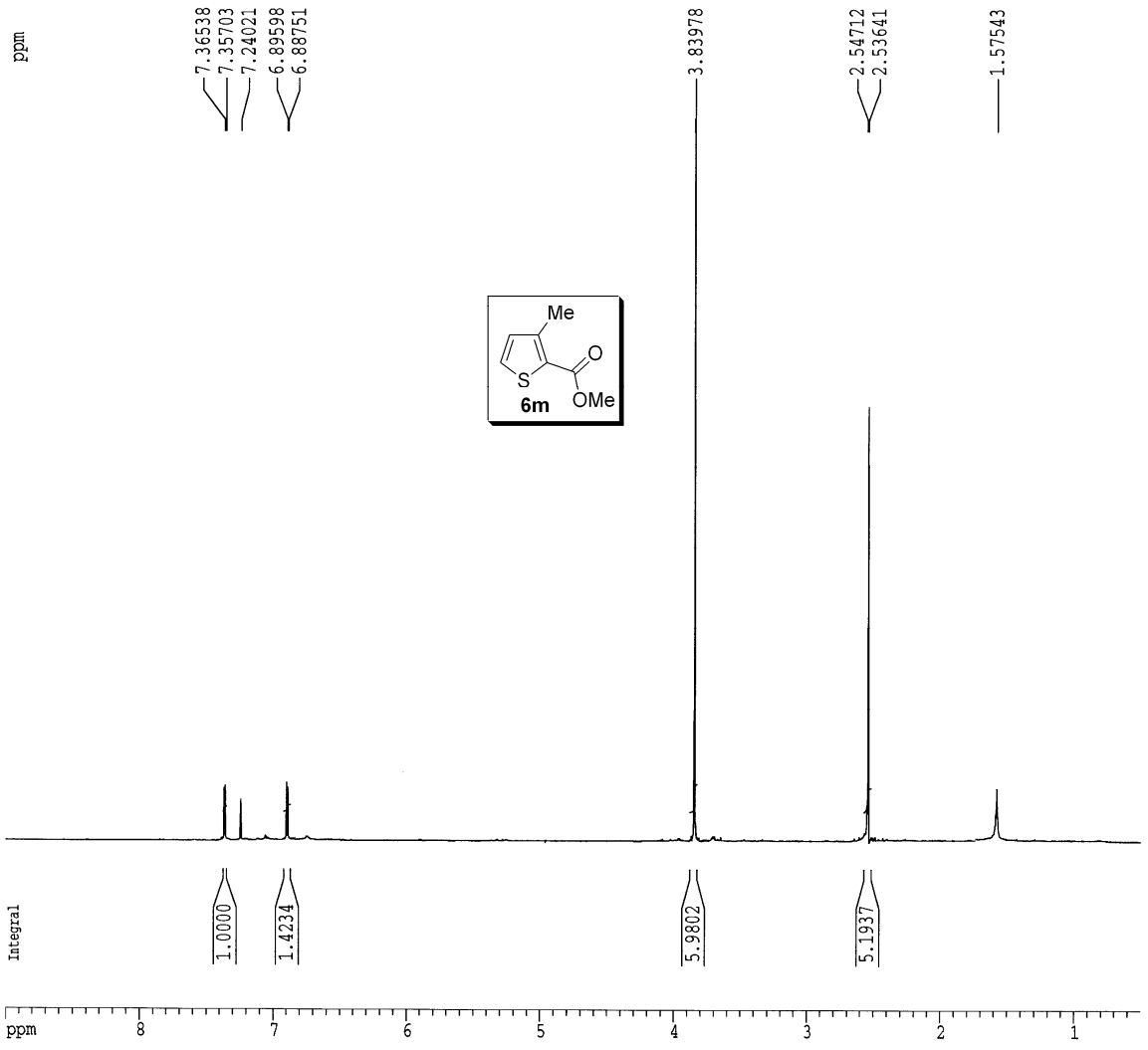

```
rrent Data Parameters
4E           Das-3-059
PNO                  2
DCNO                 1

 - Acquisition Parameters
:e_           20080923
ne                1.31
STRUM            spect
OBHD   5 mm QNP  1H/1
.PROG             zgig
                 32768
.VENT            CDCl3
