Sequential Michael addition /retro-Claisen condensation of aromatic *β*-diketones with *α, β*-unsaturated esters: an approach to 1, 5-ketoesters

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Contents of Supporting Information:

I. Experimental Section

General Information

All reactions were carried out using 20 mL sealed tube. All chemicals obtained from commercial suppliers were utilized without further purification unless otherwise noted. NMR Spectra were recorded on a Bruker 600 MHz NMR spectrometer. The chemical shift is given in dimensionless δ values and is frequency referenced relative to TMS in ^{1}H and ^{13}C NMR spectroscopy. Chemical shifts are reported relative to $CDCl_3$ (δ =7.26 ppm) for ^{1}H NMR and relative to $CDCl_3$ (δ =77 ppm) for ^{13}C NMR. FT-IR spectra were tested by Bruker RFS100/S spectrophotometer (Bio-Rad, Cambridge, MA, USA) using KBr pellets in the 400-4000 cm^{-1} range. The mass spectra were recorded on LCMS-2010A and the high resolution mass spectra (HRMS) were recorded on an Ion Spec FTICR mass spectrometer with ESI resource.

General experimental procedures for the synthesis of compounds 4 and 7

Aromatic *β*-diketones (0.5 mmol), acrylates (1 mmol), K_2CO_3 (0.1 mmol, 10 mol %) and dehydrated alcohol (0.5-2 mL) as noted were put into the 20 mL sealed tube. The reaction mixture was stirred at 85 °C for required time. After cooled to room temperature, the mixture was concentrated in vacuo. The desired products **4**/**7** were obtained in the corresponding yields after purification by flash chromatography on neutral Al_2O_3 with the eluent (EA/PE = 1/15-1/5).

General procedures for the synthesis of compounds 5

1, 3-diphenylpropane-1,3-dione (0.5 mmol), acrylates (1 mmol), K_2CO_3 (0.1 mmol, 10 mol %) and the corresponding alcohol solution (0.5-2 mL) as noted were put into the 20 mL sealed tube. The reaction mixture was stirred at 85 °C for required time. After cooled to room temperature, the mixture was concentrated via vacuum distillation. The desired products **5** were obtained in the corresponding yields after purification by flash chromatography on neutral Al_2O_3 with the eluent (EA/PE = 1/15-1/5).

II. Spectra data of the products

*Ethyl 5-oxo-5-phenylpentanoate (**4a**)*:[1] The title compound was prepared according to general procedure for the synthesis of compounds **4**, the crude product was purified by column chromatography on neutral Al_2O_3 and eluted with ethyl acetate/petroleum ether (1/5) to afford a colourless oily liquid：R_f = 0.47 (EA/PE 1/5). Isolated yield: 107 mg, 98%. ^{1}H NMR (600 MHz, $CDCl_3$, 25 °C, TMS) δ 7.97 (d, *J* = 7.6 Hz, 2H), 7.56 (t, *J* = 7.4 Hz, 1H), 7.46 (t, *J* = 7.6 Hz, 2H),

4.14 (q, *J* = 7.1 Hz, 2H), 3.06 (t, *J* = 7.2 Hz, 2H), 2.43 (t, *J* = 7.2 Hz, 2H), 2.08 (p, *J* = 7.2 Hz, 2H), 1.26 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (151 MHz, $CDCl_3$, 25 ºC, TMS) δ 199.45, 173.26, 136.96, 133.06, 128.62, 128.06, 60.37, 37.52, 33.48, 19.50, 14.25; MS (ESI): *m/z*: $[M + H]^+$ 221.1; IR (KBr) υ 3441, 3061, 2980, 2937, 2390, 1731, 1685, 1597, 1580, 1448, 1208, 1002, 691cm^{-1}.

*Ethyl 2-methyl-5-oxo-5-phenylpentanoate (**4b**):* The title compound was prepared according to general procedure for the synthesis of compounds **4** from 1,3-diphenylpropane-1,3-dione (112 mg, 0.5 mmol) and *tert*-butyl methacrylate (126 μL, 1 mmol), the crude product was purified by column chromatography on neutral Al_2O_3 and eluted with ethyl acetate/petroleum ether (1/10) to afford a colourless oily liquid：R_f = 0.62 (EA/PE 1/10). Isolated yield: 84 mg, 72%. ^{1}H NMR (600 MHz, $CDCl_3$, 25 ºC, TMS): δ 7.95 (d, J = 7.2 Hz, 2H), 7.55 (t, J = 7.4 Hz, 1H), 7.46 (t, J = 7.7 Hz, 2H), 4.14 (q, J = 7.1 Hz, 2H), 3.10-2.92 (m, 2H), 2.56 (dq, J = 14.0, 7.0 Hz, 1H), 2.13-1.87 (m, 2H), 1.30-1.17 (m, 6H); ^{13}C NMR (151 MHz, $CDCl_3$, 25 ºC, TMS) δ 199.47, 176.17, 136.93, 132.98, 128.57, 128.01, 60.29, 38.93, 36.05, 27.98, 17.28, 14.21; HRMS (ESI-TOF) m/z: $[M+H]^+$ Calcd for $C_{14}H_{19}O_3$ 235.1334; Found 235.1329; IR (KBr) υ 3440, 3062, 2977, 2936, 2390, 1729, 1686, 1449, 1159, 744, 658 cm^{-1}.

*Ethyl 5-(4-methoxyphenyl)-5-oxopentanoate (**4d**):* [2] The title compound was prepared according to general procedure for the synthesis of compounds **4** from 1,3-bis(4-methoxyphenyl) propane-1,3-dione (142 mg, 0.5 mmol) and ethyl acrylate (108 μL, 1 mmol), the crude product was purified by column chromatography on neutral Al_2O_3 and eluted with ethyl acetate/petroleum ether (1/5) to afford a white solid：R_f = 0.31 (EA/PE =1/5). Isolated yield: 175 mg, 70%; Melting point: 58-59 ºC. ^{1}H NMR (600 MHz, $CDCl_3$, 25 ºC, TMS) δ 7.95 (d, J = 8.6 Hz, 2H), 6.93 (d, J = 8.6 Hz, 2H), 4.14 (q, J = 7.1 Hz, 2H), 3.86 (s, 3H), 2.99 (t, J = 7.2 Hz, 2H), 2.42 (t, J = 7.2 Hz, 2H), 2.06 (p, J = 7.2 Hz, 2H), 1.25 (t, J = 7.1 Hz, 3H); ^{13}C NMR (151 MHz, $CDCl_3$, 25 ºC, TMS) δ 197.99, 173.27, 163.47, 130.27, 130.01, 113.72, 60.28, 55.42, 37.13, 33.49, 19.67, 14.20; HRMS (ESI-TOF) m/z: $[M+H]^+$ Calcd for $C_{14}H_{19}O_4$ 251.1283; Found 251.1277; IR (KBr) υ 3452, 3416, 2982, 2943, 2845, 1929, 1734, 1668, 1313, 1279, 1217, 1184, 987, 834, 753, 586 cm^{-1}.

*Ethyl 5-oxo-5-(pyridine-2-yl)pentanoate (**4e**):* [3] The title compound was prepared according to general procedure for the synthesis of compounds **4**, the crude product was purified by column chromatography on neutral Al_2O_3 and eluted with ethyl acetate/petroleum ether (1/5) to afford a colourless oily liquid : R_f = 0.28 (EA/PE =1/5). Isolated yield: 83 mg, 75%. ^{1}H NMR (600 MHz,

$CDCl_3$, 25 ºC, TMS) δ 8.67 (d, J = 4.7 Hz, 1H), 8.04 (d, J = 7.8 Hz, 1H), 7.84 (t, J = 7.7 Hz, 1H), 7.51-7.44 (m, 1H), 4.14 (q, J = 7.1 Hz, 2H), 3.29 (t, J = 7.3 Hz, 2H), 2.44 (t, J = 7.4 Hz, 2H), 2.08 (p, J = 7.3 Hz, 2H), 1.25 (t, J = 7.1 Hz, 3H); ^{13}C NMR (151 MHz, CDCl3, 25 ºC, TMS) δ 201.08, 173.20, 153.33, 148.91, 136.83, 127.06, 121.69, 60.24, 36.80, 33.61, 19.22, 14.19; MS (ESI) m/z: $[M]^+$ 221.0; IR (KBr) υ 3444, 3056, 2938, 2390, 1732, 1697, 1583, 1439, 1373, 994, 682 cm^{-1}.

*Ethyl 5-(4-(tert-butyl)phenyl)-5-oxopentanoate (**4f**):*[4] The title compound was prepared according to general procedure for the synthesis of compounds **4**, the crude product was purified by column chromatography on neutral Al_2O_3 and eluted with ethyl acetate/petroleum ether (1/5) to afford a colourless oily liquid : R_f = 0.53 (EA/PE =1/5). Isolated yield: 55 mg, 40%. ^{1}H NMR (600 MHz, $CDCl_3$, 25 ºC, TMS) δ 7.91 (d, J = 8.1 Hz, 2H), 7.47 (d, J = 8.1 Hz, 2H), 4.14 (q, J = 7.1 Hz, 2H), 3.03 (t, J = 7.1 Hz, 2H), 2.42 (t, J = 7.2 Hz, 2H), 2.07 (p, J = 7.1 Hz, 2H), 1.34 (s, 9H), 1.25 (t, J = 7.1 Hz, 3H); ^{13}C NMR (151 MHz, $CDCl_3$, 25 ºC, TMS) δ 199.08, 173.24, 156.76, 134.36, 128.01, 125.51, 60.29, 37.38, 35.07, 33.48, 31.07, 19.57, 14.22; HRMS (ESI-TOF) m/z: $[M+H]^+$ Calcd for $C_{17}H_{25}O_3$ 277.1804; Found 277.1799; IR (KBr) υ 3349, 3055, 2964, 2907, 2870, 1733, 1682, 1605, 1566, 1407, 1191, 1028, 988, 734, 545 cm^{-1}.

*Methyl 5-oxo-5-phenylpentanoate (**5a**):* [5] The title compound was prepared according to general procedure for the synthesis of compounds **5**, the crude product was purified by column chromatography on neutral Al_2O_3 and eluted with ethyl acetate/petroleum ether (1/5) to afford a colourless oily liquid : R_f = 0.50 (EA/PE =1/5). Isolated yield: 77 mg, 75%. ^{1}H NMR (600 MHz, $CDCl_3$, 25 ºC, TMS) δ 7.96 (d, J = 7.8 Hz, 2H), 7.56 (t, J = 7.4 Hz, 1H), 7.46 (t, J = 7.7 Hz, 2H), 3.68 (s, 3H), 3.06 (t, J = 7.2 Hz, 2H), 2.45 (t, J = 7.2 Hz, 2H), 2.08 (p, J = 7.2 Hz, 2H); ^{13}C NMR (151 MHz, $CDCl_3$, 25 ºC, TMS) δ 199.37, 173.68, 136.94, 133.07, 128.62, 128.05, 51.54, 37.49, 33.19, 19.43; MS (ESI) *m/z:* $[M + H]^+$ 207.1; IR (KBr) υ 3450, 3353, 3061, 2951, 1735, 1685, 1448, 1257, 745, 658 cm^{-1}.

*Butyl 5-oxo-5-phenylpentanoate (**5b**):* [6] The title compound was prepared according to general procedure for the synthesis of compounds **5**, the crude product was purified by column chromatography on neutral Al_2O_3 and eluted with ethyl acetate/petroleum ether (1/5) to afford a colourless oily liquid : R_f = 0.47 (EA/PE =1/5). Isolated yield: 99 mg, 80%. ^{1}H NMR (600 MHz, $CDCl_3$, 25 ºC, TMS) δ 7.96 (d, J = 8.3 Hz, 2H), 7.56 (t, J = 7.4 Hz, 1H), 7.46 (t, J = 7.7 Hz, 2H), 4.09 (t, J = 6.7 Hz, 2H), 3.05 (t, J = 7.2 Hz, 2H), 2.44 (t, J = 7.2 Hz, 2H), 2.08 (p, J = 7.2 Hz, 2H),

1.64-1.54 (m, 2H), 1.42-1.32 (m, 2H), 0.93 (t, J = 7.4 Hz, 3H); ^{13}C NMR (151 MHz, $CDCl_3$, 25 ºC, TMS) δ 199.41, 173.33, 136.96, 133.05, 128.62, 128.05, 64.31, 37.52, 33.47, 30.73, 19.52, 19.17, 13.69; HRMS (ESI-TOF) m/z: $[M+H]^+$ Calcd for $C_{15}H_{21}O_3$ 249.1491; Found 249.1489; IR (KBr) υ 3440, 3159, 2961, 2873, 2254, 1730, 1686, 1598, 1450, 1209, 1067, 912, 733 cm^{-1}.

*Hexyl 5-oxo-5-phenylpentanoate (**5c**):* [7] The title compound was prepared according to general procedure for the synthesis of compounds **5**, the crude product was purified by column chromatography on neutral Al_2O_3 and eluted with ethyl acetate/petroleum ether (1/5) to afford a colourless oily liquid : R_f = 0.47 (EA/PE =1/5). Isolated yield: 83 mg, 75%. ^{1}H NMR (600 MHz, $CDCl_3$, 25 ºC, TMS) δ 7.96 (d, J = 8.0 Hz, 2H), 7.57 (dd, J = 17.8,10.4 Hz, 1H), 7.46 (t, J = 7.5 Hz, 2H), 4.07 (t, J = 6.7 Hz, 2H), 3.06 (t, J=7.2Hz, 2H), 2.44 (t, J = 7.2 Hz, 2H), 2.12-2.03 (m, 2H), 1.65-1.57 (m, 2H), 1.37-1.24 (m, 6H), 0.88 (t, J = 6.7 Hz, 3H); ^{13}C NMR (151 MHz, $CDCl_3$, 25 ºC, TMS) δ 199.38, 173.29, 136.89, 132.99, 128.55, 127.99, 64.57, 37.46, 33.41, 31.38, 28.58, 25.56, 22.47, 19.45, 13.91; MS (ESI) *m/z:* $[M + H]^+$ 277.2; IR (KBr) υ 3029, 2921, 2857, 1734, 1684, 1958, 1540, 1452, 1235, 1071, 1015, 812, 693 cm^{-1}.

*Benzyl 5-oxo-5-phenylpentanoate (**5d**):* The title compound was prepared according to general procedure for the synthesis of compounds **5**, the crude product was purified by column chromatography on neutral Al_2O_3 and eluted with ethyl acetate/petroleum ether (1/10) to afford a colourless oily liquid : R_f = 0.38 (EA/PE 1/10). Isolated yield: 69 mg, 49%. ^{1}H NMR (600 MHz, $CDCl_3$, 25 ºC, TMS) δ 7.92 (d, J = 7.3 Hz, 2H), 7.55 (t, J = 7.4 Hz, 1H), 7.44 (t, J = 7.7 Hz, 2H), 7.37-7.28 (m, 5H), 5.13 (s, 2H), 3.03 (t, J = 7.2 Hz, 2H), 2.49 (t, J = 7.2 Hz, 2H), 2.09 (p, J = 7.2 Hz, 2H); ^{13}C NMR (151 MHz, $CDCl_3$, 25 ºC, TMS) δ 199.32, 173.03, 136.90, 136.04, 133.03, 128.59, 128.57, 128.22, 128.03, 66.23, 37.40, 33.42, 19.45; HRMS (ESI-TOF) m/z: $[M+H]^+$ Calcd for $C_{18}H_{19}O_3$ 283.1334; Found 283.1332; IR (KBr) υ 3443, 3063, 2941, 1734, 1685, 1597, 1450, 1257, 1211, 1074, 745, 694 cm^{-1}.

*tert-Butyl 5-oxo-5-phenylpentanoate (**5e**):*[8] The title compound was prepared according to general procedure for the synthesis of compounds **5**, the crude product was purified by column chromatography on neutral Al_2O_3 and eluted with ethyl acetate/petroleum ether (1/10) to afford a colourless oily liquid : R_f = 0.63 (EA/PE 1/10). Isolated yield: 112 mg, 90%. ^{1}H NMR (600 MHz, $CDCl_3$, 25 ºC, TMS) δ 7.96 (d, J = 8.4 Hz, 2H), 7.56 (t, J = 8.5 Hz, 1H), 7.46 (t, J = 7.7 Hz, 2H), 3.04 (t, J = 7.2 Hz, 2H), 2.34 (t, J = 7.2 Hz, 2H), 2.03 (p, J = 7.2 Hz, 2H), 1.45 (s, 9H); ^{13}C NMR

(151 MHz, $CDCl_3$, 25 ºC, TMS) δ 199.54, 172.57, 136.95, 132.98, 128.57, 128.02, 80.26, 37.51, 34.69, 28.12, 19.69; MS (ESI) *m/z*: $[M + Na]^+$ 271.1; IR (KBr) υ 3435, 3354, 3062, 2977, 2934, 1726, 1686, 1597, 1450, 1390, 1147, 751, 691, 658, 590 cm^{-1}.

*2-Methoxyethyl 5-oxo-5-phenylpentanoate (**5g**):* [9] The title compound was prepared according to general procedure for the synthesis of compounds **5**, the crude product was purified by column chromatography on neutral Al_2O_3 and eluted with ethyl acetate/petroleum ether (1/10) to afford a colourless oily liquid : R_f = 0.42 (EA/PE =1/10). Isolated yield: 100 mg, 80%. ^1H NMR (600 MHz, $CDCl_3$, 25 ºC, TMS) δ 7.96 (d, J = 7.3 Hz, 2H), 7.56 (t, J = 7.4 Hz, 1H), 7.46 (t, J = 7.7 Hz, 2H), 4.24 (t, 2H), 3.59 (t, 2H), 3.37 (s, 3H), 3.06 (t, J = 7.2 Hz, 2H), 2.49 (t, J = 7.2 Hz, 2H), 2.09 (p, J = 7.2 Hz, 2H); ^{13}C NMR (151 MHz, $CDCl_3$, 25 ºC, TMS) δ 199.33, 173.16, 136.87, 133.00, 128.55, 127.99, 70.42, 63.36, 58.88, 37.39, 33.23, 19.37; HRMS (ESI-TOF) m/z: $[M+H]^+$ Calcd for $C_{14}H_{19}O_4$ 251.1283; Found 251.1276; IR (KBr) υ 3451, 3353, 3061, 2934, 2893, 2821, 2393, 1733, 1684, 1580, 1449, 1179, 1100, 985, 749, 692, 658, 569 cm^{-1} .

*(Tetrahydrofuran-2-yl)ethyl-5-oxo-5-phenylpentanoate (**5h**):* The title compound was prepared according to general procedure for the synthesis of compounds **5**, the crude product was purified by column chromatography on neutral Al_2O_3 and eluted with ethyl acetate/petroleum ether (1/10) to afford a colourless oily liquid : R_f = 0.35 (EA/PE =1/10). Isolated yield: 125 mg, 91%. ^1H NMR (600 MHz, $CDCl_3$, 25 ºC, TMS) δ 7.96 (d, J = 7.8 Hz, 2H), 7.55 (t, J = 7.3 Hz, 1H), 7.46 (t, J = 7.6 Hz, 2H), 4.17 (dd, J = 11.3, 3.4 Hz, 1H), 4.11 (dt, J = 6.9, 5.2 Hz, 1H), 4.03 (dd, J = 11.3, 6.8 Hz, 1H), 3.87 (dd, J = 14.6, 7.2 Hz, 1H), 3.78 (dd, J = 14.5, 7.3 Hz, 1H), 3.06 (t, J = 7.2 Hz, 2H), 2.49 (t, J = 7.1 Hz, 2H), 2.08 (p, J = 7.1 Hz, 2H), 1.99 (dt, J = 12.4, 7.5 Hz, 1H), 1.95 – 1.83 (m, 2H), 1.60 (dt, J = 19.6, 7.5 Hz, 1H); ^{13}C NMR (151 MHz, $CDCl_3$, 25 ºC, TMS) δ 199.40, 173.19, 136.93, 133.04, 128.60, 128.05, 76.51, 68.42, 66.46, 37.47, 33.30, 28.02, 25.67, 19.45; HRMS (ESI-TOF) m/z: $[M+H]^+$ Calcd for $C_{16}H_{21}O_4$ 277.1440; Found 277.1431; IR (KBr) υ 3749, 3442, 3061, 2950, 2874, 2391, 2347, 1734, 1684, 1449, 1209, 1076, 999, 750, 692, 659, 570 cm^{-1}.

*2,2,2-Trifluoroethyl 5-oxo-5-phenylpentanoate (**5i**):* [10] The title compound was prepared according to general procedure for the synthesis of compounds **5**, the crude product was purified by column chromatography on neutral Al_2O_3 and eluted with ethyl acetate/petroleum ether (1/10) to afford a colourless oily liquid : R_f = 0.49 (EA/PE =1/10). Isolated yield: 22 mg, 16%. ^1H NMR (600 MHz, $CDCl_3$, 25 ºC, TMS) δ 7.96 (d, J = 7.2 Hz, 2H), 7.57 (t, J = 7.4 Hz, 1H), 7.47 (t, J = 7.7 Hz, 2H),

4.48 (q, J = 8.5 Hz, 2H), 3.07 (t, J = 7.1 Hz, 2H), 2.57 (t, J = 7.2 Hz, 2H), 2.12 (p, J = 7.1 Hz, 2H); ^{13}C NMR (151 MHz, $CDCl_3$, 25 ºC, TMS) δ 199.04, 171.65, 136.83, 133.19, 128.67, 128.02, 125.75, 123.92, 122.08, 120.24, 60.65, 60.40, 60.16, 59.92, 37.13, 32.78, 19.10; HRMS (ESI-TOF) m/z: $[M+H]^+$ Calcd for $C_{13}H_{14}F_3O_3$ 275.0895; Found 275.0891; IR (KBr) υ 3491, 3395, 3336, 3075, 3012, 2849, 2811, 2421, 1756, 1676, 1451, 1290, 1273, 1183, 1149, 991, 736, 658, 572 cm^{-1}.

*Benzyl 2-methyl-5-oxo-5-phenylpentanoate (**5k**):* The title compound was prepared according to general procedure for the synthesis of compounds **5**, the crude product was purified by column chromatography on neutral Al_2O_3 and eluted with ethyl acetate/petroleum ether (1/10) to afford a white solid : R_f = 0.45 (EA/PE =1/10). Isolated yield: 66 mg, 45%, melting point: 50-51 ºC. ^{1}H NMR (600 MHz, $CDCl_3$, 25 ºC, TMS) δ 7.87 (d, J = 7.8 Hz, 2H), 7.54 (t, J = 7.3 Hz, 1H), 7.42 (t, J = 7.4 Hz, 2H), 7.36-7.27 (m, 5H), 5.18-5.09 (m, 2H), 3.03-2.86 (m, 2H), 2.68-2.57 (m, 1H), 2.06 (td, J = 14.3, 8.5 Hz, 1H), 1.95 (td, J = 14.3, 6.1 Hz, 1H), 1.24 (d, J = 7.0 Hz, 3H); ^{13}C NMR (151 MHz, $CDCl_3$, 25 ºC, TMS) δ 199.40, 175.99, 136.85, 136.12, 132.98, 128.54, 128.16, 128.01, 66.16, 38.94, 35.95, 28.00, 17.29; HRMS (ESI-TOF) m/z: $[M+H]^+$ Calcd for $C_{19}H_{21}O_3$ 297.1491; Found 297.1489; IR (KBr) υ 3342, 3354, 3088, 2972, 2936, 2878, 2391, 1732, 1686, 1497, 1097, 695, 659 cm^{-1}.

