Supporting Information

Synthesis of Medium-Chain Acids or α, ω-Dicarboxylic Acids from Cellulose-Derived Platform Chemicals

Xing-Long Li, Kun Zhang, Ju-Long Jiang, Rui Zhu, Wei-Peng Wu, Jin Deng*, Yao Fu*

Hefei National Laboratory for Physical Sciences at the Microscale, iChEM, CAS Key Laboratory of Urban Pollutant Conversion, Anhui Province Key Laboratory of Biomass Clean Energy, Department of Chemistry, University of Science and Technology of China, Hefei 230026 (P. R. China)

E-mail: dengjin@ustc.edu.cn

fuyao@ustc.edu.cn

Supplementary information

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Ⅰ. General information

If not stated otherwise, the reaction was carried out in an autoclave sponsored by Anhui Kemi Machinery Technology Co., Ltd. Usually, the initial temperature is 30 °C, then heating to the required reaction temperature through intelligent temperature controller in 30 min. After that the reaction was heating preservation with a certain period of time.

If not stated otherwise, the reagents were purchased from Sinopharm group Co., Ltd. Furfural was purified by vacuum distillation. 5-HMF was supplied by Hefei Leaf Energy Biotechnology Co., Ltd. Levulinic acid was purchased from Aladdin Reagent. Methyl malonate was purchased from J & K. $Hf(OTf)_4$, Pd/C (10%) were purchased from Alfa Aesar (China) Chemicals Co., Ltd. $Sn(OTf)_2$, $La(OTf)_3$, $Fe(OTf)_3$, $Al(OTf)_3$ were purchased from Adamas Reagent Co., Ltd. The preparation methods of $Zr(OTf)_4$ and $W(OTf)_6$ are listed in Catalyst and substrate production.

If not stated otherwise, δ-furfurylidenelevulinic acid are quantitatively converted under experimental conditions. This is mainly due to the easy transformation of raw material and the strong hydrogenation capacity of Pd/C catalyst.

Analytics

Gas chromatographic (GC) analysis was acquired on a Shimadzu GC-2014 Series GC System equipped with a FFAP capillary column (30 m×0.32 mm×0.25 μm, SHIMADZU, GC-2014C).

^{1}H-NMR and ^{13}C-NMR spectra were recorded on a Bruker Avance 400 spectrometer at ambient temperature. Multiplicities are described using the following abbreviations: chemical shift (ppm, scale), multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet and/or multiplet resonances, br = broad), coupling constant (Hz), and integration. NMR spectra were analysed with MestReNova software.

ESI-MS spectrum data were acquired using a Thermo LTQ Orbitrap XL Instrument equipped with an ESI source and controlled by Xcalibur software.

Transmission electron microscopy (TEM) microphotographs were operated on JEOL-2010 electron microscope at 200 kV. The samples were suspended in ethanol.

Dielectric Constants of Solvents [1]

Table S1. The dielectric constant of reaction solvents.

Solvent	Dielectric Constants（20 °C）
n-octane	1.9
Acetic acid	6.2
Tetrahydrofuran	7.6
Methanol	32.6
Water	79.7

Typical experiment and product analysis

The catalytic conversion of aldol condensation products was carried out in a 25 mL stainless steel high pressure parr reactor equipped with a magnetic stirrer, a temperature probe and a temperature programmed controller. In a typical experiment, 0.5 mmol of substrate, 2

mol% of hydrogenation catalyst, 6 mol% of Lewis acid catalyst, and 5 mL of solvent were added to the parr reactor. Tightened the screws, replaced the gas with hydrogen three times and pressurized the hydrogen to the required pressure at room temperature. Heating the reactor to the desired temperature under 500 rpm/min magnetic stirring in a period of 30 min. Then cooling and relief pressure after holding reaction for a required reaction time. The reaction mixture in the reactor was transferred out with 20 mL of ethyl acetate and a certain amount of dimethyl phthalate (DMP) was added as an internal standard. The yield of decanoic acid was determined by internal standard method using a gas chromatograph. The yield of the product was calculated according to the following formula：

$$Y/\,\% = \frac{n_{\text{decanoic acid}}}{n_{\text{substrate}}} \times 100\%$$

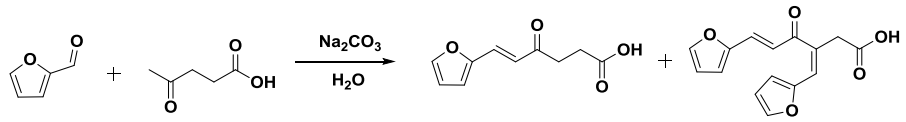
Ⅱ. Catalyst and substrate production

1. Catalyst production

$Zr(OTf)_4$ was synthesized according to the literature. [2] The synthesis procedure of $W(OTf)_6$ was showed below: Trifluoromethanesulfonic acid 20.0 g (133.3 mmol) was added to tungsten chloride 5.0 g (12.6 mmol) and the mixture was heated at 50 °C in the Schlenk tube with a drying tube for 72 h under argon. The excess trifluoromethanesulfonic acid was removed under vacuum followed by dried for 8 h under vacuum to give $W(OTf)_6$ catalyst.

