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Supporting Information

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Catalytic Asymmetric Michael Reactions with Enamides as Nucleophiles

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Experimental

General. Melting points are uncorrected. ^{1}H and ^{13}C NMR spectra were recorded on a JEOL JNM-ECX-400 or JNM-ECX-600 spectrometer in $CDCl_3$ or C_6D_6 unless otherwise noted. Tetramethylsilane (TMS) served as internal standard ($\delta = 0$) for ^{1}H NMR, and $CDCl_3$ ($\delta = 77.0$) and C_6D_6 ($\delta = 128.0$) were used as internal standard for ^{13}C NMR. IR spectra were measured on a JASCO FT/IR-610 spectrometer. Optical rotations were measured with a JASCO P-1010 polarimeter. High-performance liquid chromatography was carried out using following apparatus; SHIMADZU LC-10AT (liquid chromatograph), SHIMADZU SPD-10A (UV detector), and SHIMADZU C-R6A Chromatopac. Preparative high-performance liquid chromatography was carried out using the following apparatus; SHIMADZU LC-8A (liquid chromatograph), SHIMADZU SPD-20A (UV detector), RID-10A (refractive index detector), SCL-10A (system controller), FRC-10A (fraction collector), YMC pack SIL-06 column (250x 20 mm, 5μm, 60A), and phenomenex Luna (250 x 50 mm, 15μm, 100A). Column chromatography was conducted on Silica gel 60 (Merck) and preparative thin-layer chromatography was carried out using Wakogel B-5F. All reactions were carried out under argon atmosphere in dried glassware. All solvents were dried and distilled by standard procedures. Enecarbamates and enamides were prepared following literature known procedures.[1]

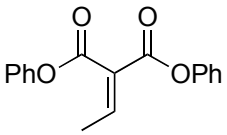
Preparation of Alkylidenemalonates. Alkylidenemalonates were prepared from

[1] R. Matsubara, P. Vital, Y. Nakamura, H. Kiyohara, S. Kobayashi, *Tetrahedron* **2004**, *60*, 9769 and the references cited therein.

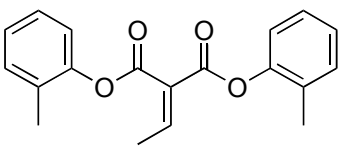
malonates. All the malonates, except bis(4-MeO-C_6H_4) malonate, were prepared using the literature known method for the preparation of diphenyl malonate.[2] This procedure gave low yields in the case of bis(4-MeO-C_6H_4) malonate so a few modifications were added to get it in a moderate yield.

Malonic acid (10.5 g, 10.1 mmol) , 4-MeO-phenol (25.2 g, 2.0 eq.) , toluene (100 mL) and DMF (7.9 mL, 10 mol%) were placed in a flask at 0 °C. $POCl_3$ (11.6 mL, 1.2 eq.) was added to the mixture. Then the mixture was stirred at 115 °C for 2 h. The reaction was cooled down to r.t., and the mixture was transferred in a separating funnel using Et_2O and a saturated solution of $NaHCO_3$. Then the two phases were separated, and the aqueous layer was extracted twice with Et_2O. The combined organic layers were dried with Na_2SO_4 and evaporated to dryness. The obtained solid was washed by MeOH and filtrated to obtain the pure product (13.4 g, 42% yield).

The condensation reactions of aldehydes and malonates were conducted following the literature procedure for the preparation of diphenyl ethylidenemalonate.[2]
In a typical experiment bis(4-methoxyphenyl) malonate (1.58g, 5 mmol), acetaldehyde (2.2 mL, 8 eq.) and acetic anhydride (0.6 mL, 1.4 eq.) were placed in a sealed tube and then the reaction was conducted stirring at 110 °C for 30 h and cooled down to r.t. The crude mixture was directly submitted to a SiO_2 chromatography column (Hexane / Ethyl Acetate : 5/1). The combined fractions were evaporated, then MeOH was added and after a few seconds of sonication the white solid appears. Filtration gave the desired product (0.9 g, 53% yield) .



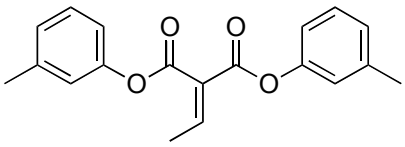
Diphenyl 2-ethylidenemalonate (2a): ^{1}H NMR ($CDCl_3$) δ = 2.18 (d, 3H, *J* = 7.3 Hz), 7.12-7.21 (m, 3H), 7.22-7.28 (m, 3H), 7.35-7.43 (m, 4H), 7.45 (q, 1H, *J* = 7.3 Hz)); ^{13}C NMR ($CDCl_3$) δ = 16.0, 121.5, 126.1, 126.2, 128.4, 129.5, 129.6, 148.9, 150.4, 150.4, 162.3, 163.4; Elemental Analysis $C_{17}H_{14}O_4$ (282.3) : calcd C 72.33, H 5.00; found C 72.12 H 5.23.



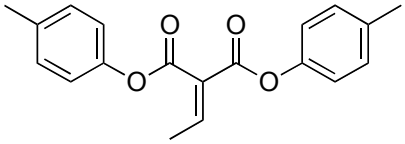
Di *o*-tolyl 2-ethylidenemalonate (2b): ^{1}H NMR ($CDCl_3$) δ = 2.18 (d, 3H, *J* = 7.3 Hz), 2.20 (s, 3H), 2.24 (s, 3H), 7.05-7.12 (m, 2H), 7.13-7.28 (m, 6H), 7.47 (q, 1H, *J* = 7.3

[2] I. Jabin, G. Revial, N. Monnier-Benoit, P. Netchitaïlo, *J. Org. Chem.* **2001**, *66*, 256.

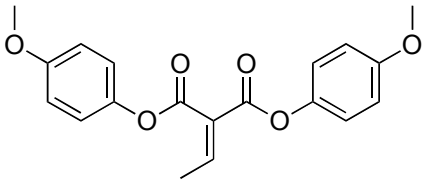
Hz); ^{13}C NMR ($CDCl_3$) δ = 16.0, 16.1, 16.3, 121.7, 121.8, 126.4, 126.4, 127.0, 127.0, 128.4, 130.2, 130.4, 131.2, 131.3, 148.7, 149.0, 162.1, 163.3; Elemental Analysis $C_{17}H_{14}O_4$ (282.3) : calcd C 73.53, H 5.85; found C 73.51 H 6.01.



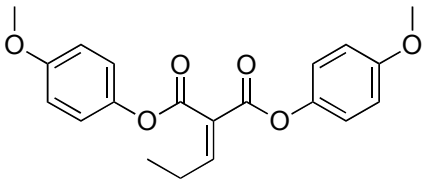
Di *m*-tolyl 2-ethylidenemalonate (2c): ^{1}H NMR $CDCl_3$) δ = 2.11 (d, 3H, *J* = 7.3 Hz), 2.32 (s, 6H), 6.92-7.05 (m, 6H), 7.20-7.26 (m, 2H), 7.39 (q, 1H, *J* = 7.3 Hz); ^{13}C NMR ($CDCl_3$) δ = 15.8, 21.1, 118.2, 121.8, 126.7, 126.8, 128.3, 129.0, 129.1, 139.5, 139.6, 148.4, 150.2, 150.2, 162.2, 163.3.



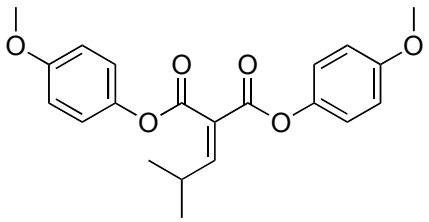
Di *p*-tolyl 2-ethylidenemalonate (2d): ^{1}H NMR ($CDCl_3$) δ = 2.16 (d, 3H, *J* = 7.3 Hz), 2.35 (s, 6H), 7.06 (t, 4H, *J* = 9.2 Hz), 7.19 (dd, 4H, *J* = 8.7, 3.2 Hz), 7.43 (q, 1H, *J* = 7.3 Hz); ^{13}C NMR ($CDCl_3$) δ = 16.0, 20.8, 121.1, 128.5, 130.0, 135.8, 135.9, 148.1, 148.2, 148.4, 150.4, 162.5, 163.7; Elemental Analysis $C_{17}H_{14}O_4$ (282.3) : calcd C 73.53, H 5.85; found C 73.29 H 5.98.



Bis(4-methoxyphenyl) 2-ethylidenemalonate (2e): ^{1}H NMR ($CDCl_3$) δ = 2.16 (d, 3H, *J* = 7.3 Hz), 3.79 (s, 6H), 6.90 (dd, 4H, *J* = 9.2, 3.2 Hz), 7.09 (t, 4H, *J* = 9.6 Hz), 7.42 (q, 1H, *J* = 7.3 Hz) ; ^{13}C NMR ($CDCl_3$) δ = 16.0, 55.6, 114.5, 114.6, 122.2, 128.5, 143.8, 143.9, 148.5, 157.4, 157.5, 162.7, 163.9.



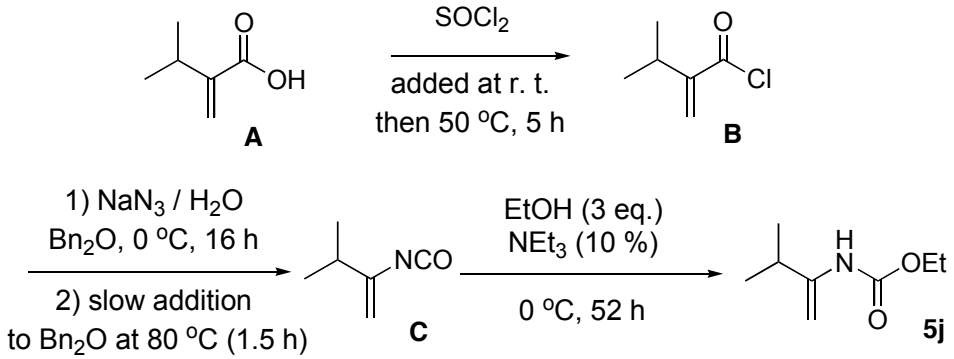
Bis(4-methoxyphenyl) 2-propylidenemalonate (2f): ^{1}H NMR ($CDCl_3$) δ = 1.21 (t, 3H, *J* = 7.6 Hz), 2.54 (quint, 2H, *J* = 7.6 Hz), 3.81 (s, 6H), 6.91 (d, 4H, *J* = 9.2 Hz), 7.10 (dd, 4H, *J* = 8.9, 5.3 Hz), 7.32 (t, 1H, *J* = 7.8 Hz); ^{13}C NMR ($CDCl_3$) δ = 12.8, 23.6, 55.6, 114.5, 114.6, 122.2, 127.0, 143.8, 143.9, 154.2, 157.4, 157.5, 162.8, 164.0.



Bis(4-methoxyphenyl) 2-(2-methylpropylidene)malonate (2g): ^{1}H NMR ($CDCl_3$) δ = 1.19 (t, 3H, *J* = 6.9 Hz), 2.90-3.01 (m, 1H), 3.81 (s, 6H), 6.91 (d, 4H, *J* = 9.2 Hz), 7.06-7.15 (m, 5H); ^{13}C NMR ($CDCl_3$) δ = 21.8, 29.8, 55.6, 114.5, 114.5, 122.2, 122.2, 125.4, 143.9, 143.9, 157.4, 157.5, 158.2, 162.8, 164.2.

Preparation of Enecarbamates and Enamides. Enecarbamates and enamides were prepared from nitriles. Most of the enecarbamates and enamides were prepared using the same procedures as described in the literature.[1]

Aliphatic ketone-derived enecarbamates can be obtained by a Curtius rearrangement as described for the acetone-derived ethyl enecarbamate.[1] Isopropyl methyl ketone-derived ethyl enecarbamate was obtained in a similar procedure, its sythesis is described in the following scheme.



Carboxylic acid **A** was synthesized according to the literature.[3, 4] To the obtained acid **A** (8.7 g, 75.2 mmol) was added $SOCl_2$ (27.8 mL, 5 eq.) and the mixture was heated for 5 h at 50 °C. Then the volatiles were evaporated at r.t. under 50 mmHg for 3 h. The remaining $SOCl_2$ was removed under 150 mmHg reduced pressure at 110°C, then the product was distilled at the same reduced pressure at 125 °C (b.p. : 84 °C; 3.88 g, 39% yield).

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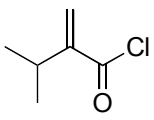
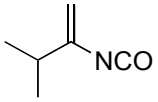
[3] G. Riehs, E. Urban, *Tetrahedron* **1996**, *52*, 1221.
[4] Y. S. Hon, Y.-W. Liu, C.-H. Hsieh, *Tetrahedron* **2004**, *60*, 4837.



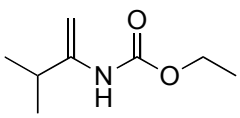
3-Methyl-2-methylenebutanoyl chloride (B) : ^{1}H NMR ($CDCl_3$) δ = 0.74 (d, 3H, *J* = 6.8 Hz), 2.52 (sept, 1H, *J* = 6.8 Hz), 5.28 (s, 1H), 6.16 (s, 1H).

To a solution of NaN_3 (2.3 g, 1.2 eq.) in water (16 mL) was added a solution of **B** (3.88 g, 29.3 mmol) in dibenzyl ether (9 mL) at 0 °C, then the mixture was strongly stirred for 18 h. The two phases were then separated, the organic phase was washed with 16 mL of a saturated solution of $NaHCO_3$, dried over Na_2SO_4 and directly placed in a dropping funnel. The mixture was very slowly added (1 h 30 addition) to 2 mL of dibenzyl ether stirred already at 80 °C. After the addition, the reaction is left stirring at 80 °C for 30 min., then the dropping funnel and condensator were replaced by a distillation apparatus. The distillation is performed under a 200 mmHg reduced pressure at 180 °C to give the desired product (0.94 g, 29% yield). The product was used imediatly in the next step.



3-Methyl-2-methylenebutanoyl chloride (C) : ^{1}H NMR (C_6D_6) δ = 0.77 (d, 3H, *J* = 6.9 Hz), 1.93 (sept, 1H, *J* = 6.8 Hz), 4.26 (s, 1H), 4.44 (s, 1H).

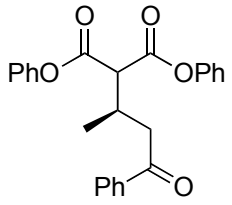
C (470.3 mg, 4.2 mmol) was placed at 0 °C and 0.75 mL of EtOH (3 eq.) was added. This solution was transfered in a flask at 0 °C containing 42.8 mg of NEt_3 (10 mol%). Then the mixture was stirred at 0 °C for 52 h. EtOH was removed at r.t. under 30 mmHg reduced pressure in a kugelröhr aparatus, the product distilled at 180 °C. A mixture of **5j** / isomerized **5j** : 84 / 16 was obtained (302.7 mg, 45% global yield).



Ethyl 3-methylbut-1-en-2-ylcarbamate (5f) : ^{1}H NMR (C_6D_6) δ = 0.78 (d, 6H, *J* = 6.8 Hz), 0.98 (t, 3H, *J* = 7.6 Hz), 1.82 (sept, 1H, *J* = 6.8 Hz), 3.99 (q, 2H, *J* = 6.9 Hz), 5.48 (s, 1H), 5.84 (s, 1H).; HRMS (ESI); Exact mass calcd for $[M+H]^+$, 158.1176. Found 158.1180.

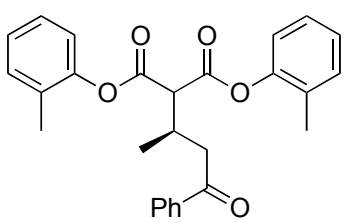
General Procedure for Reaction of Enamides with Alkylidenemalonates and Hydrolysis to the Ketone Product 4. Ligand **1f** (6.5 mg, 0.011 mmol) in CH_2Cl_2 (1.5 mL) was added to a $Cu(OTf)_2$ (3.6 mg, 0.010 mmol) flask under argon. The yellow solution was stirred for over 12 h (the colour gradually turning to brown), and cooled to

–78 °C. Alkylidenemalonate **2** (0.22 mmol) in CH_2Cl_2 (0.7 mL), and enecarbamate **3** or enamide **5** (0.20 mmol) in CH_2Cl_2 (0.8 mL) were added successively. The reaction mixture was stirred at -78 °C for the indicated reaction time, and was quenched by addition of saturated aqueous $NaHCO_3$. The reaction mixture was allowed to warm to rt, extracted with CH_2Cl_2 and dried over anhydrous Na_2SO_4. The residue obtained after removal of solvents was dissolved in THF (3 mL), and a 48% HBr aqueous solution (0.3 mL) was added to the solution. The mixture was stirred at rt for 1.5 min, and then the reaction was quenched by addition of saturated aqueous $NaHCO_3$. The mixture was extracted with CH_2Cl_2, and the organic layer was dried over anhydrous Na_2SO_4. After evaporation of the solvents, the crude product was purified by silica gel chromatography to afford the desired compound **4**.

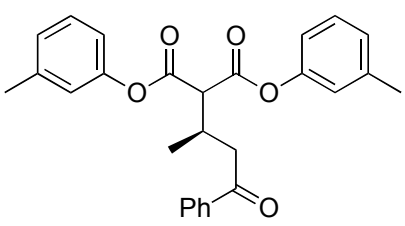


(*R*)-Diphenyl 2-(4-oxo-4-phenylbutan-2-yl)malonate (4a) : ^{1}H NMR ($CDCl_3$) δ = 1.29 (d, 3H, *J* = 6.9 Hz), 3.10 (dd, 1H, *J* = 17.0, 7.8 Hz), 3.42 (sept, 1H, *J* = 6.4 Hz), 3.44 (dd, 1H, *J* = 17.0, 5.3 Hz), 4.02 (d, 1H, *J* = 6.0 Hz), 7.06-7.28 (m, 6H), 7.30-7.47 (m, 6H), 7.52 (t, 1H, *J* = 7.4 Hz), 7.98 (d, 2H, *J* = 6.9 Hz).

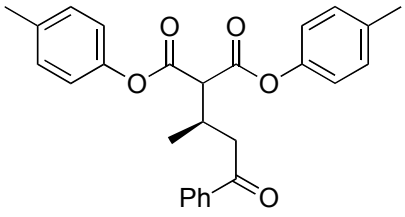
Chiral HPLC; Daicel Chiralcel AD-H; hexane/^{i}PrOH = 9/1, flow rate = 1.0 mL/min: t_R = 16.9 min (*S*), t_R = 18.7 min (*R*).



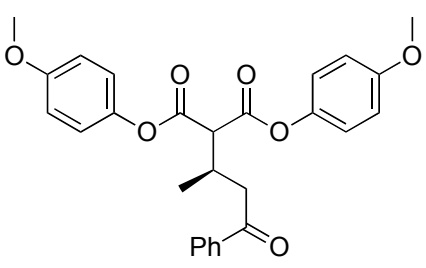
(*R*)-Di *o*-tolyl 2-(4-oxo-4-phenylbutan-2-yl)malonate (4b): ^{1}H NMR ($CDCl_3$); δ = 1.34 (d, 3H, *J* = 6.2 Hz), 2.20 (s, 3H), 2.23 (s, 3H), 3.19 (dd, 1H, *J* = 16.8, 7.2 Hz), 3.27 (sept, 1H, *J* = 6.5 Hz), 3.49 (dd, 1H, *J* = 16.5, 4.5 Hz), 4.17 (d, 1H, *J* = 5.5 Hz), 7.05 (dd, 2H, *J* = 15.8, 6.9 Hz), 7.13-7.28 (m, 6H), 7.46 (t, 2H, 7.9), 7.56 (t, 1H, *J* = 7.2 Hz), 8.00 (d, 2H, *J* = 8.2 Hz); ^{13}C NMR ($CDCl_3$) δ = 16.3, 17.8, 29.6, 42.4, 55.7, 121.5, 126.4, 127.0, 128.1, 128.6, 130.1, 131.3, 133.2, 136.8, 148.9, 166.8, 166.9, 198.5; HRMS (ESI); Exact mass calcd for $[M+Na]^+$, 453.1678. Found 453.1703.; Chiral HPLC; Daicel Chiralcel IA; hexane/CH_2Cl_2 = 15/1, flow rate = 1.0 mL/min: t_R = 47.7 min (*S*), t_R = 57.0 min (*R*).



(*R*)-Di *m*-tolyl 2-(4-oxo-4-phenylbutan-2-yl)malonate (4c): ^{1}H NMR ($CDCl_3$); δ = 1.32 (d, 3H, *J* = 6.9 Hz), 2.35 (s, 3H), 2.36 (s, 3H), 3.13 (dd, 1H, *J* = 16.8, 7.9 Hz), 3.23 (sept, 1H, *J* = 6.6 Hz), 3.48 (dd, 1H, *J* = 17.2, 4.8 Hz), 4.03 (d, 2H, *J* = 6.2 Hz), 6.90-7.00 (m, 4H), 7.06 (t, 2H, *J* = 6.2 Hz), 7.27 (q, 2H, *J* = 7.9 Hz), 7.46 (t, 2H, *J* = 7.9 Hz), 7.56 (t, 1H, *J* = 7.6 Hz), 8.02 (d, 2H, *J* = 6.9 Hz); ^{13}C NMR ($CDCl_3$) δ = 17.8, 21.3, 29.5, 42.5, 55.9, 118.2, 118.2, 121.8, 121.9, 127.1, 128.1, 128.6, 129.2, 133.2, 136.8, 139.8, 150.2, 150.3, 167.1, 167.2, 198.6; HRMS (ESI); Exact mass calcd for $[M+Na]^+$, 453.1678. Found 453.1677.; Chiral HPLC; Daicel Chiralcel IA; hexane/CH_2Cl_2 = 15/1, flow rate = 2.0 mL/min: t_R = 30.9 min (*S*), t_R = 38.9 min (*R*).

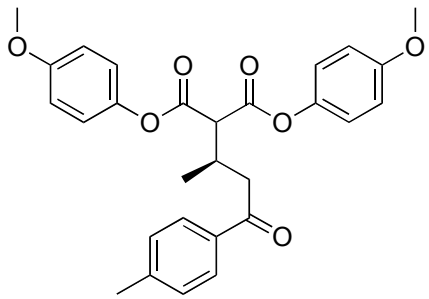


(*R*)-Di *p*-tolyl 2-(4-oxo-4-phenylbutan-2-yl)malonate (4d): ^{1}H NMR ($CDCl_3$); δ = 1.30 (d, 3H, *J* = 6.9 Hz), 2.33 (s, 3H), 2.34 (s, 3H), 3.10 (dd, 1H, *J* = 16.5, 8.2 Hz), 3.22 (sept, 1H, *J* = 6.2 Hz), 3.48 (d, 1H, *J* = 16.5, 4.8 Hz), 4.01 (d, 1H, *J* = 5.5 Hz), 7.01 (dd, 4H, *J* = 16.8, 8.6 Hz), 7.18 (t, 4H, *J* = 7.9 Hz), 7.46 (t, 2H, *J* = 7.9 Hz), 7.55 (t, 1H, *J* = 7.3 Hz), 8.01 (d, 2H, *J* = 8.3 Hz); ^{13}C NMR ($CDCl_3$) δ = 17.8, 20.8, 29.5, 42.5, 55.9, 120.9, 121.0, 128.1, 128.6, 130.0, 133.2, 135.9, 136.8, 148.0, 167.2, 167.3, 198.6; HRMS (ESI); Exact mass calcd for $[M+Na]^+$, 453.1678. Found 453.1687.; Chiral HPLC; Daicel Chiralcel AD; hexane/ ^{i}PrOH = 9/1, flow rate = 1.0 mL/min: t_R = 27.5 min (*S*), t_R = 35.6 min (*R*).

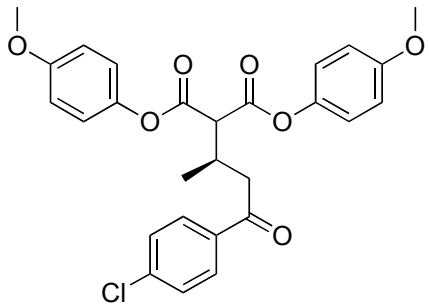


(*R*)-Bis(4-methoxyphenyl)-2-(4-oxo-4-phenylbutan-2-yl)malonate (4e): ^{1}H NMR ($CDCl_3$); δ = 1.30 (d, 3H, *J* = 6.9 Hz), 3.11 (dd, 1H, *J* = 17.2, 8.2 Hz), 3.22 (sept, 1H, *J* = 6.9 Hz), 3.46 (dd, 1H, *J* = 17.2, 5.7 Hz), 3.75-3.85 (m, 6H), 4.00 (dd, 2H, *J* = 5.5 Hz), 6.90 (t, 4H, *J* = 8.3 Hz), 7.05 (dd, 4H, *J* = 16.5, 8.9 Hz), 7.46 (t, 2H, *J* = 7.9 Hz), 7.57 (t, 1H, *J* = 7.2 Hz), 8.01 (d, 2H, *J* = 6.9 Hz); ^{13}C NMR ($CDCl_3$) δ = 17.8, 29.5, 42.5, 55.6, 55.9, 114.5, 122.1, 122.1, 128.2, 128.7, 133.2,

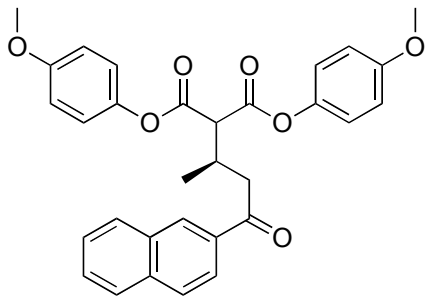
136.8, 143.8, 143.9, 157.6, 167.4, 167.5, 198.6; HRMS (ESI); Exact mass calcd for $[M+Na]^+$, 485.1571. Found 485.1564.; Chiral HPLC; Daicel Chiralcel IA; hexane/CH_2Cl_2 = 4/1, flow rate = 1.0 mL/min: t_R = 31.0 min (*S*), t_R = 40.6 min (*R*).



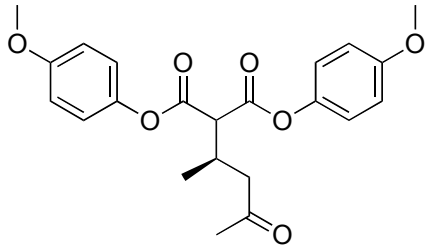
(*R*)-Bis(4-methoxyphenyl)-2-(4-oxo-4-p-tolylbutan-2-yl)malonate (4f): ^1H NMR ($CDCl_3$); δ = 1.29 (d, 3H, *J* = 6.9 Hz), 2.41 (s, 3H), 3.08 (dd, 1H, *J* = 16.9, 7.9 Hz), 3.20 (sept, 1H, *J* = 6.5 Hz), 3.43 (dd, 1H, *J* = 16.9, 5.2 Hz), 3.75-3.85 (m, 6H), 4.00 (d, 1H, *J* = 6.2 Hz), 6.90 (dd, 4H, *J* = 9.6, 6.8 Hz), 7.05 (dd, 4H, *J* = 16.5, 9.0 Hz), 7.26 (d, 2H, *J* = 8.2 Hz), 6.90 (d, 2H, *J* = 8.2 Hz); ^{13}C NMR ($CDCl_3$) δ = 17.8, 21.6, 29.6, 42.4, 55.6, 55.9, 114.5, 122.1, 122.1, 122.2, 128.3, 129.3, 134.4, 143.9, 144.1, 157.5, 167.4, 167.5, 198.3; HRMS (ESI); Exact mass calcd for $[M+Na]^+$, 499.1727. Found 499.1719.; Chiral HPLC; Daicel Chiralcel IA; hexane/CH_2Cl_2 = 4/1, flow rate = 1.0 mL/min: t_R = 33.4 min (*S*), t_R = 45.5 min (*R*).



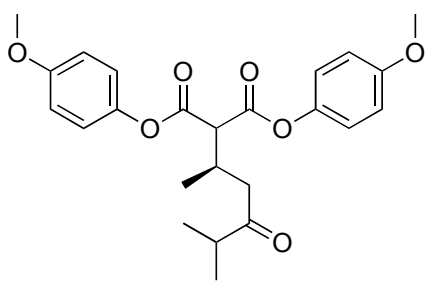
(*R*)-Bis(4-methoxyphenyl)-2-(4-(4-chlorophenyl)-4-oxobutan-2-yl)malonate (4g): ^1H NMR ($CDCl_3$); δ = 1.29 (d, 3H, *J* = 6.9 Hz), 3.05 (dd, 1H, *J* = 17.2, 8.3 Hz), 3.19 (sept, 1H, *J* = 6.5 Hz), 3.45 (dd, 1H, *J* = 17.2, 5.2 Hz), 3.75-3.85 (m, 6H), 3.98 (d, 1H, *J* = 6.2 Hz), 6.88 (dd, 4H, *J* = 9.3, 6.6 Hz), 7.05 (dd, 4H, *J* = 14.7, 9.3 Hz), 7.42 (d, 2H, *J* = 8.9 Hz), 7.95 (d, 2H, *J* = 8.9 Hz); ^{13}C NMR ($CDCl_3$) δ = 17.8, 29.4, 42.4, 55.5, 55.8, 114.5, 122.0, 122.0, 128.9, 129.5, 135.0, 139.6, 143.7, 157.5, 167.3, 167.4, 197.3; HRMS (ESI); Exact mass calcd for $[M+Na]^+$, 519.1181. Found 519.1195.; Chiral HPLC; Daicel Chiralcel IA; hexane/CH_2Cl_2 = 4/1, flow rate = 2.0 mL/min: t_R = 20.1 min (*S*), t_R = 28.1 min (*R*).



(*R*)-Bis(4-methoxyphenyl)-2-(4-(naphthalen-2-yl)-4-oxobutan-2-yl)malonate (4h): ^{1}H NMR ($CDCl_3$); δ = 1.34 (d, 3H, *J* = 6.9 Hz), 3.26 (dd, 1H, *J* = 15.0, 8.2 Hz), 3.29 (sept, 1H, *J* = 6.2 Hz), 3.61 (dd, 1H, *J* = 15.7, 4.2 Hz), 3.75-3.85 (m, 6H), 4.04 (d, 1H, *J* = 6.2 Hz), 6.90 (dd, 4H, *J* = 13.1, 8.9 Hz), 7.07 (dd, 4H, *J* = 22.0, 8.9 Hz), 7.54 (t, 1H, *J* = 7.5 Hz), 7.60 (td, 1H, *J* = 6.6, 1.3 Hz), 7.88 (dd, 2H, *J* = 15.4, 8.6 Hz), 7.96 (d, 1H, *J* = 8.2 Hz), 8.07 (dd, 1H, *J* = 8.6, 1.3 Hz), 8.54 (s, 1H); ^{13}C NMR ($CDCl_3$) δ = 17.9, 29.7, 42.6, 55.6, 55.9, 114.6, 122.1, 122.1, 123.8, 126.8, 127.7, 128.5, 129.7, 130.0, 132.5, 134.1, 135.6, 143.9, 157.6, 167.4, 167.6, 198.6; HRMS (ESI); Exact mass calcd for $[M+Na]^+$, 535.1727. Found 535.1750.; Chiral HPLC; Daicel Chiralcel IA; hexane/CH_2Cl_2 = 4/1, flow rate = 1.0 mL/min: t_R = 45.9 min (*S*), t_R = 73.6 min (*R*).

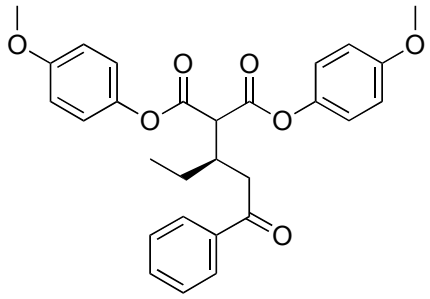


(*R*)-Bis(4-methoxyphenyl)-2-(4-oxopentan-2-yl)malonate (4i): ^{1}H NMR ($CDCl_3$); δ = 1.23 (d, 3H, *J* = 6.9 Hz), 2.17 (s, 3H), 2.60 (dd, 1H, *J* = 17.9, 7.6 Hz), 2.89 (dd, 1H, *J* = 17.9, 5.5 Hz), 3.02 (sept, 1H, *J* = 6.6 Hz), 3.75-3.85 (m, 6H), 3.88 (d, 1H, *J* = 6.2 Hz), 6.90 (d, 4H, *J* = 8.9 Hz), 7.05 (dd, 4H, *J* = 9.3, 2.4 Hz); ^{13}C NMR ($CDCl_3$) δ = 17.9, 28.9, 30.4, 47.4, 55.6, 114.5, 122.1, 143.8, 157.5, 167.3, 167.4, 207.1; HRMS (ESI); Exact mass calcd for $[M+Na]^+$, 423.1414. Found 423.1415.; Chiral HPLC; Daicel Chiralcel IA; hexane/CH_2Cl_2 = 4/1, flow rate = 1.0 mL/min: t_R = 34.1 min (*S*), t_R = 38.6 min (*R*).

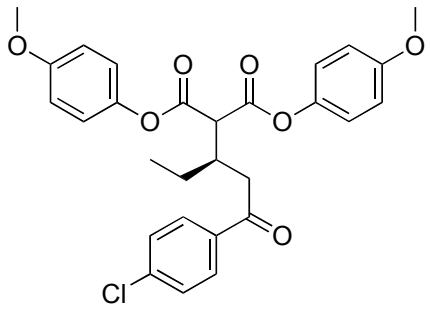


(*R*)-Bis(4-methoxyphenyl)-2-(5-methyl-4-oxohexan-2-yl)malonate (4j): ^{1}H NMR ($CDCl_3$); δ = 1.10 (d, 3H, *J* = 6.9 Hz), 1.11 (d, 3H, *J* = 6.9 Hz), 1.22 (d, 3H, *J* = 6.9 Hz), 2.61 (sept, 1H, *J* = 6.9 Hz), 2.64 (dd, 1H, *J* = 17.9, 7.6 Hz), 2.89 (dd, 1H, *J* = 17.9, 5.5 Hz), 3.03 (sept, 1H, *J* = 6.9 Hz), 3.75-3.85 (m, 6H), 4.30 (d, 1H, *J* = 5.5 Hz), 6.89 (d, 4H, *J* = 8.9 Hz), 7.04 (dd, 4H, *J* = 9.7, 3.5 Hz); ^{13}C NMR ($CDCl_3$) δ = 17.7, 18.0, 18.2, 28.8, 41.0, 44.1, 55.4, 55.5, 114.5, 122.0, 143.7,

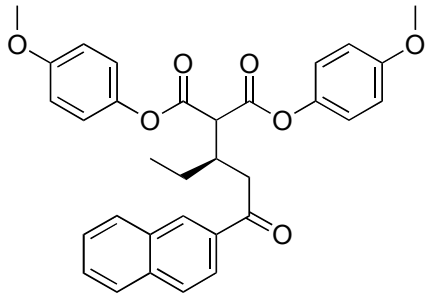
143.8, 157.5, 167.3, 167.4, 213.0; HRMS (ESI); Exact mass calcd for $[M+Na]^+$, 451.1727. Found 451.1740.; Chiral HPLC; Daicel Chiralcel IA; hexane/CH_2Cl_2 = 4/1, flow rate = 0.5 mL/min: t_R = 49.5 min (*S*), t_R = 56.2 min (*R*).



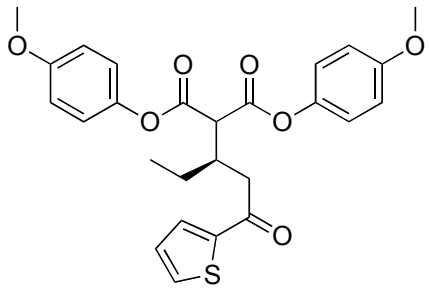
(*R*)-Bis(4-methoxyphenyl)-2-(1-oxo-1-phenylpentan-3-yl)malonate (4k): ^{1}H NMR ($CDCl_3$); δ = 1.06 (t, 3H, *J* = 7.6 Hz), 1.63-1.80 (m, 2H), 3.07 (sext, 1H, *J* = 6.2 Hz), 3.22 (dd, 1H, *J* = 17.2, 7.2 Hz), 3.48 (dd, 1H, *J* = 17.2, 5.5 Hz), 3.75-3.85 (m, 6H), 4.15 (d, 1H, *J* = 5.5 Hz), 6.88 (dd, 4H, *J* = 11.0, 9.0 Hz), 7.05 (dd, 4H, *J* = 15.8, 9.6 Hz), 7.44 (t, 2H, *J* = 7.9 Hz), 7.54 (t, 1H, *J* = 7.6 Hz), 8.00 (d, 2H, *J* = 8.3 Hz); ^{13}C NMR ($CDCl_3$) δ = 11.6, 25.1, 35.6, 39.7, 53.7, 55.5, 114.5, 122.0, 122.1, 128.1, 128.6, 133.1, 136.9, 143.8, 157.5, 167.5, 167.7, 198.9; HRMS (ESI); Exact mass calcd for $[M+Na]^+$, 499.1733. Found 499.1722.; Chiral HPLC; Daicel Chiralcel IA; hexane/CH_2Cl_2 = 4/1, flow rate = 0.5 mL/min: t_R = 42.9 min (*S*), t_R = 54.5 min (*R*).