III. References

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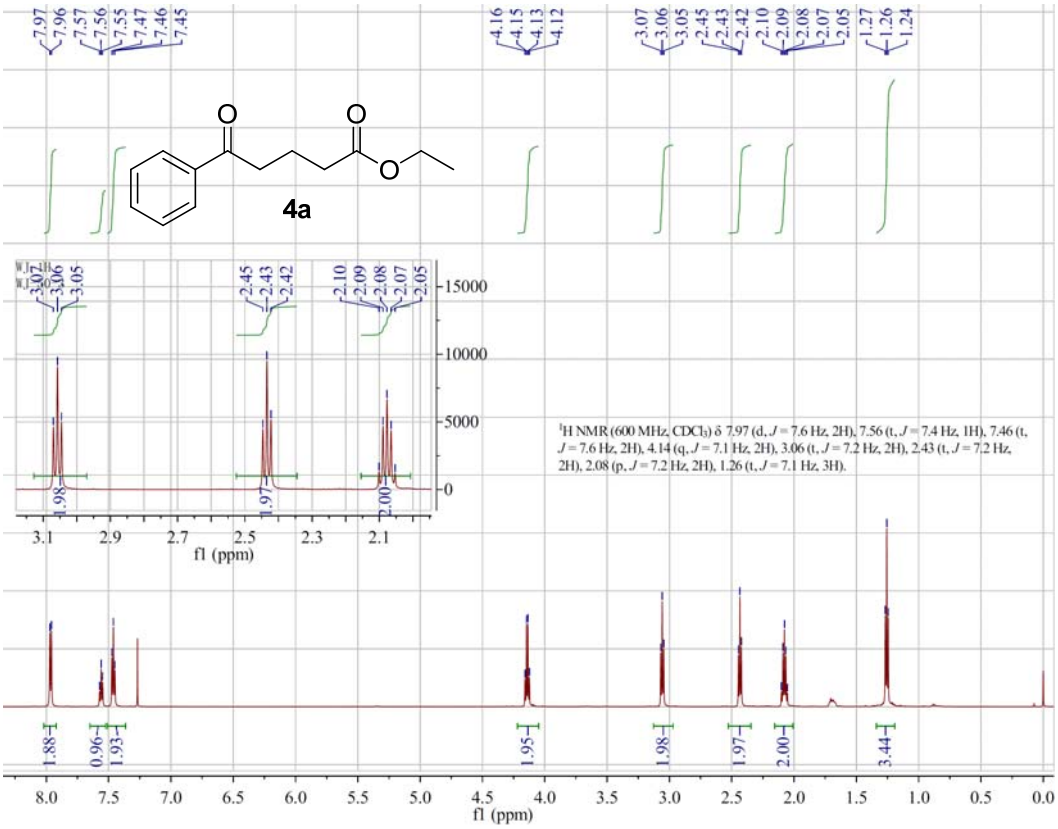
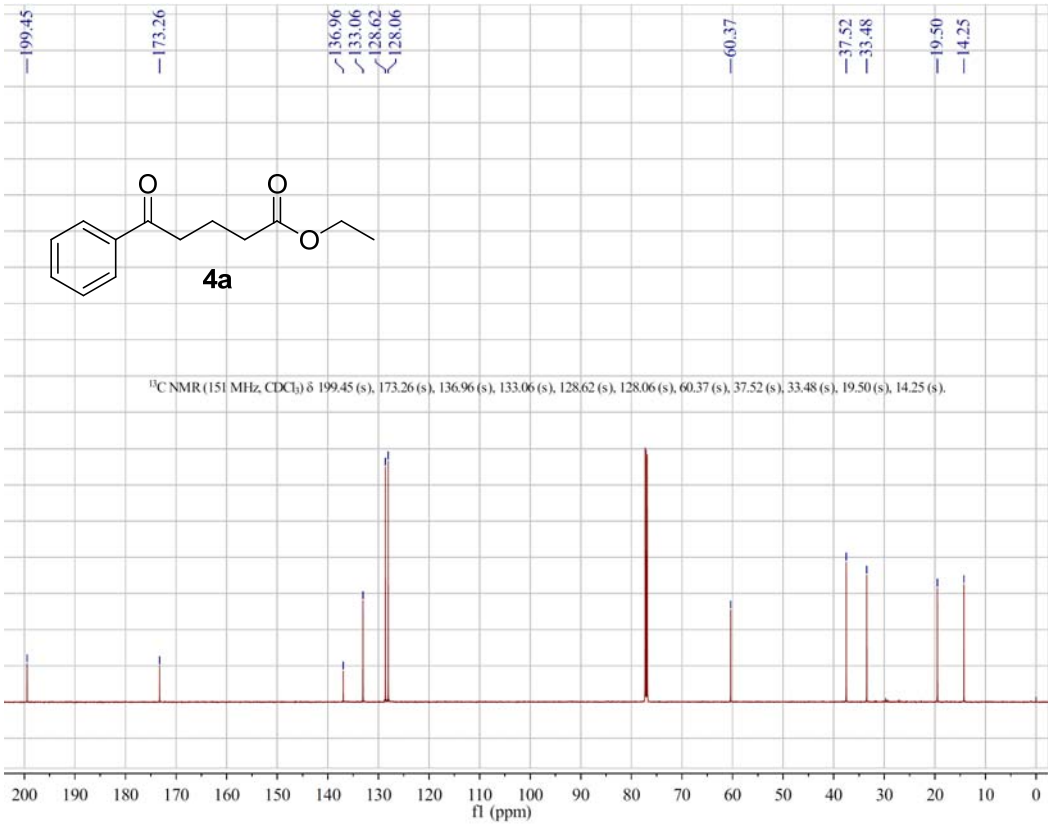
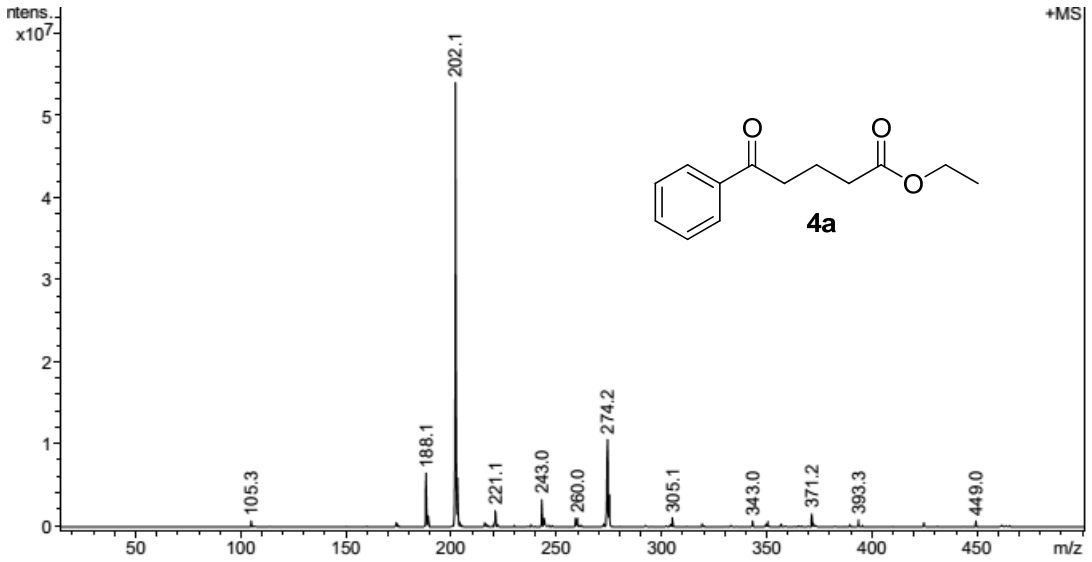
IV. Spectra

4a



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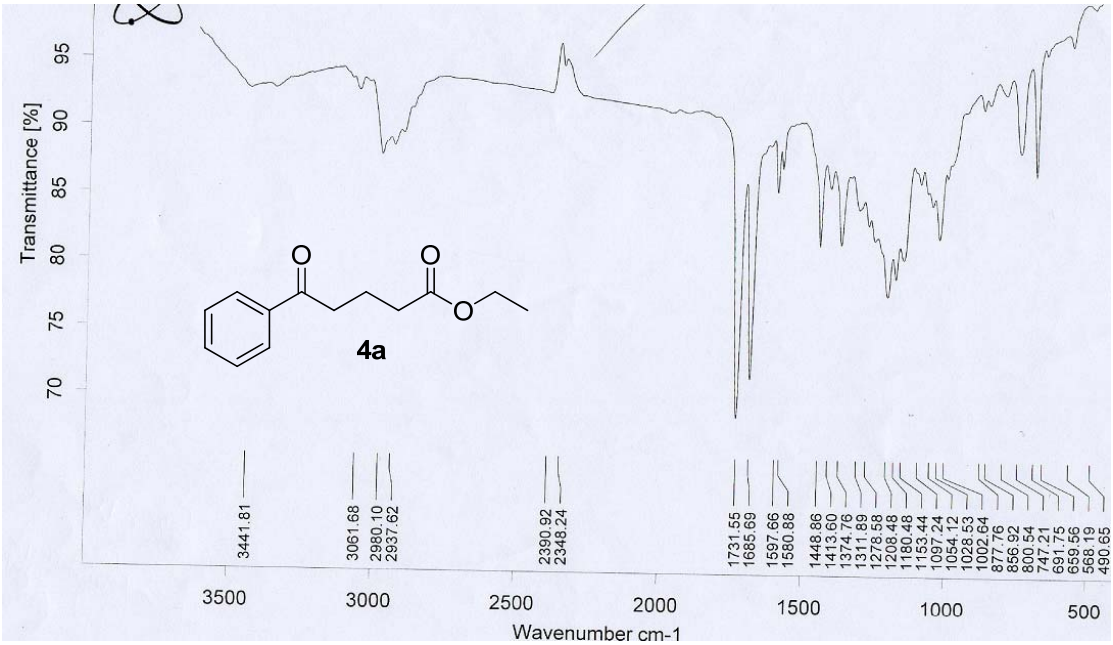
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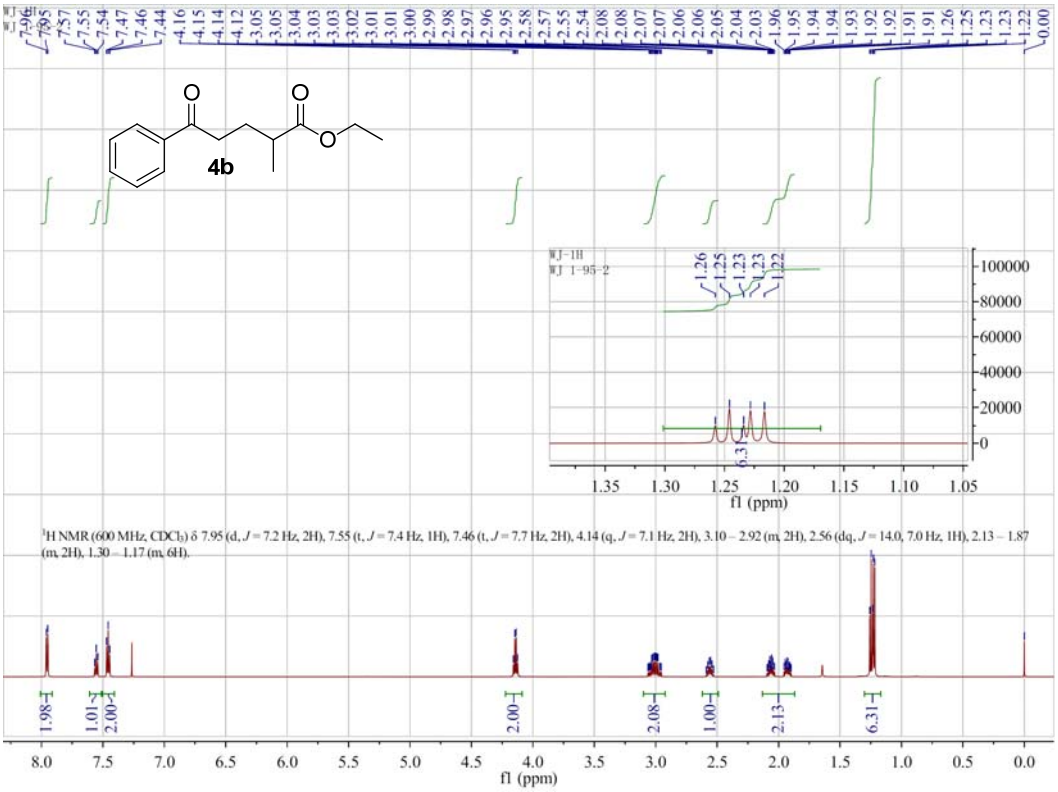
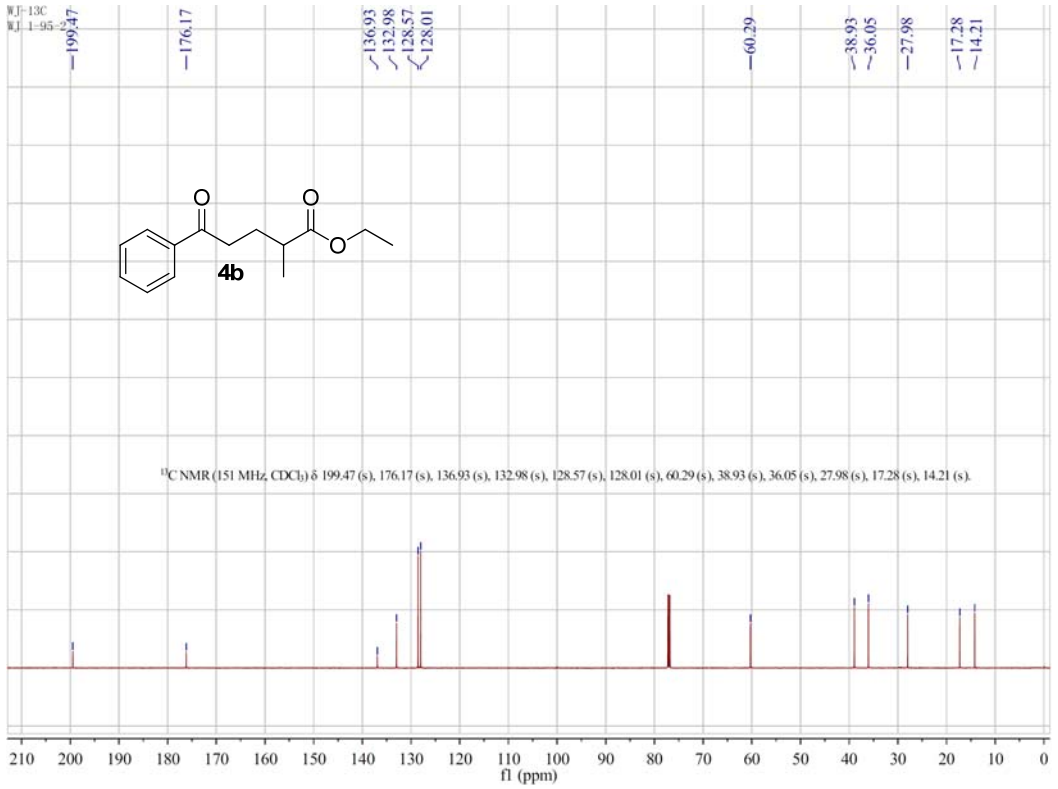
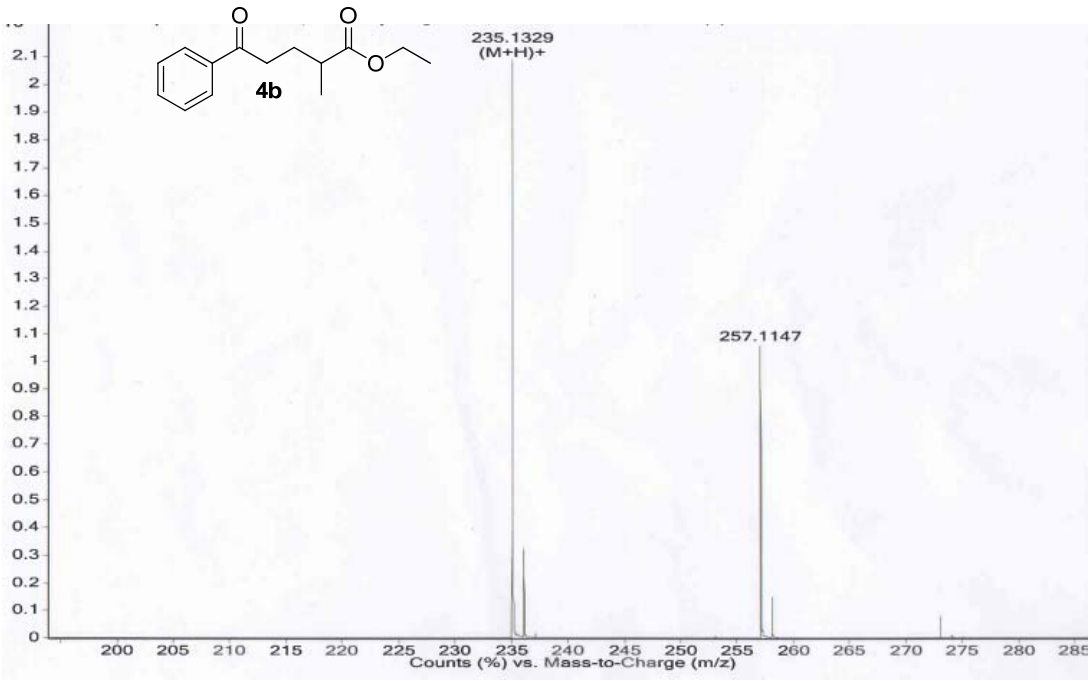
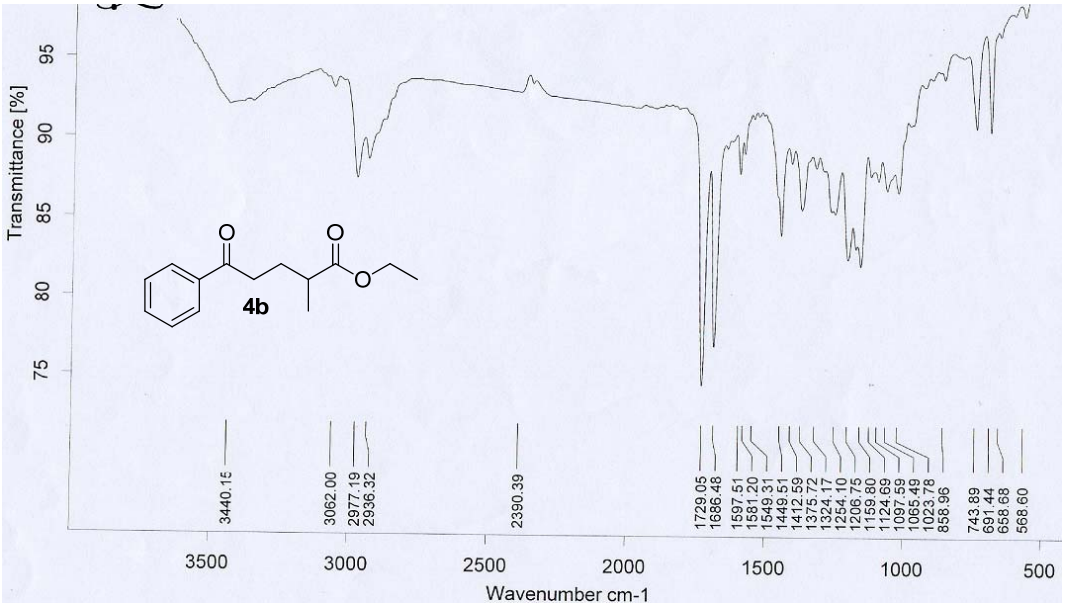
568.19
490.65
Wavenumber cm-1

4b

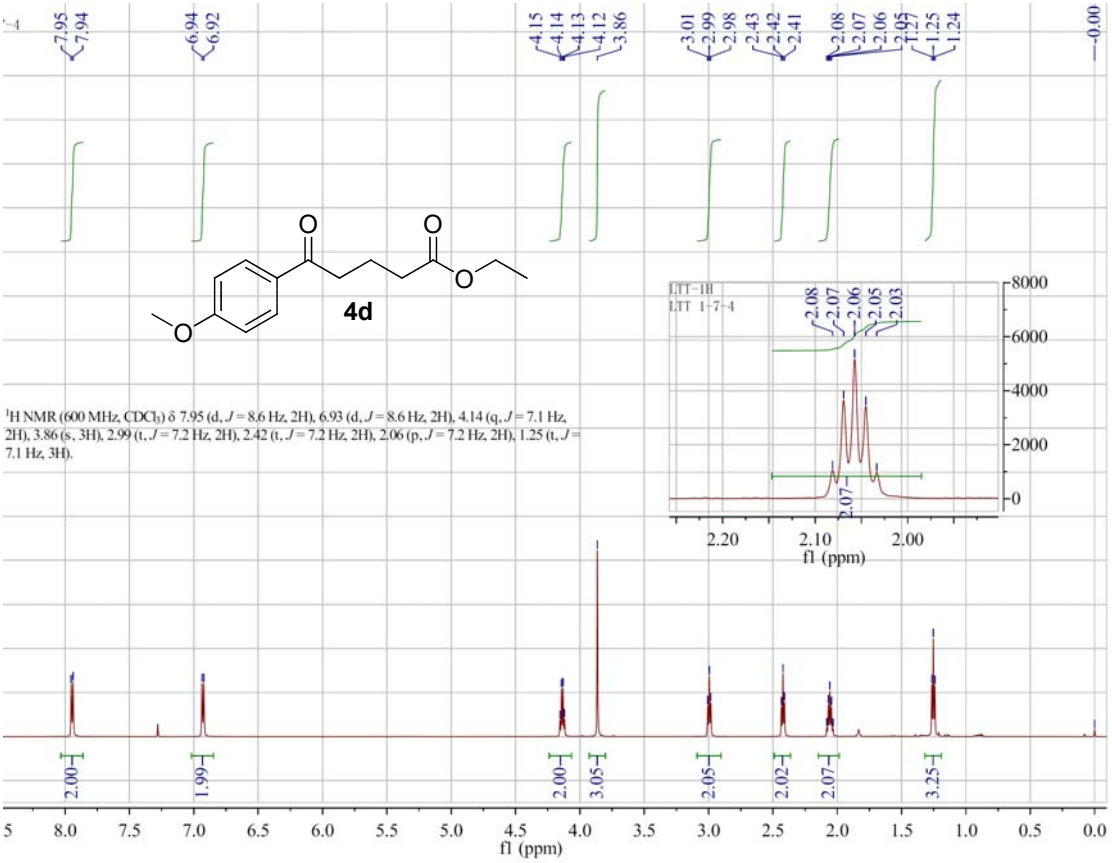

4b
^{1}H NMR (600 MHz, CDCl$_3$) δ 7.95 (d, J = 7.2 Hz, 2H), 7.55 (t, J = 7.4 Hz, 1H), 7.46 (t, J = 7.7 Hz, 2H), 4.14 (q, J = 7.1 Hz, 2H), 3.10 – 2.92 (m, 2H), 2.56 (dq, J = 14.0, 7.0 Hz, 1H), 2.13 – 1.87 (m, 2H), 1.30 – 1.17 (m, 6H).
f1 (ppm)