2. Synthesis of substrates and products

2.1 Synthesis of δ-Furfurylidenelevulinic acid

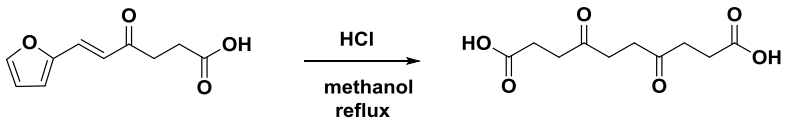


The product was prepared by aldol condensation of furfural and levulinic acid according to the literature. [3] 18.0 g (155.0 mmol) of levulinic acid, 100 mL of pure water was added to a 500 mL three-necked flask equipped with a reflux condenser and a constant pressure dropping funnel. 24.0 g (226.0 mmol) of Na_2CO_3 was added in portions at room temperature accompanied with CO_2 release. After that the reaction temperature is heated to reflux. A solution of 7.7 mL (93.0 mmol) of furfural and 16 mL of methanol were added dropwise under vigorous stirring more than 1 h. After addition, the reaction was stopped until substantially no furfural was detected by TLC (ethyl acetate: petroleum ether=1:3). Slowly pour the reaction solution into cold dilute hydrochloric acid after rapid cooling. Filtration, the solid were washed several times with ice water. Recrystallization from hot water: (1). Crystallization from water to obtain 9.0 g of a light yellow solid (**δ-Furfurylidenelevulinic acid)** with a yield of 58.0%. ^{1}H-NMR (400 MHz, $CDCl_3$): δ = 10.01 (s, 1H), 7.51 (d, *J* = 1.5 Hz, 1H), 7.36 (d, *J* = 15.8 Hz, 1H), 6.76-6.61 (m, 2H), 6.49 (dd, *J* = 3.4, 1.8 Hz, 1H), 2.96 (t, *J* = 6.6 Hz, 2H), 2.73 (t, *J* = 6.6 Hz, 2H). ^{13}C-NMR (101 MHz, $CDCl_3$): δ = 197.44, 178.42, 150.90, 145.13, 129.20, 122.73, 116.14, 112.62, 35.44, 28.00.;

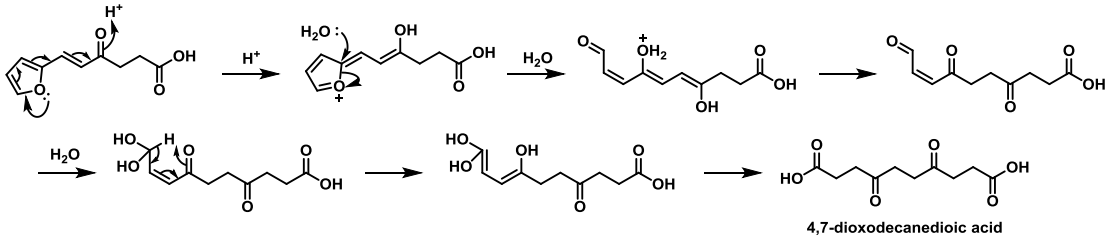
(2). The obtained insoluble matters of 3.6 g brown solids **((3Z, 5E)-6-(furan-2-yl)-3-(furan-2-ylmethylene)-4-oxohex-5-enoic acid)** with a yield of 30.0%. A new preparation method with high yield is used to obtain the later product. And the related experiments were listed below.