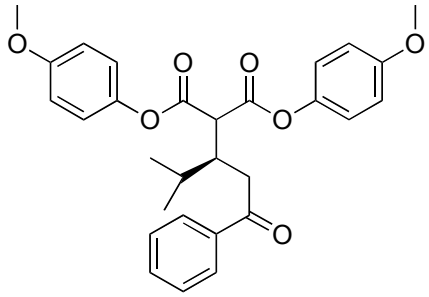
(*R*)-Bis(4-methoxyphenyl)-2-(1-(4-chlorophenyl)-1-oxopentan-3-yl)malonate (4l): ^{1}H NMR ($CDCl_3$); δ = 1.05 (t, 3H, *J* = 7.6 Hz), 1.61-1.77 (m, 2H), 3.04 (sext, 1H, *J* = 6.2 Hz), 3.15 (dd, 1H, *J* = 17.2, 7.2 Hz), 3.46 (dd, 1H, *J* = 17.2, 5.5 Hz), 3.75-3.85 (m, 6H), 4.12 (d, 1H, *J* = 4.8 Hz), 6.89 (t, 4H, *J* = 9.6, 9.0 Hz), 7.04 (dd, 4H, *J* = 13.8, 7.1 Hz), 7.42 (d, 2H, *J* = 8.3 Hz), 7.94 (d, 2H, *J* = 8.3 Hz); ^{13}C NMR ($CDCl_3$) δ = 11.7, 25.2, 35.7, 39.8, 53.6, 55.6, 114.6, 122.0, 122.1, 122.2, 128.9, 129.6, 135.2, 139.6, 143.8, 157.6, 167.5, 167.7, 197.7; HRMS (FAB); Exact mass calcd for $[M+Na]^+$, 533.1338. Found 533.1325.; Chiral HPLC; Daicel Chiralcel IA; hexane/CH_2Cl_2 = 4/1, flow rate = 1.0 mL/min: t_R = 27.8 min (*S*), t_R = 34.7 min (*R*).



(*R*)-Bis(4-methoxyphenyl-2-(1-(naphthalen-2-yl)-1-oxopentan-3-yl)malonate (4m): ^{1}H NMR ($CDCl_3$); δ = 1.09 (t, 3H, *J* = 7.6 Hz), 1.68-1.84 (m, 2H), 3.14 (sext, 1H, *J* = 5.7 Hz), 3.36 (dd, 1H, *J* = 17.6, 7.2 Hz), 3.61 (dd, 1H, *J* = 17.6, 5.9 Hz), 3.79 (s, 3H), 3.80 (s, 3H), 4.19 (d, 1H, *J* = 5.5 Hz), 6.89 (dd, 4H, *J* = 18.6, 9.0 Hz), 7.04 (dd, 4H, *J* = 22.0, 9.0 Hz), 7.54 (t, 1H, *J* = 7.6 Hz), 7.60 (t, 1H, *J* = 7.6 Hz), 7.87 (dd, 2H, *J* = 13.8, 8.3 Hz), 7.95 (d, 1H, *J* = 8.3 Hz), 8.07 (dd, 1H, *J* = 8.6, 1.6 Hz), 8.54 (s, 1H); ^{13}C NMR ($CDCl_3$) δ = 11.8, 25.2, 35.9, 39.9, 53.8, 55.6, 114.5, 114.6, 122.1, 122.2, 123.8, 126.8, 127.7, 128.5, 129.7, 132.5, 134.3, 135.6, 143.9, 143.9, 157.6, 167.7, 167.8, 198.9; HRMS (ESI); Exact mass calcd for $[M+Na]^+$, 549.1884. Found 549.1880.; Chiral HPLC; Daicel Chiralcel IA; hexane/CH_2Cl_2 = 4/1, flow rate = 1.0 mL/min: t_R = 31.6 min (*S*), t_R = 45.2 min (*R*).



(*R*)-Bis(4-methoxyphenyl)-2-(1-oxo-1-(thiophen-2-yl)pentan-3-yl)malonate (4n): ^{1}H NMR ($CDCl_3$); δ = 1.06 (t, 3H, *J* = 7.6 Hz), 1.64-1.80 (s, 2H), 3.02 (sext, 1H, *J* = 6.0 Hz), 3.15 (dd, 1H, *J* = 16.5, 7.6 Hz), 3.40 (dd, 1H, *J* = 16.5, 5.5 Hz), 3.78 (s, 3H), 3.79 (s, 3H), 4.13 (d, 1H, *J* = 5.5 Hz), 6.84 (dd, 4H, *J* = 9.0, 6.9 Hz), 7.05 (t, 4H, *J* = 9.3 Hz), 7.10 (dd, 1H, *J* = 5.2, 3.9 Hz), 7.62 (d, 1H, *J* = 4.8 Hz), 7.80 (d, 1H, *J* = 4.8 Hz); ^{13}C NMR ($CDCl_3$) δ = 11.6, 25.0, 36.11, 40.4, 53.6, 55.5, 114.5, 122.0, 122.1, 128.2, 132.2, 133.8, 143.8, 144.2, 157.5, 167.4, 167.6, 191.8; HRMS (ESI); Exact mass calcd for $[M+Na]^+$, 505.1297. Found 505.1285.; Chiral HPLC; Daicel Chiralcel IA; hexane/CH_2Cl_2 = 4/1, flow rate = 1.0 mL/min: t_R = 27.2 min (*S*), t_R = 32.1 min (*R*).

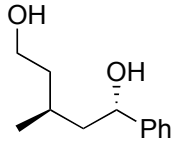


(*S*)-Bis(4-methoxyphenyl)-2-(4-methyl-1-oxo-1-phenylpentan-3-yl)malonate (4o): ^{1}H NMR ($CDCl_3$); δ = 1.00 (d, 3H, *J* = 6.9 Hz), 1.09 (d, 3H, *J* = 6.9 Hz), 2.01 (oct, 1H, *J* = 7.2 Hz), 3.22 (quint, 1H, *J* = 5.5 Hz), 3.27 (dd, 1H, *J* = 18.2, 5.9 Hz), 3.47 (dd, 1H, *J*

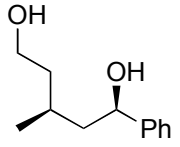
= 18.2, 5.2 Hz), 3.78 (s, 3H), 3.79 (s, 3H), 4.12 (d, 1H, *J* = 5.0 Hz), 6.88 (dd, 4H, *J* = 8.9, 6.2 Hz), 7.04 (dd, 4H, *J* = 8.9, 1.4 Hz), 7.44 (t, 2H, *J* = 7.9 Hz), 7.53 (t, 1H, *J* = 7.6 Hz), 8.00 (d, 2H, *J* = 8.2 Hz); ^{13}C NMR ($CDCl_3$) δ = 19.6, 20.6, 30.8, 37.9, 39.2, 53.2, 55.5, 114.5, 122.1, 122.1, 128.1, 128.5, 133.0, 136.9, 143.8, 143.9, 157.5, 167.8, 168.2, 198.7; HRMS (ESI); Exact mass calcd for $[M+H]^+$, 513.1884. Found 513.1874.; Chiral HPLC; Daicel Chiralcel IA; hexane/CH_2Cl_2 = 4/1, flow rate = 0.5 mL/min: t_R = 33.1 min (*R*), t_R = 36.8 min (*S*).

Determination of the Absolute Configuration of 4e. Compound **7** was prepared from **4e** following literature known procedures for transesterification,[5] and for Krapcho decarboxymethylation.[6]

Compound **7** (70.6 mg, 0.32 mmol) was dissolved with dry THF (5 mL) and stirred at 0 °C. $LiBH_4$ (35.0 mg, 5 eq.) was quickly added to the solution and the reaction was stirred for 14 h. Then the reaction was quenched by addition of 15 mL of a saturated solution of NH_4Cl, extracted three times with AcOEt, dried and evaporated. Purification by preparative TLC (Hexane / AcOEt : 1/1) gave **8** (58 mg, 93% yield). NMR showed clearly that a diastereoisomeric mixture of 1/1 was obtained. The diastereoisomers were separated on little scale on preparative TLC (Hexane / AcOEt : 1/1; 3 times eluted). Using the literature known HPLC conditions as in the literature,[7] we obtained the absolute configuration of our product. The absolute configuration of **4e** is *R*.



(1*S*, 3*S*)-3-Methyl-1-phenylpentane-1,5-diol (*Syn*-8): ^1H NMR ($CDCl_3$) δ = 0.91 (d, 3H, *J* = 6.4 Hz), 1.30-1.43 (m, 1H), 1.57-1.75 (m, 4H), 3.60-3.70 (m, 2H), 4.74 (t, 1H, *J* = 6.4 Hz), 7.2-7.5 (m, 5H).

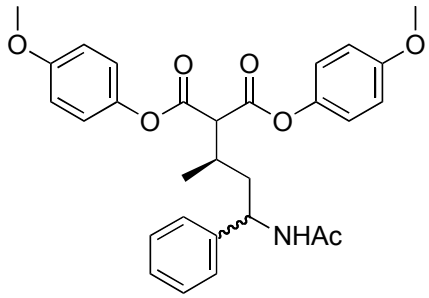


(1*R*, 3*S*)-3-Methyl-1-phenylpentane-1,5-diol (*Anti*-8): ^1H NMR ($CDCl_3$) δ = 0.94 (d, 3H, *J* = 6.4 Hz), 1.32-1.45 (m, 1H), 1.45-1.60 (m,

[5] H. R. Nahm, J. R. Potnick, P. S. White, J. S. Johnson, *J. Am. Chem. Soc.* **2006**, *128*, 2751.
[6] A. P. Krapcho, *Synthesis* **1982**, 805.
[7] E. Brenna, C. Fuganti, S. Ronzani, S. Serra, *Can. J. Chem.* **2002**, *80*, 714

2H), 1.73-1.90 (m, 2H), 3.56-3.74 (m, 2H), 4.72 (dd, 1H, *J* = 9.4, 3.9 Hz), 7.2-7.4 (m, 5H).

Reduction of the Imine. Ligand **1f** (6.5 mg, 0.011 mmol) in CH_2Cl_2 (1.5 mL) was added to a $Cu(OTf)_2$ (3.6 mg, 0.010 mmol) flask under argon. The yellow solution was stirred for over 12 h (the colour gradually turning to brown), and cooled to -78 °C. Alkylidenemalonate **2e** (75.3 mg, 0.22 mmol) in CH_2Cl_2 (0.7 mL), and then enamide **5** (32.2 mg, 0.20 mmol) in CH_2Cl_2 (0.8 mL) were added succesively. The reaction mixture was stirred at -78 °C for 10 h, and was quenched by addition of saturated aqueous $NaHCO_3$. The reaction mixture was allowed to warm to rt, extracted with CH_2Cl_2 and dried over anhydrous Na_2SO_4. The residue obtained after removal of solvents was dissolved in dry THF (3 mL) under Ar, the mixture cooled to 0 °C, and $NaBH_4$ (15.2 mg, 2 eq.) was added quickly to the solution. The reaction was allowed to proceed for 10 min. and then quenched by water. The mixture was extracted with CH_2Cl_2 and dried over anhydrous Na_2SO_4. After purification on preparative TLC (Hexane / Acetone : 2 / 1), 64 mg of a clean mixture of the 2 diastereomers (**9**, d.r. = 59/41) was obtained in 63% yield.



Bis(4-methoxyphenyl) 2-((*R*)-4-acetamido-4-phenylbutan-2-yl)malonate (a mixture of diastereoisomers) (9): ^1H NMR ($CDCl_3$); δ = 1.33 (t, 3H, *J* = 7.1 Hz), 1.60-1.75 (m, 4.2H, *J*), 1.99 (dt, 0.4H, *J* = 14.3, 8.3 Hz), 2.21 (t, 0.5H, *J* = 14.2 Hz), 2.28-2.34 (m, 0.4H), 2.57 (sept, 0.4H, *J* = 6.7 Hz), 2.62-2.75 (m, 0.6H), 3.22 (s, 6H), 3.75 (d, 0.6H, *J* = 6.7 Hz), 4.12 (d, 0.4H, *J* = 5.5 Hz), 5.39-5.53 (m, 1H), 6.22 (d, 0.4H, *J* = 8.2 Hz), 6.45 (d, 0.6H, *J* = 8.8 Hz), 6.60-6.72 (m, 4H), 6.95-7.05 (t, 1.6H, *J* = 8.2 Hz), 7.05-7.12 (dd, 2.4H, *J* = 9.1, 3.0 Hz), 7.13-7.23 (m, 3H), 7.27 (d, 1.2H, *J* = 7.3 Hz), 7.38 (d, 0.8H, *J* = 7.3 Hz); ^{13}C NMR ($CDCl_3$) δ = 17.1, 17.6, 23.3, 30.3, 30.4, 39.8, 41.7, 51.2, 51.3, 55.5, 56.3, 57.0, 114.5, 114.6, 122.0, 122.0, 126.0, 126.9, 127.2, 127.5, 127.7, 128.0, 128.6, 128.8, 140.9, 142.8, 143.6, 143.7, 143.8, 157.5, 157.6, 167.2, 167.5, 167.7, 167.8, 169.1, 169.9.

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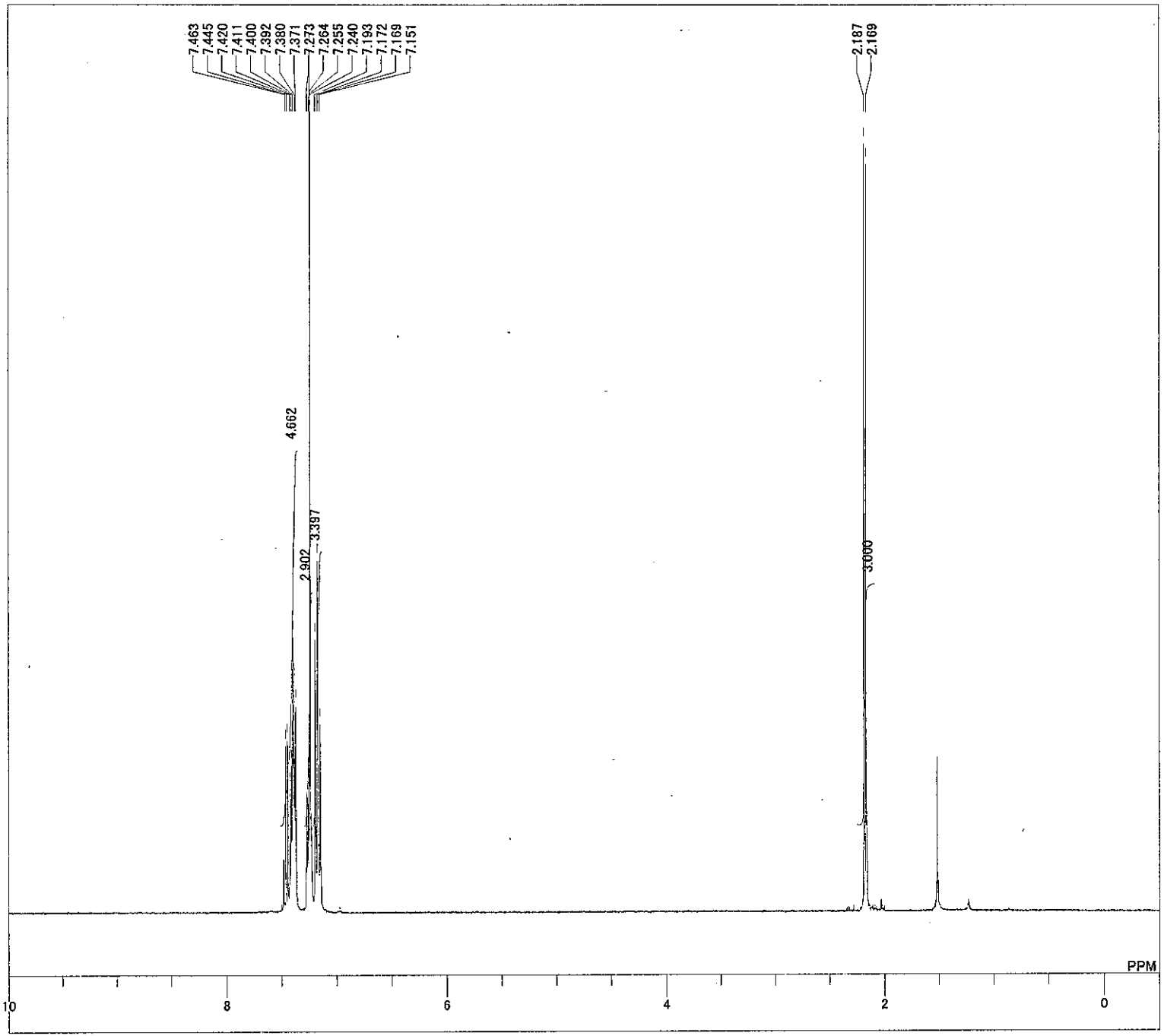

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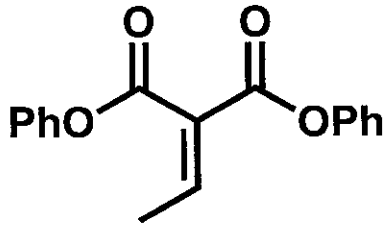
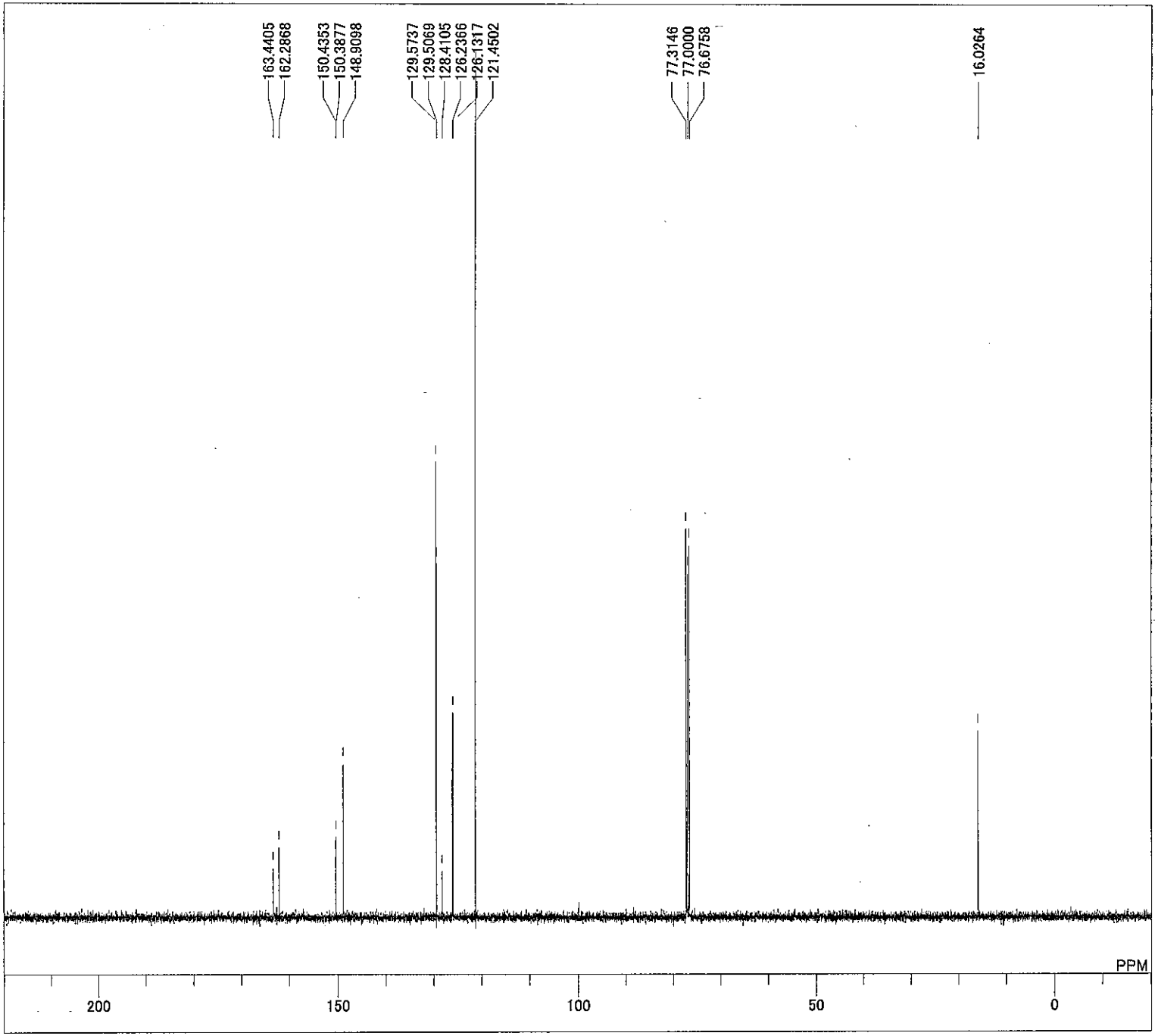
IRNUC	1H
CTEMP	22.6 c
SLVNT	CDCL3
EXREF	7.24 ppm
BF	0.12 Hz
RGAIN	38

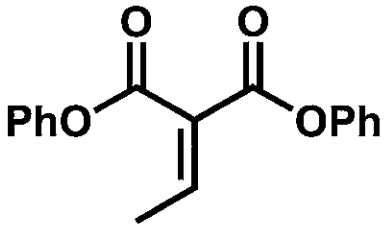


2a

¥¥Eca¥data¥fb64prana.1

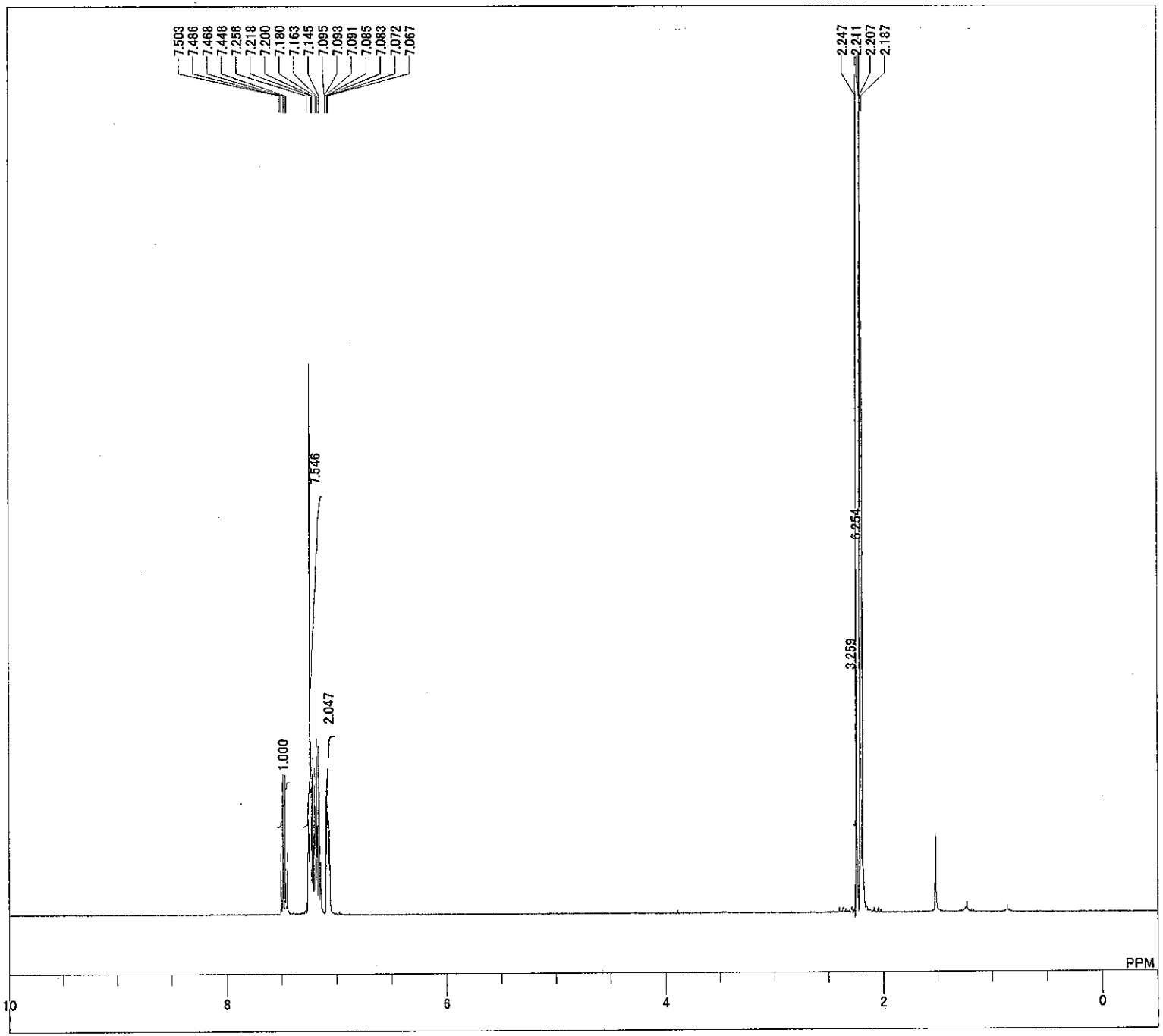


DFILE	¥¥Eca¥data¥fb64prana.1
COMNT	
DATIM	10-04-2006 08:18:20
OBNUC	13C
EXMOD	single_pulse_dec
OBFRQ	100.53 MHz
OBSET	5.35 KHz
OBFIN	5.86 Hz
POINT	32768
FREQU	31407.03 Hz
SCANS	322
ACQTM	1.0433 sec
PD	2.0000 sec
PW1	3.33 usec
IRNUC	1H
CTEMP	23.3 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	50

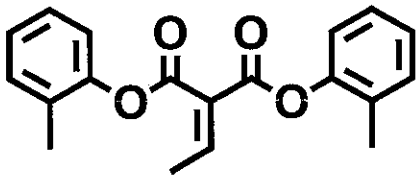


2a

C:¥Documents and Settings¥delta¥My Documents¥Personal Folder¥florian¥fb75f1recrys.als

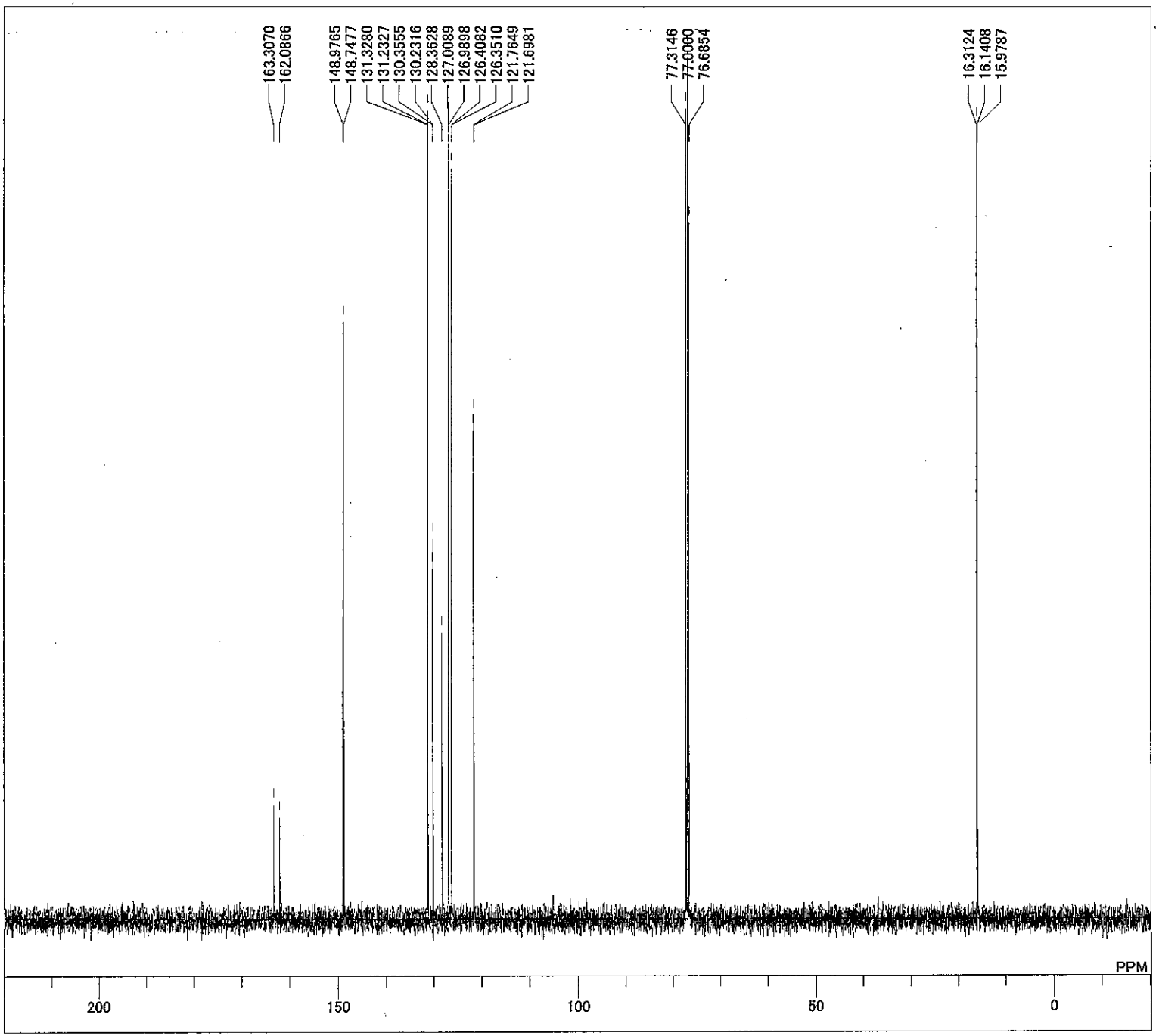


DFILE	C:¥Documents and Settings¥delta¥My Documents¥Persona
COMNT	
DATIM	03-04-2006 09:43:57
OBNUC	1H
EXMOD	single_pulse.ex2
OBFRQ	399.78 MHz
OBSET	4.19 KHz
OBFIN	7.29 Hz
POINT	13107
FREQU	6002.31 Hz
SCANS	8
ACQTM	2.1837 sec
PD	2.0000 sec
PW1	5.50 usec
IRNUC	1H
CTEMP	22.6 c
SLVNT	CDCL3
EXREF	7.24 ppm
BF	0.12 Hz
RGAIN	38

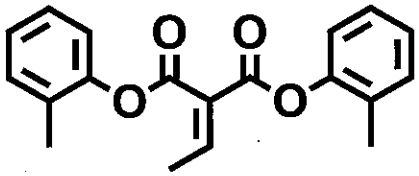


2b

¥¥Eca¥data¥michaelomeverif.2

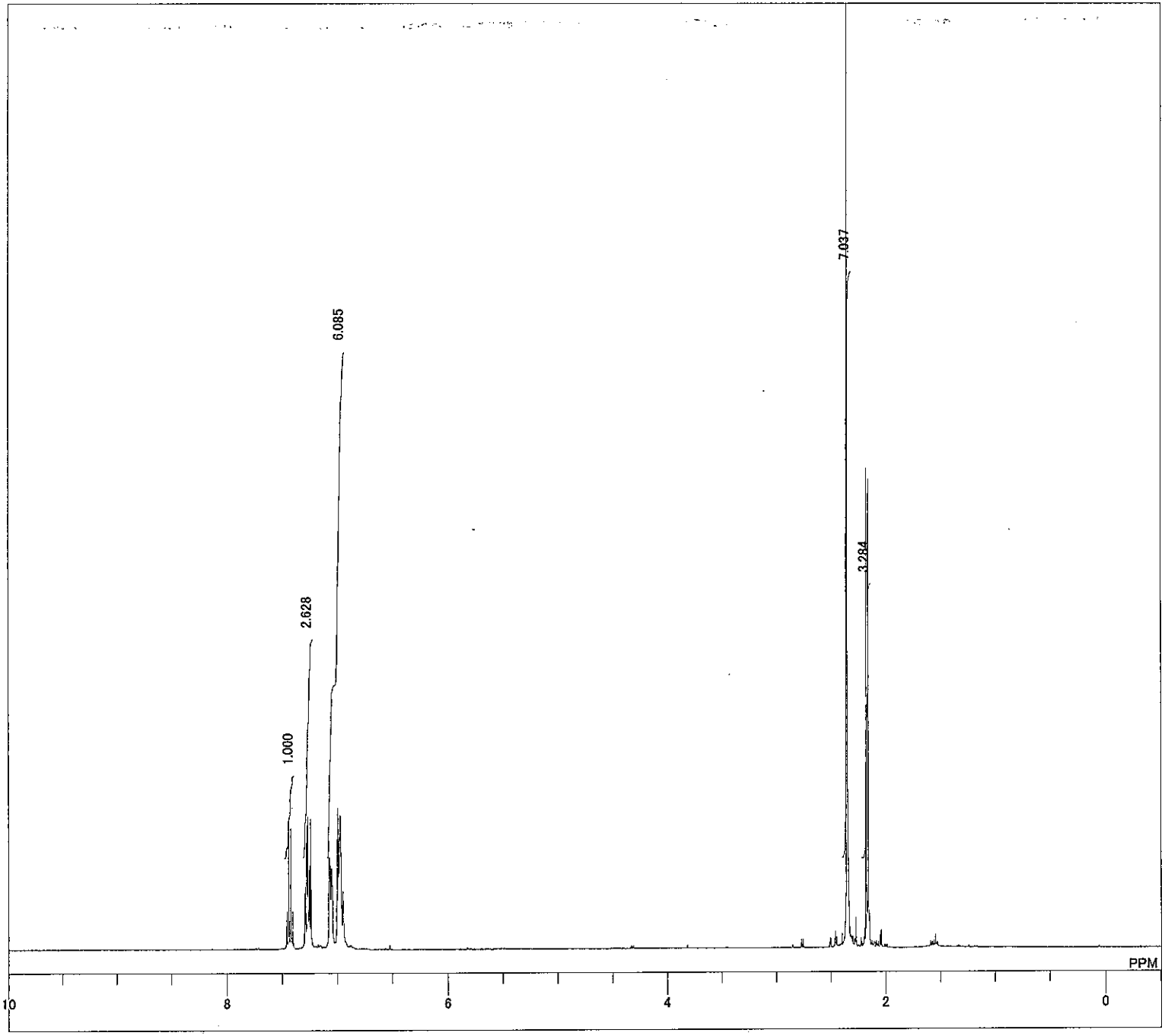


DFILE	¥¥Eca¥data¥michaelomeverif.2
COMNT	
DATIM	10-04-2006 08:38:22
OBNUC	13C
EXMOD	single_pulse_dec
OBFRQ	100.53 MHz
OBSET	5.35 KHz
OBFIN	5.86 Hz
POINT	32768
FREQU	31407.03 Hz
SCANS	253
ACQTM	1.0433 sec
PD	2.0000 sec
PW1	3.33 usec
IRNUC	1H
CTEMP	23.3 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	54

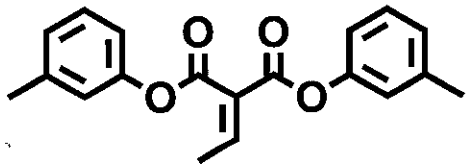


2b

C:¥Documents and Settings¥delta¥My Documents¥Personal Folder¥florian¥fb717779res.als

