Supporting Information

Iron-Catalyzed Aerobic Oxidation of Allylic Alcohols: The Issue of C=C Bond Isomerization

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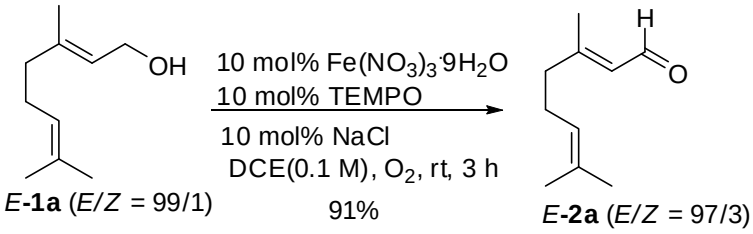
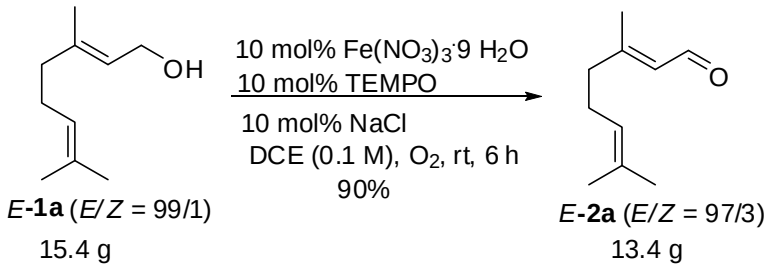
General Experimental Methods: ^{1}H and ^{13}C NMR spectra were recorded with an instrument operated at 300 MHz for ^{1}H NMR and 75 MHz for ^{13}C NMR in $CDCl_3$. Chemical shifts (δ) are given in parts per million (ppm). IR spectra were measured with a Bruker Tensor 27 instrument. MS analyses were performed with an Aglient Technologies 5975C instrument. HRMS analyses were performed with a Waters GCT Premier instrument. All the substrates were commercial available.

1. Synthesis of Geranial (*E*-**2a**, ljx-9-84)



E-**1a** (*E*/*Z* = 99/1) → *E*-**2a** (*E*/*Z* = 97/3)

Typical procedure: To a 25 mL three-necked flask were added $Fe(NO_3)_3{\cdot}9H_2O$ (40.6 mg, 0.1 mmol), TEMPO (15.6 mg, 0.1 mmol), NaCl (6.0 mg, 0.1 mmol), and DCE (10 mL). *E*-**1a** (152.6 mg, 1.0 mmol, *E*/*Z* = 99/1) was then added to the suspension and the resulting mixture was placed under the atmosphere of oxygen from a balloon and stirred at room temperature until the reaction was complete as monitored by TLC (eluent: petroleum ether/ethyl acetate = 10:1). Mesitylene (46 μl) was added for NMR analysis (*E*/*Z* = 97/3). After evaporation, the crude product was purified by column chromatography on silica gel (petroleum ether /diethyl ether = 10/1) to afford *E*-**2a**[1] (134.6 mg, 91%, *E*/*Z* = 97:3) (petroleum ether/diethyl ether = 10/1) as oil: ^{1}H NMR (300 MHz, $CDCl_3$) δ 10.01 (d, *J* = 8.1 Hz, 1 H, CHO), 5.90 (d, *J* = 8.1 Hz, 1 H, CH=), 5.15-5.05 (m, 1 H, CH=), 2.30-2.20 (m, 4 H, 2 × CH_2), 2.18 (s, 3 H, CH_3), 1.70 (s, 3 H, CH_3), 1.63 (s, 3 H, CH_3); ^{13}C NMR (75.4 MHz, $CDCl_3$) δ 190.8, 163.3, 132.4, 126.9, 122.1, 40.1, 25.23, 25.15, 17.2, 17.1; IR (neat) 2724, 1673, 1444, 1380, 1194, 1121 cm^{-1}; MS(EI) *m/z* 152 (M^+, 1.16), 41 (100).



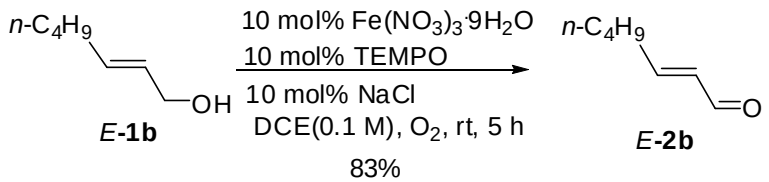
E-**1a** (*E*/*Z* = 99/1)
15.4 g

E-**2a** (*E*/*Z* = 97/3)
13.4 g

0.1 mol scale reaction: To a 2 L three-necked flask were added $Fe(NO_3)_3{\cdot}9H_2O$ (4.0561 g, 10 mmol), TEMPO (1.5640 g, 10 mmol), NaCl (0.5854 g, 10 mmol), and DCE (1000 mL). *E*-**1a** (15.4035 g, 0.1 mol, *E*/*Z* = 99/1) was then added to the suspension and the resulting mixture was placed under the atmosphere of oxygen from an oxygen bag and stirred at room temperature until the reaction was complete as monitored by TLC (eluent: petroleum ether/ethyl acetate = 10:1). The resulting mixture was concentrated in vacuo and further purified by column chromatography on silica gel (petroleum ether /diethyl ether = 10/1) to afford *E*-**2a** (13.4721 g, 90%, *E*/*Z* = 97:3) (petroleum ether/diethyl ether = 10/1) as oil.

The following compounds **2b**-**2p** and *Z*-**2a** were prepared according to the typical procedure of *E*-**2a**.

2. Synthesis of (*E*)-hept-2-enal (*E*-**2b**, ljx-9-178)



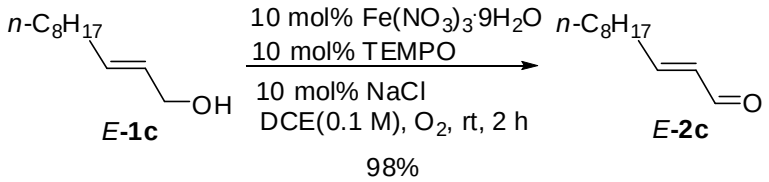
E-**1b**

E-**2b**

The reaction of (*E*)-hept-2-enol *E*-**1b** (112.7 mg, 1.0 mmol), $Fe(NO_3)_3{\cdot}9H_2O$ (40.6 mg, 0.1 mmol), TEMPO (15.7 mg, 0.1 mmol), and NaCl (5.7 mg, 0.1 mmol) in

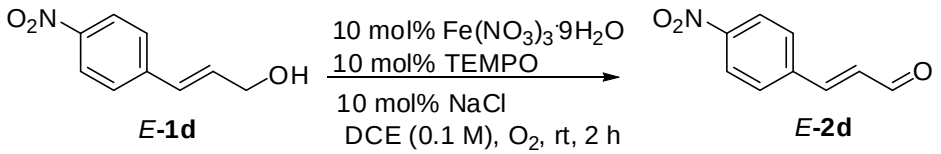
DCE (10 mL) afforded (*E*)-hept-2-enal[2] *E*-**2b** (88.1 mg, 83%, *E*/*Z* >99/1) (petroleum ether/diethyl ether = 10/1) as oil: ^{1}H NMR (300 MHz, $CDCl_3$) δ 9.48 (d, J = 8.1 Hz, 1 H, CHO), 6.84 (dt, J_1 = 15.6 Hz, J_2 = 6.9 Hz, 1 H, CH=), 6.10 (ddt, J_1 = 15.6 Hz, J_2 = 7.8 Hz, J_3 = 1.5 Hz, 1 H, CH=), 2.32 (qd, J_1 = 7.2 Hz, J_2 = 1.2 Hz, 2 H, CH_2), 1.53-1.28 (m, 4 H, 2 × CH_2), 0.91 (t, J = 7.2 Hz, 3 H, CH_3); ^{13}C NMR (75.4 MHz, $CDCl_3$) δ 194.1, 159.0, 132.9, 32.3, 29.8, 22.1, 13.7; IR (neat) 2960, 2932, 2733, 1692, 1637, 1466, 1153, 1104, 977 cm^{-1}; MS(EI) *m/z* 112 (M^+, 10.07), 83 (100).

3. Synthesis of (*E*)-undec-2-enal (*E*-**2c**, ljx-5-67)



The reaction of (*E*)-undec-2-enol *E*-**1c** (170.5 mg, 1.0 mmol), $Fe(NO_3)_3{\cdot}9H_2O$ (40.3 mg, 0.1 mmol), TEMPO (15.4 mg, 0.1 mmol), and NaCl (5.8 mg, 0.1 mmol) in DCE (10 mL) afforded (*E*)-undec-2-enal[3] *E*-**2c** (158.8 mg, 98%, *E*/*Z* >99/1) (petroleum ether/diethyl ether = 10/1) as oil: ^{1}H NMR (300 MHz, $CDCl_3$) δ 9.50 (d, J = 8.1 Hz, 1 H, CHO), 6.84 (dt, J_1 = 15.2 Hz, J_2 = 6.8 Hz, 1 H, CH=), 6.10 (ddt, J_1 = 15.9 Hz, J_2 = 8.1 Hz, J_3 = 1.5 Hz, 1 H, CH=), 2.32 (qd, J_1 = 7.2 Hz, J_2 = 1.2 Hz, 2 H, CH_2), 1.55-1.42 (m, 2 H), 1.40-1.20 (m, 10 H), 0.87 (t, J = 6.8 Hz, 3 H, CH_3); ^{13}C NMR (75.4 MHz, $CDCl_3$) δ 194.1, 159.1, 132.9, 32.7, 31.8, 29.3, 29.12, 29.10, 27.8, 22.6, 14.0; IR (neat) 2926, 2856, 2808, 2730, 1693, 1638, 1466, 1140, 1101 cm^{-1}; MS(EI) *m/z* 167 (M^+-H, 1.37), 41 (100).

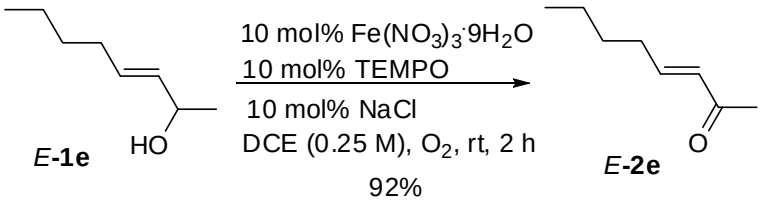
4. Synthesis of (*E*)-3-(4-nitrophenyl)acrylaldehyde (*E*-**2d**, ljx-5-70)



E-**1d** *E*-**2d**

The reaction of (*E*)-3-(4-nitrophenyl)prop-2-en-1-ol *E*-**1d** (179.9 mg, 1.0 mmol), $Fe(NO_3)_3{\cdot}9H_2O$ (40.2 mg, 0.1 mmol), TEMPO (15.8 mg, 0.1 mmol), and NaCl (6.0 mg, 0.1 mmol) in DCE (10 mL) afforded (*E*)-3-(4-nitrophenyl)acrylaldehyde[4] *E*-**2d** (172.6 mg, 99%, *E/Z* >99/1) (petroleum ether/diethyl ether = 3/1) as a yellow solid: m.p. 139-140 ^{o}C (hexane-ethyl acetate) (lit[4] 139-140 ^{o}C): ^{1}H NMR (300 MHz, $CDCl_3$) δ 9.78 (d, J = 7.5 Hz, 1 H, CHO), 8.29 (dt, J_1 = 9.0 Hz, J_2 = 2.8 Hz, 2 H, Ar-H), 7.73 (dt, J_1 = 8.7 Hz, J_2 = 1.8 Hz, 2 H, Ar-H), 7.53 (d, J = 16.2 Hz, 1 H, CH=), 6.81 (dd, J_1 = 16.2 Hz, J_2 = 7.5 Hz, 1 H, CH=); ^{13}C NMR (75.4 MHz, $CDCl_3$) δ 192.8, 148.9, 148.8, 139.9, 131.7, 129.0, 124.3; IR (neat) 2828, 2741, 1685, 1670, 1603, 1532, 1418, 1346, 1314, 1295, 1259, 1123 cm^{-1}; MS(EI) *m/z* 177 (M^+, 6.25), 77 (100).

5. Synthesis of (*E*)-oct-3-en-2-one (*E*-**2e**, ljx-9-179)

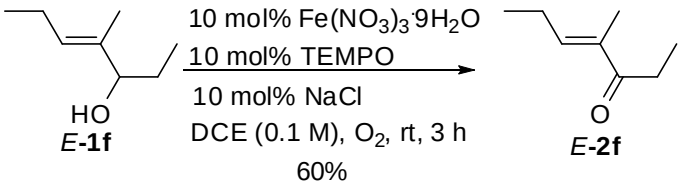


E-**1e** *E*-**2e**

The reaction of (*E*)-oct-3-en-2-ol *E*-**1e** (128.9 mg, 1.0 mmol), $Fe(NO_3)_3{\cdot}9H_2O$ (40.2 mg, 0.1 mmol), TEMPO (15.5 mg, 0.1 mmol), and NaCl (6.0 mg, 0.1 mmol) in DCE (10 mL) afforded (*E*)-oct-3-en-2-one[5] *E*-**2e** (113.4 mg, 92%, *E/Z* >99/1) (petroleum ether/diethyl ether = 10/1) as oil: ^{1}H NMR (300 MHz, $CDCl_3$) δ 6.77 (dt, J_1 = 15.9 Hz, J_2 = 6.9 Hz, 1 H, CH=), 6.03 (dt, J_1 = 15.9 Hz, J_2 = 1.5 Hz, 1 H, CH=),

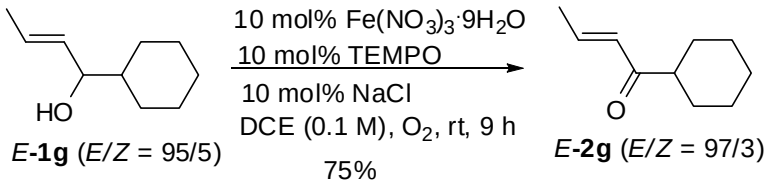
2.25-2.13 (m, 5 H, CH_2 and CH_3), 1.50-1.24 (m, 4 H, 2 × CH_2), 0.88 (t, J = 7.2 Hz, 3 H, CH_3); ^{13}C NMR (75.4 MHz, $CDCl_3$) δ 198.6, 148.4, 131.1, 32.0, 30.0, 26.6, 22.1, 13.6; IR (neat) 2959, 2931, 1699, 1675, 1627, 1466, 1432, 1361, 1253, 1178, 1102, 981 cm^{-1}; MS(EI) *m/z* 126 (M^+, 1.30), 43 (100).

6. Synthesis of (*E*)-4-methylhept-4-en-3-one (*E*-**2f**, ljx-9-177)



The reaction of (*E*)-4-methylhept-4-en-3-ol *E*-**1f** (128.2 mg, 1.0 mmol), $Fe(NO_3)_3{\cdot}9H_2O$ (40.2 mg, 0.1 mmol), TEMPO (15.7 mg, 0.1 mmol), and NaCl (5.9 mg, 0.1 mmol) in DCE (10 mL) afforded (*E*)-4-methylhept-4-en-3-one[6] *E*-**2f** (74.0 mg, 60%, *E/Z* >99/1) (petroleum ether/diethyl ether = 10/1) as oil: ^{1}H NMR (300 MHz, $CDCl_3$) δ 6.58 (t, J = 7.2 Hz, 1 H, CH=), 2.66 (q, J = 7.3 Hz, 2 H, CH_2), 2.30-2.15 (m, 2 H, CH_2), 1.75 (s, 3 H, CH_3), 1.12-1.00 (m, 6 H, 2 × CH_3); ^{13}C NMR (75.4 MHz, $CDCl_3$) δ 202.6, 143.5, 136.3, 30.2, 22.2, 13.0, 11.1, 8.8; IR (neat) 2967, 2937, 1731, 1672, 1561, 1461, 1378, 1260, 1093, 1020 cm^{-1}; MS(EI) *m/z* 126 (M^+, 3.29), 44 (100).

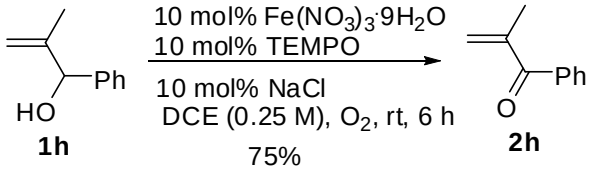
7. Synthesis of (*E*)-1-cyclohexylbut-2-en-1-one (*E*-**2g**, ljx-5-77)



The reaction of (*E*)-1-cyclohexylbut-2-en-1-ol *E*-**1g** (153.8 mg, 1.0 mmol, *E/Z* =

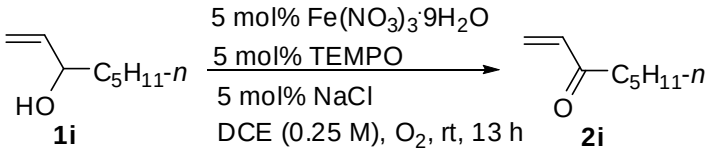
95/5), $Fe(NO_3)_3{\cdot}9H_2O$ (40.9 mg, 0.1 mmol), TEMPO (15.8 mg, 0.1 mmol), and NaCl (6.0 mg, 0.1 mmol) in DCE (4 mL) afforded (*E*)-1-cyclohexylbut-2-en-1-one[7] *E*-**2g** (114.3 mg, 75%, *E*/*Z* = 97/3) (petroleum ether/diethyl ether = 10/1) as oil: ^{1}H NMR (300 MHz, $CDCl_3$) δ 6.87 (dq, J_1 = 15.6 Hz, J_2 = 6.9 Hz, 1 H, CH=), 6.18 (dq, J_1 = 15.6 Hz, J_2 = 1.7 Hz, 1 H, CH=), 2.60-2.48 (m, 1 H), 1.88 (dd, J_1 = 6.9 Hz, J_2 = 1.5 Hz, 3 H, CH_3), 1.85-1.60 (m, 5 H), 1.42-1.10 (m, 5 H); ^{13}C NMR (75.4 MHz, $CDCl_3$) δ 203.2, 142.1, 130.2, 48.4, 28.6, 25.8, 25.7, 18.2; IR (neat) 2931, 2855, 1693, 1667, 1631, 1447, 1377, 1317, 1294, 1249, 1196, 1173, 1149, 1132, 1120, 1065, 1019 cm^{-1}; MS(EI) *m/z* 152 (M^+, 0.70), 41 (100).

8. Synthesis of 2-methyl-1-phenylprop-2-en-1-one (**2h**, ljx-9-185)



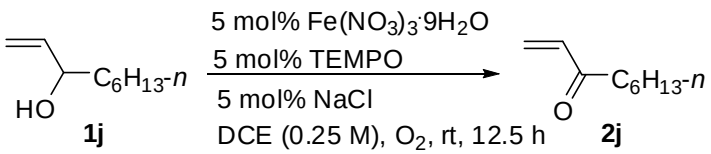
The reaction of 2-methyl-1-phenylprop-2-en-1-ol **1h** (147.5 mg, 1.0 mmol), $Fe(NO_3)_3{\cdot}9H_2O$ (40.2 mg, 0.1 mmol), TEMPO (15.7 mg, 0.1 mmol), and NaCl (5.9 mg, 0.1 mmol) in DCE (4 mL) afforded 2-methyl-1-phenylprop-2-en-1-one[8] **2h** (92.9 mg, 75%) (petroleum ether/diethyl ether = 10/1) as oil: ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.76-7.70 (m, 2 H, Ar-H), 7.53 (t, *J* = 7.4 Hz, 1 H, Ar-H), 7.43 (t, *J* = 7.4 Hz, 2 H, Ar-H), 5.91 (s, 1 H, one proton of CH_2=), 5.62 (s, 1 H, one proton of CH_2=), 2.07 (s, 3 H, CH_3); ^{13}C NMR (75.4 MHz, $CDCl_3$) δ 198.4, 143.7, 137.7, 131.9, 129.3, 128.1, 127.0, 18.6; IR (neat) 1657, 1448, 1332, 1200, 1176, 1033, 1017 cm^{-1}; MS(EI) *m/z* 146 (M^+, 3.64), 51 (100).

9. Synthesis of oct-1-en-3-one (**2i**, ljx-5-65)



The reaction of oct-1-en-3-ol **1i** (126.6 mg, 1.0 mmol), $Fe(NO_3)_3{\cdot}9H_2O$ (20.6 mg, 0.05 mmol), TEMPO (8.0 mg, 0.05 mmol), and NaCl (3.1 mg, 0.05 mmol) in DCE (4 mL) afforded oct-1-en-3-one[9] **2i** (92.2 mg, 76%) (petroleum ether/diethyl ether = 10/1) as oil: ^{1}H NMR (300 MHz, $CDCl_3$) δ 6.33 (dd, J_1 = 17.6 Hz, J_2 = 10.4 Hz, 1 H, one proton of CH_2=), 6.19 (dd, J_1 = 17.6 Hz, J_2 = 0.8 Hz, 1 H, one proton of CH_2=), 5.79 (dd, J_1 = 10.4 Hz, J_2 = 0.8 Hz, 1 H, CH=), 2.55 (t, J = 7.5 Hz, 2 H, CH_2), 1.68-1.52 (m, 2 H), 1.40-1.20 (m, 4 H, 2 × CH_2), 0.87 (t, J = 6.8 Hz, 3 H, CH_3); ^{13}C NMR (75.4 MHz, $CDCl_3$) δ 201.1, 136.6, 127.8, 39.6, 31.4, 23.6, 22.4, 13.8; IR (neat) 2958, 2932, 1683, 1616, 1466, 1402, 1187, 1132, 1078 cm^{-1}; MS(EI) *m/z* 125 (M^+-H, 1.42), 43 (100).