2.2 Synthesis of substrate and product in Table 2 Entry 1

Synthesis of δ, δ'-Dilevulinic acid [3]

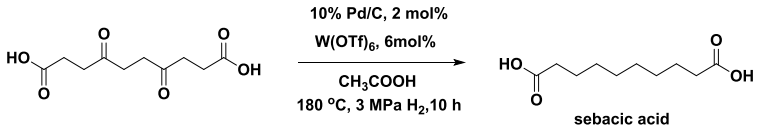


A plausible mechanism:



δ,δ'-Dilevulinic acid, the raw material of sebacic acid, was prepared according to the literature [3]. 12.0 mL of methanol, 1.8 g δ-furfurylidenelevulinic acid and 6.6 mL of 37% concentrated hydrochloric acid were added to a 250 mL single-necked flask. The reaction was heated to reflux overnight under magnetic stirring. Then evaporated the solvent, and the residue was dissolved in hot water followed by decolorization with activated carbon. Then filtration, the filtrate was evaporated until solid precipitation. Cooled, filtered and dried to give a white solid powder 1.7 g (77% yield). ^{1}H-NMR (400 MHz, DMSO): δ = 12.10 (s, 2H), 2.66 (dd, *J* = 11.4, 4.6 Hz, 8H), 2.38 (t, *J* = 6.5 Hz, 4H). ^{13}C-NMR (101 MHz, DMSO): δ = 208.30, 174.17, 37.04, 36.06, 28.12.

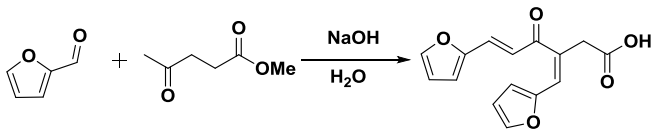
Synthesis of sebacic acid from δ, δ'-Dilevulinic acid



The catalytic conversion of **δ, δ'-Dilevulinic acid** to **sebacic acid** was carried out in a 25 mL stainless steel high pressure parr reactor equipped with a magnetic stirrer, a temperature probe and a temperature programmed controller. In a typical experiment, 0.5 mmol of **δ, δ'-Dilevulinic acid**, 2 mol% of 10% Pd/C catalyst, 6 mol% of $W(OTf)_6$, and 5 mL of acetic acid were added to the parr reactor. Tightened the screws, replaced the gas with hydrogen three times and pressurized the hydrogen to 3 MPa at room temperature. Heating the reactor from room temperature to 180 °C in a period of 30 min under 500 rpm/min magnetic stirring. Then cooling and relief pressure after holding reaction at 180 °C for 10 h. Removal of acetic acid by vacuum distillation after filtrating Pd/C catalyst. Then water was added to residue and the solution was extracted with ether several times. The ether layer was washed with saturated NaCl solution and dried by anhydrous magnesium sulfate. The solvent was evaporated and a white solid (89.0 mg, 88% mol yield) was obtained. ^{1}H-NMR (400 MHz, DMSO): δ = 11.98 (s, 2H), 2.18 (t, *J* = 7.4 Hz, 4H), 1.55 - 1.40 (m, 4H), 1.25 (s, 8H). ^{13}C-NMR (101 MHz, DMSO): δ = 174.97, 34.12, 29.03 (d, *J* = 11.7 Hz), 24.94.

2.3 Synthesis of substrate and product in Table 2 Entry 2

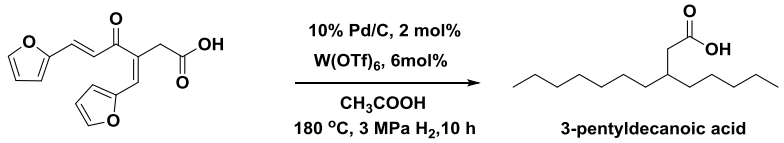
Synthesis of (3Z, 5E)-6-(furan-2-yl)-3-(furan-2-ylmethylene)-4-oxohex-5-enoic acid [4]



Methyl levulinate(1.3 g, 10 mmol) was added to a cooled NaOH (25 mL, 1.0 M) solution, then furfural (2.0 g, 21 mmol) was added dropwise. The solution were heated to 50 °C

overnight. After reaction, the solution was cooled down and neutralized with HCl solution. Then the rufous solid product was precipitated. Washed with hot water several times after filtration followed by dried at 50 ^{o}C under vacuum to obtain a rufous solid powder (2.2 g, 80% yield). ^{1}H-NMR (400 MHz, $CDCl_3$): δ = 7.63 (s, 1H), 7.52 (dd, *J* = 13.9, 5.3 Hz, 3H), 7.31 (d, *J* = 15.2 Hz, 1H), 6.91 (d, *J* = 2.9 Hz, 1H), 6.72 (d, *J* = 3.2 Hz, 1H), 6.54 (ddd, *J* = 17.0, 3.2, 1.7 Hz, 2H), 3.91 (s, 2H). ^{13}C-NMR (101 MHz, $CDCl_3$): δ = 191.23, 174.45, 151.57, 150.51, 150.27, 145.94, 145.03, 130.83, 130.13, 128.25, 118.00, 117.67, 116.49, 112.77, 33.68.