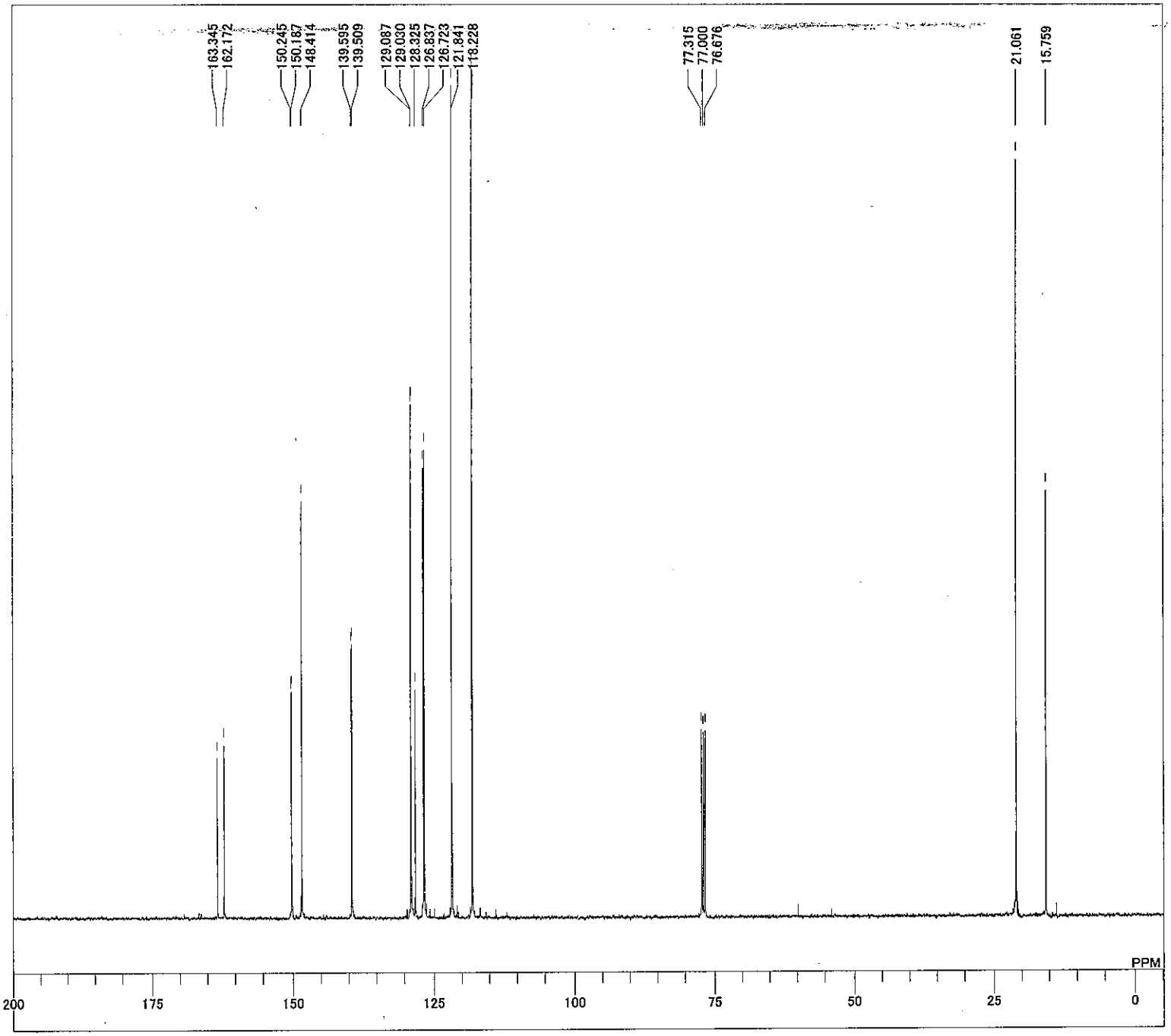


DFILE	C:¥Documents and Settings¥delta¥My Documents¥Personal
COMNT	
DATIM	06-04-2006 20:23:46
OBNUC	1H
EXMOD	single_pulse.ex2
OBFRQ	399.78 MHz
OBSET	4.19 KHz
OBFIN	7.29 Hz
POINT	13107
FREQU	6002.31 Hz
SCANS	8
ACQTM	2.1837 sec
PD	2.0000 sec
PW1	5.50 usec
IRNUC	1H
CTEMP	23.6 c
SLVNT	CDCL3
EXREF	7.24 ppm
BF	0.12 Hz
RGAIN	34

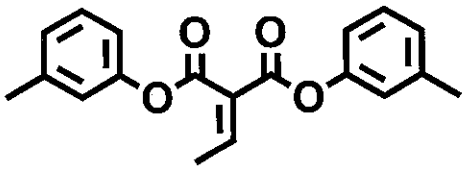


2c

C:¥Documents and Settings¥delta¥My Documents¥Personal Folder¥florian¥fb717779respuriprana.als

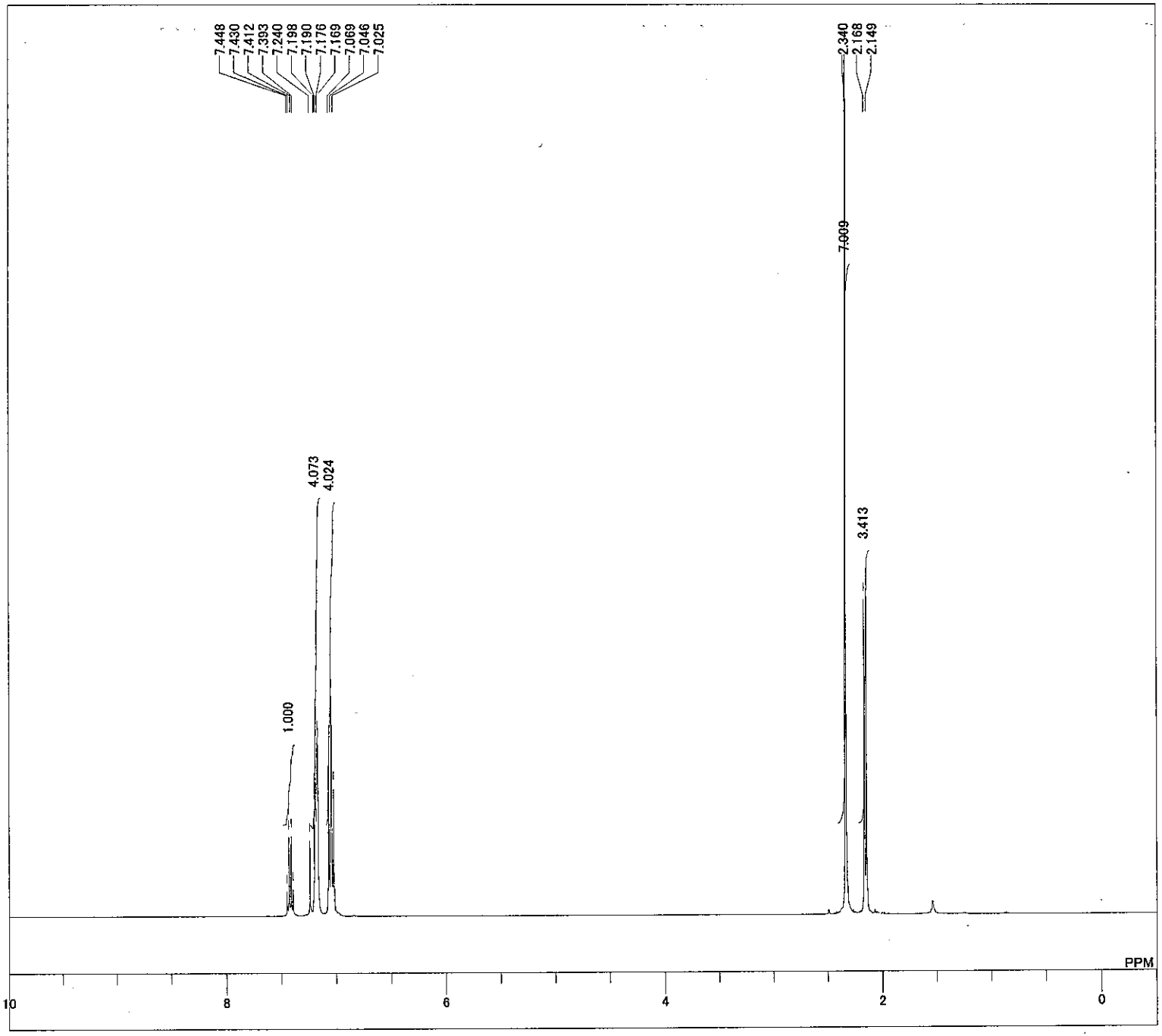


DFILE	C:¥Documents and Settings¥delta¥My Documents¥Person
COMNT	
DATIM	10-04-2006 08:58:37
OBNUC	13C
EXMOD	single_pulse_dec
OBFRQ	100.53 MHz
OBSET	5.35 KHz
OBFIN	5.86 Hz
POINT	32768
FREQU	31407.04 Hz
SCANS	273
ACQTM	1.0433 sec
PD	2.0000 sec
PW1	3.33 usec
IRNUC	1H
CTEMP	23.4 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	50

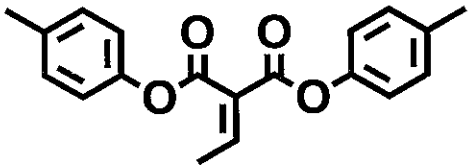


2c

C:¥Documents and Settings¥delta¥My Documents¥Personal Folder¥florian¥fb72recrys.als



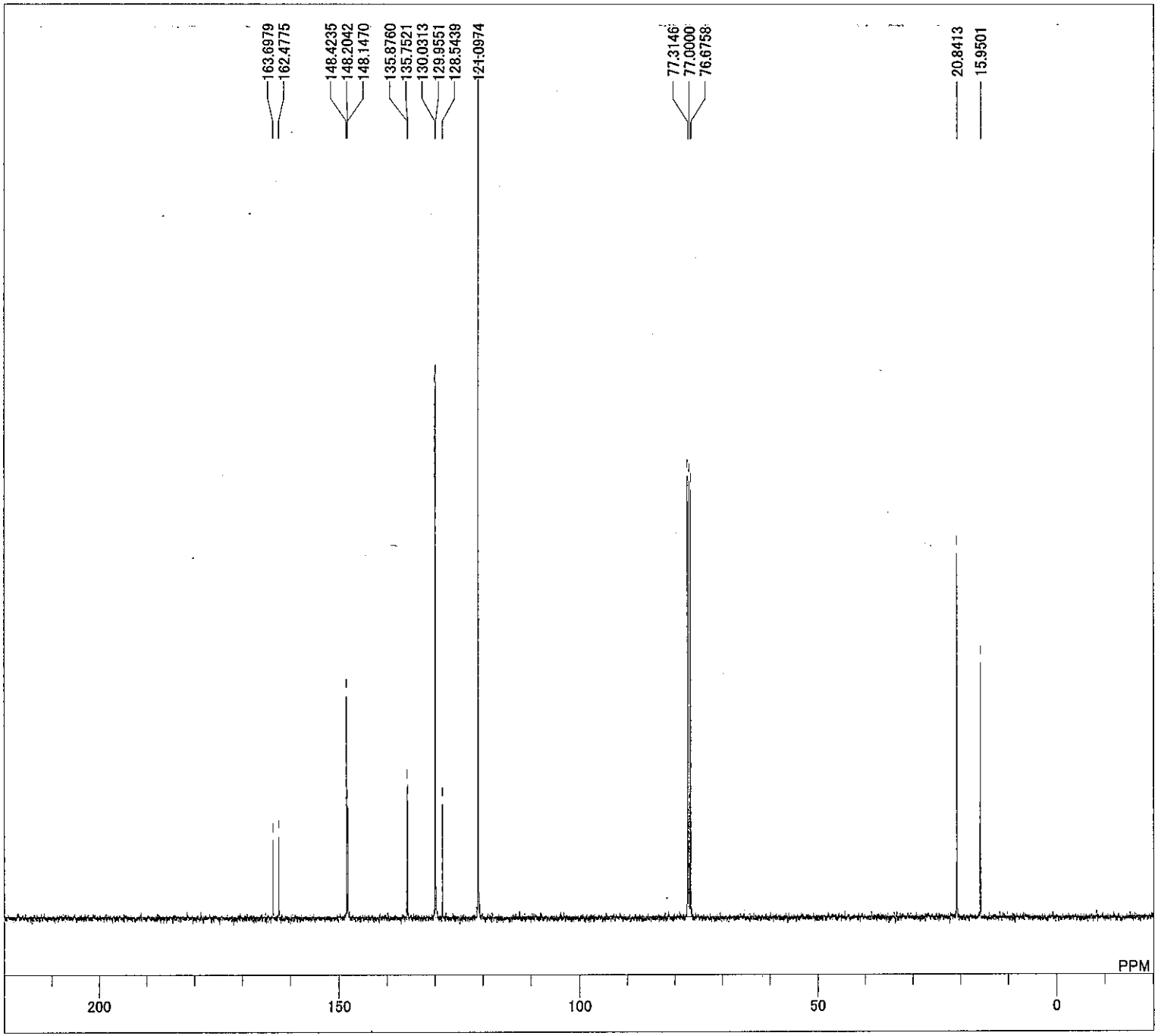
DFILE	C:¥Documents and Settings¥delta¥My Documents¥Persona
COMNT	
DATIM	31-03-2006 13:30:53
OBNUC	1H
EXMOD	single_pulse.ex2
OBFRQ	399.78 MHz
OBSET	4.19 KHz
OBFIN	7.29 Hz
POINT	13107
FREQU	6002.31 Hz
SCANS	8
ACQTM	2.1837 sec
PD	2.0000 sec
PW1	5.50 usec
IRNUC	1H
CTEMP	20.9 c
SLVNT	CDCL3
EXREF	7.24 ppm
BF	0.12 Hz
RGAIN	34



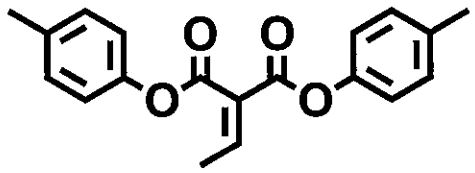
2d

2

¥¥Eca¥data¥fb74prana.3

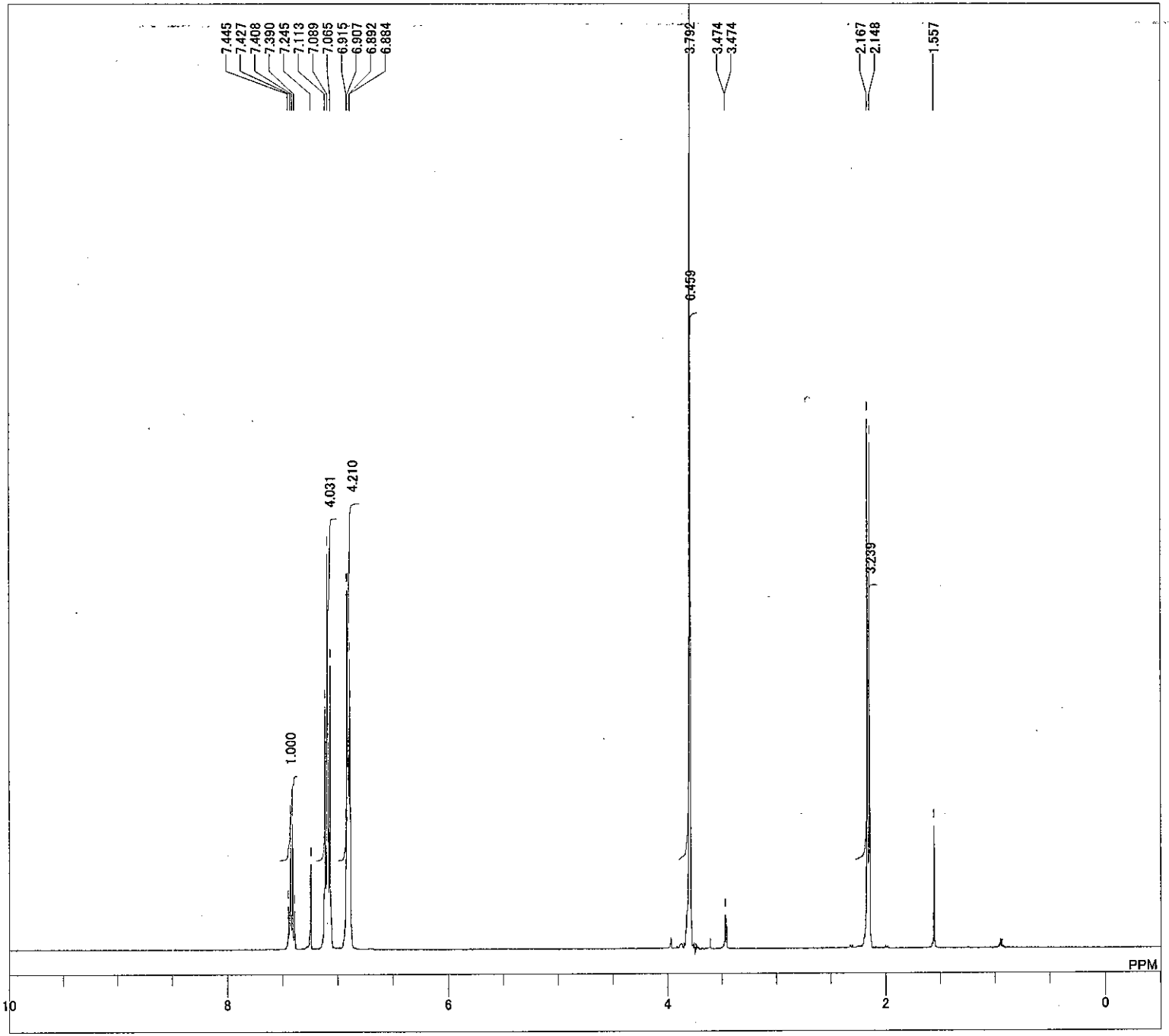


DFILE	¥¥Eca¥data¥fb74prana.3
COMNT	
DATIM	10-04-2006 09:24:48
OBNUC	13C
EXMOD	single_pulse_dec
OBFRQ	100.53 MHz
OBSET	5.35 KHz
OBFIN	5.86 Hz
POINT	40961
FREQU	39259.39 Hz
SCANS	385
ACQTM	1.0433 sec
PD	2.0000 sec
PW1	3.33 usec
IRNUC	1H
CTEMP	23.5 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	58

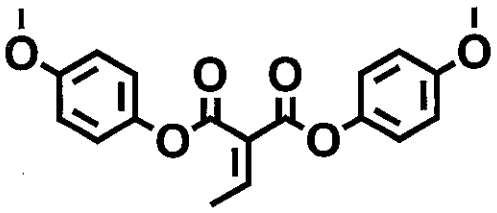


2d

C:¥Documents and Settings¥delta¥My Documents¥Personal Folder¥florian¥fb347recrys.als

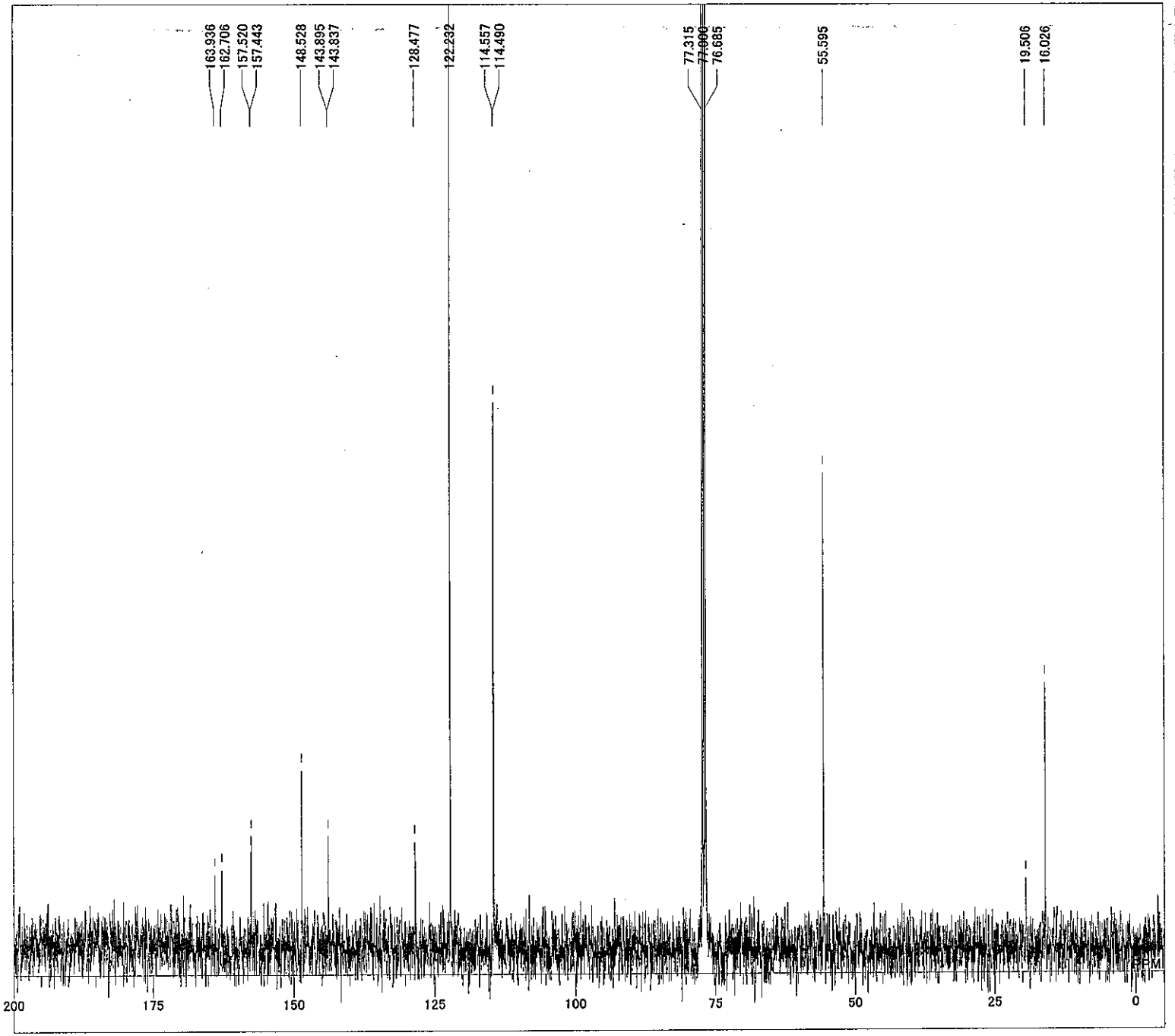


DFILE	C:¥Documents and Settings¥delta¥My Documents¥Persona
COMNT	
DATIM	21-03-2007 22:01:27
OBNUC	1H
EXMOD	single_pulse.ex2
OBFRQ	399.78 MHz
OBSET	4.19 KHz
OBFIN	7.29 Hz
POINT	16384
FREQU	7503.00 Hz
SCANS	8
ACQTM	2.1837 sec
PD	2.0000 sec
PW1	5.50 usec
IRNUC	1H
CTEMP	24.3 c
SLVNT	CDCL3
EXREF	7.24 ppm
BF	0.12 Hz
RGAIN	34

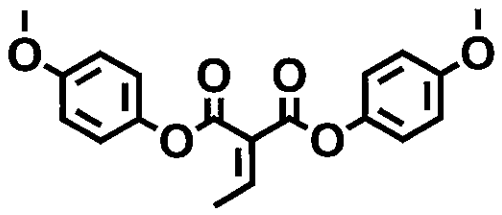


2e

C:¥Documents and Settings¥delta¥My Documents¥Personal Folder¥florian¥pmeoethylidenemalonatec13.als

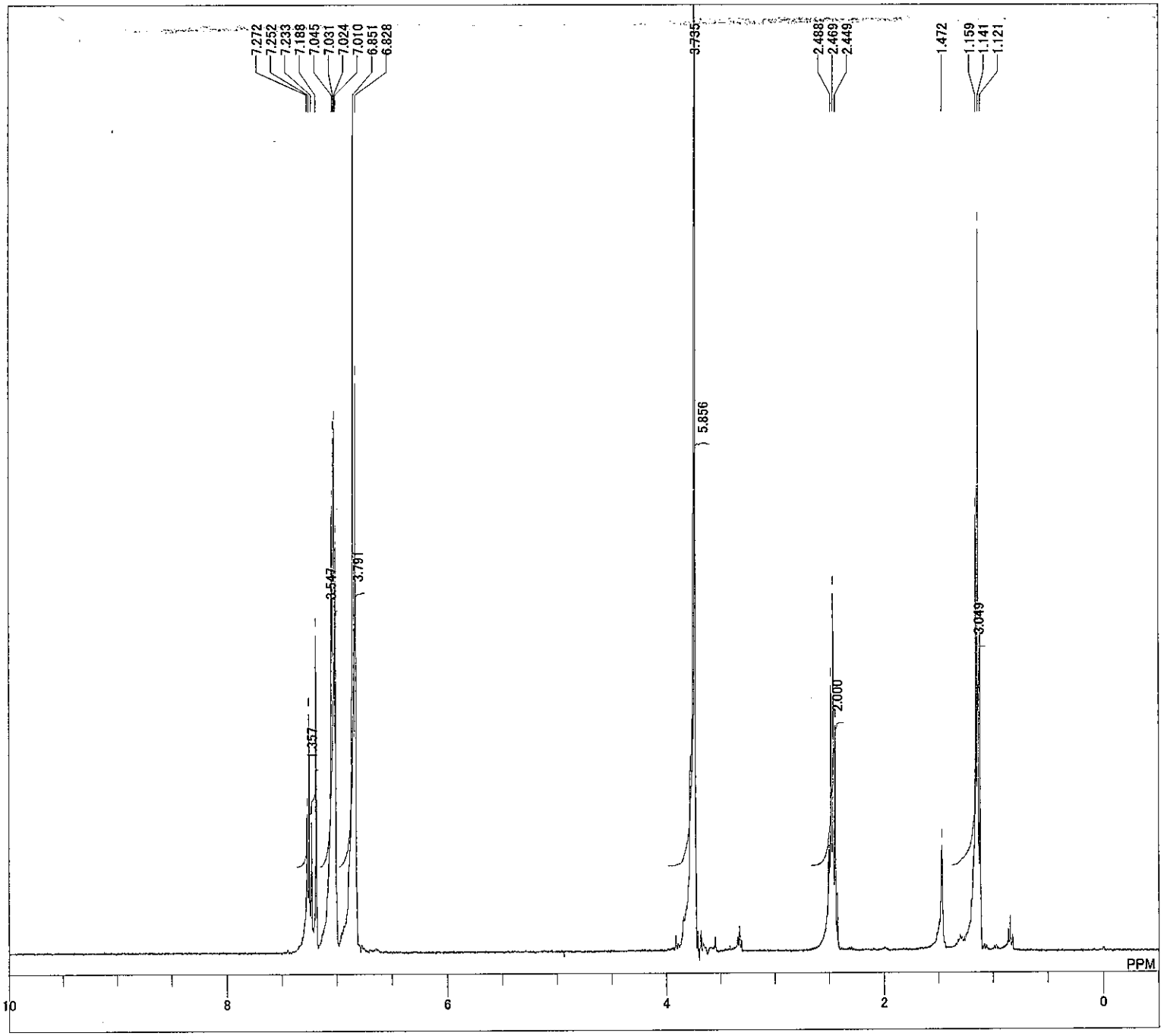


DFILE	C:¥Documents and Settings¥delta¥My Documents¥Person
COMNT	
DATIM	04-06-2007 18:01:52
OBNUC	13C
EXMOD	single_pulse_dec
OBFRQ	100.53 MHz
OBSET	5.35 KHz
OBFIN	5.86 Hz
POINT	32768
FREQU	31407.04 Hz
SCANS	139
ACQTM	1.0433 sec
PD	2.0000 sec
PW1	2.83 usec
IRNUC	1H
CTEMP	20.6 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	50

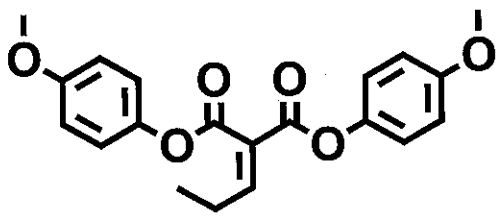


2e

C:¥Documents and Settings¥delta¥My Documents¥Personal Folder¥florian¥fb265recrys.als

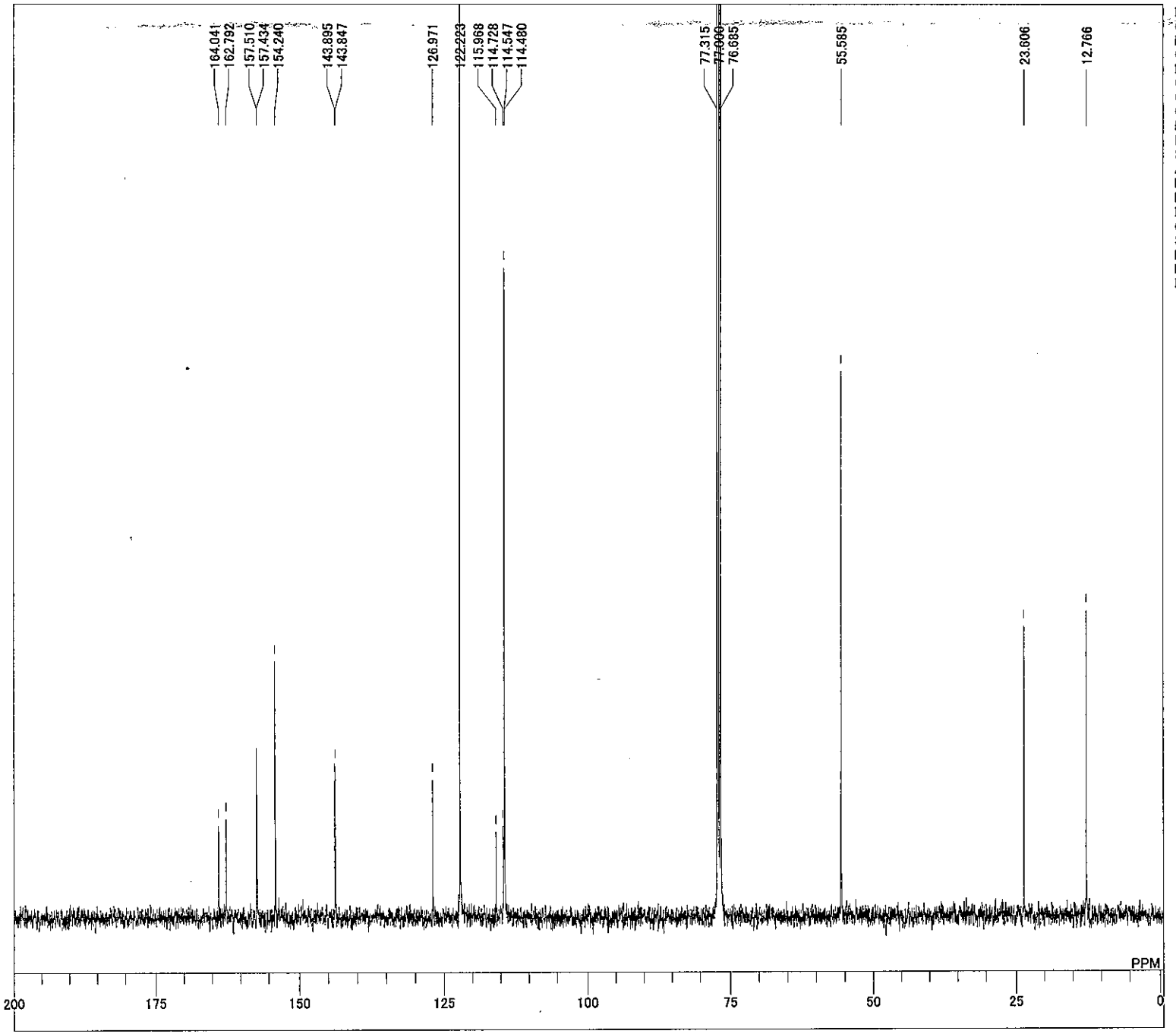


DFILE	C:¥Documents and Settings¥delta¥My Documents¥Personal
COMNT	
DATIM	08-01-2007 14:36:26
OBNUC	1H
EXMOD	single_pulse.ex2
OBFRQ	399.78 MHz
OBSET	4.19 KHz
OBFIN	7.29 Hz
POINT	16384
FREQU	7503.00 Hz
SCANS	8
ACQTM	2.1837 sec
PD	2.0000 sec
PW1	5.50 usec
IRNUC	1H
CTEMP	23.7 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	38

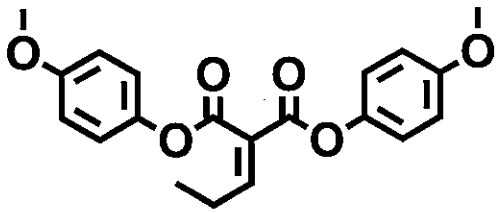


2f

C:¥Documents and Settings¥delta¥My Documents¥Personal Folder¥florian¥propylideniemalonate2.als

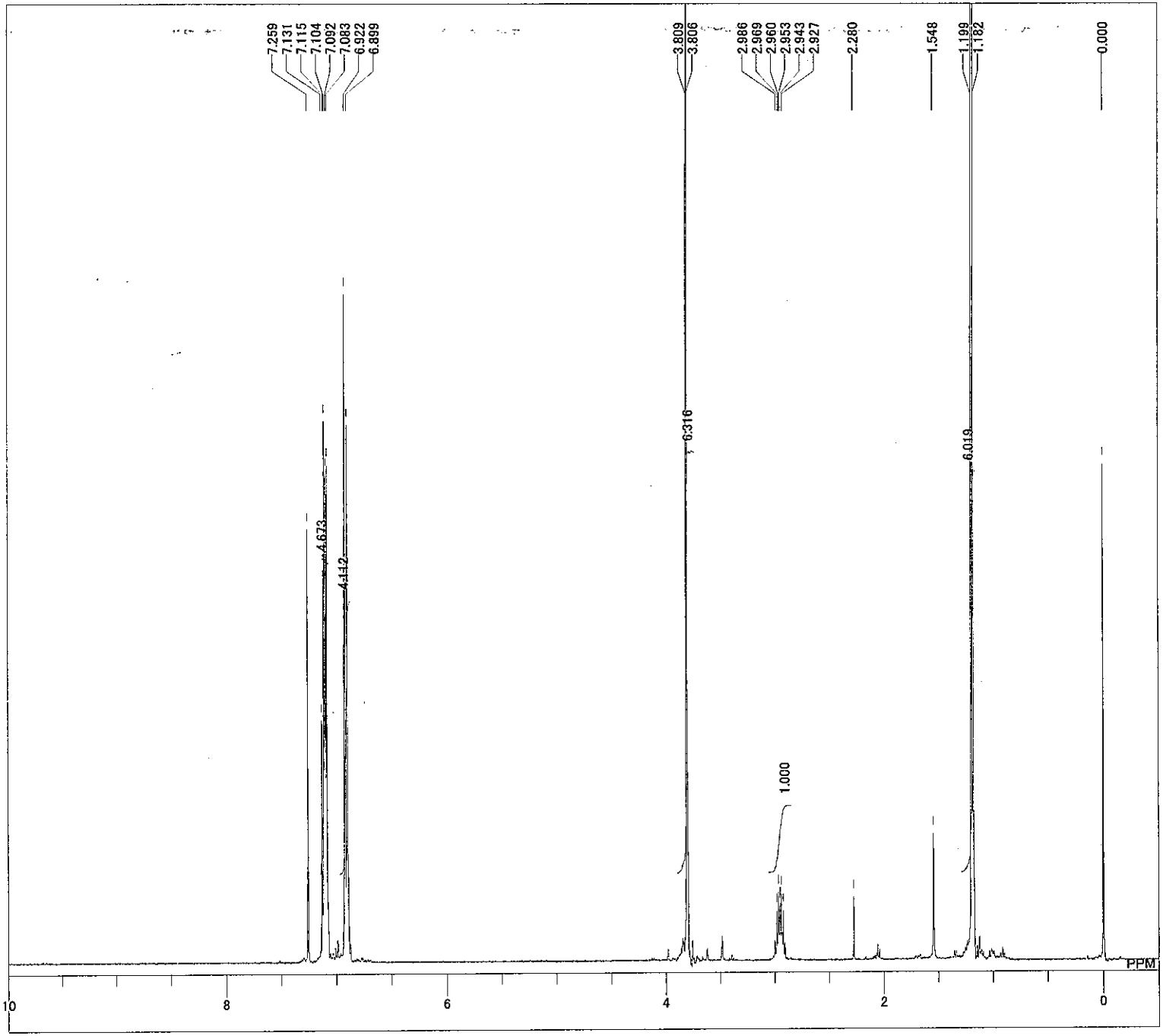


DFILE	C:¥Documents and Settings¥delta¥My Documents¥Personal
COMNT	
DATIM	05-06-2007 08:13:08
OBNUC	13C
EXMOD	single_pulse_dec
OBFRQ	100.53 MHz
OBSET	5.35 KHz
OBFIN	5.86 Hz
POINT	32768
FREQU	31407.04 Hz
SCANS	1000
ACQTM	1.0433 sec
PD	2.0000 sec
PW1	2.83 usec
IRNUC	1H
CTEMP	20.7 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	54

