

Supporting Information
for
Cyclization of alkynoic acids in water
in the presence of a vesicular self-assembled amphiphilic pincer
palladium complex catalyst

Go Hamasala,^a and Yasuhiro Uozumi^{*a,b,c}

^aInstitute for Molecular Science (IMS), Higashiyama 5-1, Myodaiji, Okazaki 444-8787,
Japan

^bJST-CREST, Higashiyama 5-1, Myodaiji, Okazaki 444-8787, Japan

^cRiken, Wako 351-0198, Japan

E-mail: uo@ims.ac.jp

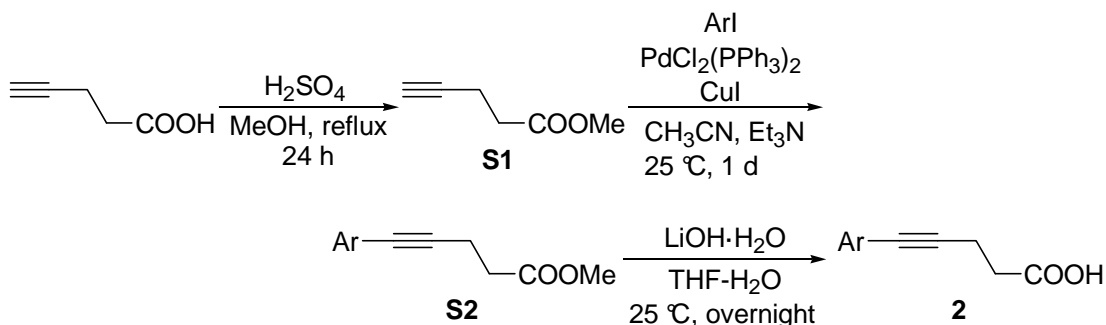
Contents:

General experiments remarks	S2
Preparation of alkynoic acids 2	S2-S10
Cyclization of alkynoic acids 2	S10-S15
Preparation of 2-phenylpropargyloxyphenol 4	S16-S18
Cyclization of 2-phenylpropargyloxyphenol 4	S18-S19
¹ H and ¹³ C{ ¹ H} NMR spectral data	S20-S61

Experimental Section

General. When manipulations were performed under a nitrogen atmosphere, nitrogen gas was dried by passage through P₂O₅. Commercially available chemicals (purchased from Aldrich, TCI, Kanto, Wako, Nakalai, and AlfaAfer) are used without further purification unless otherwise noted. NMR spectra recorded on a JEOL JNM-A500 spectrometer (500 MHz for ¹H, 125 MHz for ¹³C) or a JEOL ECS-400 spectrometer (396 MHz for ¹H, 100 MHz for ¹³C). Chemical shifts are reported in δ ppm referenced to an internal tetramethylsilane standard for ¹H NMR. Chemical shifts of ¹³C NMR are given related to CDCl₃ as an internal standard (δ 77.0). ¹H and ¹³C NMR spectra were recorded in CDCl₃ at 25 °C unless otherwise noted. HREI mass spectra were recorded on a JEOL AccuTOF GC JMS-T100GC equipped with Agilent 6890N GC. EI mass spectra were recorded on an Agilent 5973N equipped with Agilent 6890N GC. Melting points were determined using a Yanaco micro melting point apparatus MP-J3 and are uncorrected. IR spectra were obtained using a JASCO FT/IR-460plus spectrometer in ATR mode. Millipore water was obtained from a Millipore Milli-Q Biocel A10 purification unit.

Preparation of alkynoic acids **2**



Scheme S1. Preparation of alkynoic acids **2**.

Methyl-4-pentynoate (S1)



To a stirred MeOH (50 mL) solution of 4-pentynoic acid (5.00 g, 50.7 mmol) was added H₂SO₄ (1.63 mL, 30.6 mmol). After refluxing for 24 h, the reaction mixture was quenched by the addition of saturated sodium bicarbonate aqueous solution and the resulting solution was extracted with CH₂Cl₂ (30 mL × 3). The obtained organic layer was dried over magnesium sulfate. The solution was concentrated in vacuo to give

methyl-4-pentynoate (**S1**, 5.35 g, 47.7 mmol, 94.0%) as pale yellow oil.

$^1\text{H NMR}$ (396 MHz, CDCl_3) δ 1.97–1.99 (m, 1H, CCH), 2.48–1.60 (m, 4H, CH_2CH_2), 3.71 (s, 3H, COOCH_3); $^{13}\text{C}\{^1\text{H}\}$ NMR (100 MHz, CDCl_3) δ 14.2, 33.0, 51.7, 68.9, 82.3, 172.1; MS (EI) m/z = 111 ($[\text{M}+1]^+$).

General procedure for the Sonogashira coupling reaction of **S1** with **ArI**.

A typical procedure is given for the Sonogashira reaction of methyl-4-pentynoate with 4-trifluoromethyliodobenzene. To a suspension of $\text{PdCl}_2(\text{PPh}_3)_2$ (125 mg, 0.178 mmol) and copper iodide (68 mg, 0.357 mmol) in triethylamine (3 mL), methyl-4-pentynoate (**S1**, 1.00 g, 8.92 mmol) in anhydrous CH_3CN (1 mL) and 4-trifluoromethyliodobenzene (2.67 g, 9.81 mmol) were added. After being stirred for 1 day, saturated NH_4Cl aqueous solution (15 mL) was added to the reaction mixture and the resulting solution was extracted with AcOEt (15 mL, 4 times). The combined organic layer was dried over Na_2SO_4 . After evaporation of the solvent, the resulting residue was chromatographed on silica gel (eluent: 0-5% AcOEt/*n*-hexane) to give methyl 5-(4-trifluoromethylphenyl)-4-pentynoate (**S2a**, 1.85 g, 7.21 mmol, 81%) as pale yellow oil.

Methyl 5-(4-trifluoromethylphenyl)-4-pentynoate (**S2a**)



81% yield; Pale yellow oil; $^1\text{H NMR}$ (500 MHz, CDCl_3) δ 2.65 (t, J = 8 Hz, 2H, CH_2), 2.75 (t, J = 8 Hz, 2H, CH_2), 3.73 (s, 3H, CH_3), 7.48 (d, J = 8 Hz, 2H, ArH), 7.53 (d, J = 8 Hz, 2H, ArH); $^{13}\text{C}\{^1\text{H}\}$ NMR (125 MHz, CDCl_3) δ 15.3, 33.1, 51.8, 80.0, 90.7, 123.9 (q, $J_{\text{C-F}}$ = 270 Hz), 125.1 (q, $J_{\text{C-F}}$ = 4 Hz), 128.3 (q, $J_{\text{C-F}}$ = 261 Hz), 129.7, 131.8, 172.8; IR (ATR) 2999, 2955, 1738 (C=O), 1616, 1438, 1406, 1366, 1321 (CF_3), 1255, 1199, 1163, 1121, 1104, 1066, 1038, 1017, 989, 889, 840, 777, 738, 693, 597 cm^{-1} ; HR-EI-MS Calcd. for $\text{C}_{13}\text{H}_{11}\text{F}_3\text{O}_2$ $[\text{M}]^+$: m/z = 256.07111; Found 256.07061.

Methyl 5-phenyl-4-pentynoate (**S2b**)



68% yield; Colorless oil; $^1\text{H NMR}$ (500 MHz, CDCl_3) δ 2.62–2.67 (m, 2H, CH_2), 2.71–2.76 (m, 2H, CH_2), 3.72 (s, 3H, CH_3), 7.26–7.30 (m, 3H, ArH), 7.37–7.40 (m, 2H,

ArH); $^{13}\text{C}\{^1\text{H}\}$ NMR (125 MHz, CDCl_3) δ 15.3, 33.4, 51.8, 81.2, 87.9, 123.5, 127.8, 128.2, 131.6, 172.4; MS (EI) m/z = 188 ($[\text{M}^+]$).

Methyl 5-(4-methylphenyl)-4-pentynoate (S2c)



75% yield; Color less oil; ^1H NMR (395 MHz, CDCl_3) δ 2.32 (s, 3H, CH_3), 2.61–2.66 (m, 2H, CH_2), 2.70–2.75 (m, 2H, CH_2), 3.72 (s, 3H, CH_3), 7.08 (d, J = 8 Hz, 2H, ArH), 7.27 (d, J = 8 Hz, 2H, ArH); $^{13}\text{C}\{^1\text{H}\}$ NMR (100 MHz, CDCl_3) δ 15.3, 21.4, 33.5, 51.8, 81.2, 87.1, 120.3, 128.9, 131.4, 137.8, 172.4; MS (EI) m/z = 202 ($[\text{M}^+]$).

Methyl 5-(4-methoxyphenyl)-4-pentynoate (S2d)



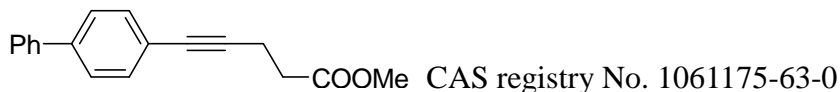
78% yield; Colorless oil; ^1H NMR (500 MHz, CDCl_3) δ 2.60–2.64 (m, 2H, CH_2), 2.69–2.73 (t, 2H, CH_2), 3.71 (s, 3H, CH_3), 3.78 (s, 3H, CH_3), 6.80 (d, J = 9 Hz, 2H, ArH), 7.31 (d, J = 9 Hz, 2H, ArH); $^{13}\text{C}\{^1\text{H}\}$ NMR (125 MHz, CDCl_3) δ 15.3, 33.5, 51.7, 55.1, 80.9, 86.3, 113.7, 115.5, 132.9, 159.1, 172.4; MS (EI) m/z = 218 ($[\text{M}^+]$).

Methyl 5-(4-tert-butylphenyl)-4-pentynoate (S2e)



47% yield (from 4-tert-butylbromobenzene: 5 mol% $\text{PdCl}_2(\text{PPh}_3)_2$ and 10 mol% CuI were used. The reaction performed in a mixture of $i\text{Pr}_2\text{NH}$ and toluene at 85 °C); Pale yellow oil; ^1H NMR (396 MHz, CDCl_3) δ 1.29 (s, 9H, $\text{C}(\text{CH}_3)_3$), 2.61–2.65 (m, 2H, CH_2), 2.70–2.75 (m, 2H, CH_2), 3.71 (s, 3H, CH_3), 7.28–7.33 (m, 4H, ArH); $^{13}\text{C}\{^1\text{H}\}$ NMR (100 MHz, CDCl_3) δ 15.3, 33.1, 33.5, 34.6, 51.8, 81.1, 87.1, 120.4, 125.1, 131.2, 150.9, 172.4; IR (ATR) 2959, 2905, 2869, 1737 (C=O), 1648, 1608, 1506, 1459, 1437, 1419, 1396, 1363, 1268, 1256, 1223, 1198, 1166, 1110, 1018, 991, 834, 779, 705, 562 cm^{-1} ; HR-EI-MS Calcd. for $\text{C}_{16}\text{H}_{20}\text{O}_2$ $[\text{M}]^+$: m/z = 244.14633; Found 244.14619.

Methyl 5-(4-phenylphenyl)-4-pentynoate (S2f)



75% yield; Pale yellow oil; $^1\text{H NMR}$ (500 MHz, CDCl_3) δ 2.65 (t, $J = 8$ Hz, 2H, CH_2), 2.76 (t, $J = 8$ Hz, 2H, CH_2), 3.72 (s, 3H, CH_3), 7.32–7.36 (m, 1H, ArH), 7.41–7.46 (m, 4H, ArH), 7.52 (d, $J = 8$ Hz, 2H, ArH), 7.57 (d, $J = 8$ Hz, 2H, ArH); $^{13}\text{C}\{^1\text{H}\}$ NMR (125 MHz, CDCl_3) δ 15.4, 33.4, 51.8, 81.0, 88.6, 122.4, 126.8, 126.9, 127.4, 128.7, 131.9, 140.3, 140.5, 172.3; MS (EI) $m/z = 264$ ($[\text{M}]^+$).

Methyl 5-(4-nitrophenyl)-4-pentynoate (S2g)



85% yield; Yellow solids; **Mp.** 88.5-89.5 °C; $^1\text{H NMR}$ (500 MHz, CDCl_3) δ 2.66 (t, $J = 7$ Hz, 2H, CH_2), 2.78 (t, $J = 7$ Hz, 2H, CH_2), 3.74 (s, 3H, CH_3), 7.51 (d, $J = 9$, 4 Hz, 2H, ArH), 8.15 (d, $J = 9$ Hz, 2H, ArH); $^{13}\text{C}\{^1\text{H}\}$ NMR (125 MHz, CDCl_3) δ 15.3, 32.9, 51.8, 79.6, 94.0, 123.4, 130.4, 132.2, 146.7, 171.9; **IR (ATR)** 3113, 2951, 2844, 2219 (CC), 1730 (C=O), 1591, 1509 (NO_2), 1491, 1434, 1372, 1339, 1309, 1300, 1285, 1273, 1256, 1198, 1173, 1108, 1054, 1011, 986, 971, 887, 852, 831, 775, 749, 687, 591, 580 cm^{-1} ; **HR-EI-MS** Calcd. for $\text{C}_{12}\text{H}_{11}\text{NO}_4$ $[\text{M}]^+$: $m/z = 233.06881$; Found 233.06890.

Methyl 5-(4-fluorophenyl)-4-pentynoate (S2h)



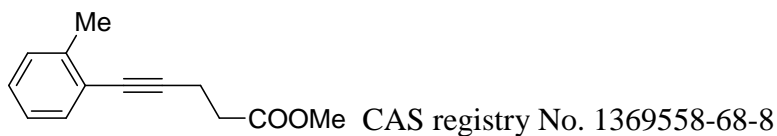
74% yield; Colorless oil; $^1\text{H NMR}$ (500 MHz, CDCl_3) δ 2.63 (t, $J = 8$ Hz, 2H, CH_2), 2.72 (t, $J = 8$ Hz, 2H, CH_2), 3.72 (s, 3H, CH_3), 6.94–6.99 (m, 2H, ArH), 7.33–7.37 (m, 2H, ArH); $^{13}\text{C}\{^1\text{H}\}$ NMR (125 MHz, CDCl_3) δ 15.2, 33.3, 51.8, 80.1, 87.6, 115.3 (d, $J_{\text{C-F}} = 22$ Hz), 119.5 (d, $J_{\text{C-F}} = 4$ Hz), 133.3 (d, $J_{\text{C-F}} = 8$ Hz), 162.1 (d, $J_{\text{C-F}} = 248$ Hz), 172.3; MS (EI) $m/z = 206$ ($[\text{M}]^+$).

Methyl 5-(4-chlorophenyl)-4-pentynoate (S2i)



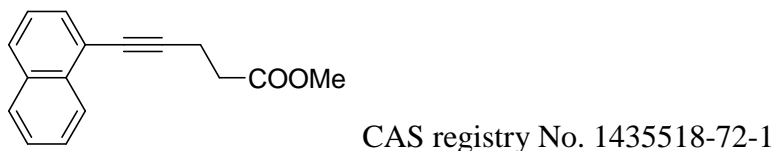
79% yield; Colorless oil; $^1\text{H NMR}$ (500 MHz, CDCl_3) δ 2.62 (t, $J = 8$ Hz, 2H, CH_2), 2.72 (t, $J = 8$ Hz, 2H, CH_2), 3.71 (s, 3H, CH_3), 7.24 (d, $J = 9$ Hz, 2H, ArH), 7.30 (d, $J = 9$ Hz, 2H, ArH); $^{13}\text{C}\{^1\text{H}\}$ NMR (125 MHz, CDCl_3) δ 15.3, 33.2, 51.8, 80.1, 89.0, 122.0, 128.5, 132.8, 133.7, 172.2; MS (EI) $m/z = 222$ ($[\text{M}]^+$).

Methyl 5-(2-methylphenyl)-4-pentynoate (S2j)



74% yield; Colorless oil; $^1\text{H NMR}$ (500 MHz, CDCl_3) δ 2.39 (s, 3H, CH_3), 2.65 (t, $J = 7$ Hz, 2H, CH_2), 2.77 (t, $J = 7$ Hz, 2H, CH_2), 3.72 (s, 3H, CH_3), 7.07–7.12 (m, 1H, ArH), 7.15–7.18 (m, 2H, ArH), 7.34 (d, $J = 7$ Hz, 1H, ArH); $^{13}\text{C}\{^1\text{H}\}$ NMR (125 MHz, CDCl_3) δ 15.5 (CH_2), 20.6 (CH_3), 33.6 (CH_2), 51.7 (CH_3), 80.1 (CC), 91.9 (CC), 123.2 (ArC), 125.3 (ArC), 127.8 (ArC), 129.3 (ArC), 131.8 (ArC), 140.0 (ArC), 172.4 (C=O); MS (EI) $m/z = 202$ ($[\text{M}^+]$).

Methyl 5-(1-naphthyl)-4-pentynoate (S2k)



71% yield; Colorless oil; $^1\text{H NMR}$ (500 MHz, CDCl_3) δ 2.73 (t, $J = 8$ Hz, 2H, CH_2), 2.89 (t, $J = 8$ Hz, 2H, CH_2), 3.74 (s, 3H, CH_3), 7.38 (dd, $J = 8, 8$ Hz, 1H, ArH), 7.49 (ddd, $J = 1, 7, 8$ Hz, 1H, ArH), 7.54 (ddd, $J = 1, 8, 9$ Hz, 1H, ArH), 7.60 (dd, $J = 1, 7$ Hz, 1H, ArH), 7.77 (d, $J = 8$ Hz, ArH), 7.82 (d, $J = 8$ Hz, 1H, ArH), 8.30 (d, $J = 9$ Hz, 1H, ArH); $^{13}\text{C}\{^1\text{H}\}$ NMR (125 MHz, CDCl_3) δ 15.7, 33.6, 51.8, 79.2, 92.9, 121.1, 125.1, 126.1, 126.2, 126.5, 128.1, 128.2, 130.1, 133.1, 133.4, 172.3; MS (EI) $m/z = 238$ ($[\text{M}^+]$).

Methyl 5-(2-thienyl)-4-pentynoate (S2l)



67% yield; Colorless oil; $^1\text{H NMR}$ (500 MHz, CDCl_3) δ 2.63 (t, $J = 8$ Hz, 2H, CH_2), 2.75 (t, $J = 8$ Hz, 2H, CH_2), 3.72 (s, 3H, CH_3), 6.92–6.94 (dd, $J = 4, 4$ Hz, 1H, ArH), 7.13 (d, $J = 4$ Hz, 1H, ArH), 7.18 (d, $J = 4$ Hz, 1H, ArH); $^{13}\text{C}\{^1\text{H}\}$ NMR (125 MHz, CDCl_3) δ 15.5, 33.1, 51.8, 74.3, 91.9, 123.5, 126.2, 126.7, 131.3, 172.4; IR (ATR) 3107, 2996, 2952, 2920, 2844, 1735 (C=O), 1519, 1436, 1362, 1290, 1256, 1239, 1192, 1165, 1081, 1038, 1019, 980, 942, 880, 865, 832, 779, 698, 656, 592, 572 cm^{-1} ; HR-EI-MS Calcd. for $\text{C}_{10}\text{H}_{10}\text{O}_2\text{S}$ $[\text{M}]^+$: $m/z = 194.0401$; Found 194.0400.

General procedure for hydrolysis of methyl esters

A typical procedure is given for the reaction of methyl 5-(4-trifluoromethylphenyl)-4-pentynoate (**S2a**). To a solution of methyl 5-(4-trifluoromethylphenyl)-4-pentynoate (1.84 g, 7.19 mmol) in a mixture of THF (80 mL) and H₂O (20 mL), lithium hydroxide monohydrate (603 mg, 14.4 mmol) was added. After being stirred at 25 °C for overnight, the mixture was washed with *tert*-butyl methyl ether (20 mL, 3 times) and acidified with 4N HCl aqueous solution. The organic layer was extracted with *tert*-butyl methyl ether (20 mL, 3 times), dried over Na₂SO₄, and concentrated in *vacuo* to give 5-(4-trifluoromethylphenyl)-4-pentynoic acid (**2a**, 1.64 g, 6.77 mmol, 94%) as white solids.

5-[(4-Trifluoromethyl)phenyl]pent-4-ynoic acid (**2a**)



94% yield; White solids; ¹H NMR (396 MHz, CDCl₃) δ 2.69–2.79 (m, 4H, CH₂), 7.47 (d, *J* = 8 Hz, 2H, ArH), 7.53 (d, *J* = 8 Hz, 2H, ArH); ¹³C{¹H} NMR (100 MHz, CDCl₃) δ 15.0, 33.2, 80.2, 90.3, 123.2 (q, *J*_{C-F} = 272 Hz₃), 125.1 (q, *J*_{C-F} = 4 Hz), 127.2, 129.6 (q, *J*_{C-F} = 32 Hz), 131.8, 178.1; MS (EI) *m/z* = 242 ([M⁺]).

5-Phenylpent-4-ynoic acid (**2b**)



91% yield; White solids; ¹H NMR (396 MHz, CDCl₃) δ 2.66–2.78 (m, 4H, CH₂), 7.25–7.30 (m, 3H, ArH), 7.36–7.42 (m, 2H, ArH); ¹³C{¹H} NMR (100 MHz, CDCl₃) δ 15.0, 33.4, 81.3, 87.5, 123.3, 127.9, 128.2, 131.6, 178.2; EI (70eV) *m/z* = 174 ([M⁺]).

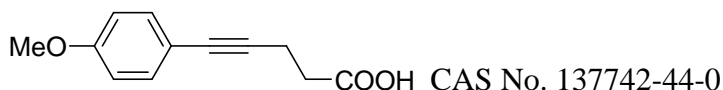
5-[(4-Methyl)phenyl]pent-4-ynoic acid (**2c**)



92% yield; White solids; **Mp.** 130.0–131.0 °C; ¹H NMR (396 MHz, CDCl₃) δ 2.33 (s, 3H, CH₃), 2.66–2.76 (m, 4H, CH₂), 7.08 (d, *J* = 8 Hz, 2H, ArH), 7.28 (d, *J* = 8 Hz, 2H, ArH); ¹³C{¹H} NMR (100 MHz, CDCl₃) δ 15.1, 21.4, 33.5, 81.4, 86.7, 120.2, 128.9, 131.4, 137.9, 178.2; IR (ATR) 3097, 3042, 3031, 2988, 2935, 2918, 2859, 2653, 2629, 2604, 2557, 2504, 1922, 1704 (C=O), 1508, 1424, 1407, 1357, 1303, 1261, 1211, 1175, 1106,

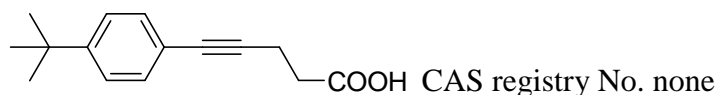
1045, 1021, 930, 822, 767, 692, 634 cm^{-1} ; **HR-EI-MS** Calcd. for $\text{C}_{12}\text{H}_{12}\text{O}_2$ $[\text{M}]^+$: $m/z = 188.08373$; Found 188.08348.

5-[(4-Methoxy)phenyl]pent-4-ynoic acid (2d)



92% yield; White solids; $^1\text{H NMR}$ (396 MHz, CDCl_3) δ 2.66–2.75 (m, 4H, CH_2), 3.80 (s, CH_3), 6.81 (d, $J = 9$ Hz, 2H, ArH), 7.32 (d, $J = 9$ Hz, 2H, ArH); $^{13}\text{C}\{^1\text{H}\}$ NMR (100 MHz, CDCl_3) δ 15.1, 33.5, 55.3, 81.1, 86.0, 113.8, 115.5, 133.0, 159.2, 178.1; **MS (EI)** $m/z = 204$ ($[\text{M}^+]$).

5-[(4-tert-Butyl)phenyl]pent-4-ynoic acid (2e)



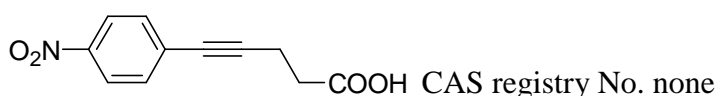
80% yield; White solids; **Mp.** 133.5–134.5 $^\circ\text{C}$; $^1\text{H NMR}$ (396 MHz, CDCl_3) δ 1.30 (s, 9H, $\text{C}(\text{CH}_3)_3$), 2.65–2.77 (m, 4H, CH_2), 7.27–7.36 (m, 4H, ArH); $^{13}\text{C}\{^1\text{H}\}$ NMR (100 MHz, CDCl_3) δ 15.1, 31.2, 33.4, 34.7, 81.3, 86.8, 120.3, 125.2, 131.2, 151.0, 177.9; **IR (ATR)** 3037, 2952, 2901, 2865, 2633, 2596, 2552, 2503, 1703 (C=O), 1505, 1462, 1428, 1409, 1362, 1299, 1267, 1259, 1213, 1109, 1019, 936, 837, 775, 754, 682, 562 cm^{-1} ; **HR-EI-MS** Calcd. for $\text{C}_{15}\text{H}_{18}\text{O}_2$ $[\text{M}]^+$: $m/z = 230.13068$; Found 230.13072.

5-[(4-Phenyl)phenyl]pent-4-ynoic acid (2f)



74% yield; Pale yellow solids; $^1\text{H NMR}$ (396 MHz, CDCl_3) δ 2.69–2.80 (m, 4H, CH_2), 7.32–7.37 (m, 1H, ArH), 7.40–7.48 (m, 4H, ArH), 7.50–7.54 (m, 2H, ArH), 7.55–7.59 (m, 2H, ArH); $^{13}\text{C}\{^1\text{H}\}$ NMR (100 MHz, CDCl_3) δ 15.2, 33.3, 81.2, 88.2, 122.3, 126.9, 127.0, 127.5, 128.76, 128.81, 132.0, 140.4, 140.6, 177.2; **EI (70eV)** $m/z = 250$ ($[\text{M}^+]$).

5-[(4-Nitro)phenyl]pent-4-ynoic acid (2g)



89% yield; Yellow solids; **Mp.** 157.0–158.0 $^\circ\text{C}$; **NMR (396 MHz, CDCl_3)** δ 2.70–2.75 (m, 2H, CH_2), 2.77–2.82 (m, 2H, CH_2), 7.52 (d, $J = 9$ Hz, 1H, ArH), 8.15 (d, $J = 9$ Hz, 1H,

ArH); $^{13}\text{C}\{^1\text{H}\}$ NMR (100 MHz, CDCl_3) δ 15.1, 32.9, 79.9, 93.5, 123.5, 130.3, 132.4, 146.8, 177.3; IR (ATR) 3110, 3086, 3028, 2978, 2930, 2845, 2808, 2792, 2710, 2647, 2614, 2560, 2511, 2465, 2445, 1694 (C=O), 1592, 1510 (NO_2), 1432, 1407, 1339, 1317, 1305, 1286, 1275, 1261, 1212, 1172, 1103, 1096, 1011, 980, 922, 854, 776, 750, 689, 677 cm^{-1} ; HR-EI-MS Calcd. for $\text{C}_{11}\text{H}_9\text{NO}_4$ $[\text{M}]^+$: m/z = 219.05316; Found 219.05341.

5-[(4-Fluoro)phenyl]pent-4-ynoic acid (2h)



88% yield; White solids; **Mp** 112.5–113.5 °C; ^1H NMR (396 MHz, CDCl_3) δ 2.65–2.76 (m, 4H, CH_2), 6.94–7.00 (m, 2H, ArH), 7.33–7.39 (m, 2H, ArH); $^{13}\text{C}\{^1\text{H}\}$ NMR (100 MHz, CDCl_3) δ 15.0, 33.4, 80.3, 87.2, 115.4 (d, $J_{\text{C-F}}$ = 22 Hz), 119.4 (d, $J_{\text{C-F}}$ = 4 Hz), 133.4 (d, $J_{\text{C-F}}$ = 9 Hz), 162.2 (d, $J_{\text{C-F}}$ = 249 Hz), 178.4; IR (ATR) 3102, 3064, 3045, 3033, 2979, 2964, 2936, 2919, 2852, 2710, 2632, 2560, 2512, 1703 (C=O), 1597, 1504, 1432, 1409, 1360, 1308, 1259, 1214, 1161, 1095, 1014, 979, 918, 841, 829, 814, 776, 699, 634 cm^{-1} ; HR-EI-MS Calcd. for $\text{C}_{11}\text{H}_9\text{FO}_2$ $[\text{M}]^+$: m/z = 192.05866; Found 192.05871.

5-[(4-Chloro)phenyl]pent-4-ynoic acid (2i)



93% yield; White solids; ^1H NMR (396 MHz, CDCl_3) δ 2.65–2.76 (m, 4H, CH_2), 7.24 (d, J = 8 Hz, 2H, ArH), 7.30 (d, J = 8 Hz, 1H, ArH); $^{13}\text{C}\{^1\text{H}\}$ NMR (100 MHz, CDCl_3) δ 15.0, 33.3, 80.3, 88.6, 121.8, 128.5, 132.8, 133.8, 178.4; EI (70eV) m/z = 208 ($[\text{M}]^+$).

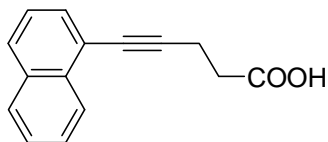
5-[(2-Methyl)phenyl]pent-4-ynoic acid (2j)



100% yield; White solids; **Mp** 61.0–62.0 °C; ^1H NMR (500 MHz, CDCl_3) δ 2.38 (s, 3H, CH_3), 2.69–2.73 (m, 2H, CH_2), 2.77–2.81 (m, 2H, CH_2), 7.07–7.12 (m, 1H, ArH), 7.14–7.19 (m, 2H, ArH), 7.34–7.36 (m, 1H, ArH); $^{13}\text{C}\{^1\text{H}\}$ NMR (125 MHz, CDCl_3) δ 15.3, 20.6, 33.7, 80.3, 91.4, 123.1, 125.4, 127.8, 129.3, 131.8, 140.1, 178.1; IR (ATR) 3021, 2967, 2921, 2852, 2735, 2701, 2635, 2551, 2503, 1693 (C=O), 1487, 1432, 1411, 1351, 1302, 1289, 1253, 1212, 1176, 1161, 1114, 1044, 1017, 978, 918, 862, 803, 777, 748, 715,

664 cm⁻¹; **HR-EI-MS** Calcd. for C₁₂H₁₂O₂ [M]⁺: *m/z* = 188.08373; Found 188.08375.

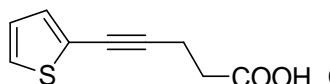
5-(1-Naphthyl)pent-4-ynoic acid (2k)



CAS registry No. 928768-34-7

91% yield; White solids; ¹H NMR (396 MHz, CDCl₃) δ 2.78–2.84 (m, 2H, CH₂), 2.88–2.93 (m, 2H, CH₂), 7.39 (t, *J* = 8 Hz, 1H, ArH), 7.49 (td, *J* = 1, 7 Hz, 1H, ArH), 7.54 (td, *J* = 1, 7 Hz, 1H, ArH), 7.61 (d, *J* = 7 Hz, 1H, ArH), 7.78 (d, *J* = 8 Hz, ArH), 7.82 (d, *J* = 8 Hz, 1H, ArH), 8.32 (d, *J* = 8 Hz, 1H, ArH); ¹³C{¹H} NMR (100 MHz, CDCl₃) δ 15.4, 33.6, 79.5, 92.5, 121.0, 125.2, 126.1, 126.3, 126.6, 128.2, 128.3, 130.2, 133.1, 133.4, 178.1; EI (70eV) *m/z* = 224 ([M⁺]).

5-(2-Thienyl)pent-4-ynoic acid (2l)



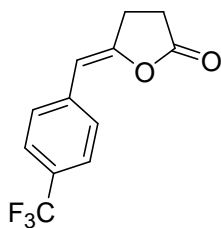
CAS No. 928768-35-8

86% yield; Pale brown solids; ¹H NMR (500 MHz, CDCl₃) δ 2.68–2.72 (m, 2H, CH₂), 2.74–2.78 (m, 2H, CH₂), 6.93–6.95 (m, 1H, ArH), 7.13–7.14 (m, 1H, ArH), 7.18–7.20 (m, 1H, ArH); ¹³C{¹H} NMR (125 MHz, CDCl₃) δ 15.3, 33.1, 74.6, 91.5, 123.4, 126.4, 126.8, 131.5, 177.9; EI (70eV) *m/z* = 180 ([M⁺]).

General procedure for cyclization of alkynoic acids

A typical procedure is given for the reaction with 5-(4-trifluoromethylphenyl)pentynoic acid (**2a**) in the presence of **1a_{vscI}** and Et₃N in water (Table 1, entry 2). 5-(4-Trifluoromethylphenyl)pentynoic acid (**2a**, 29.1 mg, 0.12 mmol) was placed in a vial, to which was added 1 mL aqueous suspension of **1a_{vscI}** (2.6 mg, 2.4 × 10⁻³ mmol) and Et₃N (1 mL, 7.2 × 10⁻³ mmol). The reaction mixture was agitated by shaking at 50 °C for 1 h, and then extracted with t-butyl methyl ether (MTBE) (1.5 mL, 4 times). The combined extract was dried over Na₂SO₄ and concentrated in *vacuo*. The residue was chromatographed on silica gel (eluent: CHCl₃) to give **3a** (17.6 mg, 0.727 mmol, 61%).

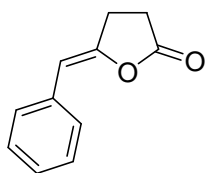
(Z)- γ -(4-Trifluoromethylbenzylidene)- γ -butyrolactone (3a)



CAS registry No. none

White solids; **Mp** 56.5–57.0 °C; **$^1\text{H NMR}$ (396 MHz, CDCl_3)** δ 2.73–2.78 (m, 2H, CH_2), 3.05–3.11 (m, 2H, CH_2), 5.58–5.60 (m, 1H, $\text{C}=\text{CH}$), 7.56 (d, $J = 8$ Hz, 2H, ArH), 7.64 (d, $J = 8$ Hz, 2H, ArH); **$^{13}\text{C}\{^1\text{H}\}$ NMR (100 MHz, CDCl_3)** δ 26.4, 26.7, 103.7, 124.2 (q, $J_{\text{C-F}} = 272$ Hz), 125.3 (q, $J_{\text{C-F}} = 4$ Hz), 128.3 (q, $J_{\text{C-F}} = 32$ Hz), 128.4, 137.5, 150.2, 174.5; **IR (ATR)** 2956, 2936, 1804 (C=O), 1680, 1614, 1443, 1415, 1323 (CF_3), 1290, 1227, 1165, 1093, 1064, 1015, 939, 853, 815, 755, 708, 652, 597 cm^{-1} ; **HR-EI-MS** Calcd. for $\text{C}_{12}\text{H}_9\text{F}_3\text{O}_2$ $[\text{M}]^+$: $m/z = 242.05546$; Found 242.05518.

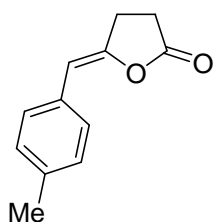
(Z)- γ -Benzylidene- γ -butyrolactone (3b)



CAS registry No. 69063-20-3

White solids; **$^1\text{H NMR}$ (396 MHz, CDCl_3)** δ 2.69–2.74 (m, 2H, CH_2), 3.01–3.07 (m, 2H, CH_2), 5.54–5.56 (m, 1H, $\text{C}=\text{CH}$), 7.21 (t, $J = 7$ Hz, 1H, ArH), 7.33 (t, $J = 7$ Hz, 2H, ArH), 7.54 (d, $J = 7$ Hz, 2H, ArH); **$^{13}\text{C}\{^1\text{H}\}$ NMR (100 MHz, CDCl_3)** δ 26.3, 26.9, 104.9, 126.7, 128.3, 128.4, 133.8, 148.1, 174.9; **EIMS (70eV)** $m/z = 174$ ($[\text{M}]^+$).

(Z)- γ -(4-Methylbenzylidene)- γ -butyrolactone (3c)

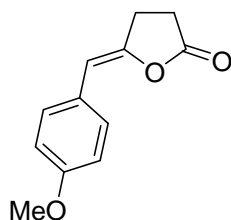


CAS registry No. none

White solids; **Mp** 85.0–86.0 °C; **$^1\text{H NMR}$ (396 MHz, CDCl_3)** δ 2.34 (s, 3H, CH_3), 2.68–2.73 (m, 2H, CH_2), 3.00–3.06 (m, 2H, CH_2), 5.51–5.53 (m, 1H, $\text{C}=\text{CH}$), 7.13 (d, $J = 8$ Hz, 2H, ArH), 7.44 (d, $J = 8$ Hz, 2H, ArH); **$^{13}\text{C}\{^1\text{H}\}$ NMR (100 MHz, CDCl_3)** δ 21.1, 26.2, 27.0, 104.7, 128.1, 129.1, 131.0, 136.4, 147.3, 175.1; **IR (ATR)** 3024, 2982, 2923, 2872, 1792 (C=O), 1682, 1609, 1508, 1444, 1413, 1355, 1290, 1227, 1208, 1176, 1102,

1023, 1011, 992, 942, 857, 841, 803, 758, 714, 702, 656, 634, 563 cm^{-1} ; **HR-EI-MS** Calcd. for $\text{C}_{12}\text{H}_{12}\text{O}_2$ $[\text{M}]^+$: $m/z = 188.08373$; Found 188.08367.

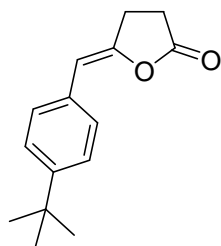
(Z)- γ -(4-Methoxybenzylidene)- γ -butyrolactone (3d)



CAS registry No. none

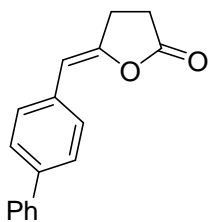
Pale yellow oil; $^1\text{H NMR}$ (396 MHz, CDCl_3) δ 2.68–2.73 (m, 2H, CH_2), 2.99–3.05 (m, 2H, CH_2), 3.81 (s, OCH_3), 5.48–5.51 (m, 1H, $\text{C}=\text{CH}$), 6.86 (d, $J = 9$ Hz, 2H, ArH), 7.49 (d, $J = 9$ Hz, 2H, ArH); $^{13}\text{C}\{^1\text{H}\}$ NMR (100 MHz, CDCl_3) δ 26.2, 27.0, 55.2, 104.3, 113.80, 126.6, 129.5, 146.4, 158.2, 175.1; **IR (ATR)** 3003, 2958, 2934, 2838, 1792 ($\text{C}=\text{O}$), 1732, 1685, 1606, 1577, 1509, 1463, 1443, 1419, 1350, 1294, 1248, 1175, 1114, 1096, 1029, 941, 906, 848, 830, 815, 756, 722, 704, 657, 628, 597, 554 cm^{-1} ; **HR-EI-MS** Calcd. for $\text{C}_{12}\text{H}_{12}\text{O}_3$ $[\text{M}]^+$: $m/z = 204.07864$ Found 204.07866.

(Z)- γ -(4-*tert*-Butylbenzylidene)- γ -butyrolactone (3e)



White solids; **Mp.** 34.0–35.0 $^\circ\text{C}$; $^1\text{H NMR}$ (396 MHz, CDCl_3) δ 1.31 (s, 9H, $\text{C}(\text{CH}_3)_3$), 2.68–2.73 (m, 2H, CH_2), 3.00–3.05 (m, 2H, CH_2), 5.53–5.54 (m, 1H, $\text{C}=\text{CH}$), 7.35 (d, $J = 9$ Hz, 2H, ArH), 7.48 (d, $J = 9$ Hz, 2H, ArH); $^{13}\text{C}\{^1\text{H}\}$ NMR (100 MHz, CDCl_3) δ 26.2, 27.1, 31.2, 34.5, 104.7, 125.3, 128.0, 131.0, 147.5, 149.7, 175.0; **IR (ATR)** 2959, 2928, 2868, 1799 ($\text{C}=\text{O}$), 1726, 1685, 1511, 1475, 1462, 1445, 1415, 1395, 1362, 1293, 1270, 1229, 1201, 1173, 1123, 1096, 1018, 941, 851, 836, 807, 705, 656, 575 cm^{-1} ; **HR-EI-MS** Calcd. for $\text{C}_{15}\text{H}_{18}\text{O}_2$ $[\text{M}]^+$: $m/z = 230.13068$; Found 230.13070.

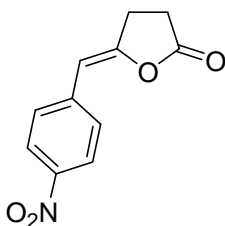
(Z)- γ -(4-Phenylbenzylidene)- γ -butyrolactone (3f)



CAS registry No. none

Light brown solids; **Mp** 116.5–117.5 °C; $^1\text{H NMR}$ (396 MHz, CDCl_3) δ 2.71–2.76 (m, 2H, CH_2), 3.04–3.10 (m, 2H, CH_2), 5.59–5.60 (m, 1H, $\text{C}=\text{CH}$), 7.33–7.36 (m, 1H, ArH), 7.41–7.44 (m, 2H, ArH), 7.56–7.65 (m, 6H, ArH); $^{13}\text{C}\{^1\text{H}\}$ NMR (100 MHz, CDCl_3) δ 26.3, 26.9, 104.5, 126.9, 127.1, 127.2, 128.7, 128.7, 128.8, 132.9, 139.3, 140.7, 148.3, 174.9; **IR (ATR)** 3031, 2932, 1793 ($\text{C}=\text{O}$), 1678, 1604, 1593, 1554, 1486, 1449, 1414, 1294, 1234, 1218, 1208, 1181, 1097, 1103, 940, 913, 876, 852, 838, 809, 762, 723, 688, 659, 607, 570 cm^{-1} ; **HR-EI-MS** Calcd. for $\text{C}_{17}\text{H}_{14}\text{O}_2$ $[\text{M}]^+$: m/z = 250.09938 Found 250.09936.

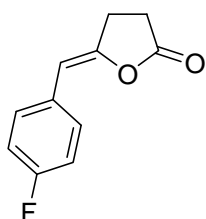
(Z)- γ -(4-Nitrobenzylidene)- γ -butyrolactone (3g)



CAS registry No. none

Yellow solids; **Mp.** 120.0–121.0 °C; $^1\text{H NMR}$ (396 MHz, CDCl_3) δ 2.75–2.80 (m, 2H, CH_2), 3.08–3.14 (m, 2H, CH_2), 5.63–5.64 (m, 1H, $\text{C}=\text{CH}$), 7.68 (d, J = 9 Hz, 2H, ArH), 8.17 (d, J = 9 Hz, 2H, ArH); $^{13}\text{C}\{^1\text{H}\}$ NMR (100 MHz, CDCl_3) δ 26.5, 26.6, 103.2, 123.8, 128.6, 140.6, 145.9, 151.9, 174.0; **IR (ATR)** 3361, 2955, 2923, 2852, 1821 ($\text{C}=\text{O}$), 1666, 1587, 1504, 1469, 1437, 1414, 1335, 1289, 1224, 1180, 1085, 1009, 933, 863, 820, 801, 751, 705, 691, 654, 637, 626, 559 cm^{-1} ; **HR-EI-MS** Calcd. for $\text{C}_{11}\text{H}_9\text{NO}_4$ $[\text{M}]^+$: m/z = 219.05316 Found 219.05319.

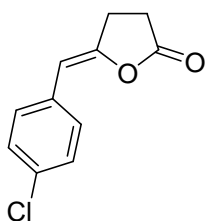
(Z)- γ -(4-Fluorobenzylidene)- γ -butyrolactone (3h)



CAS registry No. none

White solids; **Mp** 78.0–79.0 °C; **¹H NMR (396 MHz, CDCl₃)** δ 2.70–2.74 (m, 2H, CH₂), 3.00–3.06 (m, 2H, CH₂), 5.51–5.53 (m, 1H, C=CH), 6.98–7.04 (m, 2H, ArH), 7.50–7.54 (m, 2H, ArH); **¹³C{¹H} NMR (100 MHz, CDCl₃)** δ 26.2, 26.9, 103.7, 115.3 (d, *J*_{C-F} = 22 Hz), 129.8 (d, *J*_{C-F} = 8 Hz), 130.0 (d, *J*_{C-F} = 4 Hz), 147.7 (d, *J*_{C-F} = 3 Hz), 161.4 (d, *J*_{C-F} = 247 Hz), 174.8; **IR (ATR)** 3006, 2936, 2852, 1788 (C=O), 1687, 1601, 1504, 1443, 1416, 1352, 1297, 1226, 1173, 1159, 1101, 1091, 1013, 1002, 935, 855, 833, 818, 769, 706, 658, 626, 562 cm⁻¹; **HR-EI-MS** Calcd. for C₁₁H₉FO₂ [M]⁺: *m/z* = 192.05866; Found 192.05866.

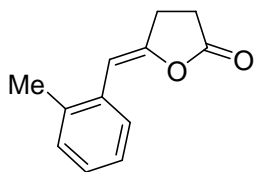
(Z)-γ-(4-Chlorobenzylidene)-γ-butyrolactone (3i)



CAS registry No. none

White solids; **Mp** 81.5–82.5 °C; **¹H NMR (396 MHz, CDCl₃)** δ 2.70–2.75 (m, 2H, CH₂), 3.01–3.07 (m, 2H, CH₂), 5.50–5.52 (m, 1H, C=CH), 7.28 (d, *J* = 8 Hz, 2H, ArH), 7.48 (d, *J* = 8 Hz, 2H, ArH); **¹³C{¹H} NMR (100 MHz, CDCl₃)** δ 26.3, 26.8, 103.8, 128.6, 129.5, 132.2, 132.4, 148.6, 174.7; **IR (ATR)** 2956, 2925, 2851, 1793 (C=O), 1680, 1657, 1486, 1438, 1411, 1353, 1294, 1227, 1176, 1109, 1096, 1010, 944, 851, 833, 804, 714, 708, 679, 653, 625, 559 cm⁻¹; **HR-EI-MS** Calcd. for C₁₁H₉ClO₂ [M]⁺: *m/z* = 208.02911; Found 208.02920.