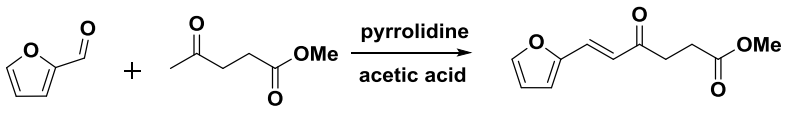
Synthesis of 3-pentyldecanoic acid from (3Z, 5E)-6-(furan-2-yl)-3-(furan-2-ylmethylene) -4-oxohex-5-enoic acid



The catalytic conversion of **(3Z, 5E)-6-(furan-2-yl)-3-(furan-2-ylmethylene)-4-oxohex -5-enoic acid** to **3-pentyldecanoic acid** was carried out in a 25 mL stainless steel high pressure parr reactor equipped with a magnetic stirrer, a temperature probe and a temperature programmed controller. In a typical experiment, 0.5 mmol of **(3Z, 5E)-6-(furan-2-yl)-3-(furan-2-ylmethylene)-4-oxohex-5-enoic acid**, 2 mol% of 10% Pd/C catalyst, 6 mol% of $W(OTf)_6$, and 5 mL of acetic acid were added to the parr reactor. Tightened the screws, replaced the gas with hydrogen three times and pressurized the hydrogen to 3 MPa at room temperature. Heating the reactor from room temperature to 180 °C in a period of 30 min under 500 rpm/min magnetic stirring. Then cooling and relief pressure after holding reaction at 180 °C for 10 h. Removal of acetic acid by vacuum distillation after filtrating Pd/C catalyst. Then water was added to residue and the solution was extracted with ether several times. The ether layer was washed with saturated NaCl solution and dried by anhydrous magnesium sulfate. The solvent was evaporated and a white solid (87.3 mg, 72% mol yield) was obtained. ^{1}H-NMR (400 MHz, $CDCl_3$): δ = 10.98 (s, 1H), 2.27 (d, *J* = 6.8 Hz, 1H), 2.05 (d, *J* = 2.9 Hz, 1H), 1.59 (dd, *J* = 13.5, 6.6 Hz, 1H), 1.27 (s, 20H), 0.88 (t, *J* = 6.7 Hz, 6H). ^{13}C-NMR (101 MHz, $CDCl_3$): δ = 179.75, 38.96, 34.87, 33.78, 33.74, 32.06, 31.87, 29.82, 29.28, 26.51, 26.17, 22.68, 22.63, 14.11, 14.07.

2.4 Synthesis of substrate and product in Table 2 Entry 3

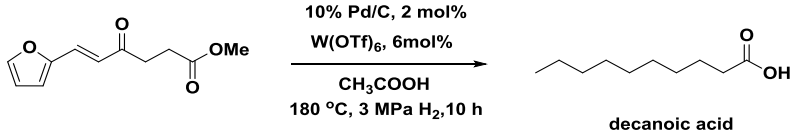
Synthesis of δ-Furfurylidenelevulinic acid methyl ester [5]



340 μL (4.2 mmol) of pyrrolidine and 240 μL of acetic acid (4.2 mmol) were sequentially added to an iced solution of 2.7 g (20.8 mmol) methyl levulinate and 2.0 g of furfural (20.8 mmol) in a Schlenk tube. The resultant solution was allowed to stay at ambient temperature and stir until the reaction was completed by TLC. The resultant solution was washed with petroleum ether several times. Then the residue were added by ethyl acetate and purified by a

short silica column. The solvent was evaporated and a reddish cast oil (4.4 g, 90% yield) was obtained. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.51 (s, 1H), 7.38 – 7.32 (m, 1H), 6.66 (dd, *J* = 12.5, 9.7 Hz, 2H), 6.49 (dd, *J* = 3.3, 1.8 Hz, 1H), 3.68 (s, 3H), 2.96 (t, *J* = 6.7 Hz, 2H), 2.67 (t, *J* = 6.7 Hz, 2H). ^{13}C NMR (101 MHz, $CDCl_3$): δ = 197.42, 173.19, 150.85, 145.03, 128.85, 122.86, 115.89, 112.54, 51.65, 35.49, 27.86.