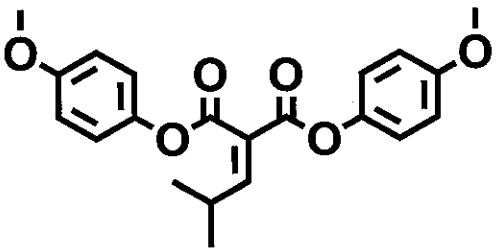


2f

C:¥Documents and Settings¥delta¥My Documents¥Personal Folder¥florian¥fb271recrys.als

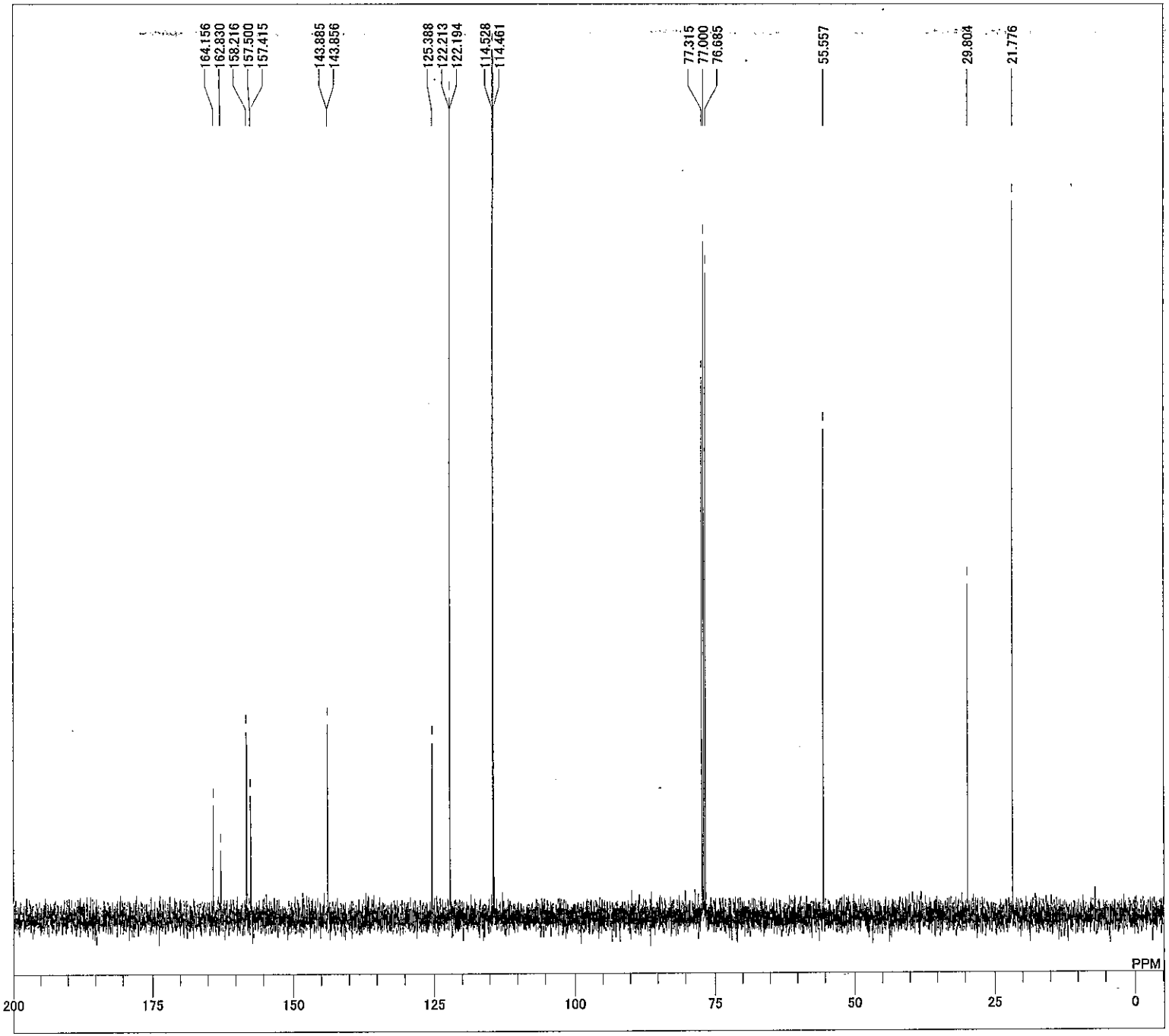


DFILE	C:¥Documents and Settings¥delta¥My Documents¥Persona
COMNT	
DATIM	18-01-2007 10:11:24
OBNUC	1H
EXMOD	single_pulse.ex2
OBFRQ	399.78 MHz
OBSET	4.19 KHz
OBFIN	7.29 Hz
POINT	16384
FREQU	7503.00 Hz
SCANS	8
ACQTM	2.1837 sec
PD	2.0000 sec
PW1	5.50 usec
IRNUC	1H
CTEMP	22.9 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	38

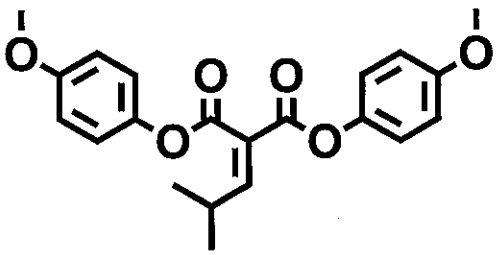


2g

C:¥Documents and Settings¥delta¥My Documents¥Personal Folder¥florian¥isobutylidenemalonate.als

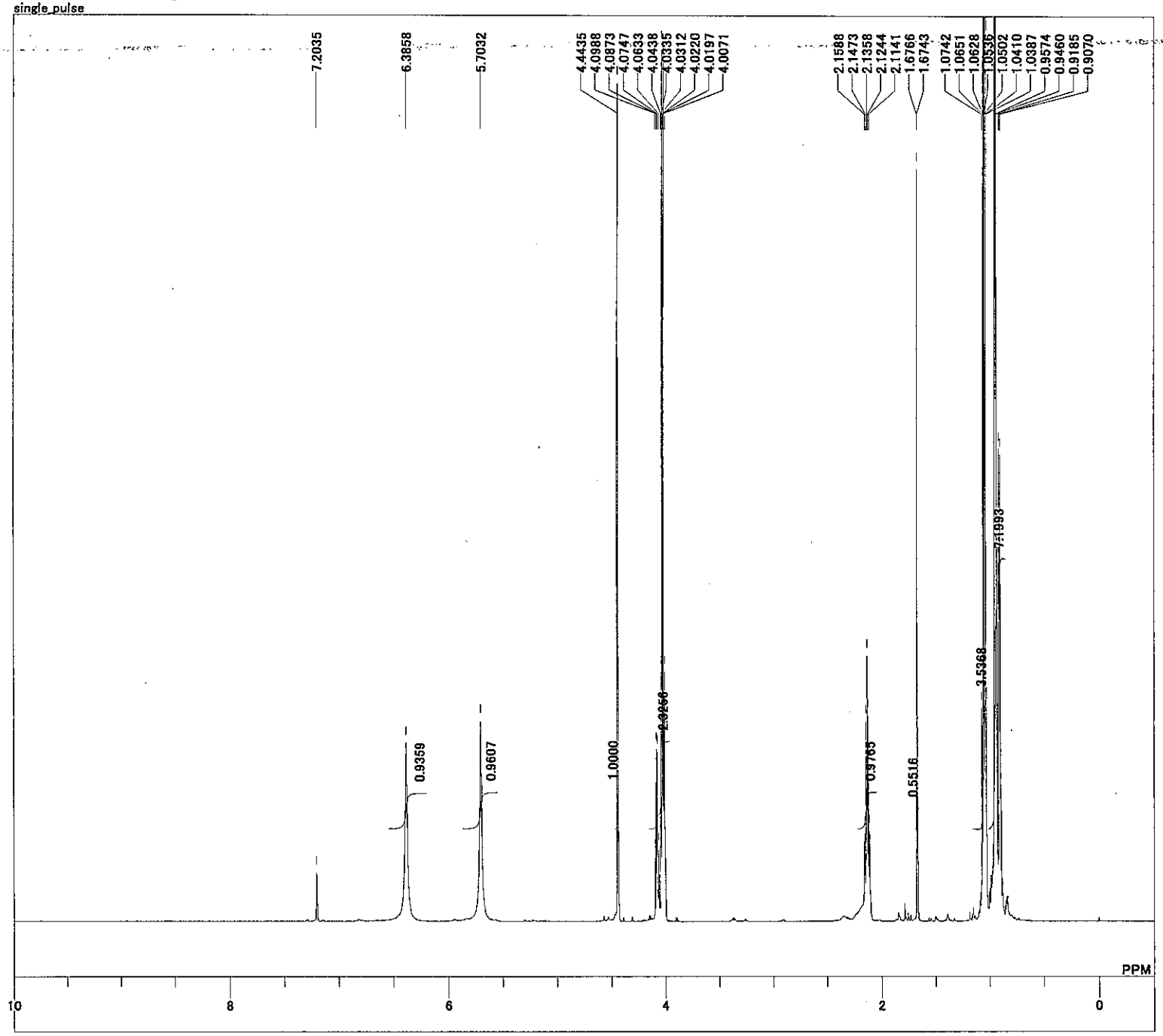


DFILE	C:¥Documents and Settings¥delta¥My Documents¥Personal
COMNT	
DATIM	04-06-2007 18:10:09
OBNUC	13C
EXMOD	single_pulse_dec
OBFRQ	100.53 MHz
OBSET	5.35 KHz
OBFIN	5.86 Hz
POINT	26214
FREQU	25125.24 Hz
SCANS	91
ACQTM	1.0433 sec
PD	2.0000 sec
PW1	2.83 usec
IRNUC	1H
CTEMP	20.2 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	50

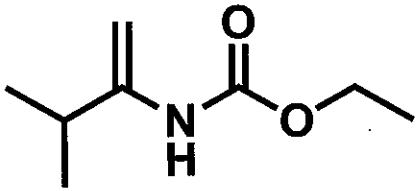


2g

C:¥Documents and Settings¥All Users¥Documents¥florian¥fb3491h.als
single_pulse

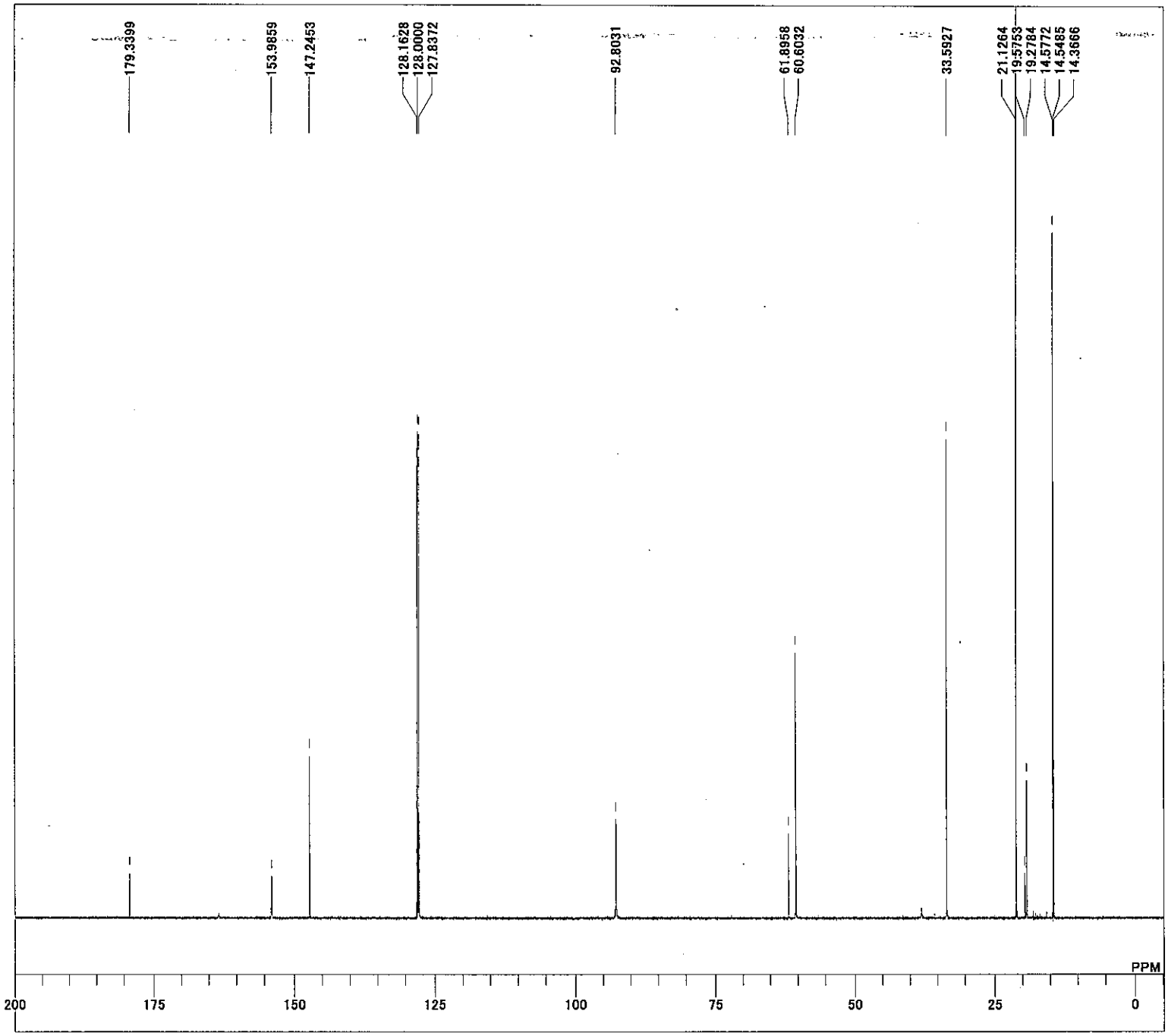


DFILE	C:¥Documents and Settings¥All Users¥Documents¥floria
COMNT	single_pulse
DATIM	11-05-2007 21:17:41
OBNUC	1H
EXMOD	single_pulse.ex2
OBFRQ	600.17 MHz
OBSET	5.30 KHz
OBFIN	5.47 Hz
POINT	13107
FREQU	9008.87 Hz
SCANS	8
ACQTM	1.4549 sec
PD	2.0000 sec
PW1	7.30 usec
IRNUC	1H
CTEMP	19.4 c
SLVNT	C6D6
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	20

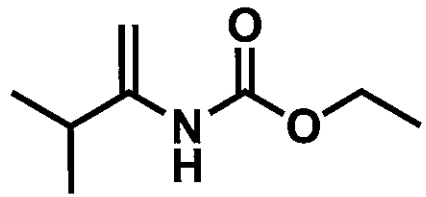


5j

C:¥Documents and Settings¥All Users¥Documents¥florian¥fb34913c.als

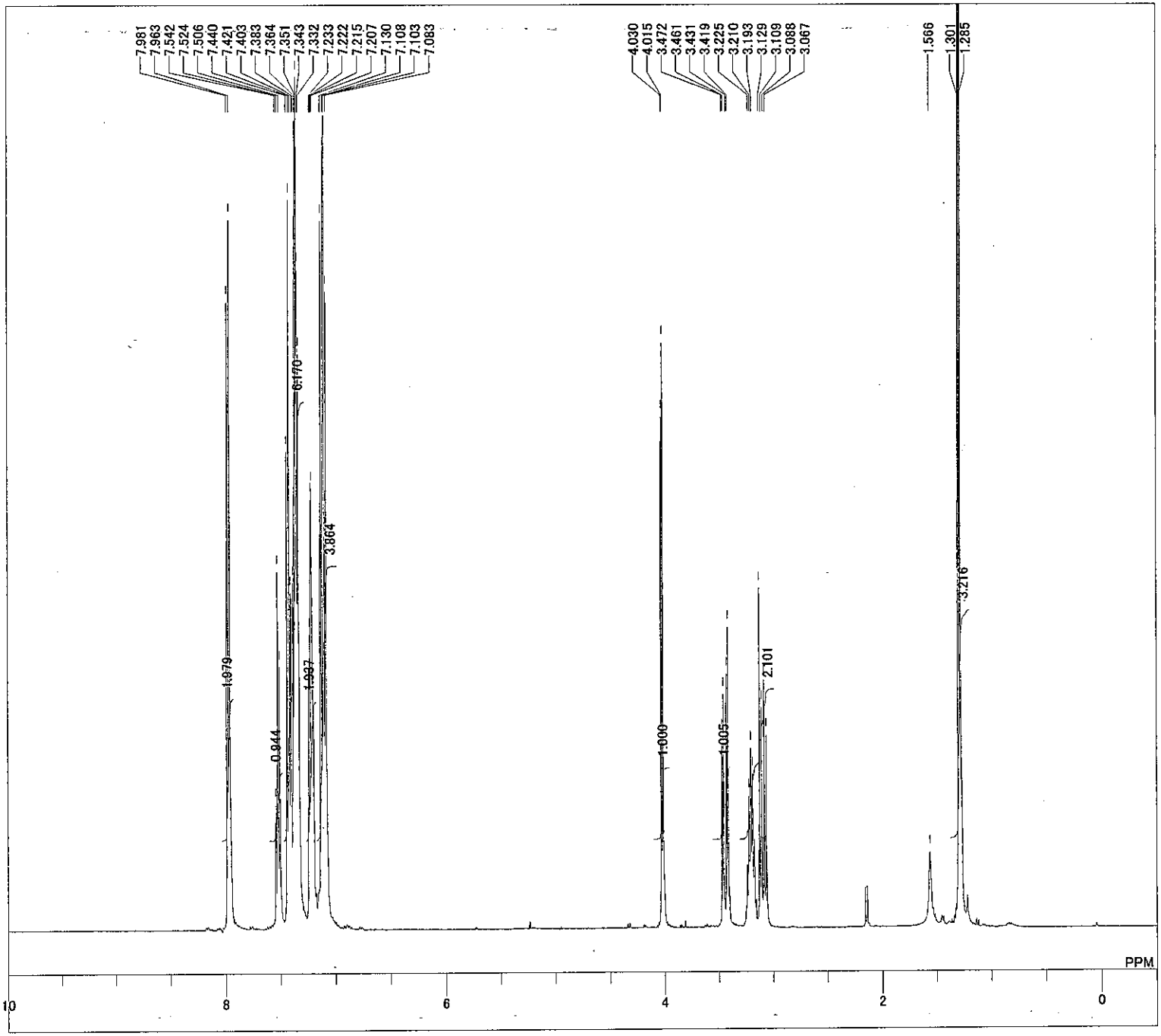


DFILE	C:¥Documents and Settings¥All Users¥Documents¥floria
COMNT	
DATIM	11-05-2007 21:25:49
OBNUC	13C
EXMOD	single_pulse_dec
OBFRQ	150.92 MHz
OBSET	8.52 KHz
OBFIN	1.74 Hz
POINT	26214
FREQU	37878.21 Hz
SCANS	128
ACQTM	0.6921 sec
PD	2.0000 sec
PW1	3.45 usec
IRNUC	1H
CTEMP	20.5 c
SLVNT	C6D6
EXREF	128.00 ppm
BF	0.12 Hz
RGAIN	58

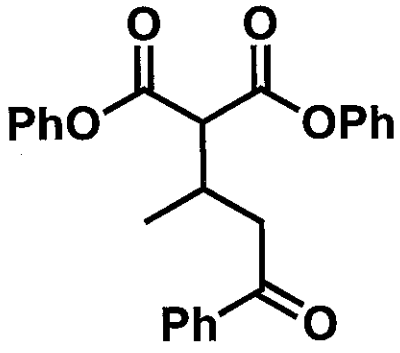


5j

C:¥Documents and Settings¥delta¥My Documents¥Personal Folder¥florian¥fb129puriptlc.als

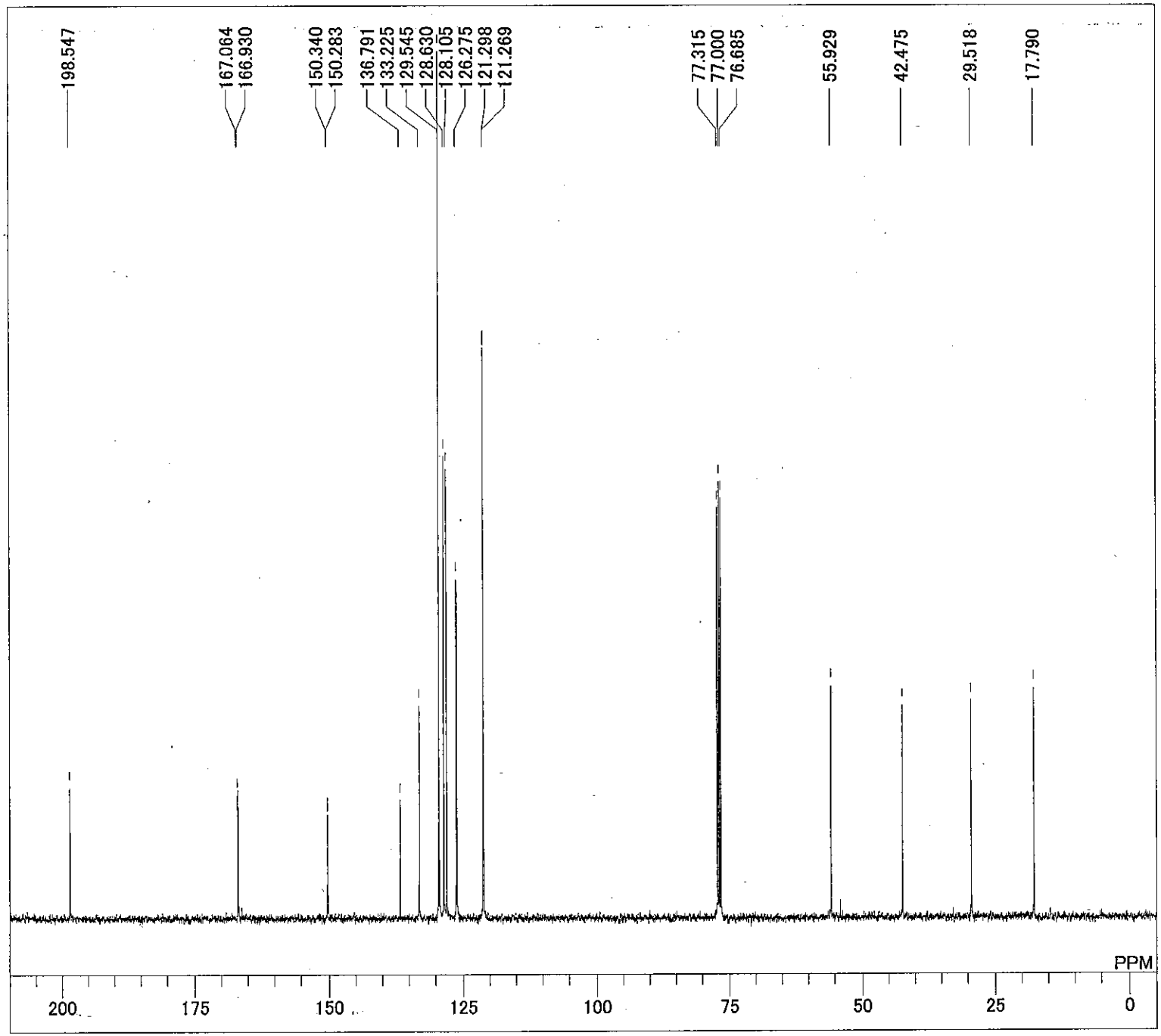


DFILE	C:¥Documents and Settings¥delta¥My Documents¥Personal
COMNT	
DATIM	07-06-2006 16:30:23
OBNUC	1H
EXMOD	single_pulse.ex2
OBFRQ	399.78 MHz
OBSET	4.19 KHz
OBFIN	7.29 Hz
POINT	16384
FREQU	7503.00 Hz
SCANS	8
ACQTM	2.1837 sec
PD	2.0000 sec
PW1	5.50 usec
IRNUC	1H
CTEMP	25.5 c
SLVNT	CDCL3
EXREF	7.24 ppm
BF	1.20 Hz
RGAIN	32

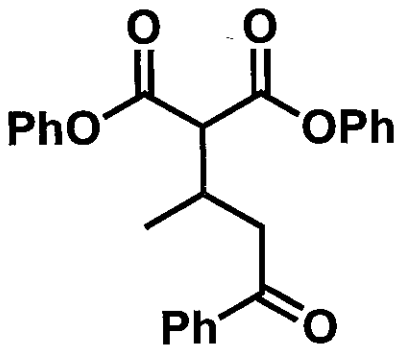


4a

C:¥Documents and Settings¥delta¥My Documents¥Personal Folder¥florian¥fb81verifc13.als

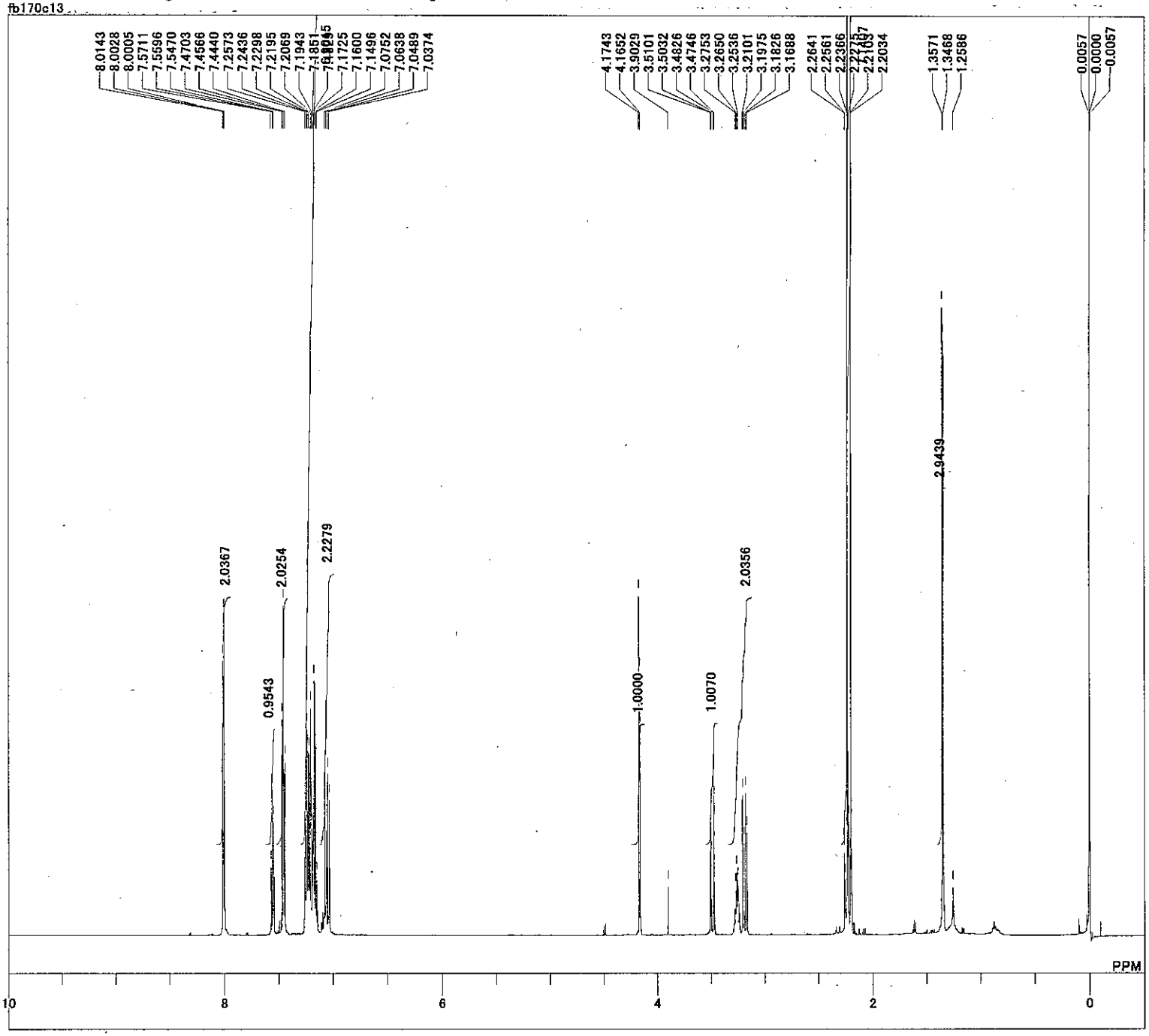


DFILE	C:¥Documents and Settings¥delta¥My D
COMNT	
DATIM	15-03-2007 19:16:59
OBNUC	13C
EXMOD	single_pulse_dec
OBFRQ	100.53 MHz
OBSET	5.35 KHz
OBFIN	5.86 Hz
POINT	32768
FREQU	31407.04 Hz
SCANS	274
ACQTM	1.0433 sec
PD	2.0000 sec
PW1	3.33 usec
IRNUC	1H
CTEMP	24.5 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	56

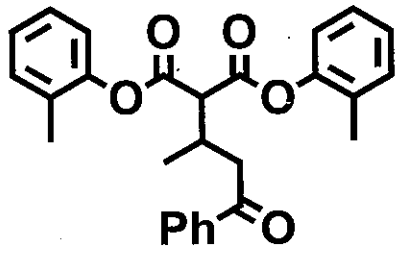


4a

C:¥Documents and Settings¥All Users¥Documents¥florian¥fb170c13_PROTON.als
fb170c13

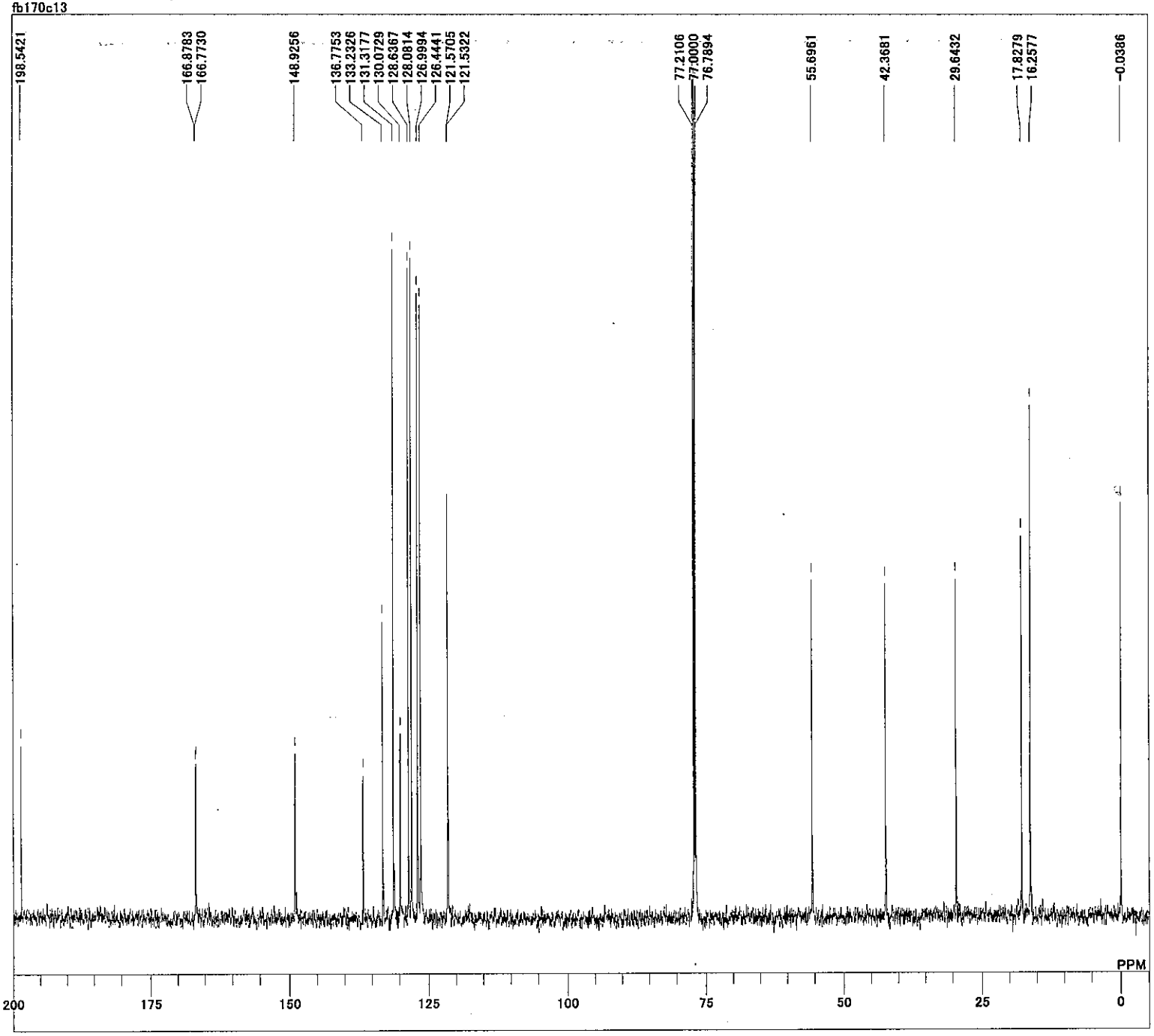


DFILE	C:¥Documents and Settings¥All Users¥D
COMNT	fb170c13
DATIM	28-04-2007 04:36:21
OBNUC	1H
EXMOD	single_pulse.ex2
OBFRQ	600.17 MHz
OBSET	5.30 KHz
OBFIN	5.47 Hz
POINT	16384
FREQU	11261.26 Hz
SCANS	8
ACQTM	1.4549 sec
PD	4.0000 sec
PW1	7.30 usec
IRNUC	1H
CTEMP	19.1 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	36

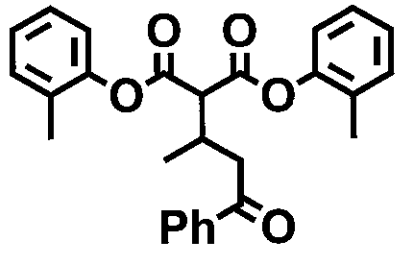


4b

C:¥Documents and Settings¥All Users¥Documents¥florian¥fb170c13_CARBON.als
fb170c13