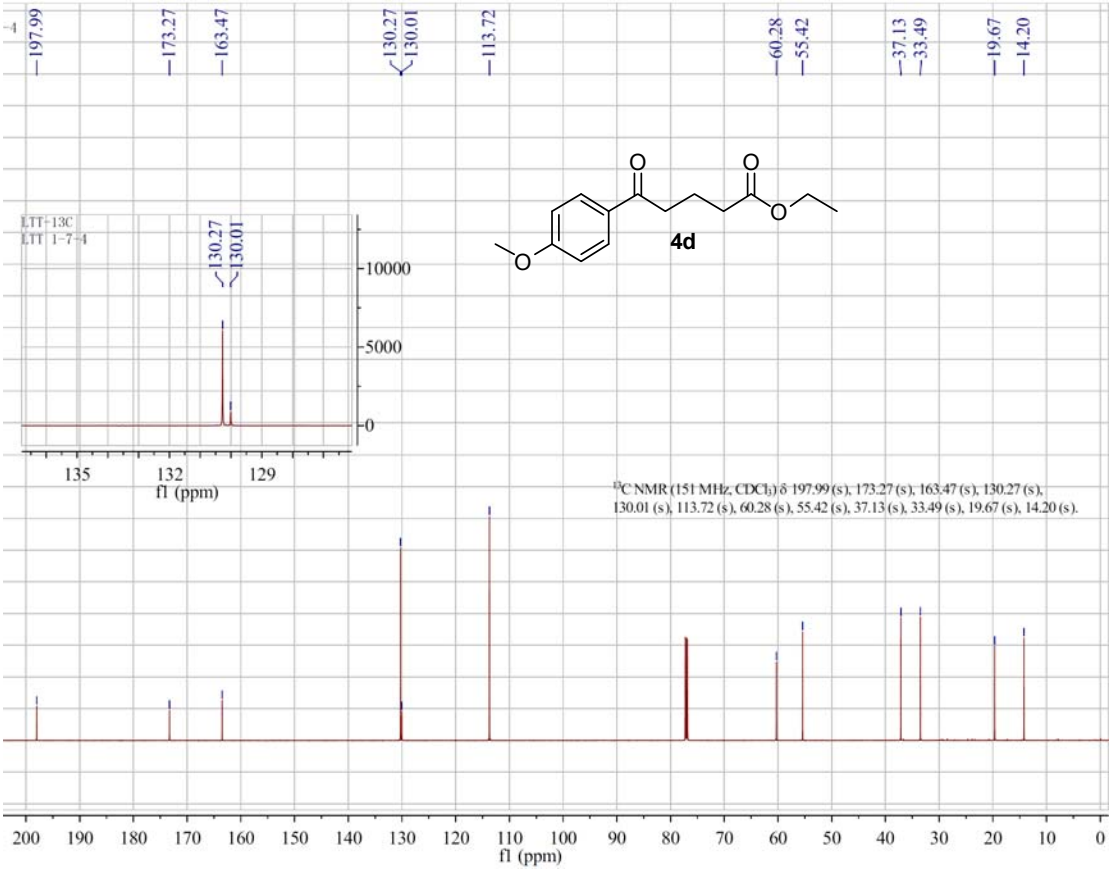

4b
^{13}C NMR (151 MHz, CDCl$_3$) δ 199.47 (s), 176.17 (s), 136.93 (s), 132.98 (s), 128.57 (s), 128.01 (s), 60.29 (s), 38.93 (s), 36.05 (s), 27.98 (s), 17.28 (s), 14.21 (s).
f1 (ppm)

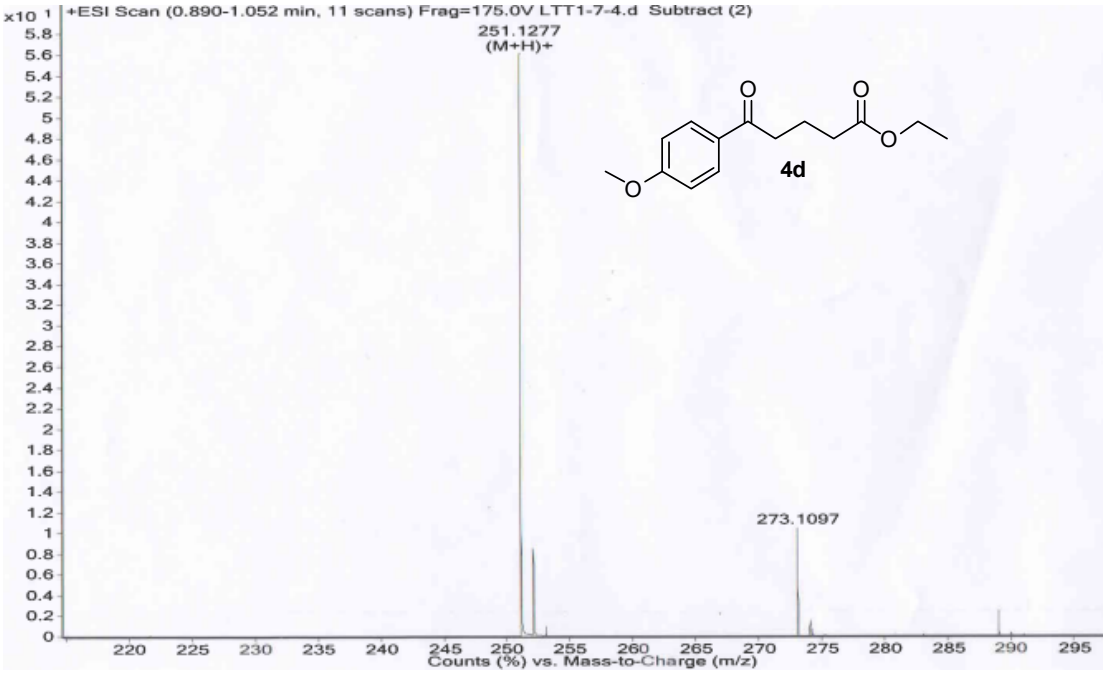

4b
235.1329
(M+H)+
257.1147
Counts (%) vs. Mass-to-Charge (m/z)

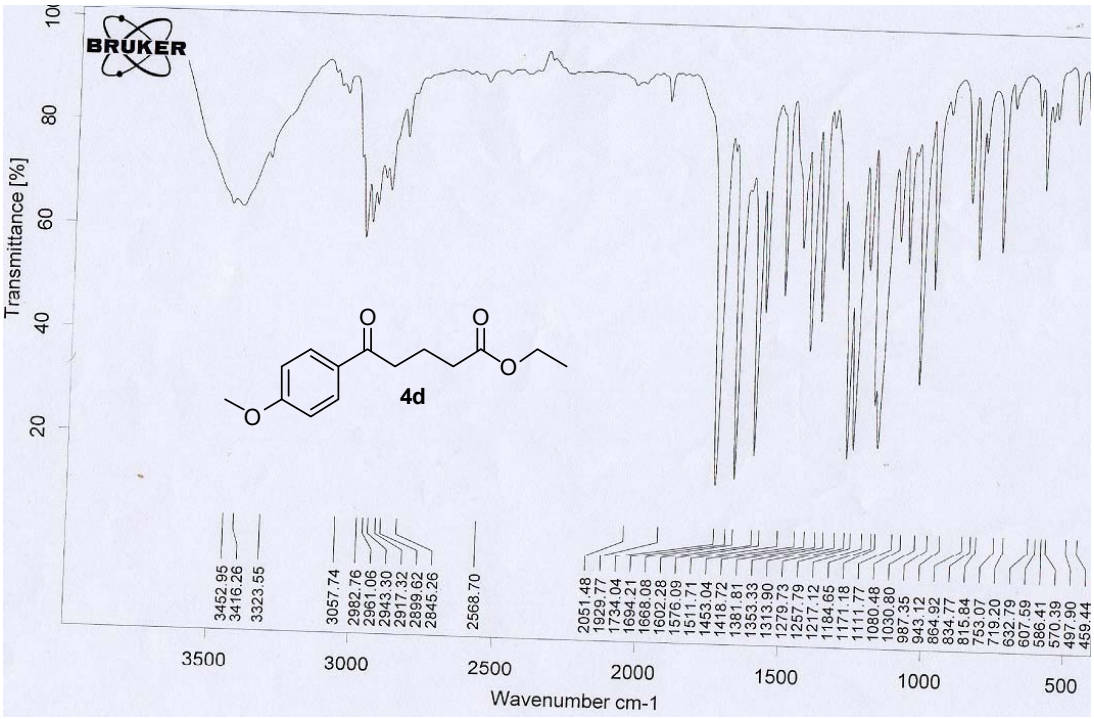

4b
Transmittance [%]
3440.15
3062.00
2977.19
2936.32
2390.39
1729.05
1686.48
1597.51
1581.20
1549.31
1449.51
1412.59
1375.72
1324.17
1254.10
1206.75
1159.80
1124.69
1097.59
1065.49
1023.78
858.96
743.89
691.44
658.68
568.60
Wavenumber cm-1

4d

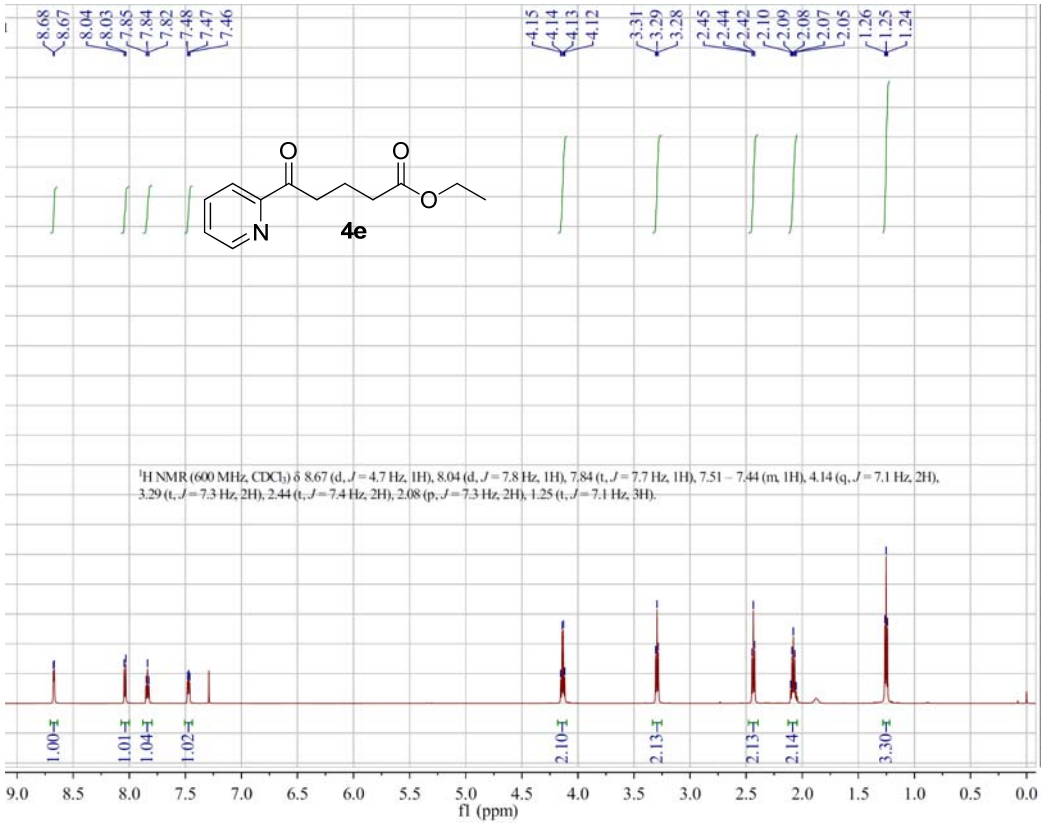

4d
^{1}H NMR (600 MHz, CDCl$_3$) δ 7.95 (d, J = 8.6 Hz, 2H), 6.93 (d, J = 8.6 Hz, 2H), 4.14 (q, J = 7.1 Hz, 2H), 3.86 (s, 3H), 2.99 (t, J = 7.2 Hz, 2H), 2.42 (t, J = 7.2 Hz, 2H), 2.06 (p, J = 7.2 Hz, 2H), 1.25 (t, J = 7.1 Hz, 3H).
f1 (ppm)


4d
^{13}C NMR (151 MHz, CDCl$_3$) δ 197.99 (s), 173.27 (s), 163.47 (s), 130.27 (s), 130.01 (s), 113.72 (s), 60.28 (s), 55.42 (s), 37.13 (s), 33.49 (s), 19.67 (s), 14.20 (s).
f1 (ppm)

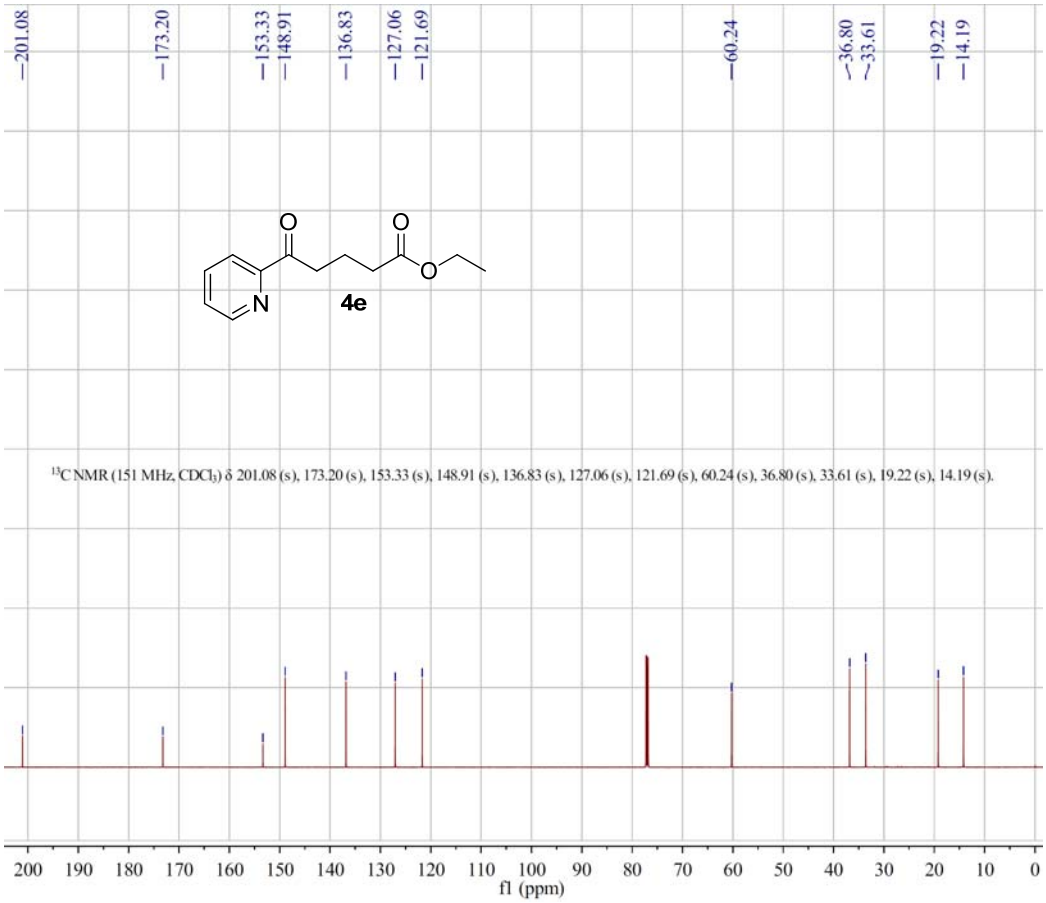

+ESI Scan (0.890-1.052 min, 11 scans) Frag=175.0V LTT1-7-4.d Subtract (2)
251.1277
(M+H)+
273.1097
4d
Counts (%) vs. Mass-to-Charge (m/z)


BRUKER
4d
Transmittance [%]
Wavenumber cm-1
3452.95
3416.26
3323.55
3057.74
2982.76
2961.06
2943.30
2917.32
2899.62
2845.26
2568.70
2051.48
1929.77
1734.04
1694.21
1668.08
1602.28
1576.09
1511.71
1453.04
1418.72
1381.81
1353.33
1313.90
1279.73
1257.79
1217.12
1184.65
1171.18
1111.77
1080.48
1030.80
987.35
943.12
864.92
834.77
815.84
753.07
719.20
632.79
607.59
586.41
570.39
497.90
459.44

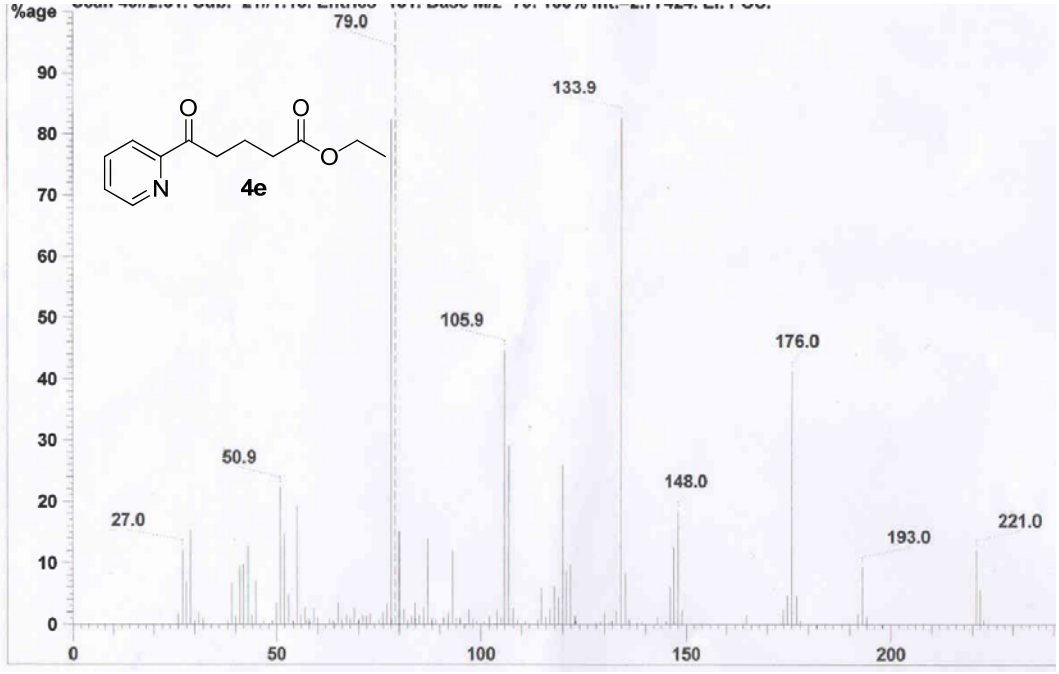
4e

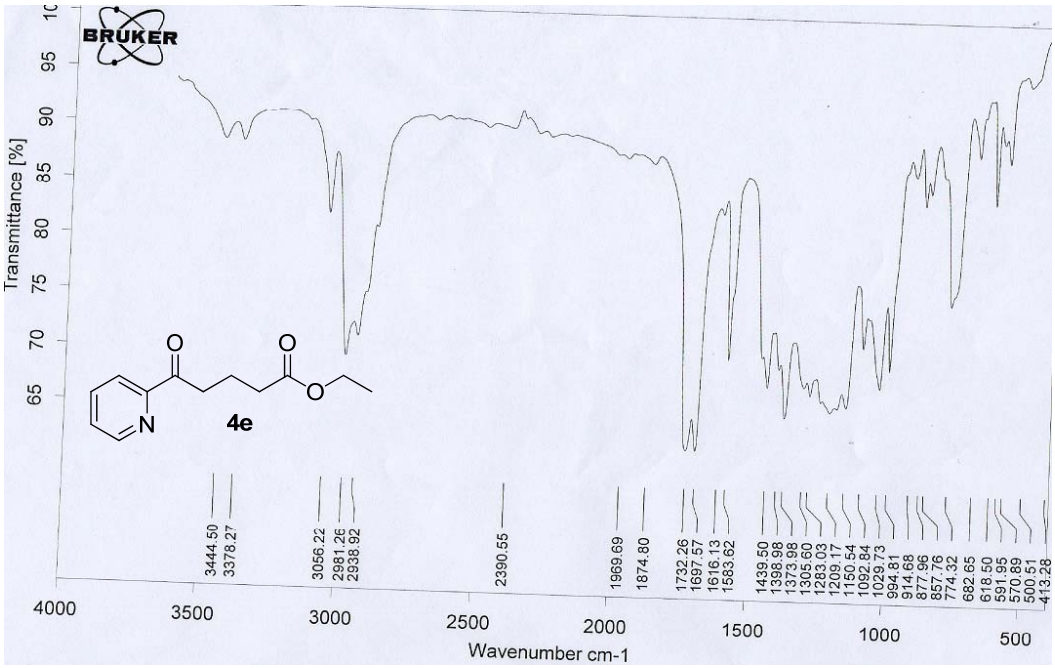


^{1}H NMR (600 MHz, $CDCl_3$) δ 8.67 (d, J = 4.7 Hz, 1H), 8.04 (d, J = 7.8 Hz, 1H), 7.84 (t, J = 7.7 Hz, 1H), 7.51 – 7.44 (m, 1H), 4.14 (q, J = 7.1 Hz, 2H), 3.29 (t, J = 7.3 Hz, 2H), 2.44 (t, J = 7.4 Hz, 2H), 2.08 (p, J = 7.3 Hz, 2H), 1.25 (t, J = 7.1 Hz, 3H).

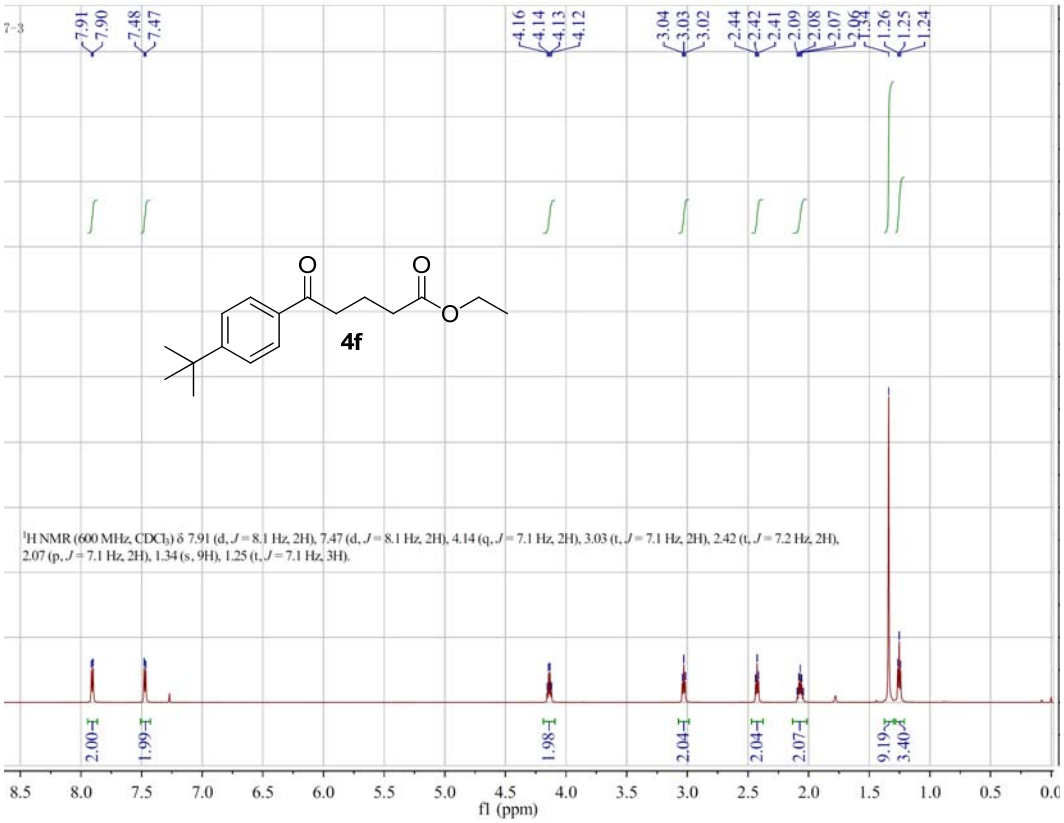


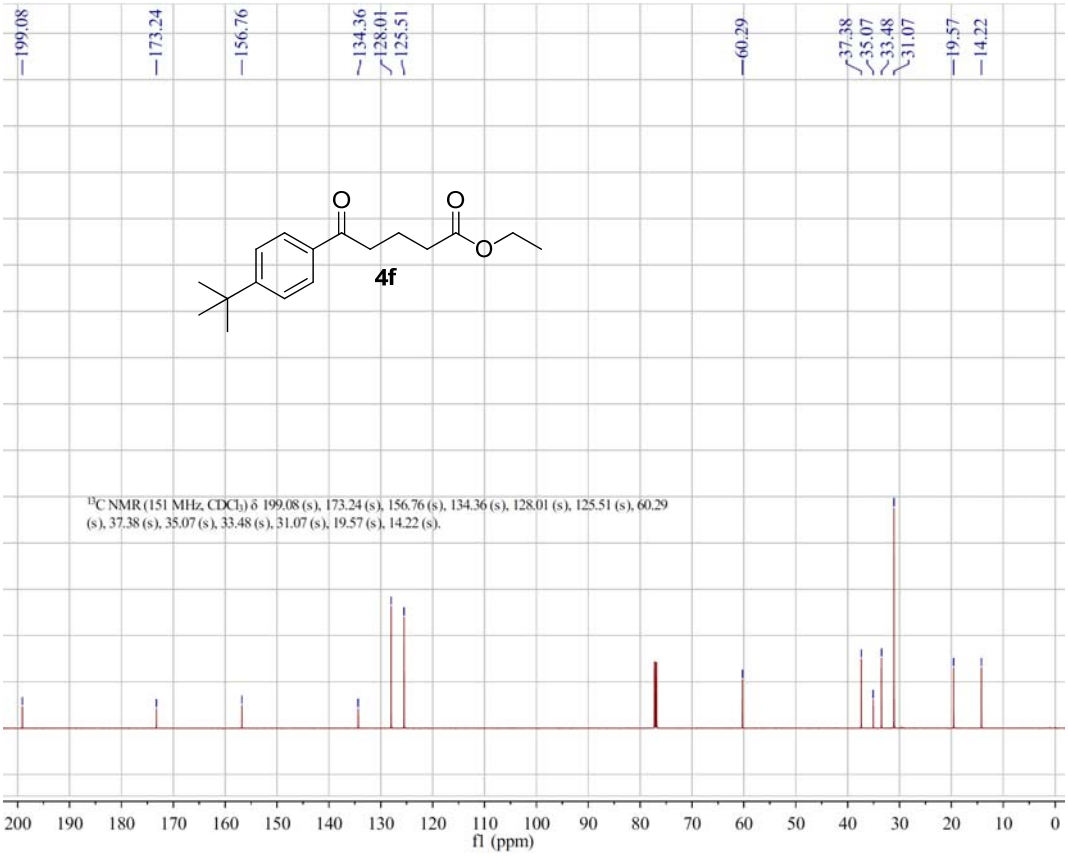
^{13}C NMR (151 MHz, $CDCl_3$) δ 201.08 (s), 173.20 (s), 153.33 (s), 148.91 (s), 136.83 (s), 127.06 (s), 121.69 (s), 60.24 (s), 36.80 (s), 33.61 (s), 19.22 (s), 14.19 (s).

