Angewandte Chemie
Eine Zeitschrift der Gesellschaft Deutscher Chemiker

Supporting Information

69451 Weinheim, Germany

Supporting Information

Iron-Catalyzed C-C Bond Formation via Direct Functionalization of C-H Bonds Adjacent to Heteroatoms

Zhiping Li,* Rong Yu, and Haijun Li

Department of Chemistry, Renmin University of China, Beijing 100872, China

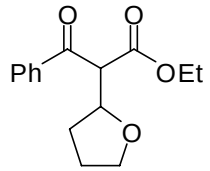
Fax: (+86)10-62516444
Email: zhipingli@ruc.edu.cn

Contents

1) Experimental details and characterization data for all compounds

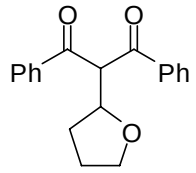
General information: ^{1}H NMR spectra were recorded on JEOL 300 MHz spectrometer and the chemical shifts were reported in parts per million (δ) relative to internal standard TMS (0 ppm) for $CDCl_3$. The peak patterns are indicated as follows: s, singlet; d, doublet; dd, doublet of doublet; ddd, doublet of doublet of doublet; t, triplet; m, multiplet; q, quartet. The coupling constants, *J*, are reported in Hertz (Hz). ^{13}C NMR spectra were obtained at JEOL 75.4 MHz and referenced to the internal solvent signals (central peak is 77.0 ppm in $CDCl_3$). $CDCl_3$ was used as the NMR solvent. Mass spectra were determined with AEI-MS 50 for EI-MS. APEX Ⅱ(Bruker Inc.) for HR-MS and ESI-MS. IR spectra were recorded by a Nicolet 5MX-S infrared spectrometer. Flash column chromatography was performed over silica gel 200-300. All reagents were weighed and handled in air at room temperature. Unless otherwise noted, all reactions were performed under a nitrogen atmosphere. All reagents were purchased from Acros, Aldrich, TCI, and Strem and used without further purification.

General procedure for products 3: To a mixture of THF **1a** (1 mL) and $Fe_2(CO)_9$ (9.2 mg, 0.025 mmol), ethyl benzoylacetate **2a** (0.25 mmol) was added under a nitrogen atmosphere at room temperature. *tert*-Butyl peroxide (0.139 mL, 0.75 mmol) was added dropwise into the mixture. The resulting mixture was stirred under reflux condition for 1 h or as noted in the text. The resulting reaction mixture was mixed with a small amount of silica gel and concentrated, then purified by flash column chromatography (ethyl acetate/petroleum ether = 1:20). The fraction with an R_f = 0.3 (ethyl acetate/petroleum ether = 1:6) was collected and to give the desired product **3a**.

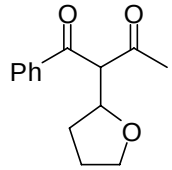


Ethyl 3-oxo-3-phenyl-2-(tetrahydrofuran-2-yl)propanoate (3a)[1]. Isolated by flash column chromatography (ethyl acetate/petroleum ether = 1:6, R_f = 0.3). The ratio of two diasteromers is 1:1. **Two diasteromers:** ^{1}H NMR (ppm) *δ* 8.05-8.01(m, 4H), 7.62-7.55(m, 2H), 7.50-7.44(m, 4H), 4.74-4.64(m, 2H), 4.46(d, *J* = 9.0Hz, 1H), 4.41(d, *J* = 9.0Hz, 1H), 4.17(q, *J* = 7.2Hz, 2×2H), 3.92-3.69(m, 4H), 2.28-2.15(m, 2H), 1.99-1.84(m, 4H), 1.57-1.45(m, 2H), 1.18(t, *J* = 6.9Hz, 3H), 1.17(t, *J* = 6.9Hz, 3H); ^{13}C NMR (ppm) *δ* 193.5, 193.2, 167.8, 167.4, 136.7, 136.2, 133.7, 133.3, 128.7,

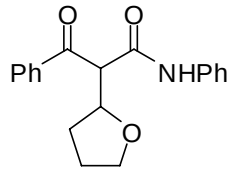
128.6, 128.5, 78.0, 77.6, 68.1, 68.0, 61.5, 61.3, 60.1, 59.2, 30.1, 29.9, 25.4, 25.3, 13.9, 13.8; MS(EI) *m/z*(%): 216, 193, 189, 157, 147, 105(100), 77, 71, 28.



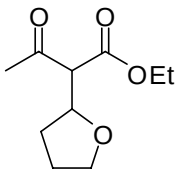
1,3-diphenyl-2-(tetrahydrofuran-2-yl)propane-1,3-dione (3b)[2]. Isolated by flash column chromatography (ethyl acetate/petroleum ether = 1:6, R_f = 0.4). ^{1}H NMR (ppm) δ 8.01-7.95(m, 4H), 7.58-7.38(m, 6H), 5.43(d, J = 8.4Hz, 1H), 4.83(ddd, J = 8.1, 6.9, 6.9Hz, 1H), 3.84-3.69(m, 2H), 2.35-2.26(m, 1H), 1.93-1.71(m, 2H), 1.69-1.65(m, 1H); ^{13}C NMR (ppm) δ 194.6, 194.3, 136.8, 136.4, 133.6, 133.3, 128.9, 128.8, 128.7, 79.3, 68.0, 62.7, 30.5, 25.4; MS(EI) *m/z*(%): 224, 189, 105(100), 77, 71, 51, 27.



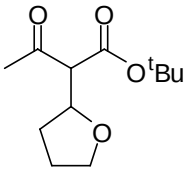
1-phenyl-2-(tetrahydrofuran-2-yl)butane-1,3-dione (3c)[3]. Isolated by flash column chromatography (ethyl acetate/petroleum ether = 1:6, R_{f1} = 0.3, R_{f2} = 0.25). The ratio of two diasteromers is 1:1. **Two diasteromers:** IR (neat): ν_{max} 1717, 1665, 1590, 1570, 1449, 1354, 1283, 1209, 1167, 1068, 1001, 968, 923, 806, 761, 690cm^{-1}; **One diasteromer:** ^{1}H NMR (ppm) δ 8.03-8.00(m, 2H), 7.63-7.57(m, 1H), 7.51-7.46(m, 2H), 4.80-4.72(m, 1H), 4.55(d, J = 9.3Hz, 1H), 3.92-3.73(m, 2H), 2.28(s, 3H), 2.22-2.12(m, 1H), 1.95-1.85(m, 2H), 1.50-1.38(m, 1H); ^{13}C NMR (ppm) δ 202.5, 194.2, 136.5, 133.9, 128.8, 128.7, 78.4, 69.4, 68.1, 30.5, 27.9, 25.2; **The other diasteromer:** ^{1}H NMR (ppm) δ 8.05-7.99(m, 2H), 7.62-7.54(m, 1H), 7.52-7.42(m, 2H), 4.73-4.65(m, 1H), 4.60(d, J = 8.7Hz, 1H), 3.85-3.67(m, 2H), 2.17(s, 3H), 2.00-1.89(m, 2H), 1.69-1.60(m, 2H); ^{13}C NMR (ppm) δ 202.0, 194.9, 137.0, 133.6, 128.8, 128.7, 78.1, 68.7, 67.9, 29.9, 29.2, 25.5.



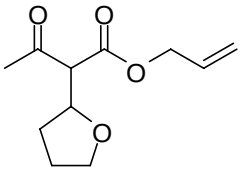
3-oxo-N,3-diphenyl-2-(tetrahydrofuran-2-yl)propanamide (3d). Isolated by flash column chromatography (ethyl acetate/petroleum ether = 1:2, R_{f1} = 0.4, R_{f2} = 0.35). The ratio of two diasteromers is 1:1. **Two diasteromers:** IR (neat): ν_{max} 1686, 1648, 1594, 1545, 1495, 1441, 1325, 1213, 1180, 1064, 1001, 902, 757, 690cm^{-1}; **One diasteromer:** ^{1}H NMR (ppm) δ 8.81(s, 1H), 8.06-8.02(m, 2H), 7.63-7.46(m, 5H), 7.34-7.25(m, 2H), 7.13-7.08(m, 1H), 4.61-4.51(m, 2H), 3.92-3.71(m, 2H), 2.20-2.11(m, 1H), 2.04-1.88(m, 2H), 1.76-1.68(m, 1H); ^{13}C NMR (ppm) δ 199.1, 164.3, 137.5, 137.1, 134.0, 129.0, 128.9, 128.8, 128.7, 124.5, 119.8, 80.7, 68.4, 60.9, 29.4, 25.4; **The other diasteromer:** ^{1}H NMR (ppm) δ 8.93(s, 1H), 8.04(d, J = 8.1Hz, 2H), 7.63-7.46(m, 5H), 7.32-7.26(m, 2H), 7.11-7.06(m, 1H), 4.61-4.53(m, 2H), 3.97-3.82(m, 2H), 2.19-2.09(m, 1H), 1.97-1.86(m, 2H), 1.75-1.64(m, 1H); ^{13}C NMR (ppm) δ 197.0, 165.5, 137.7, 136.6, 133.9, 128.9, 128.8, 124.3, 119.9, 78.7, 68.7, 60.4, 30.3, 25.4; MS (EI) *m/z* (%): 309(M^+), 291, 239, 204, 147, 120, 119, 105(100), 93, 77, 71, 66, 51; HRMS calcd for $C_{19}H_{19}NO_3$: 309.1365; found: 309.1367.



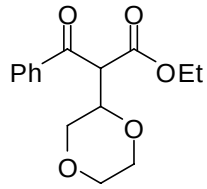
Ethyl 3-oxo-2-(tetrahydrofuran-2-yl)butanoate (3e)[4]. Isolated by flash column chromatography (ethyl acetate/petroleum ether = 1:6, R_f = 0.3). The ratio of two diasteromers is 2:1. **Two diasteromers:** ^{1}H NMR (ppm) δ 4.48-4.40(m, 3H), 4.28-4.15(m, 6H), 3.89-3.71(m, 6H), 3.58(d, J = 8.7Hz, 1H), 3.51(d, J = 9.3Hz, 2H), 2.31(s, 6H), 2.25(s, 3H), 2.21-2.10(m, 3H), 1.96-1.83(m, 9H), 1.32-1.24(m, 9H); ^{13}C NMR (ppm) δ 201.9, 201.3, 167.8, 167.4, 76.9, 76.6, 68.0, 67.9, 65.2, 64.9, 61.4, 61.2, 30.2, 29.8, 29.7, 29.6, 25.4, 25.2, 25.1, 13.9; MS (EI) *m/z* (%): 200(M^+), 199, 157, 127, 111, 85, 71(100), 43, 29.



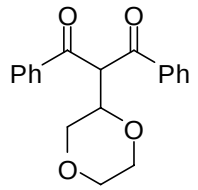
Tert-butyl 3-oxo-2-(tetrahydrofuran-2-yl)butanoate (3f). Isolated by flash column chromatography (ethyl acetate/petroleum ether = 1:6, R_f = 0.4). The ratio of two diasteromers is 2:1. **Two diasteromers:** IR (neat): ν_{max} 1740, 1711, 1362, 1254, 1147, 1064, 844cm^{-1}; ^{1}H NMR (ppm) δ 4.44-4.35(m, 3H), 3.87-3.71(m, 6H), 3.48(d, J = 8.7Hz, 1H), 3.41(d, J = 9.6Hz, 2H), 2.29(s, 6H), 2.24(s, 3H), 2.20-2.11(m, 6H), 1.95-1.86(m, 6H), 1.48(s, 9H), 1.45(s, 18H); ^{13}C NMR (ppm) δ 202.3, 201.9, 167.0, 166.6, 82.1, 82.0, 79.9, 77.4, 76.6, 68.1, 67.9, 66.2, 66.1, 30.2, 29.7, 29.4, 27.9, 27.8, 25.5, 25.2; MS (EI) *m/z* (%):182, 172, 129, 127, 113, 111, 97, 85, 71(100), 57, 43, 41; MS(ESI) *m/z*(%): 228.1(M^+); HRMS calcd for $C_{12}H_{20}O_4$: 228.1362; found: 228.1361.



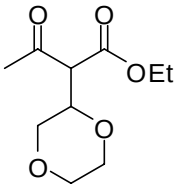
Allyl 3-oxo-2-(tetrahydrofuran-2-yl)butanoate (3g). Isolated by flash column chromatography (ethyl acetate/petroleum ether = 1:6, R_f = 0.2). The ratio of two diasteromers is 1:1. **Two diasteromers:** IR (neat): ν_{max} 1744, 1715, 1648, 1354, 1267, 1151, 1064, 985, 918cm^{-1}; ^{1}H NMR (ppm) δ 5.98-5.83(m, 2H), 5.38-5.24(m, 4H), 4.67(d, J = 5.7Hz, 2H), 4.62(d, J = 5.7Hz, 2H), 4.49-4.41(m, 2H), 3.88-3.71(m, 4H), 3.62(d, J = 8.4Hz, 1H), 3.55(d, J = 9.6Hz, 1H), 2.31(s, 3H), 2.25(s, 3H), 2.24-2.12(m, 2H), 1.96-1.84(m, 4H), 1.68-1.53(m, 2H); ^{13}C NMR (ppm) δ 201.8, 201.2, 167.5, 167.1, 131.4, 131.2, 118.9, 118.7, 76.9, 76.6, 68.1, 67.9, 65.9, 65.8, 65.1, 64.8, 30.3, 29.9, 29.8, 29.7, 25.4, 25.2; MS (EI) *m/z* (%): 212(M^+), 211, 181, 169, 153, 149, 127, 111, 71(100), 43, 41; HRMS calcd for $C_{11}H_{16}O_4$: 212.1049; found: 212.1023.



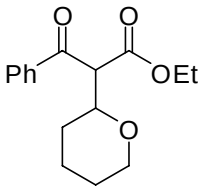
Ethyl 2-(1,4-dioxan-2-yl)-3-oxo-3-phenylpropanoate (3h). Isolated by flash column chromatography (ethyl acetate/petroleum ether = 1:6, R_f = 0.3). The ratio of two diasteromers is 1:1. **Two diasteromers:** IR (neat): ν_{max} 1735, 1682, 1590, 1445, 1271, 1205, 1113, 993, 881cm^{-1}; ^{1}H NMR (ppm) δ 7.96-7.91 (m, 4H), 7.55-7.48 (m, 2H), 7.43-7.38 (m, 4H), 4.46-4.36 (m, 4H), 4.15-4.01 (m, 4H), 3.85-3.25 (m, 12H), 1.09 (t, J = 7.2Hz, 3H), 1.08 (t, J = 7.2Hz, 3H) ; ^{13}C NMR (ppm) δ 192.8; 191.8, 167.2, 166.5, 136.7, 135.9, 133.9, 133.5, 128.8, 128.7, 128.6, 74.0, 73.9, 69.4, 69.2, 66.8, 66.7, 66.4, 66.3, 61.7, 61.6, 56.7, 55.7, 13.8, 13.7; MS (EI) *m/z* (%): 278(M^+), 276, 235, 232, 205, 192, 173, 149, 147, 105(100), 86, 77, 73, 51; HRMS calcd for $C_{15}H_{18}O_5$: 278.1154; found: 278.1153.



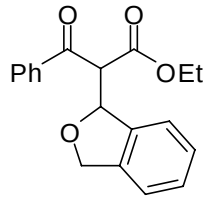
2-(1,4-dioxan-2-yl)-1,3-diphenylpropane-1,3-dione (3i). Isolated by flash column chromatography (ethyl acetate/petroleum ether = 1:6, R_f = 0.2). IR (neat): ν_{max} 1694, 1669, 1590, 1441, 1279, 1205, 1118, 985, 910cm^{-1}; ^{1}H NMR (ppm) δ 8.00-7.91(m, 4H), 7.59-7.38(m, 6H), 5.42 (d, J = 8.7 Hz, 1H), 4.70(ddd, J = 9.3, 9.3, 2.4Hz, 1H), 4.00(dd, J = 11.4, 2.4Hz, 1H), 3.79-3.57(m, 4H), 3.41(dd, J = 11.1, 9.6Hz, 1H); ^{13}C NMR (ppm) δ 193.8, 192.8, 136.9, 136.2, 133.8, 133.5, 128.8, 128.7, 75.6, 69.9, 67.1, 66.5, 59.4; MS (EI) *m/z* (%): 310(M^+), 308, 224, 205, 171, 105(100), 86, 77, 51; MS(ESI) *m/z*(%):310.1(M^+); HRMS calcd for $C_{19}H_{18}O_4$: 310.1205; found: 310.1195.



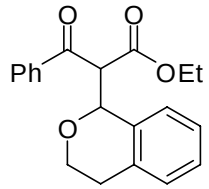
Ethyl 2-(1,4-dioxan-2-yl)-3-oxobutanoate (3j). Isolated by flash column chromatography (ethyl acetate/petroleum ether = 1:6, R_f = 0.2). The ratio of two diasteromers is 2:1. **Two diasteromers:** IR (neat): ν_{max} 1744, 1715, 1441, 1358, 1275, 1180, 1113, 1080, 1026, 885cm^{-1}; ^{1}H NMR (ppm) δ 4.33-4.15(m, 9H), 3.94-3.86(m, 3H), 3.79-3.54(m, 15H), 3.39(d, J = 11.1Hz, 2H), 3.36(d, J = 11.4Hz, 1H), 2.29(s, 6H), 2.25(s, 3H), 1.32-1.24(m, 9H); ^{13}C NMR (ppm) δ 201.0, 200.2, 167.1, 166.5, 73.6, 73.0, 69.3, 69.0, 66.7, 66.6, 66.4, 66.3, 61.9, 61.7, 61.6, 61.1, 29.9, 29.8, 14.0, 13.9; MS (EI) *m/z* (%): 216(M^+), 214, 197, 174, 173, 145, 130, 116, 101, 87, 86(100), 85, 73, 43; HRMS calcd for $C_{10}H_{16}O_5$: 216.0998; found: 216.0997.



Ethyl 3-oxo-3-phenyl-2-(tetrahydro-2H-pyran-2-yl)propanoate (3k)[1]. Isolated by flash column chromatography (ethyl acetate/petroleum ether = 1:6, R_{f1} = 0.4, R_{f2} = 0.3). The ratio of two diasteromers is 1:1. **One diasteromer:** ^{1}H NMR (ppm) δ 8.03-8.00(m, 2H), 7.59-7.54(m, 1H), 7.50-7.43(m, 2H), 4.47(d, J = 9.3Hz, 1H); 4.25-4.10(m, 3H), 3.86-3.82(m, 1H), 3.43(ddd, J = 11.4, 11.4, 3.0Hz, 1H), 1.87-1.79(m, 2H), 1.60-1.44(m, 4H), 1.18(t, J = 7.2Hz, 3H); ^{13}C NMR (ppm) δ 193.7, 167.1, 137.1, 133.3, 128.7, 128.5, 76.6, 68.7, 61.4, 59.8, 29.7, 25.7, 23.1, 14.0; **The other diasteromer:** ^{1}H NMR (ppm) δ 8.06-8.03(m, 2H), 7.62-7.57(m, 1H), 7.51-7.44(m, 2H), 4.46(d, J = 9.6Hz, 1H); 4.25-4.10(m, 3H), 4.03-3.99(m, 1H), 3.51(ddd, J = 11.1, 11.1, 3.0Hz, 1H), 1.81-1.50(m, 6H), 1.18(t, J = 6.9Hz, 3H); ^{13}C NMR (ppm) δ 192.6, 167.8, 136.5, 133.7, 128.7, 128.6, 77.1, 68.9, 61.5, 60.7, 29.8, 25.7, 23.1, 14.0; MS (EI) *m/z* (%): 230, 204, 203, 192, 171, 147, 125, 105(100), 77, 69, 57, 41, 29.

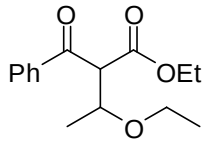


Ethyl 2-(1,3-dihydroisobenzofuran-1-yl)-3-oxo-3-phenylpropanoate (3l). Isolated by flash column chromatography (ethyl acetate/petroleum ether = 1:6, R_f = 0.3). The ratio of two diasteromers is 1:1. **Two diasteromers:** IR (neat): ν_{max} 1735, 1682, 1590, 1445, 1279, 1196, 1035, 1018, 748cm^{-1}; ^{1}H NMR (ppm) δ 8.03-7.97(m, 4H), 7.59-7.12(m, 14H), 6.18(d, J = 9.3Hz, 1H), 6.15(d, J = 8.4Hz, 1H), 5.15-4.98(m, 4H), 4.73(d, J = 8.1Hz, 1H), 4.67(d, J = 9.0Hz, 1H)，4.20(q, J = 7.2Hz, 2H), 4.14(q, J = 7.2Hz, 2H), 1.19(t, J = 7.2Hz, 3H), 1.13(t, J = 7.2Hz, 3H); ^{13}C NMR (ppm) δ 193.1, 193.0, 167.1, 167.0, 139.5, 139.4, 139.3, 139.2, 136.4, 133.8, 133.5, 128.9, 128.8, 128.7, 128.6, 128.2, 128.1, 127.5, 127.4, 122.7, 122.5, 121.1, 121.0, 82.8, 82.7, 72.6, 72.5, 61.7, 61.5, 61.2, 60.0, 13.9, 13.9; MS (EI) *m/z* (%): 310(M^+), 237, 205, 171, 149, 119, 118(100), 105, 90, 89, 77, 63, 51; HRMS calcd for $C_{19}H_{18}O_4$: 310.1205; found: 310.1207.

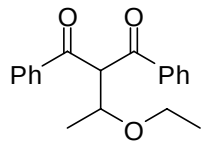


Ethyl 2-(isochroman-1-yl)-3-oxo-3-phenylpropanoate (3m). Isolated by flash column chromatography (ethyl acetate/petroleum ether = 1:6, R_f = 0.3). The ratio of two diasteromers is 1:1. **Two diasteromers:** IR (neat): ν_{max} 1740, 1682, 1590, 1491, 1445, 1362, 1275, 1205, 1147, 1088, 1001, 748, 686cm^{-1}; ^{1}H NMR (ppm) δ 8.00-7.90(m, 4H), 7.58-7.49(m, 2H), 7.46-7.38(m, 4H); 7.18-7.01(m, 8H), 5.76(d, J = 7.8Hz, 1H), 5.75(d, J = 7.8Hz, 1H), 4.93(d, J = 7.8Hz, 1H), 4.88(d, J = 6.9Hz, 1H), 4.20-4.08(m, 4H), 3.72-3.61(m, 2H), 3.56-3.45(m, 2H), 3.01-2.54(m, 4H), 1.15(t, J = 7.2Hz, 3H), 1.13(t, J=7.2Hz, 3H); ^{13}C NMR (ppm) δ 193.2, 193.0, 168.0, 167.0, 137.2, 136.4, 135.8, 135.6, 134.3, 133.9, 133.5, 133.1, 128.9, 128.6, 128.5, 128.4, 127.0, 126.9, 126.2, 125.1, 124.9, 74.5, 73.8, 63.3, 63.2, 61.6, 61.4, 61.3, 60.3, 28.7, 28.5, 13.8, 13.8; MS (EI) *m/z* (%):

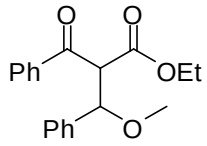
324(M^+), 279, 251, 219, 173, 149, 133, 105(100), 77, 51; HRMS calcd for $C_{20}H_{20}O_4$: 324.1362; found: 324.1360.



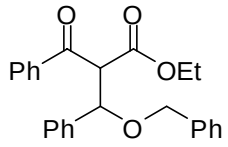
Ethyl 2-benzoyl-3-ethoxybutanoate (3n). Isolated by flash column chromatography (ethyl acetate/petroleum ether = 1:6, R_f = 0.4). The ratio of two diasteromers is 2:1. **Two diasteromers:** IR (neat): ν_{max} 2980, 1735, 1677, 1603, 1445, 1362, 1217, 1093, cm^{-1}; ^1H NMR (ppm) δ 8.07-7.88(m, 6H), 7.61-7.54(m, 3H), 7.50-7.41(m, 6H), 4.49(d, J = 9.6Hz, 1H), 4.48(d, J = 9.3Hz, 2H), 4.31(qd, J = 9.1, 6.0Hz, 2H), 4.28(dq, J = 9.1, 6.0Hz, 1H), 4.21-4.11(m, 6H), 3.73-3.18(m, 6H), 1.31(d, J = 6.0Hz, 6H), 1.26-1.10(m, 12H), 1.06(t, J = 7.2Hz, 3H), 0.98(t, J = 7.2Hz, 6H); ^{13}C NMR (ppm) δ 194.1, 167.5, 143.4, 137.3, 133.7, 133.6, 133.5, 133.2, 132.7, 129.0, 128.9, 128.7, 128.6, 128.5, 128.4, 128.3, 128.1, 128.0, 74.6, 74.5, 64.9, 64.7, 63.7, 61.6, 61.4, 61.3, 61.1, 61.0, 60.6, 55.5, 45.9, 39.3, 18.2, 18.1, 16.4, 15.4, 15.3, 15.2, 15.1, 15.0, 14.0, 13.9, 13.8, 13.7, 13.1; MS (EI) *m/z* (%): 264(M^+), 220, 191, 173, 159, 144, 115, 105(100), 100, 77, 73, 45. HRMS calcd for $C_{15}H_{20}O_4$: 264.1362; found: 264.1361.



2-(1-ethoxyethyl)-1,3-diphenylpropane-1,3-dione (3o)[5]. Isolated by flash column chromatography (ethyl acetate/petroleum ether = 1:6, R_f = 0.3). ^1H NMR (ppm) δ 8.02-7.93(m, 4H), 7.58-7.50(m, 2H), 7.47-7.37(m, 4H), 5.46(d, J = 9.0Hz, 1H), 4.51(dq, J = 9.0, 6.0Hz, 1H), 3.59(dq, J = 9.3, 9.3Hz, 1H), 3.30(dq, J = 9.0, 9.0Hz, 1H), 1.28(d, J = 6.0Hz, 3H), 0.95(t, J = 6.9Hz, 3H); ^{13}C NMR (ppm) δ 194.9, 193.8, 137.5, 136.6, 133.5, 133.1, 128.7, 128.7, 128.5, 76.0, 64.9, 63.6, 19.0, 15.1.

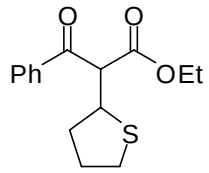


Ethyl 2-benzoyl-3-methoxy-3-phenylpropanoate (3p)[6]. Isolated by flash column chromatography(ethyl acetate/petroleum ether = 1:6, R_{f1} = 0.3, R_{f2} = 0.25). The ratio of two diasteromers is 2:1. **One diasteromer:** ^{1}H NMR (ppm) δ 8.09(d, *J*=8.7Hz, 2H), 7.59-7.17(m, 8H), 5.07(d, *J* = 9.9Hz, 1H), 4.79(d, *J* = 9.9Hz, 1H), 3.86(q, *J* = 6.9Hz, 2H), 3.14(s, 3H), 0.93(t, *J* = 6.9Hz, 3H); ^{13}C NMR (ppm) δ 192.8, 166.3, 138.4, 137.0, 133.5, 128.9, 128.6, 128.5, 128.4, 127.9, 82.2, 61.5, 61.3, 56.8, 13.7; **The other diasteromer:** ^{1}H NMR (ppm) δ 7.83(d, *J* = 8.7Hz, 2H), 7.51-7.15(m, 8H), 5.06(d, *J* = 9.9Hz, 1H), 4.82(d, *J* = 10.2Hz, 1H); 4.31-4.17(m, 2H), 3.22(s, 3H), 1.24(t, *J* = 6.9Hz, 3H); ^{13}C NMR (ppm) δ 192.1, 167.3, 138.4, 136.2, 133.5, 128.6, 128.5, 128.3, 128.2, 127.9, 82.2, 62.3, 61.7, 56.8, 14.1; MS (EI) *m/z* (%): 294, 280, 251, 239, 207, 178, 161, 131, 121, 105(100), 91, 77, 51, 29.

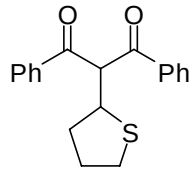


Ethyl 2-benzoyl-3-(benzyloxy)-3-phenylpropanoate (3q). Isolated by flash column chromatography (ethyl acetate/petroleum ether = 1:6, R_f = 0.4). The ratio of two diasteromers is 1:1. **Two diasteromers:** IR (neat): ν_{max} 1735, 1677, 1445, 1292, 1205, 1123, 1084, 1059, 1022, 740cm^{-1}; ^{1}H NMR (ppm) δ 8.11-8.08(m, 2H), 7.85-7.82(m, 2H), 7.60-7.17(m, 24H), 7.07-7.04(m, 2H), 5.32(d, *J* = 9.9Hz, 1H); 5.31(d, *J* = 10.2Hz, 1H), 4.93(d, *J* = 10.2Hz, 1H), 4.88(d, *J* = 9.9Hz, 1H), 4.38(q, *J* = 12.0Hz, 2H), 4.32(s, 2H), 4.19(q, *J* = 7.2Hz, 2H), 3.87(dq, *J* = 7.2, 1.8Hz, 2H), 1.18(t, *J* = 7.2Hz, 3H), 0.93(t, *J* = 7.2Hz, 3H); ^{13}C NMR (ppm) δ 192.8, 192.0, 167.3, 166.3, 138.5, 137.8, 137.7, 137.1, 136.2, 133.5, 133.4, 128.8, 128.7, 128.6, 128.5, 128.4, 128.3, 128.2, 128.1, 128.0, 127.9, 127.8, 127.7, 127.6, 127.5, 127.4, 80.4, 80.3, 70.9, 70.7, 62.3, 61.6, 61.5, 61.3, 14.0, 13.6;

MS (EI) *m/z* (%): 297, 281, 251, 207, 178, 146, 105(100), 91, 77, 51; HRMS calcd for $C_{18}H_{17}O_4(M^+-CH_2Ph)$: 297.1127; found: 297.1124.

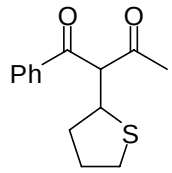


Ethyl 3-oxo-3-phenyl-2-(tetrahydrothiophen-2-yl)propanoate (3r). Isolated by flash column chromatography (ethyl acetate/petroleum ether = 1:6, R_{f1} = 0.45, R_{f2} = 0.4). The ratio of two diasteromers is 1:1. **Two diasteromers:** IR (neat): ν_{max} 1735, 1682, 1596, 1447, 1367, 1278, 1212, 1175, 1159, 1098, 1026, 1001, 984, 701, 683cm^{-1}; **One diasteromer:** ^{1}H NMR (ppm) δ 8.08-8.02(m, 2H), 7.64-7.58(m, 1H), 7.56-7.45(m, 2H), 4.47(d, J = 10.5Hz, 1H), 4.30-4.10(m, 3H), 2.94-2.82(m, 2H), 2.18-1.92(m, 3H), 1.55-1.46(m, 1H), 1.18(t, J = 6.9Hz, 3H); ^{13}C NMR (ppm) δ 194.0, 168.0, 136.4, 133.8, 128.9, 128.8, 128.6, 61.7, 61.6, 47.5, 35.0, 32.3, 30.1, 13.9; **The other diasteromer:** ^{1}H NMR (ppm) δ 8.07-8.02(m, 2H), 7.61-7.54(m, 1H), 7.52-7.44(m, 2H), 4.47(d, J = 9.9Hz, 1H), 4.24-4.09(m, 3H), 2.91-2.81(m, 2H), 2.23-1.93(m, 4H), 1.16(t, J = 6.9Hz, 3H); ^{13}C NMR (ppm) δ 193.2, 167.8, 136.1, 133.6, 128.9, 128.8, 128.7, 128.6, 62.3, 61.6, 46.6, 34.5, 32.3, 30.2, 13.9; MS (EI) *m/z* (%): 278(M^+), 233, 205, 192, 173, 149, 127, 105(100), 101, 85, 77, 58; HRMS calcd for $C_{15}H_{18}O_3S$: 278.0977; found: 278.0976.

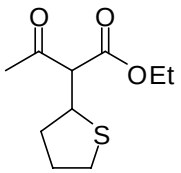


1,3-diphenyl-2-(tetrahydrofuran-2-yl)propane-1,3-dione (3s). Isolated by flash column chromatography (ethyl acetate/petroleum ether = 1:6, R_f = 0.4). IR (neat): ν_{max} 1722, 1694, 1663, 1597, 1578, 1447, 1276, 1228, 1198, 1180, 979, 732, 715, 689cm^{-1}; ^{1}H NMR (ppm) δ 8.04-7.96(m, 4H), 7.60-7.37(m, 6H), 5.36(d, J = 10.2Hz, 1H), 4.44-4.37(m, 1H), 2.93-2.81(m, 2H), 2.69-2.57(m, 1H), 2.23-1.86(m, 2H), 1.70-1.58(m, 1H); ^{13}C NMR (ppm) δ 194.3, 194.2, 136.3, 136.2, 133.8,

133.4, 128.9, 128.6, 65.6, 48.5, 35.0, 32.2, 30.2; MS (EI) *m/z* (%): 310(M^+), 224, 205(100), 186, 147, 105, 85, 77, 51; HRMS calcd for $C_{19}H_{18}O_2S$: 310.1028; found: 310.1030.

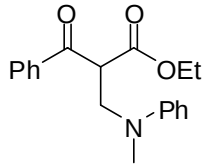


1-phenyl-2-(tetrahydrothiophen-2-yl)butane-1,3-dione (3t). Isolated by flash column chromatography (ethyl acetate/petroleum ether = 1:6, R_{f1} = 0.4, R_{f2} = 0.35). The ratio of two diasteromers is 1:1. **Two diasteromers:** IR (neat): ν_{max} 1721, 1671, 1596, 1447, 1355, 1278, 1208, 1182, 1156, 969, 933, 845, 817, 690cm^{-1}; **One diasteromer:** ^{1}H NMR (ppm) δ 8.06-8.02(m, 2H), 7.65-7.60(m, 1H), 7.54-7.38(m, 2H), 4.60(d, J = 10.8Hz, 1H), 4.31-4.22(m, 1H), 2.92-2.87(m, 2H), 2.19(s, 3H), 2.15-1.86(m, 3H), 1.52-1.43(m, 1H); ^{13}C NMR (ppm) δ 201.9, 194.9, 134.0, 129.0, 128.9, 128.6, 127.0, 70.7, 47.6, 35.3, 32.5, 30.0, 28.3; **The other diasteromer:** ^{1}H NMR (ppm) δ 8.04-8.00(m, 2H), 7.63-7.57(m, 1H), 7.54-7.42(m, 2H), 4.59(d, J = 10.8Hz, 1H), 4.33-4.24(m, 1H), 2.86-2.82(m, 2H), 2.14(s, 3H), 2.11-1.93(m, 3H), 1.71-1.62(m, 1H); ^{13}C NMR (ppm) δ 202.5, 194.8, 133.9, 129.0, 128.8, 128.6, 127.0, 72.1, 46.8, 34.4, 32.2, 30.2, 27.7; MS (EI) *m/z* (%): 248(M^+), 223, 205(100), 171, 149, 143, 127, 105, 85, 77, 43; HRMS calcd for $C_{14}H_{16}O_2S$: 248.0871; found: 248.0872.



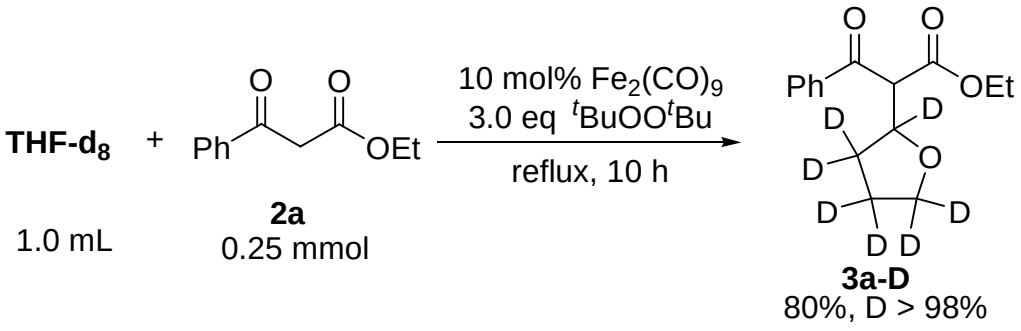
Ethyl 3-oxo-2-(tetrahydrothiophen-2-yl)butanoate (3u)[8]. Isolated by flash column chromatography (ethyl acetate/petroleum ether = 1:6, R_f = 0.5). The ratio of two diasteromers is 1:1. **Two diasteromers:** IR (neat): ν_{max} 2948, 1740, 1715, 1443, 1358, 1270, 1205, 1172, 1020cm^{-1}; ^{1}H NMR (ppm) δ 4.25-4.16(m, 4H), 4.03-3.91(m, 2H), 3.60(d, J = 10.5Hz, 2×1H), 2.86-2.82(m, 4H), 2.27(s, 3H), 2.26(s, 3H), 2.21-1.91(m, 6H), 1.68-1.53(m, 2H), 1.29(t, J = 7.2Hz, 3H), 1.28(t, J =

7.2Hz, 3H); ^{13}C NMR (ppm) δ 201.5, 201.3, 167.9, 167.7, 67.4, 66.9, 61.6, 61.5, 46.0, 45.8, 34.7, 34.5, 32.3, 32.1, 30.1, 29.9, 29.6, 29.2, 14.0, 13.9; MS (EI) *m/z* (%): 216(M^+), 190, 173, 143, 127(100), 99, 87, 85, 59, 43; HRMS calcd for $C_{10}H_{16}O_3S$: 216.0820; found: 216.0822.



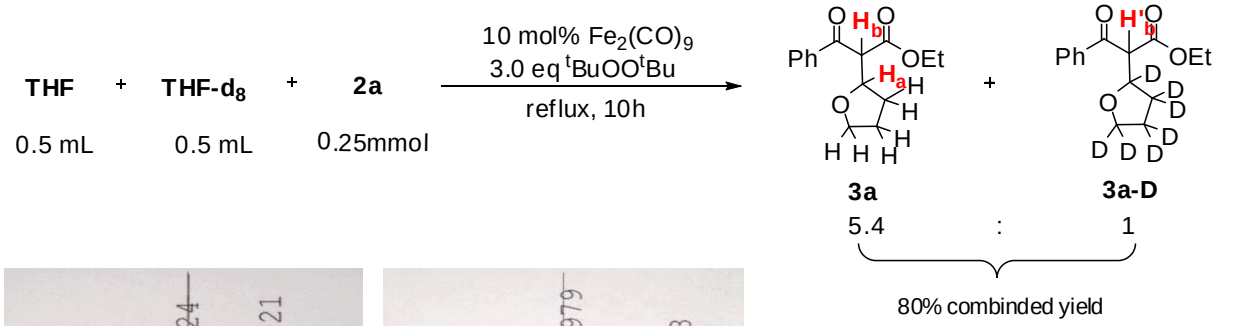
Ethyl 2-((methyl(phenyl)amino)methyl)-3-oxo-3-phenylpropanoate (3v). Isolated by flash column chromatography (ethyl acetate/petroleum ether = 1:6, R_f = 0.5). IR (neat): ν_{max} 2987, 1733, 1684, 1599, 1506, 1448, 1328, 1222, 1118, 1028, 988, 945, 744, 679cm^{-1}; ^{1}H NMR (ppm) δ 7.94-7.91(m, 2H), 7.58-7.54(m, 1H), 7.46-7.40(m, 2H), 7.26-7.21(m, 2H), 6.76-6.70(m, 3H), 4.77(t, J = 6.6Hz, 1H), 4.17-4.02(m, 4H), 2.90(s, 3H), 1.15(t, J = 7.2Hz, 3H); ^{13}C NMR (ppm) δ 194.6, 168.7, 148.2, 136.3, 133.7, 129.3, 128.7, 128.6, 116.9, 112.4, 61.7, 52.2, 52.0, 39.2, 13.9; MS (EI) *m/z* (%): 311 (M^+), 207, 175, 158, 133, 107, 105(100), 77; HRMS calcd for $C_{19}H_{21}NO_3$: 311.1521; found: 311.1519.