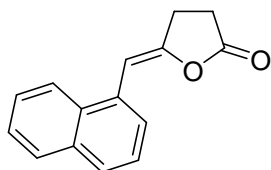
(Z)-γ-(2-Methylbenzylidene)-γ-butyrolactone (3j)



CAS registry No. none

White solids; **Mp** 90.0–91.0 °C; **¹H NMR (396 MHz, CDCl₃)** δ 2.32 (s, 3H, CH₃), 2.70–2.75 (m, 2H, CH₂), 3.04–3.10 (m, 2H, CH₂), 5.68–5.70 (m, 1H, C=CH), 7.11–7.22 (m, 3H, ArH), 7.76 (d, *J* = 8 Hz, 2H, ArH); **¹³C{¹H} NMR (100 MHz, CDCl₃)** δ 20.2, 26.3, 27.1, 102.1, 126.0, 126.8, 129.0, 129.9, 132.3, 135.0, 148.2, 175.0; **IR (ATR)** 3069, 2956, 2930, 2862, 1793 (C=O), 1679, 1597, 1489, 1480, 1461, 1437, 1409, 1377, 1362, 1292, 1226, 1202, 1174, 1151, 1110, 1096, 1053, 1038, 1002, 945, 836, 769, 718, 650, 610, 568 cm⁻¹; **HR-EI-MS** Calcd. for C₁₂H₁₂O₂ [M]⁺: *m/z* = 188.08373; Found 188.08370.

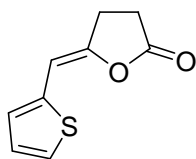
(Z)- γ -(1-Naphthalenylmethylene)- γ -butyrolactone (3k)



CAS registry No. none

White solids; **Mp** 91.0–92.0 °C; **^1H NMR (396 MHz, CDCl_3)** δ 2.76–2.81 (m, 2H, CH_2), 3.16–3.21 (m, 2H, CH_2), 6.24–6.25 (m, 1H, $\text{C}=\text{CH}$), 7.46–7.54 (m, 3H, ArH), 7.75 (d, $J = 8$ Hz, 1H, ArH), 7.85 (dd, $J = 3, 7$ Hz, 1H, ArH), 7.89 (d, $J = 7$ Hz, 1H, ArH), 8.03 (dd, $J = 2, 8$ Hz, 1H, ArH); **$^{13}\text{C}\{^1\text{H}\}$ NMR (100 MHz, CDCl_3)** δ 26.3, 27.2, 101.1, 123.6, 125.5, 125.6, 125.9, 127.1, 127.3, 128.7, 129.8, 131.0, 133.6, 149.4, 174.9; **IR (ATR)** 3048, 2929, 2868, 1789 (C=O), 1686, 1589, 1579, 1506, 1443, 1414, 1397, 1353, 1293, 1222, 1194, 1166, 1105, 1038, 1017, 959, 941, 910, 866, 832, 801, 775, 748, 740, 693, 650, 640, 612 cm^{-1} ; **HR-EI-MS** Calcd. for $\text{C}_{15}\text{H}_{12}\text{O}_2$ $[\text{M}]^+$: $m/z = 224.08373$; Found 224.08369.

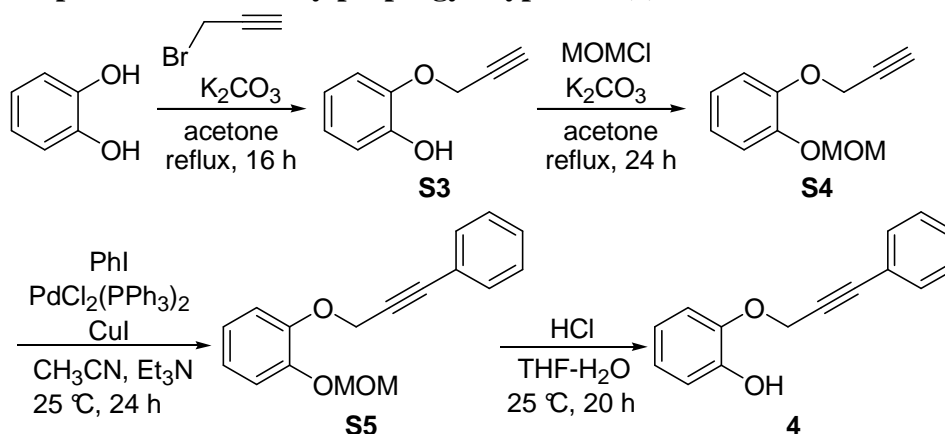
(Z)- γ -(2-Thienylmethylene)- γ -butyrolactone (3l)



CAS registry No. none

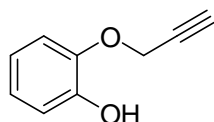
White solids; **Mp** 96.5–97.5 °C; **^1H NMR (396 MHz, CDCl_3)** δ 2.73–2.78 (m, 2H, CH_2), 3.00–3.05 (m, 2H, CH_2), 5.84–5.86 (m, 1H, $\text{C}=\text{CH}$), 6.99 (dd, $J = 4, 5$ Hz, 1H, ArH), 7.08 (d, $J = 4$ Hz, 1H, ArH), 7.24 (d, $J = 5$ Hz, 1H, ArH); **$^{13}\text{C}\{^1\text{H}\}$ NMR (100 MHz, CDCl_3)** δ 25.5, 27.3, 99.0, 125.1, 125.7, 126.8, 136.5, 146.6, 174.2; **IR (ATR)** 3093, 3069, 2961, 2928, 2852, 1786 (C=O), 1683, 1610, 1442, 1425, 1364, 1297, 1242, 1202, 1167, 1142, 1091, 1045, 1023, 939, 903, 846, 824, 819, 806, 780, 752, 698, 673, 653, 611, 588 cm^{-1} ; **HR-EI-MS** Calcd. for $\text{C}_9\text{H}_8\text{O}_2\text{S}$ $[\text{M}]^+$: $m/z = 180.02450$; Found 180.02446.

Preparation of 2-Propargyloxyphenol (4)



Scheme S2. Preparation of 2-phenylpropargyloxyphenol (4).

2-Propargyloxyphenol (S3)

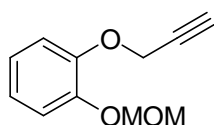


CAS registry No. 14817-38-0

Under a nitrogen atmosphere, a mixture of pyrocatechol (2.00 g, 18.2 mmol) and K_2CO_3 (1.38 g, 9.99 mmol) in anhydrous acetone (90 mL) was stirred for 1 h at 25 °C. Propargyl bromide (2.16 g, 18.2 mmol) in dry acetone (1.5 mL) was slowly added at 25 °C during 1 h. The mixture was refluxed at 65 °C for 16 h. After being cooled to 25 °C, acetone was evaporated from the mixture. To the resulting residue was added water (50 mL). The suspension was extracted with CHCl_3 (15 mL, 4 times). The combined organic layer was dried over Na_2SO_4 and concentrated in vacuo. The resulting crude material was chromatographed on silica gel (eluent: 5-20% $\text{AcOEt}/\text{n-hexane}$) to give 2-propargyloxyphenol (1.90 g, 12.8 mmol, 71%) as white solids.

^1H NMR (396 MHz) δ 2.56 (t, $J = 2.3$ Hz, 1H, $\text{HC}\equiv\text{C}-$), 4.75 (d, $J = 2.3$ Hz, 2H, $\text{CH}\equiv\text{C}-\text{CH}_2-$), 5.64 (s, 1H, $-\text{OH}$), 6.83–6.88 (m, 1H, ArH), 6.90–7.00 (m, 3H, ArH); $^{13}\text{C}\{^1\text{H}\}$ NMR (100 MHz) δ 56.9, 76.1, 78.1, 112.8, 115.2, 120.1, 122.6, 144.6, 146.0; MS (EI) $m/z = 148$ ($[\text{M}^+]$).

1-Methoxymethoxy-2-propargyloxybenzene (S4)

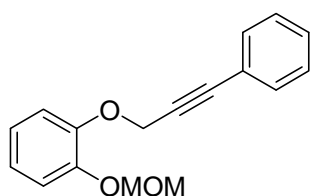


CAS registry No. none

Under a nitrogen atmosphere, to a mixture of 2-propargyloxyphenol (700 mg, 4.72 mmol)

and K_2CO_3 (979 mg, 7.09 mmol) was added acetone (20 mL) at 25 °C. After being stirred at 25 °C for 1 h, methoxymethyl chloride (0.53 mL, 7.09 mmol) was slowly added to the resulting suspension at 25 °C. The reaction mixture stirred at 65 °C for 24 h. After being cooled to 25 °C, saturated NH_4Cl aqueous solution (15 mL) was added to the reaction mixture and then the resulting mixture was evaporated. The solution was extracted with MTBE (15 mL, 3 times). The combined organic layer was dried over Na_2SO_4 , and concentrated in vacuo. The resulting crude mixture was chromatographed on silica gel (eluent: 5-10% AcOEt/n-hexane) to give 1-methoxymethoxy-2-propargyloxybenzene (720 mg, 3.75 mmol, 79%) as pale yellow oil. 1H NMR (396 MHz, $CDCl_3$) δ 2.51 (t, $J = 2$ Hz, 1H, CCH), 3.52 (d, $J = 0.9$ Hz, 3H, OCH_2OCH_3), 4.77 (d, $J = 2$ Hz, 2H, CH_2CCH), 5.22 (d, $J = 0.9$ Hz, 2H, OCH_2OCH_3), 6.93–7.02 (m, 2H, ArH), 7.07 (dd, $J = 2, 8$ Hz, 1H, ArH), 7.17 (dd, $J = 2, 8$ Hz, 1H, ArH); $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$) δ 56.2, 56.7, 75.7, 78.6, 95.5, 114.7, 117.0, 122.3, 122.4, 147.2, 147.6; IR (ATR) 3284 (CCH), 3071, 2993, 2956, 2934, 2827, 2120 (CC), 1594, 1499, 1456, 1374, 1324, 1302, 1242 (Ar-O- CH_2), 1216, 1188, 1153, 1119, 1078, 994, 922, 835, 745, 682, 671, 646, 639 cm^{-1} ; HR-EI-MS Calcd. for $C_{11}H_{12}O_3$ $[M]^+$: $m/z = 192.07864$; Found 192.07883.

1-Methoxymethoxy-2-phenylpropargyloxybenzene (S5)

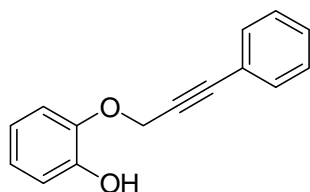


CAS registry No. none

Under a nitrogen atmosphere, to a suspension of $PdCl_2(PPh_3)_2$ (64 mg, 0.091 mmol) and copper iodide (35 mg, 0.182 mmol) in triethylamine (2 mL), 1-methoxymethoxy-2-propargyloxybenzene (350 mg, 1.82 mmol) in CH_3CN (2 mL) and iodobenzene (0.22 mL, 2.00 mmol) were added at 25 °C. After being stirred at 25 °C for 24 h, saturated NH_4Cl aqueous solution (20 mL) was added to the reaction mixture and the resulting solution was extracted with AcOEt (20 mL, 3 times). The combined organic layer was dried over Na_2SO_4 . After evaporation of the solvent, the resulting mixture was chromatographed on silica gel to give 1-methoxymethoxy-2-phenylpropargyloxybenzene (255.6 mg, 0.953 mmol, 52%) as pale yellow oil.

^1H NMR (396 MHz, CDCl_3) δ 3.53 (s, 3H, OCH_3), 4.98 (s, 2H, CH_2), 5.24 (s, 2H, OCH_2OCH_3), 6.93–7.02 (m, 2H, ArH), 7.13–7.20 (m, 2H, ArH), 7.28–7.32 (m, 3H, ArH), 7.39–7.43 (m, 2H, ArH); $^{13}\text{C}\{^1\text{H}\}$ NMR (100 MHz, CDCl_3) δ 56.2, 56.6, 84.0, 87.3, 95.6, 114.9, 117.0, 122.1, 122.3, 122.4, 128.2, 128.6, 131.7, 147.2, 147.9; IR (ATR) 3063, 2993, 2953, 2930, 2898, 2848, 2825, 2237 (CC), 1592, 1498, 1490, 1455, 1441, 1403, 1371, 1322, 1300, 1240 (Ar-O- CH_2), 1214, 1187, 1152, 1118, 1076, 1051, 1030, 991, 920, 837, 817, 742, 690, 648, 607 cm^{-1} ; HR-EI-MS Calcd. for $\text{C}_{17}\text{H}_{16}\text{O}_3$ $[\text{M}]^+$: m/z = 268.10994; Found 268.10991.

2-[(3-phenylprop-2-yn-1-yl)oxy]phenol (**4**)



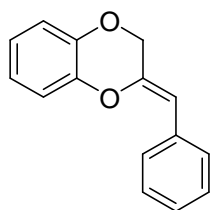
CAS registry No. 73222-90-9

HCl aqueous solution (4 mol/L, 6 mL) was added to a solution of 1-methoxymethoxy-2-phenylpropargyloxybenzene (225 mg, 0.839 mmol) in THF (6 mL) at 25 °C and the reaction mixture was stirred for 20 h at 25 °C. The resulting solution was extracted with *t*-butyl methyl ether (10 mL, 3 times). The combined organic layer was dried over Na_2SO_4 . The organic layer was concentrated under reduced pressure to give 2-phenylpropargyloxyphenol (175.5 mg, 0.783 mmol, 93%) as brown oil.

^1H NMR (396 MHz, CDCl_3) δ 4.97 (s, 2H, CH_2), 5.70 (s, 1H, OH), 6.84–6.99 (m, 3H, ArH), 7.04–7.08 (m, 1H, ArH), 7.29–7.35 (m, 3H, ArH), 7.42–7.45 (m, 2H, ArH); $^{13}\text{C}\{^1\text{H}\}$ NMR (100 MHz, CDCl_3) δ 57.9, 83.3, 87.7, 113.0, 115.1, 120.1, 121.9, 122.5, 128.3, 128.8, 131.8, 144.9, 146.1; MS (EI) m/z = 224 ($[\text{M}]^+$).

Cyclization reaction of 2-phenylpropargyloxyphenol (**4**) (Scheme 1)

2,3-Dihydro-2-(Z)-phenylmethylene-1,4-benzodioxin (**5**)



CAS registry No. 73249-12-4

2-Phenylpropargyloxyphenol (**4**, 13.5 mg, 0.060 mmol) was placed in a vial, to which was

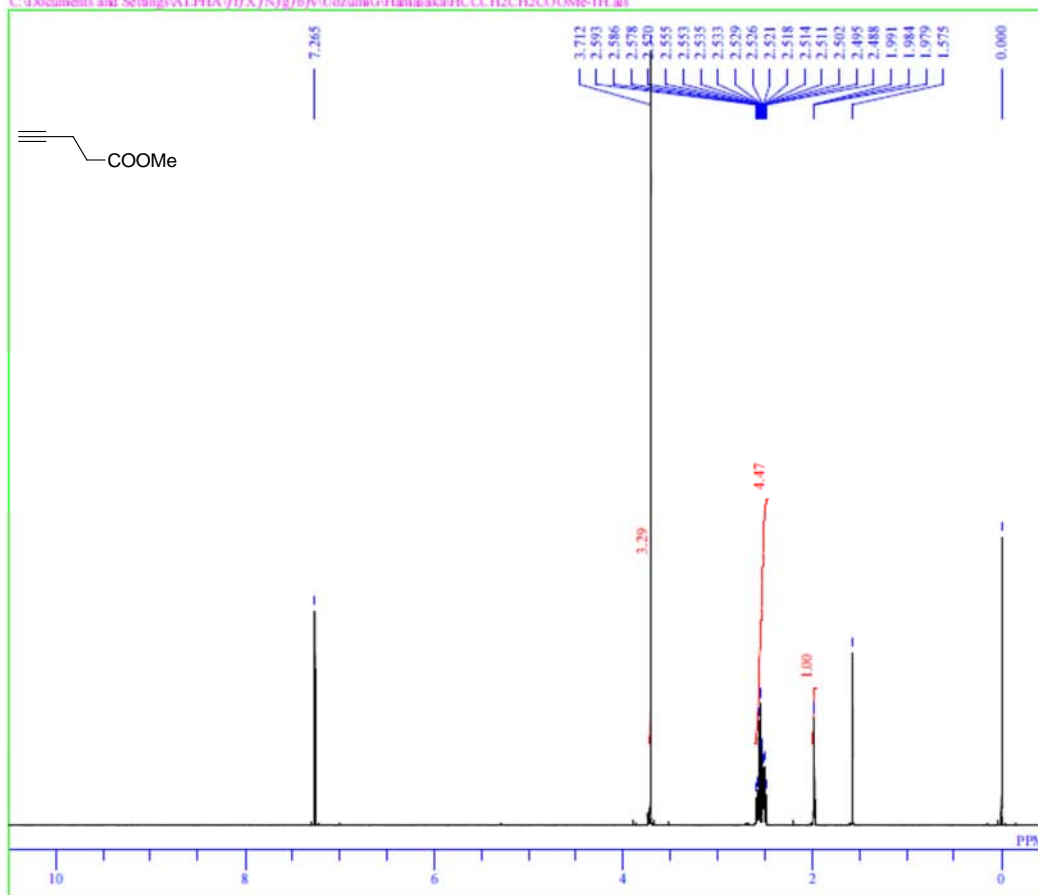
added 0.5 mL aqueous suspension of **1a_{vsc1}** (1.3 mg, 1.2×10^{-3} mmol) and Et₃N (0.5 μ L, 3.6×10^{-3} mmol). The reaction mixture was agitated by shaking at 50 °C for 4 h, and then extracted with *t*-butyl methyl ether (1.5 mL, 4 times). The combined extract was dried over Na₂SO₄ and concentrated in *vacuo*. The residue was chromatographed on silica gel (eluent: 5-15% AcOEt/*n*-hexane) to give **5** (5.6 mg, 0.025 mmol, 42%) as colorless oil.

¹H NMR (396 MHz, CDCl₃) δ 4.62 (s, 2H, CH₂), 5.59 (s, 1H, C=CH), 6.93–6.99 (m, 3H, ArH), 7.12–7.15 (m, 1H, ArH), 7.21–7.25 (m, 1H, ArH), 7.34–7.38 (m, 2H, ArH), 7.68–7.71 (m, 2H, ArH); **¹³C{¹H} NMR (100 MHz, CDCl₃)** δ 66.1, 107.0, 116.8, 117.4, 122.3, 122.6, 126.9, 128.4, 128.9, 134.2, 142.5, 143.4, 144.1; **MS (EI)** m/z = 224 ([M⁺]).

¹H and ¹³C NMR spectra
Methyl-4-pentynoate (S1)

HCCCH₂CH₂COOMe-1H

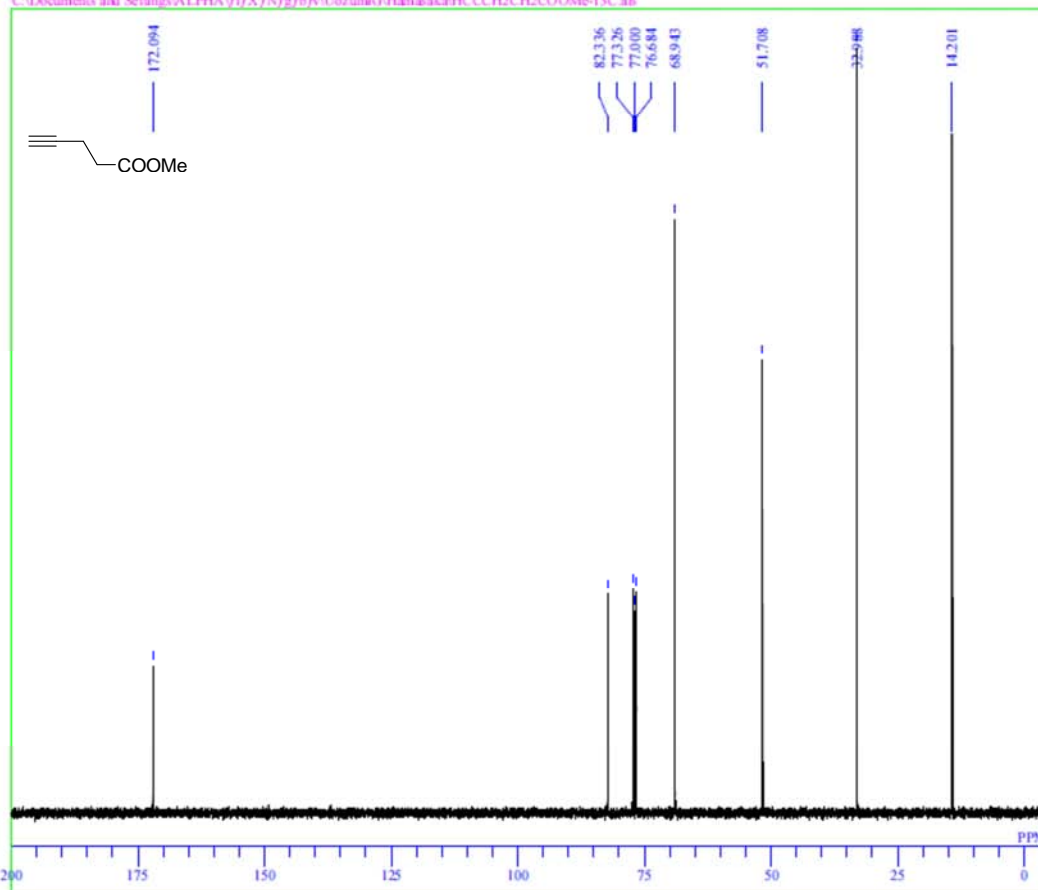
C:\Documents and Settings\ALPHA\I\X\N/g/b/A/UozumiG\Hanaoka\HCCCH₂CH₂COOMe-1H.als



DFILE HCCCH₂CH₂COOMe-1H.als
COMNT HCCCH₂CH₂COOMe-1H
DATIM 2013-07-04 22:36:31
OBNUC 1H
EXMOD proton.jsp
OBFREQ 395.88 MHz
OBSET 6.28 KHz
OBFIN 0.87 Hz
POINT 16384
FREQU 7422.80 Hz
SCANS 8
ACQTM 2.2073 sec
PD 5.0000 sec
PWI 3.12 usec
IRNUC 1H
CTEMP 18.9 c
SLVNT CDCL₃
EXREF 0.00 ppm
BF 0.00 Hz
RGAIN 42

HCCCH₂CH₂COOMe-13C

C:\Documents and Settings\ALPHA\I\X\N/g/b/A/UozumiG\Hanaoka\HCCCH₂CH₂COOMe-13C.als

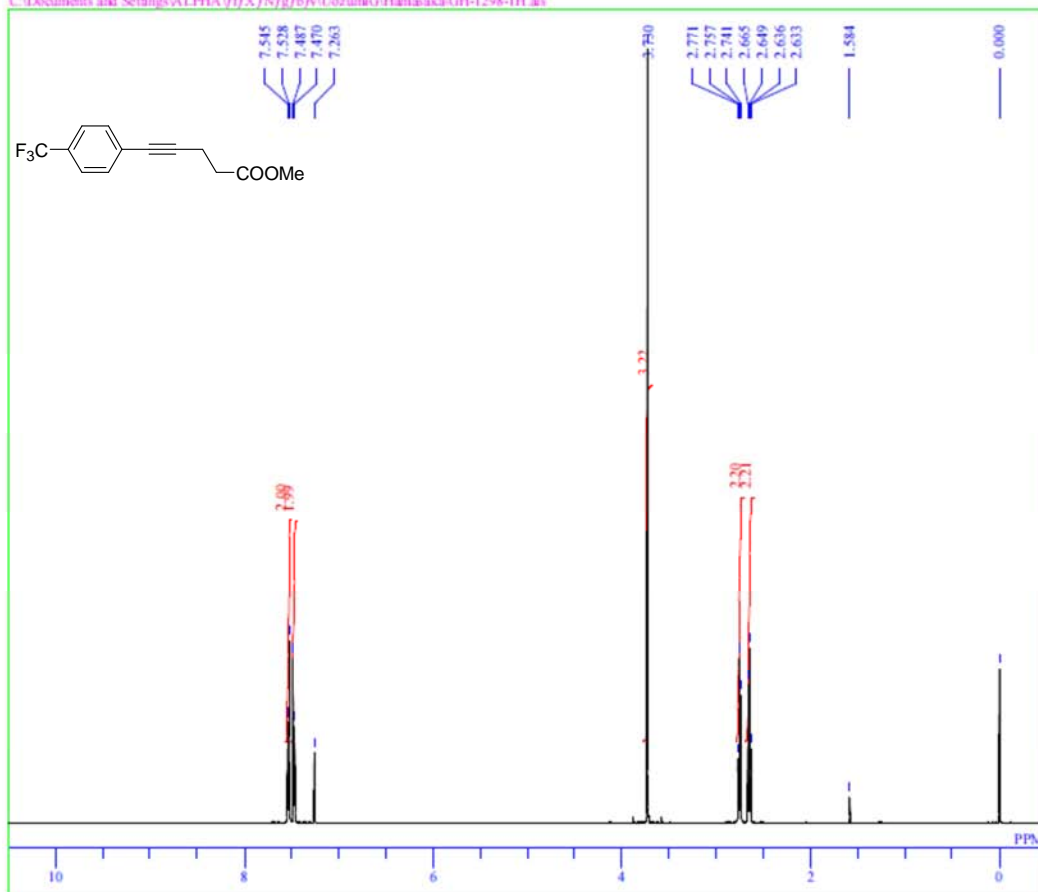


DFILE HCCCH₂CH₂COOMe-13C.als
COMNT HCCCH₂CH₂COOMe-13C
DATIM 2013-07-04 22:23:09
OBNUC 13C
EXMOD carbon.jsp
OBFREQ 99.55 MHz
OBSET 5.13 KHz
OBFIN 0.98 Hz
POINT 32767
FREQU 31250.00 Hz
SCANS 112
ACQTM 1.0486 sec
PD 2.0000 sec
PWI 3.42 usec
IRNUC 1H
CTEMP 19.0 c
SLVNT CDCL₃
EXREF 77.00 ppm
BF 0.50 Hz
RGAIN 60

Methyl 5-(4-trifluoromethylphenyl)-4-pentynoate (S2a)

GH-1298-1H

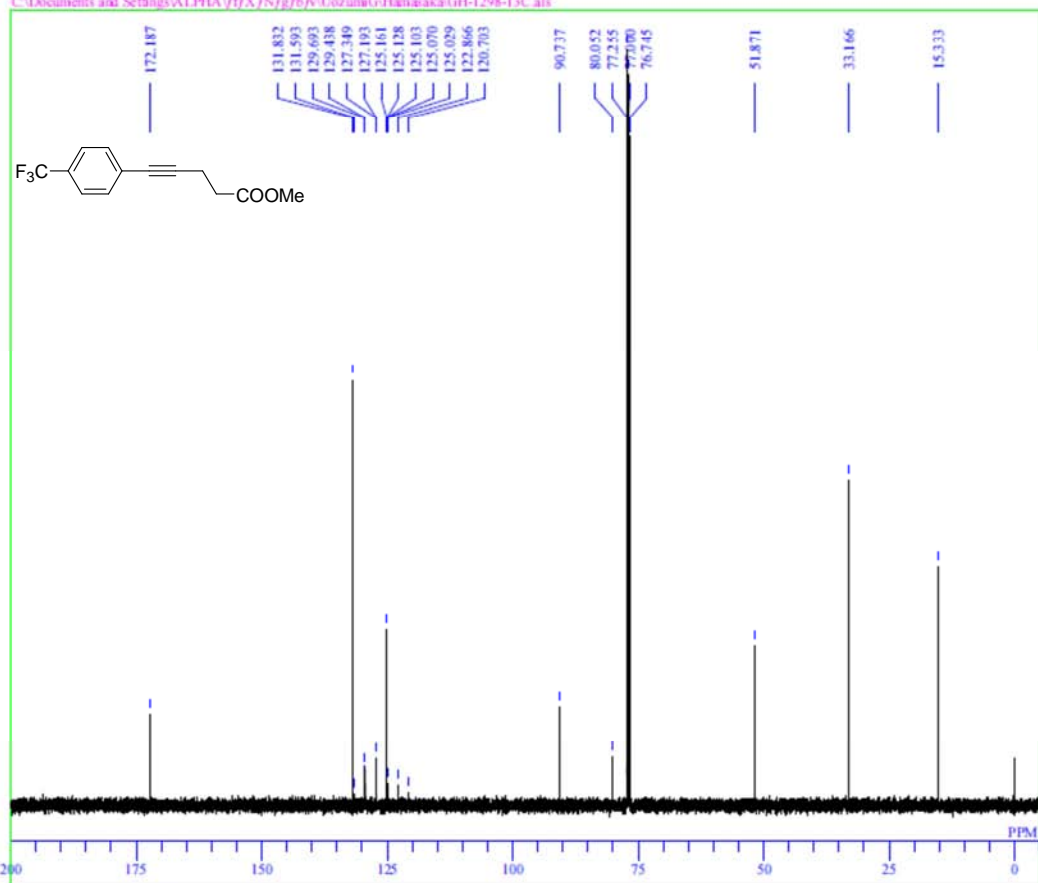
C:\Documents and Settings\ALPHA\ffx\N/g/b/A/Uozumi/G/Hamaoka/GH-1298-1H.als



DFILE GH-1298-1H.als
 COMNT GH-1298-1H
 DATIM Mon Oct 01 19:29:48 2012
 OBNUC 1H
 EXMOD SINGL
 OBFREQ 500.00 MHz
 OBSET 0.00 KHz
 OBFIN 162410.00 Hz
 POINT 16384
 FREQU 10000.00 Hz
 SCANS 16
 ACQTM 1.6384 sec
 PD 2.0000 sec
 PWI 6.40 usec
 IRNUC 1H
 CTEMP 22.5 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.00 Hz
 RGAIN 17

GH-1298-13C

C:\Documents and Settings\ALPHA\ffx\N/g/b/A/Uozumi/G/Hamaoka/GH-1298-13C.als

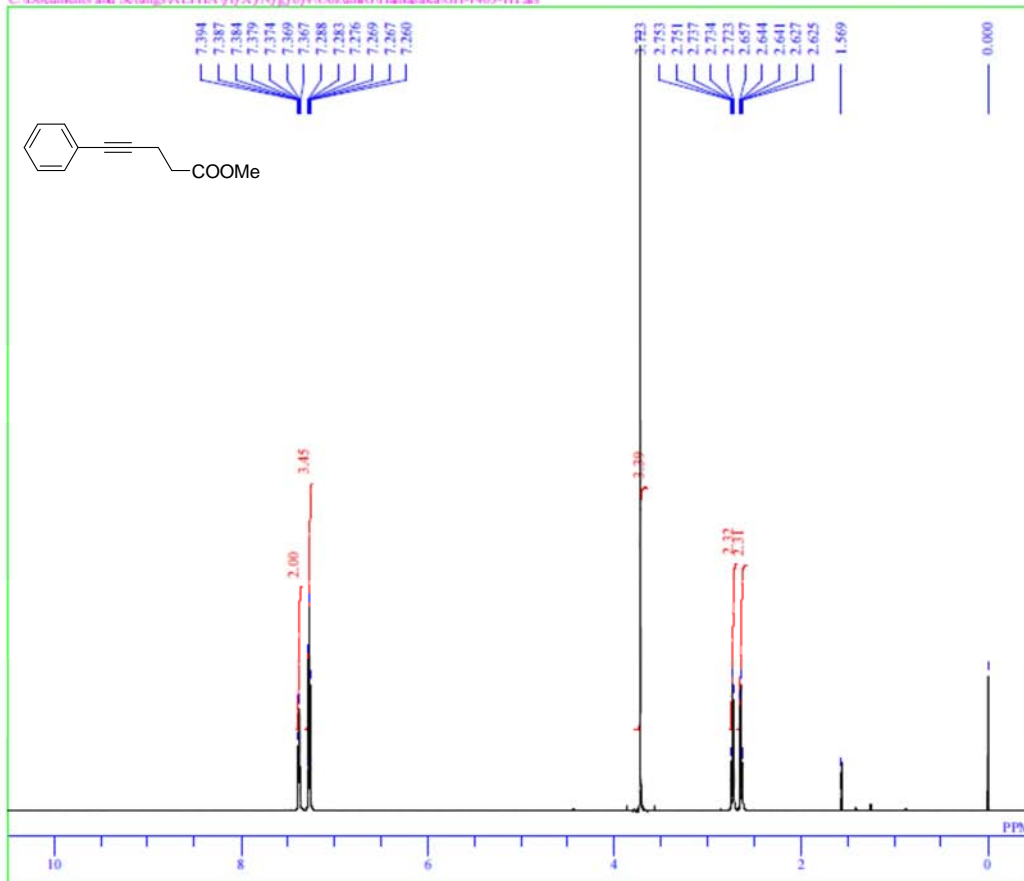


DFILE GH-1298-13C.als
 COMNT GH-1298-13C
 DATIM Mon Oct 01 20:38:05 2012
 OBNUC 13C
 EXMOD SINGL
 OBFREQ 125.65 MHz
 OBSET 0.00 KHz
 OBFIN 127958.00 Hz
 POINT 32768
 FREQU 33898.30 Hz
 SCANS 2000
 ACQTM 0.9667 sec
 PD 1.0000 sec
 PWI 5.75 usec
 IRNUC 13C
 CTEMP 23.9 c
 SLVNT CDCL3
 EXREF 77.00 ppm
 BF 0.00 Hz
 RGAIN 26

Methyl 5-phenyl-4-pentynoate (S2b)

GH-1463-1H

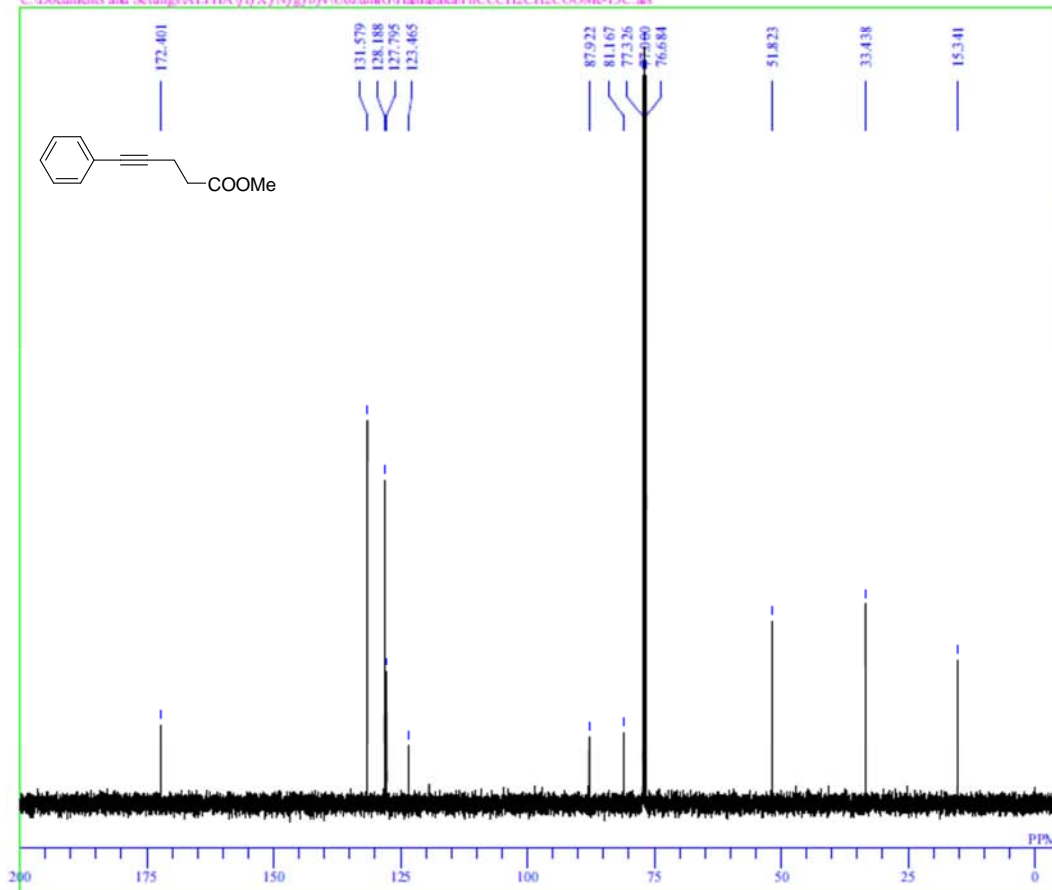
C:\Documents and Settings\ALPHA\ff\X\N\g\b\Uozumi\G\Hamazaki\GH-1463-1H.als



DFILE GH-1463-1H.als
 COMNT GH-1463-1H
 DATIM Fri Feb 15 21:39:57 2013
 OBNUC 1H
 EXMOD SINGL
 OBFREQ 500.00 MHz
 OBSET 0.00 KHz
 OBFIN 162410.00 Hz
 POINT 16384
 FREQU 10000.00 Hz
 SCANS 16
 ACQTM 1.6384 sec
 PD 2.0000 sec
 PWI 6.40 usec
 IRNUC 1H
 CTEMP 21.6 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.00 Hz
 RGAIN 20

PhCCCH2CH2COOMe-13C

C:\Documents and Settings\ALPHA\ff\X\N\g\b\Uozumi\G\Hamazaki\PhCCCH2CH2COOMe-13C.als

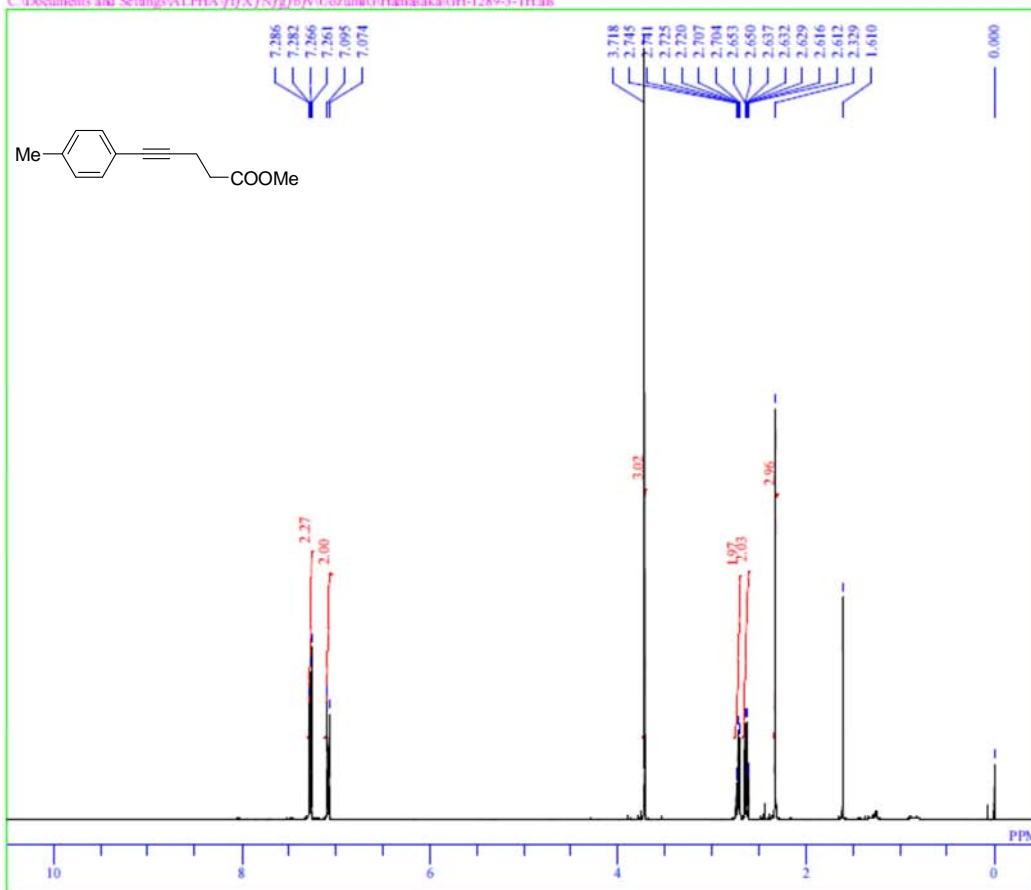


DFILE PhCCCH2CH2COOMe-13C.als
 COMNT PhCCCH2CH2COOMe-13C
 DATIM 2013-07-05 19:15:02
 OBNUC 13C
 EXMOD carbon.jsp
 OBFREQ 99.55 MHz
 OBSET 5.13 KHz
 OBFIN 0.98 Hz
 POINT 32767
 FREQU 31250.00 Hz
 SCANS 239
 ACQTM 1.0486 sec
 PD 2.0000 sec
 PWI 3.42 usec
 IRNUC 13C
 CTEMP 21.3 c
 SLVNT CDCL3
 EXREF 77.00 ppm
 BF 0.50 Hz
 RGAIN 60

Methyl 5-(4-methylphenyl)-4-pentynoate (S2c)

GH-1289-3-1H

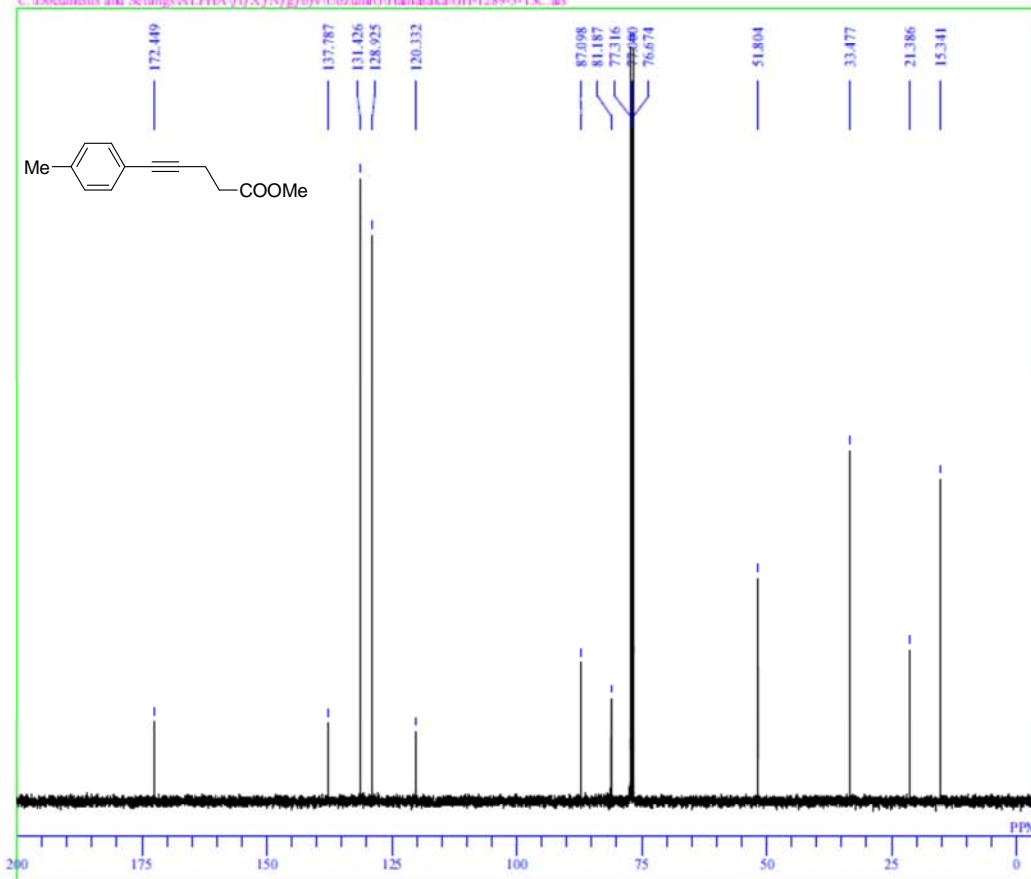
C:\Documents and Settings\ALPHA\ff\X\N\g\b\A\Uozumi\GHanasaki\GH-1289-3-1H.als



DFILE GH-1289-3-1H.als
 COMNT GH-1289-3-1H
 DATIM 2013-06-18 21:49:22
 OBNUC 1H
 EXMOD proton.jsp
 OBFREQ 395.88 MHz
 OBSSET 6.28 KHz
 OBFIN 0.87 Hz
 POINT 16384
 FREQU 7422.80 Hz
 SCANS 8
 ACQTM 2.2073 sec
 PD 5.0000 sec
 PW1 3.12 usec
 BRNUC 1H
 CTEMP 19.1 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.00 Hz
 RGAIN 34

GH-1289-3-13C

C:\Documents and Settings\ALPHA\ff\X\N\g\b\A\Uozumi\GHanasaki\GH-1289-3-13C.als

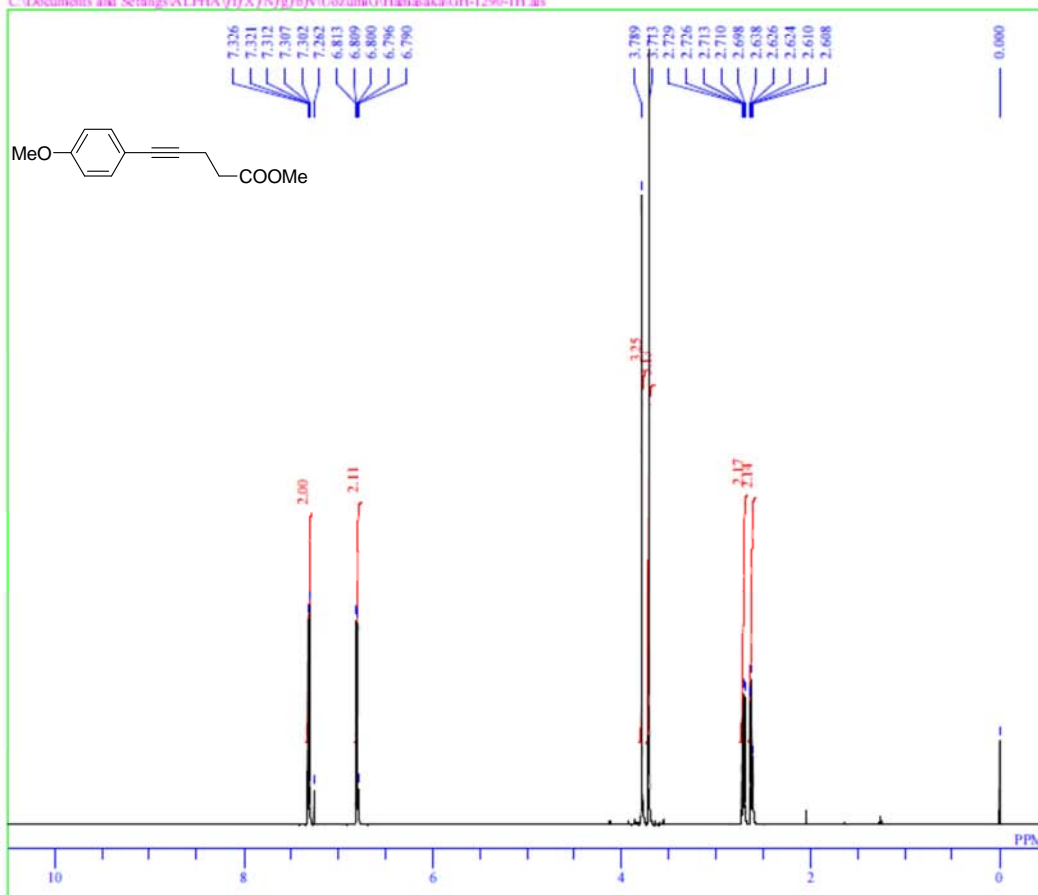


DFILE GH-1289-3-13C.als
 COMNT GH-1289-3-13C
 DATIM 2013-06-18 21:53:45
 OBNUC 13C
 EXMOD carbon.jsp
 OBFREQ 99.55 MHz
 OBSSET 5.13 KHz
 OBFIN 0.98 Hz
 POINT 32767
 FREQU 31250.00 Hz
 SCANS 864
 ACQTM 1.0486 sec
 PD 2.0000 sec
 PW1 3.42 usec
 BRNUC 13C
 CTEMP 19.1 c
 SLVNT CDCL3
 EXREF 77.00 ppm
 BF 0.40 Hz
 RGAIN 60