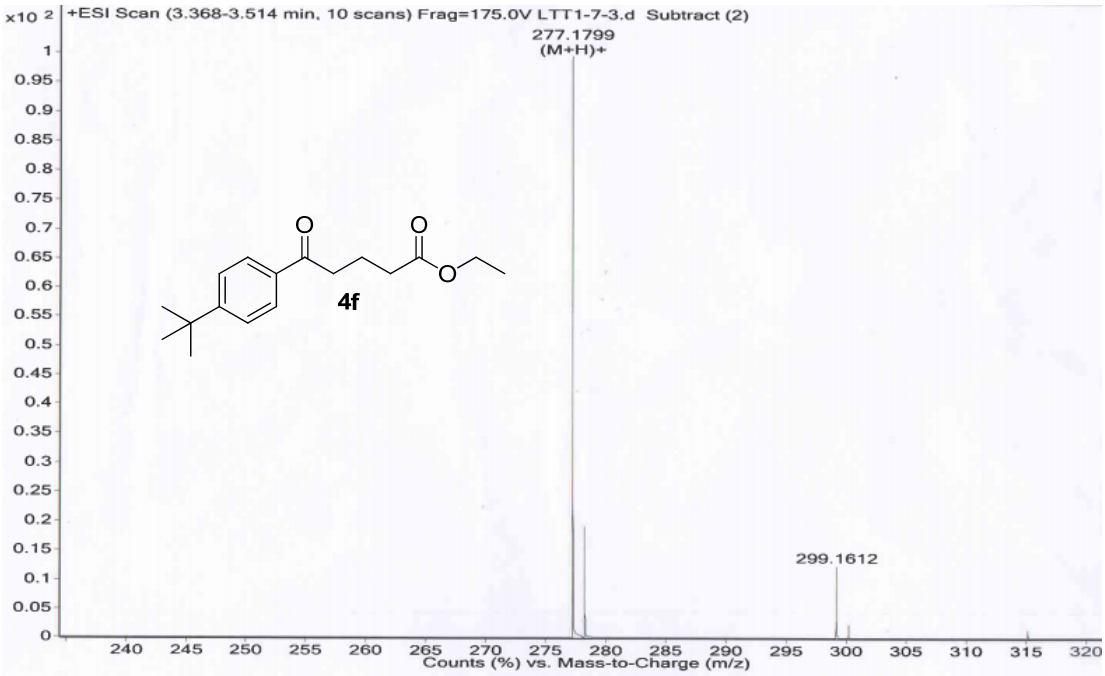

%age
79.0
133.9
105.9
176.0
50.9
148.0
27.0
193.0
221.0
4e

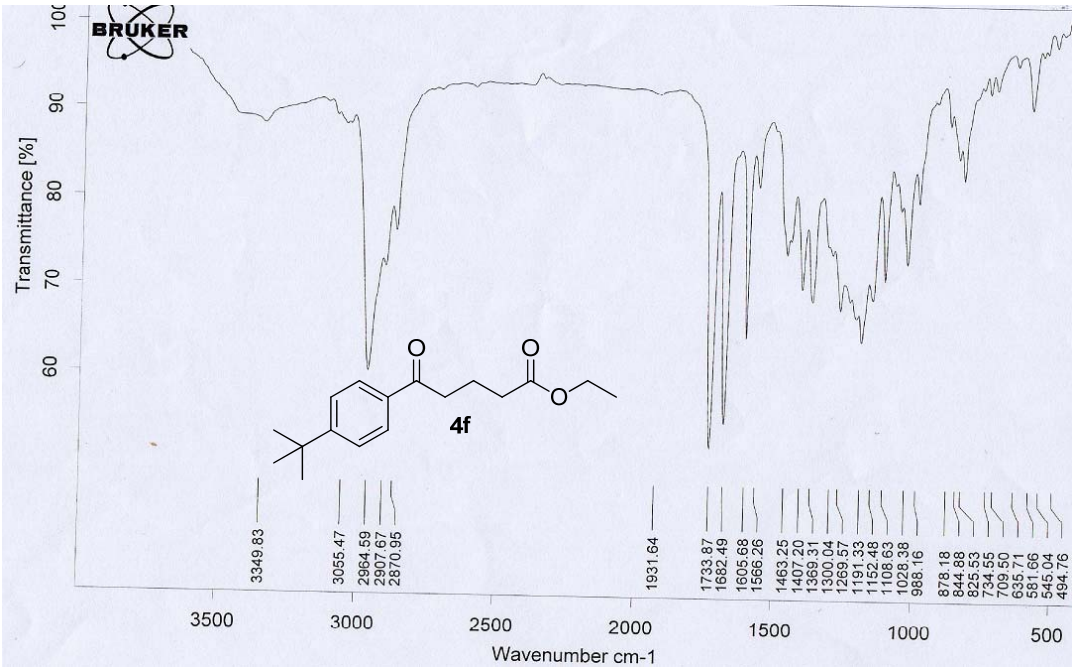

BRUKER
Transmittance [%]
4e
3444.50
3378.27
3056.22
2981.26
2938.92
2390.55
1969.69
1874.80
1732.26
1697.57
1616.13
1583.62
1439.50
1398.98
1373.98
1305.60
1283.03
1209.17
1150.54
1092.84
1029.73
994.81
914.68
877.96
857.76
774.32
682.65
618.50
591.95
570.89
500.51
413.28
Wavenumber cm-1

4f

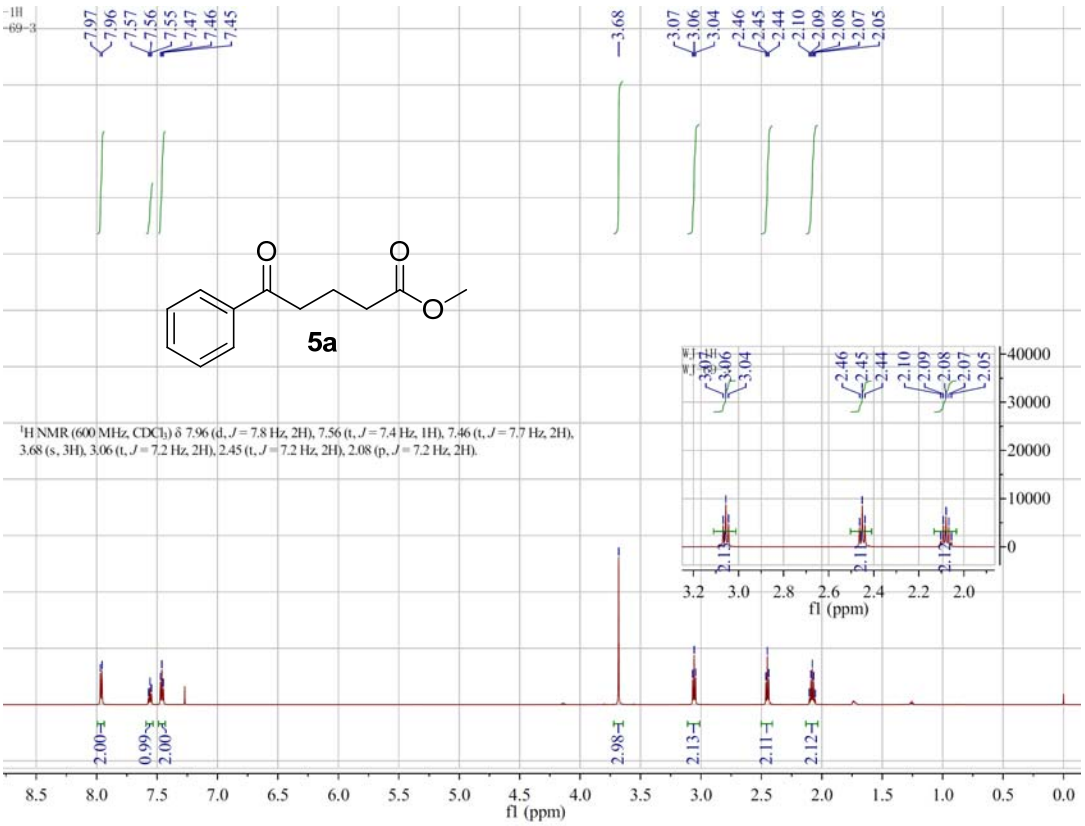

7.91
7.90
7.48
7.47
4.16
4.14
4.13
4.12
3.04
3.03
3.02
2.44
2.42
2.41
2.09
2.08
2.07
2.06
1.34
1.26
1.25
1.24
4f
^{1}H NMR (600 MHz, CDCl$_3$) δ 7.91 (d, J = 8.1 Hz, 2H), 7.47 (d, J = 8.1 Hz, 2H), 4.14 (q, J = 7.1 Hz, 2H), 3.03 (t, J = 7.1 Hz, 2H), 2.42 (t, J = 7.2 Hz, 2H), 2.07 (p, J = 7.1 Hz, 2H), 1.34 (s, 9H), 1.25 (t, J = 7.1 Hz, 3H).
2.00
1.99
1.98
2.04
2.04
2.07
9.19
3.40
f1 (ppm)

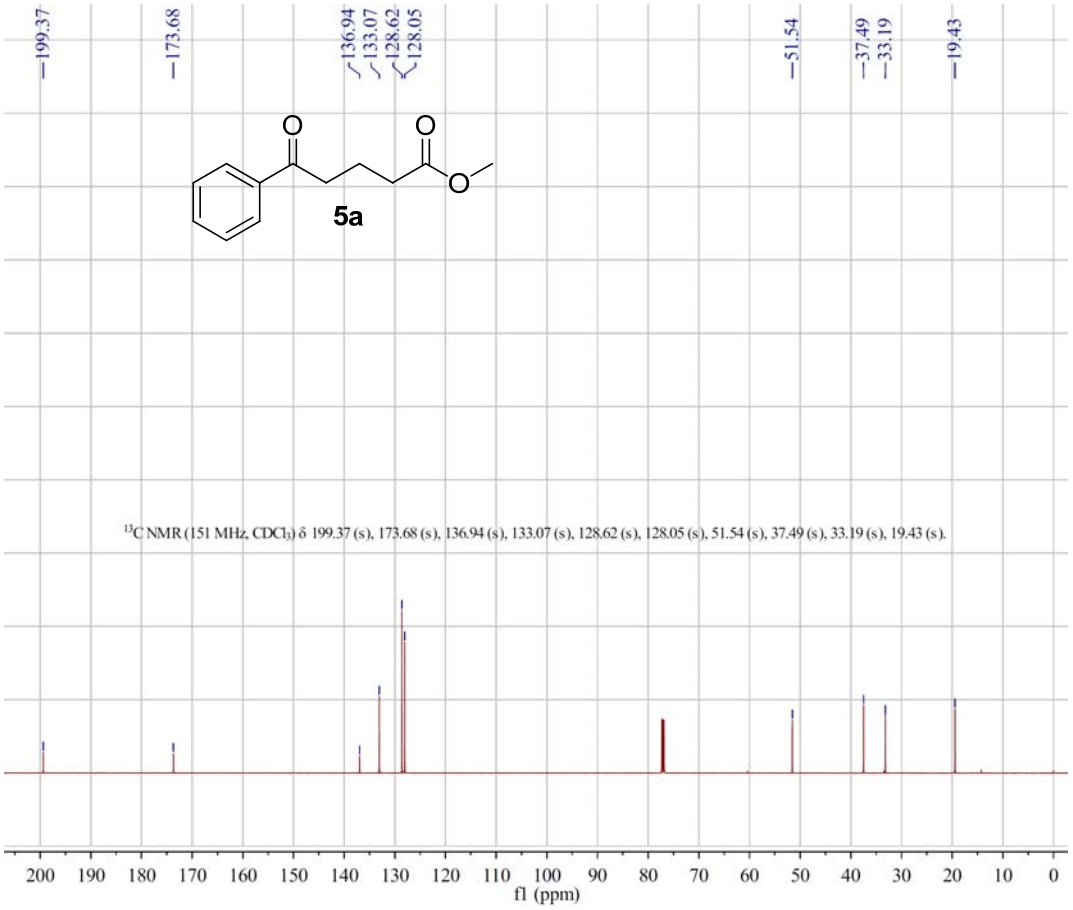

199.08
173.24
156.76
134.36
128.01
125.51
60.29
37.38
35.07
33.48
31.07
19.57
14.22
4f
^{13}C NMR (151 MHz, CDCl$_3$) δ 199.08 (s), 173.24 (s), 156.76 (s), 134.36 (s), 128.01 (s), 125.51 (s), 60.29 (s), 37.38 (s), 35.07 (s), 33.48 (s), 31.07 (s), 19.57 (s), 14.22 (s).
f1 (ppm)

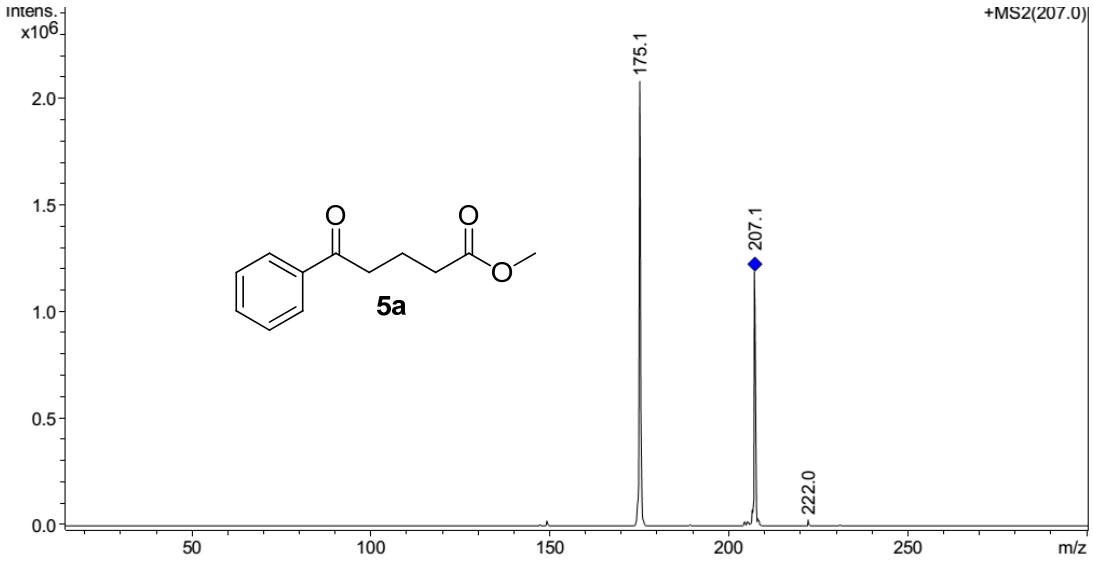

x10 2
+ESI Scan (3.368-3.514 min, 10 scans) Frag=175.0V LTT1-7-3.d Subtract (2)
277.1799
(M+H)+
4f
299.1612
Counts (%) vs. Mass-to-Charge (m/z)

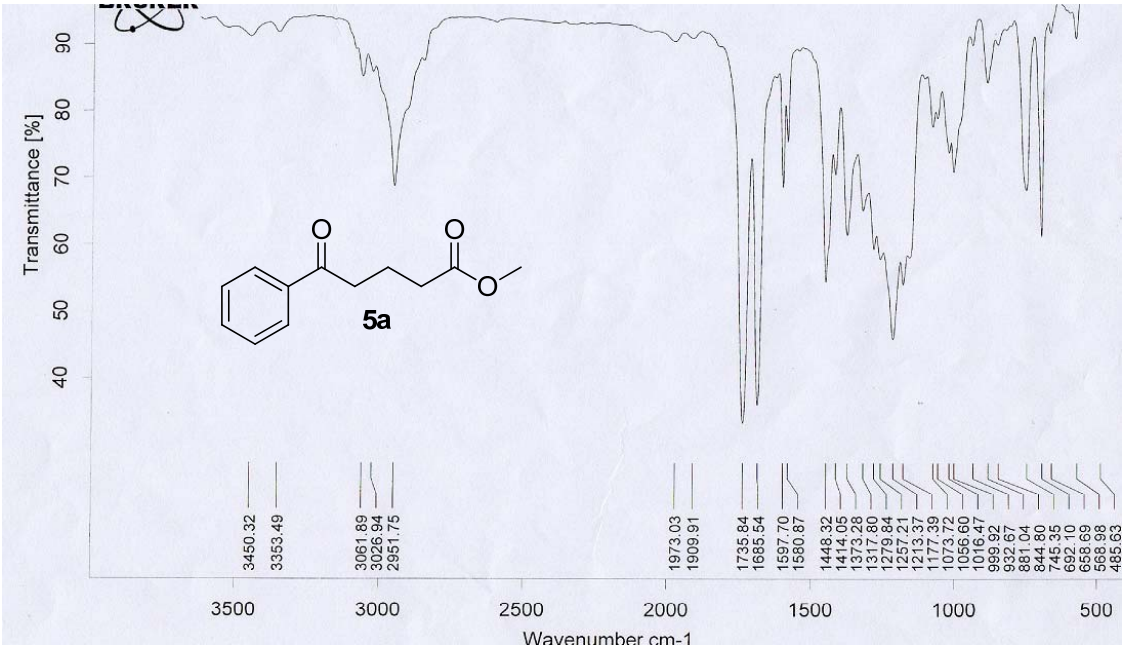

BRUKER
Transmittance [%]
4f
3349.83
3055.47
2964.59
2907.67
2870.95
1931.64
1733.87
1682.49
1605.68
1566.26
1463.25
1407.20
1369.31
1300.04
1269.57
1191.33
1152.48
1108.63
1028.38
988.16
878.18
844.88
825.53
734.55
709.50
635.71
581.66
545.04
494.76
Wavenumber cm-1

5a

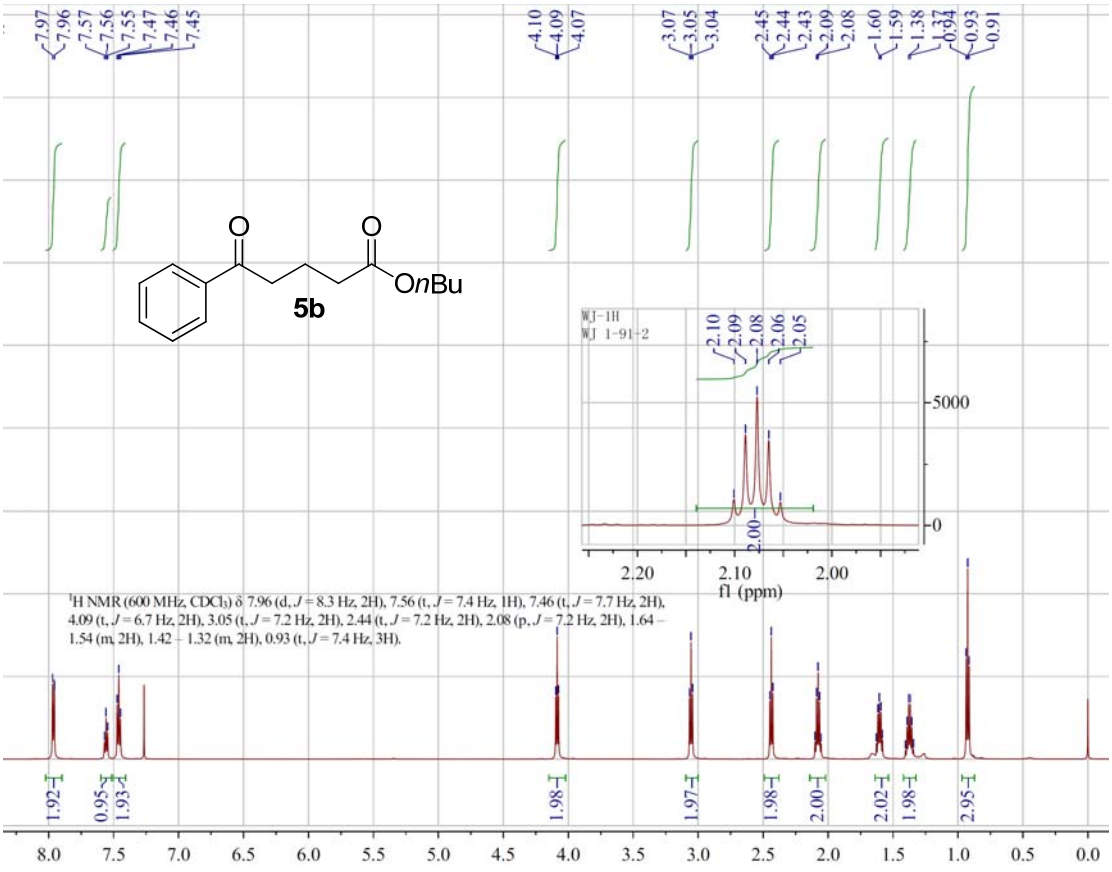

7.97
7.96
7.57
7.56
7.55
7.47
7.46
7.45
3.68
3.07
3.06
3.04
2.46
2.45
2.44
2.10
2.09
2.08
2.07
2.05
5a
^{1}H NMR (600 MHz, CDCl$_3$) δ 7.96 (d, J = 7.8 Hz, 2H), 7.56 (t, J = 7.4 Hz, 1H), 7.46 (t, J = 7.7 Hz, 2H), 3.68 (s, 3H), 3.06 (t, J = 7.2 Hz, 2H), 2.45 (t, J = 7.2 Hz, 2H), 2.08 (p, J = 7.2 Hz, 2H).
2.00
0.99
2.00
2.98
2.13
2.11
2.12
f1 (ppm)