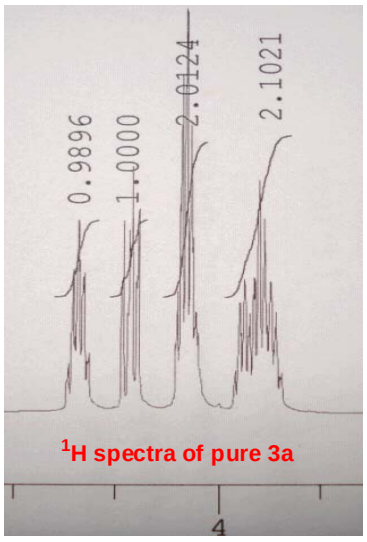
General procedure for products 3a-D: To a mixture of **THF-d$_8$** (1 mL) and $Fe_2(CO)_9$ (9.2 mg, 0.025 mmol), ethyl benzoylacetate **2a** (0.25 mmol) was added under a nitrogen atmosphere at room temperature. *tert*-Butyl peroxide (0.139 mL, 0.75 mmol) was added dropwise into the mixture. The resulting mixture was stirred under reflux condition for 10 h. The resulting reaction mixture was mixed with few silica gel and concentrated, then purified by flash column chromatography (ethyl acetate/petroleum ether = 1:20). The fraction with an R_f = 0.3 (ethyl acetate/petroleum ether = 1:6) was collected and to give the desired product **3a-D**.

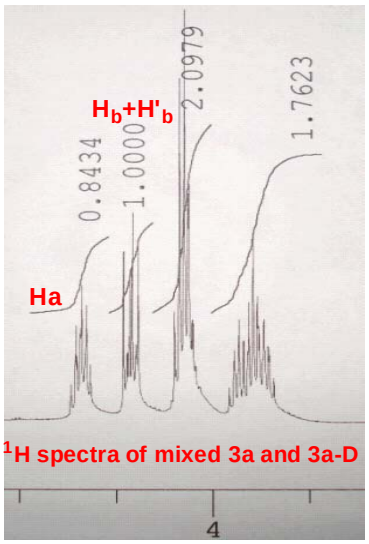


Ethyl 3-oxo-3-phenyl-2-(2,3,3,4,4,5,5,-D-tetrahydrofuran-2-yl)propanoate (3a-D). Isolated by flash column chromatography (ethyl acetate/petroleum ether = 1:6, R_f = 0.3). The ratio of two diasteromers is 1:1. **Two diasteromers:** IR (neat): ν_{max} 1737, 1682, 1597, 1448, 1368, 1298, 1232, 1184, 1052, 1033, 1001, 758, 706, 690, 685cm^{-1}; ^{1}H NMR (ppm) δ 8.05-8.01(m, 4H), 7.62-7.55(m, 2H), 7.51-7.44(m, 4H), 4.45(s, 1H), 4.40(s, 1H), 4.21-4.11(m, 4H), 1.18(t, J = 7.2Hz, 3H), 1.17(t, J = 7.2Hz, 3H); ^{13}C NMR (ppm) δ 193.6, 193.3, 167.9, 167.5, 136.8, 136.3, 133.7, 133.4, 128.8, 128.7, 128.6, 61.6, 61.4, 60.0, 59.2, 13.9, 13.8; MS(EI) m/z(%): 269(M^+), 250, 223, 196, 164, 117, 105(100), 77, 51; HRMS calcd for $C_{15}H_{11}D_7O_4$: 269.1644; found: 269.1645.

General procedure for experiment of KIE: To a mixture of **THF** (0.5 mL), **THF-d$_8$** (0.5 mL) and $Fe_2(CO)_9$ (9.2 mg, 0.025 mmol), ethyl benzoylacetate **2a** (0.25 mmol) was added under a nitrogen atmosphere at room temperature. *tert*-Butyl peroxide (0.139 mL, 0.75 mmol) was added dropwise into the mixture. The resulting mixture was stirred under reflux condition for 10 h. The resulting reaction mixture was mixed with few silica gel and concentrated, then purified by flash column chromatography (ethyl acetate/petroleum ether = 1:20). The fraction with an R_f= 0.3 (ethyl acetate/petroleum ether = 1:6) was collected and to give the desired product **3a** and **3a-D**.







Entry	Cat. M	KIE
1	$Fe_2(CO)_9$ (10%)	5.4

H_a=0.8434

H_b+H'_b=1.0000

H_a=H_b

H'_b=1.0000-H_b=1.0000-0.8434=0.1566

KIE=3a/3a-D=H_b/H'_b=0.8434/0.1566=5.4

References:

[1] A. R. Katritzky, A. A. A. Abdel-Fattah, K. R. Idzik, B. E.-D. M. Ei-Gendy, J. Soloducho, *Tetrahedron.* **2007**, *63*, 6477-6484.

[2] R.-V. Nguyen, X.-Q. Yao, D. S. Bohle, C.-J. Li, *Org. Lett.* **2005**, *7*, 673-675.

[3] O.Moriya, Y. Urata, Y. Ikeda, Y. Ueno, T. Endo, *J. Org. Chem.* **1986**, *51*, 4708-4709.

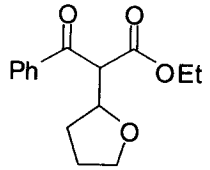
[4] L. A. F. De Godoy, N. S. Camilo, R. A. Pilli, *Tetrahedron Lett.* **2006**, *47*, 7853-7856.

[5] R. Antonioletti, F. Bonadies, A. Scettri, *J. Org. Chem.* **1988**, *53*, 5540-5542.

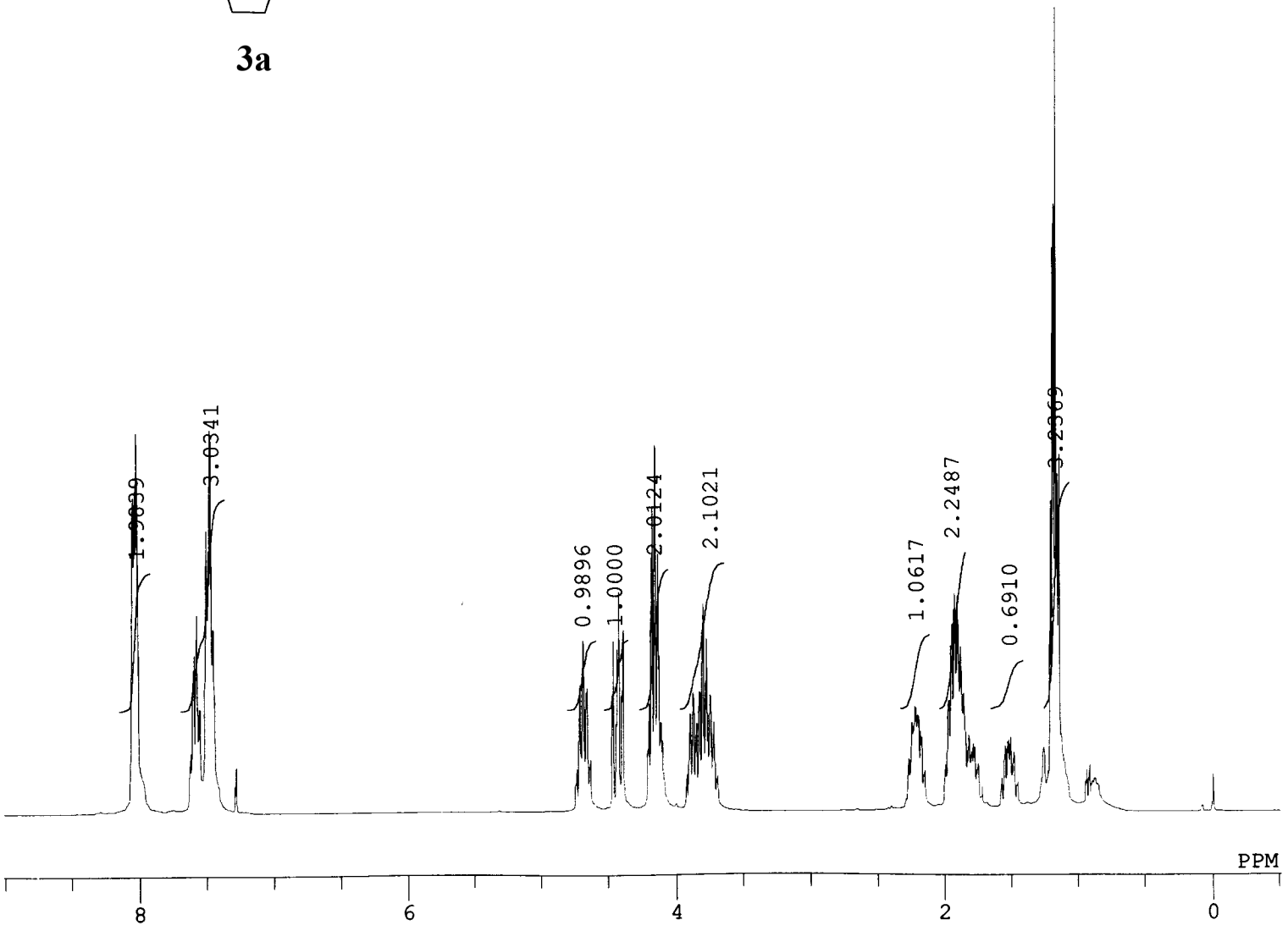
[6] A. Soriente, R. Arienzo, M. De Rosa, L. Palombi, A. Spinella, A. Scettri, *Green Chem*. **1999**, *1*, 157-162.

[7] C. G. Kruse, E. K. Poels, A. van der Gen, *J. Org. Chem.* **1979**, *44*, 2911-2915.

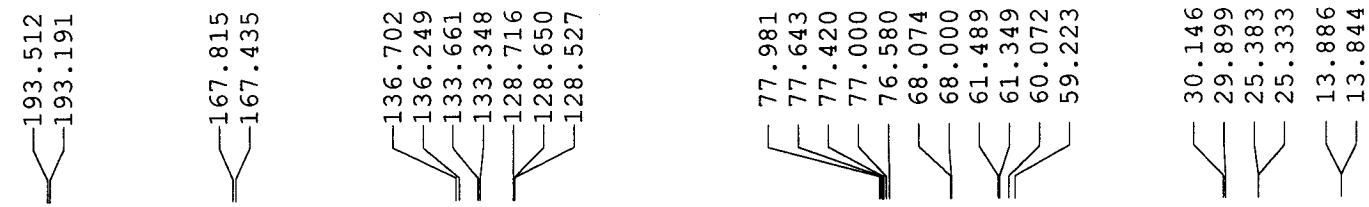
2) Copies of ^{1}H NMR and ^{13}C NMR spectra for all compounds.

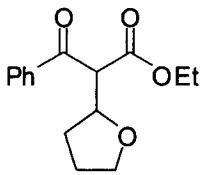


3a



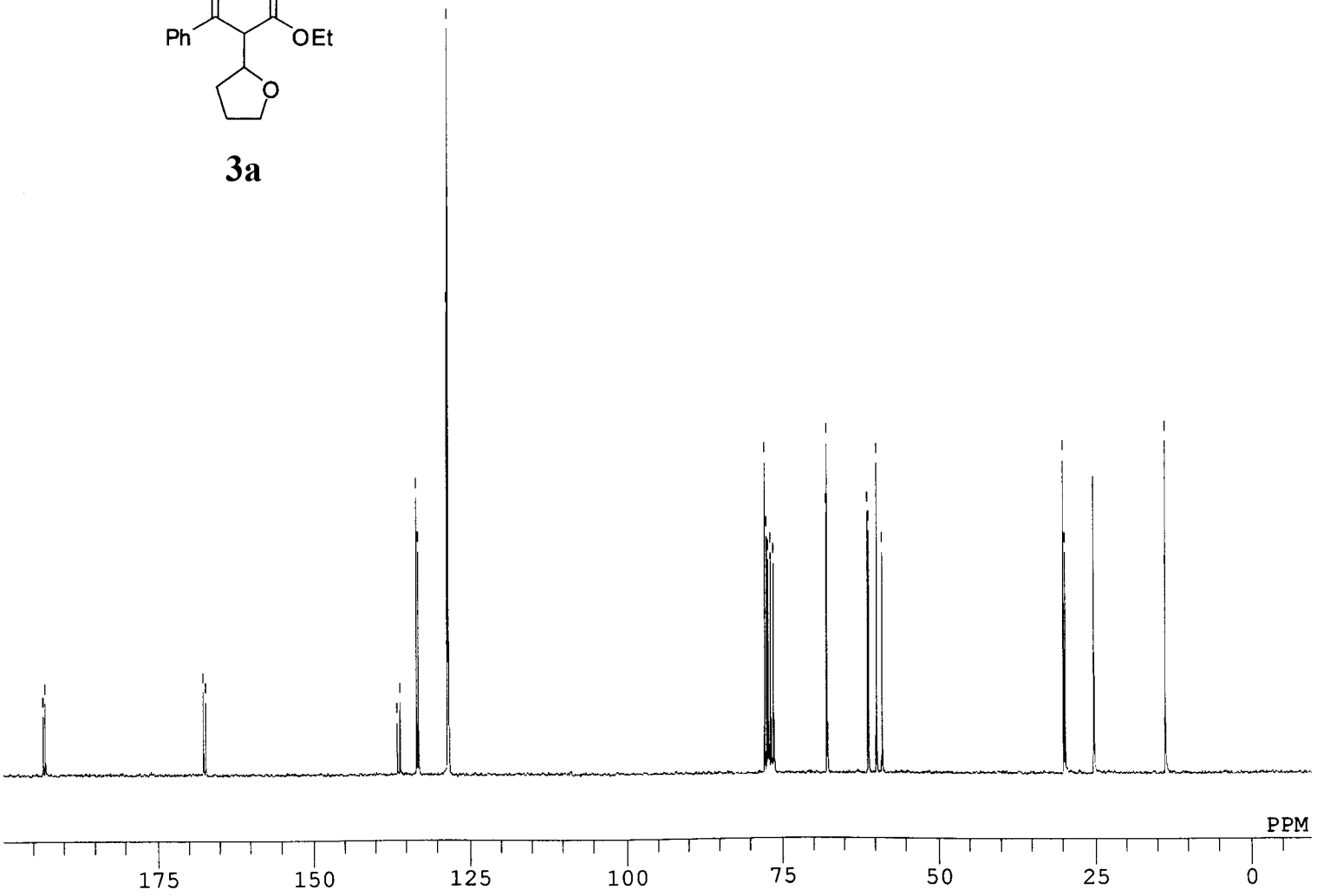

```
DFILE   D:\YuRong\2008\2-H.als
COMNT
DATIM   Mon Jan 14 00:52:32 2008
OBNUC   1H
EXMOD   NON
OBFRQ         300.40 MHz
OBSET         130.00 KHz
OBFIN         1150.0 Hz
POINT          32768
FREQU         6013.2 Hz
SCANS             36
ACQTM          5.449 sec
PD             1.551 sec
PW1              5.8 us
IRNUC   1H
CTEMP           22.1 c
SLVNT   CDCL3
EXREF          0.00 ppm
BF             0.06 Hz
RGAIN             14
```

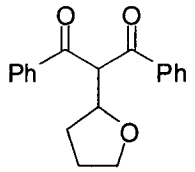




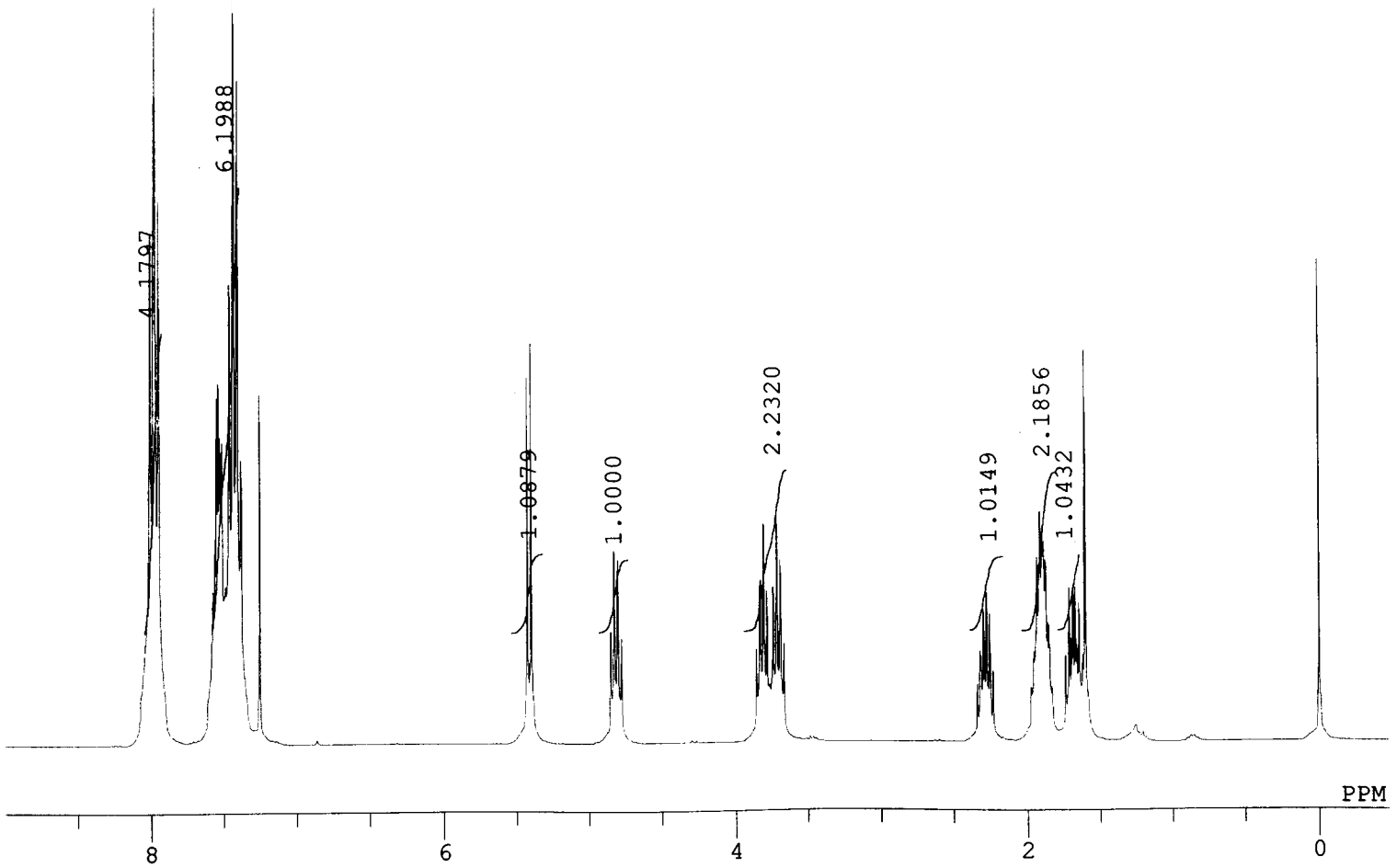
3a

```
DFILE   D:\YuRong\2008\2-C.als
COMNT
DATIM   Fri Dec 14 16:11:00 2007
OBNUC   13C
EXMOD   BCM
OBFRQ         75.45 MHz
OBSET        124.00 KHz
OBFIN        1840.0 Hz
POINT         32768
FREQU       20408.1 Hz
SCANS           320
ACQTM         1.606 sec
PD            1.394 sec
PW1             5.0 us
IRNUC   1H
CTEMP          22.7 c
SLVNT   CDCL3
EXREF         77.00 ppm
BF             0.62 Hz
RGAIN            26
```

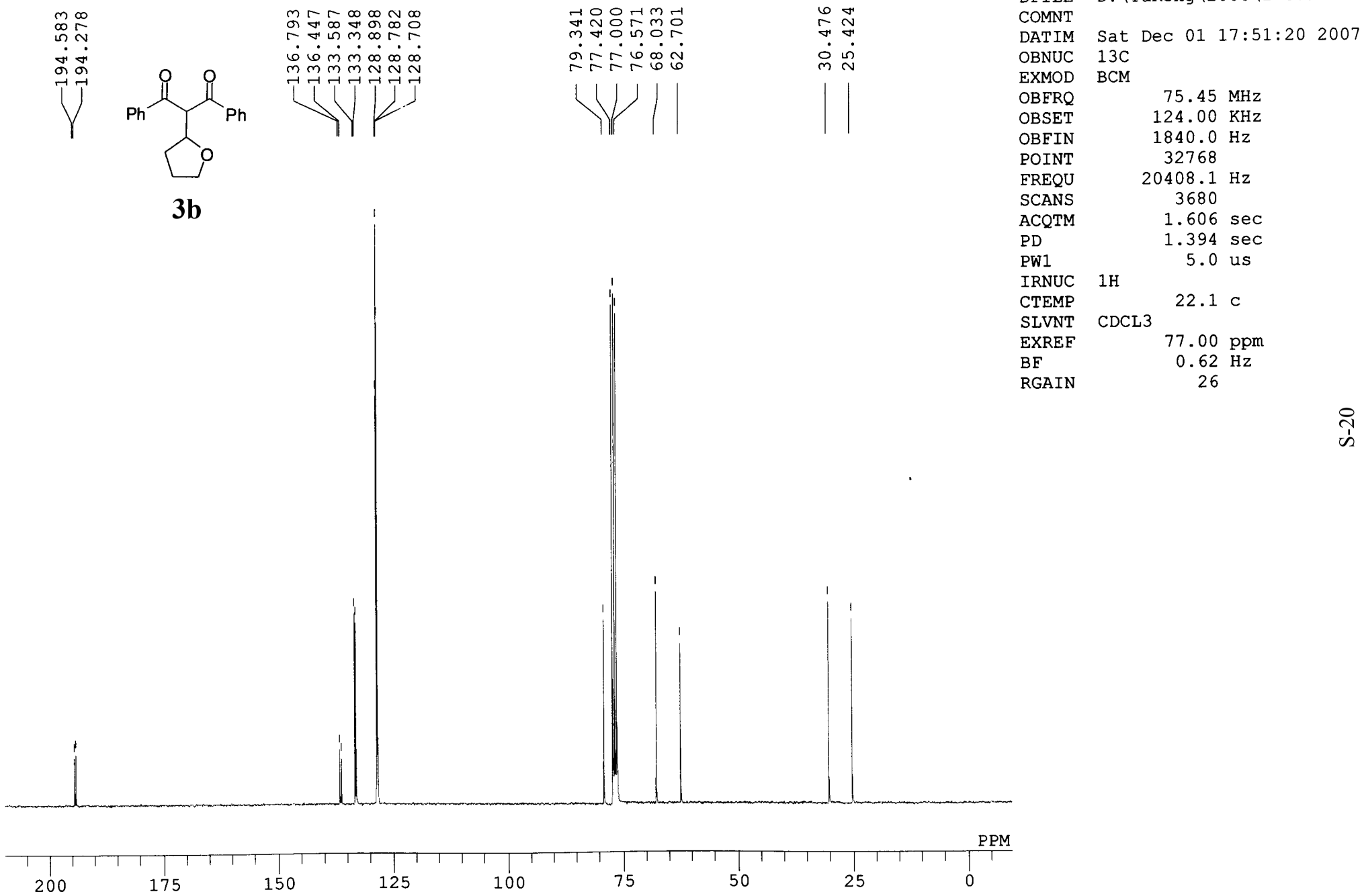




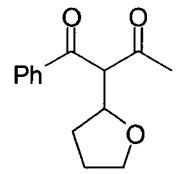
3b




```
DFILE   D:\YuRong\2008\1-H.als
COMNT
DATIM   Mon Jan 14 00:37:23 2008
OBNUC   1H
EXMOD   NON
OBFRQ         300.40 MHz
OBSET         130.00 KHz
OBFIN         1150.0 Hz
POINT          32768
FREQU         6013.2 Hz
SCANS             16
ACQTM          5.449 sec
PD             1.551 sec
PW1              5.8 us
IRNUC   1H
CTEMP           21.8 c
SLVNT   CDCL3
EXREF          0.00 ppm
BF             0.09 Hz
RGAIN             21
```



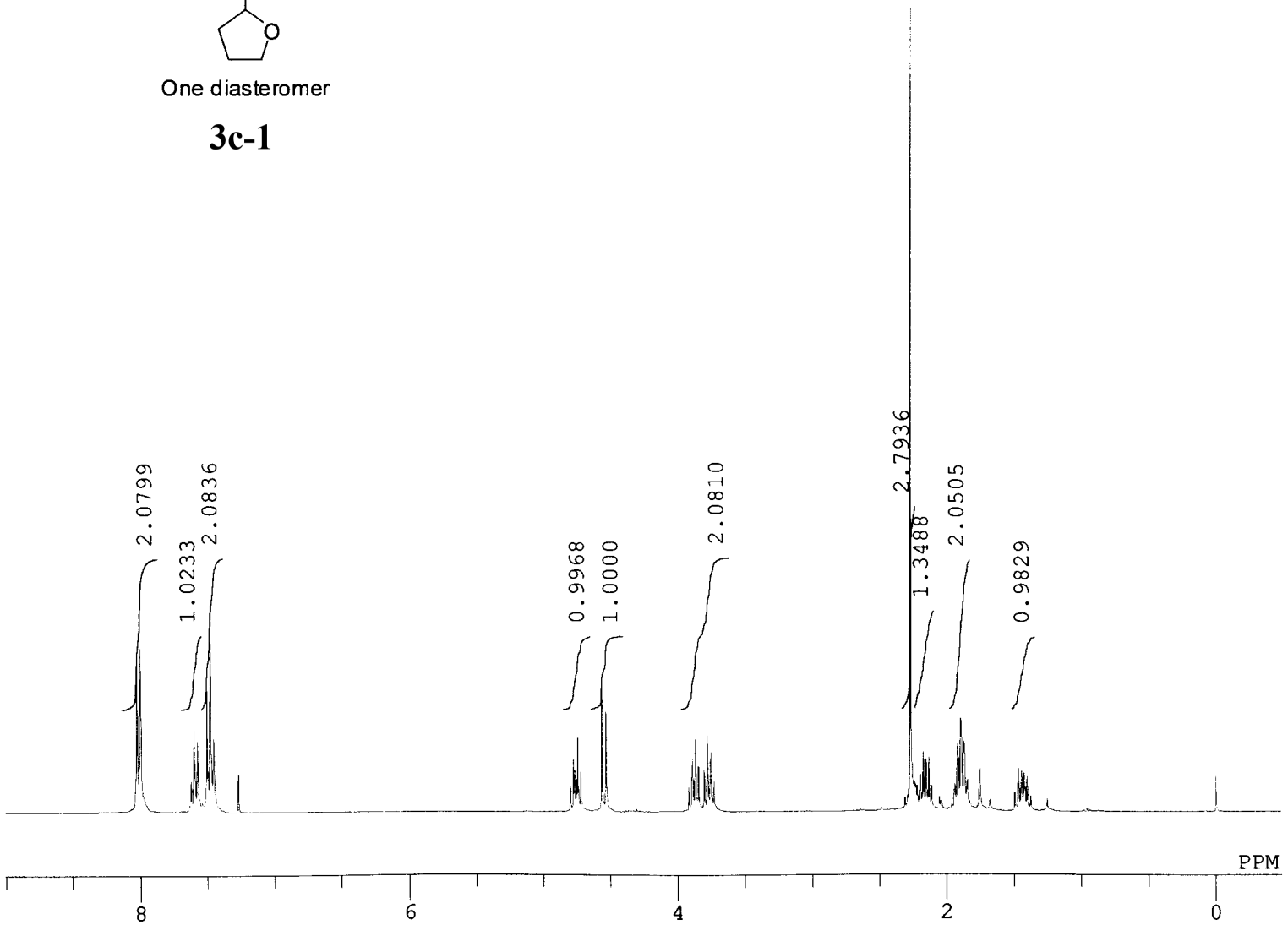
DFILE	D:\YuRong\2008\1-C.als
COMNT	
DATIM	Sat Dec 01 17:51:20 2007
OBNUC	13C
EXMOD	BCM
OBFRQ	75.45 MHz
OBSET	124.00 KHz
OBFIN	1840.0 Hz
POINT	32768
FREQU	20408.1 Hz
SCANS	3680
ACQTM	1.606 sec
PD	1.394 sec
PW1	5.0 us
IRNUC	1H
CTEMP	22.1 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.62 Hz
RGAIN	26

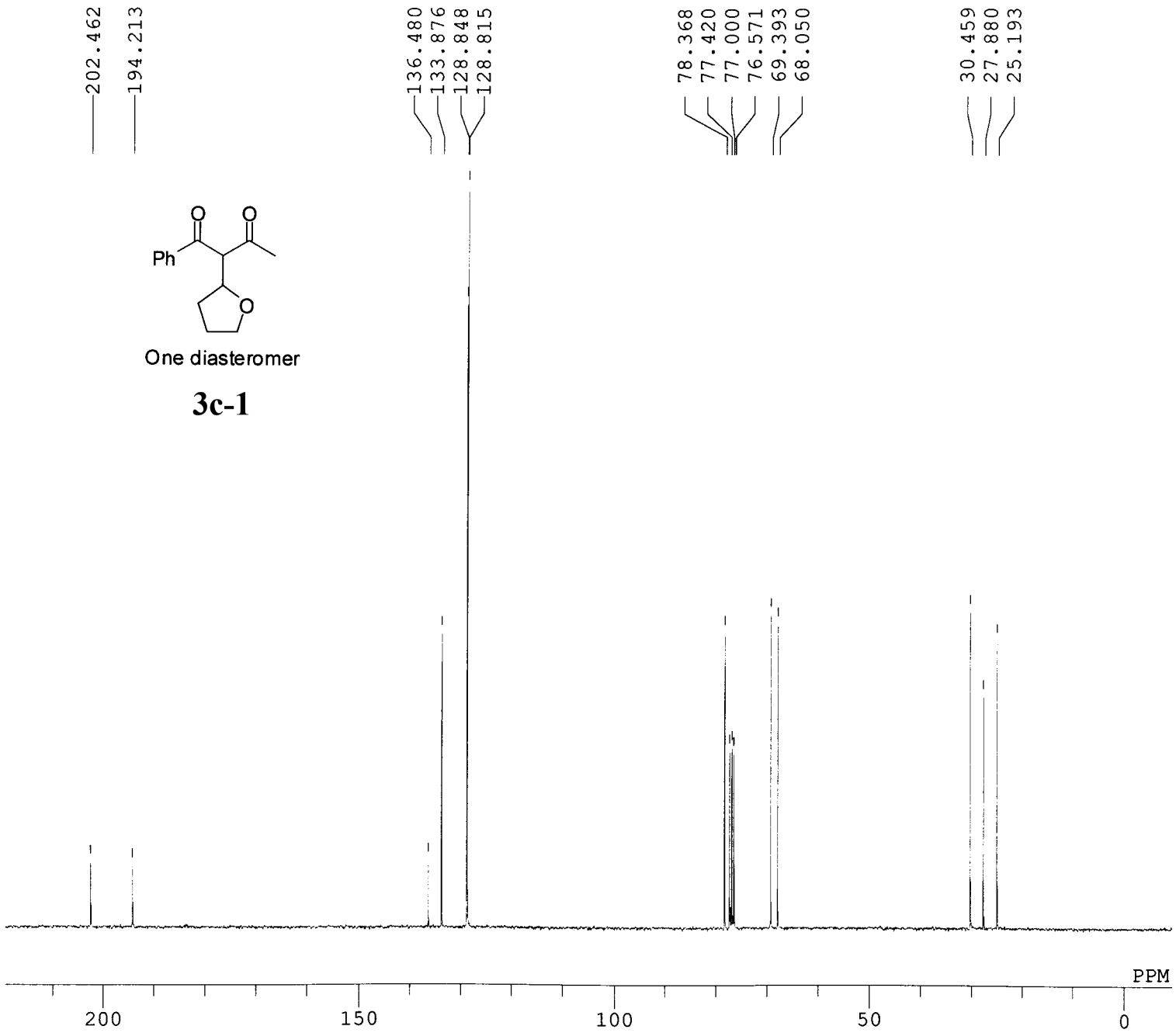


One diasteromer

3c-1

```
DFILE   D:\YuRong\2008\21-1H.als
COMNT
DATIM   Sun May 04 22:53:33 2008
OBNUC   1H
EXMOD   NON
OBFRQ       300.40 MHz
OBSET       130.00 KHz
OBFIN       1150.0 Hz
POINT        32768
FREQU       6013.2 Hz
SCANS           16
ACQTM        5.449 sec
PD           1.551 sec
PW1            5.8 us
IRNUC   1H
CTEMP         22.9 c
SLVNT   CDCL3
EXREF         0.00 ppm
BF            0.09 Hz
RGAIN           16
```

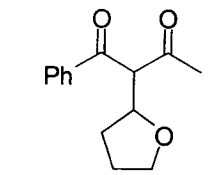




One diasteromer

3c-1

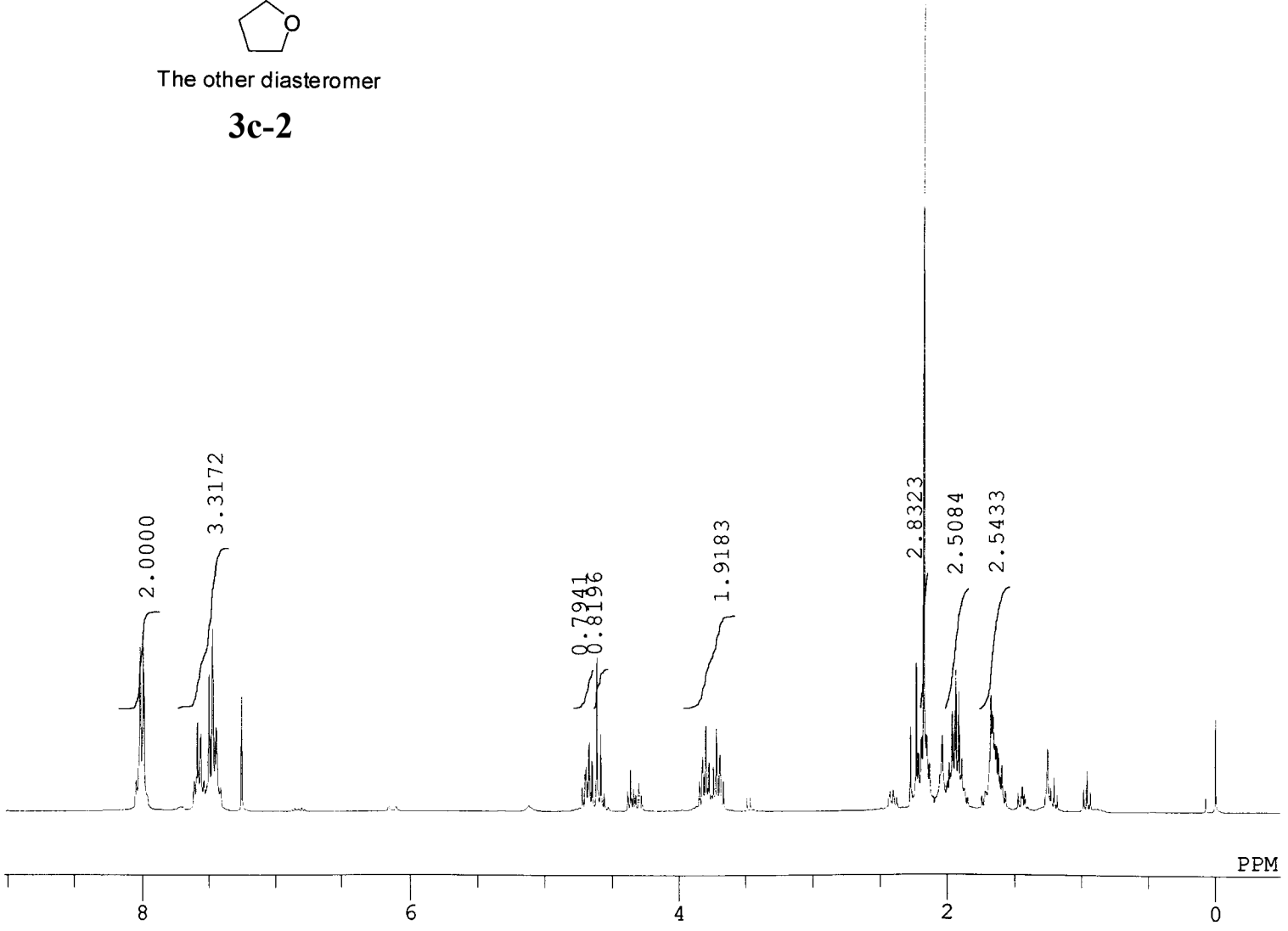
DFILE	D:\YuRong\2008\21-1C.als
COMNT	
DATIM	Sun May 04 22:50:03 2008
OBNUC	13C
EXMOD	BCM
OBFRQ	75.45 MHz
OBSET	124.00 KHz
OBFIN	1840.0 Hz
POINT	32768
FREQU	20408.1 Hz
SCANS	200
ACQTM	1.606 sec
PD	1.394 sec
PW1	5.0 us
IRNUC	1H
CTEMP	23.0 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.62 Hz
RGAIN	26