Methyl 5-(4-methoxyphenyl)-4-pentynoate (**S2d**)

GH-1290-1H

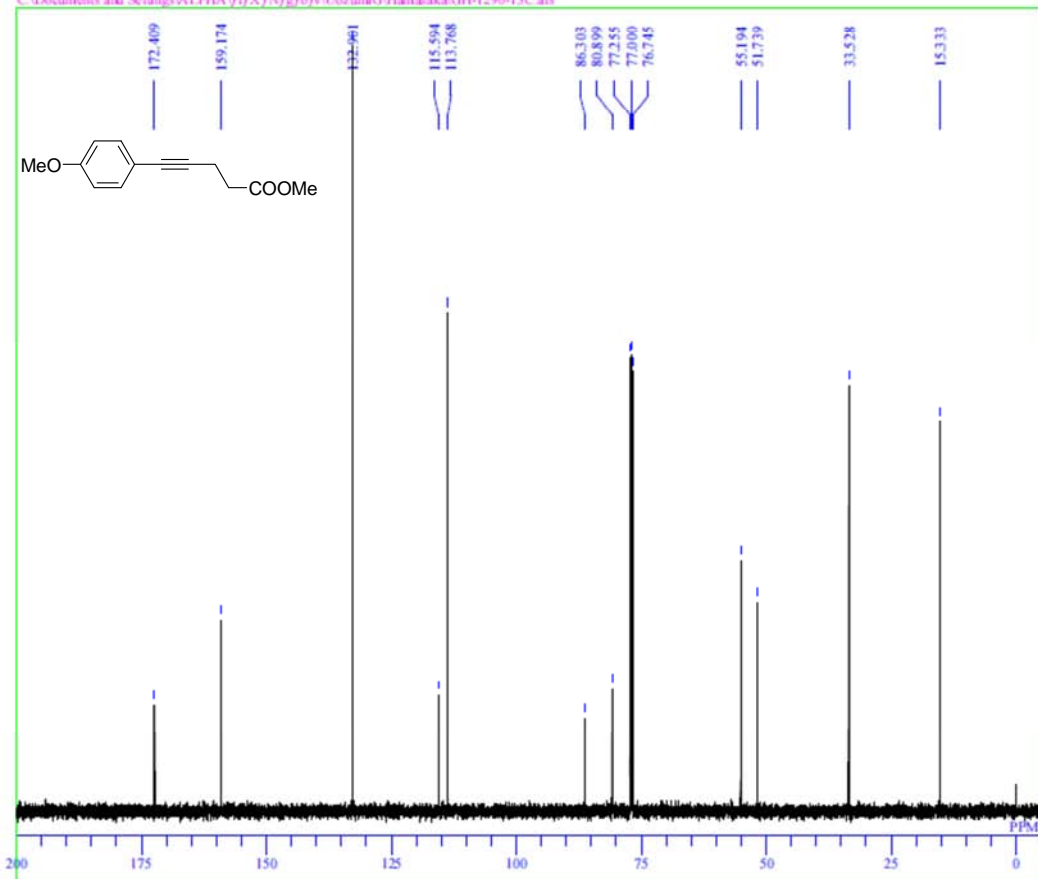
C:\Documents and Settings\ALPHA\p\X\N\p\6\Uozumi\G\Hamaoka\GH-1290-1H.als



DFILE GH-1290-1H.als
 COMNT GH-1290-1H
 DATIM Thu Sep 27 19:23:00 2012
 OBNUC 1H
 EXMOD SINGL
 OBFREQ 500.00 MHz
 OBSET 0.00 KHz
 OBFIN 162410.00 Hz
 POINT 16384
 FREQU 10000.00 Hz
 SCANS 16
 ACQTM 1.6384 sec
 PD 2.0000 sec
 PWI 6.40 usec
 IRNUC 1H
 CTEMP 22.0 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.00 Hz
 RGAIN 14

GH-1290-13C

C:\Documents and Settings\ALPHA\p\X\N\p\6\Uozumi\G\Hamaoka\GH-1290-13C.als

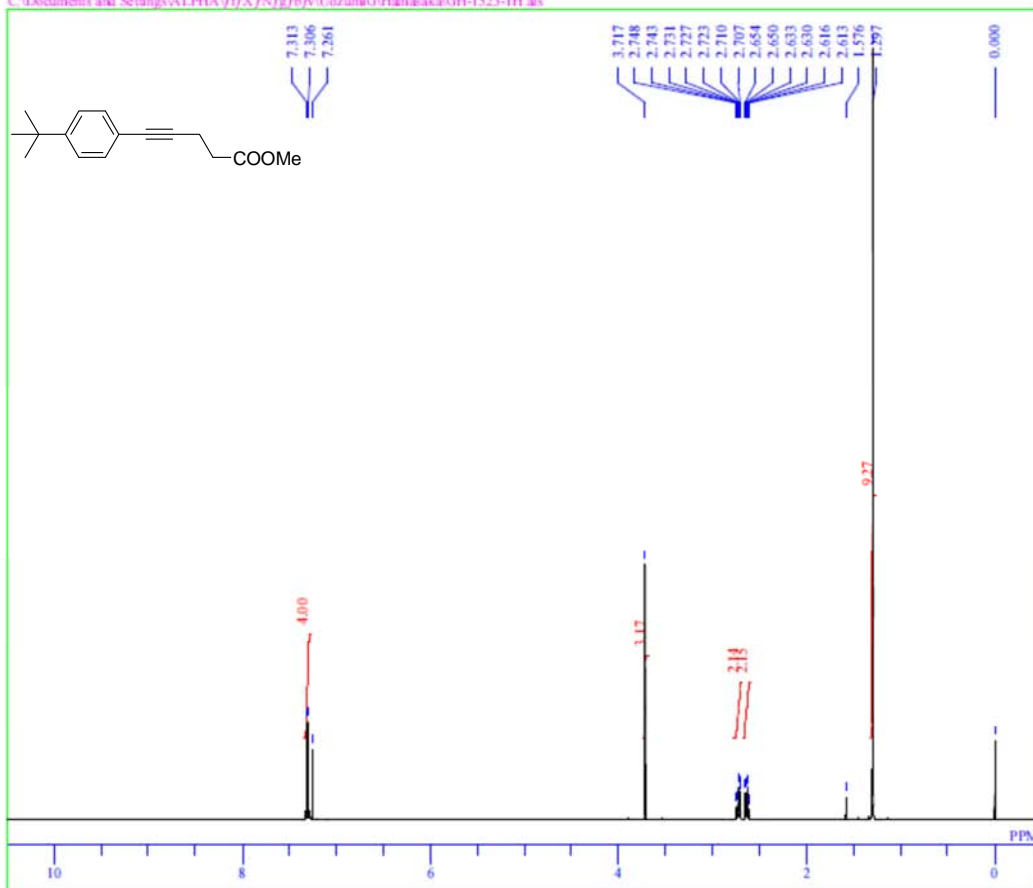


DFILE GH-1290-13C.als
 COMNT GH-1290-13C
 DATIM Thu Sep 27 19:42:14 2012
 OBNUC 13C
 EXMOD SINGL
 OBFREQ 125.65 MHz
 OBSET 0.00 KHz
 OBFIN 127958.00 Hz
 POINT 32768
 FREQU 33898.30 Hz
 SCANS 531
 ACQTM 0.9667 sec
 PD 1.0000 sec
 PWI 5.75 usec
 IRNUC 13C
 CTEMP 23.5 c
 SLVNT CDCL3
 EXREF 77.00 ppm
 BF 0.40 Hz
 RGAIN 26

Methyl 5-(4-*tert*-butylphenyl)-4-pentynoate (**S2e**)

GH-1523-1H

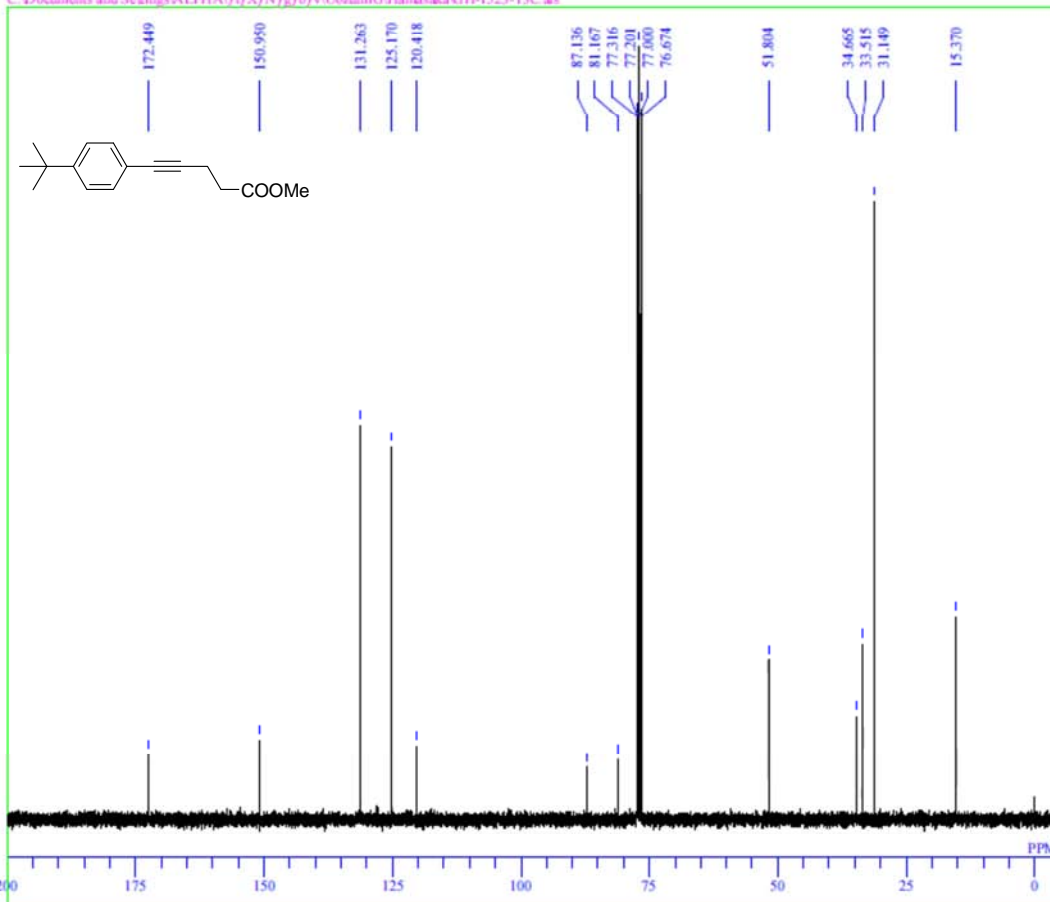
C:\Documents and Settings\ALPHA\I\X\N\g\h\Uozumi\G\Hamasaki\GH-1523-1H.als



DFILE GH-1523-1H.als
 COMNT GH-1523-1H
 DATIM 2013-05-01 21:24:31
 IRNUC 1H
 EXMOD proton.jsp
 OBFREQ 395.88 MHz
 OBSET 6.28 KHz
 OBFIN 0.87 Hz
 POINT 13107
 FREQU 5938.24 Hz
 SCANS 8
 ACQTM 2.2073 sec
 PD 5.0000 sec
 PW1 3.12 usec
 IRNUC 1H
 CTEMP 19.1 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.00 Hz
 RGAIN 36

GH-1523-13C

C:\Documents and Settings\ALPHA\I\X\N\g\h\Uozumi\G\Hamasaki\GH-1523-13C.als

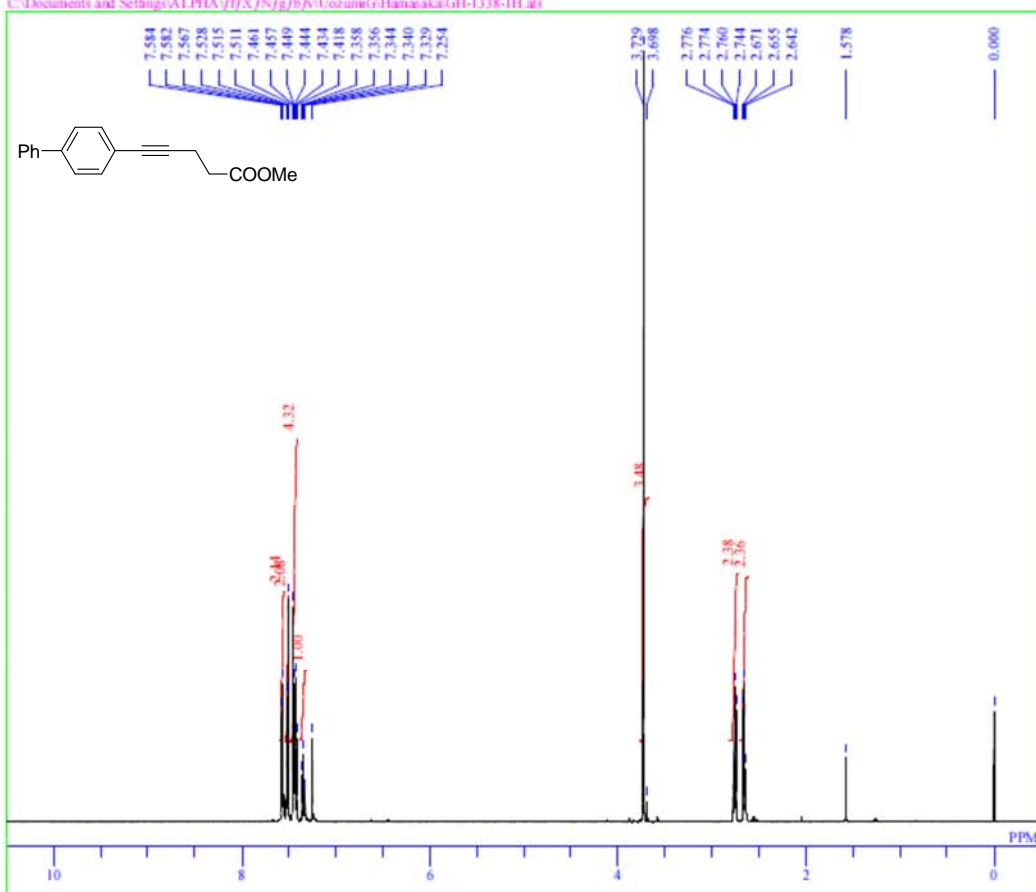


DFILE GH-1523-13C.als
 COMNT GH-1523-13C
 DATIM 2013-05-01 21:27:54
 IRNUC 13C
 EXMOD carbon.jsp
 OBFREQ 99.55 MHz
 OBSET 5.13 KHz
 OBFIN 0.98 Hz
 POINT 32767
 FREQU 31250.00 Hz
 SCANS 544
 ACQTM 1.0486 sec
 PD 2.0000 sec
 PW1 3.42 usec
 IRNUC 1H
 CTEMP 18.6 c
 SLVNT CDCL3
 EXREF 77.00 ppm
 BF 0.50 Hz
 RGAIN 60

Methyl 5-(4-phenylphenyl)-4-pentynoate (**S2f**)

GH-1338-1H

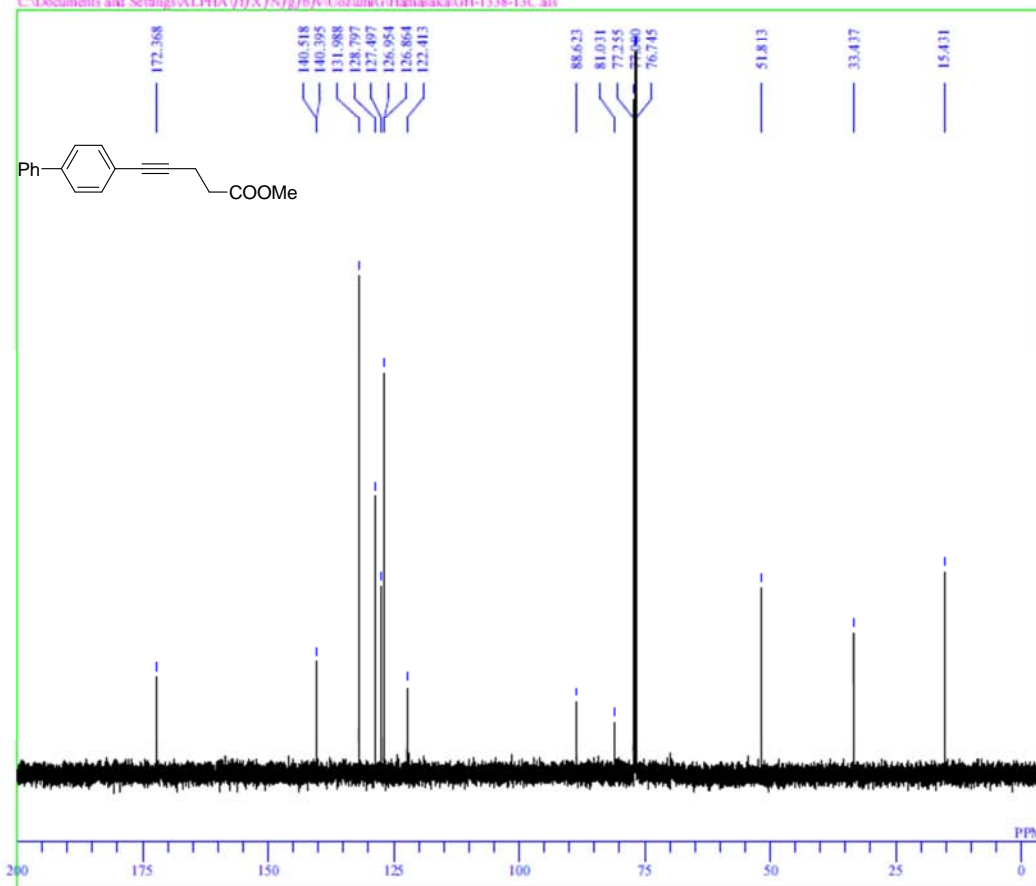
C:\Documents and Settings\ALPHA\ffX\N\g\b\A\Uozumi\GH\Hanaoka\GH-1338-1H.als



DFILE GH-1338-1H.als
 COMNT GH-1338-1H
 DATIM Thu Nov 22 21:33:51 2012
 OBNUC 1H
 EXMOD SINGL
 OBFREQ 500.00 MHz
 OBSSET 0.00 KHz
 OBFIN 162410.00 Hz
 POINT 16384
 FREQU 10000.00 Hz
 SCANS 16
 ACQTM 1.6384 sec
 PD 2.0000 sec
 PWI 6.40 usec
 IRNUC 1H
 CTEMP 21.5 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.00 Hz
 RGAIN 17

GH-1338-13C

C:\Documents and Settings\ALPHA\ffX\N\g\b\A\Uozumi\GH\Hanaoka\GH-1338-13C.als

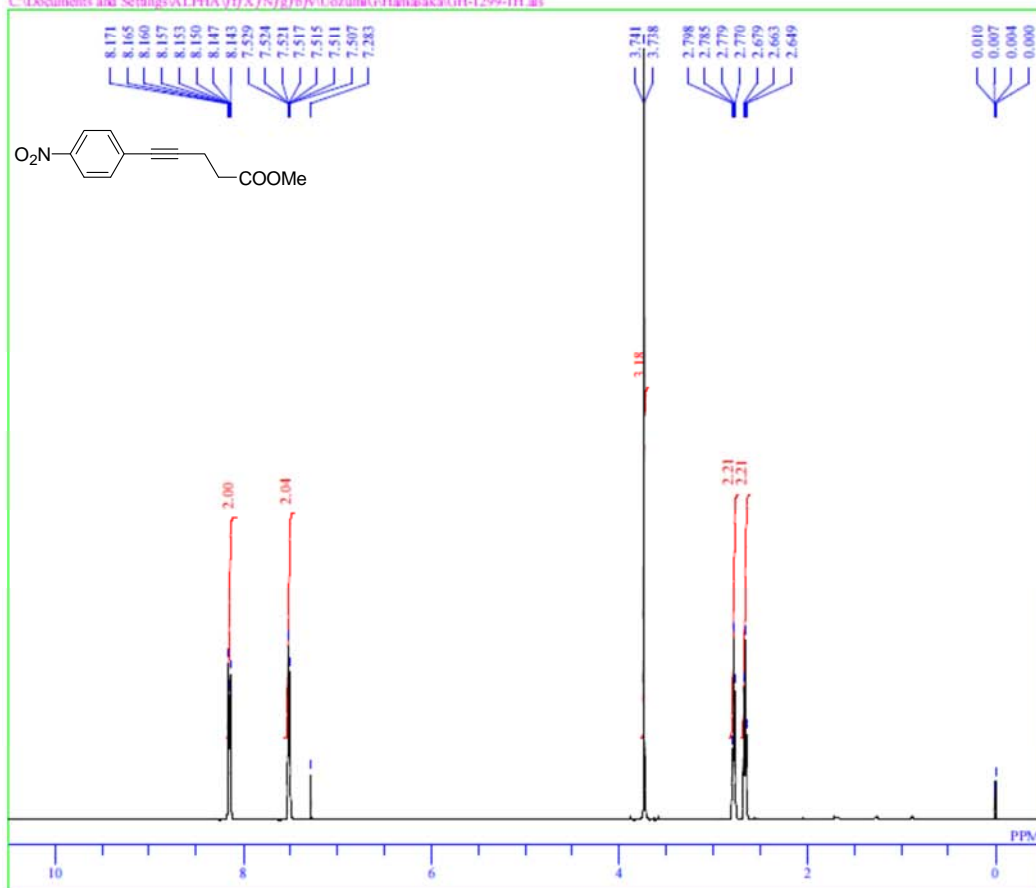


DFILE GH-1338-13C.als
 COMNT GH-1338-13C
 DATIM Tue Nov 27 10:32:00 2012
 OBNUC 13C
 EXMOD SINGL
 OBFREQ 125.65 MHz
 OBSSET 0.00 KHz
 OBFIN 127958.00 Hz
 POINT 32768
 FREQU 33898.30 Hz
 SCANS 463
 ACQTM 0.9667 sec
 PD 1.0000 sec
 PWI 5.75 usec
 IRNUC 1H
 CTEMP 23.8 c
 SLVNT CDCL3
 EXREF 77.00 ppm
 BF 0.20 Hz
 RGAIN 26

Methyl 5-(4-nitrophenyl)-4-pentynoate (**S2g**)

GH-1299-1H

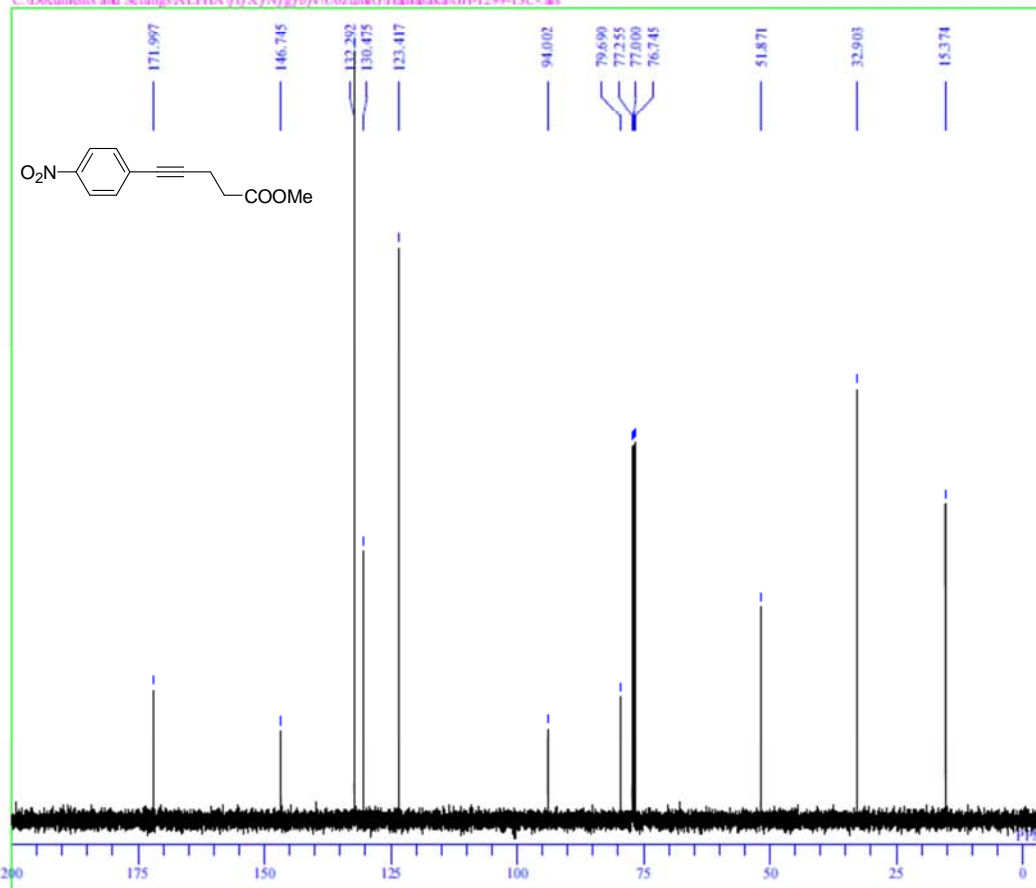
C:\Documents and Settings\ALPHA\ff\X\N/g/b/A/UozumiG\Hanawaka\GH-1299-1H.xls



DFILE GH-1299-1H.xls
 COMNT GH-1299-1H
 DATIM Mon Oct 01 20:55:22 2012
 OBNUC 1H
 EXMOD SINGL
 OBFREQ 500.00 MHz
 OBSET 0.00 KHz
 OBFIN 162410.00 Hz
 POINT 16384
 FREQU 10000.00 Hz
 SCANS 16
 ACQTM 1.6384 sec
 PD 2.0000 sec
 PWI 6.40 usec
 IRNUC 1H
 CTEMP 22.8 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.00 Hz
 RGAIN 15

GH-1299-13C

C:\Documents and Settings\ALPHA\ff\X\N/g/b/A/UozumiG\Hanawaka\GH-1299-13C.xls

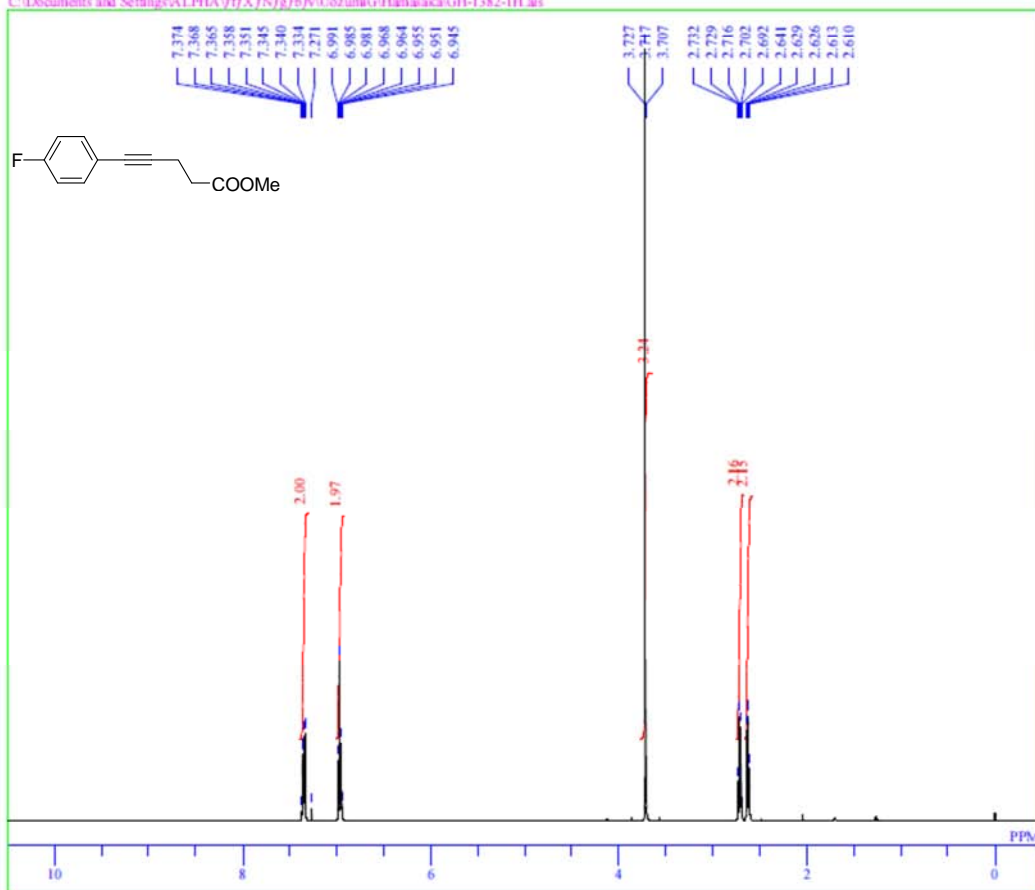


DFILE GH-1299-13C-.xls
 COMNT GH-1299-13C
 DATIM Mon Oct 01 21:16:13 2012
 OBNUC 13C
 EXMOD SINGL
 OBFREQ 125.65 MHz
 OBSET 0.00 KHz
 OBFIN 127958.00 Hz
 POINT 32768
 FREQU 33898.30 Hz
 SCANS 58
 ACQTM 0.9667 sec
 PD 1.0000 sec
 PWI 5.75 usec
 IRNUC 1H
 CTEMP 23.7 c
 SLVNT CDCL3
 EXREF 77.00 ppm
 BF 0.00 Hz
 RGAIN 27

Methyl 5-(4-fluorophenyl)-4-pentynoate (**S2h**)

GH-1382-1H

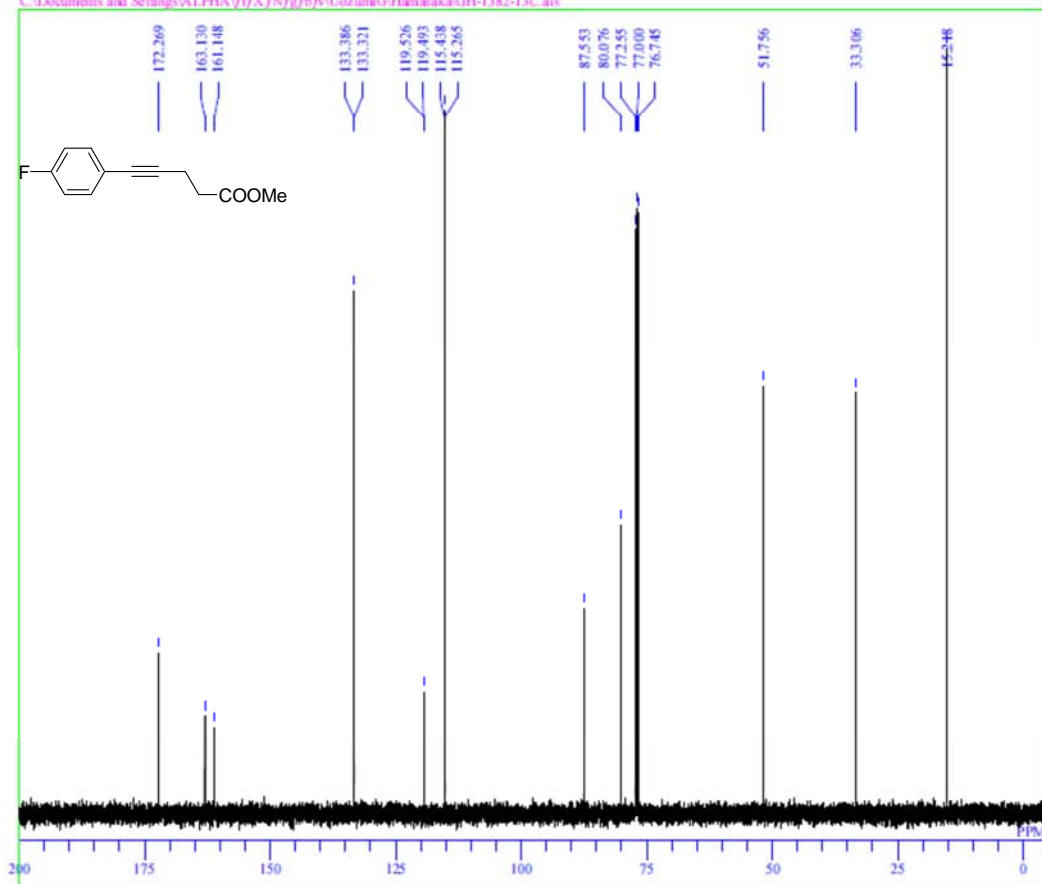
C:\Documents and Settings\ALPHA\ffX\N/g/b/A/UozumiGHanawaka\GH-1382-1H.als



DFILE GH-1382-1H.als
 COMNT GH-1382-1H
 DATIM Thu Dec 20 10:54:28 2012
 OBNUC 1H
 EXMOD SINGL
 OBFREQ 500.00 MHz
 OBSSET 0.00 KHz
 OBFIN 162410.00 Hz
 POINT 16384
 FREQU 10000.00 Hz
 SCANS 16
 ACQTM 1.6384 sec
 PD 2.0000 sec
 PW1 6.40 usec
 IRNUC 1H
 CTEMP 22.1 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.00 Hz
 RGAIN 14

GH-1382-13C

C:\Documents and Settings\ALPHA\ffX\N/g/b/A/UozumiGHanawaka\GH-1382-13C.als

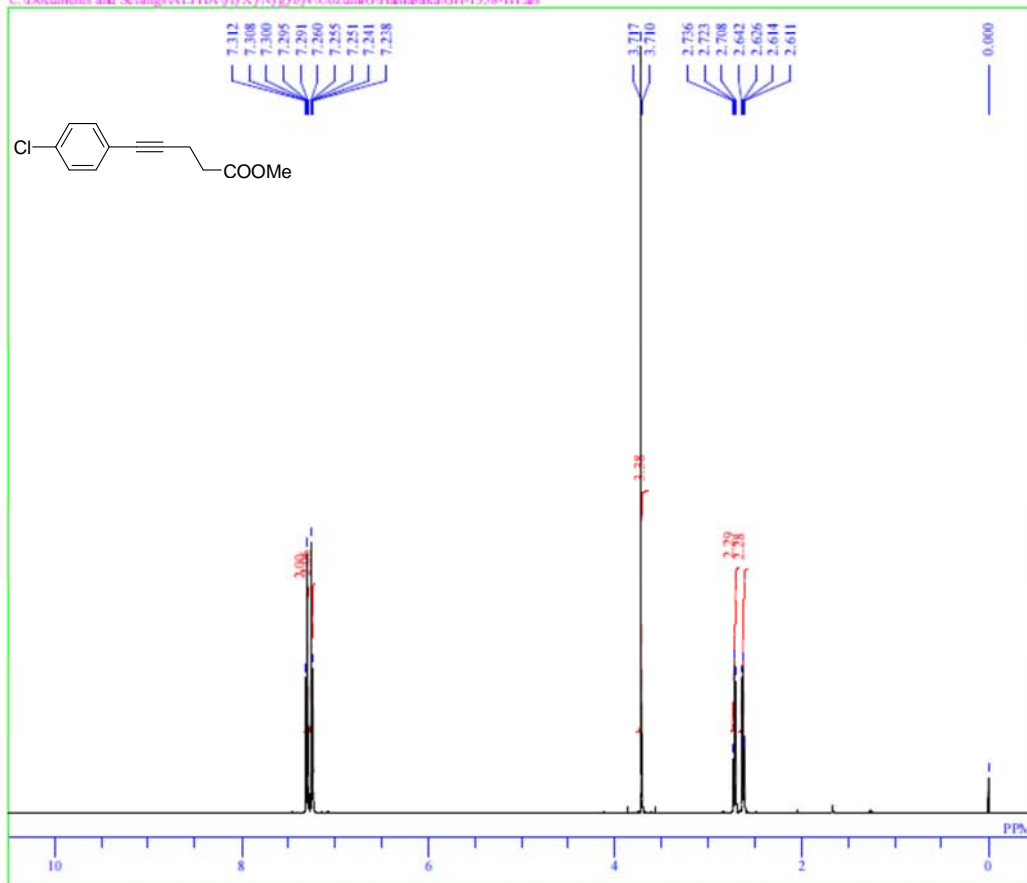


DFILE GH-1382-13C.als
 COMNT GH-1382-13C
 DATIM Thu Dec 20 11:08:18 2012
 OBNUC 13C
 EXMOD SINGL
 OBFREQ 125.65 MHz
 OBSSET 0.00 KHz
 OBFIN 127958.00 Hz
 POINT 32768
 FREQU 33898.30 Hz
 SCANS 350
 ACQTM 0.9667 sec
 PD 1.0000 sec
 PW1 5.75 usec
 IRNUC 1H
 CTEMP 23.3 c
 SLVNT CDCL3
 EXREF 77.00 ppm
 BF 0.20 Hz
 RGAIN 27

Methyl 5-(4-chlorophenyl)-4-pentynoate (S2i)

GH-1358-1H

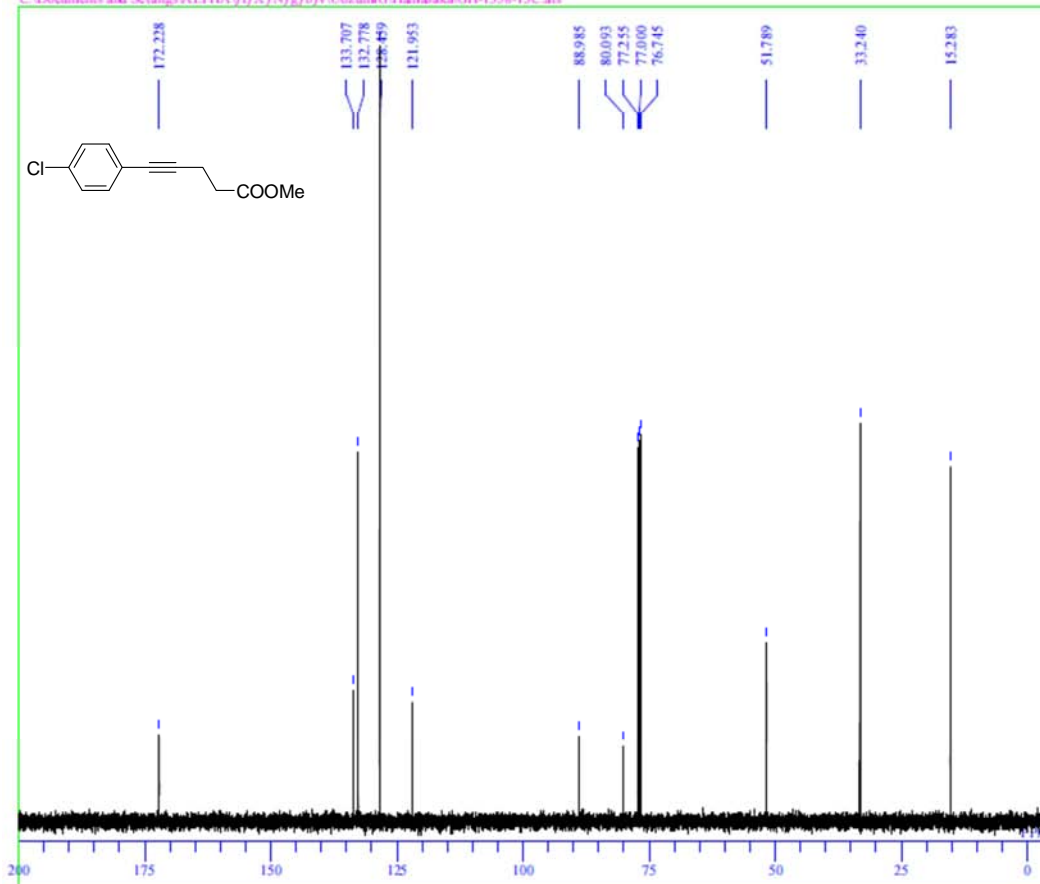
C:\Documents and Settings\ALPHA\ff\X\N/g/b\N\Uozumi\G\Hamazaki\GH-1358-1H.als



DFILE GH-1358-1H.als
 COMNT GH-1358-1H
 DATIM Fri Dec 07 17:18:22 2012
 OBNUC 1H
 EXMOD SINGL
 OBFREQ 500.00 MHz
 OBSSET 0.00 KHz
 OBFIN 162410.00 Hz
 POINT 16384
 FREQU 10000.00 Hz
 SCANS 16
 ACQTM 1.6384 sec
 PD 2.0000 sec
 PW1 6.40 usec
 IRNUC 1H
 CTEMP 22.0 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.00 Hz
 RGAIN 14

GH-1358-13C

C:\Documents and Settings\ALPHA\ff\X\N/g/b\N\Uozumi\G\Hamazaki\GH-1358-13C.als

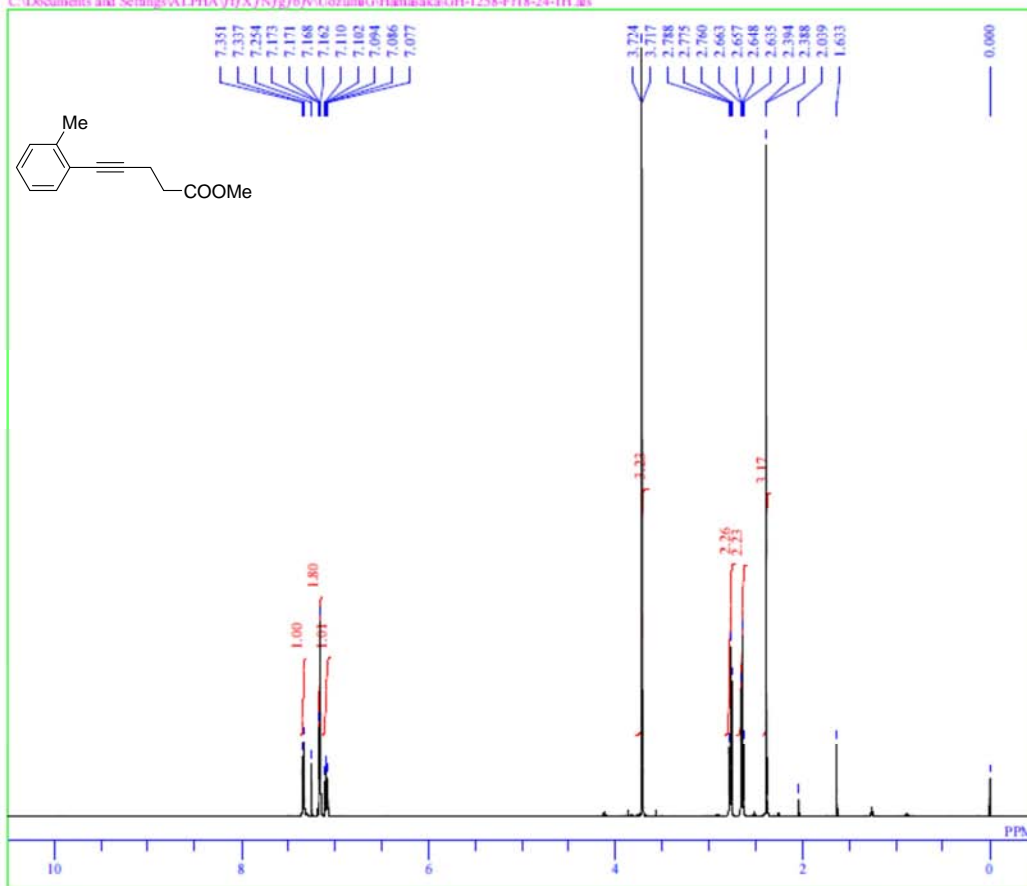


DFILE GH-1358-13C.als
 COMNT GH-1358-13C
 DATIM Fri Dec 07 17:27:01 2012
 OBNUC 13C
 EXMOD SINGL
 OBFREQ 125.65 MHz
 OBSSET 0.00 KHz
 OBFIN 127958.00 Hz
 POINT 32768
 FREQU 33898.30 Hz
 SCANS 177
 ACQTM 0.9667 sec
 PD 1.0000 sec
 PW1 5.75 usec
 IRNUC 13C
 CTEMP 24.1 c
 SLVNT CDCL3
 EXREF 77.00 ppm
 BF 0.40 Hz
 RGAIN 27

Methyl 5-(2-Methylphenyl)-4-pentynoate (**S2j**)