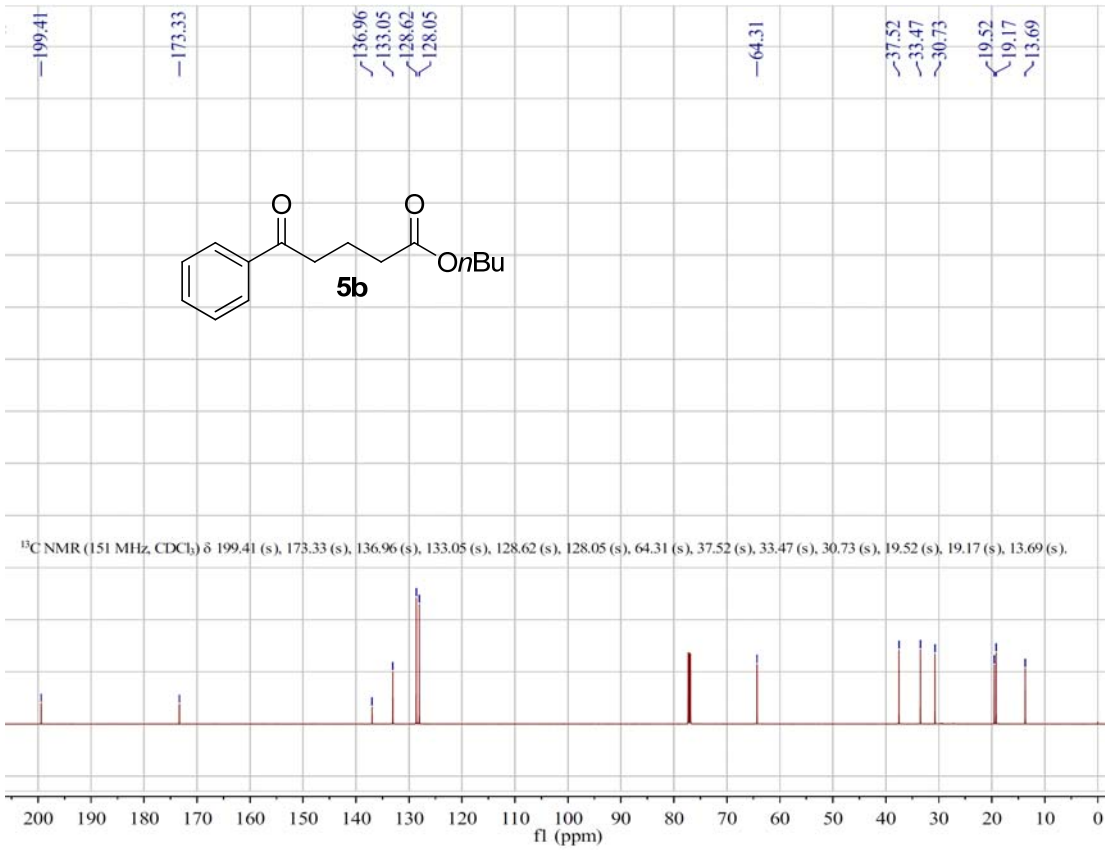

199.37
173.68
136.94
133.07
128.62
128.05
51.54
37.49
33.19
19.43
5a
^{13}C NMR (151 MHz, CDCl$_3$) δ 199.37 (s), 173.68 (s), 136.94 (s), 133.07 (s), 128.62 (s), 128.05 (s), 51.54 (s), 37.49 (s), 33.19 (s), 19.43 (s).
f1 (ppm)

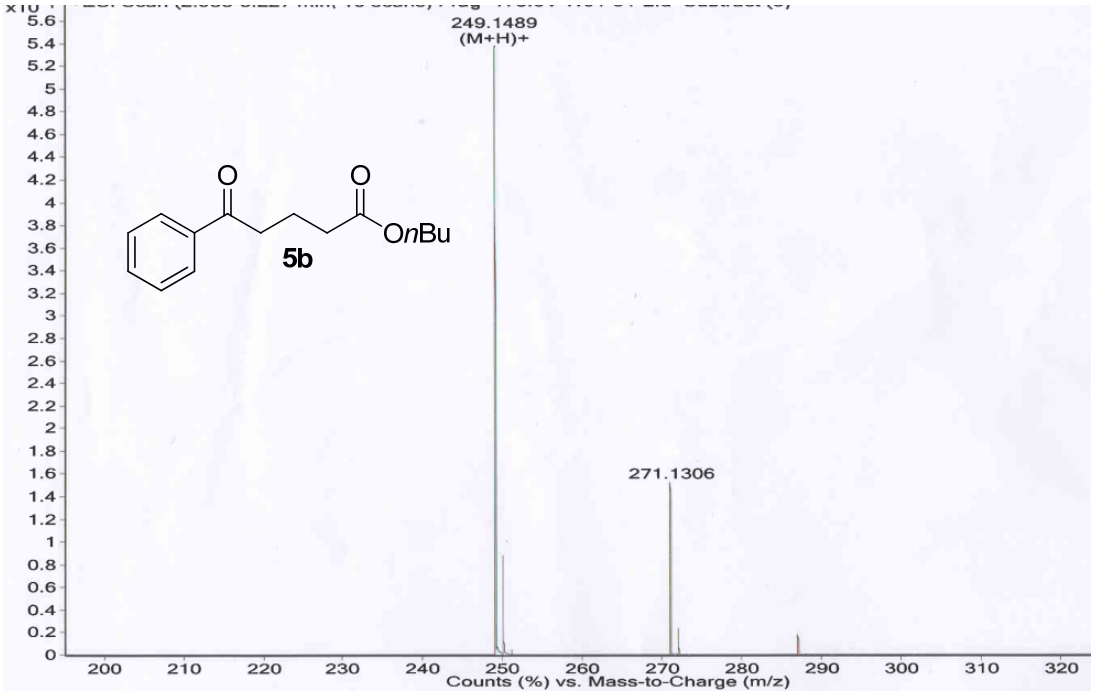

Intens.
x10^6
+MS2(207.0)
175.1
207.1
222.0
5a
m/z

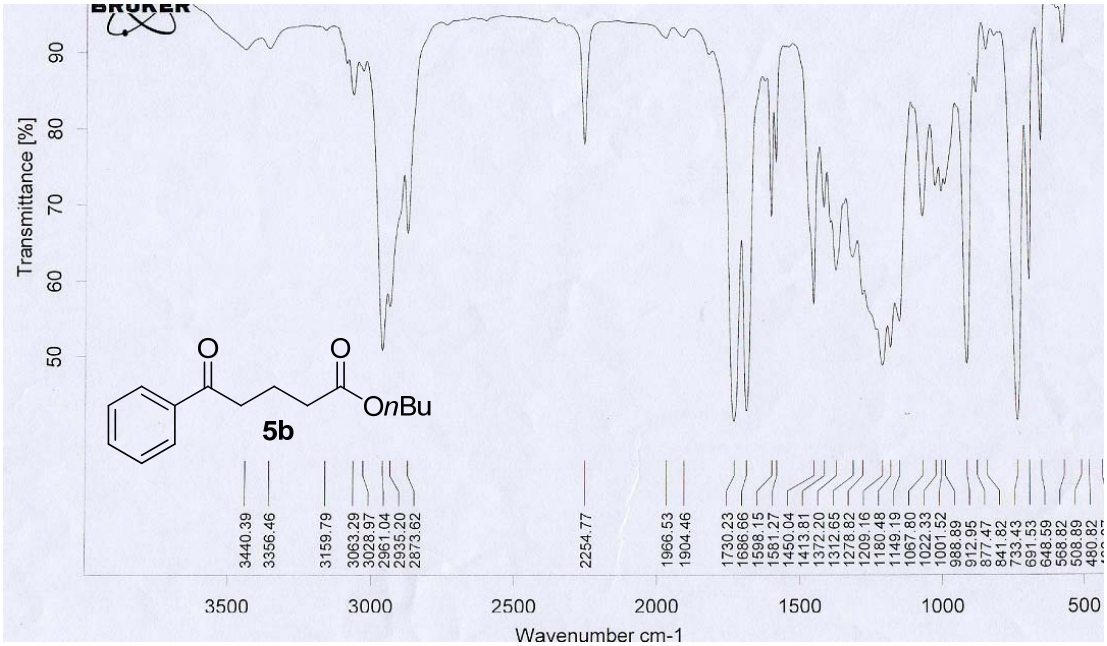

Transmittance [%]
5a
3450.32
3353.49
3061.89
3026.94
2951.75
1973.03
1909.91
1735.84
1685.54
1597.70
1580.87
1448.32
1414.05
1373.28
1317.80
1279.84
1257.21
1213.37
1177.39
1073.72
1056.60
1016.47
999.92
932.67
881.04
844.80
745.35
692.10
658.69
568.98
485.63
Wavenumber cm-1

5b

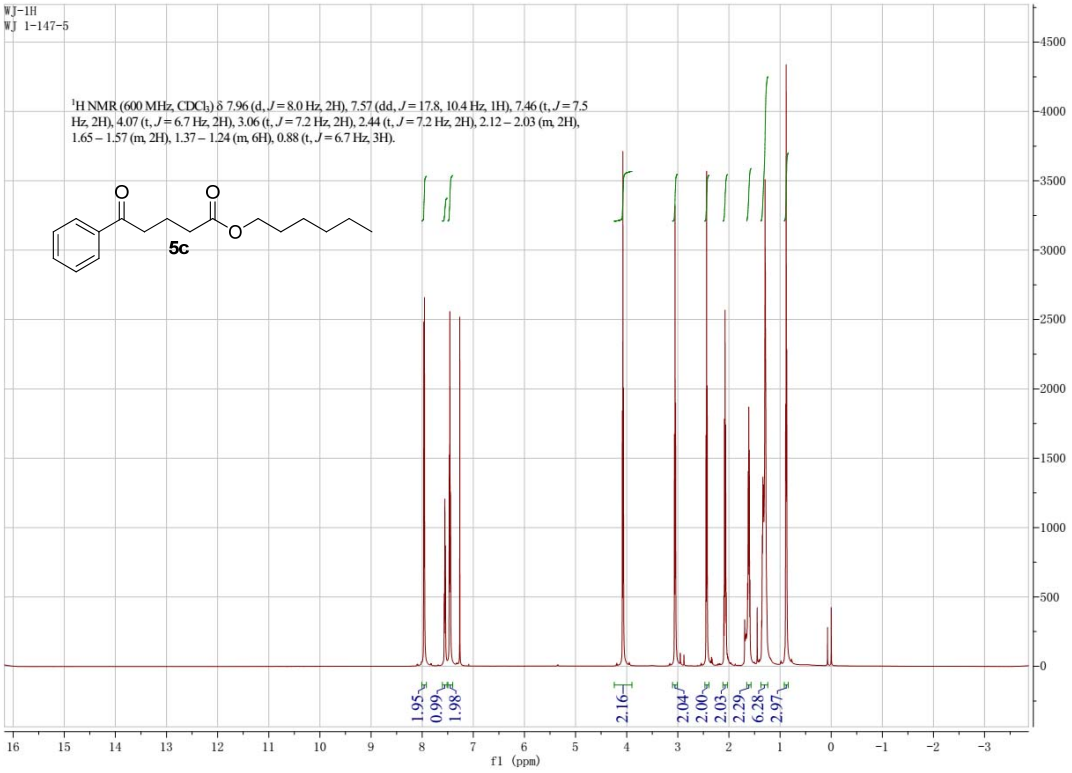

7.97
7.96
7.57
7.56
7.55
7.47
7.46
7.45
4.10
4.09
4.07
3.07
3.05
3.04
2.45
2.44
2.43
2.09
2.08
1.60
1.59
1.38
1.37
0.94
0.93
0.91
O
O
OnBu
5b
WJ-1H
WJ 1-91-2
2.10
2.09
2.08
2.06
2.05
5000
0
2.00
2.20
2.10
2.00
f1 (ppm)
1H NMR (600 MHz, CDCl3) δ 7.96 (d, J = 8.3 Hz, 2H), 7.56 (t, J = 7.4 Hz, 1H), 7.46 (t, J = 7.7 Hz, 2H), 4.09 (t, J = 6.7 Hz, 2H), 3.05 (t, J = 7.2 Hz, 2H), 2.44 (t, J = 7.2 Hz, 2H), 2.08 (p, J = 7.2 Hz, 2H), 1.64 – 1.54 (m, 2H), 1.42 – 1.32 (m, 2H), 0.93 (t, J = 7.4 Hz, 3H).
1.92
0.95
1.93
1.98
1.97
1.98
2.00
2.02
1.98
2.95
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0

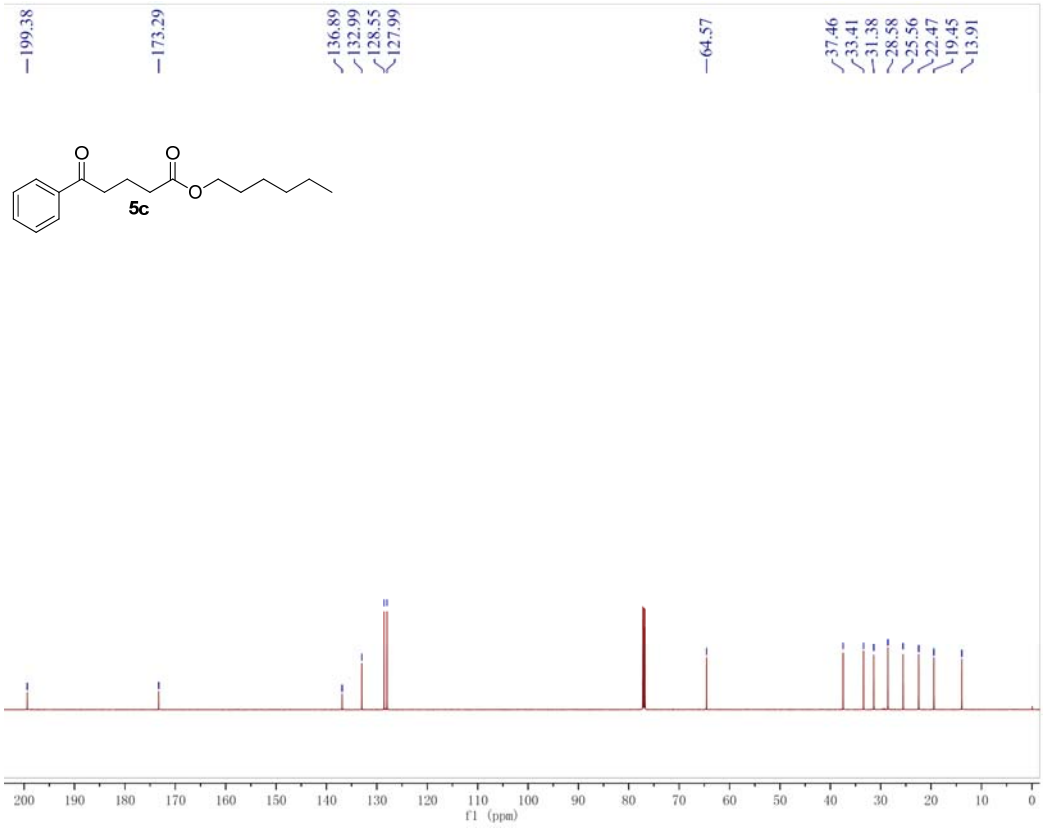

199.41
173.33
136.96
133.05
128.62
128.05
64.31
37.52
33.47
30.73
19.52
19.17
13.69
O
O
OnBu
5b
13C NMR (151 MHz, CDCl3) δ 199.41 (s), 173.33 (s), 136.96 (s), 133.05 (s), 128.62 (s), 128.05 (s), 64.31 (s), 37.52 (s), 33.47 (s), 30.73 (s), 19.52 (s), 19.17 (s), 13.69 (s).
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

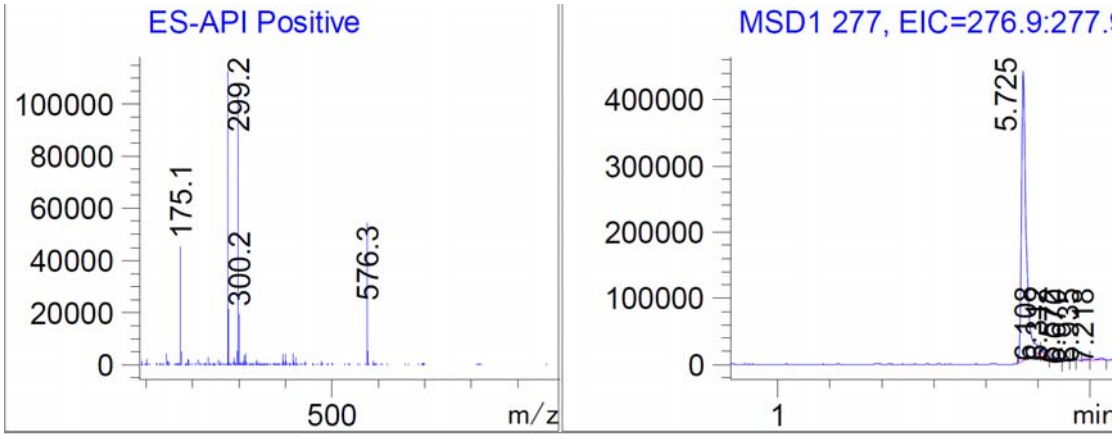

249.1489
(M+H)+
271.1306
OnBu
5b
Counts (%) vs. Mass-to-Charge (m/z)


Transmittance [%]
OnBu
5b
3440.39
3356.46
3159.79
3063.29
3028.97
2961.04
2935.20
2873.62
2254.77
1966.53
1904.46
1730.23
1686.66
1598.15
1581.27
1450.04
1413.81
1372.20
1312.65
1278.82
1209.16
1180.48
1149.19
1067.80
1022.33
1001.52
988.89
912.95
877.47
841.82
733.43
691.53
648.59
568.82
508.89
480.82
Wavenumber cm-1

5c


WJ-1H
WJ 1-147-5
^{1}H NMR (600 MHz, $CDCl_3$) δ 7.96 (d, J = 8.0 Hz, 2H), 7.57 (dd, J = 17.8, 10.4 Hz, 1H), 7.46 (t, J = 7.5 Hz, 2H), 4.07 (t, J = 6.7 Hz, 2H), 3.06 (t, J = 7.2 Hz, 2H), 2.44 (t, J = 7.2 Hz, 2H), 2.12 – 2.03 (m, 2H), 1.65 – 1.57 (m, 2H), 1.37 – 1.24 (m, 6H), 0.88 (t, J = 6.7 Hz, 3H).
5c
1.95
0.99
1.98
2.16
2.04
2.00
2.03
2.29
6.28
2.97
f1 (ppm)


199.38
173.29
136.89
132.99
128.55
127.99
64.57
37.46
33.41
31.38
28.58
25.56
22.47
19.45
13.91
5c
f1 (ppm)

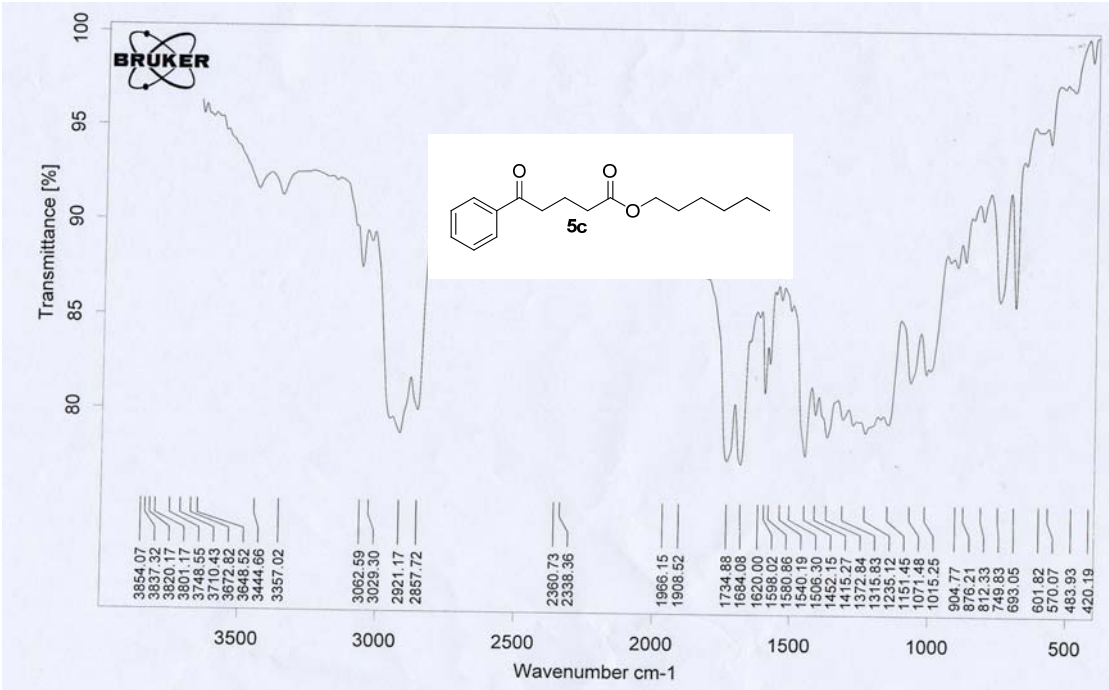


Base Peak Ion Ratios:

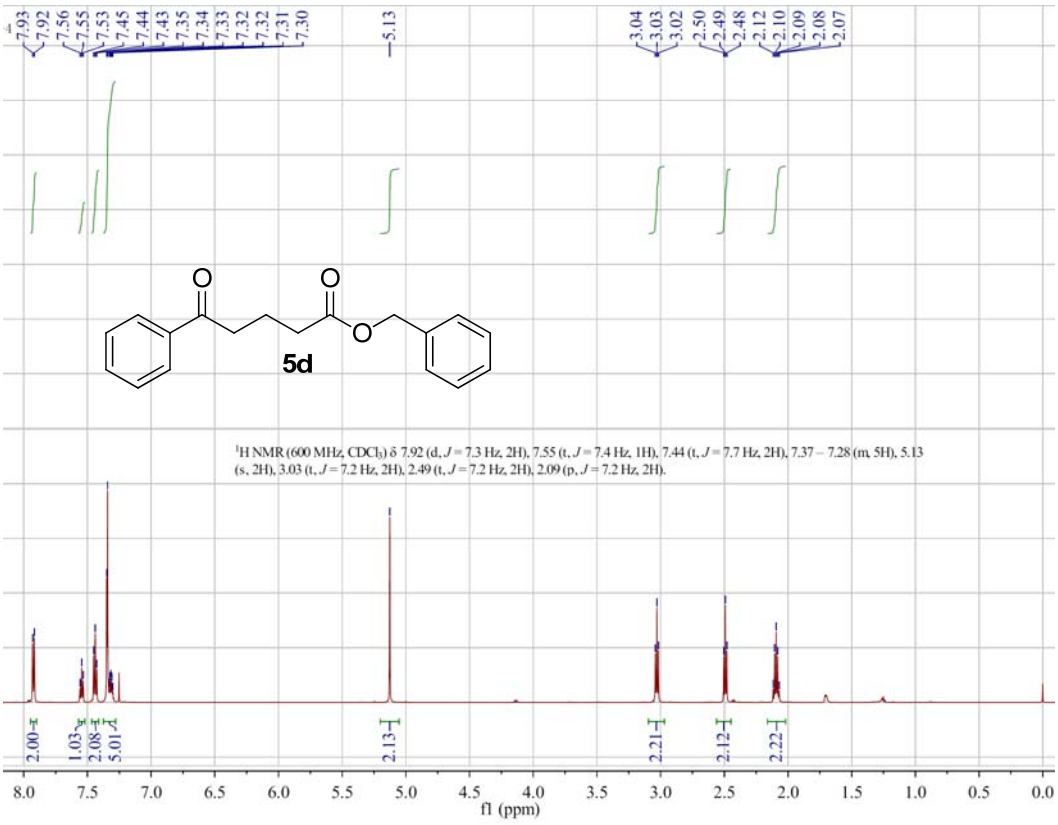
Ion	Mass	Abundance	Ratio
A	277.20	112830	100.00
A + 1	278.20	21420	18.98
A + 2	279.20	4159	3.69
A + 3	280.20	635	0.56
A + 4	281.10	167	0.15
A + 5	282.05	101	0.09