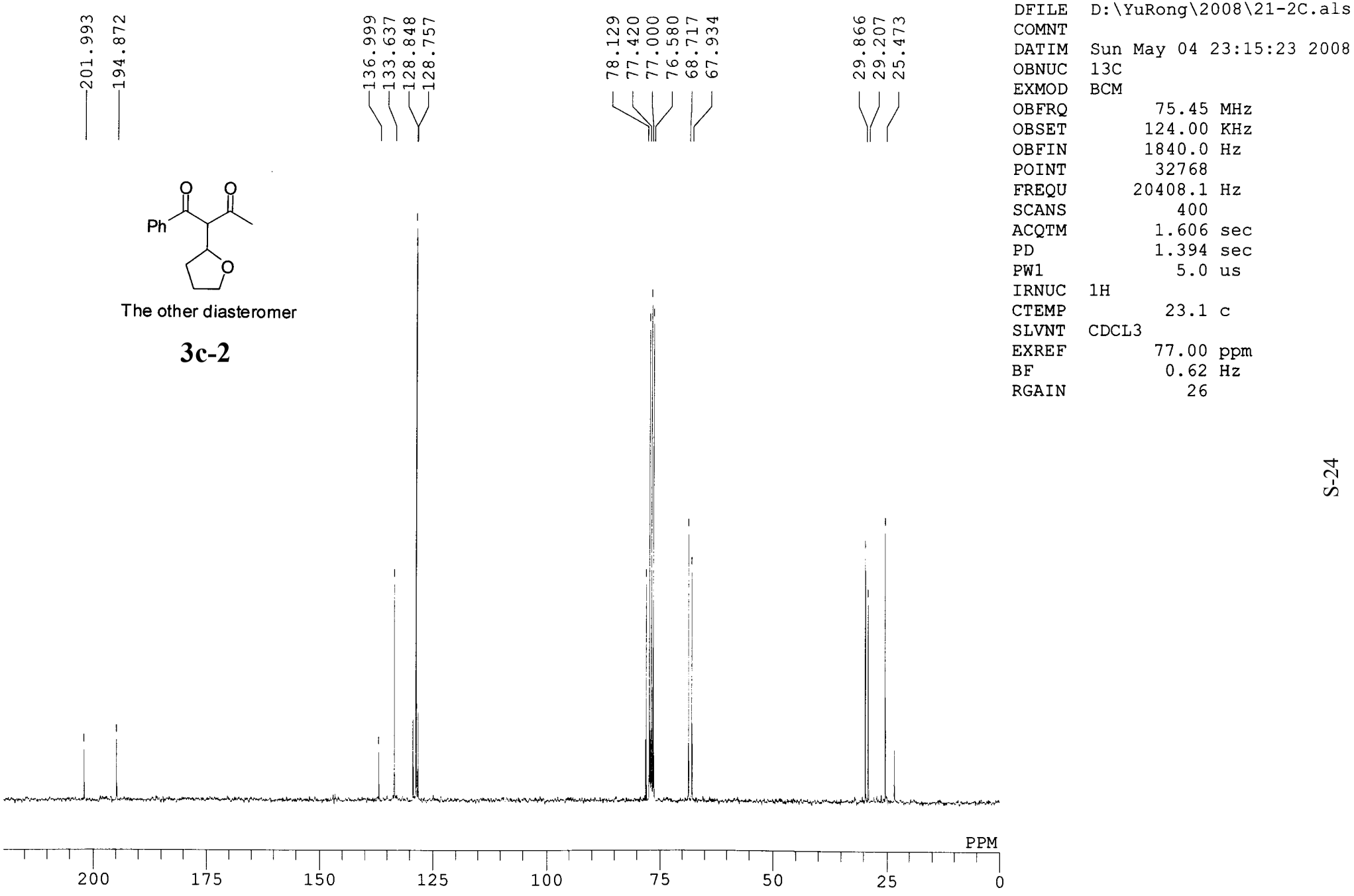


The other diasteromer

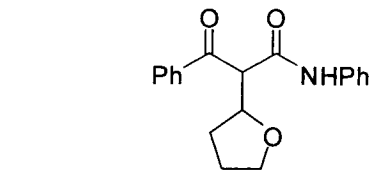
3c-2

```
DFILE   D:\YuRong\2008\21-2H.als
COMNT
DATIM   Sun May 04 23:21:48 2008
OBNUC   1H
EXMOD   NON
OBFRQ         300.40 MHz
OBSET         130.00 KHz
OBFIN         1150.0 Hz
POINT          32768
FREQU         6013.2 Hz
SCANS             16
ACQTM          5.449 sec
PD             1.551 sec
PW1              5.8 us
IRNUC   1H
CTEMP           22.7 c
SLVNT   CDCL3
EXREF             0.00 ppm
BF                0.09 Hz
RGAIN               18
```



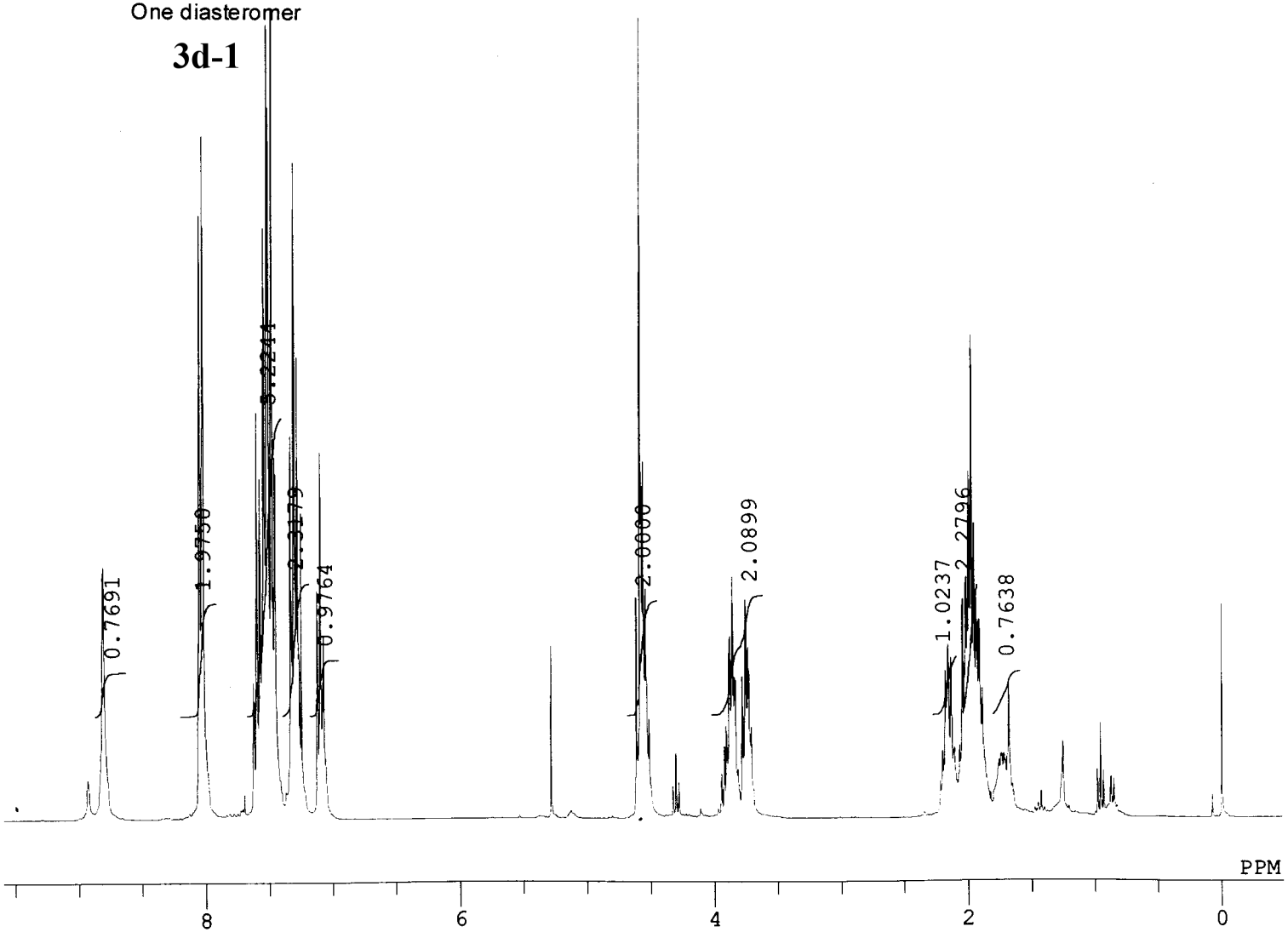


DFILE	D:\YuRong\2008\21-2C.als
COMNT	
DATIM	Sun May 04 23:15:23 2008
OBNUC	13C
EXMOD	BCM
OBFRQ	75.45 MHz
OBSET	124.00 KHz
OBFIN	1840.0 Hz
POINT	32768
FREQU	20408.1 Hz
SCANS	400
ACQTM	1.606 sec
PD	1.394 sec
PW1	5.0 us
IRNUC	1H
CTEMP	23.1 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.62 Hz
RGAIN	26

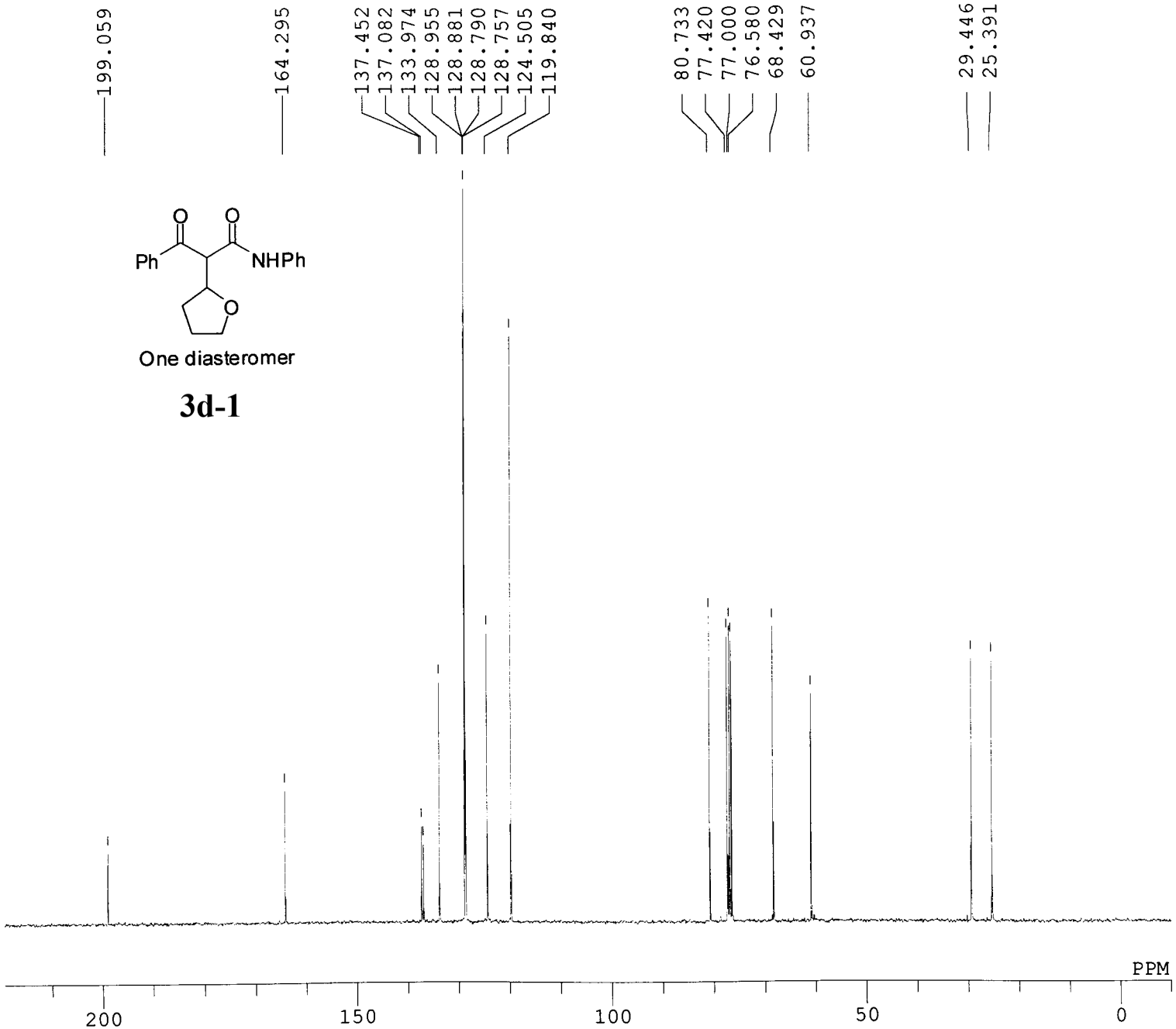


One diasteromer

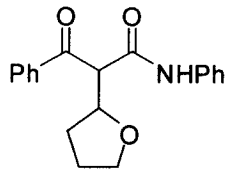
3d-1




```
DFILE   D:\YuRong\2008\22-1H.als
COMNT
DATIM   Sun May 04 23:47:14 2008
OBNUC   1H
EXMOD   NON
OBFRQ         300.40 MHz
OBSET         130.00 KHz
OBFIN         1150.0 Hz
POINT          32768
FREQU         6013.2 Hz
SCANS             16
ACQTM          5.449 sec
PD             1.551 sec
PW1              5.8 us
IRNUC   1H
CTEMP           22.3 c
SLVNT   CDCL3
EXREF           0.00 ppm
BF              0.09 Hz
RGAIN             16
```

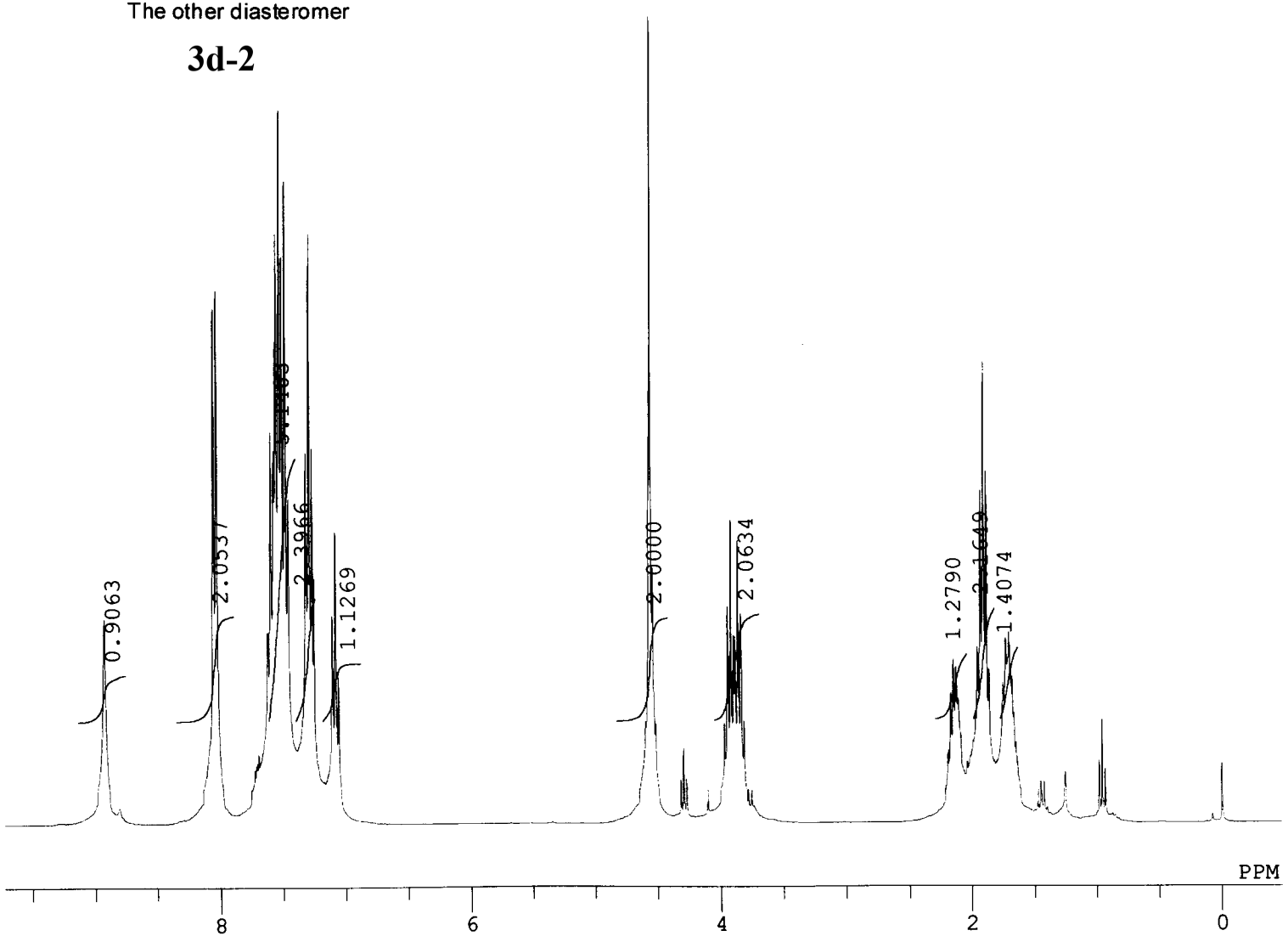


DFILE D:\YuRong\2008\22-1C.als
COMNT
DATIM Sun May 04 23:42:04 2008
OBNUC 13C
EXMOD BCM
OBFRQ 75.45 MHz
OBSET 124.00 KHz
OBFIN 1840.0 Hz
POINT 32768
FREQU 20408.1 Hz
SCANS 370
ACQTM 1.606 sec
PD 1.394 sec
PW1 5.0 us
IRNUC 1H
CTEMP 23.2 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.62 Hz
RGAIN 26

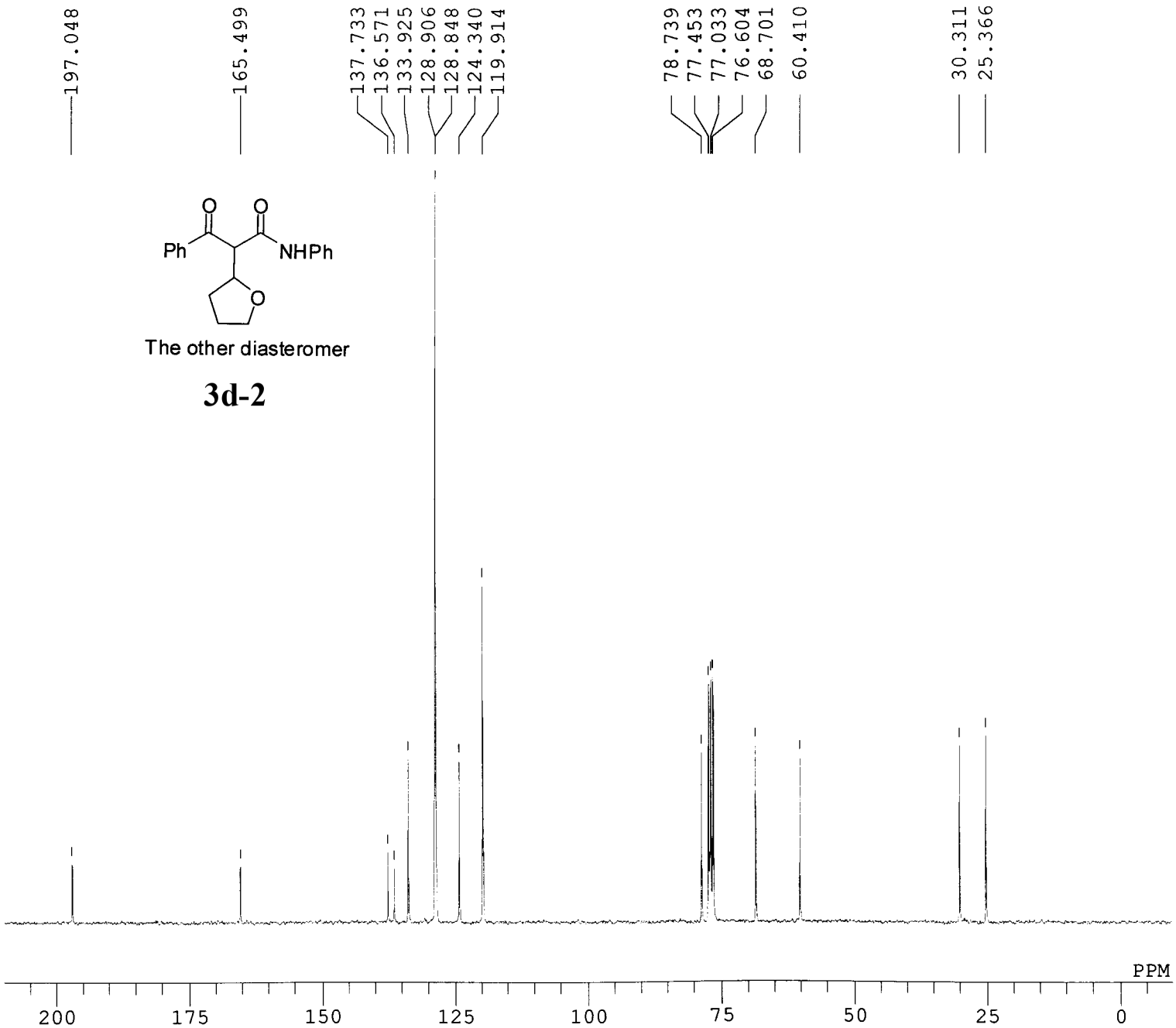


The other diasteromer

3d-2



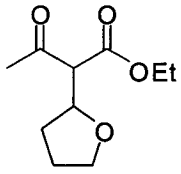

```
DFILE   D:\YuRong\2008\22-2H.als
COMNT
DATIM   Wed Apr 16 07:15:29 2008
OBNUC   1H
EXMOD   NON
OBFRQ         300.40 MHz
OBSET         130.00 KHz
OBFIN         1150.0 Hz
POINT          32768
FREQU         6013.2 Hz
SCANS             16
ACQTM          5.449 sec
PD             1.551 sec
PW1              5.8 us
IRNUC   1H
CTEMP           23.5 c
SLVNT   CDCL3
EXREF           0.00 ppm
BF              0.09 Hz
RGAIN             16
```



The other diasteromer

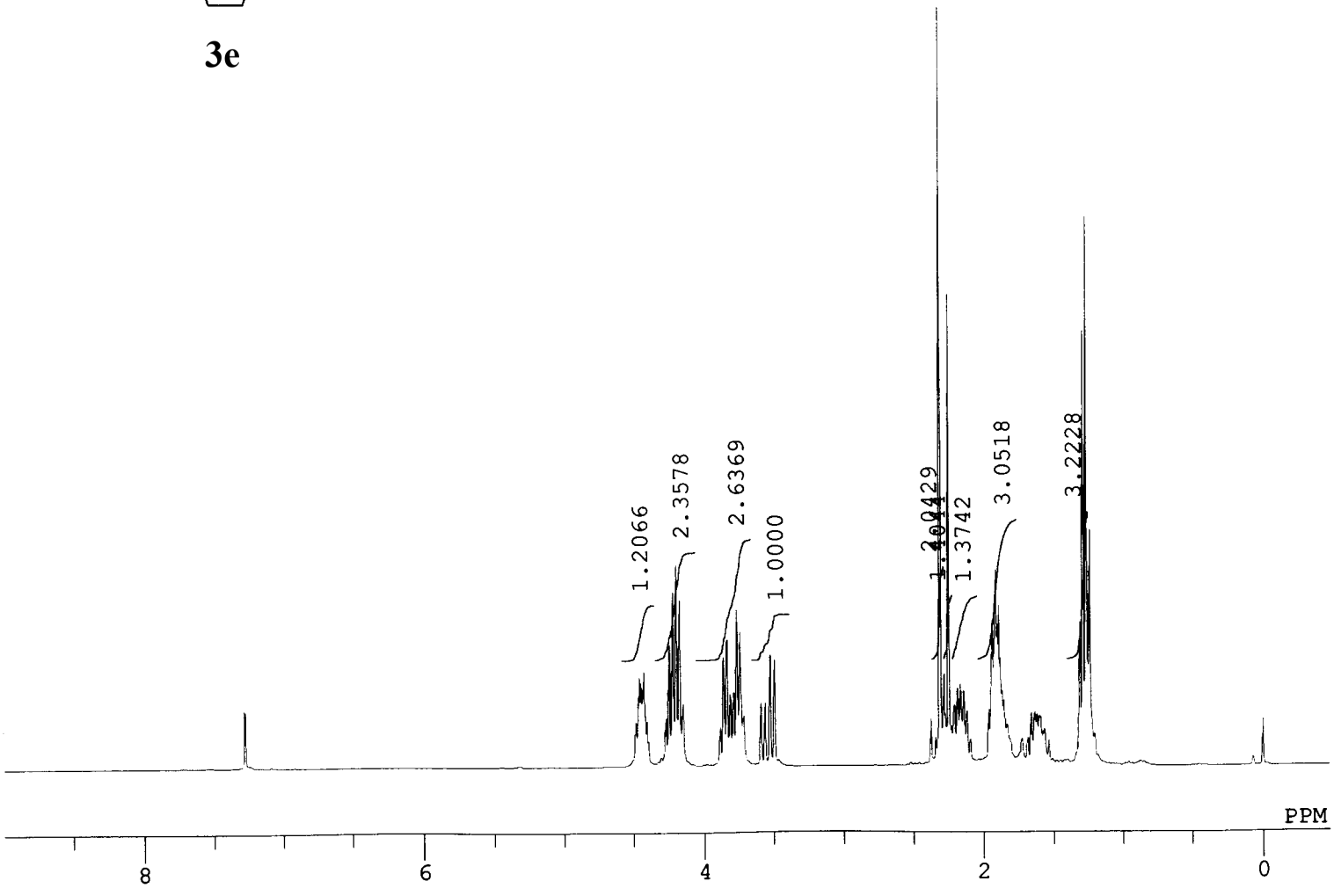
3d-2

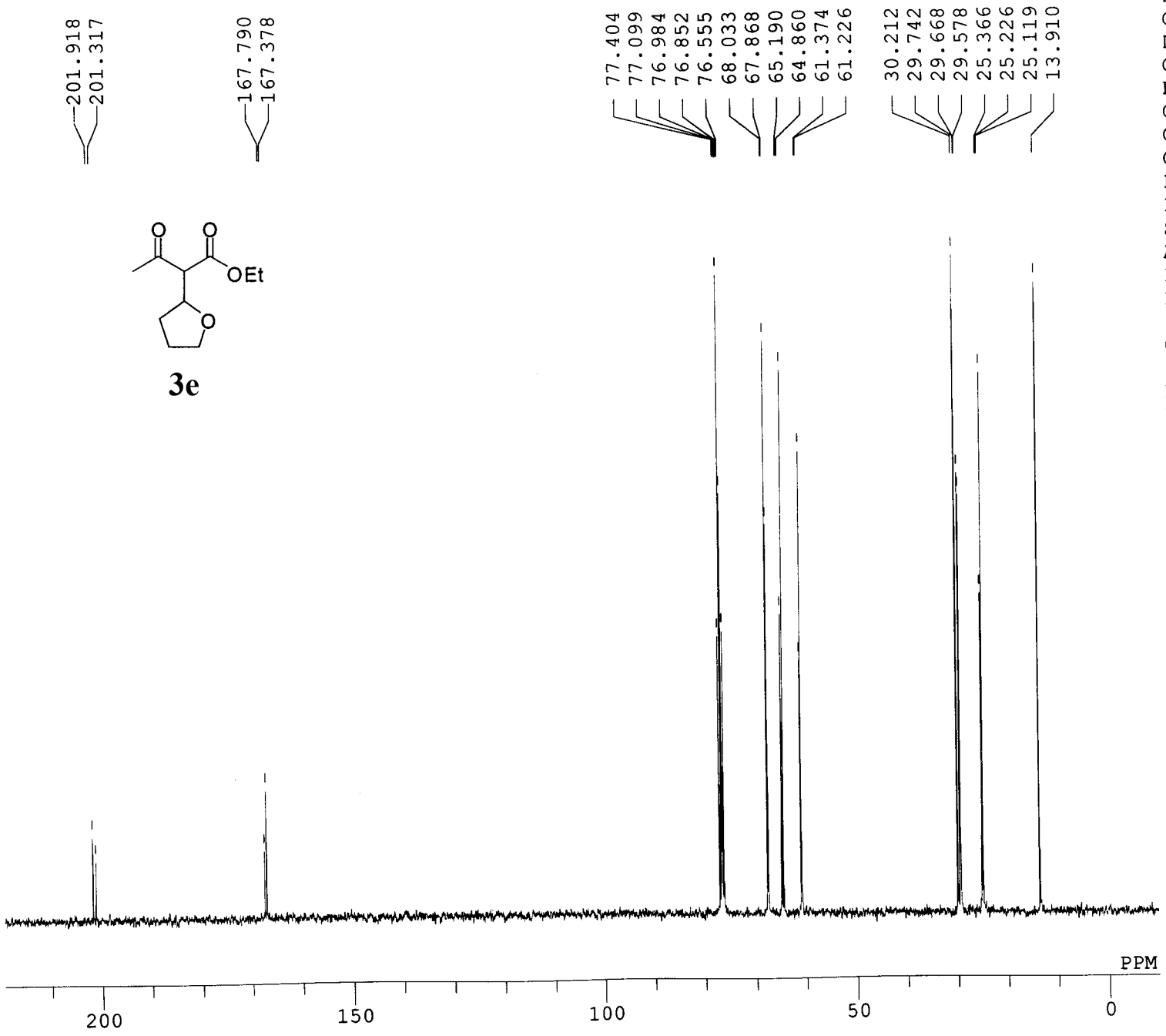
```
DFILE   D:\YuRong\2008\22-2C.als
COMNT
DATIM   Wed Apr 16 10:19:20 2008
OBNUC   13C
EXMOD   BCM
OBFRQ         75.45 MHz
OBSET        124.00 KHz
OBFIN        1840.0 Hz
POINT         32768
FREQU       20408.1 Hz
SCANS           360
ACQTM         1.606 sec
PD            1.394 sec
PW1             5.0 us
IRNUC   1H
CTEMP          23.8 c
SLVNT   CDCL3
EXREF         77.00 ppm
BF             0.62 Hz
RGAIN            26
```



3e

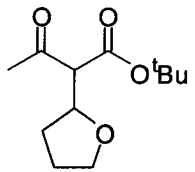
```
DFILE   D:\YuRong\2008\8-H.als
COMNT
DATIM   Mon Mar 17 23:16:52 2008
OBNUC   1H
EXMOD   NON
OBFRQ        300.40 MHz
OBSET        130.00 KHz
OBFIN        1150.0 Hz
POINT         32768
FREQU        6013.2 Hz
SCANS            16
ACQTM         5.449 sec
PD            1.551 sec
PW1             5.8 us
IRNUC   1H
CTEMP          23.2 c
SLVNT   CDCL3
EXREF          0.00 ppm
BF             0.09 Hz
RGAIN            16
```





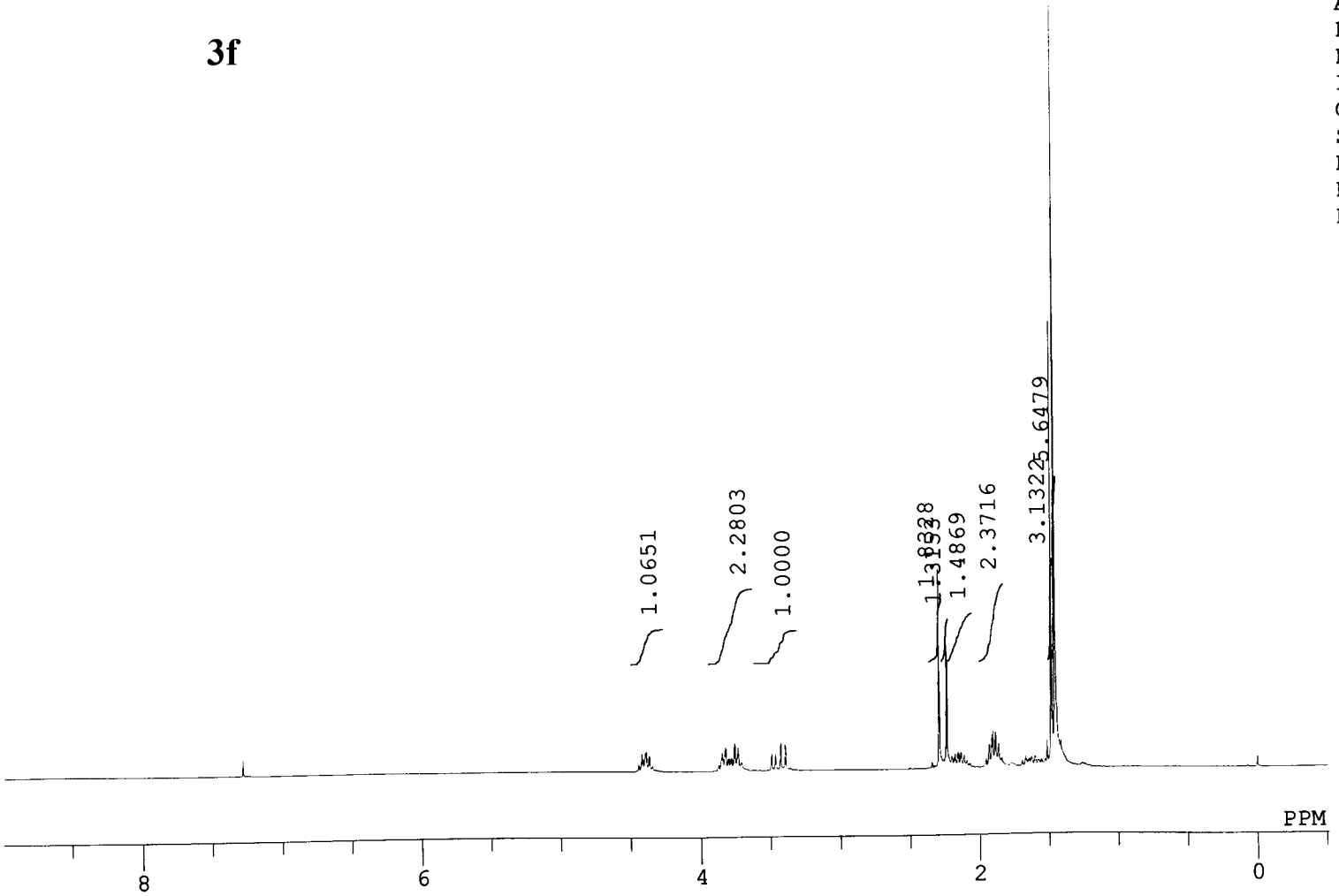
3e

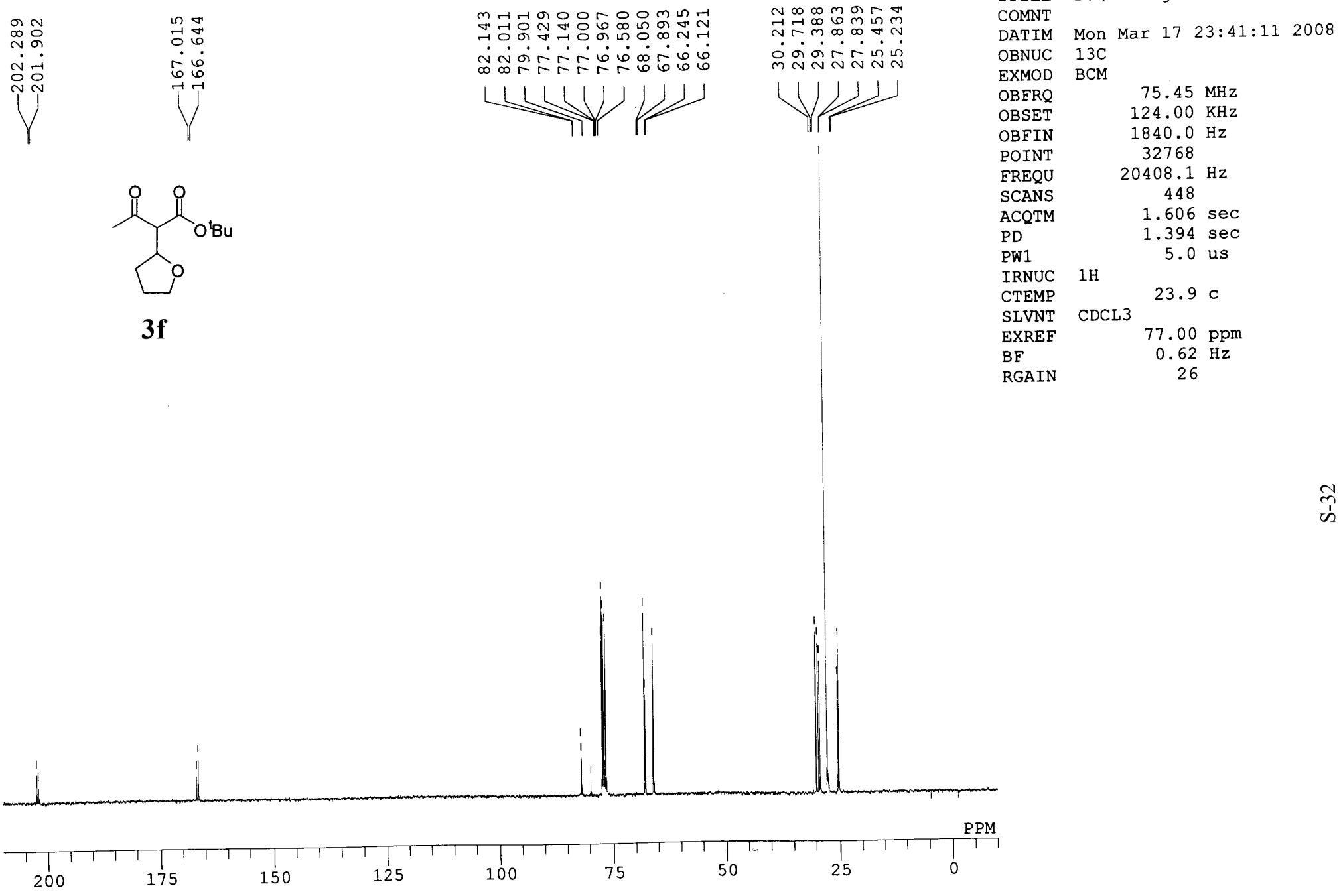
```
DFILE   D:\YuRong\2008\8-C.als
COMNT
DATIM   Wed Apr 16 09:33:19 2008
OBNUC   13C
EXMOD   BCM
OBFRQ         75.45 MHz
OBSET        124.00 KHz
OBFIN        1840.0 Hz
POINT         32768
FREQU       20408.1 Hz
SCANS           100
ACQTM         1.606 sec
PD            1.394 sec
PW1             5.0 us
IRNUC   1H
CTEMP          23.1 c
SLVNT   CDCL3
EXREF         77.00 ppm
BF             0.62 Hz
RGAIN            26
```



3f

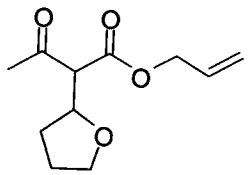
DFILE	D:\YuRong\2008\9-H.als
COMNT	
DATIM	Mon Mar 17 23:45:16 2008
OBNUC	1H
EXMOD	NON
OBFRQ	300.40 MHz
OBSET	130.00 KHz
OBFIN	1150.0 Hz
POINT	32768
FREQU	6013.2 Hz
SCANS	16
ACQTM	5.449 sec
PD	1.551 sec
PW1	5.8 us
IRNUC	1H
CTEMP	23.2 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.09 Hz
RGAIN	15