GH-1258-Fr18-24-1H

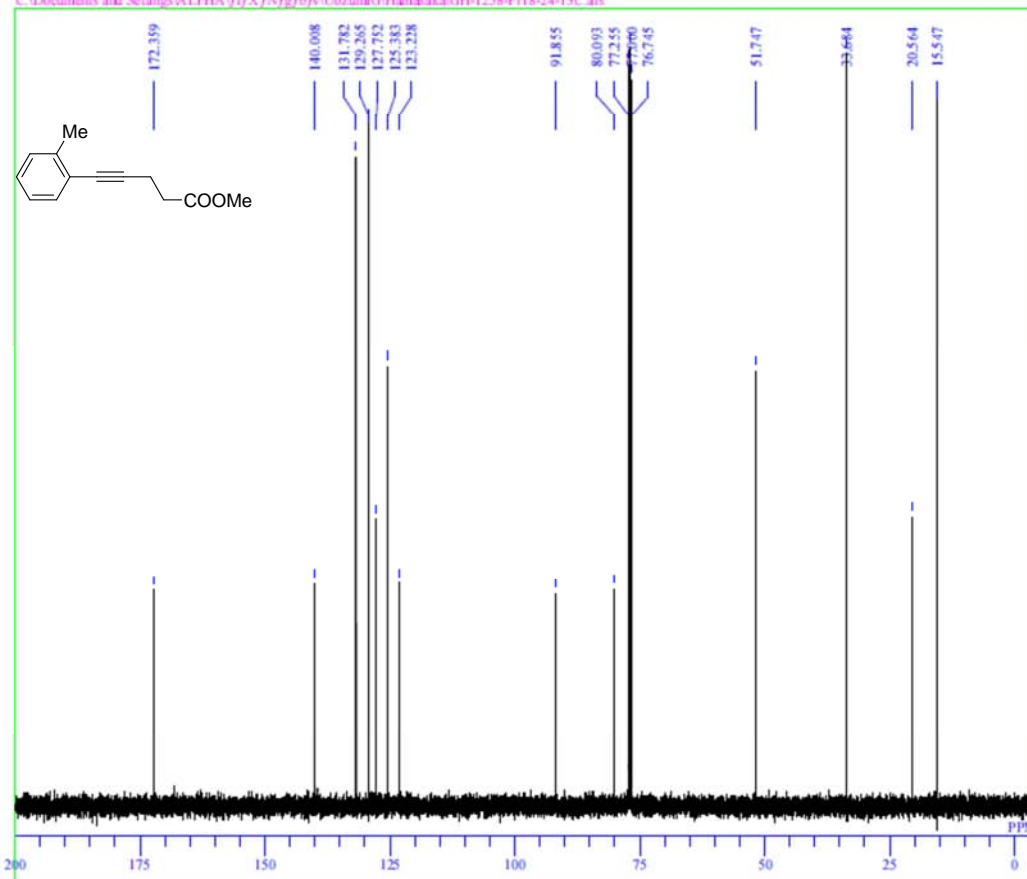
C:\Documents and Settings\ALPHA\ffX\N/g/b/v/Uozumi\G\Hamakata\GH-1258-Fr18-24-1H.als



DFILE GH-1258-Fr18-24-1H.als
 COMNT GH-1258-Fr18-24-1H
 DATIM Wed Aug 08 17:39:40 2012
 OBNUC 1H
 EXMOD SINGL
 OBFREQ 500.00 MHz
 OBSET 0.00 KHz
 OBFIN 162410.00 Hz
 POINT 16384
 FREQU 10000.00 Hz
 SCANS 16
 ACQTM 1.6384 sec
 PD 2.0000 sec
 PWI 6.40 usec
 IRNUC 1H
 CTEMP 24.9 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.00 Hz
 RGAIN 15

GH-1258-Fr18-24-13C

C:\Documents and Settings\ALPHA\ffX\N/g/b/v/Uozumi\G\Hamakata\GH-1258-Fr18-24-13C.als

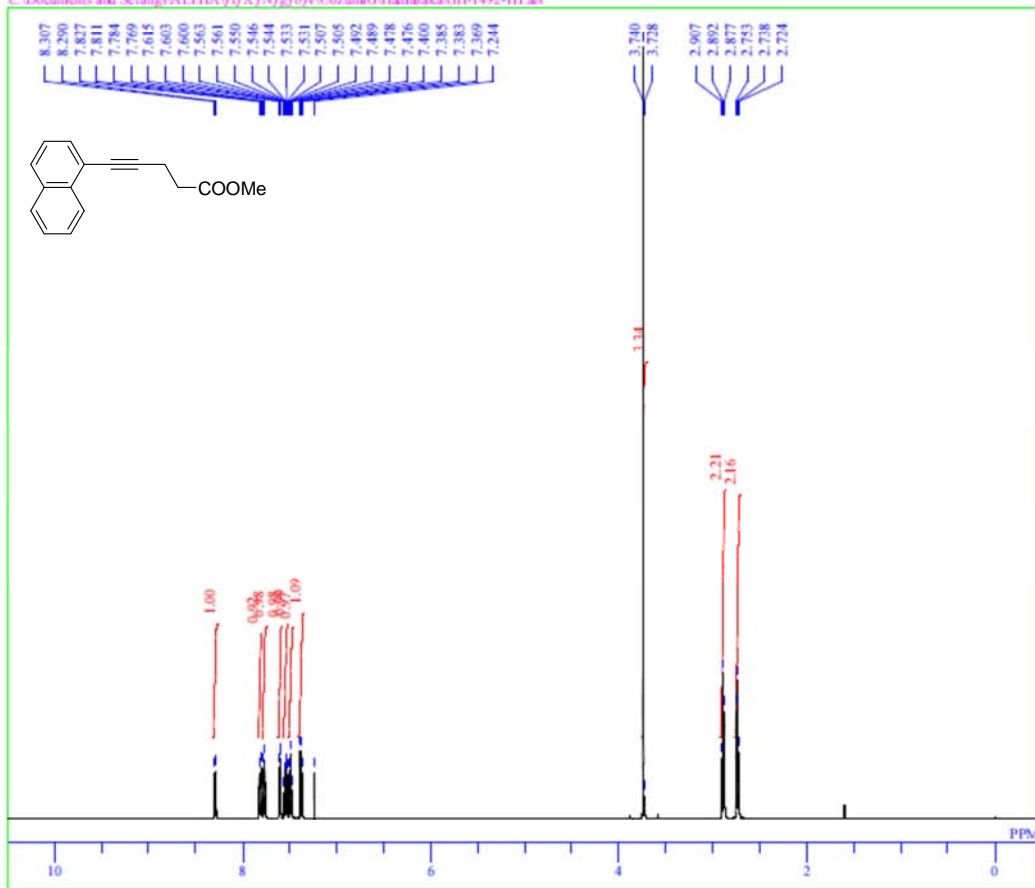


DFILE GH-1258-Fr18-24-13C.als
 COMNT GH-1258-Fr18-24-13C
 DATIM Wed Aug 08 18:01:03 2012
 OBNUC 13C
 EXMOD SINGL
 OBFREQ 125.65 MHz
 OBSET 0.00 KHz
 OBFIN 127958.00 Hz
 POINT 32768
 FREQU 33898.30 Hz
 SCANS 474
 ACQTM 0.9667 sec
 PD 1.0000 sec
 PWI 5.75 usec
 IRNUC 13C
 CTEMP 26.7 c
 SLVNT CDCL3
 EXREF 77.00 ppm
 BF 0.20 Hz
 RGAIN 26

Methyl 5-(1-naphthyl)-4-pentynoate (S2k)

GH-1492-1H

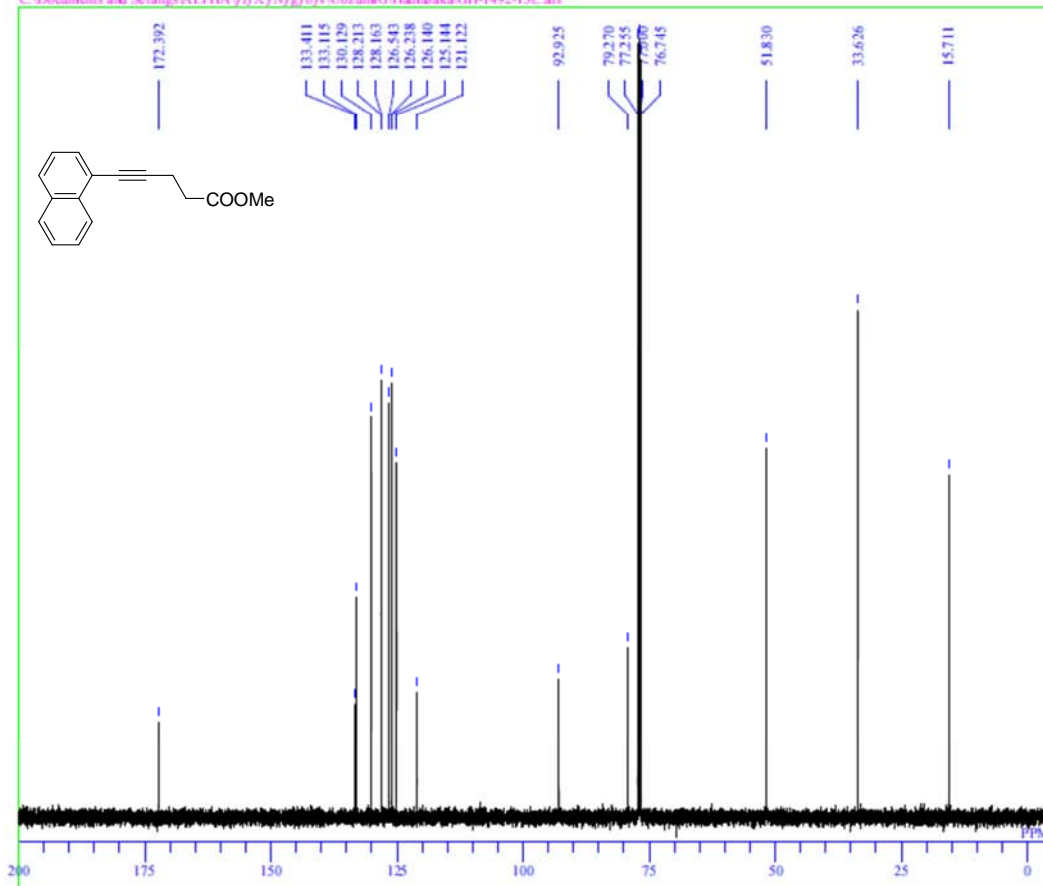
C:\Documents and Settings\ALPHA\ff\X\N/g/b/v/UozumiGHamasaka\GH-1492-1H.als



DFILE GH-1492-1H.als
 COMNT GH-1492-1H
 DATIM Fri Mar 15 19:35:51 2013
 OBNUC 1H
 EXMOD SINGL
 OBFREQ 500.00 MHz
 OBSET 0.00 KHz
 OBFIN 162410.00 Hz
 POINT 16384
 FREQU 10000.00 Hz
 SCANS 16
 ACQTM 1.6384 sec
 PD 2.0000 sec
 PWI 6.40 usec
 IRNUC 1H
 CTEMP 22.2 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.00 Hz
 RGAIN 16

GH-1492-13C

C:\Documents and Settings\ALPHA\ff\X\N/g/b/v/UozumiGHamasaka\GH-1492-13C.als

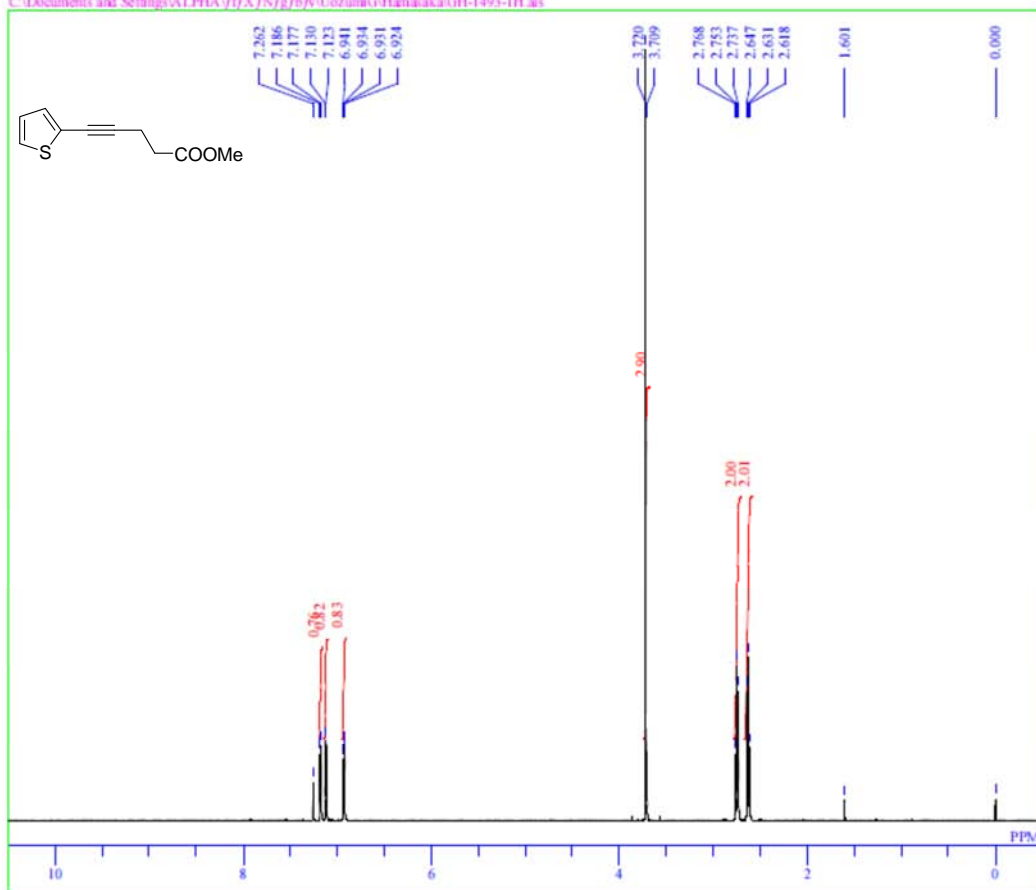


DFILE GH-1492-13C.als
 COMNT GH-1492-13C
 DATIM Fri Mar 15 20:05:17 2013
 OBNUC 13C
 EXMOD SINGL
 OBFREQ 125.65 MHz
 OBSET 0.00 KHz
 OBFIN 127958.00 Hz
 POINT 32768
 FREQU 33898.30 Hz
 SCANS 795
 ACQTM 0.9667 sec
 PD 1.0000 sec
 PWI 5.75 usec
 IRNUC 1H
 CTEMP 23.7 c
 SLVNT CDCL3
 EXREF 77.00 ppm
 BF 0.30 Hz
 RGAIN 26

Methyl 5-(2-thienyl)-4-pentynoate (**S21**)

GH-1493-1H

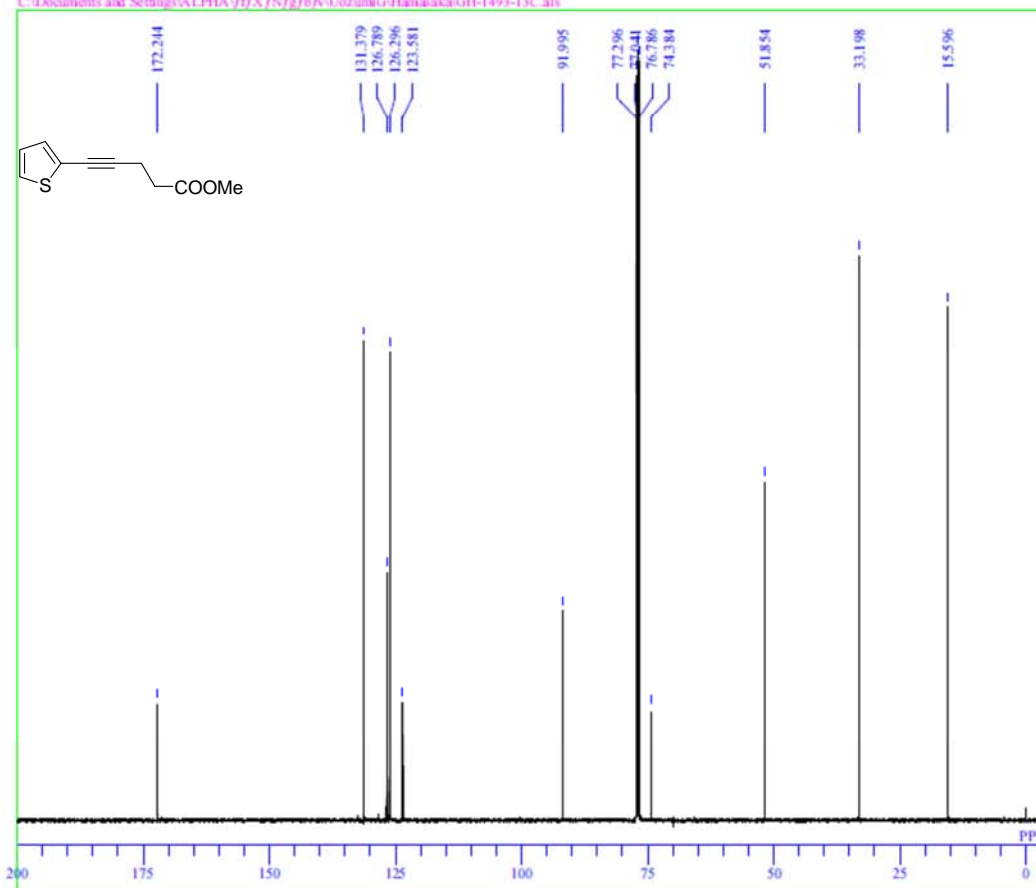
C:\Documents and Settings\ALPHA\ff\X\N\g\b\A\Uozumi\GHanaka\GH-1493-1H.als



DFILE GH-1493-1H.als
 COMNT GH-1493-1H
 DATIM Mon Mar 04 22:50:27 2013
 OBNUC 1H
 EXMOD SINGL
 OBFREQ 500.00 MHz
 OBSET 0.00 KHz
 OBFIN 162410.00 Hz
 POINT 16384
 FREQU 10000.00 Hz
 SCANS 16
 ACQTM 1.6384 sec
 PD 2.0000 sec
 PWI 6.40 usec
 IRNUC 1H
 CTEMP 22.3 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.00 Hz
 RGAIN 16

GH-1493-13C

C:\Documents and Settings\ALPHA\ff\X\N\g\b\A\Uozumi\GHanaka\GH-1493-13C.als

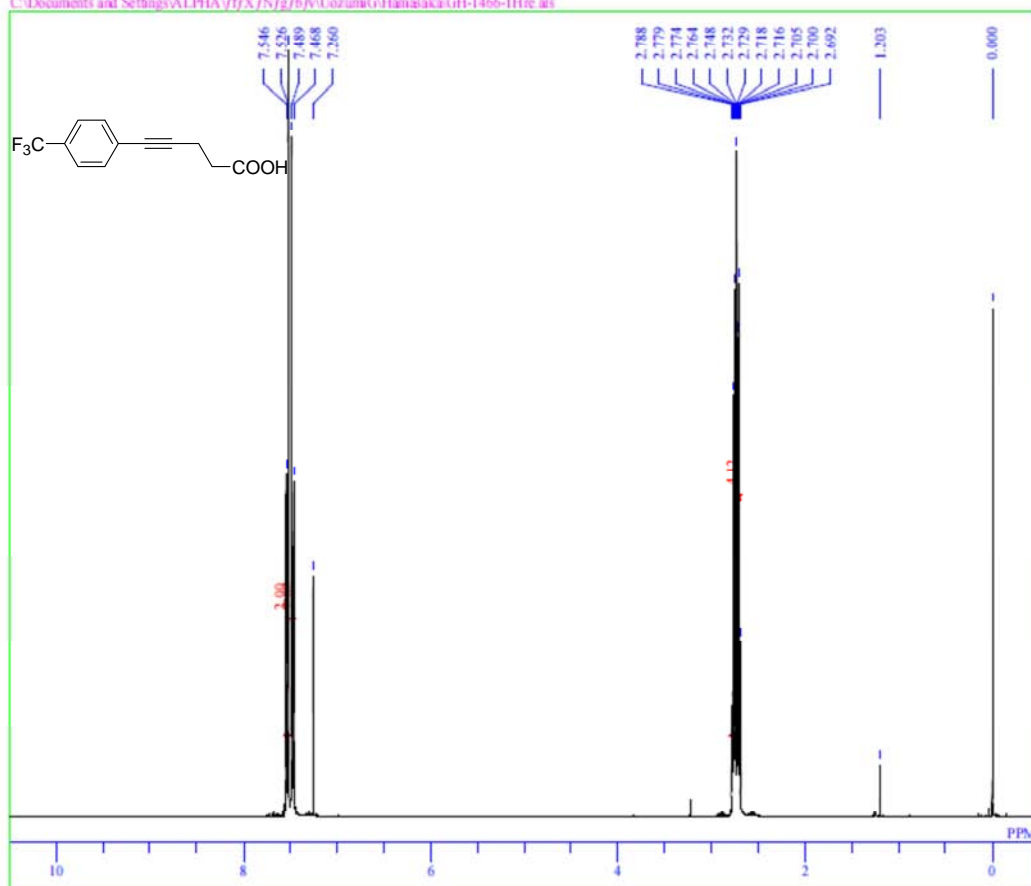


DFILE GH-1493-13C.als
 COMNT GH-1493-13C
 DATIM Tue Mar 05 09:47:03 2013
 OBNUC 13C
 EXMOD SINGL
 OBFREQ 125.65 MHz
 OBSET 0.00 KHz
 OBFIN 127958.00 Hz
 POINT 32768
 FREQU 33898.30 Hz
 SCANS 17701
 ACQTM 0.9667 sec
 PD 1.0000 sec
 PWI 5.75 usec
 IRNUC 13C
 CTEMP 23.9 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.40 Hz
 RGAIN 27

5-(4-trifluoromethylphenyl)-4-pentynoic acid (**2a**)

GH-1466-1H

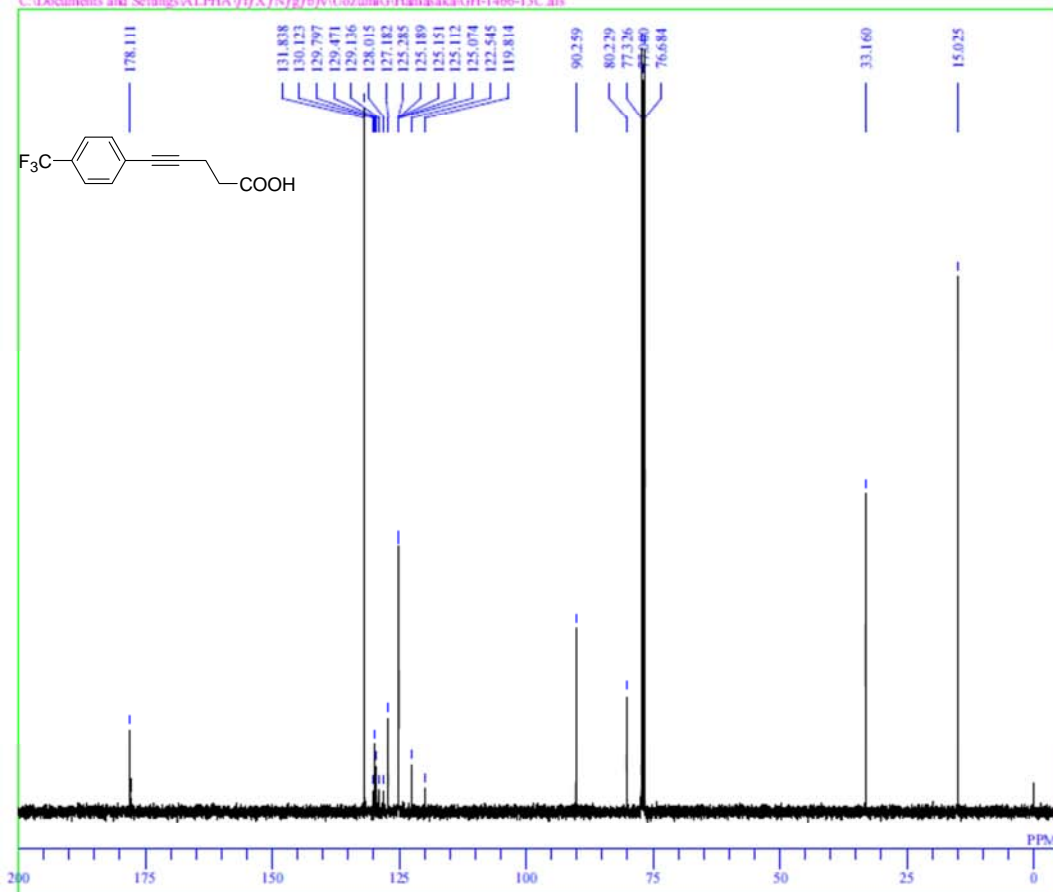
C:\Documents and Settings\ALPHA\fx\N/g/b/A/UozumiGHamasaka\GH-1466-1Hre.als



DFILE GH-1466-1Hre.als
 COMNT GH-1466-1H
 DATIM 2013-07-03 18:53:14
 OBNUC 1H
 EXMOD proton.jxp
 OBFREQ 395.88 MHz
 OBSET 6.28 KHz
 OBFIN 0.87 Hz
 POINT 16384
 FREQU 7422.80 Hz
 SCANS 8
 ACQTM 2.2073 sec
 PD 5.0000 sec
 PWI 3.12 usec
 IRNUC 1H
 CTEMP 19.0 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.00 Hz
 RGAIN 34

GH-1466-13C

C:\Documents and Settings\ALPHA\fx\N/g/b/A/UozumiGHamasaka\GH-1466-13C.als

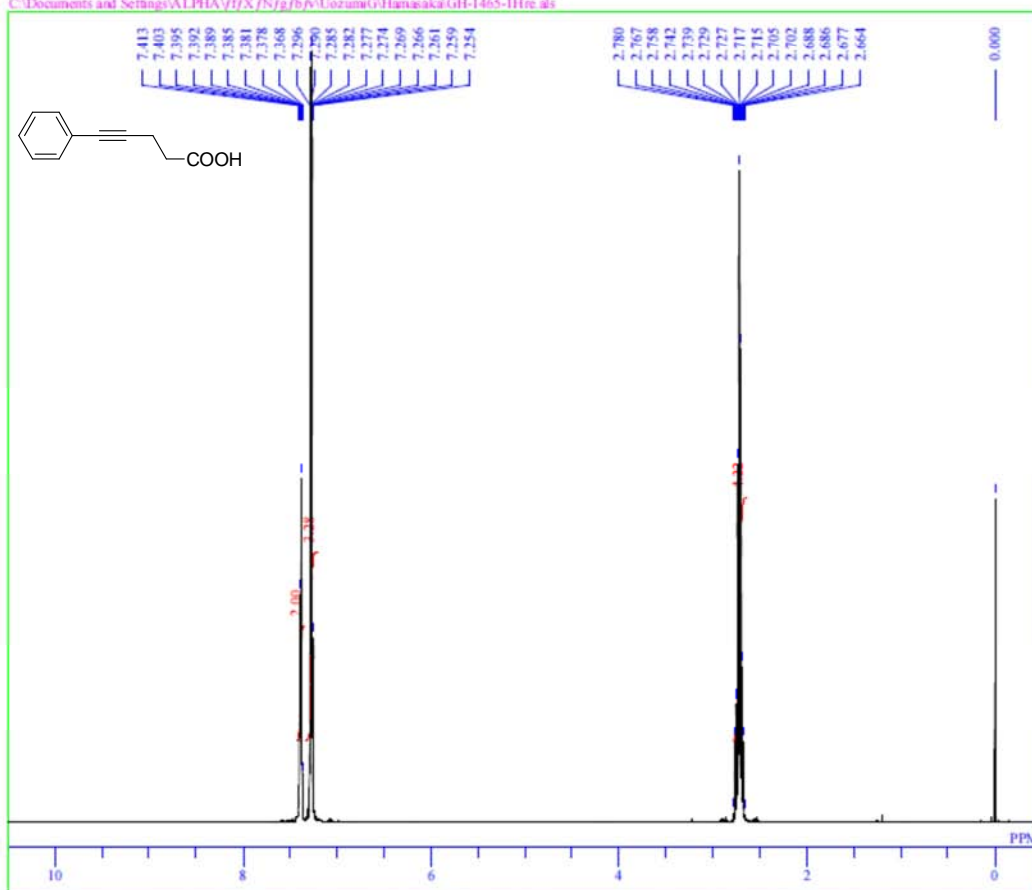


DFILE GH-1466-13C.als
 COMNT GH-1466-13C
 DATIM 2013-07-03 18:55:23
 OBNUC 13C
 EXMOD carbon.jxp
 OBFREQ 99.55 MHz
 OBSET 5.13 KHz
 OBFIN 0.98 Hz
 POINT 32767
 FREQU 31250.00 Hz
 SCANS 614
 ACQTM 1.0486 sec
 PD 2.0000 sec
 PWI 3.42 usec
 IRNUC 13C
 CTEMP 20.1 c
 SLVNT CDCL3
 EXREF 77.00 ppm
 BF 0.50 Hz
 RGAIN 60

5-Phenyl-4-pentynoic acid (**2b**)

GH-1465-1H

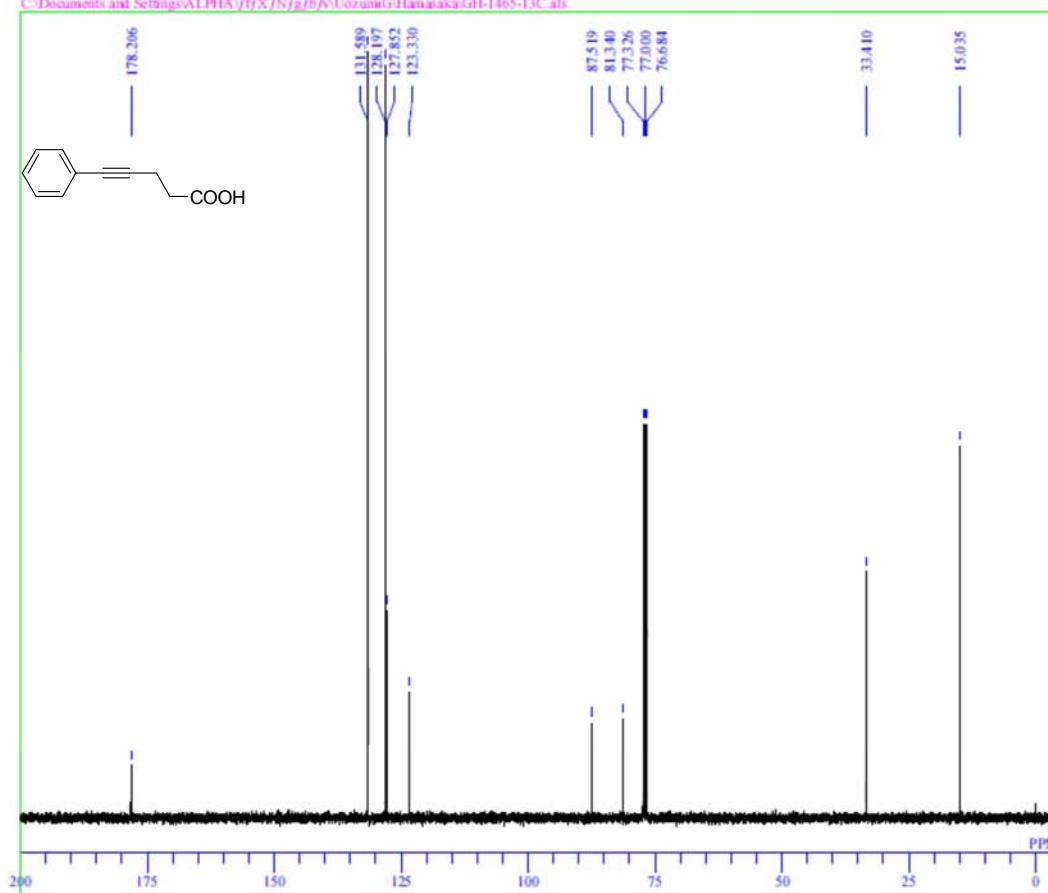
C:\Documents and Settings\ALPHA\1\1X\N/g/b/b/UozumiGHamasaka\GH-1465-1Hre.als



DFILE GH-1465-1Hre.als
 COMNT GH-1465-1H
 DATIM 2013-07-03 22:14:05
 OBNUC 1H
 EXMOD proton.jxp
 OBFREQ 395.88 MHz
 OBSET 6.28 KHz
 OBFIN 0.87 Hz
 POINT 16384
 FREQU 7422.80 Hz
 SCANS 8
 ACQTM 2.2073 sec
 PD 5.0000 sec
 PWI 3.12 usec
 IRNUC 1H
 CTEMP 19.2 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.00 Hz
 RGAIN 32

GH-1465-13C

C:\Documents and Settings\ALPHA\1\1X\N/g/b/b/UozumiGHamasaka\GH-1465-13C.als

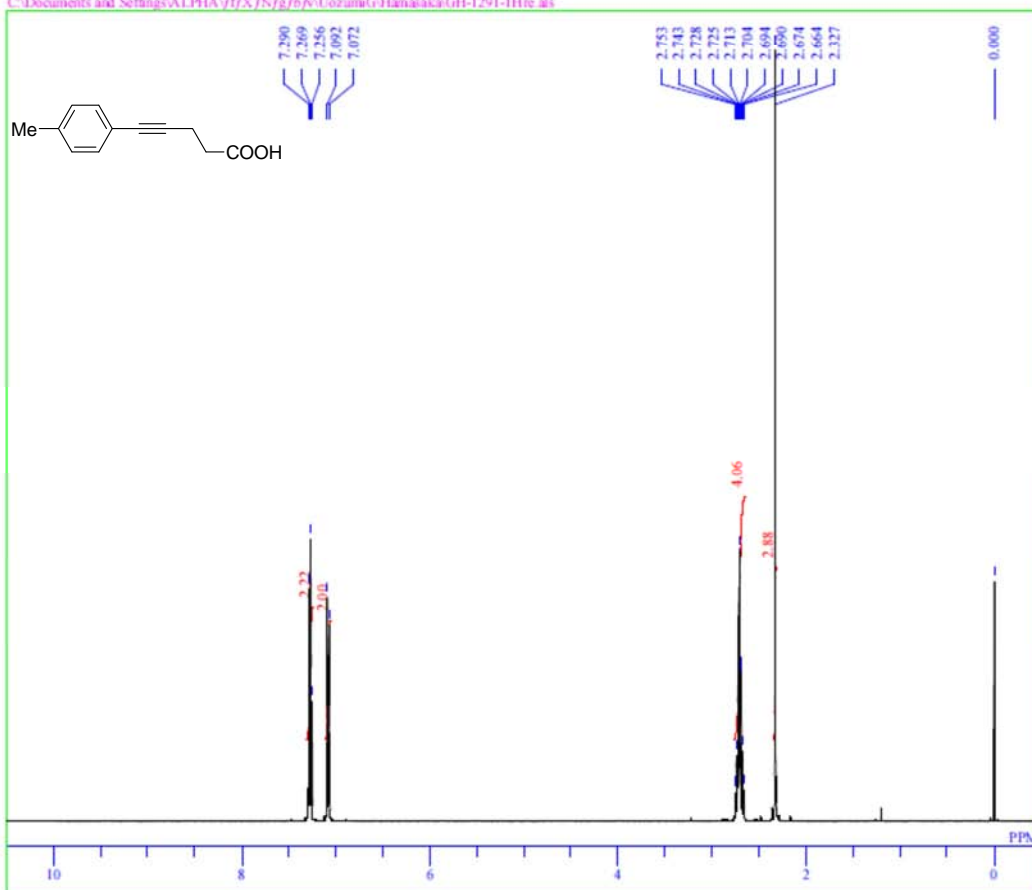


DFILE GH-1465-13C.als
 COMNT GH-1465-13C
 DATIM 2013-07-03 22:16:24
 OBNUC 13C
 EXMOD carbon.jxp
 OBFREQ 99.55 MHz
 OBSET 5.13 KHz
 OBFIN 0.98 Hz
 POINT 32767
 FREQU 31250.00 Hz
 SCANS 317
 ACQTM 1.0486 sec
 PD 2.0000 sec
 PWI 3.42 usec
 IRNUC 13C
 CTEMP 19.3 c
 SLVNT CDCL3
 EXREF 77.00 ppm
 BF 0.50 Hz
 RGAIN 60

5-(4-methylphenyl)-4-pentynoic acid (**2c**)

GH-1291-1H

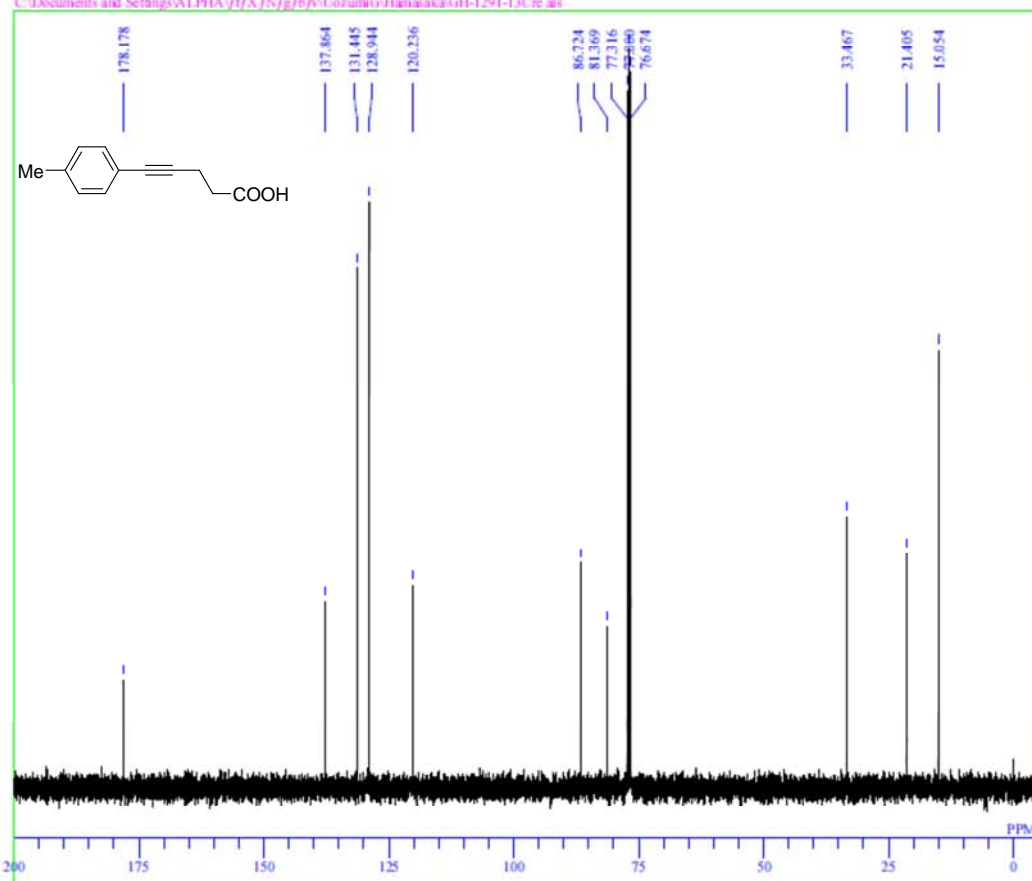
C:\Documents and Settings\ALPHA\ff\X\N\g\b\A\Uozumi\G\Hanaka\GH-1291-1Hre.als



DFILE GH-1291-1Hre.als
 COMNT GH-1291-1H
 DATM 2013-07-04 12:27:30
 OBNUC 1H
 EXMOD proton.jsp
 OBFREQ 395.88 MHz
 OBSFET 6.28 KHz
 OBFIN 0.87 Hz
 POINT 16384
 FREQU 7422.80 Hz
 SCANS 8
 ACQTM 2.2073 sec
 PD 5.0000 sec
 PWI 3.12 usec
 IRNUC 1H
 CTEMP 19.5 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.00 Hz
 RGAIN 32

GH-1291-13C

C:\Documents and Settings\ALPHA\ff\X\N\g\b\A\Uozumi\G\Hanaka\GH-1291-13Cre.als

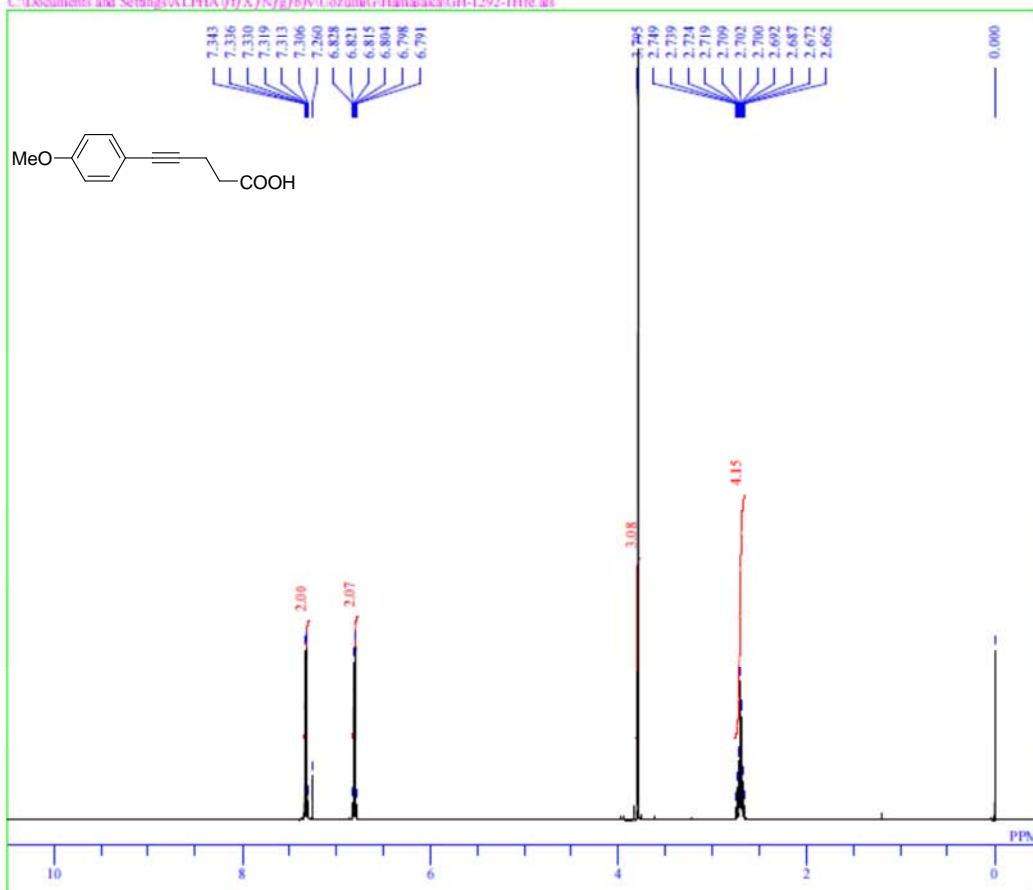


DFILE GH-1291-13Cre.als
 COMNT GH-1291-13C
 DATM 2013-07-04 12:29:52
 OBNUC 13C
 EXMOD carbon.jsp
 OBFREQ 99.55 MHz
 OBSFET 5.13 KHz
 OBFIN 0.98 Hz
 POINT 32767
 FREQU 31250.00 Hz
 SCANS 129
 ACQTM 1.0486 sec
 PD 2.0000 sec
 PWI 3.42 usec
 IRNUC 13C
 CTEMP 19.2 c
 SLVNT CDCL3
 EXREF 77.00 ppm
 BF 0.50 Hz
 RGAIN 60

5-(4-methoxyphenyl)-4-pentynoic acid (**2d**)

GH-1292-1H

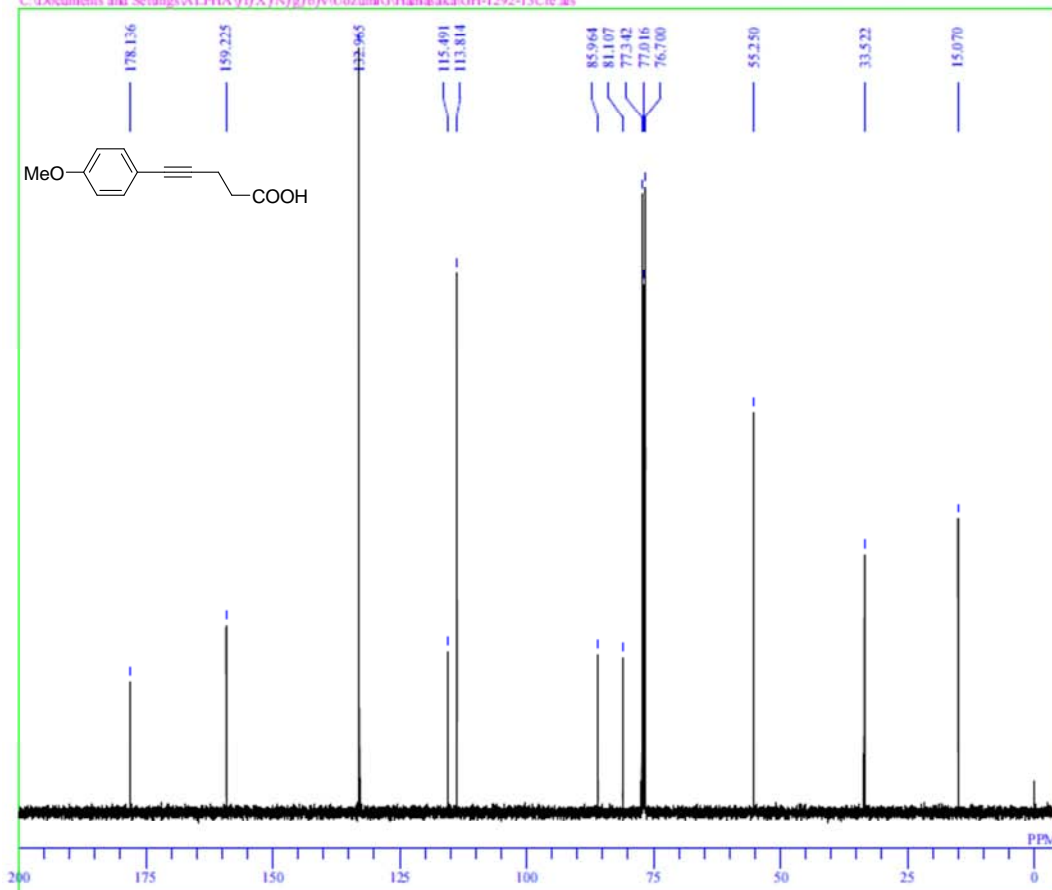
C:\Documents and Settings\ALPHA\ffX\N/g/b/A/UozumiG\Hanasaka\GH-1292-1Hre als



DFILE GH-1292-1Hre als
 COMNT GH-1292-1H
 DATIM 2013-07-04 12:42:22
 OBNUC 1H
 EXMOD proton.jsp
 OBFREQ 395.88 MHz
 OBSET 6.28 KHz
 OBFIN 0.87 Hz
 POINT 13107
 FREQU 5938.24 Hz
 SCANS 8
 ACQTM 2.2073 sec
 PD 5.0000 sec
 PWI 3.12 usec
 IRNUC 1H
 CTEMP 19.0 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.00 Hz
 RGAIN 34