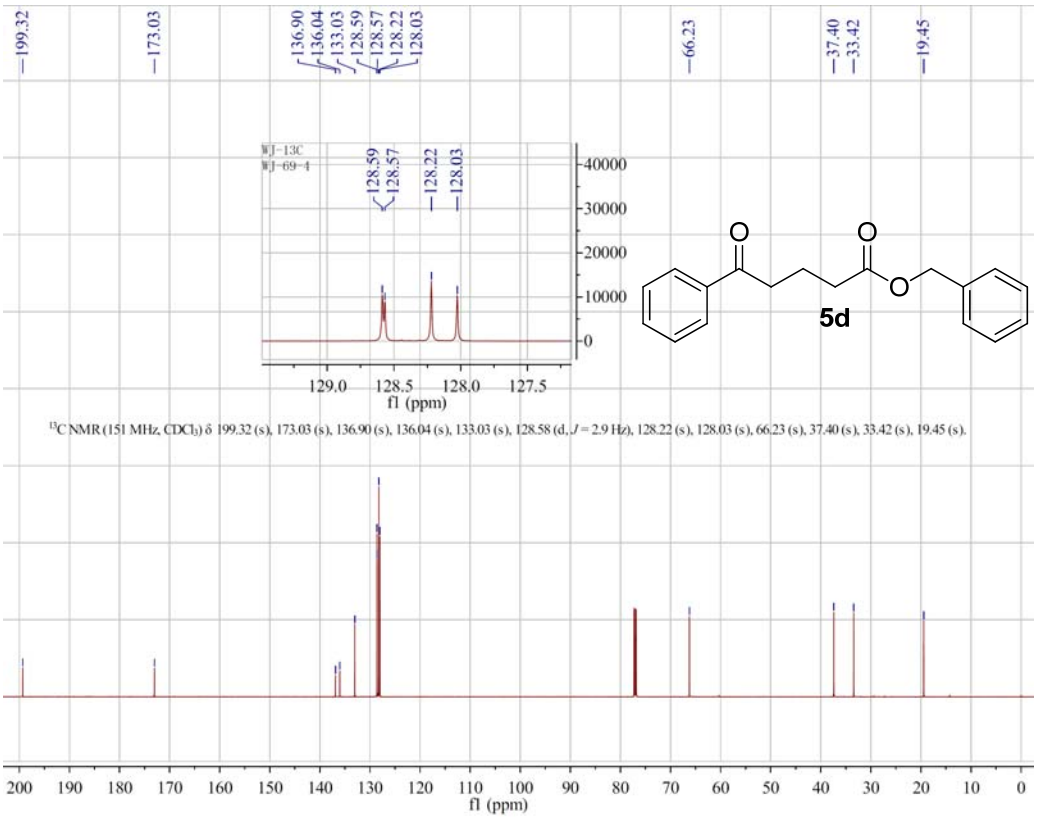
2nd Largest Peak Ion Ratios:

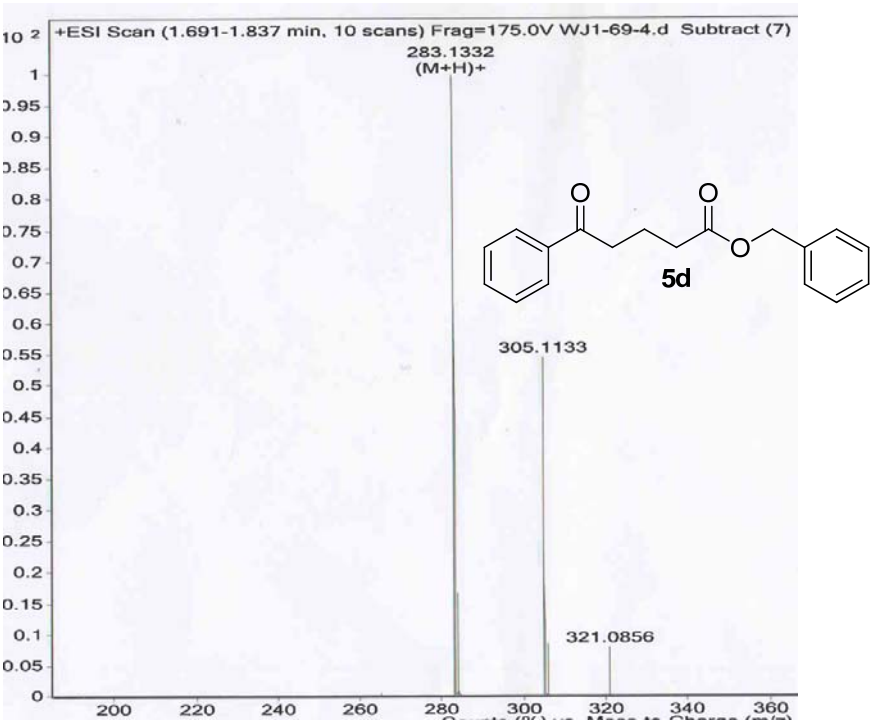
Ion	Mass	Abundance	Ratio
B	299.20	104152	100.00
B + 1	300.20	19318	18.55
B + 2	301.10	3885	3.73
B + 3	302.05	583	0.56
B + 4	303.25	144	0.14
B + 5	304.35	30	0.03

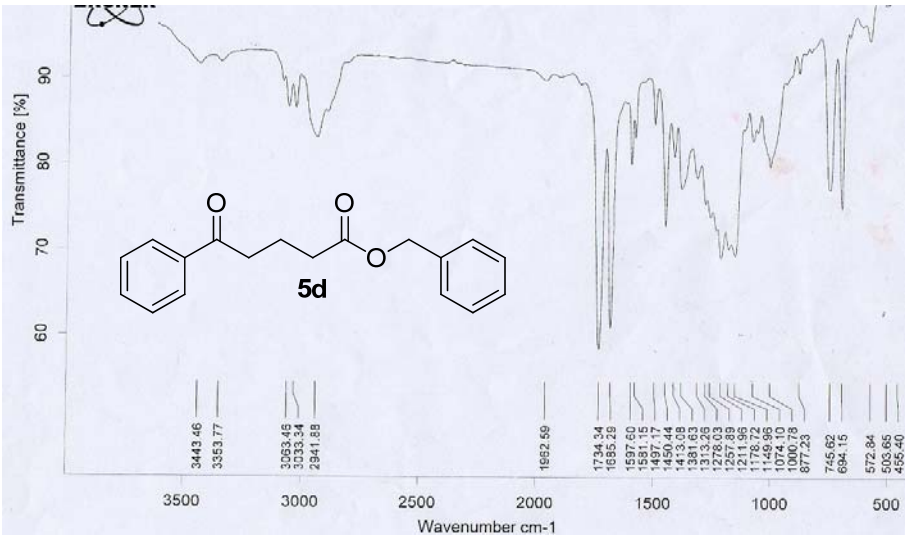


5d

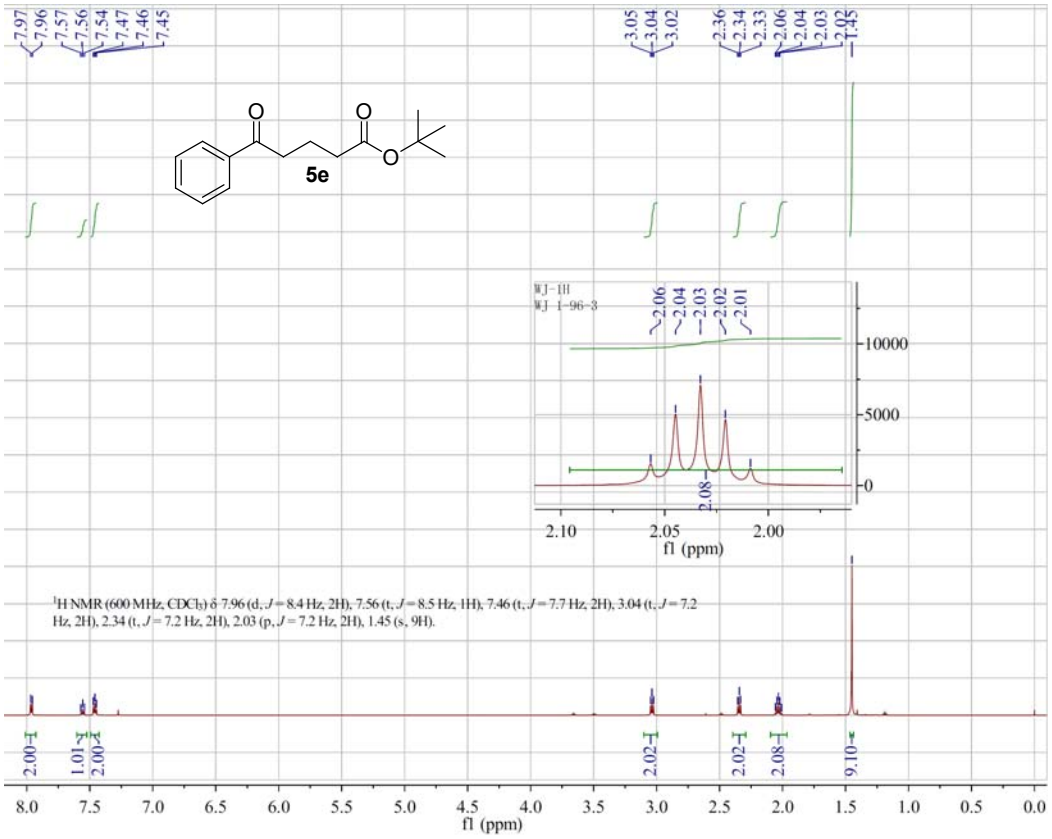

7.93
7.92
7.56
7.55
7.53
7.45
7.44
7.43
7.35
7.34
7.33
7.32
7.32
7.31
7.30
5.13
3.04
3.03
3.02
2.50
2.49
2.48
2.12
2.10
2.09
2.08
2.07
5d
¹H NMR (600 MHz, CDCl₃) δ 7.92 (d, J = 7.3 Hz, 2H), 7.55 (t, J = 7.4 Hz, 1H), 7.44 (t, J = 7.7 Hz, 2H), 7.37 – 7.28 (m, 5H), 5.13 (s, 2H), 3.03 (t, J = 7.2 Hz, 2H), 2.49 (t, J = 7.2 Hz, 2H), 2.09 (p, J = 7.2 Hz, 2H).
2.00
1.03
2.08
5.01
2.13
2.21
2.12
2.22
f1 (ppm)

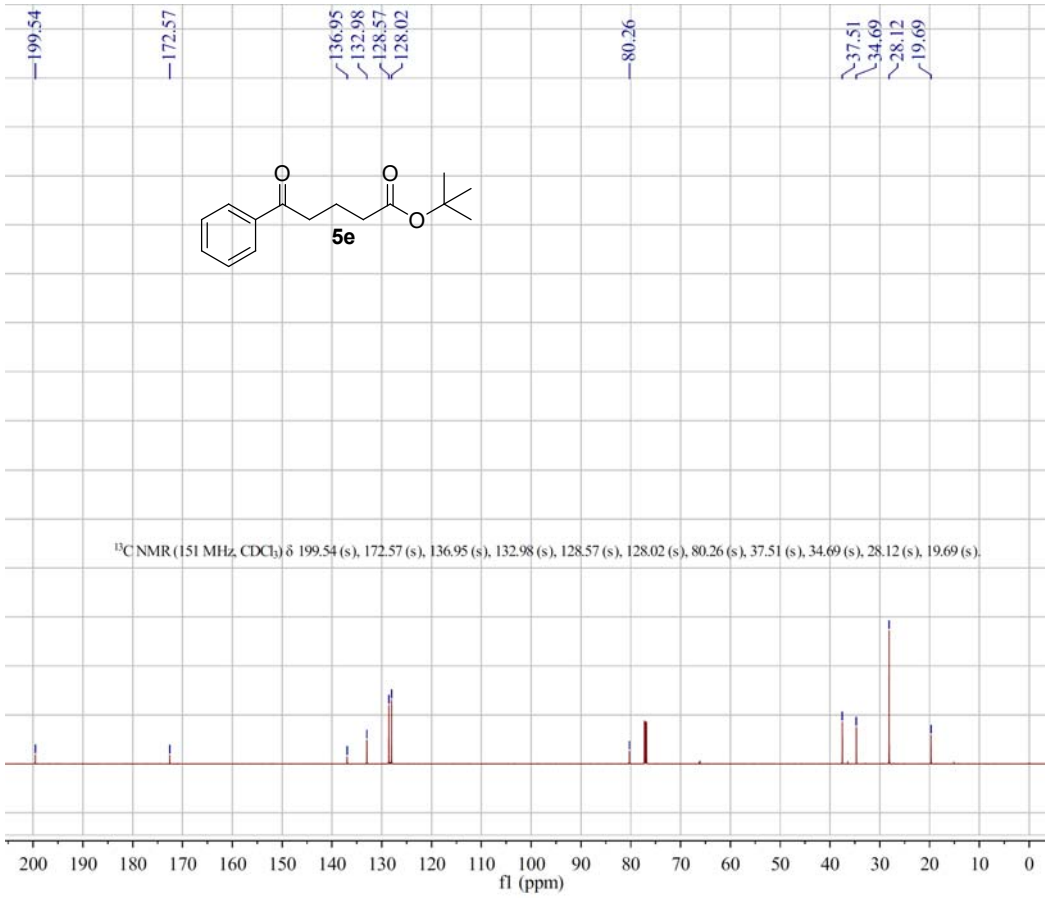

199.32
173.03
136.90
136.04
133.03
128.59
128.57
128.22
128.03
66.23
37.40
33.42
19.45
WJ-13C
WJ-69-4
128.59
128.57
128.22
128.03
40000
30000
20000
10000
0
129.0
128.5
128.0
127.5
f1 (ppm)
5d
¹³C NMR (151 MHz, CDCl₃) δ 199.32 (s), 173.03 (s), 136.90 (s), 136.04 (s), 133.03 (s), 128.58 (d, J = 2.9 Hz), 128.22 (s), 128.03 (s), 66.23 (s), 37.40 (s), 33.42 (s), 19.45 (s).
f1 (ppm)

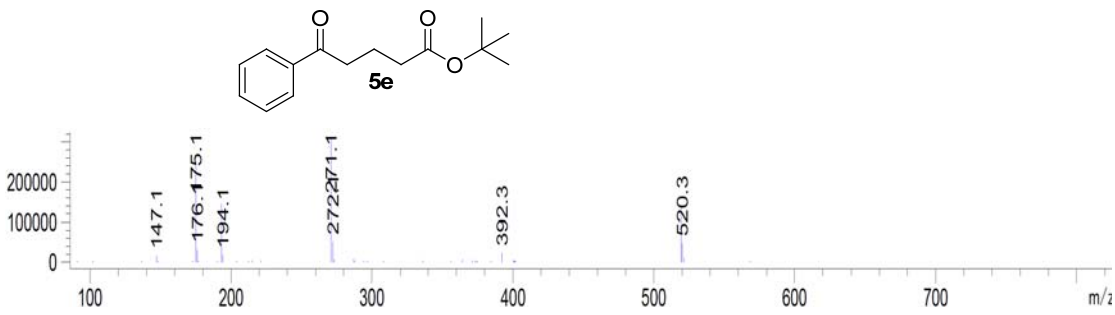

+ESI Scan (1.691-1.837 min, 10 scans) Frag=175.0V WJ1-69-4.d Subtract (7)
283.1332
(M+H)+
305.1133
321.0856
5d

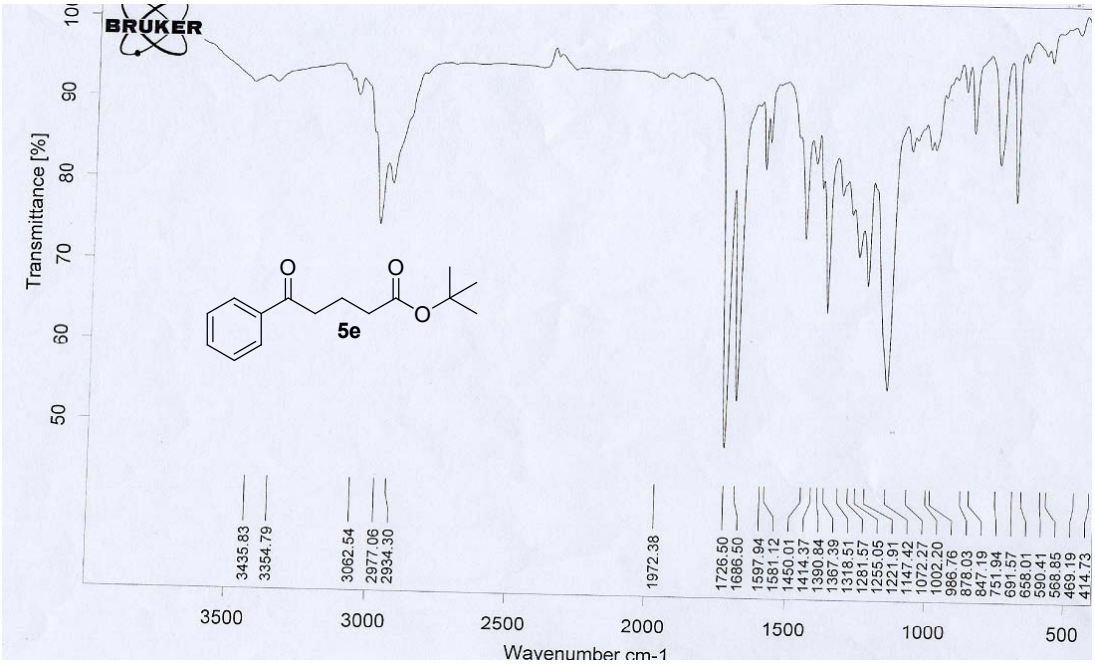

Transmittance [%]
Wavenumber cm-1
5d

5e

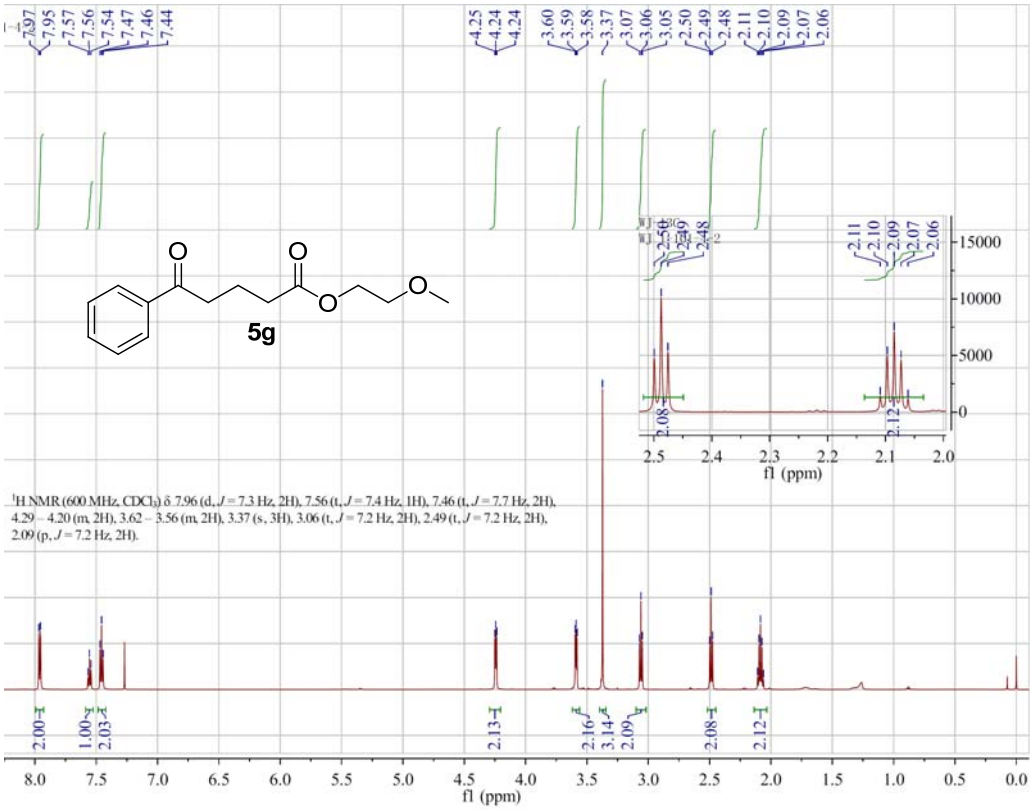

5e
^{1}H NMR (600 MHz, $CDCl_3$) δ 7.96 (d, J = 8.4 Hz, 2H), 7.56 (t, J = 8.5 Hz, 1H), 7.46 (t, J = 7.7 Hz, 2H), 3.04 (t, J = 7.2 Hz, 2H), 2.34 (t, J = 7.2 Hz, 2H), 2.03 (p, J = 7.2 Hz, 2H), 1.45 (s, 9H).
f1 (ppm)

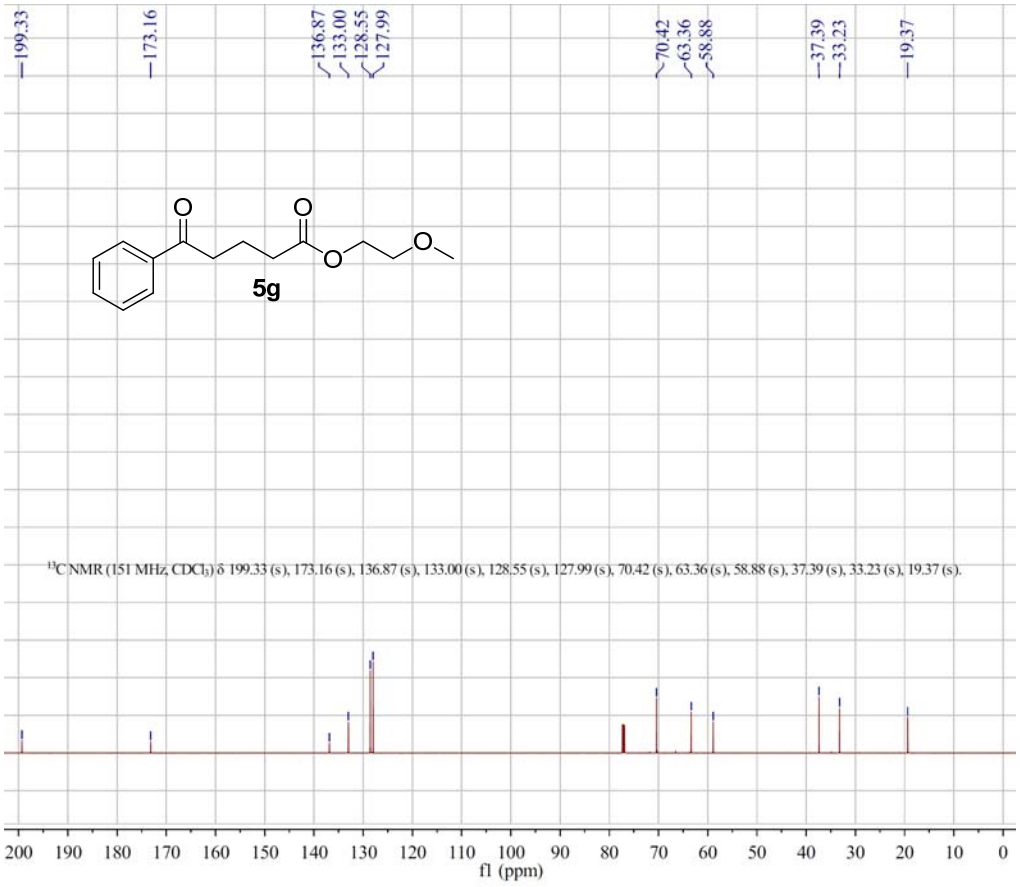

5e
^{13}C NMR (151 MHz, $CDCl_3$) δ 199.54 (s), 172.57 (s), 136.95 (s), 132.98 (s), 128.57 (s), 128.02 (s), 80.26 (s), 37.51 (s), 34.69 (s), 28.12 (s), 19.69 (s).
f1 (ppm)