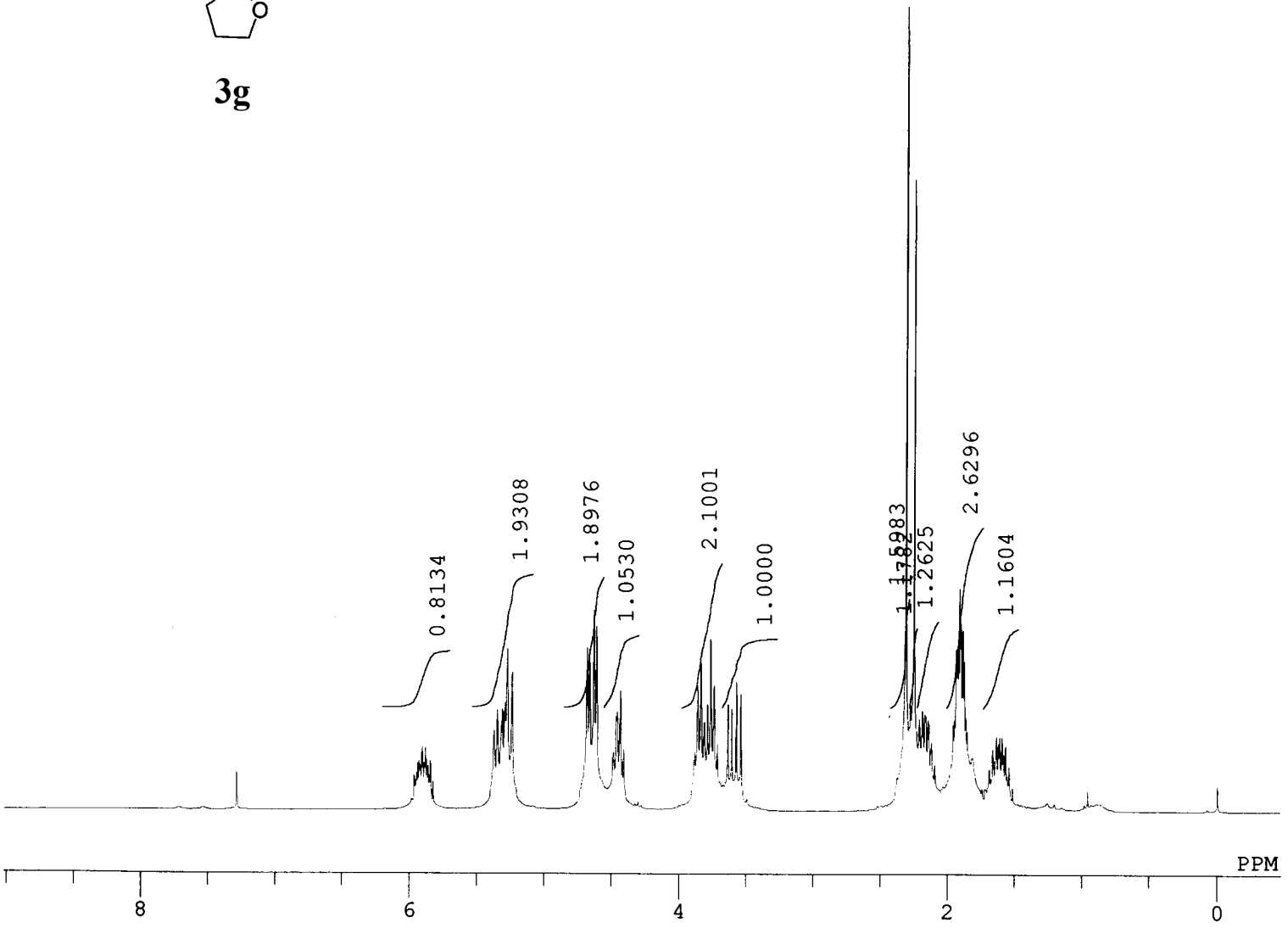
3f

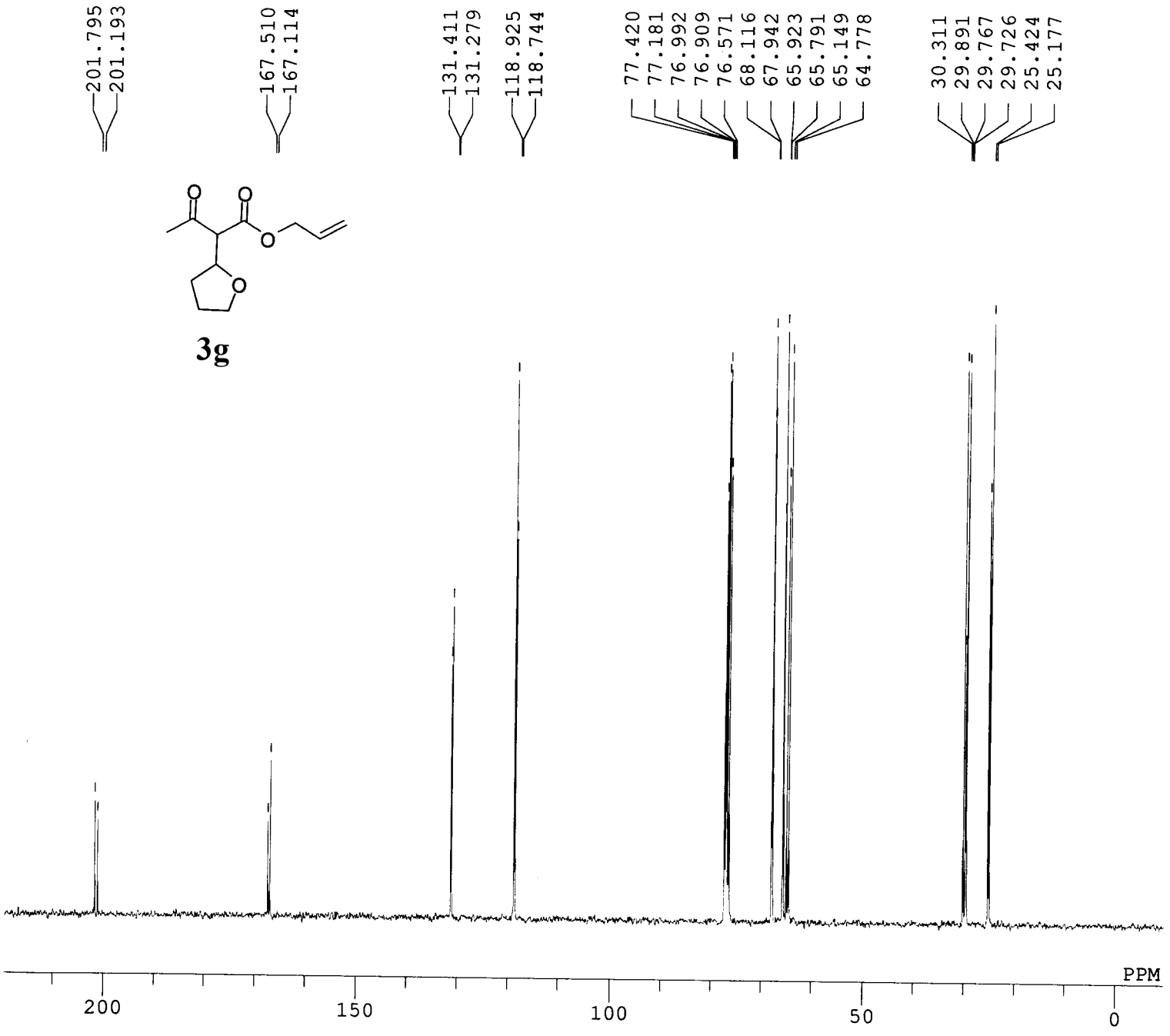
```
DFILE   D:\YuRong\2008\9-C.als
COMNT
DATIM   Mon Mar 17 23:41:11 2008
OBNUC   13C
EXMOD   BCM
OBFRQ         75.45 MHz
OBSET        124.00 KHz
OBFIN        1840.0 Hz
POINT         32768
FREQU       20408.1 Hz
SCANS           448
ACQTM         1.606 sec
PD            1.394 sec
PW1             5.0 us
IRNUC   1H
CTEMP          23.9 c
SLVNT   CDCL3
EXREF         77.00 ppm
BF             0.62 Hz
RGAIN            26
```



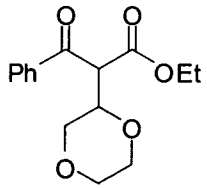
3g

DFILE	D:\YuRong\2008\20-H.als
COMNT	
DATIM	Wed Apr 16 06:47:16 2008
OBNUC	1H
EXMOD	NON
OBFRQ	300.40 MHz
OBSET	130.00 KHz
OBFIN	1150.0 Hz
POINT	32768
FREQU	6013.2 Hz
SCANS	16
ACQTM	5.449 sec
PD	1.551 sec
PW1	5.8 us
IRNUC	1H
CTEMP	23.5 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.06 Hz
RGAIN	15



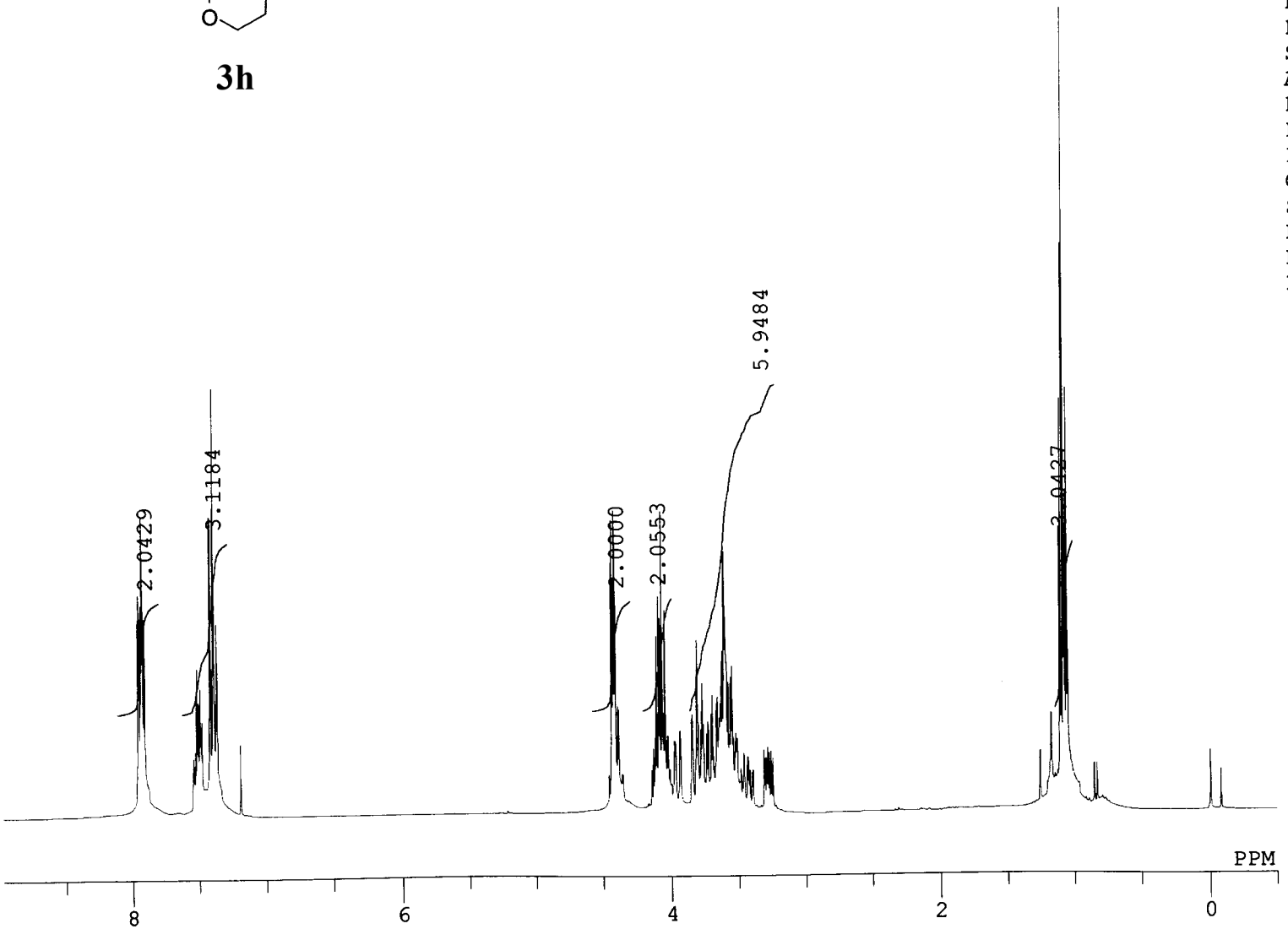


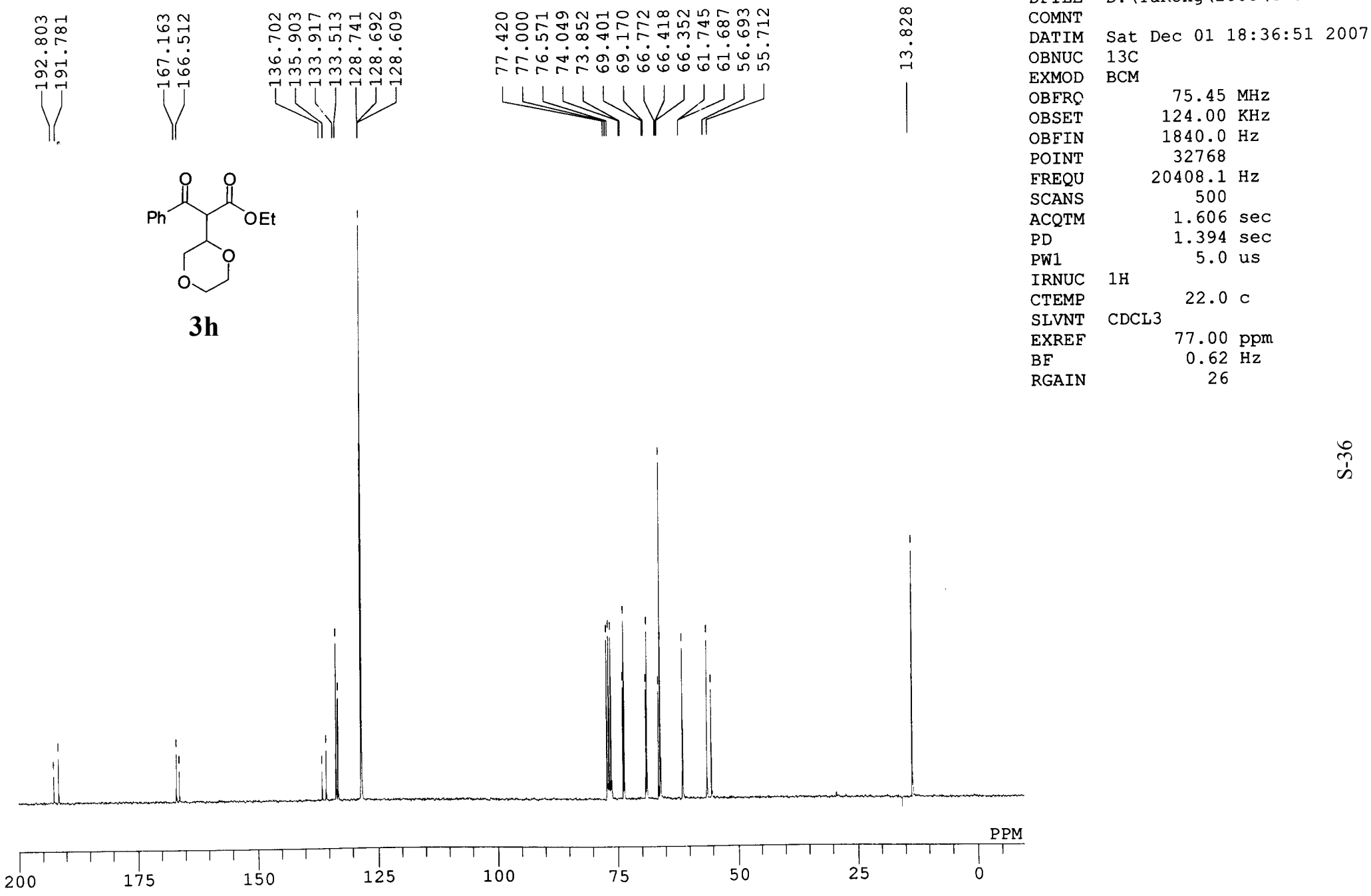
DFILE	D:\YuRong\2008\20-C.als
COMNT	
DATIM	Wed Apr 16 06:58:25 2008
OBNUC	13C
EXMOD	BCM
OBFRQ	75.45 MHz
OBSET	124.00 KHz
OBFIN	1840.0 Hz
POINT	32768
FREQU	20408.1 Hz
SCANS	200
ACQTM	1.606 sec
PD	1.394 sec
PW1	5.0 us
IRNUC	1H
CTEMP	24.1 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.62 Hz
RGAIN	26



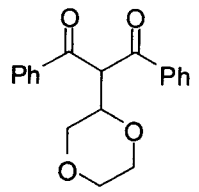
3h

```
DFILE   D:\YuRong\2008\4-H.als
COMNT
DATIM   Mon Jan 14 01:18:35 2008
OBNUC   1H
EXMOD   NON
OBFRQ         300.40 MHz
OBSET         130.00 KHz
OBFIN         1150.0 Hz
POINT          32768
FREQU         6013.2 Hz
SCANS             16
ACQTM          5.449 sec
PD             1.551 sec
PW1              5.8 us
IRNUC   1H
CTEMP           21.6 c
SLVNT   CDCL3
EXREF          0.00 ppm
BF             0.09 Hz
RGAIN             14
```

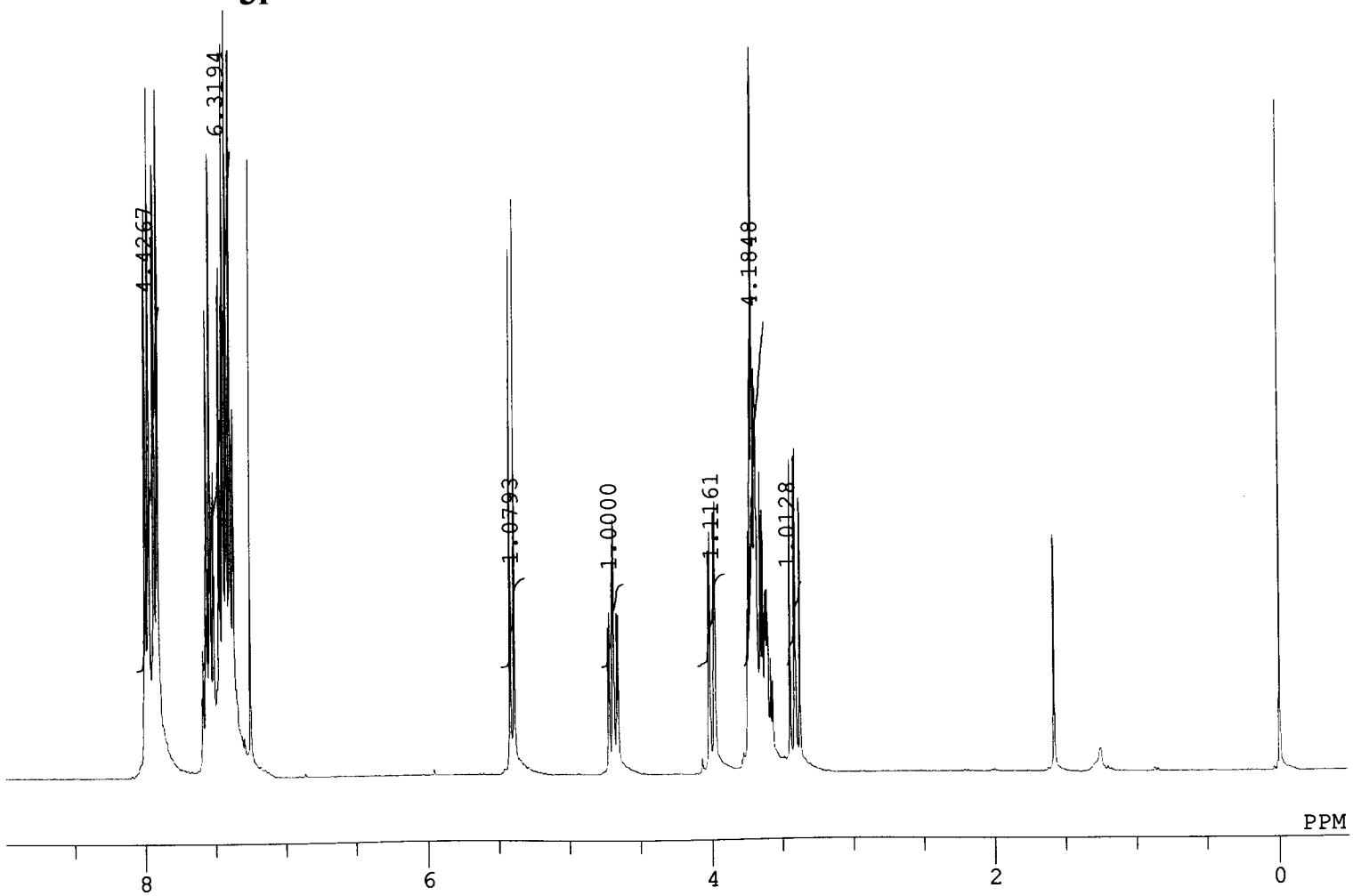




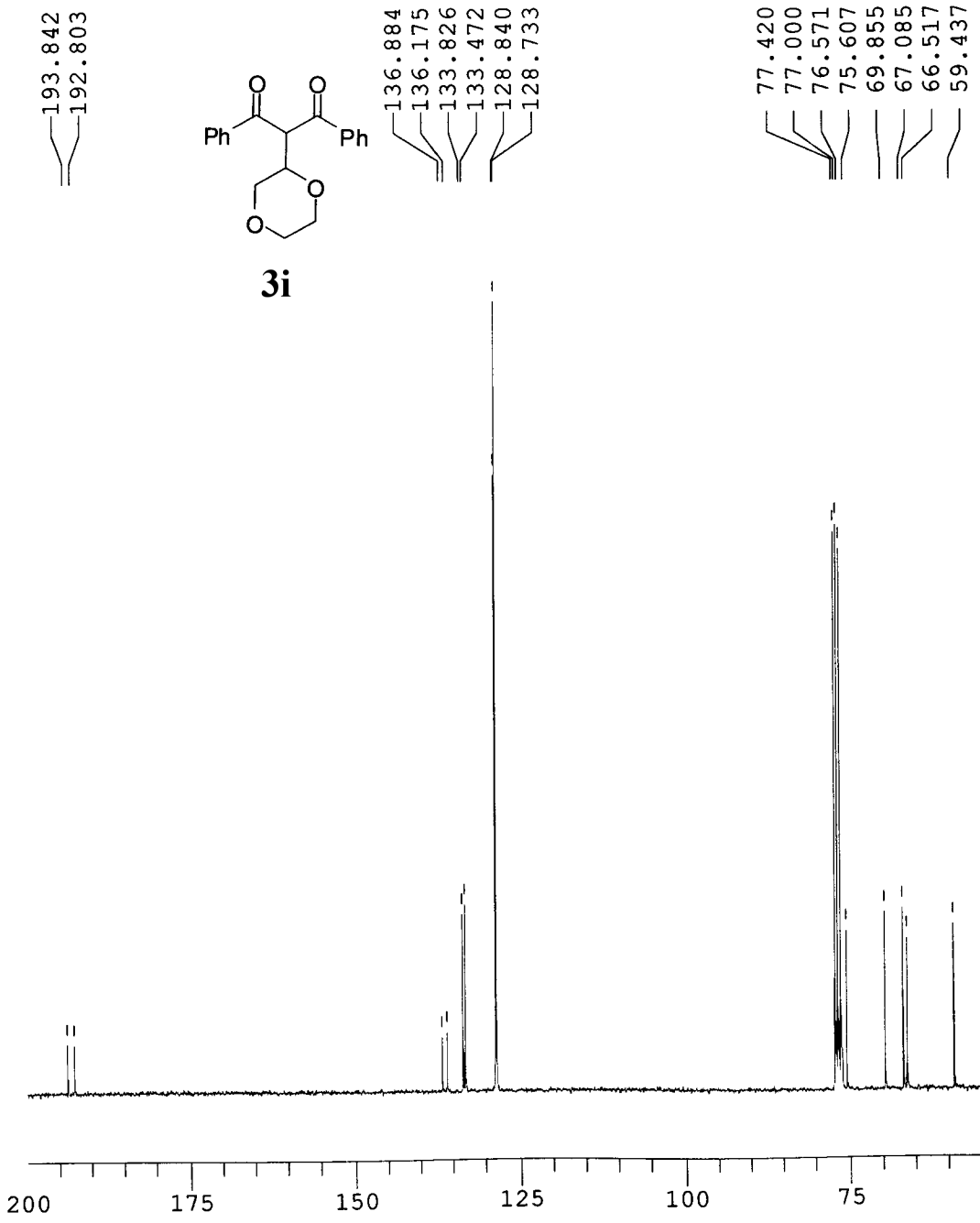
DFILE D:\YuRong\2008\4-C.als
COMNT
DATIM Sat Dec 01 18:36:51 2007
OBNUC 13C
EXMOD BCM
OBFRQ 75.45 MHz
OBSET 124.00 KHz
OBFIN 1840.0 Hz
POINT 32768
FREQU 20408.1 Hz
SCANS 500
ACQTM 1.606 sec
PD 1.394 sec
PW1 5.0 us
IRNUC 1H
CTEMP 22.0 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.62 Hz
RGAIN 26



3i

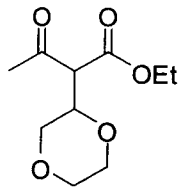



```
DFILE   D:\YuRong\2008\3-H.als
COMNT
DATIM   Wed Nov 28 14:46:26 2007
OBNUC   1H
EXMOD   NON
OBFRQ         300.40 MHz
OBSET         130.00 KHz
OBFIN         1150.0 Hz
POINT          32768
FREQU         6013.2 Hz
SCANS              4
ACQTM          5.449 sec
PD             1.551 sec
PW1              5.8 us
IRNUC   1H
CTEMP           22.7 c
SLVNT   CDCL3
EXREF          0.00 ppm
BF             0.09 Hz
RGAIN            21
```



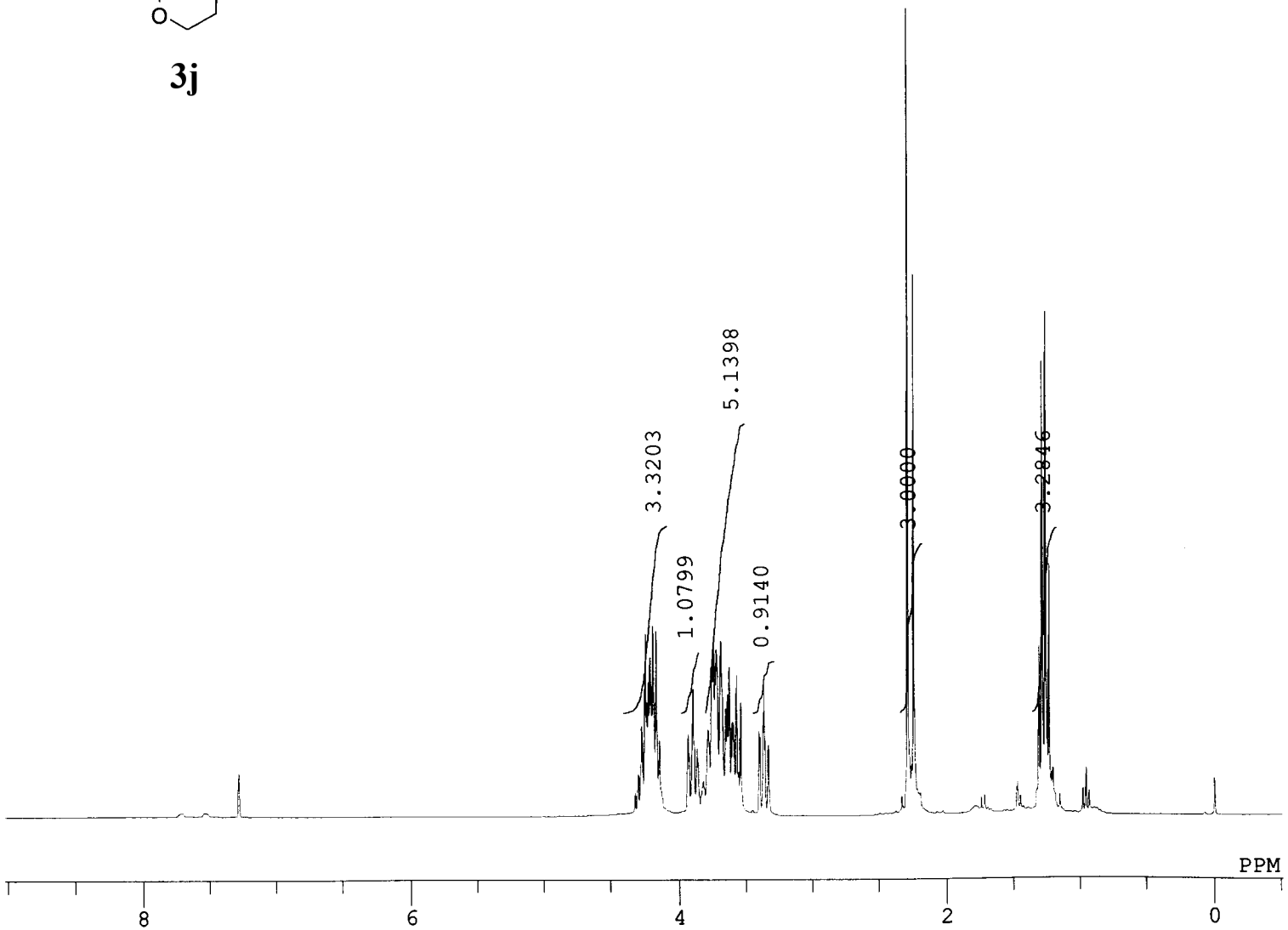
3i

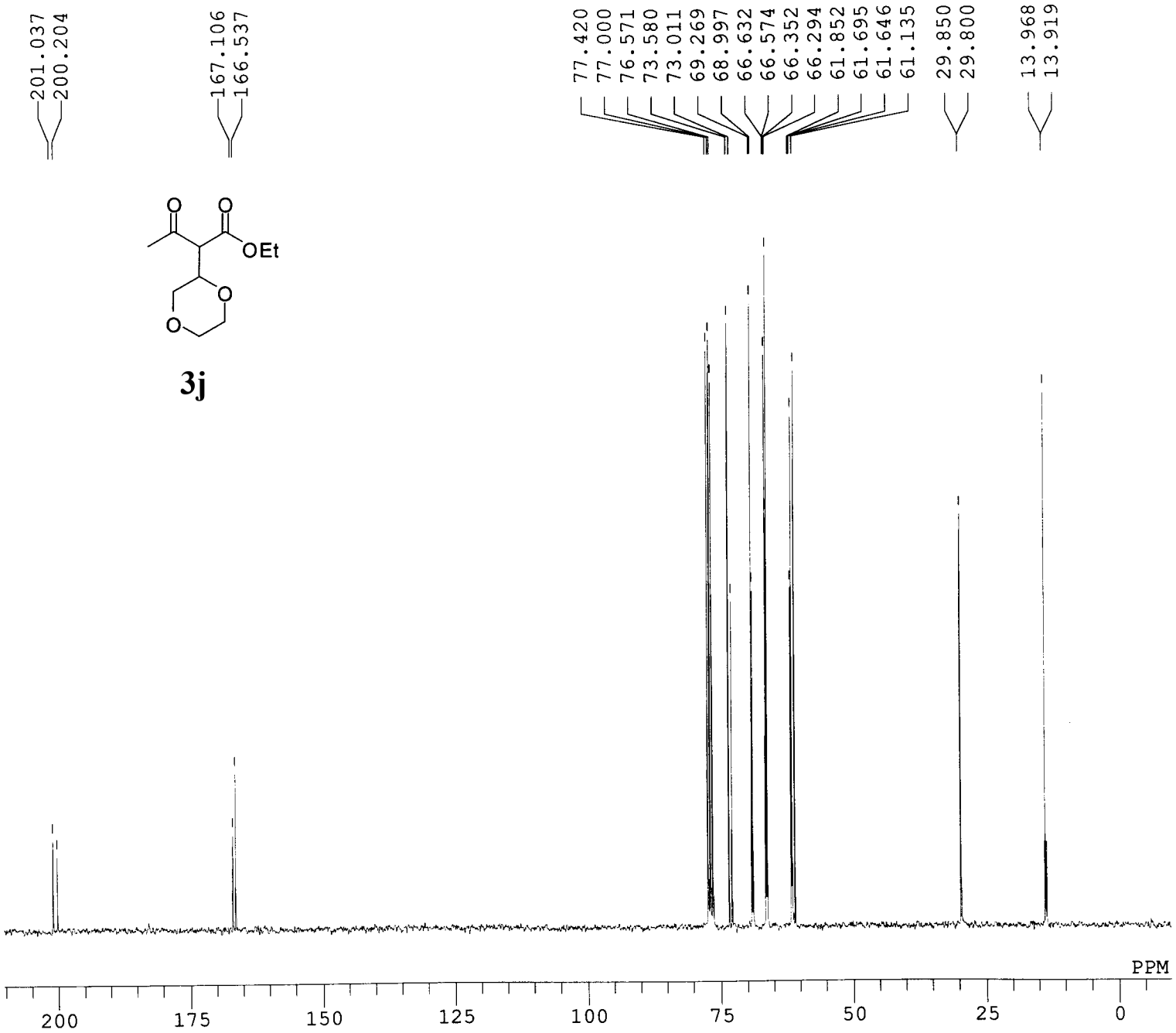
```
DFILE   D:\YuRong\2008\3-C.als
COMNT
DATIM   Wed Nov 28 14:42:12 2007
OBNUC   13C
EXMOD   BCM
OBFRQ         75.45 MHz
OBSET        124.00 KHz
OBFIN        1840.0 Hz
POINT         32768
FREQU       20408.1 Hz
SCANS          1640
ACQTM         1.606 sec
PD            1.394 sec
PW1             5.0 us
IRNUC   1H
CTEMP          22.9 c
SLVNT   CDCL3
EXREF         77.00 ppm
BF             0.62 Hz
RGAIN            26
```



3j

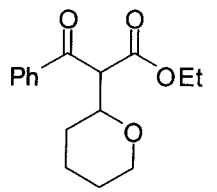
DFILE	D:\YuRong\2008\17-H.als
COMNT	
DATIM	Wed Apr 16 07:53:17 2008
OBNUC	1H
EXMOD	NON
OBFRQ	300.40 MHz
OBSET	130.00 KHz
OBFIN	1150.0 Hz
POINT	32768
FREQU	6013.2 Hz
SCANS	16
ACQTM	5.449 sec
PD	1.551 sec
PW1	5.8 us
IRNUC	1H
CTEMP	23.3 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.09 Hz
RGAIN	15





3j

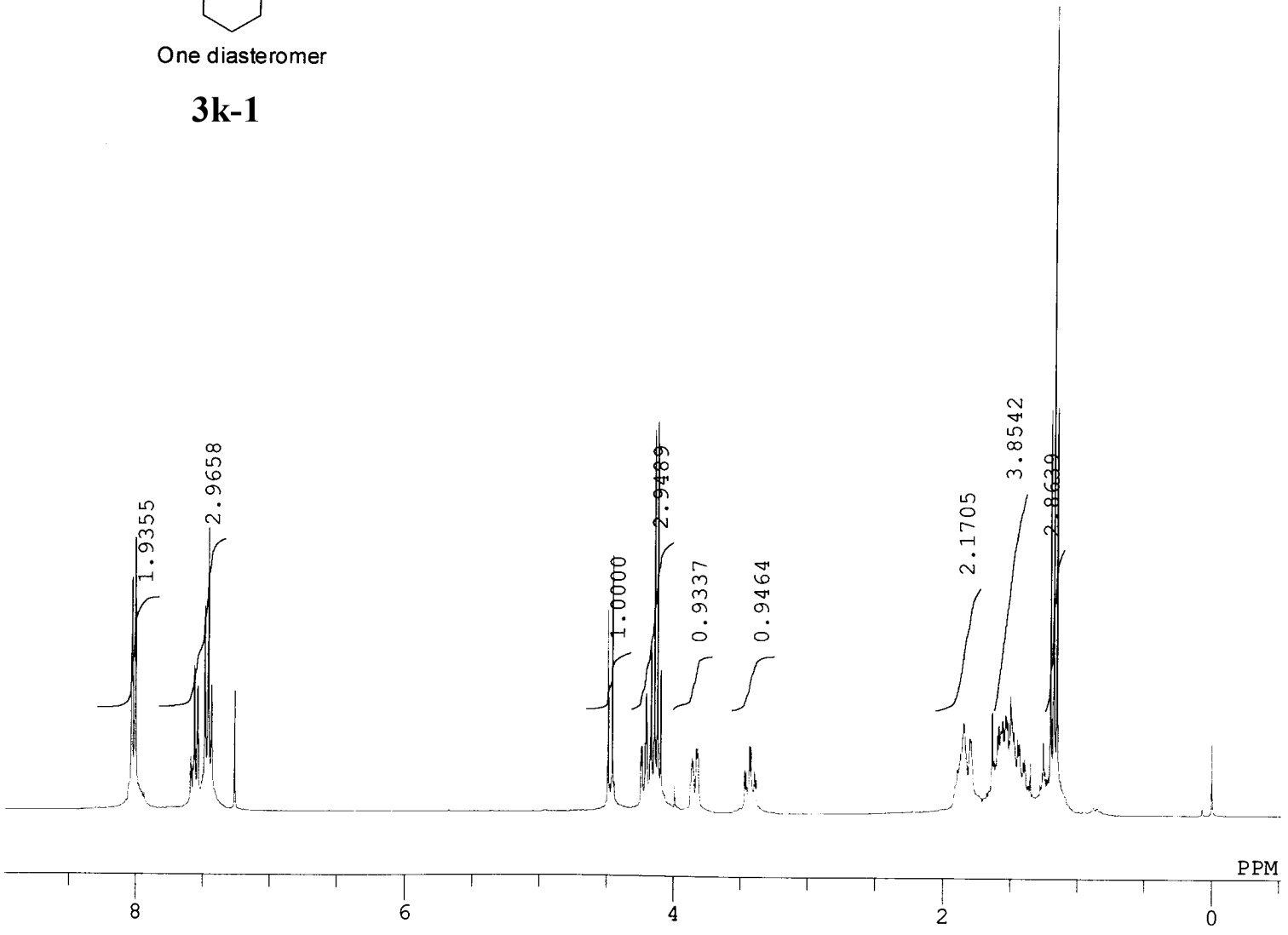
DFILE	D:\YuRong\2008\17-C.als
COMNT	
DATIM	Wed Apr 16 08:02:14 2008
OBNUC	13C
EXMOD	BCM
OBFRQ	75.45 MHz
OBSET	124.00 KHz
OBFIN	1840.0 Hz
POINT	32768
FREQU	20408.1 Hz
SCANS	160
ACQTM	1.606 sec
PD	1.394 sec
PW1	5.0 us
IRNUC	1H
CTEMP	23.5 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.62 Hz
RGAIN	26