GH-1292-13C

C:\Documents and Settings\ALPHA\ffX\N/g/b/A/UozumiG\Hanasaka\GH-1292-13Cre als

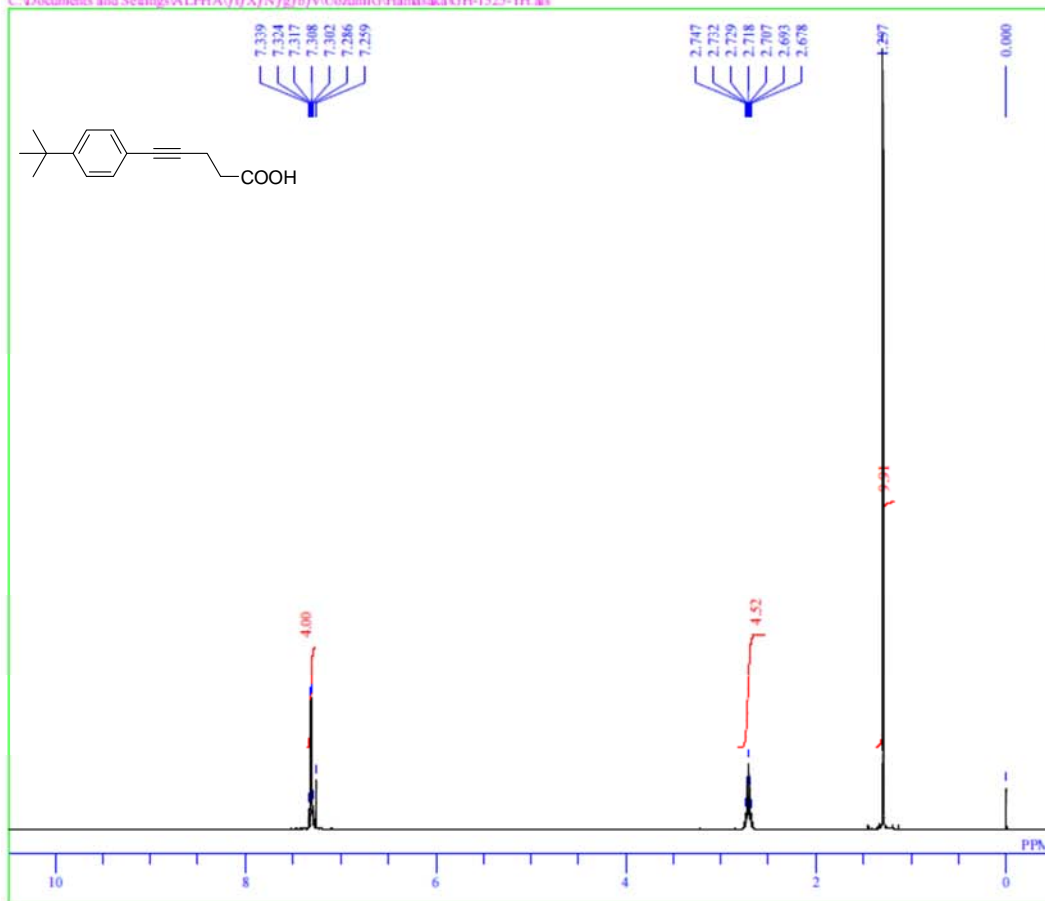


DFILE GH-1292-13Cre als
 COMNT GH-1292-13C
 DATIM 2013-07-04 12:44:42
 OBNUC 13C
 EXMOD carbon.jsp
 OBFREQ 99.55 MHz
 OBSET 5.13 KHz
 OBFIN 0.98 Hz
 POINT 32767
 FREQU 31250.00 Hz
 SCANS 477
 ACQTM 1.0486 sec
 PD 2.0000 sec
 PWI 3.42 usec
 IRNUC 13C
 CTEMP 19.0 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.50 Hz
 RGAIN 60

5-(4-tert-butylphenyl)-4-pentynoic acid (**2e**)

GH-1525-1H

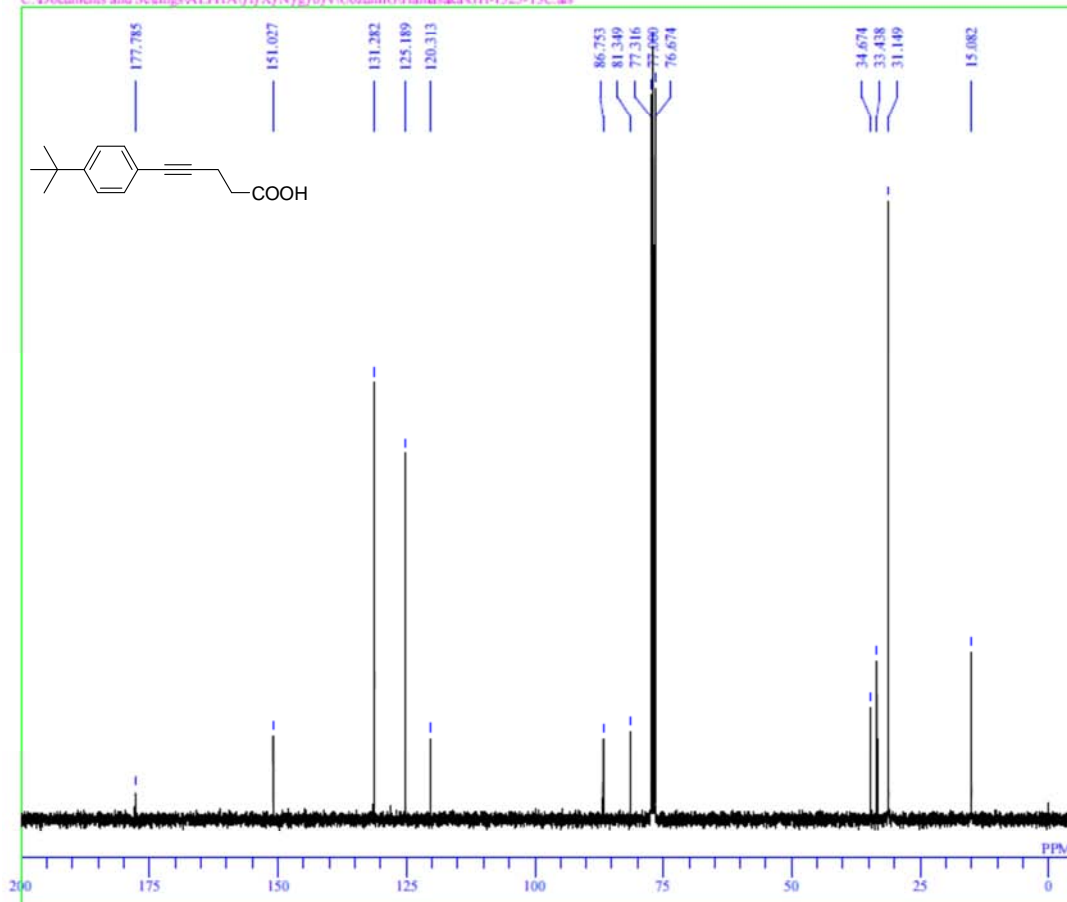
C:\Documents and Settings\ALPHA\ff\X\N\g\fb\Uozumi\GHamasaka\GH-1525-1H.als



DFILE GH-1525-1H.als
 COMNT GH-1525-1H
 DATIM 2013-05-07 22:24:21
 OBNUC 1H
 EXMOD proton.jpg
 OBFRQ 395.88 MHz
 OBSET 6.28 KHz
 OBFIN 0.87 Hz
 POINT 16384
 FREQU 7422.80 Hz
 SCANS 8
 ACQTM 2.2073 sec
 PD 5.0000 sec
 PWI 3.12 usec
 IRNUC 1H
 CTEMP 18.9 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.00 Hz
 RGAIN 34

GH-1525-13C

C:\Documents and Settings\ALPHA\ff\X\N\g\fb\Uozumi\GHamasaka\GH-1525-13C.als

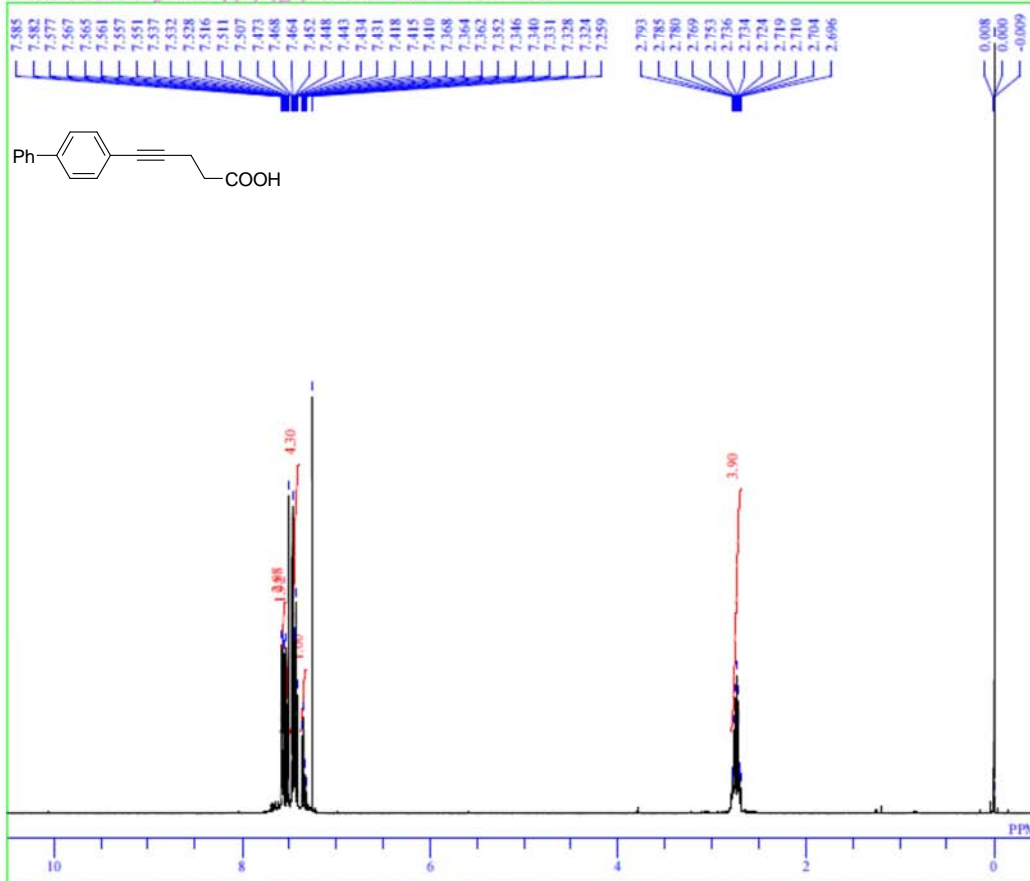


DFILE GH-1525-13C.als
 COMNT GH-1525-13C
 DATIM 2013-05-07 22:27:23
 OBNUC 13C
 EXMOD carbon.jpg
 OBFRQ 99.55 MHz
 OBSET 5.13 KHz
 OBFIN 0.98 Hz
 POINT 32767
 FREQU 31250.00 Hz
 SCANS 458
 ACQTM 1.0486 sec
 PD 2.0000 sec
 PWI 3.42 usec
 IRNUC 13C
 CTEMP 18.8 c
 SLVNT CDCL3
 EXREF 77.00 ppm
 BF 1.00 Hz
 RGAIN 60

5-(4-phenylphenyl)-4-pentynoic acid (**2f**)

GH-1342-1H

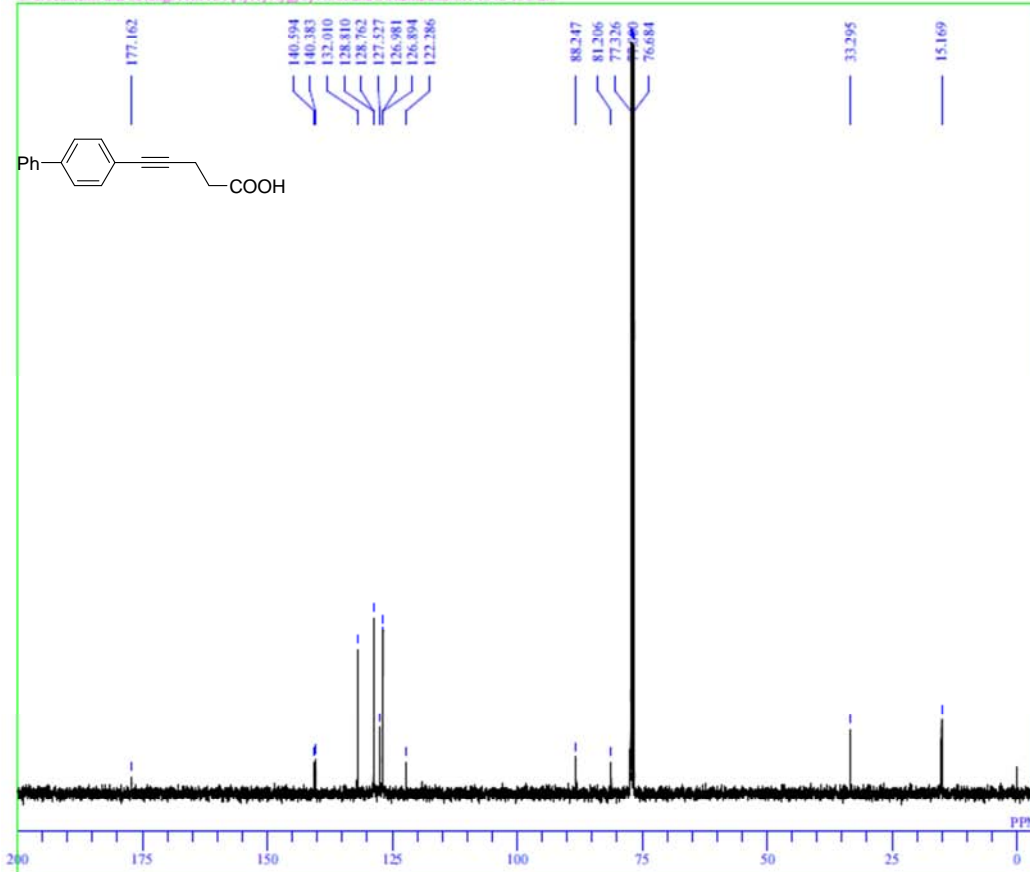
C:\Documents and Settings\ALPHA\ff\X\N\g\fb\Uozumi\GH-1342-1Hre.als



DFILE GH-1342-1Hre.als
 COMNT GH-1342-1H
 DATIM 2013-07-04 13:48:58
 OBNUC 1H
 EXMOD proton.jsp
 OBFREQ 395.88 MHz
 OBSSET 6.28 KHz
 OBFIN 0.87 Hz
 POINT 16384
 FREQU 7422.80 Hz
 SCANS 8
 ACQTM 2.2073 sec
 PD 5.0000 sec
 PWI 3.12 usec
 IRNUC 1H
 CTEMP 19.1 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.00 Hz
 RGAIN 40

GH-1342-13C

C:\Documents and Settings\ALPHA\ff\X\N\g\fb\Uozumi\GH-1342-13C.als

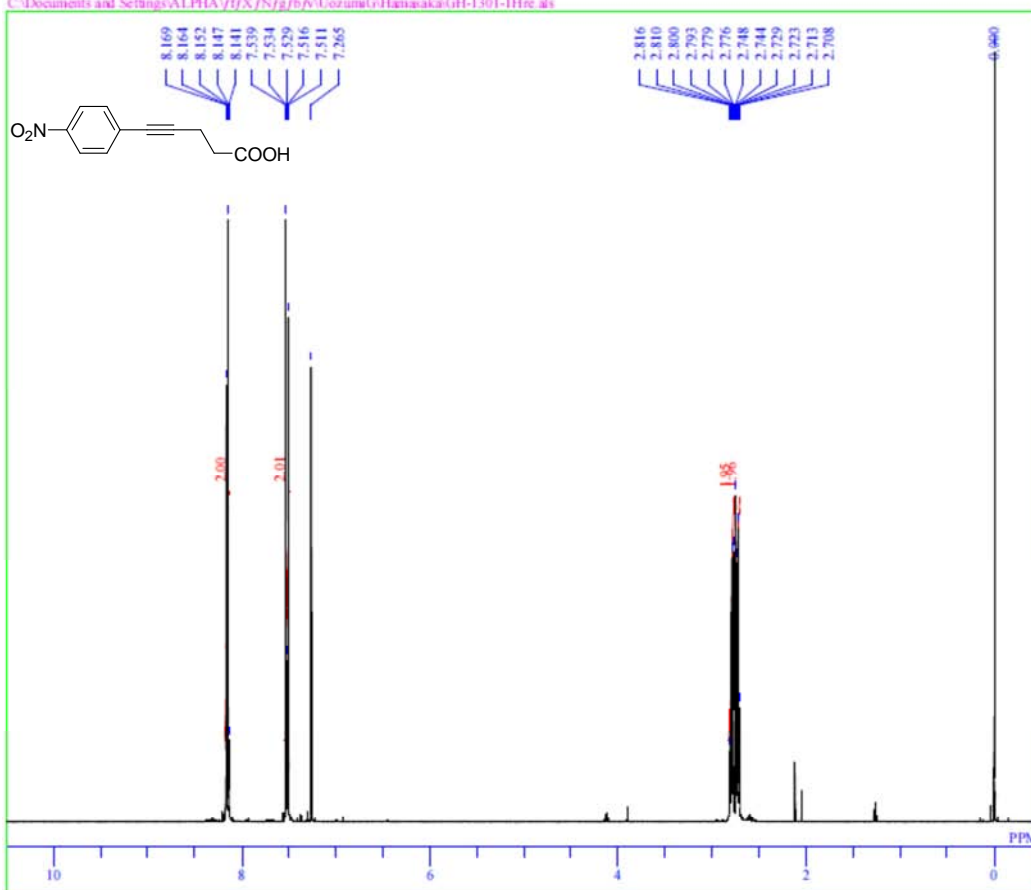


DFILE GH-1342-13C.als
 COMNT GH-1342-13C
 DATIM 2013-07-04 13:50:57
 OBNUC 13C
 EXMOD carbon.jsp
 OBFREQ 99.55 MHz
 OBSSET 5.13 KHz
 OBFIN 0.98 Hz
 POINT 32767
 FREQU 31250.00 Hz
 SCANS 461
 ACQTM 1.0486 sec
 PD 2.0000 sec
 PWI 3.42 usec
 IRNUC 1H
 CTEMP 19.7 c
 SLVNT CDCL3
 EXREF 77.00 ppm
 BF 1.50 Hz
 RGAIN 60

5-(4-nitrophenyl)-4-pentynoic acid (**2g**)

GH-1301-1H

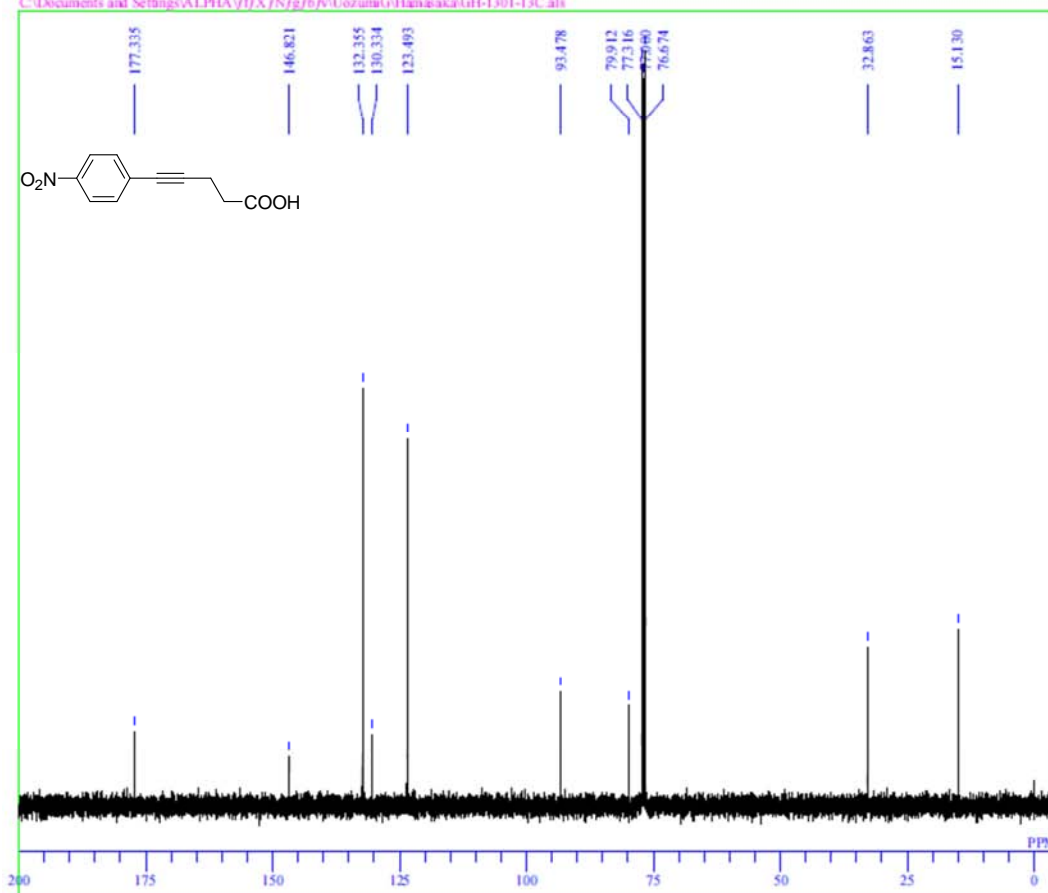
C:\Documents and Settings\ALPHA\I\X\N/g/b/v/UozumiG\Hamasaki\GH-1301-1Hre.als



DFILE GH-1301-1Hre.als
 COMNT GH-1301-1H
 DATIM 2013-07-04 12:07:13
 OBNUC 1H
 EXMOD proton.jsp
 OBFRQ 395.88 MHz
 OBSET 6.28 KHz
 OBFIN 0.87 Hz
 POINT 16384
 FREQU 7422.80 Hz
 SCANS 8
 ACQTM 2.2073 sec
 PD 5.0000 sec
 PWI 3.12 usec
 IRNUC 1H
 CTEMP 19.5 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.00 Hz
 RGAIN 40

GH-1301-13C

C:\Documents and Settings\ALPHA\I\X\N/g/b/v/UozumiG\Hamasaki\GH-1301-13C.als

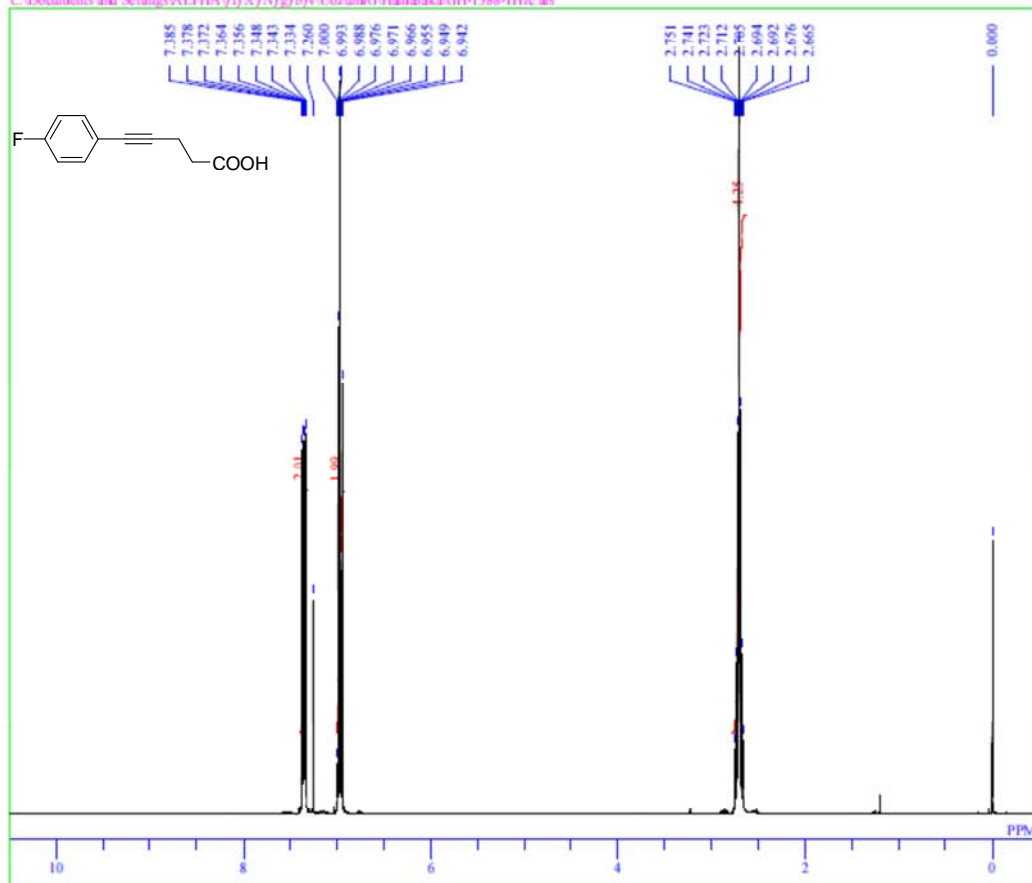


DFILE GH-1301-13C.als
 COMNT GH-1301-13C
 DATIM 2013-07-04 12:09:46
 OBNUC 13C
 EXMOD carbon.jsp
 OBFRQ 99.55 MHz
 OBSET 5.13 KHz
 OBFIN 0.98 Hz
 POINT 32767
 FREQU 31250.00 Hz
 SCANS 215
 ACQTM 1.0486 sec
 PD 2.0000 sec
 PWI 3.42 usec
 IRNUC 13H
 CTEMP 19.1 c
 SLVNT CDCL3
 EXREF 77.00 ppm
 BF 0.50 Hz
 RGAIN 60

5-(4-fluorophenyl)-4-pentynoic acid (**2h**)

GH-1388-1H1

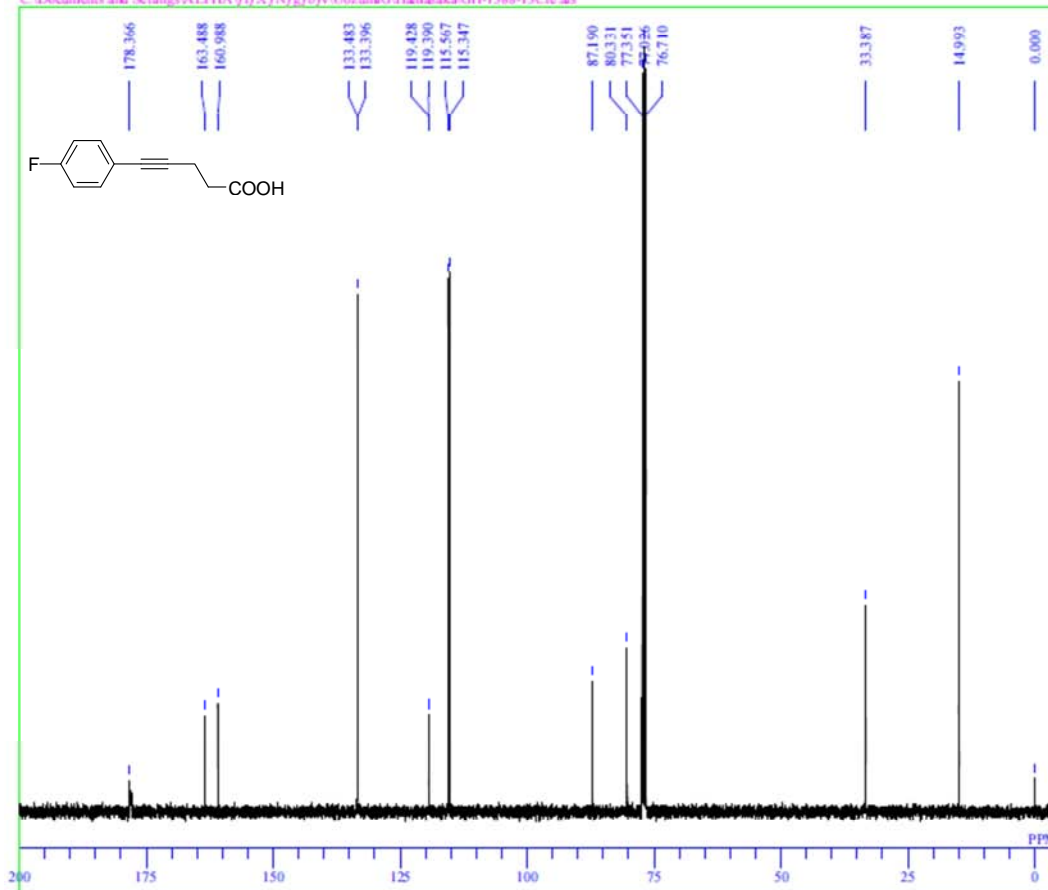
C:\Documents and Settings\ALPHA\1\1\X\N/g/b/b/Uozumi\GHamasaka\GH-1388-1Hre.als



DFILE GH-1388-1Hre.als
 COMNT GH-1388-1H
 DATIM 2013-07-03 18:16:02
 OBNUC 1H
 EXMOD proton.jsp
 OBFREQ 395.88 MHz
 OBSSET 6.28 KHz
 OBFIN 0.87 Hz
 POINT 16384
 FREQU 7422.80 Hz
 SCANS 8
 ACQTM 2.2073 sec
 PD 5.0000 sec
 PWI 3.12 usec
 IRNUC 1H
 CTEMP 19.4 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.00 Hz
 RGAIN 34

GH-1388-13C

C:\Documents and Settings\ALPHA\1\1\X\N/g/b/b/Uozumi\GHamasaka\GH-1388-13Cre.als

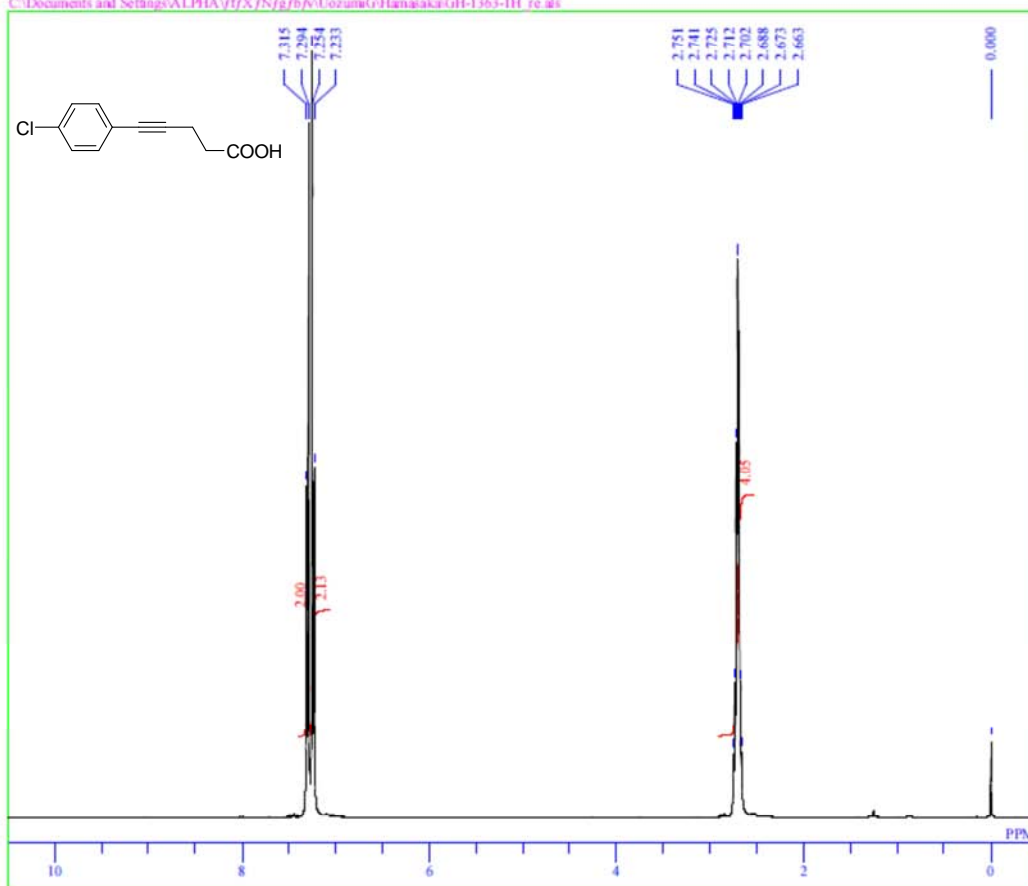


DFILE GH-1388-13Cre.als
 COMNT GH-1388-13C
 DATIM 2013-07-03 18:18:14
 OBNUC 13C
 EXMOD carbon.jsp
 OBFREQ 99.55 MHz
 OBSSET 5.13 KHz
 OBFIN 0.98 Hz
 POINT 32767
 FREQU 31250.00 Hz
 SCANS 565
 ACQTM 1.0486 sec
 PD 2.0000 sec
 PWI 3.42 usec
 IRNUC 1H
 CTEMP 19.6 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 1.00 Hz
 RGAIN 60

5-(4-chlorophenyl)-4-pentynoic acid (**2i**)

GH-1363-1H

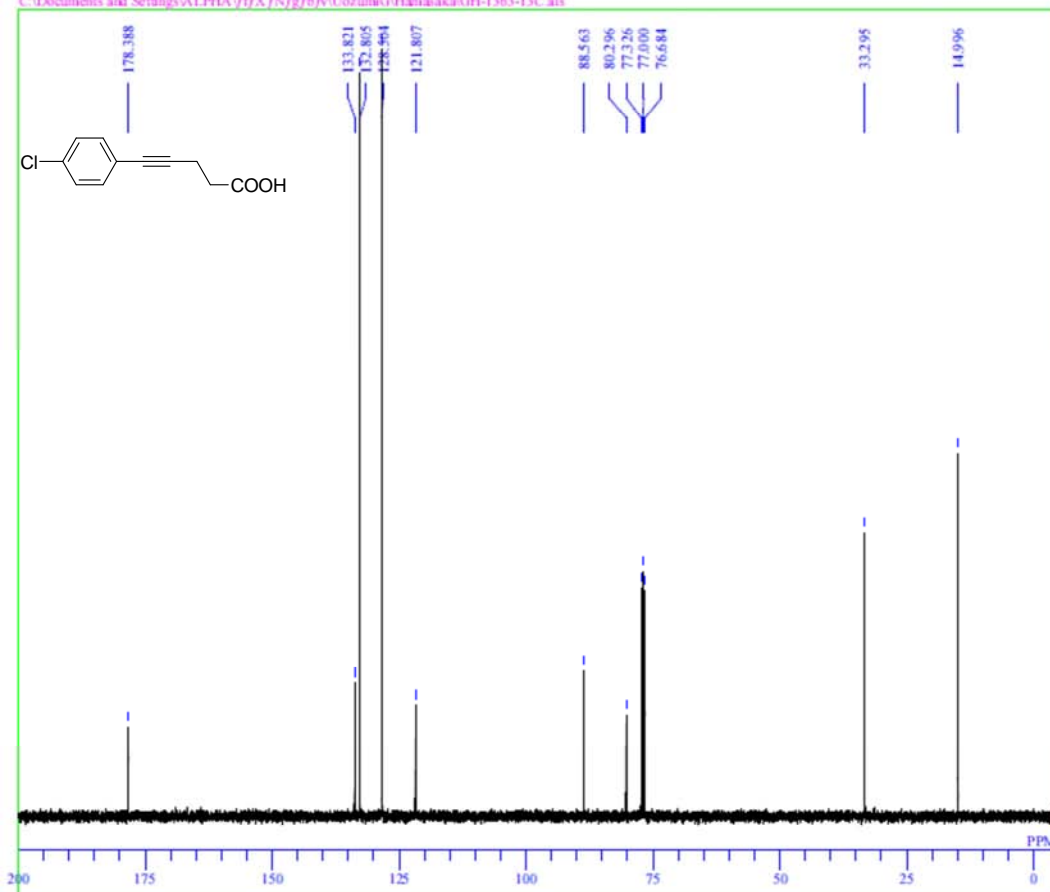
C:\Documents and Settings\ALPHA\fx\N/g/b/A/UozumiG\Hamasaki\GH-1363-1H_re.als



DFILE GH-1363-1H_re.als
 COMNT GH-1363-1H
 DATIM 2013-07-03 16:51:51
 OBNUC 1H
 EXMOD proton.jxp
 OBFREQ 395.88 MHz
 OBSET 6.28 KHz
 OBFIN 0.87 Hz
 POINT 16384
 FREQU 7422.80 Hz
 SCANS 8
 ACQTM 2.2073 sec
 PD 5.0000 sec
 PWI 3.12 usec
 IRNUC 1H
 CTEMP 20.7 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.00 Hz
 RGAIN 28

GH-1363-13C

C:\Documents and Settings\ALPHA\fx\N/g/b/A/UozumiG\Hamasaki\GH-1363-13C.als

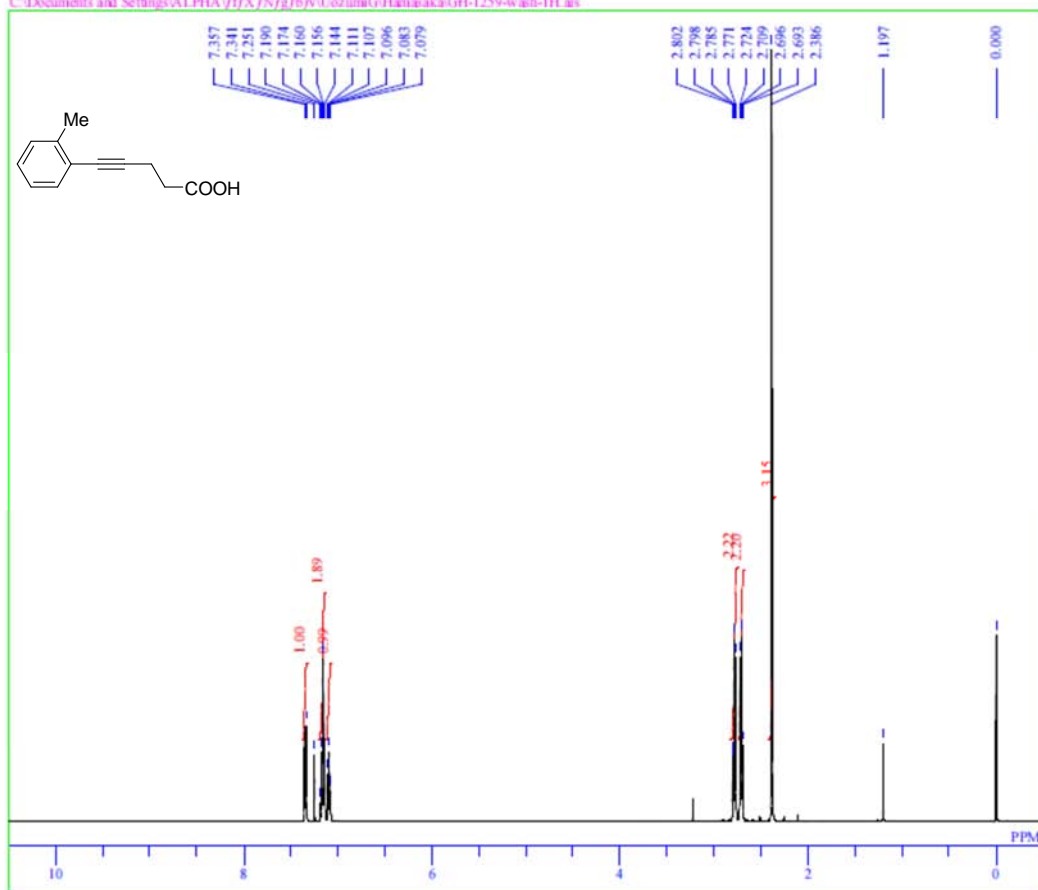


DFILE GH-1363-13C.als
 COMNT GH-1363-13C
 DATIM 2013-07-03 16:53:46
 OBNUC 13C
 EXMOD carbon.jxp
 OBFREQ 99.55 MHz
 OBSET 5.13 KHz
 OBFIN 0.98 Hz
 POINT 32767
 FREQU 31250.00 Hz
 SCANS 112
 ACQTM 1.0486 sec
 PD 2.0000 sec
 PWI 3.42 usec
 IRNUC 1H
 CTEMP 19.6 c
 SLVNT CDCL3
 EXREF 77.00 ppm
 BF 0.50 Hz
 RGAIN 60

5-(2-methylphenyl)-4-pentynoic acid (**2j**)

GH-1259-wash-1H

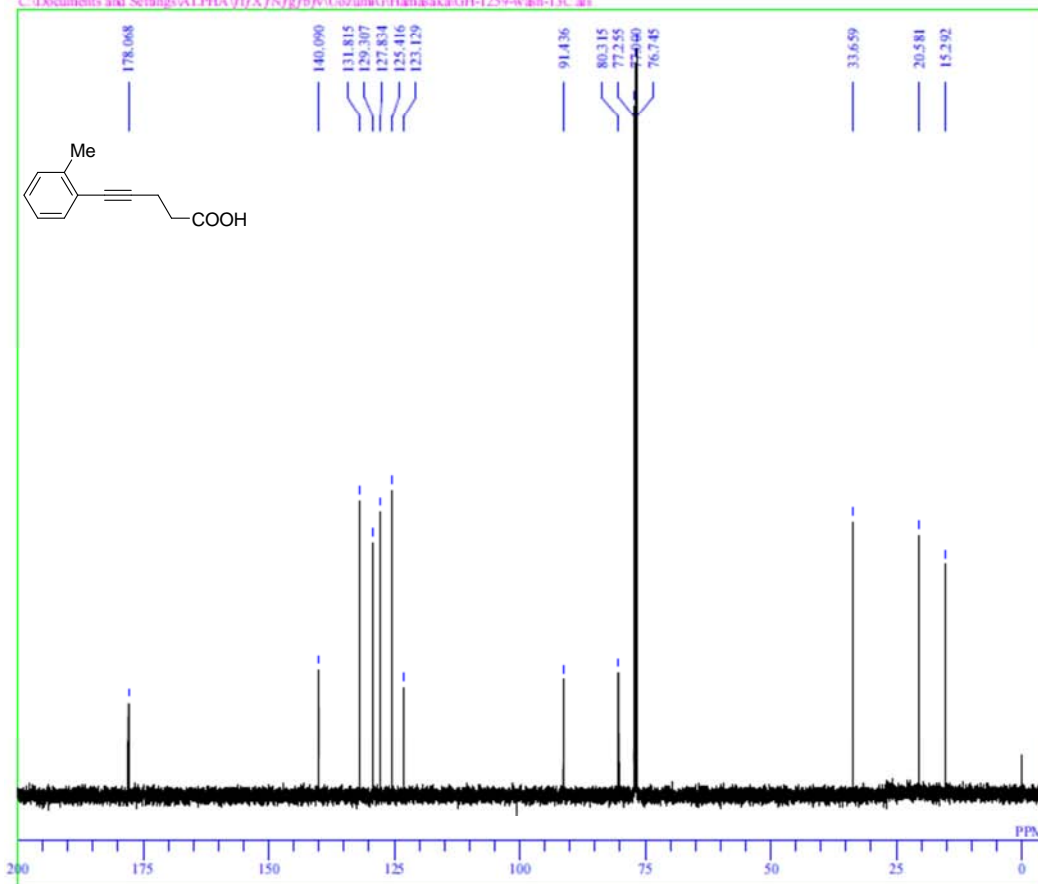
C:\Documents and Settings\ALPHA\ff\X\N\g\fb\A\Uozumi\GH\Hamakaki\GH-1259-wash-1H.als



DFILE GH-1259-wash-1H.als
 COMNT GH-1259-wash-1H
 DATIM Mon Aug 20 16:18:46 2012
 OBNUC 1H
 EXMOD SINGL
 OBFREQ 500.00 MHz
 OBSSET 0.00 KHz
 OBFIN 162410.00 Hz
 POINT 16384
 FREQU 10000.00 Hz
 SCANS 16
 ACQTM 1.6384 sec
 PD 2.0000 sec
 PWI 6.40 usec
 IRNUC 1H
 CTEMP 24.3 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.00 Hz
 RGAIN 16

GH-1259-wash-13C

C:\Documents and Settings\ALPHA\ff\X\N\g\fb\A\Uozumi\GH\Hamakaki\GH-1259-wash-13C.als

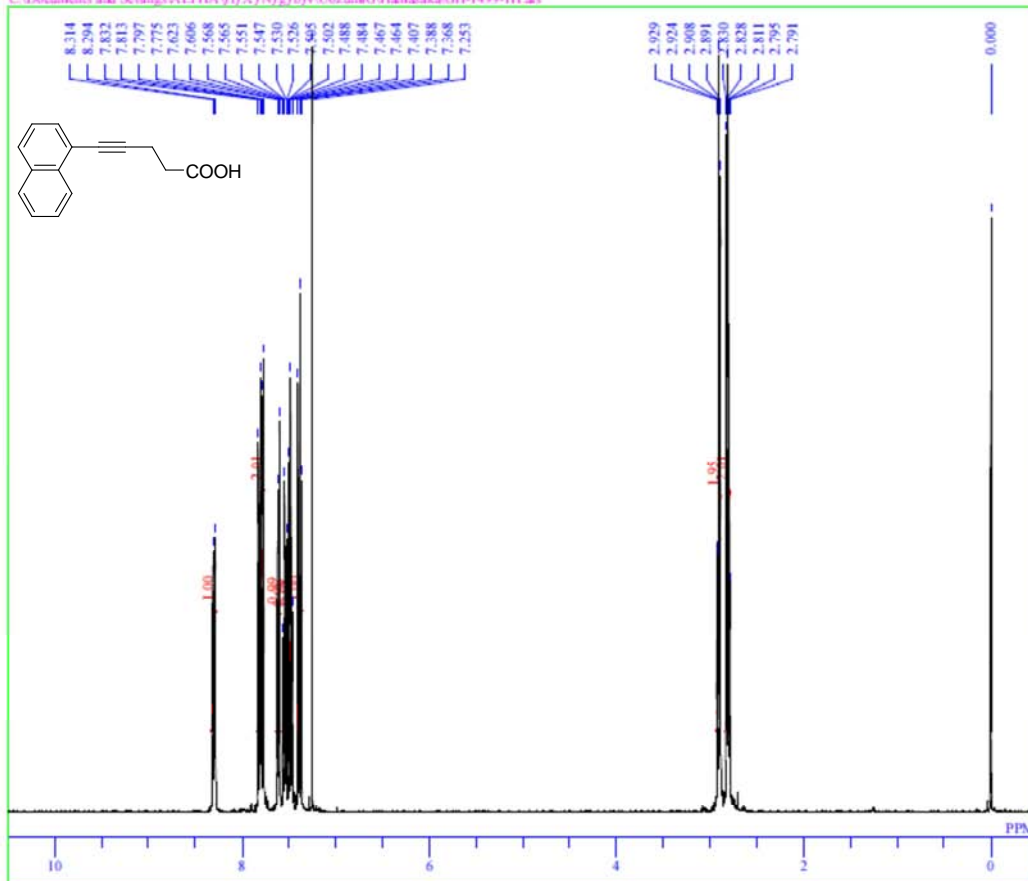


DFILE GH-1259-wash-13C.als
 COMNT GH-1259-wash-13C
 DATIM Mon Aug 20 16:48:13 2012
 OBNUC 13C
 EXMOD SINGL
 OBFREQ 125.65 MHz
 OBSSET 0.00 KHz
 OBFIN 127958.00 Hz
 POINT 32768
 FREQU 33898.30 Hz
 SCANS 63
 ACQTM 0.9667 sec
 PD 1.0000 sec
 PWI 5.75 usec
 IRNUC 13C
 CTEMP 26.4 c
 SLVNT CDCL3
 EXREF 77.00 ppm
 BF 0.50 Hz
 RGAIN 26