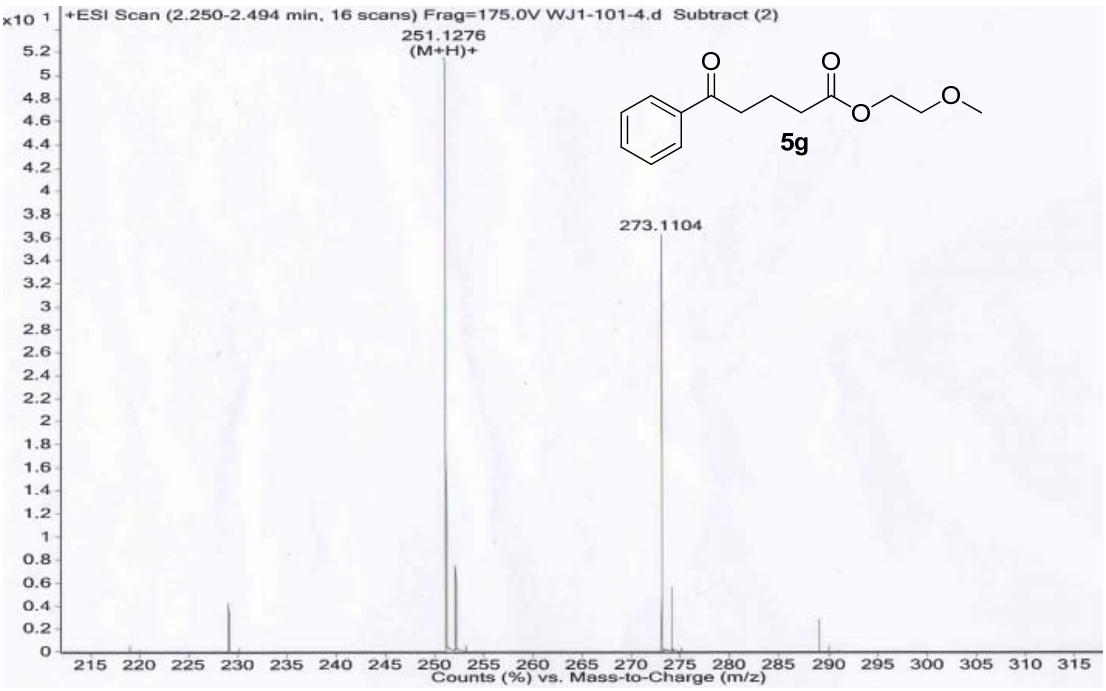

5e
200000
100000
0
147.1
176.1
175.1
194.1
272.1
271.1
392.3
520.3
100
200
300
400
500
600
700
m/z

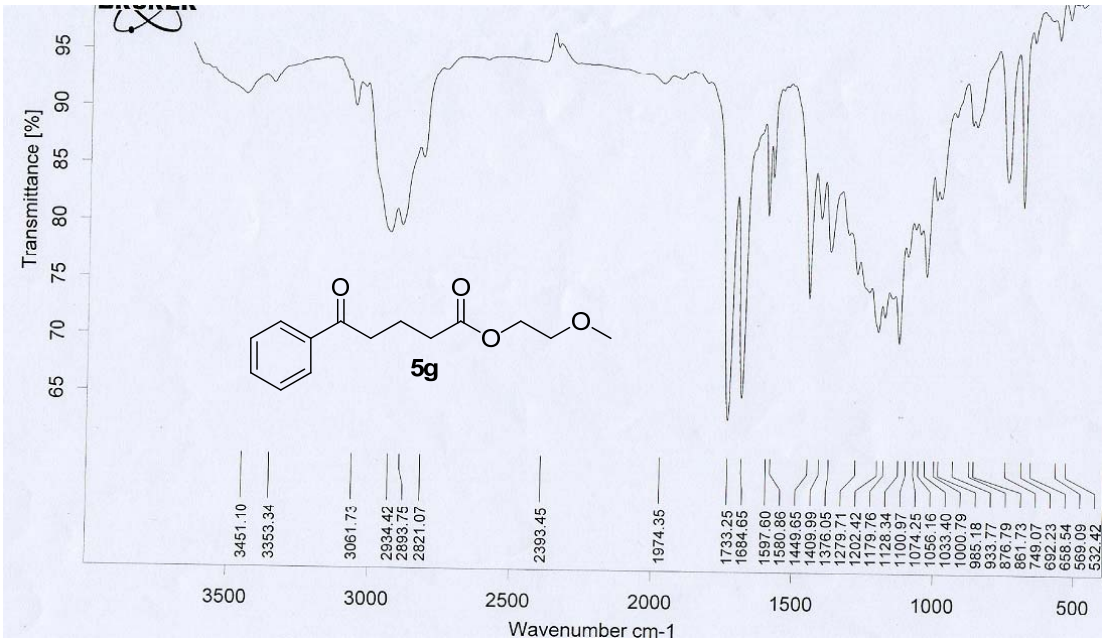

BRUKER
Transmittance [%]
100
90
80
70
60
50
5e
3435.83
3354.79
3062.54
2977.06
2934.30
1972.38
1726.50
1686.50
1597.94
1581.12
1450.01
1414.37
1390.84
1367.39
1318.51
1281.57
1255.05
1221.91
1147.42
1072.27
1002.20
986.76
878.03
847.19
751.94
691.57
658.01
590.41
568.85
469.19
414.73
3500
3000
2500
2000
1500
1000
500
Wavenumber cm-1

5g

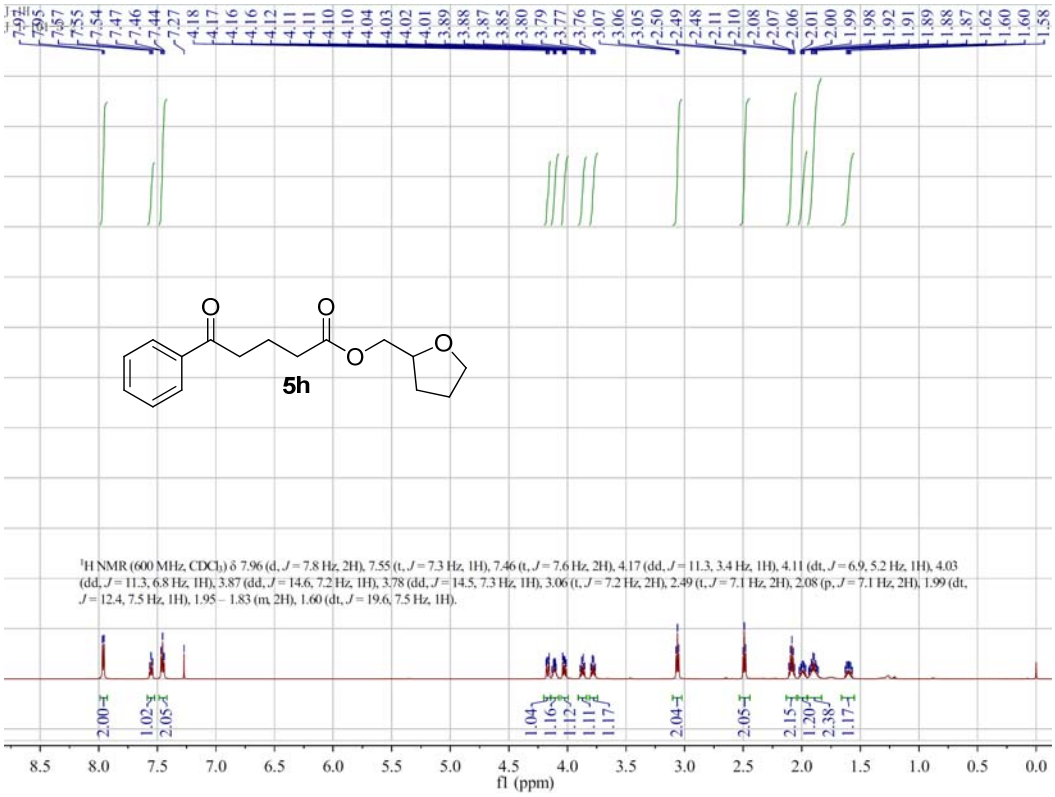

5g
^{1}H NMR (600 MHz, $CDCl_3$) δ 7.96 (d, J = 7.3 Hz, 2H), 7.56 (t, J = 7.4 Hz, 1H), 7.46 (t, J = 7.7 Hz, 2H), 4.29 – 4.20 (m, 2H), 3.62 – 3.56 (m, 2H), 3.37 (s, 3H), 3.06 (t, J = 7.2 Hz, 2H), 2.49 (t, J = 7.2 Hz, 2H), 2.09 (p, J = 7.2 Hz, 2H).
f1 (ppm)

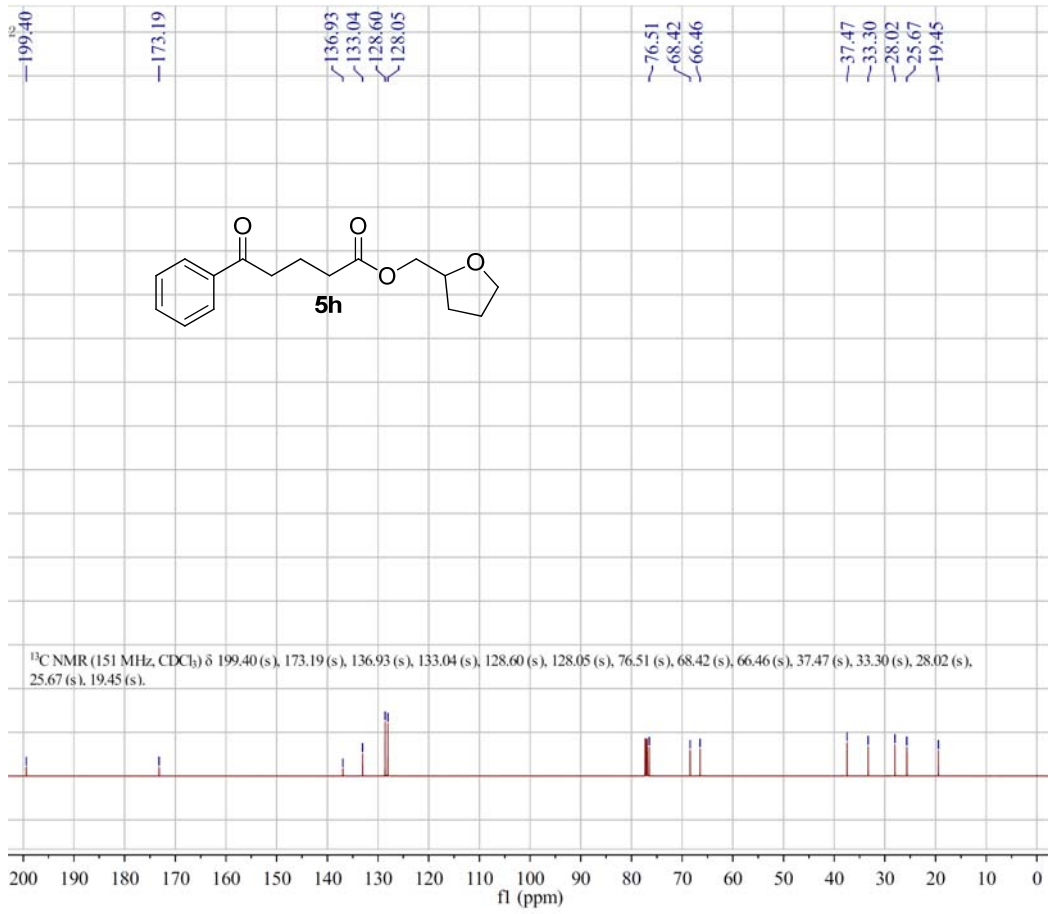

5g
^{13}C NMR (151 MHz, $CDCl_3$) δ 199.33 (s), 173.16 (s), 136.87 (s), 133.00 (s), 128.55 (s), 127.99 (s), 70.42 (s), 63.36 (s), 58.88 (s), 37.39 (s), 33.23 (s), 19.37 (s).
f1 (ppm)

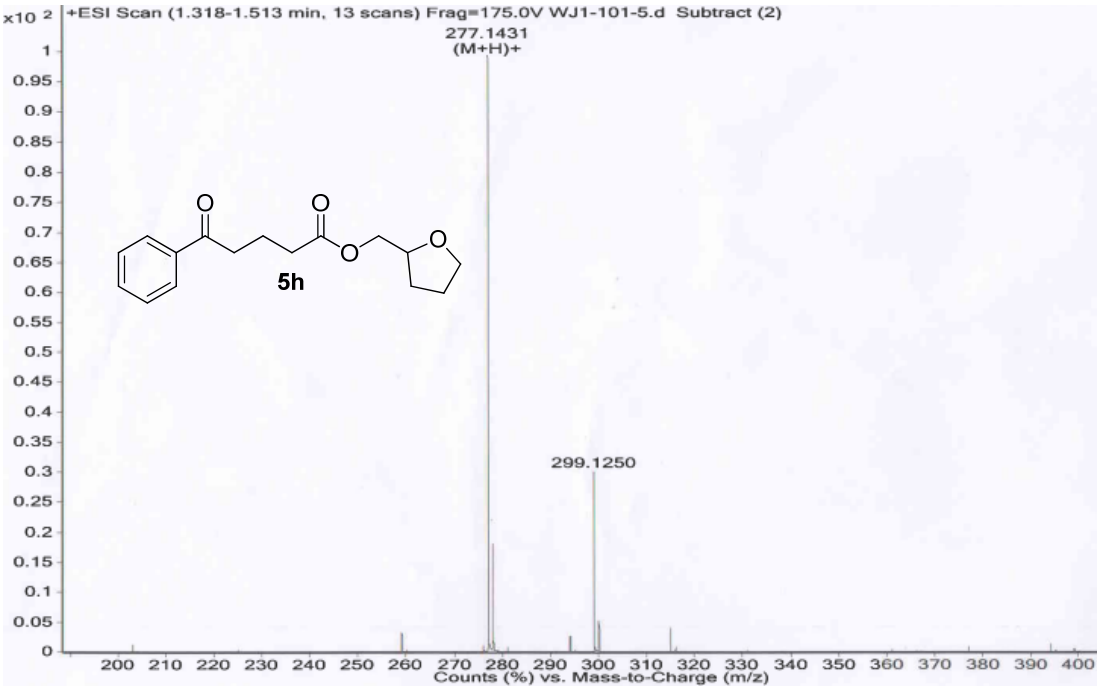

+ESI Scan (2.250-2.494 min, 16 scans) Frag=175.0V WJ1-101-4.d Subtract (2)
251.1276
(M+H)+
273.1104
5g
Counts (%) vs. Mass-to-Charge (m/z)

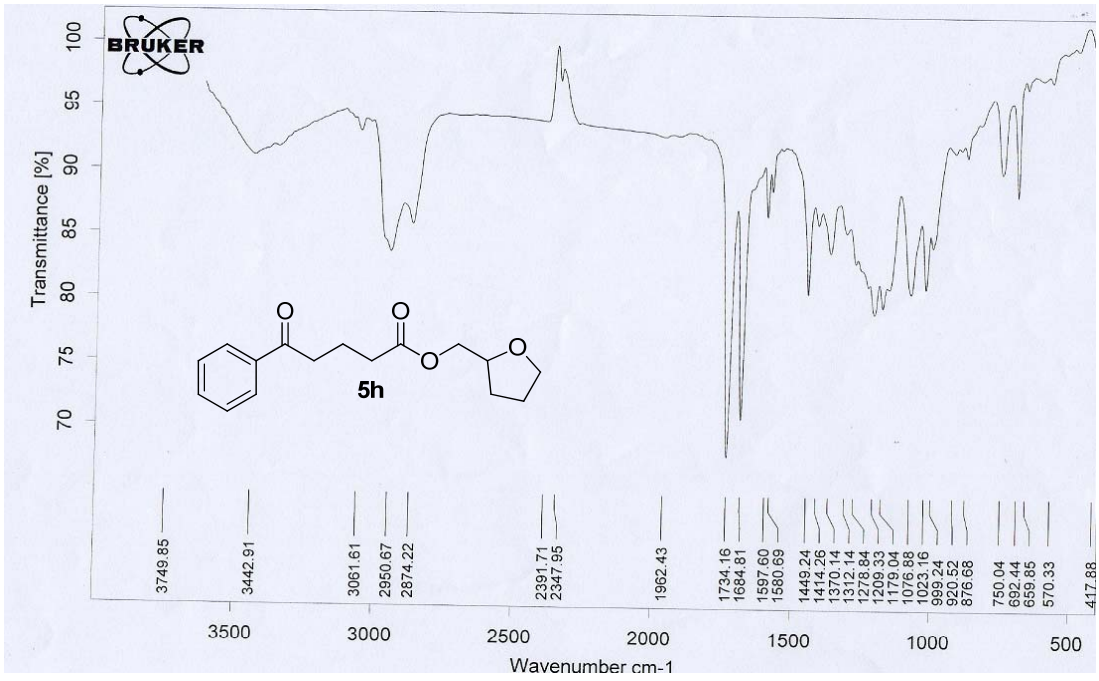

Transmittance [%]
5g
3451.10
3353.34
3061.73
2934.42
2893.75
2821.07
2393.45
1974.35
1733.25
1684.65
1597.60
1580.86
1449.65
1409.99
1376.05
1279.71
1202.42
1179.76
1128.34
1100.97
1074.25
1056.16
1033.40
1000.79
985.18
933.77
876.79
861.73
749.07
692.23
658.54
569.09
532.42
Wavenumber cm-1

5h

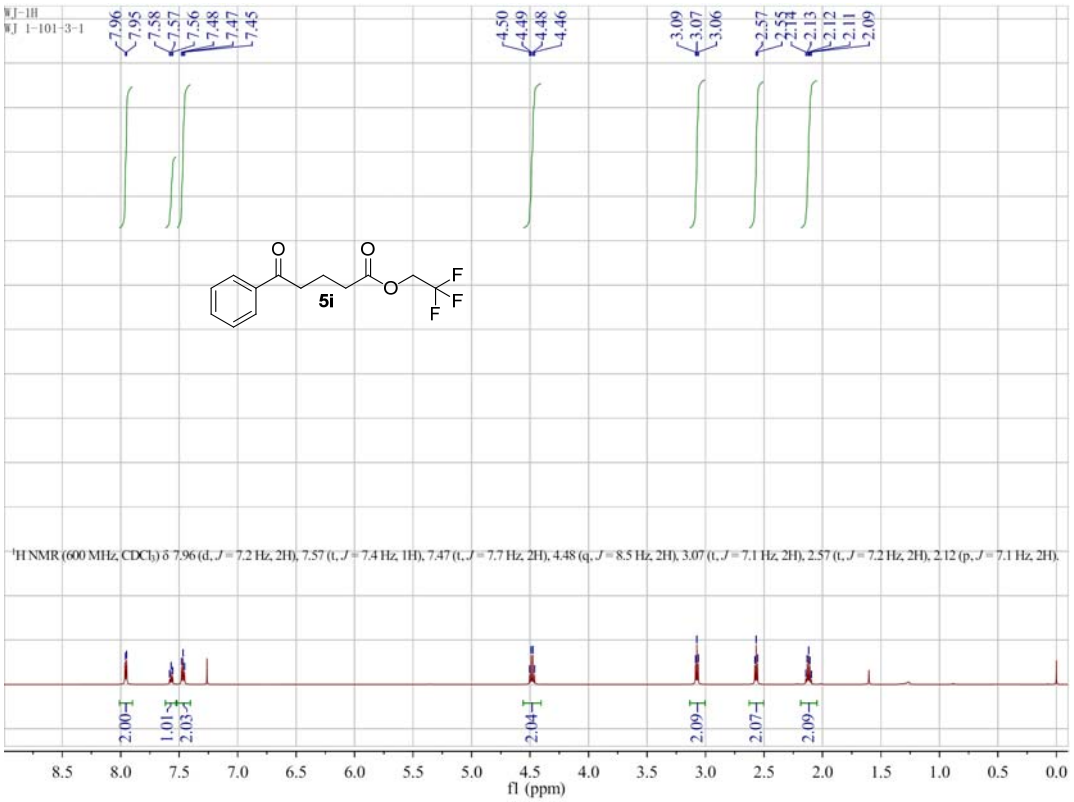

5h
^{1}H NMR (600 MHz, CDCl$_3$) δ 7.96 (d, J = 7.8 Hz, 2H), 7.55 (t, J = 7.3 Hz, 1H), 7.46 (t, J = 7.6 Hz, 2H), 4.17 (dd, J = 11.3, 3.4 Hz, 1H), 4.11 (dt, J = 6.9, 5.2 Hz, 1H), 4.03 (dd, J = 11.3, 6.8 Hz, 1H), 3.87 (dd, J = 14.6, 7.2 Hz, 1H), 3.78 (dd, J = 14.5, 7.3 Hz, 1H), 3.06 (t, J = 7.2 Hz, 2H), 2.49 (t, J = 7.1 Hz, 2H), 2.08 (p, J = 7.1 Hz, 2H), 1.99 (dt, J = 12.4, 7.5 Hz, 1H), 1.95 – 1.83 (m, 2H), 1.60 (dt, J = 19.6, 7.5 Hz, 1H).
f1 (ppm)