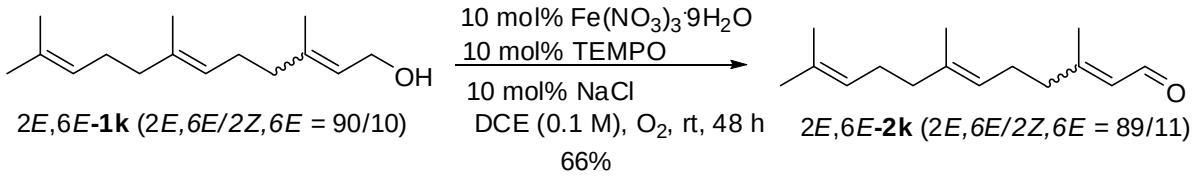
10. Synthesis of non-1-en-3-one (**2j**, ljx-5-83)



The reaction of non-1-en-3-ol **1j** (143.8 mg, 1.0 mmol), $Fe(NO_3)_3{\cdot}9H_2O$ (20.5 mg, 0.05 mmol), TEMPO (7.8 mg, 0.05 mmol), and NaCl (3.1 mg, 0.05 mmol) in DCE (4 mL) afforded non-1-en-3-one[10] **2j** (119.3 mg, 86%) (petroleum ether/diethyl ether =

10/1) as oil: ^{1}H NMR (300 MHz, $CDCl_3$) δ 6.34 (dd, J_1 = 17.6 Hz, J_2 = 10.4 Hz, 1 H, one proton of CH_2=), 6.19 (dd, J_1 = 17.6 Hz, J_2 = 1.1 Hz, 1 H, one proton of CH_2=), 5.79 (dd, J_1 = 10.4 Hz, J_2 = 1.1 Hz, 1 H, CH=), 2.56 (t, J = 7.5 Hz, 2 H, CH_2), 1.68-1.50 (m, 2 H, CH_2), 1.40-1.20 (m, 6 H, 3 × CH_2), 0.86 (t, J = 6.5 Hz, 3 H, CH_3); ^{13}C NMR (75.4 MHz, $CDCl_3$) δ 201.1, 136.6, 127.8, 39.6, 31.6, 28.9, 23.9, 22.4, 14.0; IR (neat) 2957, 2931, 2859, 1702, 1685, 1617, 1467, 1402, 1260, 1082, 1014 cm^{-1}; MS(EI) *m/z* 140 (M^+, 0.65), 70 (100).

11. Synthesis of farnesal (2*E,6E*-**2k**, ljx-5-41)

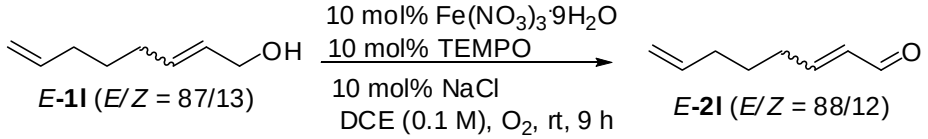


2*E*,6*E*-**1k** (2*E,6E/2Z,6E* = 90/10) → 2*E*,6*E*-**2k** (2*E,6E/2Z,6E* = 89/11)

The reaction of farnesol 2*E,*6*E*-**1k** (223.1 mg, 1.0 mmol, 2*E,*6*E*-**1k** /2*Z,*6*E*-**1k** = 90/10), $Fe(NO_3)_3{\cdot}9H_2O$ (40.0 mg, 0.1 mmol), TEMPO (15.5 mg, 0.1 mmol), and NaCl (5.5 mg, 0.1 mmol) in DCE (10 mL) afforded farnesal[11] 2*E,*6*E*-**2k** (138.4 mg, 66%, 2*E,*6*E*-**2k** /2*Z,*6*E*-**2k** = 89:11) (petroleum ether (30-60 ^{o}C)/diethyl ether = 10/1) as oil: ^{1}H NMR (300 MHz, $CDCl_3$) (2*E,*6*E*-**2k**): δ 10.01 (d, J = 8.1 Hz, 1 H, CHO), 5.90 (d, J = 7.8 Hz, 1 H, CH=), 5.20-5.00 (m, 2 H, 2 × CH=), 2.30-2.12 (m, 6 H), 2.10-1.95 (m, 4 H), 1.75-1.55 (m, 9 H); the following signals are discernible for （2*Z,*6*E*）-**2k**: 9.93(d, J = 8.1 Hz, 1 H, CHO), 2.61(t, J =7.5 Hz, 2 H, CH_2); ^{13}C NMR (75.4 MHz, $CDCl_3$) (2*E,*6*E*-**2k**): δ 190.9, 163.5, 136.2, 131.1, 127.0, 123.7, 122.1, 40.2, 39.2, 26.2, 25.3, 25.1, 17.3, 17.2, 15.7; the following signals are discernible for 2*Z,*6*E*-**2k**: 190.4,

163.4, 136.3, 131.4, 123.7, 122.8, 40.5, 32.2, 31.6, 26.1, 25.3, 22.9, 17.2; IR (neat) 2916, 2726, 1675, 1443, 1379, 1194, 1120 cm^{-1}; MS(EI) *m/z* 220 (M^+, 0.79), 41 (100).

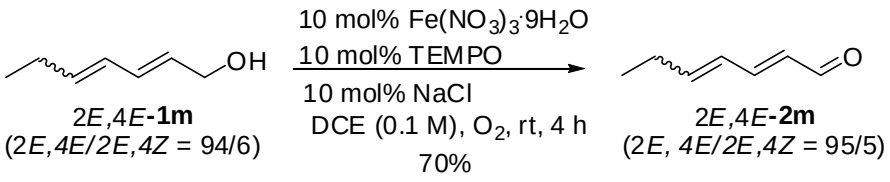
12. Synthesis of (*E*)-octa-2,7-dienal (*E*-**2l**, ljx-5-76)



E-**1l** (*E*/*Z* = 87/13) → *E*-**2l** (*E*/*Z* = 88/12)

The reaction of (*E*)-octa-2,7-dienol *E*-**1l** (125.4 mg, 1.0 mmol), $Fe(NO_3)_3{\cdot}9H_2O$ (40.6 mg, 0.1 mmol), TEMPO (15.2 mg, 0.1 mmol), and NaCl (6.0 mg, 0.1 mmol) in DCE (10 mL) afforded (*E*)-octa-2,7-dienal[12] *E*-**2l** (84.4 mg, 72%, *E*/*Z* = 88/12) (petroleum ether/diethyl ether = 10/1) as oil: ^{1}H NMR (300 MHz, $CDCl_3$) δ 9.51 (d, *J* = 8.1 Hz, 1 H, CHO), 6.86 (dt, J_1 = 15.5 Hz, J_2 = 6.8 Hz,1 H, CH=), 6.13 (ddt, J_1 = 15.9 Hz, J_2 = 8.1 Hz, J_3 = 1.2 Hz, 1 H, CH=), 5.88-5.70 (m, 1 H, CH=), 5.10-4.98 (m, 2 H, CH_2=), 2.36 (q, *J* = 6.9 Hz, 2 H, CH_2), 2.12 (q, *J* = 7.2 Hz, 2 H, CH_2), 1.70-1.52 (m, 2 H, CH_2); the following signals are discernible for *Z*-**2l**: 10.07 (d, *J* = 8.1 Hz, 1 H, CHO), 6.63 (dt, J_1 = 11.4 Hz, J_2 = 8.1 Hz,1 H, CH=), 6.02-5.95 (m, 1 H, CH=), 2.63 (q, *J* = 7.8 Hz, 2 H); ^{13}C NMR (75.4 MHz, $CDCl_3$) δ 193.8, 158.2, 137.4, 132.9, 115.3, 32.8, 31.7, 26.7; IR (neat) 2733, 1692, 1639, 1134, 1102 cm^{-1}; MS(EI) *m/z* 124 (M^+, 0.88), 55 (100).

13. Synthesis of (2*E*,4*E*)-hepta-2,4-dienal (2*E*,4*E*-**2m**, ljx-9-180)

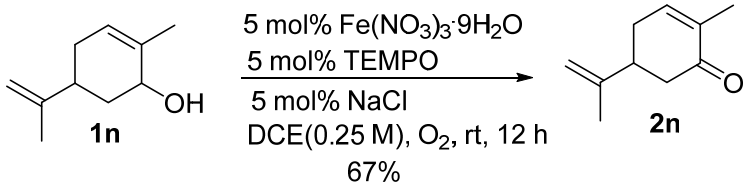


2*E*,4*E*-**1m** (2*E*,4*E*/2*E*,4*Z* = 94/6)

2*E*,4*E*-**2m** (2*E*, 4*E*/2*E*,4*Z* = 95/5)

The reaction of (2*E*,4*E*)-hepta-2,4-dienol 2*E*,4*E*-**1m** (111.6 mg, 1.0 mmol, 2*E*,4*E*-**1m**/2*E*,4*Z*-**1m** = 94/6), $Fe(NO_3)_3{\cdot}9H_2O$ (40.5 mg, 0.1 mmol), TEMPO (15.6 mg, 0.1 mmol), and NaCl (5.8 mg, 0.1 mmol) in DCE (10 mL) afforded (2*E*,4*E*)-hepta-2,4-dienal[13] 2*E*,4*E*-**2m** (77.2 mg, 70%, (2*E*,4*E*)-**2m**/(2*E*,4*Z*)-**2m** = 95/5) (petroleum ether/diethyl ether = 10/1) as oil: ^{1}H NMR (300 MHz, $CDCl_3$) δ 9.57 (d, *J* = 8.1 Hz, 1 H, CHO), 7.20-7.00 (m, 1 H, CH=), 6.45-6.35 (m, 2 H, two 2 × CH=), 6.11 (dd, J_1 = 15.3 Hz, J_2 = 8.1 Hz, 1 H, CH=), 2.35-2.20 (m, 2 H), 1.11 (t, *J* = 7.4 Hz, 3 H, CH_3); the following signal are discernible for 2*Z*,4*E*-**2m**: 9.64 (d, *J* = 8.1 Hz, 1 H, CHO); ^{13}C NM`R (75.4 MHz, $CDCl_3$) δ 193.6, 152.6, 148.2, 129.7, 127.3, 25.8, 12.3; IR (neat) 2742, 1683, 1641, 1462, 1166, 1121, 1107, 1013, 988 cm^{-1}; MS(EI) *m/z* 110 (M^+, 11.95), 81 (100).

14. Synthesis of 2-methyl-5-(prop-1-en-2-yl)cyclohex-2-enone (**2n**, ljx-5-78)

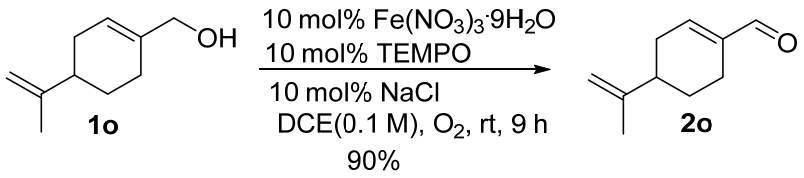


1n **2n**

The reaction of 2-methyl-5-(prop-1-en-2-yl)cyclohex-2-enol **1n** (151.7 mg, 1.0 mmol), $Fe(NO_3)_3{\cdot}9H_2O$ (40.5 mg, 0.1 mmol), TEMPO (15.9 mg, 0.1 mmol), and NaCl (6.0 mg, 0.1 mmol) in DCE (10 mL) afforded 2-methyl-5-(prop-1-en-2-yl)cyclohex-2-enone[14] **2n** (100.2 mg, 67%) (petroleum

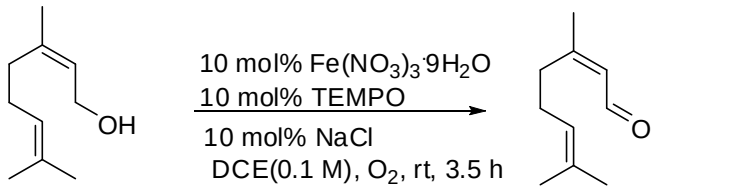
ether/diethyl ether = 10/1) as oil: ^{1}H NMR (300 MHz, $CDCl_3$) δ 6.75-6.68 (m, 1 H, CH=), 4.78 (s, 1 H, one proton of CH_2=), 4.73 (s, 1 H, one proton of CH_2=), 2.71-2.19 (m, 5 H, 2 × CH_2 and 1 × CH), 1.76 (s, 3 H, CH_3), 1.73 (s, 3 H, CH_3); ^{13}C NMR (75.4 MHz, $CDCl_3$) δ 199.6, 146.6, 144.5, 135.3, 110.3, 43.0, 42.3, 31.1, 20.4, 15.6; IR (neat) 1673, 1645, 1434, 1367, 1247, 1110, 1059 cm^{-1}; MS(EI) *m/z* 150 (M^+, 6.79), 41 (100).

15. Synthesis of 4-(prop-1-en-2-yl)cyclohex-1-enecarbaldehyde (**2o**, ljx-5-102)



The reaction of (4-(prop-1-en-2-yl)cyclohex-1-enyl)carbinol **1o** (153.1 mg, 1.0 mmol), $Fe(NO_3)_3·9H_2O$ (40.4 mg, 0.1 mmol), TEMPO (15.6 mg, 0.1 mmol), and NaCl (5.7 mg, 0.1 mmol) in DCE (10 mL) afforded 4-(prop-1-en-2-yl)cyclohex-1-enecarbaldehyde[15] **2o** (129.9 mg, 90%) (petroleum ether/diethyl ether = 10/1) as oil: ^{1}H NMR (300 MHz, $CDCl_3$) δ 9.44 (s, 1 H, CHO), 6.85-6.78 (m, 1 H, CH=), 4.78 (s, 1 H, one proton of CH_2=), 4.73 (s, 1 H, one proton of CH_2=), 2.59-2.40 (m, 2 H), 2.32-1.82 (m, 4 H, 2 × CH_2), 1.76 (s, 3 H, CH_3), 1.50-1.36 (m, 1 H); ^{13}C NMR (75.4 MHz, $CDCl_3$) δ 193.8, 150.5, 148.2, 141.1, 109.4, 40.5, 31.6, 26.2, 21.4, 20.5; IR (neat) 2717, 1683, 1644, 1434, 1165 cm^{-1}; MS(EI) *m/z* 149 (M^+-H, 3.71), 133 (100).

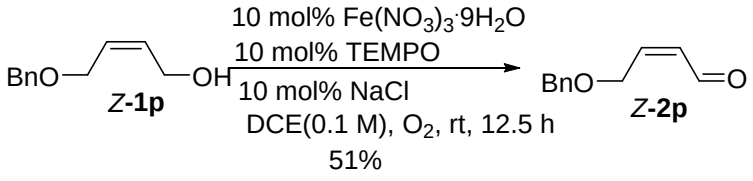
16. Synthesis of Neral (*Z*-**2a**, ljx-5-43)



Z-**1a** (*E*/*Z* =99/1) *Z*-**2a** (*E*/*Z* = 96/4)

The reaction of nerol *Z*-**1a** (154.1 mg, 1.0 mmol, *E*/*Z* =99/1), $Fe(NO_3)_3{\cdot}9H_2O$ (40.3 mg, 0.1 mmol), TEMPO (15.9 mg, 0.1 mmol), and NaCl (5.9 mg, 0.1 mmol) in DCE (10 mL) afforded neral[16] *Z*-**2a** (128.2 mg, 87%, *Z*/*E* = 96:4) (petroleum ether/diethyl ether = 10/1) as oil: ^{1}H NMR (300 MHz, $CDCl_3$) δ 9.91 (d, *J* = 8.4 Hz, 1 H, CHO), 5.89 (d, *J* = 8.1 Hz, 1 H, CH=), 5.11 (t, *J* = 7.4 Hz, 1 H, CH=), 2.60 (t, *J* = 7.4 Hz, 2 H, CH_2), 2.25 (q, *J* = 7.4 Hz, 2 H, CH_2), 2.00 (s, 3 H, CH_3), 1.70 (s, 3 H, CH_3), 1.61 (s, 3 H, CH_3); ^{13}C NMR (75.4 MHz, $CDCl_3$) δ 190.4, 163.4, 133.3, 128.3, 121.8, 32.2, 26.6, 25.2, 24.7, 17.3; IR (neat) 2729, 1674, 1631, 1445, 1377, 1154, 1095, 1042 cm^{-1}; MS(EI) *m*/*z* 152 (M^+, 1.05), 41 (100).

17. Synthesis of (*Z*)-4-(benzyloxy)but-2-enal (*Z*-**2p**, ljx-5-85)



Z-**1p** *Z*-**2p**

The reaction of (*Z*)-4-(benzyloxy)but-2-enol *Z*-**1p** (176.6 mg, 1.0 mmol), $Fe(NO_3)_3{\cdot}9H_2O$ (40.9 mg, 0.1 mmol), TEMPO (15.5 mg, 0.1 mmol), and NaCl (6.0 mg, 0.1 mmol) in DCE (10 mL) afforded (*Z*)-4-(benzyloxy)but-2-enal[17] *Z*-**2p** (84.1 mg, 51%, *Z*/*E* >99/1) (petroleum ether/diethyl ether = 10/1) as oil: ^{1}H NMR (300 MHz, $CDCl_3$) δ 10.05 (d, *J* = 6.9 Hz, 1 H, CHO), 7.42-7.28 (m, 5 H, Ar-H), 6.64 (dt,

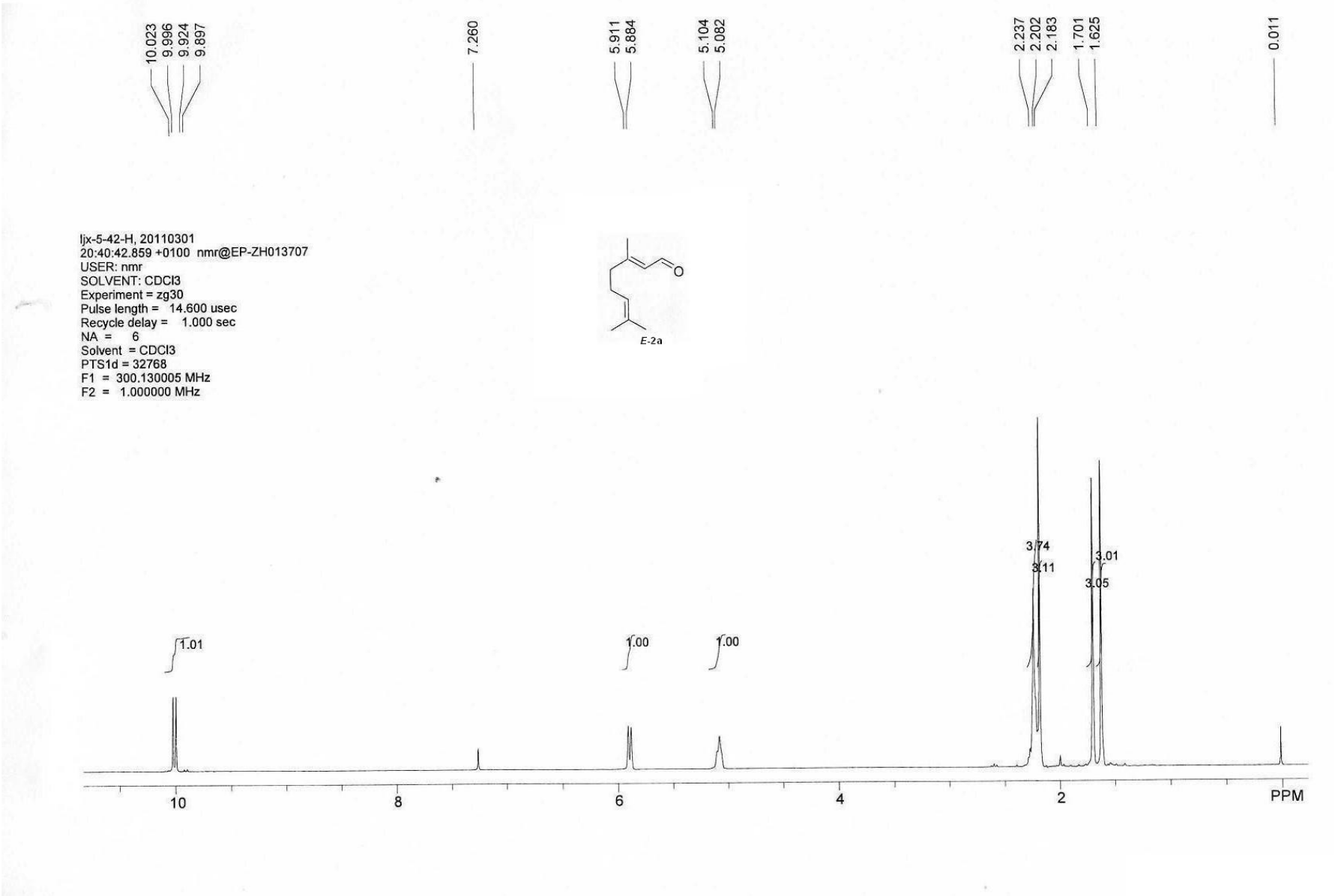
J_1 = 11.7 Hz, J_2 = 5.7 Hz, 1 H, CH=), 6.06 (ddt, J_1 = 11.7 Hz, J_2 = 6.6 Hz, J_3 = 2.1 Hz, 1 H, CH=), 4.59 (s, 2 H, CH_2), 4.53 (dd, J_1 = 5.6 Hz, J_2 = 2.0 Hz, 2 H, CH_2); ^{13}C NMR (75.4 MHz, $CDCl_3$) δ 191.4, 147.5, 137.3, 129.7, 128.5, 128.0, 127.8, 73.0, 66.9; IR (neat) 1683, 1496, 1454, 1366, 1216, 1073 cm^{-1}; MS(EI) *m/z* 176 (M^+, 0.99), 133 (91).

Notes and References

1. Velusamy, S.; Ahamed, M.; Punniyamurthy, T. *Org. Lett.* **2004**, *6*, 4821.
2. Baillargeon, V. P.; Stille, J. K. *J. Am. Chem. Soc.* **1986**, *108*, 452.
3. Seo, S.-M.; Kim, J.; Kim, E.; Park, H.-M.; Kim, Y.-J.; Park, I.-K. J. Agric. Food Chem. 2010, 58, 1823.
4. Tichotová, L.; Matoušová, E.; Špulák, M.; Kuneš, J.; Votruba, I.; Buchta, V.; Pour, M. *Bioorg. Med. Chem. Lett.* **2011**, *21*, 6062.
5. Kon, Y.; Yazawa, H.; Usui, Y.; Sato, K. *Chem. Asian J.* **2008**, *3*, 1642.
6. Das, B.; Banerjee, J.; Chowdhury, N.; Majhi, A.; Holla, H. *Synlett* **2006**, 1879.
7. Snowden, R. L.; Linder, S. M.; Muller, B. L.; Schulte-Elte, K. H. *Helv. Chim. Acta* **1987**, *70*, 1858.
8. DeShong, P.; Dicken, C. M.; Staib, R. R.; Freyer, A. J.; Weinreb, S. M. *J. Org. Chem.* **1982**, *47*, 4397.
9. Venturello, C.; Gambaro, M. J. Org. Chem. 1991, 56, 5924.
10. Nakahira, H.; Ryu, I.; Masanobu, I.; Oku, Y.; Ogawa, A.; Kambe, N.; Sonoda, N.;

Murai, S. *J. Org. Chem.* **1992**, *57*, 17.