                  5120
                     0
!             45045.047 Hz
DRES          1.374666 Hz
              0.3637748 sec
                  1024
                11.100 usec
                 6.50 usec
                300.5 K
             3.00000000 sec
             0.03000000 sec
EST          0.00000000 sec
RK           0.01500000 sec

===== CHANNEL f1 ========
:1                13C
                 4.50 usec
                 0.00 dB
)1         150.6855336 MHz

===== CHANNEL f2 ========
)PRG2          waltz16
:2                1H
)D2              92.00 usec
                120.00 dB
:2               11.30 dB
)2         599.2029960 MHz

 - Processing parameters
                65536
          150.6689654 MHz
                   EM
                    0
                3.00 Hz
                    0
                1.00

NMR plot parameters
               20.00 cm
               10.00 cm
             200.000 ppm
            30133.79 Hz
               0.000 ppm
                0.00 Hz
CM           10.00000 ppm/cm
M          1506.68958 Hz/cm
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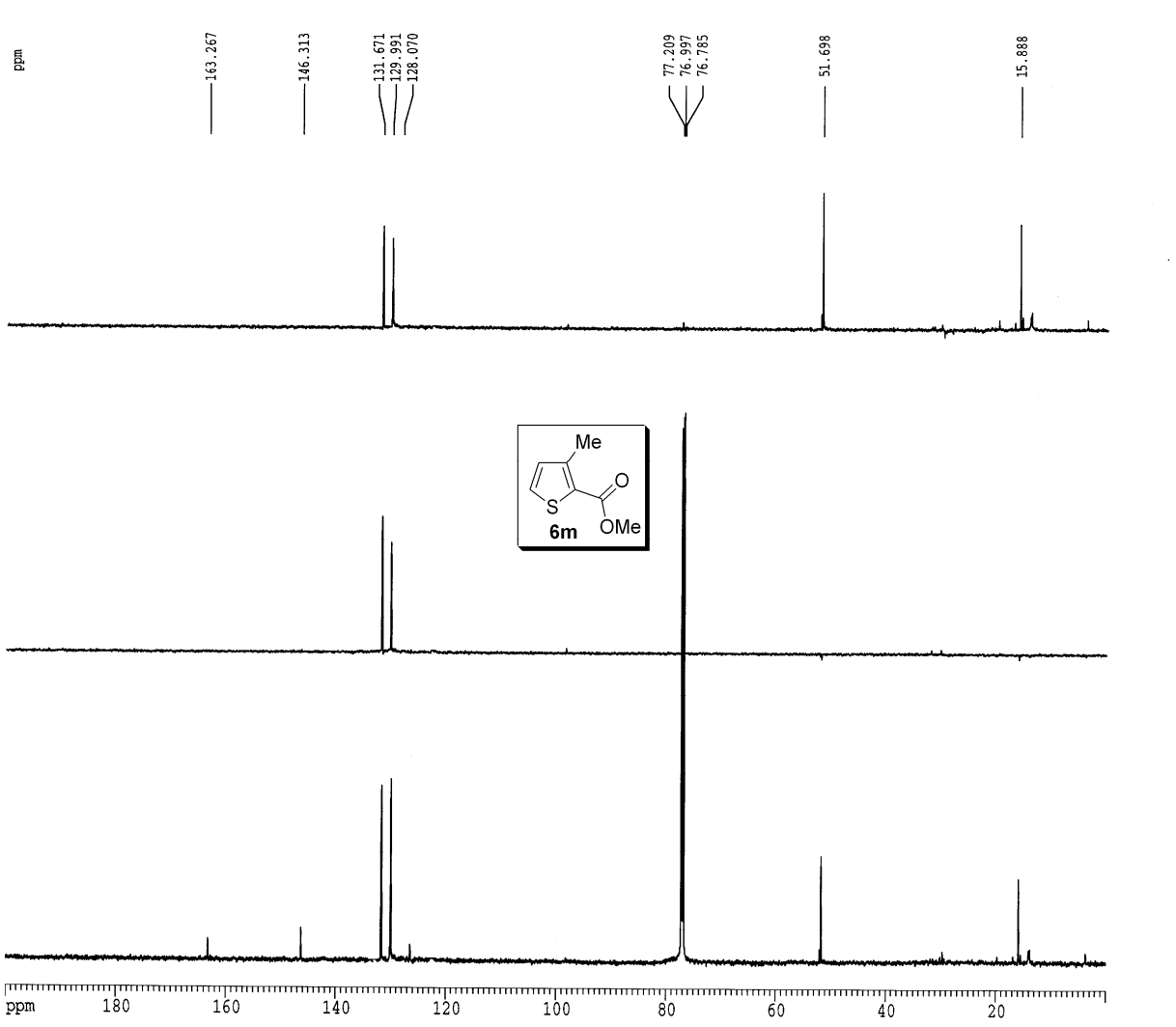

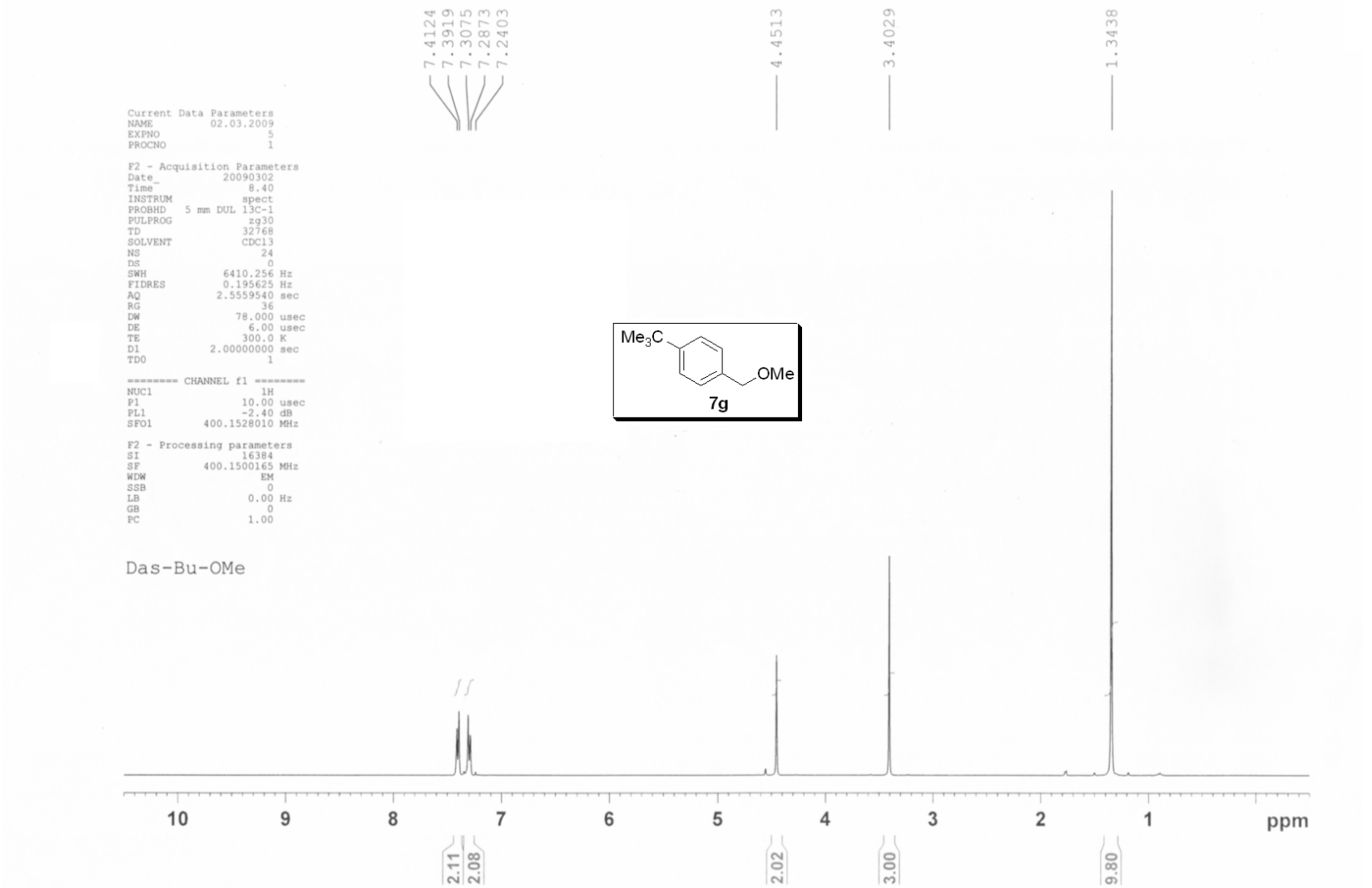

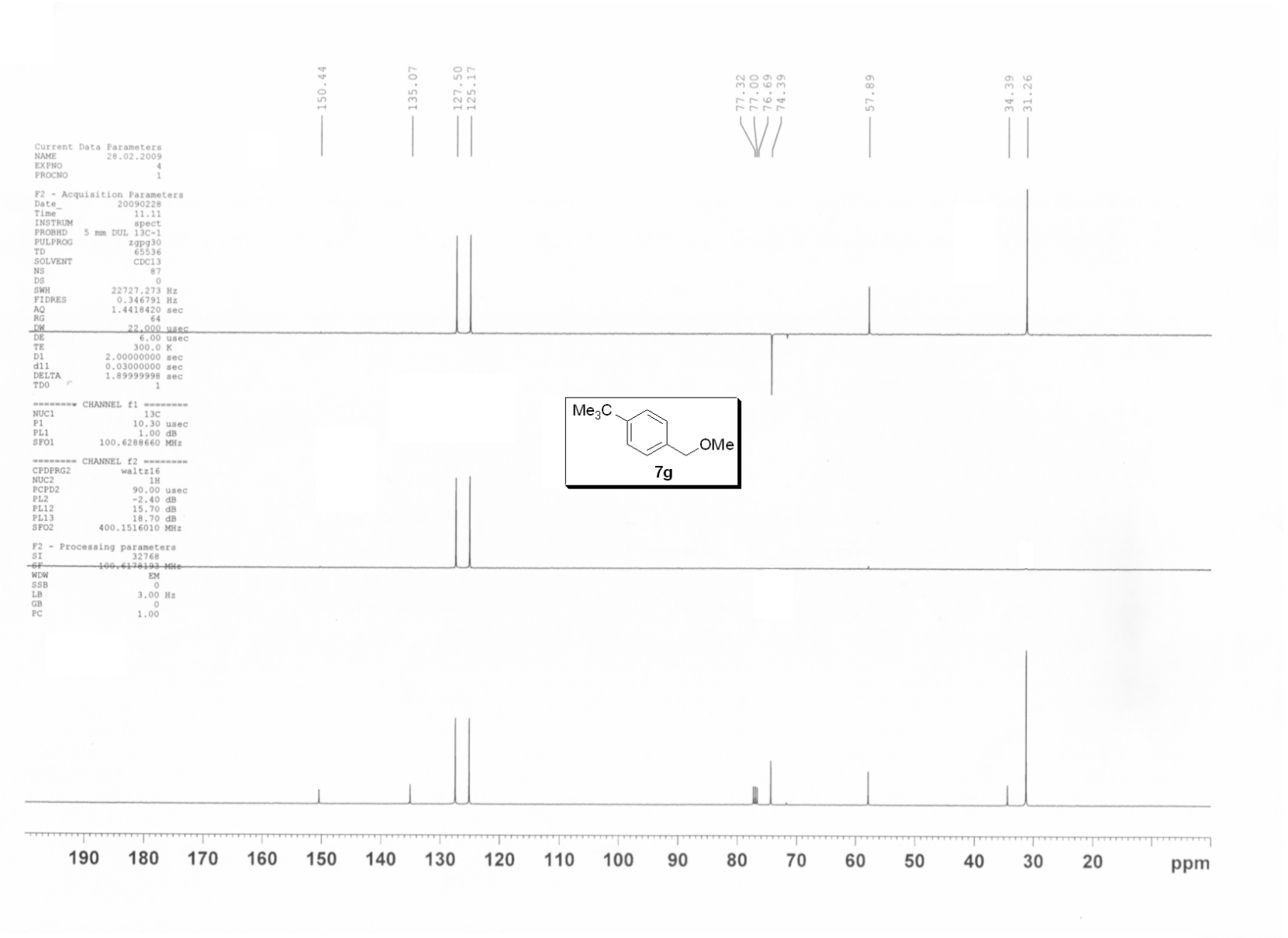