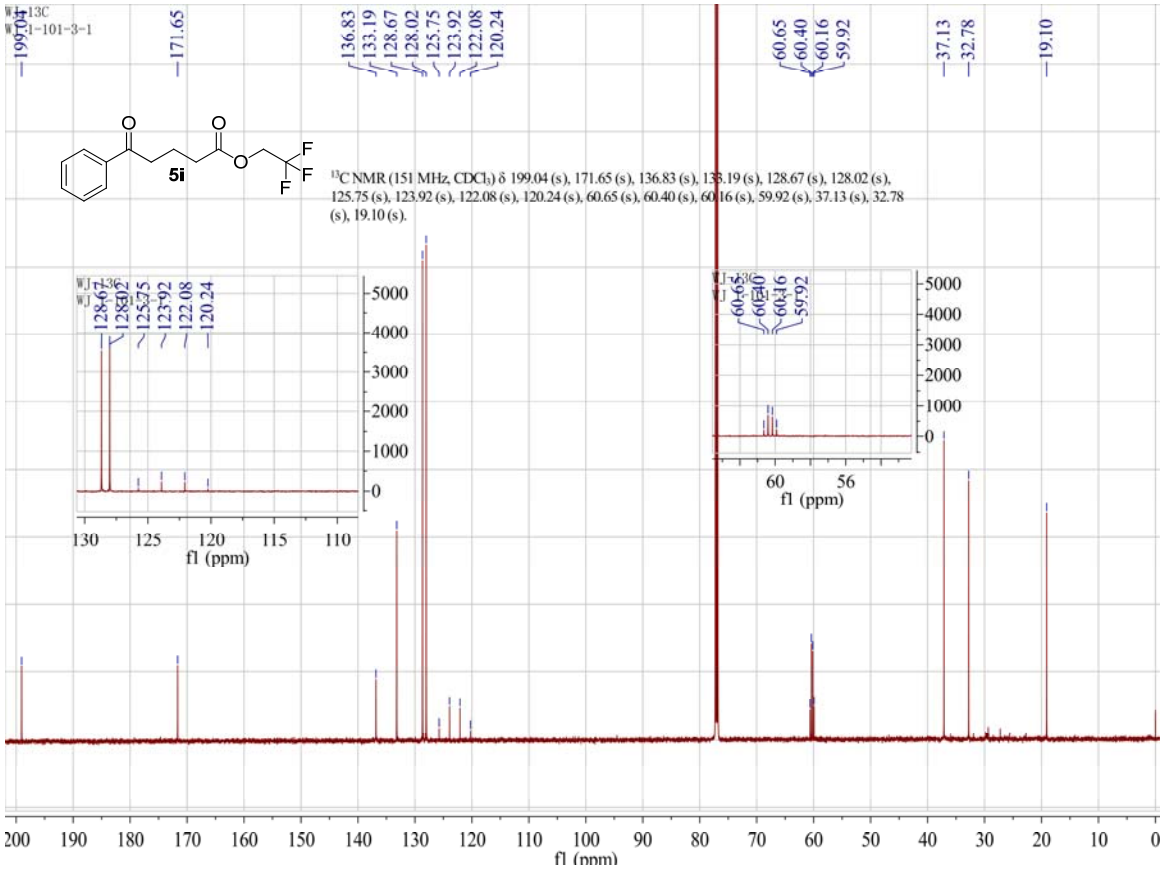

5h
^{13}C NMR (151 MHz, CDCl$_3$) δ 199.40 (s), 173.19 (s), 136.93 (s), 133.04 (s), 128.60 (s), 128.05 (s), 76.51 (s), 68.42 (s), 66.46 (s), 37.47 (s), 33.30 (s), 28.02 (s), 25.67 (s), 19.45 (s).
f1 (ppm)

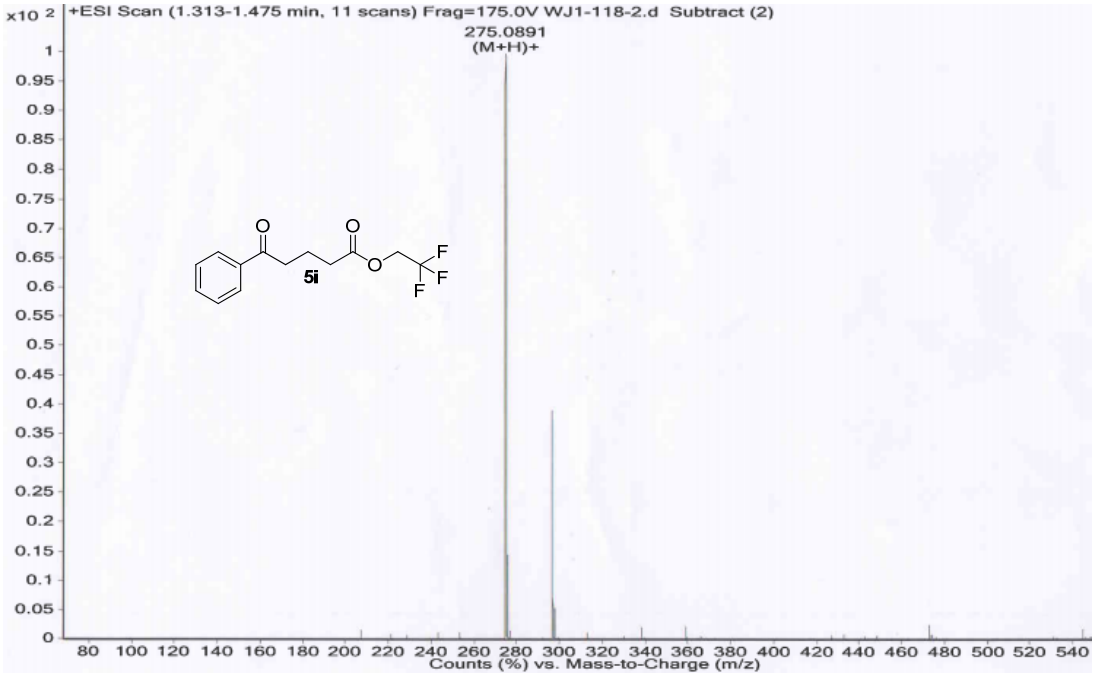

x10 2
+ESI Scan (1.318-1.513 min, 13 scans) Frag=175.0V WJ1-101-5.d Subtract (2)
277.1431
(M+H)+
299.1250
5h
Counts (%) vs. Mass-to-Charge (m/z)

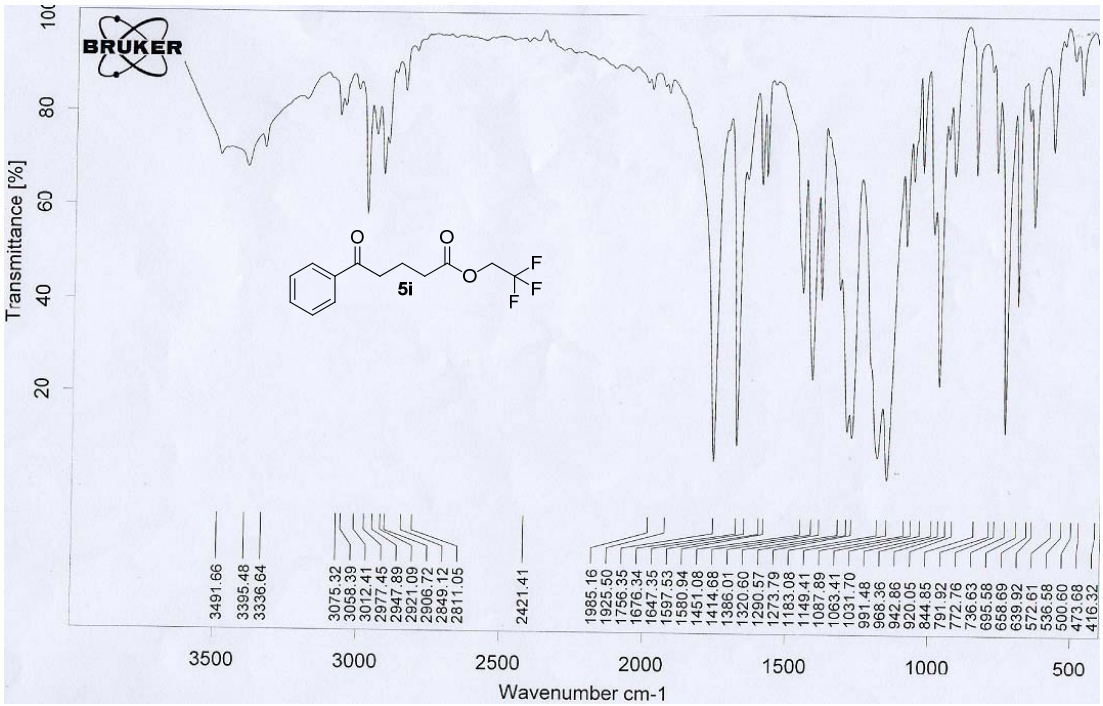

BRUKER
Transmittance [%]
5h
3749.85
3442.91
3061.61
2950.67
2874.22
2391.71
2347.95
1962.43
1734.16
1684.81
1597.60
1580.69
1449.24
1414.26
1370.14
1312.14
1278.84
1209.33
1179.04
1076.88
1023.16
999.24
920.52
876.68
750.04
692.44
659.85
570.33
417.88
Wavenumber cm-1

5i

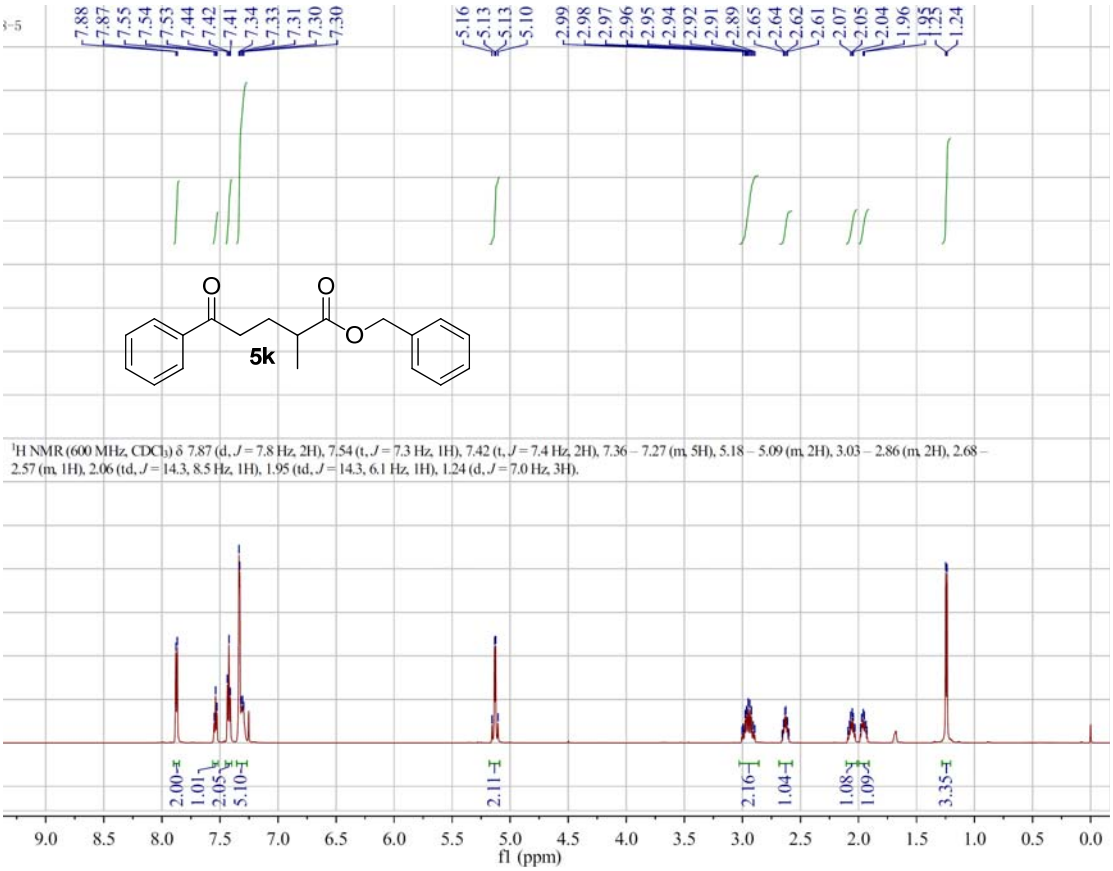

WJ-1H
WJ 1-101-3-1
7.96
7.95
7.58
7.57
7.56
7.48
7.47
7.45
4.50
4.49
4.48
4.46
3.09
3.07
3.06
2.57
2.55
2.14
2.13
2.12
2.11
2.09
5i
F
F
F
O
O
O
^{1}H NMR (600 MHz, CDCl$_3$) δ 7.96 (d, J = 7.2 Hz, 2H), 7.57 (t, J = 7.4 Hz, 1H), 7.47 (t, J = 7.7 Hz, 2H), 4.48 (q, J = 8.5 Hz, 2H), 3.07 (t, J = 7.1 Hz, 2H), 2.57 (t, J = 7.2 Hz, 2H), 2.12 (p, J = 7.1 Hz, 2H).
2.00
1.01
2.03
2.04
2.09
2.07
2.09
f1 (ppm)

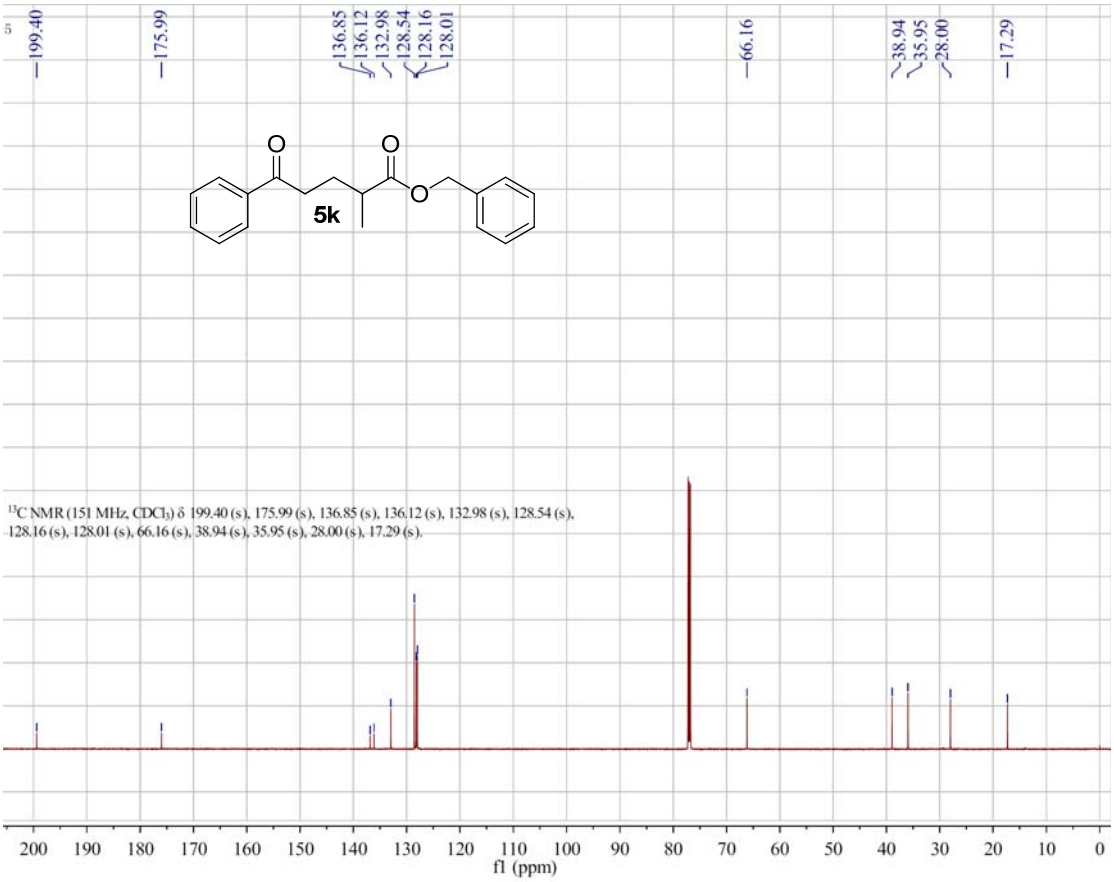

WJ-13C
WJ 1-101-3-1
199.04
171.65
136.83
133.19
128.67
128.02
125.75
123.92
122.08
120.24
60.65
60.40
60.16
59.92
37.13
32.78
19.10
5i
F
F
F
O
O
O
^{13}C NMR (151 MHz, CDCl$_3$) δ 199.04 (s), 171.65 (s), 136.83 (s), 133.19 (s), 128.67 (s), 128.02 (s), 125.75 (s), 123.92 (s), 122.08 (s), 120.24 (s), 60.65 (s), 60.40 (s), 60.16 (s), 59.92 (s), 37.13 (s), 32.78 (s), 19.10 (s).
f1 (ppm)

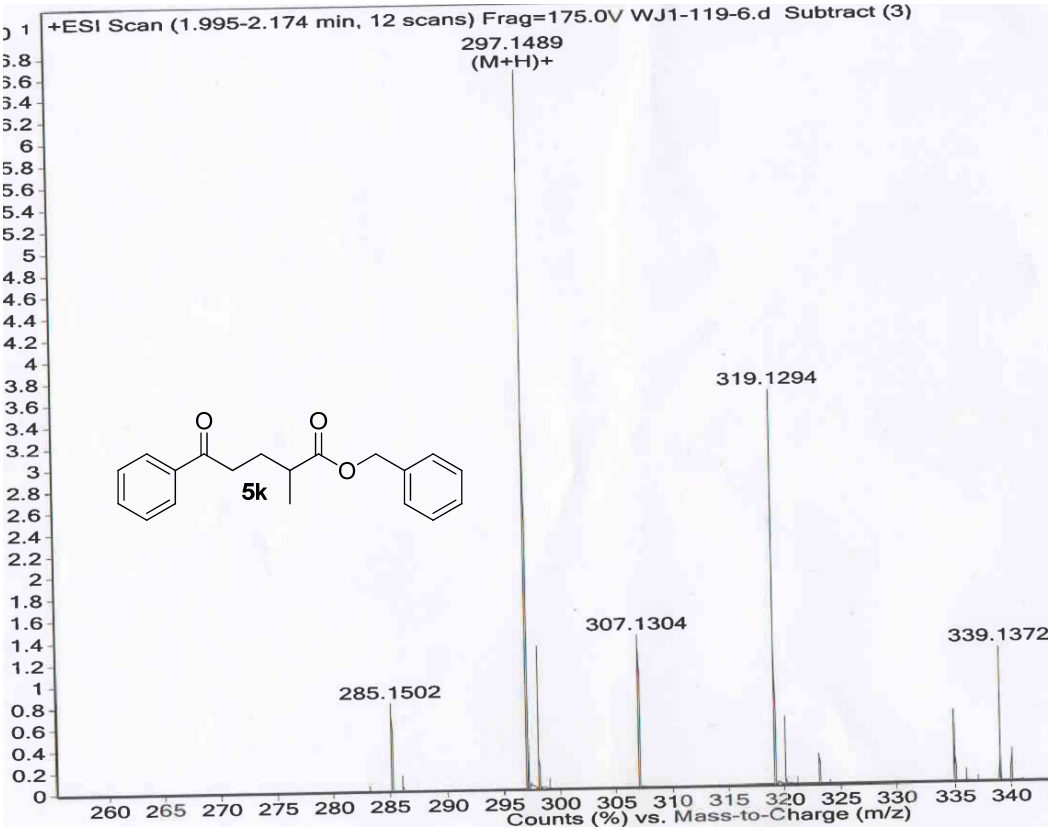

+ESI Scan (1.313-1.475 min, 11 scans) Frag=175.0V WJ1-118-2.d Subtract (2)
275.0891
(M+H)+
x10 2
5i
Counts (%) vs. Mass-to-Charge (m/z)

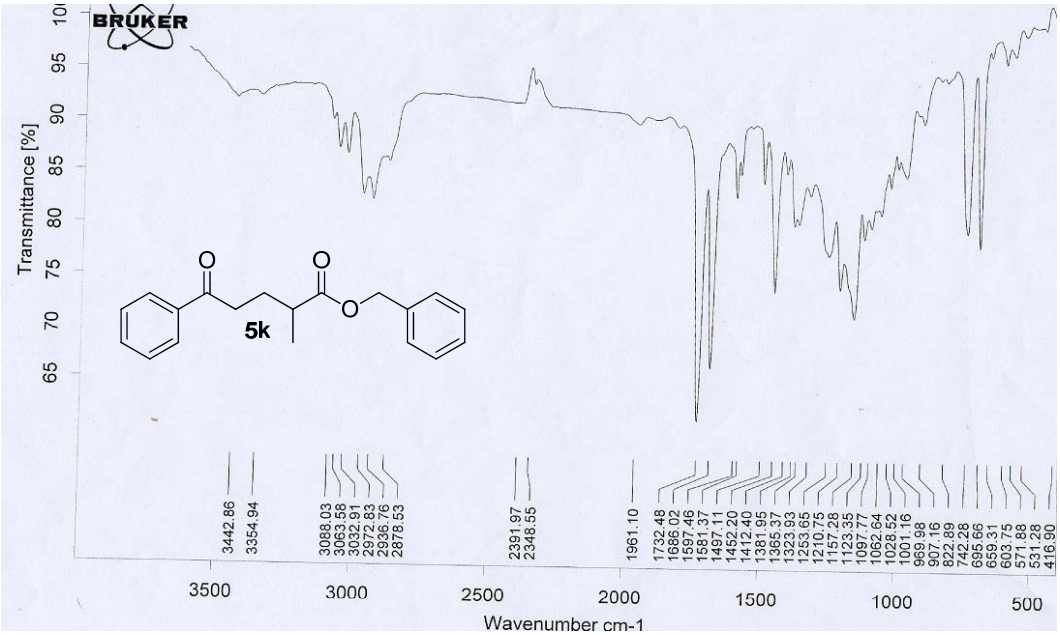

BRUKER
5i
Transmittance [%]
3491.66
3385.48
3336.64
3075.32
3058.39
3012.41
2977.45
2947.89
2921.09
2906.72
2849.12
2811.05
2421.41
1985.16
1925.50
1756.35
1676.34
1647.35
1597.53
1580.94
1451.08
1414.68
1386.01
1320.60
1290.57
1273.79
1183.08
1149.41
1087.89
1063.41
1031.70
991.48
968.36
942.86
920.05
844.85
791.92
772.76
736.63
695.58
658.69
639.92
572.61
536.58
500.60
473.68
416.32
Wavenumber cm-1

5k


7.88
7.87
7.55
7.54
7.53
7.44
7.42
7.41
7.34
7.33
7.31
7.30
7.30
5.16
5.13
5.13
5.10
2.99
2.98
2.97
2.96
2.95
2.94
2.92
2.91
2.89
2.65
2.64
2.62
2.61
2.07
2.05
2.04
1.96
1.25
1.24
5k
¹H NMR (600 MHz, CDCl₃) δ 7.87 (d, J = 7.8 Hz, 2H), 7.54 (t, J = 7.3 Hz, 1H), 7.42 (t, J = 7.4 Hz, 2H), 7.36 – 7.27 (m, 5H), 5.18 – 5.09 (m, 2H), 3.03 – 2.86 (m, 2H), 2.68 – 2.57 (m, 1H), 2.06 (td, J = 14.3, 8.5 Hz, 1H), 1.95 (td, J = 14.3, 6.1 Hz, 1H), 1.24 (d, J = 7.0 Hz, 3H).
2.00
1.01
2.05
5.10
2.11
2.16
1.04
1.08
1.09
3.35
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


199.40
175.99
136.85
136.12
132.98
128.54
128.16
128.01
66.16
38.94
35.95
28.00
17.29
5k
¹³C NMR (151 MHz, CDCl₃) δ 199.40 (s), 175.99 (s), 136.85 (s), 136.12 (s), 132.98 (s), 128.54 (s), 128.16 (s), 128.01 (s), 66.16 (s), 38.94 (s), 35.95 (s), 28.00 (s), 17.29 (s).
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


+ESI Scan (1.995-2.174 min, 12 scans) Frag=175.0V WJ1-119-6.d Subtract (3)
297.1489
(M+H)+
319.1294
307.1304
339.1372
285.1502
5k
Counts (%) vs. Mass-to-Charge (m/z)


BRUKER
Transmittance [%]
5k
3442.86
3354.94
3088.03
3063.58
3032.91
2972.83
2936.76
2878.53
2391.97
2348.55
1961.10
1732.48
1686.02
1597.46
1581.37
1497.11
1452.20
1412.40
1381.95
1365.37
1323.93
1253.65
1210.75
1157.28
1123.35
1097.77
1062.64
1028.52
1001.16
969.98
907.16
822.89
742.28
695.66
659.31
603.75
571.88
531.28
416.90
Wavenumber cm-1