11. (a) Altman, L. J.; Kowerski, R. C.; Laungani, D. R. *J. Am. Chem. Soc.* **1978**, *100*, 6174; (b) Leal, W. S.; Kuwahara, Y.; Suzuki, T.; Kurosa, K. *Agric. Biol. Chem.* **1989**, *53*, 2703.
12. Hartikka, A.; Arvidsson, P. I. *J. Org. Chem.* **2007**, *72*, 5874.
13. Binns, F.; Hodgetts, K. J.; Saengchantara, S. T.; Wallace, T. W.; Wallis, C. J. *Tetrahedron* **1996**, *52*, 3631.
14. Shen, S.-S.; Kartika, V.; Tan, Y. S.; Webster, R. D.; Narasaka, K. *Tetrahedron Lett.* **2012**, *53*, 986.
15. Zou, Y.; Wang, Q.; Goeke, A. *Chem. Eur. J.* **2008**, *14*, 5335.
16. Singh, S.; Jayaram, R. V. *Synth. Commun.* **2012**, *42*, 299.
17. Hoover, J. M.; Stahl, S. *J. Am. Chem. Soc.* **2011**, *133*, 16901.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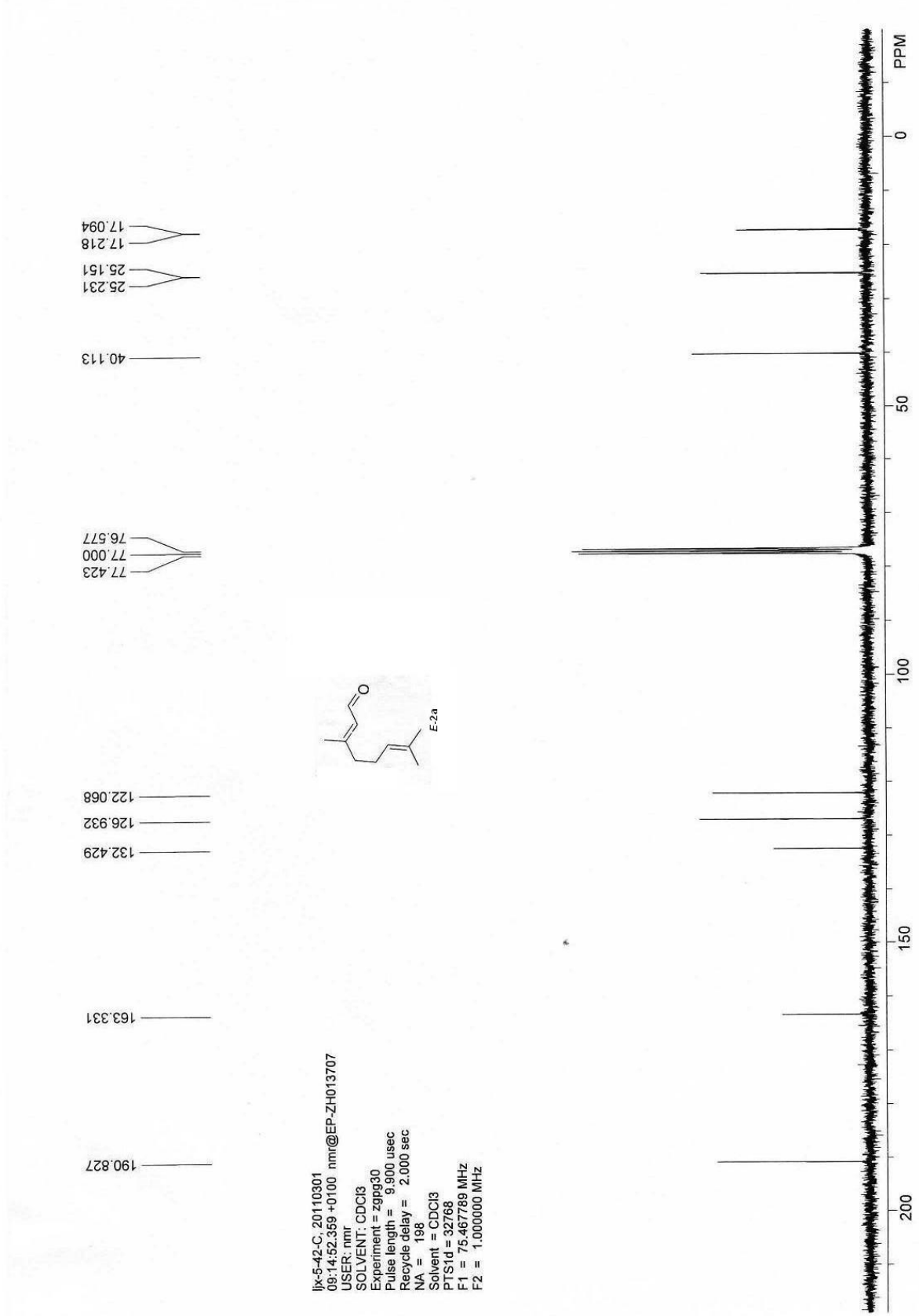
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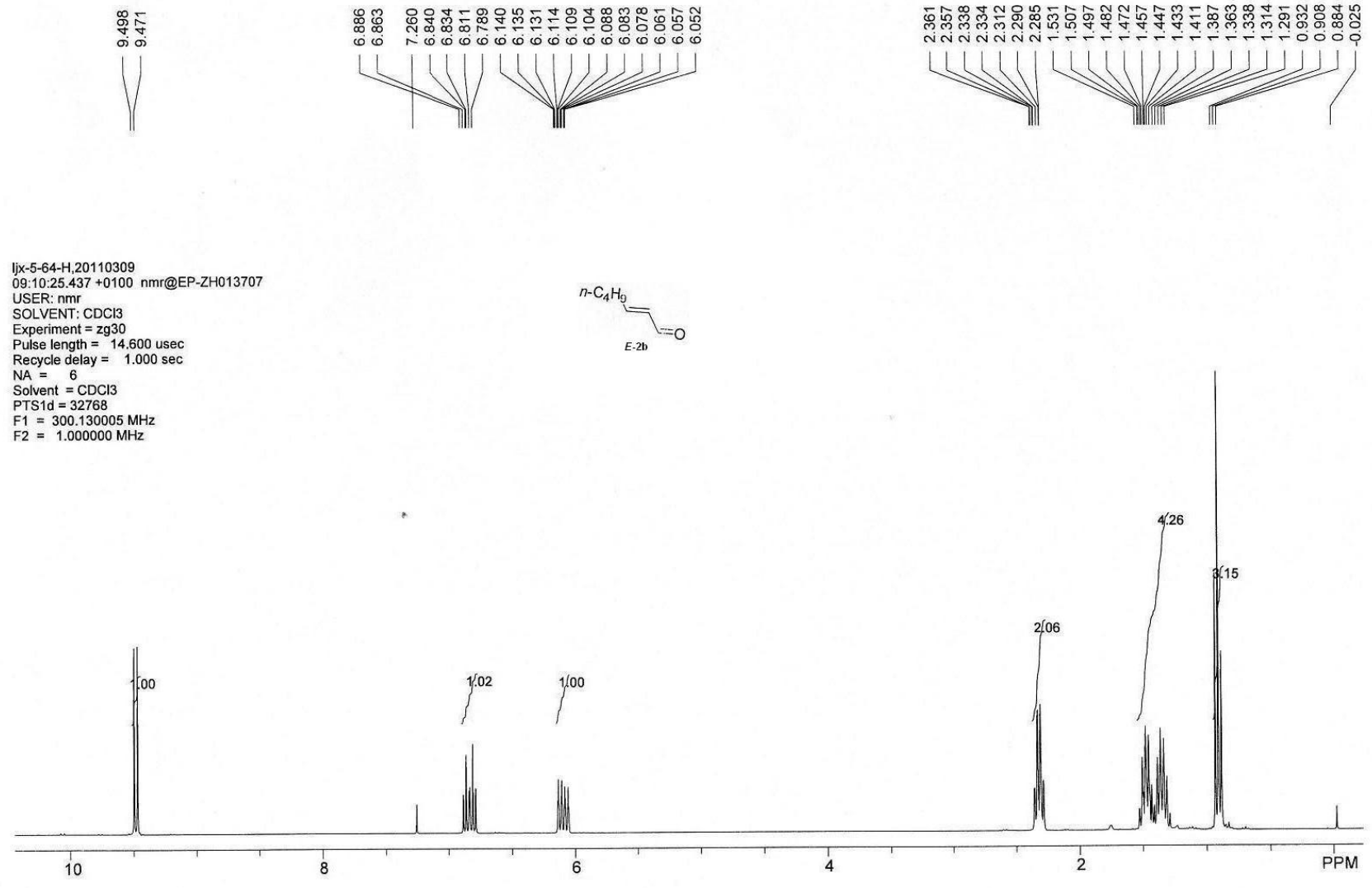
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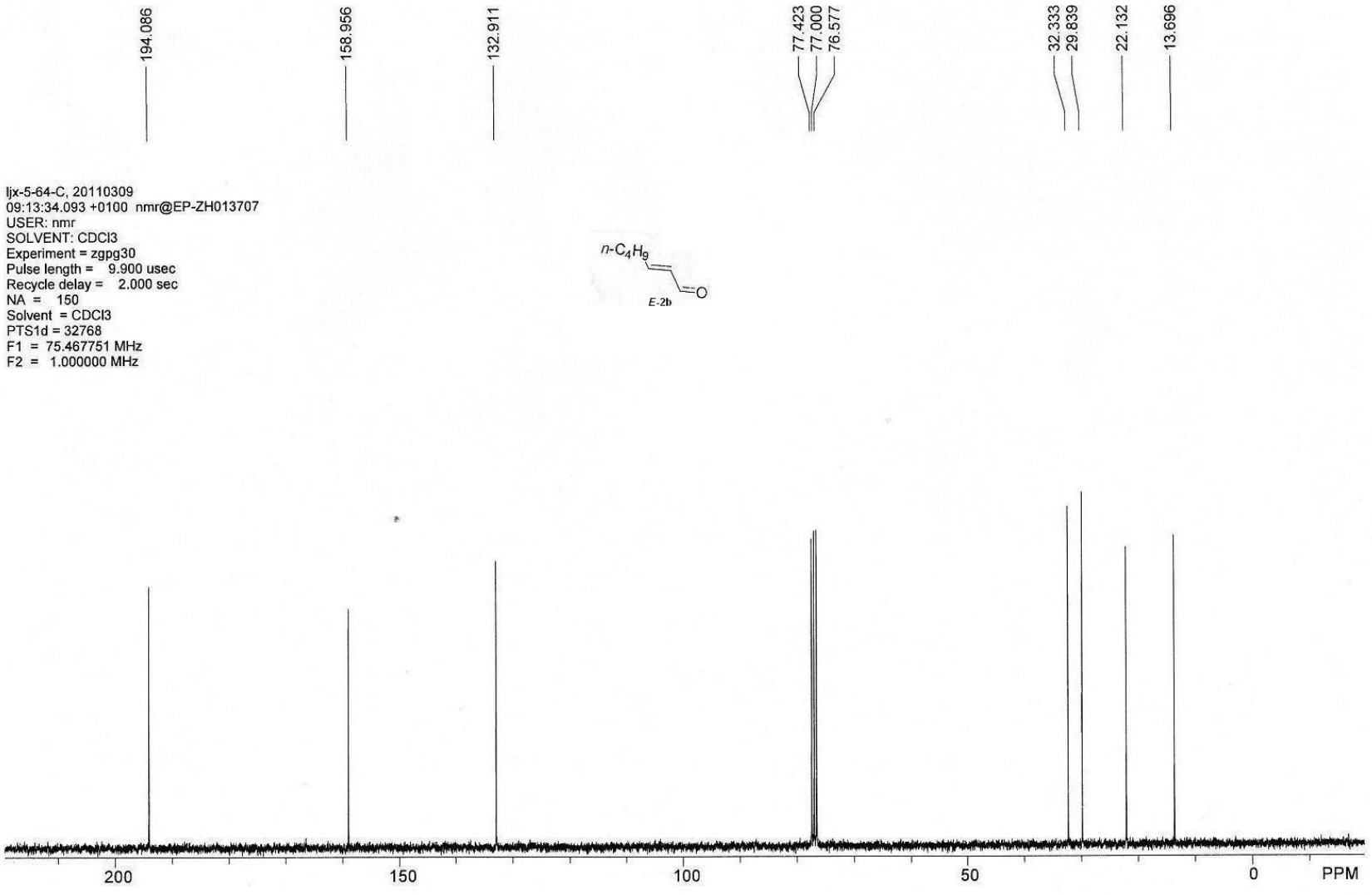
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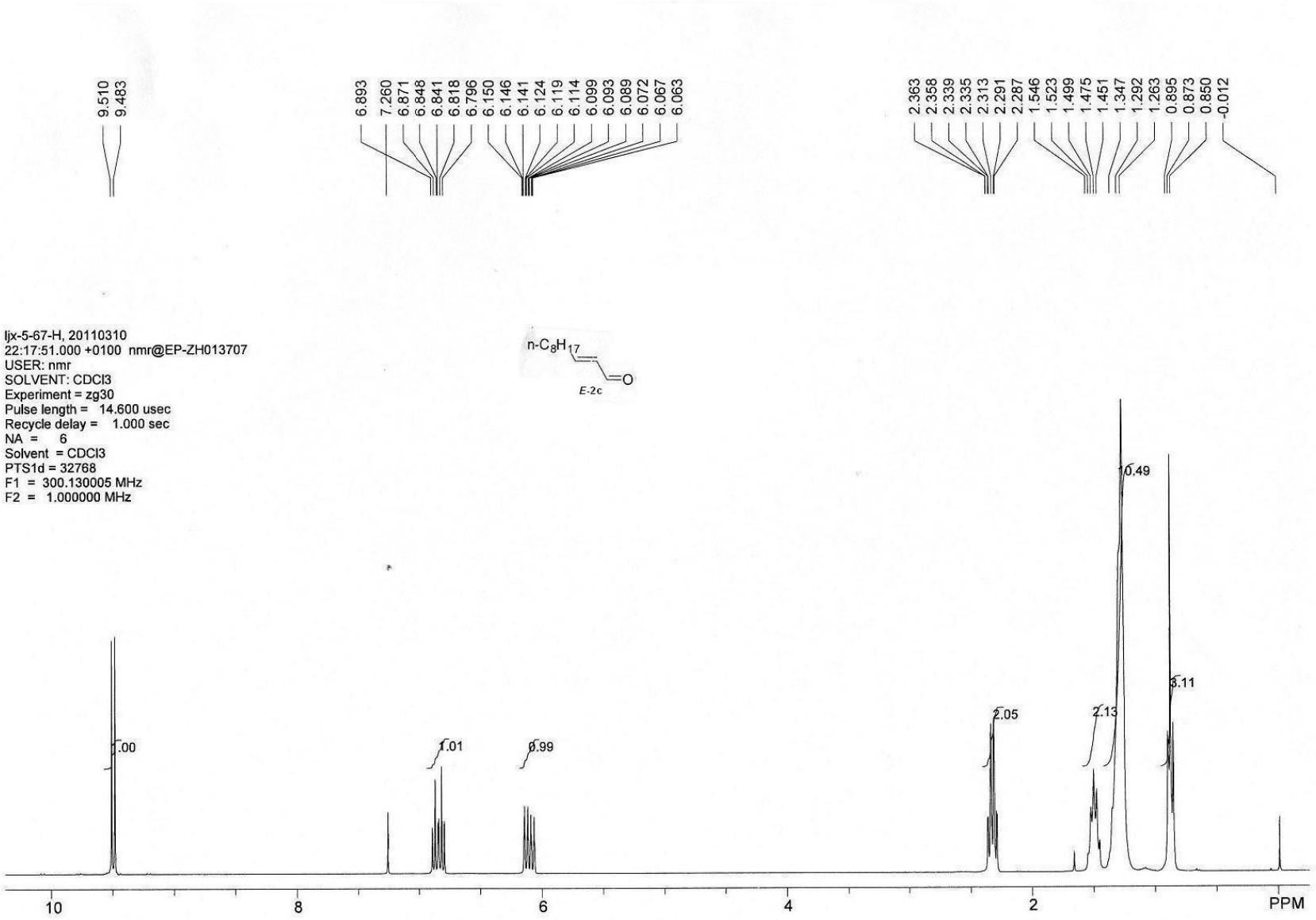
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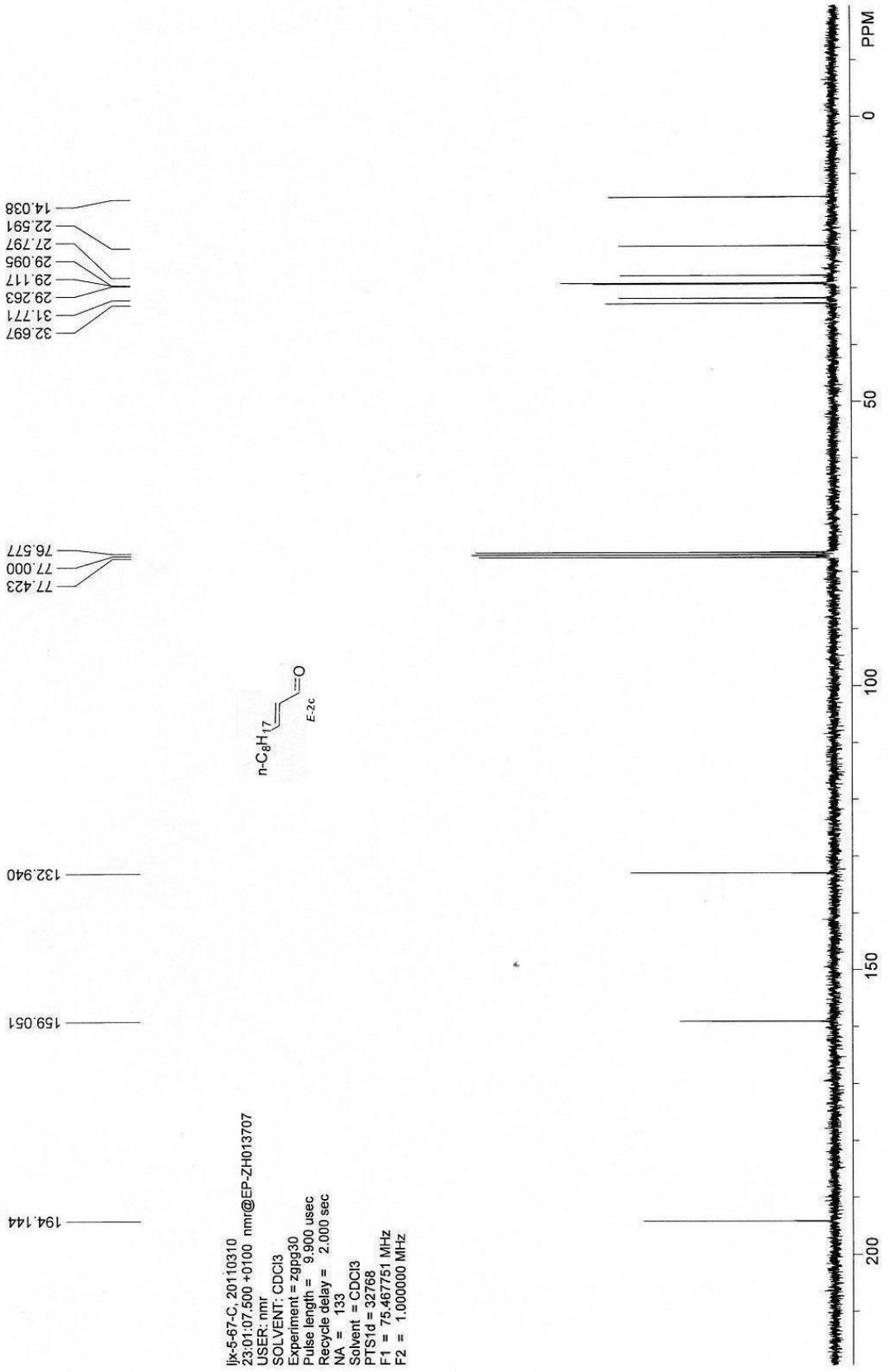
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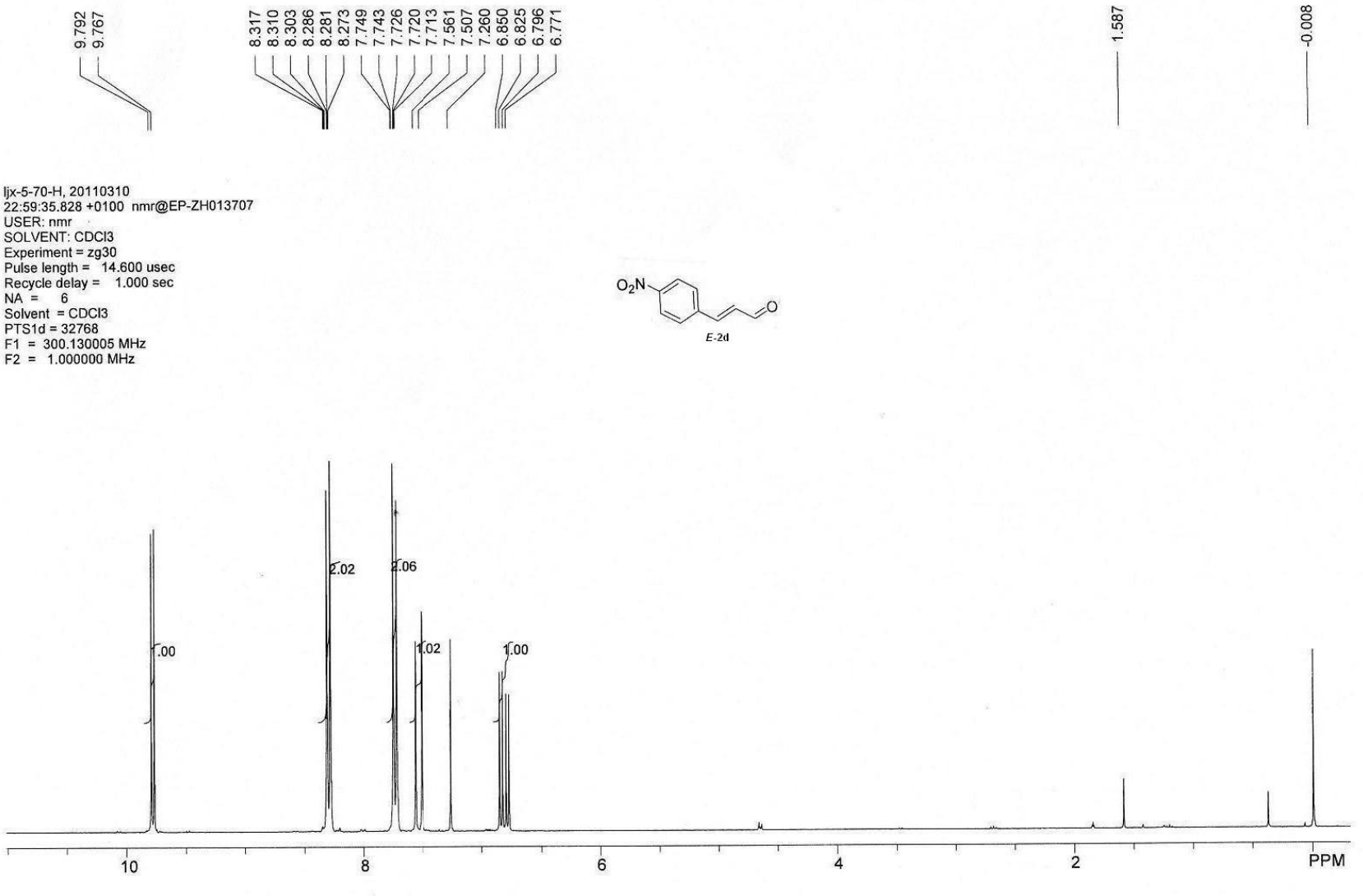
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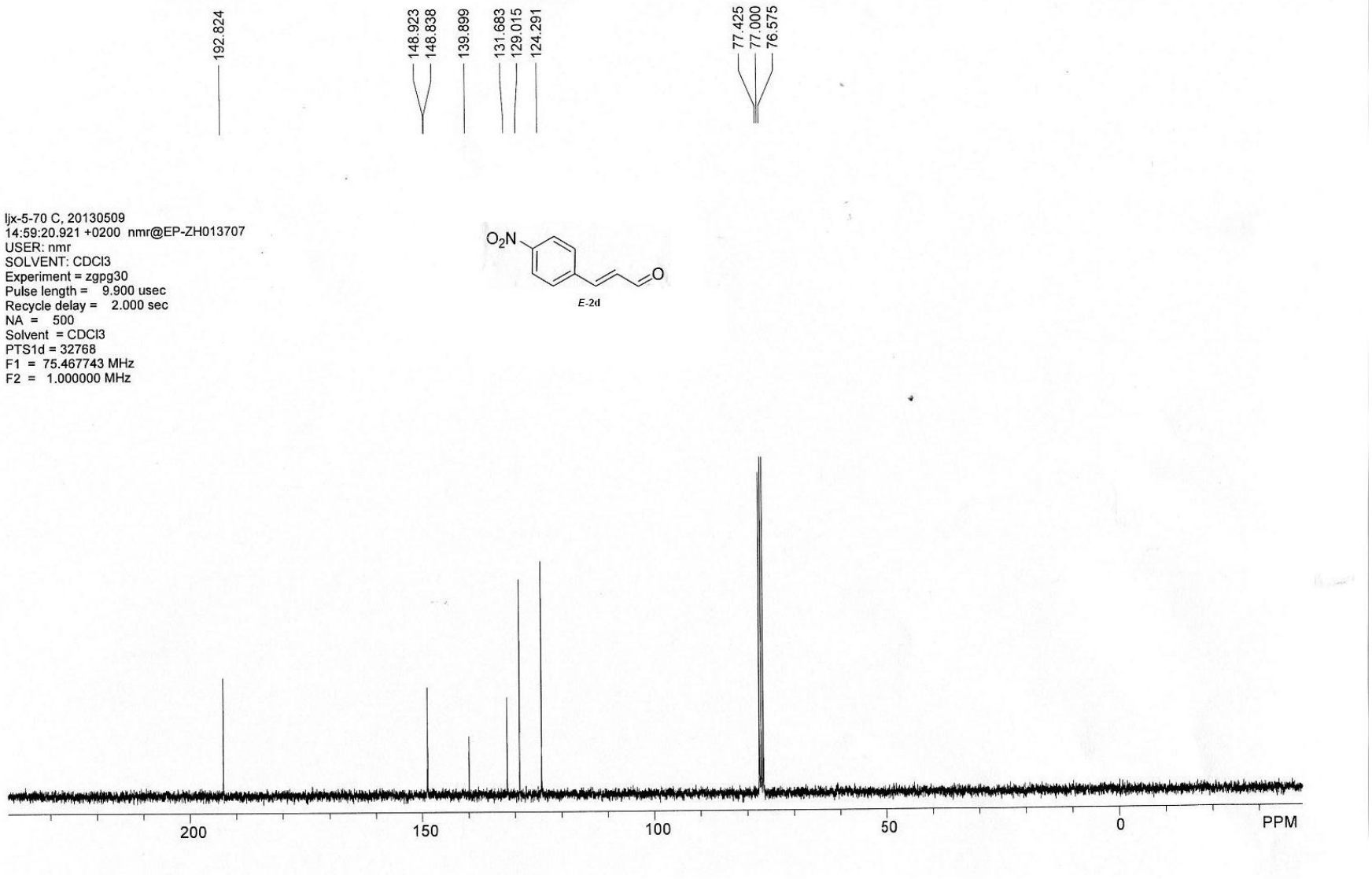
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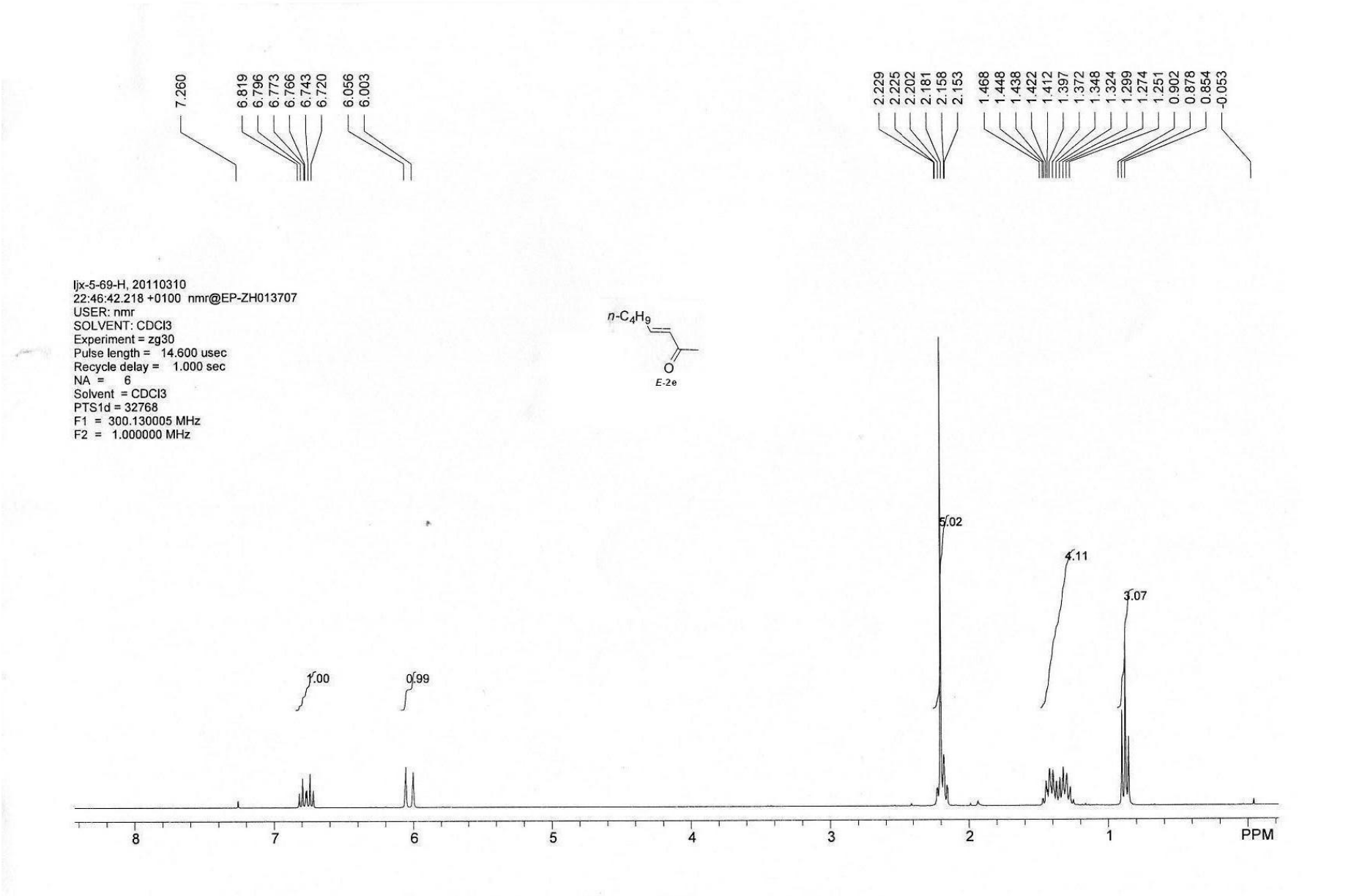
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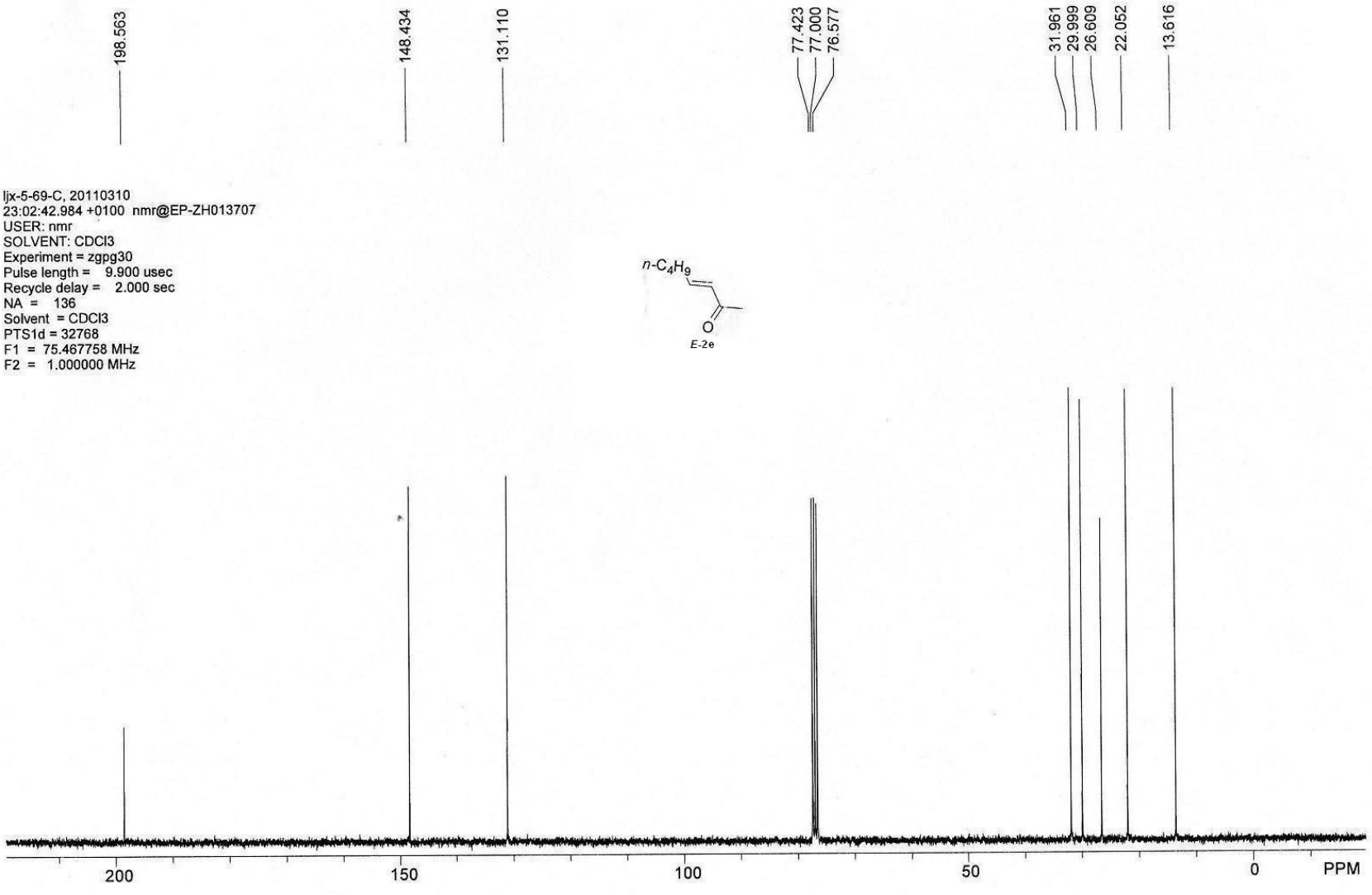
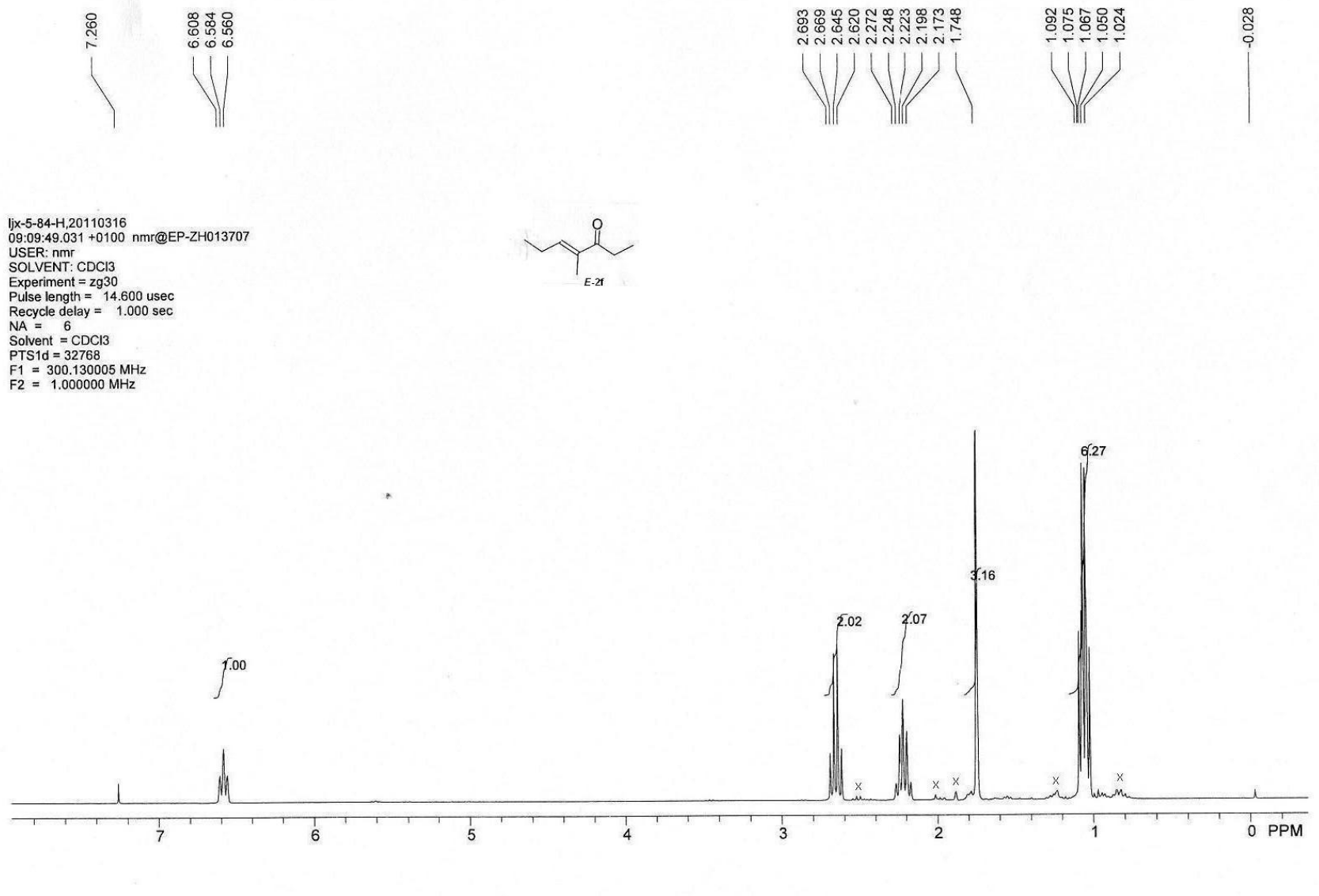
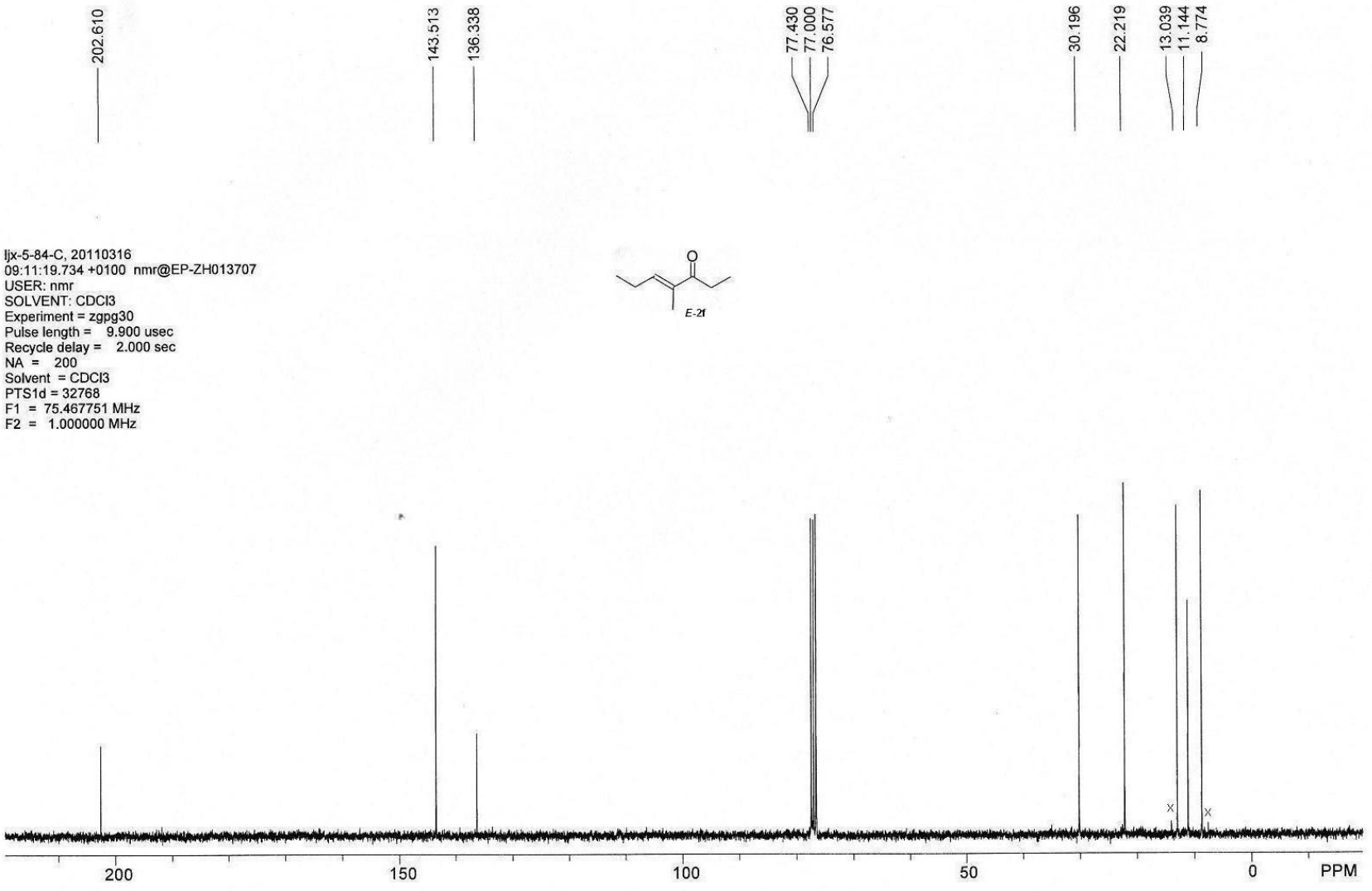
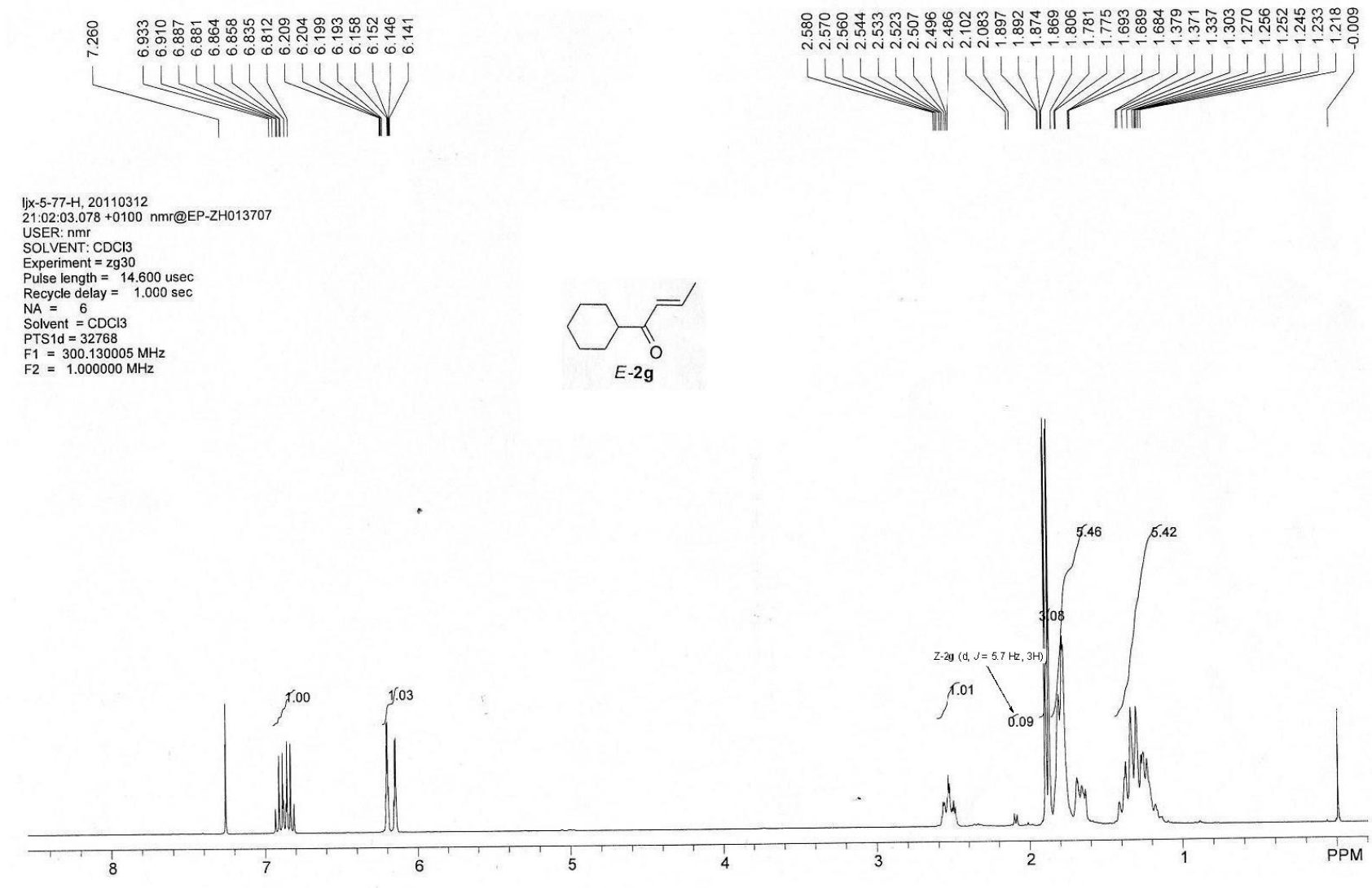
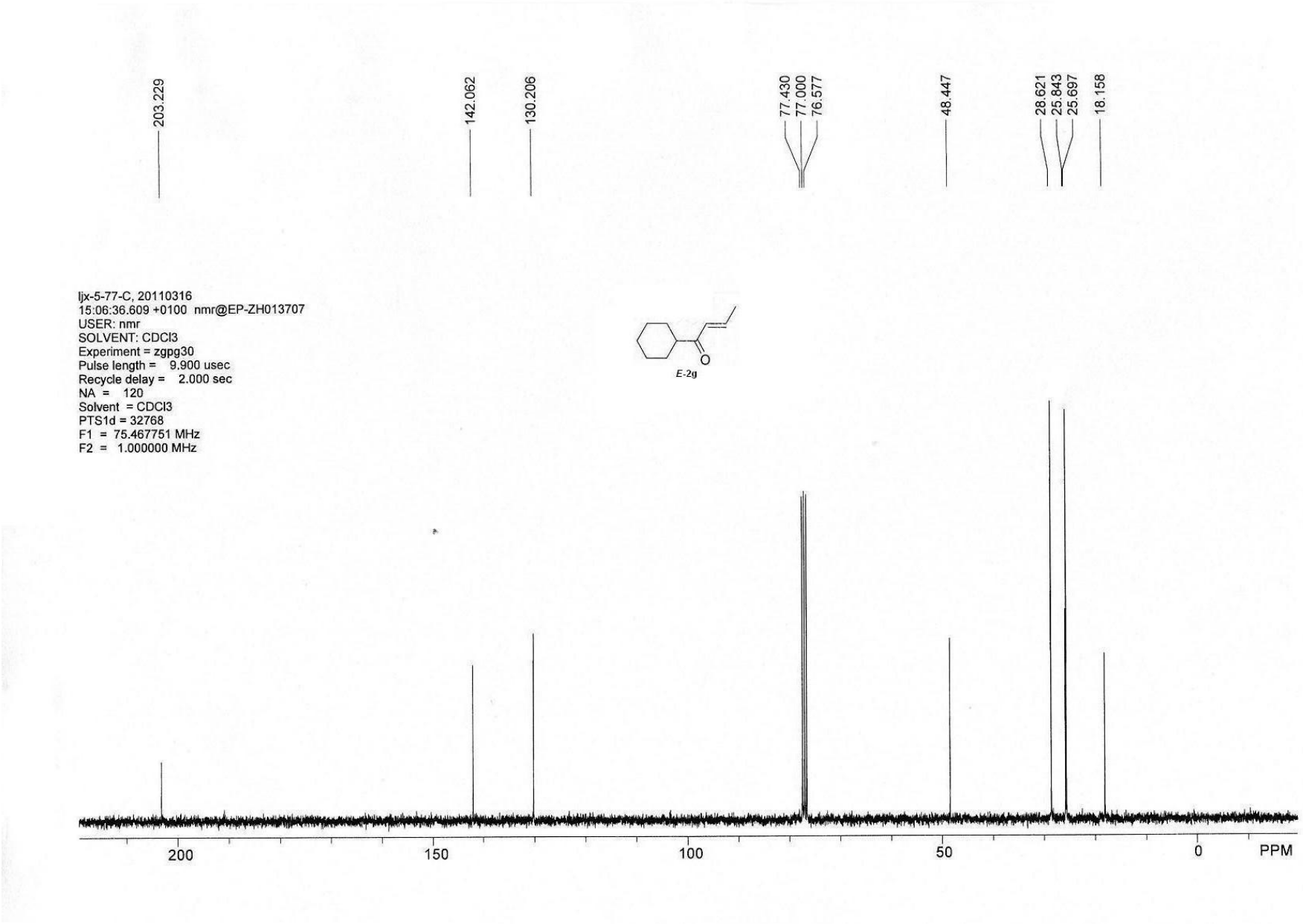
1.299
1.274
1.251
0.902
0.878
0.854
-0.053
ljx-5-69-H, 20110310
22:46:42.218 +0100 nmr@EP-ZH013707
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.600 usec
Recycle delay = 1.000 sec
NA = 6
Solvent = CDCl3
PTS1d = 32768
F1 = 300.130005 MHz
F2 = 1.000000 MHz
n-C4H9
O
E-2e
1.00
0.99
5.02
4.11
3.07
8
7
6
5
4
3
2
1
PPM

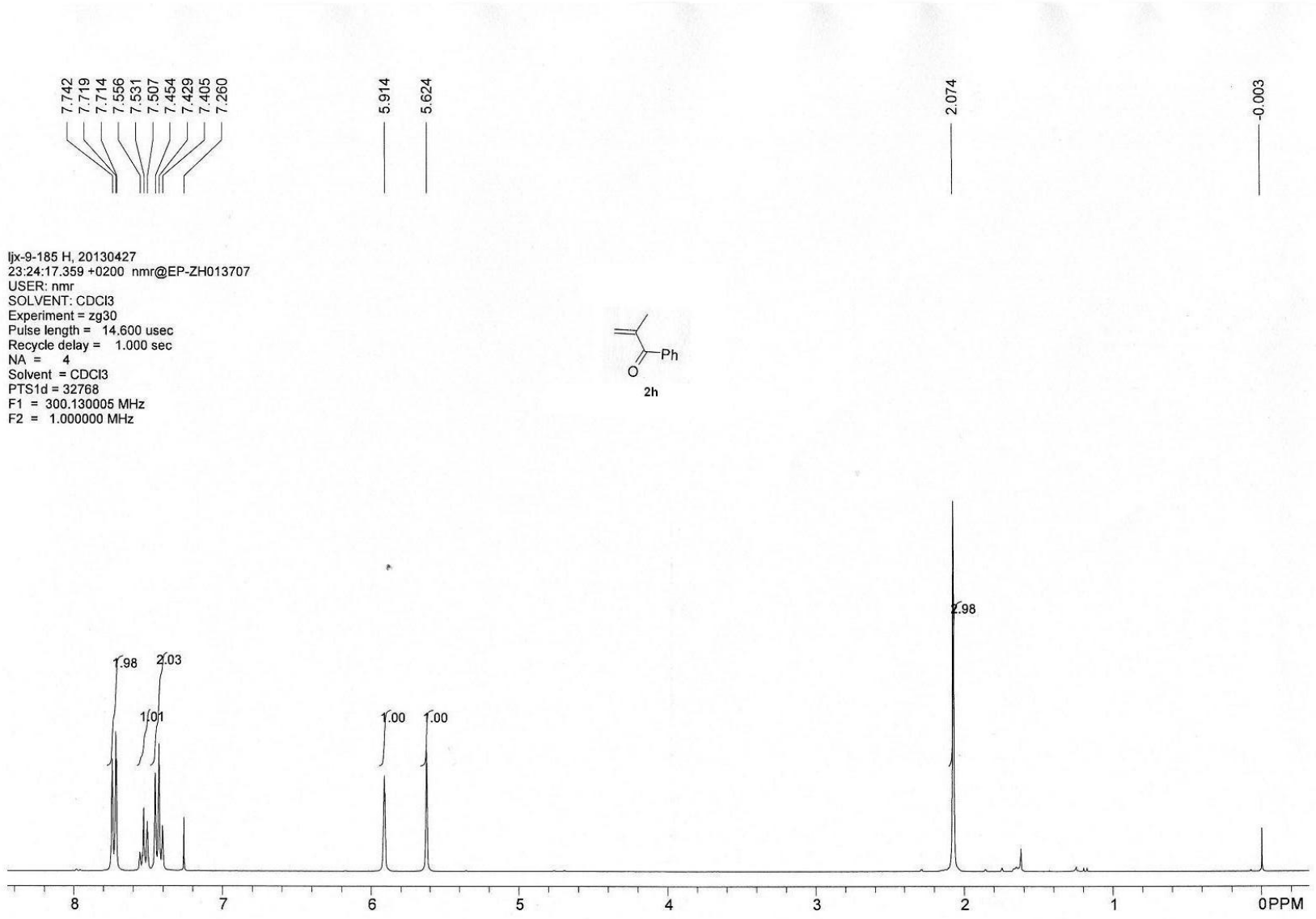

198.563
148.434
131.110
77.423
77.000
76.577
31.961
29.999
26.609
22.052
13.616
ljx-5-69-C, 20110310
23:02:42.984 +0100 nmr@EP-ZH013707
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.900 usec
Recycle delay = 2.000 sec
NA = 136
Solvent = CDCl3
PTS1d = 32768
F1 = 75.467758 MHz
F2 = 1.000000 MHz
n-C4H9
O
E-2e
200
150
100
50
0
PPM

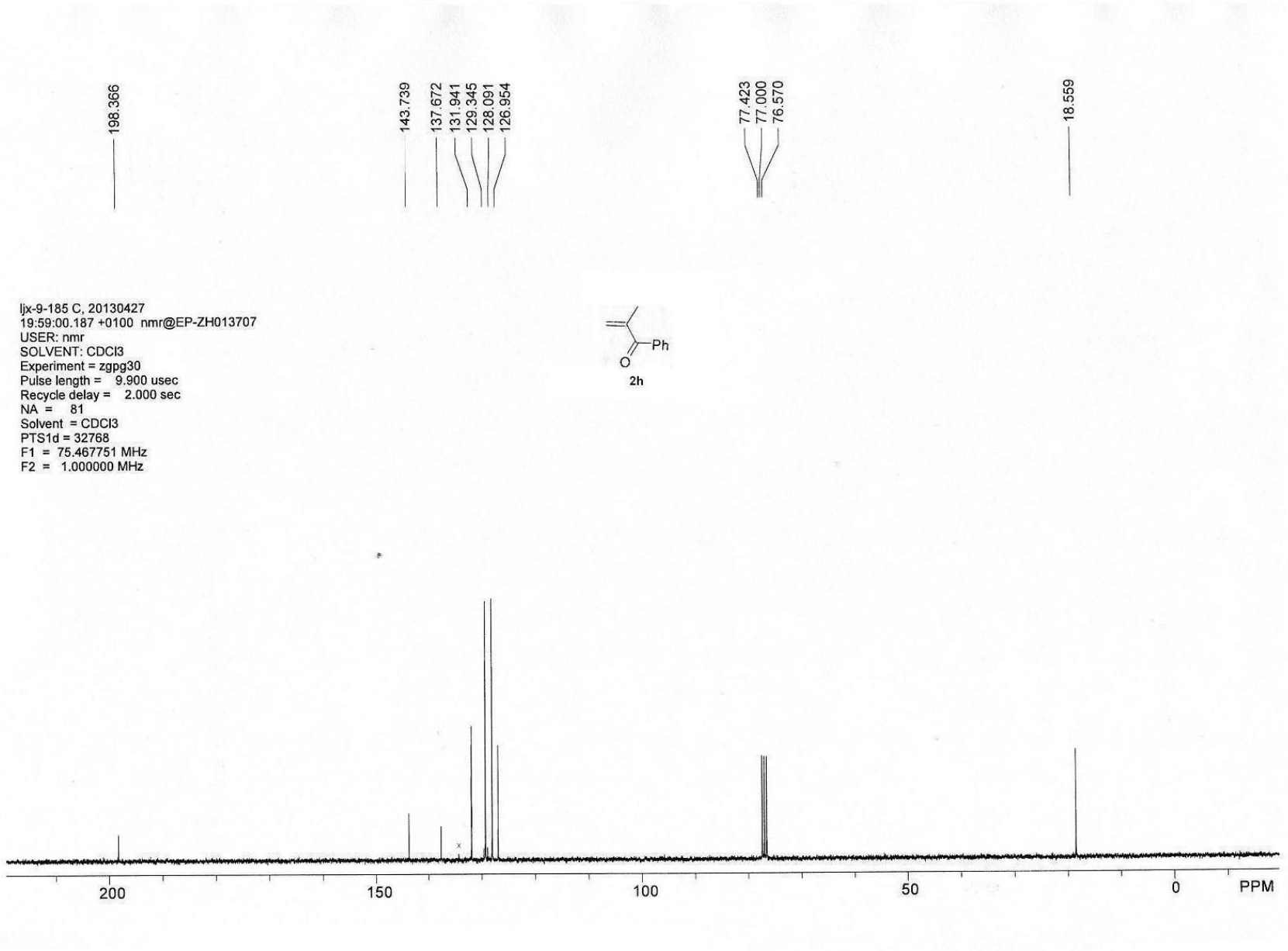

7.260
6.608
6.584
6.560
2.693
2.669
2.645
2.620
2.272
2.248
2.223
2.198
2.173
1.748
1.092
1.075
1.067
1.050
1.024
-0.028
ljx-5-84-H,20110316
09:09:49.031 +0100 nmr@EP-ZH013707
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.600 usec
Recycle delay = 1.000 sec
NA = 6
Solvent = CDCl3
PTS1d = 32768
F1 = 300.130005 MHz
F2 = 1.000000 MHz
O
E-2f
1.00
2.02
2.07
3.16
6.27
7
6
5
4
3
2
1
0 PPM

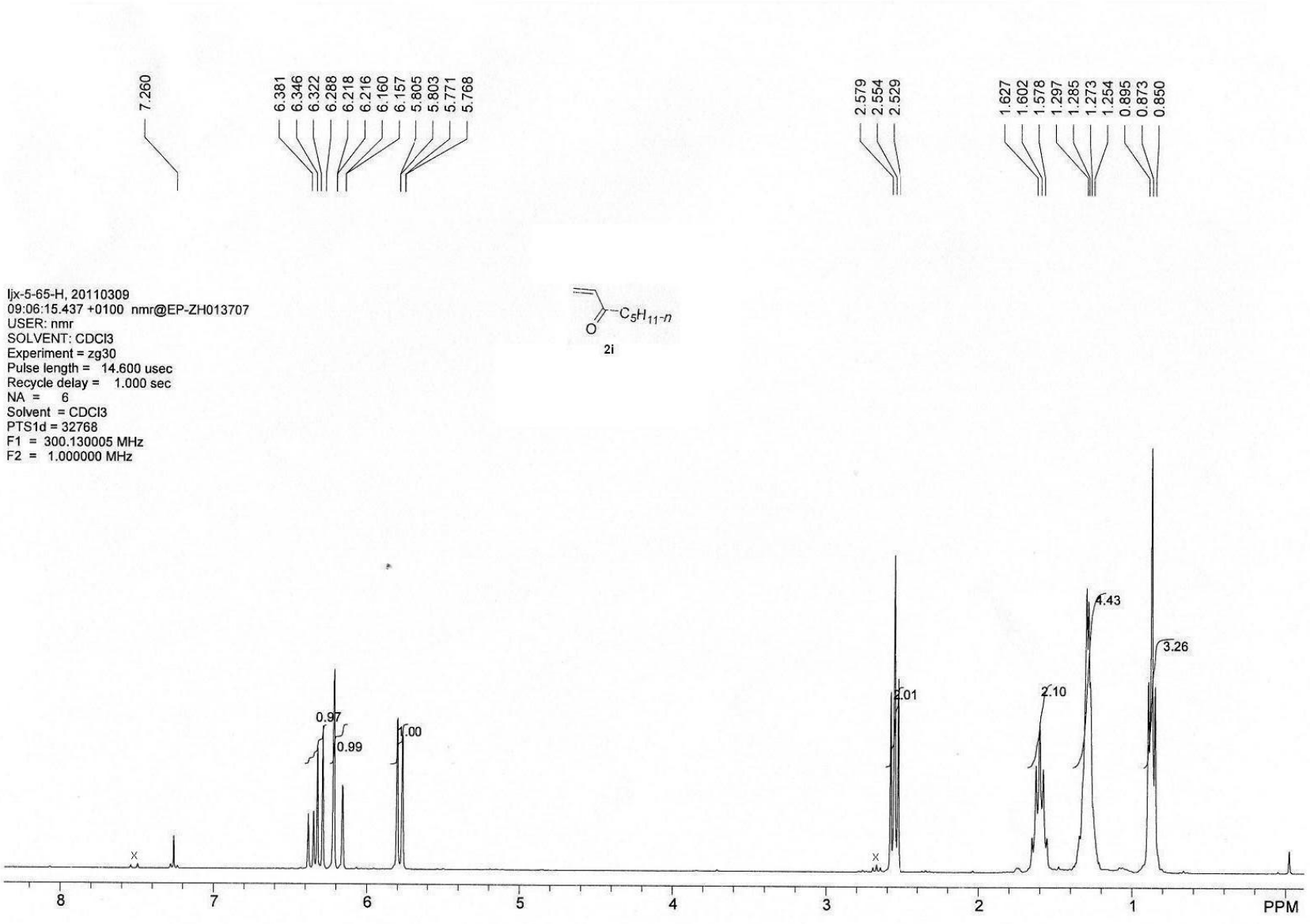

202.610
143.513
136.338
77.430
77.000
76.577
30.196
22.219
13.039
11.144
8.774
ljx-5-84-C, 20110316
09:11:19.734 +0100 nmr@EP-ZH013707
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.900 usec
Recycle delay = 2.000 sec
NA = 200
Solvent = CDCl3
PTS1d = 32768
F1 = 75.467751 MHz
F2 = 1.000000 MHz
O
E-2f
X
X
200
150
100
50
0
PPM

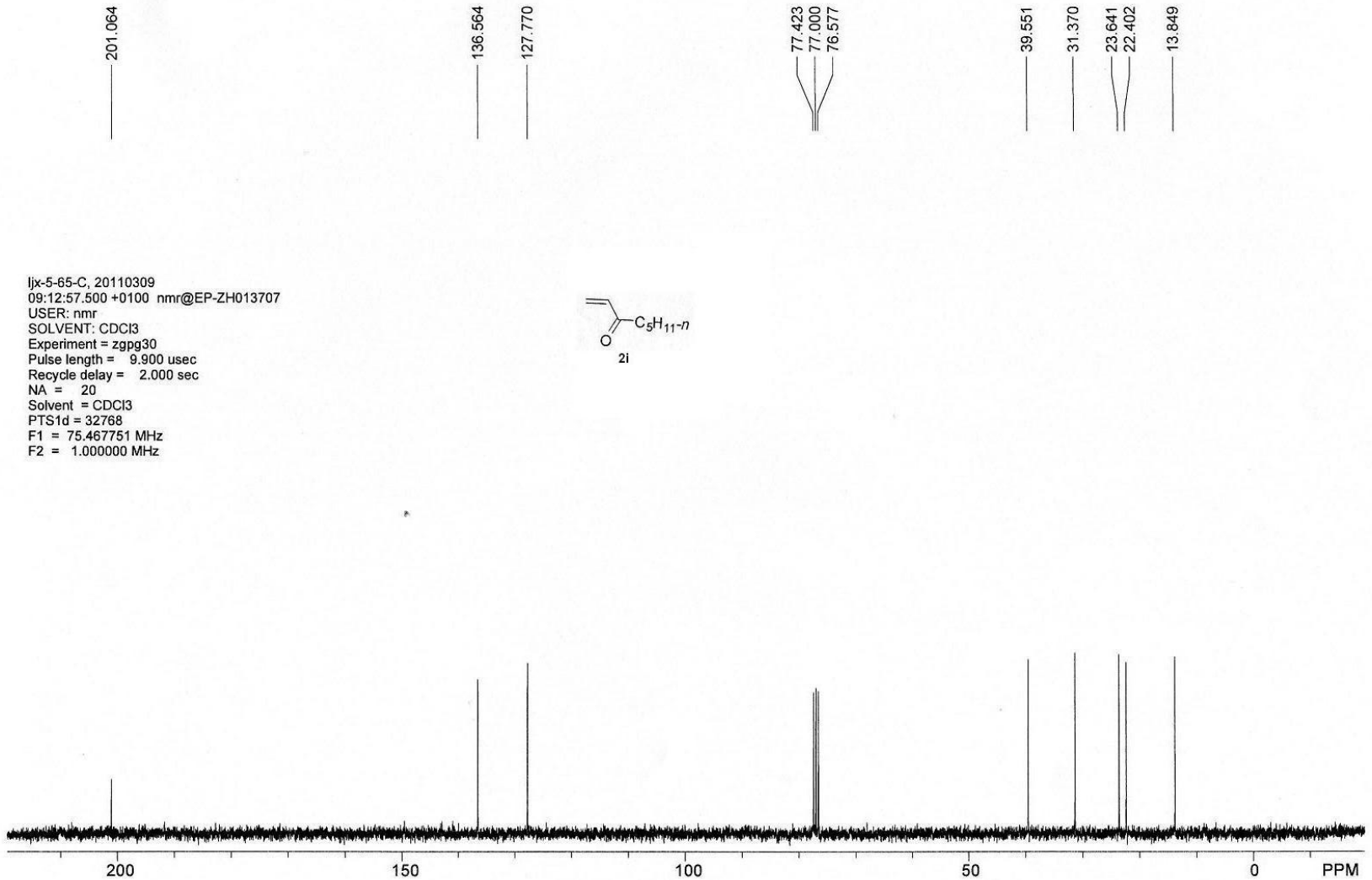

7.260
6.933
6.910
6.887
6.881
6.864
6.858
6.835
6.812
6.209
6.204
6.199
6.193
6.158
6.152
6.146
6.141
2.580
2.570
2.560
2.544
2.533
2.523
2.507
2.496
2.486
2.102
2.083
1.897
1.892
1.874
1.869
1.806
1.781
1.775
1.693
1.689
1.684
1.379
1.371
1.337
1.303
1.270
1.256
1.252
1.245
1.233
1.218
-0.009
ljx-5-77-H, 20110312
21:02:03.078 +0100 nmr@EP-ZH013707
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.600 usec
Recycle delay = 1.000 sec
NA = 6
Solvent = CDCl3
PTS1d = 32768
F1 = 300.130005 MHz
F2 = 1.000000 MHz
O
E-2g
1.00
1.03
1.01
0.09
3.08
5.46
5.42
Z-2g (d, J = 5.7 Hz, 3H)
8
7
6
5
4
3
2
1
PPM

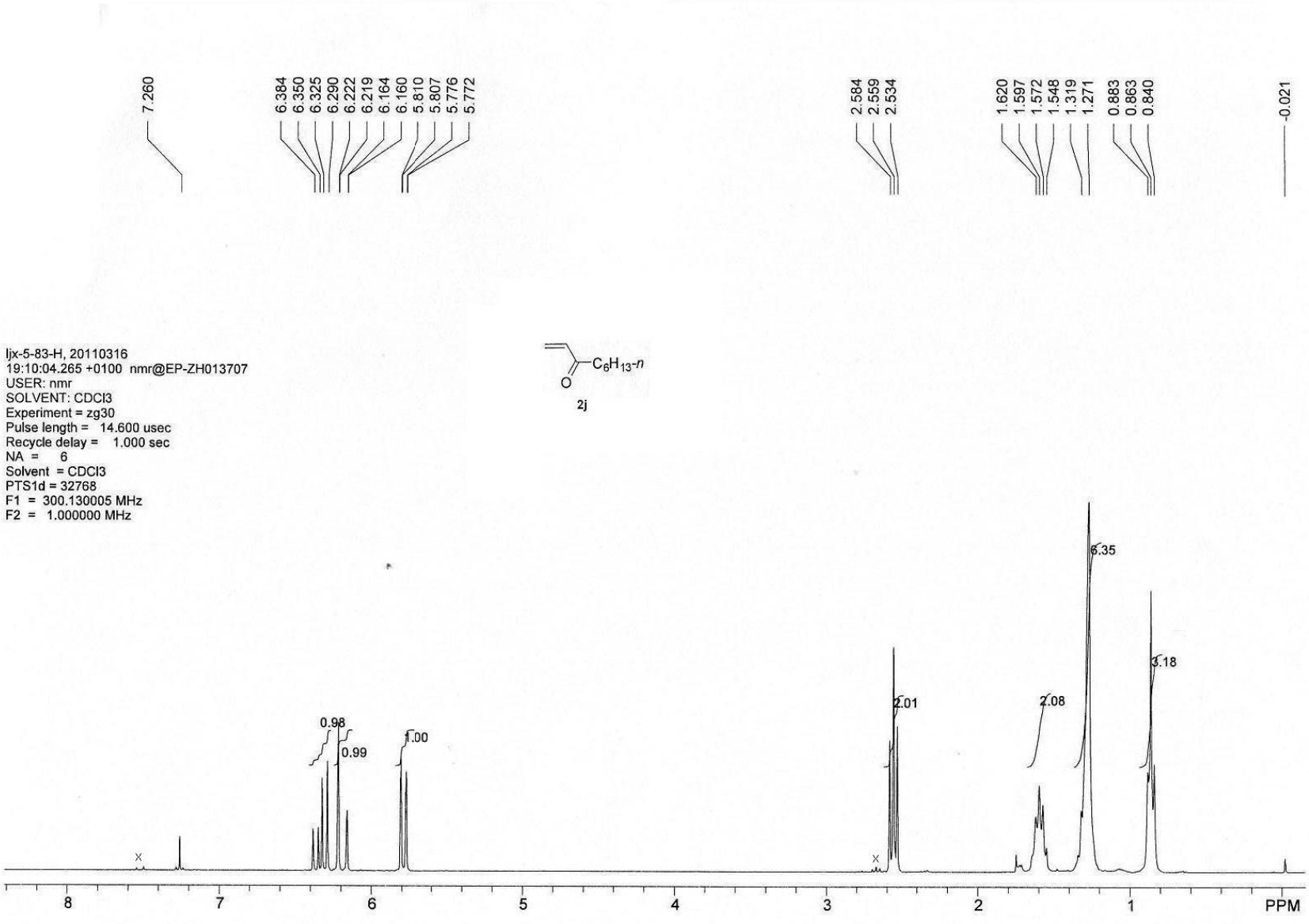

203.229
142.062
130.206
77.430
77.000
76.577
48.447
28.621
25.843
25.697
18.158
ljx-5-77-C, 20110316
15:06:36.609 +0100 nmr@EP-ZH013707
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.900 usec
Recycle delay = 2.000 sec
NA = 120
Solvent = CDCl3
PTS1d = 32768
F1 = 75.467751 MHz
F2 = 1.000000 MHz
O
E-2g
200
150
100
50
0
PPM

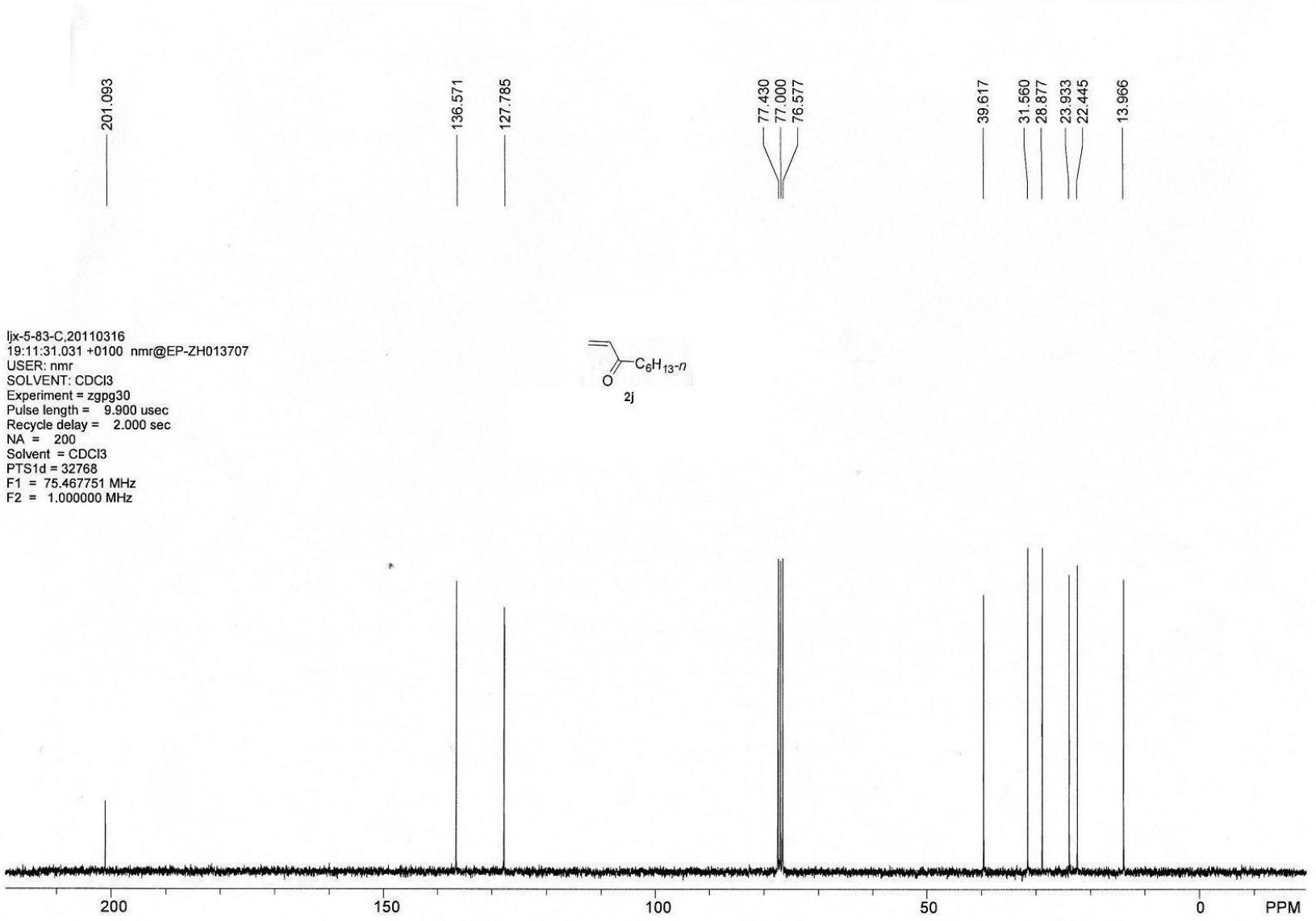

7.742
7.719
7.714
7.556
7.531
7.507
7.454
7.429
7.405
7.260
5.914
5.624
2.074
-0.003
ljx-9-185 H, 20130427
23:24:17.359 +0200 nmr@EP-ZH013707
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.600 usec
Recycle delay = 1.000 sec
NA = 4
Solvent = CDCl3
PTS1d = 32768
F1 = 300.130005 MHz
F2 = 1.000000 MHz
Ph
O
2h
1.98
2.03
1.01
1.00
1.00
2.98
8
7
6
5
4
3
2
1
0PPM

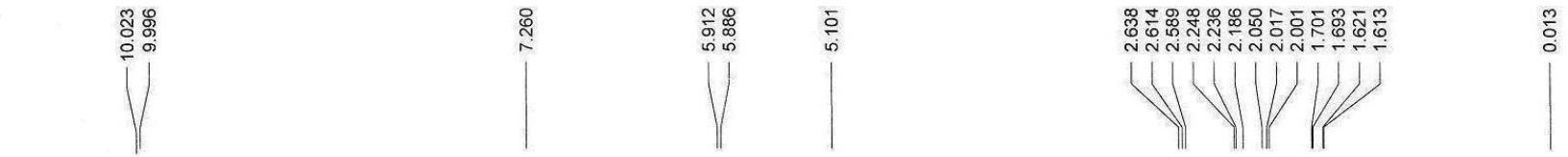

198.366
143.739
137.672
131.941
129.345
128.091
126.954
77.423
77.000
76.570
18.559
ljx-9-185 C, 20130427
19:59:00.187 +0100 nmr@EP-ZH013707
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.900 usec
Recycle delay = 2.000 sec
NA = 81
Solvent = CDCl3
PTS1d = 32768
F1 = 75.467751 MHz
F2 = 1.000000 MHz
Ph
O
2h
200
150
100
50
0
PPM


7.260
6.381
6.346
6.322
6.288
6.218
6.216
6.160
6.157
5.805
5.803
5.771
5.768
2.579
2.554
2.529
1.627
1.602
1.578
1.297
1.285
1.273
1.254
0.895
0.873
0.850
ljx-5-65-H, 20110309
09:06:15.437 +0100 nmr@EP-ZH013707
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.600 usec
Recycle delay = 1.000 sec
NA = 6
Solvent = CDCl3
PTS1d = 32768
F1 = 300.130005 MHz
F2 = 1.000000 MHz
O
C5H11-n
2i
0.97
0.99
1.00
2.01
2.10
4.43
3.26
X
X
8
7
6
5
4
3
2
1
PPM

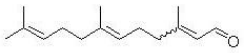

201.064
136.564
127.770
77.423
77.000
76.577
39.551
31.370
23.641
22.402
13.849
ljx-5-65-C, 20110309
09:12:57.500 +0100 nmr@EP-ZH013707
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.900 usec
Recycle delay = 2.000 sec
NA = 20
Solvent = CDCl3
PTS1d = 32768
F1 = 75.467751 MHz
F2 = 1.000000 MHz
C_5H_{11}-n
O
2i
200
150
100
50
0
PPM


7.260
6.384
6.350
6.325
6.290
6.222
6.219
6.164
6.160
5.810
5.807
5.776
5.772
2.584
2.559
2.534
1.620
1.597
1.572
1.548
1.319
1.271
0.883
0.863
0.840
-0.021
ljx-5-83-H, 20110316
19:10:04.265 +0100 nmr@EP-ZH013707
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.600 usec
Recycle delay = 1.000 sec
NA = 6
Solvent = CDCl3
PTS1d = 32768
F1 = 300.130005 MHz
F2 = 1.000000 MHz
C6H13-n
O
2j
0.98
0.99
1.00
2.01
2.08
6.35
3.18
X
X
8
7
6
5
4
3
2
1
PPM

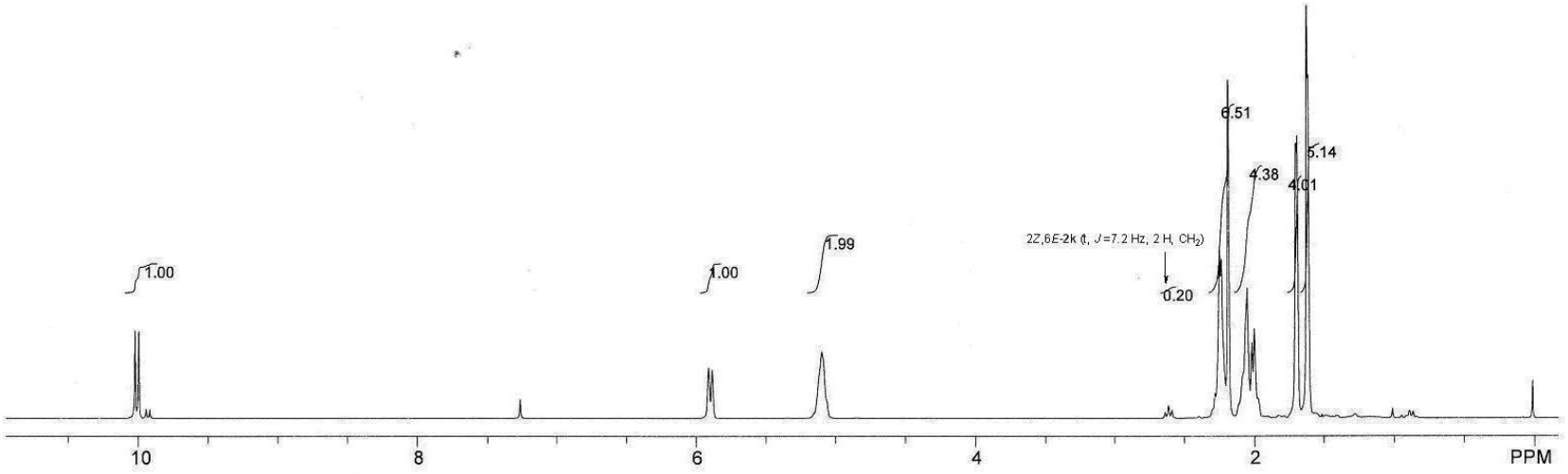

201.093
136.571
127.785
77.430
77.000
76.577
39.617
31.560
28.877
23.933
22.445
13.966
ljx-5-83-C,20110316
19:11:31.031 +0100 nmr@EP-ZH013707
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.900 usec
Recycle delay = 2.000 sec
NA = 200
Solvent = CDCl3
PTS1d = 32768
F1 = 75.467751 MHz
F2 = 1.000000 MHz
O
C6H13-n
2j
200
150
100
50
0
PPM



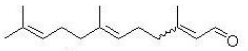
ljx-5-41-H, 20110301
21:08:51.687 +0100 nmr@EP-ZH013707
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.600 usec
Recycle delay = 1.000 sec
NA = 6
Solvent = CDCl3
PTS1d = 32768
F1 = 300.130005 MHz
F2 = 1.000000 MHz



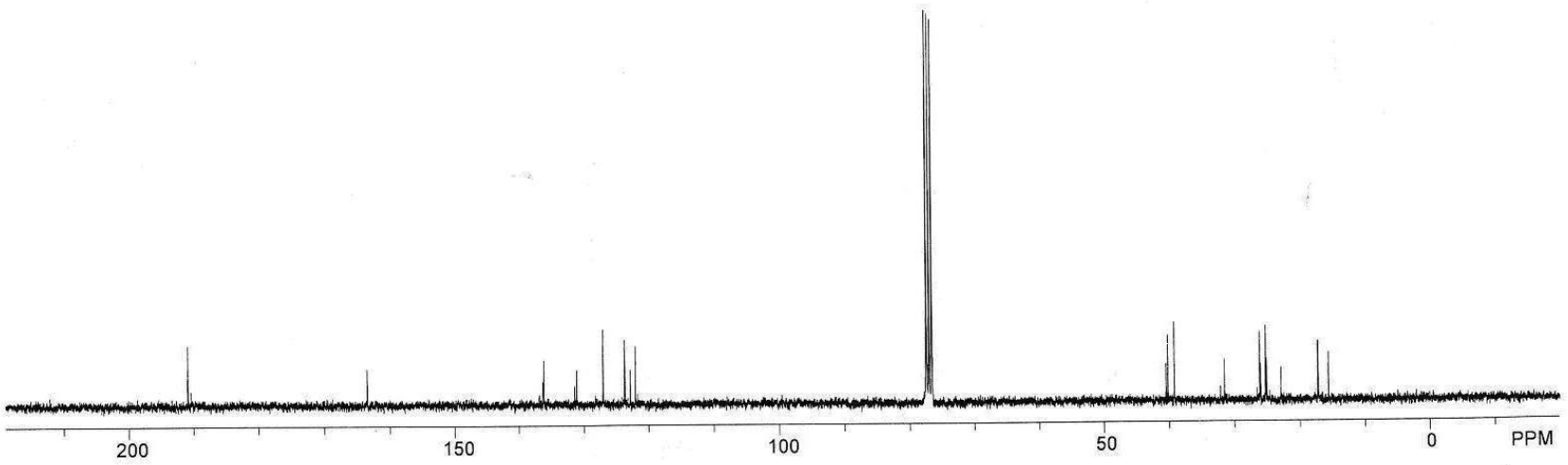
2*E*,6*E*-**2k** (2*E*,6*E*/2*Z*,6*E* = 89/11)

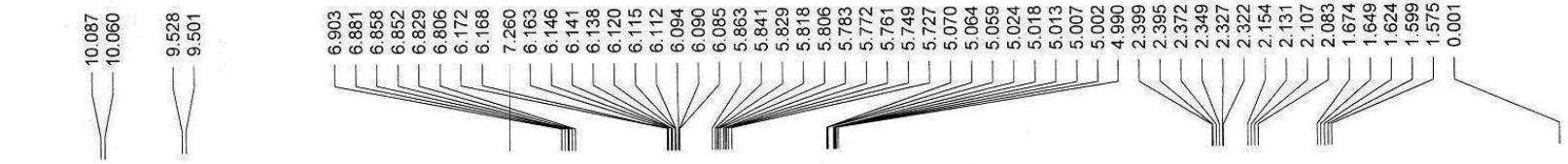


ljx-5-41-C, 20110301
22:05:02.296 +0100 nmr@EP-ZH013707
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.900 usec
Recycle delay = 2.000 sec
NA = 172
Solvent = CDCl3
PTS1d = 32768
F1 = 75.467751 MHz
F2 = 1.000000 MHz

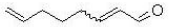


2E,6E-**2k** (*2E,6E*/*2Z,6E* = 89/11)

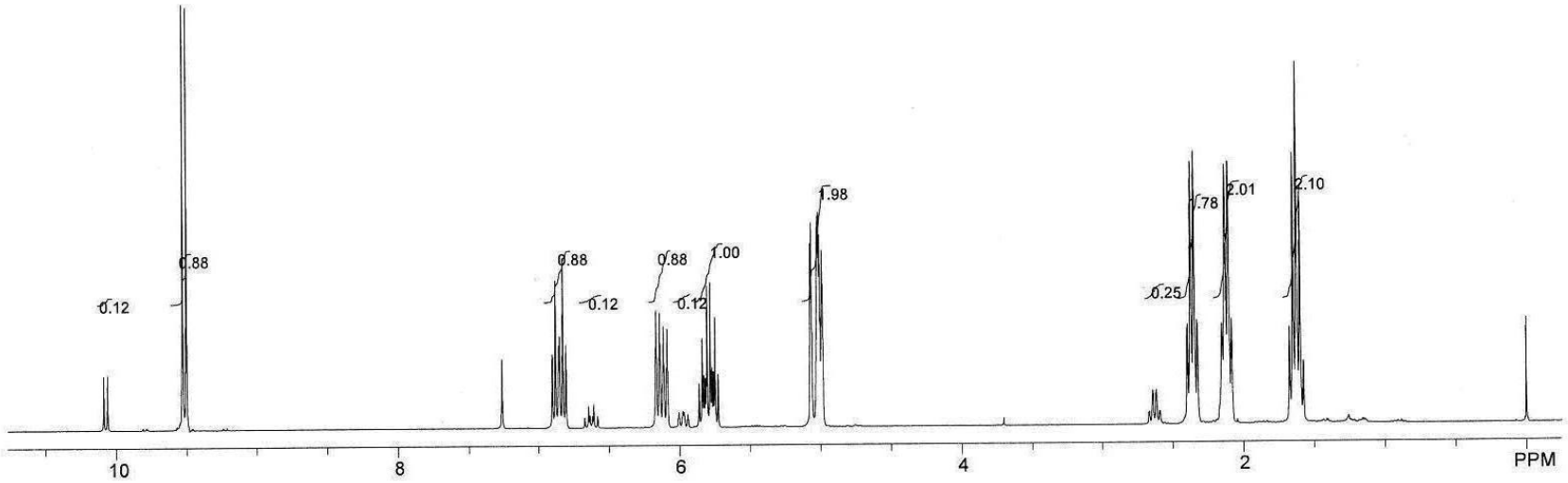


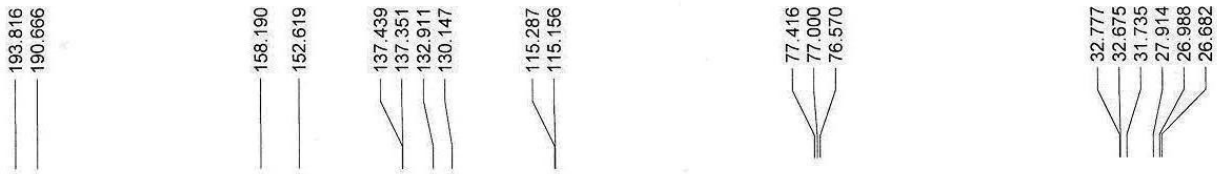


ljx-5-76-H,20110311
15:22:23.531 +0100 nmr@EP-ZH013707
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.600 usec
Recycle delay = 1.000 sec
NA = 6
Solvent = CDCl3
PTS1d = 32768
F1 = 300.130005 MHz
F2 = 1.000000 MHz



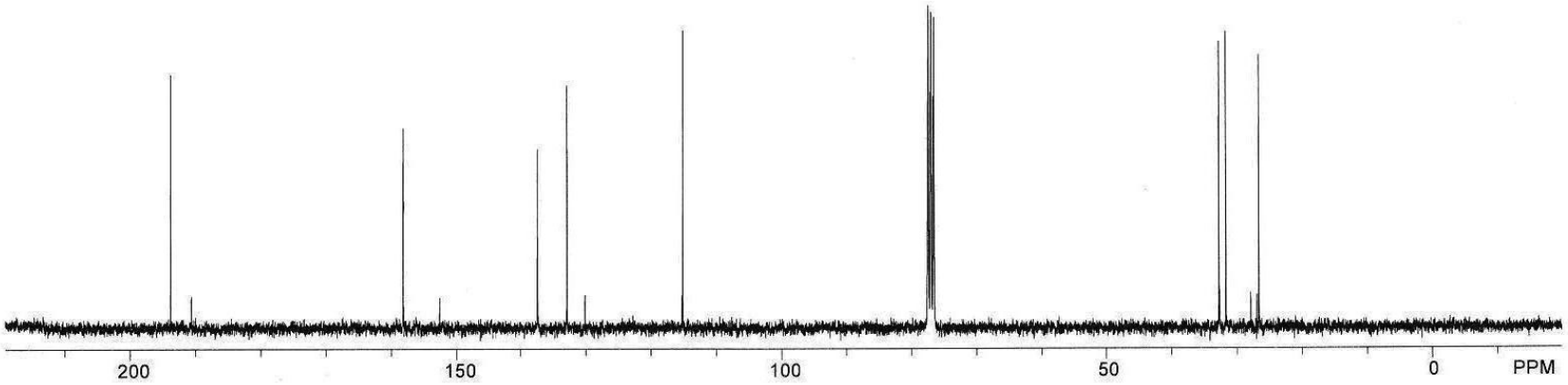
E-**2l** (*E*/*Z* = 88/12)

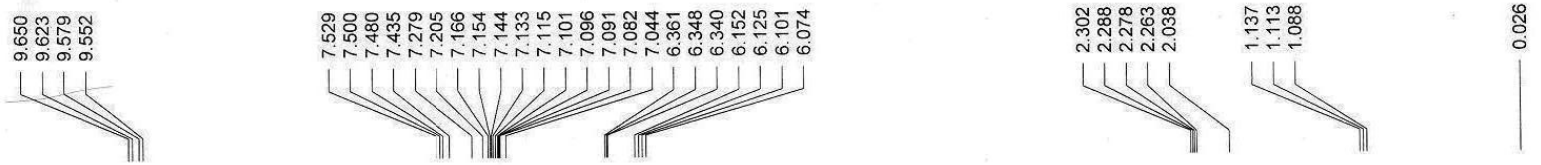




ljx-5-76-C, 20110311
15:30:31.687 +0100 nmr@EP-ZH013707
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.900 usec
Recycle delay = 2.000 sec
NA = 200
Solvent = CDCl3
PTS1d = 32768
F1 = 75.467773 MHz
F2 = 1.000000 MHz

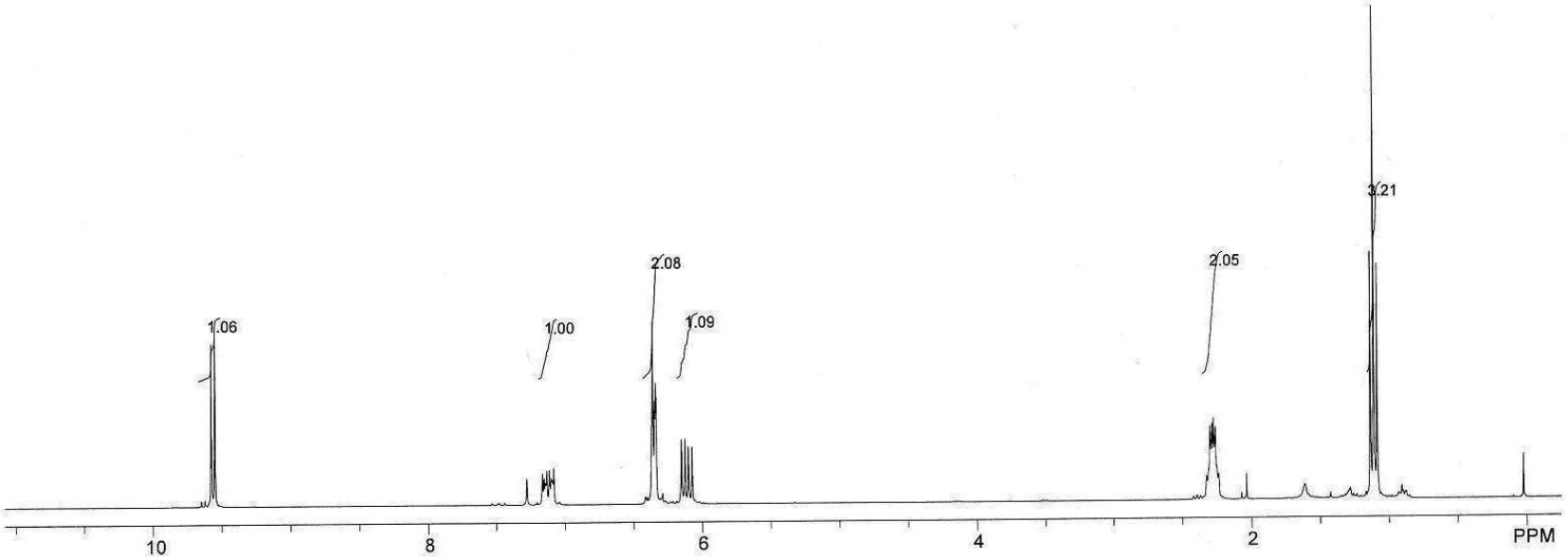
E-**2l** (*E*/*Z* = 88/12)

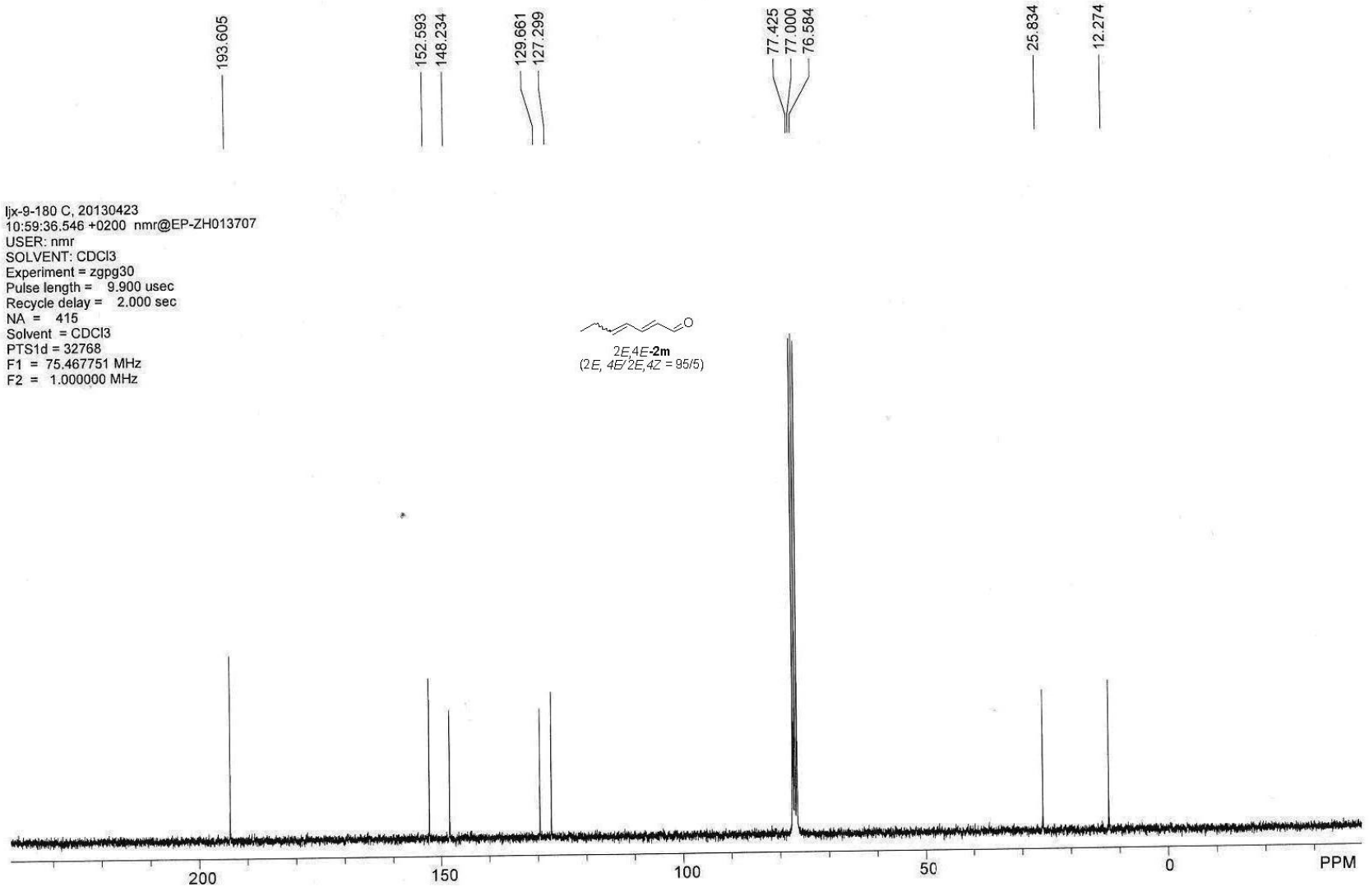


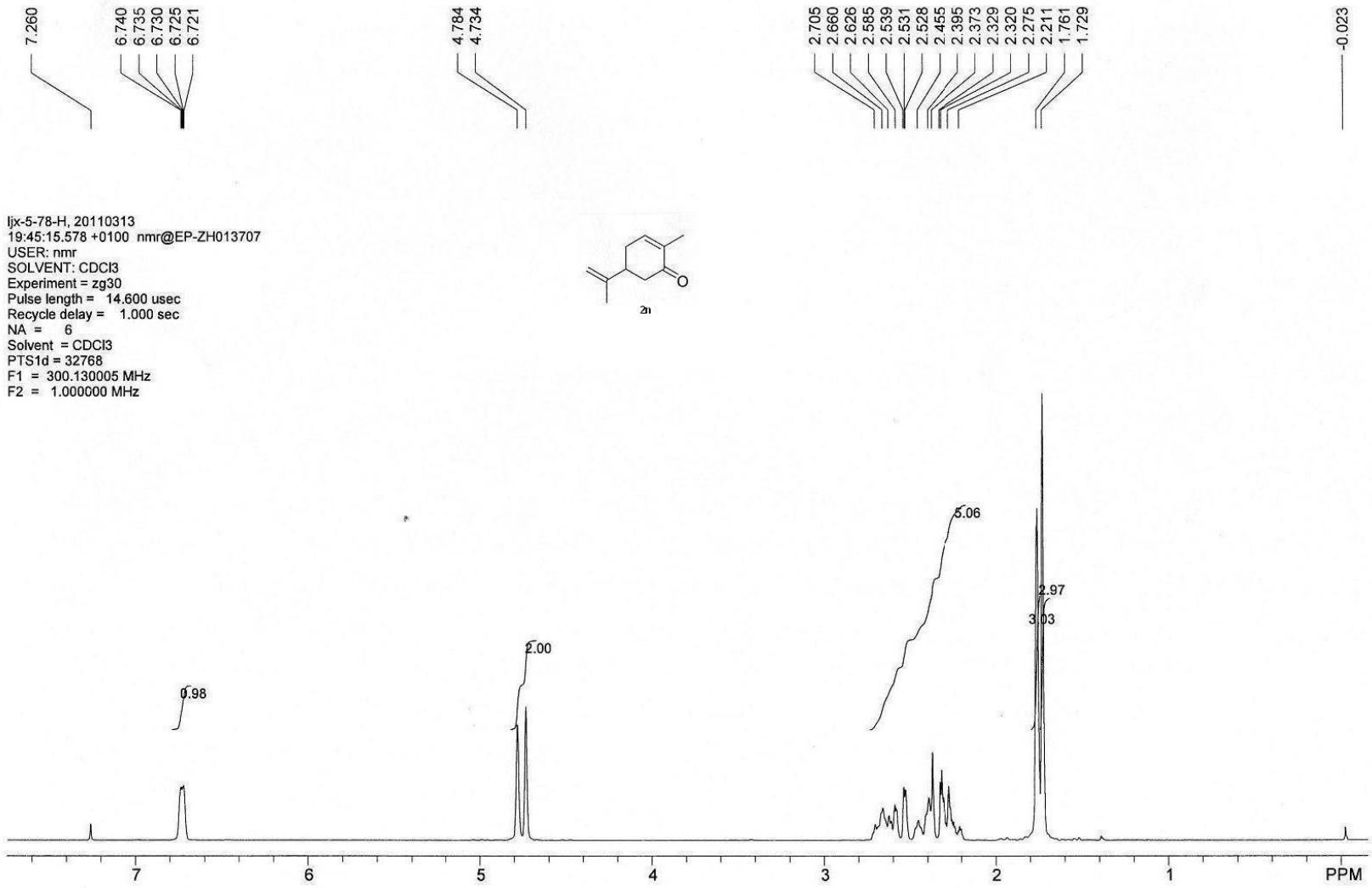


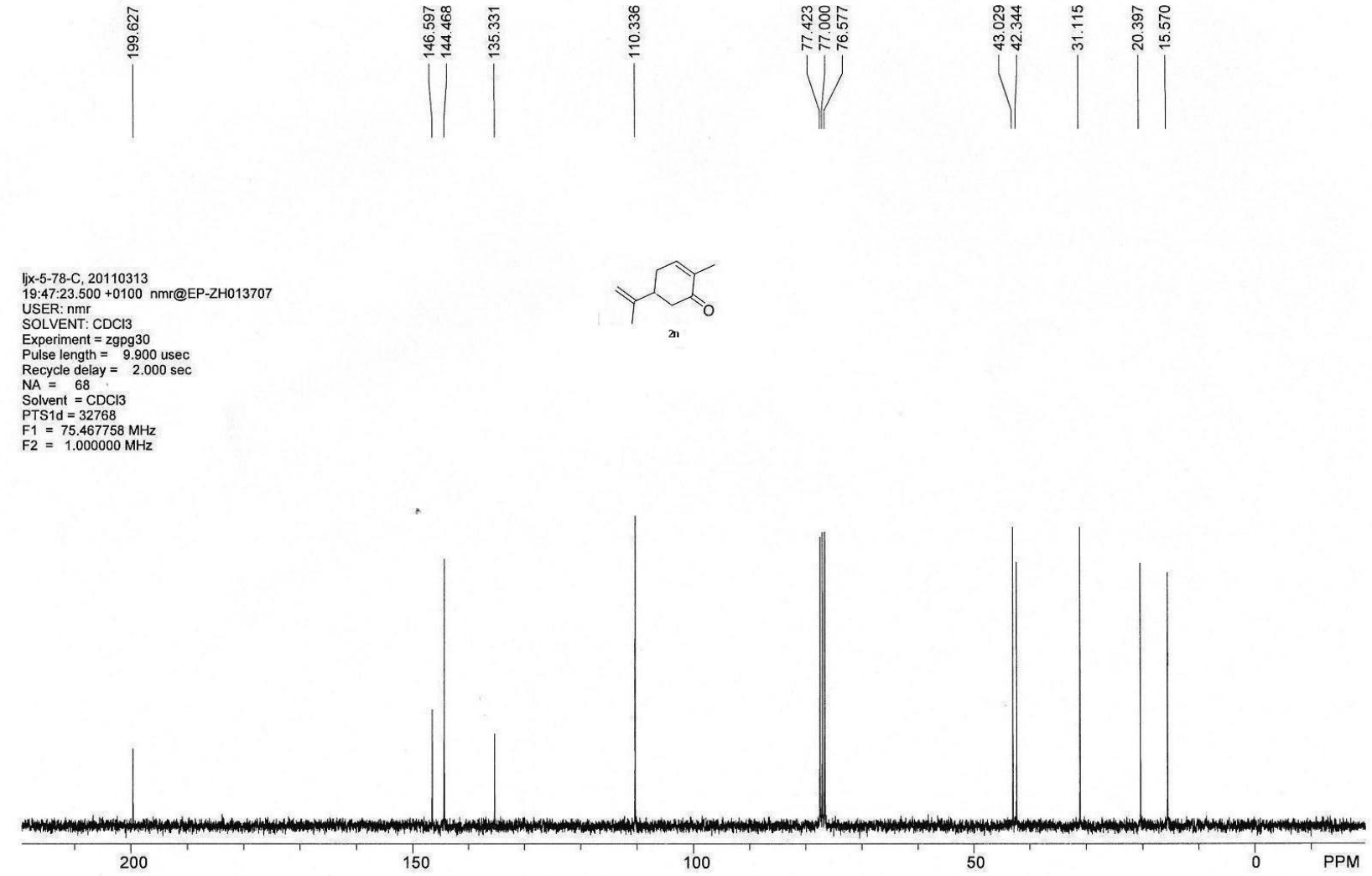
ljx-9-180 H, 20130423
10:59:07.218 +0200 nmr@EP-ZH013707
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.600 usec
Recycle delay = 1.000 sec
NA = 4
Solvent = CDCl3
PTS1d = 32768
F1 = 300.130005 MHz
F2 = 1.000000 MHz

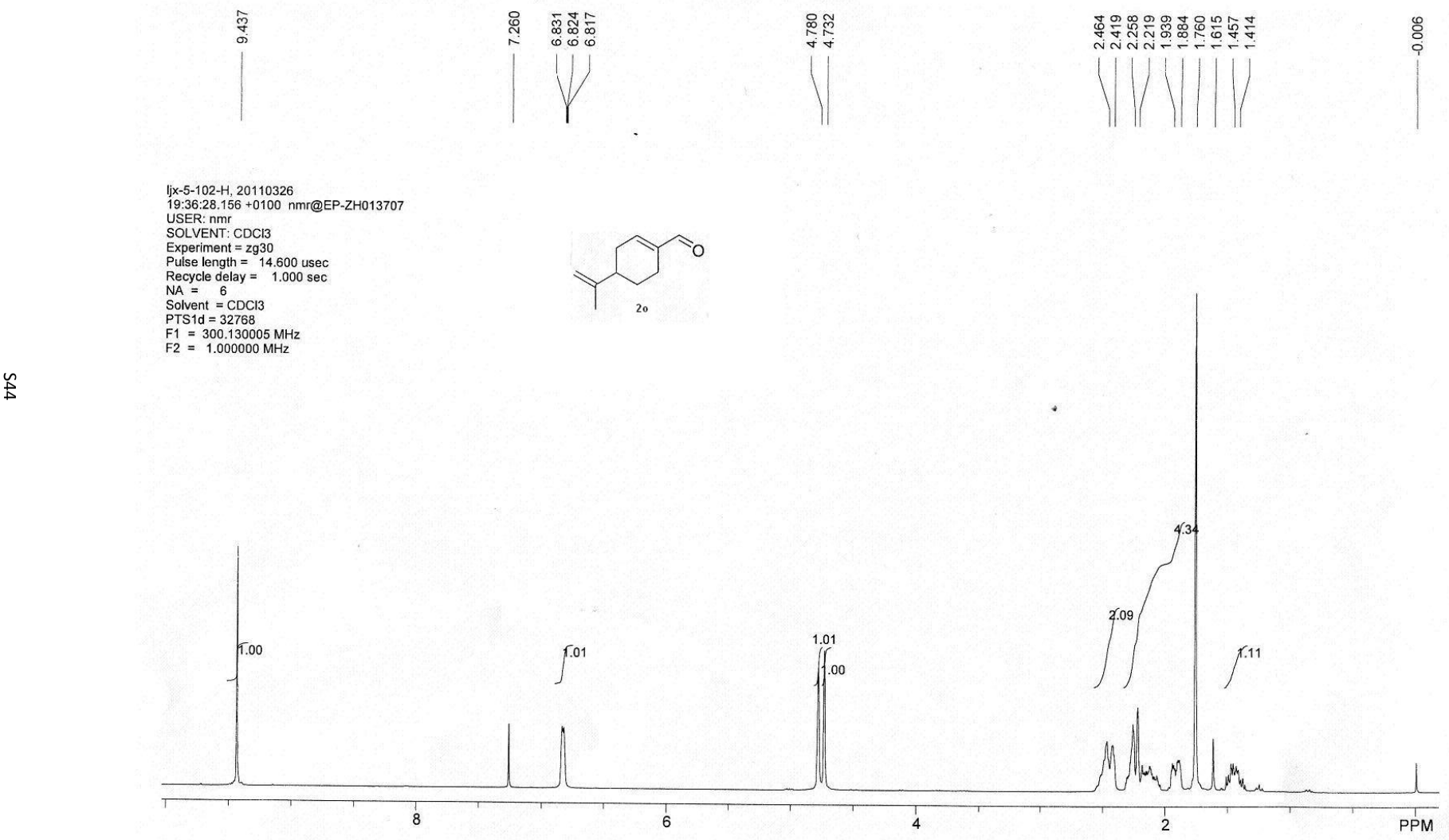
2*E*,4*E*-**2m**
(2*E*, 4*E*/2*E*,4*Z* = 95/5)

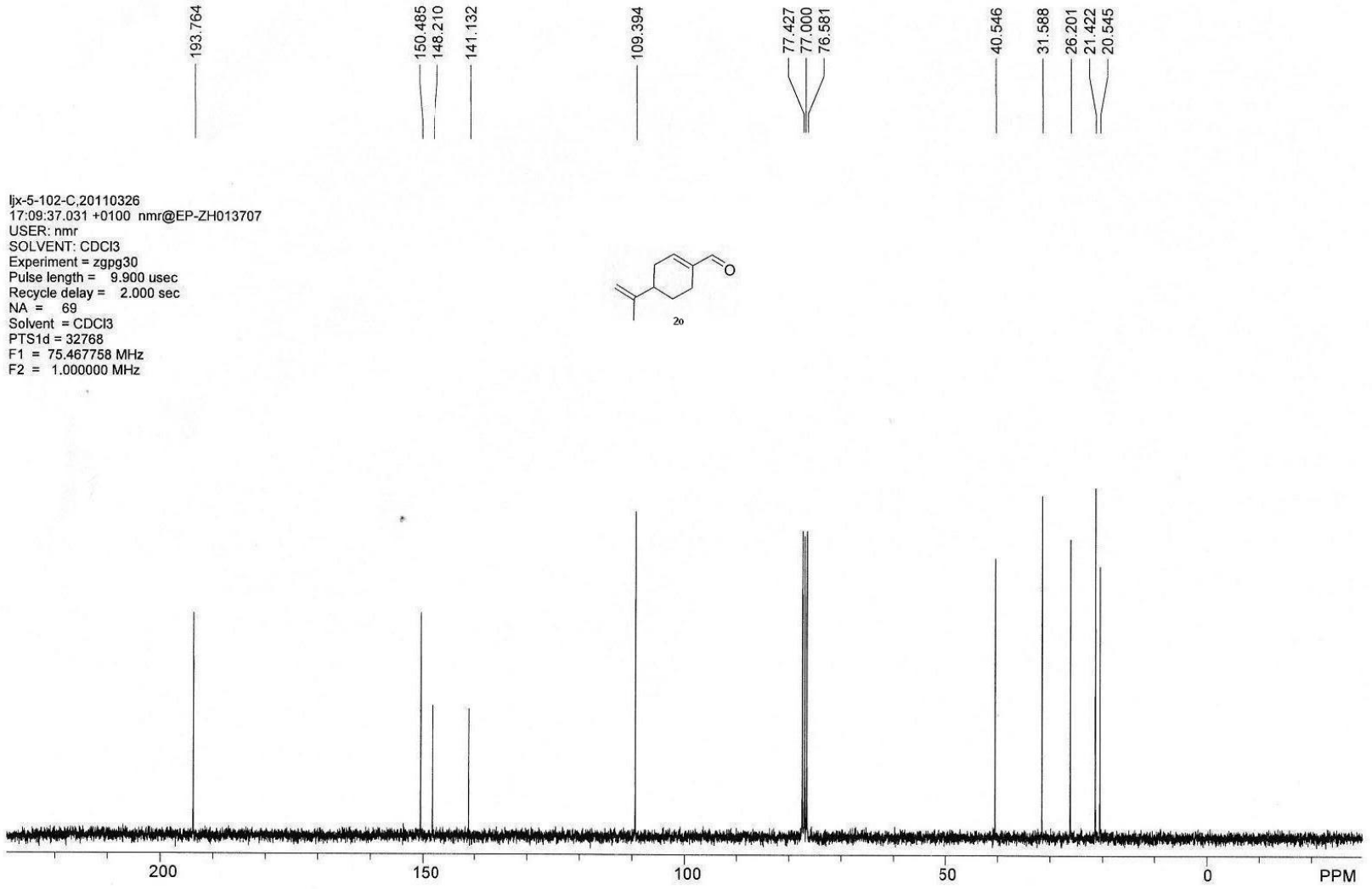


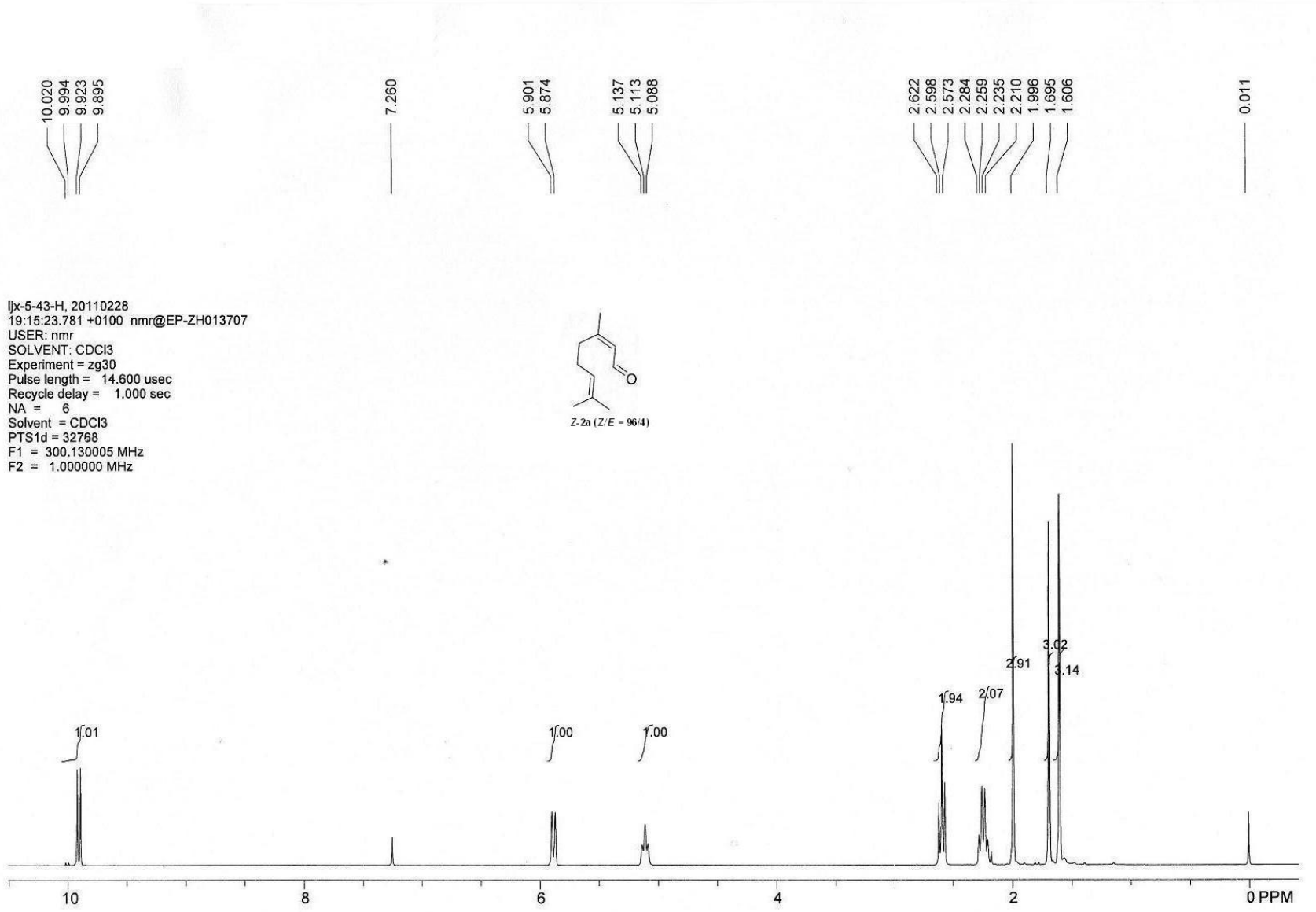

193.605
152.593
148.234
129.661
127.299
77.425
77.000
76.584
25.834
12.274
ljx-9-180 C, 20130423
10:59:36.546 +0200 nmr@EP-ZH013707
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.900 usec
Recycle delay = 2.000 sec
NA = 415
Solvent = CDCl3
PTS1d = 32768
F1 = 75.467751 MHz
F2 = 1.000000 MHz
O
2E,4E-2m
(2E, 4E/2E,4Z = 95/5)
200
150
100
50
0
PPM

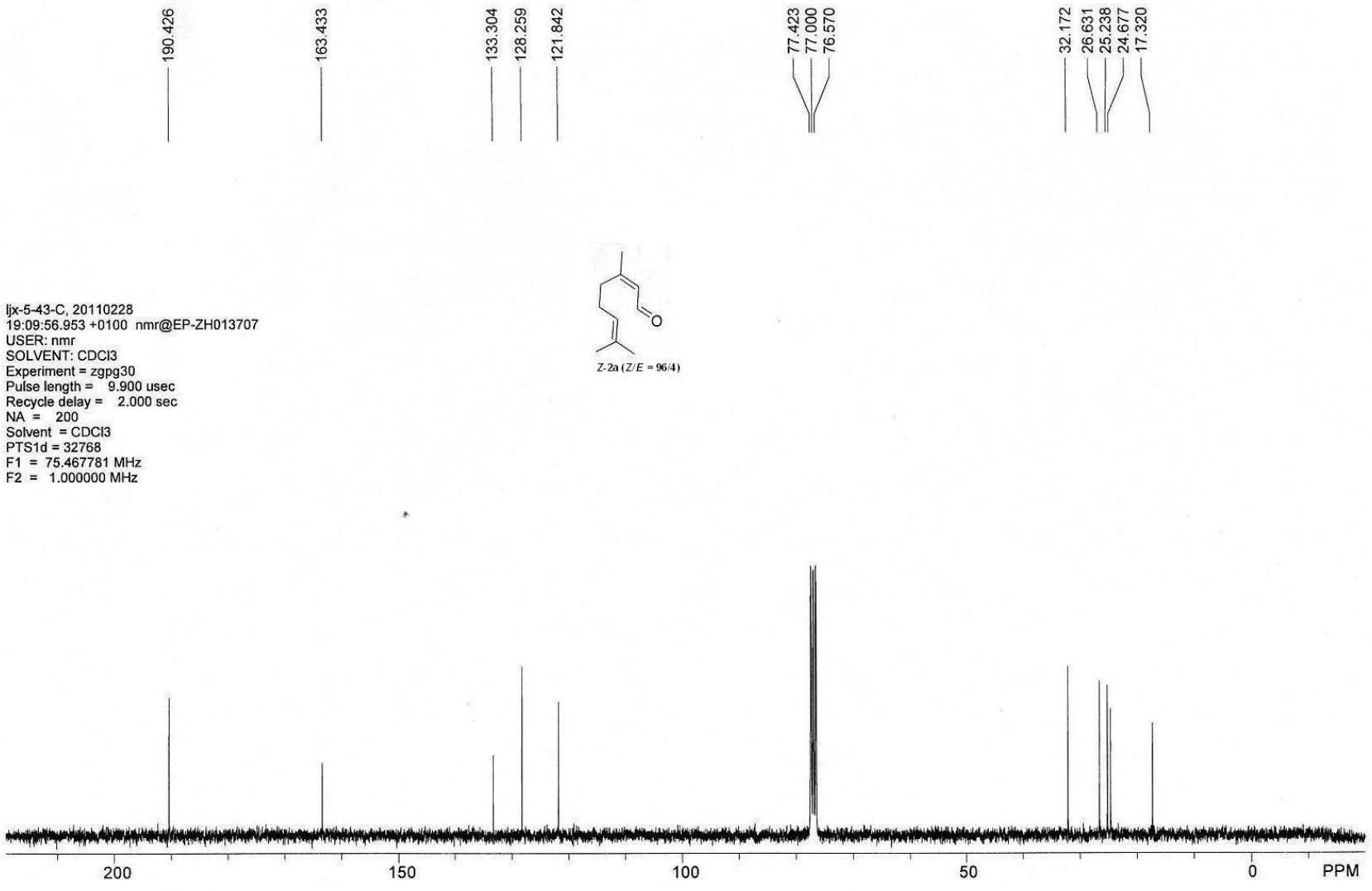

7.260
6.740
6.735
6.730
6.725
6.721
4.784
4.734
2.705
2.660
2.626
2.585
2.539
2.531
2.528
2.455
2.395
2.373
2.329
2.320
2.275
2.211
1.761
1.729
-0.023
ljx-5-78-H, 20110313
19:45:15.578 +0100 nmr@EP-ZH013707
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.600 usec
Recycle delay = 1.000 sec
NA = 6
Solvent = CDCl3
PTS1d = 32768
F1 = 300.130005 MHz
F2 = 1.000000 MHz
O
2n
0.98
2.00
5.06
2.97
3.03
7
6
5
4
3
2
1
PPM

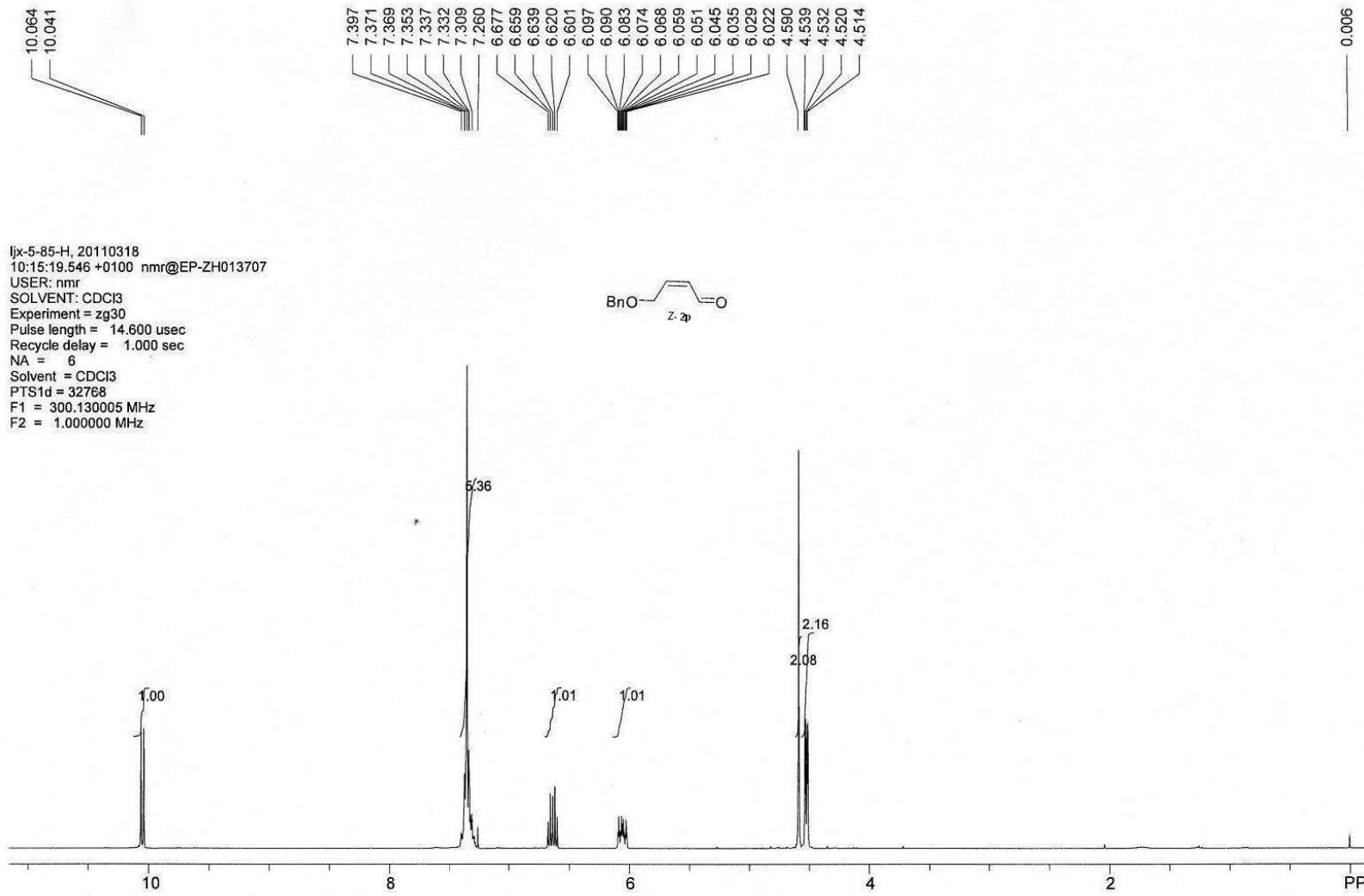

199.627
146.597
144.468
135.331
110.336
77.423
77.000
76.577
43.029
42.344
31.115
20.397
15.570
ljx-5-78-C, 20110313
19:47:23.500 +0100 nmr@EP-ZH013707
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.900 usec
Recycle delay = 2.000 sec
NA = 68
Solvent = CDCl3
PTS1d = 32768
F1 = 75.467758 MHz
F2 = 1.000000 MHz
O
2n
200
150
100
50
0
PPM

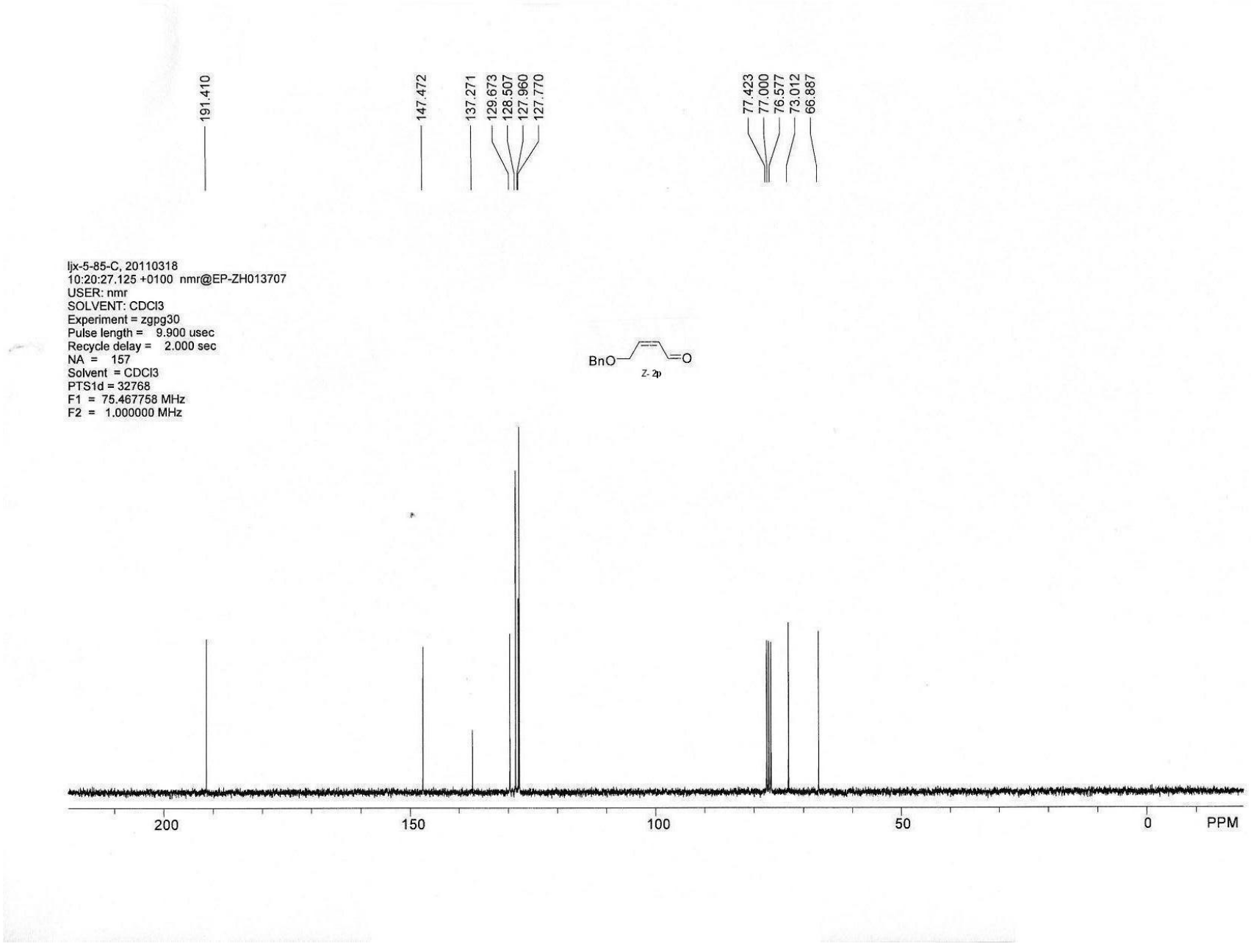

9.437
7.260
6.831
6.824
6.817
4.780
4.732
2.464
2.419
2.258
2.219
1.939
1.884
1.760
1.615
1.457
1.414
-0.006
ljx-5-102-H, 20110326
19:36:28.156 +0100 nmr@EP-ZH013707
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.600 usec
Recycle delay = 1.000 sec
NA = 6
Solvent = CDCl3
PTS1d = 32768
F1 = 300.130005 MHz
F2 = 1.000000 MHz
O
2o
1.00
1.01
1.01
1.00
2.09
4.34
1.11
8
6
4
2
PPM


193.764
150.485
148.210
141.132
109.394
77.427
77.000
76.581
40.546
31.588
26.201
21.422
20.545
ljx-5-102-C,20110326
17:09:37.031 +0100 nmr@EP-ZH013707
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.900 usec
Recycle delay = 2.000 sec
NA = 69
Solvent = CDCl3
PTS1d = 32768
F1 = 75.467758 MHz
F2 = 1.000000 MHz
O
2o
200
150
100
50
0
PPM


10.020
9.994
9.923
9.895
7.260
5.901
5.874
5.137
5.113
5.088
2.622
2.598
2.573
2.284
2.259
2.235
2.210
1.996
1.695
1.606
0.011
ljx-5-43-H, 20110228
19:15:23.781 +0100 nmr@EP-ZH013707
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.600 usec
Recycle delay = 1.000 sec
NA = 6
Solvent = CDCl3
PTS1d = 32768
F1 = 300.130005 MHz
F2 = 1.000000 MHz
O
Z-2a (Z/E = 96/4)
1.01
1.00
1.00
1.94
2.07
2.91
3.02
3.14
10
8
6
4
2
0 PPM


190.426
163.433
133.304
128.259
121.842
77.423
77.000
76.570
32.172
26.631
25.238
24.677
17.320
O
Z-2a (Z/E = 96/4)
ljx-5-43-C, 20110228
19:09:56.953 +0100 nmr@EP-ZH013707
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.900 usec
Recycle delay = 2.000 sec
NA = 200
Solvent = CDCl3
PTS1d = 32768
F1 = 75.467781 MHz
F2 = 1.000000 MHz
200
150
100
50
0
PPM


10.064
10.041
7.397
7.371
7.369
7.353
7.337
7.332
7.309
7.260
6.677
6.659
6.639
6.620
6.601
6.097
6.090
6.083
6.074
6.068
6.059
6.051
6.045
6.035
6.029
6.022
4.590
4.539
4.532
4.520
4.514
0.006
ljx-5-85-H, 20110318
10:15:19.546 +0100 nmr@EP-ZH013707
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.600 usec
Recycle delay = 1.000 sec
NA = 6
Solvent = CDCl3
PTS1d = 32768
F1 = 300.130005 MHz
F2 = 1.000000 MHz
BnO
O
Z-2p
5.36
2.16
2.08
1.00
1.01
1.01
10
8
6
4
2
PPM


191.410
147.472
137.271
129.673
128.507
127.960
127.770
77.423
77.000
76.577
73.012
66.887
ljx-5-85-C, 20110318
10:20:27.125 +0100 nmr@EP-ZH013707
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.900 usec
Recycle delay = 2.000 sec
NA = 157
Solvent = CDCl3
PTS1d = 32768
F1 = 75.467758 MHz
F2 = 1.000000 MHz
BnO
O
Z- 2p
200
150
100
50
0
PPM