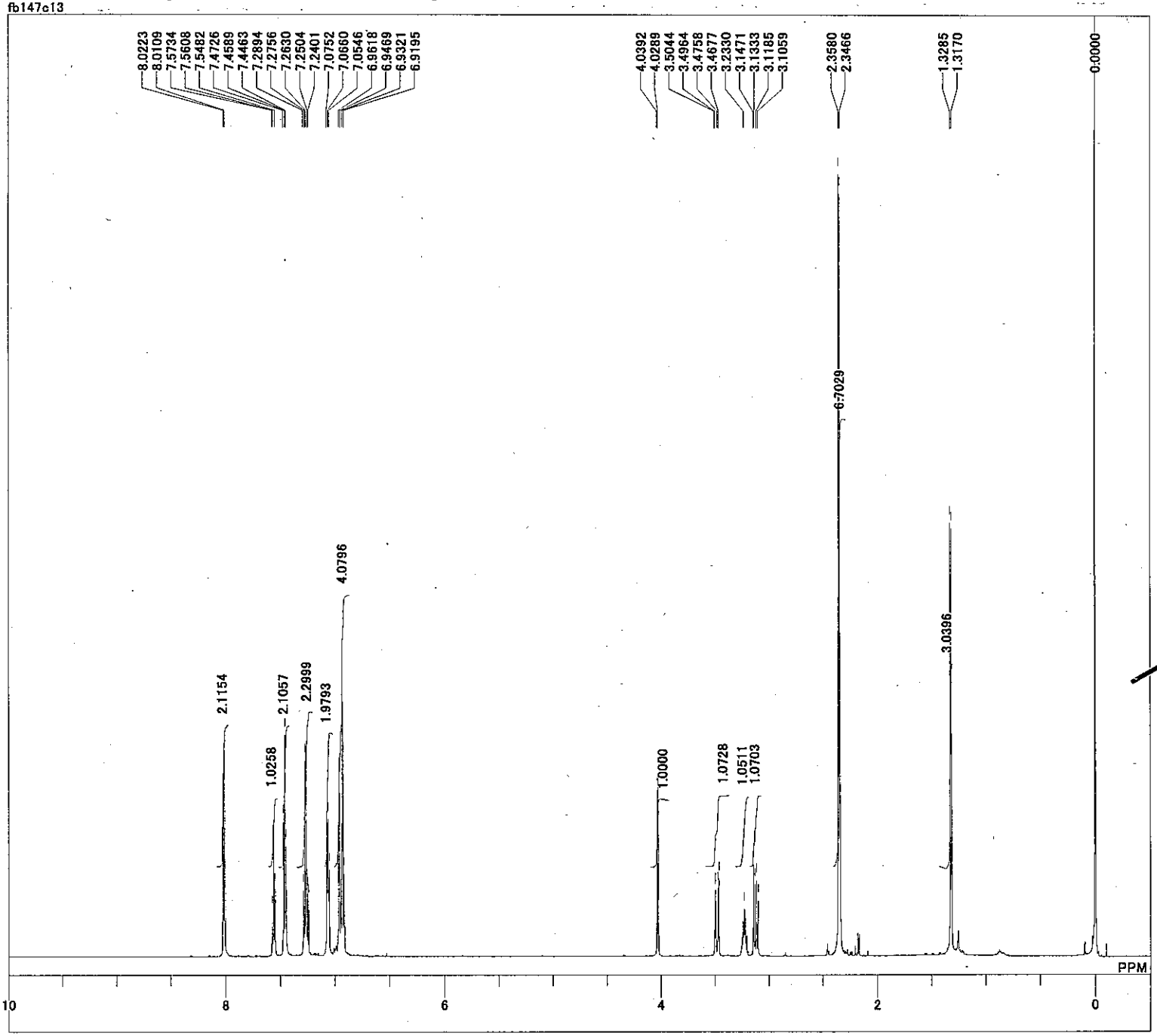


DFILE	C:¥Documents and Settings¥All Users¥Documents¥floria
COMNT	fb170c13
DATIM	28-04-2007 04:42:50
OBNUC	13C
EXMOD	single_pulse_dec
OBFRQ	150.92 MHz
OBSET	8.52 KHz
OBFIN	1.74 Hz
POINT	32768
FREQU	47348.48 Hz
SCANS	128
ACQTM	0.6921 sec
PD	2.0000 sec
PW1	3.45 usec
IRNUC	1H
CTEMP	20.0 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	60

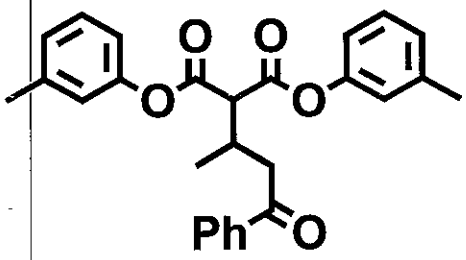


4b

C:¥Documents and Settings¥All Users¥Documents¥florian¥fb147c13_PROTON.als
fb147c13

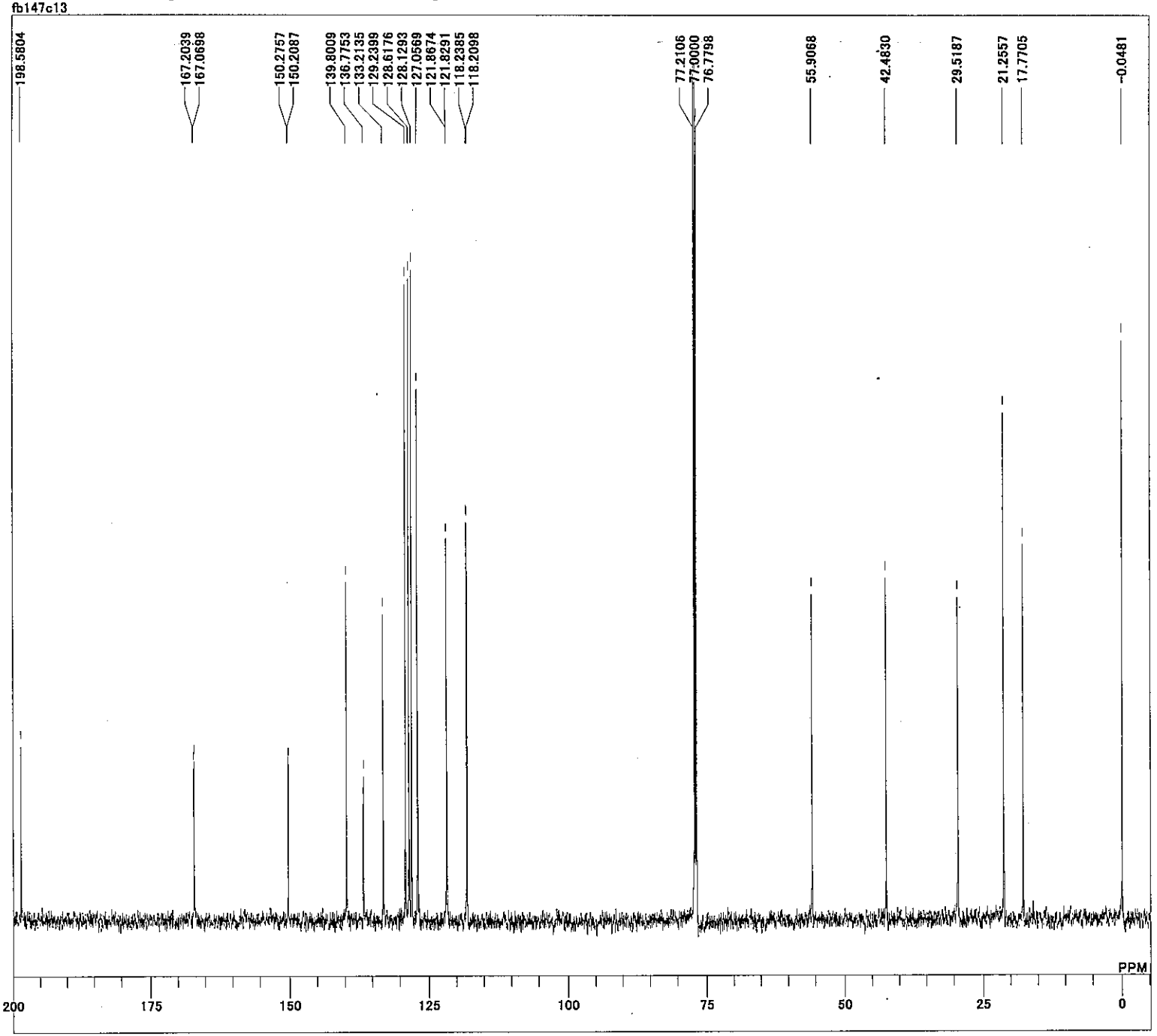


DFILE	C:¥Documents and Settings¥All Users¥D
COMNT	fb147c13
DATIM	28-04-2007 04:52:47
OBNUC	1H
EXMOD	single_pulse.ex2
OBFRQ	600.17 MHz
OBSET	5.30 KHz
OBFIN	5.47 Hz
POINT	16384
FREQU	11261.26 Hz
SCANS	8
ACQTM	1.4549 sec
PD	4.0000 sec
PW1	7.30 usec
IRNUC	1H
CTEMP	19.2 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	36

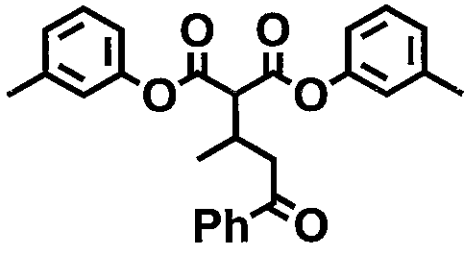


4c

C:¥Documents and Settings¥All Users¥Documents¥florian¥fb147c13_CARBON.als
fb147c13

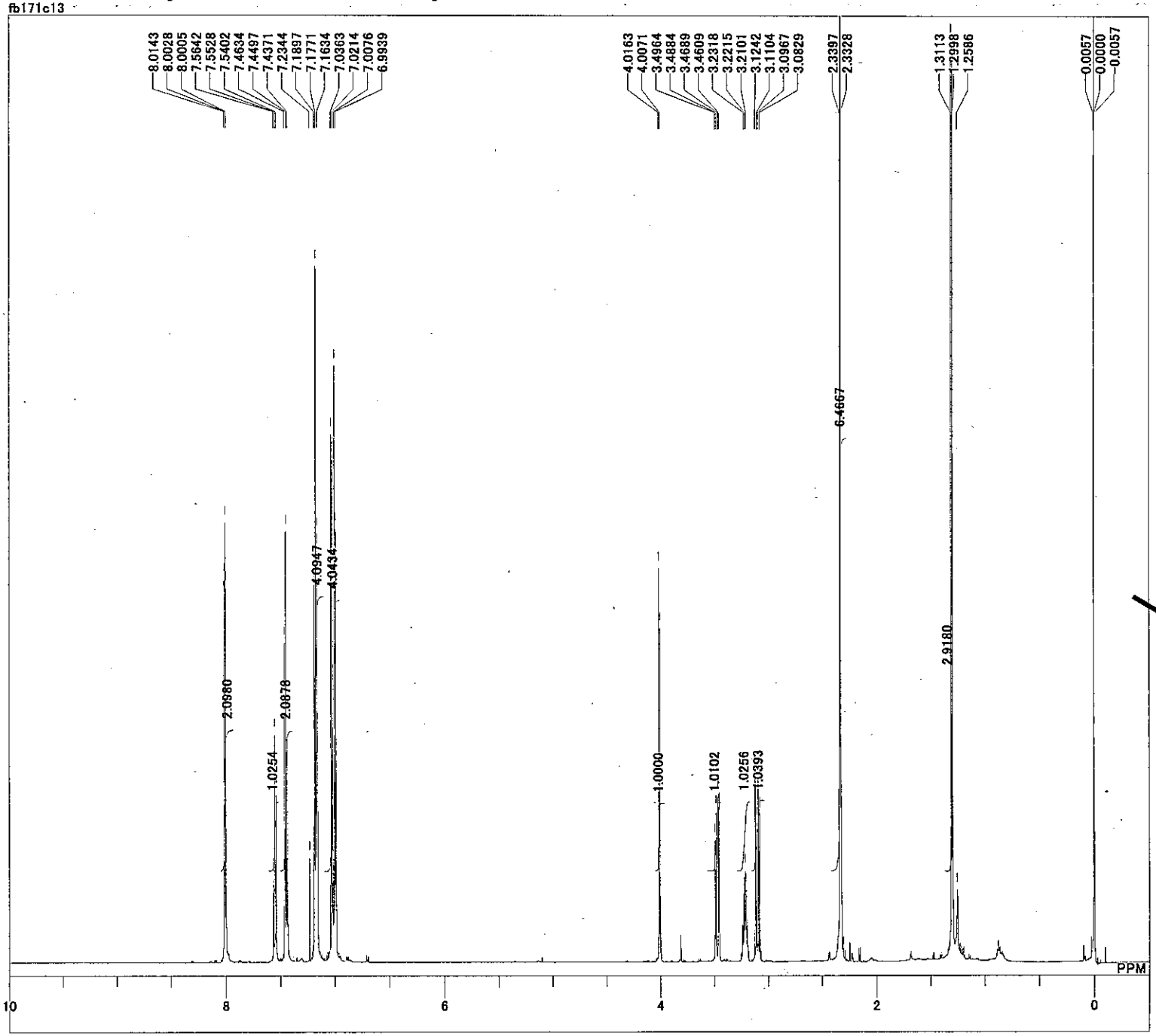


DFILE	C:¥Documents and Settings¥All Users¥Documents¥floria
COMNT	fb147c13
DATIM	28-04-2007 04:59:16
OBNUC	13C
EXMOD	single_pulse_dec
OBFRQ	150.92 MHz
OBSET	8.52 KHz
OBFIN	1.74 Hz
POINT	32768
FREQU	47348.48 Hz
SCANS	128
ACQTM	0.6921 sec
PD	2.0000 sec
PW1	3.45 usec
IRNUC	1H
CTEMP	20.1 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	60

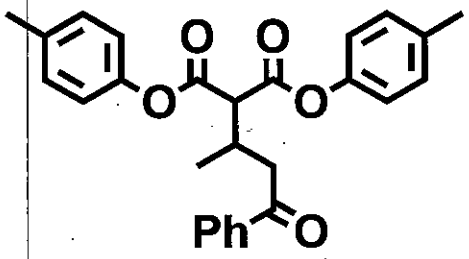


4c

C:¥Documents and Settings¥All Users¥Documents¥florian¥fb171c13_PROTON.als
fb171c13

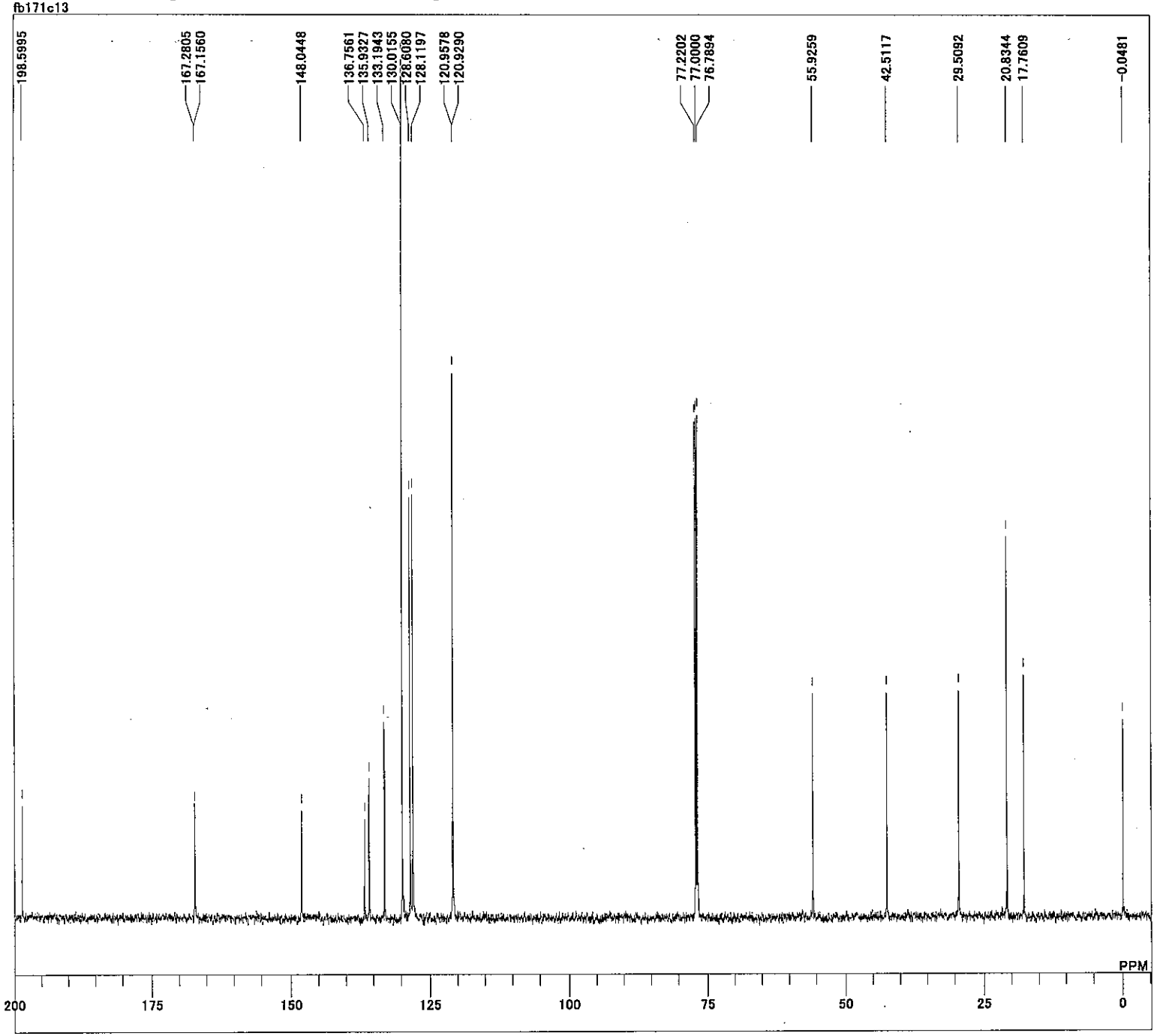


DFILE	C:¥Documents and Settings¥All Users¥D
COMNT	fb171c13
DATIM	28-04-2007 22:35:37
OBNUC	1H
EXMOD	single_pulse.ex2
OBFRQ	600.17 MHz
OBSET	5.30 KHz
OBFIN	5.47 Hz
POINT	16384
FREQU	11261.26 Hz
SCANS	8
ACQTM	1.4549 sec
PD	4.0000 sec
PW1	7.30 usec
IRNUC	1H
CTEMP	19.1 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	34

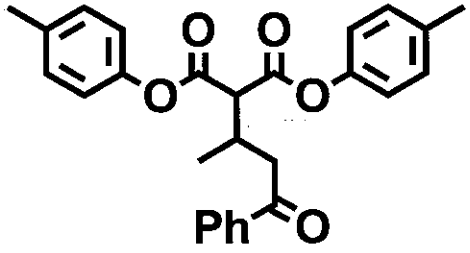


4d

C:¥Documents and Settings¥All Users¥Documents¥florian¥fb171c13_CARBON.als
fb171c13

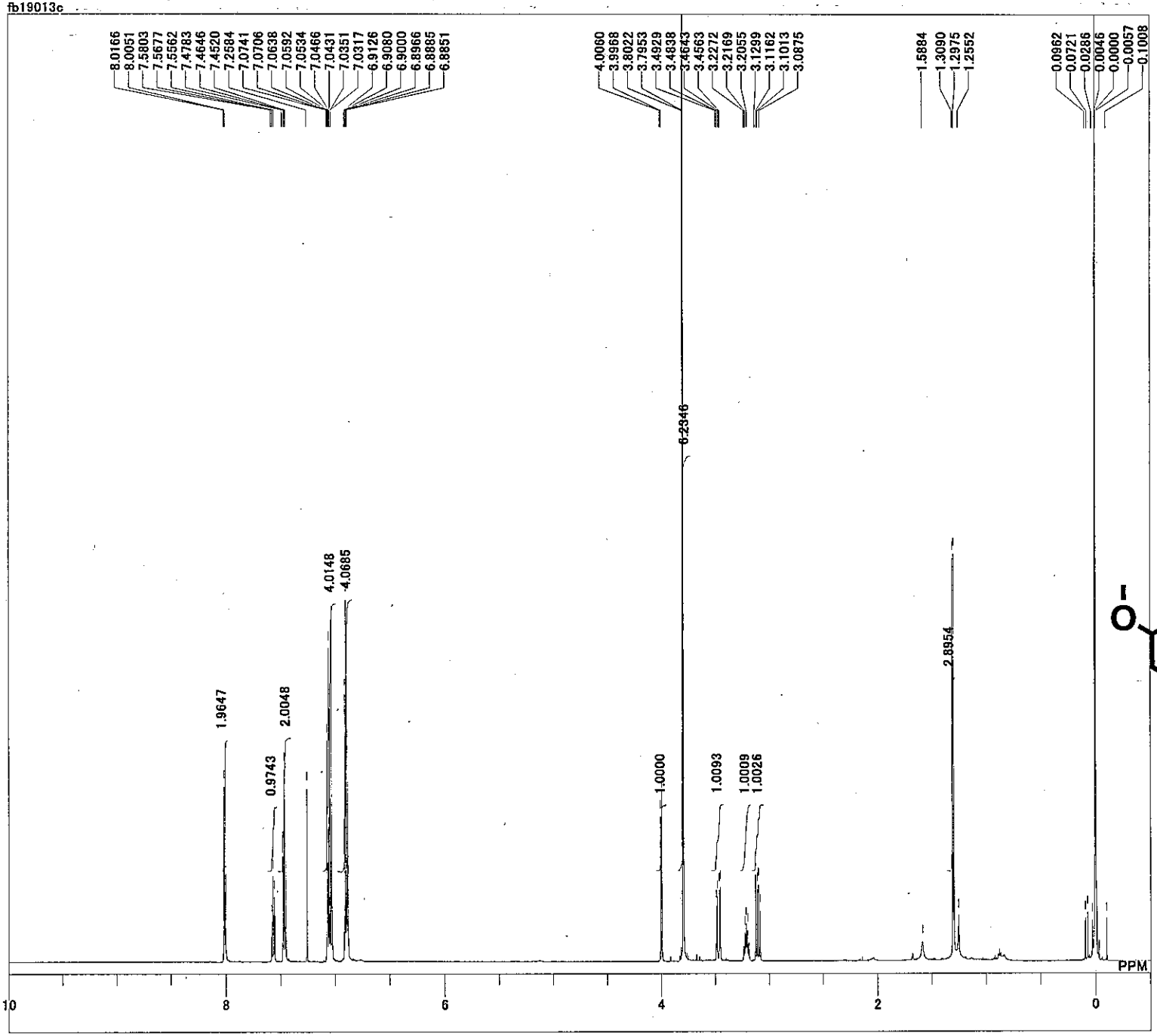


DFILE	C:¥Documents and Settings¥All Users¥Documents¥floria
COMNT	fb171c13
DATIM	28-04-2007 22:42:06
OBNUC	13C
EXMOD	single_pulse_dec
OBFRQ	150.92 MHz
OBSET	8.52 KHz
OBFIN	1.74 Hz
POINT	32768
FREQU	47348.48 Hz
SCANS	128
ACQTM	0.6921 sec
PD	2.0000 sec
PW1	3.45 usec
IRNUC	1H
CTEMP	20.0 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	60

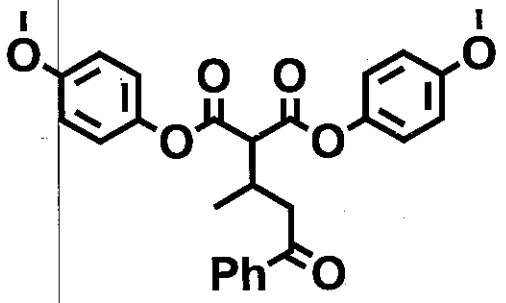


4d

C:¥Documents and Settings¥All Users¥Documents¥florian¥fb19013c_PROTON.als
fb19013c

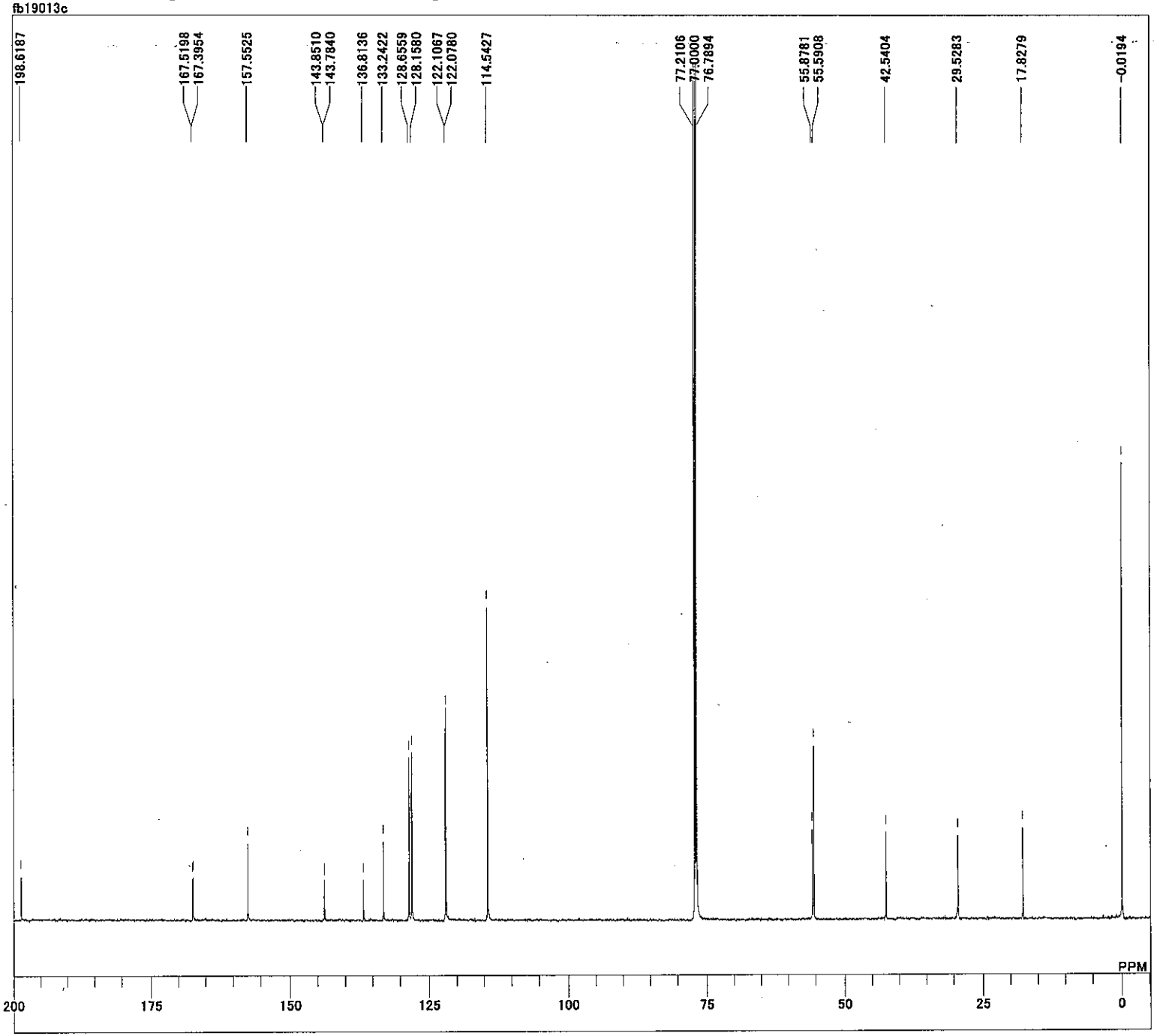


DFILE	C:¥Documents and Settings¥All Users¥D
COMNT	fb19013c
DATIM	29-04-2007 08:01:47
OBNUC	1H
EXMOD	single_pulse.ex2
OBFRQ	600.17 MHz
OBSET	5.30 KHz
OBFIN	5.47 Hz
POINT	16384
FREQU	11261.26 Hz
SCANS	16
ACQTM	1.4549 sec
PD	4.0000 sec
PW1	7.30 usec
IRNUC	1H
CTEMP	18.9 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	40

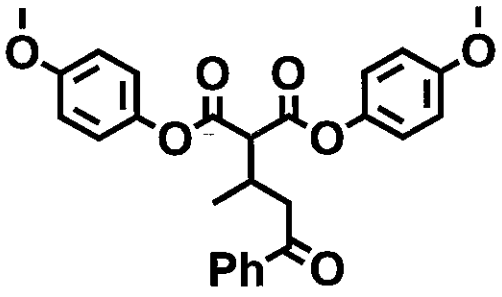


4e

C:¥Documents and Settings¥All Users¥Documents¥florian¥fb19013c_CARBON.als
fb19013c

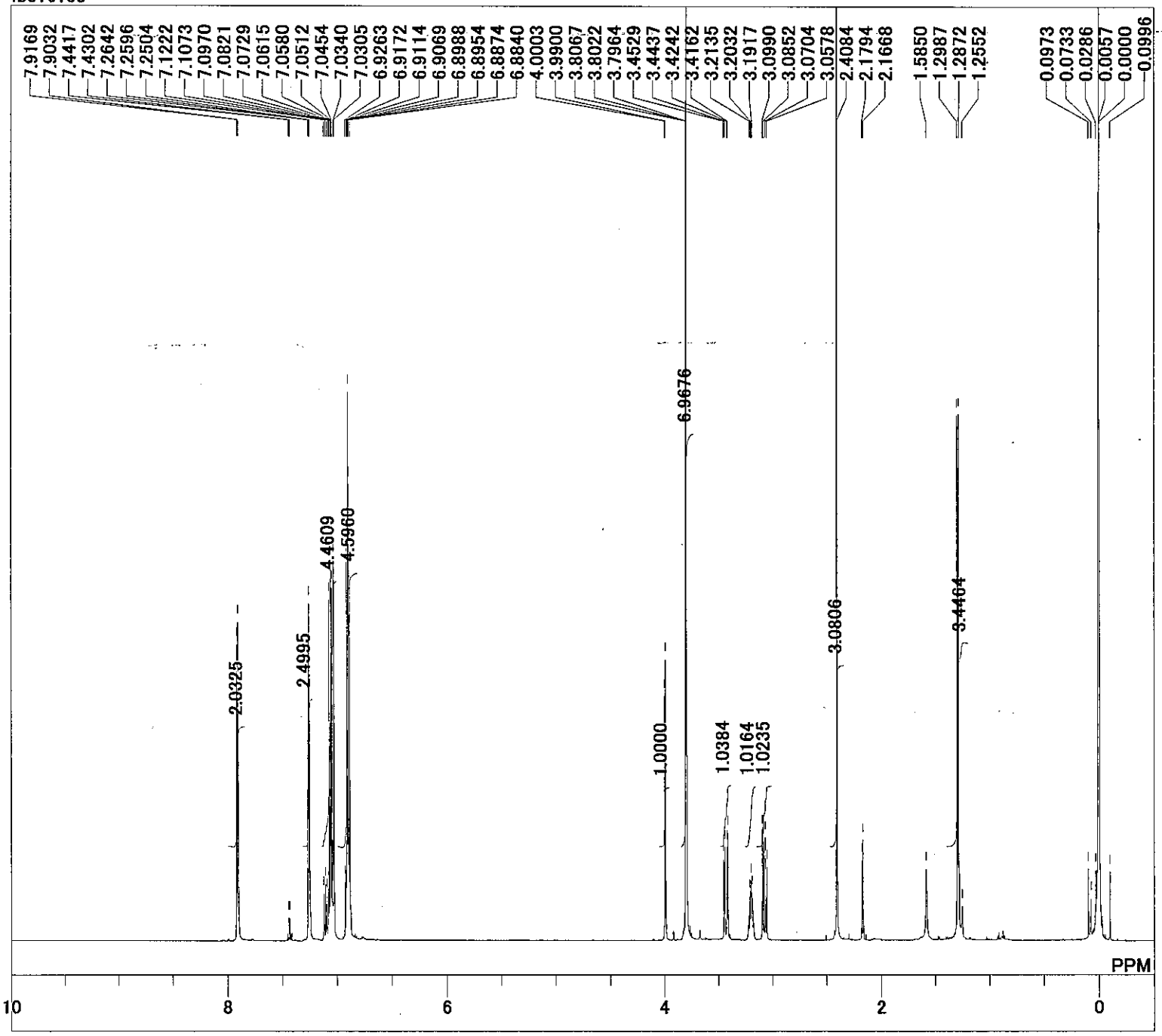


DFILE	C:¥Documents and Settings¥All Users¥Documents¥floria
COMNT	fb19013c
DATIM	29-04-2007 11:06:17
OBNUC	13C
EXMOD	single_pulse_dec
OBFRQ	150.92 MHz
OBSET	8.52 KHz
OBFIN	1.74 Hz
POINT	32768
FREQU	47348.48 Hz
SCANS	4096
ACQTM	0.6921 sec
PD	2.0000 sec
PW1	3.45 usec
IRNUC	1H
CTEMP	20.4 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	60

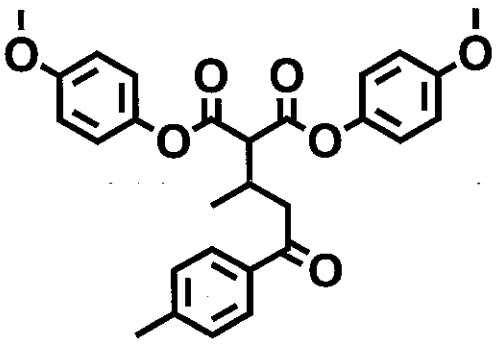


4e

¥¥Eca¥data¥data¥fb31613c_PROTON.3
fb31613c

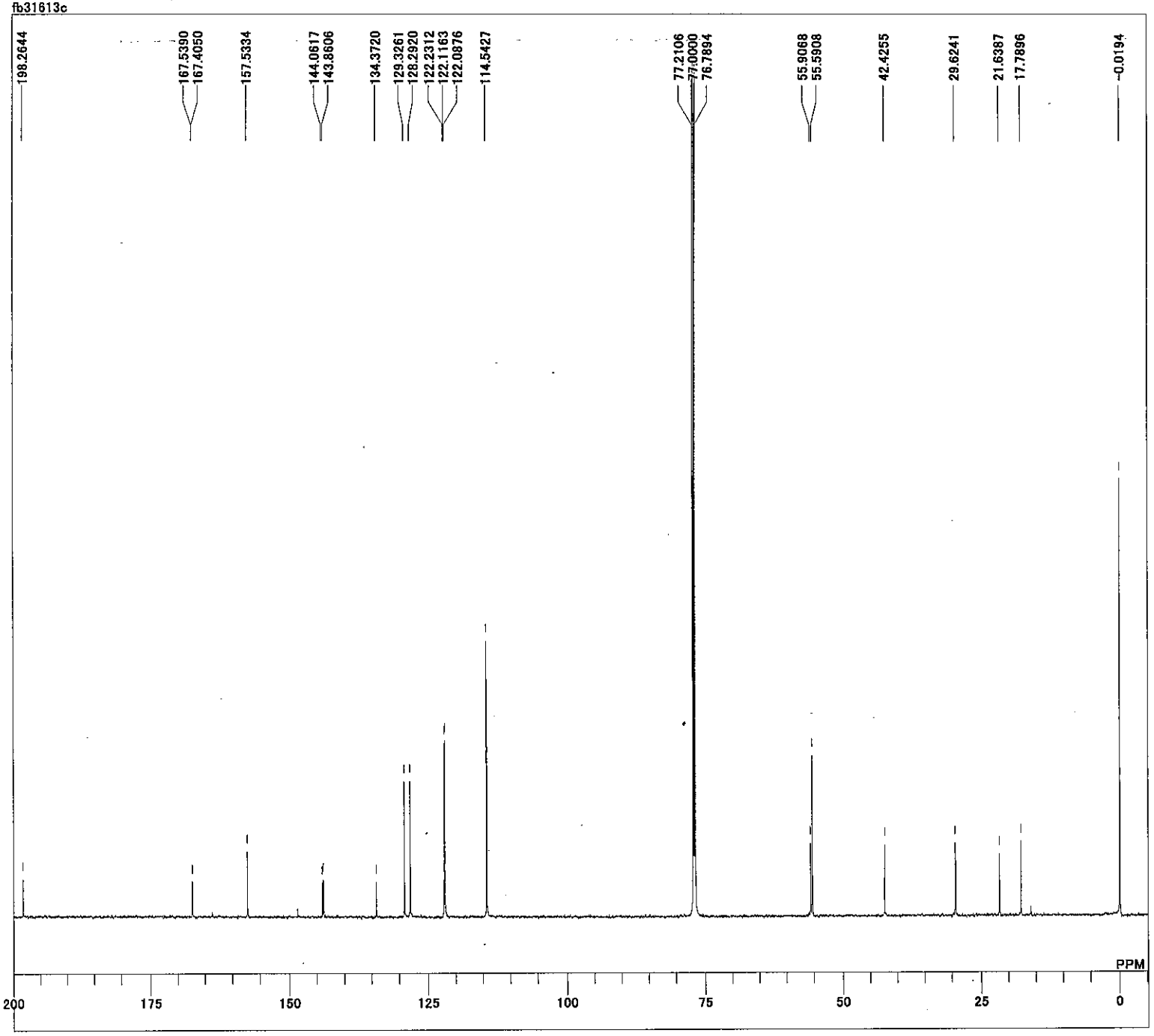


DFILE	¥¥Eca¥data¥data¥fb31613c_PROTON.3
COMNT	fb31613c
DATIM	29-04-2007 14:32:58
OBNUC	1H
EXMOD	single_pulse.ex2
OBFRQ	600.17 MHz
OBSET	5.30 KHz
OBFIN	5.47 Hz
POINT	20480
FREQU	14076.79 Hz
SCANS	16
ACQTM	1.4549 sec
PD	4.0000 sec
PW1	7.30 usec
IRNUC	1H
CTEMP	19.2 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	40

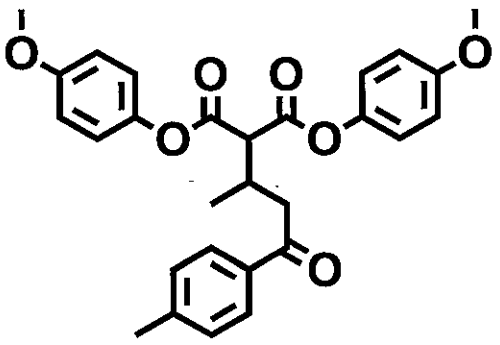


4f

C:¥Documents and Settings¥All Users¥Documents¥florian¥fb31613c_CARBON.als
fb31613c

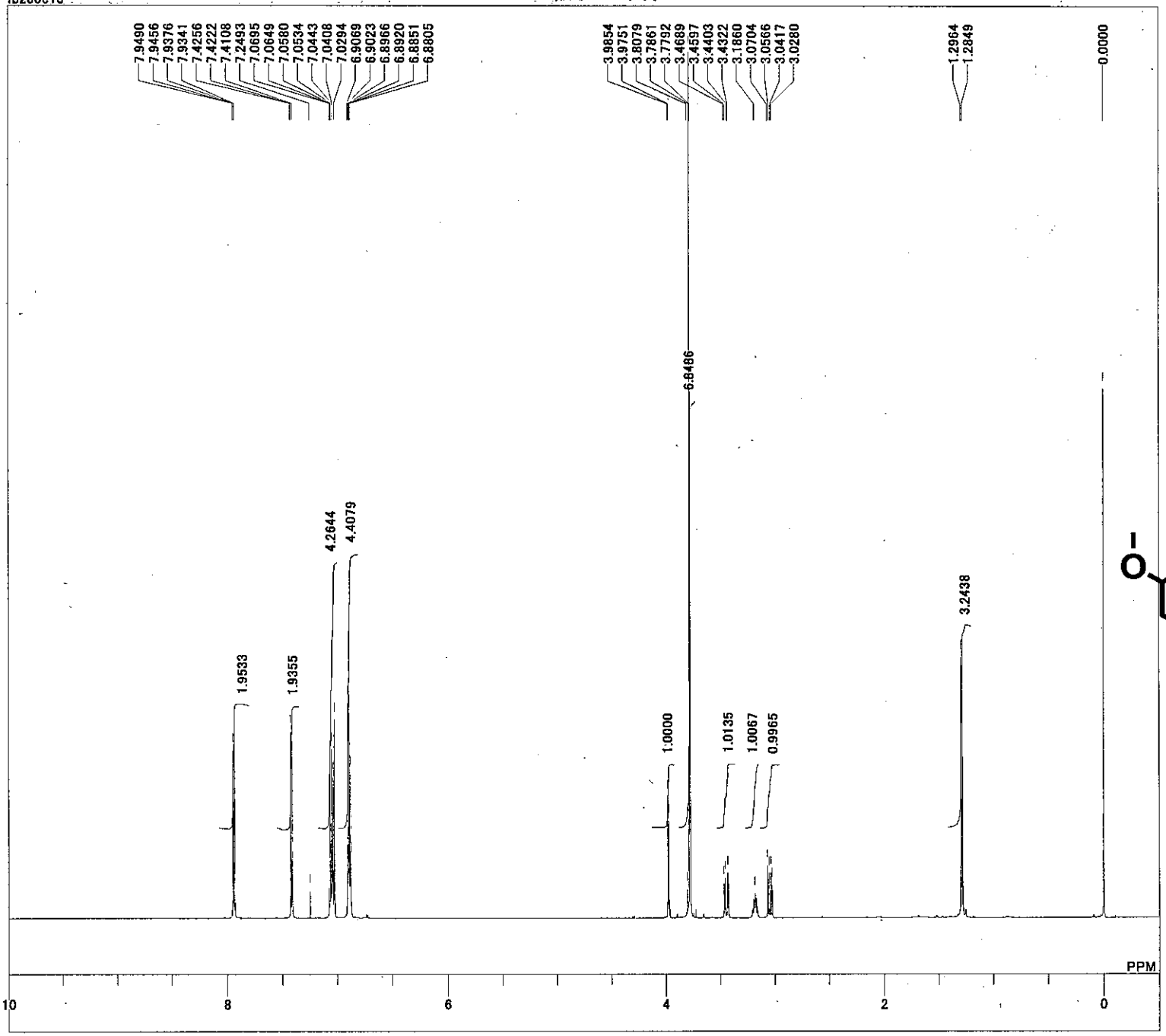


DFILE	C:¥Documents and Settings¥All Users¥Documents¥floria
COMNT	fb31613c
DATIM	29-04-2007 17:37:28
OBNUC	13C
EXMOD	single_pulse_dec
OBFRQ	150.92 MHz
OBSET	8.52 KHz
OBFIN	1.74 Hz
POINT	32768
FREQU	47348.48 Hz
SCANS	4096
ACQTM	0.6921 sec
PD	2.0000 sec
PW1	3.45 usec
IRNUC	1H
CTEMP	20.6 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	60

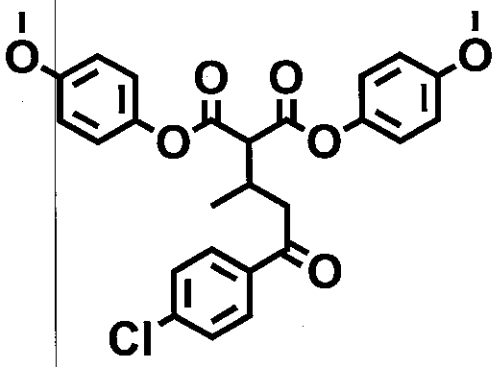


4f

C:¥Documents and Settings¥All Users¥Documents¥florian¥fb238c13_PROTON.als
fb238c13

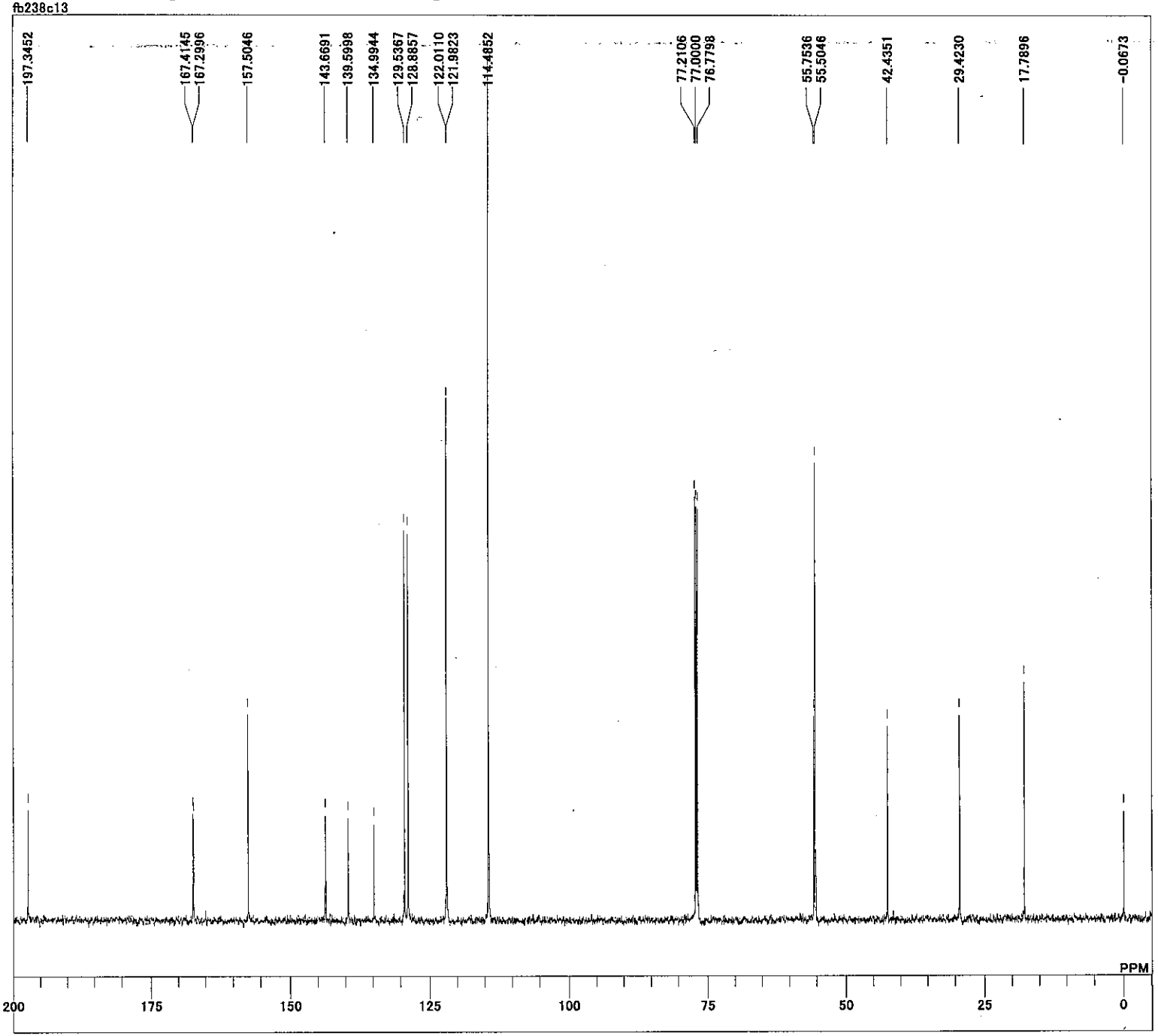


DFILE	C:¥Documents and Settings¥All Users¥D
COMNT	fb238c13
DATIM	29-04-2007 02:05:07
OBNUC	1H
EXMOD	single_pulse.ex2
OBFRQ	600.17 MHz
OBSET	5.30 KHz
OBFIN	5.47 Hz
POINT	16384
FREQU	11261.26 Hz
SCANS	8
ACQTM	1.4549 sec
PD	4.0000 sec
PW1	7.30 usec
IRNUC	1H
CTEMP	18.8 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	34

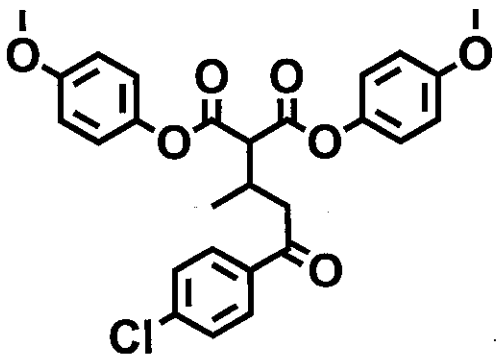


4g

C:¥Documents and Settings¥All Users¥Documents¥florian¥fb238c13_CARBON.als
fb238c13

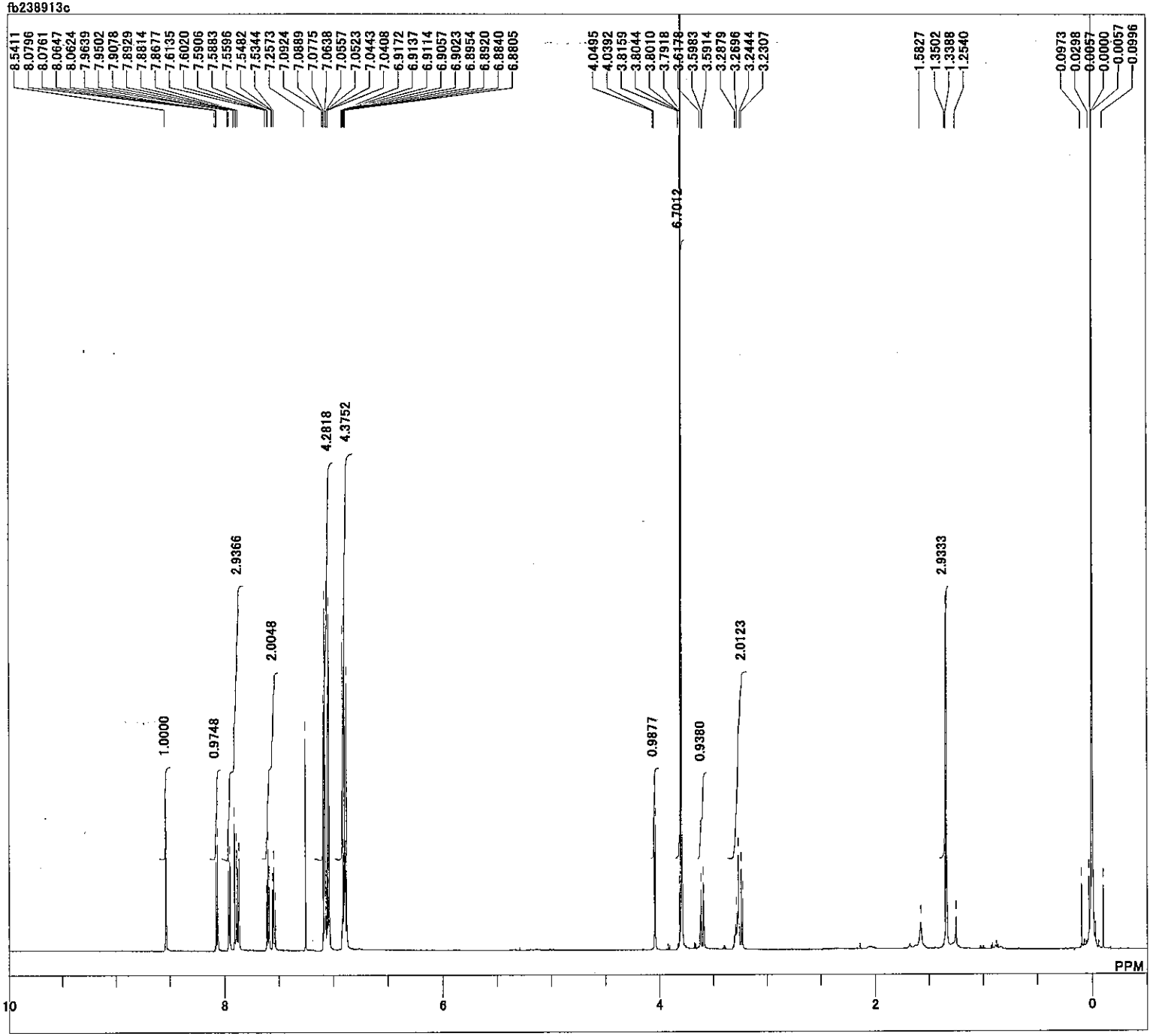


DFILE	C:¥Documents and Settings¥All Users¥Documents¥floria
COMNT	fb238c13
DATIM	29-04-2007 02:11:36
OBNUC	13C
EXMOD	single_pulse_dec
OBFRQ	150.92 MHz
OBSET	8.52 KHz
OBFIN	1.74 Hz
POINT	32768
FREQU	47348.48 Hz
SCANS	128
ACQTM	0.6921 sec
PD	2.0000 sec
PW1	3.45 usec
IRNUC	1H
CTEMP	19.9 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	60

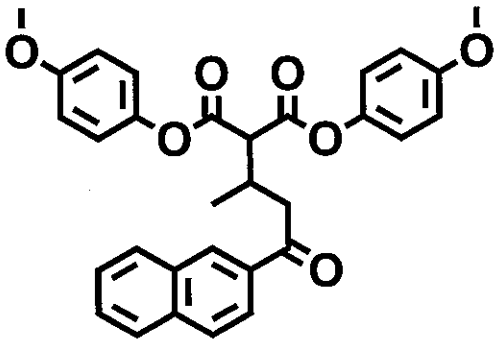


4g

C:¥Documents and Settings¥All Users¥Documents¥florian¥fb23913c_PROTON.als
fb238913c

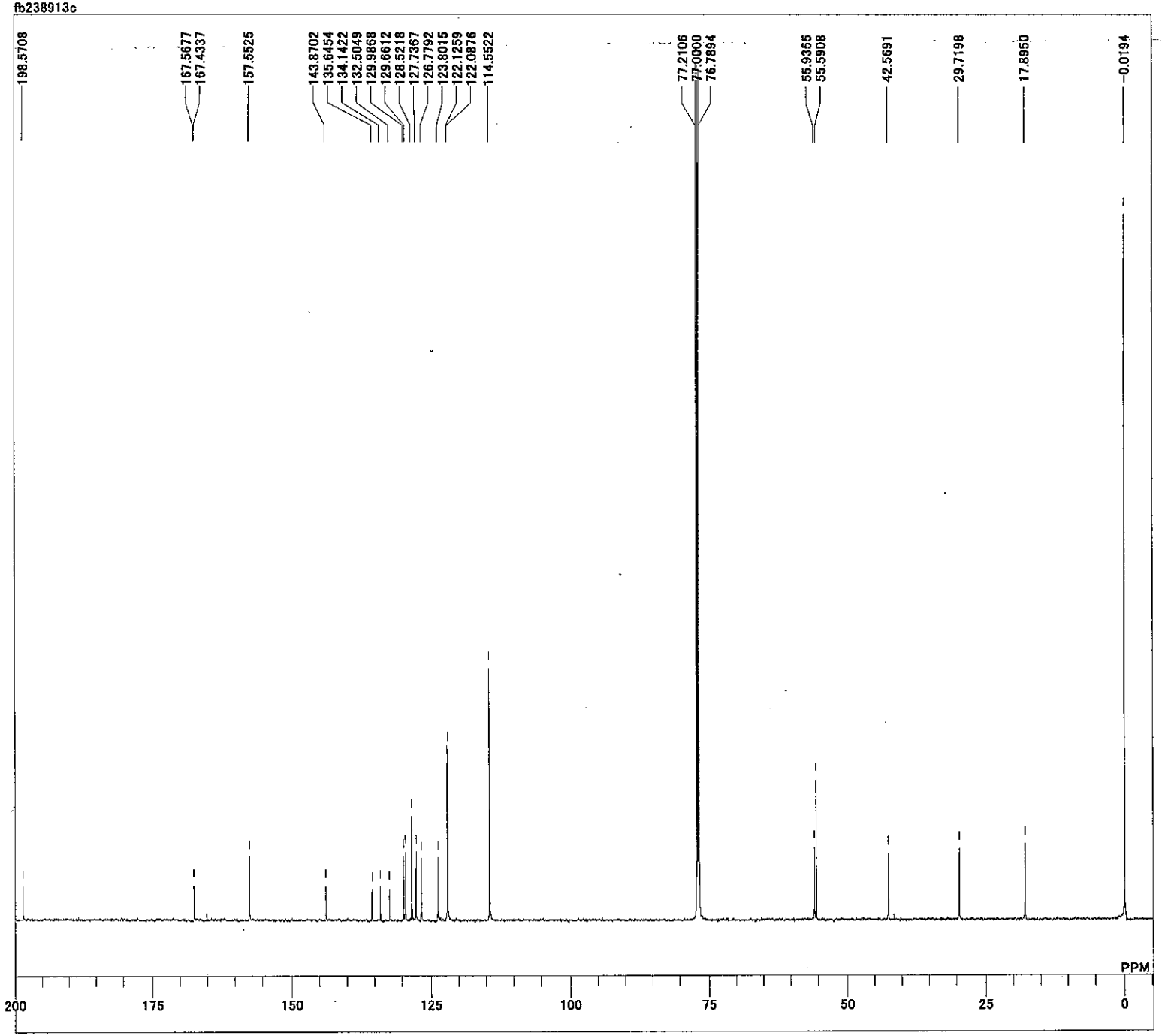


DFILE	C:¥Documents and Settings¥All Users¥Documents¥floria
COMNT	fb238913c
DATIM	29-04-2007 11:17:18
OBNUC	1H
EXMOD	single_pulse.ex2
OBFRQ	600.17 MHz
OBSET	5.30 KHz
OBFIN	5.47 Hz
POINT	16384
FREQU	11261.26 Hz
SCANS	16
ACQTM	1.4549 sec
PD	4.0000 sec
PW1	7.30 usec
IRNUC	1H
CTEMP	19.3 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	40

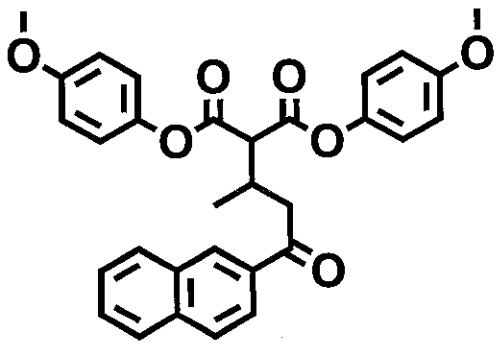


4h

C:¥Documents and Settings¥All Users¥Documents¥florian¥fb23913c_CARBON.als
fb238913c

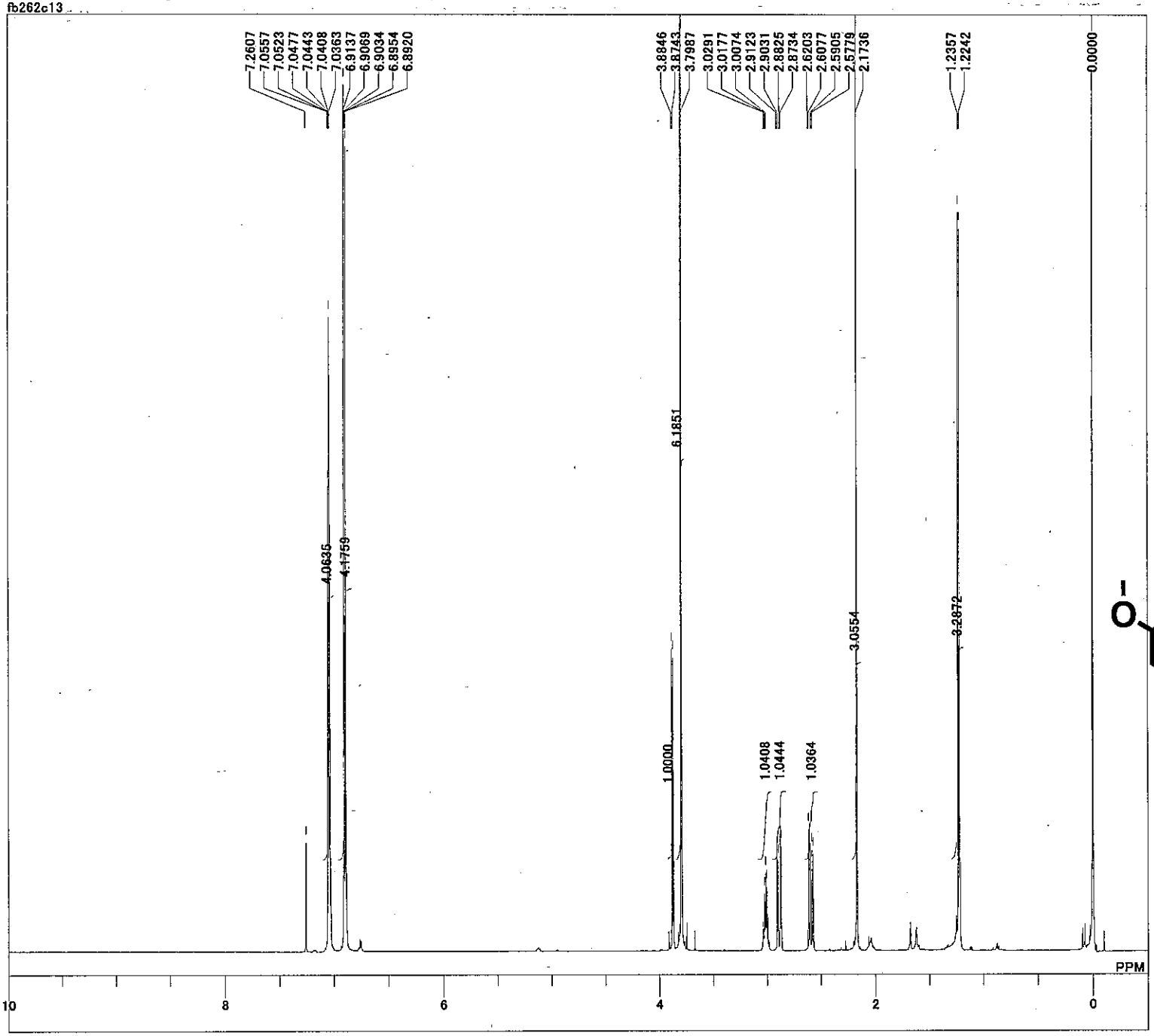


DFILE	C:¥Documents and Settings¥All Users¥Documents¥floria
COMNT	fb238913c
DATIM	29-04-2007 14:21:48
OBNUC	13C
EXMOD	single_pulse_dec
OBFRQ	150.92 MHz
OBSET	8.52 KHz
OBFIN	1.74 Hz
POINT	32768
FREQU	47348.48 Hz
SCANS	4096
ACQTM	0.6921 sec
PD	2.0000 sec
PW1	3.45 usec
IRNUC	1H
CTEMP	20.4 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	60

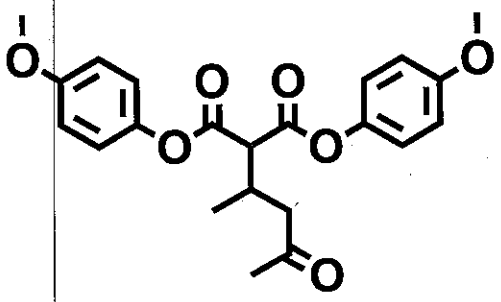


4h

C:¥Documents and Settings¥All Users¥Documents¥florian¥fb262c13_PROTON.als
fb262c13

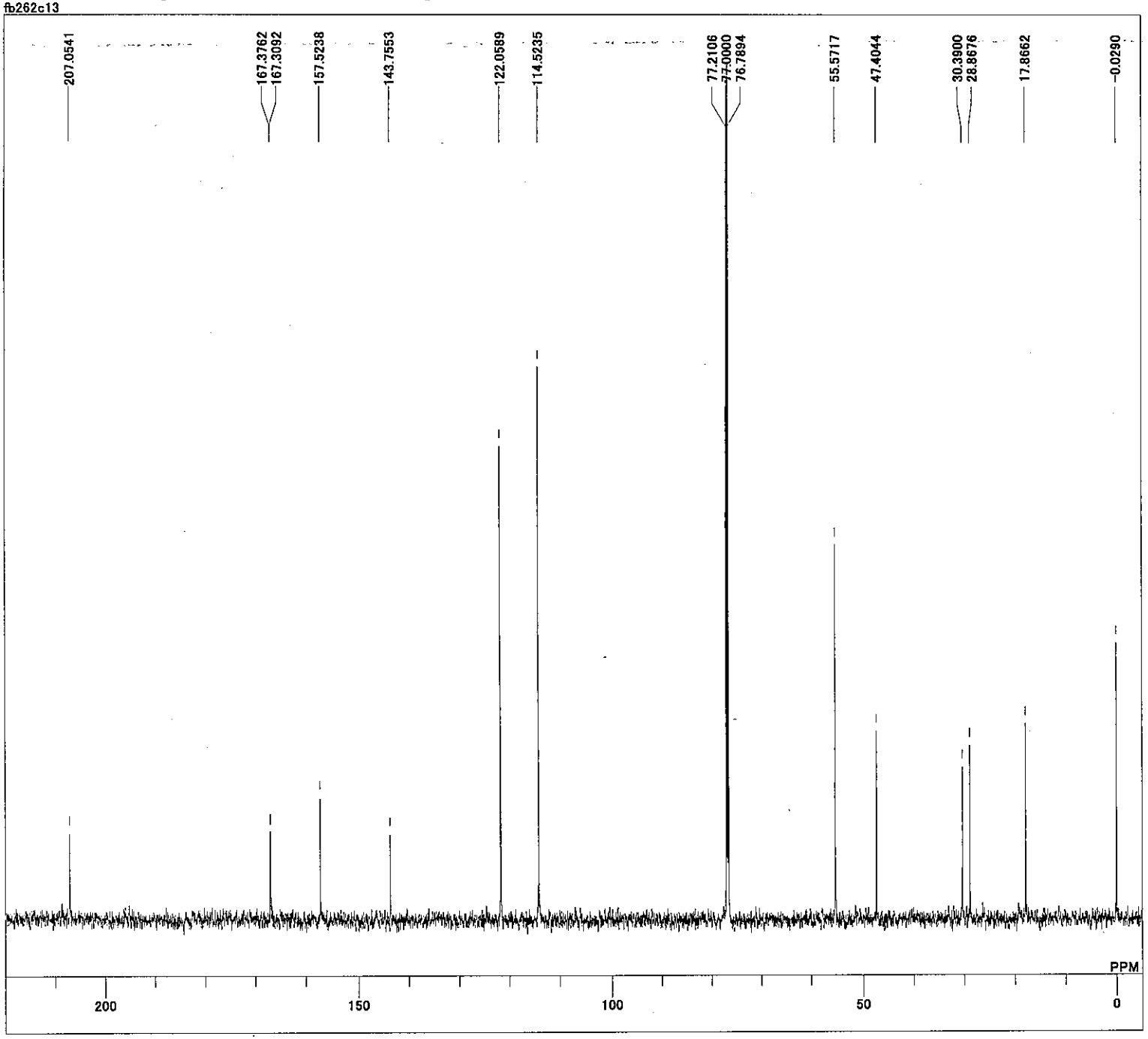


DFILE	C:¥Documents and Settings¥All Users¥D
COMNT	fb262c13
DATIM	30-04-2007 02:00:21
OBNUC	1H
EXMOD	single_pulse.ex2
OBFRQ	600.17 MHz
OBSET	5.30 KHz
OBFIN	5.47 Hz
POINT	16384
FREQU	11261.26 Hz
SCANS	8
ACQTM	1.4549 sec
PD	4.0000 sec
PW1	7.30 usec
IRNUC	1H
CTEMP	18.9 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	38

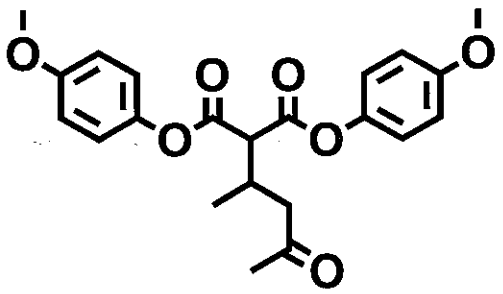


4i

C:¥Documents and Settings¥All Users¥Documents¥florian¥fb262c13_CARBON.als
fb262c13

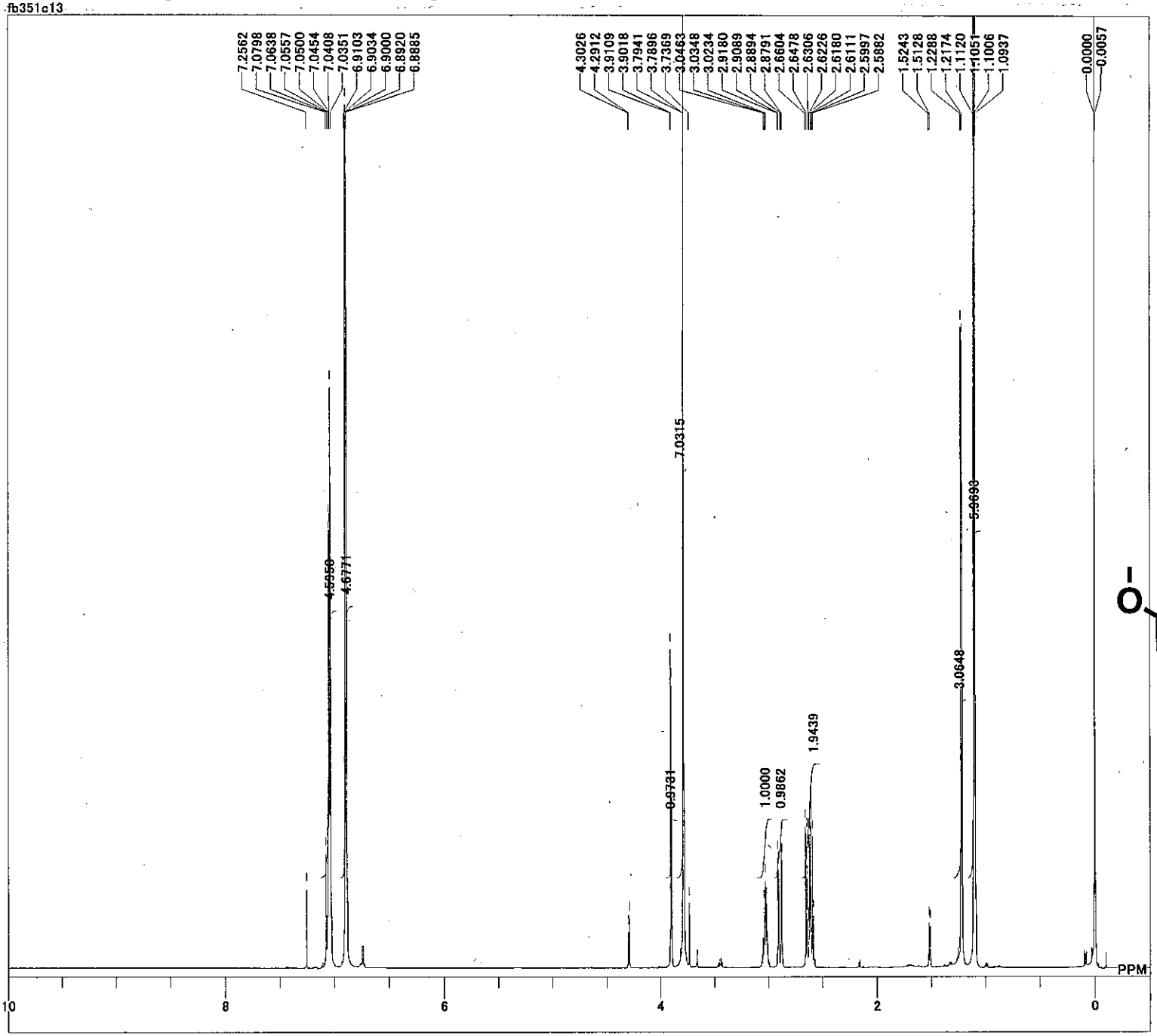


DFILE	C:¥Documents and Settings¥All Users¥Documents¥floria
COMNT	fb262c13.
DATIM	30-04-2007 02:06:50
OBNUC	13C
EXMOD	single_pulse_dec
OBFRQ	150.92 MHz
OBSET	8.52 KHz
OBFIN	1.74 Hz
POINT	32768
FREQU	47348.48 Hz
SCANS	128
ACQTM	0.6921 sec
PD	2.0000 sec
PW1	3.45 usec
IRNUC	1H
CTEMP	19.9 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	60

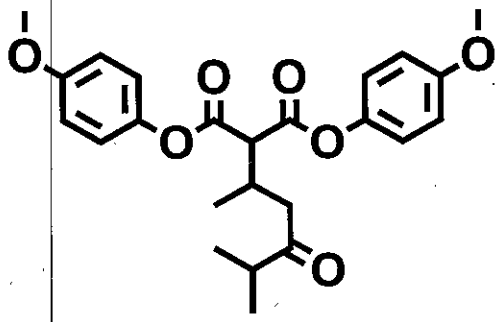


4i

C:¥Documents and Settings¥All Users¥Documents¥florian¥fb351c13_PROTON.als
fb351c13

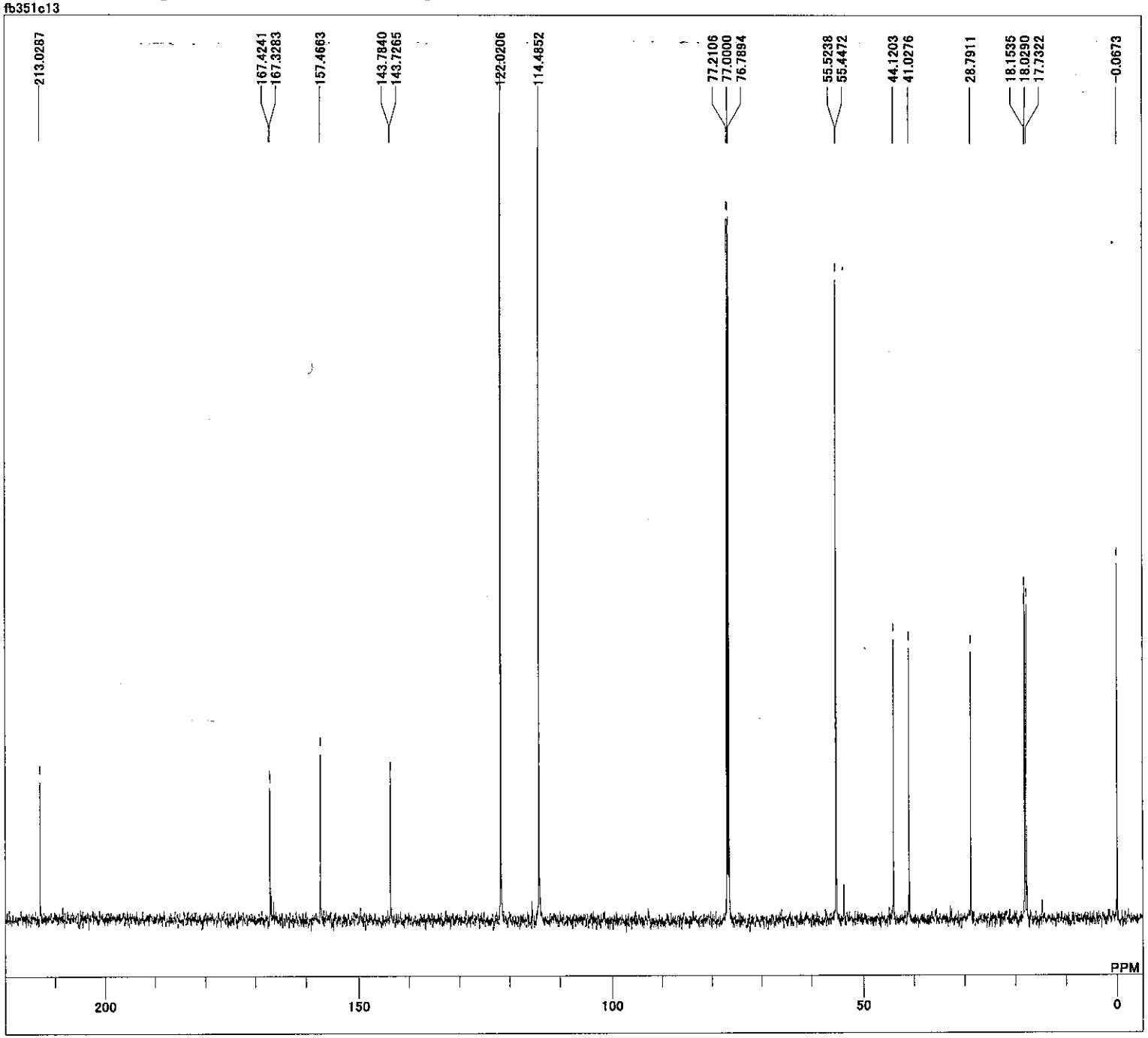


DFILE C:¥Documents and Settings¥All Users¥D
COMNT fb351c13
DATIM 30-04-2007 02:17:36
OBNUC 1H
EXMOD single_pulse.ex2
OBFRQ 600.17 MHz
OBSET 5.30 KHz
OBFIN 5.47 Hz
POINT 16384
FREQU 11261.26 Hz
SCANS 8
ACQTM 1.4549 sec
PD 4.0000 sec
PW1 7.30 usec
IRNUC 1H
CTEMP 19.0 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 32

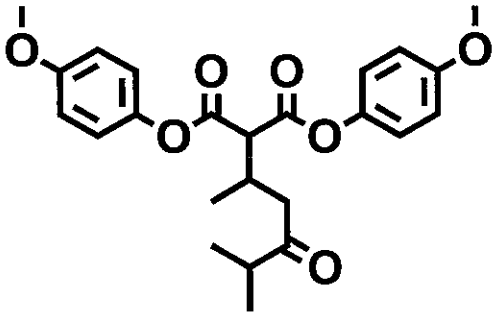


4j

C:¥Documents and Settings¥All Users¥Documents¥florian¥fb351c13_CARBON.als
fb351c13

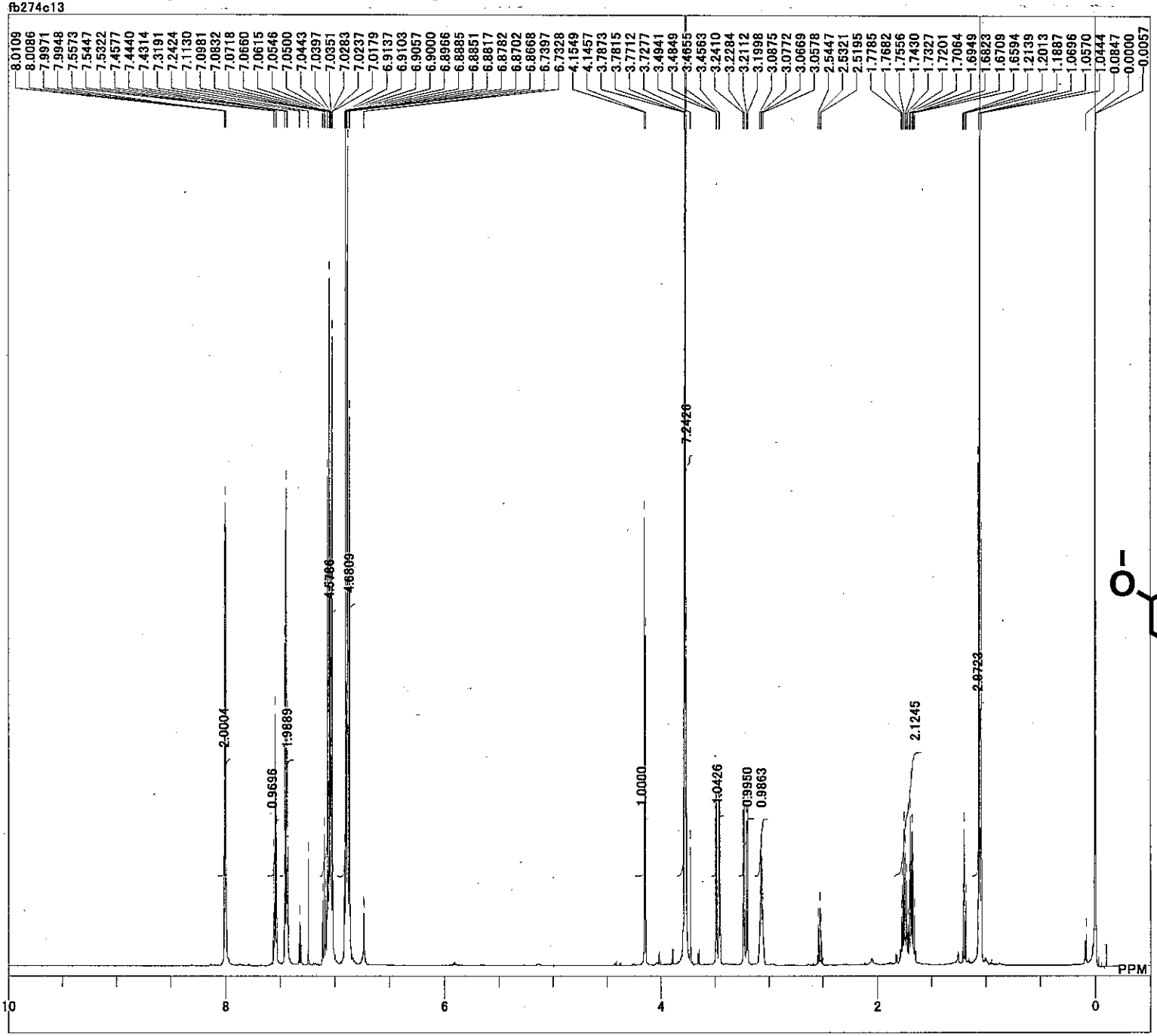


DFILE	C:¥Documents and Settings¥All Users¥Documents¥floria
COMNT	fb351c13
DATIM	30-04-2007 02:24:05
OBNUC	13C
EXMOD	single_pulse_dec
OBFRQ	150.92 MHz
OBSET	8.52 KHz
OBFIN	1.74 Hz
POINT	32768
FREQU	47348.48 Hz
SCANS	128
ACQTM	0.6921 sec
PD	2.0000 sec
PW1	3.45 usec
IRNUC	1H
CTEMP	19.9 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	60

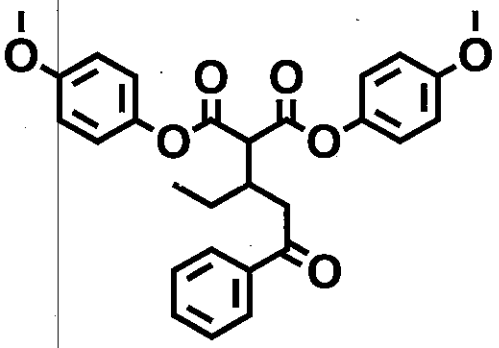


4j

C:¥Documents and Settings¥All Users¥Documents¥florian¥fb274c13_PROTON.als
fb274c13

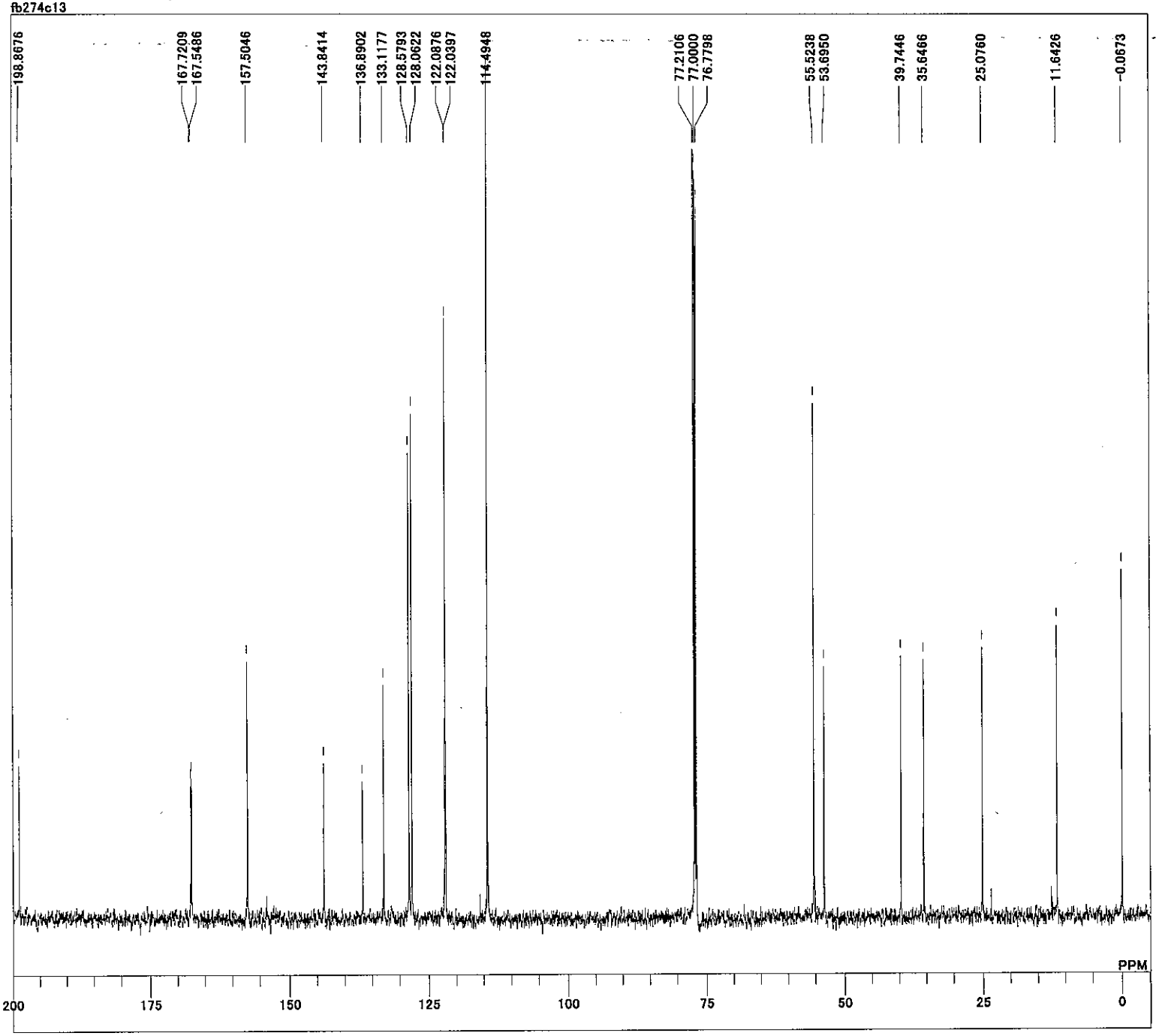


DFILE C:¥Documents and Settings¥All Users¥D
COMNT fb274c13
DATIM 30-04-2007 09:41:16
OBNUC 1H
EXMOD single_pulse.ex2
OBFRQ 600.17 MHz
OBSET 5.30 KHz
OBFIN 5.47 Hz
POINT 16384
FREQU 11261.26 Hz
SCANS 8
ACQTM 1.4549 sec
PD 4.0000 sec
PW1 7.30 usec
IRNUC 1H
CTEMP 24.9 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 32

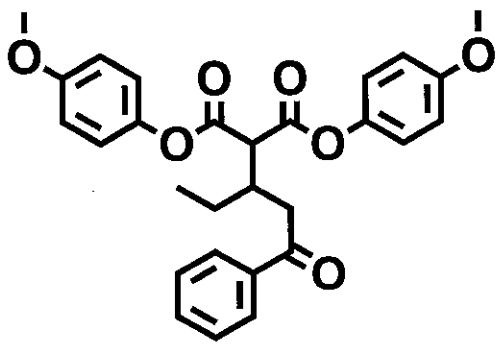


4k

C:¥Documents and Settings¥All Users¥Documents¥florian¥fb274c13_CARBON.als
fb274c13

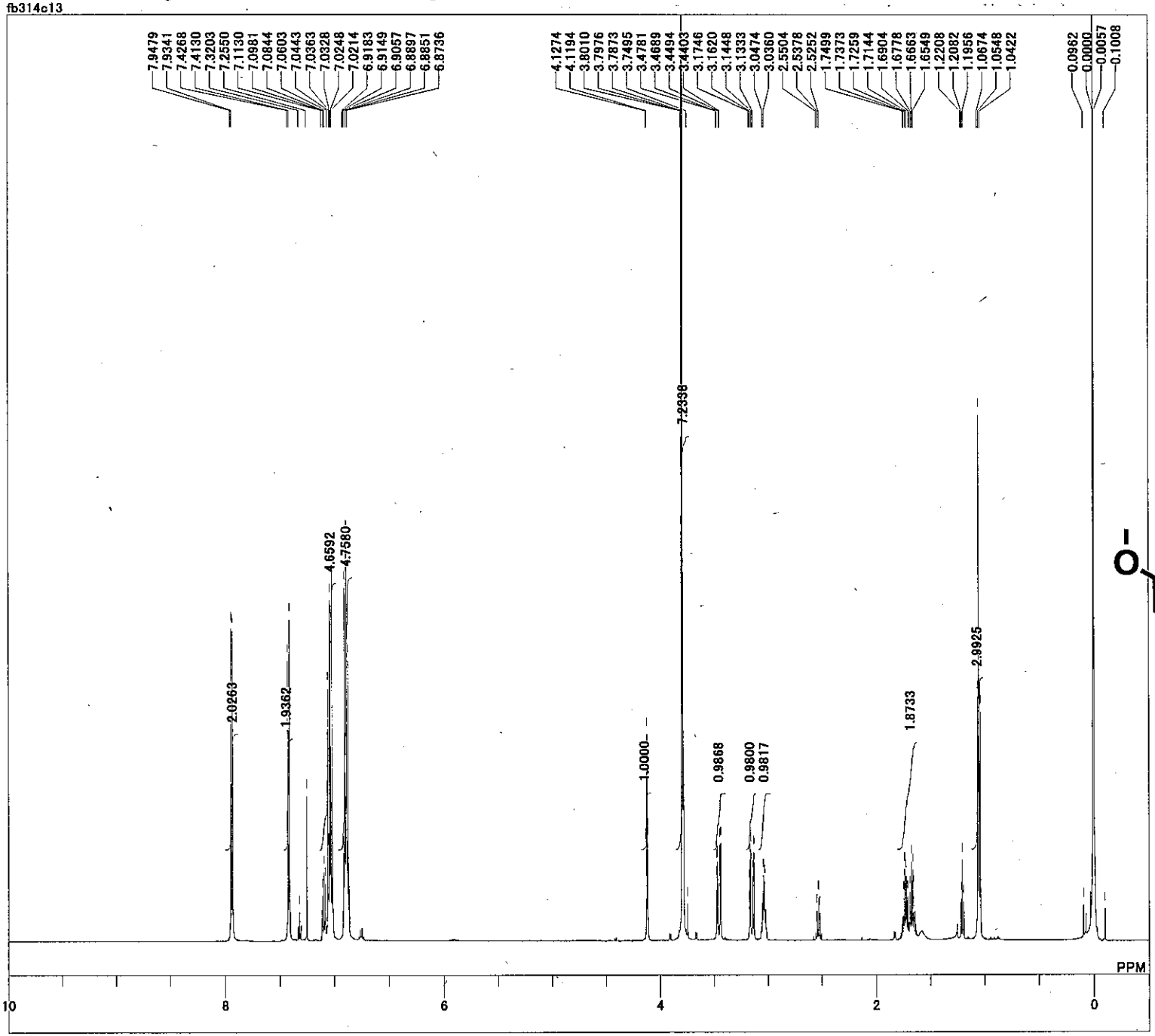


DFILE	C:¥Documents and Settings¥All Users¥Documents¥floria
COMNT	fb274c13
DATIM	30-04-2007 09:47:45
OBNUC	13C
EXMOD	single_pulse_dec
OBFRQ	150.92 MHz
OBSET	8.52 KHz
OBFIN	1.74 Hz
POINT	32768
FREQU	47348.48 Hz
SCANS	128
ACQTM	0.6921 sec
PD	2.0000 sec
PW1	3.45 usec
IRNUC	1H
CTEMP	25.9 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	60

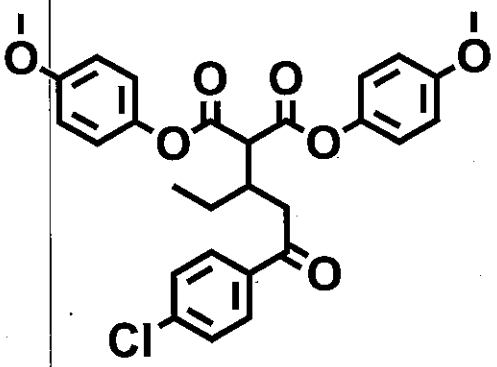


4k

C:¥Documents and Settings¥All Users¥Documents¥florian¥fb314c13_PROTON.als
fb314c13

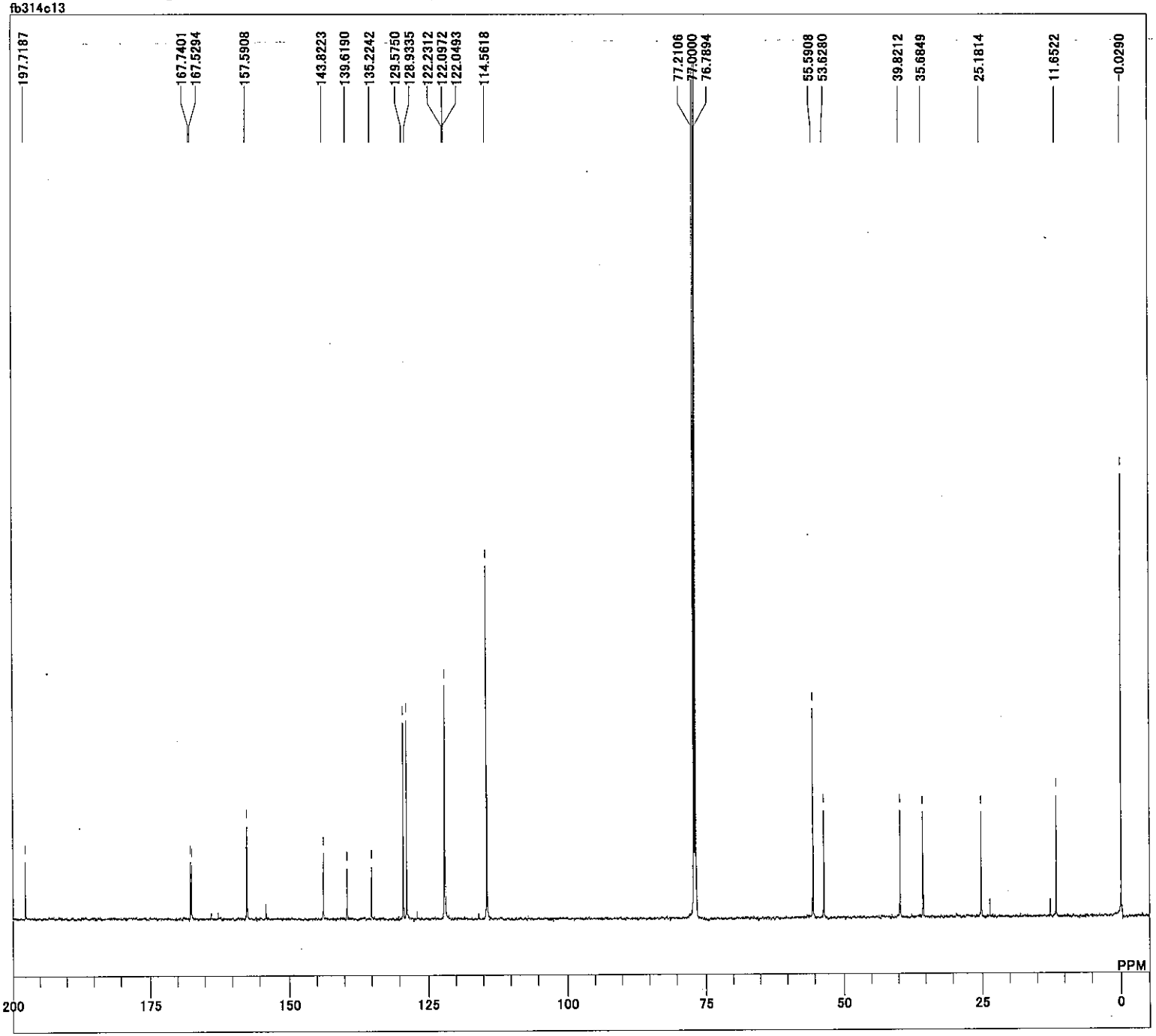


DFILE	C:¥Documents and Settings¥All Users¥D
COMNT	fb314c13
DATIM	30-04-2007 09:59:10
OBNUC	1H
EXMOD	single_pulse.ex2
OBFRQ	600.17 MHz
OBSET	5.30 KHz
OBFIN	5.47 Hz
POINT	16384
FREQU	11261.26 Hz
SCANS	16
ACQTM	1.4549 sec
PD	4.0000 sec
PW1	7.30 usec
IRNUC	1H
CTEMP	25.3 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	38

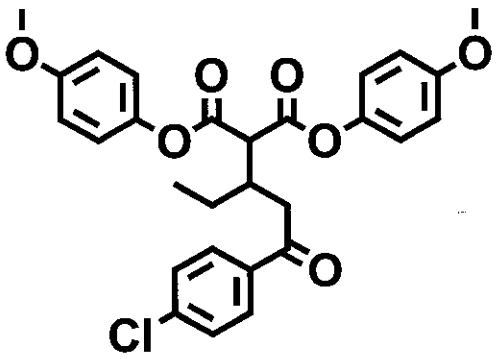


4l

C:¥Documents and Settings¥All Users¥Documents¥florian¥fb314c13_CARBON.als
fb314c13

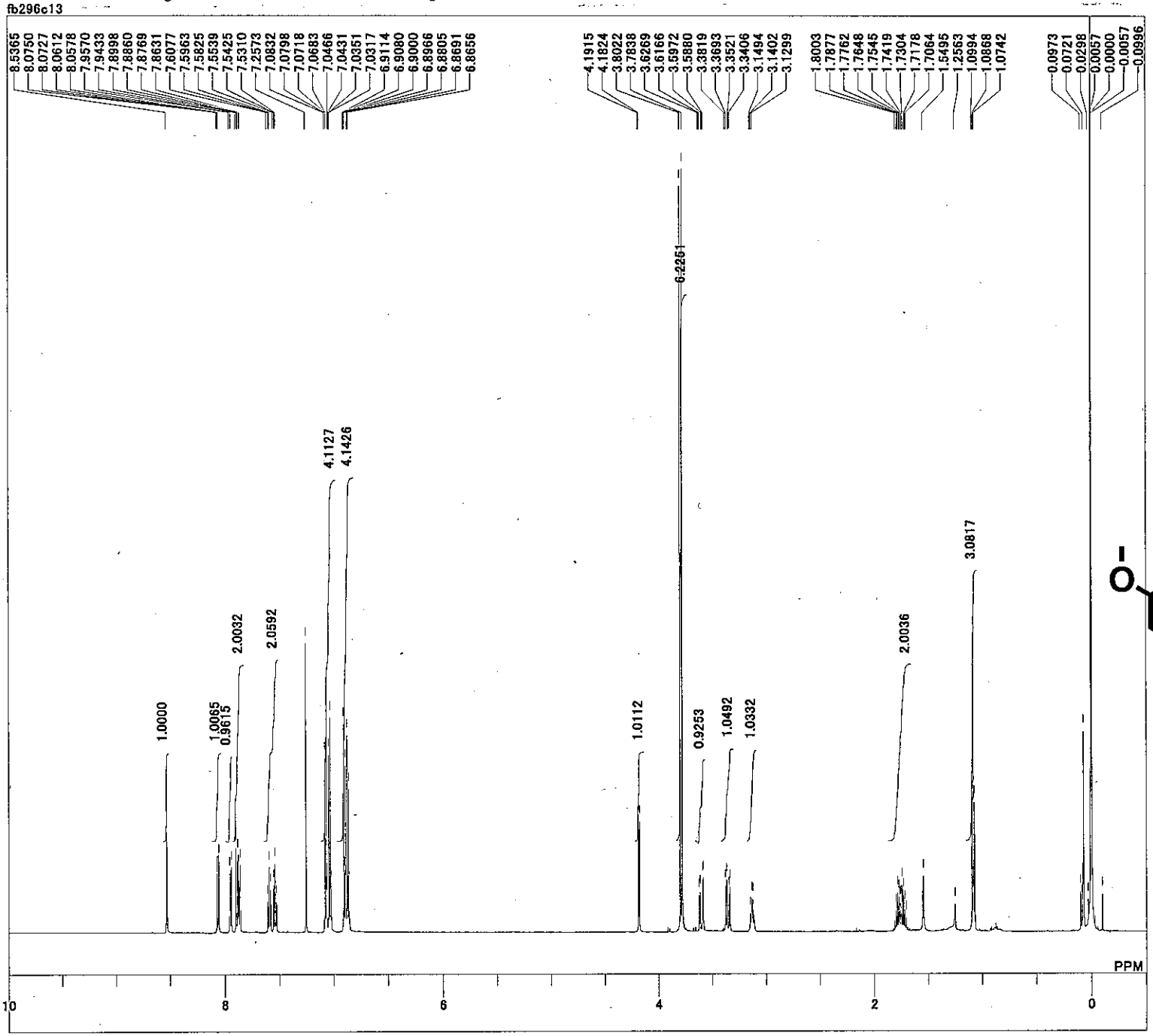


DFILE	C:¥Documents and Settings¥All Users¥Documents¥floria
COMNT	fb314c13
DATIM	30-04-2007 13:03:40
OBNUC	13C
EXMOD	single_pulse_dec
OBFRQ	150.92 MHz
OBSET	8.52 KHz
OBFIN	1.74 Hz
POINT	32768
FREQU	47348.48 Hz
SCANS	4096
ACQTM	0.6921 sec
PD	2.0000 sec
PW1	3.45 usec
IRNUC	1H
CTEMP	25.7 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	60

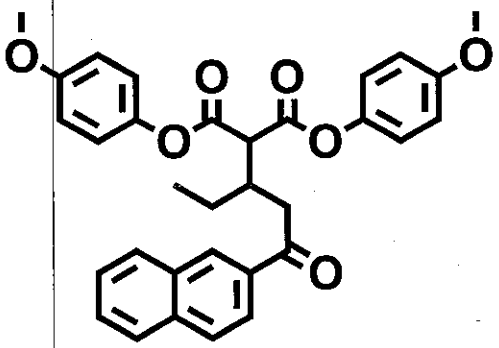


4l

C:¥Documents and Settings¥All Users¥Documents¥florian¥fb296c13_PROTON.als
fb296c13

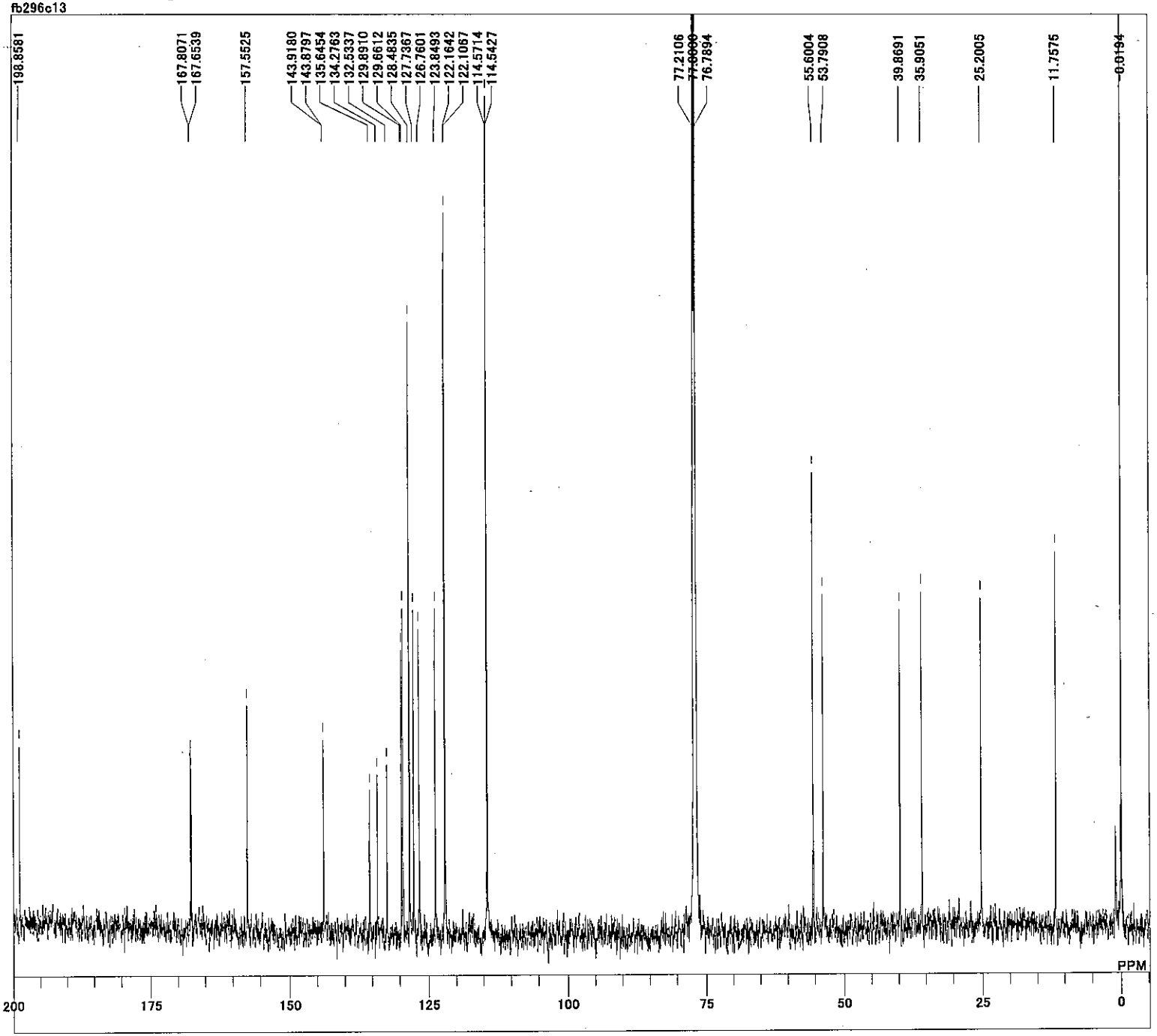


DFILE C:¥Documents and Settings¥All Users¥D
COMNT fb296c13
DATIM 30-04-2007 13:14:38
OBNUC 1H
EXMOD single_pulse.ex2
OBFRQ 600.17 MHz
OBSET 5.30 KHz
OBFIN 5.47 Hz
POINT 16384
FREQU 11261.26 Hz
SCANS 16
ACQTM 1.4549 sec
PD 4.0000 sec
PW1 7.30 usec
IRNUC 1H
CTEMP 24.7 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 44

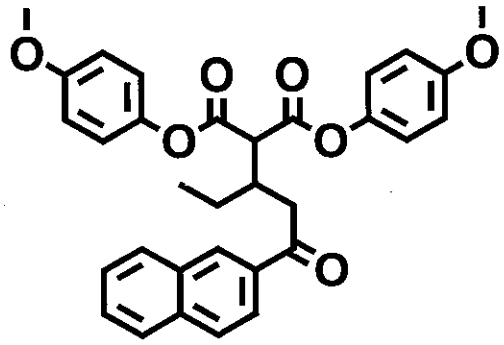


4m

C:¥Documents and Settings¥All Users¥Documents¥florian¥fb296c13_CARBON.als
fb296c13

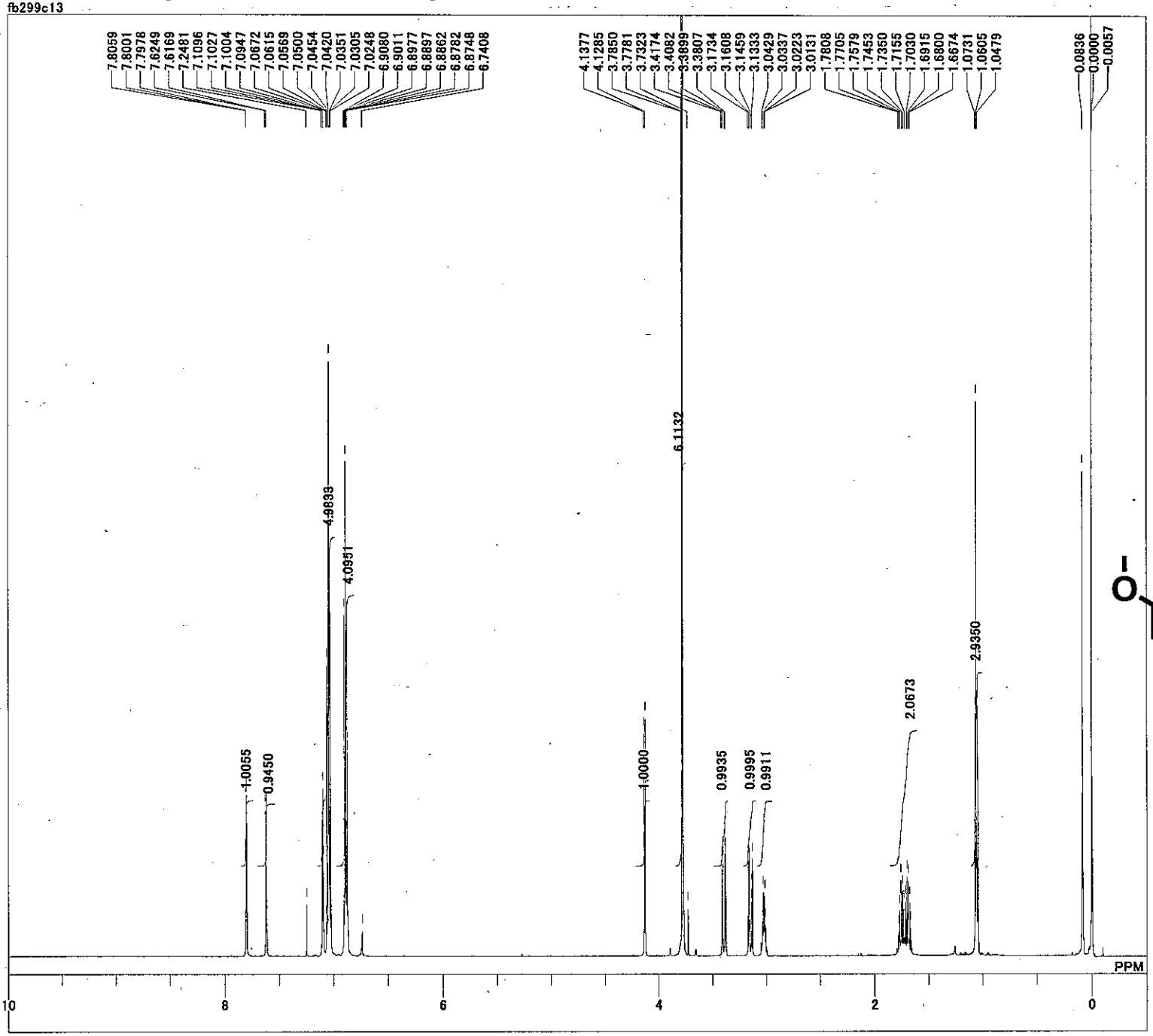


DFILE	C:¥Documents and Settings¥All Users¥Documents¥floria
COMNT	fb296c13
DATIM	30-04-2007 16:19:08
OBNUC	13C
EXMOD	single_pulse_dec
OBFRQ	150.92 MHz
OBSET	8.52 KHz
OBFIN	1.74 Hz
POINT	32768
FREQU	47348.48 Hz
SCANS	4096
ACQTM	0.6921 sec
PD	2.0000 sec
PW1	3.45 usec
IRNUC	1H
CTEMP	25.7 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	60

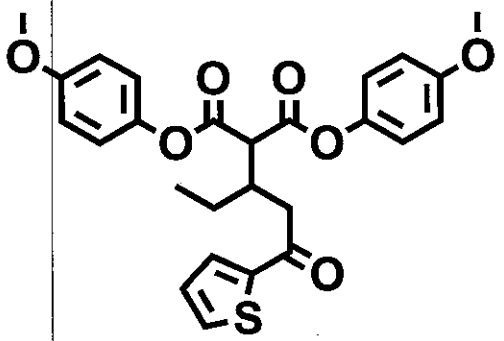


4m

C:¥Documents and Settings¥All Users¥Documents¥florian¥fb299c13_PROTON.als
fb299c13

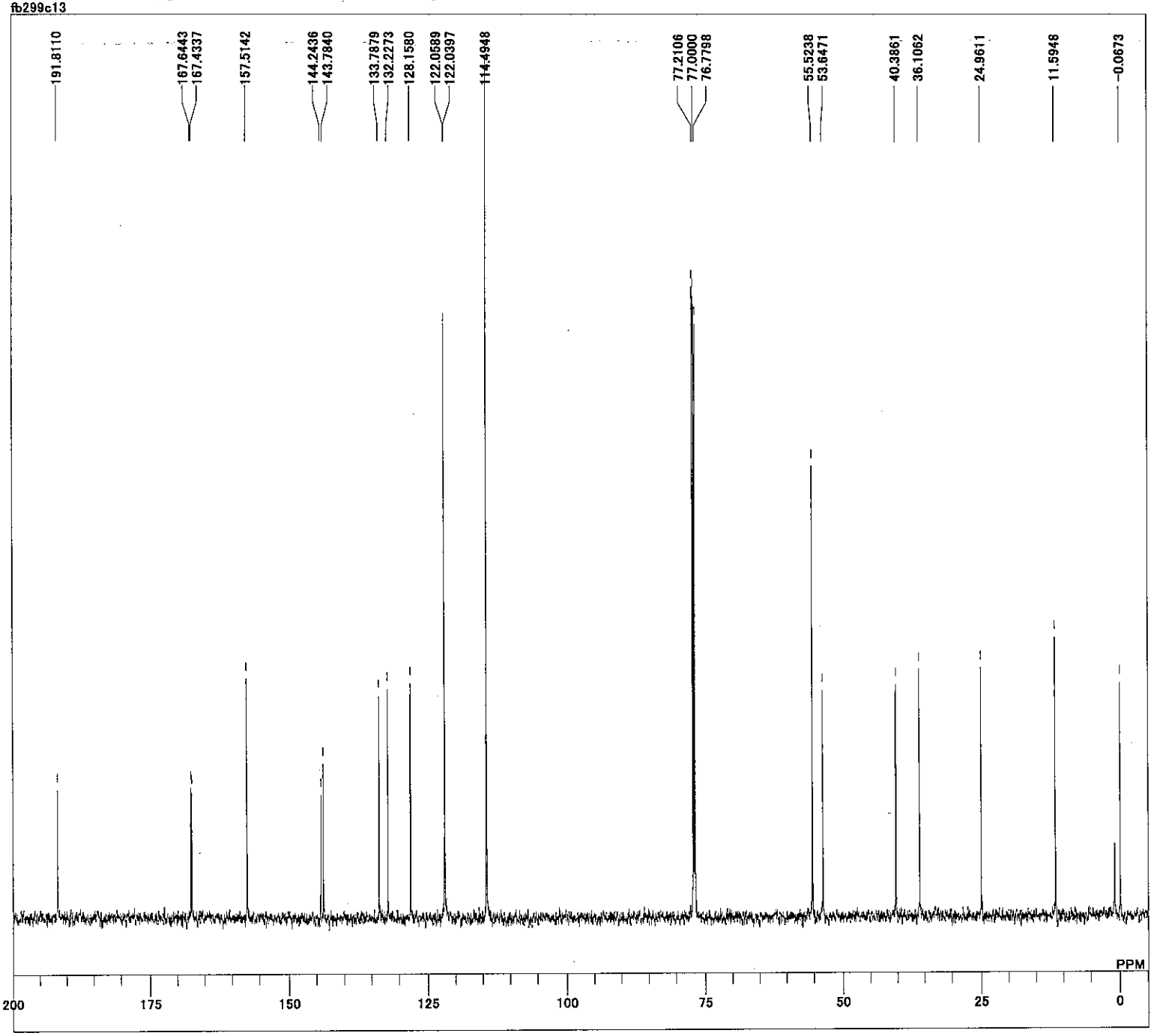


DFILE	C:¥Documents and Settings¥All Users¥D
COMNT	fb299c13
DATIM	30-04-2007 16:29:17
OBNUC	1H
EXMOD	single_pulse.ex2
OBFRQ	600.17 MHz
OBSET	5.30 KHz
OBFIN	5.47 Hz
POINT	16384
FREQU	11261.26 Hz
SCANS	8
ACQTM	1.4549 sec
PD	4.0000 sec
PW1	7.30 usec
IRNUC	1H
CTEMP	24.7 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	34

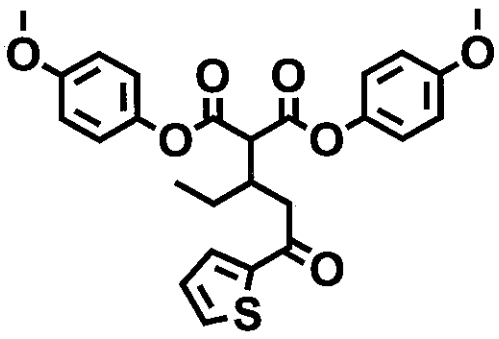


4n

C:¥Documents and Settings¥All Users¥Documents¥florian¥fb299c13_CARBON.als
fb299c13

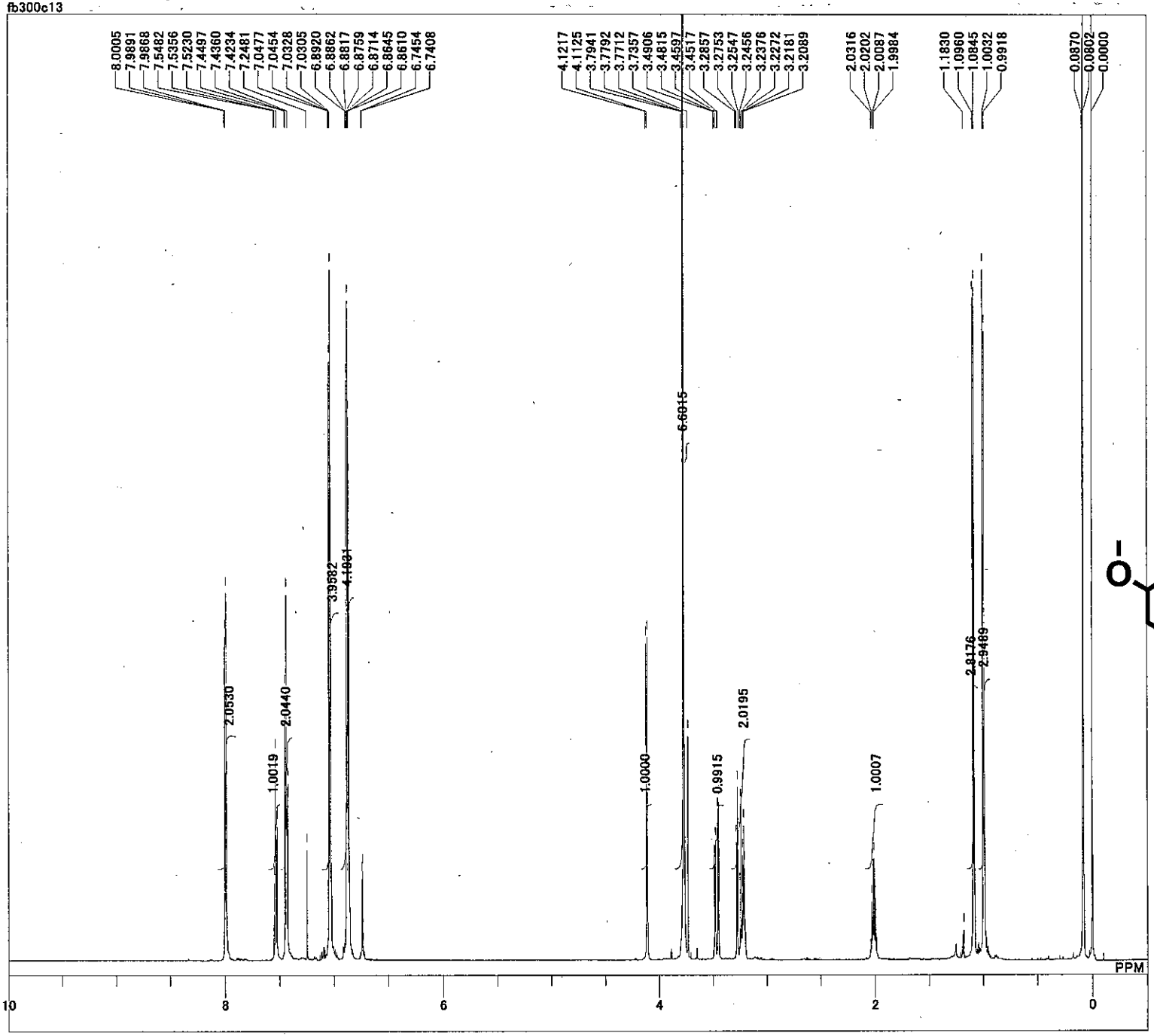


DFILE	C:¥Documents and Settings¥All Users¥Documents¥floria
COMNT	fb299c13
DATIM	30-04-2007 16:35:46
OBNUC	13C
EXMOD	single_pulse_dec
OBFRQ	150.92 MHz
OBSET	8.52 KHz
OBFIN	1.74 Hz
POINT	32768
FREQU	47348.48 Hz
SCANS	128
ACQTM	0.6921 sec
PD	2.0000 sec
PW1	3.45 usec
IRNUC	1H
CTEMP	25.6 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	60

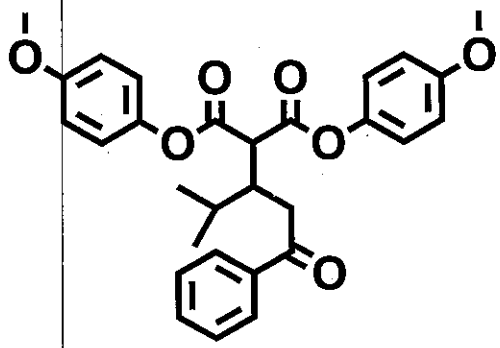


4n

C:¥Documents and Settings¥All Users¥Documents¥florian¥fb300c13_PROTON.als
fb300c13

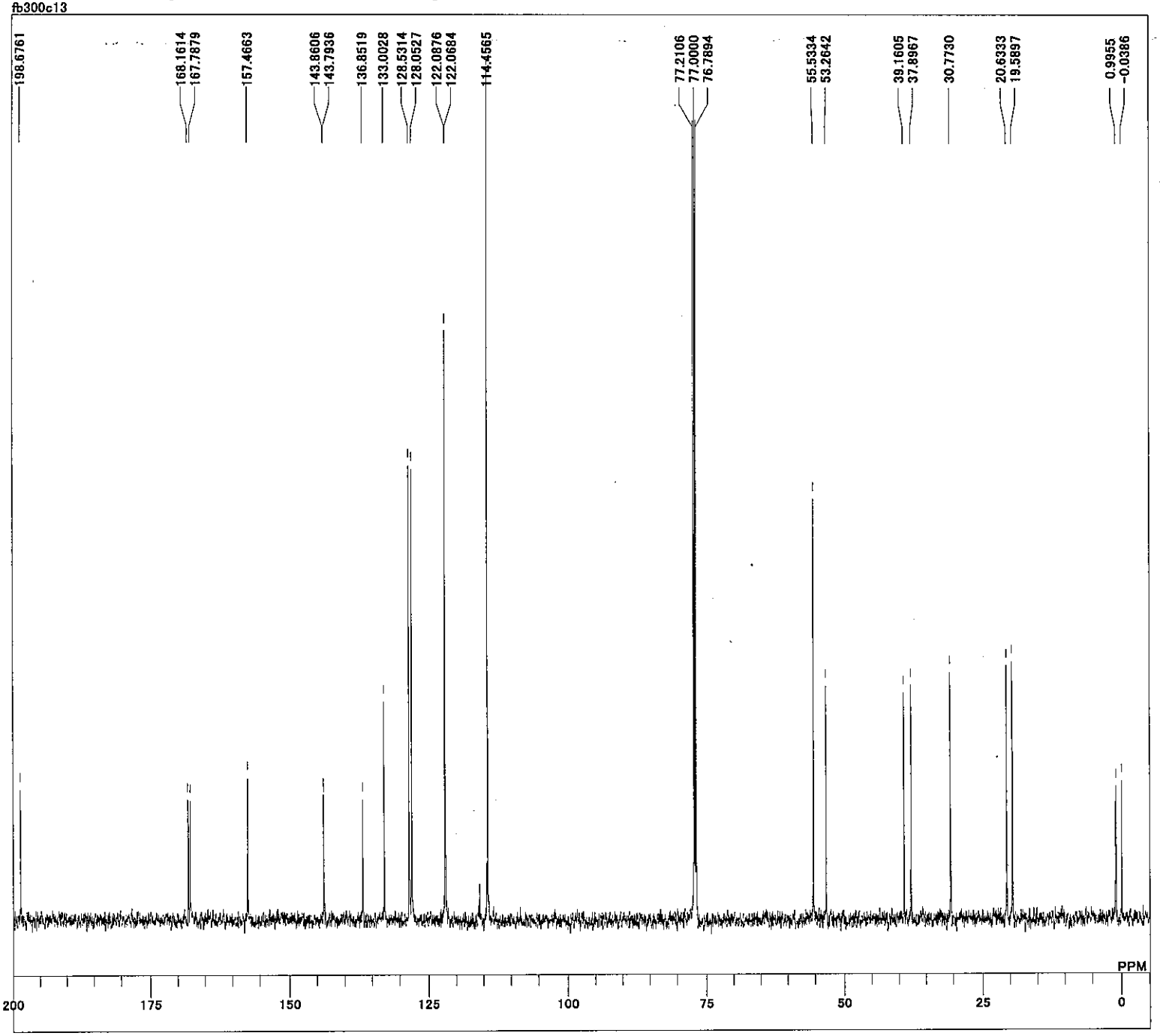


DFILE	C:¥Documents and Settings¥All Users¥D
COMNT	fb300c13
DATIM	30-04-2007 02:36:14
OBNUC	1H
EXMOD	single_pulse.ex2
OBFRQ	600.17 MHz
OBSET	5.30 KHz
OBFIN	5.47 Hz
POINT	16384
FREQU	11261.26 Hz
SCANS	8
ACQTM	1.4549 sec
PD	4.0000 sec
PW1	7.30 usec
IRNUC	1H
CTEMP	19.0 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	34

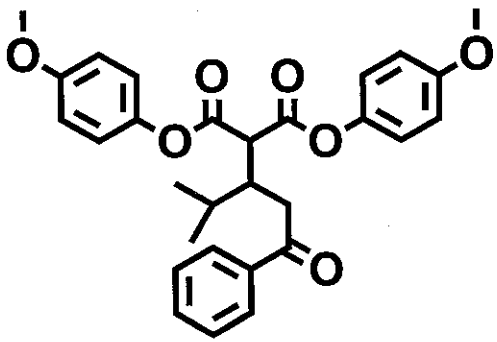


4o

C:¥Documents and Settings¥All Users¥Documents¥florian¥fb300c13_CARBON.als
fb300c13

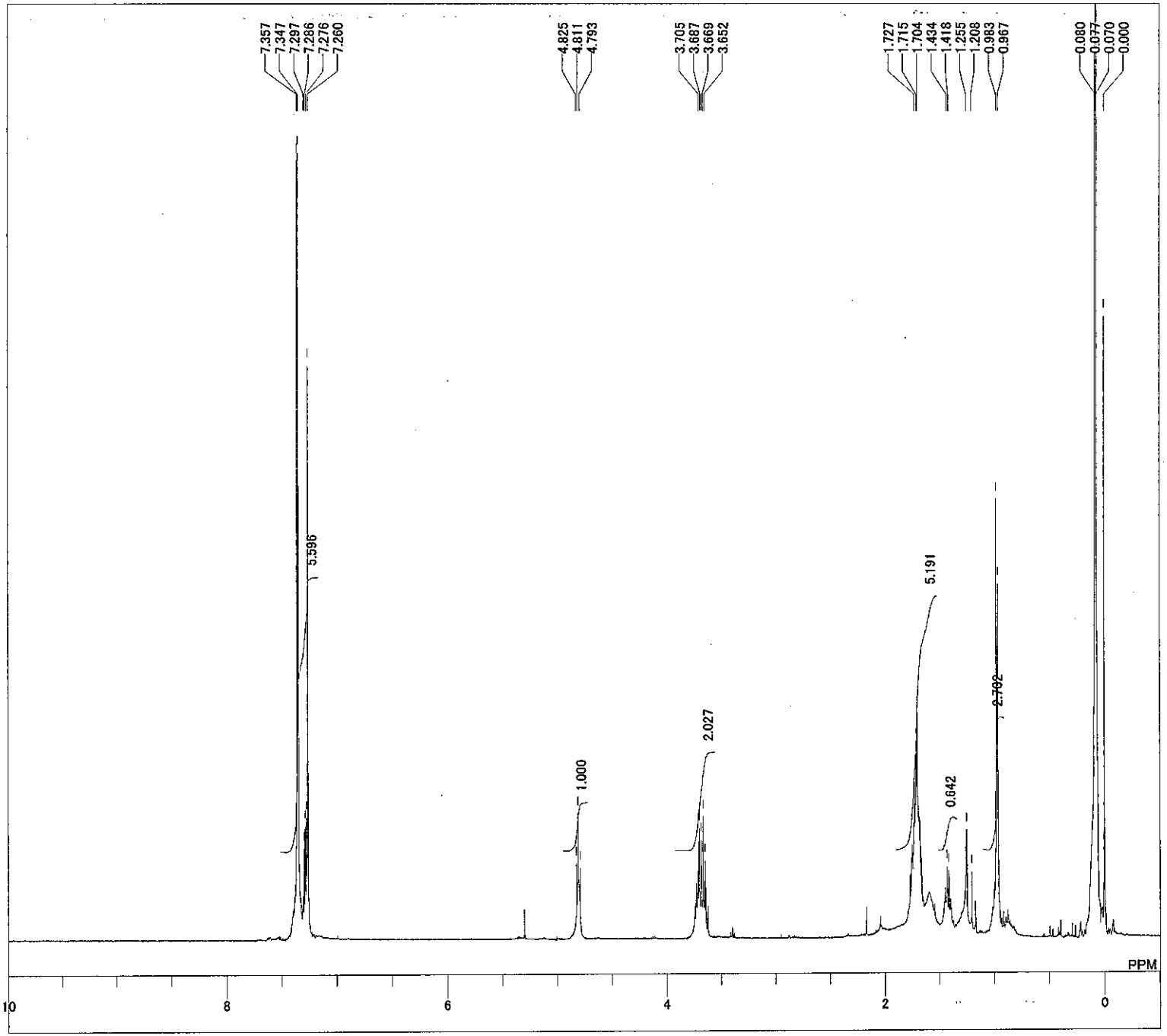


DFILE	C:¥Documents and Settings¥All Users¥Documents¥floria
COMNT	fb300c13
DATIM	30-04-2007 02:42:43
OBNUC	13C
EXMOD	single_pulse_dec
OBFRQ	150.92 MHz
OBSET	8.52 KHz
OBFIN	1.74 Hz
POINT	32768
FREQU	47348.48 Hz
SCANS	128
ACQTM	0.6921 sec
PD	2.0000 sec
PW1	3.45 usec
IRNUC	1H
CTEMP	20.1 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	60

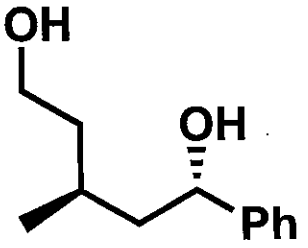


4o

C:¥Documents and Settings¥delta¥My Documents¥Personal Folder¥florian¥fb292ptlccommercup.als

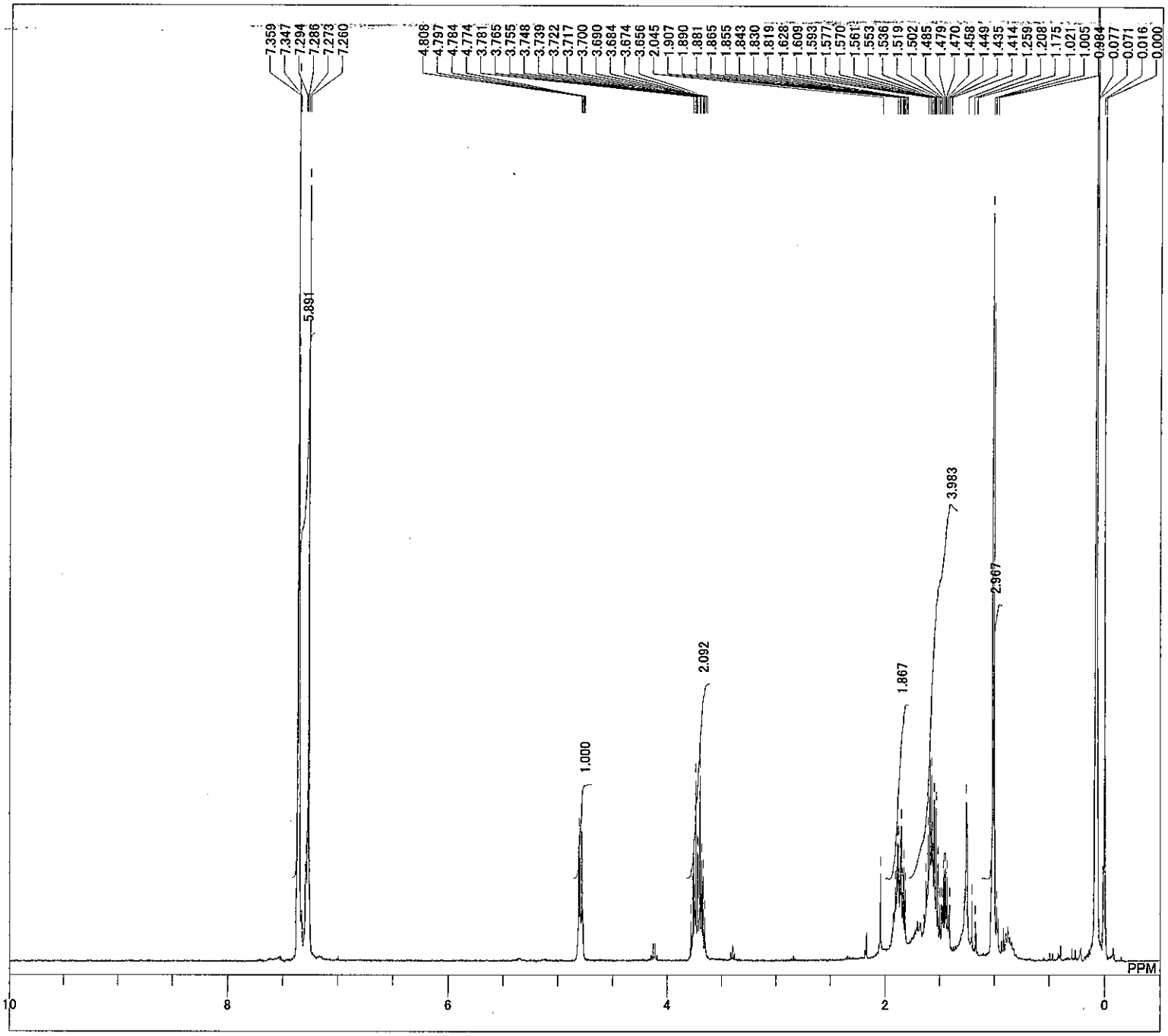


DFILE	C:¥Documents and Settings¥delta¥My Documents¥Personal
COMNT	
DATIM	08-02-2007 16:08:34
OBNUC	1H
EXMOD	single_pulse.ex2
OBFRQ	399.78 MHz
OBSET	4.19 KHz
OBFIN	7.29 Hz
POINT	16384
FREQU	7503.00 Hz
SCANS	8
ACQTM	2.1837 sec
PD	2.0000 sec
PW1	5.50 usec
IRNUC	1H
CTEMP	24.0 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	36

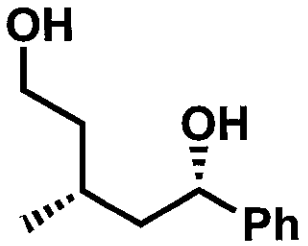


***Syn*-8**

C:¥Documents and Settings¥delta¥My Documents¥Personal Folder¥florian¥fb292ptlcommercdown.als

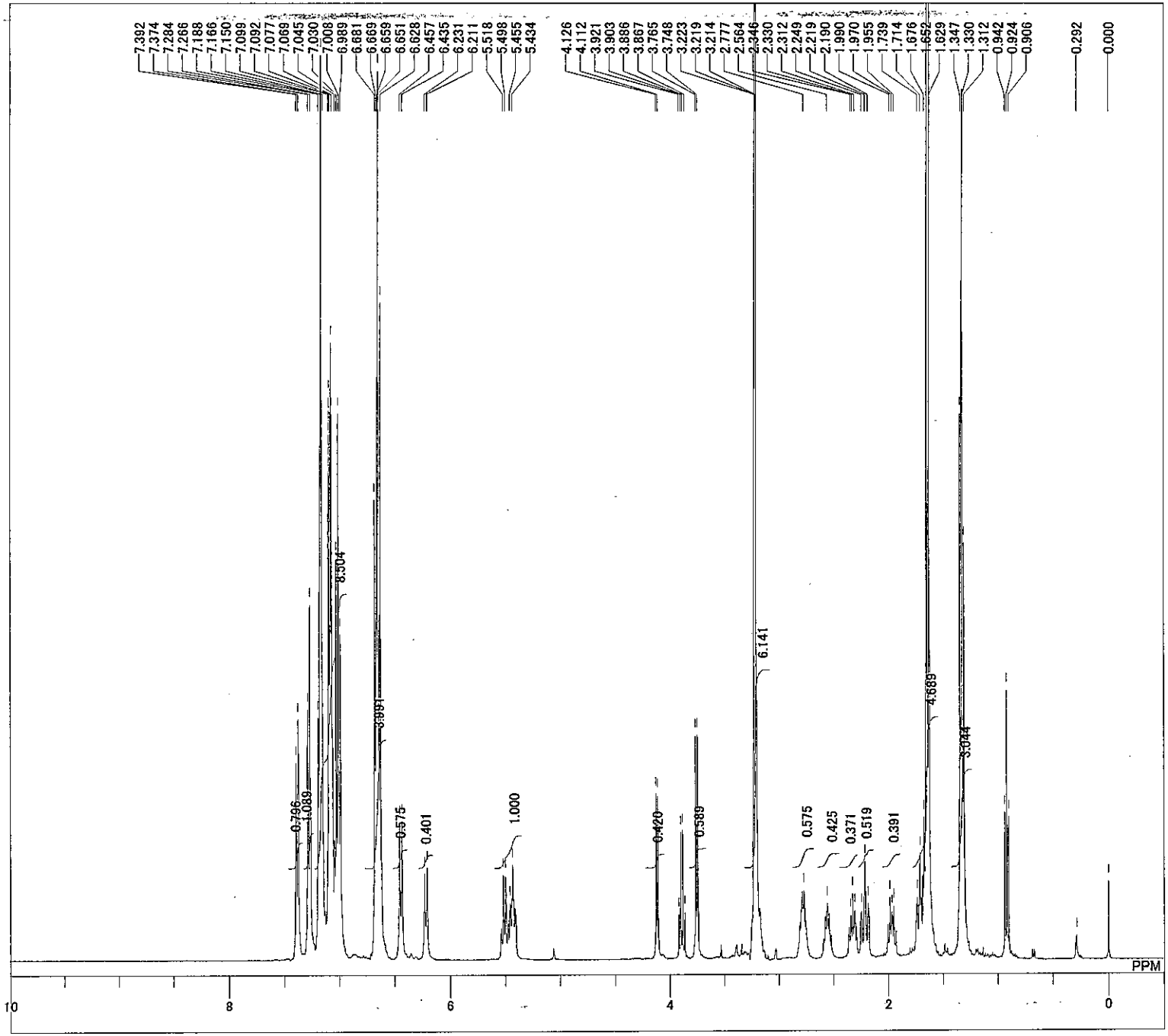


DFILE	C:¥Documents and Settings¥delta¥My Documents¥Person...
COMNT	
DATIM	08-02-2007 16:19:03
OBNUC	1H
EXMOD	single_pulse.ex2
OBFRQ	399.78 MHz
OBSET	4.19 KHz
OBFIN	7.29 Hz
POINT	16384
FREQU	7503.00 Hz
SCANS	8
ACQTM	2.1837 sec
PD	2.0000 sec
PW1	5.50 usec
IRNUC	1H
CTEMP	24.0 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	38

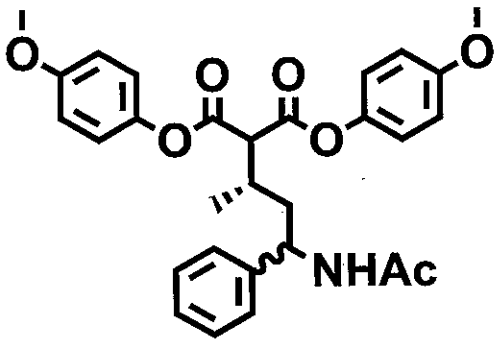


***Anti*-8**

C:¥Documents and Settings¥delta¥My Documents¥Personal Folder¥florian¥fb400ptlc.als

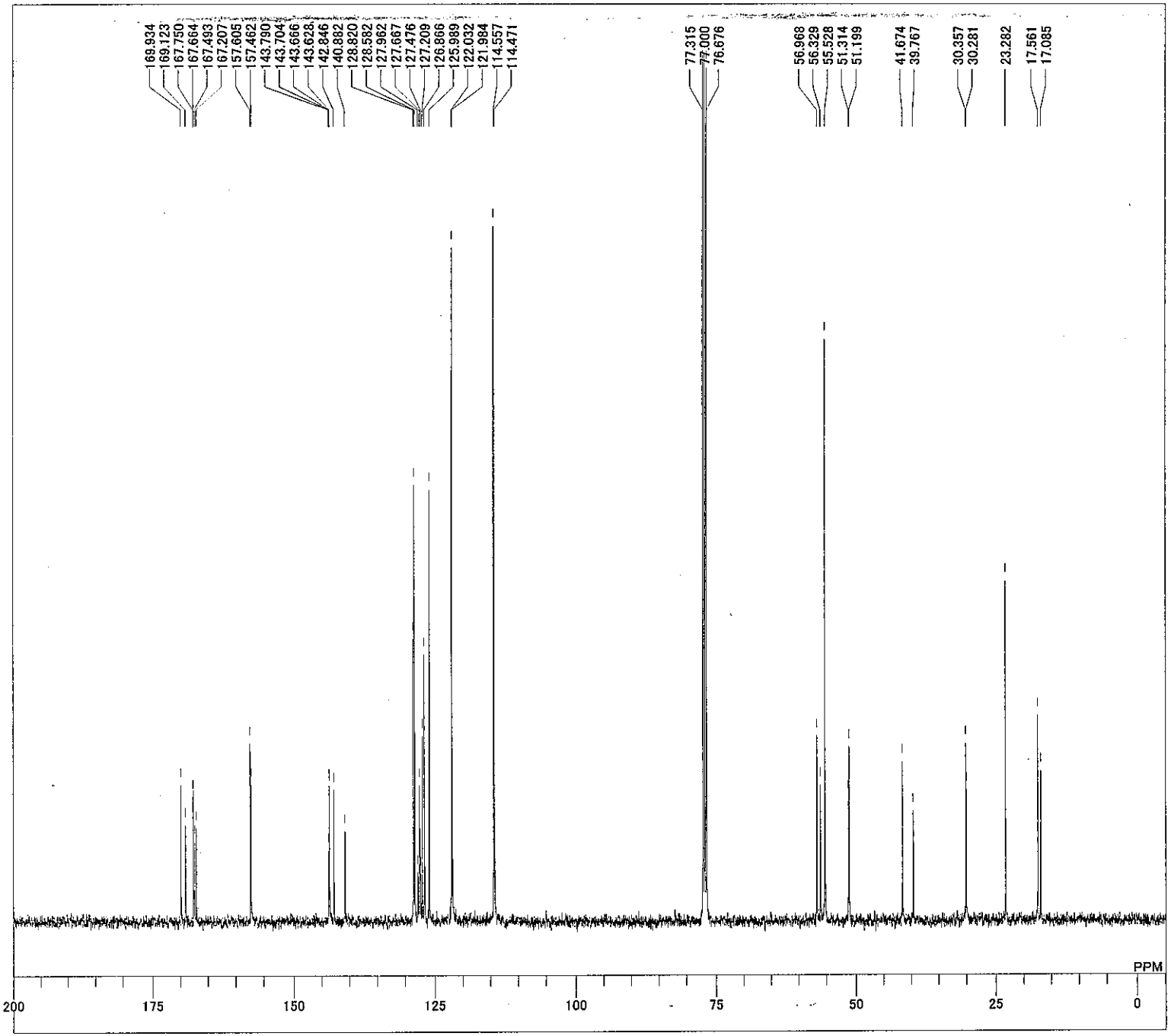


DFILE C:¥Documents-and-Settings¥delta¥My Documents¥Persona
COMNT
DATIM 26-05-2007 17:02:14
OBNUC 1H
EXMOD single_pulse.ex2
OBFRQ 399.78 MHz
OBSET 4.19 KHz
OBFIN 7.29 Hz
POINT 16384
FREQU 7503.00 Hz
SCANS 8
ACQTM 2.1837 sec
PD 2.0000 sec
PW1 5.90 usec
IRNUC 1H
CTEMP 20.2 c
SLVNT C6D6
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 28

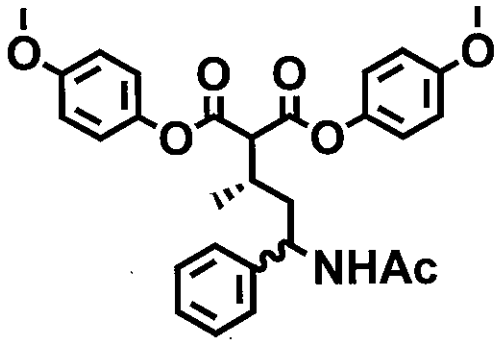


9

¥¥Eca¥data¥fb400c13.2



DFILE ¥¥Eca¥data¥fb400c13.2
COMNT
DATIM 07-06-2007 08:58:31
OBNUC 13C
EXMOD single_pulse_dec
OBFRQ 100.53 MHz
OBSET 5.35 KHz
OBFIN 5.86 Hz
POINT 40961
FREQU 39259.39 Hz
SCANS 655
ACQTM 1.0433 sec
PD 2.0000 sec
PW1 2.83 usec
IRNUC 1H
CTEMP 23.9 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 50



9