5-(1-naphthyl)-4-pentynoic acid (**2k**)

GH-1499-1H

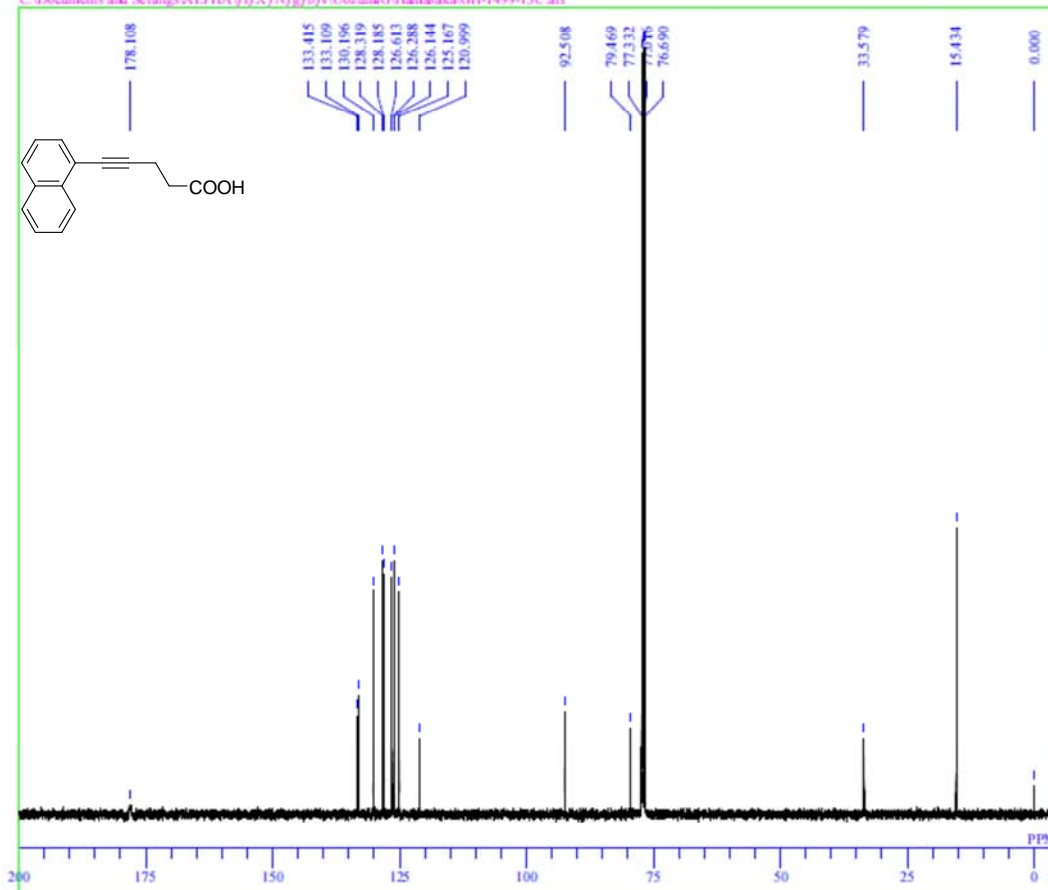
C:\Documents and Settings\ALPHA\ff\X\N/g/b/A/UozumiG\Hamasaki\GH-1499-1H.als



DFILE GH-1499-1H.als
 COMNT GH-1499-1H
 DATIM 2013-07-03 17:05:56
 OBNUC 1H
 EXMOD proton.jxp
 OBFREQ 395.88 MHz
 OBSET 6.28 KHz
 OBFIN 0.87 Hz
 POINT 16384
 FREQU 7422.80 Hz
 SCANS 8
 ACQTM 2.2073 sec
 PD 5.0000 sec
 PW1 3.12 usec
 IRNUC 1H
 CTEMP 19.3 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.00 Hz
 RGAIN 38

GH-1499-13C

C:\Documents and Settings\ALPHA\ff\X\N/g/b/A/UozumiG\Hamasaki\GH-1499-13C.als

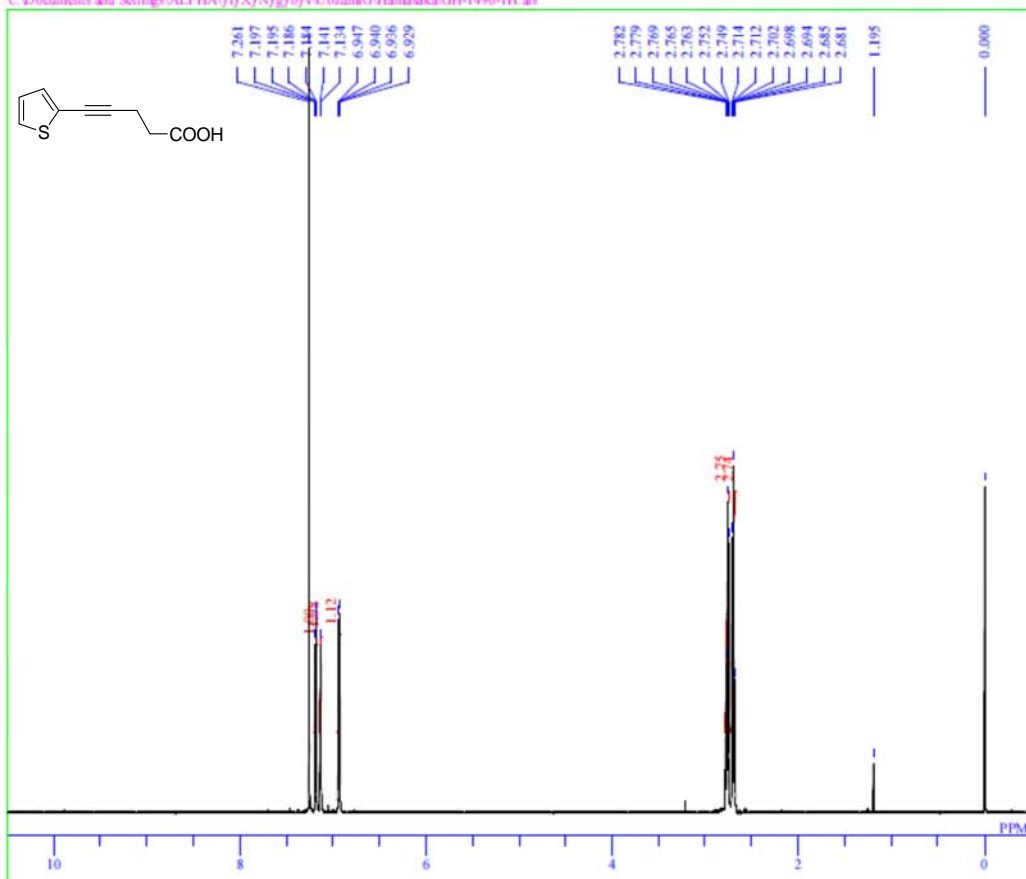


DFILE GH-1499-13C.als
 COMNT GH-1499-13C
 DATIM 2013-07-03 17:08:03
 OBNUC 13C
 EXMOD carbon.jxp
 OBFREQ 99.55 MHz
 OBSET 5.13 KHz
 OBFIN 0.98 Hz
 POINT 32767
 FREQU 31250.00 Hz
 SCANS 1174
 ACQTM 1.0486 sec
 PD 2.0000 sec
 PW1 3.42 usec
 IRNUC 1H
 CTEMP 19.2 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 1.00 Hz
 RGAIN 60

5-(2-thienyl)-4-pentynoic acid (**21**)

GH-1496-1H

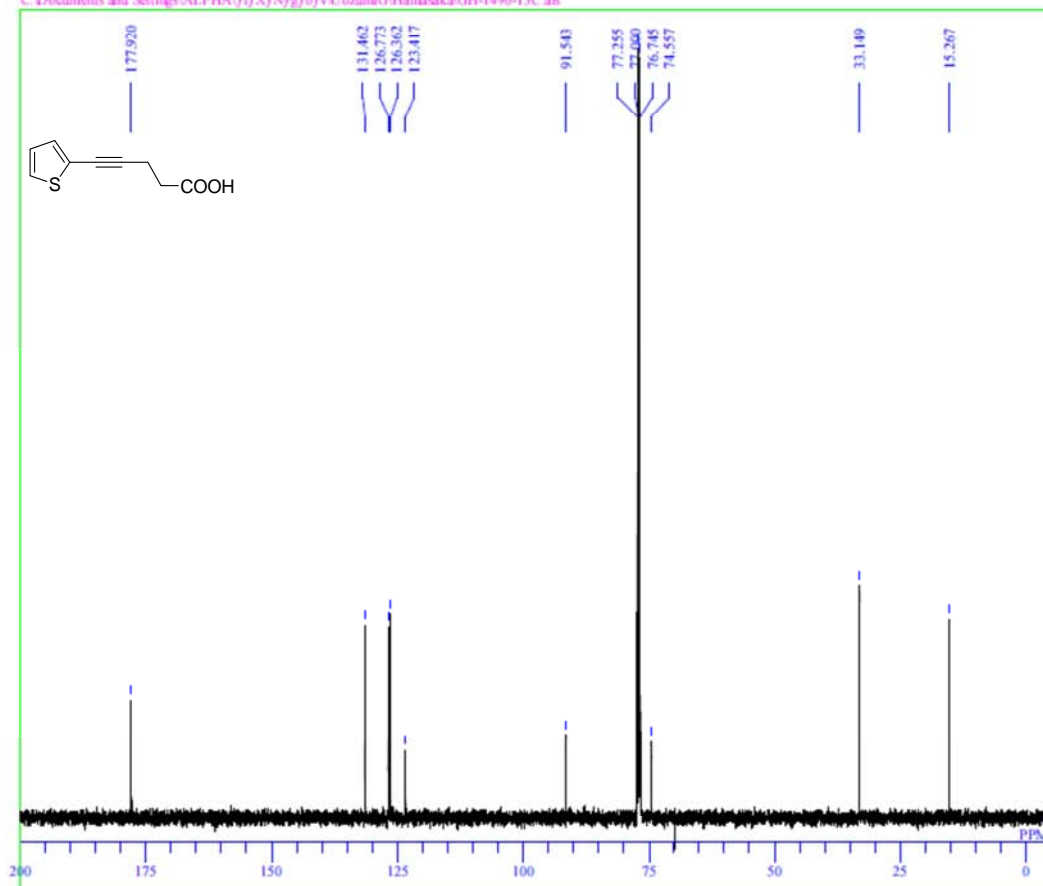
C:\Documents and Settings\ALPHA\ff\X\N\g\b\U\ozami\G\Hamasaki\GH-1496-1H.als



DFILE GH-1496-1H.als
 COMNT GH-1496-1H
 DATIM Thu Mar 07 21:18:32 2013
 OBNUC 1H
 EXMOD SINGL
 OBFREQ 500.00 MHz
 OBSET 0.00 KHz
 OBFIN 162410.00 Hz
 POINT 16384
 FREQU 10000.00 Hz
 SCANS 16
 ACQTM 1.6384 sec
 PD 2.0000 sec
 PWI 6.40 usec
 IRNUC 1H
 CTEMP 21.9 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.00 Hz
 RGAIN 26

GH-1496-13C

C:\Documents and Settings\ALPHA\ff\X\N\g\b\U\ozami\G\Hamasaki\GH-1496-13C.als

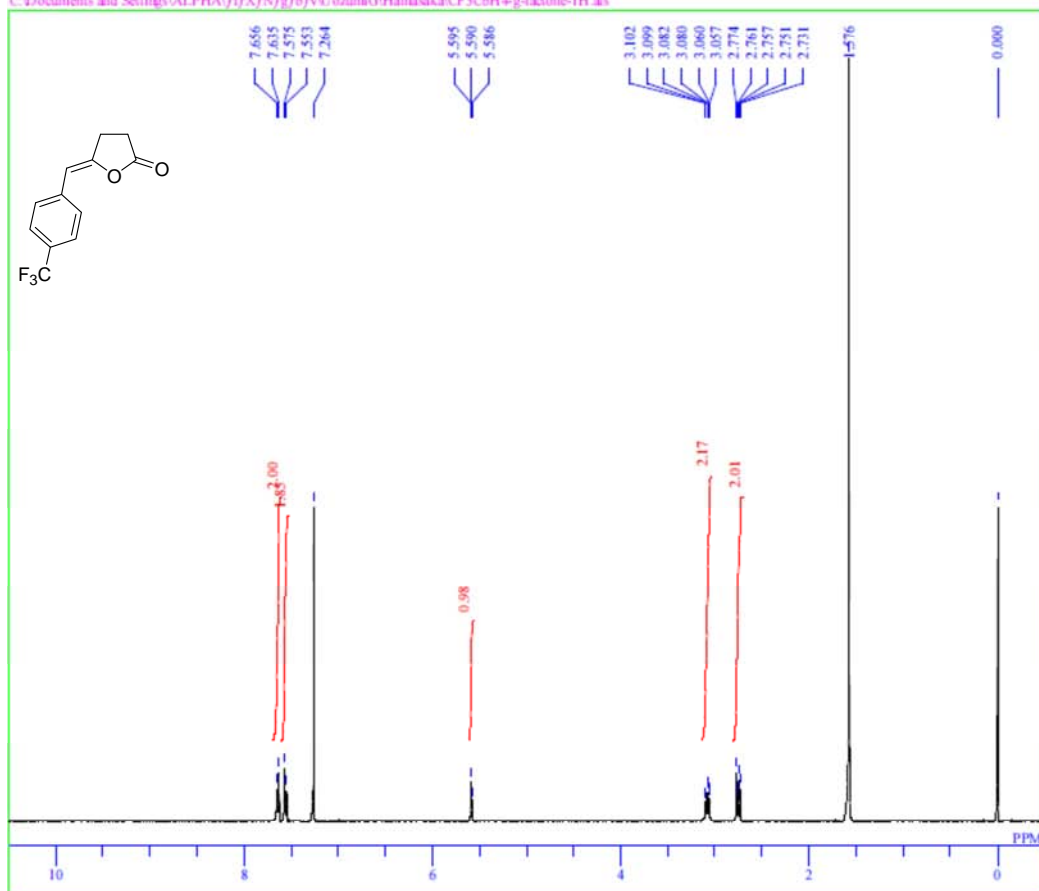


DFILE GH-1496-13C.als
 COMNT GH-1496-13C
 DATIM Mon Mar 11 14:31:58 2013
 OBNUC 13C
 EXMOD SINGL
 OBFREQ 125.65 MHz
 OBSET 0.00 KHz
 OBFIN 127958.00 Hz
 POINT 32768
 FREQU 33898.30 Hz
 SCANS 1101
 ACQTM 0.9667 sec
 PD 1.0000 sec
 PWI 5.75 usec
 IRNUC 1H
 CTEMP 23.8 c
 SLVNT CDCL3
 EXREF 77.00 ppm
 BF 1.50 Hz
 RGAIN 26

(Z)- γ -(4-Trifluoromethylbenzylidene)- γ -butyrolactone (**3a**)

F3CC6H4-g-lactone-1H

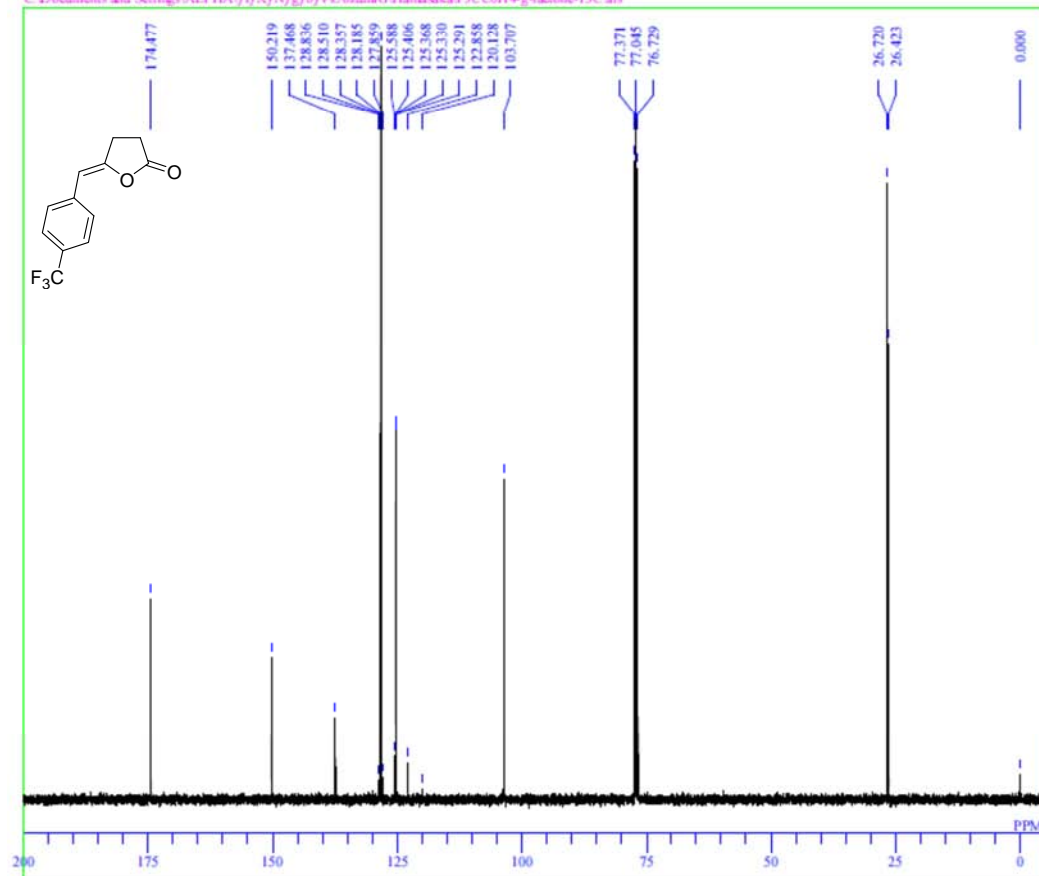
C:\Documents and Settings\ALPHA\ff\X\N\g\h\y\U\ozumi\G\Hamasaki\F3CC6H4-g-lactone-1H.als



DFILE F3CC6H4-g-lactone-1H.als
COMNT F3CC6H4-g-lactone-1H
DATIM 2013-08-01 12:03:42
OBNUC 1H
EXMOD proton.jsp
OBFRQ 395.88 MHz
OBSET 6.28 KHz
OBFIN 0.87 Hz
POINT 16384
FREQU 7422.80 Hz
SCANS 8
ACQTM 2.2073 sec
PD 5.0000 sec
PWI 3.12 usec
IRNUC 1H
CTEMP 20.7 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.30 Hz
RGAIN 46

F3CC6H4-g-lactone-13C

C:\Documents and Settings\ALPHA\ff\X\N\g\h\y\U\ozumi\G\Hamasaki\F3CC6H4-g-lactone-13C.als

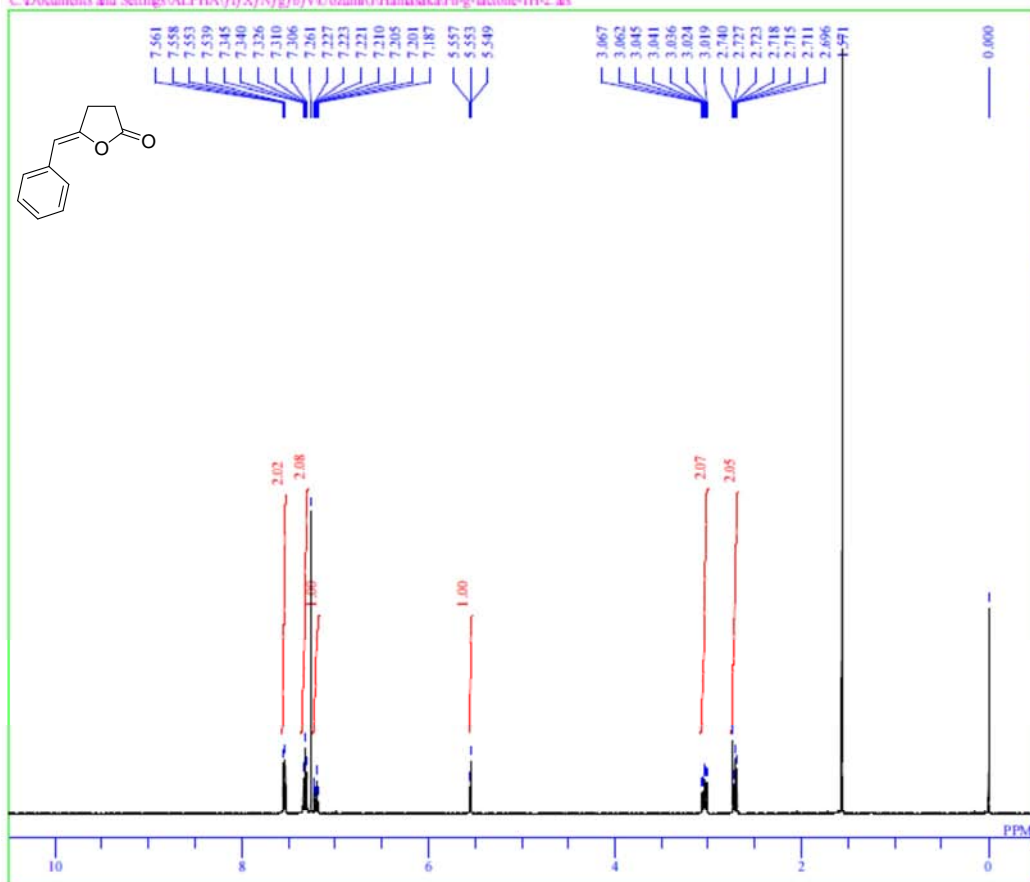


DFILE F3CC6H4-g-lactone-13C.als
COMNT F3CC6H4-g-lactone-13C
DATIM 2013-07-08 18:11:37
OBNUC 13C
EXMOD carbon.jsp
OBFRQ 99.55 MHz
OBSET 5.13 KHz
OBFIN 0.98 Hz
POINT 32767
FREQU 31250.00 Hz
SCANS 806
ACQTM 1.0486 sec
PD 2.0000 sec
PWI 3.42 usec
IRNUC 1H
CTEMP 21.9 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.50 Hz
RGAIN 60

(Z)- γ -Phenylbenzylidene- γ -butyrolactone (**3b**)

Ph-g-lactone-1H

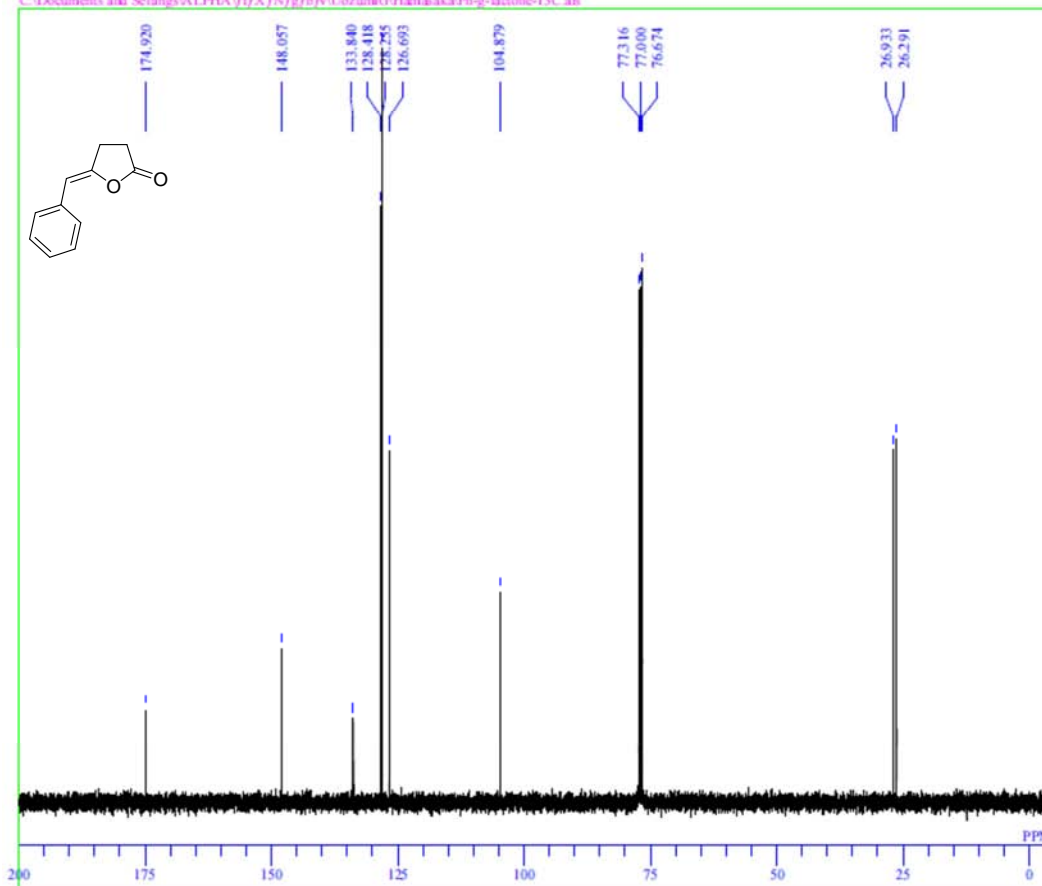
C:\Documents and Settings\ALPHA\ff\X\N\g\fb\Uozumi\G\Hamazaki\Ph-g-lactone-1H-2.als



DFILE Ph-g-lactone-1H-2.als
COMNT Ph-g-lactone-1H
DATIM 2013-08-01 16:20:34
OBNUC 1H
EXMOD proton.jxp
OBFRQ 395.88 MHz
OBSET 6.28 KHz
OBFIN 0.87 Hz
POINT 16384
FREQU 7422.80 Hz
SCANS 8
ACQTM 2.2073 sec
PD 5.0000 sec
PWI 3.12 usec
IRNUC 1H
CTEMP 21.9 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.00 Hz
RGAIN 46

Ph-g-lactone-13C

C:\Documents and Settings\ALPHA\ff\X\N\g\fb\Uozumi\G\Hamazaki\Ph-g-lactone-13C.als

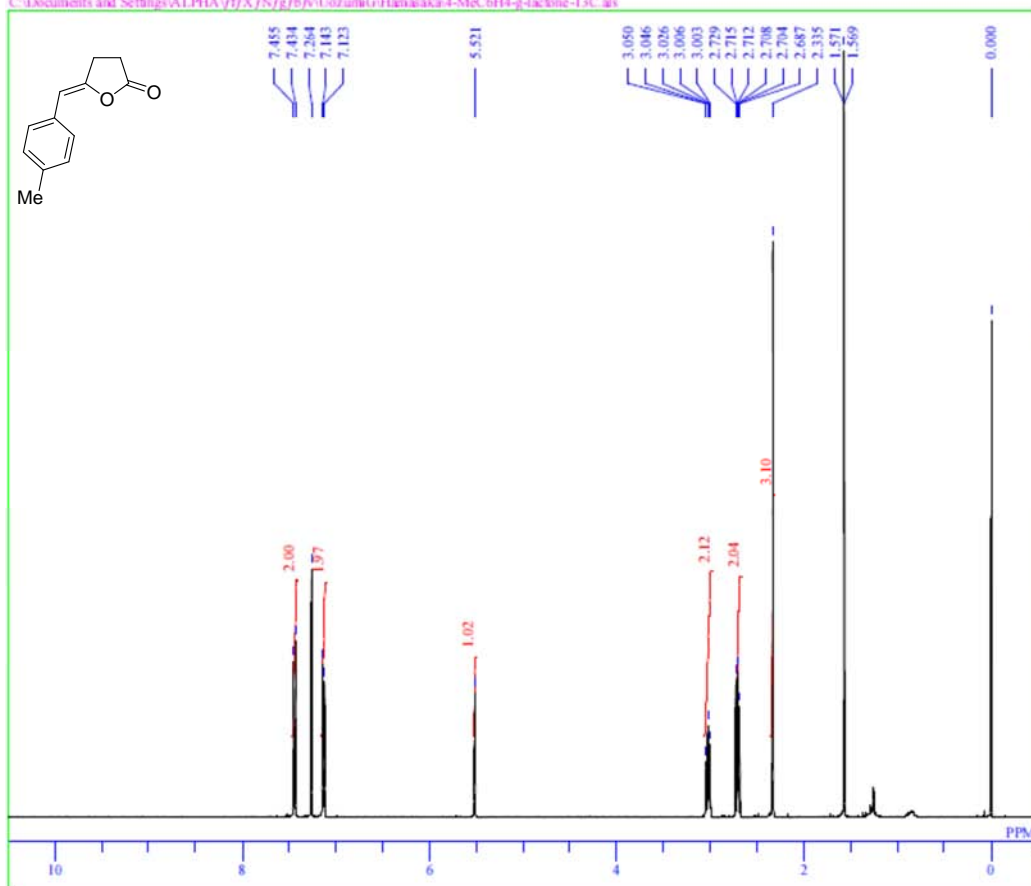


DFILE Ph-g-lactone-13C.als
COMNT Ph-g-lactone-13C
DATIM 2013-07-09 11:13:56
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 99.55 MHz
OBSET 5.13 KHz
OBFIN 0.98 Hz
POINT 32767
FREQU 31250.00 Hz
SCANS 163
ACQTM 1.0486 sec
PD 2.0000 sec
PWI 3.42 usec
IRNUC 1H
CTEMP 21.4 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.50 Hz
RGAIN 60

(Z)- γ -(4-Methylbenzylidene)- γ -butyrolactone (**3c**)

4-MeC6H4-g-lactone-1H

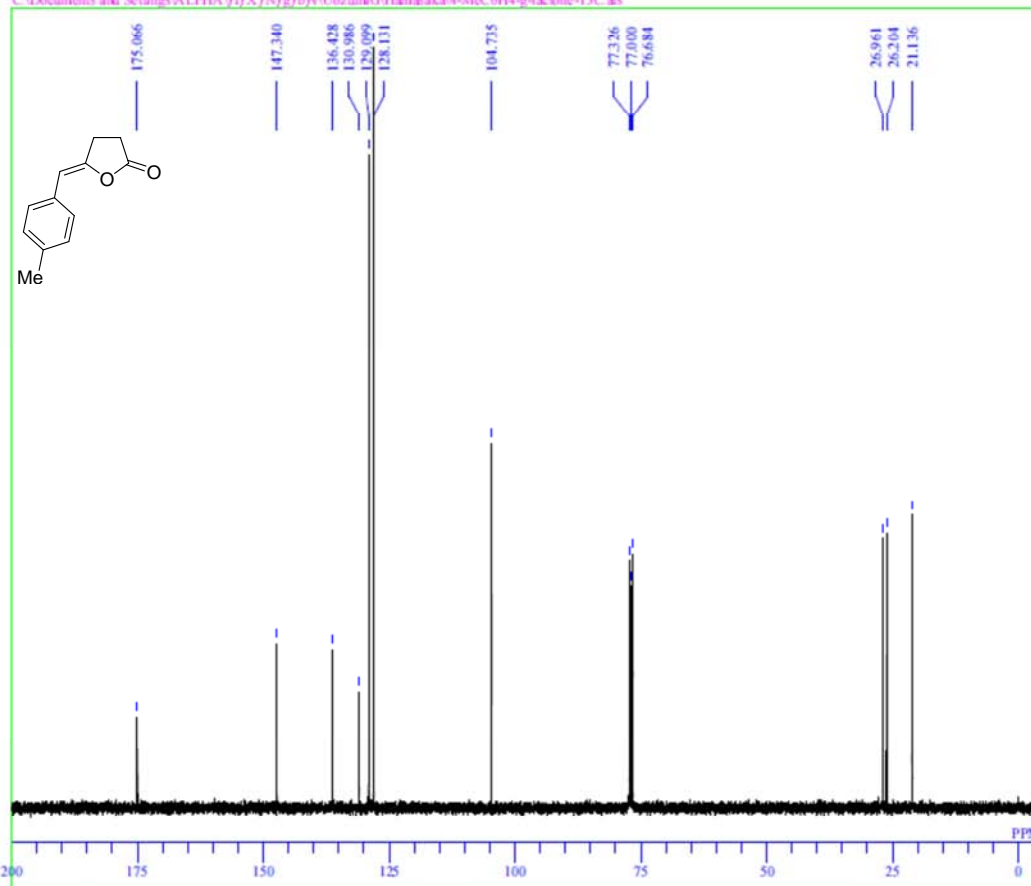
C:\Documents and Settings\ALPHA\ff\X\N\g\b\U\ozumi\G\Hamazaki\4-MeC6H4-g-lactone-13C.als



DFILE 4-MeC6H4-g-lactone-13C.als
COMNT 4-MeC6H4-g-lactone-1H
DATIM 2013-07-09 22:06:38
OBNUC 1H
EXMOD proton.jxp
OBFREQ 395.88 MHz
OBSET 6.28 KHz
OBFIN 0.87 Hz
POINT 16384
FREQU 7422.80 Hz
SCANS 8
ACQTM 2.2073 sec
PD 5.0000 sec
PWI 3.12 usec
IRNUC 1H
CTEMP 21.2 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.00 Hz
RGAIN 44

4-MeC6H4-g-lactone-13C

C:\Documents and Settings\ALPHA\ff\X\N\g\b\U\ozumi\G\Hamazaki\4-MeC6H4-g-lactone-13C.als

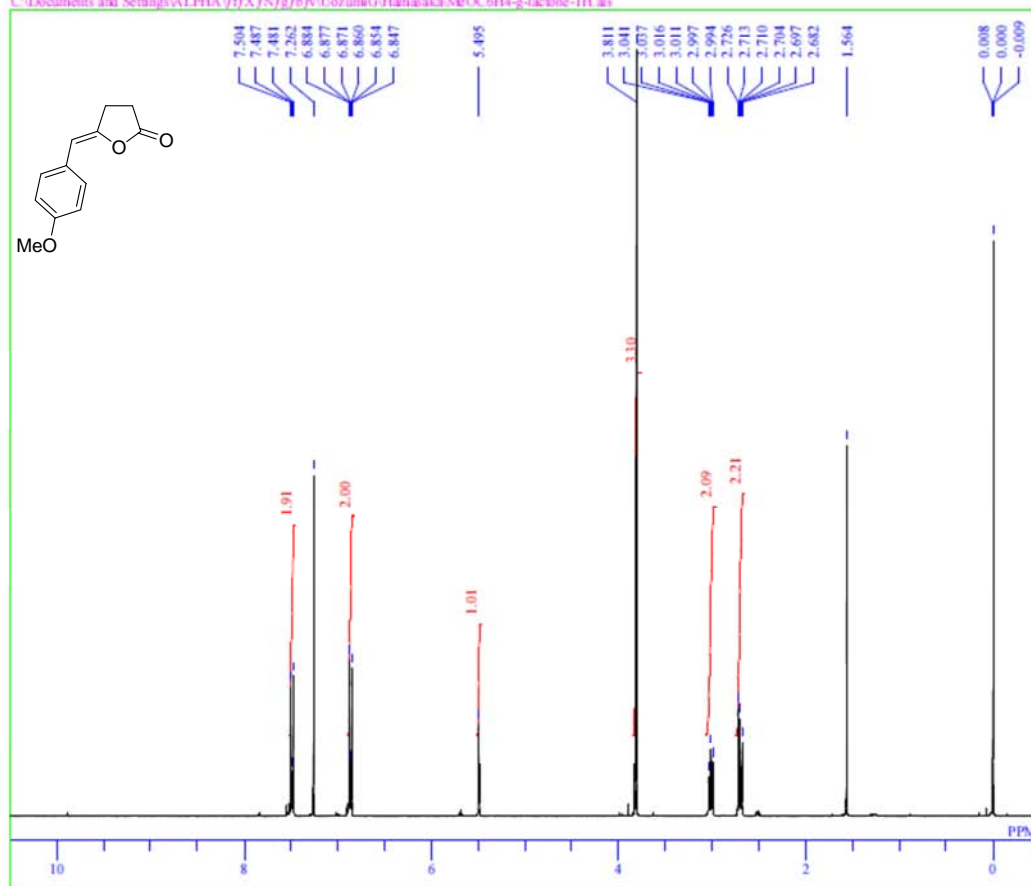


DFILE 4-MeC6H4-g-lactone-13C.als
COMNT 4-MeC6H4-g-lactone-13C
DATIM 2013-07-17 23:27:08
OBNUC 13C
EXMOD carbon.jxp
OBFREQ 99.55 MHz
OBSET 5.13 KHz
OBFIN 0.98 Hz
POINT 26214
FREQU 25000.00 Hz
SCANS 136
ACQTM 1.0486 sec
PD 2.0000 sec
PWI 3.42 usec
IRNUC 1H
CTEMP 19.9 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.50 Hz
RGAIN 60

(Z)- γ -(4-Methoxybenzylidene)- γ -butyrolactone (**3d**)

MeOC6H4-g-lactone

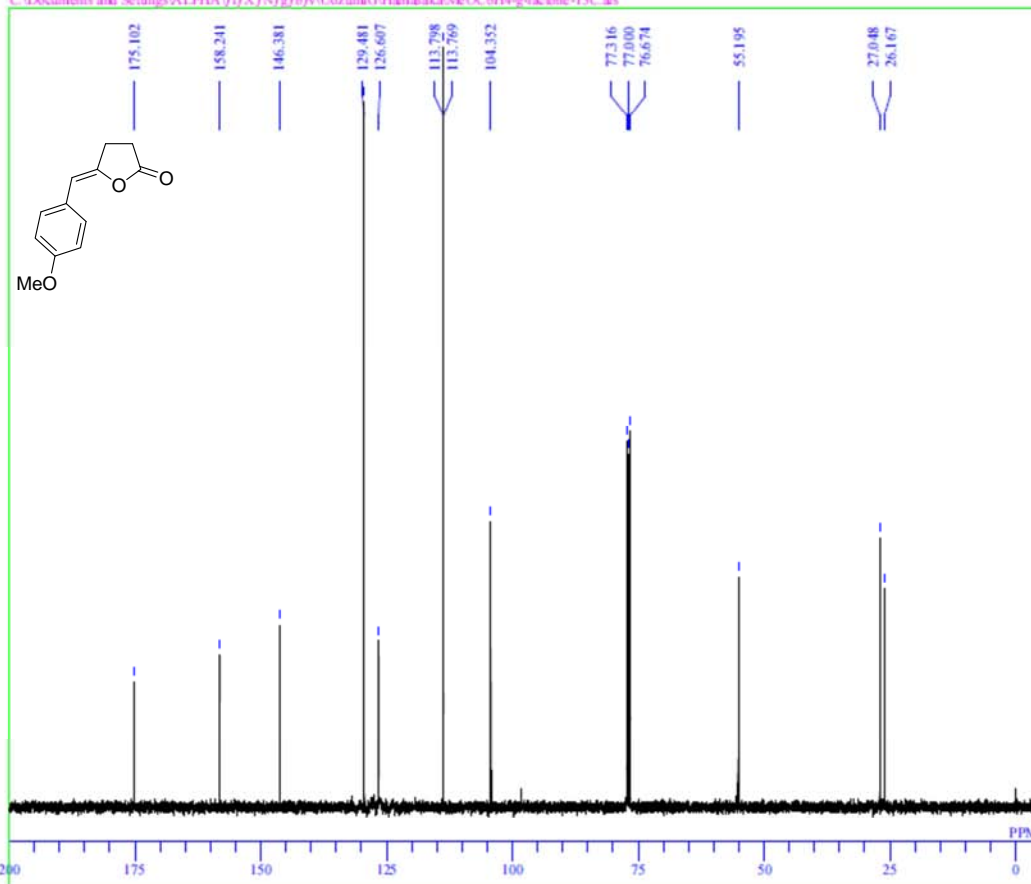
C:\Documents and Settings\ALPHA\ffx\N/g/b/b/Uozumi\G\Hamazaki\MeOC6H4-g-lactone-1H.als



DFILE MeOC6H4-g-lactone-1H.als
COMNT MeOC6H4-g-lactone
DATIM 2013-07-06 19:41:56
OBNUC 1H
EXMOD proton.jxp
OBFREQ 395.88 MHz
OBSET 6.28 KHz
OBFIN 0.87 Hz
POINT 16384
FREQU 7422.80 Hz
SCANS 8
ACQTM 2.2073 sec
PD 5.0000 sec
PWI 3.12 usec
IRNUC 1H
CTEMP 19.8 c
SLVNT CDCl3
XREF 0.00 ppm
BF 0.00 Hz
RGAIN 44

MeOC6H4-g-lactone-13C

C:\Documents and Settings\ALPHA\ffx\N/g/b/b/Uozumi\G\Hamazaki\MeOC6H4-g-lactone-13C.als

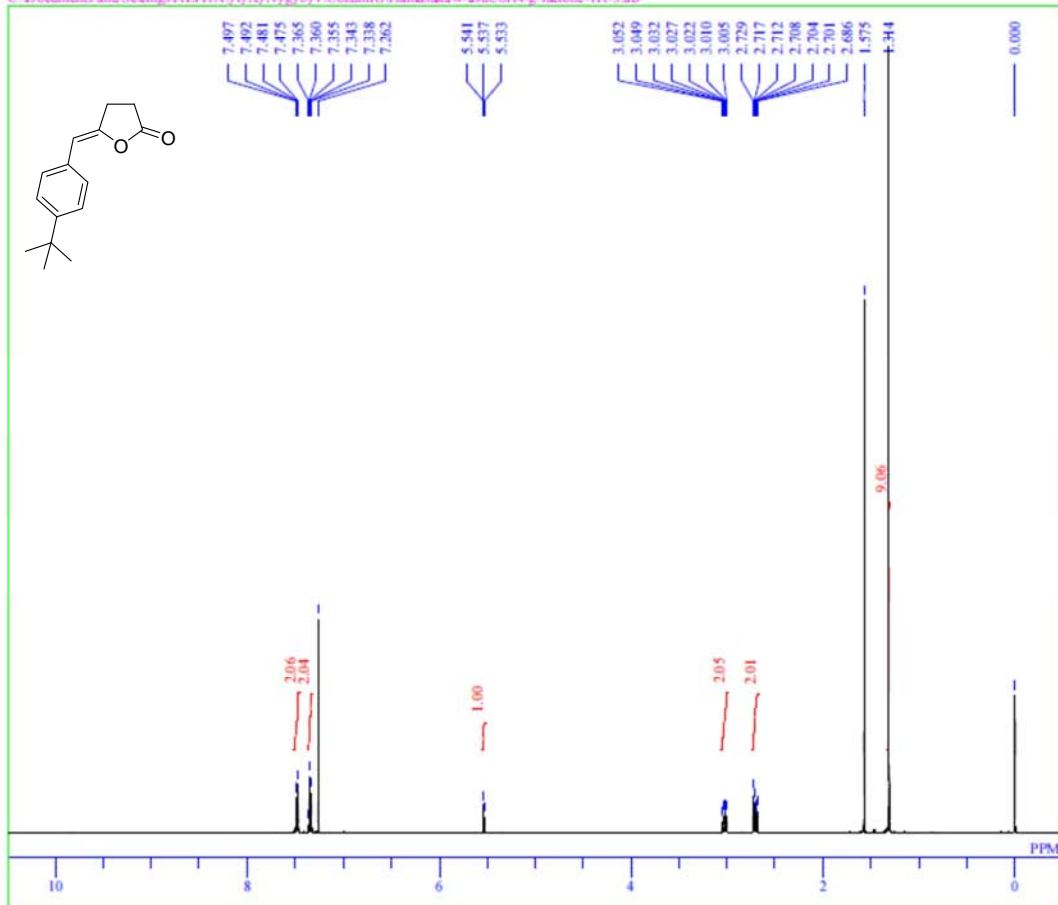


DFILE MeOC6H4-g-lactone-13C.als
COMNT MeOC6H4-g-lactone-13C
DATIM 2013-07-06 20:27:13
OBNUC 13C
EXMOD carbon.jxp
OBFREQ 99.55 MHz
OBSET 5.13 KHz
OBFIN 0.98 Hz
POINT 32767
FREQU 31250.00 Hz
SCANS 273
ACQTM 1.0486 sec
PD 2.0000 sec
PWI 3.42 usec
IRNUC 13C
CTEMP 19.7 c
SLVNT CDCl3
XREF 77.00 ppm
BF 0.50 Hz
RGAIN 60

(Z)- γ -(4-*tert*-Butylbenzylidene)- γ -butyrolactone (**3e**)