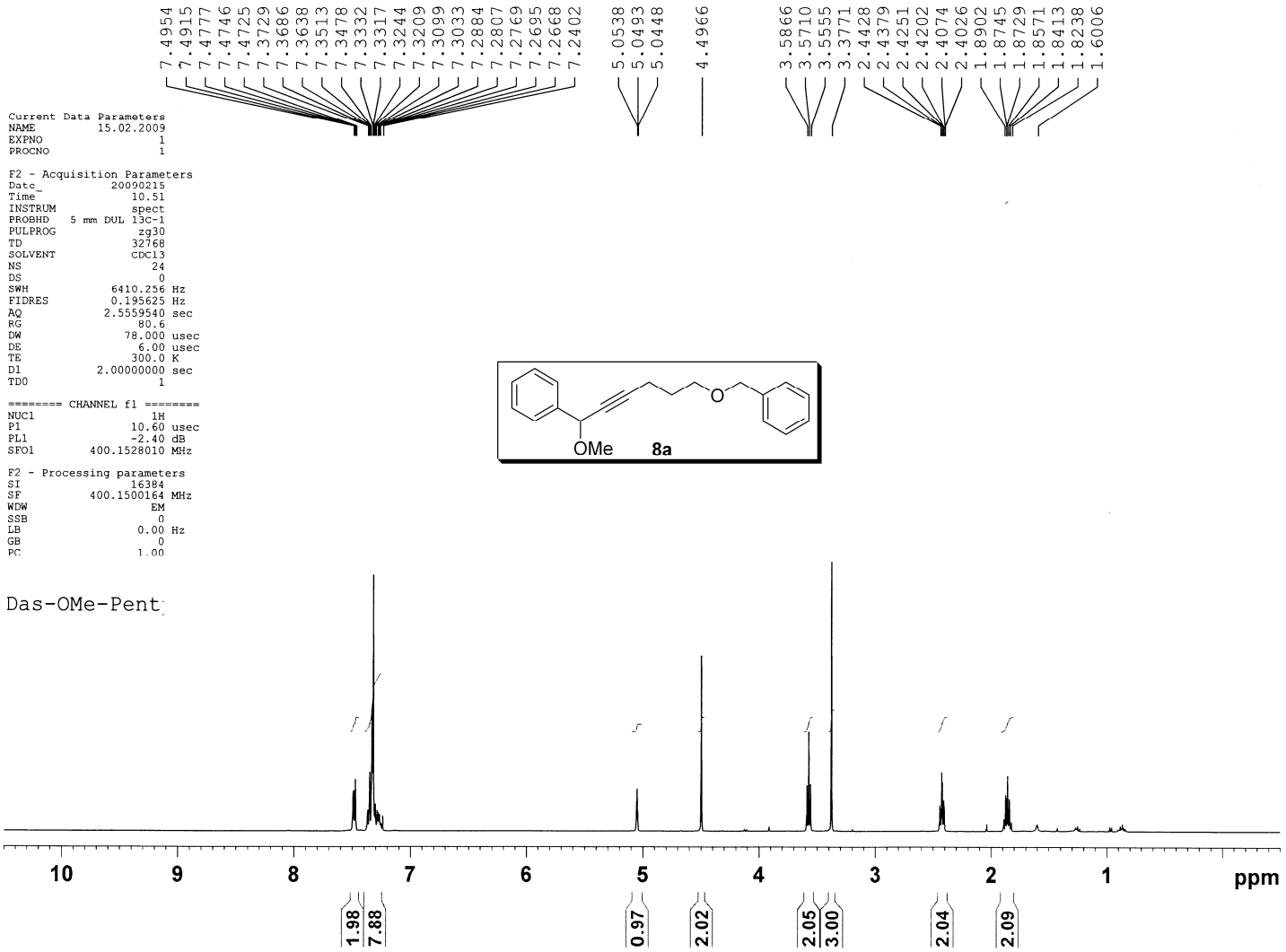

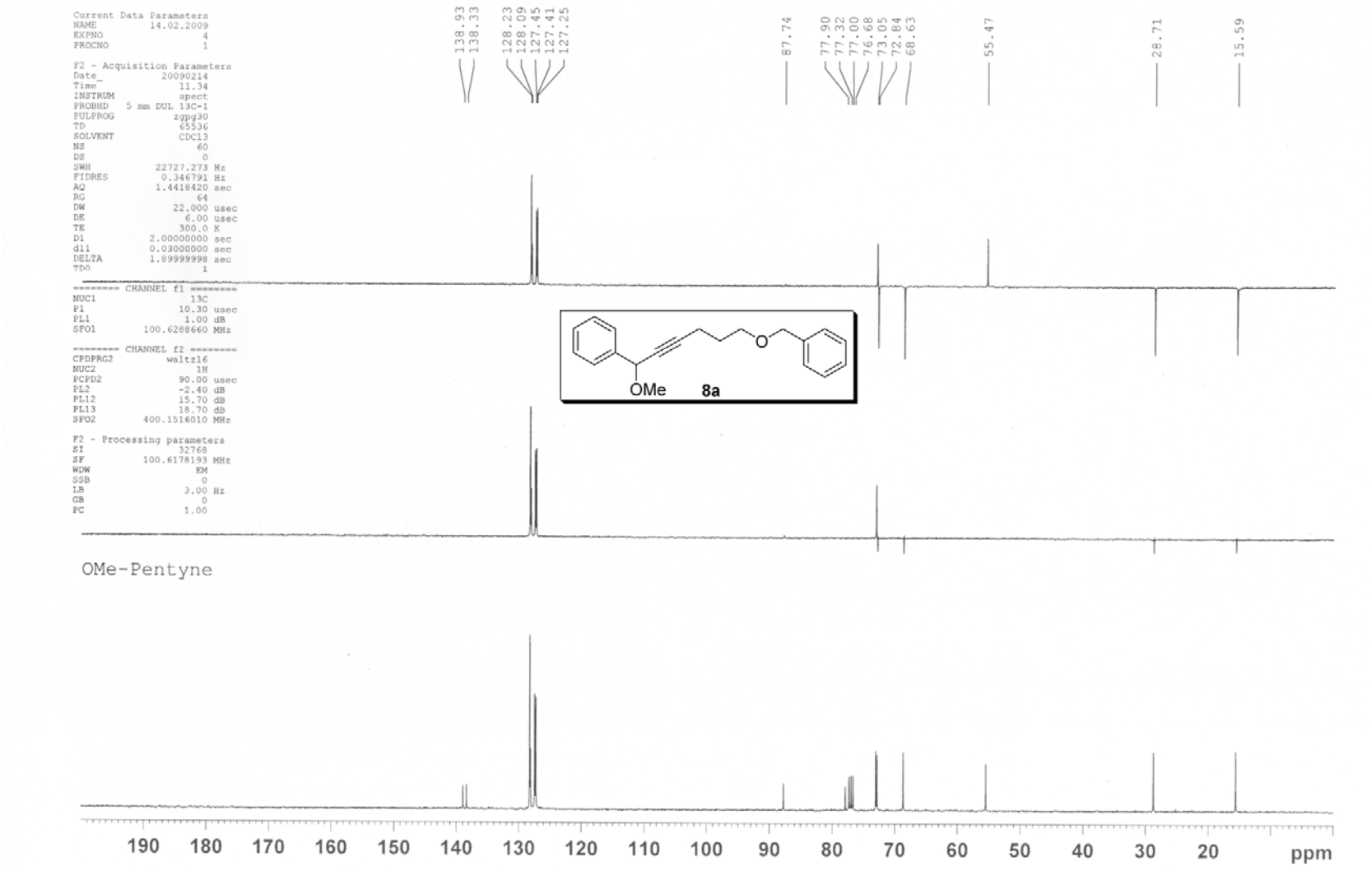

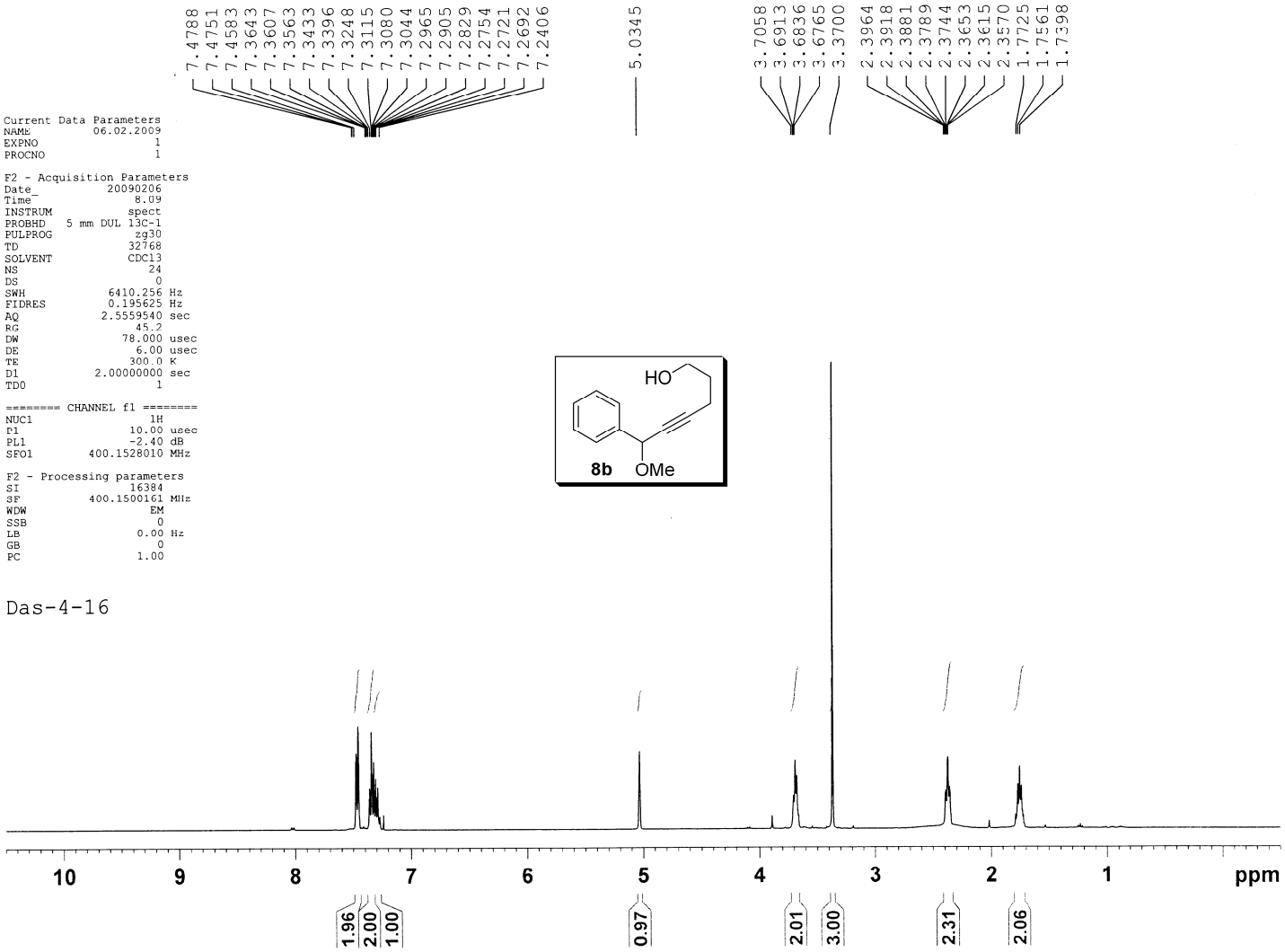

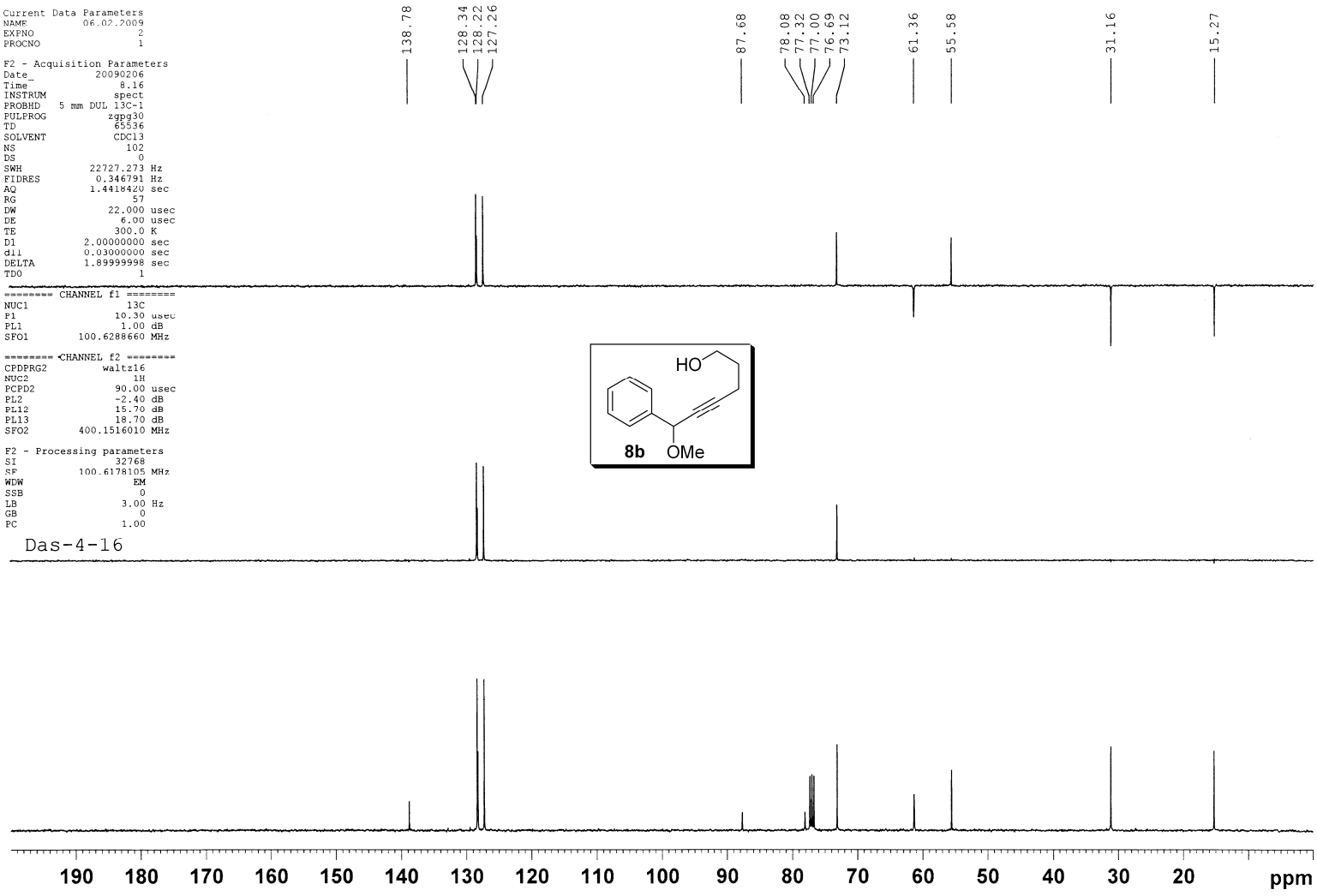

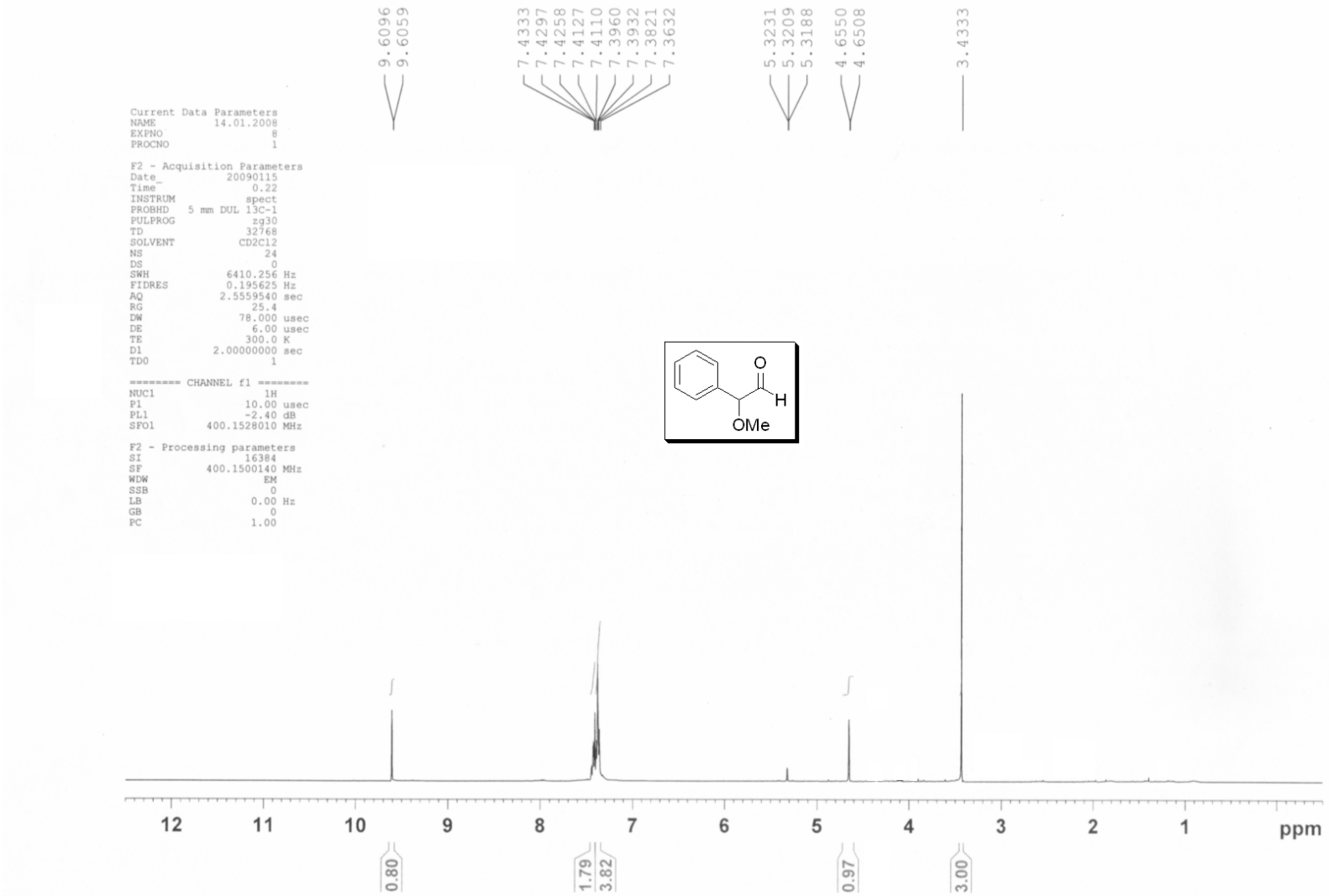

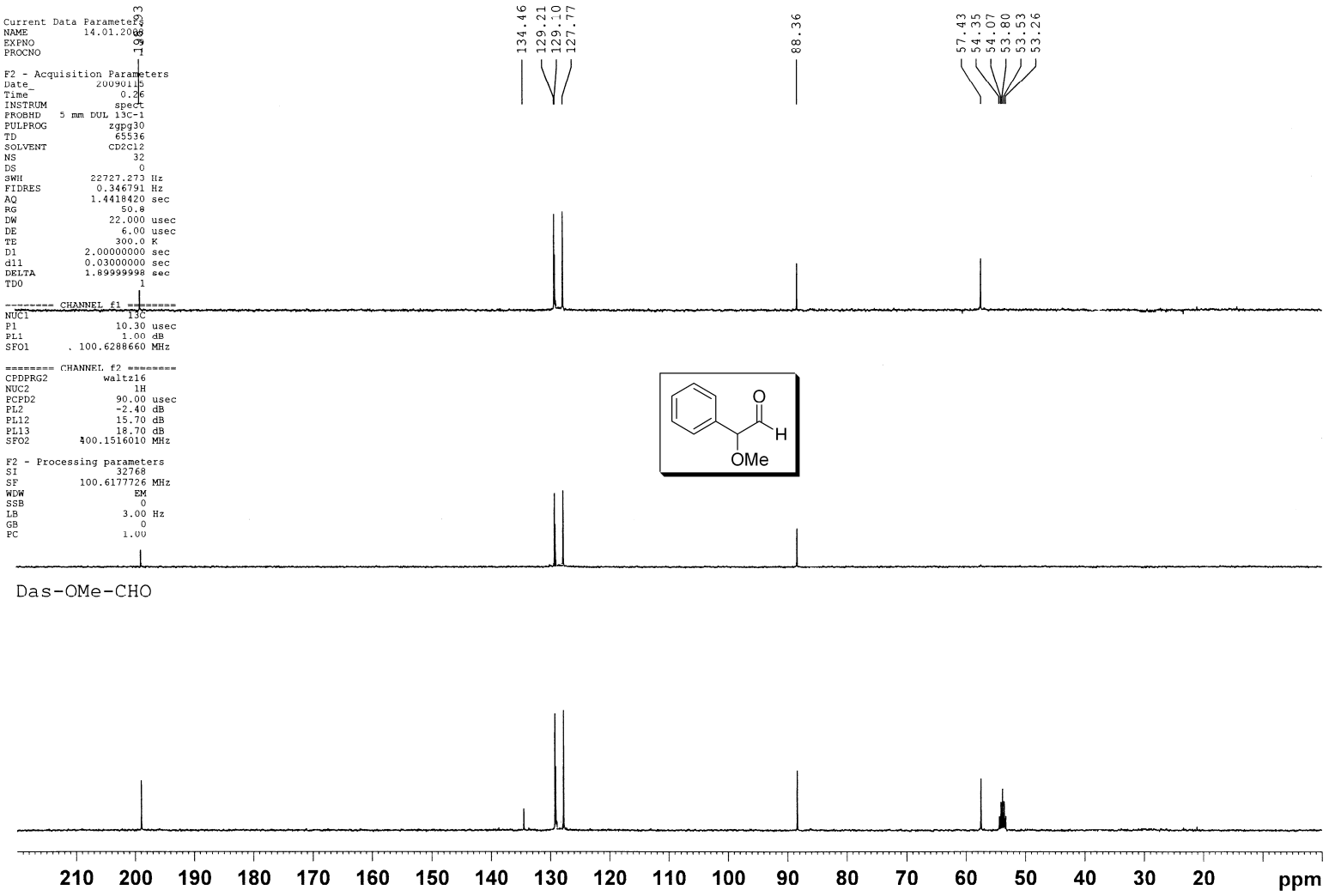
Current Data Parameters
NAME 14.01.2009
EXPNO 9
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090115
Time 0.26
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CD2Cl2
NS 32
DS 0
SWH 22727.273 Hz
FIDRES 0.346791 Hz
AQ 1.4418420 sec
RG 50.8
DW 22.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 10.30 usec
PL1 1.00 dB
SFO1 100.6288660 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -2.40 dB
PL12 15.70 dB
PL13 18.70 dB
SFO2 400.1516010 MHz
F2 - Processing parameters
SI 32768
SF 100.6177726 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.00
134.46
129.21
129.10
127.77
88.36
57.43
54.35
54.07
53.80
53.53
53.26
O
H
OMe
Das-OMe-CHO
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm