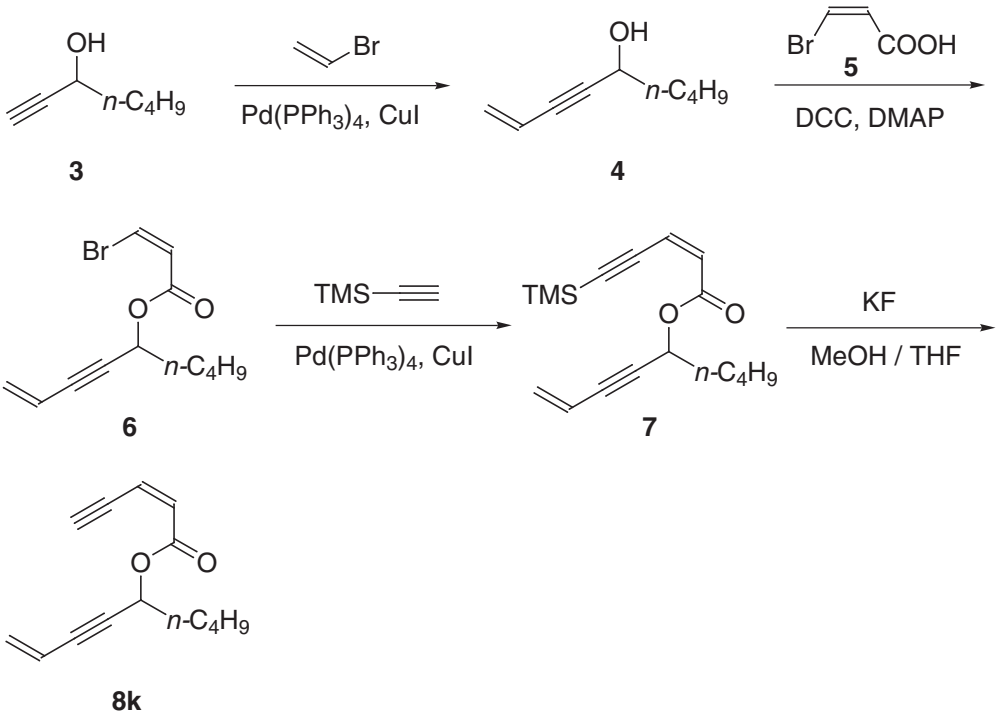
Supporting Information

Preparation of Bis-Enynes 8a-f, 8j-k, 18a-c, 20. A Representative Procedure.

Scheme S1



1-Nonen-3-yn-5-ol (4). To a mixture of $Pd(PPh_3)_4$ (99 mg, 0.086 mmol) and CuI (98 mg, 0.51 mmol) were added Et_2NH (20 mL) and 1-heptyn-3-ol **3** (4.7 mL, 37 mmol) under an argon atmosphere at room temperature. The reaction mixture was cooled with water bath, and vinyl bromide in THF (1.0 M, 47 mL, 47 mmol) was added. After 5 min, the water bath was removed and the reaction mixture was stirred at room temperature for 1.5 h. The mixture was poured into ice water, and extracted with Et_2O. The organic layer was washed with 2 N HCl aq. and dried over $MgSO_4$. Evaporation of the solvent gave an oil, which was further purified by silica gel column chromatography (hexane : AcOEt = 10 : 1) to give **4** (4.5 g, 89%): yellow oil; ^{1}H NMR (300 MHz, $CDCl_3$) 5.80 (ddd, J = 17.6, 11.0, 1.6 Hz, 1H), 5.62 (dd, J = 17.5, 2.2 Hz, 1H), 5.46 (dd, J = 10.9, 2.3 Hz, 1H), 4.46 (m, 1H), 1.75-1.62 (m, 3H), 1.48-1.28 (m, 4H), 0.90 (dd, J = 7.2, 7.2 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) 127.1, 116.6, 90.9, 83.3, 62.7, 37.4, 27.2, 22.3, 13.9; IR (neat) 3335, 3101, 3047, 3013, 2959, 2934, 2862, 2222, 1468, 1412, 1161, 1030, 1009 cm^{-1}; HMRS Calcd for $C_9H_{14}O$: 138.1044. Found: 138.1040.

1-Butyl-4-penten-2-ynyl (*Z*)-3-bromo-2-propenoate (6). To a mixture of (*Z*)-3-bromo-2-propenoic acid **5**[1] (4.5 g, 30 mmol) and DMAP (0.37 g, 3.0 mmol) were added CH_2Cl_2 (30 mL) and a CH_2Cl_2 (10 mL) solution of **4** (4.2 g, 30 mmol) under an argon atmosphere at room temperature. The reaction mixture was cooled to 0 _, and DCC (7.4 g, 36 mmol) was added and the mixture was stirred for 5 min. After stirring for additional 1 h at room temperature, the reaction mixture was filtered with celite and the residue was washed with Et_2O. The organic layer was washed with 0.5 N HCl aq. and saturated $NaHCO_3$ aq., and dried over $MgSO_4$. Evaporation of the solvent gave an oil, which was further purified by silica gel column chromatography (hexane : AcOEt = 30 : 1) to give **6** (6.8 g, 84%): colorless oil; ^{1}H NMR (300 MHz, $CDCl_3$) 7.02 (d, J = 8.5 Hz,

1H), 6.62 (d, *J* = 8.3 Hz, 1H), 5.80 (ddd, *J* = 17.5, 10.9, 1.7 Hz, 1H), 5.65 (dd, *J* = 17.5, 2.5 Hz, 1H), 5.57 (ddd, *J* = 6.6, 6.6, 1.0 Hz, 1H), 5.49 (dd, *J* = 10.9, 2.4 Hz, 1H), 1.85-1.75 (m, 2H), 1.48 –1.24 (m, 4H), 0.90 (dd, *J* = 7.2, 7.2 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) 162.7, 127.9, 124.0, 122.2, 116.3, 86.7, 84.1, 64.8, 34.4, 27.1, 22.1, 13.8; IR (neat) 3074, 3049, 3013, 2957, 2934, 2864, 2149, 1736, 1612, 1333, 1204, 1159 cm^{-1}; Anal. Calcd for $C_{12}H_{15}BrO_2$: C, 53.15; H, 5.58. Found: C, 53.41; H, 5.70.

1-Butyl-4-penten-2-ynyl (*Z*)-5-trimethylsilyl-2-penten-4-ynoate (7). To a mixture of $Pd(PPh_3)_4$ (300 mg, 0.26 mmol) and CuI (100 mg, 0.52 mmol) were added Et_3N (40 mL), a THF (20 mL) solution of **6** (5.4 g, 20 mmol), and (trimethylsilyl)acetylene (3.4 mL, 24 mmol) under an argon atmosphere at room temperature. The reaction mixture was stirred for 1 h. Then the mixture was poured into ice water, and extracted with Et_2O. The organic layer was washed with 0.5 N HCl aq., and dried over $MgSO_4$. Evaporation of the solvent gave an oil, which was further purified by silica gel column chromatography (hexane : AcOEt = 50 : 1) to give **7** (5.3 g, 92%): yellow oil; ^{1}H NMR (300 MHz, $CDCl_3$) 6.16 (d, *J* = 11.7 Hz, 1H), 6.07 (d, *J* = 11.7 Hz, 1H), 5.79 (ddd, *J* = 17.5, 10.9, 1.8 Hz, 1H), 5.67-5.56 (m, 2H), 5.48 (dd, *J* = 10.9, 2.4 Hz, 1H), 1.85-1.78 (m, 2H), 1.46-1.28 (m, 4H), 0.90 (dd, *J* = 7.2, 7.2 Hz, 3H), 0.22 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$) 163.3, 128.8, 127.7, 123.2, 116.5, 108.8, 100.6, 87.2, 83.9, 64.5, 34.5, 27.1, 22.2, 13.9, -0.4; IR (neat) 3015, 2959, 2934, 2864, 2151, 1736, 1717, 1607, 1252, 1161, 1034 cm^{-1}; HMRS Calcd for $C_{17}H_{24}O_2Si$: 288.1544. Found: 288.1553.

1-Butyl-4-penten-2-ynyl (*Z*)-2-penten-4-ynoate (8k): To a mixture of KF (1.5 g, 26 mmol) in MeOH (40 mL) was slowly added a THF (20 mL) solution of **7** (3.8 g, 13 mmol) at 0 °C under an argon atmosphere and the mixture was kept stirring for 40 min. The mixture was poured into water, and extracted with Et_2O. The organic layer was washed with saturated brine, and dried over $MgSO_4$. Evaporation of the solvent gave an oil, which was further purified by silica gel column chromatography (hexane : AcOEt = 50 : 1) to give **8k** (2.8 g, 98%): yellow oil; ^{1}H NMR (300 MHz, $CDCl_3$) 6.21 (d, *J* = 11.5 Hz, 1H), 6.14 (dd, *J* = 11.5, 2.4 Hz, 1H), 5.79 (ddd, *J* = 17.5, 10.9, 1.6 Hz, 1H), 5.64 (dd, *J* = 17.4, 2.3 Hz, 1H), 5.57 (ddd, *J* = 6.6, 6.6, 1.5 Hz, 1H), 5.48 (dd, *J* = 10.7, 2.4 Hz, 1H), 3.62 (d, *J* = 2.2 Hz, 1H), 1.87-1.75 (m, 2H), 1.51-1.28 (m, 4H), 0.90 (dd, *J* = 7.2, 7.2 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) 163.2, 130.2, 127.9, 122.7, 116.4, 89.7, 86.9, 84.0, 79.5, 64.8, 34.4, 27.1, 22.2, 13.9; IR (neat) 3292, 3101, 3080, 3032, 3013, 2959, 2934, 2864, 1732, 1611, 1541, 1508, 1458, 1398, 1277, 1221, 1165 cm^{-1}; HMRS Calcd for $C_{14}H_{16}O_2$: 216.1149. Found: 216.1140.

4-Penten-2-ynyl (*Z*)-2-penten-4-ynoate (8a): yellow oil; ^{1}H NMR (300 MHz, $CDCl_3$) 6.24 (dd, *J* = 11.6, 0.8 Hz, 1H), 6.17 (dd, *J* = 11.5, 2.5 Hz, 1H), 5.80 (dddd, *J* = 17.6, 10.7, 1.8, 1.8 Hz, 1H), 5.67 (dd, *J* = 17.6, 2.5 Hz, 1H), 5.52 (dd, *J* = 10.8, 2.6 Hz, 1H), 4.89 (d, *J* = 1.8 Hz, 2H), 3.65 (dd, *J* = 2.4, 0.8 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) 163.5, 129.6, 128.4, 123.2, 116.2, 90.0, 85.3, 83.2, 79.4, 52.8; IR (neat) 3287, 2943, 2235, 2098, 1732, 1653, 1614, 1558, 1506, 1456, 1435, 1404, 1362, 1281, 1221, 1167, 1040, 1024 cm^{-1}; HMRS Calcd for $C_{10}H_8O_2$: 160.0524. Found: 160.0560.

4-Penten-2-ynyl (*Z*)-2-undecen-4-ynoate (8b): brown oil; ^{1}H NMR (300 MHz, $CDCl_3$) 6.19 (ddd, *J* = 11.4, 2.4, 2.4 Hz, 1H), 6.04 (d, *J* = 11.3 Hz, 1H), 5.80 (dddd, *J* = 17.6, 10.6, 1.8, 1.8 Hz, 1H), 5.66 (dd, *J* = 17.6, 2.6 Hz, 1H), 5.51 (dd, *J* = 10.7, 2.5 Hz, 1H), 4.86 (d, *J* = 1.9 Hz, 2H), 2.43 (ddd, *J* = 7.0, 7.0, 2.3 Hz, 2H), 1.63-1.26 (m, 8H), 0.87 (dd, *J* = 6.9, 6.9 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) 164.0, 128.2, 126.2, 125.3, 116.3, 105.4, 85.1, 83.6, 77.6, 52.4, 31.3, 28.6, 28.3, 22.5, 20.1, 14.0; IR (neat) 3101,

3013, 2955, 2932, 2858, 2239, 2208, 1732, 1716, 1609, 1456, 1431, 1412, 1362, 1277, 1229, 1209, 1163, 1040, 1020 cm^{-1}; HMRS Calcd for $C_{16}H_{20}O_2$: 244.1462. Found: 244.1463; Anal. Calcd for $C_{16}H_{20}O_2$: C, 78.65; H, 8.25. Found: C, 78.58; H, 8.48.

4-Penten-2-ynyl (*Z*)-5-phenyl-2-penten-4-ynoate (8c): yellow oil; ^{1}H NMR (300 MHz, $CDCl_3$) 7.59-7.53 (m, 2H), 7.35-7.28 (m, 3H), 6.41 (d, *J* = 11.3 Hz, 1H), 6.15 (d, *J* = 11.3 Hz, 1H), 5.80 (dddd, *J* = 17.5, 10.9, 1.8, 1.8 Hz, 1H), 5.66 (dd, *J* = 17.6, 2.5 Hz, 1H), 5.51 (dd, *J* = 10.8, 2.5 Hz, 1H), 4.92 (d, *J* = 1.6 Hz, 2H); ^{13}C NMR (75 MHz, $CDCl_3$) 163.9, 132.2, 129.3, 128.4, 128.3, 126.9, 124.1, 122.4, 116.3, 102.1, 86.2, 85.3, 83.5, 52.7; IR (neat) 3101, 3080, 3057, 3034, 3015, 2961, 2937, 2872, 2855, 2235, 2199, 1730, 1715, 1609, 1489, 1443, 1412, 1362, 1281, 1266, 1207, 1163, 1030 cm^{-1}; HMRS Calcd for $C_{16}H_{12}O_2$: 236.0837. Found: 236.0812.

4-Penten-2-ynyl (*Z*)-6-hydroxy-2-hexen-4-ynoate (8d): brown oil; ^{1}H NMR (300 MHz, $CDCl_3$) 6.22 (ddd, *J* = 11.5, 1.8, 1.8 Hz, 1H), 6.14 (d, *J* = 11.5 Hz, 1H), 5.81 (dddd, *J* = 17.4, 10.8, 1.8, 1.8 Hz, 1H), 5.68 (dd, *J* = 17.6, 2.5 Hz, 1H), 5.53 (dd, *J* = 10.7, 2.6 Hz, 1H), 4.87 (d, *J* = 1.8 Hz, 2H), 4.49 (s, 2H), 2.03 (s, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) 163.9, 128.5, 127.4, 124.1, 116.1, 101.2, 85.3, 83.2, 82.0, 52.8, 51.5; IR (neat); 3449, 3101, 3044, 3013, 2928, 2856, 2235, 2201, 1717, 1609, 1435, 1410, 1362, 1277, 1229, 1169, 1119, 1018 cm^{-1}; HMRS Calcd for $C_{11}H_{10}O_3$: 190.0630. Found: 190.0629.

4-Methyl-4-penten-2-ynyl (*Z*)-2-penten-4-ynoate (8e): pale yellow oil; ^{1}H NMR (300 MHz, $CDCl_3$) 6.24 (dd, *J* = 11.5, 0.6 Hz, 1H), 6.16 (dd, *J* = 11.5, 2.4 Hz, 1H), 5.33 (s, 1H), 5.26 (dd, *J* = 1.6, 1.6 Hz, 1H), 4.89 (s, 2H), 3.63 (dd, *J* = 2.4, 0.8 Hz, 1H), 1.87 (dd, *J* = 1.1, 1.1 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) 163.4, 129.7, 125.8, 123.1, 123.0, 89.9, 87.8, 81.5, 79.3, 52.7, 23.0; IR (neat) 3288, 3098, 2932, 2856, 2233, 2118, 1732, 1612, 1404, 1362, 1281, 1221, 1167 cm^{-1}; HMRS Calcd for $C_{11}H_{10}O_2$: 174.0680. Found: 174.0668.

4-Methyl-4-penten-2-ynyl (*Z*)-2-octen-4-ynoate (8f): brown oil; ^{1}H NMR (300 MHz, $CDCl_3$) 6.19 (ddd, *J* = 11.3, 2.4, 2.4 Hz, 1H), 6.05 (d *J* = 11.3 Hz, 1H), 5.32 (s, 1H), 5.25 (dddd, *J* = 1.6, 1.6, 1.6, 1.6 Hz, 1H), 4.86 (s, 2H), 2.42 (ddd, *J* = 7.1, 7.1, 2.2 Hz, 2H), 1.87 (dd, *J* = 1.2, 1.2 Hz, 3H), 1.61 (ddddd, *J* = 7.3, 7.3, 7.3, 7.3, 7.3 Hz, 2H), 1.01 (dd, *J* = 7.4, 7.4 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) 163.9, 126.3, 125.9, 125.1, 122.9, 105.0, 87.6, 81.9, 77.7, 52.5, 23.1, 22.0, 21.8, 13.5; IR (neat) 3098, 2964, 2936, 2874, 2235, 2208, 1732, 1609, 1433, 1412, 1362, 1339, 1277, 1229, 1209, 1163, 1088, 1030, 1011 cm^{-1}; HMRS Calcd for $C_{14}H_{16}O_2$: 216.1149. Found: 216.1129.

1-Phenyl-4-penten-2-ynyl (*Z*)-2-penten-4-ynoate (8j) : yellow oil; ^{1}H NMR (300 MHz, $CDCl_3$) 7.56-7.53 (m, 2H), 7.40-7.31 (m, 3H), 6.66 (d, *J* = 1.4 Hz, 1H), 6.22 (dd, *J* = 11.7 Hz, 1H), 6.15 (dd, *J* = 11.5, 2.4 Hz, 1H), 5.84 (ddd, *J* = 17.5, 10.9, 1.7 Hz, 1H), 5.70 (dd, *J* = 17.5, 2.4 Hz, 1H), 5.53 (dd, *J* = 10.7, 2.4 Hz, 1H), 3.63 (d, *J* = 2.4 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) 162.9, 136.7, 129.9, 129.0, 128.6, 128.5, 127.8, 123.2, 116.2, 90.1, 85.9, 85.8, 79.4, 66.2; IR (neat) 3287, 3090, 3067, 3036, 3013, 2973, 2941, 2228, 2098, 1732, 1611, 1495, 1456, 1398, 1313, 1273, 1219, 1171, 1151, 1001 cm^{-1}; HMRS Calcd for $C_{16}H_{12}O_2$: 236.0837. Found: 236.0844.

5-Hexen-3-ynyl (*Z*)-2-penten-4-ynoate (18a): pale yellow oil; ^{1}H NMR (300 MHz, $CDCl_3$) 6.22 (dd, *J* = 11.5, 0.6 Hz, 1H), 6.14 (dd, *J* = 11.6, 2.5 Hz, 1H), 5.75 (dddd, *J* = 17.5, 10.9, 2.1, 2.1 Hz, 1H), 5.57 (dd, *J* = 17.5, 2.2 Hz, 1H), 5.41 (dd, *J* = 10.9, 2.4 Hz, 1H), 4.28 (dd, *J* = 6.9, 6.9 Hz, 2H), 3.61 (dd, *J* = 2.4, 0.8 Hz, 1H), 2.69 (ddd, *J* = 6.9, 6.9, 2.0 Hz, 2H); ^{13}C NMR (75 MHz, $CDCl_3$) 164.0, 130.3, 126.5, 122.5, 117.1, 89.5,

86.1, 80.7, 79.4, 62.5, 19.7; IR (neat) 3285, 3099, 3045, 3011, 2964, 2910, 2230, 2098, 1732, 1614, 1404, 1286, 1227, 1177, 1013 cm^{-1}; HMRS Calcd for $C_{11}H_9O_2$ (M^+ - H): 173.0602. Found: 173.0599.

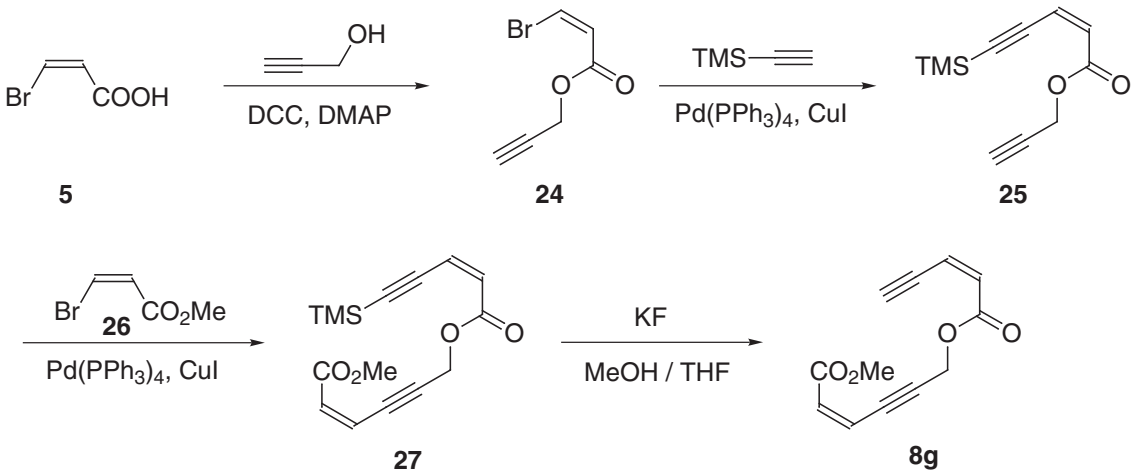
5-Hexen-3-ynyl (*Z*)-2-octen-4-ynoate (18b): brown oil; ^1H NMR (300 MHz, $CDCl_3$) 6.16 (ddd, *J* = 11.5, 2.3, 2.3 Hz, 1H), 6.03 (d, *J* = 11.5 Hz, 1H), 5.75 (dddd, *J* = 17.6, 10.8, 2.0, 2.0 Hz, 1H), 5.56 (dd, *J* = 17.6, 2.1 Hz, 1H), 5.40 (dd, *J* = 10.8, 2.3 Hz, 1H), 4.25 (dd, *J* = 7.0, 7.0 Hz, 2H), 2.67 (ddd, *J* = 7.0, 7.0, 1.7 Hz, 2H), 2.42 (ddd, *J* = 7.1, 7.1, 2.4 Hz, 2H), 1.60 (ddddd, *J* = 7.3, 7.3, 7.3, 7.3, 7.3 Hz, 2H), 1.01 (dd, *J* = 7.4, 7.4 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) 164.5, 126.8, 126.4, 124.6, 117.1, 104.6, 86.2, 80.6, 77.8, 62.1, 22.0, 21.8, 19.8, 13.5; IR (neat) 3099, 3011, 2964, 2936, 2905, 2874, 2833, 2259, 2208, 1728, 1609, 1456, 1414, 1381, 1339, 1277, 1231, 1173, 1119, 1070, 1013 cm^{-1}; Anal. Calcd for $C_{14}H_{16}O_2$: C, 77.75; H, 7.46. Found: C, 77.65; H, 7.59.

5-Hexen-3-ynyl (*Z*)-5-phenyl-2-penten-4-ynoate (18c): colorless oil; ^1H NMR (300 MHz, $CDCl_3$) 7.54-7.50 (m, 2H), 7.36-7.29 (m, 3H), 6.38 (d, *J* = 11.5 Hz, 1H), 6.14 (d, *J* = 11.3 Hz, 1H), 5.73 (dddd, *J* = 17.5, 10.9, 2.0, 2.0 Hz, 1H), 5.55 (dd, *J* = 17.5, 2.4 Hz, 1H), 5.39 (dd, *J* = 10.9, 2.4 Hz, 1H), 4.30 (dd, *J* = 7.0, 7.0 Hz, 2H), 2.70 (ddd, *J* = 7.0, 7.0, 1.8 Hz, 2H); ^{13}C NMR (75 MHz, $CDCl_3$) 164.4, 132.0, 129.2, 128.3, 127.5, 126.5, 123.5, 122.5, 117.0, 101.7, 86.3, 86.1, 80.7, 62.3, 19.8; IR (neat) 3099, 3080, 3057, 3011, 2961, 2907, 2230, 2201, 1724, 1609, 1489, 1443, 1412, 1337, 1286, 1265, 1209, 1169, 1070, 1015 cm^{-1}; Anal. Calcd for $C_{17}H_{14}O_2$: C, 81.58; H, 5.64. Found: C, 81.46; H, 5.80.

6-Hepten-4-ynyl (*Z*)-2-penten-4-ynoate (20): pale yellow oil; ^1H NMR (300 MHz, $CDCl_3$) 6.19 (d, *J* = 11.7 Hz, 1H), 6.12 (dd, *J* = 11.5, 2.4 Hz, 1H), 5.74 (dddd, *J* = 17.5, 10.9, 2.1, 2.1 Hz, 1H), 5.54 (dd, *J* = 17.5, 2.2 Hz, 1H), 5.38 (dd, *J* = 10.9, 2.2 Hz, 1H), 4.27 (dd, *J* = 6.1, 6.1 Hz, 2H), 3.60 (d, *J* = 2.4 Hz, 1H), 2.43 (ddd, *J* = 7.0, 7.0, 2.0 Hz, 2H), 1.90 (dddd, *J* = 6.6, 6.6, 6.6, 6.6 Hz, 2H); ^{13}C NMR (75 MHz, $CDCl_3$) 164.3, 130.6, 125.9, 122.0, 117.3, 89.2, 80.0, 79.5, 63.3, 27.6, 16.1; IR (neat) 3287, 3099, 3044, 3009, 2961, 2899, 2845, 2228, 2098, 1728, 1614, 1404, 1288, 1223, 1178 cm^{-1}; HMRS Calcd for $C_{12}H_{12}O_2$: 188.0837. Found: 188.0783.

Preparation of Bis-Enynes 8g-i, 18d. A Representative Procedure.

Scheme S2



2-Propynyl (*Z*)-3-bromo-2-propenoate (24). To a mixture of (*Z*)-3-bromo-2-propenoic acid **5** (10.5 g, 70 mmol) and DMAP (0.86 g, 7.0 mmol) were added CH_2Cl_2 (90 mL) and 2-propyn-1-ol (4.4 mL, 77 mmol) under an argon atmosphere at room

temperature. The reaction mixture was cooled to 0 _, and DCC (17.3 g, 84 mmol) was added and the mixture was stirred for 5 min. After stirring for additional 45 min at room temperature, the reaction mixture was filtered with celite and the residue was washed with Et_2O. The organic layer was washed with 0.5 N HCl aq. and saturated $NaHCO_3$ aq., and dried over $MgSO_4$. Evaporation of the solvent gave an oil, which was further purified by silica gel column chromatography (hexane : AcOEt = 30 : 1) to give **23** (11.2 g, 84%): colorless oil; ^{1}H NMR (300 MHz, $CDCl_3$) 7.08 (d, *J* = 8.3 Hz, 1H), 6.65 (d, *J* = 8.5 Hz, 1H), 4.77 (d, *J* = 2.4 Hz, 2H), 2.49 (dd, *J* = 2.5, 2.5 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) 162.8, 123.3, 123.2, 77.1, 75.2, 52.1; IR (neat) 3296, 3078, 3053, 2945, 2129, 1732, 1614, 1435, 1371, 1333, 1275, 1205, 1159, 1024 cm^{-1}; Anal. Calcd for $C_6H_5BrO_2$: C, 38.13; H, 2.67. Found: C, 38.33; H, 2.78.

2-Propynyl (*Z*)-5-trimethylsilyl-2-penten-4-ynoate (25). To a mixture of $Pd(PPh_3)_4$ (150 mg, 0.13 mmol) and CuI (50 mg, 0.26 mmol) were added THF (6 mL), Et_3N (20 mL) and (trimethylsilyl)acetylene (7.0 mL, 50 mmol). Then a THF (4 mL) soution of **24** (1.9 g, 10 mmol) was added for 1 h under an argon atmosphere at room temperature. The reaction mixture was stirred for additional 30 min at this temperature. Then the mixture was poured into ice water, and extracted with Et_2O. The organic layer was washed with 0.5 N HCl aq. and dried over $MgSO_4$. Evaporation of the solvent gave an oil, which was further purified by silica gel column chromatography (hexane : AcOEt = 30 : 1) to give **25** (1.3 g, 63%): colorless oil; ^{1}H NMR (300 MHz, $CDCl_3$) 6.19 (d, *J* = 11.5 Hz, 1H), 6.10 (d, *J* = 11.5 Hz, 1H), 4.76 (d, *J* = 2.4 Hz, 2H), 2.46 (dd, *J* = 2.5 Hz, 1H), 0.23 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$) 163.5, 128.1, 123.9, 109.4, 100.4, 77.5, 75.0, 51.9, -0.4; IR (neat) 3294, 3078, 3034, 2961, 2901, 2151, 2131, 1736, 1717, 1607, 1404, 1279, 1252, 1221, 1161, 1042 cm^{-1}; Anal. Calcd for $C_{11}H_{14}O_2Si$: C, 64.04; H, 6.84. Found: C, 64.12; H, 6.96.

Methyl (*Z*)-3-bromo-2-propenoate (26) was prepared according to the literature.[1]

(*Z*)-5-Methoxycarbonyl-4-penten-2-ynyl (*Z*)-5-trimethylsilyl-2-penten-4-ynoate (27). To a mixture of $Pd(PPh_3)_4$ (45 mg, 0.039 mmol) and CuI (15 mg, 0.078 mmol) were added Et_3N (6 mL) and a THF (2 mL) solution of **25** (0.62 g, 3.0 mmol) and methyl (*Z*)-3-bromo-2-propenoate **26** (0.58 g, 3.6 mmol) under an argon atmosphere at room temperature. The rection mixture was stirred for 40 min. Then the mixture was poured into ice water, and extracted with Et_2O. The organic layer was washed with 0.5 N HCl aq. and dried over $MgSO_4$. Evaporation of the solvent gave an oil, which was further purified by silica gel column chromatography (hexane : AcOEt = 10 : 1) to give **27** (0.54 g, 63%): brown oil; ^{1}H NMR (300 MHz, $CDCl_3$) 6.21-6.10 (m, 4H), 5.00 (d, *J* = 1.4 Hz, 2H), 3.75 (s, 3H), 0.22 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$) 164.8, 163.4, 129.0, 128.1, 124.0, 122.1, 109.4, 100.4, 95.0, 82.9, 52.6, 51.5, -0.5; IR (neat) 3078, 3032, 2957, 2901, 2856, 2214, 2149, 1732, 1718, 1609, 1439, 1404, 1360, 1279, 1252, 1232, 1198, 1163, 1038 cm^{-1}; Anal. Calcd for $C_{15}H_{18}O_4Si$: C, 62.04; H, 6.25. Found: C, 61.87; H, 6.36.

(*Z*)-5-Methoxycarbonyl-4-penten-2-ynyl (*Z*)-2-penten-4-ynoate (8g). To a mixture of KF (0.20 g, 3.4 mmol) in MeOH (6 mL) was slowly added a THF (4 mL) soution of **27** (0.49 g, 1.7 mmol) at 0 °C under an argon atmosphere and the mixture was kept stirring for 40 min. The mixture was poured into water, and extracted with Et_2O. The organic layer was washed with saturated brine, and dried over $MgSO_4$. Evaporation of the solvent gave an oil, which was further purified by silica gel column chromatography (hexane : AcOEt = 7 : 1) to give **8g** (0.29 g, 77%): white solid, mp

97.7-98.3°C; ^{1}H NMR (300 MHz, $CDCl_3$) 6.27-6.10 (m, 4H), 5.00 (d, *J* = 1.2 Hz, 2H), 3.75 (s, 3H), 3.67 (d, *J* = 2.4 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) 164.7, 163.2, 129.4, 129.0, 123.3, 122.1, 94.7, 90.2, 83.0, 79.2, 52.7, 51.5; IR (KBr) 3258, 3084, 2997, 2951, 2212, 2097, 1728, 1716, 1616, 1560, 1441, 1402, 1360, 1232, 1213, 1178 cm^{-1}; HMRS Calcd for $C_{11}H_7O_2$ (M^+ - CH_3): 203.0344. Found: 203.0327.

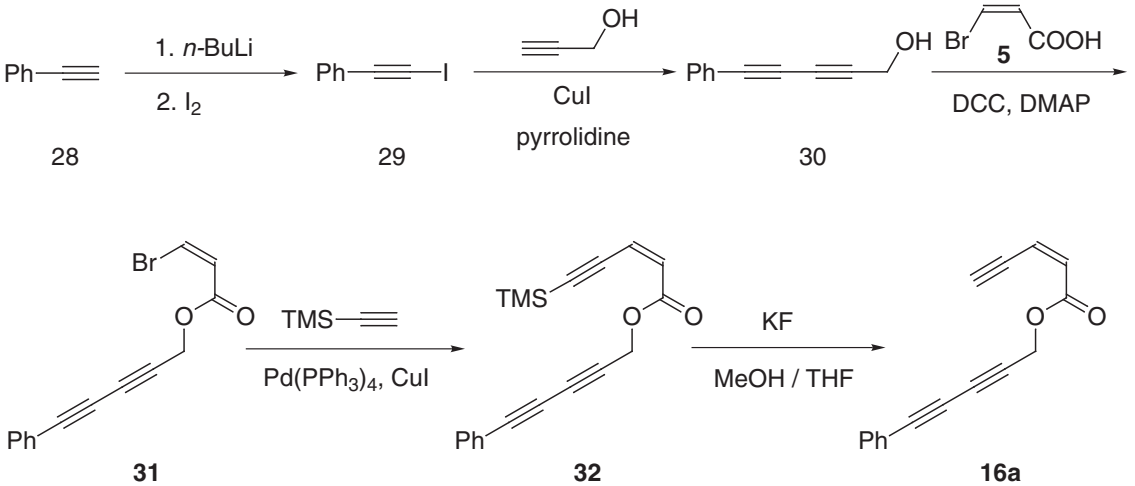
(*Z*)-5-Methoxycarbonyl-4-penten-2-ynyl (*Z*)-2-octen-4-ynoate (8h): brown oil; ^{1}H NMR (300 MHz, $CDCl_3$) 6.23-6.05 (m, 4H), 4.98 (d, *J* = 1.4 Hz, 2H), 3.75 (s, 3H), 2.42 (ddd, *J* = 7.1, 7.1, 2.4 Hz, 2H), 1.60 (ddddd, *J* = 7.3, 7.3, 7.3, 7.3, 7.3 Hz, 2H), 1.01 (dd, *J* = 7.4, 7.4 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) 164.8, 163.7, 128.9, 125.9, 125.4, 122.1, 105.2, 95.2, 82.8, 77.7, 52.4, 51.4, 22.0, 21.7, 13.4; IR (neat) 3080, 3020, 2966, 2907, 2874, 2245, 2208, 1732, 1609, 1437, 1410, 1360, 1279, 1232, 1197, 1161 cm^{-1}; HMRS Calcd for $C_{15}H_{16}O_4$: 260.1048. Found: 260.1051.

(*E*)-6,6,7,7,8,8,9,9,10,10,11,11,11-Tridecafluoro-4-undecen-2-ynyl (*Z*)-2-octen-4-ynoate (8i): colorless oil; ^{1}H NMR (300 MHz, $CDCl_3$) 6.30 (ddd, *J* = 15.9, 2.1, 2.1 Hz, 1H), 6.22 (ddd, *J* = 11.5, 2.4, 2.4 Hz, 1H), 6.11 (ddd, *J* = 15.7, 12.0, 12.0 Hz, 1H), 6.05 (d, *J* = 11.3 Hz, 1H), 4.90 (s, 2H), 2.42 (ddd, *J* = 7.0, 7.0, 2.4 Hz, 2H), 1.61 (ddddd, *J* = 7.5, 7.3, 7.3, 7.3, 7.1 Hz, 2H), 1.01 (dd, *J* = 7.4, 7.4 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) 163.6, 127.6 (dd, $J_{C\text{-}F}$ = 22.6, 22.6 Hz), 125.7, 125.6, 123.5-106.9 (m), 120.0 (dd, $J_{C\text{-}F}$ = 11.3, 11.3 Hz), 105.4, 90.9, 81.3, 77.8, 51.9, 22.2, 21.9, 13.5; IR (neat) 3074, 3040, 2968, 2939, 2878, 2210, 1734, 1641, 1609, 1431, 1414, 1364, 1240, 1202, 1167, 1146, 1121, 1067, 1032 cm^{-1}; HMRS Calcd for $C_{19}H_{13}F_{13}O_2$: 520.0707. Found: 520.0688.

(*Z*)-6-Methoxycarbonyl-5-hexen-3-ynyl (*Z*)-2-penten-4-ynoate (18d): yellow solid, mp 46.5-47.5°C; ^{1}H NMR (300 MHz, $CDCl_3$) 6.25-6.04 (m, 4H), 4.33 (dd, *J* = 6.9, 6.9 Hz, 2H), 3.73 (s, 3H), 3.62 (d, *J* = 2.0 Hz, 1H), 2.84 (ddd, *J* = 6.8, 6.8, 2.0 Hz, 2H); ^{13}C NMR (75 MHz, $CDCl_3$) 165.0, 163.9, 130.2, 127.9, 123.4, 122.5, 98.7, 89.6, 79.4, 78.8, 62.1, 51.4, 20.4; IR (KBr) 3294, 3092, 3038, 2959, 2905, 2214, 2097, 1720, 1605, 1439, 1406, 1238, 1192, 1128 cm^{-1}; HMRS Calcd for $C_{13}H_{11}O_4$ (M^+ - H): 231.0657. Found: 231.0669.

Preparation of Enyne-Diynes 16. A Representative Procedure.

Scheme S3



5-Phenyl-2,4-pentadiyn-1-ol (30) was prepared according to the literature.[2]

5-Phenyl-2,4-pentadiynyl (*Z*)-3-bromo-2-propenoate (31). To a mixture of (*Z*)-3-bromo-2-propenoic acid **5** (1.8 g, 12 mmol) and DMAP (0.15 g, 1.2 mmol) were added

CH_2Cl_2 (5.3 mL) and a CH_2Cl_2 (10 mL) solution of 5-phenyl-2,4-pentadiyn-1-ol **30** (1.9 g, 12 mmol) under an argon atmosphere at room temperature. The reaction mixture was cooled to 0 _, and DCC (2.9 g, 14 mmol) was added and the mixture was stirred for 5 min. After stirring for additional 35 min at room temperature, the reaction mixture was filtered with celite and the residue was washed with Et_2O. The organic layer was washed with 0.5 N HCl aq. and saturated $NaHCO_3$ aq. and dried over $MgSO_4$. Evaporation of the solvent gave a solid, which was further purified by silica gel column chromatography (hexane : AcOEt = 20 : 1) to give **31** (1.3 g, 38%): yellow solid, mp 63.5-64.2 °C; ^{1}H NMR (300 MHz, $CDCl_3$) 7.49-7.46 (m, 2H), 7.39-7.20 (m, 3H), 7.09 (d, *J* = 8.5 Hz, 1H), 6.66 (d, *J* = 8.5 Hz, 1H), 4.91 (s, 2H); ^{13}C NMR (75 MHz, $CDCl_3$) 162.8, 132.7, 129.5, 128.4, 123.3, 121.1, 79.0, 75.6, 73.0, 71.6, 52.7; IR (KBr) 3082, 3065, 3049, 2928, 2251, 2224, 1732, 1686, 1655, 1609, 1578, 1560, 1541, 1508, 1491, 1437, 1373, 1337, 1204, 1165, 1070, 1024 cm^{-1}; HMRS Calcd for $C_{14}H_9BrO_2$: 287.9786. Found: 287.9795.

5-Phenyl-2,4-pentadiynyl (*Z*)-5-trimethylsilyl-2-penten-4-ynoate (32). To a mixture of $Pd(PPh_3)_4$ (30 mg, 0.026 mmol) and CuI (10 mg, 0.052 mmol) were added Et_3N (4 mL), a THF (1 mL) solution of **31** (0.29 g, 1.0 mmol), and (trimethylsilyl)acetylene (0.21 mL, 1.5 mmol) under an argon atmosphere at room temperature. The reaction mixture was stirred for 3.5 h. Then the mixture was poured into ice water, and extracted with Et_2O. The organic layer was washed with 0.5 N HCl aq. and dried over $MgSO_4$. Evaporation of the solvent gave a solid, which was further purified by silica gel column chromatography (hexane : AcOEt = 50 : 1) to give **32** (0.19 g, 63%): yellow solid, mp 43.2-44.2 °C; ^{1}H NMR (300 MHz, $CDCl_3$) 7.49-7.45 (m, 2H), 7.39-7.27 (m, 3H), 6.21 (d, *J* = 11.7 Hz, 1H), 6.10 (d, *J* = 11.7 Hz, 1H), 4.91 (s, 2H), 0.24 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$) 163.4, 132.6, 129.4, 128.4, 128.0, 124.0, 121.1, 109.6, 100.4, 78.8, 76.0, 73.1, 71.4, 52.5, -0.4; IR (KBr) 3084, 3065, 3038, 2957, 2928, 2901, 2864, 2251, 2224, 2147, 1730, 1605, 1489, 1443, 1402, 1364, 1250, 1223, 1161, 1034 cm^{-1}; Anal. Calcd for $C_{19}H_{18}O_2Si$: C, 74.47; H, 5.92. Found: C, 74.35; H, 6.02.

5-Phenyl-2,4-pentadiynyl (*Z*)-2-penten-4-ynoate (16a). To a mixture of KF (62 mg, 1.1 mmol) in MeOH (1.3 mL) was slowly added a THF (1.3 mL) solution of **32** (111 mg, 0.36 mmol) at 0 °C under an argon atmosphere and the mixture was kept stirring for 45 min. The mixture was poured into water, and extracted with Et_2O. The organic layer was washed with saturated brine, and dried over $MgSO_4$. Evaporation of the solvent gave a solid, which was further purified by silica gel column chromatography (hexane : AcOEt = 20 : 1) to give **16a** (43 mg, 51%): white solid, mp 86.2-87.7 °C; ^{1}H NMR (300 MHz, $CDCl_3$) 7.49-7.46 (m, 2H), 7.39-7.27(m, 3H), 6.24 (d, *J* = 11.9 Hz, 1H), 6.18 (dd, *J* = 11.5, 2.2 Hz, 1H), 4.91 (s, 2H), 3.66 (d, *J* = 2.2 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) 163.2, 132.6, 129.5, 129.3, 128.4, 123.5, 121.1, 90.2, 79.3, 78.8, 75.8, 73.0, 71.4, 52.6; IR (KBr) 3269, 3055, 3038, 2930, 2255, 2225, 2093, 1728, 1686, 1611, 1491, 1441, 1402, 1367, 1223, 1175, 1119, 1072, 1026, 1003 cm^{-1}; HMRS Calcd for $C_{16}H_{10}O_2$: 234.0680. Found: 234.0700.

5-Phenyl-2,4-pentadiynyl (*Z*)-2-octen-4-ynoate (16b): yellow solid, mp 32.5-33.5 °C; ^{1}H NMR (300 MHz, $CDCl_3$) 7.49-7.45 (m, 2H), 7.38-7.27 (m, 3H), 6.21 (ddd, *J* = 11.5, 2.4, 2.4 Hz, 1H), 6.05 (d, *J* = 11.3 Hz, 1H), 4.89 (s, 2H), 2.43 (ddd, *J* = 7.1, 7.1, 2.4 Hz, 2H), 1.62 (ddddd, *J* = 7.3, 7.3, 7.3, 7.3, 7.3 Hz, 2H), 1.02 (dd, *J* = 7.4, 7.4 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) 163.8, 132.6, 129.4, 128.4, 125.9, 125.6, 121.1, 105.5,

78.6, 77.8, 76.1, 73.1, 71.2, 52.3, 22.0, 21.8, 13.5; IR (KBr) 3080, 3065, 3036, 2963, 2934, 2909, 2874, 2831, 2249, 2208, 1732, 1607, 1491, 1412, 1364, 1275, 1231, 1207, 1155, 1117 cm^{-1}; HMRS Calcd for $C_{19}H_{16}O_2$: 276.1149. Found: 276.1153.

6-Vinyl-4,5-dihydro-3*H*-benzo[c]oxepin-1-one (21). A mixture of $Pd(PPh_3)_4$ (140 mg, 0.12 mmol) and DPPF (130 mg, 0.32 mmol) in dry toluene (595 mL) in 1 L flask was kept at 100 _. To this mixture was slowly added a toluene (5 mL) solution of **20** (56 mg, 0.30 mmol) for 5 h with syringe pump. After stirring for additional 1.5 h, toluene was evaporated and the resulting mixture was passed through a short silica gel column chromatography (hexane and Et_2O). The residue was further purified by silica gel column chromatography (hexane : AcOEt = 10 : 1) to give **21** (12 mg, 22%): yellow oil; ^1H NMR (300 MHz, $CDCl_3$) 7.63-7.57 (m, 2H), 7.31 (dd, *J* = 7.7, 7.7 Hz, 1H), 6.99 (dd, *J* = 17.3, 11.1 Hz, 1H), 5.63 (dd, *J* = 17.3, 1.0 Hz, 1H), 5.40 (dd, 11.1, 1.2 Hz, 1H), 4.12 (dd, *J* = 6.2, 6.2 Hz, 2H), 2.94 (dd, *J* = 7.1, 7.1 Hz, 2H), 2.07 (dddd, *J* = 7.1, 6.9, 6.5, 6.4 Hz, 2H); ^{13}C NMR (75 MHz, $CDCl_3$) 172.5, 136.5, 134.2, 133.6, 132.7, 130.3, 129.3, 127.2, 118.1, 66.2, 27.4, 24.3; IR (neat) 3067, 2961, 2926, 2878, 2856, 1722, 1470, 1285, 1259, 1182, 1111, 1059 cm^{-1}; HMRS Calcd for $C_{12}H_{12}O_2$: 188.0837. Found: 188.0835.

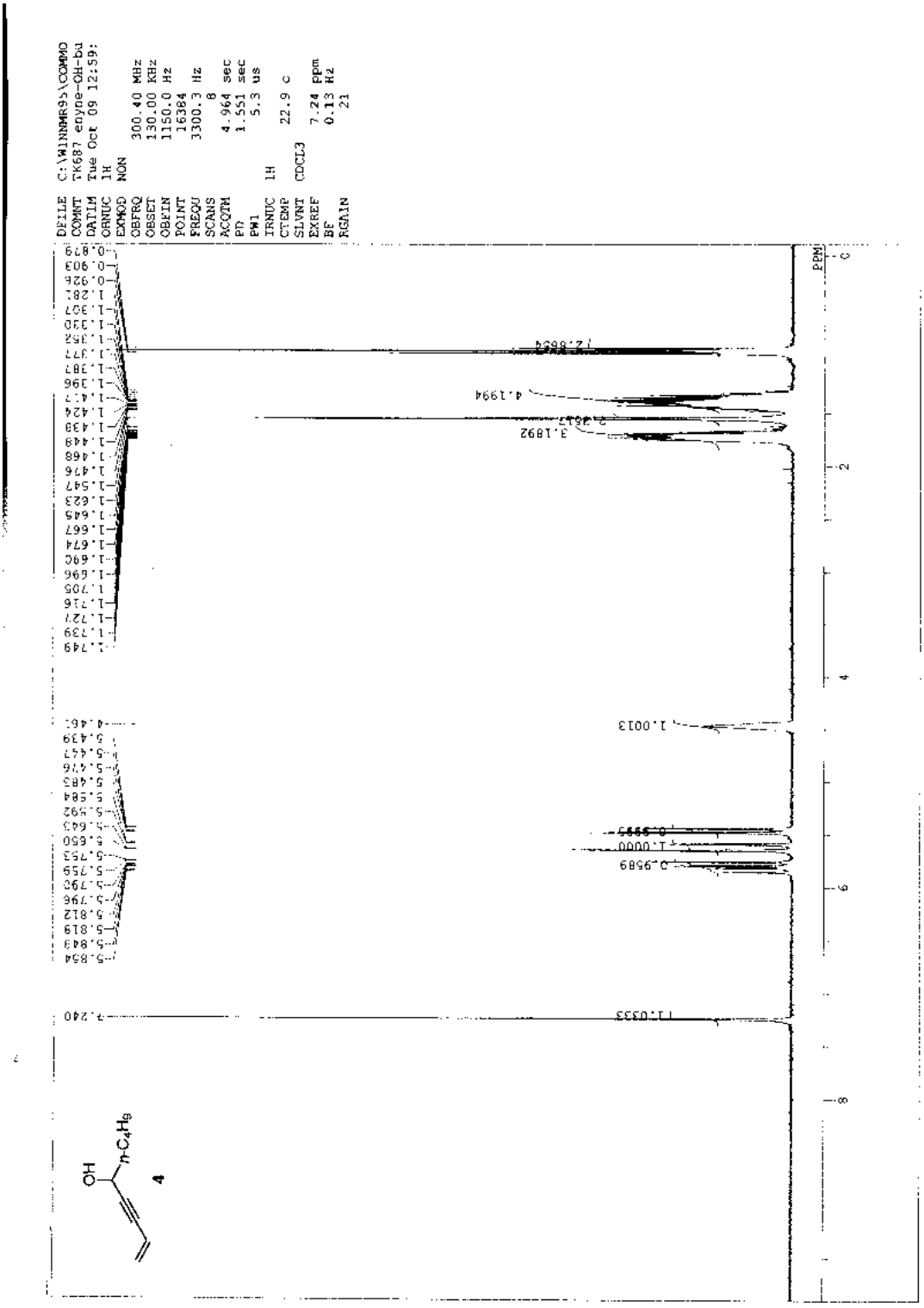
Synthesis of 3-*n*-Butylphthalide.

3-Butyl-4-formylphthalide (22). Into a methanol (15 mL) solution of **9k** (520 mg, 2.4 mmol) at –78 °C was bubbled a stream of O_3 in O_2 until the solution became blue and this color maintained for 2 min. The reaction was purged with O_2 until the blue color disappeared, then dimethyl sulfide (1.0 mL) was added. The mixture warmed to room temperature with stirring. Then the mixture was poured into water, and extracted with Et_2O. The organic layer was washed with water, dried over Na_2SO_4, and evaporated to give **22** (500 mg, 96%): yellow solid, mp 51.0-51.8 °C; ^1H NMR (300 MHz, $CDCl_3$) 10.13 (s, 1H), 8.12 (d, *J* = 7.5 Hz, 1H), 8.11 (d, *J* = 7.5 Hz, 1H), 7.75 (dd, *J* = 7.6, 7.6 Hz, 1H), 5.92 (dd, *J* = 7.8, 2.3 Hz, 1H), 2.31-2.23 (m, 1H), 1.72-1.60 (m, 1H), 1.53-1.24 (m, 4H), 0.86 (dd, *J* = 7.0, 7.0 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) 190.8, 169.2, 149.9, 138.3, 131.1, 130.6, 129.8, 127.8, 82.7, 32.8, 27.0, 22.1, 13.7; IR (neat) 3069, 3034, 2959, 2932, 2862, 2743, 1757, 1701, 1595, 1350, 1265, 1223, 1086, 1045, 1003 cm^{-1}; HMRS Calcd for $C_{13}H_{14}O_3$: 218.0942. Found: 218.0943.

3-Butylphthalide (23). To a mixture of $RhCl(PPh_3)_3$ (460 mg, 0.50 mmol) in toluene (1.0 mL) was added a toluene (1.0 mL) solution of **22** (110 mg, 0.50 mmol) at room temperature. Then the mixture was stirred at 120 °C for 1 h. The mixture was passed through a short silica gel column chromatography (hexane and Et_2O). The residue was further purified by silica gel column chromatography (hexane : EtOAc = 10 : 1) to give **23** (75 mg, 78%): colorless oil; ^1H NMR (300 MHz, $CDCl_3$) 7.88 (d, *J* = 7.7 Hz, 1H), 7.65 (dd, *J* = 7.5, 7.5 Hz, 1H), 7.50 (dd, *J* = 7.6, 7.6 Hz, 1H), 7.41 (d, *J* = 7.7 Hz, 1H), 5.46 (dd, *J* = 7.8, 3.9 Hz, 1H), 2.09-1.97 (m, 1H), 1.81-1.69 (m, 1H), 1.53-1.29 (m, 4H), 0.89 (dd, *J* = 7.1, 7.1 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) 170.6, 150.1, 133.9, 129.0, 126.2, 125.7, 121.7, 81.4, 34.4, 26.8, 22.4, 13.8; IR (neat) 3069, 3057, 3032, 2957, 2934, 2872, 1763, 1466, 1286, 1213, 1063 cm^{-1}; HMRS Calcd for $C_{12}H_{14}O_2$: 190.0993. Found: 190.0995.

References

(1) Weir, J. R.; Patal, B. A.; Heck, R. F. *J. Org. Chem.* **1980**, *45*, 4926-4931.
(2) Alami, M.; Ferri, F. *Tetrahedron. Lett.* **1996**, *37*, 2763-2766.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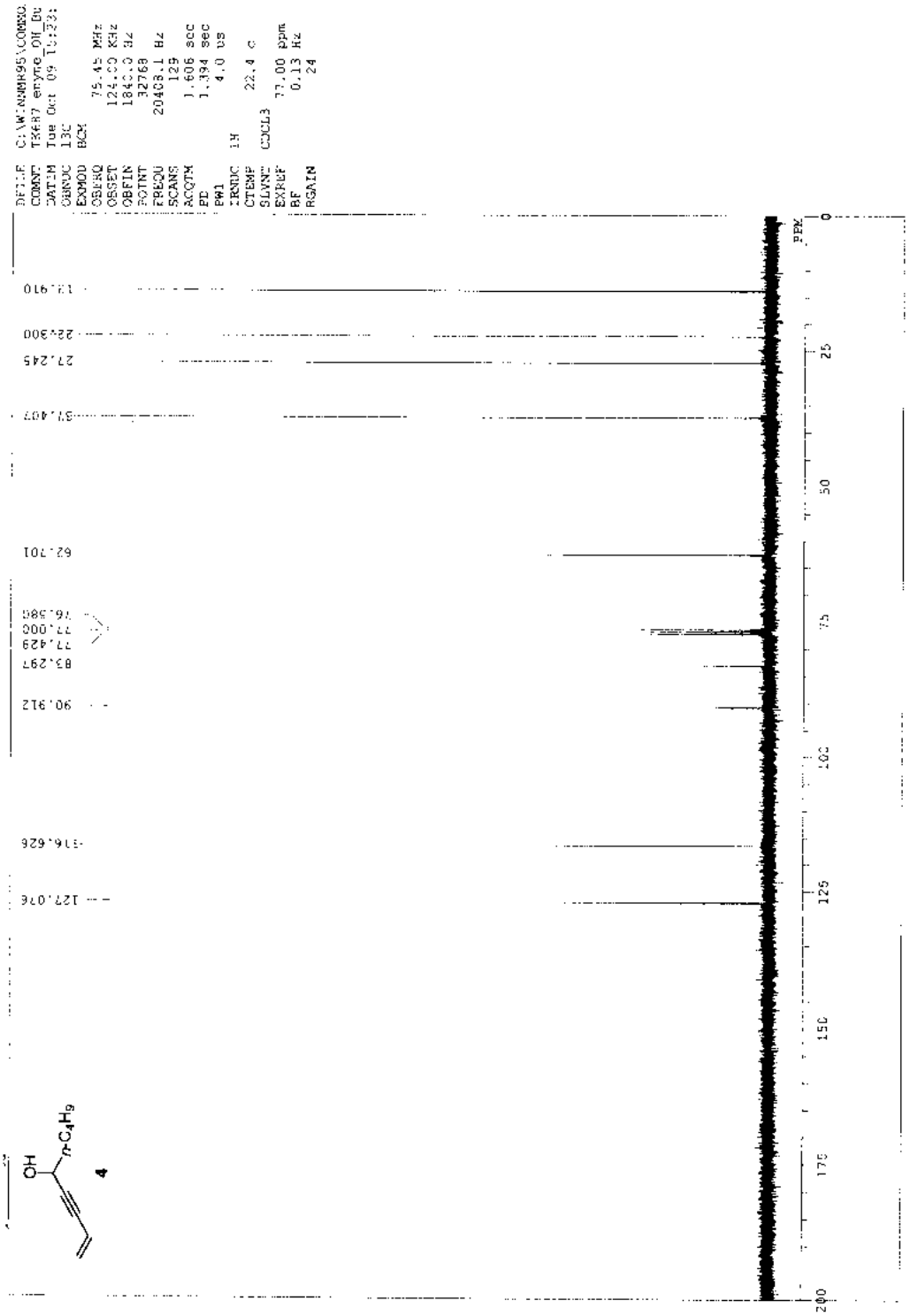
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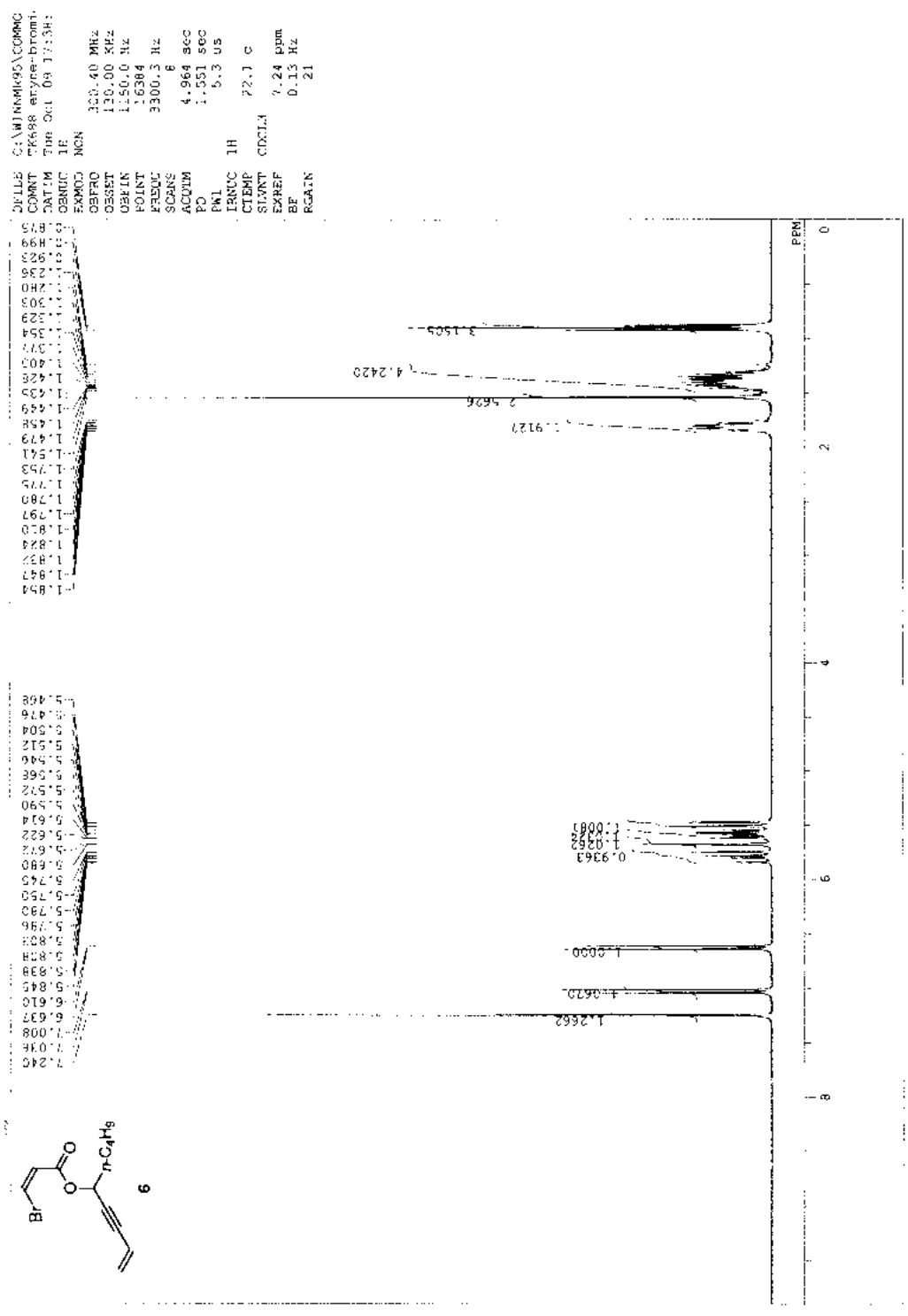
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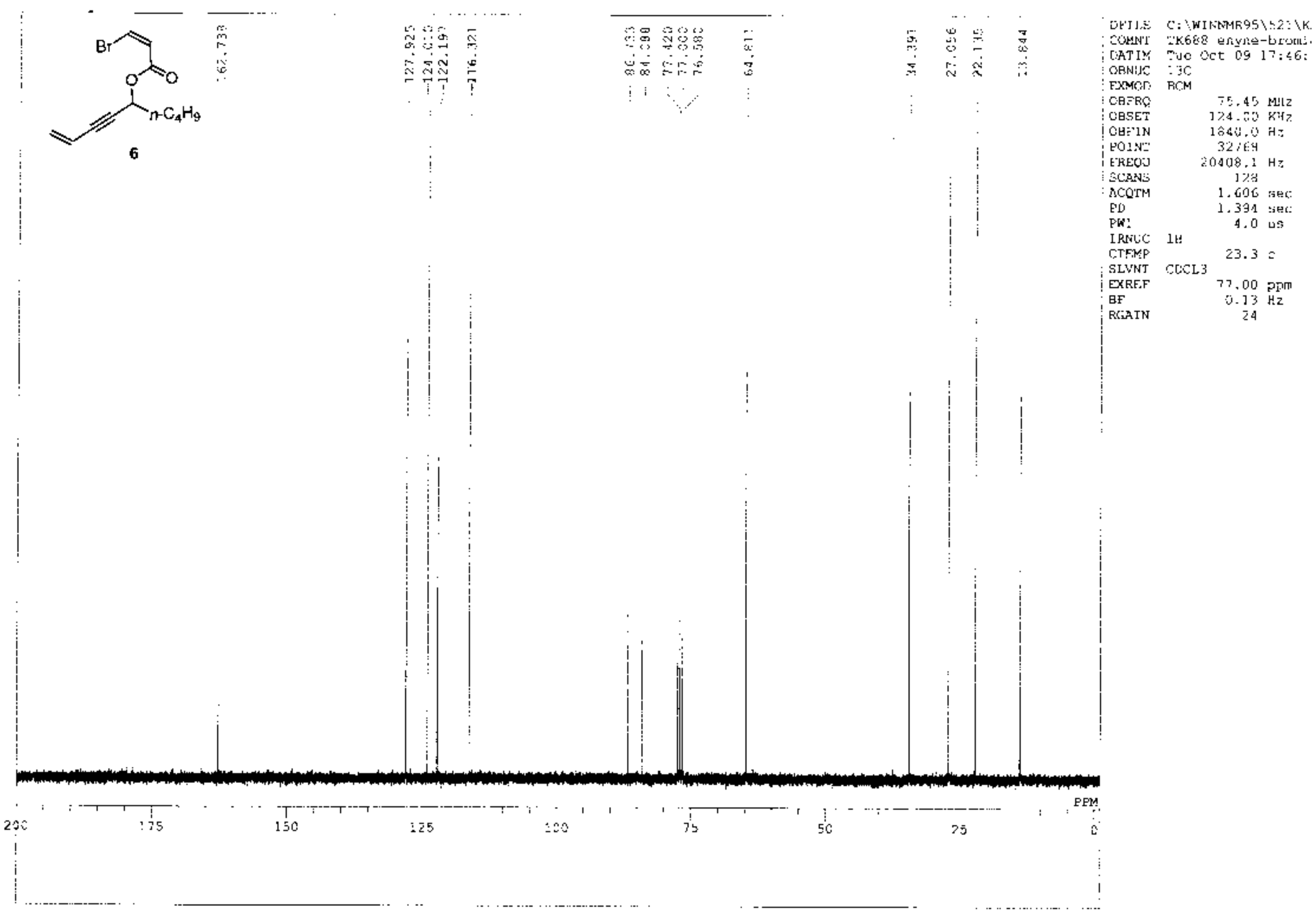
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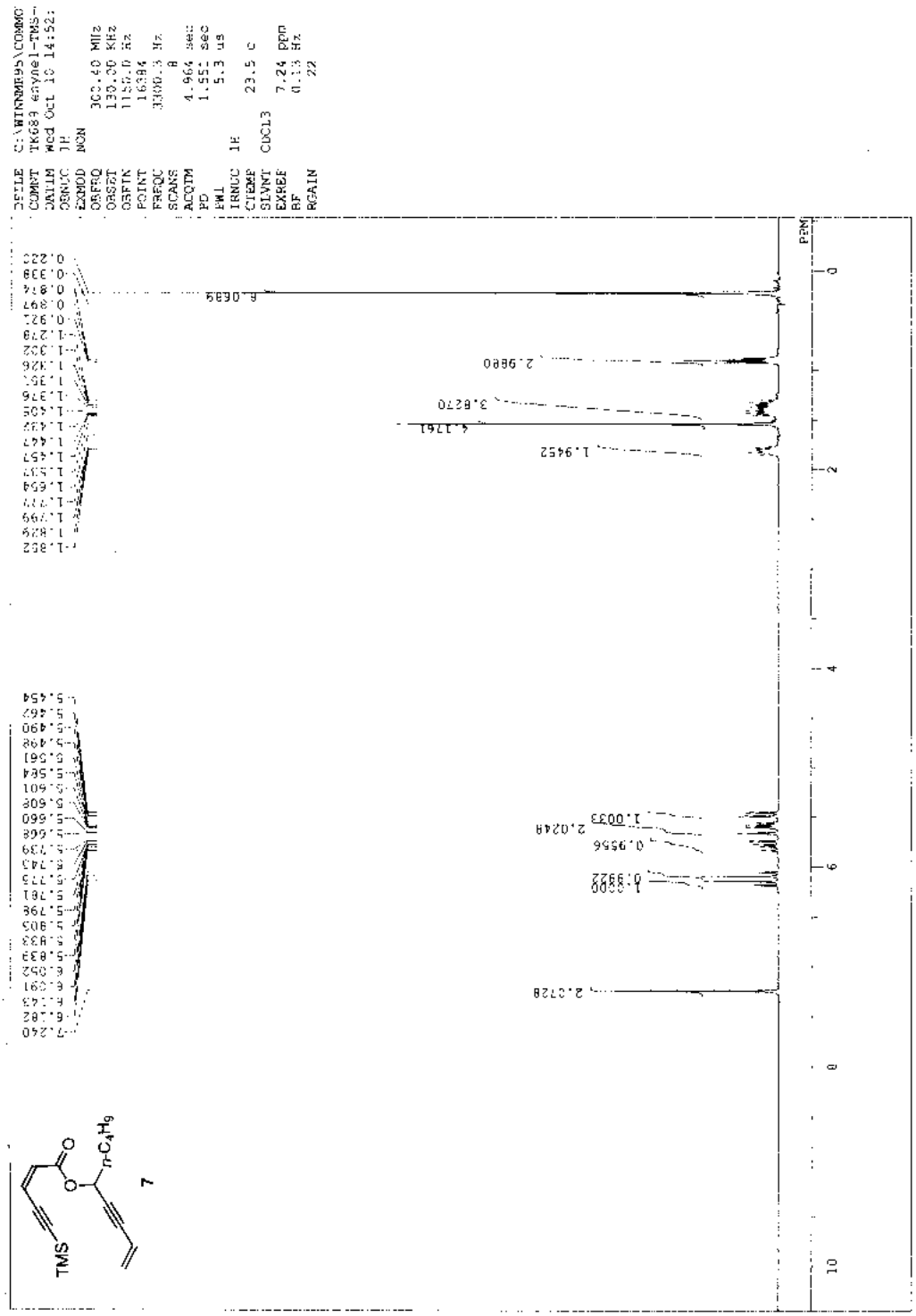
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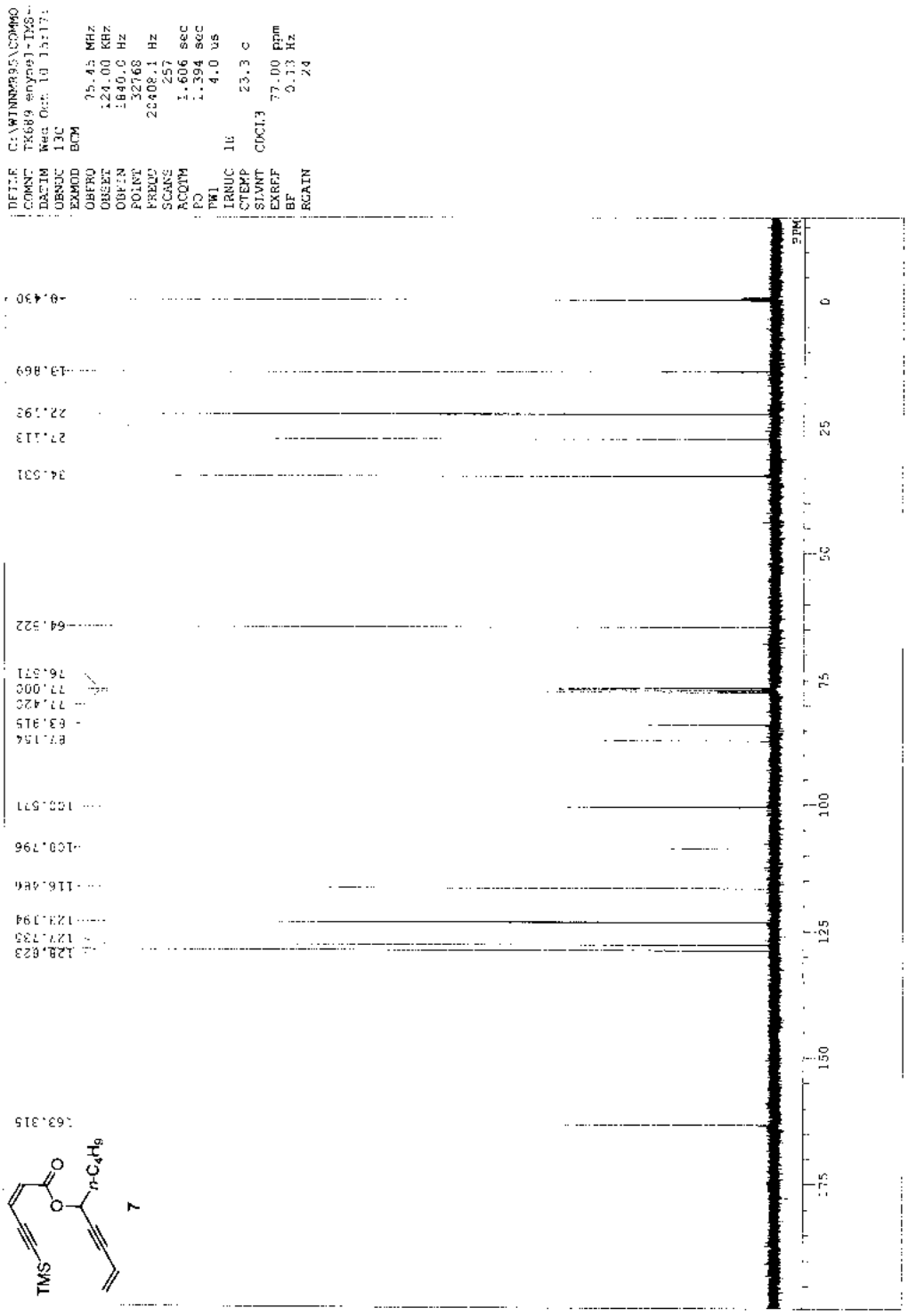
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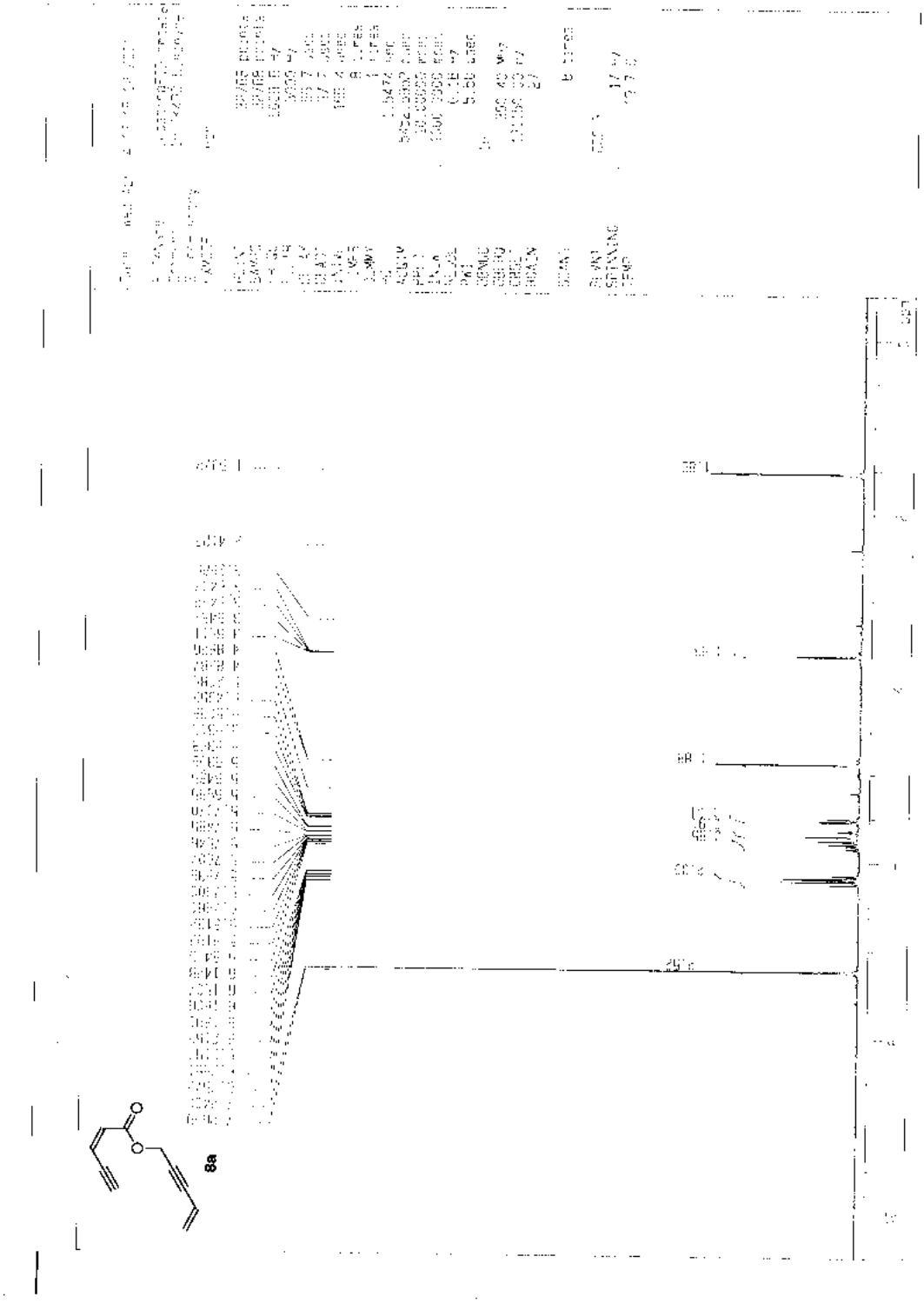
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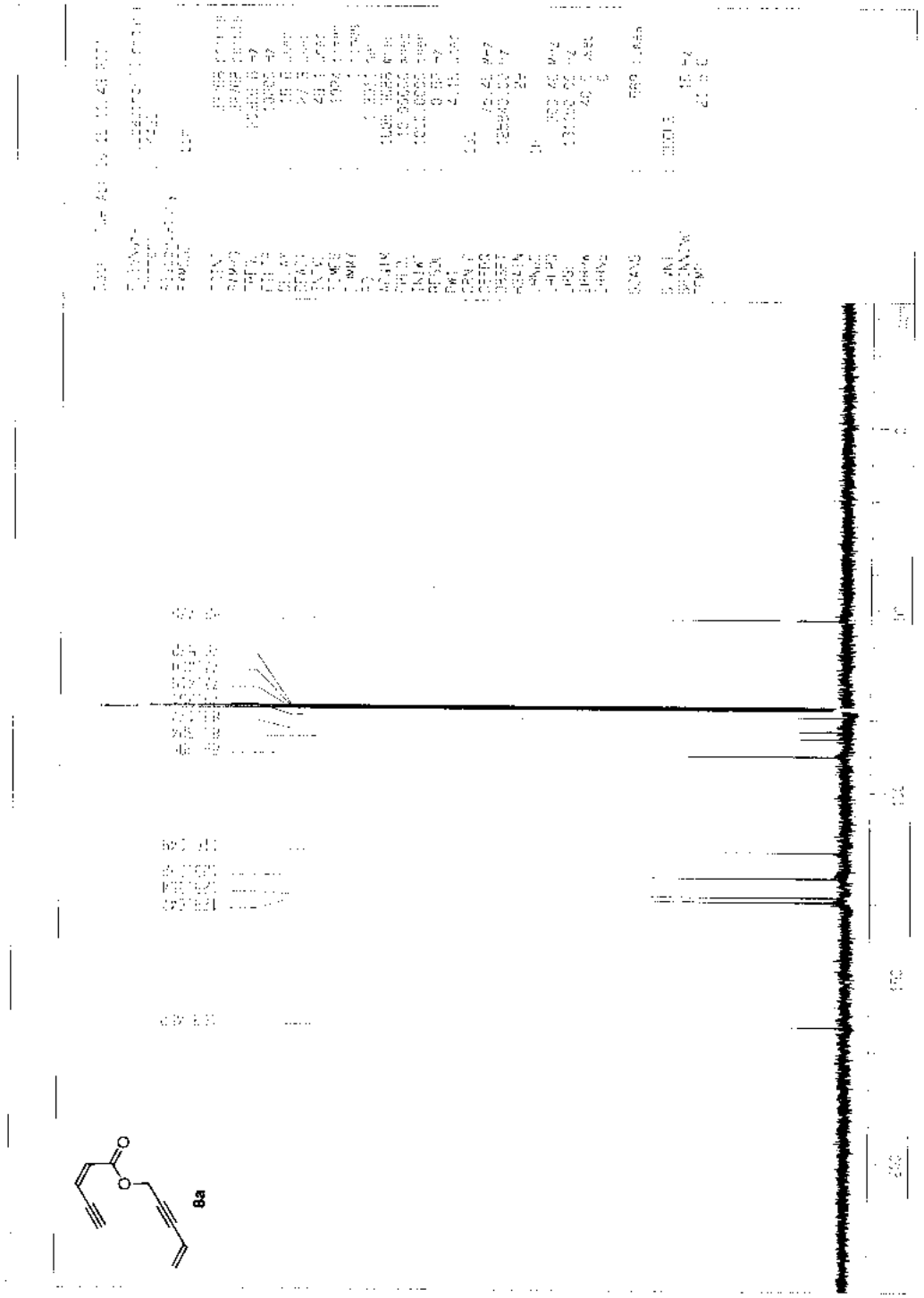
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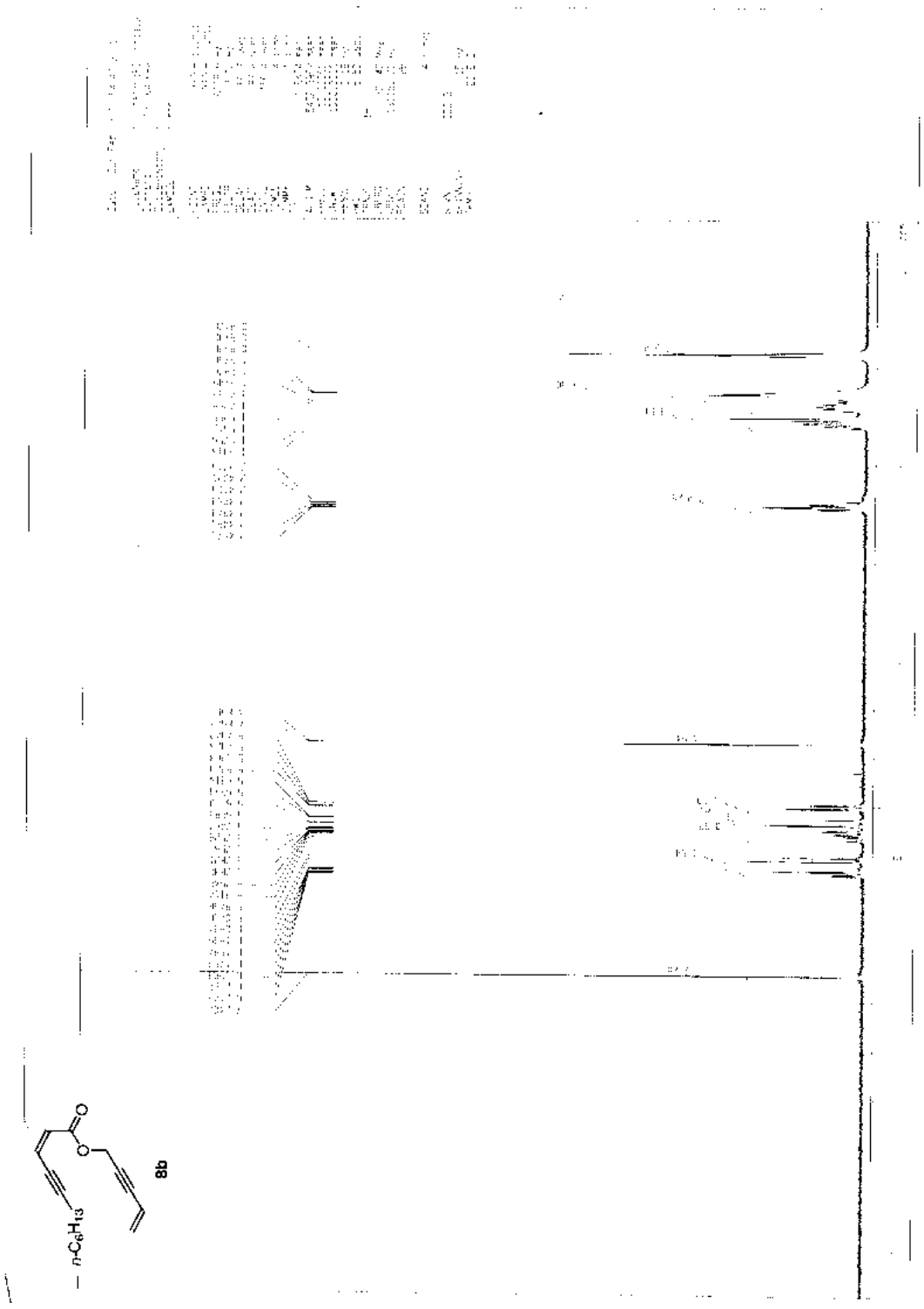
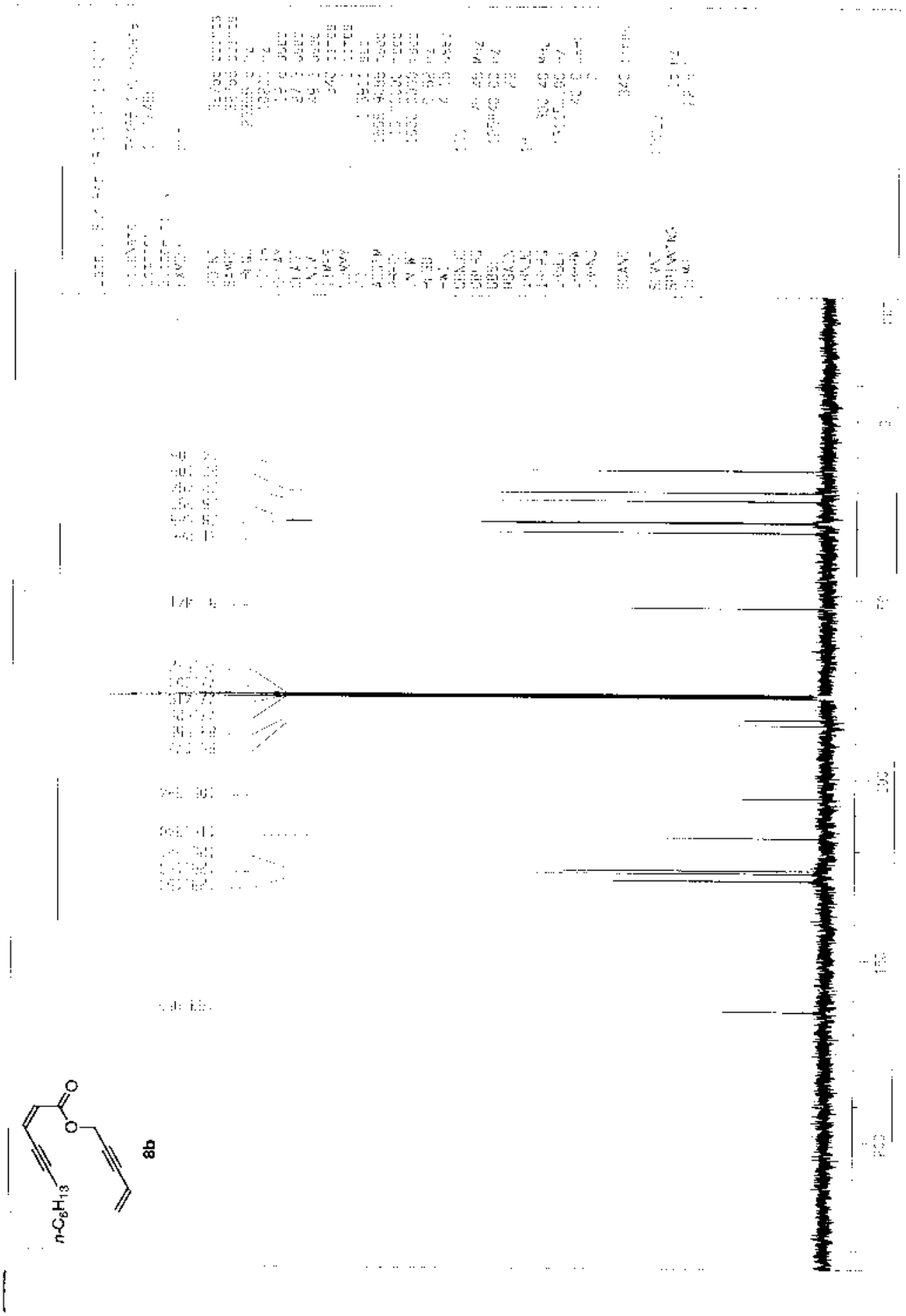
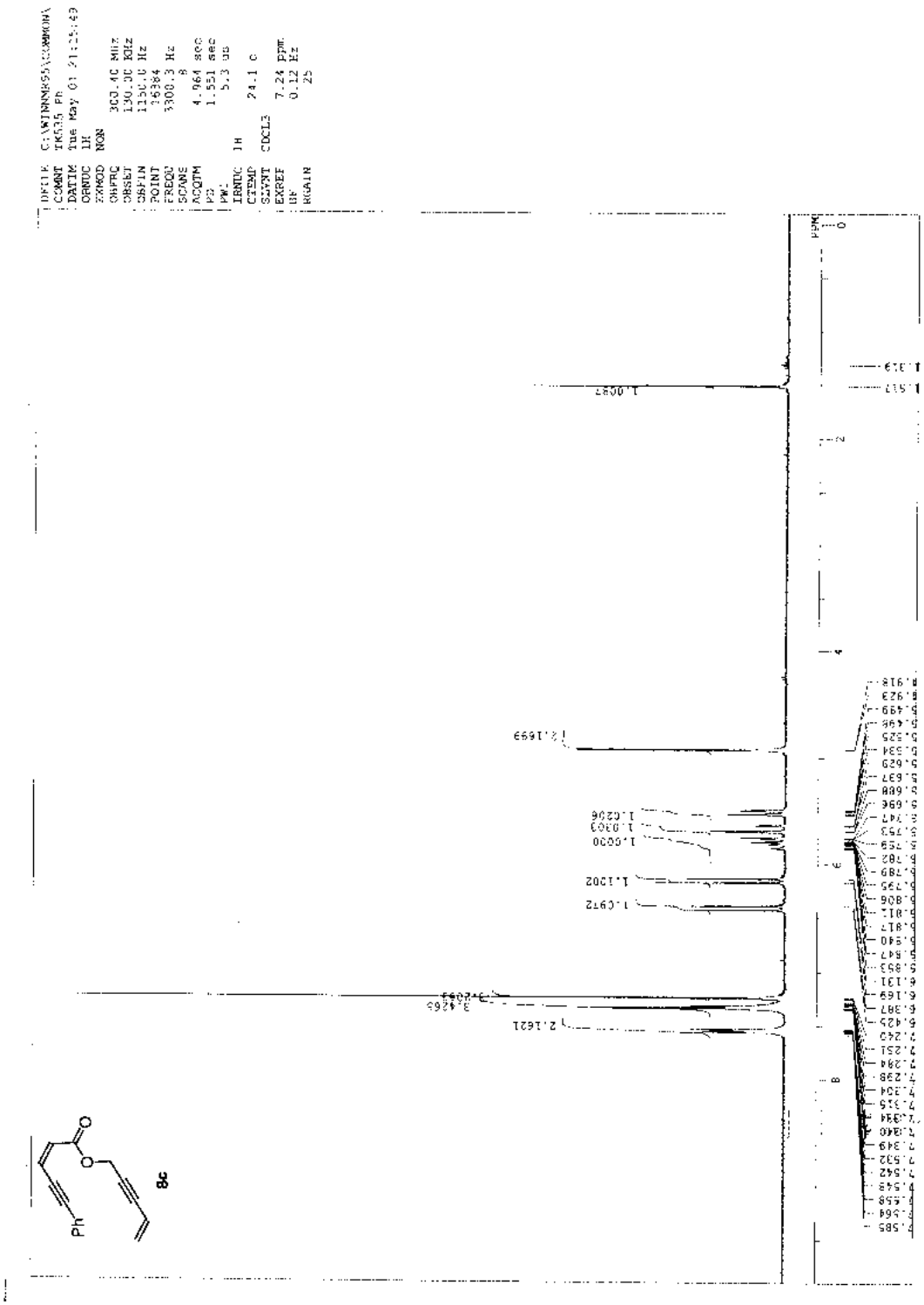
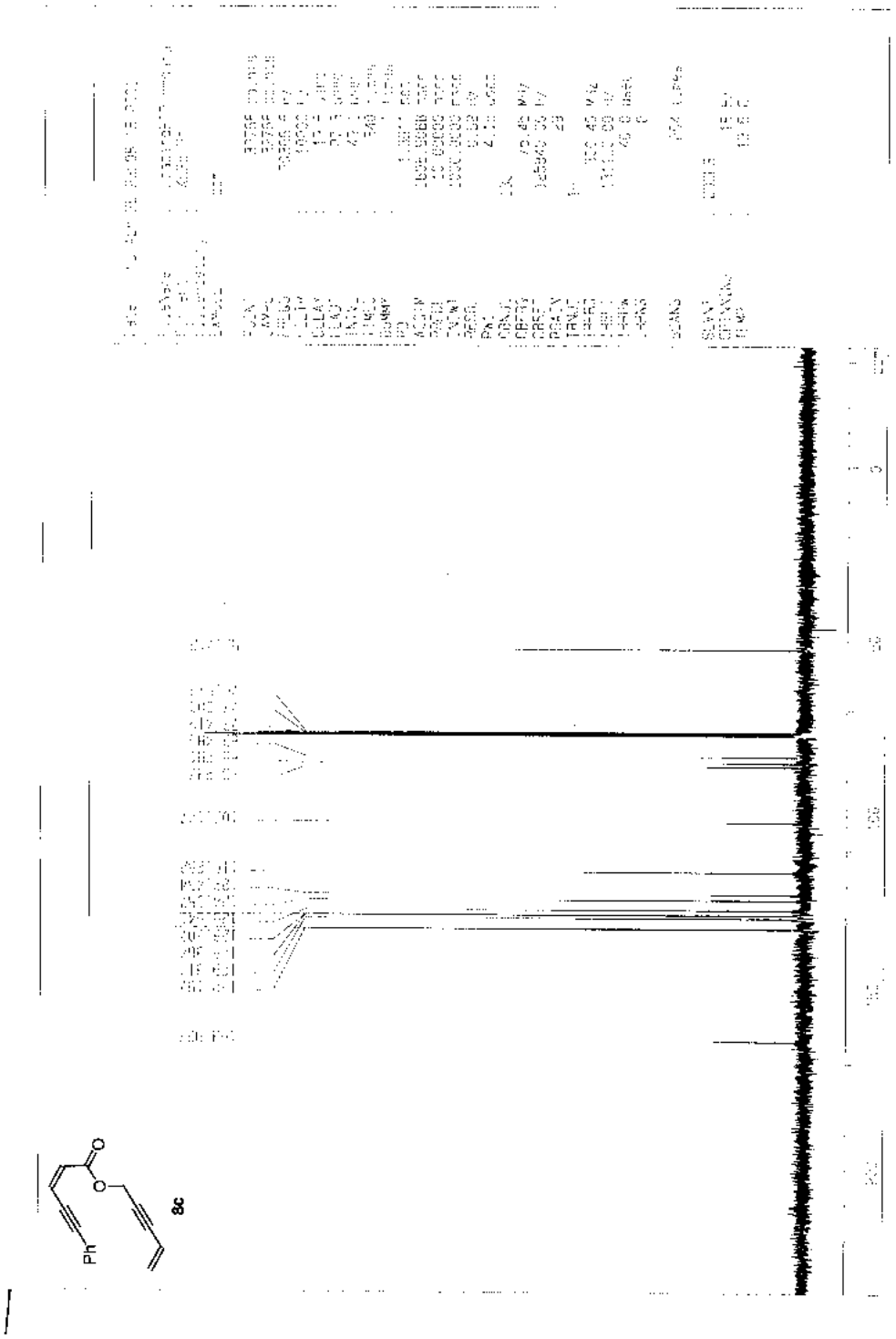
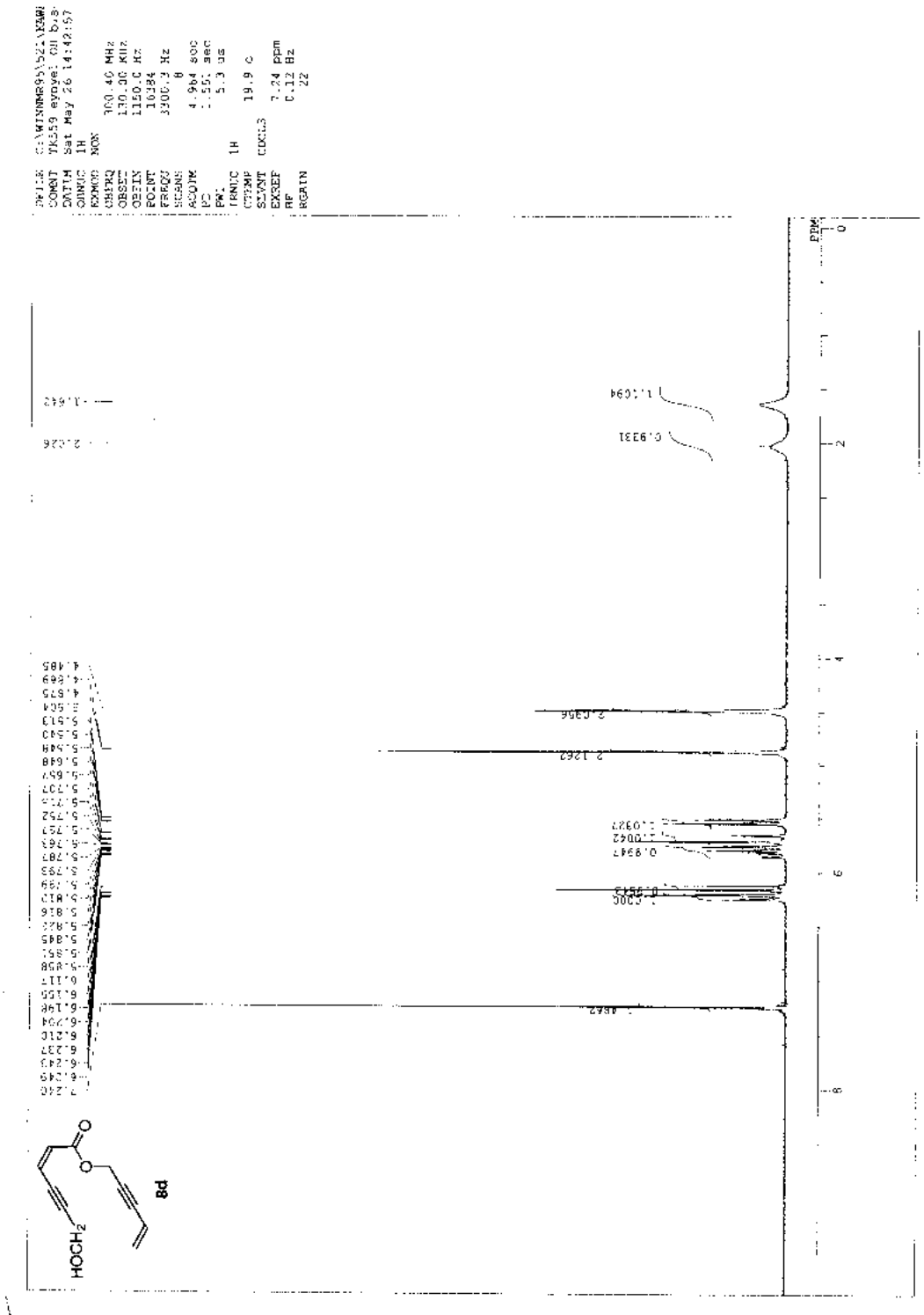
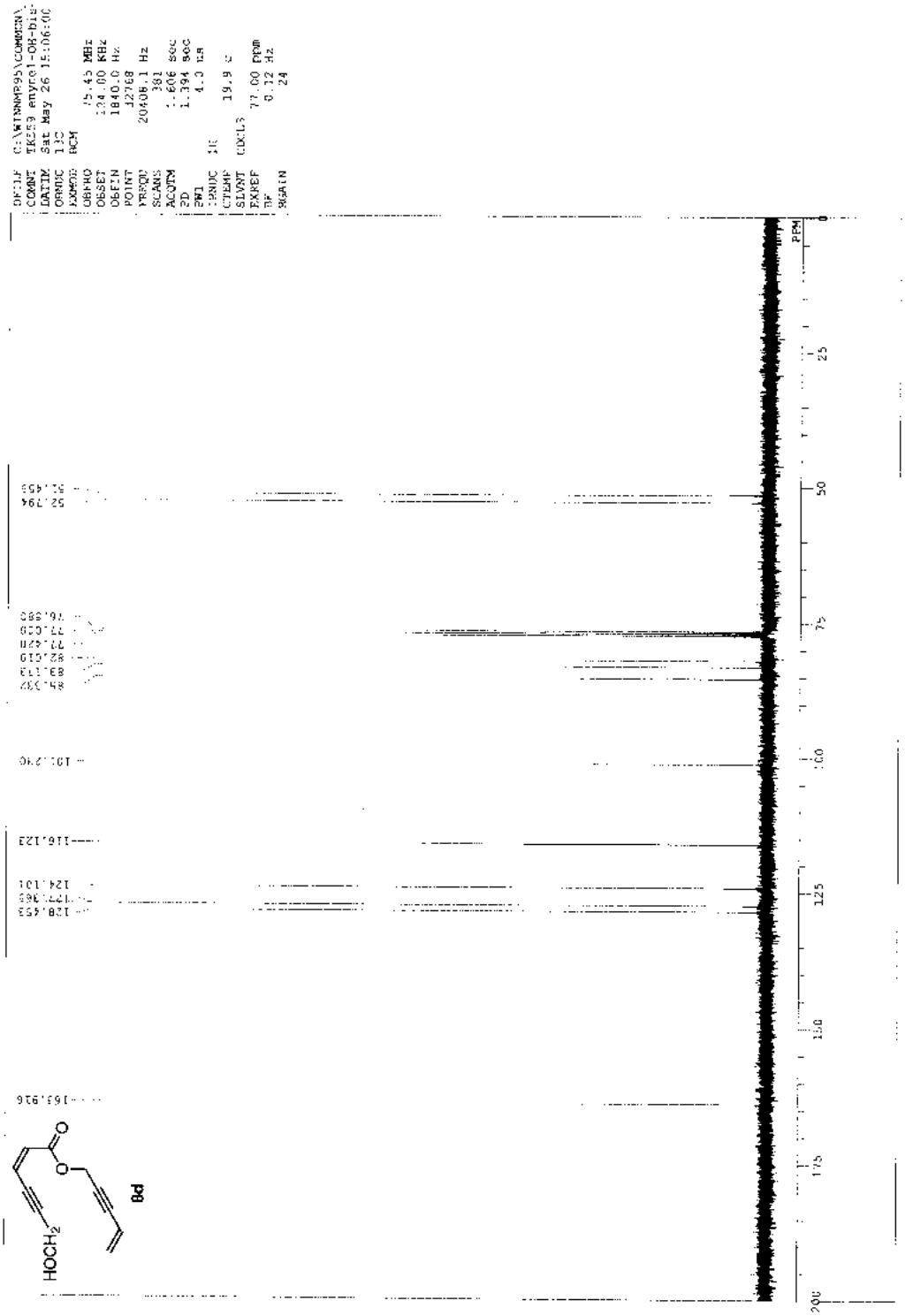

8b
n-C6H13

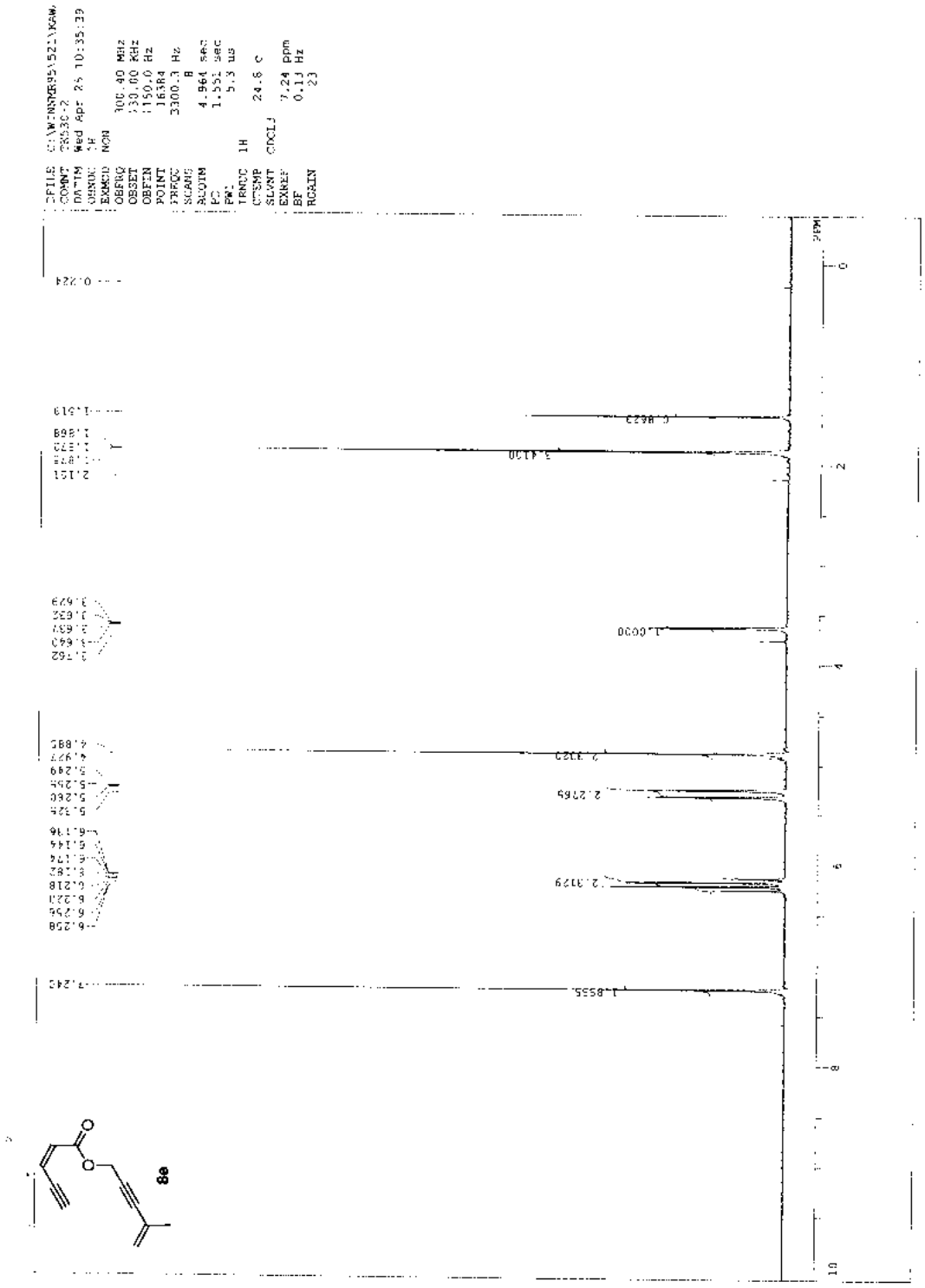

8b

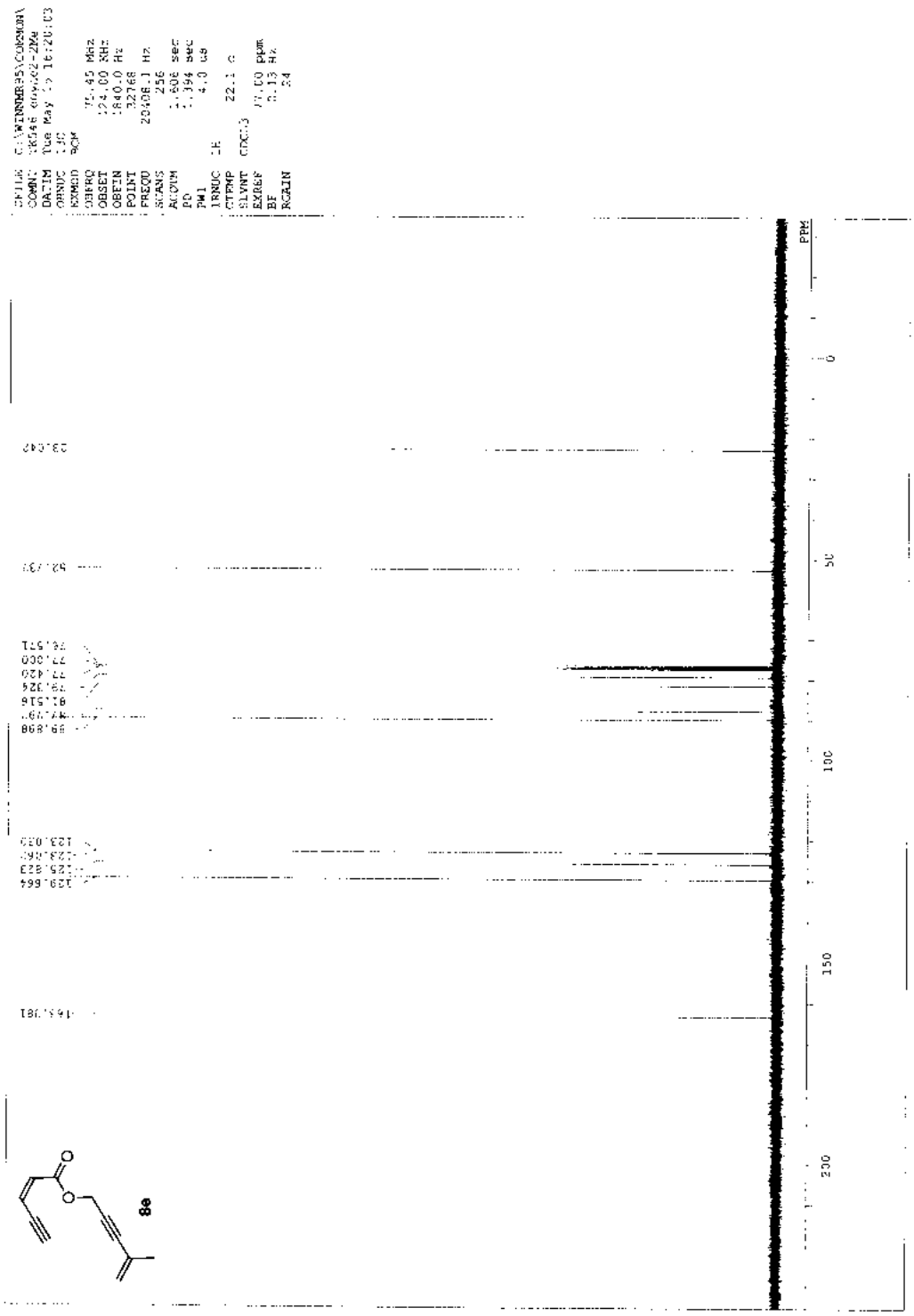

8c
Ph
O
O

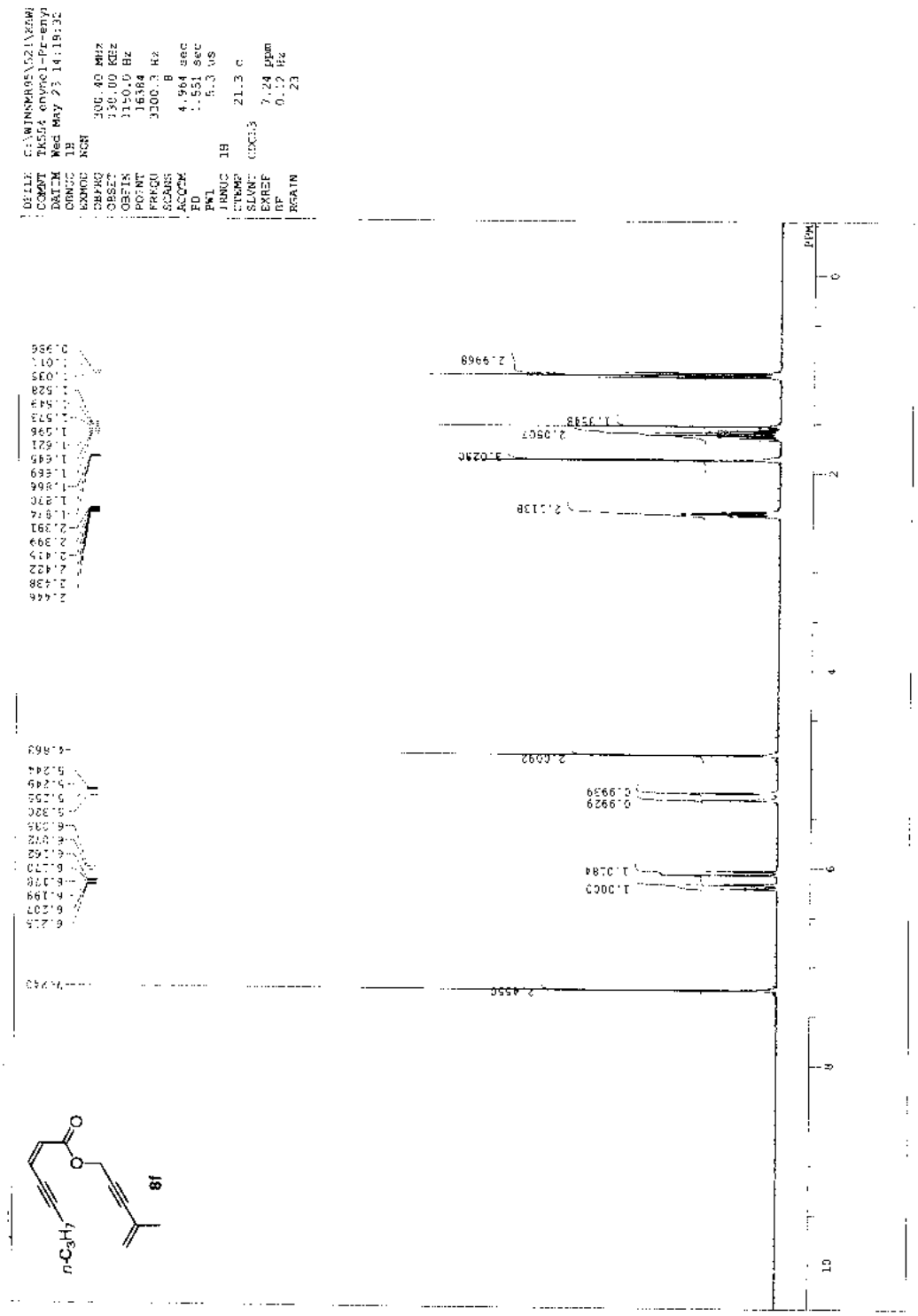

O
O
Ph
8c

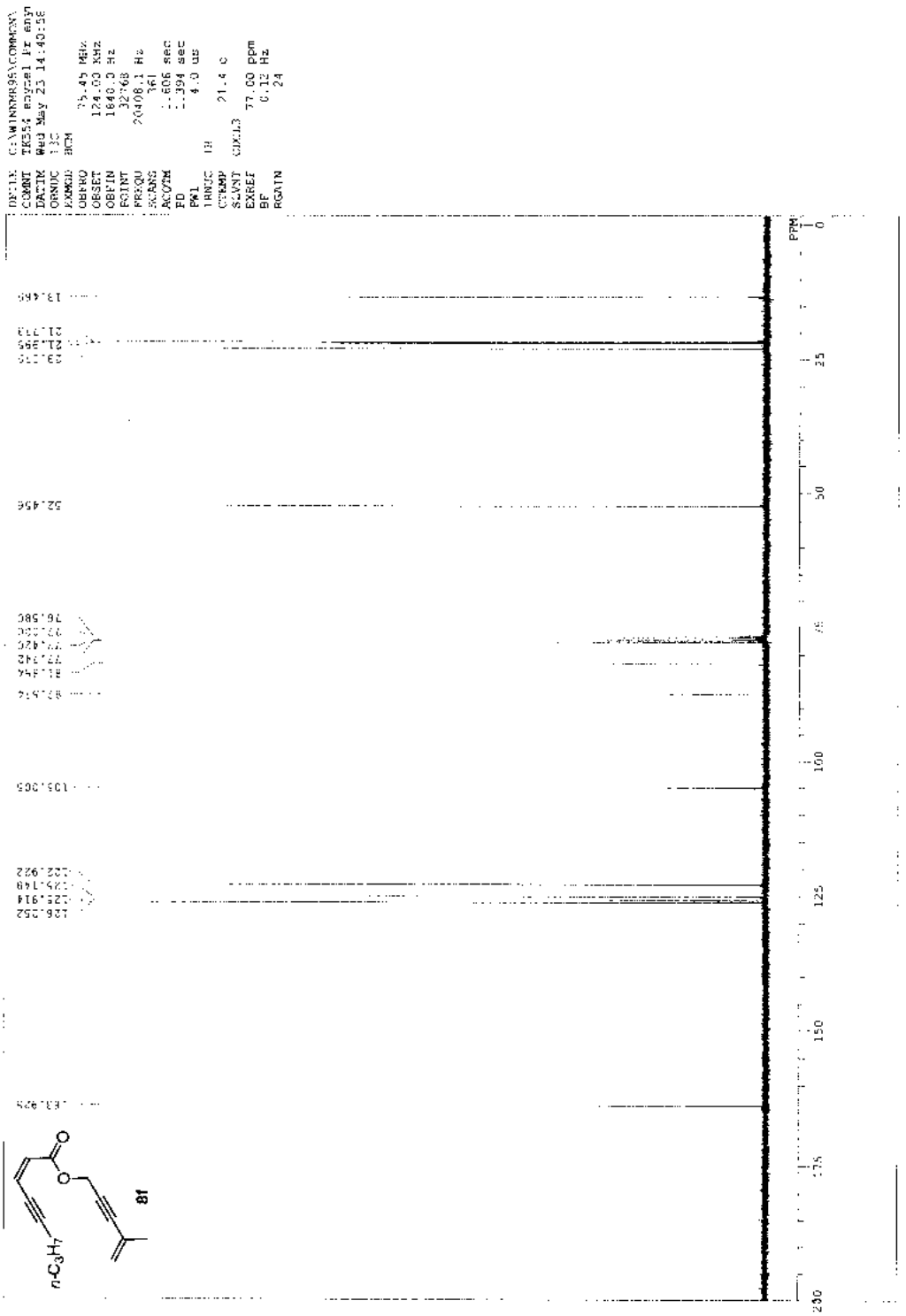

HOCH2
8d

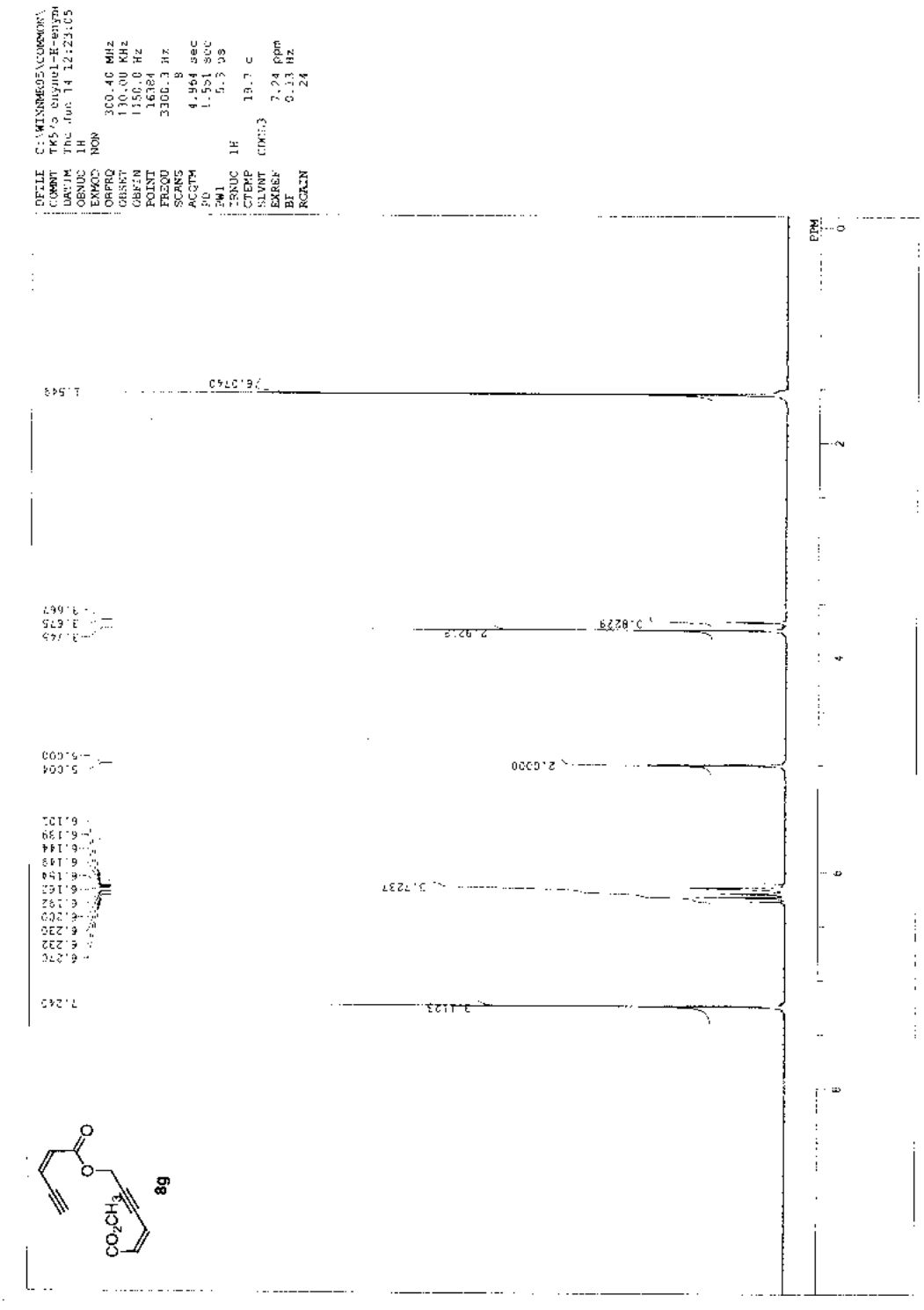

HOCH2
8d
PPM

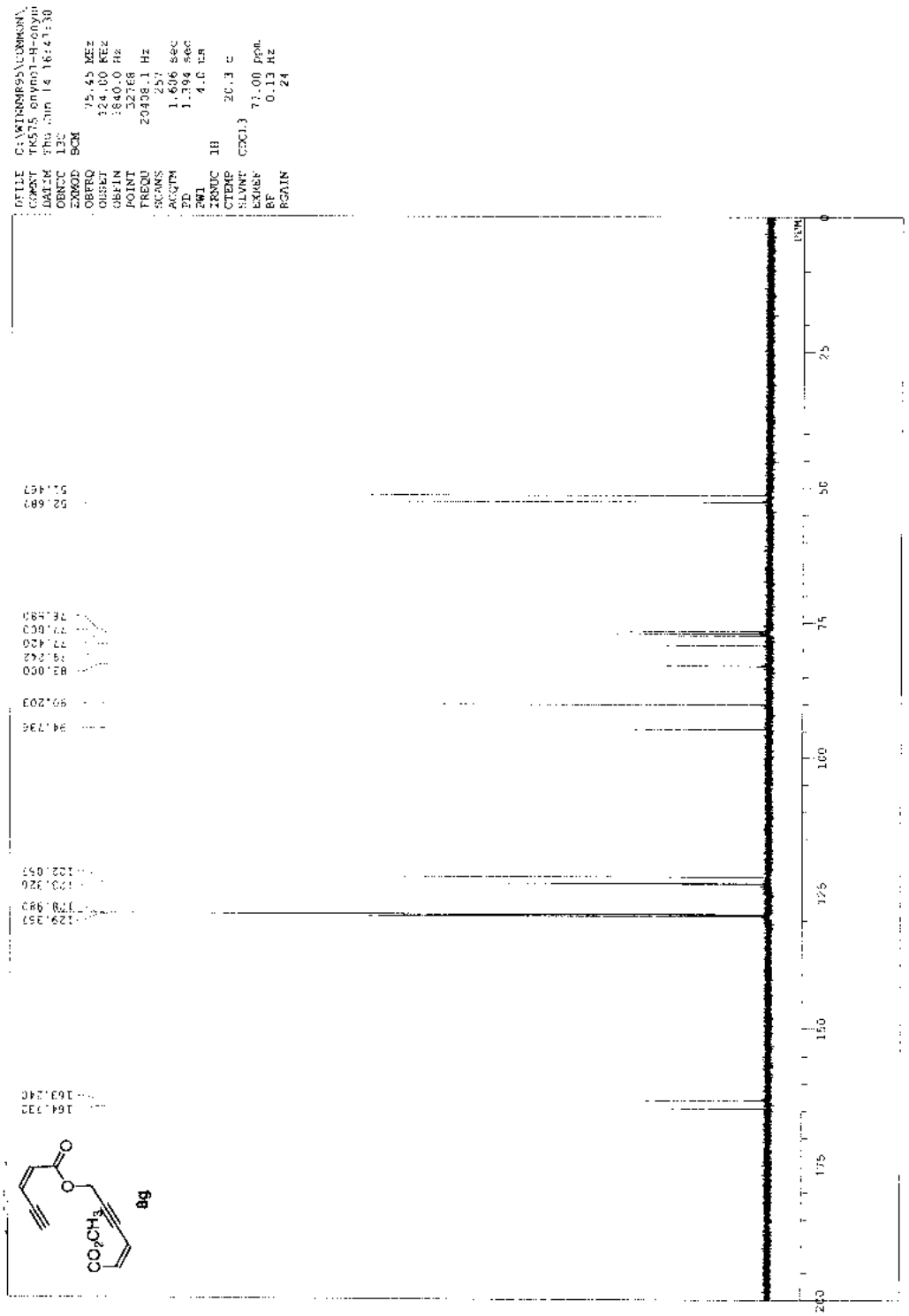

8e

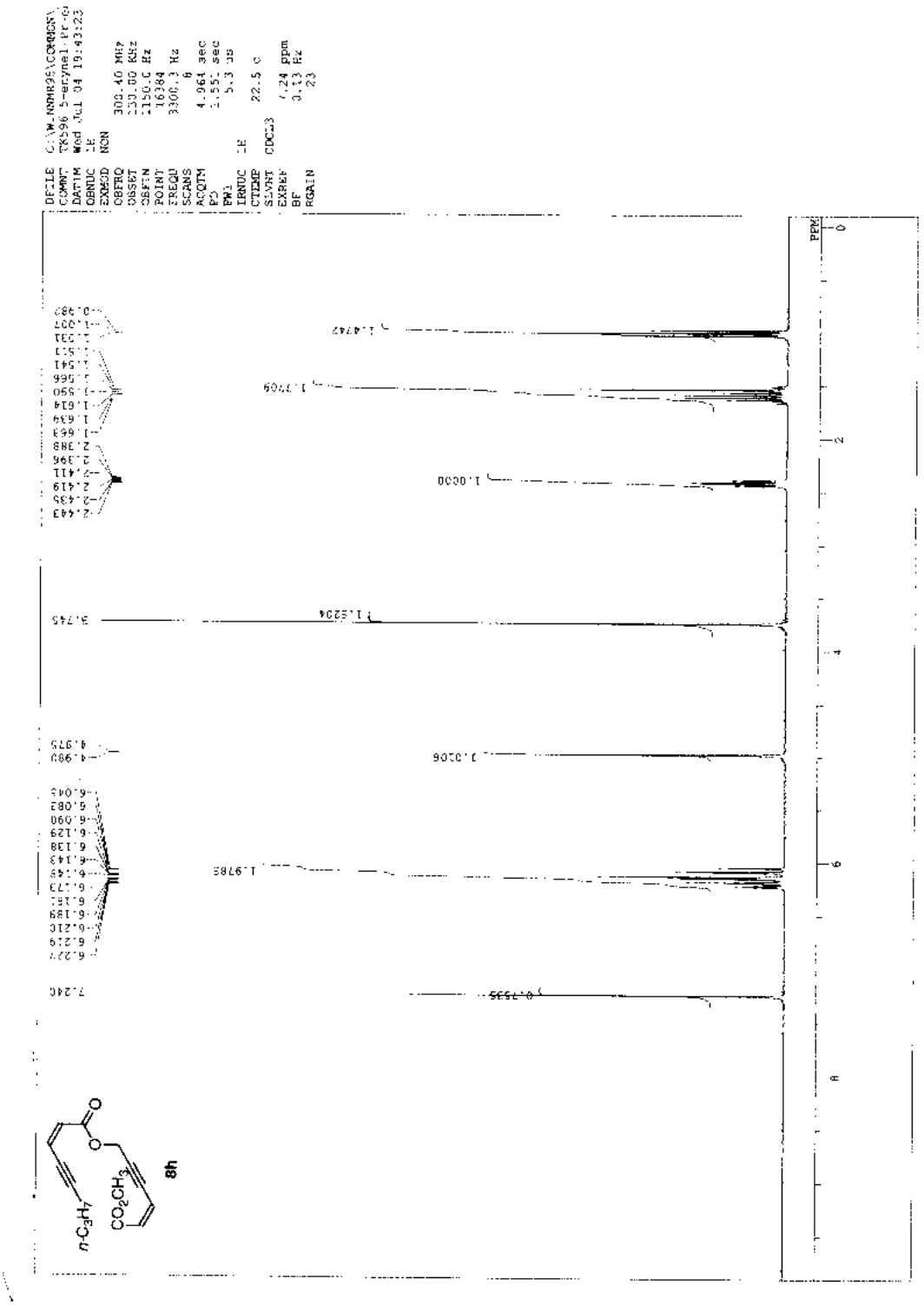

8e
PPM
0
50
100
150
200

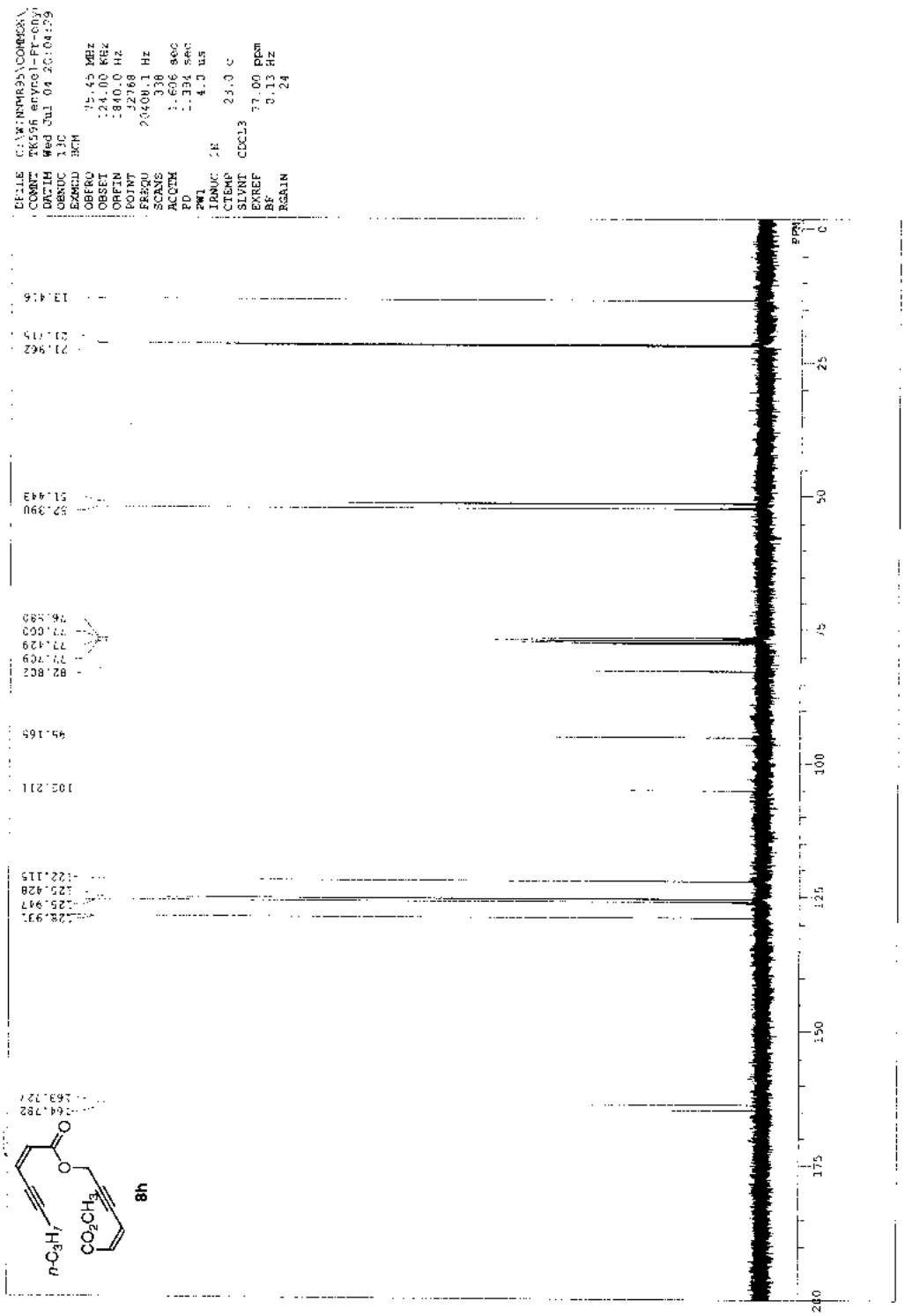

8f
n-C3H7
PPM

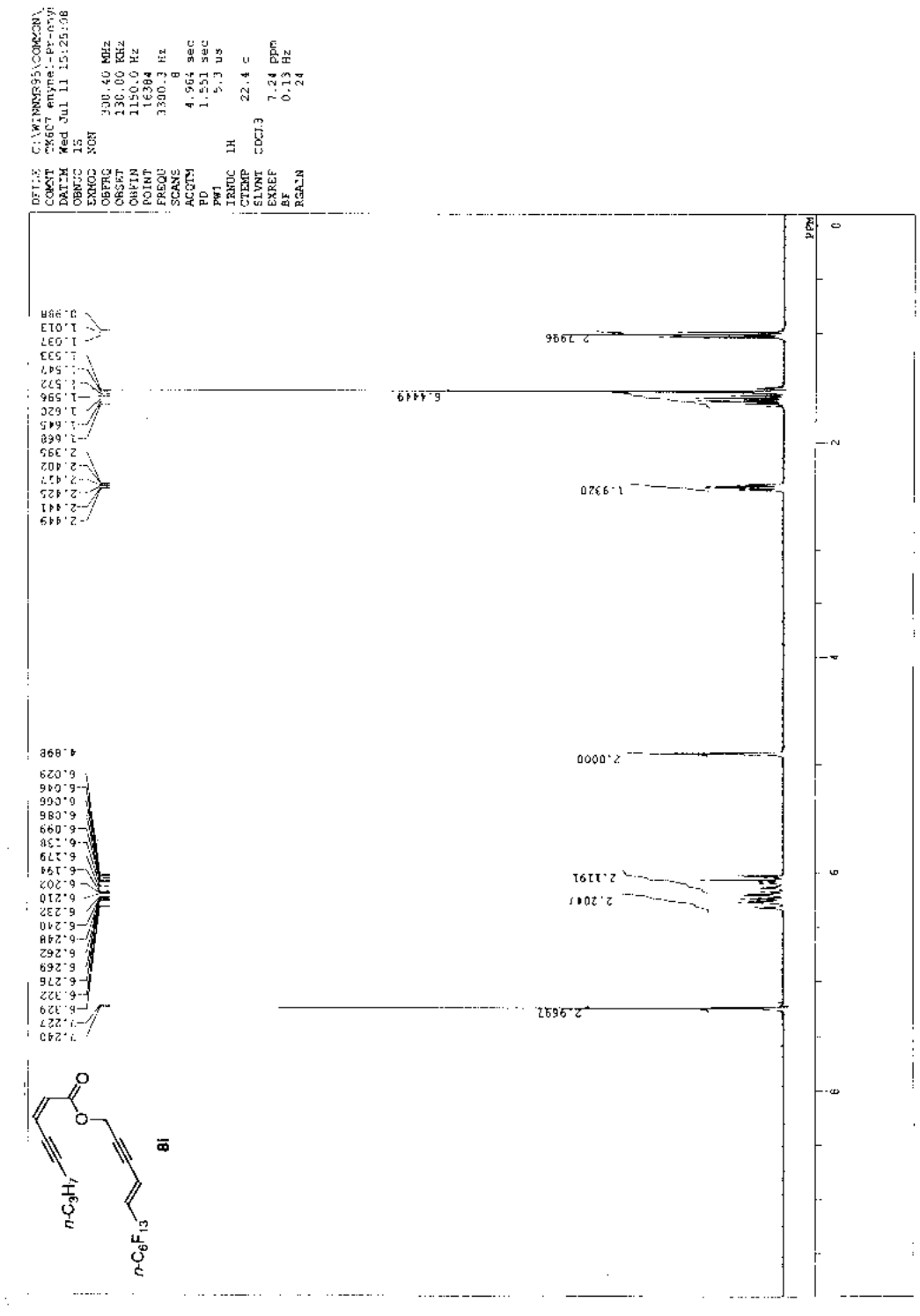

81
n-C3H7
PPM

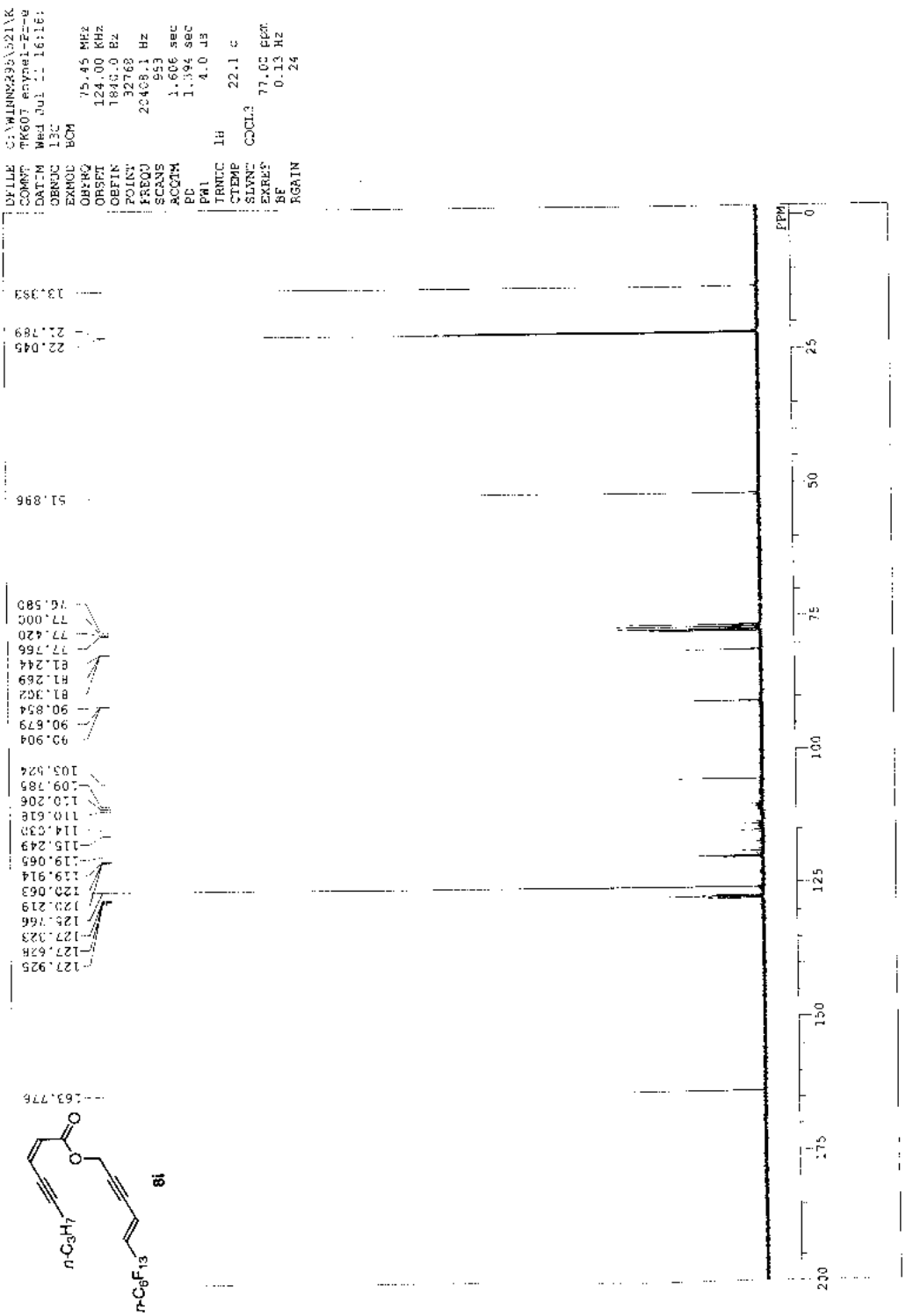

8g
CO2CH3
PPM

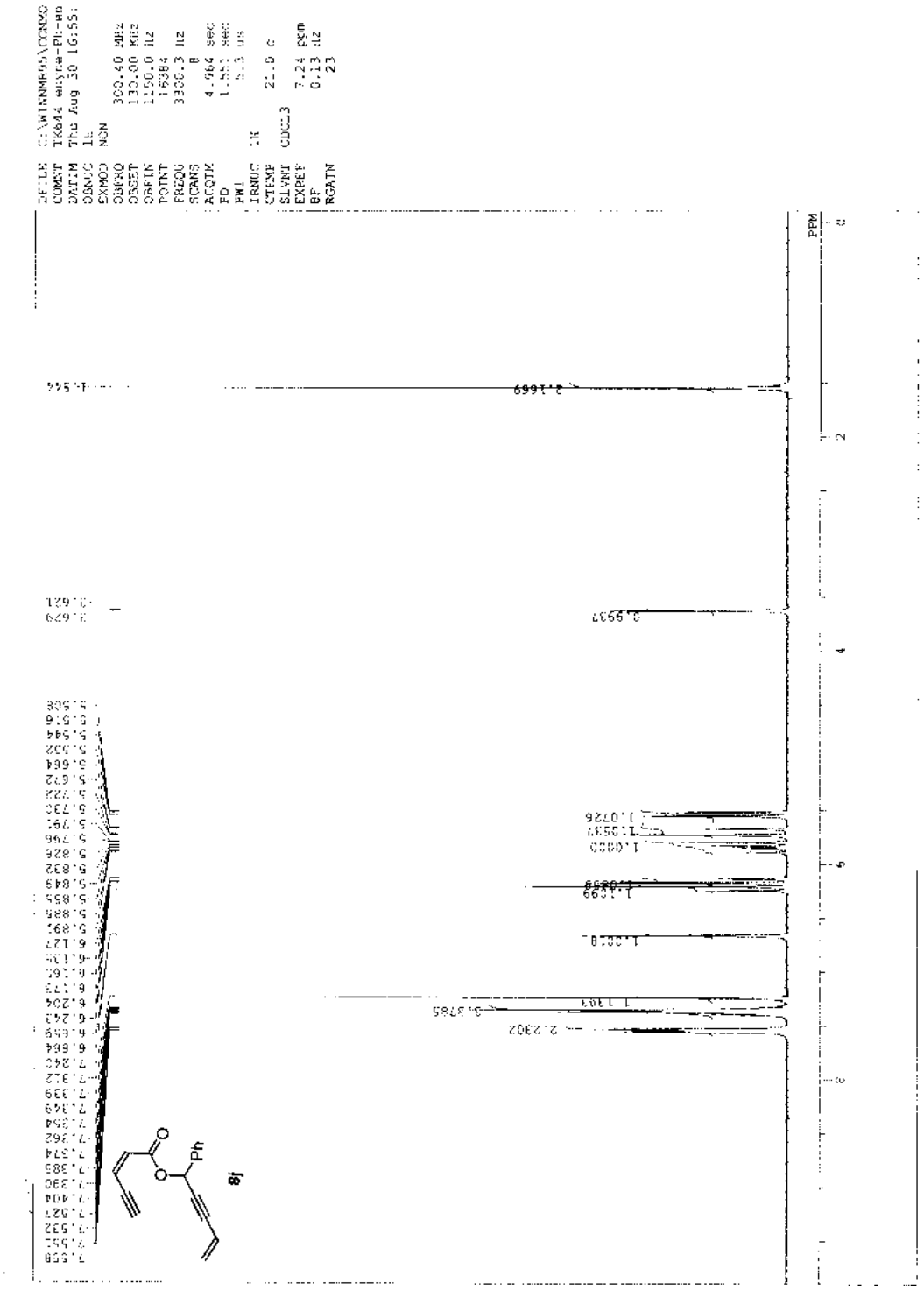

8g
CO2CH3

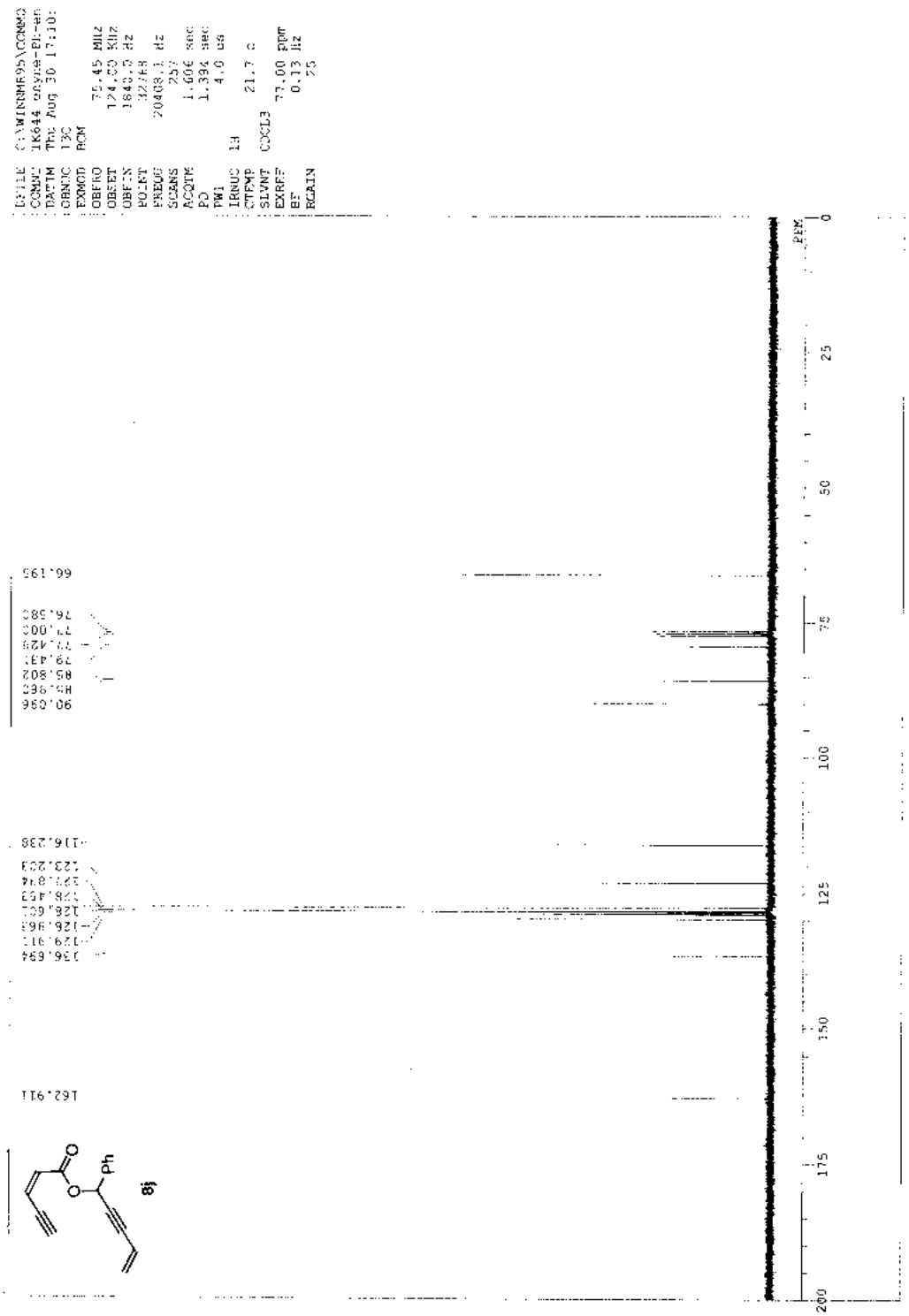

8h
n-C_3H_7
CO_2CH_3
O
O
PPM
0
2
4
6
8

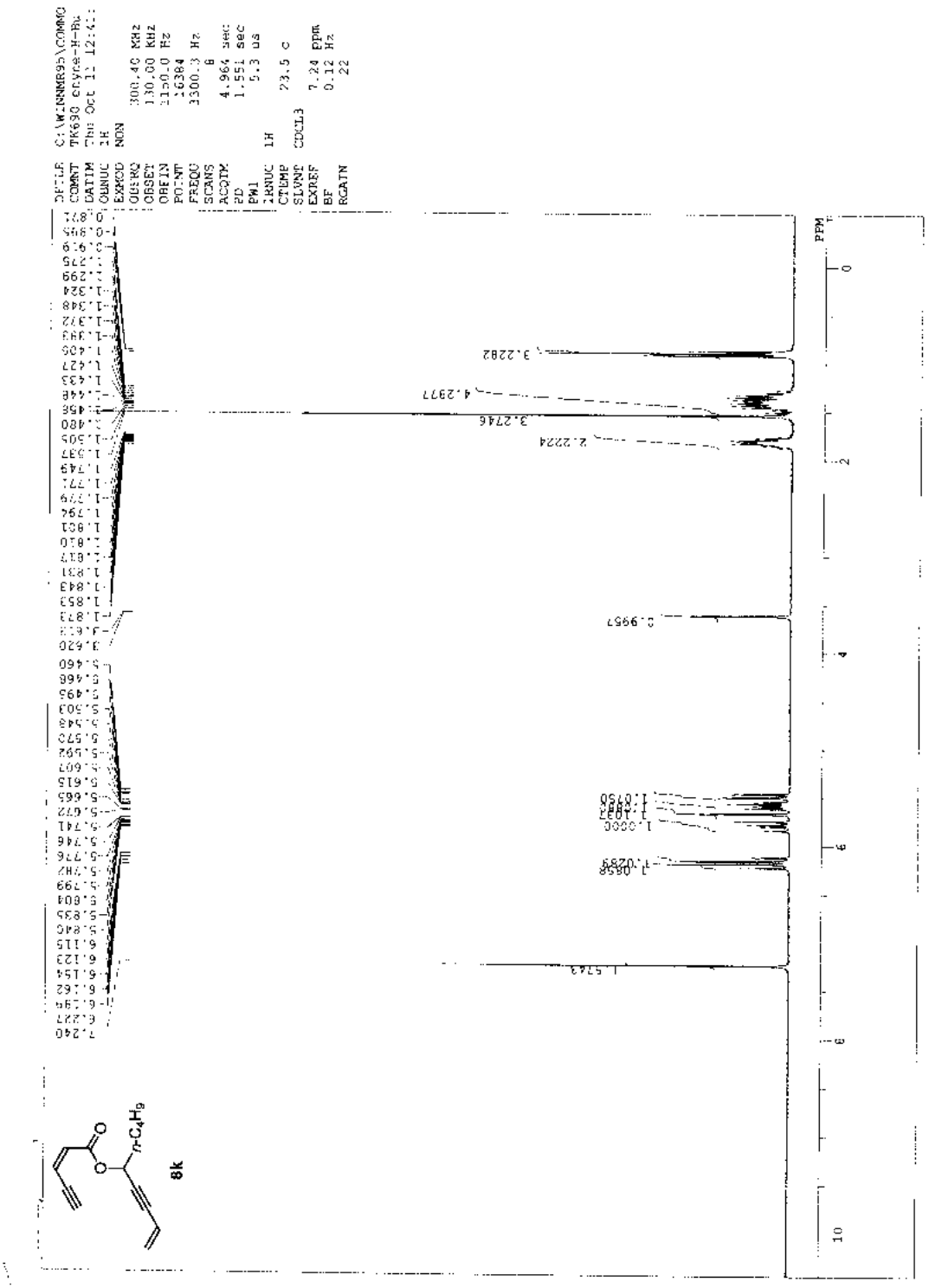

8h

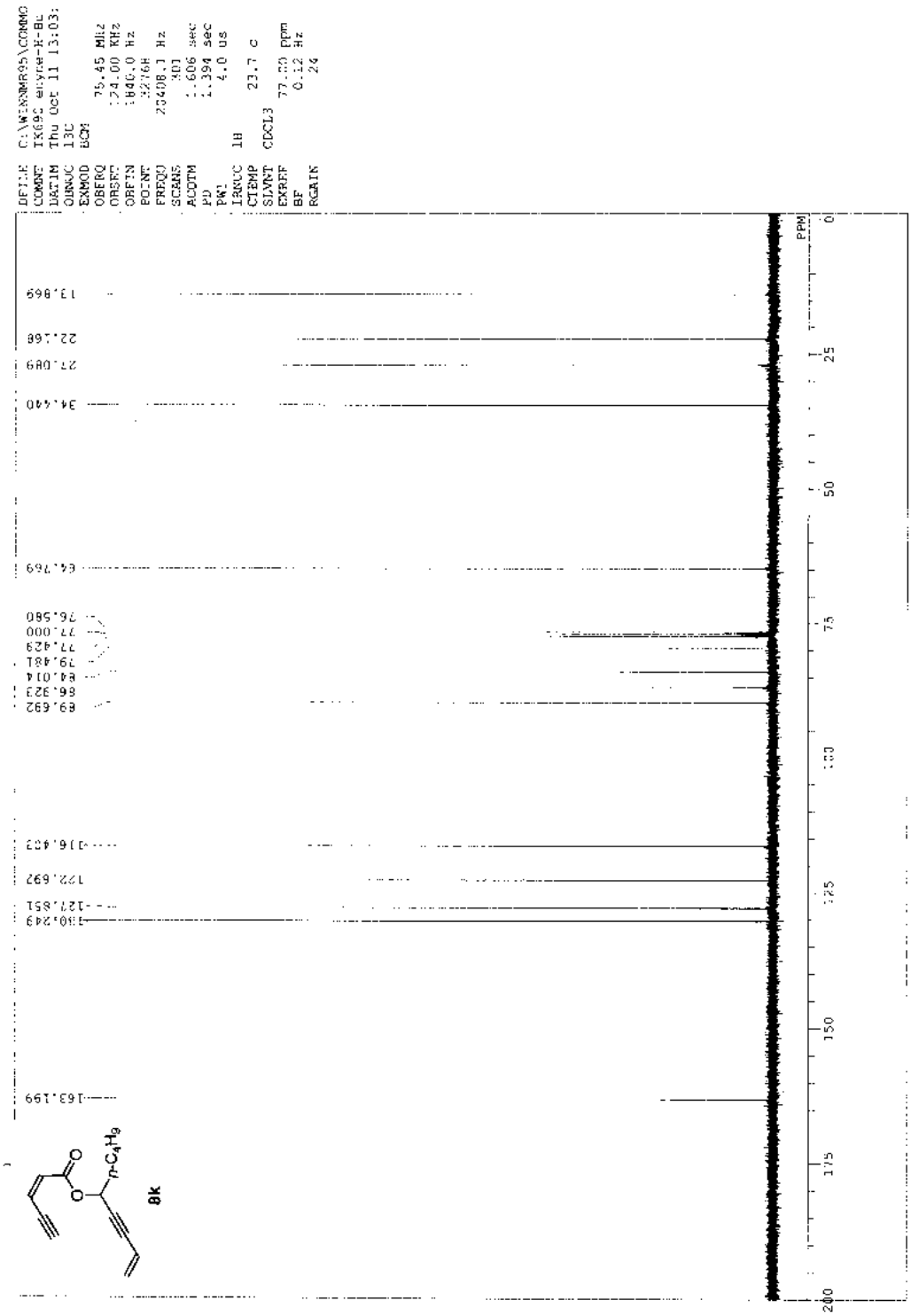

8i
n-C3H7
n-C6F13

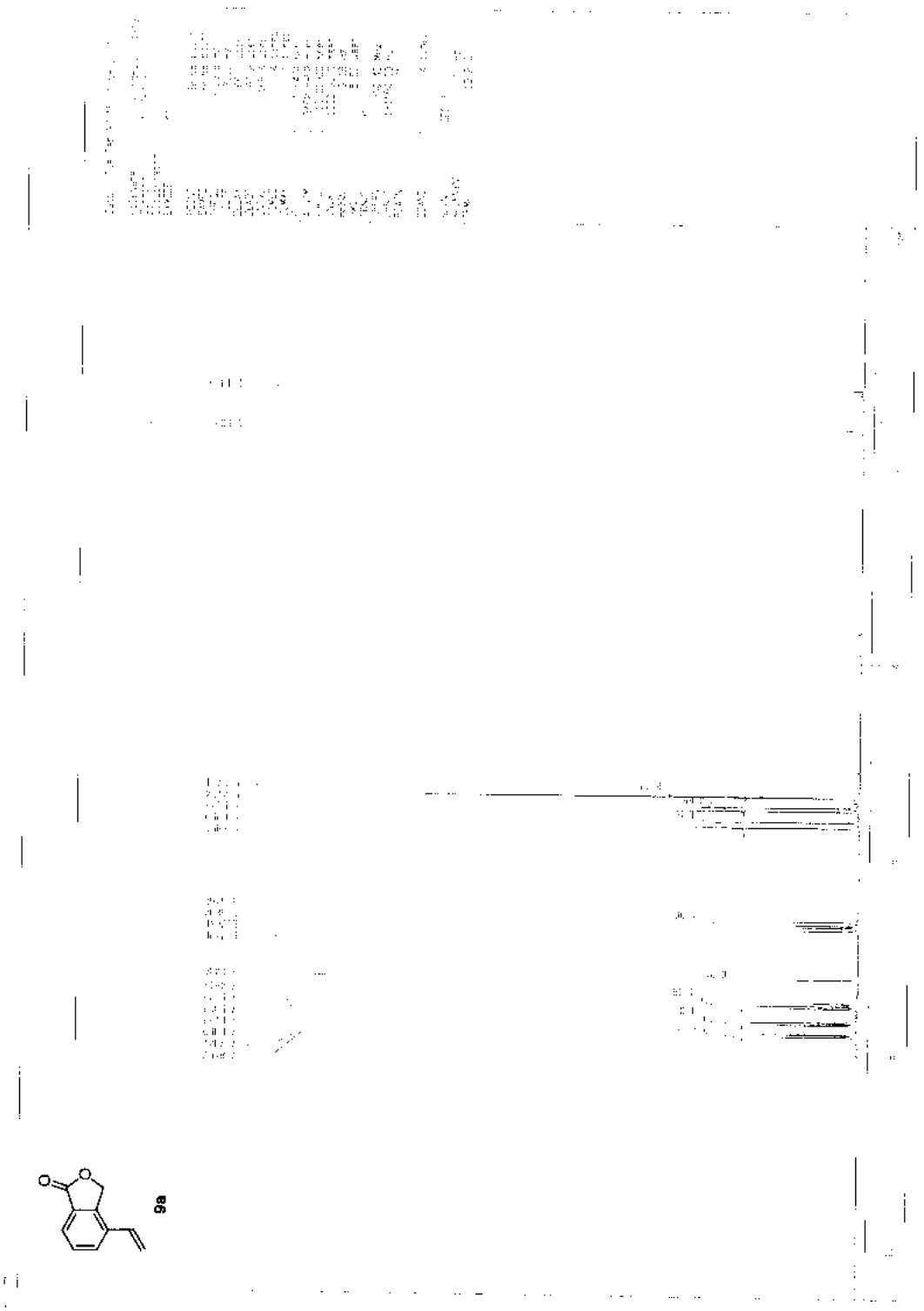

8i
n-C3H7
n-C6F13


8j
Ph
PPM

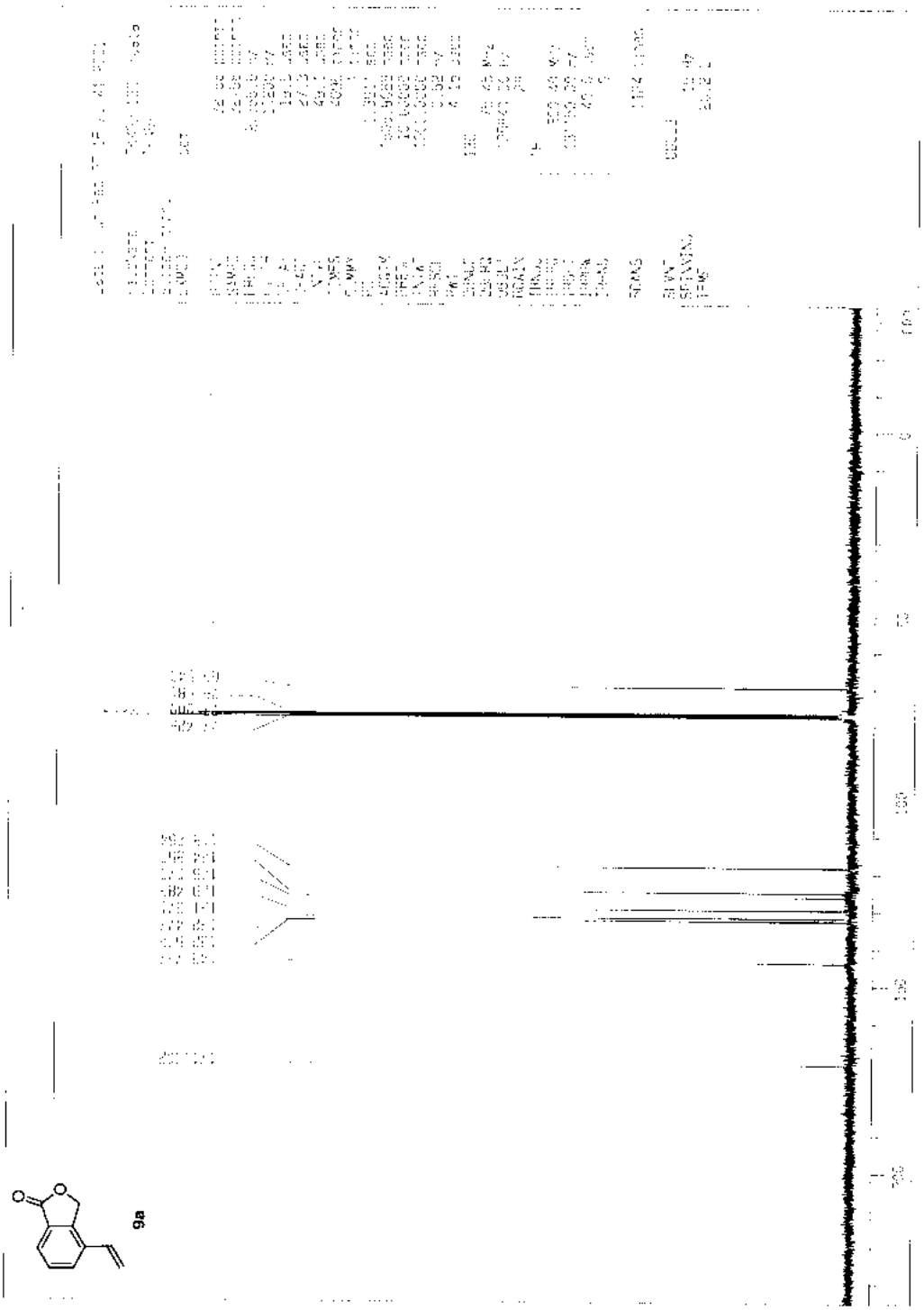

8j
Ph
O
O
162.911
136.694
129.911
128.963
128.601
128.459
127.894
123.259
116.238
90.096
85.962
85.802
79.437
77.428
77.000
76.580
66.195
PPM
0
25
50
75
100
125
150
175
200


8k
n-C4H9
PPM

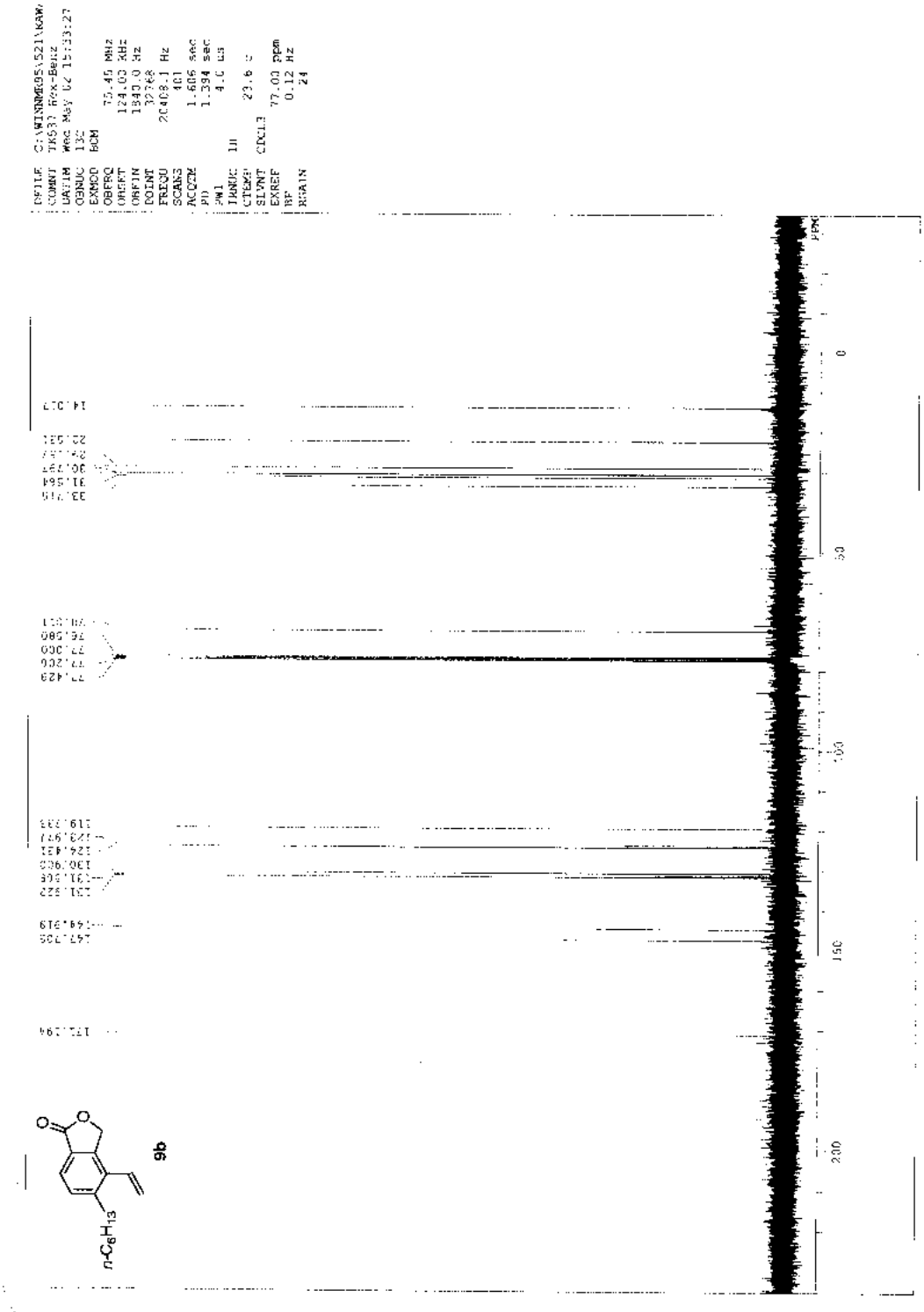

DATIM Thu Oct 11 13:03:
OBSFRQ 75.45 MHz
SWH 24154.6 Hz
FREQU 25408.1 Hz
SCANS 401
ACQTM 1.606 sec
PD 1.394 sec
PW1 4.0 us
TEMP 23.7 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 24
13.869
22.168
27.089
34.440
64.769
76.580
77.000
77.429
79.481
84.014
86.323
89.692
116.423
122.692
127.851
130.249
163.199
PPM
8k

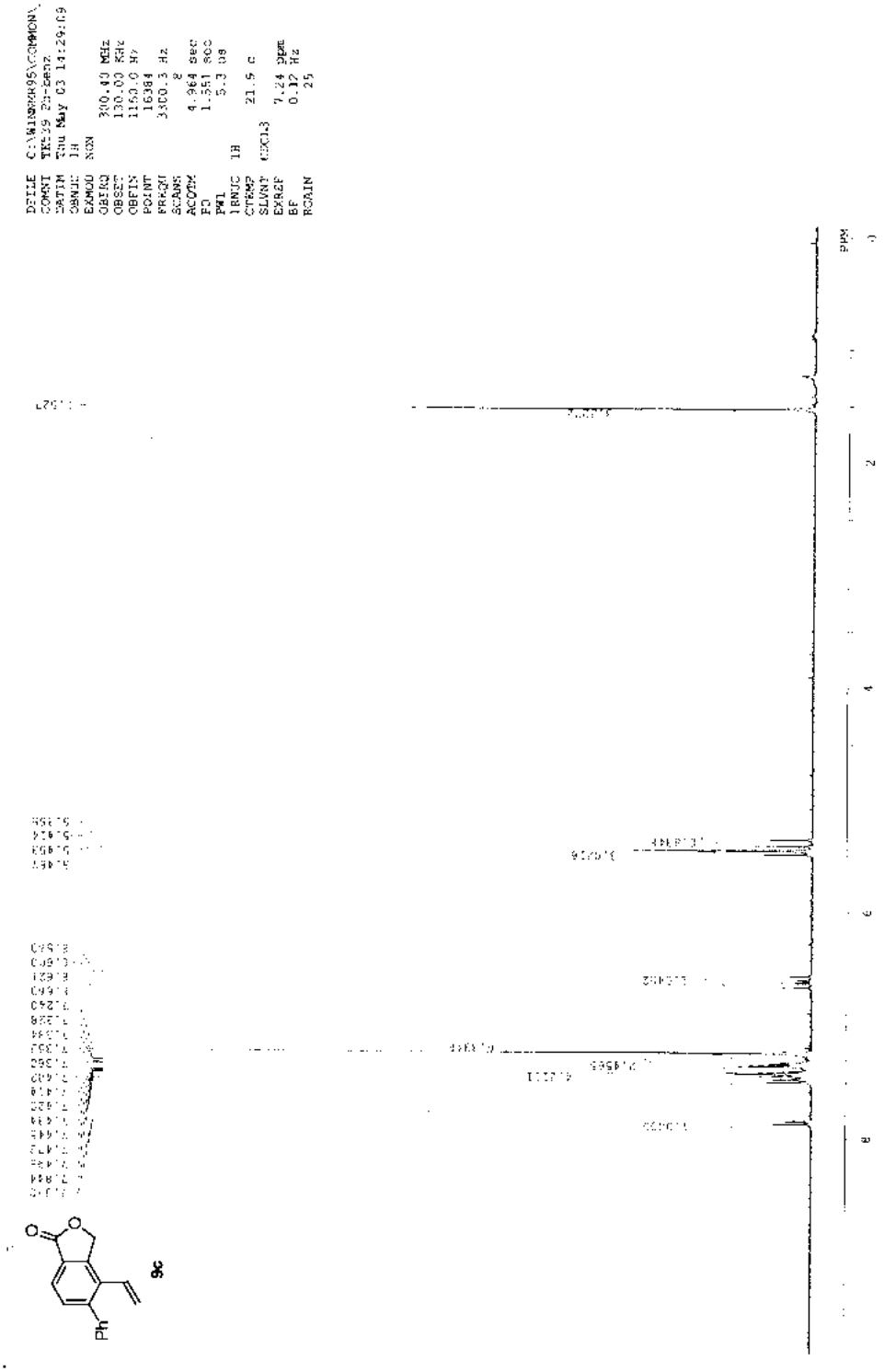


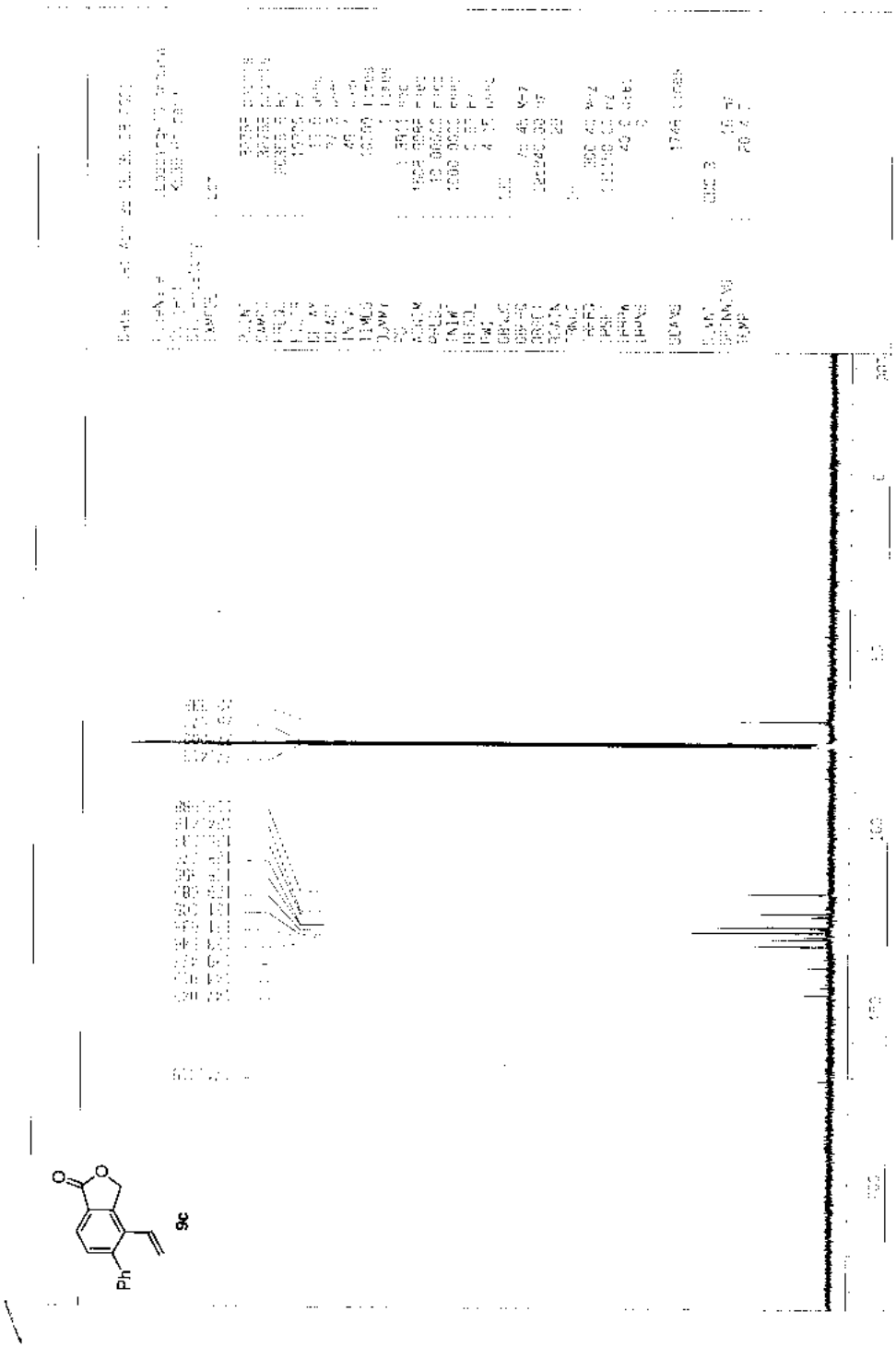
9a

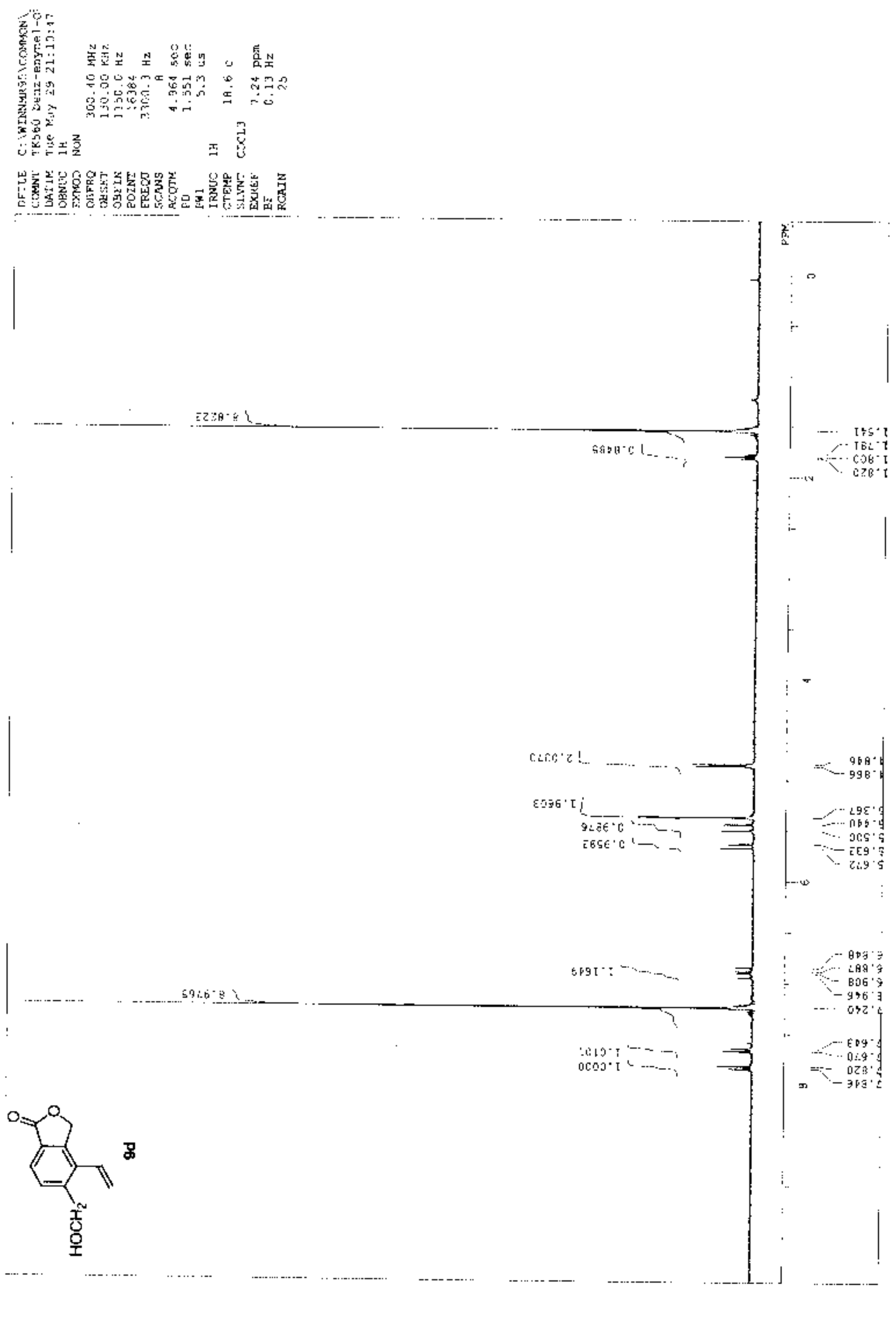


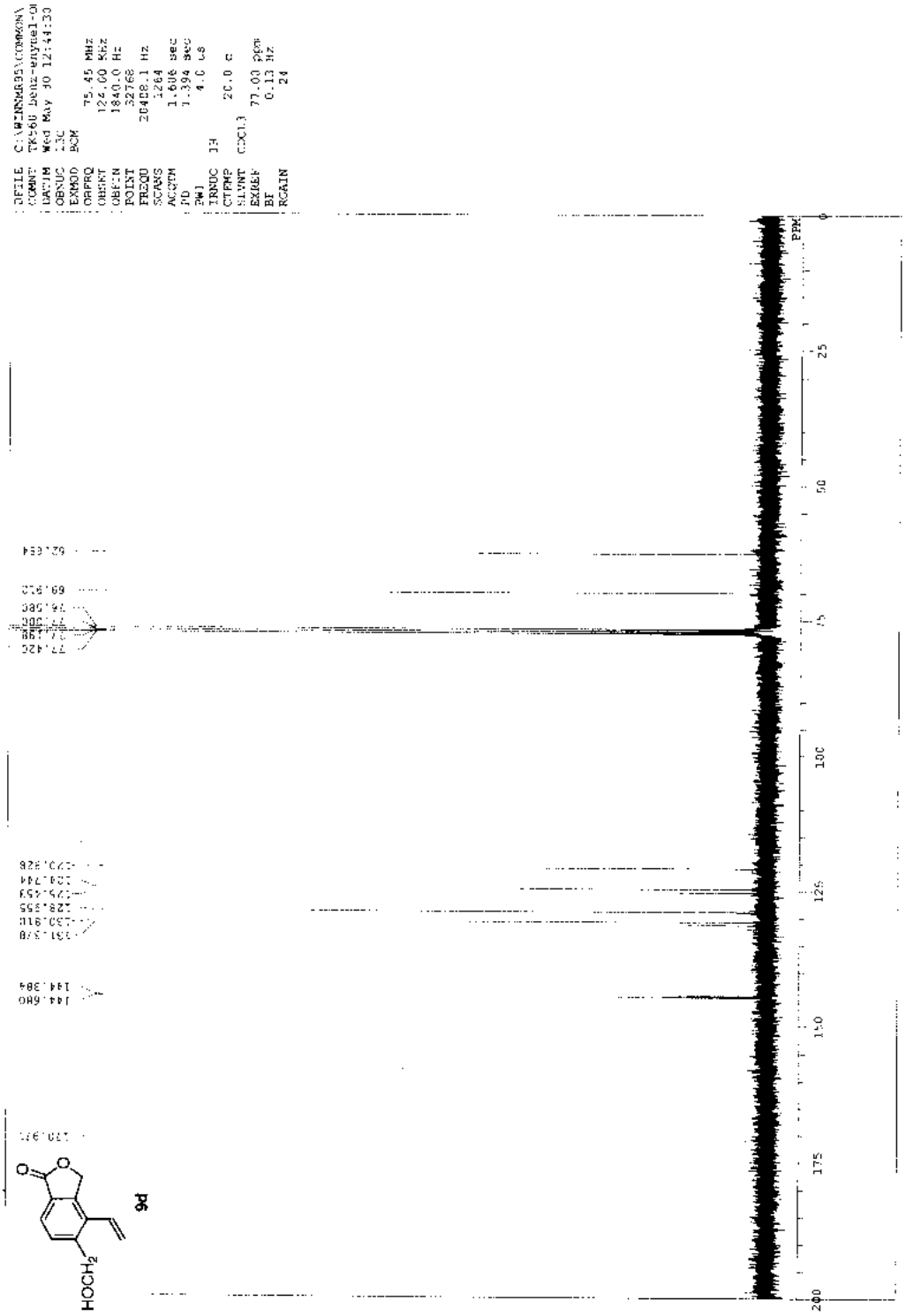

9b
n-C6H13

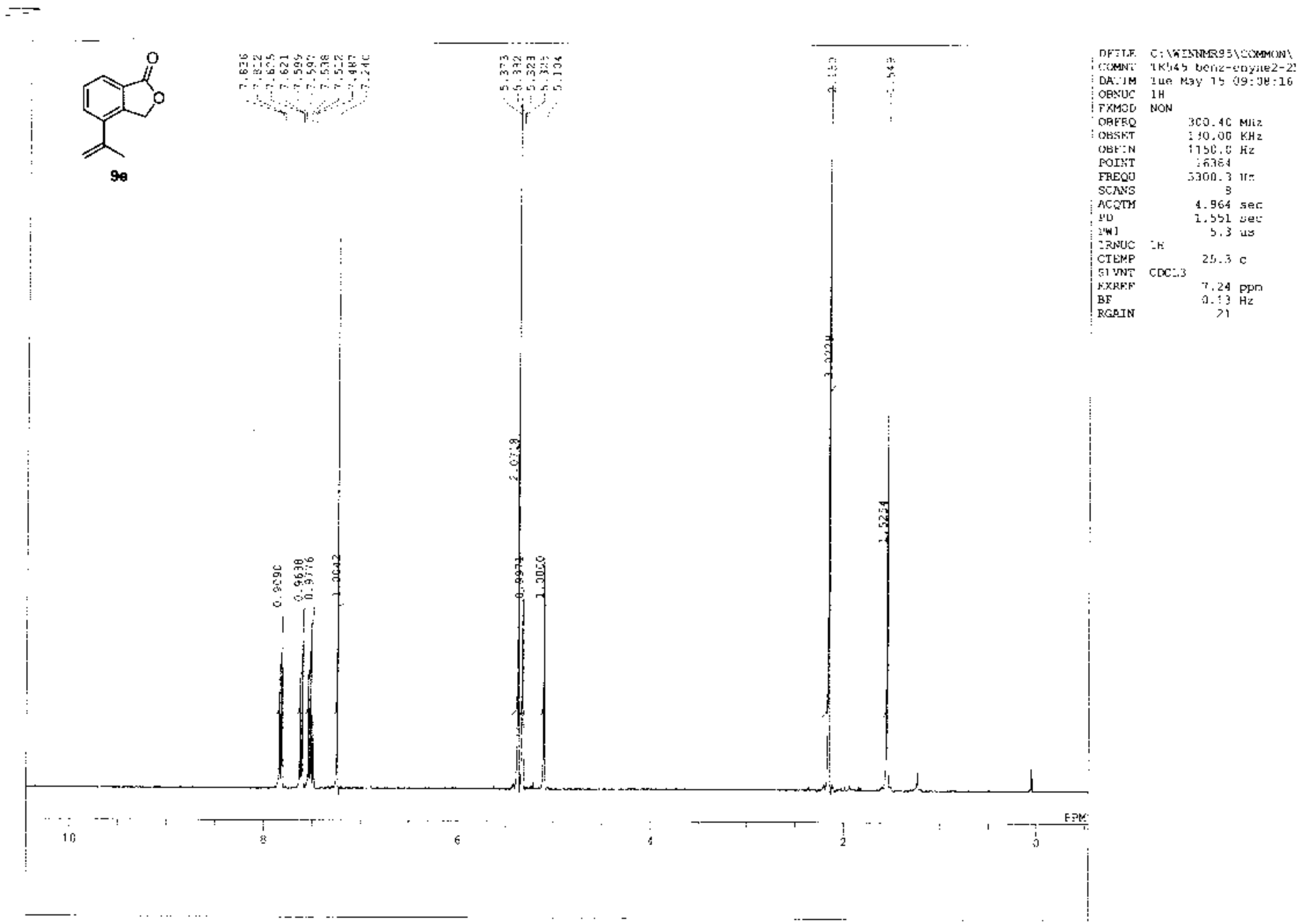

9b
n-C6H13

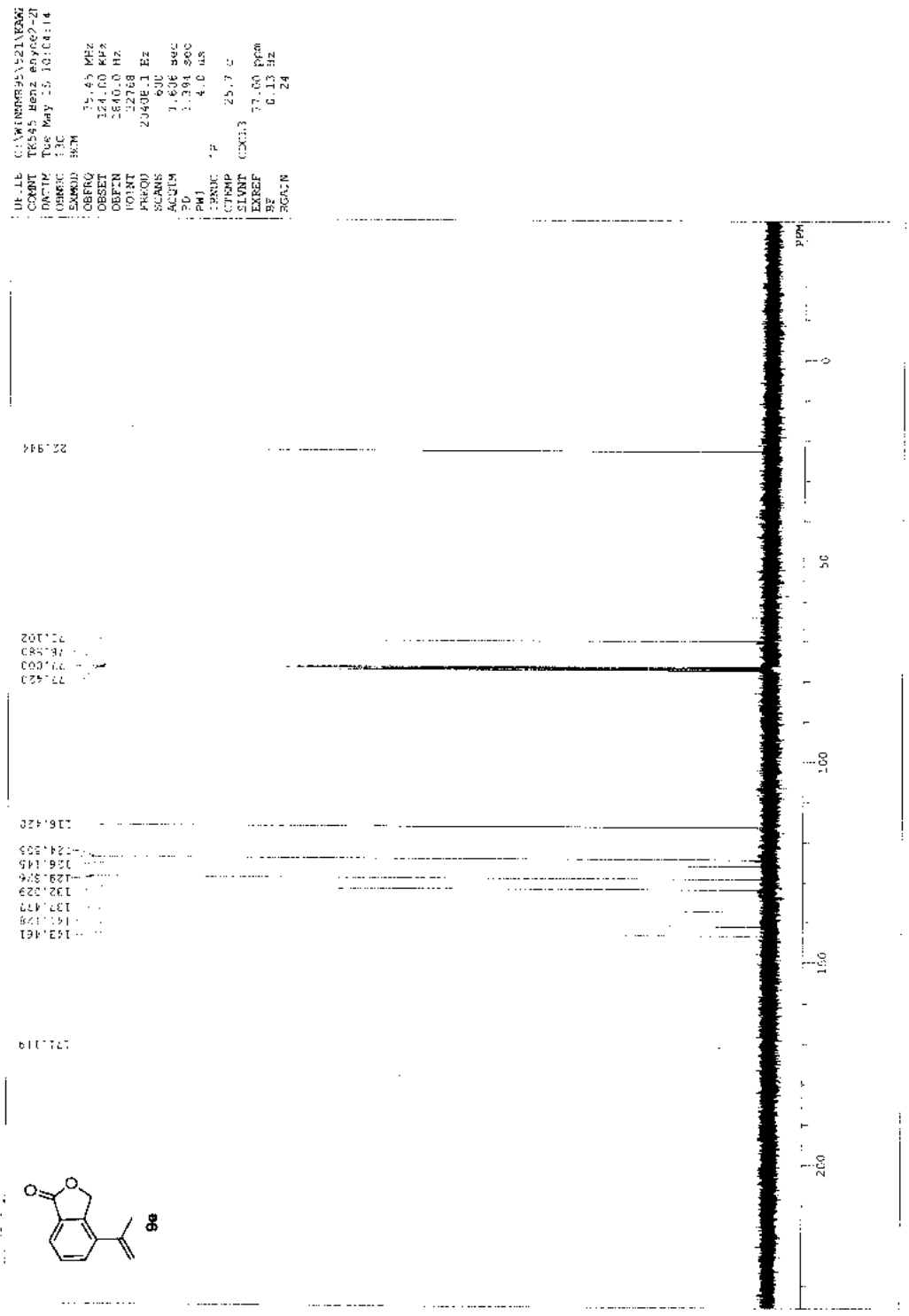

DATIM Thu May 03 14:24:09
OBSERVE 1H
EXMOD NON
OBSFRQ 300.40 MHz
OBSET 130.00 KHz
OBFIN 115.0 Hz
POINT 16384
FRQDM 3300.3 Hz
SCANS 8
ACQTM 4.964 sec
PD 1.551 sec
PW1 5.3 us
IRNUC 1H
CTEMP 21.5 c
SLVNT CDCl3
EXREF 7.24 PPM
BF 0.12 Hz
RGAIN 25
PPM
9c
Ph

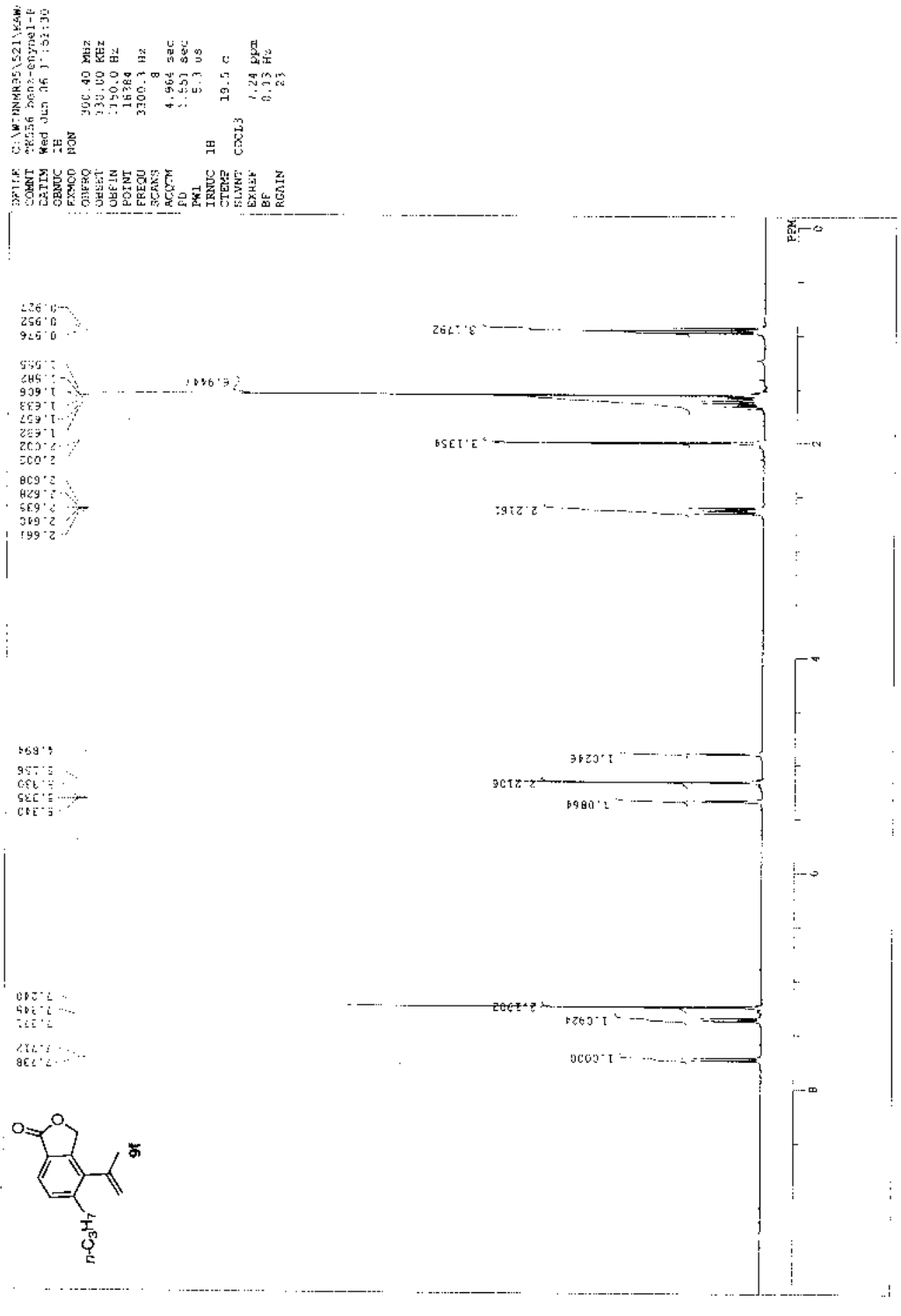

O
O
Ph
9e

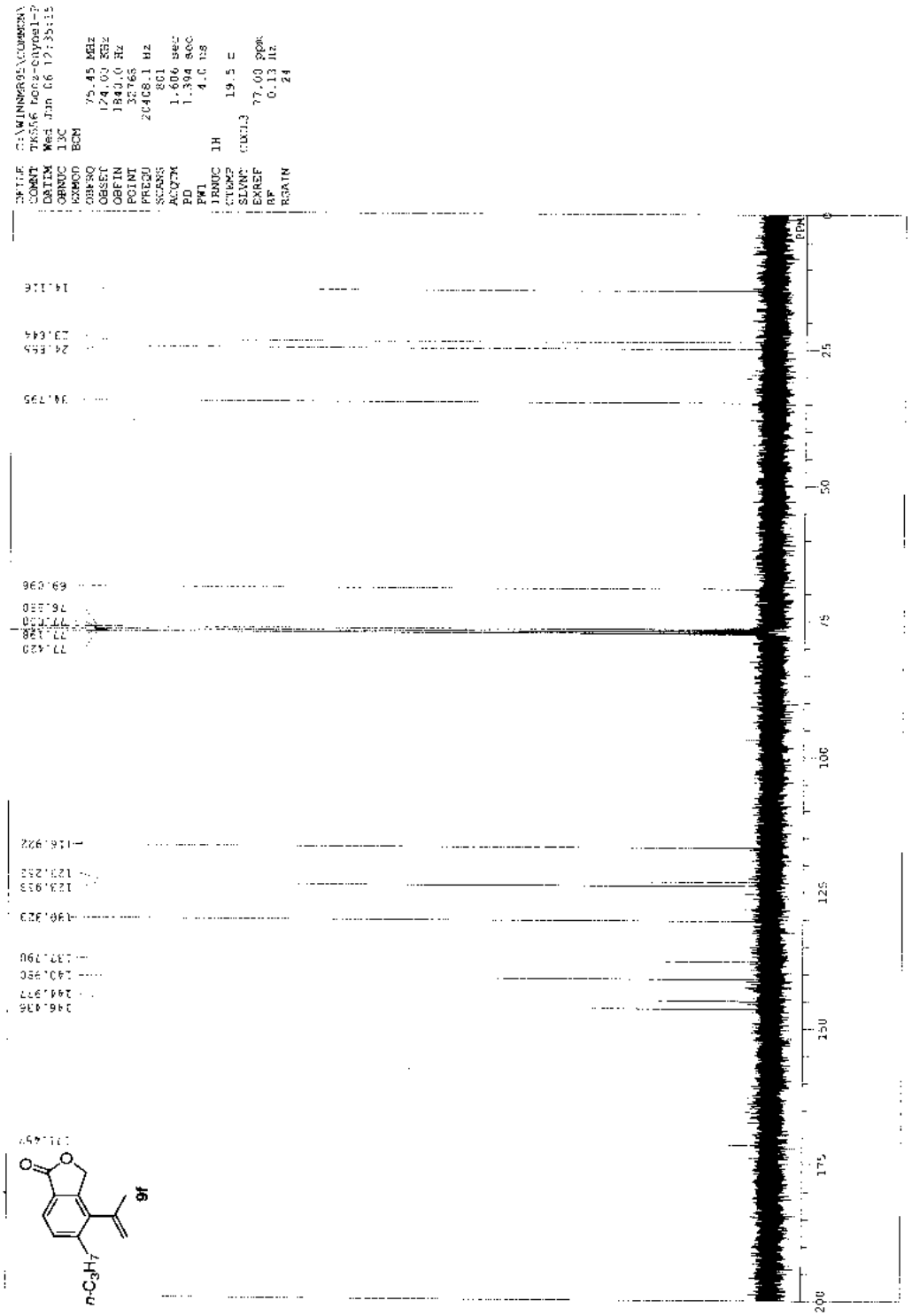

DFILE C:\WINNMR95\COMMON\
DATIM Tue May 29 21:10:17
OBNUC 1H
EXMOD NON
OBFRQ 300.40 MHz
OBSET 130.00 KHz
OBFIN 1350.0 Hz
POINT 16384
FREQU 3300.3 Hz
SCANS 8
ACQTM 4.964 sec
PD 1.551 sec
PW1 5.3 us
IRNUC 1H
CTEMP 18.6 c
SLVNT CDCL3
EXREF 7.24 ppm
BF 0.13 Hz
RGAIN 25
HOCH2
P6
PPM


HOCH2
p6
PPM


9e
7.836
7.812
7.625
7.621
7.599
7.597
7.538
7.512
7.487
7.240
5.373
5.332
5.323
5.325
5.104
2.153
1.549
0.9290
0.9638
0.9776
1.0042
2.0718
0.9971
1.0050
3.0224
1.5254
DFILE C:\WINNMR95\COMMON\
COMNT TK545 benz-enyne2-2
DATIM Tue May 15 09:08:16
OBNUC 1H
EXMOD NON
OBFRQ 300.40 MHz
OBSET 130.00 KHz
OBFIN 1150.0 Hz
POINT 16384
FREQU 5300.3 Hz
SCANS 8
ACQTM 4.964 sec
PD 1.551 sec
PW1 5.3 us
IRNUC 1H
CTEMP 25.3 c
SLVNT CDCL3
EXREF 7.24 ppm
BF 0.13 Hz
RGAIN 21
10
8
6
4
2
0
PPM


22.944
73.420
116.426
173.119
9g


9f
n-C3H7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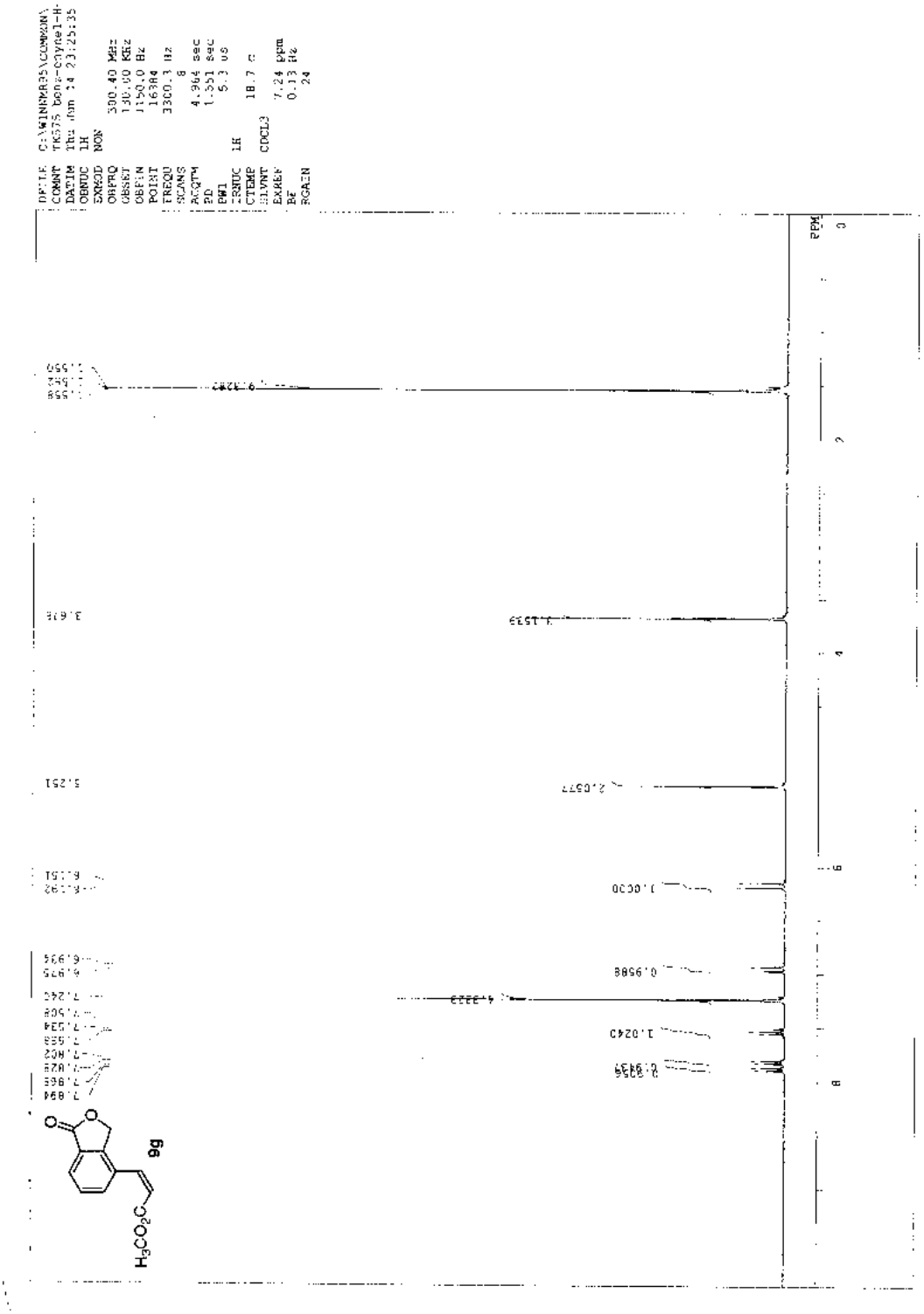
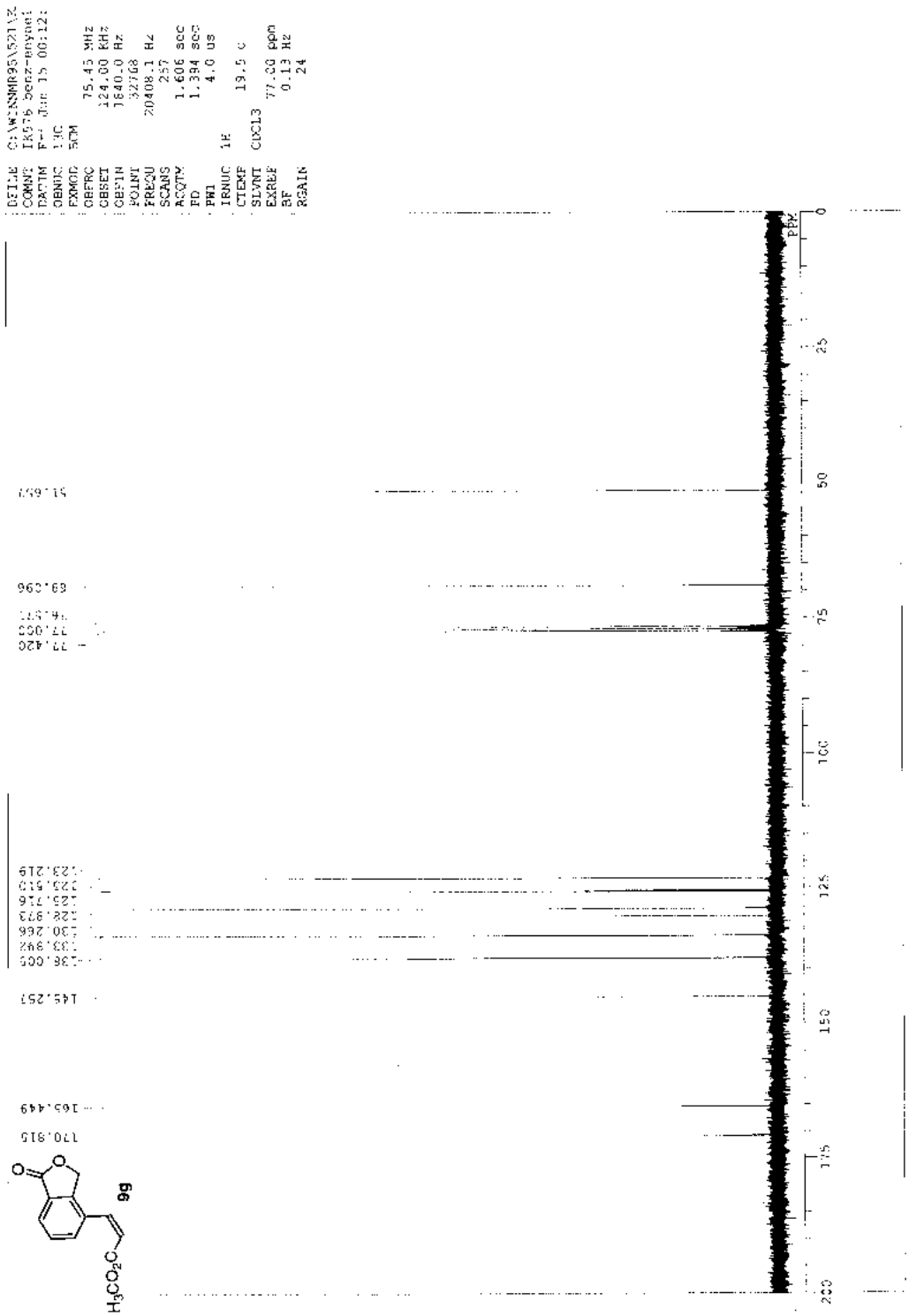
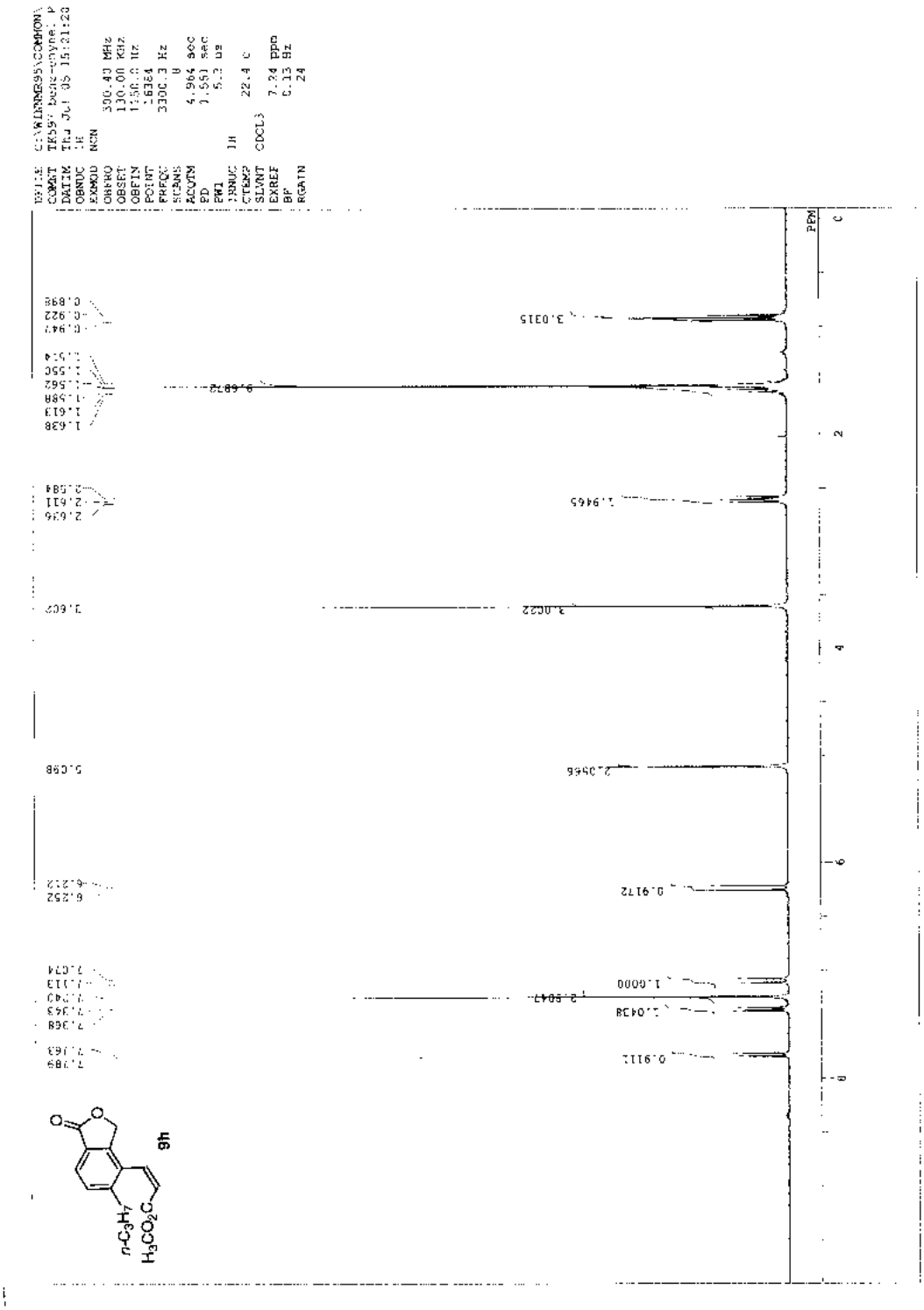
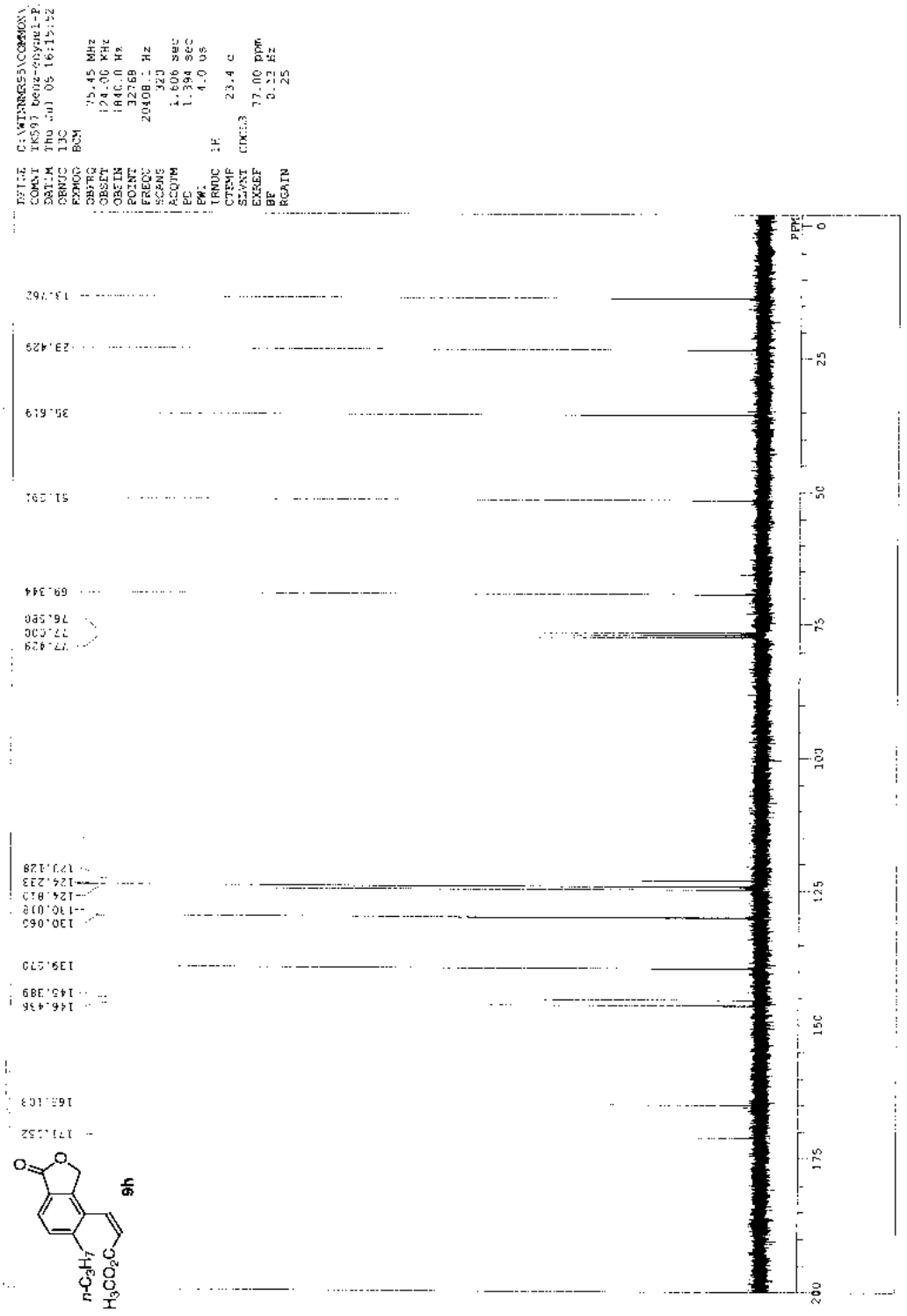
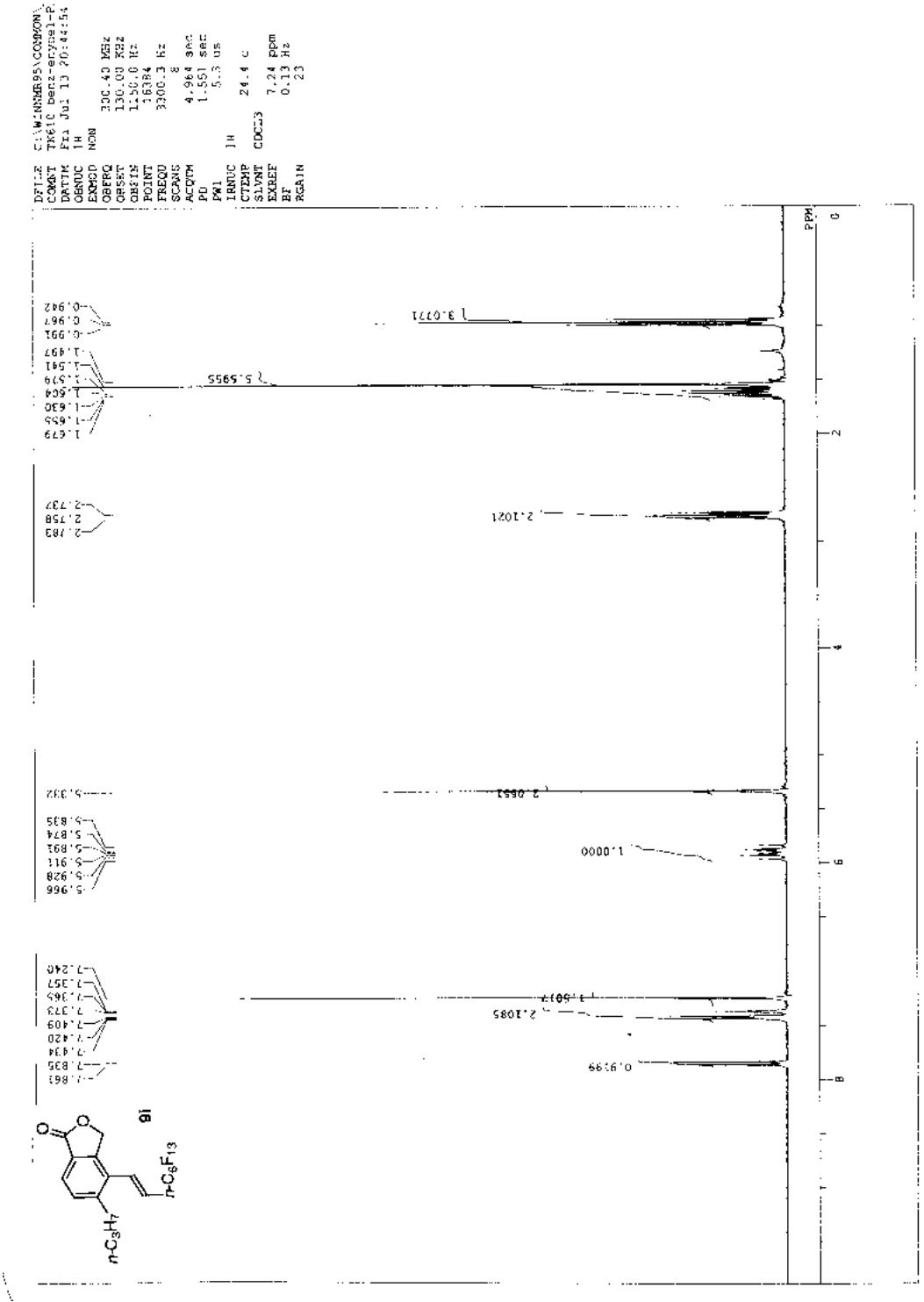
n-C3H7

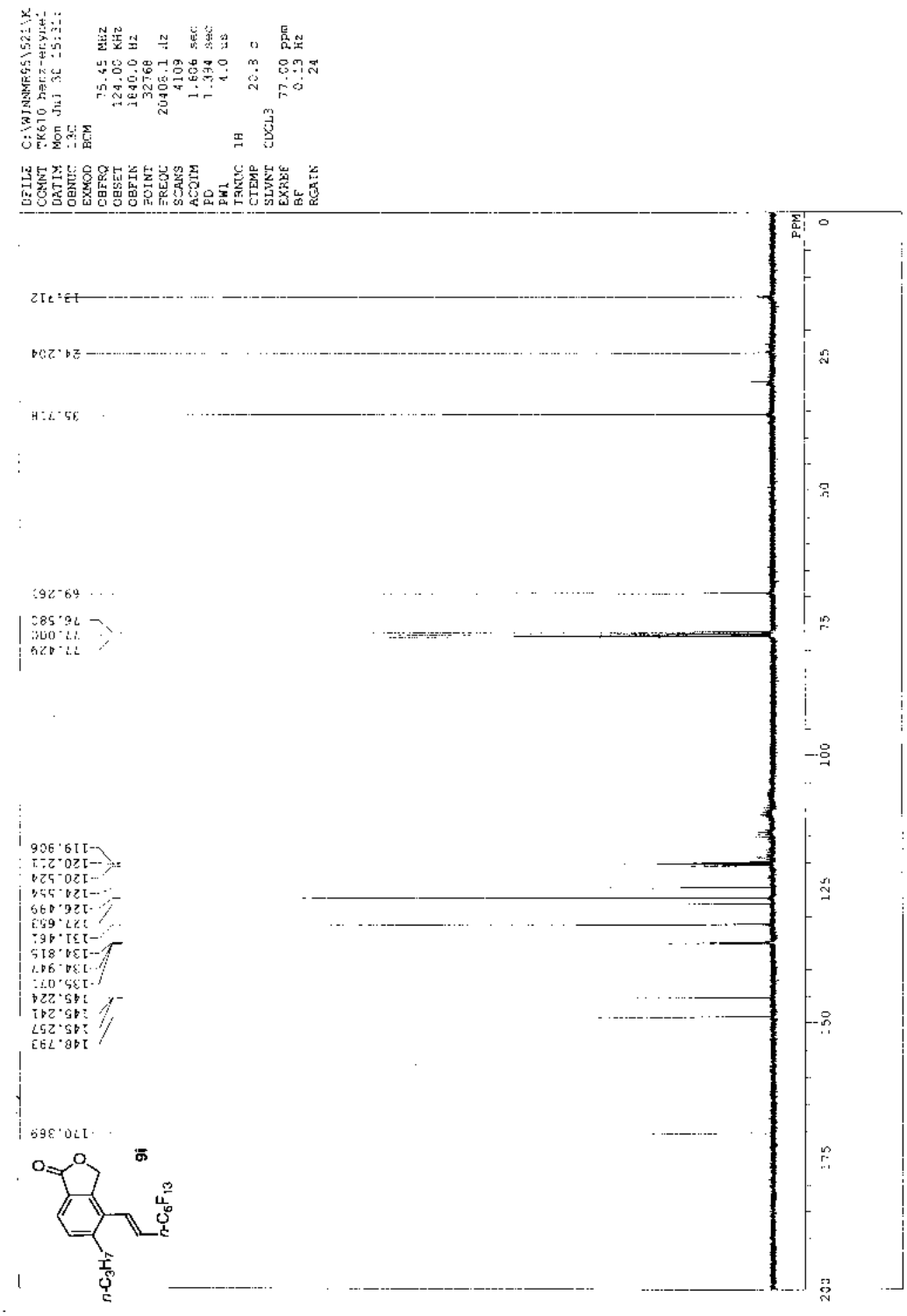

9g
H_3CO_2C
PPM

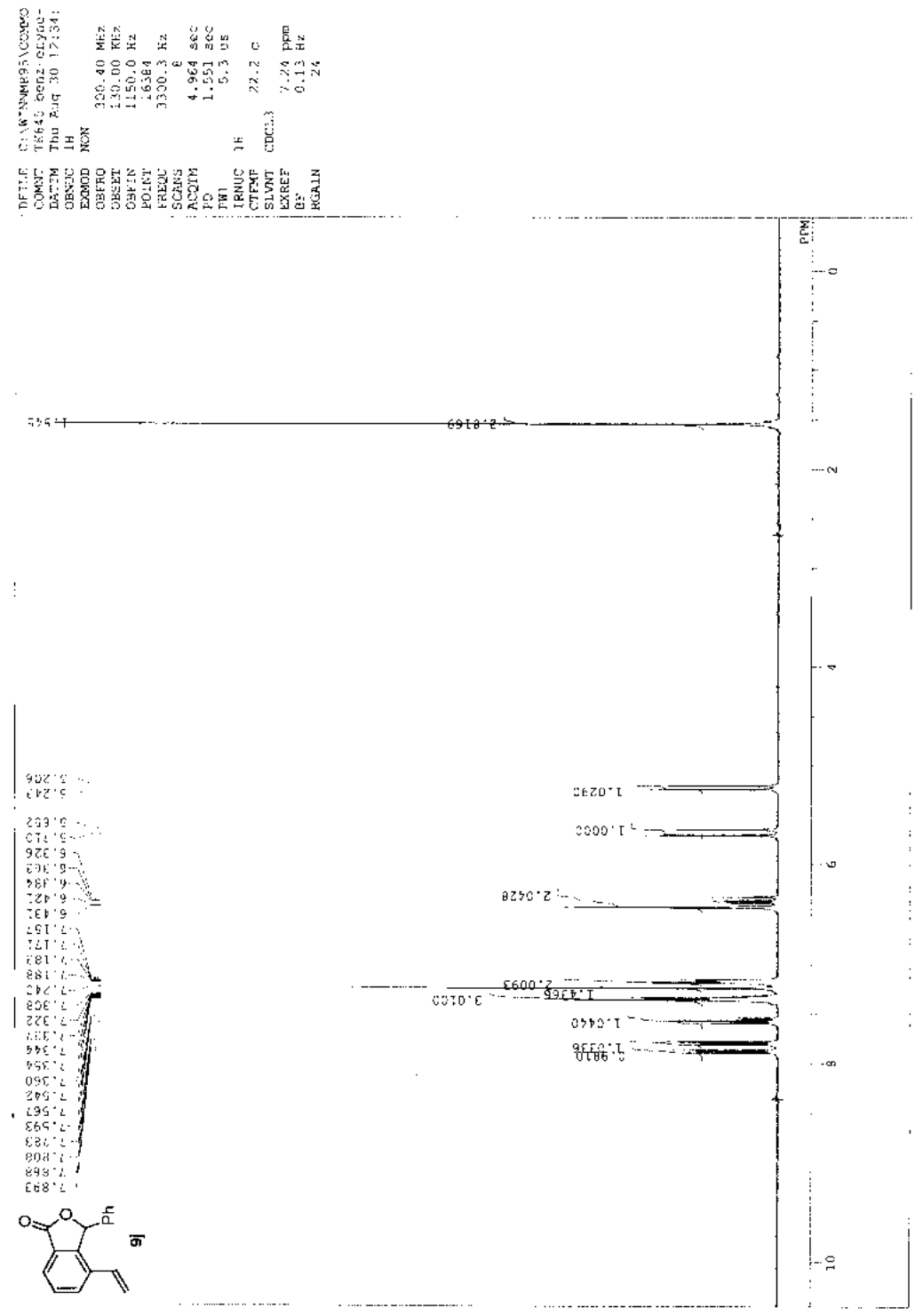

9g
H_3CO_2C
170.815
165.449
145.257
136.005
133.992
130.266
128.973
126.716
125.619
123.219
77.000
73.420
69.096
51.659
PPM


9h
n-C3H7
H3CO2C
PPM


9h
n-C5H7
H3CO2C


DATA C:\W-NMR\95\COMMON\
COMMT TK610 benz-eryzel-P
DATIM Fri Jul 13 20:44:54
OBNUC 1H
EXMOD NOE
OBFRQ 300.40 MHz
OBSET 130.00 KHz
OBFIN 1.56E+0 Hz
POINT 16384
FREQU 3300.3 Hz
SCANS 8
ACQTM 4.964 sec
PD 1.551 sec
PW1 5.3 us
IRNUC 1H
CTEMP 24.4 c
SLVNT CDCl3
EXREF 7.24 ppm
BF 0.13 Hz
RGAIN 23
PPM
n-C3H7
n-C6F13
9i


DATA C:\WINNMR65\DATA\K
COMNT TK610 benz-enyne2
DATIM Mon Jul 30 15:32:1
OBNUC 13C
EXMOD BCM
OBFRQ 75.45 MHz
OBSET 124.05 KHz
OBFIN 1640.0 Hz
POINT 32768
FREQU 20408.1 Hz
SCANS 4109
ACQTM 1.606 sec
PD 1.394 sec
PW1 4.0 us
TNUC 1H
CTEMP 20.3 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.13 Hz
RGAIN 24
PPM
0
25
50
75
100
125
150
175
200
13.912
24.204
35.718
69.267
76.582
77.000
77.429
119.906
120.211
120.524
124.554
126.499
127.653
131.461
134.815
134.947
135.071
145.224
145.241
145.257
148.793
170.389
9i
n-C6F13
n-C3H7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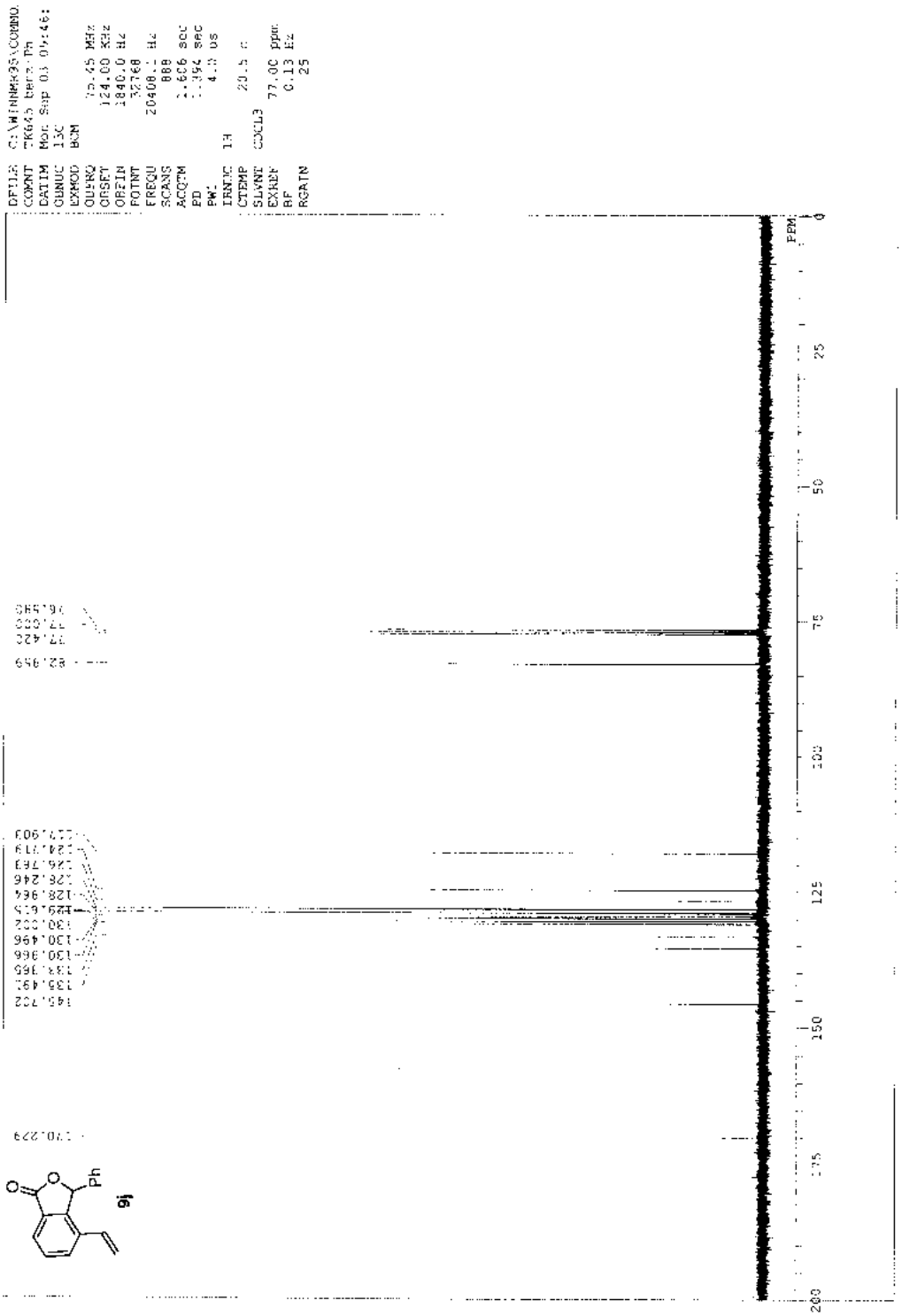
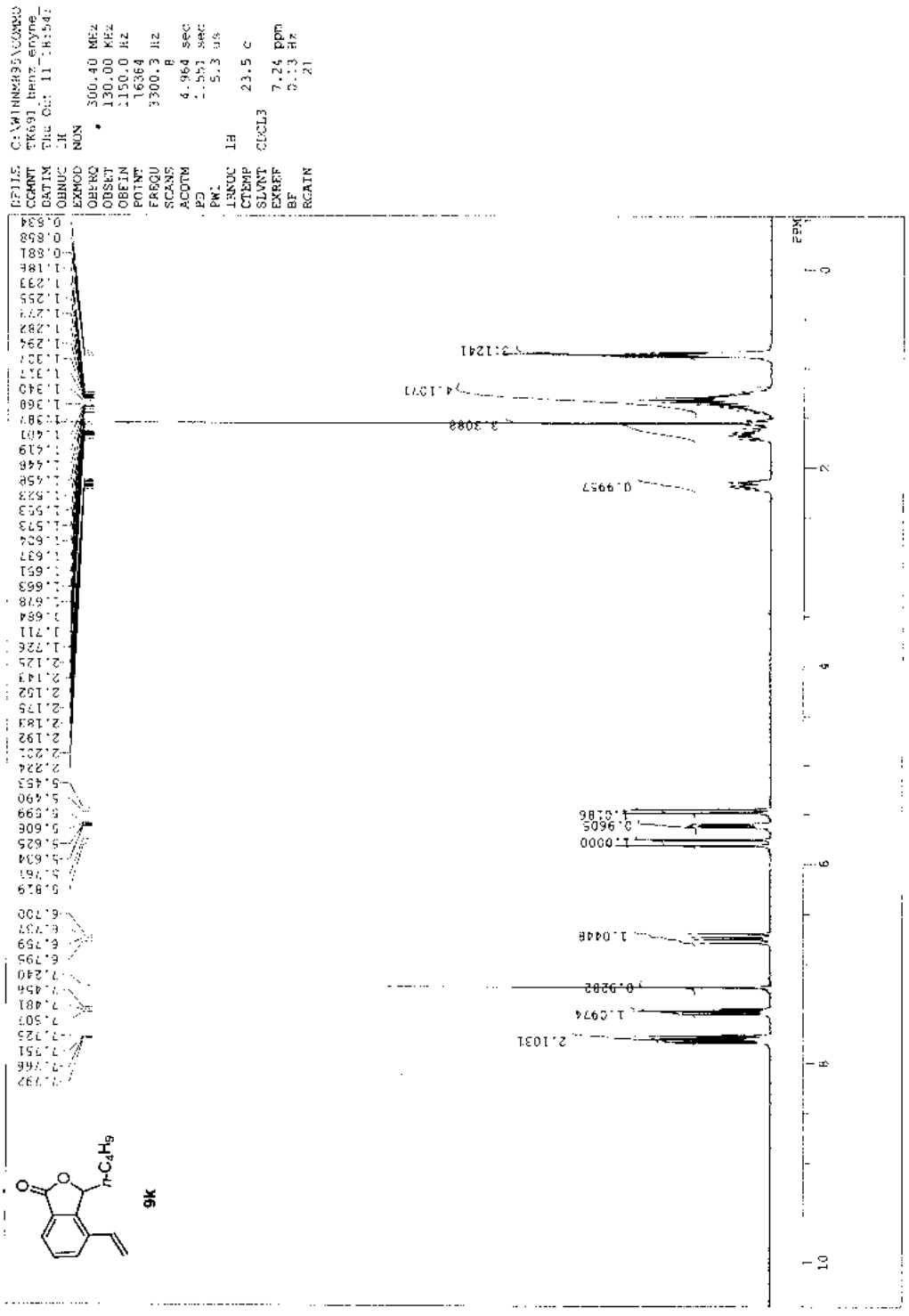
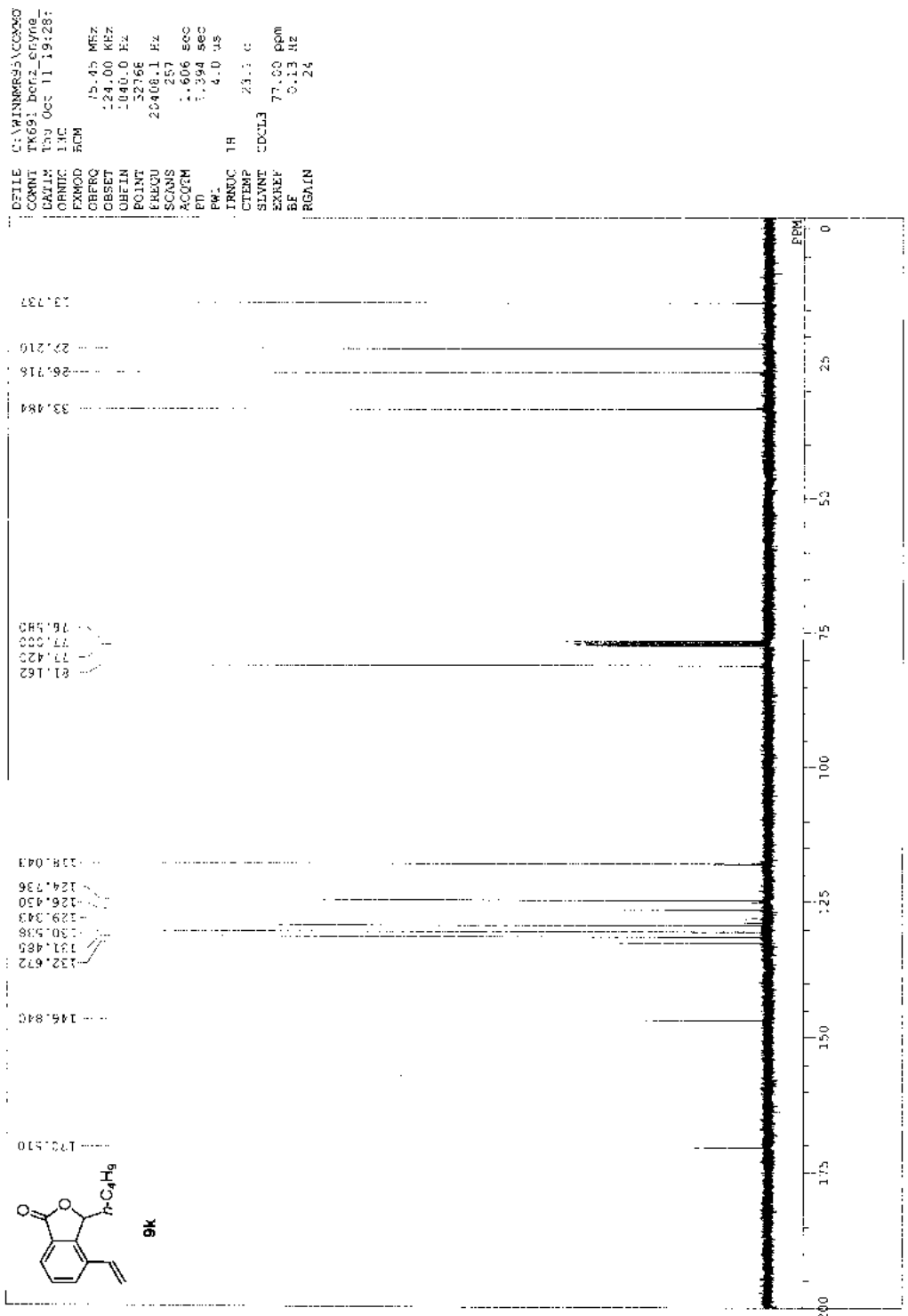
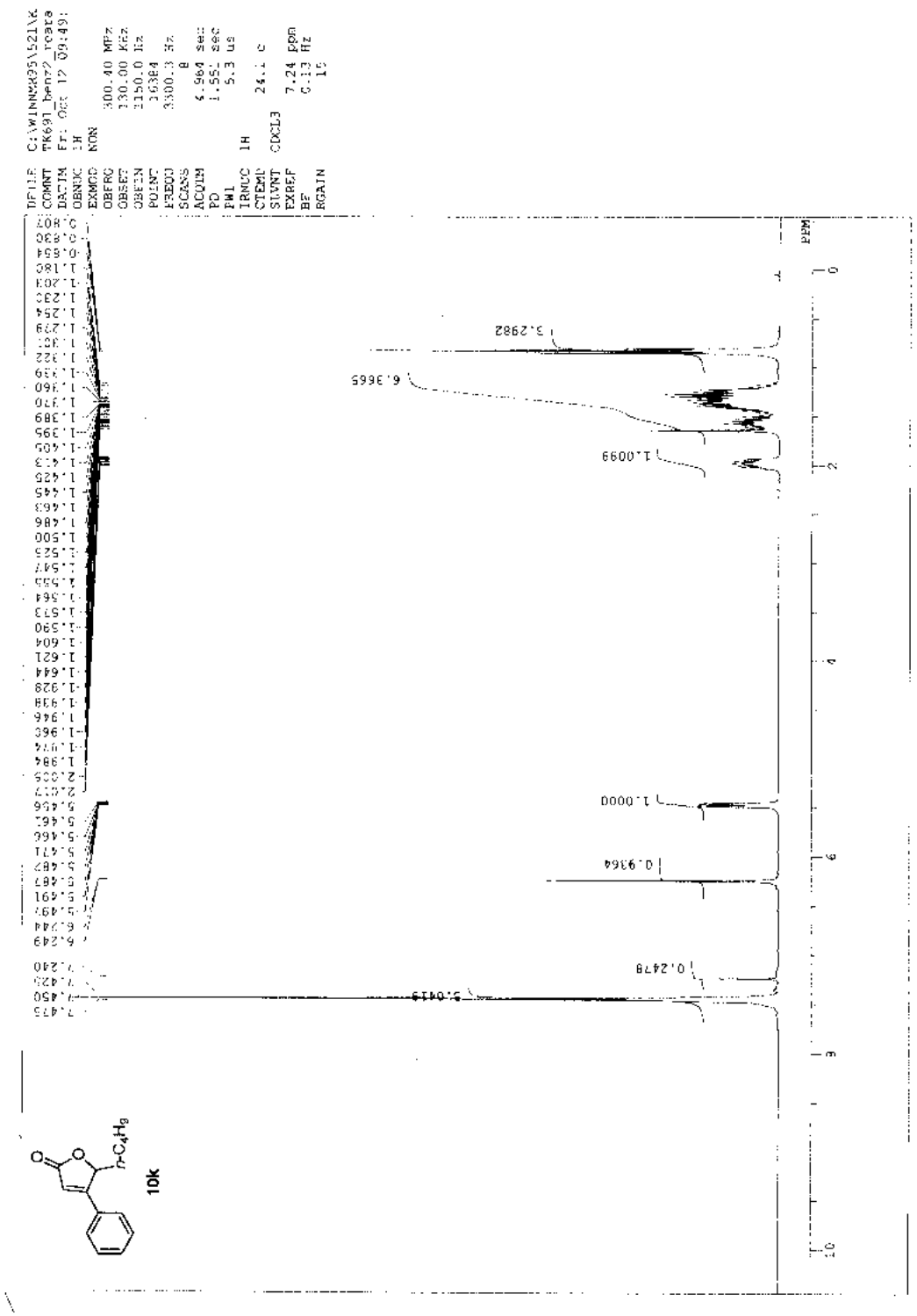
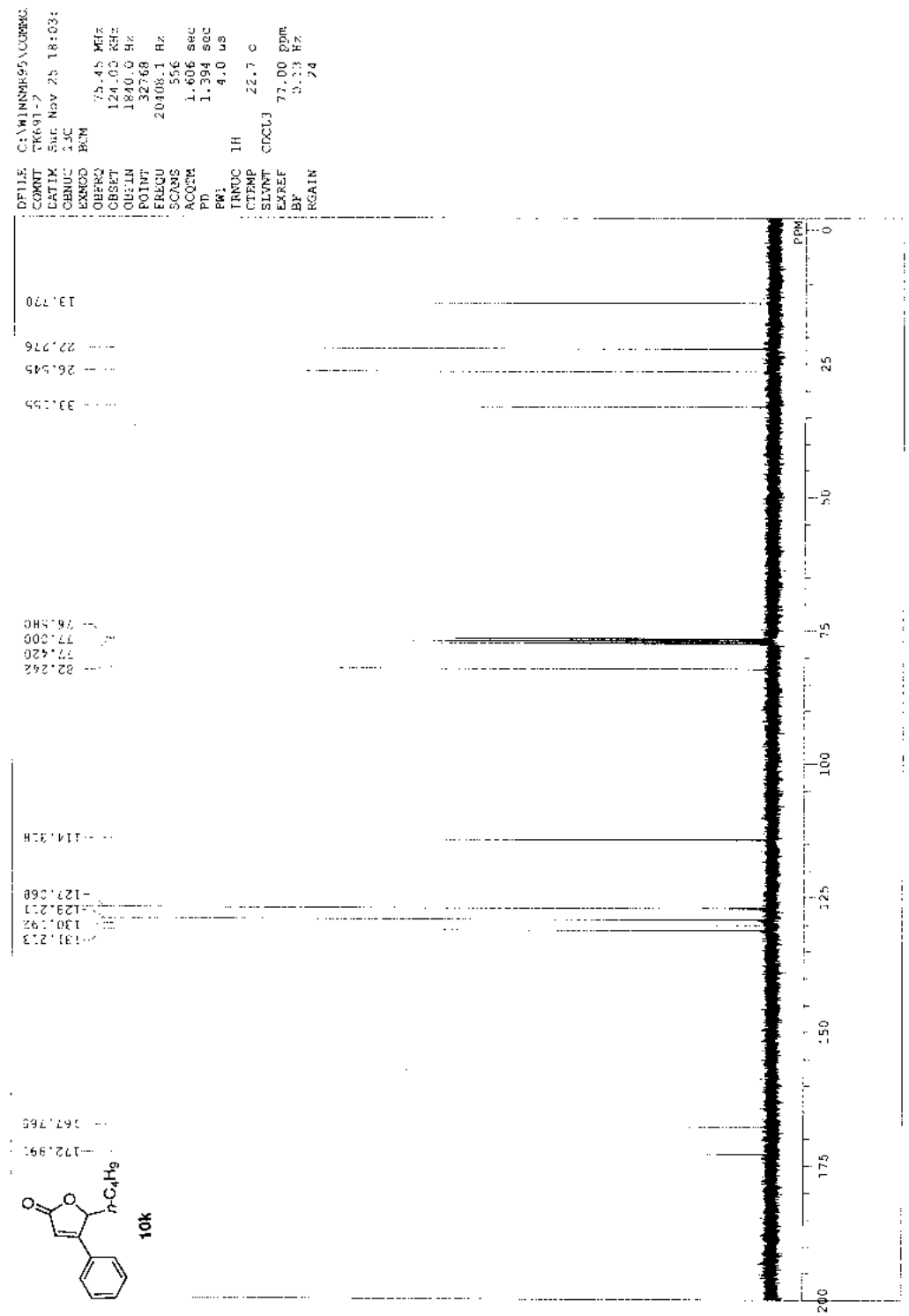
1.0290
1.0000
2.0528
3.0120
2.0093
1.3766
1.0460
1.0238
0.9810
PPM
0
2
4
6
8
10
Ph
O
O
9j

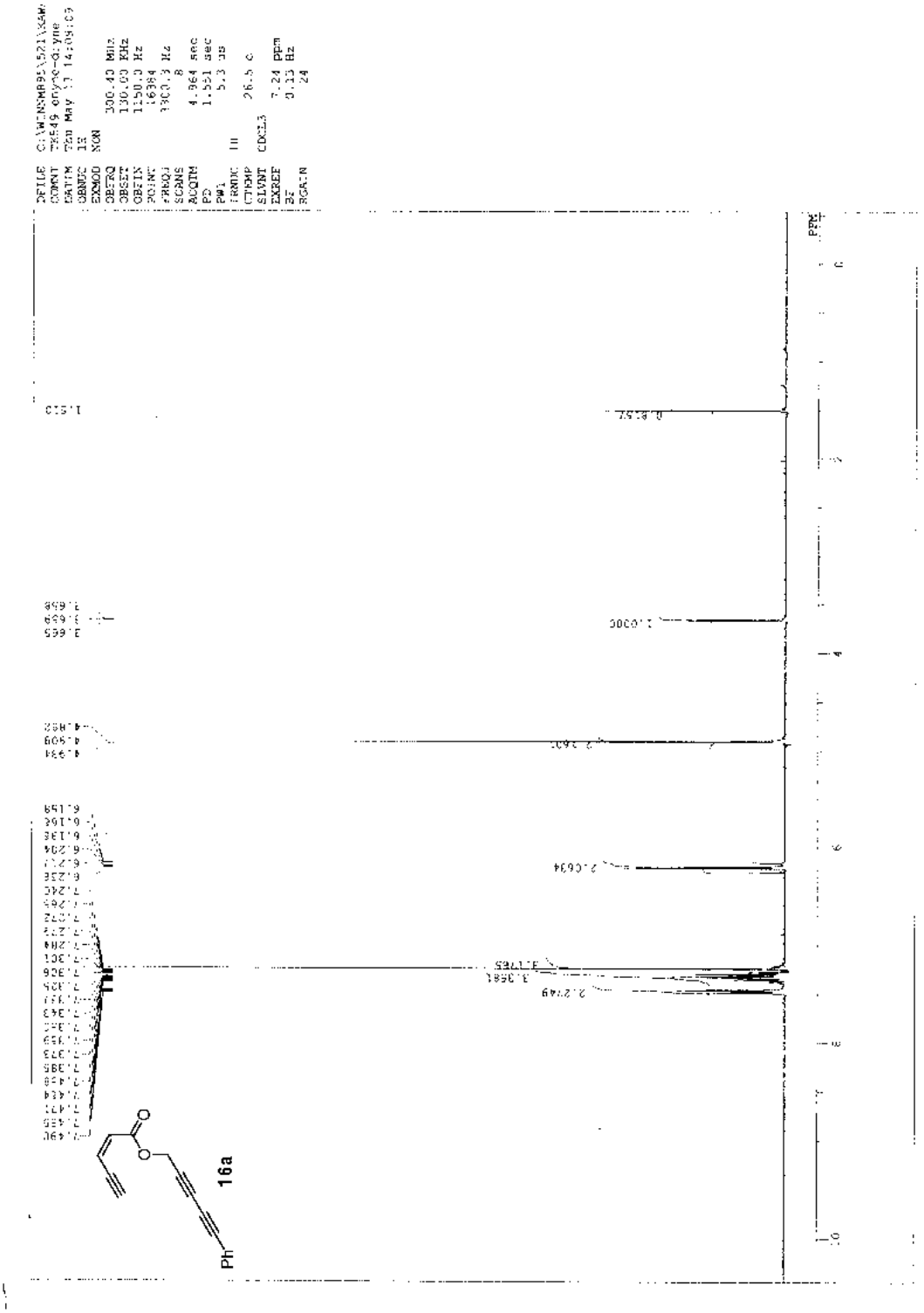

9j
Ph
O
O
PPM

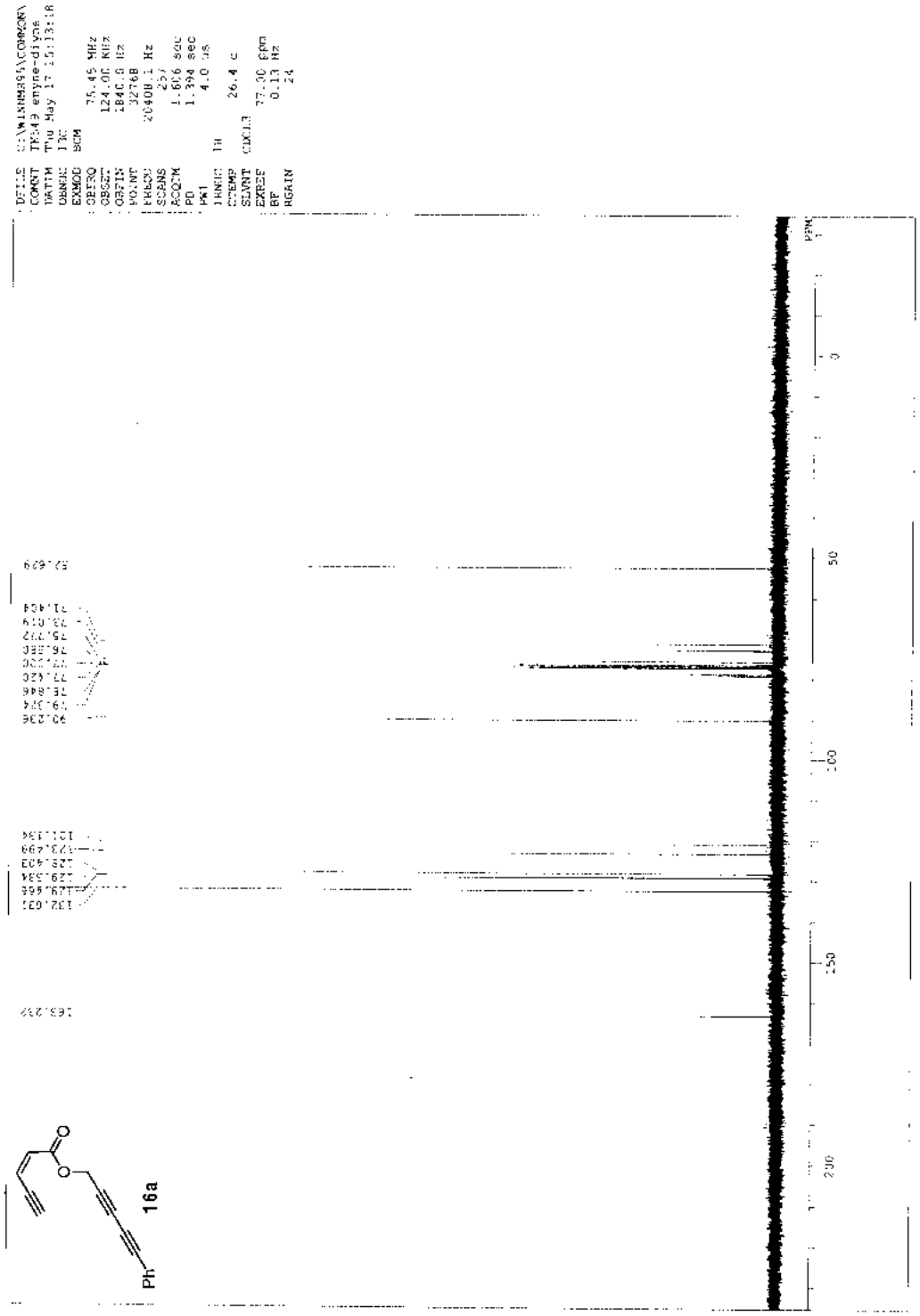

9k
n-C4H9
O
O
3.1241
4.1371
3.3088
0.9957
1.0000
0.9605
1.0186
1.0448
0.9282
1.0974
2.1031
PPM

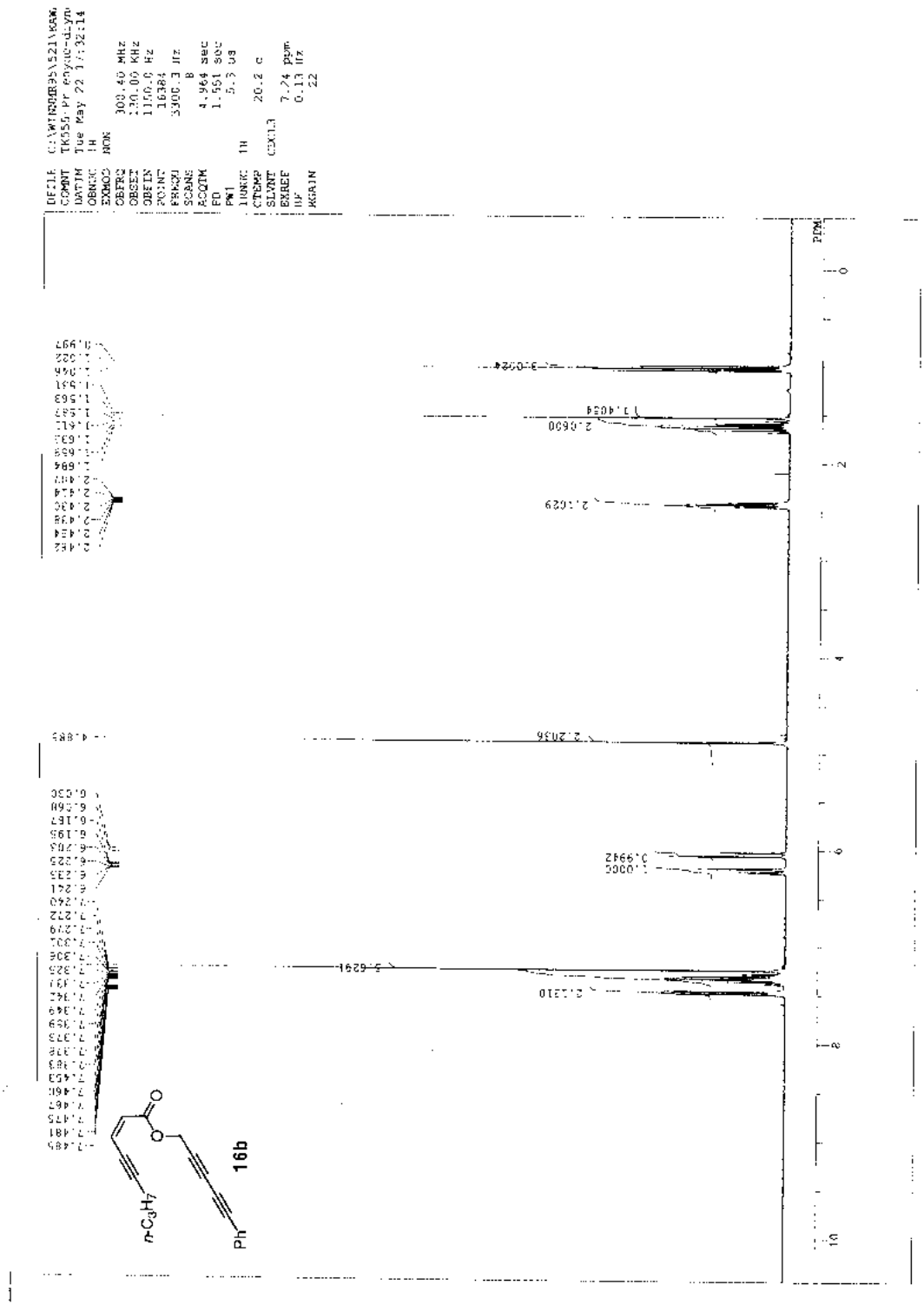

9k
n-C4H9
PPM

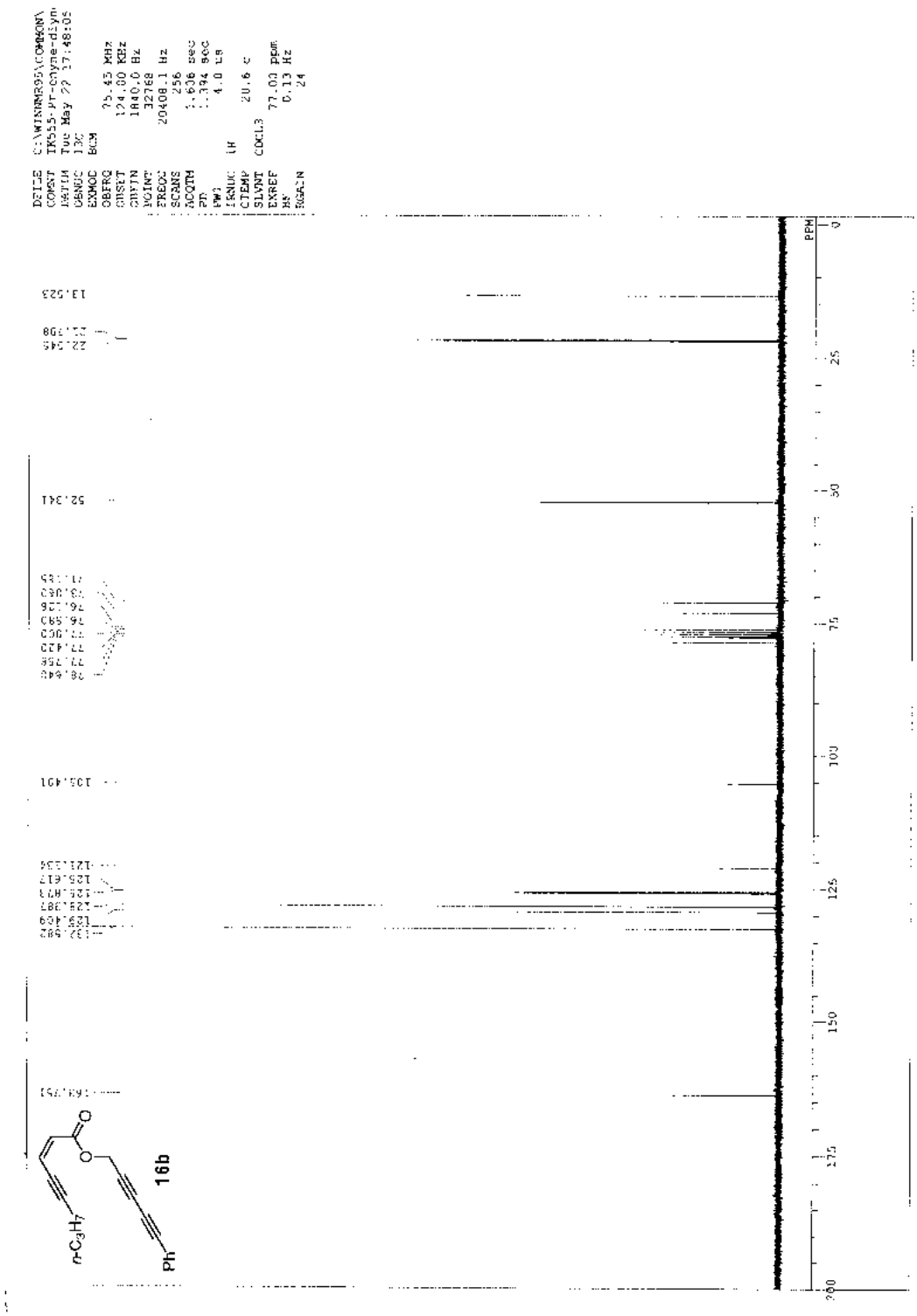

3.2982
6.3665
1.0099
1.0000
0.9864
0.2478
5.0419
PPM
10k


13.770
22.976
26.545
33.155
76.580
77.000
77.420
82.262
114.314
127.268
128.217
130.192
131.213
167.766
172.992
PPM
0
25
50
75
100
125
150
175
200
10k

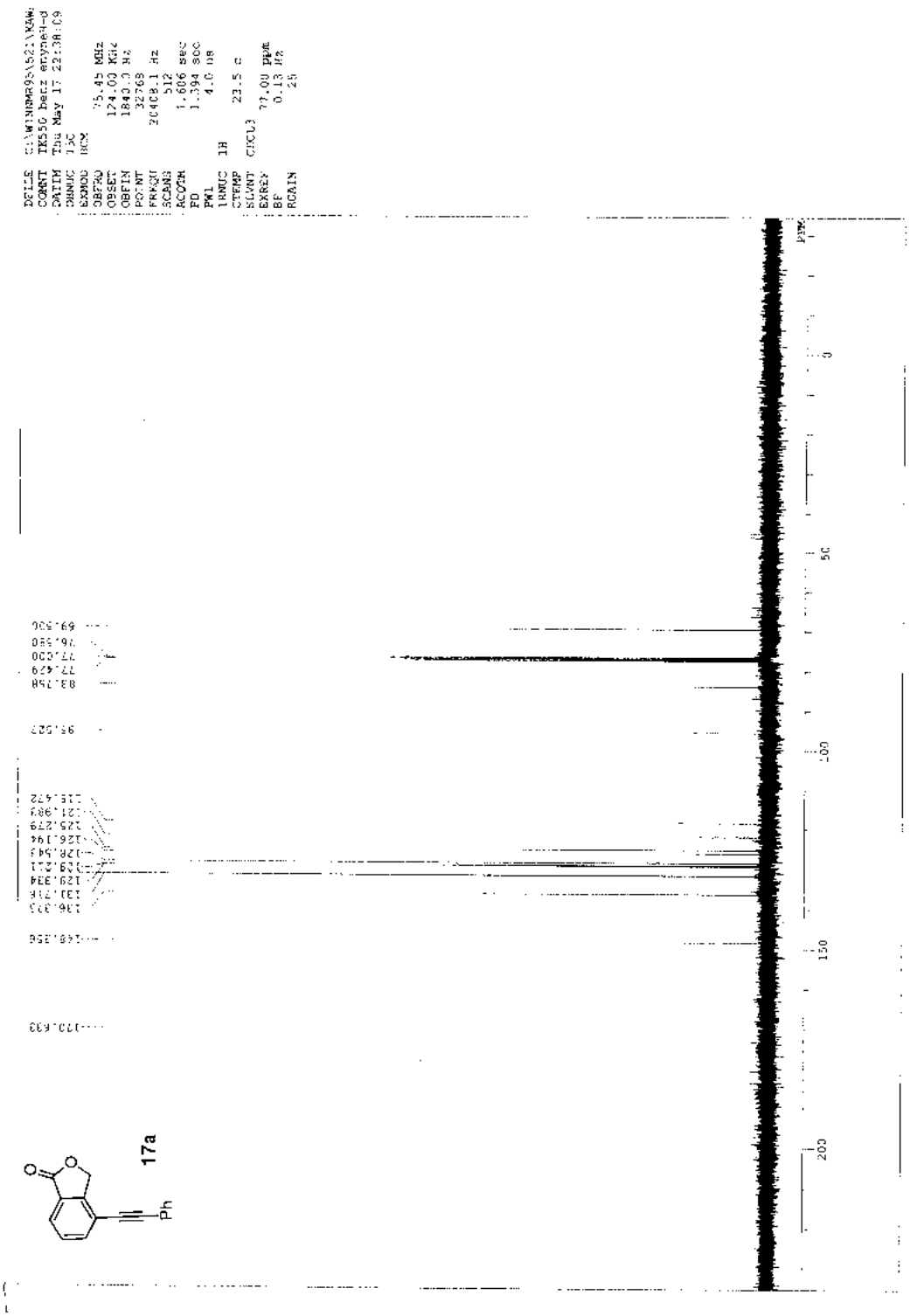

16a
Ph

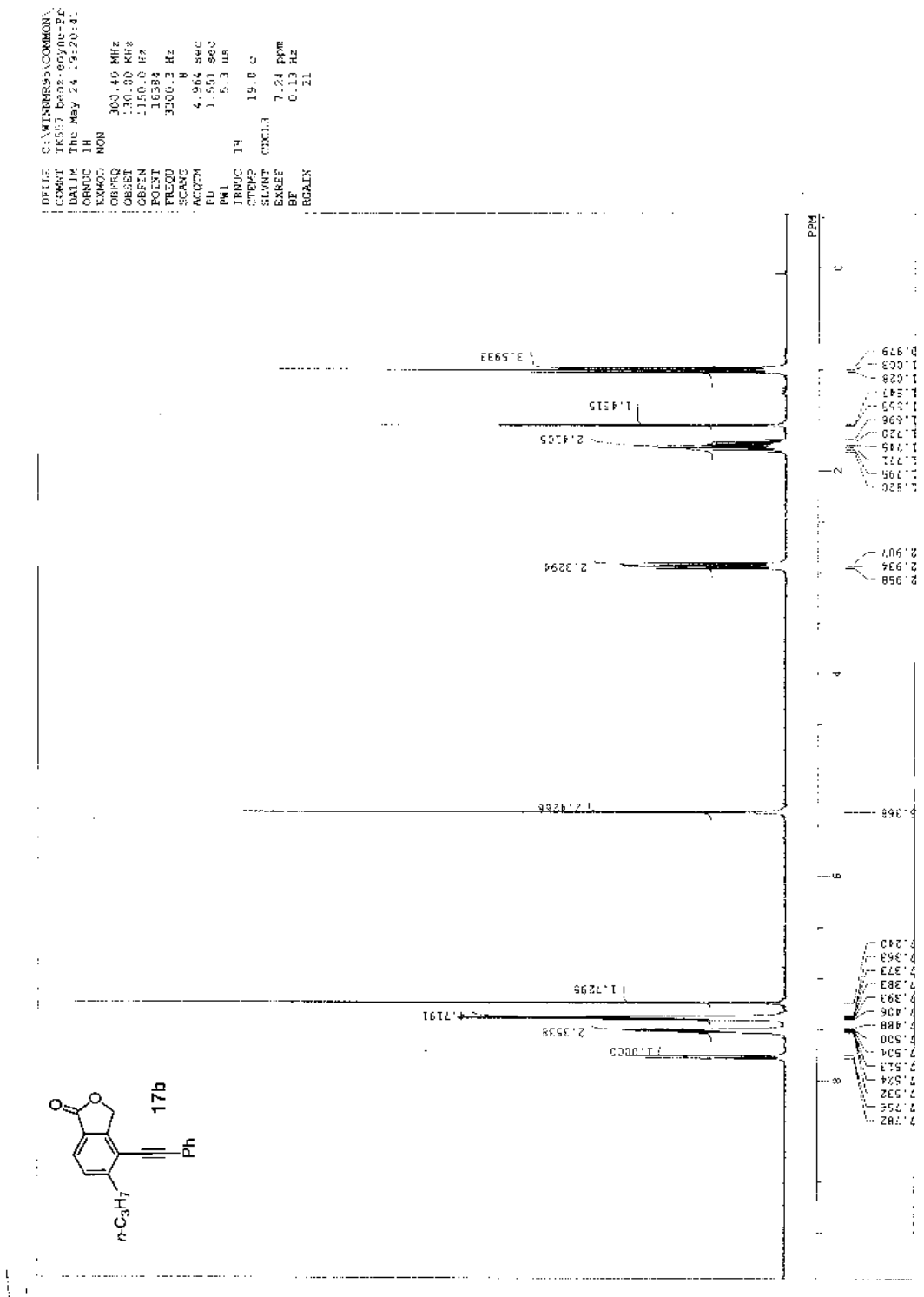

16a
Ph

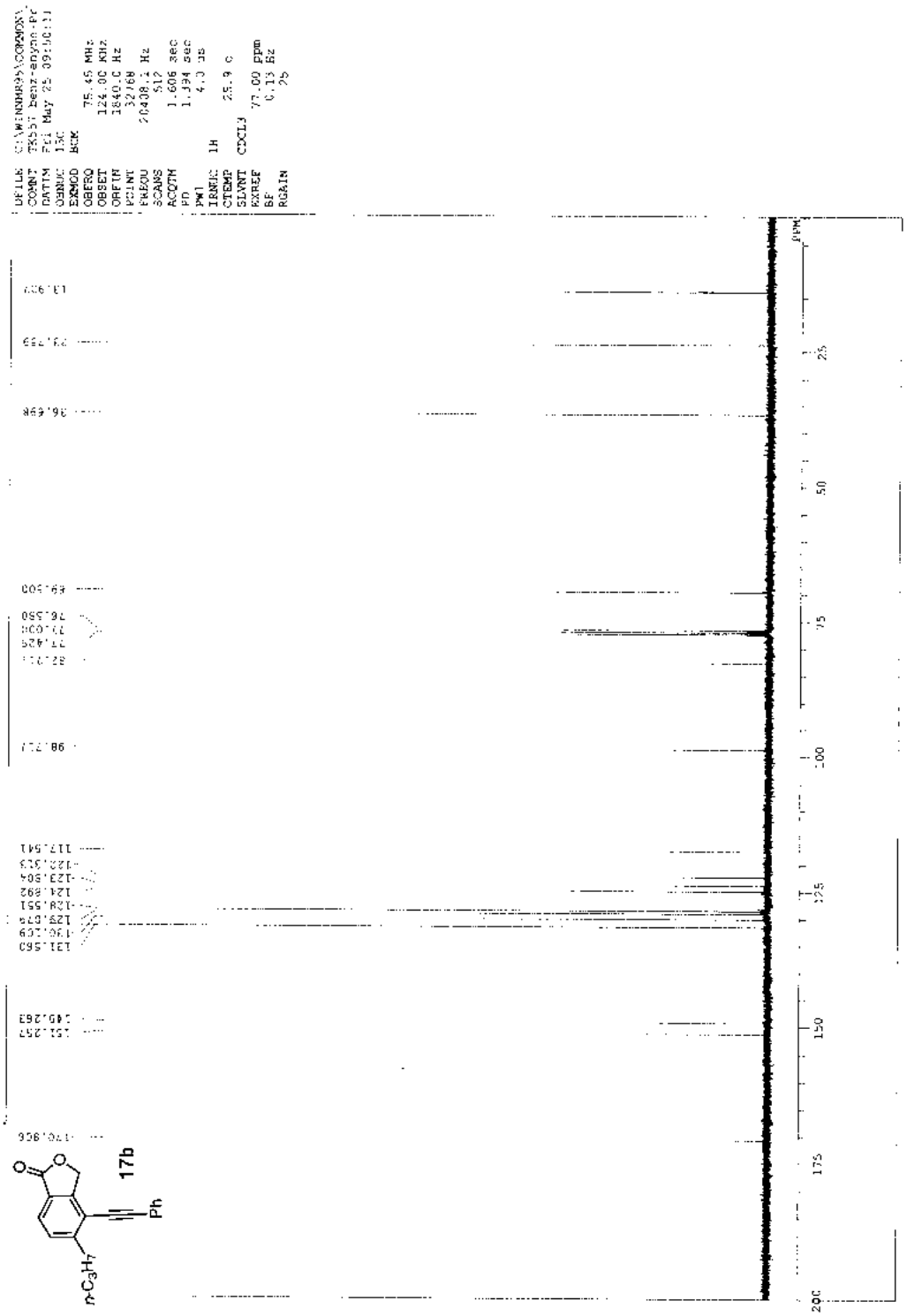

16b
n-C3H7
Ph
PPM

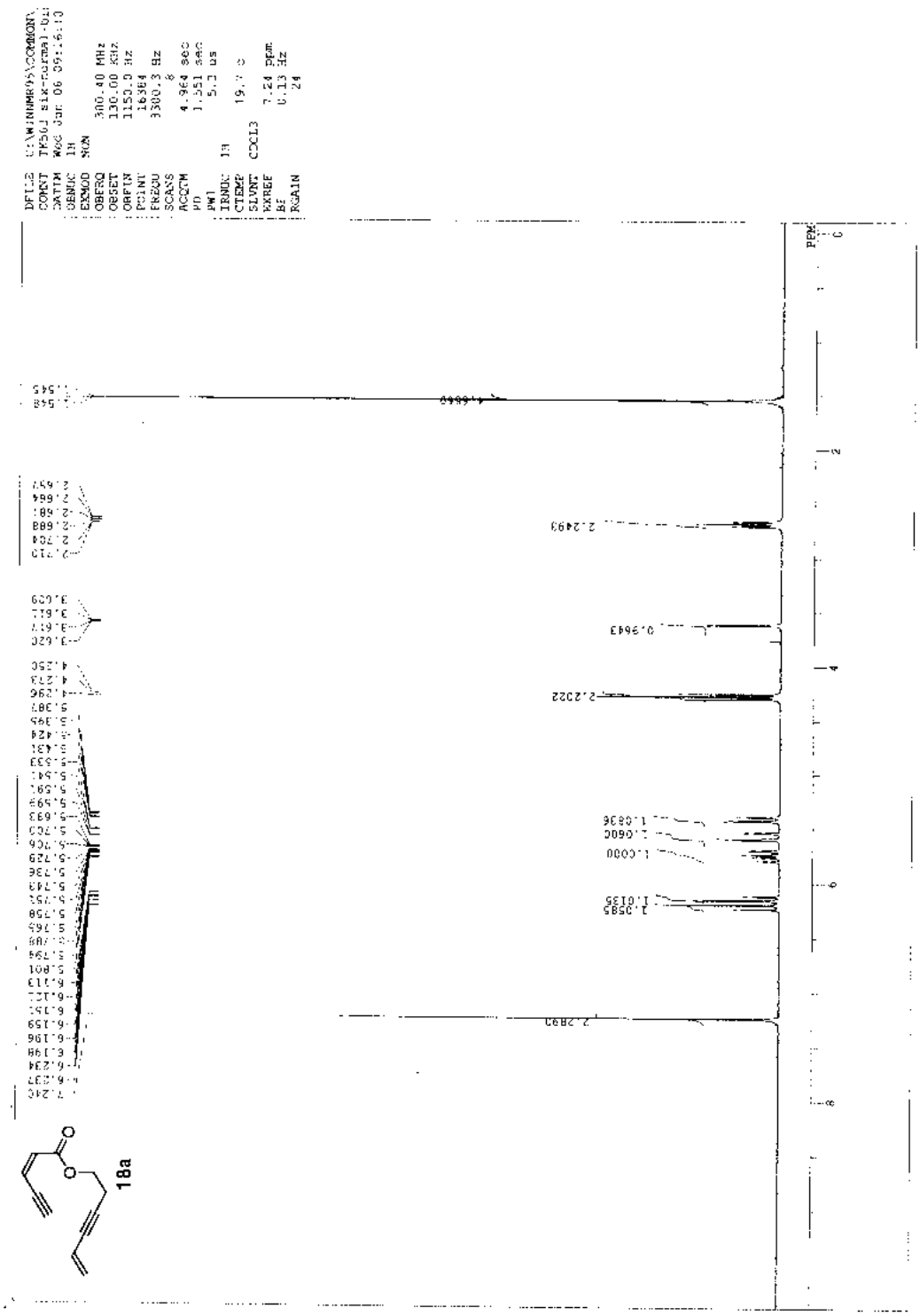

16b
n-C3H7
Ph
PPM

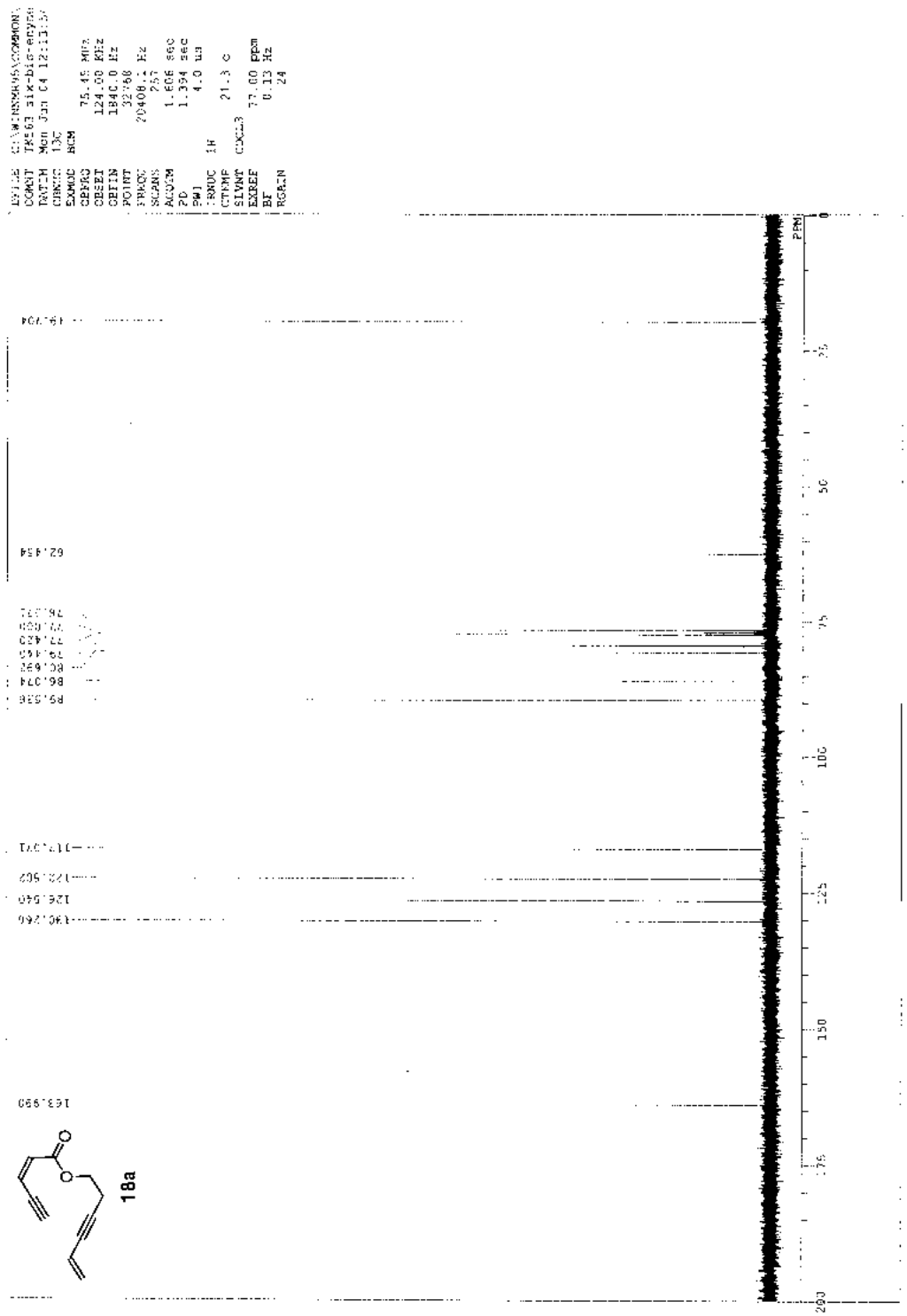

17a
Ph


17a
Ph


17b
Ph
n-C3H7


17b
Ph
n-C3H7


18a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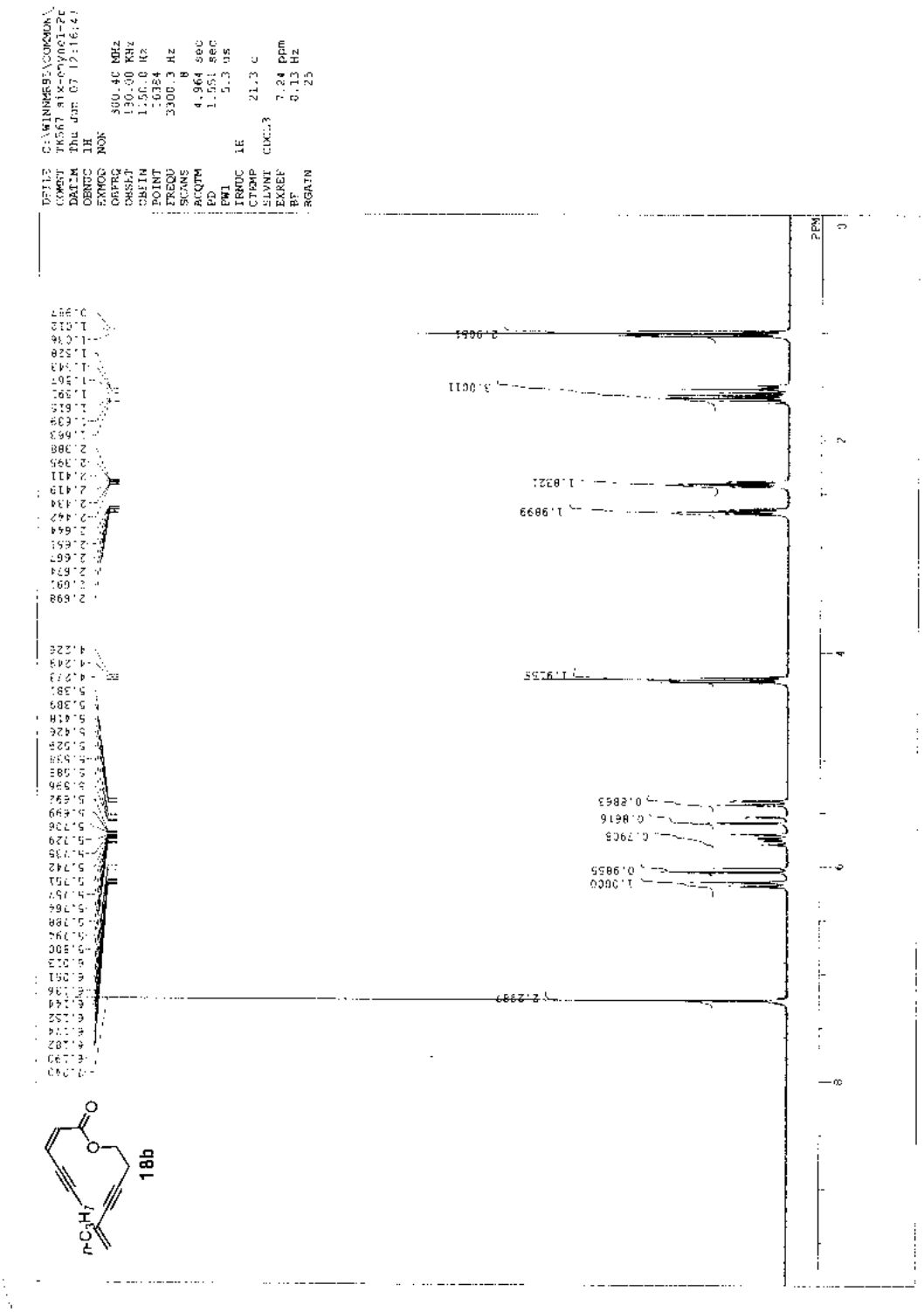
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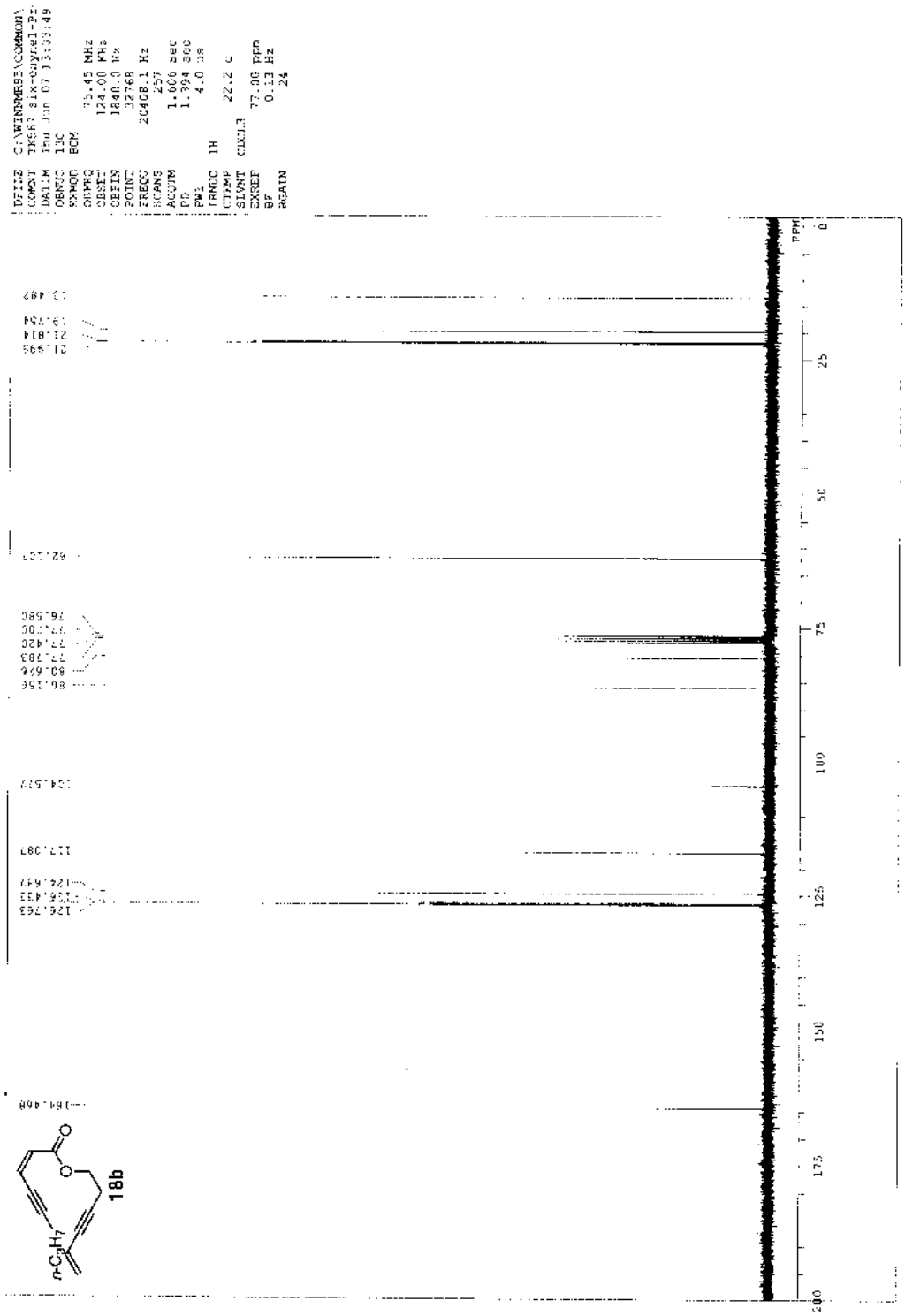
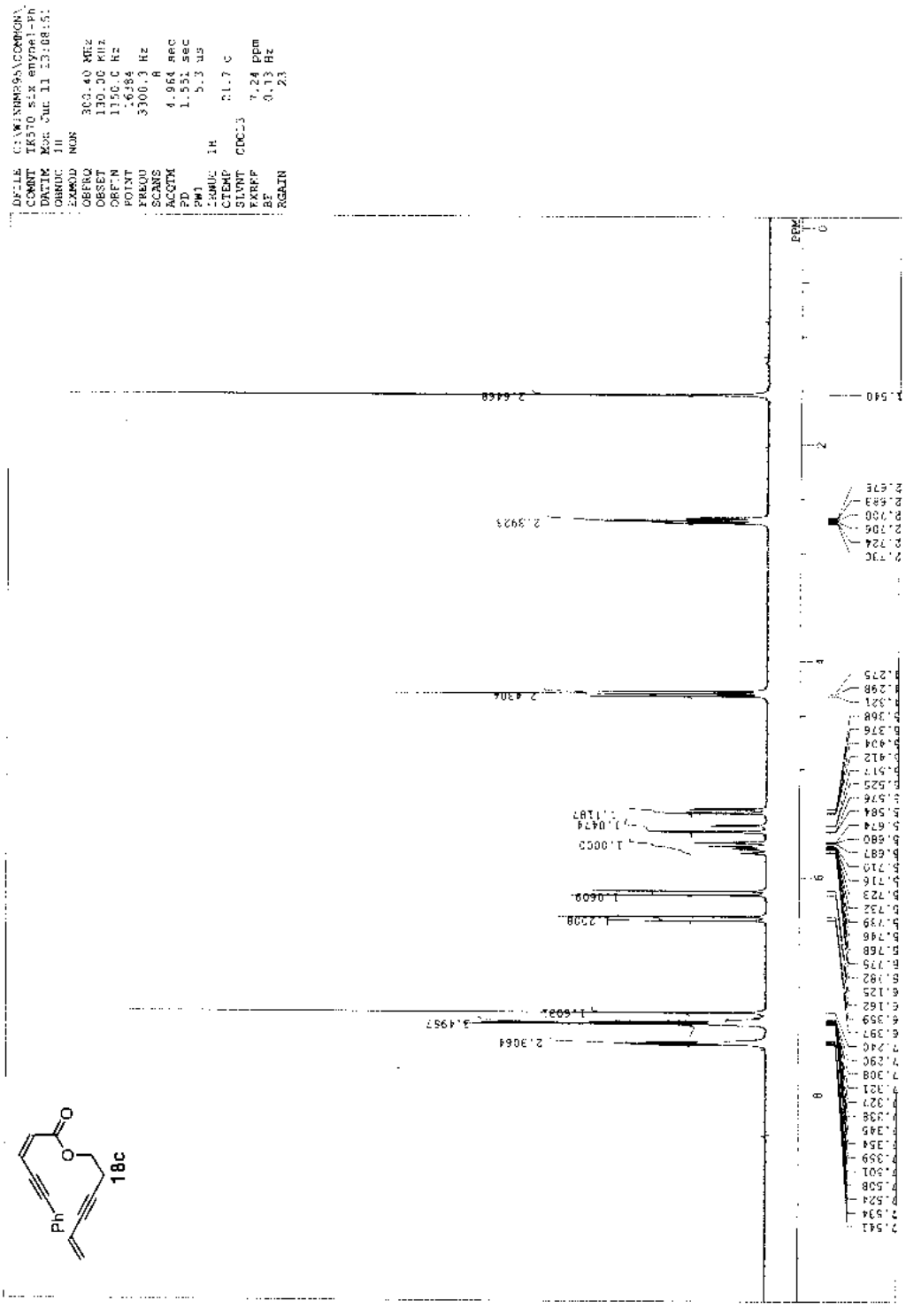
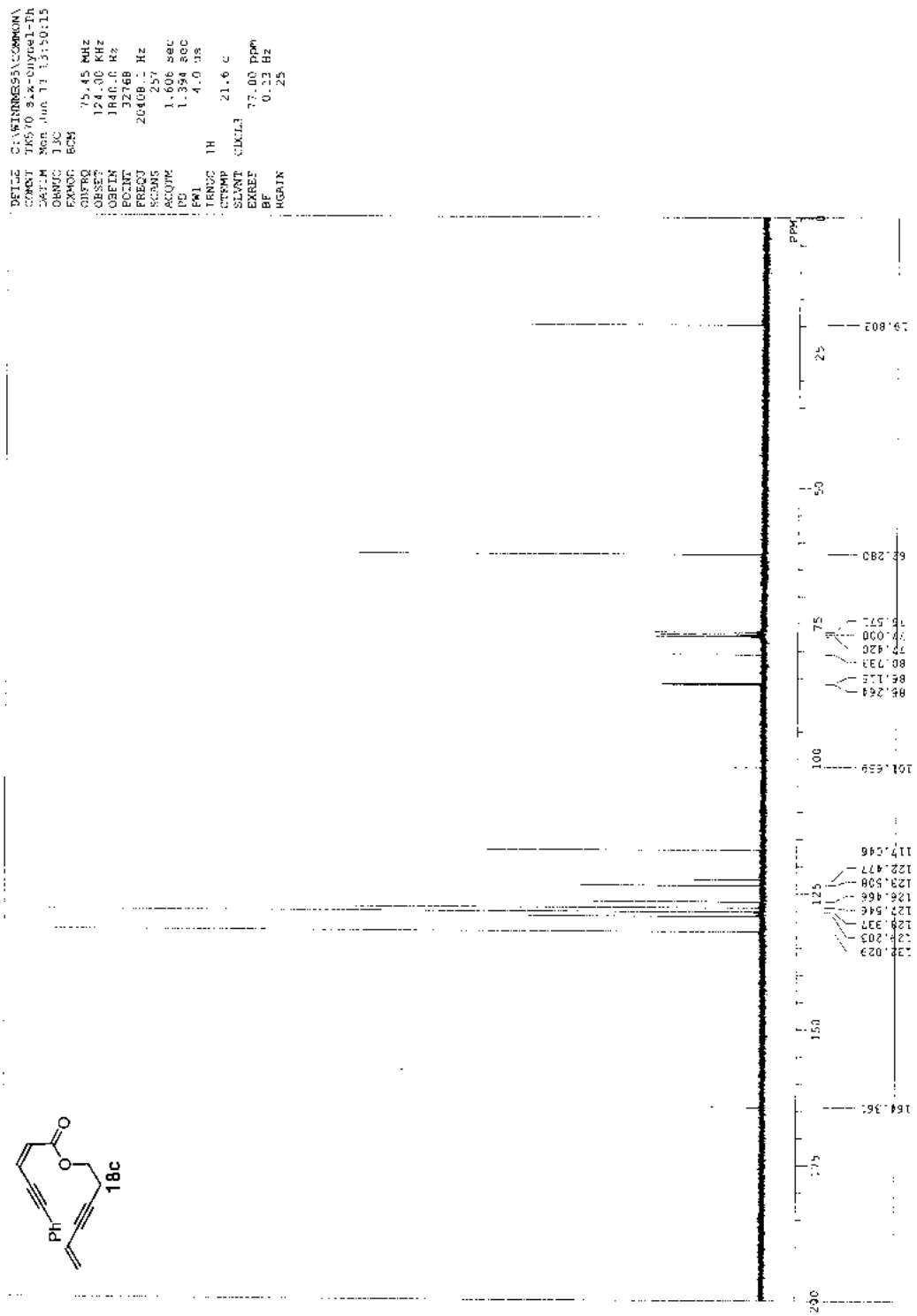
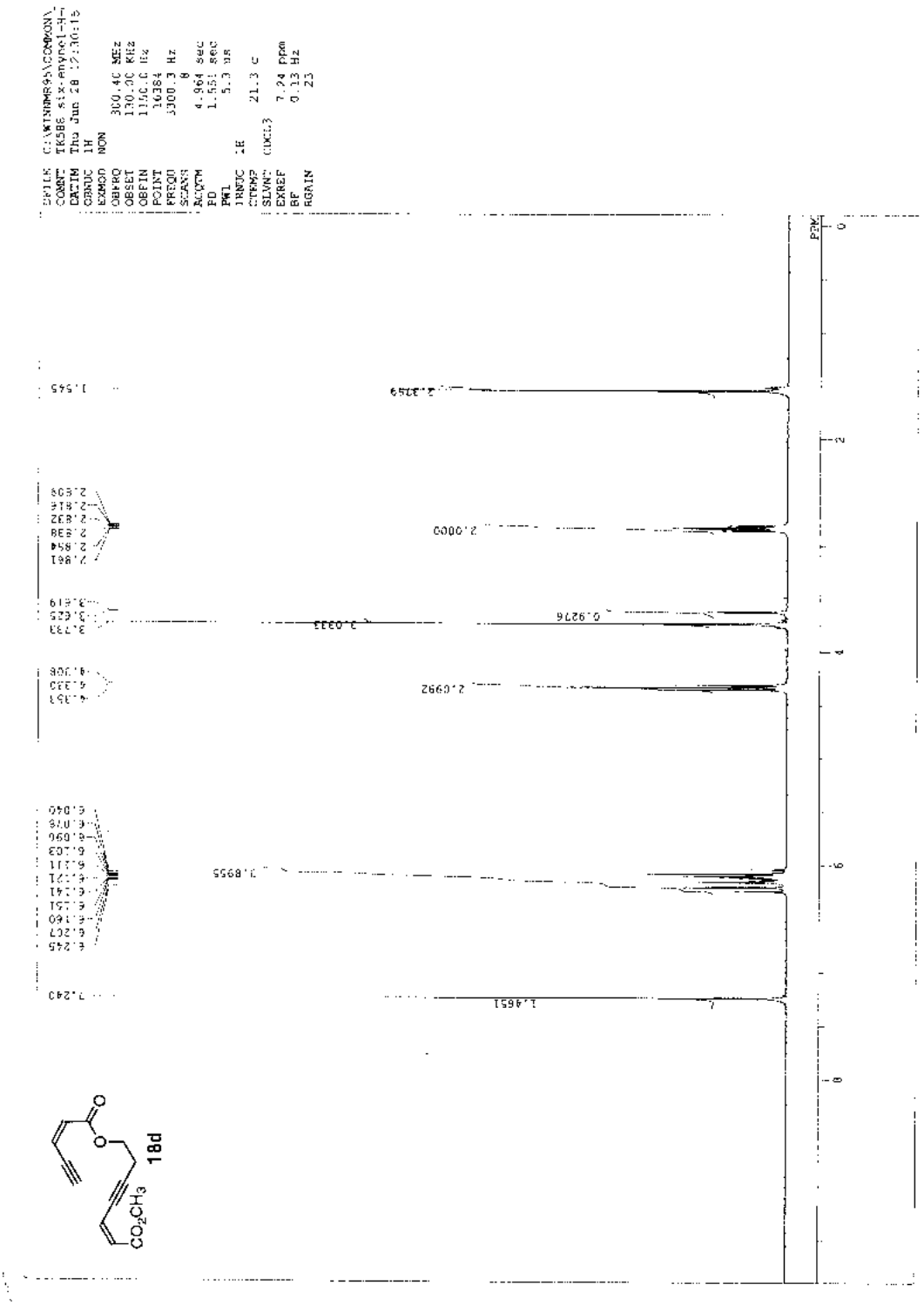
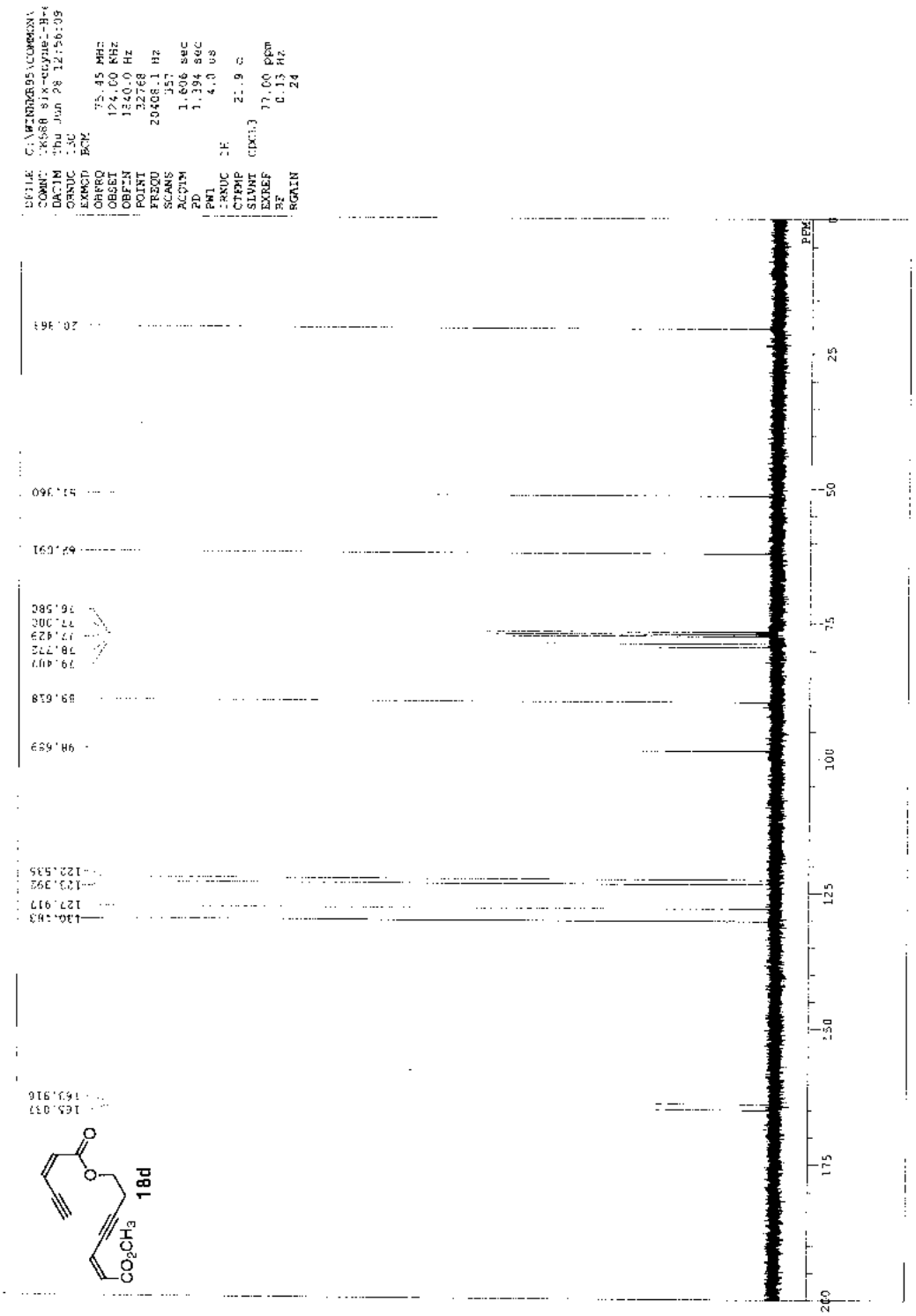
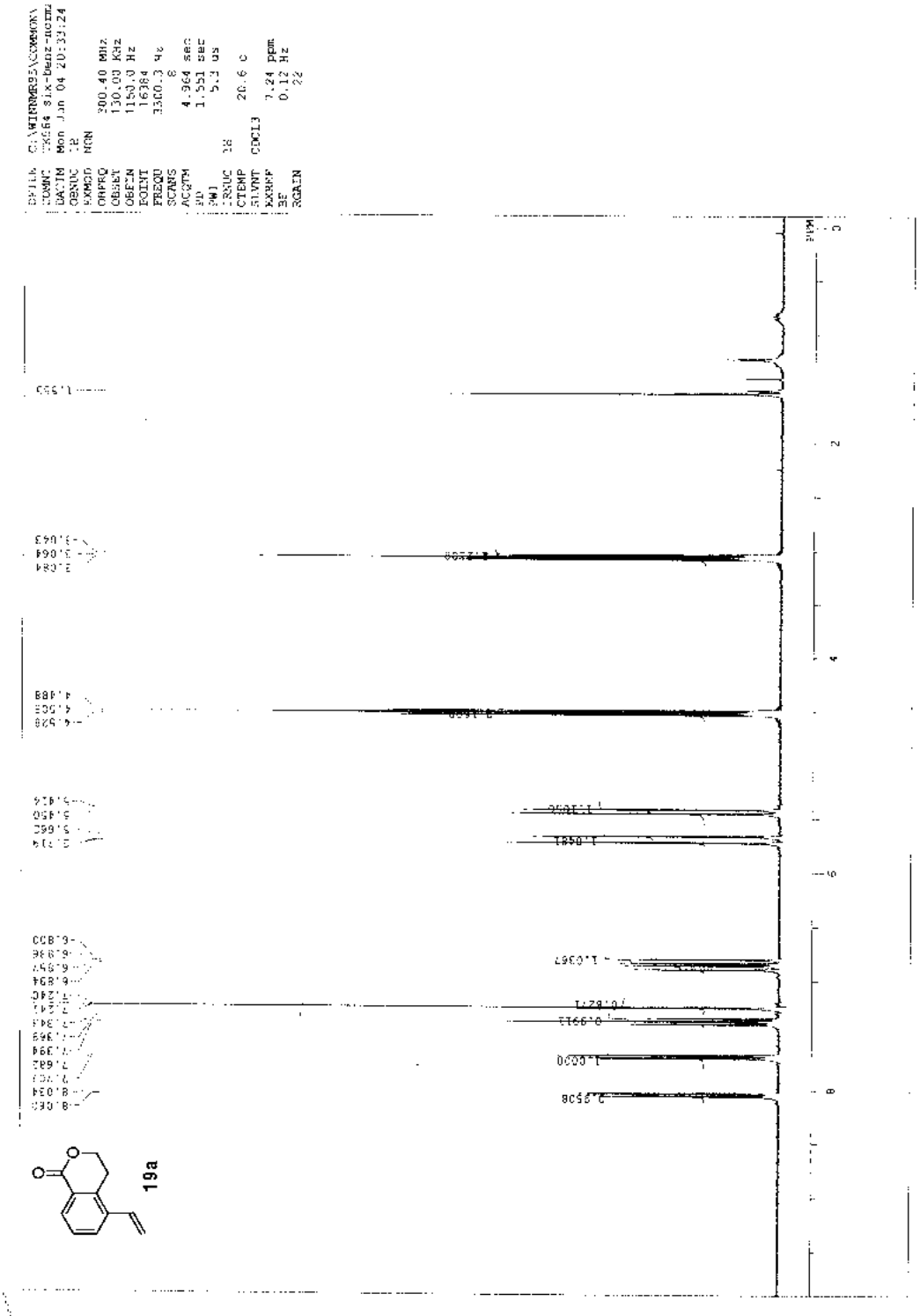

18b
n-C5H7

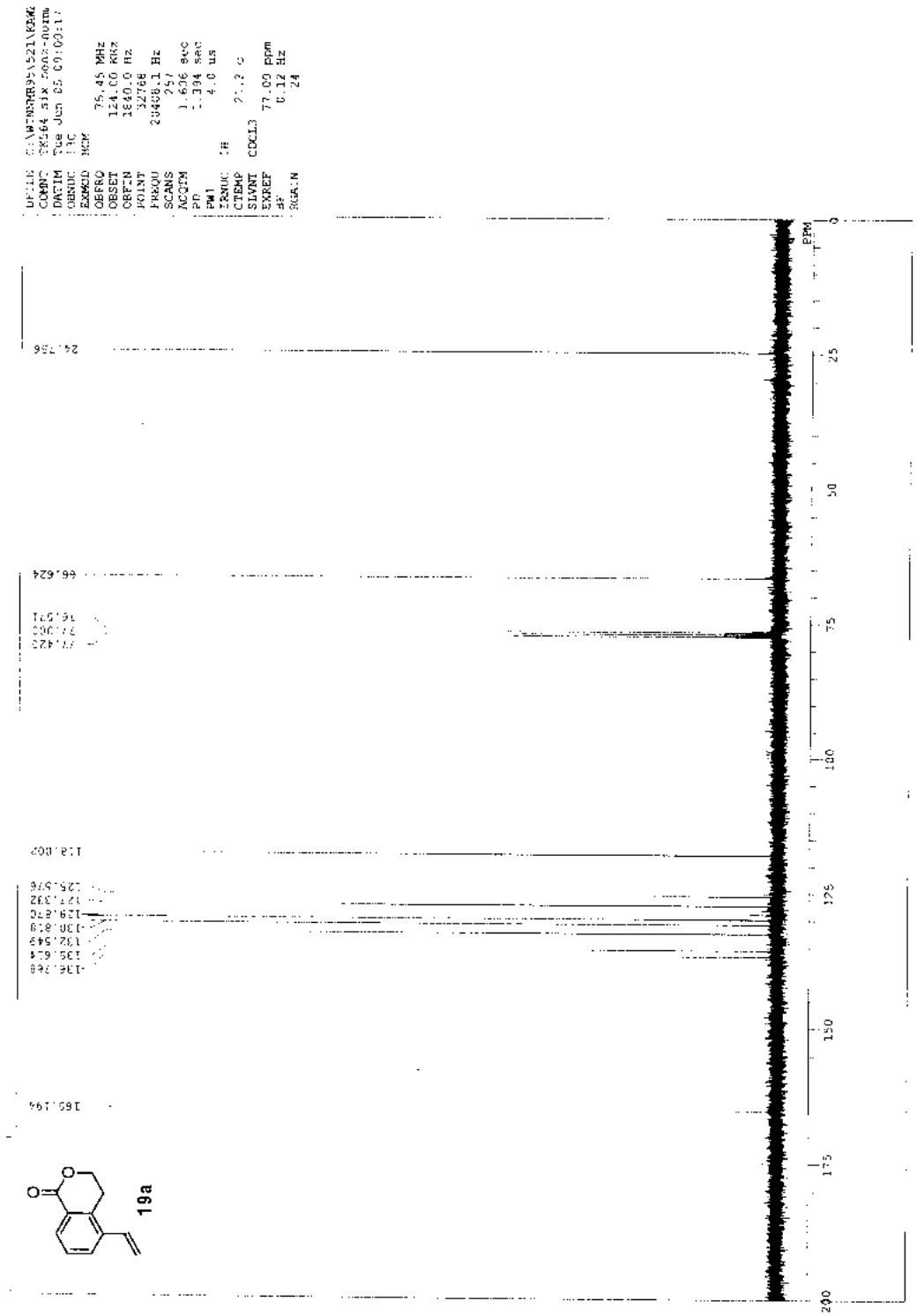

18c
Ph
O
O

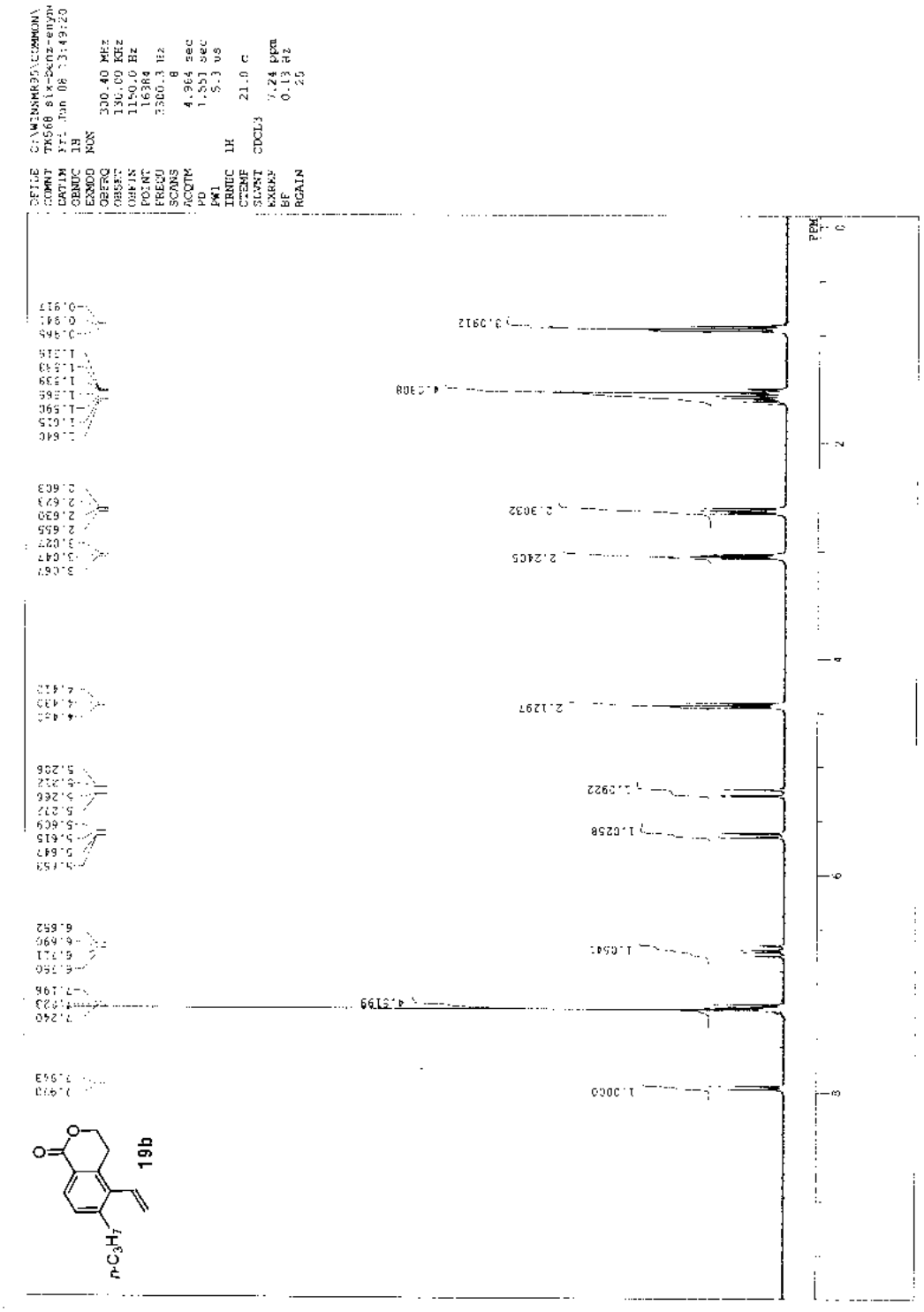

18c
Ph

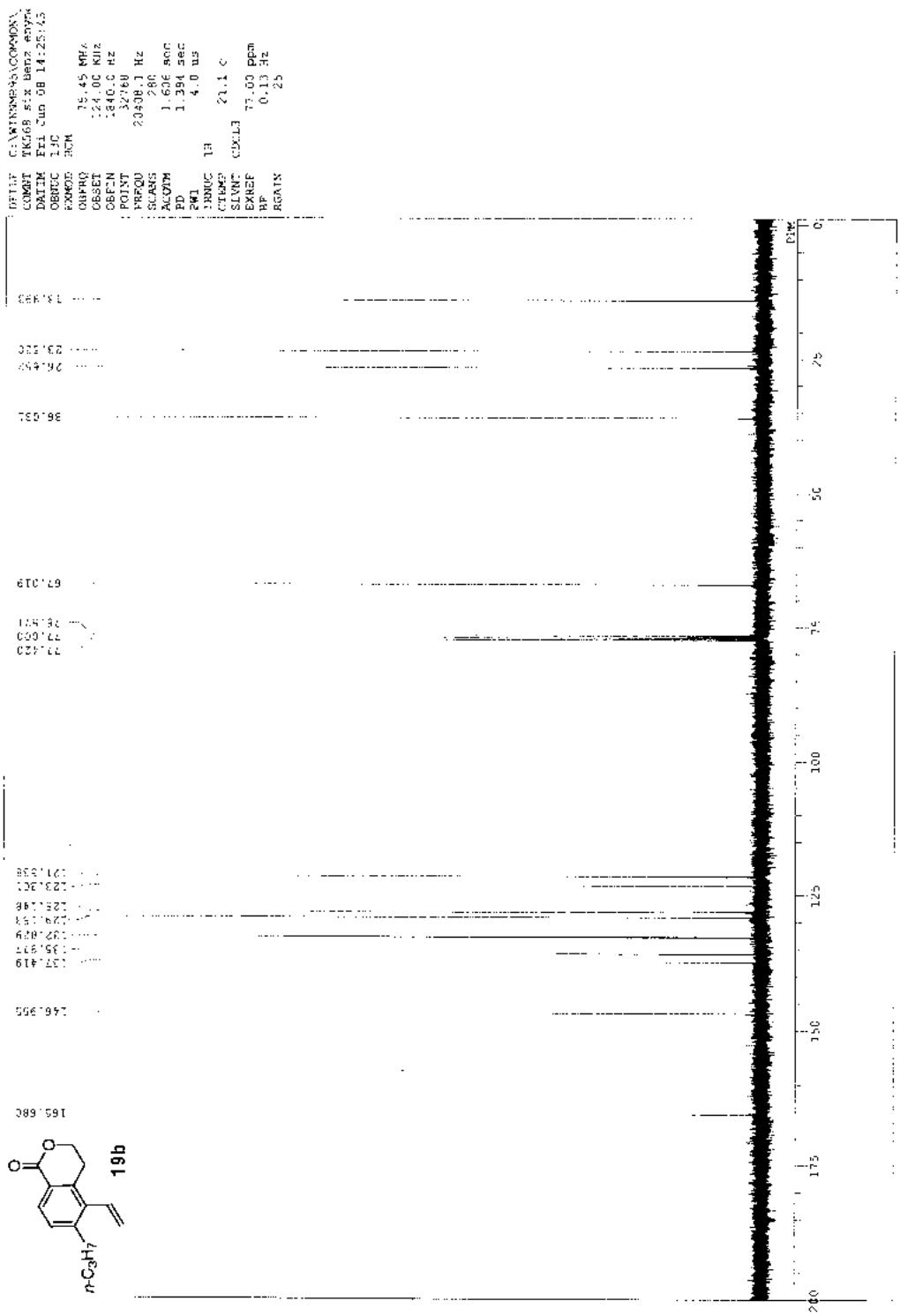

18d
CO2CH3


18d
CO2CH3
PPM

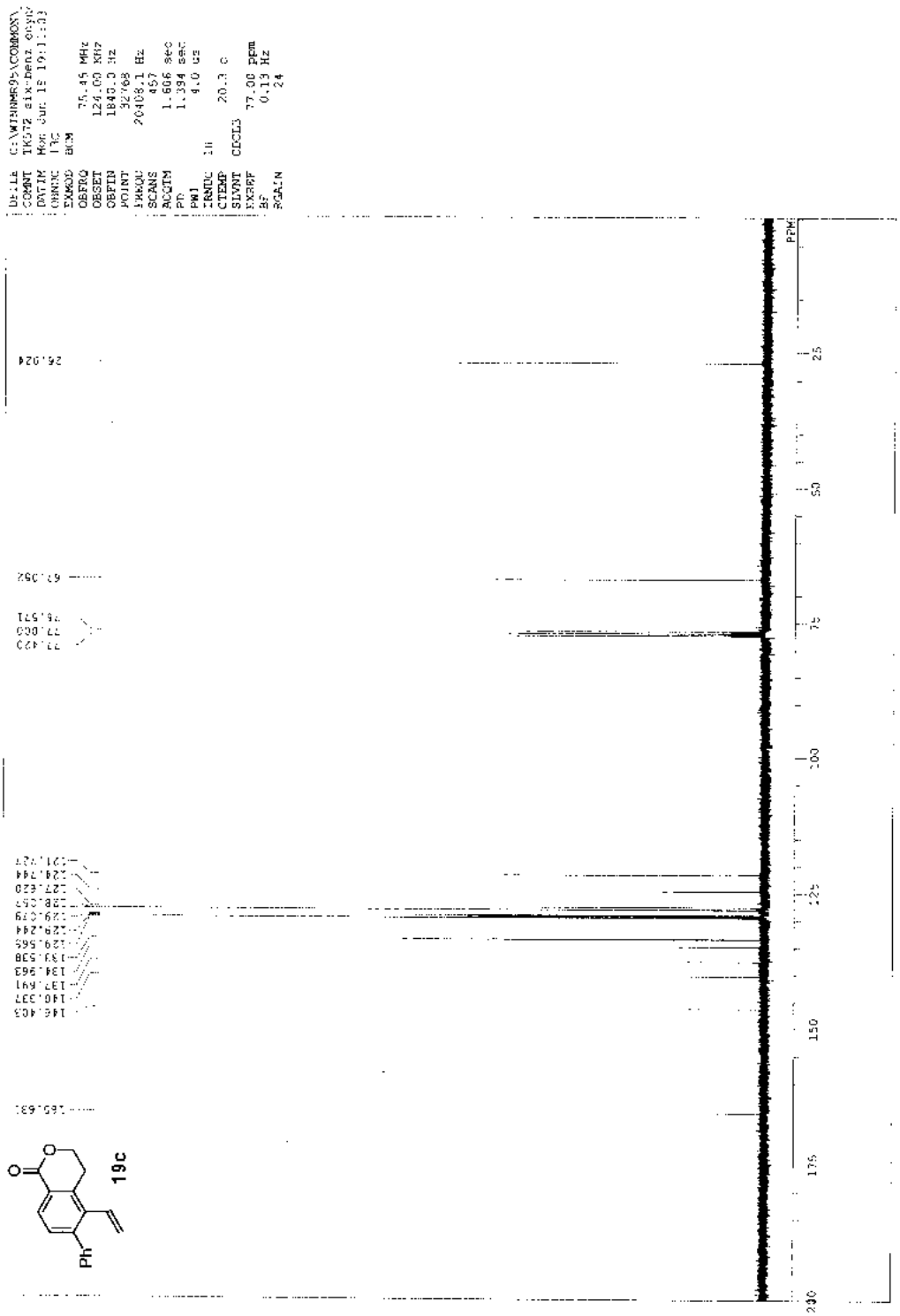

19a

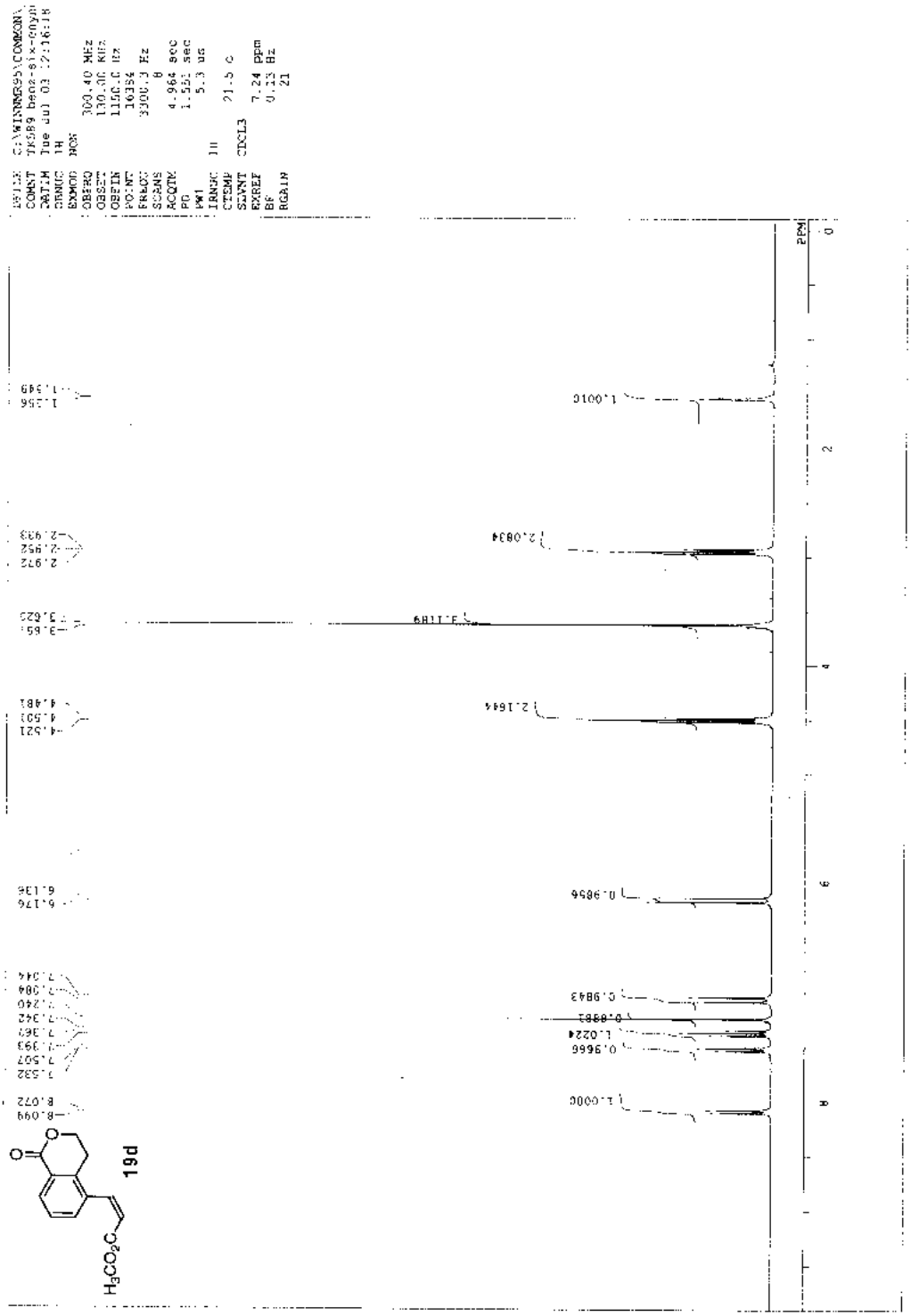

19a
CDCl3
77.00 ppm
PPM


19b
n-C3H7


19b
n-C3H7


19c
Ph


19c
Ph
O
O
PPM


19d
H_3CO_2C
O
O
PPM
0
2
4
6
8
1.000
2.083
3.118
2.164
1.000
0.985
0.984
0.988
1.022
0.966
1.000
1.256
1.349
2.972
2.952
2.933
3.625
4.521
4.501
4.481
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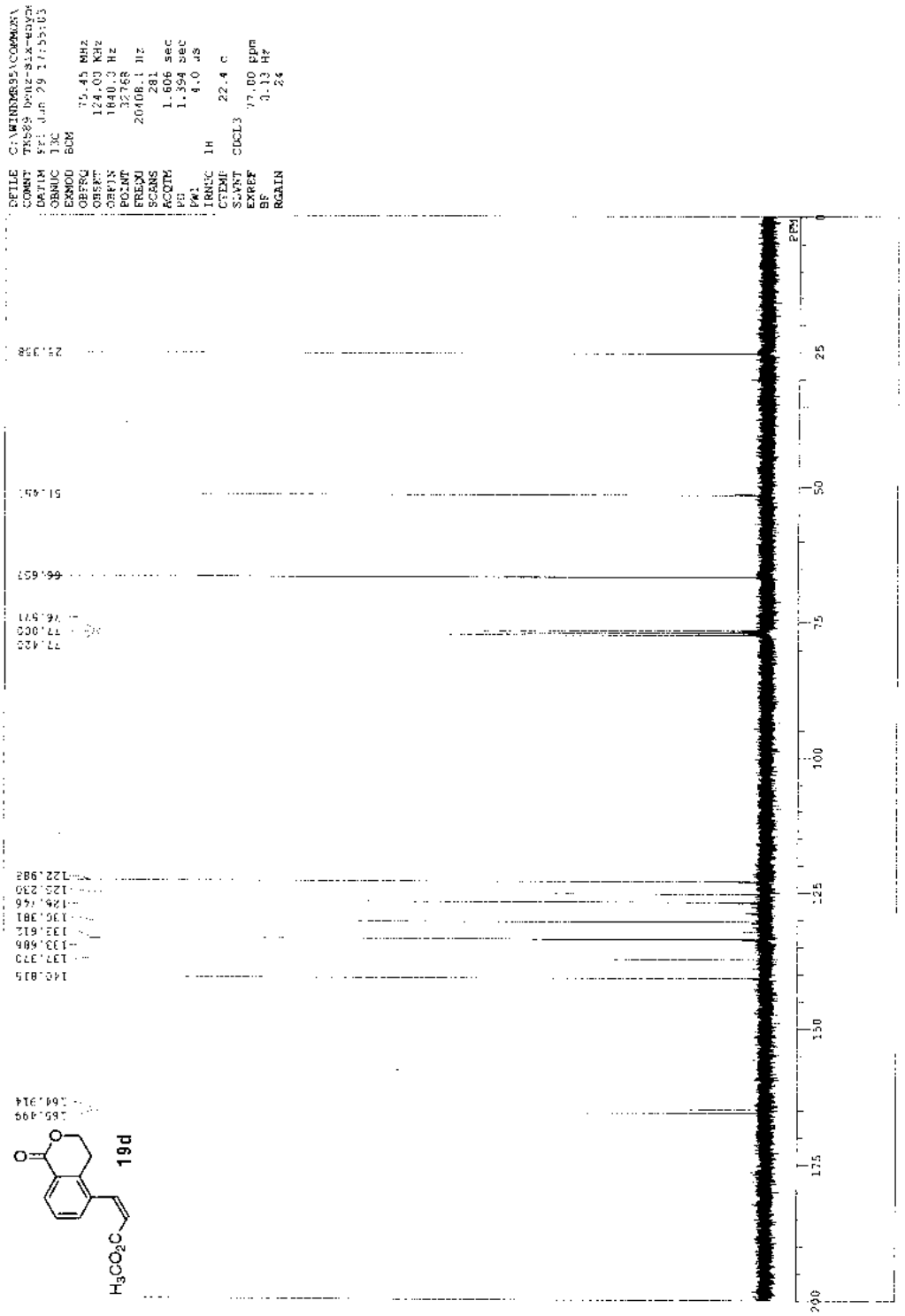
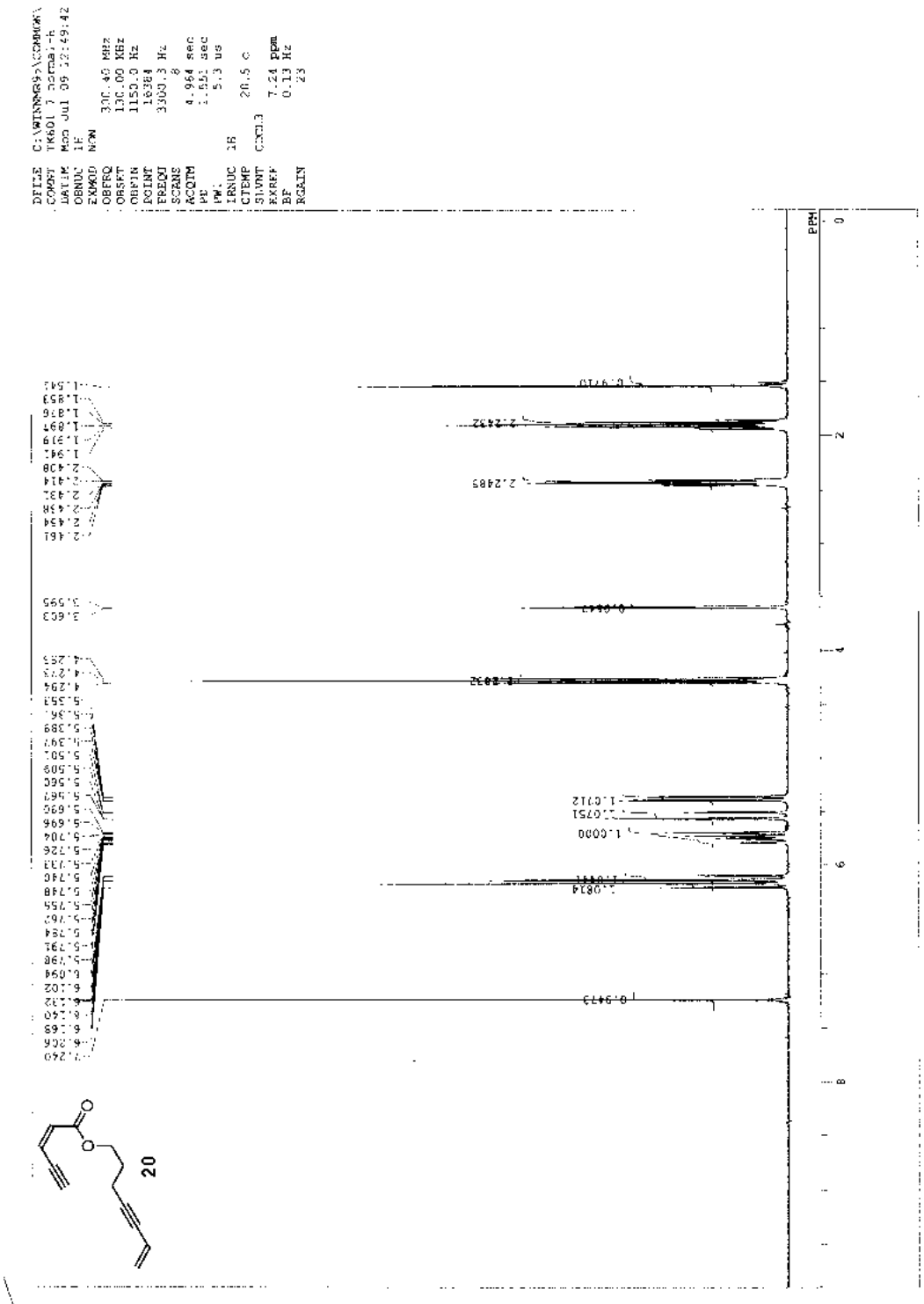
7.342
7.240
7.084
7.044
8.099
8.072


19d
H_3CO_2C
PPM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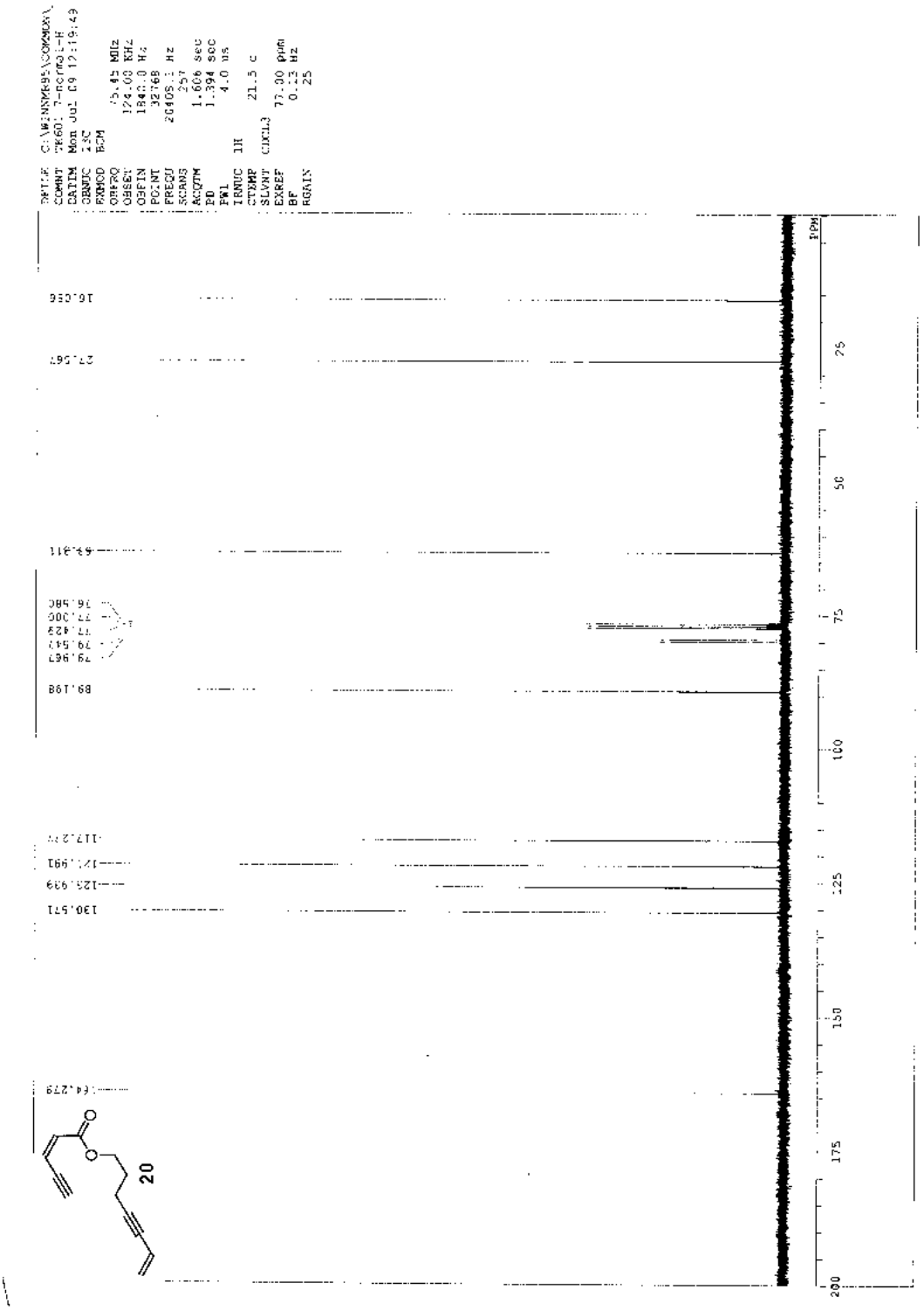
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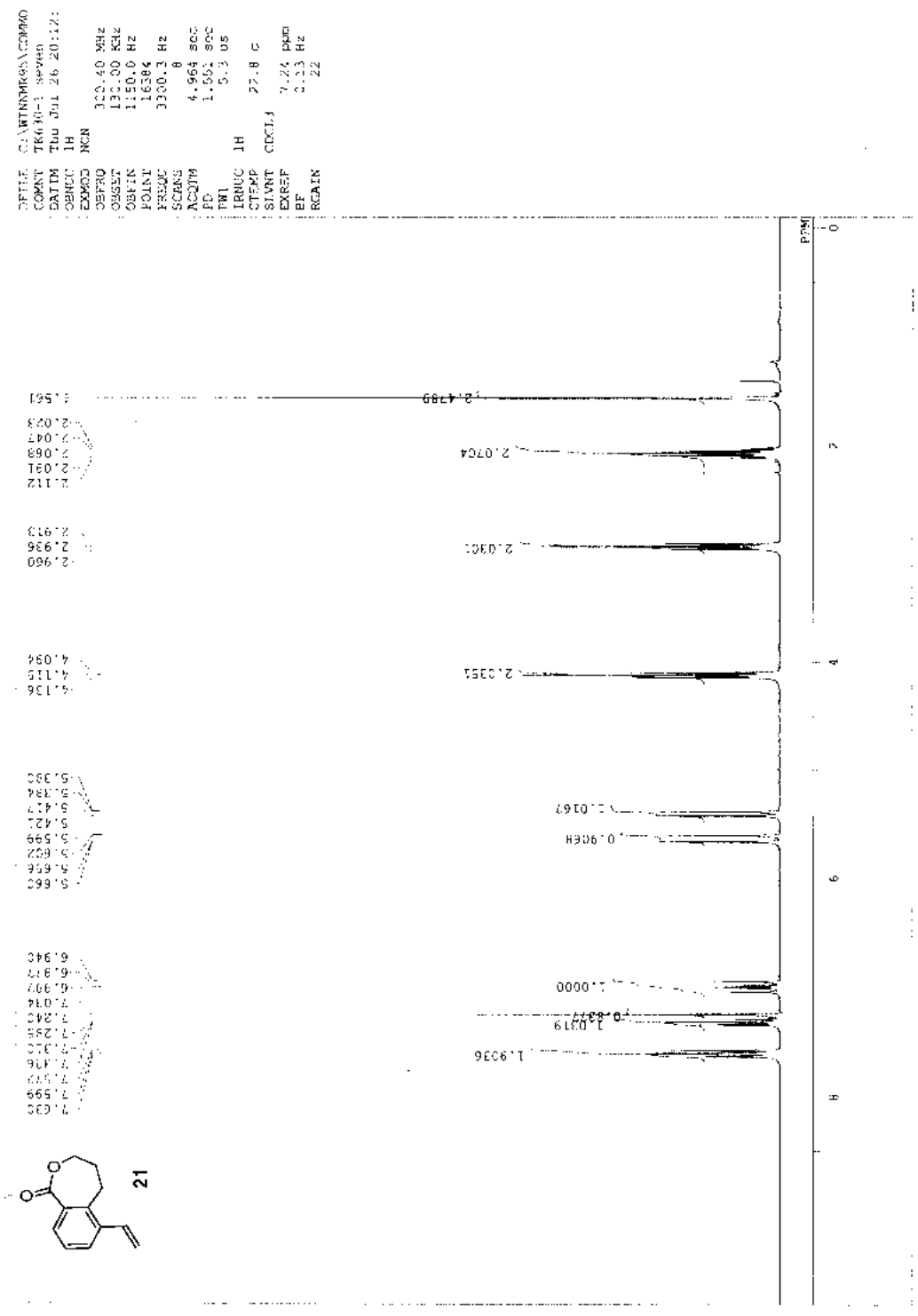
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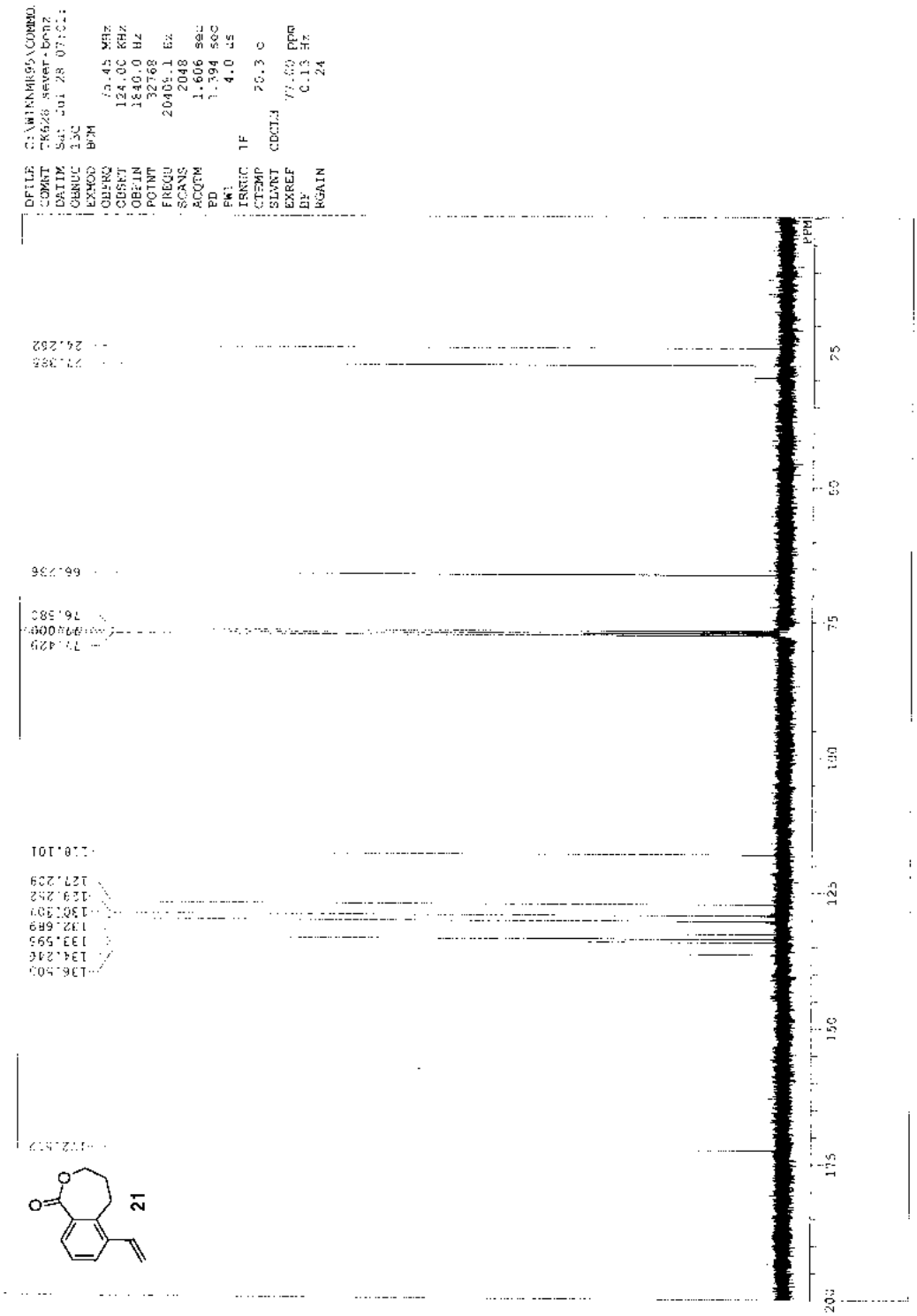
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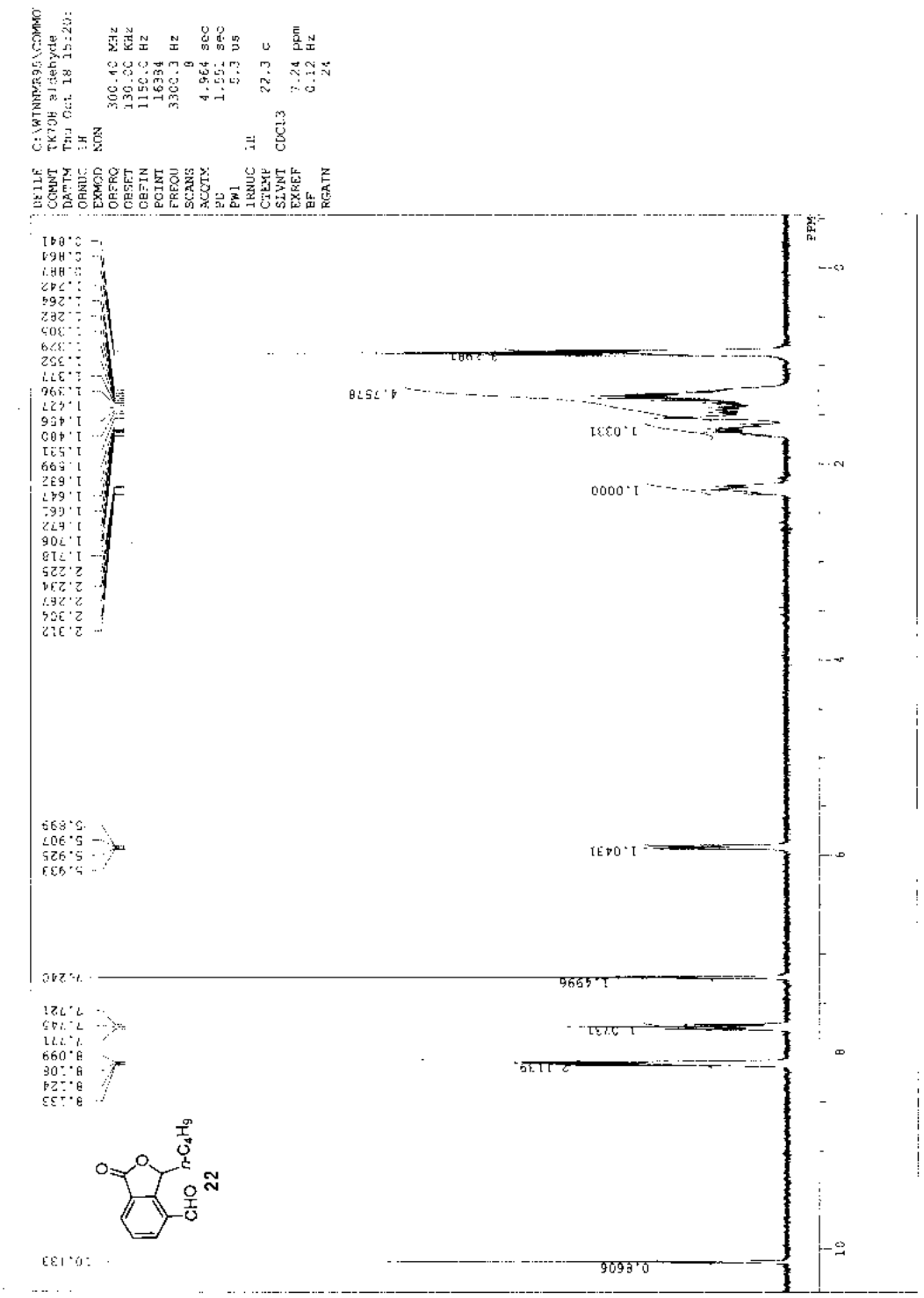
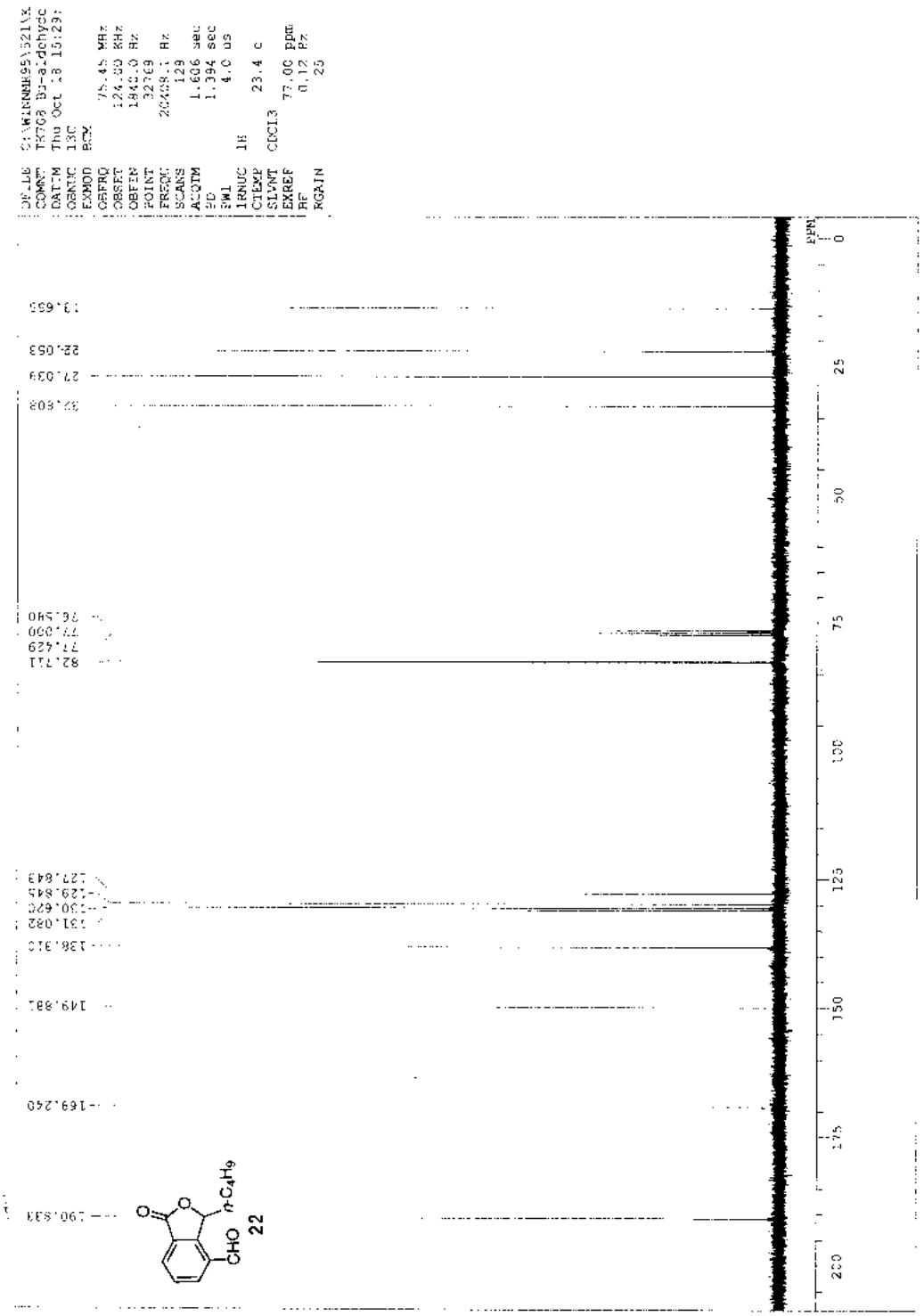

22
CHO
n-C4H9
O
PPM


22
CHO
n-C4H9
O
O
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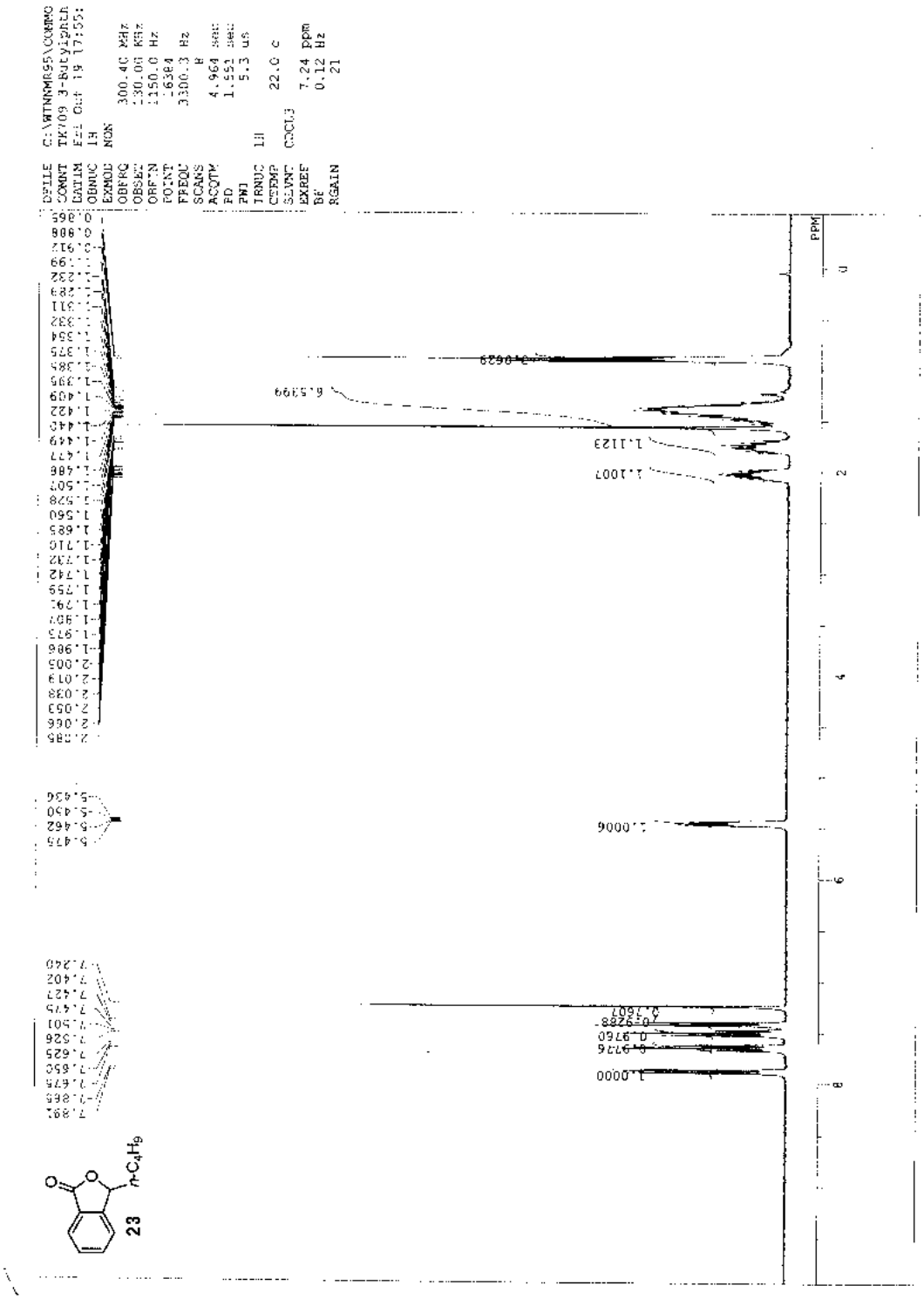
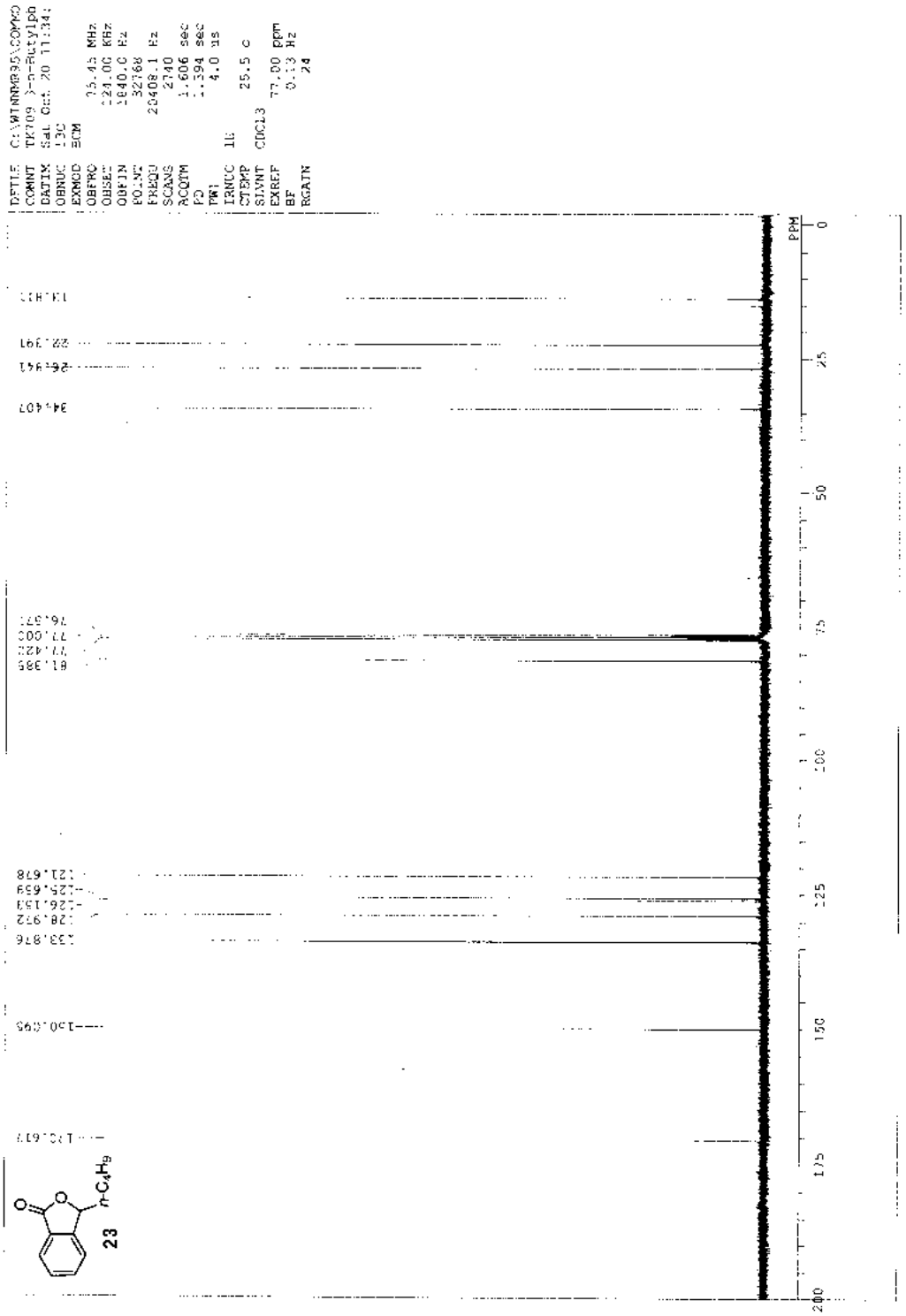
169.260
149.861
138.310
131.082
130.670
129.845
127.843
82.711
77.429
77.000
76.580
27.039
22.053
13.685
PPM
0
25
50
75
100
125
150
175
200


23
n-C4H9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DATIM Sat Oct 20 11:34:
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EXMOD EOM
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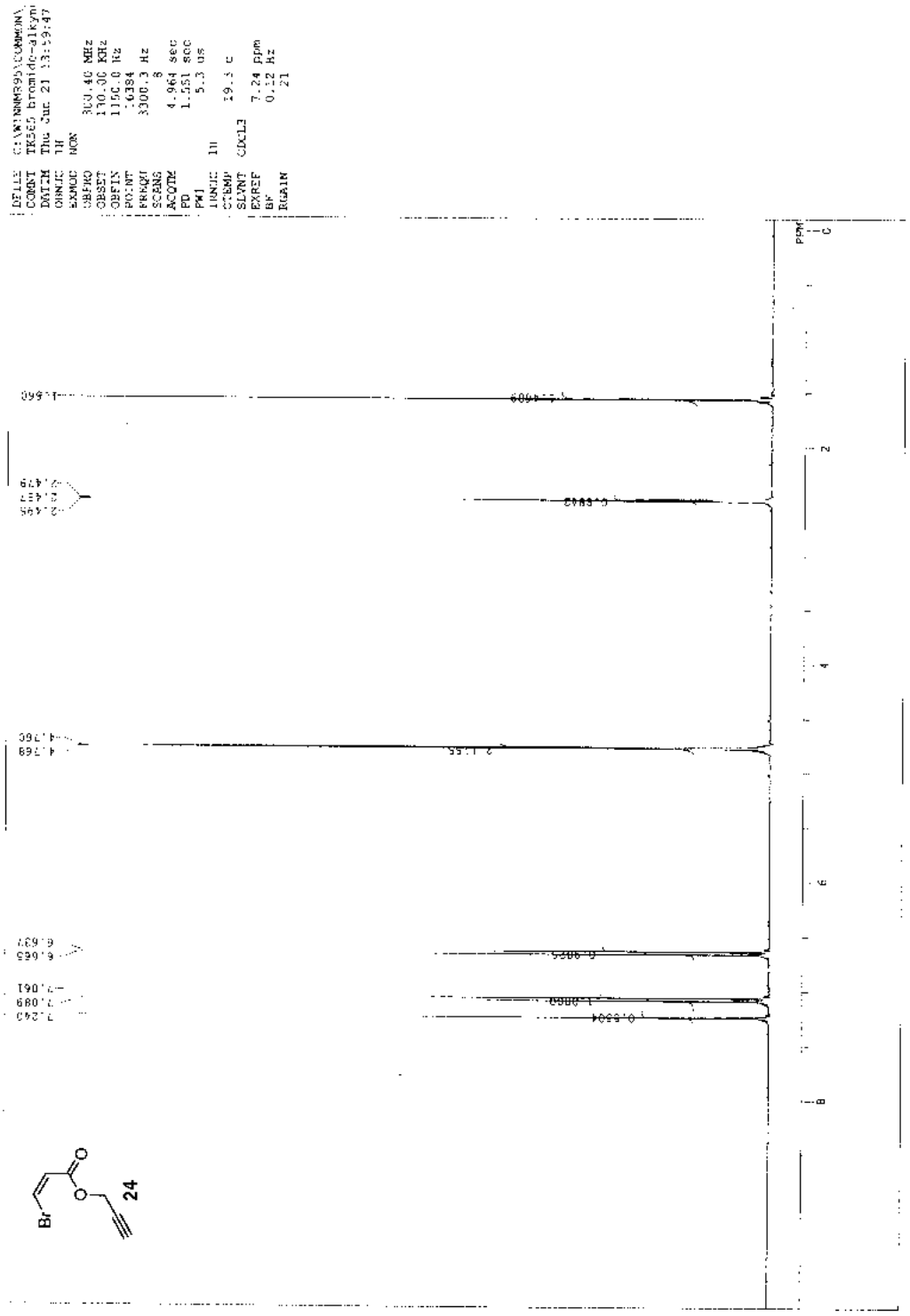
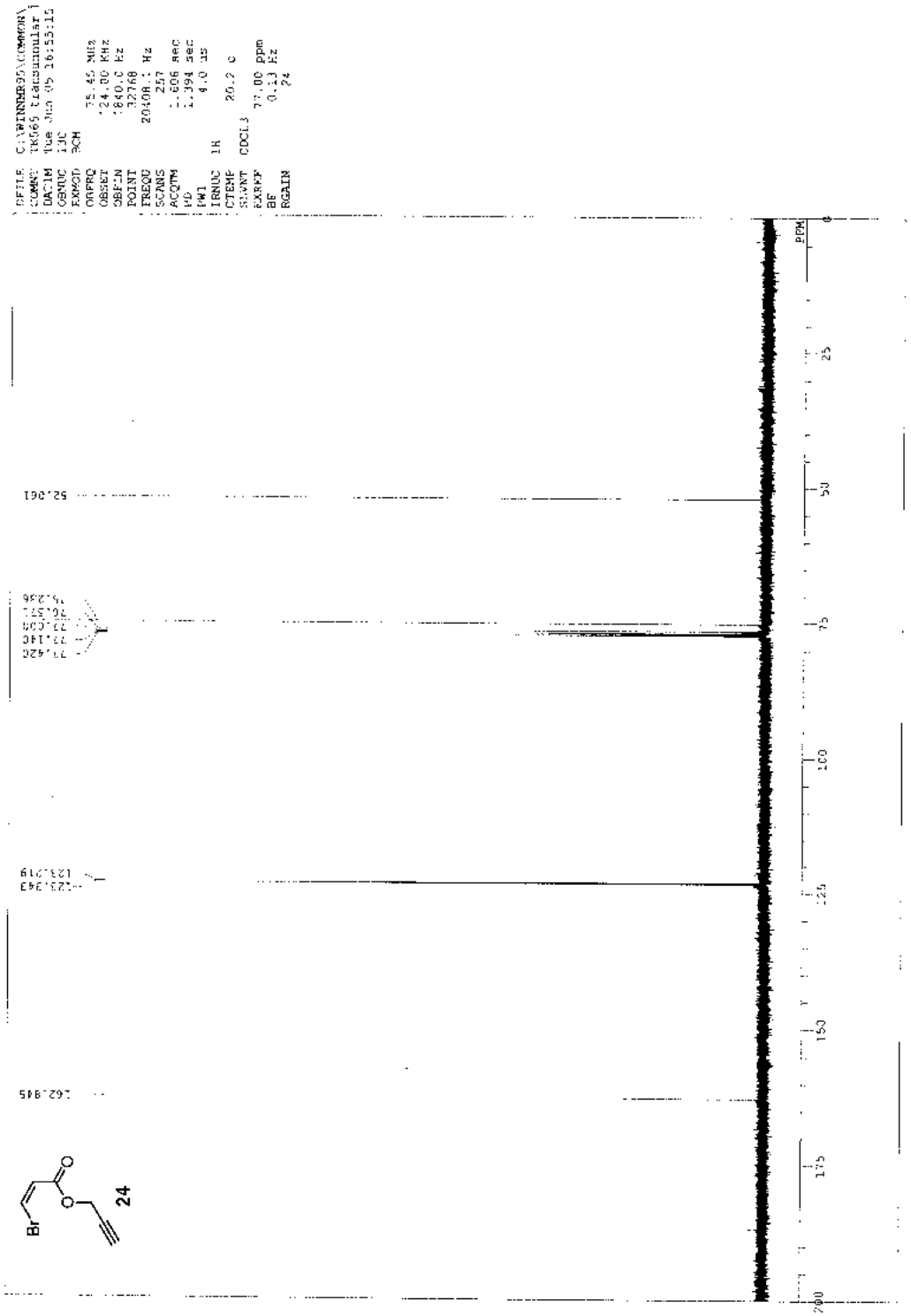
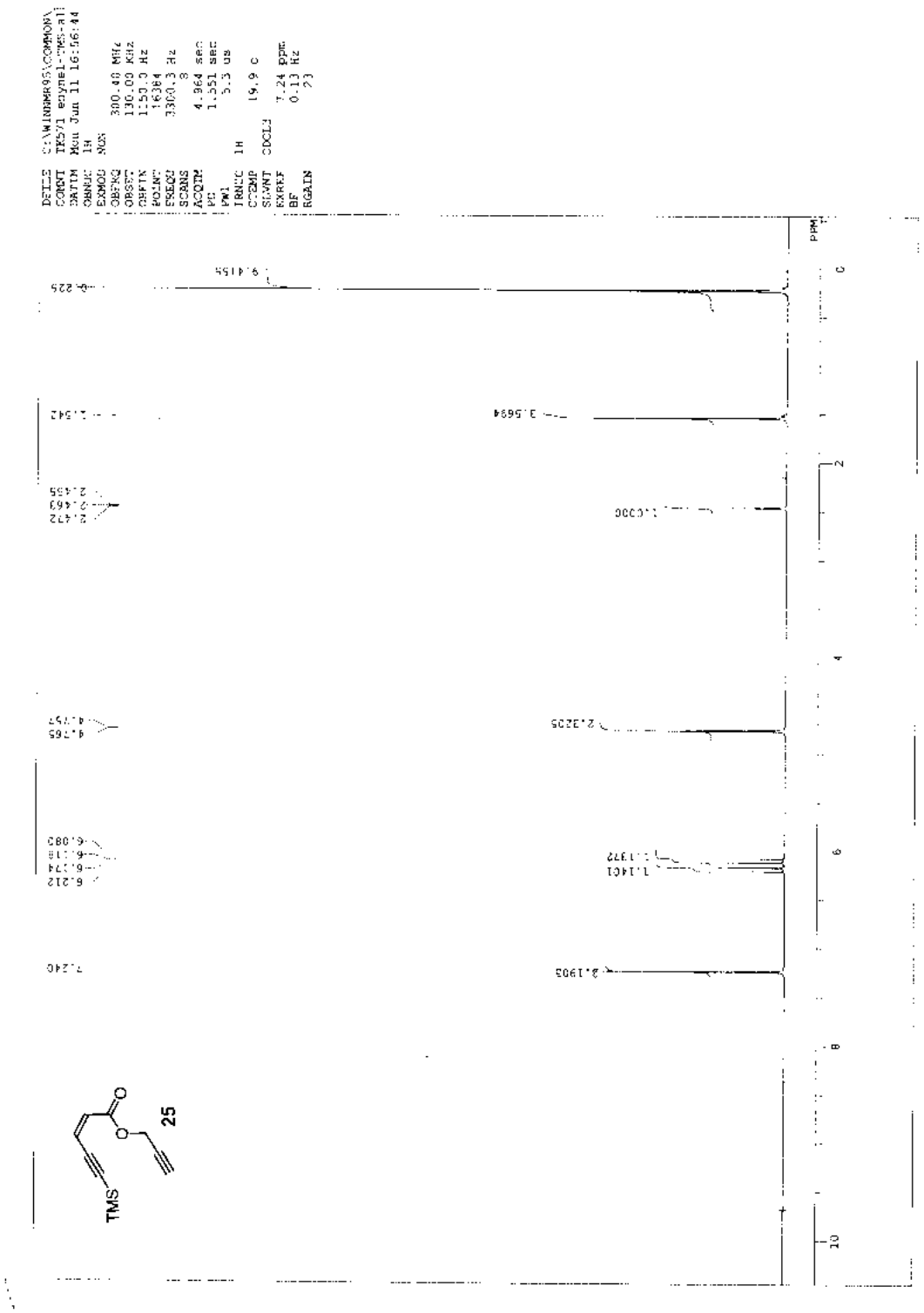
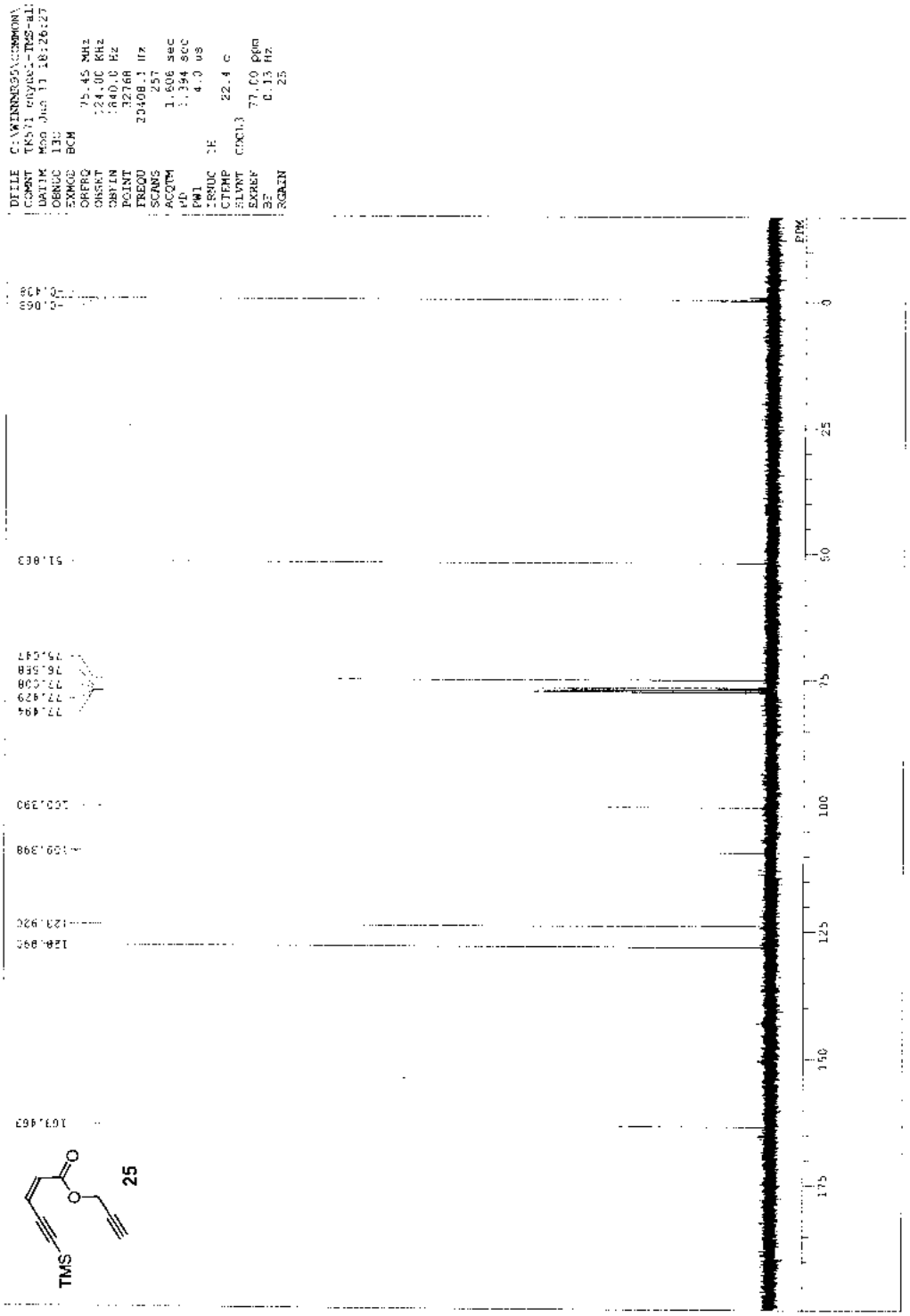
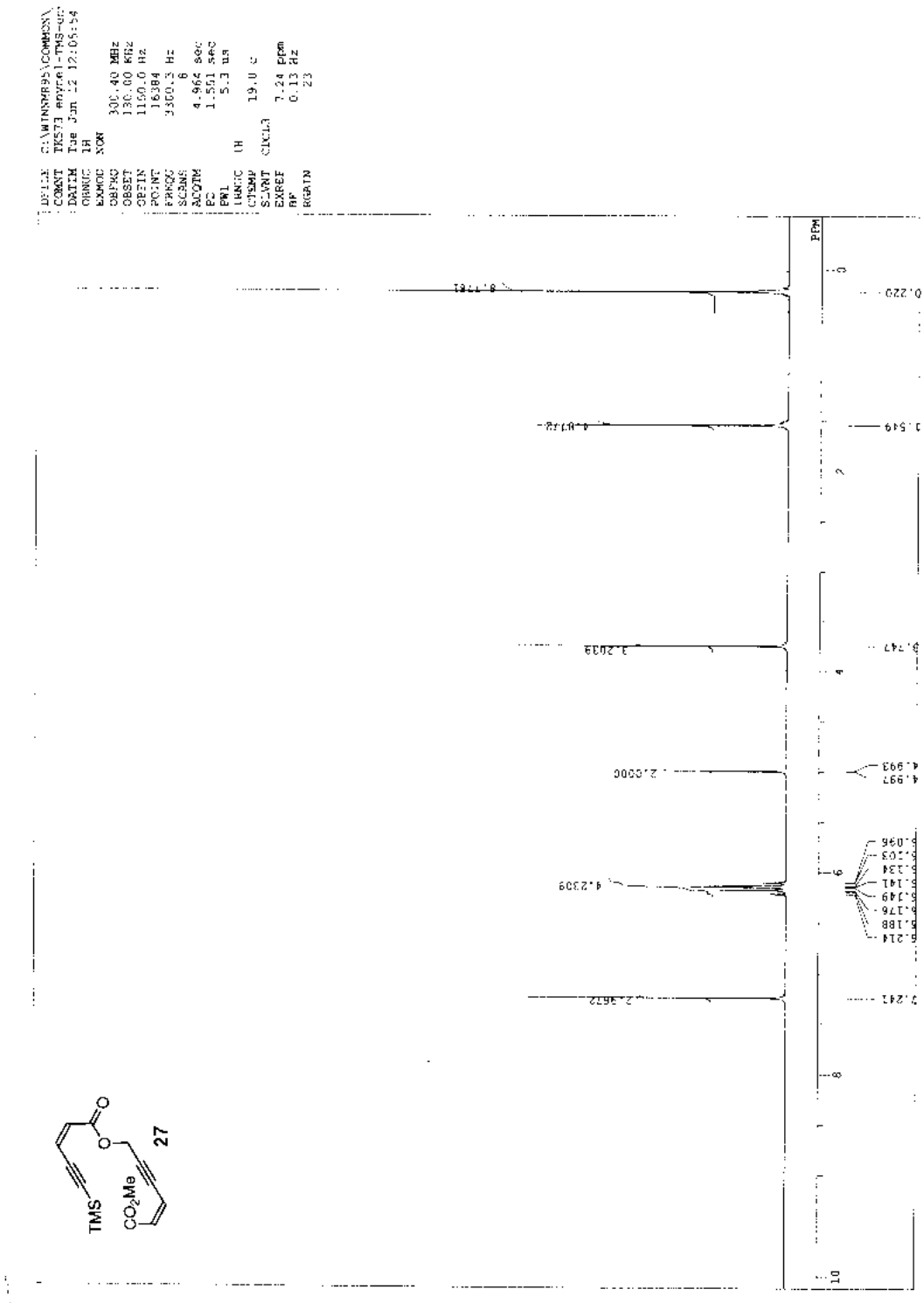
OBSET 124.00 KHz
ODFIN 1840.0 Hz
POINT 32768
FWIDTH 20408.1 Hz
SCANS 2740
ACQTM 1.606 sec
PD 1.394 sec
PW1 4.0 us
TRNUC 1H
CTEMP 25.5 c
SLVNT CDCl3
EXREF 77.00 ppm
BF 0.13 Hz
RGAIN 24
PPM
0
25
50
75
100
125
150
175
200
n-C4H9
23

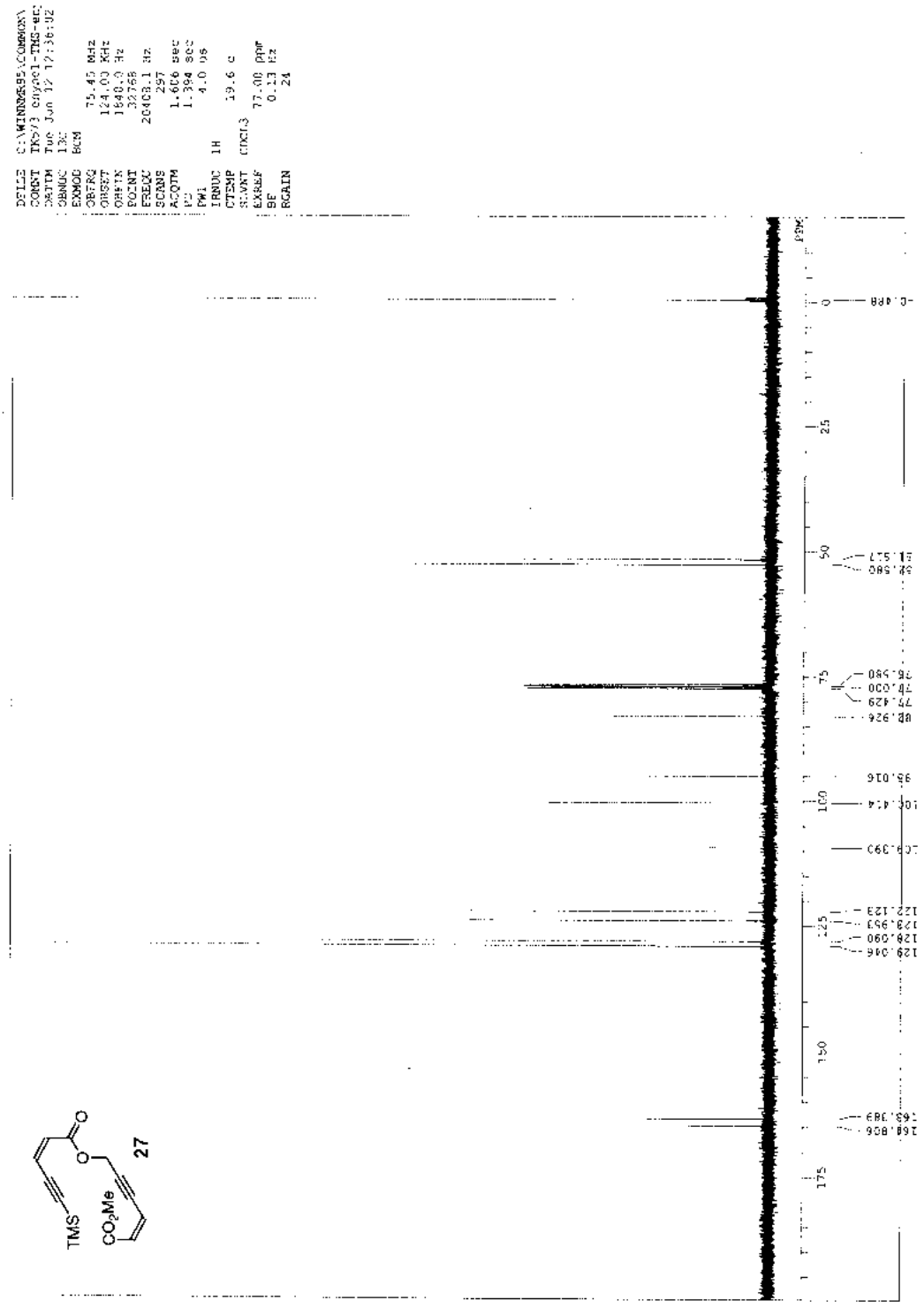

Br
O
O
24
PPM

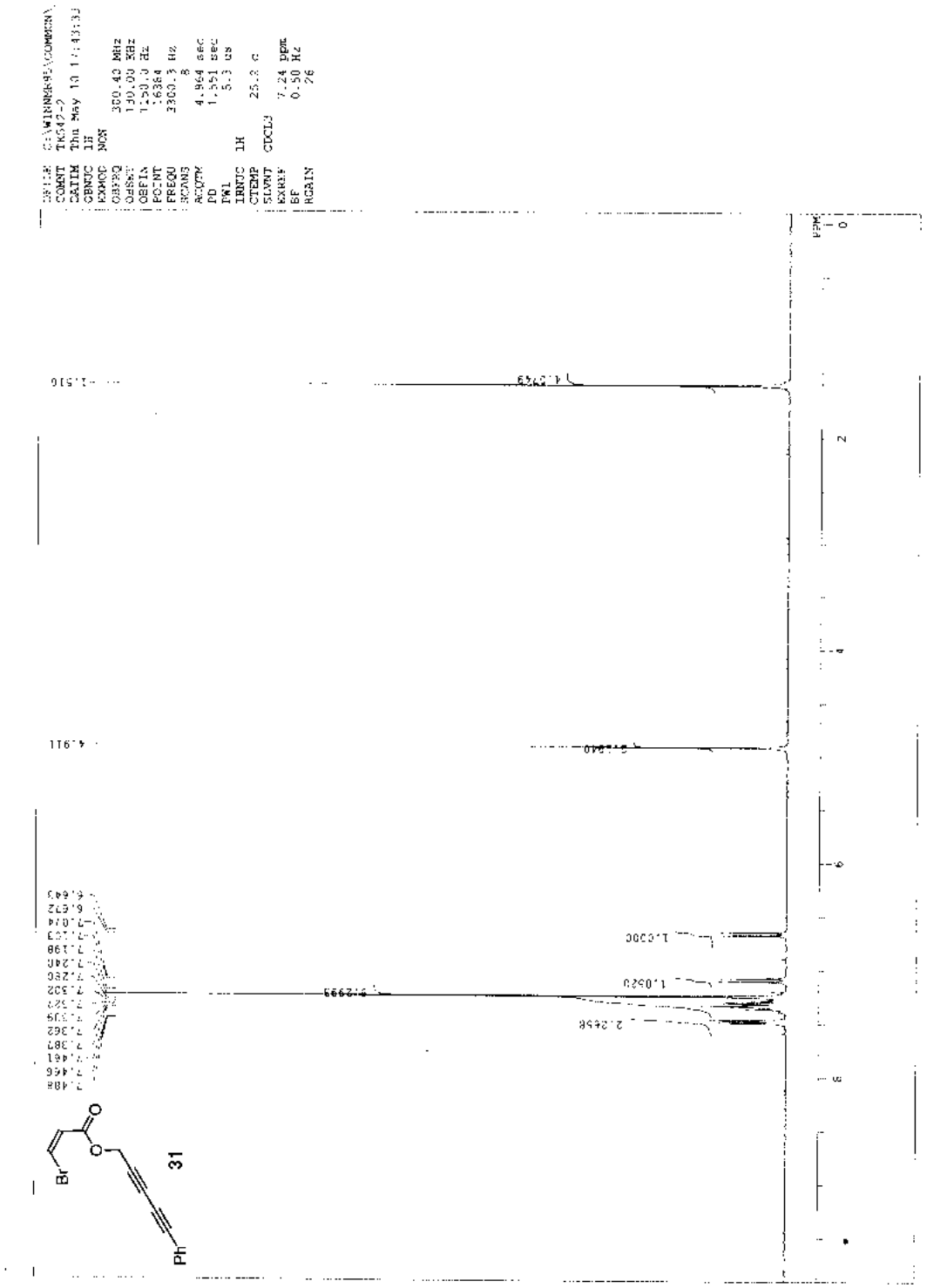

Br
24
PPM

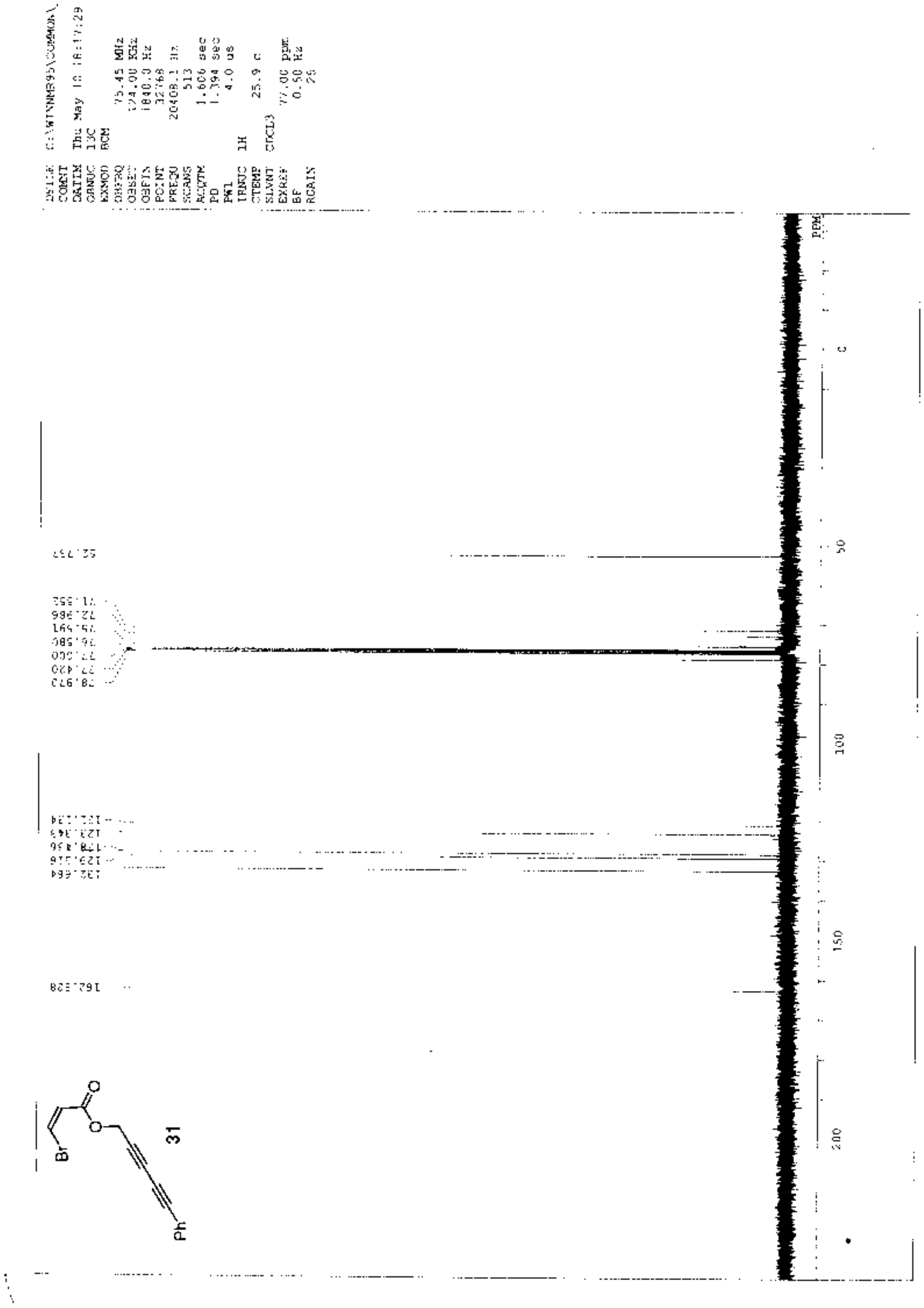

TMS
25


TMS
25
PPM


TMS
CO2Me
27


TMS
CO2Me
27


Br
O
O
Ph
31



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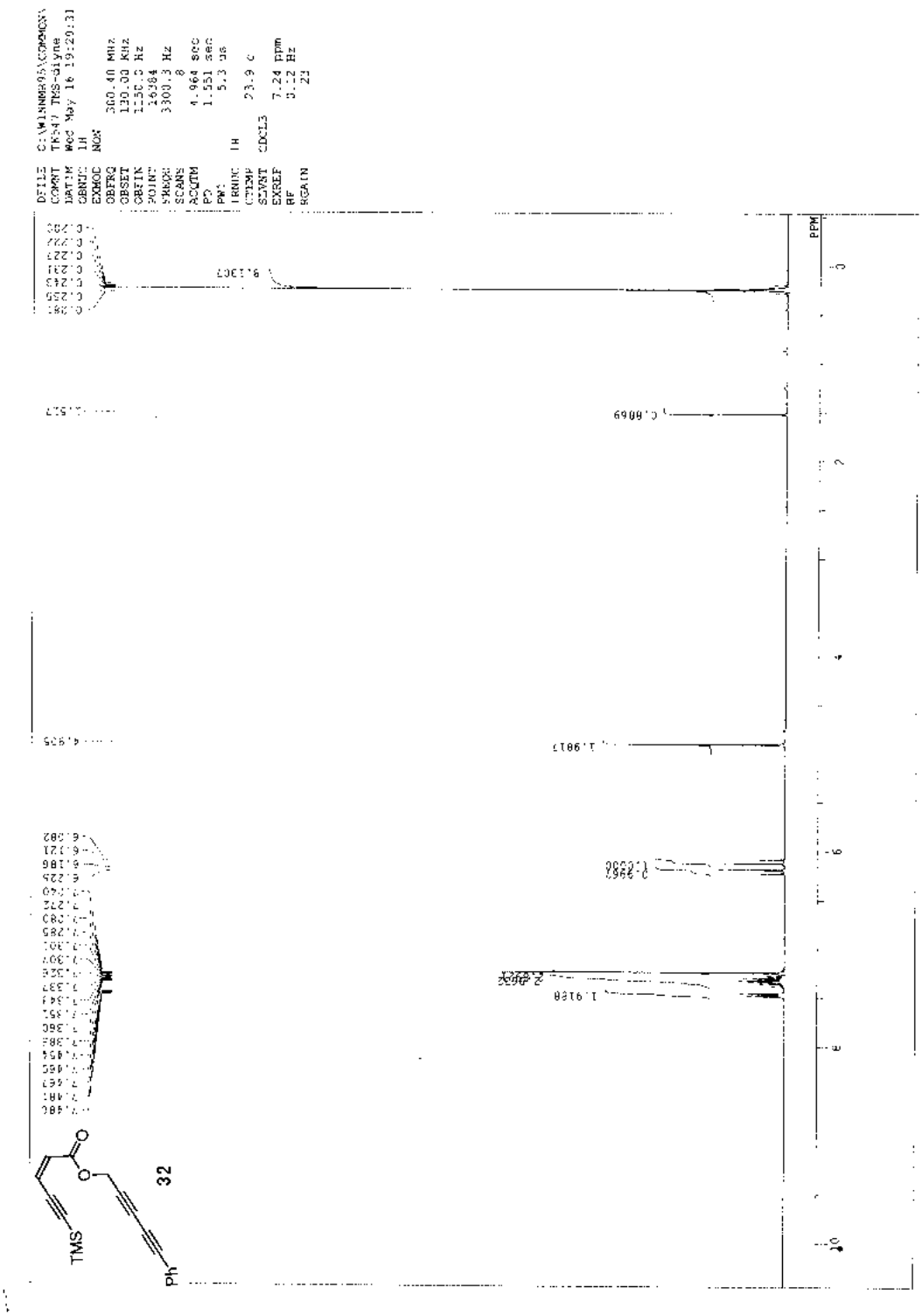
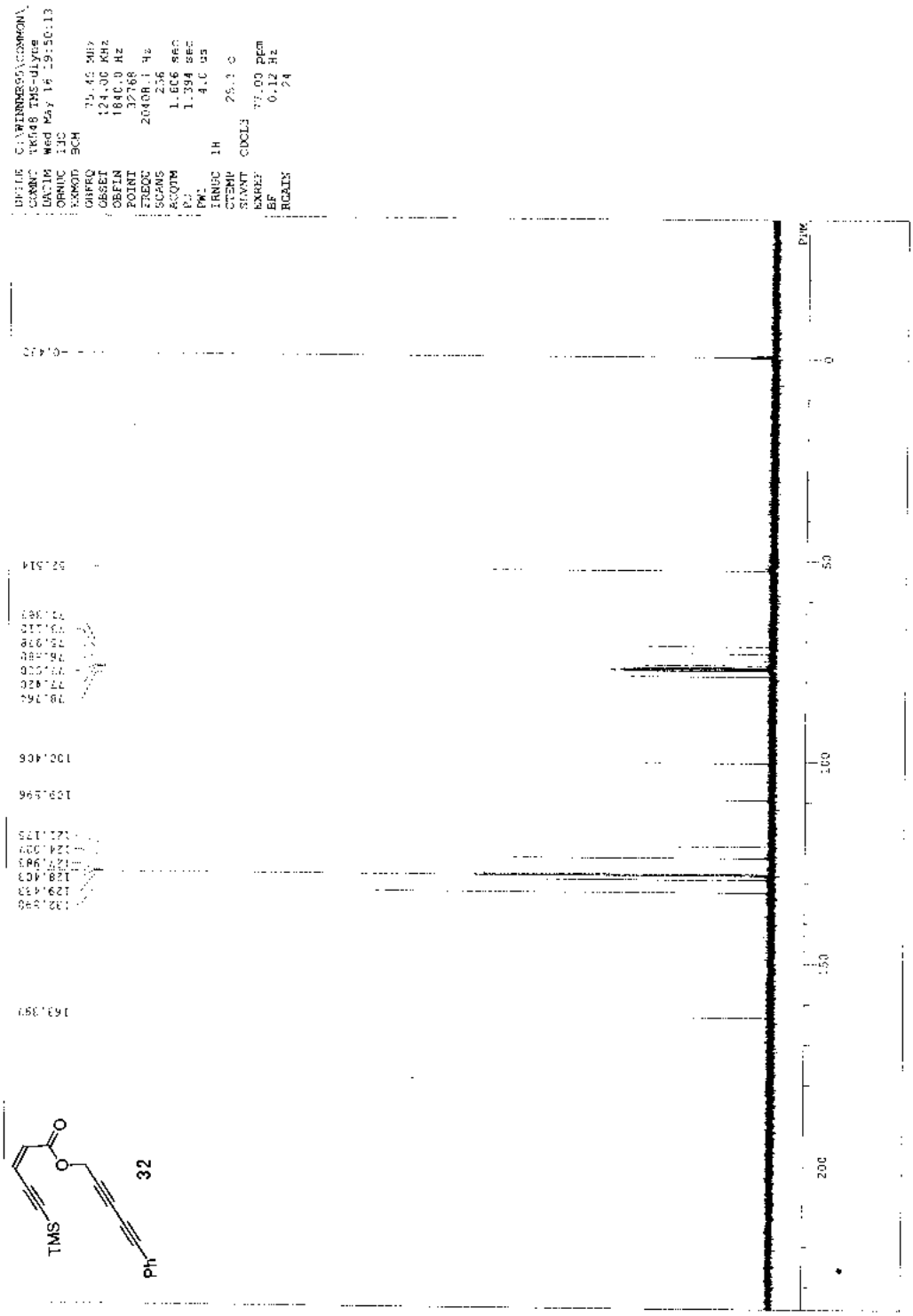
OBSET 124.00 KHz
OBFIN 18840.0 Hz
POINT 32768
FREQU 20408.1 Hz
SCANS 513
ACQTM 1.606 sec
PD 1.394 sec
PW1 4.0 us
IRNUC 1H
CTEMP 25.9 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.50 Hz
RGAIN 26
52.757
71.352
72.966
75.991
76.580
77.000
77.420
78.970
121.134
123.343
128.436
129.316
132.664
162.328
PPM
0
50
100
150
200
Br
O
Ph
31


32
TMS
Ph
PPM


TMS
Ph
32