4-tBuC6H4-g-lactone-1H

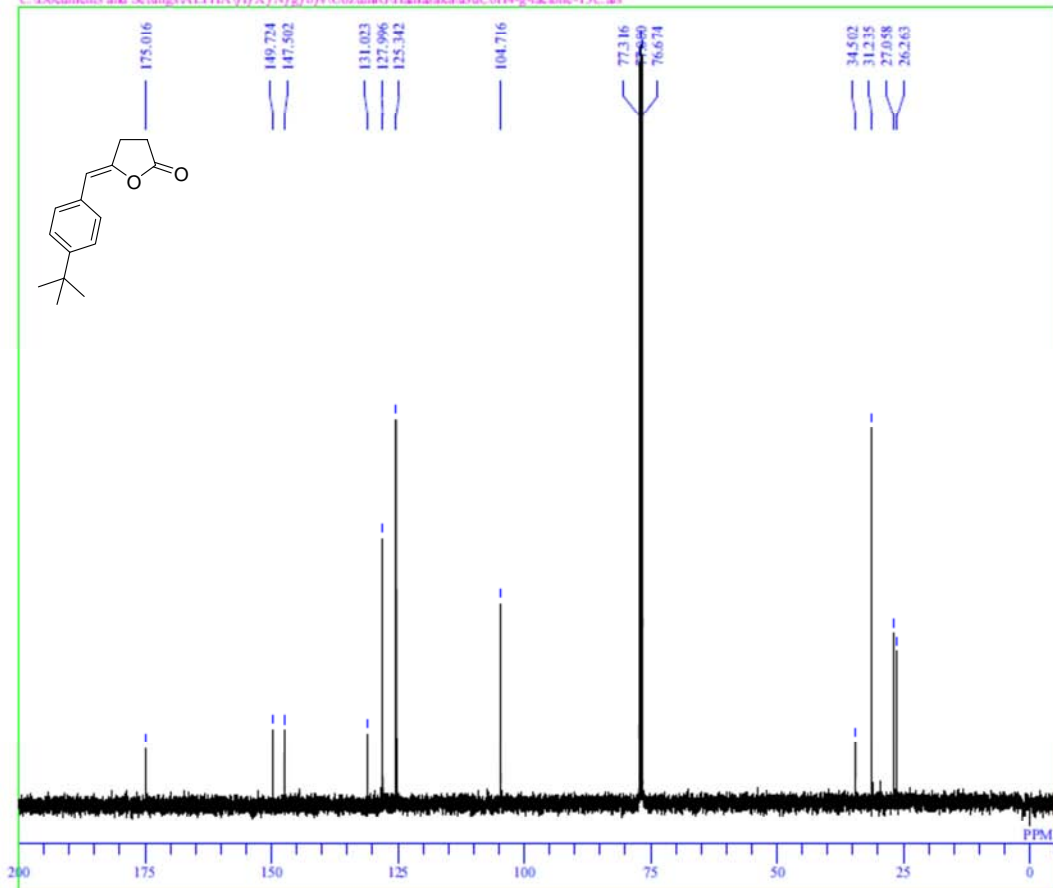
C:\Documents and Settings\ALPHA\ff\X\N/g/b/v/UozumiG\Hanasaka\4-tBuC6H4-g-lactone-1H-3.als



DFILE 4-tBuC6H4-g-lactone-1H-3.als
COMNT 4-tBuC6H4-g-lactone-1H
DATIM 2013-08-03 15:44:31
1H
EXMOD proton.jsp
OBFREQ 395.88 MHz
OBSET 6.28 KHz
OBFIN 0.87 Hz
POINT 16384
FREQU 7422.80 Hz
SCANS 8
ACQTM 2.2073 sec
PD 5.0000 sec
PW1 3.12 usec
IRNUC 1H
CTEMP 20.9 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.00 Hz
RGAIN 46

tBuC6H4-g-lactone-13C

C:\Documents and Settings\ALPHA\ff\X\N/g/b/v/UozumiG\Hanasaka\4-tBuC6H4-g-lactone-13C.als

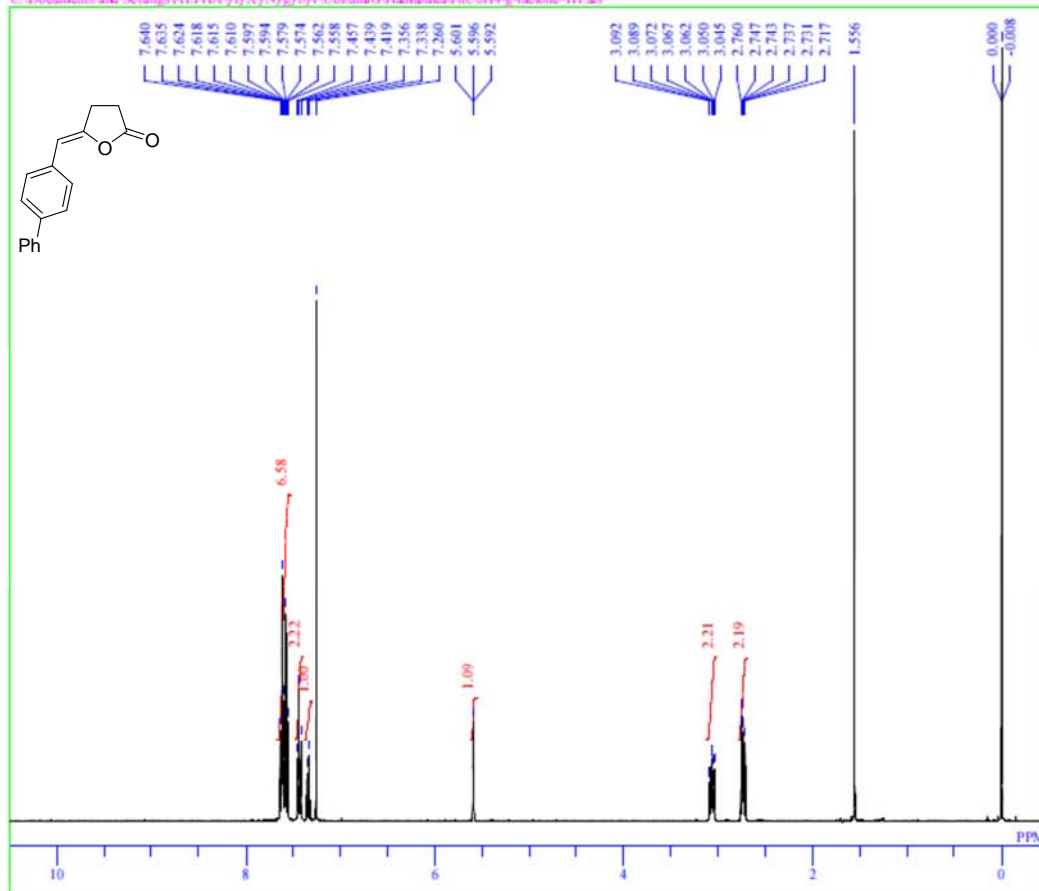


DFILE tBuC6H4-g-lactone-13C.als
COMNT tBuC6H4-g-lactone-13C
DATIM 2013-07-09 10:31:25
13C
EXMOD carbon.jsp
OBFREQ 99.55 MHz
OBSET 5.13 KHz
OBFIN 0.98 Hz
POINT 32767
FREQU 31250.00 Hz
SCANS 315
ACQTM 1.0486 sec
PD 2.0000 sec
PW1 3.42 usec
IRNUC 1H
CTEMP 21.3 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.50 Hz
RGAIN 60

(Z)- γ -(4-Phenylbenzylidene)- γ -butyrolactone (**3f**)

PhC6H4-g-lactone-1H

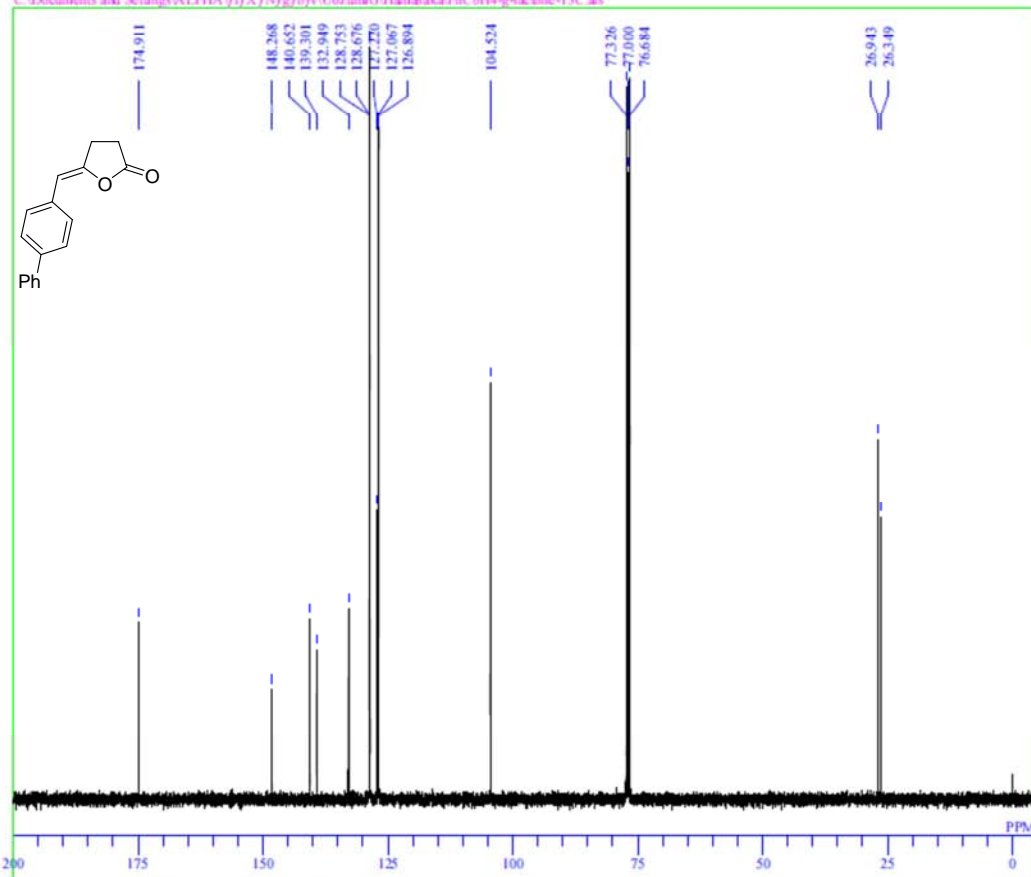
C:\Documents and Settings\ALPHA\ff\X\N\g\b\Uozumi\G\Hamana\PhC6H4-g-lactone-1H.als



DFILE PhC6H4-g-lactone-1H.als
COMNT PhC6H4-g-lactone-1H
DATIM 2013-07-09 21:46:11
OBNUC 1H
EXMOD proton.jxp
OBFREQ 395.88 MHz
OBSET 6.28 KHz
OBFIN 0.87 Hz
POINT 16384
FREQU 7422.80 Hz
SCANS 8
ACQTM 2.2073 sec
PD 5.0000 sec
PWI 3.12 usec
IRNUC 1H
CTEMP 21.2 c
SLVNT CDCl3
EXREF 0.00 ppm
BF 0.00 Hz
RGAIN 44

PhC6H4-g-lactone-13C

C:\Documents and Settings\ALPHA\ff\X\N\g\b\Uozumi\G\Hamana\PhC6H4-g-lactone-13C.als

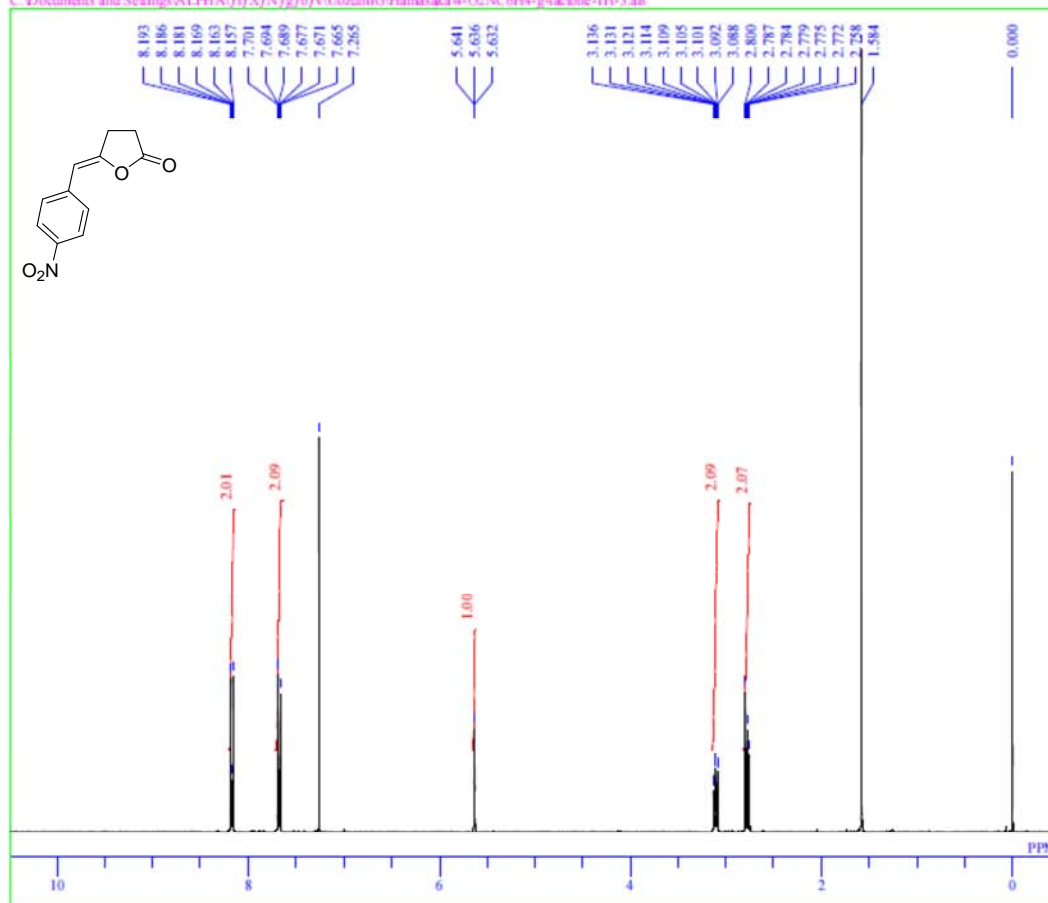


DFILE PhC6H4-g-lactone-13C.als
COMNT PhC6H4-g-lactone-13C
DATIM 2013-07-09 22:26:07
OBNUC 13C
EXMOD carbon.jxp
OBFREQ 99.55 MHz
OBSET 5.13 KHz
OBFIN 0.98 Hz
POINT 32767
FREQU 31250.00 Hz
SCANS 668
ACQTM 1.0486 sec
PD 2.0000 sec
PWI 3.42 usec
IRNUC 13C
CTEMP 21.0 c
SLVNT CDCl3
EXREF 77.00 ppm
BF 0.50 Hz
RGAIN 60

(Z)- γ -(4-Nitrobenzylidene)- γ -butyrolactone (**3g**)

4-O2NC6H4-g-lactone-1H

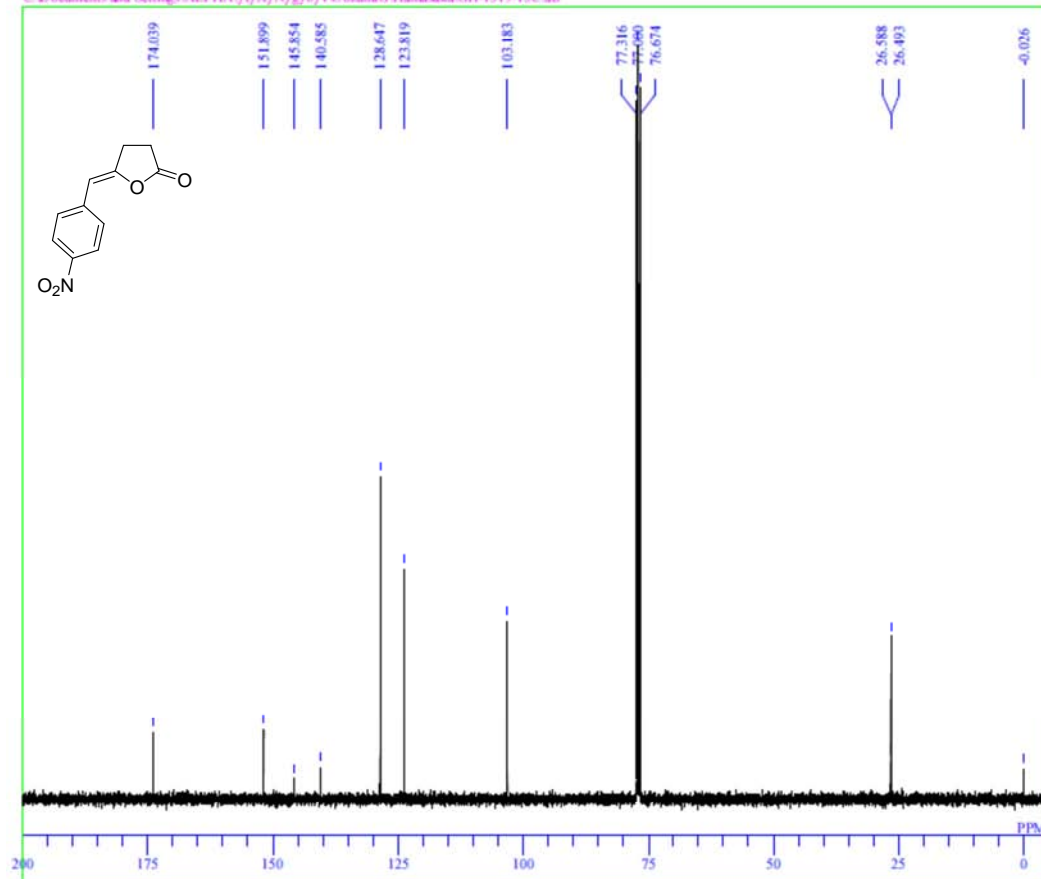
C:\Documents and Settings\ALPHA\ff\X\N\g\b\U\ozumi\G\Hamazaka\4-O2NC6H4-g-lactone-1H-3.als



DFILE 4-O2NC6H4-g-lactone-1H-3.als
COMNT 4-O2NC6H4-g-lactone-1H
DATIM 2013-08-03 15:31:55
OBNUC 1H
EXMOD proton.jpg
OBFREQ 395.88 MHz
OBSET 6.28 KHz
OBFIN 0.87 Hz
POINT 16384
FREQU 7422.80 Hz
SCANS 8
ACQTM 2.2073 sec
PD 5.0000 sec
PW1 3.12 usec
IRNUC 1H
CTEMP 20.9 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.00 Hz
RGAIN 46

GH-1319-13C

C:\Documents and Settings\ALPHA\ff\X\N\g\b\U\ozumi\G\Hamazaka\GH-1319-13C.als

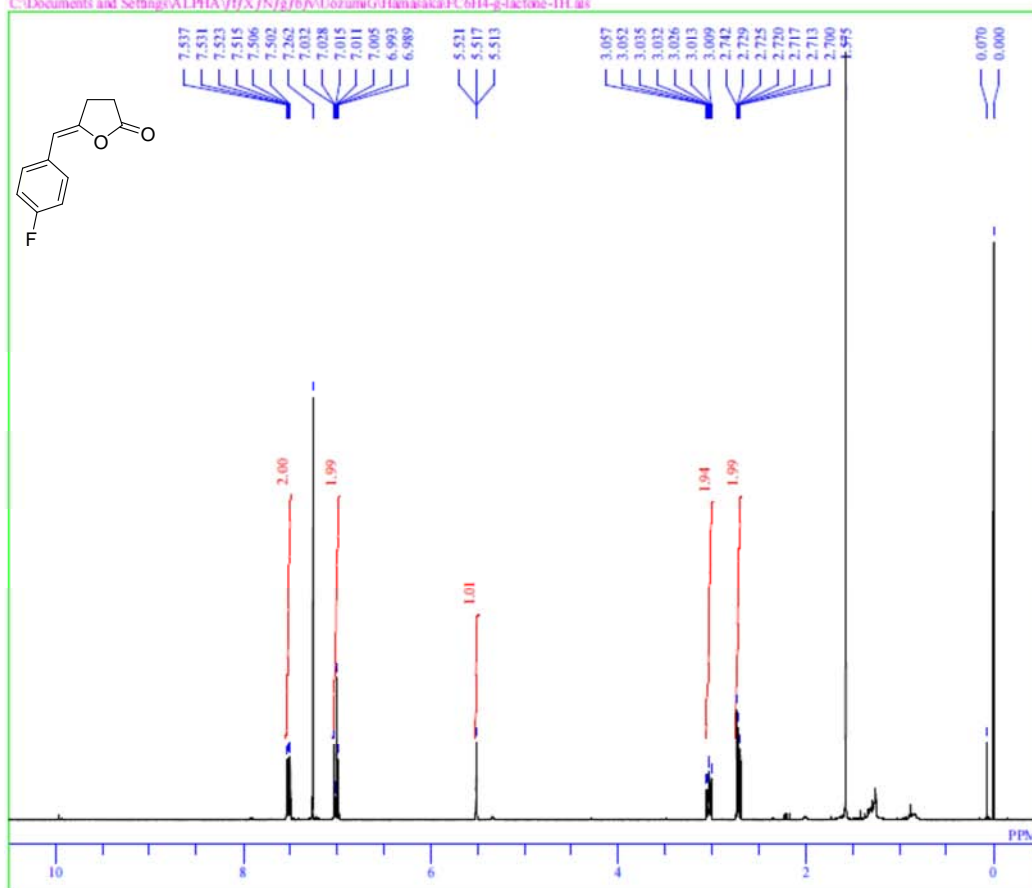


DFILE GH-1319-13C.als
COMNT GH-1319-13C
DATIM 2013-07-05 22:44:58
OBNUC 13C
EXMOD carbon.jpg
OBFREQ 99.55 MHz
OBSET 5.13 KHz
OBFIN 0.98 Hz
POINT 32767
FREQU 31250.00 Hz
SCANS 612
ACQTM 1.0486 sec
PD 2.0000 sec
PW1 3.42 usec
IRNUC 1H
CTEMP 20.5 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.50 Hz
RGAIN 60

(Z)- γ -(4-Fluorobenzylidene)- γ -butyrolactone (**3h**)

FC6H4-g-lactone

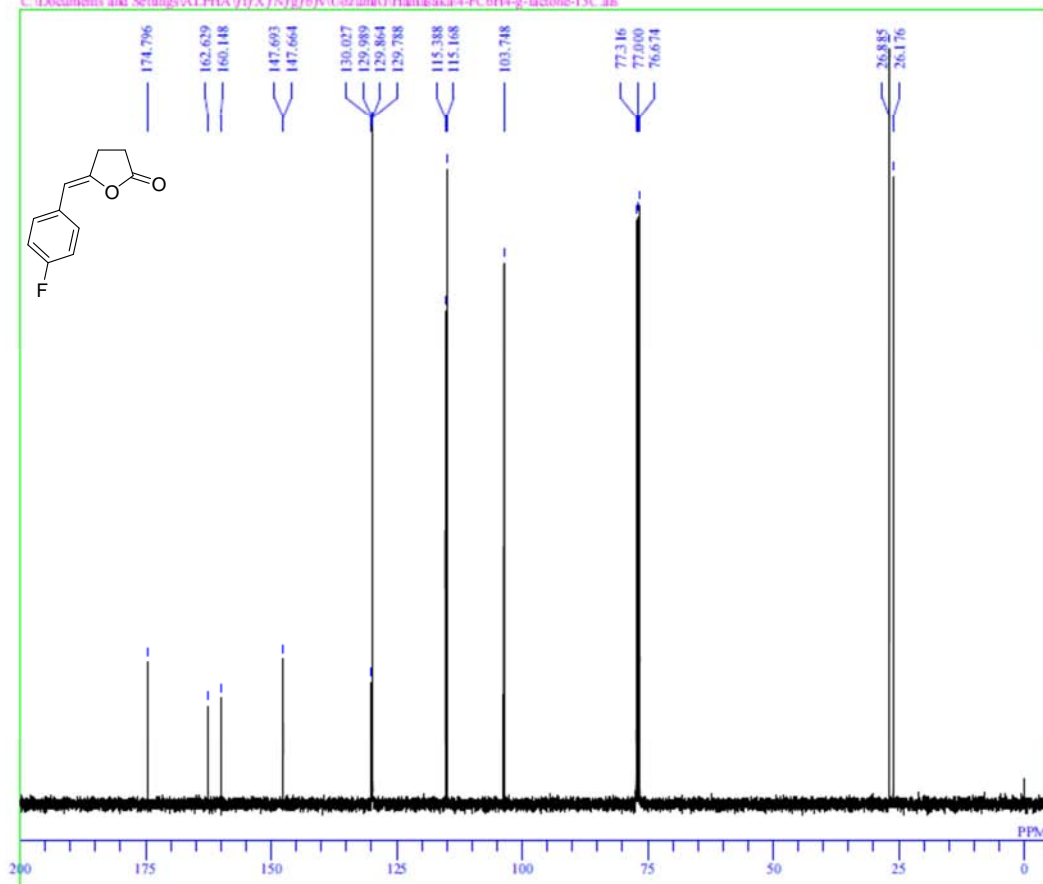
C:\Documents and Settings\ALPHA\ff\X\N\g\b\A\Uozumi\G\Hanagaki\FC6H4-g-lactone-1H.als



DFILE FC6H4-g-lactone-1H.als
COMNT FC6H4-g-lactone
DATIM 2013-07-08 14:01:44
OBNUC 1H
EXMOD proton.jsp
OBFREQ 395.88 MHz
OBSET 6.28 kHz
OBFIN 0.87 Hz
POINT 16384
FREQU 7422.80 Hz
SCANS 8
ACQTM 2.2073 sec
PD 5.0000 sec
PW1 3.12 usec
IRNUC 1H
CTEMP 22.3 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.00 Hz
RGAIN 46

4-FC6H4-g-lactone-13C

C:\Documents and Settings\ALPHA\ff\X\N\g\b\A\Uozumi\G\Hanagaki\4-FC6H4-g-lactone-13C.als

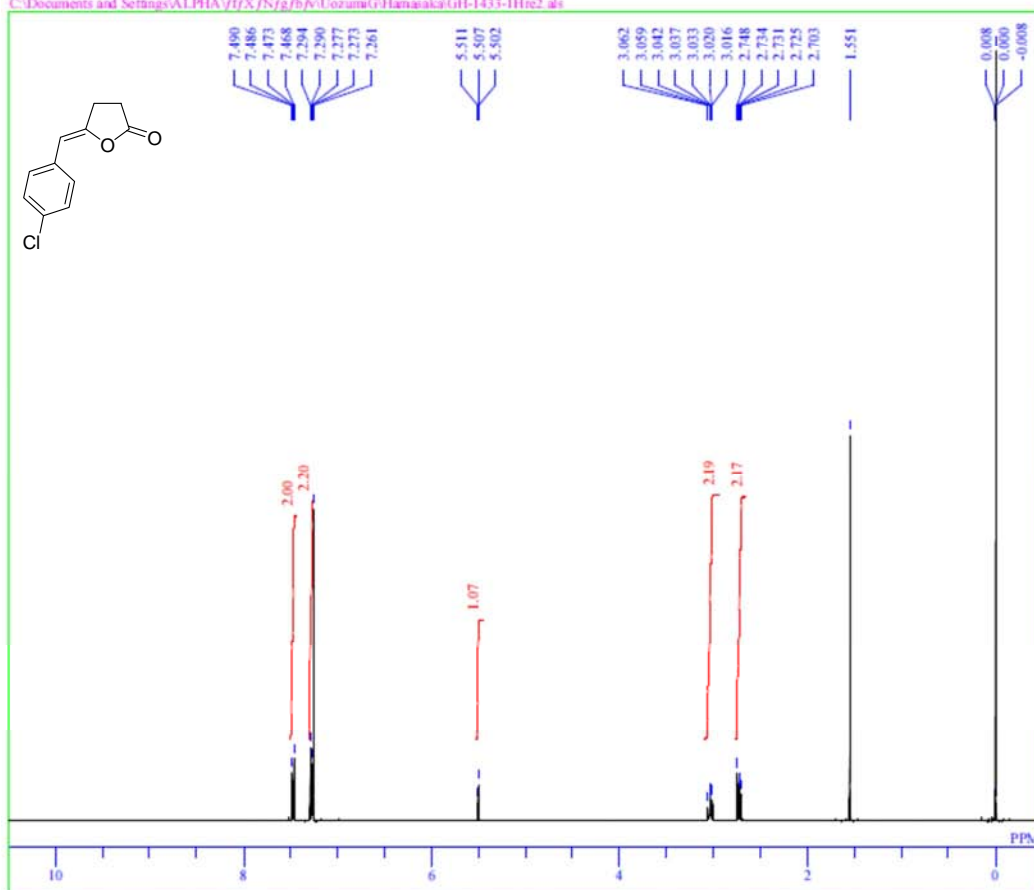


DFILE 4-FC6H4-g-lactone-13C.als
COMNT 4-FC6H4-g-lactone-13C
DATIM 2013-07-17 19:05:31
OBNUC 13C
EXMOD carbon.jsp
OBFREQ 99.55 MHz
OBSET 5.13 kHz
OBFIN 0.98 Hz
POINT 32767
FREQU 31250.00 Hz
SCANS 451
ACQTM 1.0486 sec
PD 2.0000 sec
PW1 3.42 usec
IRNUC 13C
CTEMP 20.2 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.50 Hz
RGAIN 60

(Z)- γ -(4-Chlorobenzylidene)- γ -butyrolactone (**3i**)

GH-1433-1H

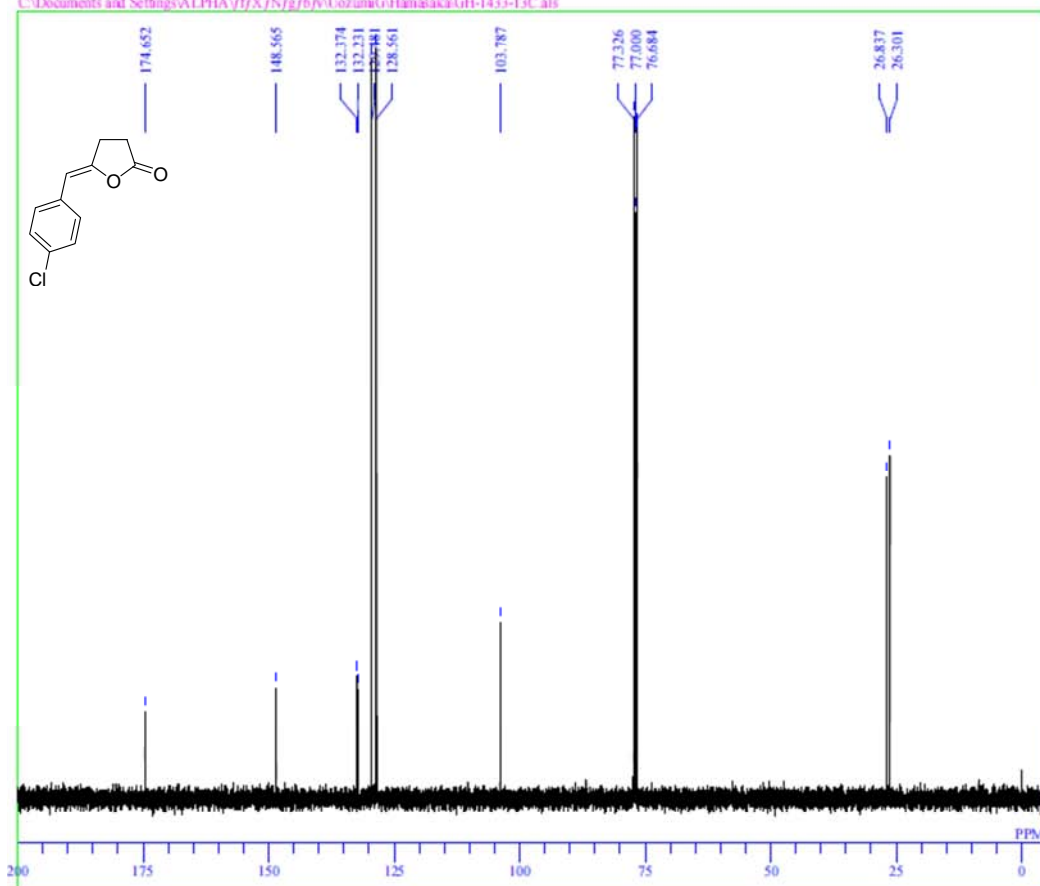
C:\Documents and Settings\ALPHA\ffX\N/g/b/v/UozumiGHamasaki\GH-1433-1Hre2.als



DFILE GH-1433-1Hre2.als
COMNT GH-1433-1H
DATIM 2013-07-05 16:39:30
OBNUC 1H
EXMOD proton.jxp
OBFRQ 395.88 MHz
OBSET 6.28 KHz
OBFIN 0.87 Hz
POINT 16384
FREQU 7422.80 Hz
SCANS 8
ACQTM 2.2073 sec
PD 5.0000 sec
PWI 3.12 usec
IRNUC 1H
CTEMP 21.5 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.00 Hz
RGAIN 48

GH-1433-13C

C:\Documents and Settings\ALPHA\ffX\N/g/b/v/UozumiGHamasaki\GH-1433-13C.als

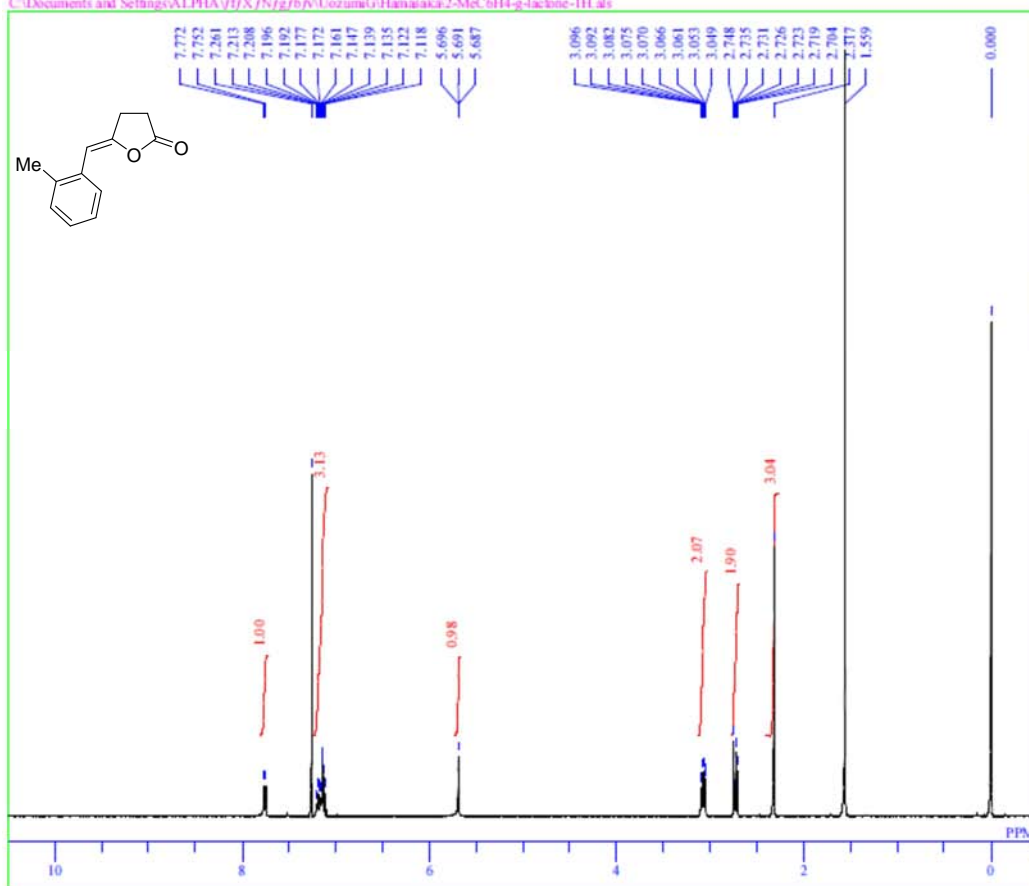


DFILE GH-1433-13C.als
COMNT GH-1433-13C
DATIM 2013-07-05 15:47:02
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 99.55 MHz
OBSET 5.13 KHz
OBFIN 0.98 Hz
POINT 32767
FREQU 31250.00 Hz
SCANS 162
ACQTM 1.0486 sec
PD 2.0000 sec
PWI 3.42 usec
IRNUC 1H
CTEMP 21.6 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.50 Hz
RGAIN 60

(Z)- γ -(2-Methylbenzylidene)- γ -butyrolactone (**3j**)

2-MeC6H4-g-lactone-1H

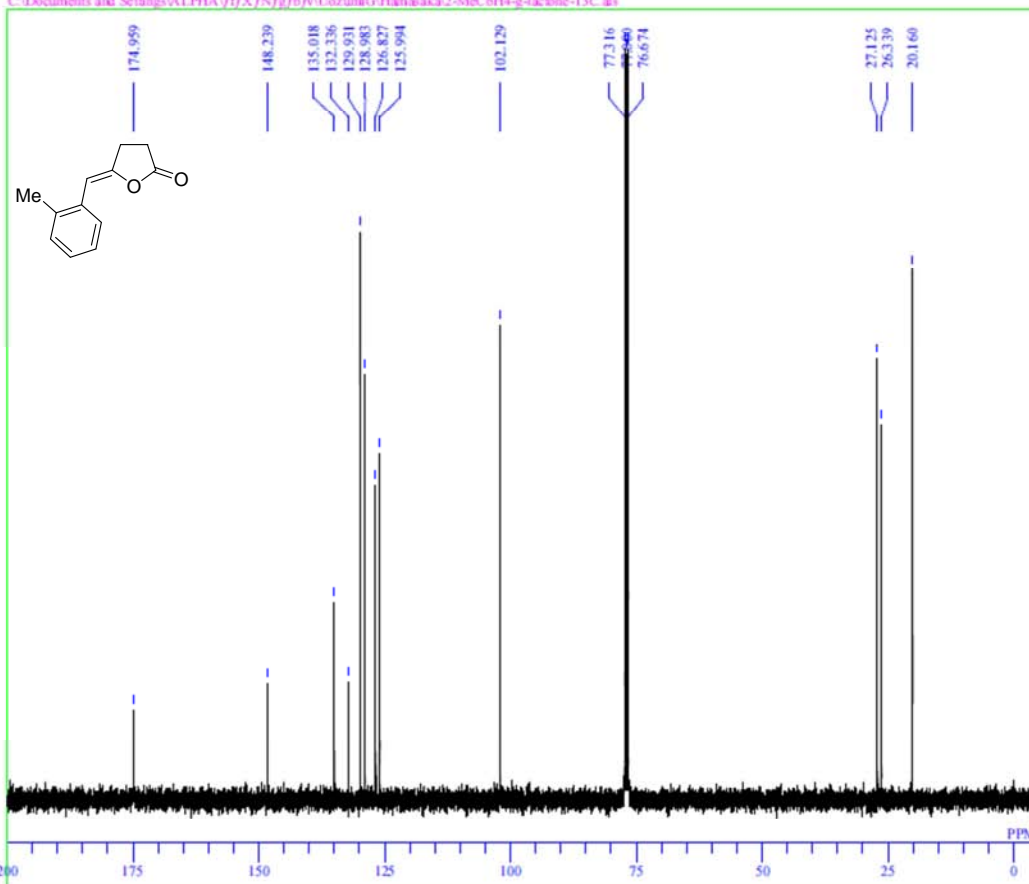
C:\Documents and Settings\ALPHA\ff\X\N\g\b\Uozumi\G\Hamazaki\2-MeC6H4-g-lactone-1H.als



DFILE 2-MeC6H4-g-lactone-1H.als
COMNT 2-MeC6H4-g-lactone-1H
DATIM 2013-07-18 14:36:49
OBNUC 1H
EXMOD proton.jsp
OBFREQ 395.88 MHz
OBSET 6.28 KHz
OBFIN 0.87 Hz
POINT 13107
FREQU 5938.24 Hz
SCANS 8
ACQTM 2.2073 sec
PD 5.0000 sec
PWI 3.12 usec
IRNUC 1H
CTEMP 21.8 c
SLVNT CDCl3
EXREF 0.00 ppm
BF 0.00 Hz
RGAIN 48

2-MeC6H4-g-lactone-13C

C:\Documents and Settings\ALPHA\ff\X\N\g\b\Uozumi\G\Hamazaki\2-MeC6H4-g-lactone-13C.als

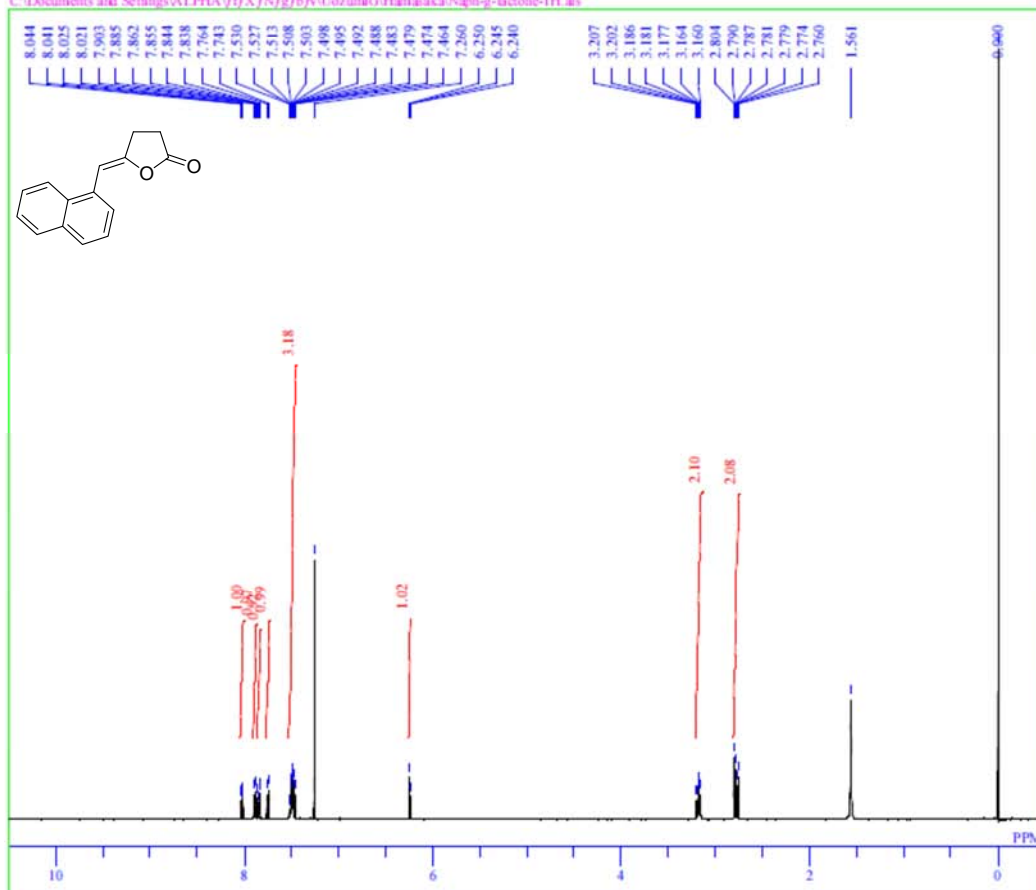


DFILE 2-MeC6H4-g-lactone-13C.als
COMNT 2-MeC6H4-g-lactone-13C
DATIM 2013-07-18 17:38:28
OBNUC 13C
EXMOD carbon.jsp
OBFREQ 99.55 MHz
OBSET 5.13 KHz
OBFIN 0.98 Hz
POINT 32767
FREQU 31250.00 Hz
SCANS 343
ACQTM 1.0486 sec
PD 2.0000 sec
PWI 3.42 usec
IRNUC 1H
CTEMP 21.8 c
SLVNT CDCl3
EXREF 77.00 ppm
BF 0.50 Hz
RGAIN 60

(Z)- γ -(1-Naphthalenylmethylene)- γ -butyrolactone (**3k**)

Naph-g-lactone-1H

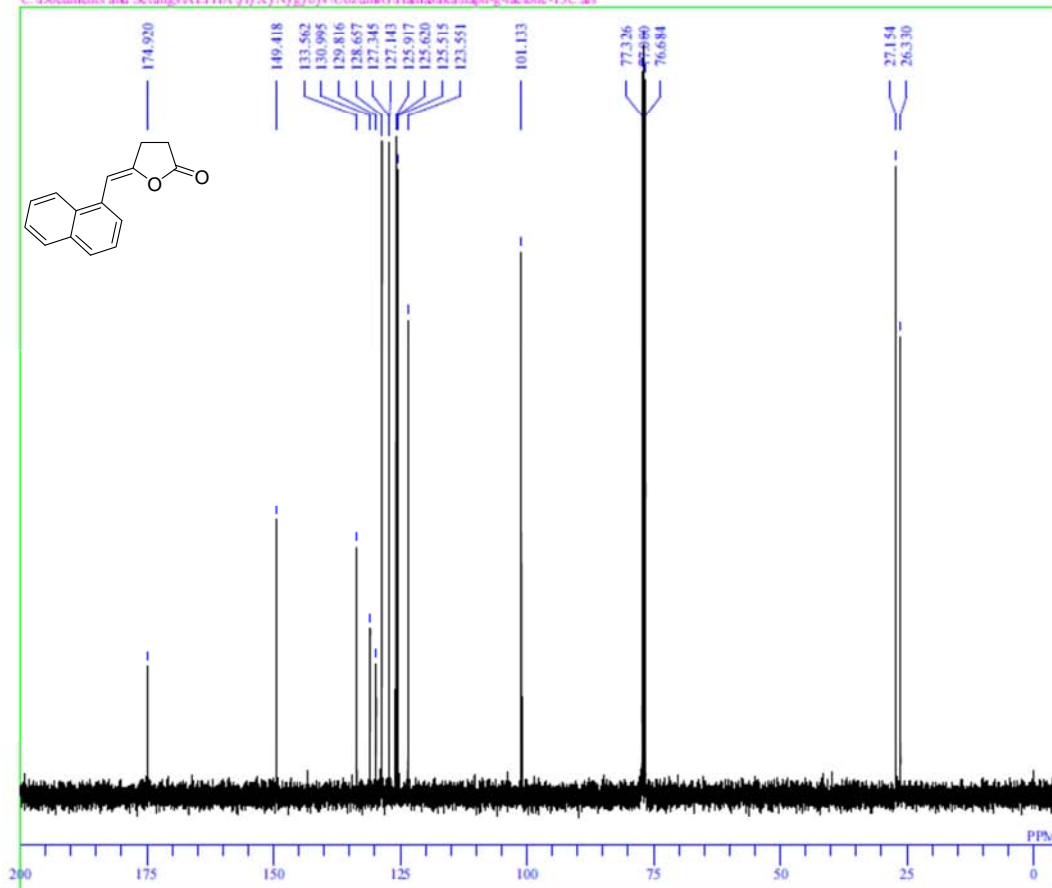
C:\Documents and Settings\ALPHA\ff\X\N\g\fb\Uozumi\G\Hamazaki\Naph-g-lactone-1H.als