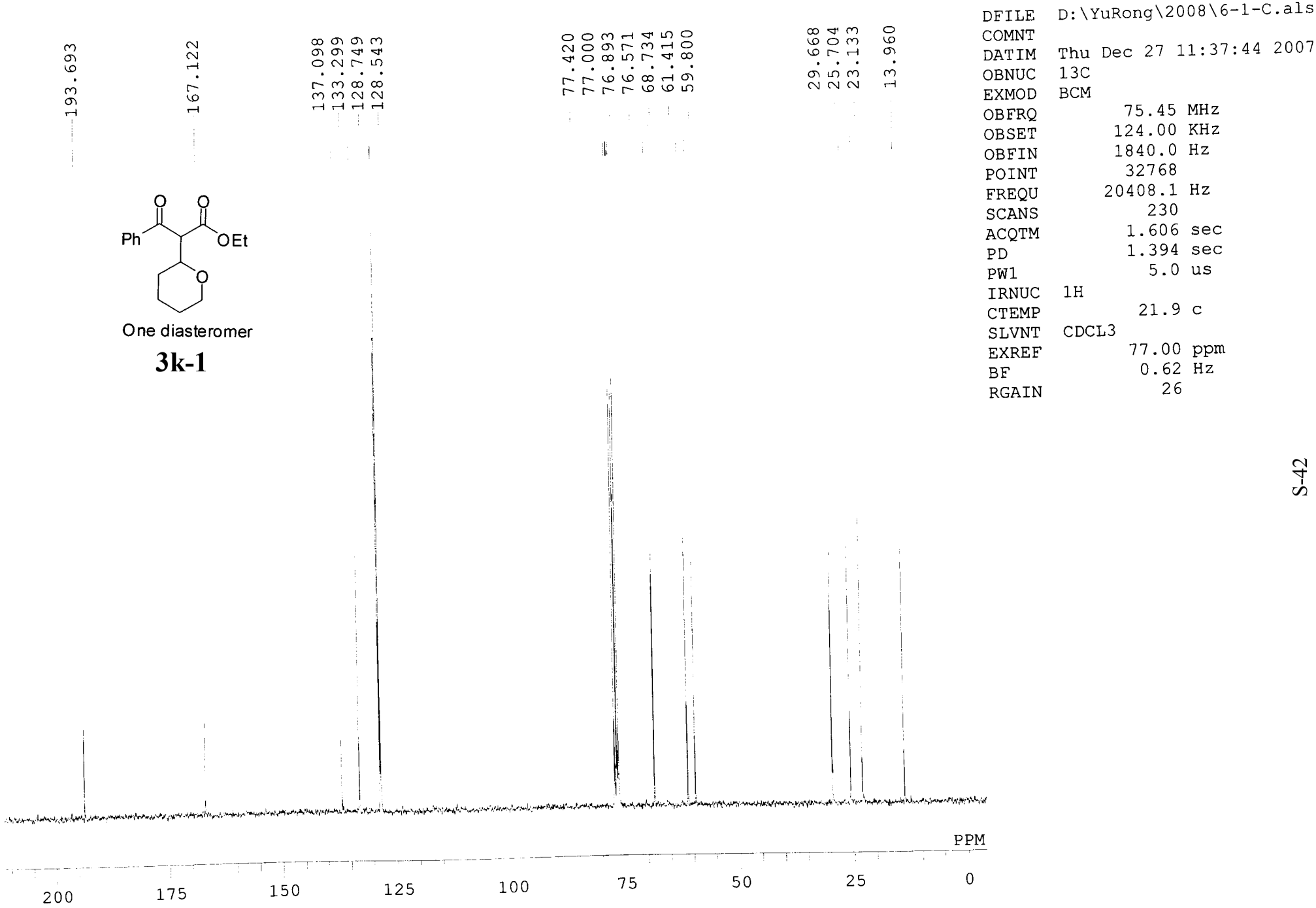


One diasteromer

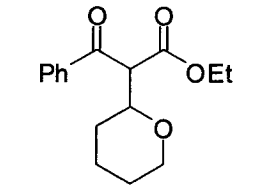
3k-1

```
DFILE   D:\YuRong\2008\6-1-H.als
COMNT
DATIM   Thu Dec 27 11:24:32 2007
OBNUC   1H
EXMOD   NON
OBFRQ         300.40 MHz
OBSET         130.00 KHz
OBFIN         1150.0 Hz
POINT          32768
FREQU         6013.2 Hz
SCANS             12
ACQTM          5.449 sec
PD             1.551 sec
PW1              5.8 us
IRNUC   1H
CTEMP           21.5 c
SLVNT   CDCL3
EXREF           0.00 ppm
BF              0.12 Hz
RGAIN             19
```





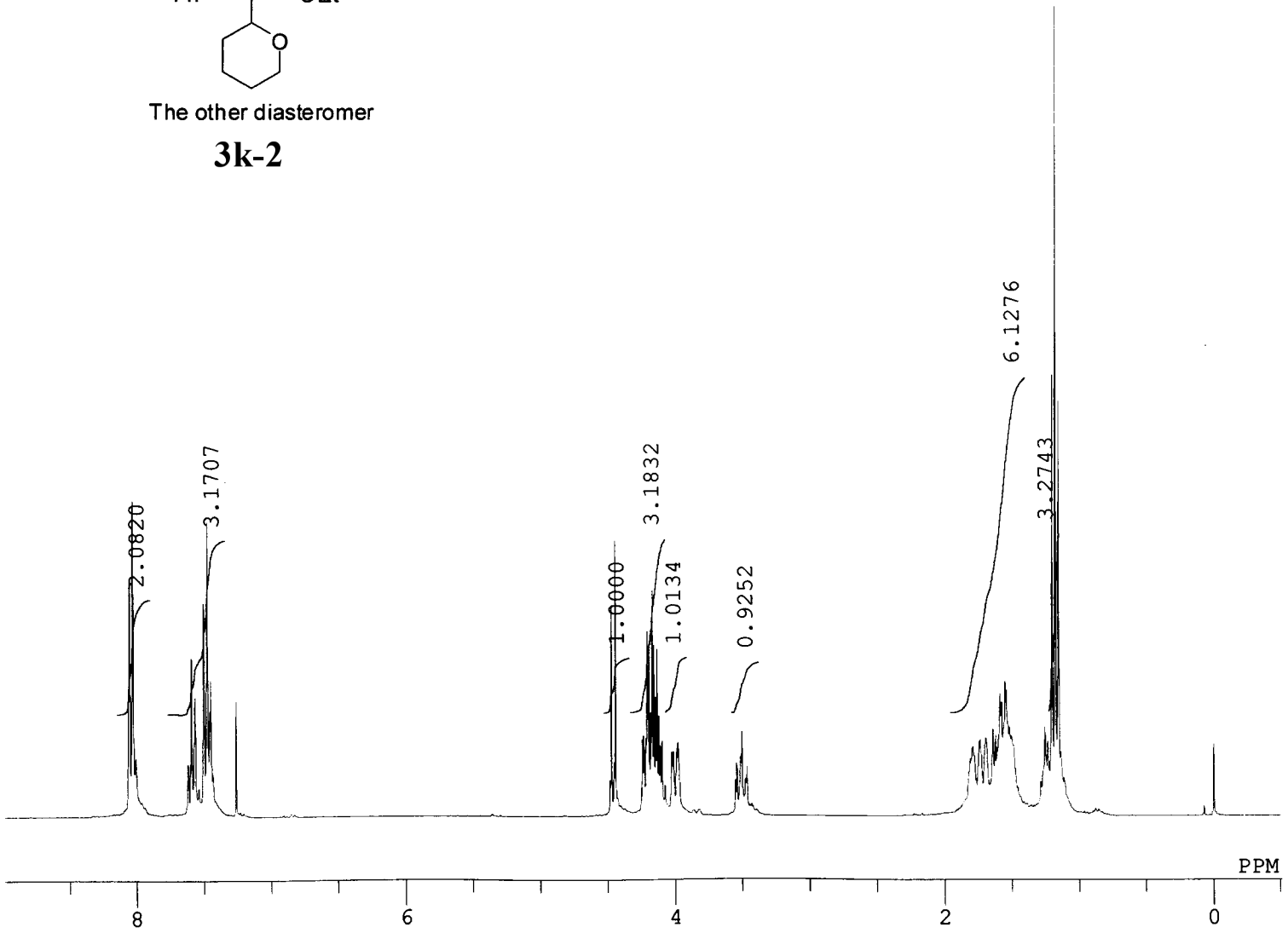

```
DFILE   D:\YuRong\2008\6-1-C.als
COMNT
DATIM   Thu Dec 27 11:37:44 2007
OBNUC   13C
EXMOD   BCM
OBFRQ         75.45 MHz
OBSET        124.00 KHz
OBFIN        1840.0 Hz
POINT         32768
FREQU       20408.1 Hz
SCANS           230
ACQTM         1.606 sec
PD            1.394 sec
PW1             5.0 us
IRNUC   1H
CTEMP          21.9 c
SLVNT   CDCL3
EXREF         77.00 ppm
BF             0.62 Hz
RGAIN            26
```

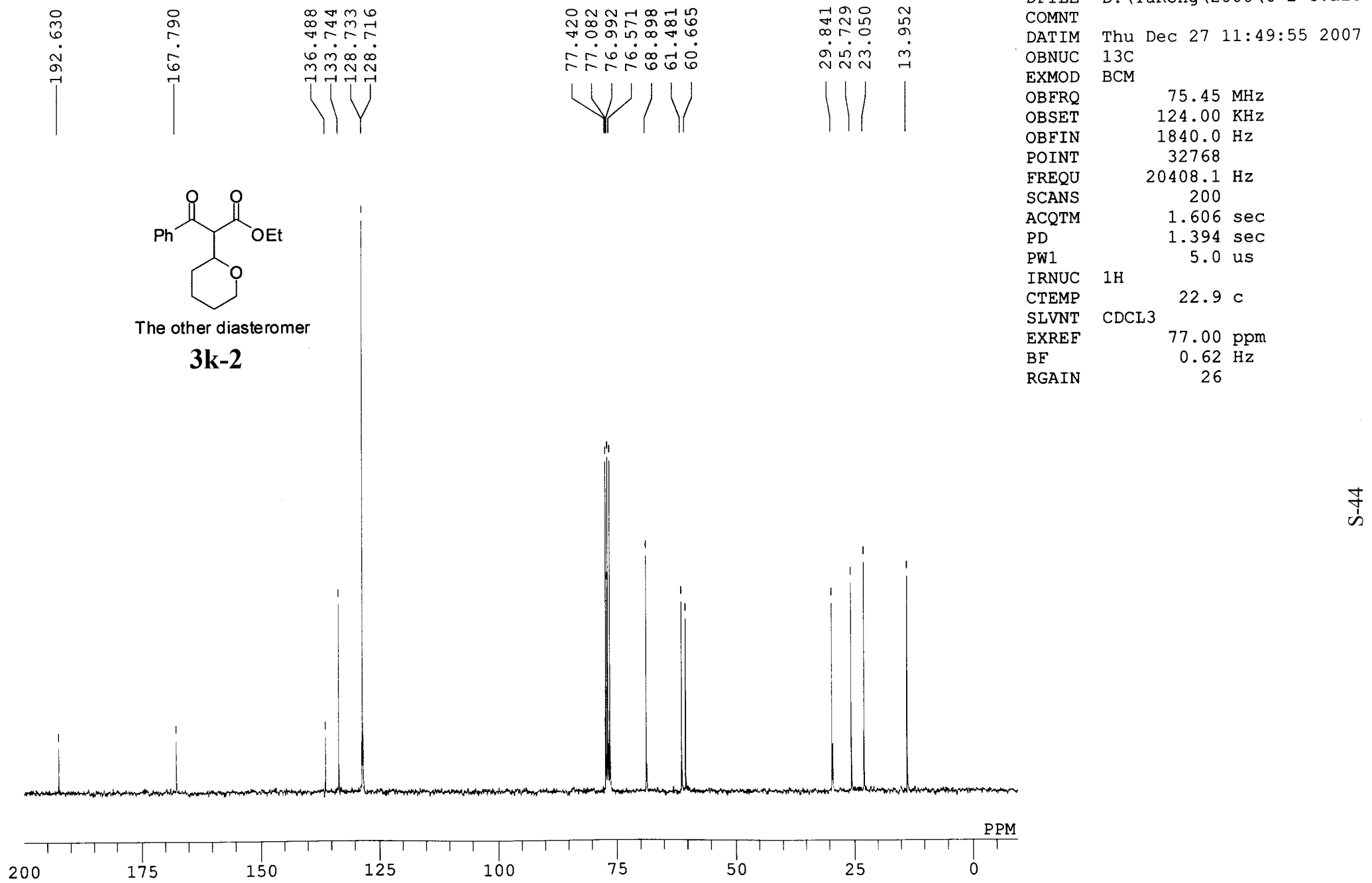


The other diasteromer

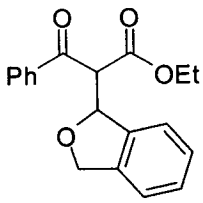
3k-2

```
DFILE   D:\YuRong\2008\6-2-H.als
COMNT
DATIM   Thu Dec 27 11:51:30 2007
OBNUC   1H
EXMOD   NON
OBFRQ         300.40 MHz
OBSET         130.00 KHz
OBFIN         1150.0 Hz
POINT          32768
FREQU         6013.2 Hz
SCANS             4
ACQTM          5.449 sec
PD             1.551 sec
PW1              5.8 us
IRNUC   1H
CTEMP           22.7 c
SLVNT   CDCL3
EXREF          0.00 ppm
BF             0.09 Hz
RGAIN            17
```



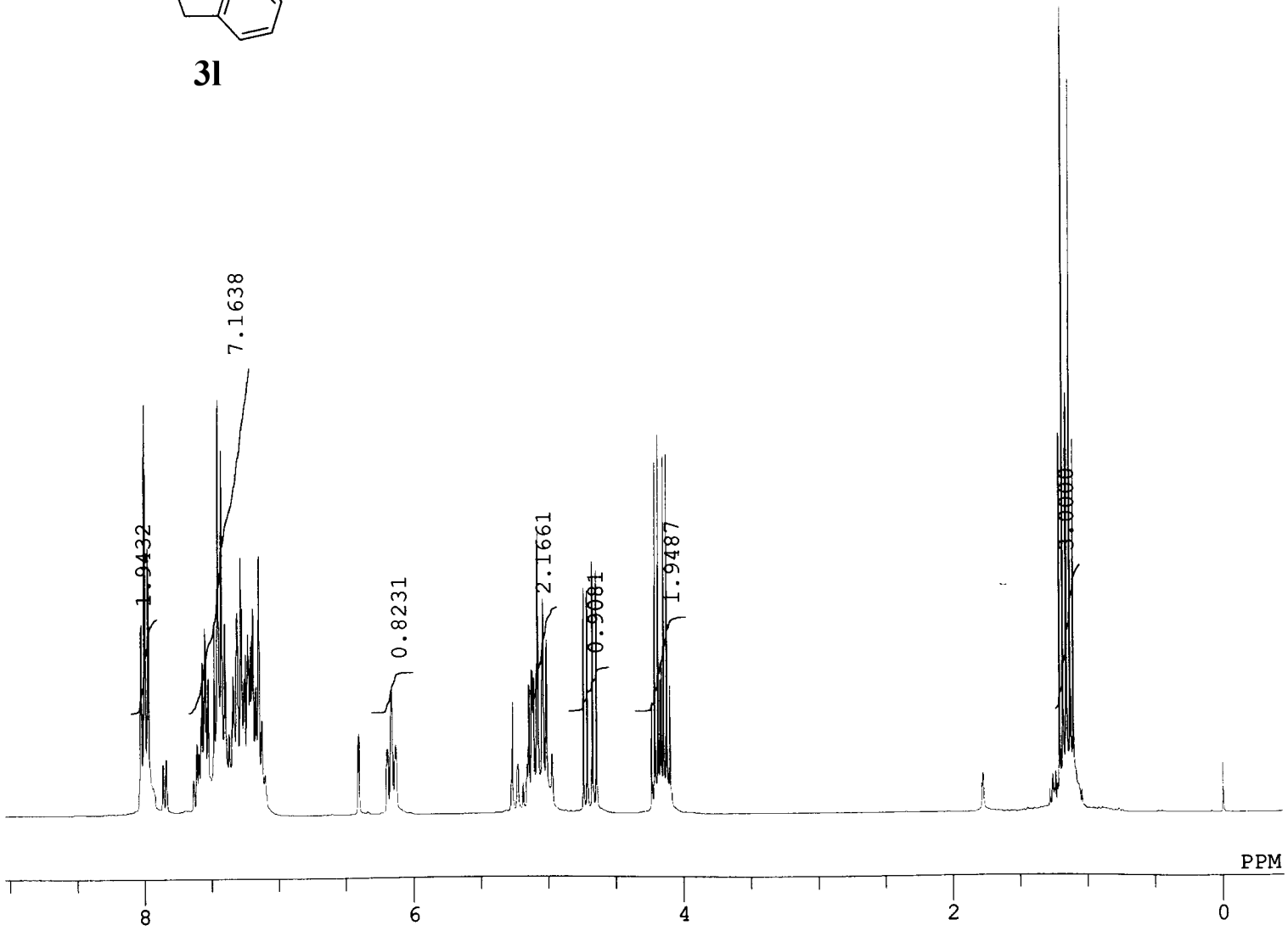


DFILE	D:\YuRong\2008\6-2-C.als
COMNT	
DATIM	Thu Dec 27 11:49:55 2007
OBNUC	13C
EXMOD	BCM
OBFRQ	75.45 MHz
OBSET	124.00 KHz
OBFIN	1840.0 Hz
POINT	32768
FREQU	20408.1 Hz
SCANS	200
ACQTM	1.606 sec
PD	1.394 sec
PW1	5.0 us
IRNUC	1H
CTEMP	22.9 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.62 Hz
RGAIN	26

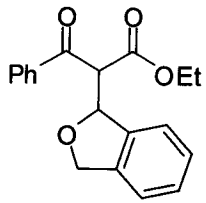


3l

```
DFILE   D:\YuRong\2008\15-H.als
COMNT
DATIM   Sun Mar 23 16:55:39 2008
OBNUC   1H
EXMOD   NON
OBFRQ         300.40 MHz
OBSET         130.00 KHz
OBFIN         1150.0 Hz
POINT          32768
FREQU         6013.2 Hz
SCANS             16
ACQTM          5.449 sec
PD             1.551 sec
PW1              5.8 us
IRNUC   1H
CTEMP           23.7 c
SLVNT   CDCL3
EXREF           0.00 ppm
BF              0.09 Hz
RGAIN             14
```

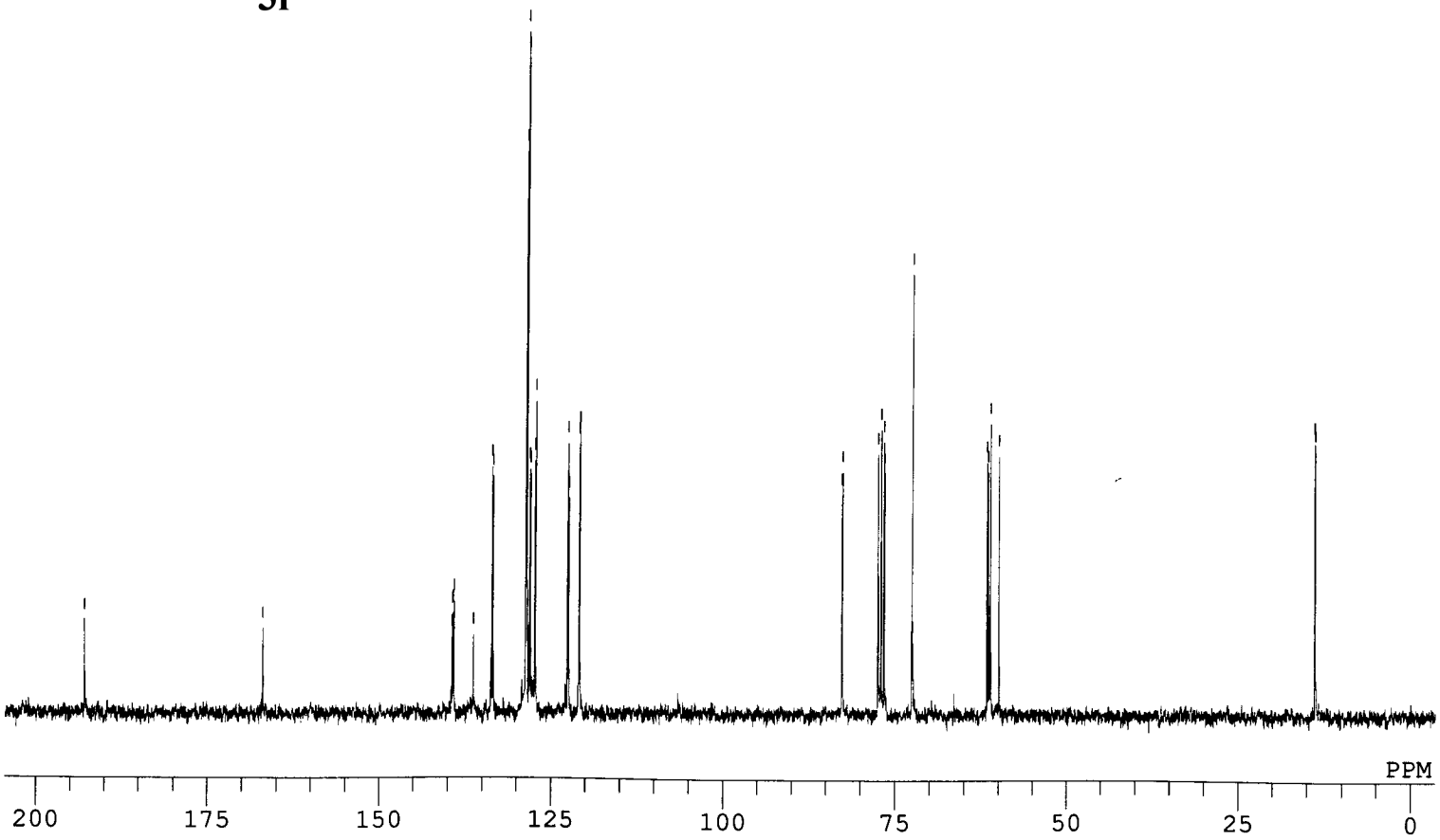


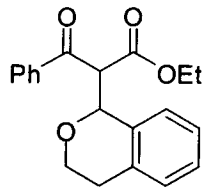
192.993
192.968
167.032
139.505
139.364
139.274
139.183
136.373
133.768
133.529
128.799
128.774
128.692
128.659
128.230
128.115
127.455
127.414
122.650
122.510
121.019
120.953
82.777
82.728
77.470
77.049
76.621
72.632
72.459
61.736
61.522
61.242
59.998
13.943
13.894



3l

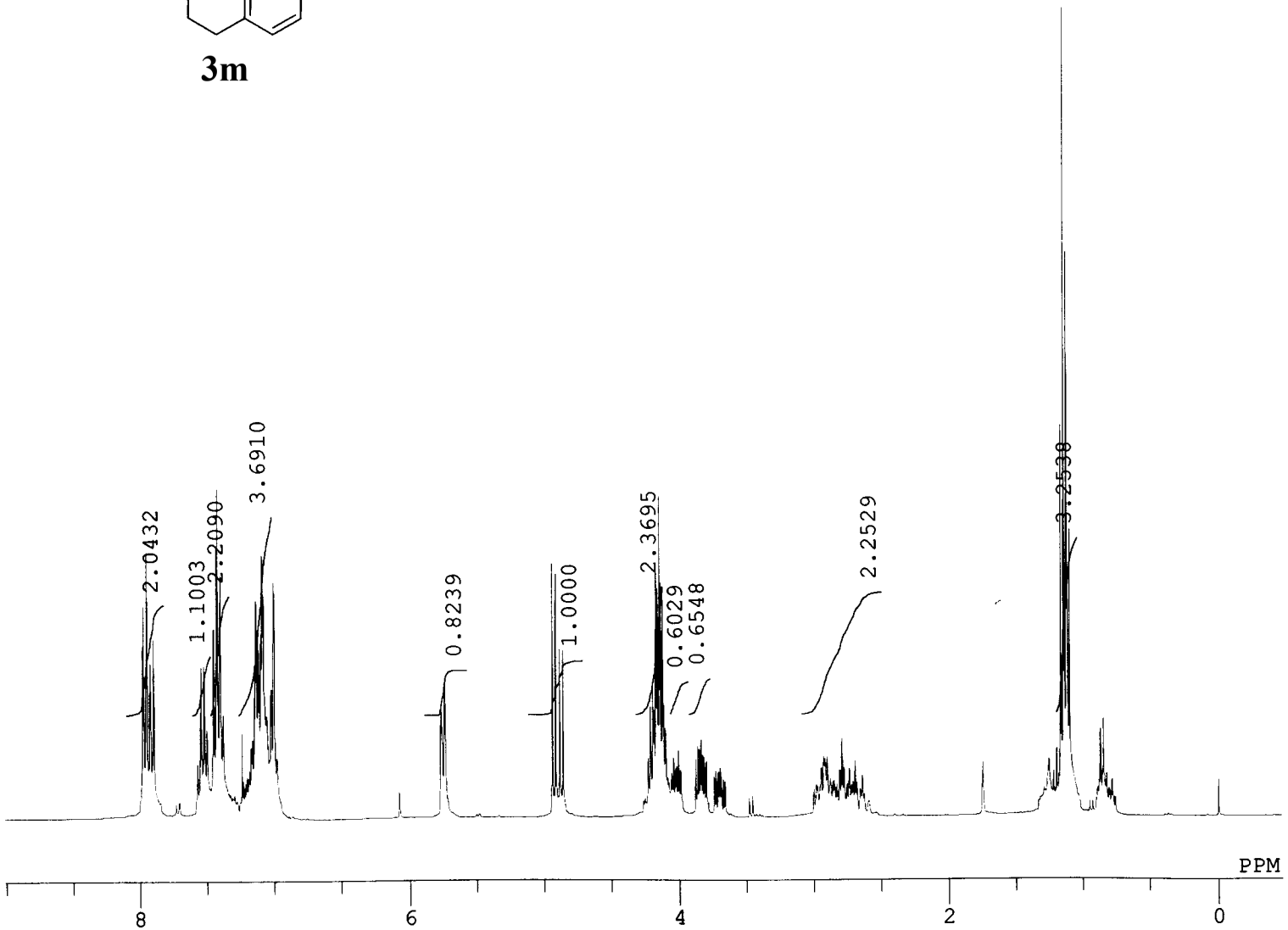
DFILE	D:\YuRong\2008\15-C.als
COMNT	
DATIM	Wed Apr 16 12:50:58 2008
OBNUC	13C
EXMOD	BCM
OBFRQ	75.45 MHz
OBSET	124.00 KHz
OBFIN	1840.0 Hz
POINT	32768
FREQU	20408.1 Hz
SCANS	140
ACQTM	1.606 sec
PD	1.394 sec
PW1	5.0 us
IRNUC	1H
CTEMP	24.1 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.62 Hz
RGAIN	26

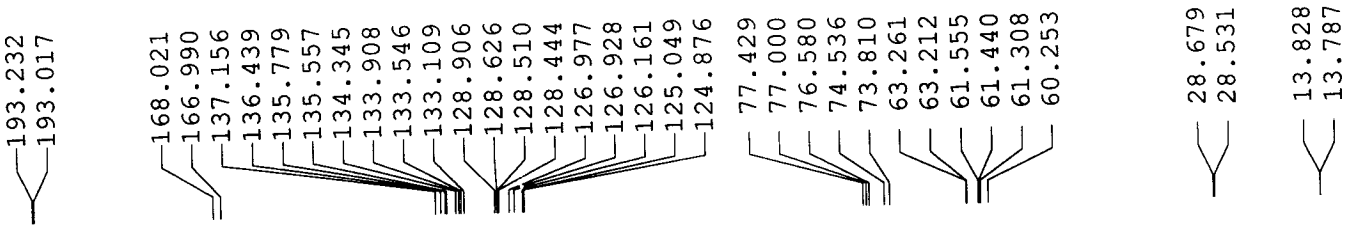


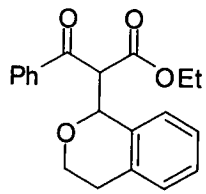


3m

```
DFILE   D:\YuRong\2008\16-H.als
COMNT
DATIM   Sun Mar 23 15:09:38 2008
OBNUC   1H
EXMOD   NON
OBFRQ         300.40 MHz
OBSET         130.00 KHz
OBFIN         1150.0 Hz
POINT          32768
FREQU         6013.2 Hz
SCANS             16
ACQTM          5.449 sec
PD             1.551 sec
PW1              5.8 us
IRNUC   1H
CTEMP           23.5 c
SLVNT   CDCL3
EXREF          0.00 ppm
BF             0.09 Hz
RGAIN             14
```

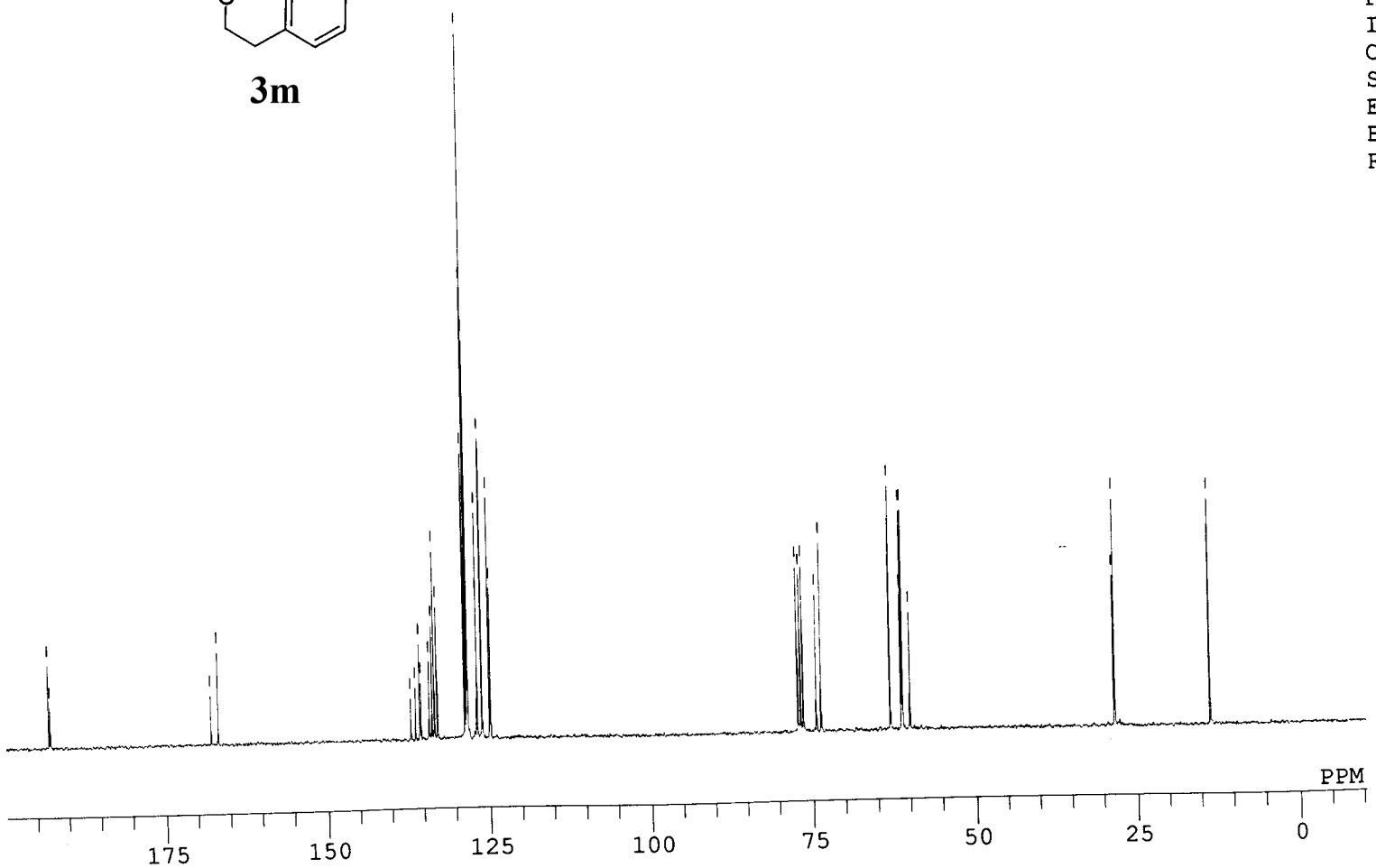


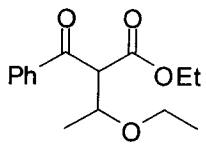




3m

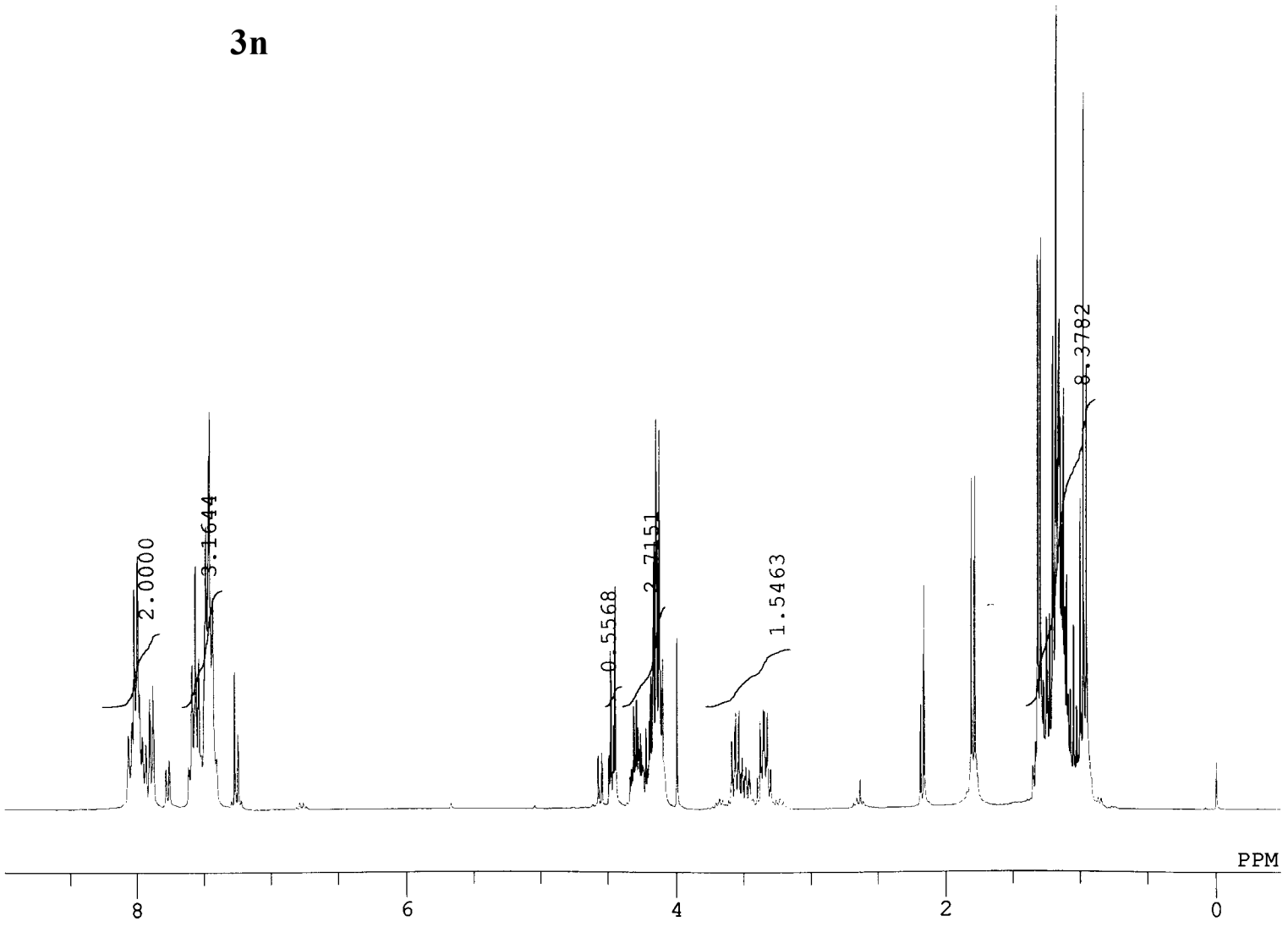
```
DFILE   D:\YuRong\2008\16-C.als
COMNT
DATIM   Sun Mar 23 17:07:46 2008
OBNUC   13C
EXMOD   BCM
OBFRQ         75.45 MHz
OBSET        124.00 KHz
OBFIN        1840.0 Hz
POINT         32768
FREQU       20408.1 Hz
SCANS           200
ACQTM         1.606 sec
PD            1.394 sec
PW1             5.0 us
IRNUC   1H
CTEMP          23.9 c
SLVNT   CDCL3
EXREF         77.00 ppm
BF             0.62 Hz
RGAIN            28
```

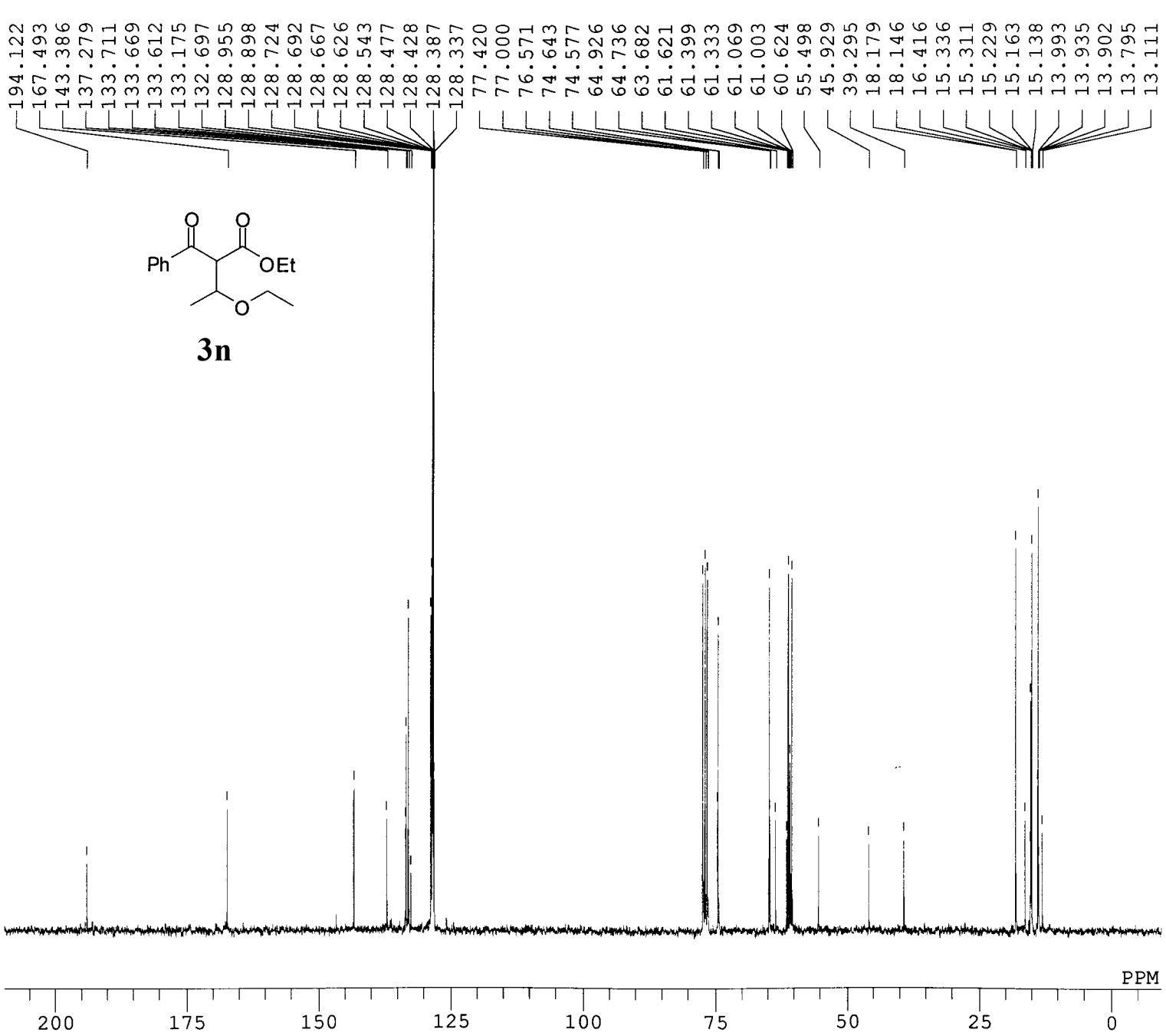




3n

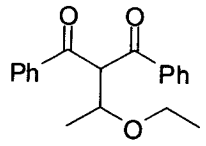
```
DFILE   D:\YuRong\2008\10-H.als
COMNT
DATIM   Sun May 04 21:57:47 2008
OBNUC   1H
EXMOD   NON
OBFRQ         300.40 MHz
OBSET         130.00 KHz
OBFIN         1150.0 Hz
POINT          32768
FREQU         6013.2 Hz
SCANS             16
ACQTM          5.449 sec
PD             1.551 sec
PW1              5.8 us
IRNUC   1H
CTEMP           22.7 c
SLVNT   CDCL3
EXREF          0.00 ppm
BF             0.12 Hz
RGAIN             15
```





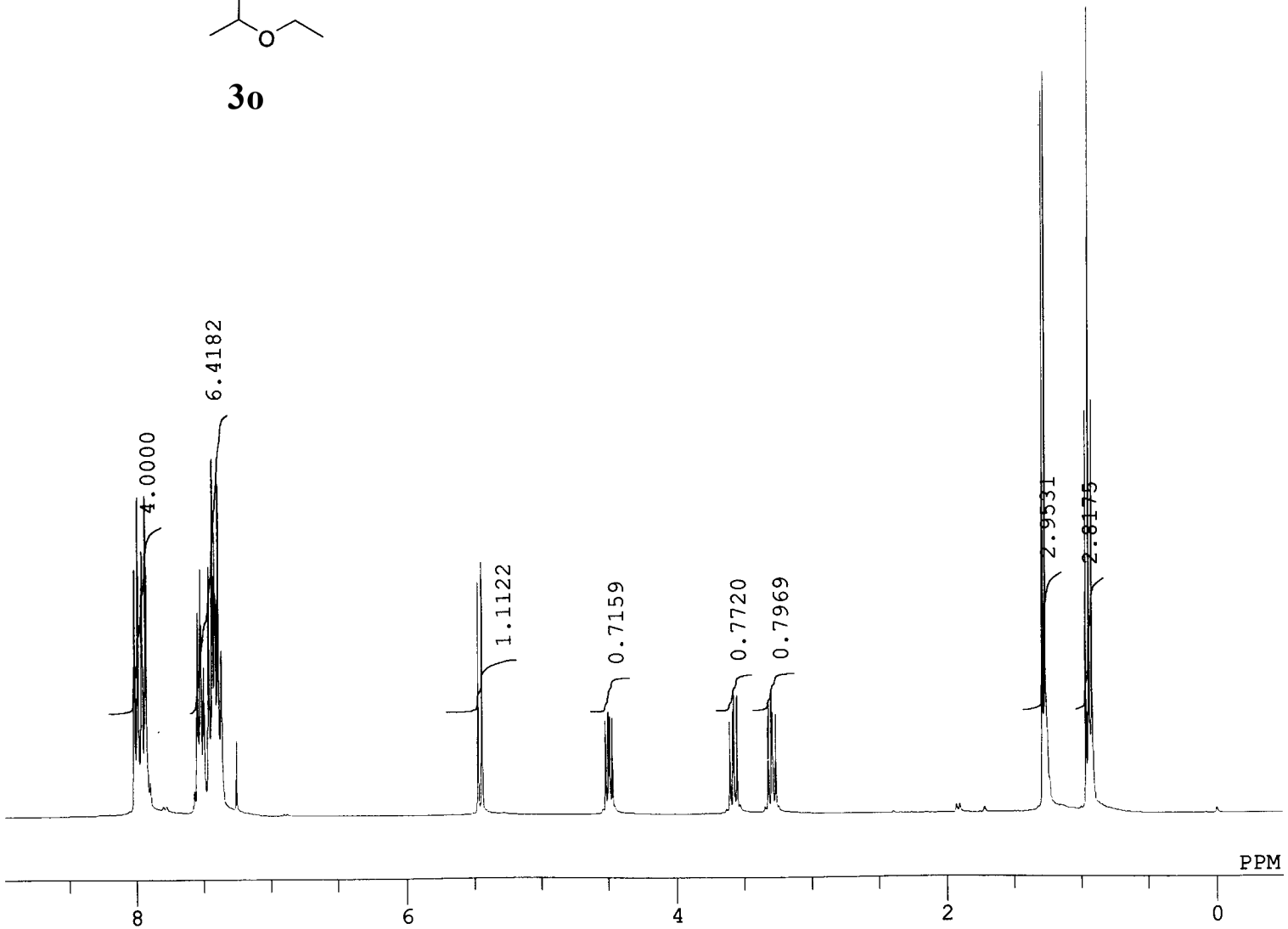
3n

DFILE	D:\YuRong\2008\10-C.als
COMNT	
DATIM	Sun May 04 21:54:31 2008
OBNUC	13C
EXMOD	BCM
OBFRQ	75.45 MHz
OBSET	124.00 KHz
OBFIN	1840.0 Hz
POINT	32768
FREQU	20408.1 Hz
SCANS	340
ACQTM	1.606 sec
PD	1.394 sec
PW1	5.0 us
IRNUC	1H
CTEMP	23.0 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.62 Hz
RGAIN	26



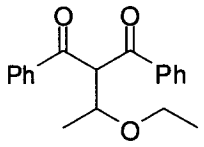
3o

DFILE	D:\YuRong\2008\7-H.als
COMNT	1
DATIM	Mon Dec 17 18:28:43 2007
OBNUC	1H
EXMOD	NON
OBFRQ	300.40 MHz
OBSET	130.00 KHz
OBFIN	1150.0 Hz
POINT	32768
FREQU	6013.2 Hz
SCANS	16
ACQTM	5.449 sec
PD	1.551 sec
PW1	5.8 us
IRNUC	1H
CTEMP	22.7 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	15

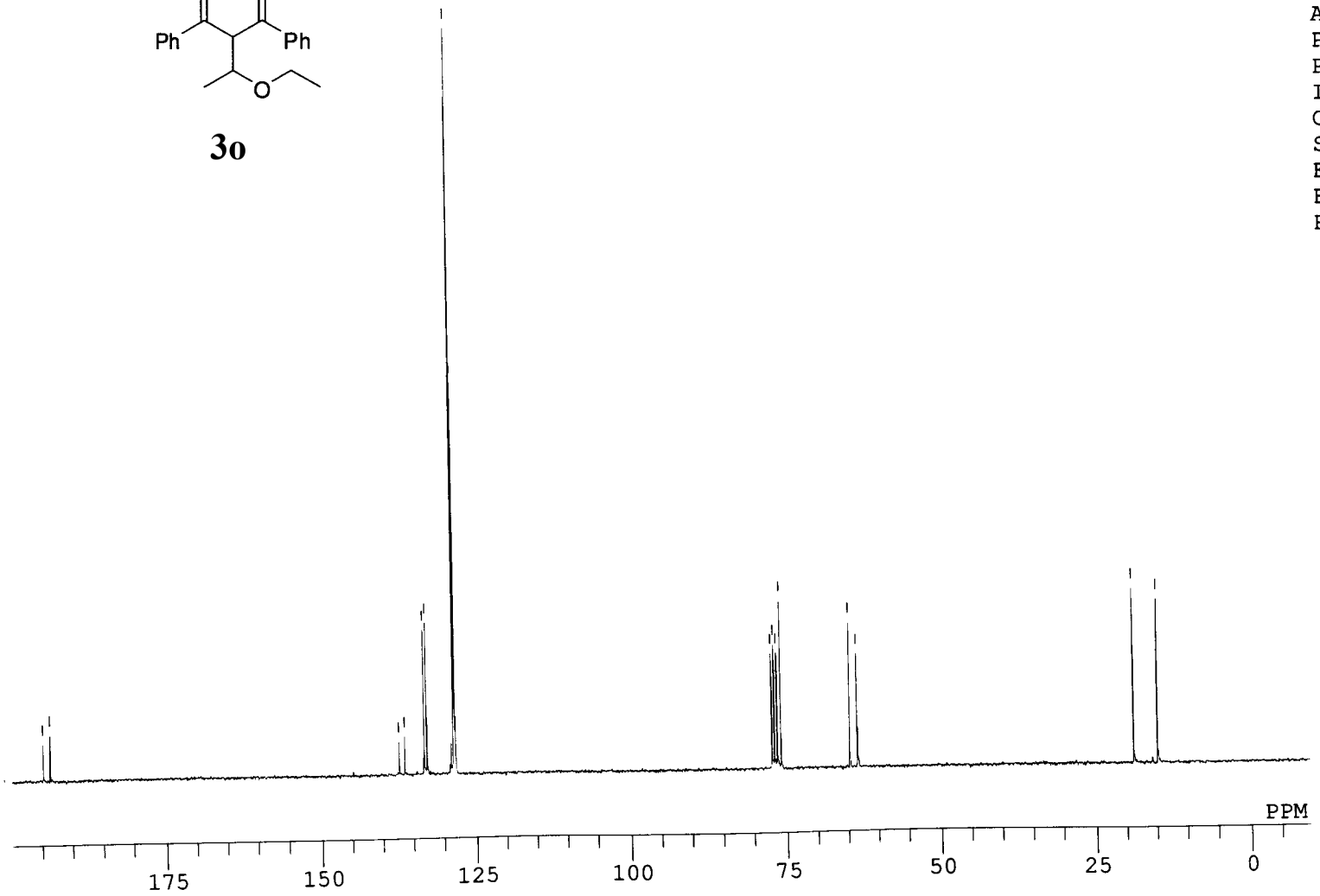


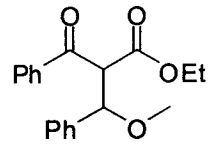
194.913
193.792
137.469
136.579
133.521
133.051
128.741
128.667
128.535
77.420
77.000
76.580
75.995
64.918
63.616
18.962
15.081

DFILE D:\YuRong\2008\7-C.als
COMNT 1
DATIM Mon Dec 17 18:21:22 2007
OBNUC 13C
EXMOD BCM
OBFRQ 75.45 MHz
OBSET 124.00 KHz
OBFIN 1840.0 Hz
POINT 32768
FREQU 20408.1 Hz
SCANS 260
ACQTM 1.606 sec
PD 1.394 sec
PW1 5.0 us
IRNUC 1H
CTEMP 22.9 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.62 Hz
RGAIN 26



3o

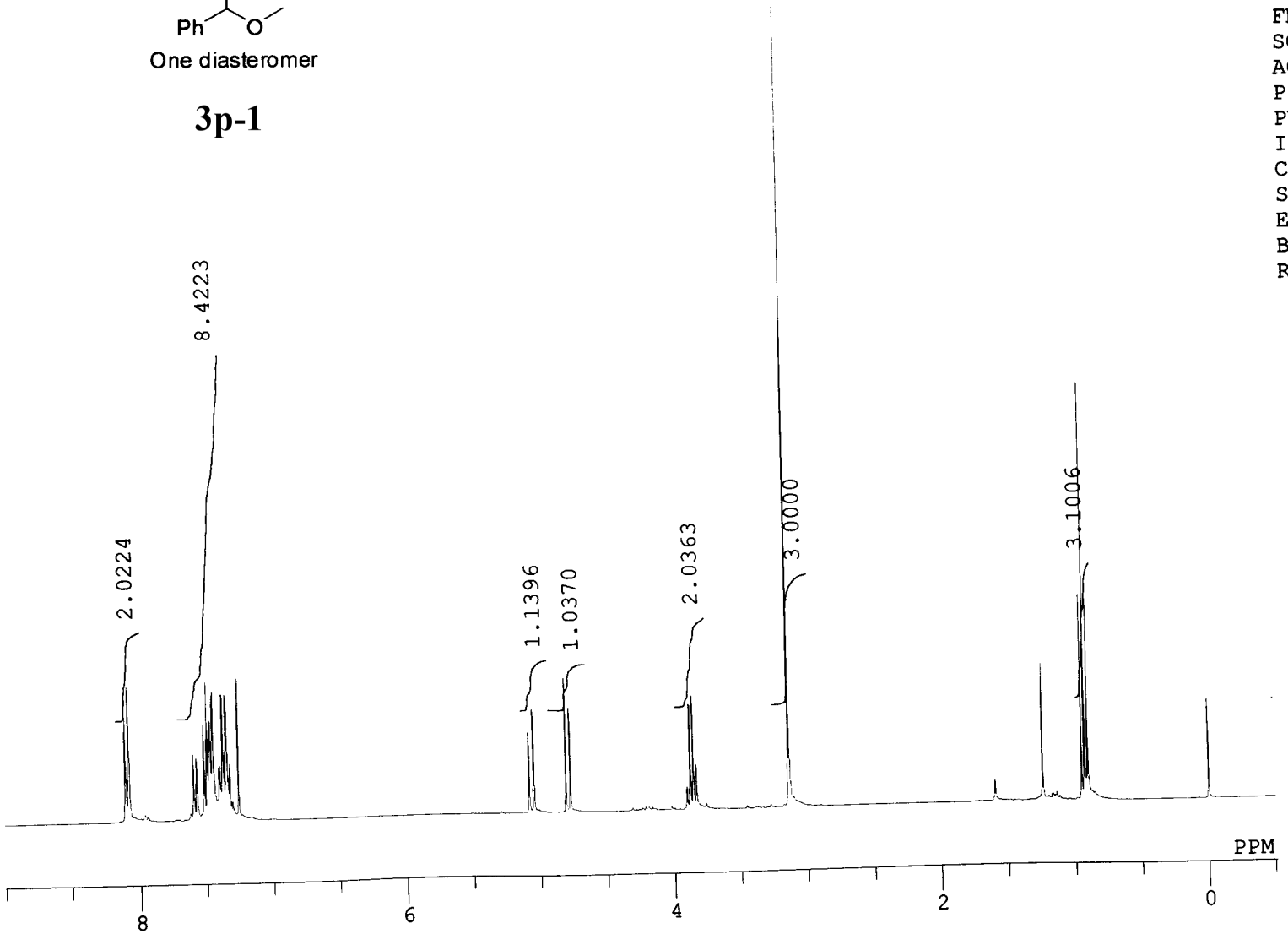


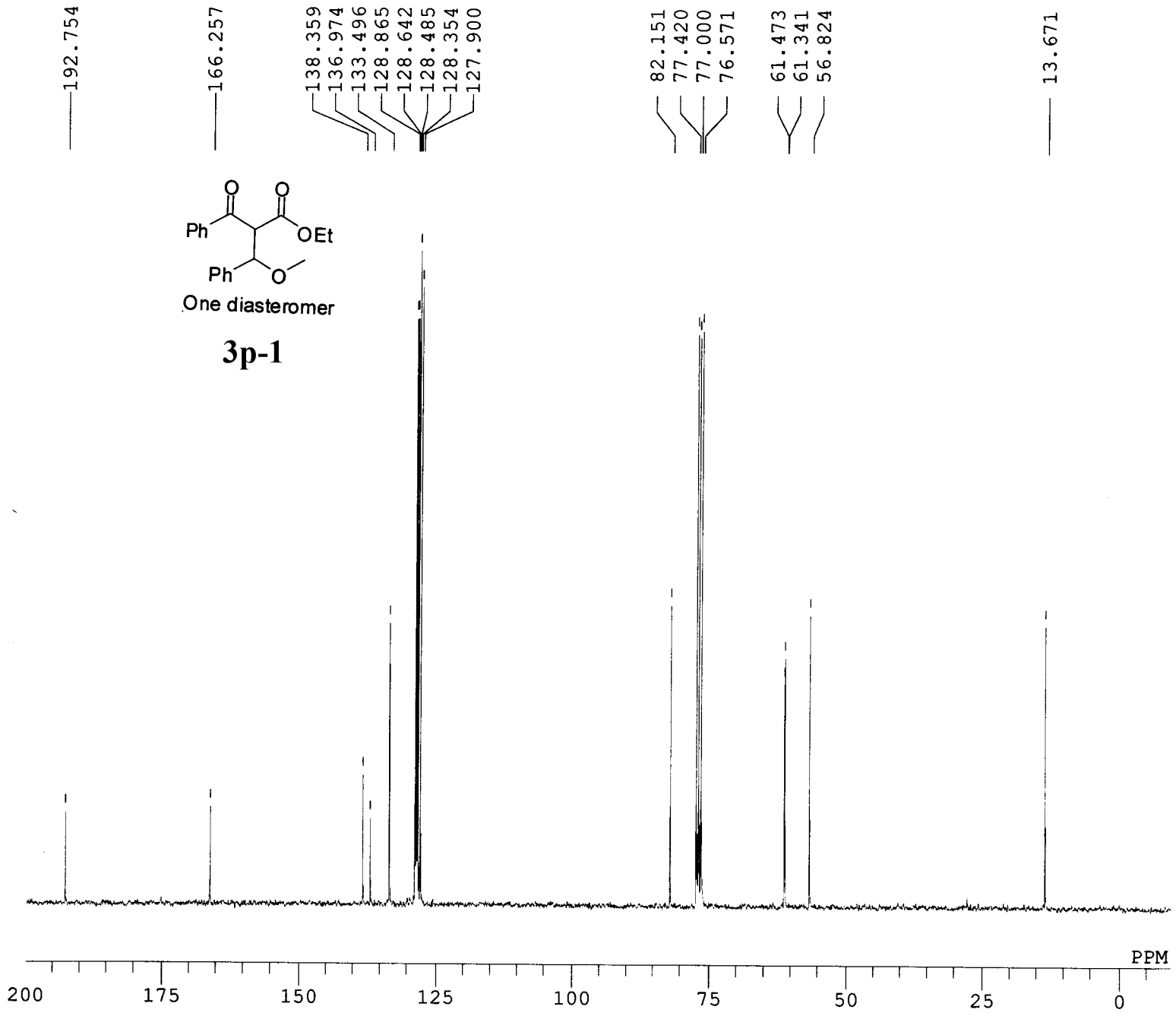


One diasteromer

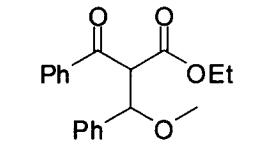
3p-1

```
DFILE   D:\YuRong\2008\12-1H.als
COMNT
DATIM   Sat Apr 26 22:29:17 2008
OBNUC   1H
EXMOD   NON
OBFRQ        300.40 MHz
OBSET        130.00 KHz
OBFIN        1150.0 Hz
POINT         32768
FREQU        6013.2 Hz
SCANS            16
ACQTM         5.449 sec
PD            1.551 sec
PW1             5.8 us
IRNUC   1H
CTEMP          23.1 c
SLVNT   CDCL3
EXREF          0.00 ppm
BF             0.09 Hz
RGAIN            20
```





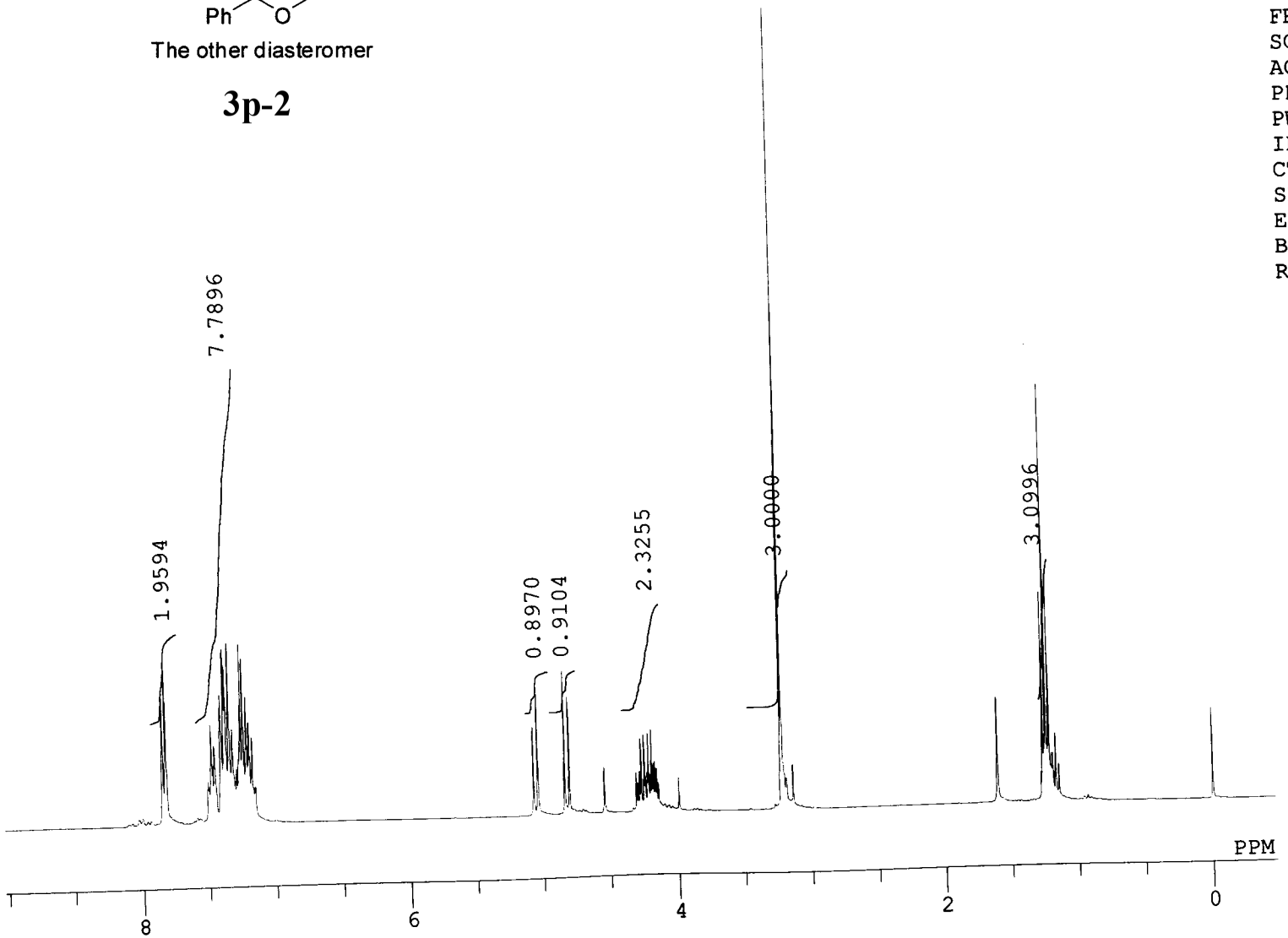
DFILE	D:\YuRong\2008\12-1C.als
COMNT	
DATIM	Sat Apr 26 22:24:57 2008
OBNUC	13C
EXMOD	BCM
OBFRQ	75.45 MHz
OBSET	124.00 KHz
OBFIN	1840.0 Hz
POINT	32768
FREQU	20408.1 Hz
SCANS	800
ACQTM	1.606 sec
PD	1.394 sec
PW1	5.0 us
IRNUC	1H
CTEMP	23.5 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.62 Hz
RGAIN	26

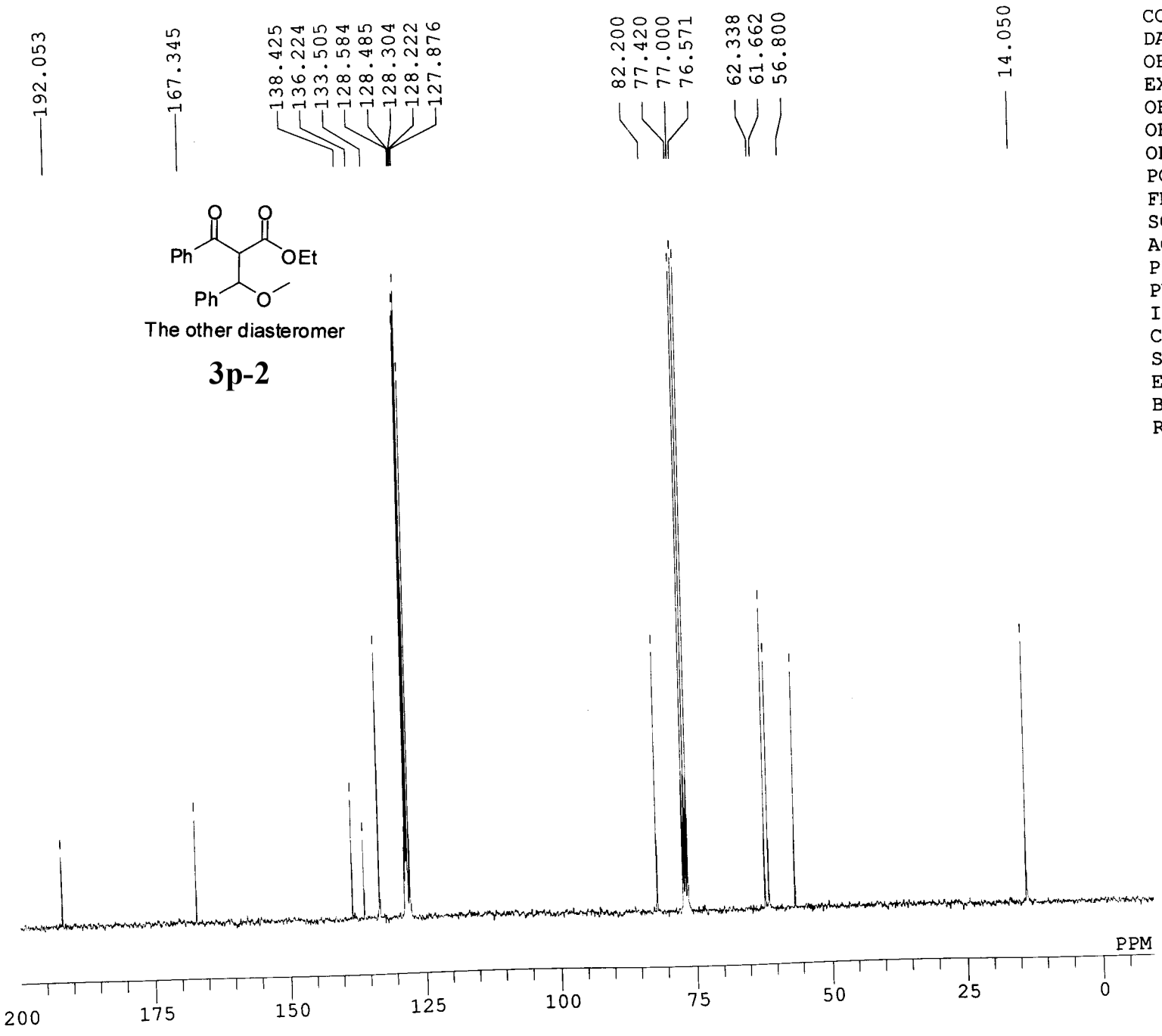


The other diasteromer

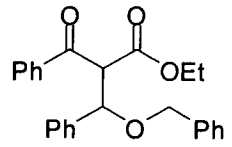
3p-2

```
DFILE   D:\YuRong\2008\12-2H.als
COMNT
DATIM   Tue Apr 15 14:30:22 2008
OBNUC   1H
EXMOD   NON
OBFRQ       300.40 MHz
OBSET       130.00 KHz
OBFIN       1150.0 Hz
POINT        32768
FREQU       6013.2 Hz
SCANS           16
ACQTM        5.449 sec
PD           1.551 sec
PW1            5.8 us
IRNUC   1H
CTEMP         23.5 c
SLVNT   CDCL3
EXREF         0.00 ppm
BF            0.09 Hz
RGAIN           20
```

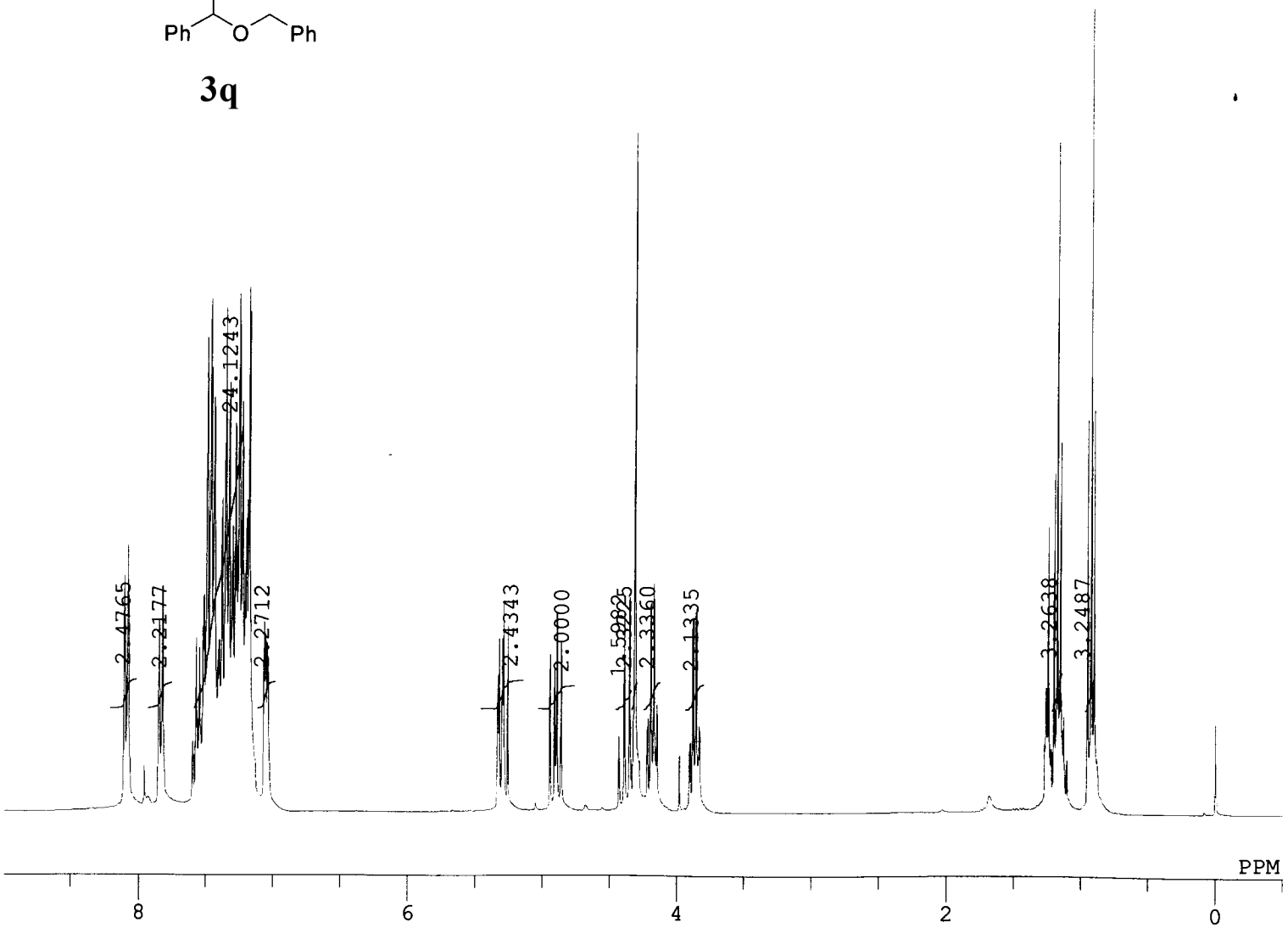




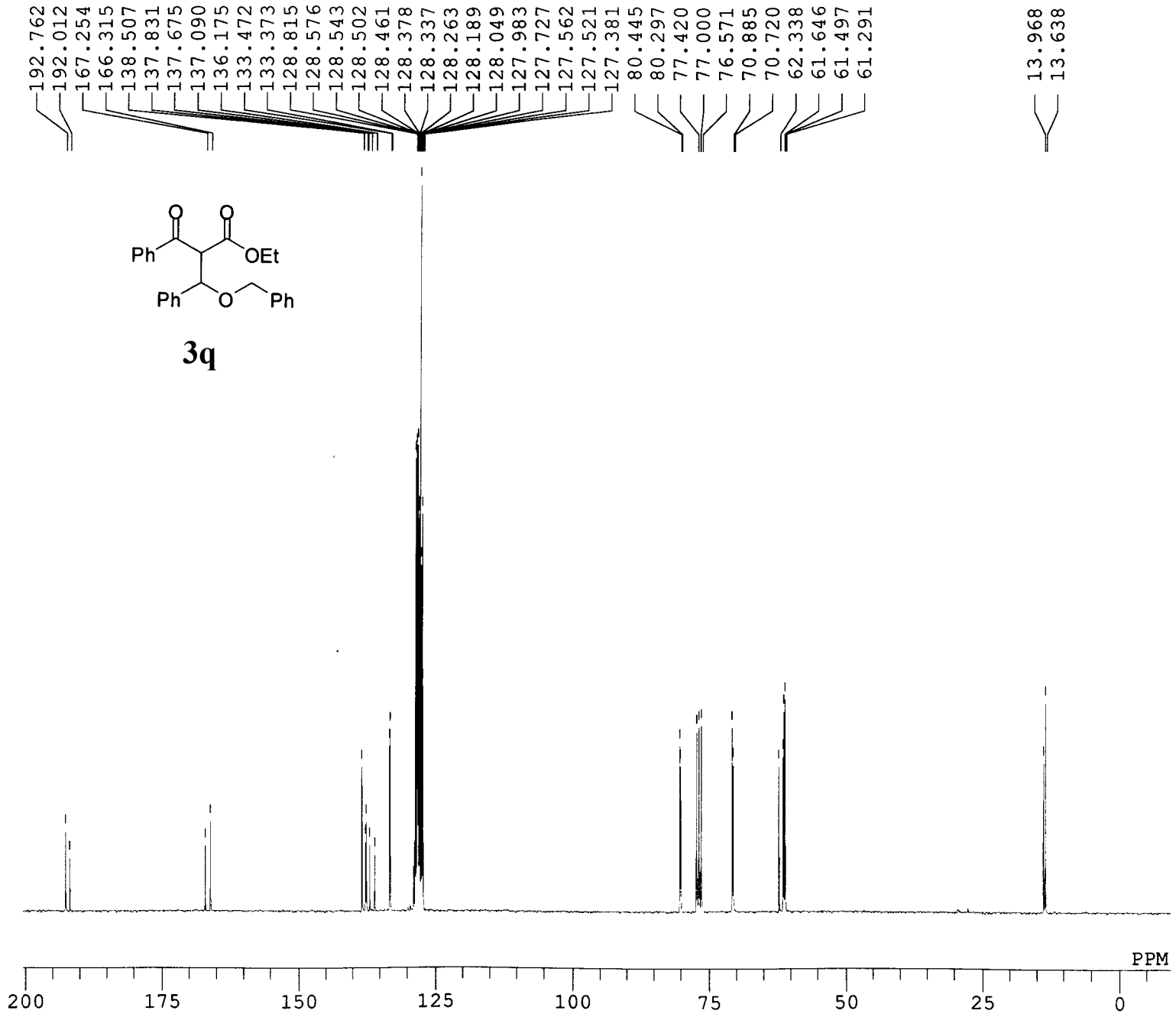

```
DFILE   D:\YuRong\2008\12-2C.als
COMNT
DATIM   Tue Apr 15 14:27:12 2008
OBNUC   13C
EXMOD   BCM
OBFRQ         75.45 MHz
OBSET        124.00 KHz
OBFIN        1840.0 Hz
POINT         32768
FREQU       20408.1 Hz
SCANS           400
ACQTM         1.606 sec
PD            1.394 sec
PW1             5.0 us
IRNUC   1H
CTEMP          24.0 c
SLVNT   CDCL3
EXREF         77.00 ppm
BF             0.62 Hz
RGAIN            26
```



3q

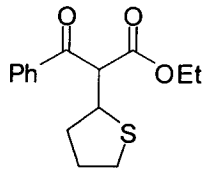


DFILE	D:\YuRong\2008\11-H-80.als	
COMNT		
DATIM	Thu May 08 15:36:44 2008	
OBNUC	1H	
EXMOD	NON	
OBFRQ	300.40	MHz
OBSET	130.00	KHz
OBFIN	1150.0	Hz
POINT	32768	
FREQU	6013.2	Hz
SCANS	16	
ACQTM	5.449	sec
PD	1.551	sec
PW1	5.8	us
IRNUC	1H	
CTEMP	22.7	c
SLVNT	CDCL3	
EXREF	0.00	ppm
BF	0.09	Hz
RGAIN	15	



3q

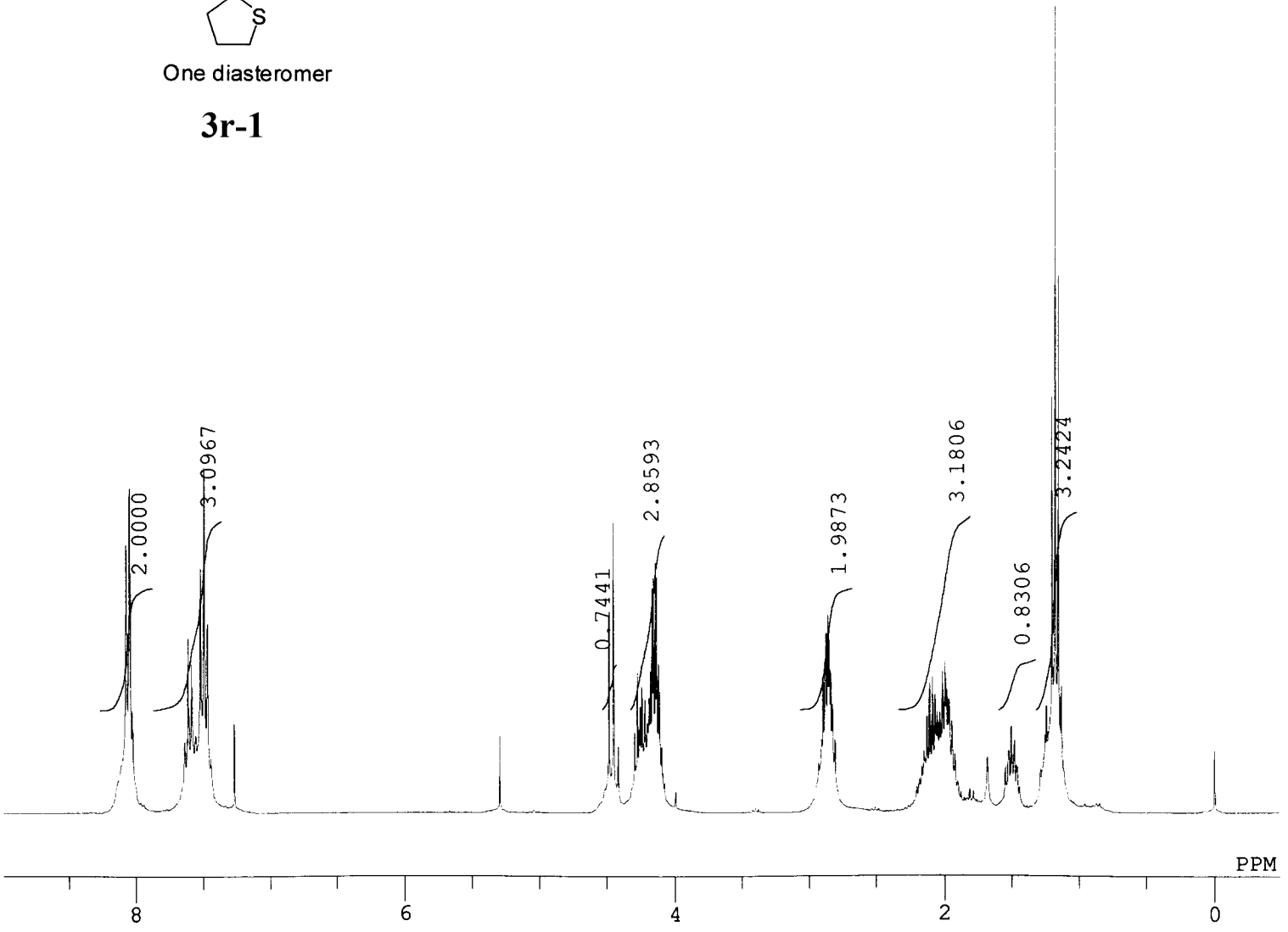
DFILE	D:\YuRong\11-C-80.als
COMNT	
DATIM	Thu May 08 19:17:38 2008
OBNUC	13C
EXMOD	BCM
OBFRQ	75.45 MHz
OBSET	124.00 KHz
OBFIN	1840.0 Hz
POINT	32768
FREQU	20408.1 Hz
SCANS	440
ACQTM	1.606 sec
PD	1.394 sec
PW1	5.0 us
IRNUC	1H
CTEMP	23.5 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.62 Hz
RGAIN	26

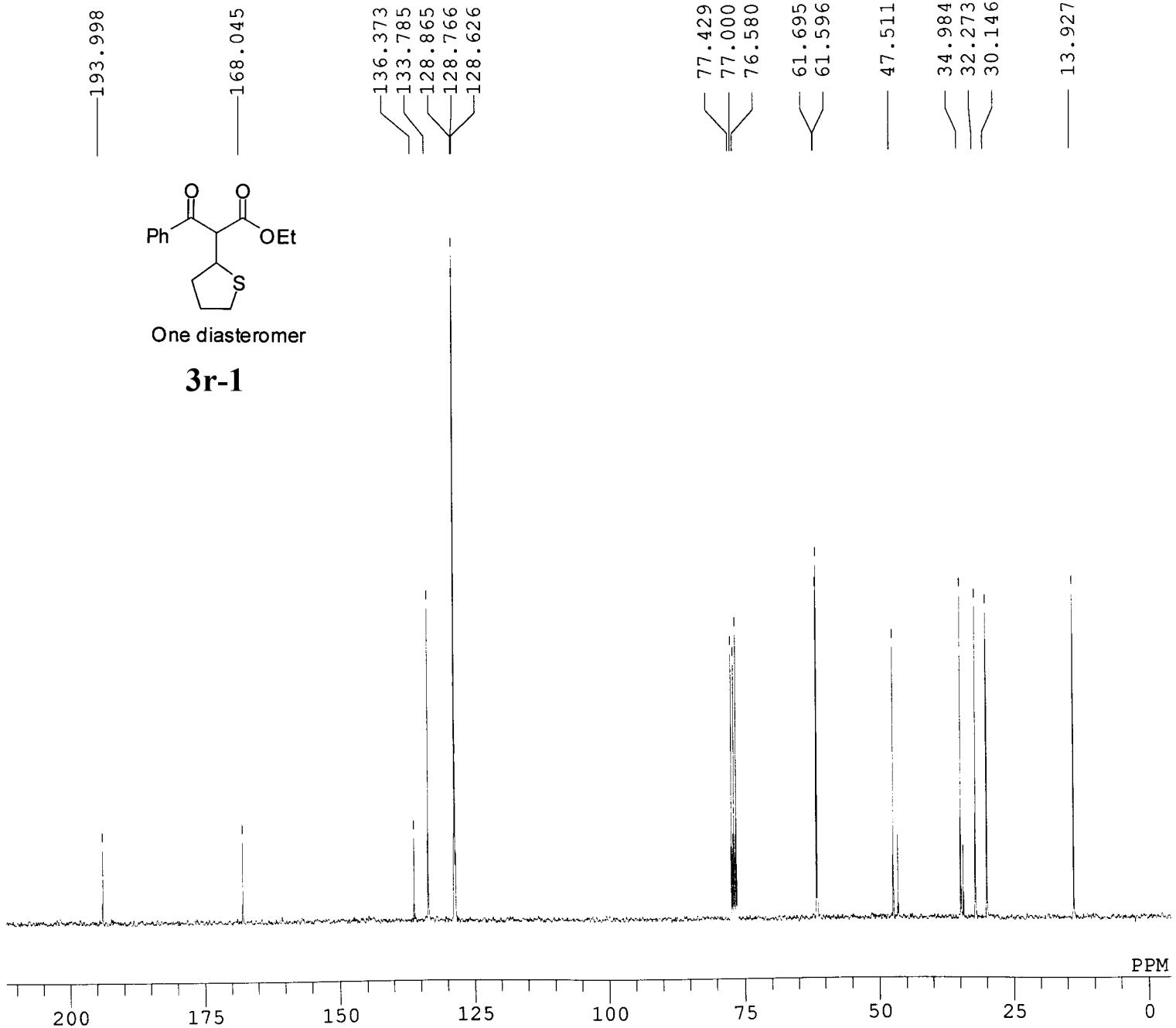


One diasteromer

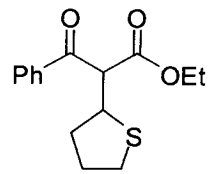
3r-1

```
DFILE   D:\YuRong\2008\28-1H.als
COMNT
DATIM   Sat Apr 19 15:02:10 2008
OBNUC   1H
EXMOD   NON
OBFRQ       300.40 MHz
OBSET       130.00 KHz
OBFIN       1150.0 Hz
POINT        32768
FREQU       6013.2 Hz
SCANS           16
ACQTM        5.449 sec
PD           1.551 sec
PW1            5.8 us
IRNUC   1H
CTEMP         24.0 c
SLVNT   CDCL3
EXREF         0.00 ppm
BF            0.09 Hz
RGAIN           17
```





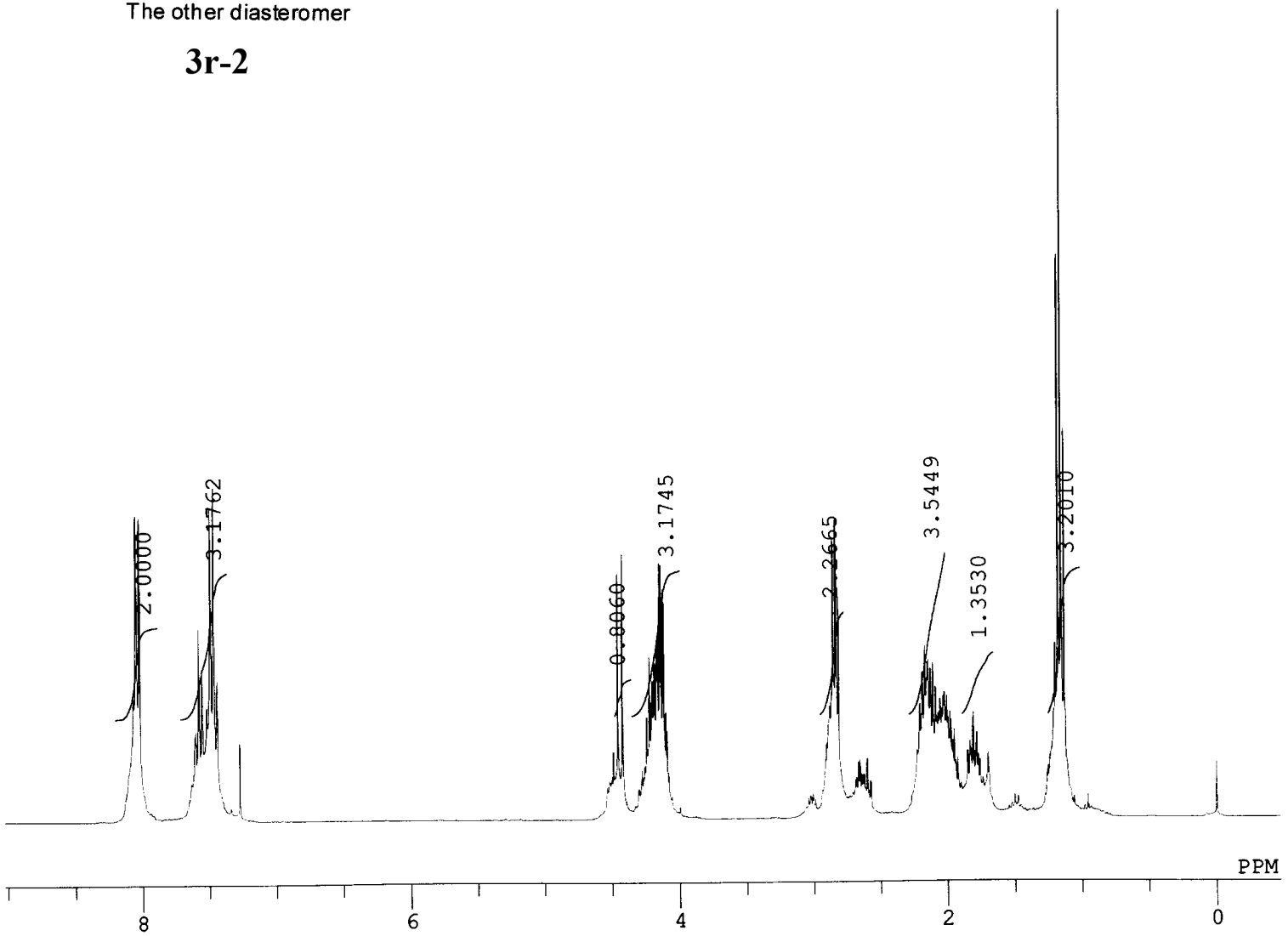
DFILE	D:\YuRong\2008\28-1C.als
COMNT	
DATIM	Sat Apr 19 15:48:26 2008
OBNUC	13C
EXMOD	BCM
OBFRQ	75.45 MHz
OBSET	124.00 KHz
OBFIN	1840.0 Hz
POINT	32768
FREQU	20408.1 Hz
SCANS	100
ACQTM	1.606 sec
PD	1.394 sec
PW1	5.0 us
IRNUC	1H
CTEMP	24.7 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.62 Hz
RGAIN	27

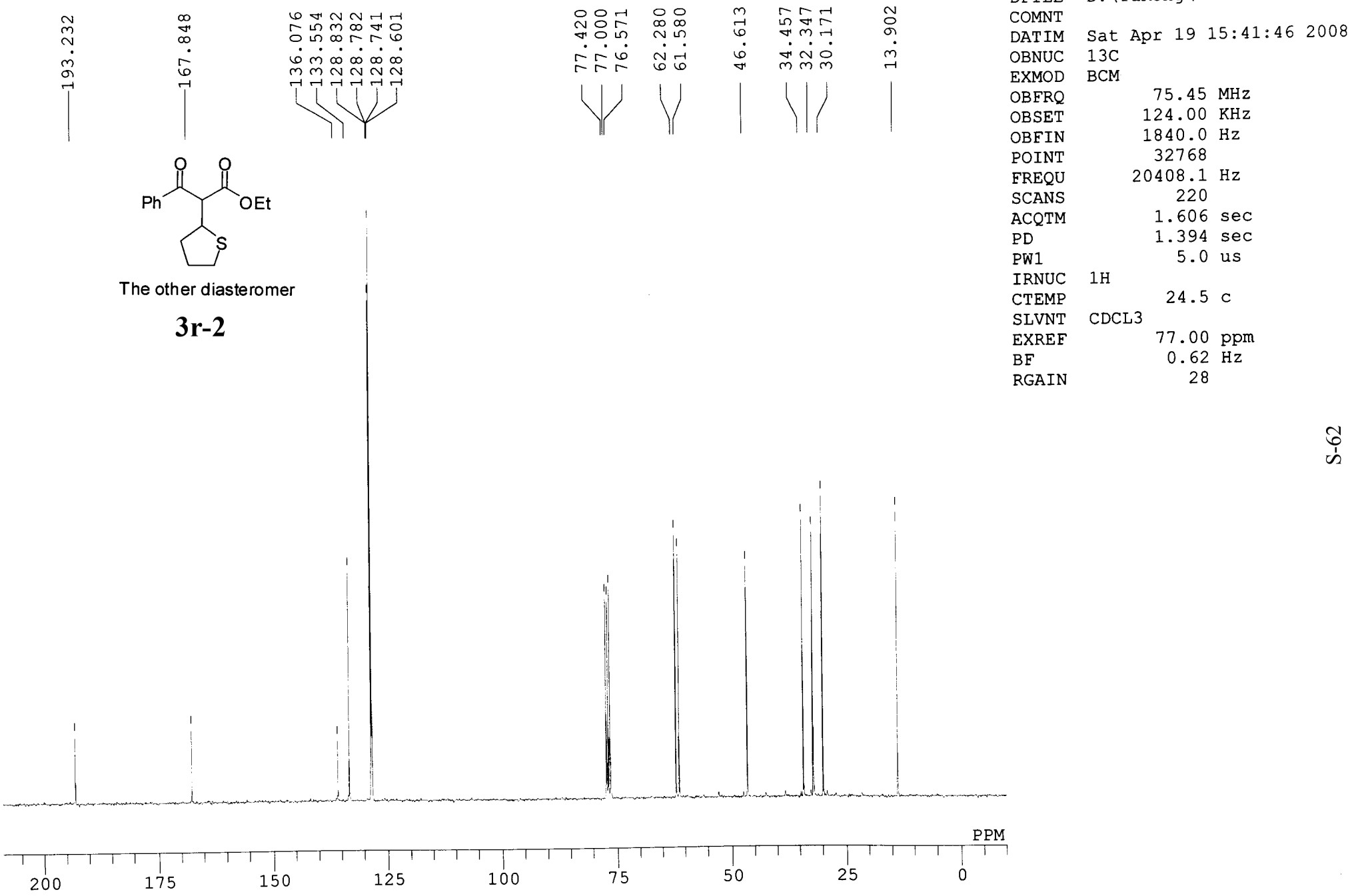


The other diasteromer

3r-2

```
DFILE   D:\YuRong\2008\28-2H.als
COMNT
DATIM   Sat Apr 19 15:07:03 2008
OBNUC   1H
EXMOD   NON
OBFRQ         300.40 MHz
OBSET         130.00 KHz
OBFIN         1150.0 Hz
POINT          32768
FREQU         6013.2 Hz
SCANS             16
ACQTM          5.449 sec
PD             1.551 sec
PW1              5.8 us
IRNUC   1H
CTEMP           24.0 c
SLVNT   CDCL3
EXREF           0.00 ppm
BF              0.09 Hz
RGAIN             16
```

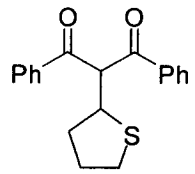




The other diasteromer

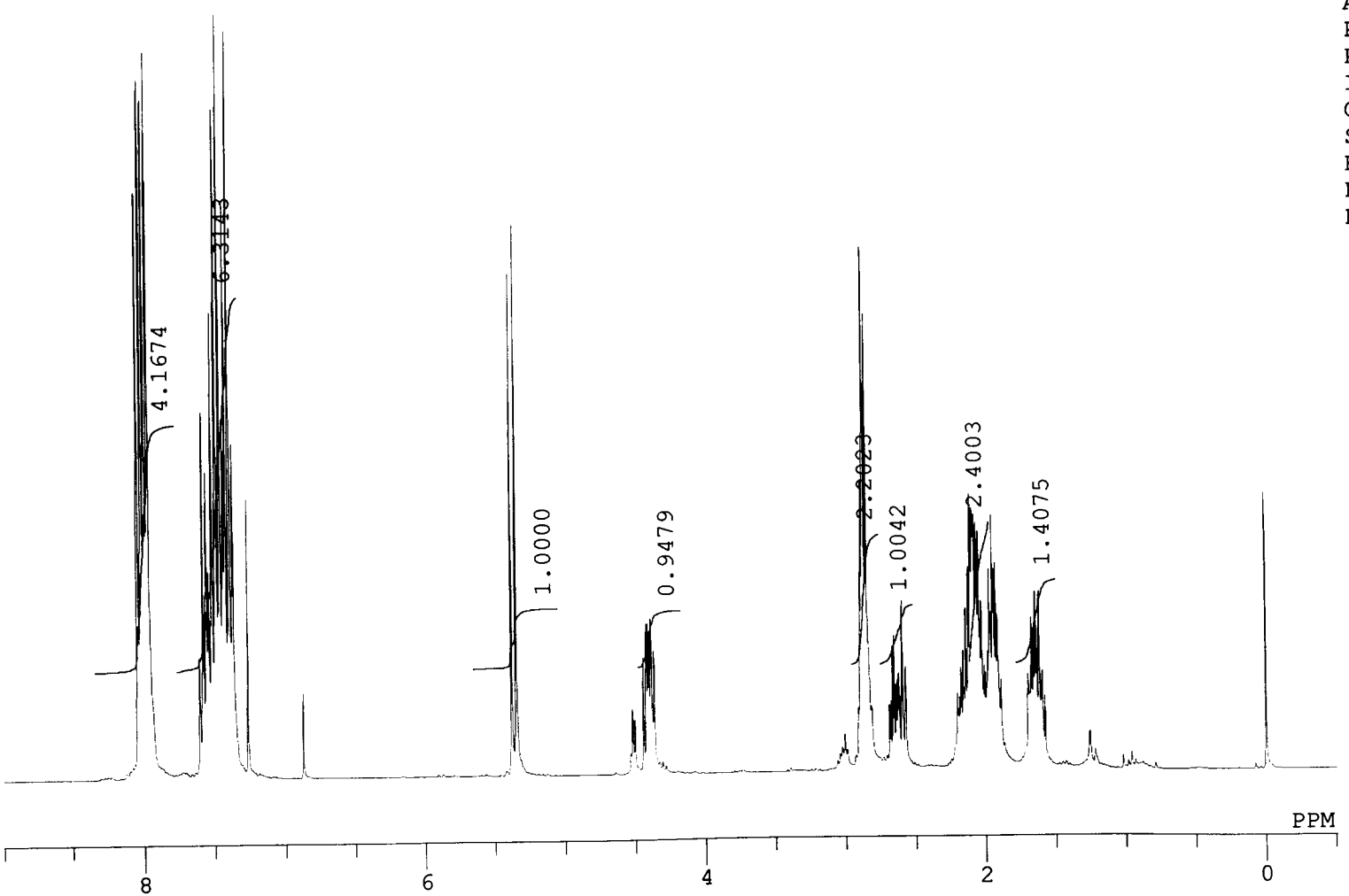
3r-2

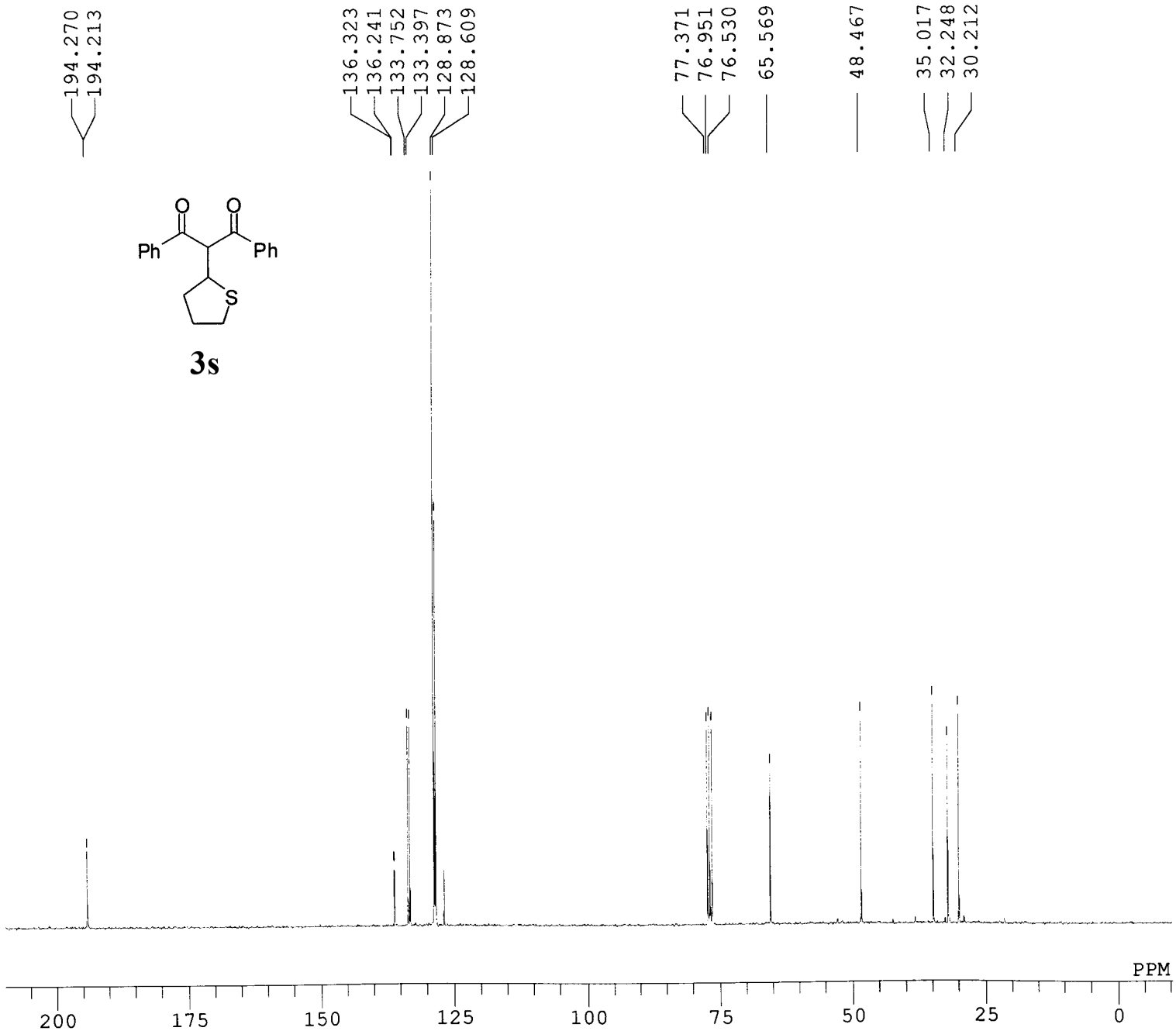
```
DFILE   D:\YuRong\2008\28-2C.als
COMNT
DATIM   Sat Apr 19 15:41:46 2008
OBNUC   13C
EXMOD   BCM
OBFRQ         75.45 MHz
OBSET        124.00 KHz
OBFIN        1840.0 Hz
POINT         32768
FREQU       20408.1 Hz
SCANS           220
ACQTM         1.606 sec
PD            1.394 sec
PW1             5.0 us
IRNUC   1H
CTEMP          24.5 c
SLVNT   CDCL3
EXREF         77.00 ppm
BF             0.62 Hz
RGAIN            28
```



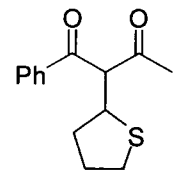
3s

```
DFILE   D:\YuRong\2008\29-H.als
COMNT
DATIM   Mon May 05 00:19:49 2008
OBNUC   1H
EXMOD   NON
OBFRQ         300.40 MHz
OBSET         130.00 KHz
OBFIN         1150.0 Hz
POINT          32768
FREQU         6013.2 Hz
SCANS             12
ACQTM          5.449 sec
PD             1.551 sec
PW1              5.8 us
IRNUC   1H
CTEMP           23.0 c
SLVNT   CDCL3
EXREF           0.00 ppm
BF              0.09 Hz
RGAIN             18
```





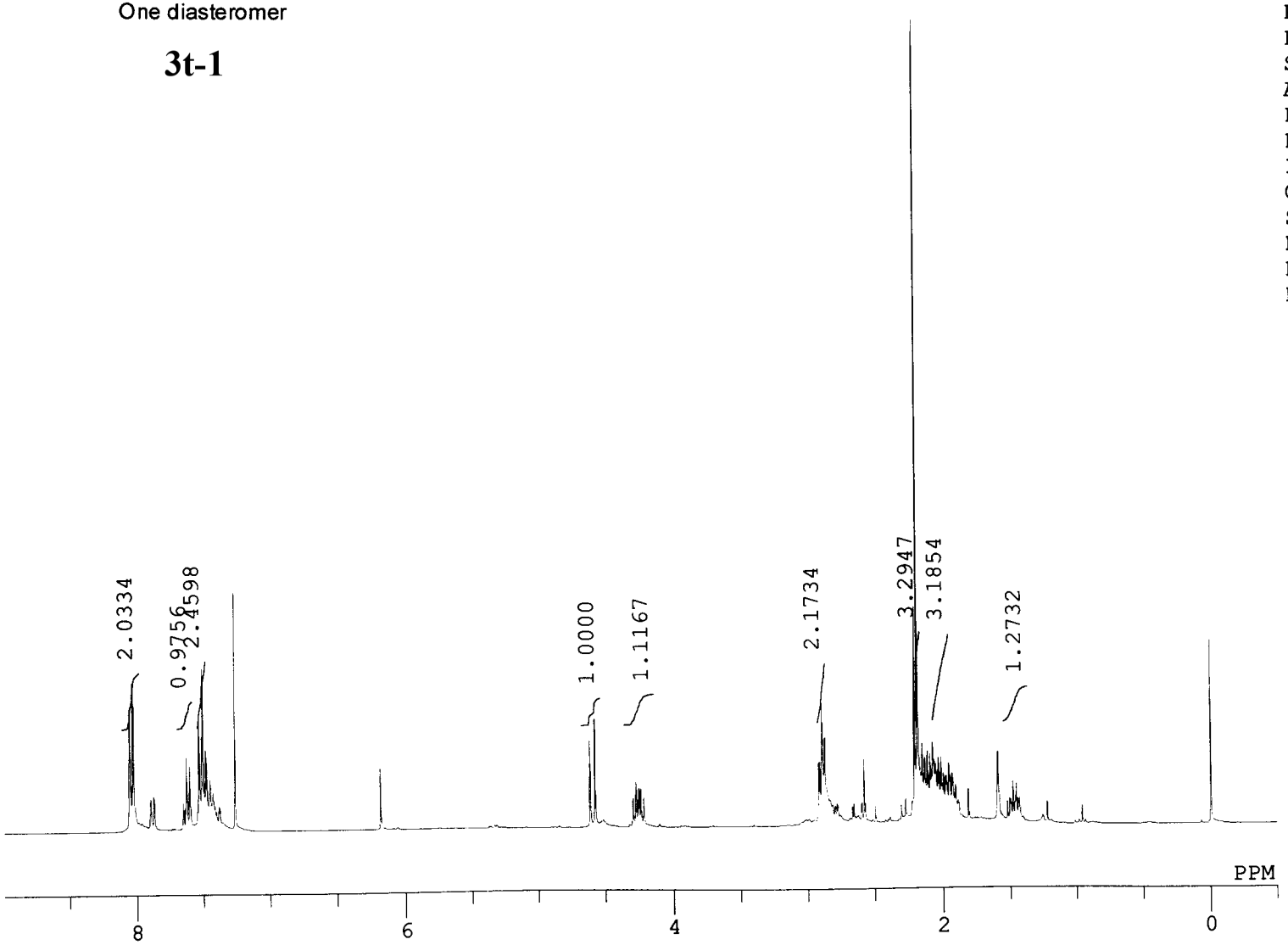
DFILE	D:\YuRong\2008\29-C.als
COMNT	
DATIM	Mon May 05 00:16:25 2008
OBNUC	13C
EXMOD	BCM
OBFRQ	75.45 MHz
OBSET	124.00 KHz
OBFIN	1840.0 Hz
POINT	32768
FREQU	20408.1 Hz
SCANS	240
ACQTM	1.606 sec
PD	1.394 sec
PW1	5.0 us
IRNUC	1H
CTEMP	23.0 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.62 Hz
RGAIN	26

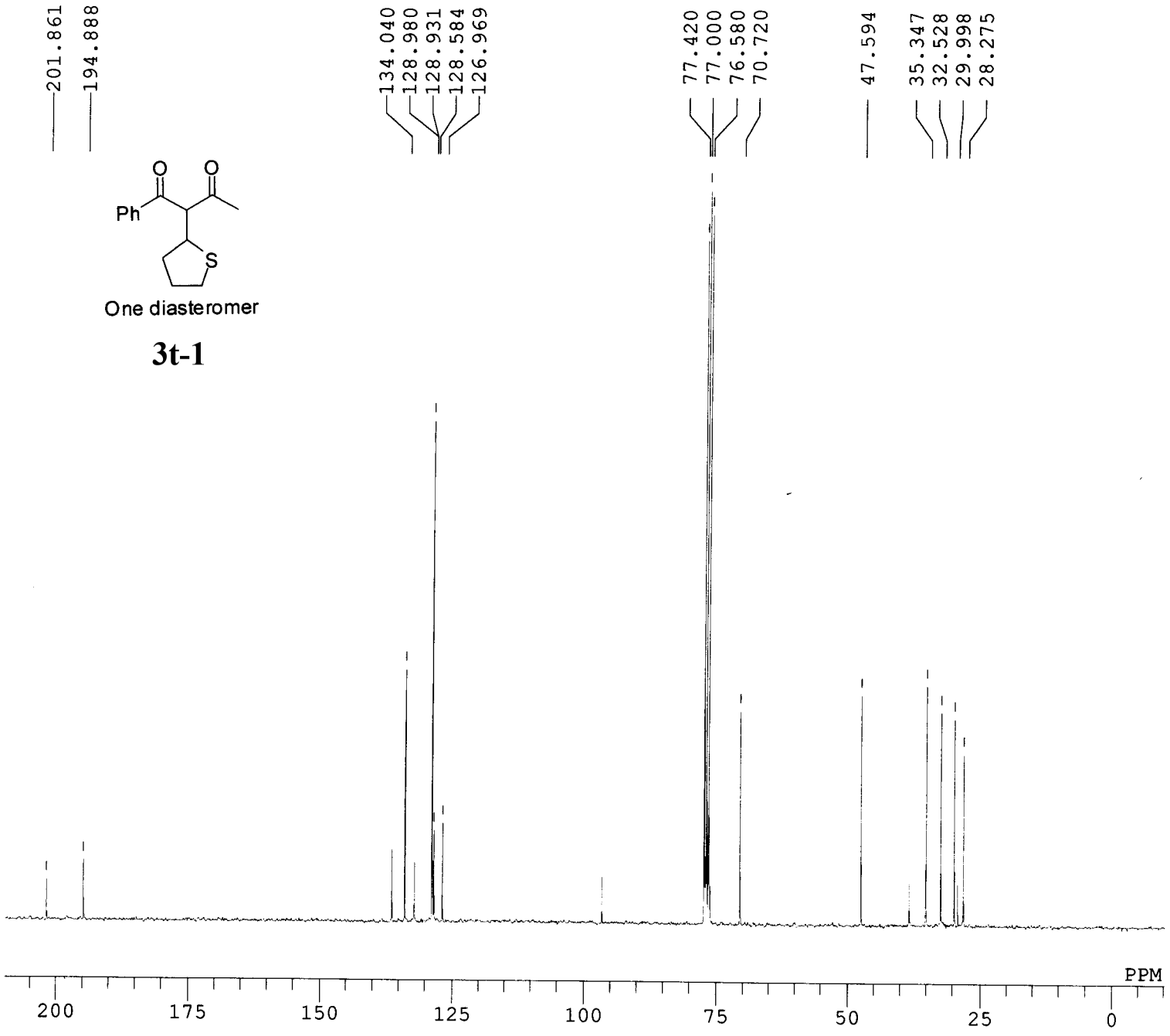


One diasteromer

3t-1

```
DFILE   D:\YuRong\2008\31-1H.als
COMNT
DATIM   Sat Apr 26 22:36:19 2008
OBNUC   1H
EXMOD   NON
OBFRQ        300.40 MHz
OBSET        130.00 KHz
OBFIN        1150.0 Hz
POINT         32768
FREQU        6013.2 Hz
SCANS            16
ACQTM         5.449 sec
PD            1.551 sec
PW1             5.8 us
IRNUC   1H
CTEMP          23.1 c
SLVNT   CDCL3
EXREF          0.00 ppm
BF             0.09 Hz
RGAIN            22
```

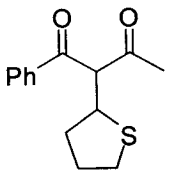




One diasteromer

3t-1

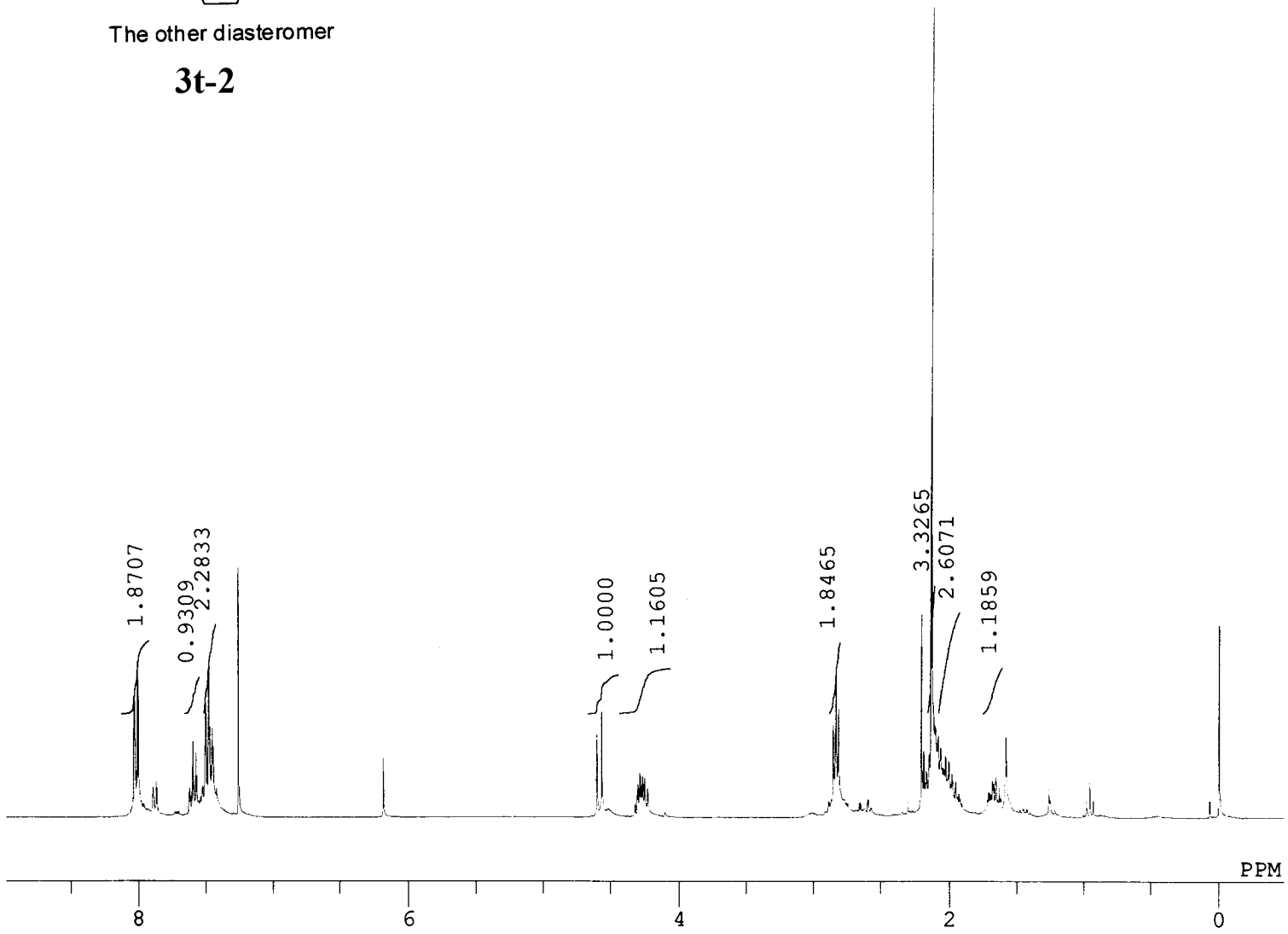
DFILE	D:\YuRong\2008\31-1C.als
COMNT	
DATIM	Mon May 05 01:01:26 2008
OBNUC	13C
EXMOD	BCM
OBFRQ	75.45 MHz
OBSET	124.00 KHz
OBFIN	1840.0 Hz
POINT	32768
FREQU	20408.1 Hz
SCANS	480
ACQTM	1.606 sec
PD	1.394 sec
PW1	5.0 us
IRNUC	1H
CTEMP	23.2 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.62 Hz
RGAIN	26

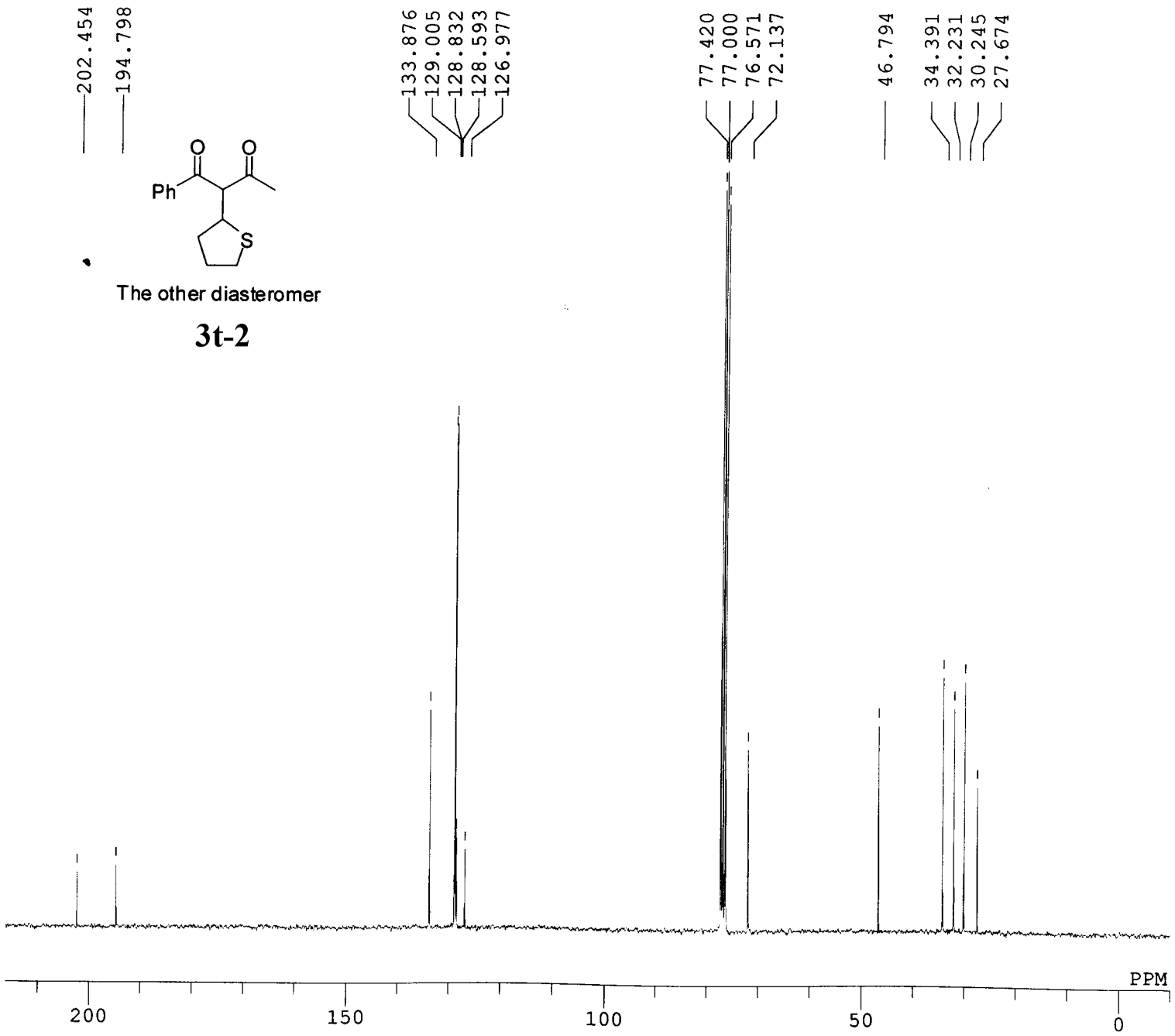


The other diasteromer

3t-2

DFILE	D:\YuRong\2008\31-2H.als
COMNT	
DATIM	Sat Apr 26 22:42:33 2008
OBNUC	1H
EXMOD	NON
OBFRQ	300.40 MHz
OBSET	130.00 KHz
OBFIN	1150.0 Hz
POINT	32768
FREQU	6013.2 Hz
SCANS	16
ACQTM	5.449 sec
PD	1.551 sec
PW1	5.8 us
IRNUC	1H
CTEMP	23.1 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.09 Hz
RGAIN	21

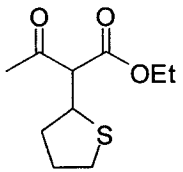




The other diasteromer

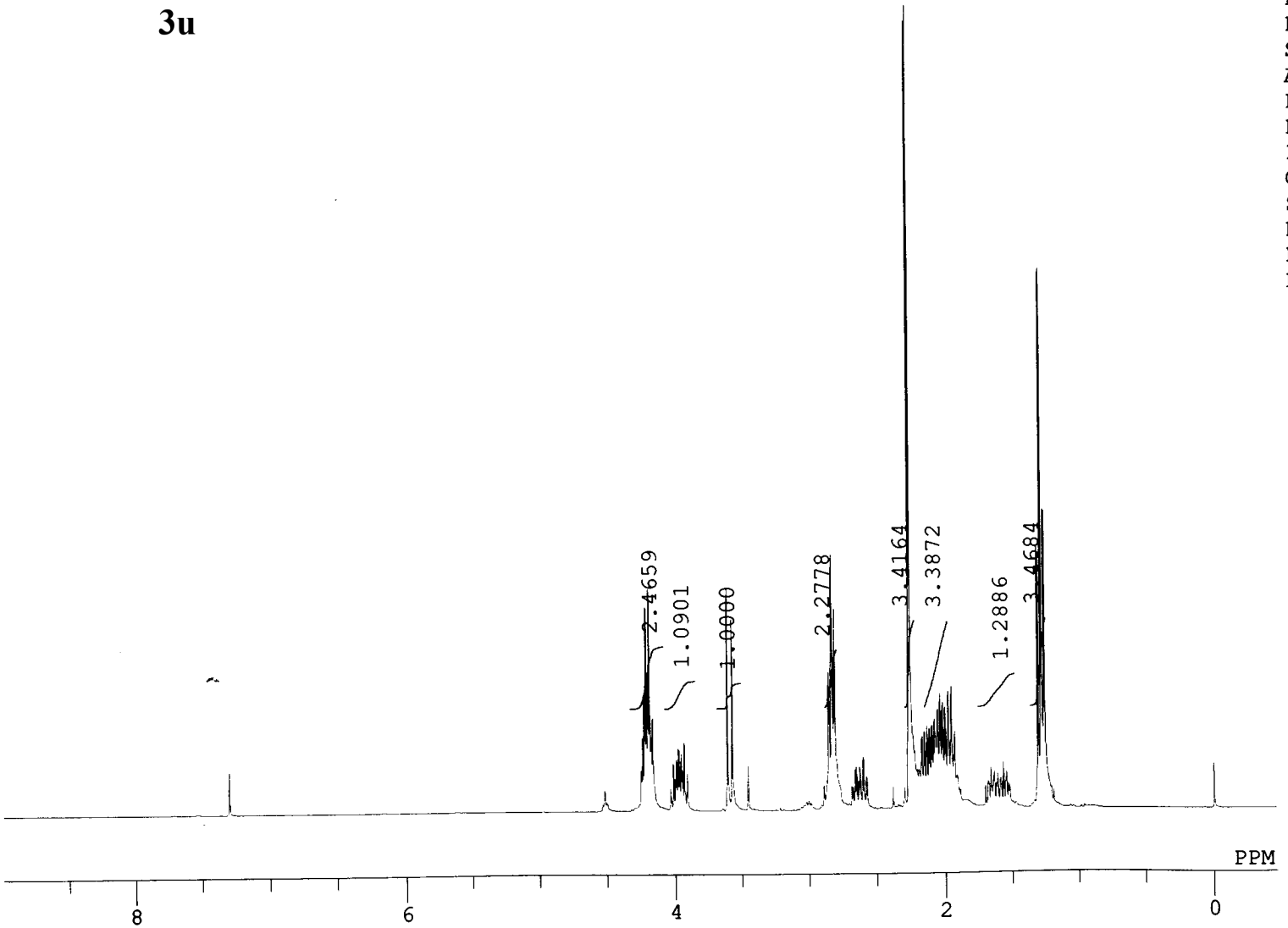
3t-2

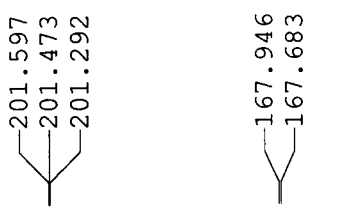
DFILE	D:\YuRong\2008\31-2C.als
COMNT	
DATIM	Mon May 05 01:37:55 2008
OBNUC	13C
EXMOD	BCM
OBFRQ	75.45 MHz
OBSET	124.00 KHz
OBFIN	1840.0 Hz
POINT	32768
FREQU	20408.1 Hz
SCANS	510
ACQTM	1.606 sec
PD	1.394 sec
PW1	5.0 us
IRNUC	1H
CTEMP	23.2 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.62 Hz
RGAIN	26

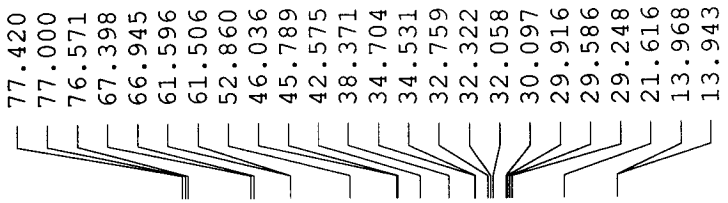


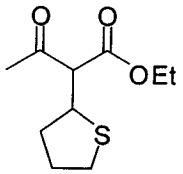
3u

DFILE	D:\YuRong\2008\30-H.als
COMNT	
DATIM	Mon May 05 00:35:49 2008
OBNUC	1H
EXMOD	NON
OBFRQ	300.40 MHz
OBSET	130.00 KHz
OBFIN	1150.0 Hz
POINT	32768
FREQU	6013.2 Hz
SCANS	16
ACQTM	5.449 sec
PD	1.551 sec
PW1	5.8 us
IRNUC	1H
CTEMP	22.5 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.09 Hz
RGAIN	14



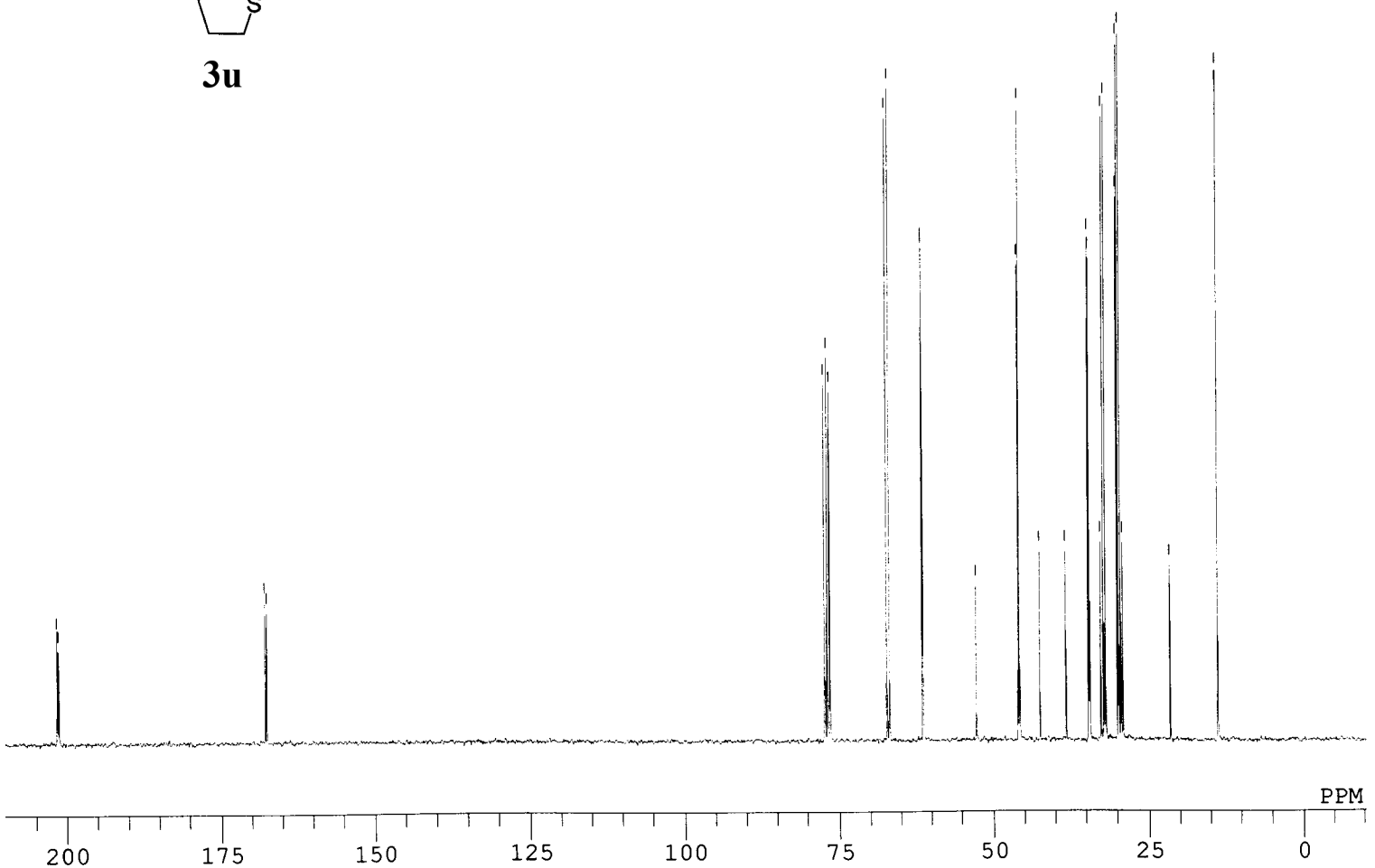


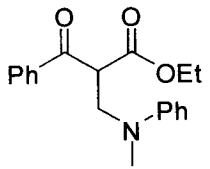




3u

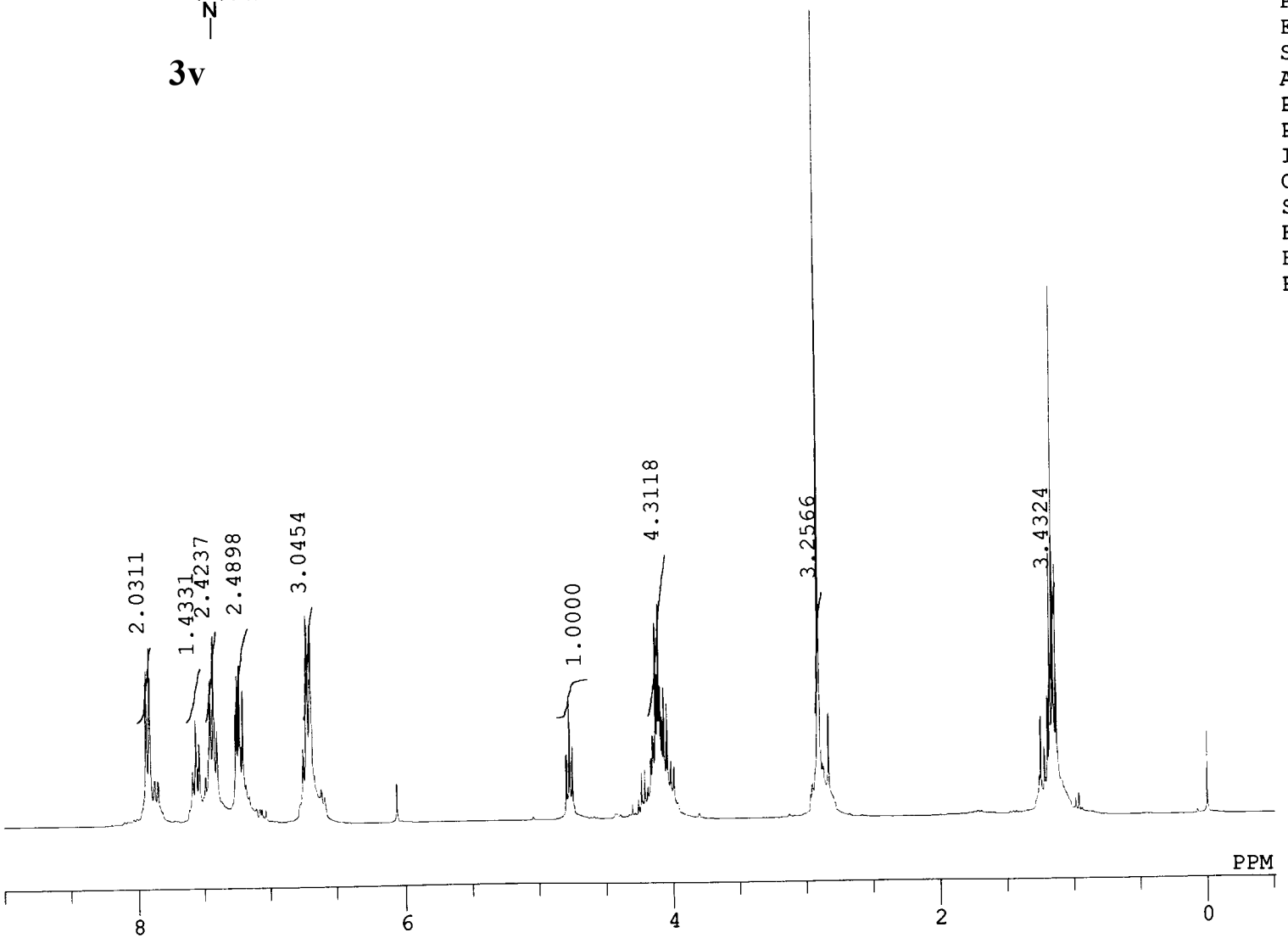
```
DFILE   D:\YuRong\2008\30-C.als
COMNT
DATIM   Mon May 05 00:32:35 2008
OBNUC   13C
EXMOD   BCM
OBFRQ         75.45 MHz
OBSET        124.00 KHz
OBFIN        1840.0 Hz
POINT         32768
FREQU       20408.1 Hz
SCANS           200
ACQTM         1.606 sec
PD            1.394 sec
PW1             5.0 us
IRNUC   1H
CTEMP          23.2 c
SLVNT   CDCL3
EXREF         77.00 ppm
BF             0.62 Hz
RGAIN            26
```

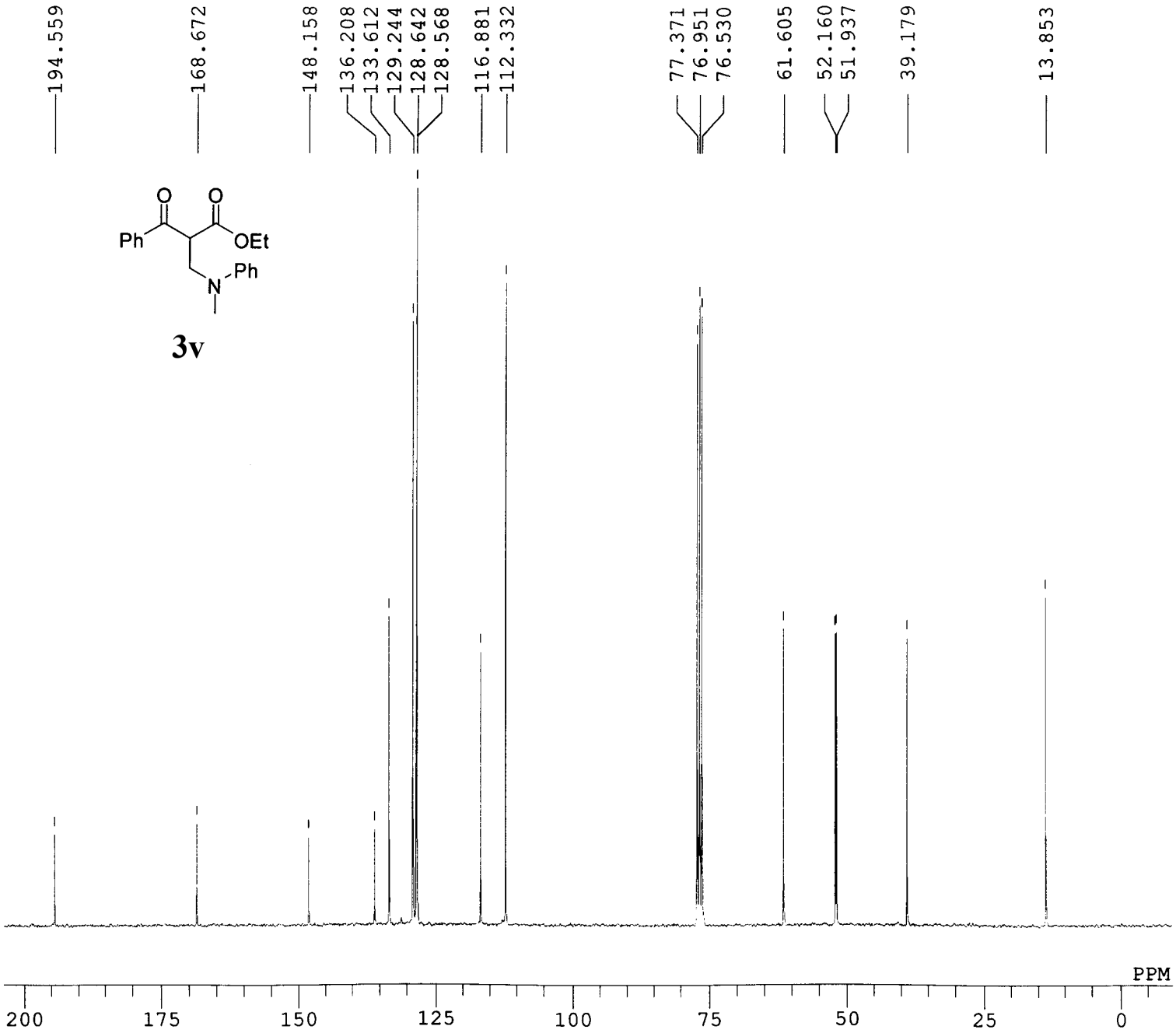




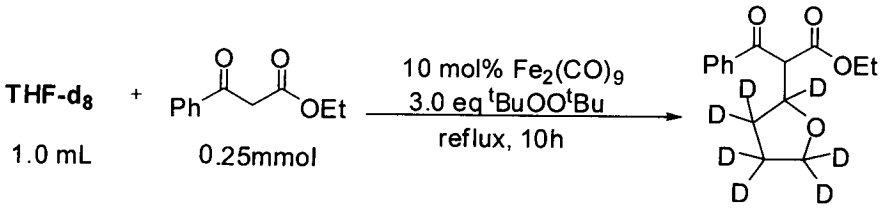
3v

```
DFILE   D:\YuRong\2008\33-H.als
COMNT
DATIM   Wed May 07 15:26:40 2008
OBNUC   1H
EXMOD   NON
OBFRQ        300.40 MHz
OBSET        130.00 KHz
OBFIN        1150.0 Hz
POINT         32768
FREQU        6013.2 Hz
SCANS            16
ACQTM         5.449 sec
PD            1.551 sec
PW1             5.8 us
IRNUC   1H
CTEMP          22.9 c
SLVNT   CDCL3
EXREF          0.00 ppm
BF             0.09 Hz
RGAIN            20
```

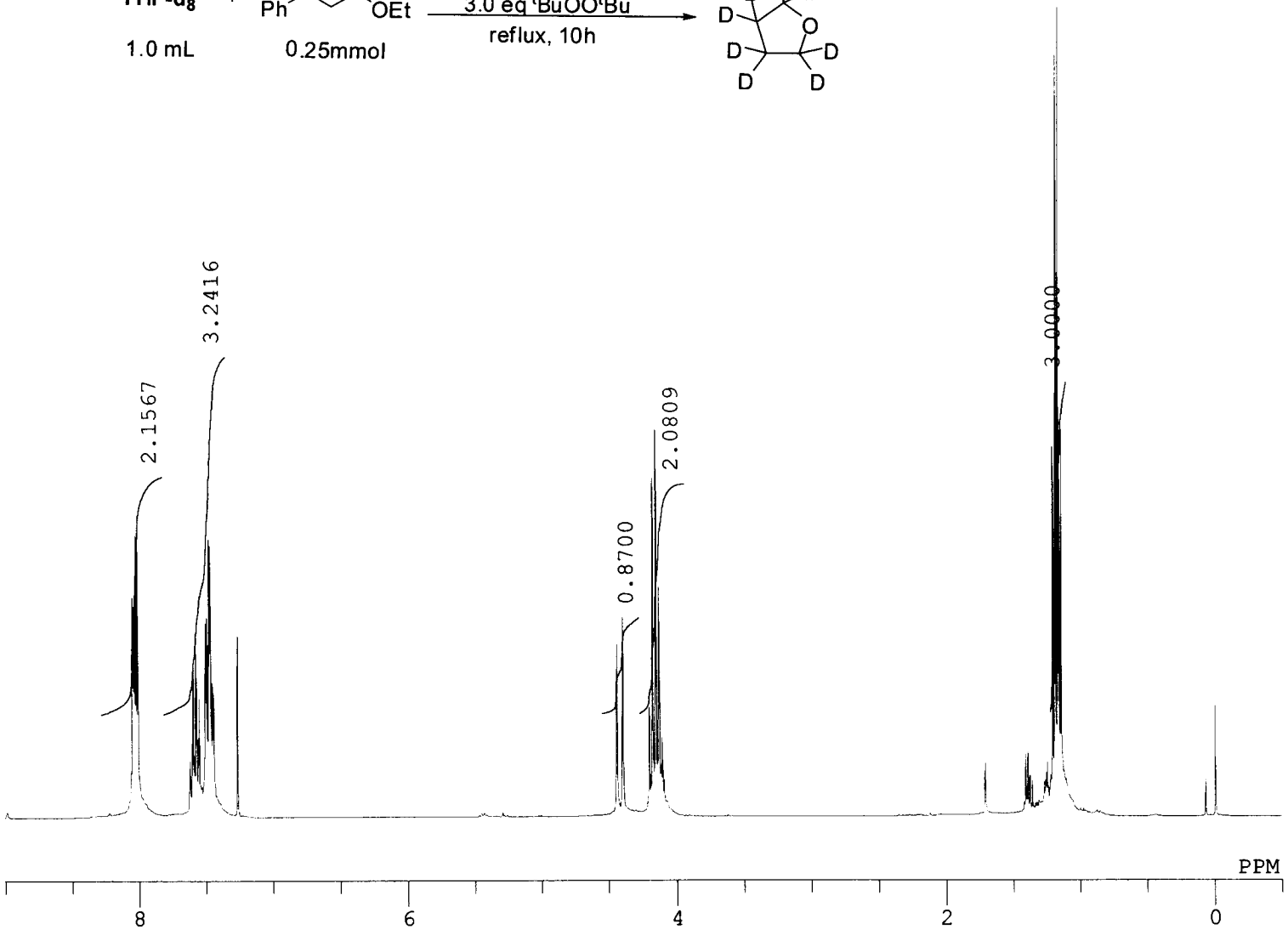


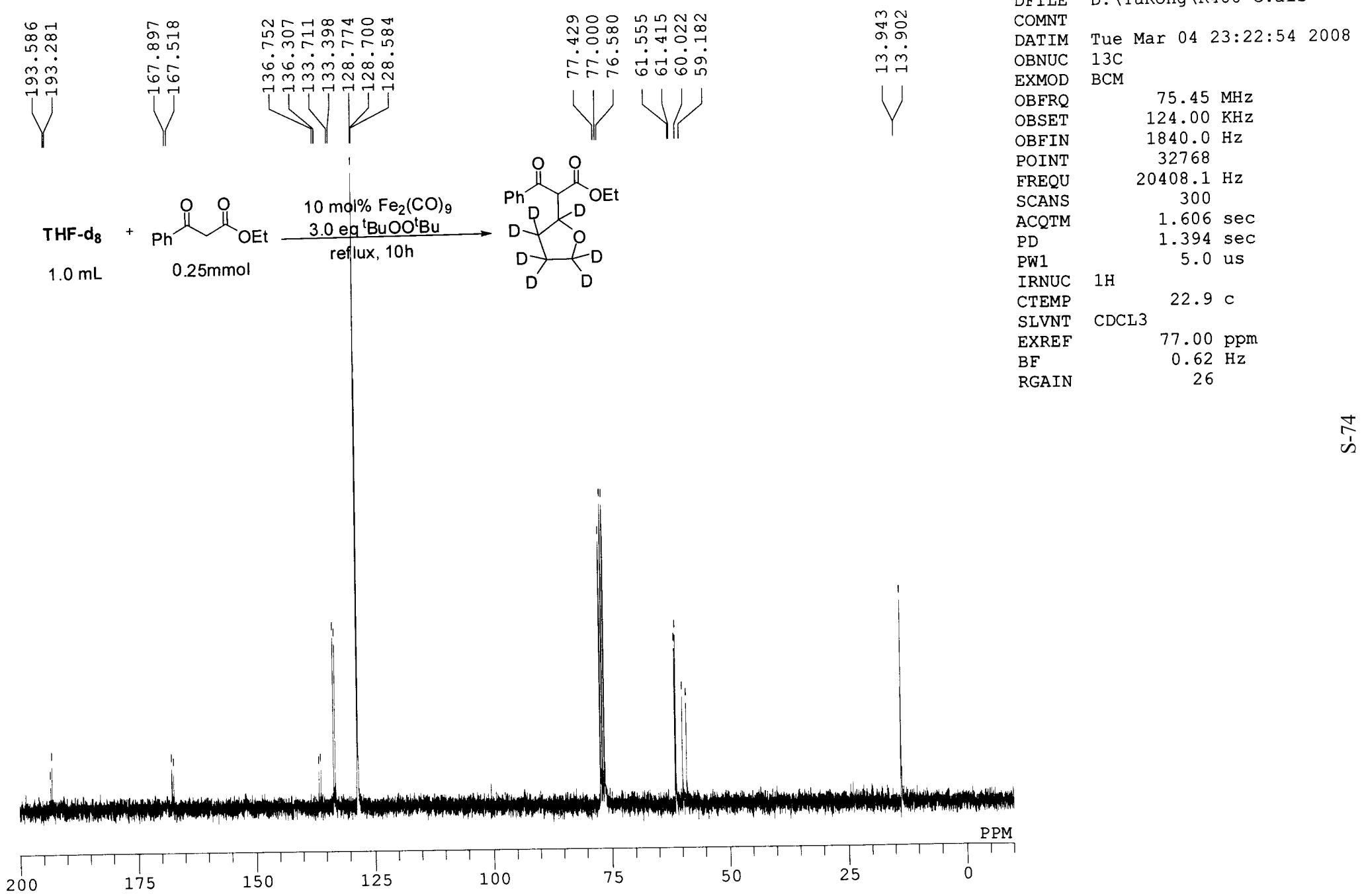


DFILE D:\YuRong\2008\33-C.als
COMNT
DATIM Wed May 07 15:20:31 2008
OBNUC 13C
EXMOD BCM
OBFRQ 75.45 MHz
OBSET 124.00 KHz
OBFIN 1840.0 Hz
POINT 32768
FREQU 20408.1 Hz
SCANS 1600
ACQTM 1.606 sec
PD 1.394 sec
PW1 5.0 us
IRNUC 1H
CTEMP 23.7 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.62 Hz
RGAIN 26

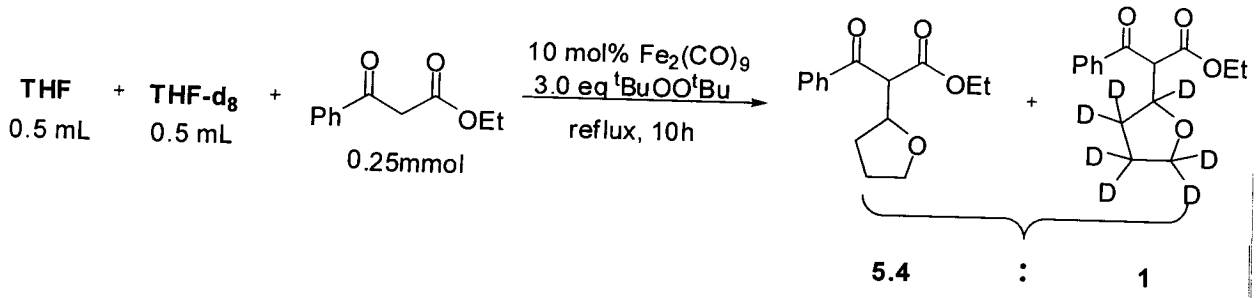



```
DFILE   D:\YuRong\R400-H.als
COMNT
DATIM   Tue Mar 04 23:06:12 2008
OBNUC   1H
EXMOD   NON
OBFRQ         300.40 MHz
OBSET         130.00 KHz
OBFIN         1150.0 Hz
POINT          32768
FREQU         6013.2 Hz
SCANS             16
ACQTM          5.449 sec
PD             1.551 sec
PW1              5.8 us
IRNUC   1H
CTEMP           22.1 c
SLVNT   CDCL3
EXREF           0.00 ppm
BF              0.09 Hz
RGAIN             17
```

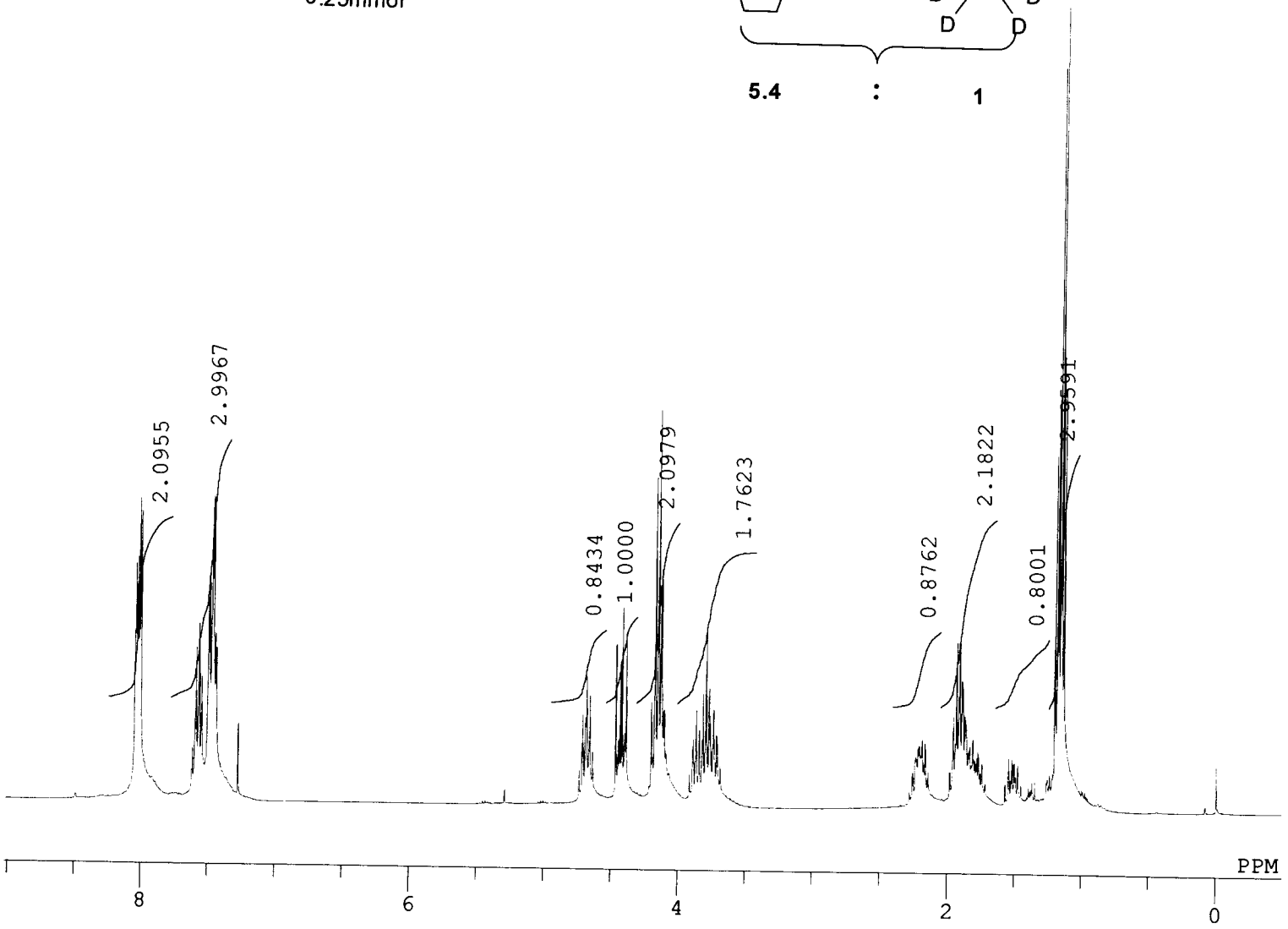


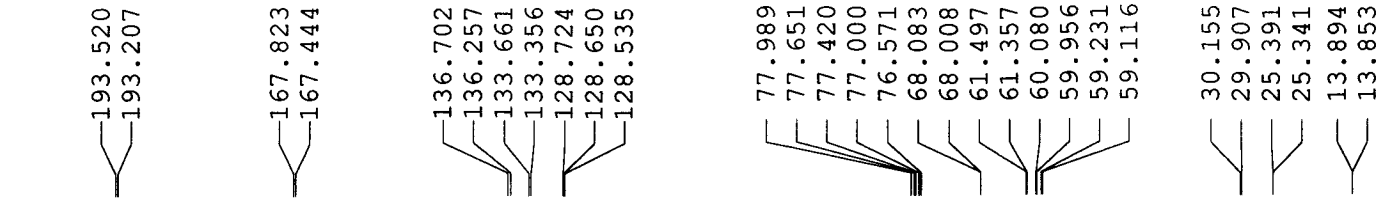


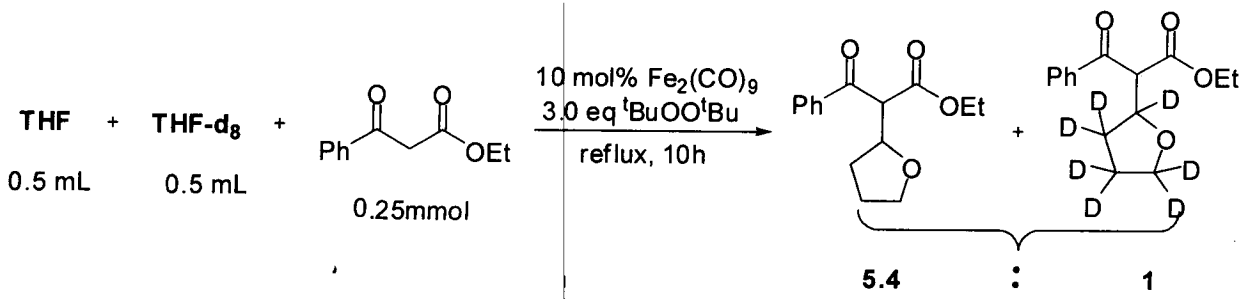
DFILE D:\YuRong\R400-C.als
COMNT
DATIM Tue Mar 04 23:22:54 2008
OBNUC 13C
EXMOD BCM
OBFRQ 75.45 MHz
OBSET 124.00 KHz
OBFIN 1840.0 Hz
POINT 32768
FREQU 20408.1 Hz
SCANS 300
ACQTM 1.606 sec
PD 1.394 sec
PW1 5.0 us
IRNUC 1H
CTEMP 22.9 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.62 Hz
RGAIN 26



DFILE D:\YuRong\R401-H.als
COMNT
DATIM Tue Mar 04 22:45:20 2008
OBNUC 1H
EXMOD NON
OBFRQ 300.40 MHz
OBSET 130.00 KHz
OBFIN 1150.0 Hz
POINT 32768
FREQU 6013.2 Hz
SCANS 16
ACQTM 5.449 sec
PD 1.551 sec
PW1 5.8 us
IRNUC 1H
CTEMP 22.3 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.09 Hz
RGAIN 15







DFILE D:\YuRong\R401-C.als
COMNT
DATIM Tue Mar 04 22:58:58 2008
OBNUC 13C
EXMOD BCM
OBFRQ 75.45 MHz
OBSET 124.00 KHz
OBFIN 1840.0 Hz
POINT 32768
FREQU 20408.1 Hz
SCANS 260
ACQTM 1.606 sec
PD 1.394 sec
PW1 5.0 us
IRNUC 1H
CTEMP 22.9 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 1.20 Hz
RGAIN 26