DFILE Naph-g-lactone-1H.als
COMNT Naph-g-lactone-1H
DATIM 2013-07-18 12:22:46
OBNUC 1H
EXMOD proton.jxp
OBFRQ 395.88 MHz
OBSET 6.28 KHz
OBFIN 0.87 Hz
POINT 16384
FREQU 7422.80 Hz
SCANS 8
ACQTM 2.2073 sec
PD 5.0000 sec
PWI 3.12 usec
IRNUC 1H
CTEMP 21.2 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.00 Hz
RGAIN 46

naph-g-lactone-13C

C:\Documents and Settings\ALPHA\ff\X\N\g\fb\Uozumi\G\Hamazaki\Naph-g-lactone-13C.als

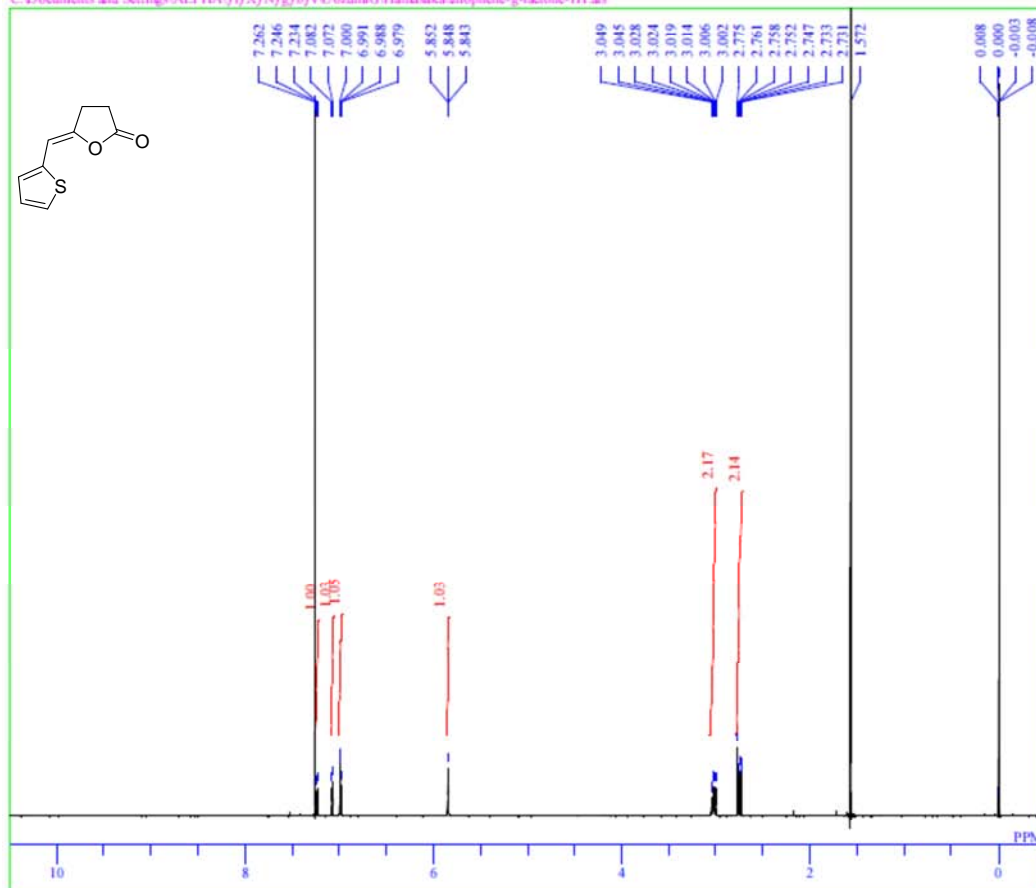


DFILE naph-g-lactone-13C.als
COMNT naph-g-lactone-13C
DATIM 2013-07-18 17:16:19
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 99.55 MHz
OBSET 5.13 KHz
OBFIN 0.98 Hz
POINT 32767
FREQU 31250.00 Hz
SCANS 198
ACQTM 1.0486 sec
PD 2.0000 sec
PWI 3.42 usec
IRNUC 13C
CTEMP 21.9 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.50 Hz
RGAIN 60

(Z)- γ -(2-Thienylmethylene)- γ -butyrolactone (**31**)

thiophene-g-lactone-1H

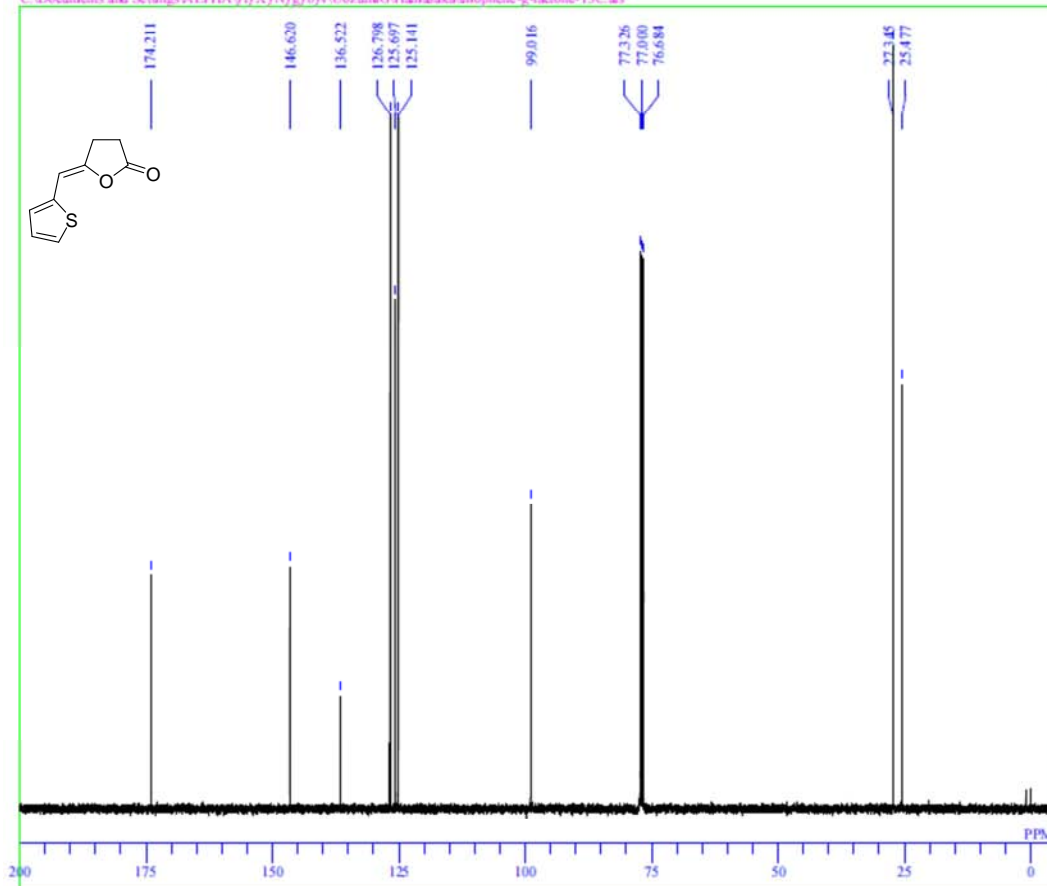
C:\Documents and Settings\ALPHA\ff\X\N\g\h\Uozumi\G\Hamaoka\thiophene-g-lactone-1H.als



DFILE thiophene-g-lactone-1H.als
COMNT thiophene-g-lactone-1H
DATIM 2013-08-01 18:27:50
OBNUC 1H
EXMOD proton.jsp
OBFRQ 395.88 MHz
OBSET 6.28 KHz
OBFIN 0.87 Hz
POINT 16384
FREQU 7422.80 Hz
SCANS 8
ACQTM 2.2073 sec
PD 5.0000 sec
PWI 3.12 usec
IRNUC 1H
CTEMP 21.9 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.00 Hz
RGAIN 48

thiophene-g-lactone-13C

C:\Documents and Settings\ALPHA\ff\X\N\g\h\Uozumi\G\Hamaoka\thiophene-g-lactone-13C.als

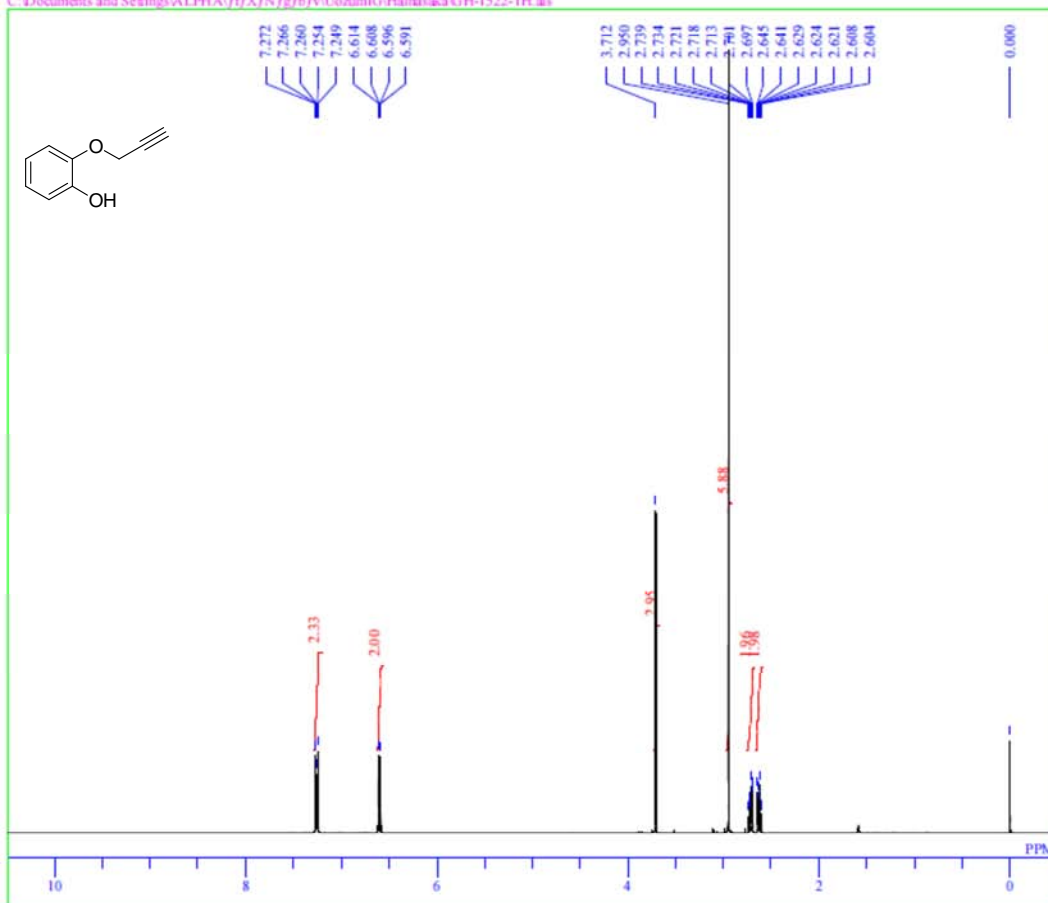


DFILE thiophene-g-lactone-13C.als
COMNT thiophene-g-lactone-13C
DATIM 2013-07-20 19:33:58
OBNUC 13C
EXMOD carbon.jsp
OBFRQ 99.55 MHz
OBSET 5.13 KHz
OBFIN 0.98 Hz
POINT 32767
FREQU 31250.00 Hz
SCANS 909
ACQTM 1.0486 sec
PD 2.0000 sec
PWI 3.42 usec
IRNUC 1H
CTEMP 19.6 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.40 Hz
RGAIN 60

2-Propargyloxyphenol (S3)

GH-1522-1H

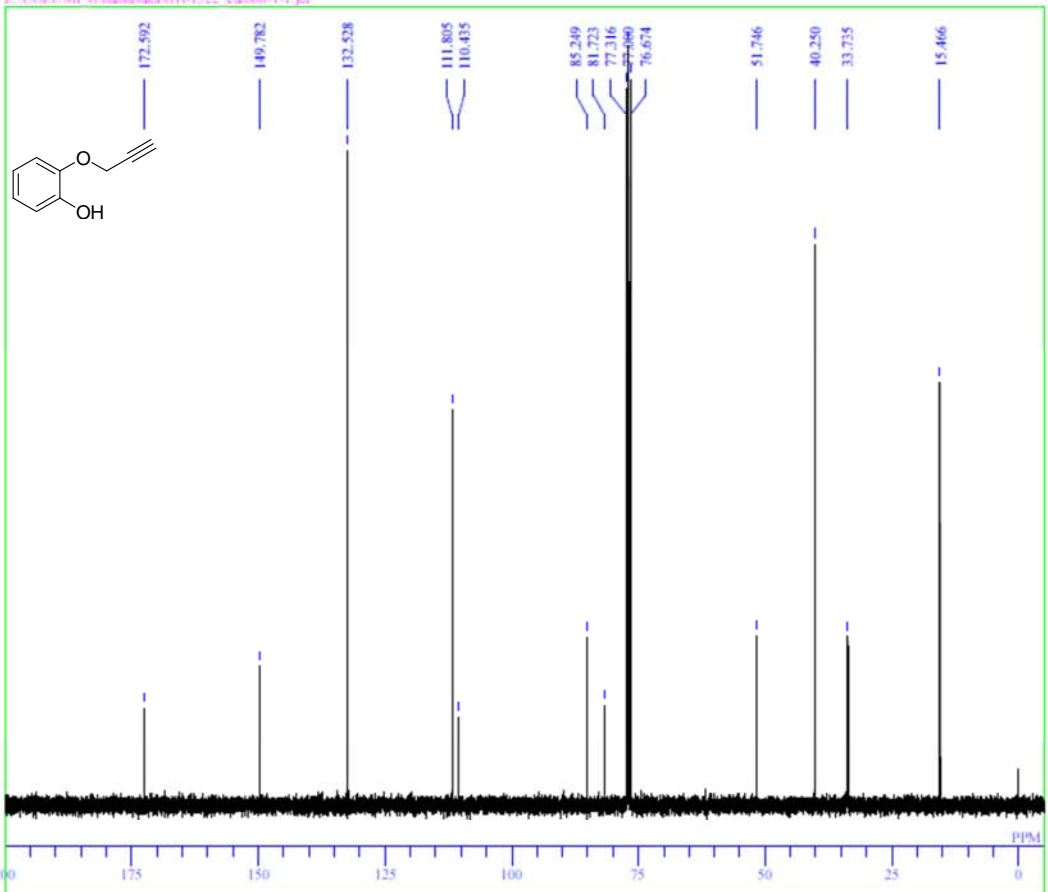
C:\Documents and Settings\ALPHA\ffx\N/g/b/v/Uozumi\G\hamasaka\GH-1522-1H.als



DFILE GH-1522-1H.als
 COMNT GH-1522-1H
 DATIM 2013-05-01 09:59:37
 OBNUC 1H
 EXMOD proton_jsp
 OBFREQ 395.88 MHz
 OBSET 6.28 KHz
 OBFHN 0.87 Hz
 POINT 16384
 FREQU 7422.80 Hz
 SCANS 8
 ACQTM 2.2073 sec
 PD 5.0000 sec
 PW1 3.12 usec
 IRNUC 1H
 CTEMP 19.6 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.00 Hz
 RGAIN 36

GH-1522-13C

Z:\UOZUMI\G\hamasaka\GH-1522_carbon-1-1.jdf

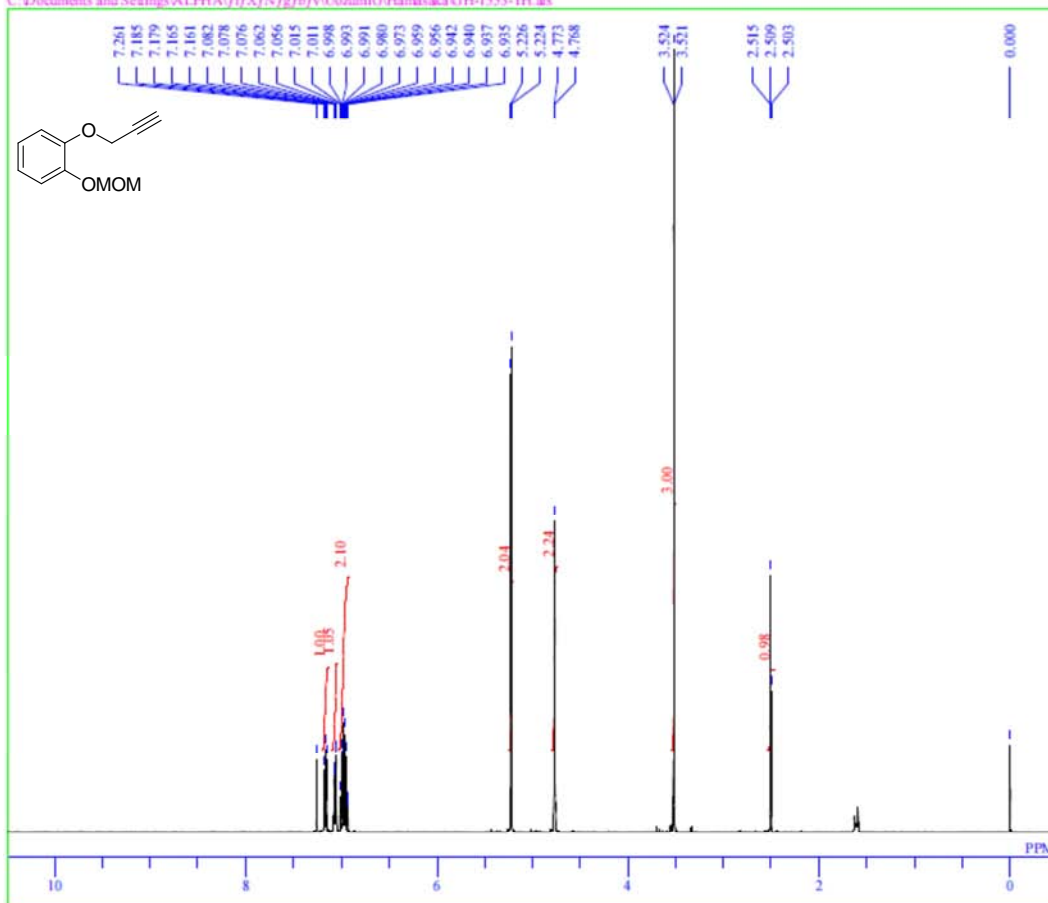


DFILE GH-1522_carbon-1-1.jdf
 COMNT GH-1522-13C
 DATIM 2013-05-01 10:03:13
 OBNUC 13C
 EXMOD carbon_jsp
 OBFREQ 99.55 MHz
 OBSET 5.13 KHz
 OBFHN 0.98 Hz
 POINT 32767
 FREQU 31250.00 Hz
 SCANS 488
 ACQTM 1.0486 sec
 PD 2.0000 sec
 PW1 3.42 usec
 IRNUC 13C
 CTEMP 18.6 c
 SLVNT CDCL3
 EXREF 77.00 ppm
 BF 0.20 Hz
 RGAIN 60

1-Methoxymethoxy-2-propargyloxybenzene (S4)

GH-1553-1H

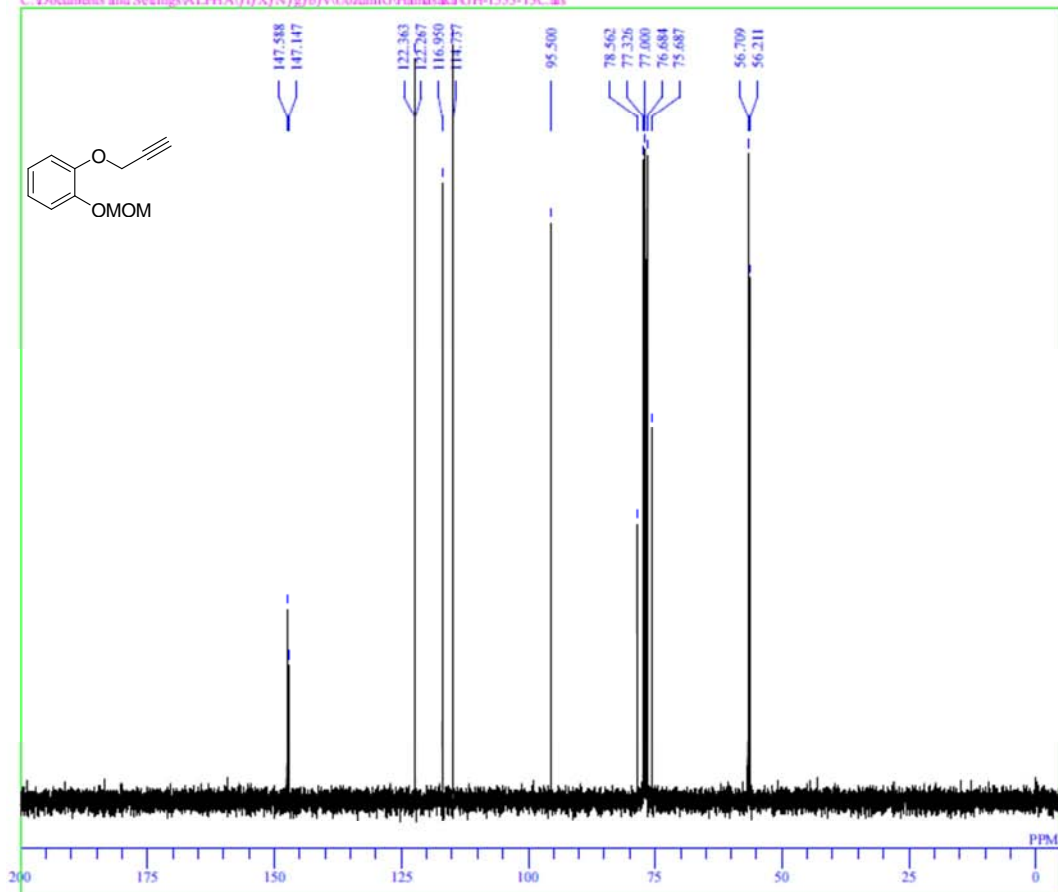
C:\Documents and Settings\ALPHA\ff\X(N\g\h\Uozumi\G\Hamusaka\GH-1553-1H.als



DFILE GH-1553-1H.als
 COMNT GH-1553-1H
 DATIM 2013-05-30 16:45:32
 OBNUC 1H
 EXMOD proton.jsp
 OBFRQ 395.88 MHz
 OBSET 6.28 KHz
 OBFIN 0.87 Hz
 POINT 16384
 FREQU 7422.80 Hz
 SCANS 8
 ACQTM 2.2073 sec
 PD 5.0000 sec
 PW1 3.12 usec
 IRNUC 1H
 CTEMP 18.7 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.00 Hz
 RGAIN 30

GH-1553-13C

C:\Documents and Settings\ALPHA\ff\X(N\g\h\Uozumi\G\Hamusaka\GH-1553-13C.als

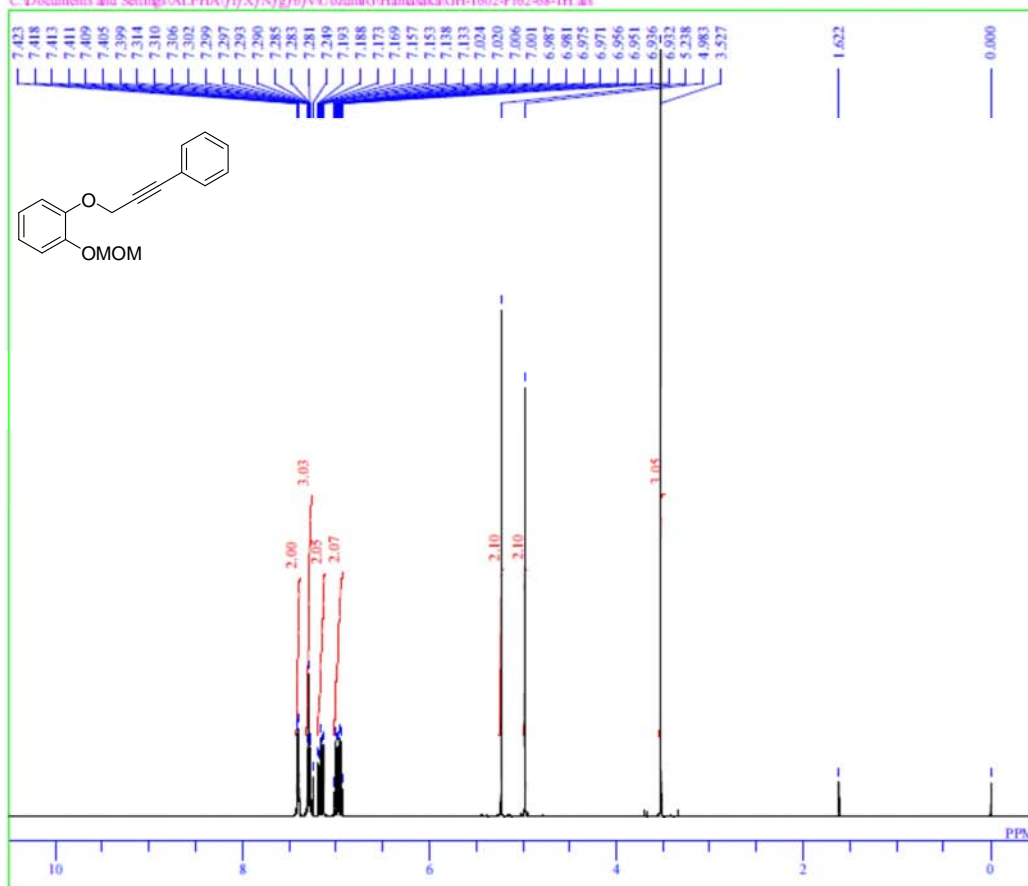


DFILE GH-1553-13C.als
 COMNT GH-1553-13C
 DATIM 2013-05-30 16:48:06
 OBNUC 13C
 EXMOD carbon.jsp
 OBFRQ 99.55 MHz
 OBSET 5.13 KHz
 OBFIN 0.98 Hz
 POINT 32767
 FREQU 31250.00 Hz
 SCANS 252
 ACQTM 1.0486 sec
 PD 2.0000 sec
 PW1 3.42 usec
 IRNUC 1H
 CTEMP 19.0 c
 SLVNT CDCL3
 EXREF 77.00 ppm
 BF 0.50 Hz
 RGAIN 60

1-Methoxymethoxy-2-phenylpropargyloxybenzene (S5)

GH-1602-Fr62-68-1H

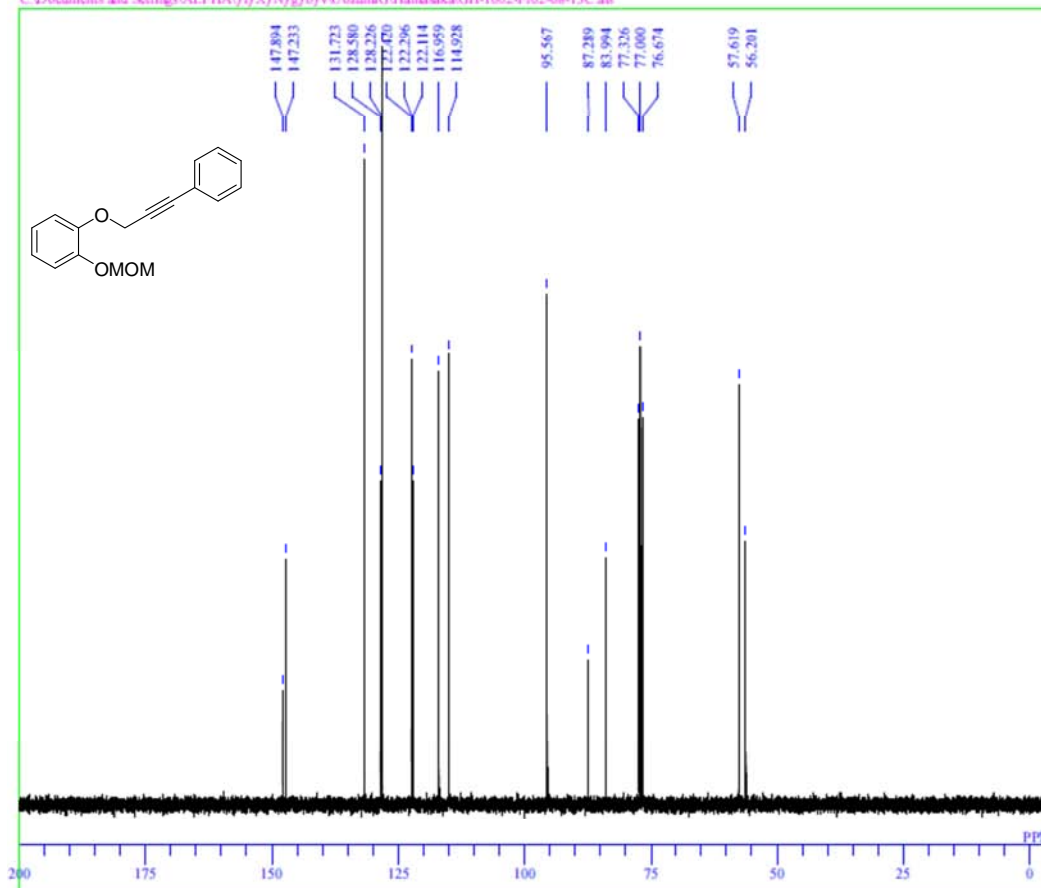
C:\Documents and Settings\ALPHA\ff\X\N\g\fb\U\ozumiG\Hansuka\GH-1602-Fr62-68-1H.als



DFILE GH-1602-Fr62-68-1H.als
 COMNT GH-1602-Fr62-68-1H
 DATIM 2013-08-01 18:41:22
 OBNUC 1H
 EXMOD proton_jxp
 OBFREQ 395.88 MHz
 OBSET 6.28 KHz
 OBFIN 0.87 Hz
 POINT 16384
 FREQU 7422.80 Hz
 SCANS 8
 ACQTM 2.2073 sec
 PD 5.0000 sec
 PWI 3.12 usec
 IRNUC 1H
 CTEMP 21.8 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.00 Hz
 RGAIN 28

GH-1602-Fr62-68-13C

C:\Documents and Settings\ALPHA\ff\X\N\g\fb\U\ozumiG\Hansuka\GH-1602-Fr62-68-13C.als

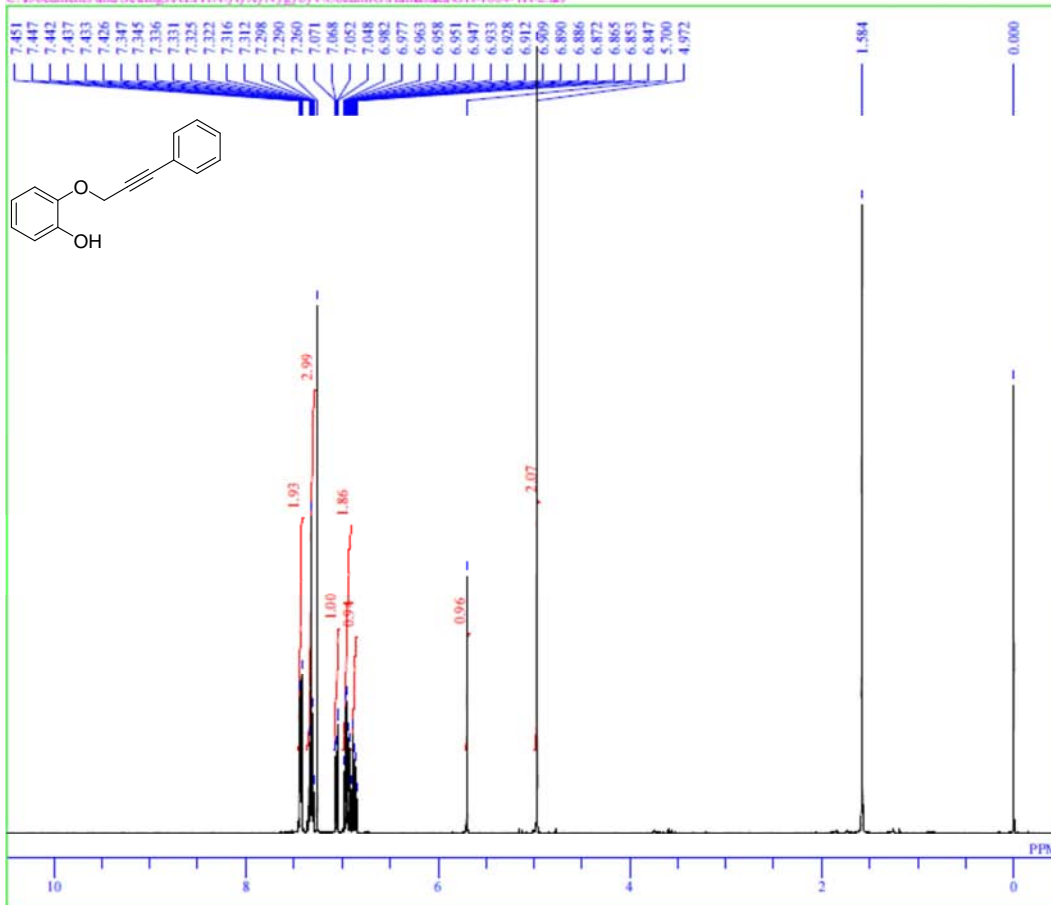


DFILE GH-1602-Fr62-68-13C.als
 COMNT GH-1602-Fr62-68-13C
 DATIM 2013-08-01 18:44:15
 OBNUC 13C
 EXMOD carbon_jxp
 OBFREQ 99.55 MHz
 OBSET 5.13 KHz
 OBFIN 0.98 Hz
 POINT 32767
 FREQU 31250.00 Hz
 SCANS 251
 ACQTM 1.0486 sec
 PD 2.0000 sec
 PWI 3.42 usec
 IRNUC 1H
 CTEMP 21.8 c
 SLVNT CDCL3
 EXREF 77.00 ppm
 BF 0.40 Hz
 RGAIN 60

2-Phenylpropargyloxyphenol (4)

GH-1604-1H

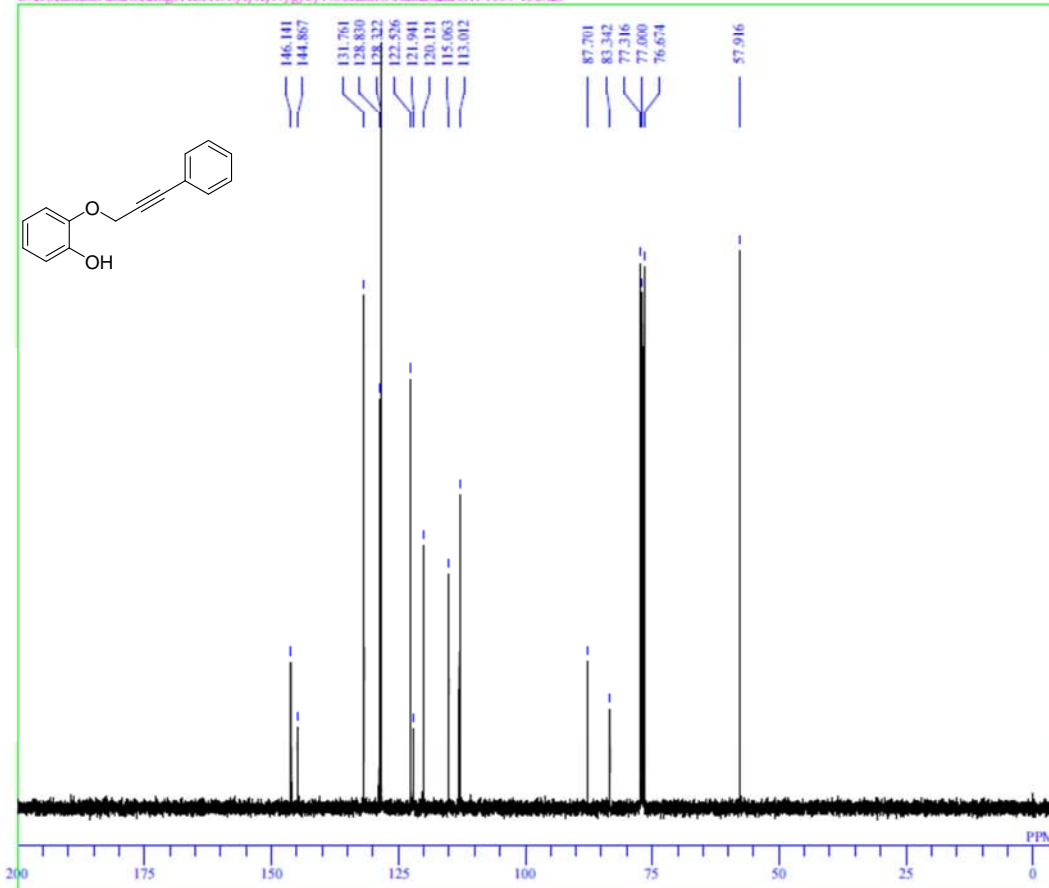
C:\Documents and Settings\ALPHA\ff\X\N\g\fb\vl\ozumi\GHamasaka\GH-1604-1H-2.als



DFILE GH-1604-1H-2.als
 COMNT GH-1604-1H
 DATIM 2013-08-02 23:29:22
 OBNUC 1H
 EXMOD proton.jsp
 OBFRQ 395.88 MHz
 OBSET 6.28 KHz
 OBFIN 0.87 Hz
 POINT 16384
 FREQU 7422.80 Hz
 SCANS 8
 ACQTM 2.2073 sec
 PD 5.0000 sec
 PW1 3.12 usec
 IRNUC 1H
 CTEMP 20.7 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.00 Hz
 RGAIN 42

GH-1604-13C

C:\Documents and Settings\ALPHA\ff\X\N\g\fb\vl\ozumi\GHamasaka\GH-1604-13C.als

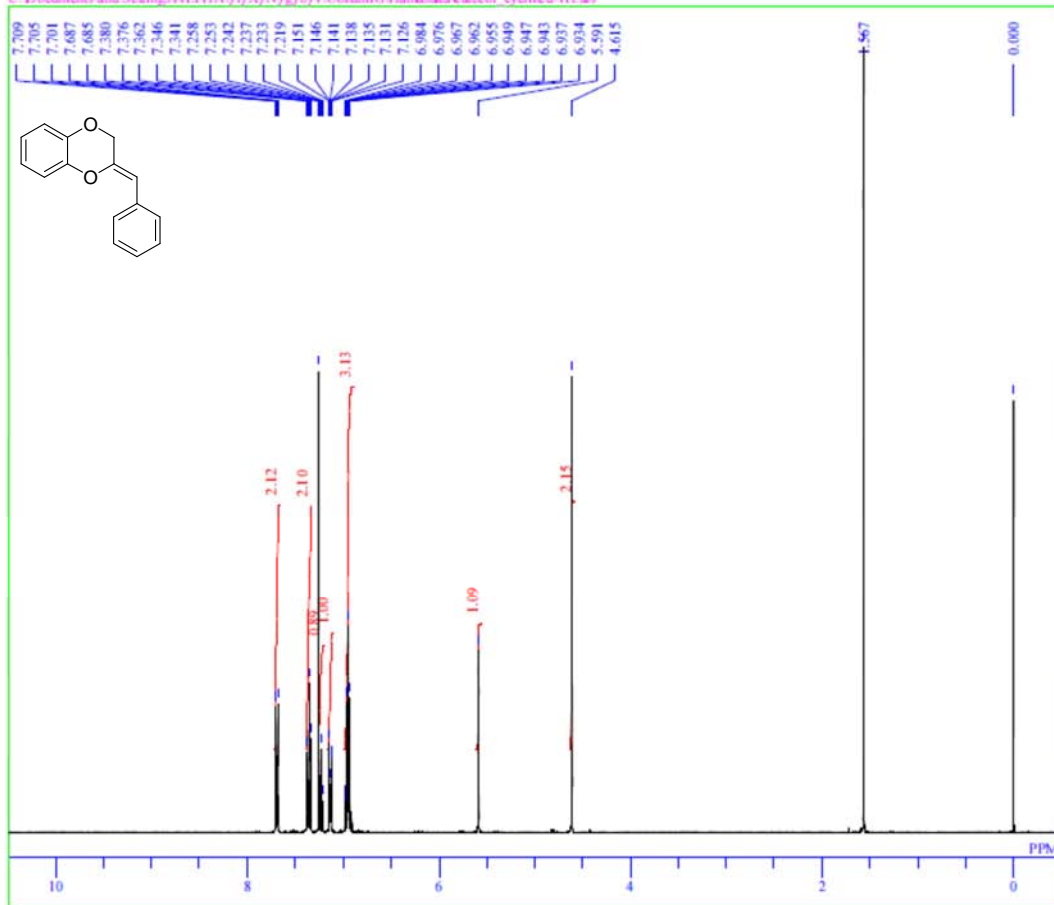


DFILE GH-1604-13C.als
 COMNT GH-1604-13C
 DATIM 2013-08-02 22:20:16
 OBNUC 13C
 EXMOD carbon.jsp
 OBFRQ 99.55 MHz
 OBSET 5.13 KHz
 OBFIN 0.98 Hz
 POINT 32767
 FREQU 31250.00 Hz
 SCANS 481
 ACQTM 1.0486 sec
 PD 2.0000 sec
 PW1 3.42 usec
 IRNUC 1H
 CTEMP 20.8 c
 SLVNT CDCL3
 EXREF 77.00 ppm
 BF 0.50 Hz
 RGAIN 60

2,3-Dihydro-2-(Z)-phenylmethylene-1,4-benzodioxin (**5**)

catecol_cyclized-1H

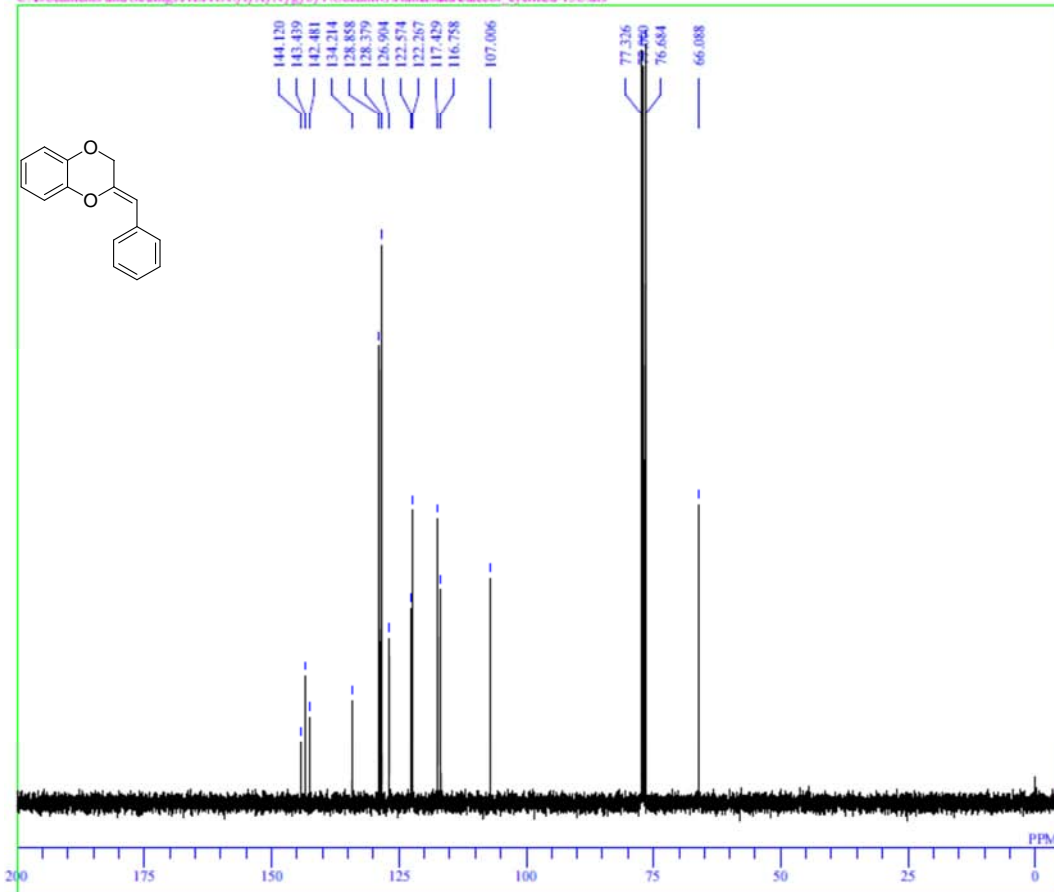
C:\Documents and Settings\ALPHA\ff\X\N\g\h\y\Uozumi\G\Hamsaka\catecol_cyclized-1H.als



DFILE catecol_cyclized-1H.als
 COMNT catecol_cyclized-1H
 DATIM 2013-08-03 15:14:60
 OBNUC 1H
 EXMOD proton_jsp
 OBFREQ 395.88 MHz
 OBSET 6.28 KHz
 OBFIN 0.87 Hz
 POINT 13107
 FREQU 5938.24 Hz
 SCANS 8
 ACQTM 2.2073 sec
 PD 5.0000 sec
 PW1 3.12 usec
 IRNUC 1H
 CTEMP 20.9 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.00 Hz
 RGAIN 42

catecol_cyclized-13C

C:\Documents and Settings\ALPHA\ff\X\N\g\h\y\Uozumi\G\Hamsaka\catecol_cyclized-13C.als



DFILE catecol_cyclized-13C.als
 COMNT catecol_cyclized-13C
 DATIM 2013-08-03 15:57:16
 OBNUC 13C
 EXMOD carbon_jsp
 OBFREQ 99.55 MHz
 OBSET 5.13 KHz
 OBFIN 0.98 Hz
 POINT 32767
 FREQU 31250.00 Hz
 SCANS 313
 ACQTM 1.0486 sec
 PD 2.0000 sec
 PW1 3.42 usec
 IRNUC 13C
 CTEMP 21.0 c
 SLVNT CDCL3
 EXREF 77.00 ppm
 BF 0.50 Hz
 RGAIN 60