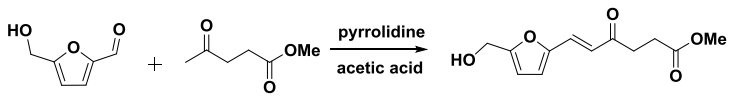
Synthesis of decanoic acid from δ-Furfurylidenelevulinic acid methyl ester



The catalytic conversion of **δ-Furfurylidenelevulinic acid methyl ester** to **decanoic acid** was carried out in a 25 mL stainless steel high pressure parr reactor equipped with a magnetic stirrer, a temperature probe and a temperature programmed controller. In a typical experiment, 0.5 mmol of **δ-Furfurylidenelevulinic acid methyl ester**, 2 mol% of 10% Pd/C catalyst, 6 mol% of $W(OTf)_6$, and 5 mL of acetic acid were added to the parr reactor. Tightened the screws, replaced the gas with hydrogen three times and pressurized the hydrogen to 3 MPa at room temperature. Heating the reactor from room temperature to 180 °C in a period of 30 min under 500 rpm/min magnetic stirring. Then cooling and relief pressure after holding reaction at 180 °C for 10 h. Removal of acetic acid by vacuum distillation after filtrating Pd/C catalyst. Then water was added to residue and the solution was extracted with ether several times. The ether layer was washed with saturated NaCl solution and dried by anhydrous magnesium sulfate. The solvent was evaporated and a white solid (80.8 mg, 94% mol yield) was obtained. ^{1}H-NMR (400 MHz, $CDCl_3$): δ = 9.25 (s, 1H), 2.35 (t, *J* = 7.5 Hz, 2H), 1.69 - 1.58 (m, 2H), 1.31 (dd, *J* = 21.8, 10.9 Hz, 12H), 0.88 (t, *J* = 6.9 Hz, 3H). ^{13}C-NMR (101 MHz, $CDCl_3$): δ = 180.36, 34.10, 31.86, 29.40, 29.26, 29.06, 24.68, 22.67, 14.11.

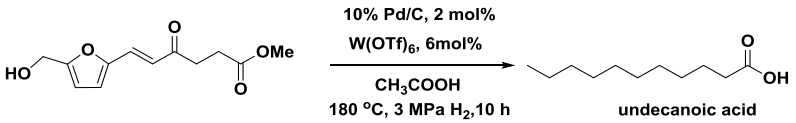
2.5 Synthesis of substrate and product in Table 2 Entry 4

Synthesis of methyl (E)-6-(5-(hydroxymethyl)furan-2-yl)-4-oxohex-5-enoate [5]



340 μL (4.2 mmol) of pyrrolidine and 240 μL of acetic acid (4.2 mmol) were sequentially added to an iced solution of 2.7 g (20.8 mmol) methyl levulinate and 2.6 g of 5-hydroxymethylfurfural (20.8 mmol) in a Schlenk tube. The resultant solution was allowed to stay at ambient temperature and stir until the reaction was completed by TLC. The resultant solution was washed with petroleum ether several times. Then the residue were added by ethyl acetate and purified by a short silica column. The solvent was evaporated and a reddish cast oil (4.4 g, 90% yield) was obtained. ^{1}H-NMR (400 MHz, $CDCl_3$): δ = 7.31 (d, *J* = 15.9 Hz, 1H), 6.64 (dd, *J* = 12.7, 9.6 Hz, 2H), 6.39 (d, *J* = 3.2 Hz, 1H), 4.65 (s, 2H), 3.69 (s, 3H), 2.94 (t, *J* = 6.7 Hz, 2H), 2.68 (t, *J* = 6.6 Hz, 2H), 2.10 (s, 1H). ^{13}C-NMR (101 MHz, $CDCl_3$): δ = 197.50, 173.36, 156.76, 150.88, 128.78, 122.84, 116.88, 110.52, 57.67, 51.85, 35.84, 27.99.

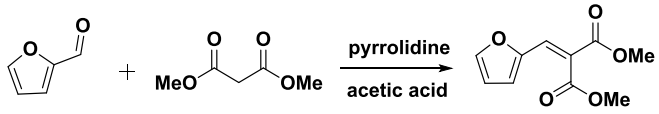
Synthesis of undecanoic acid from methyl (E)-6-(5-(hydroxymethyl)furan-2-yl)-4-oxohex-5-enoate



The catalytic conversion of **methyl (E)-6-(5-(hydroxymethyl)furan-2-yl)-4-oxohex-5-enoate** to **undecanoic acid** was carried out in a 25 mL stainless steel high pressure parr reactor equipped with a magnetic stirrer, a temperature probe and a temperature programmed controller. In a typical experiment, 0.5 mmol of **methyl (E)-6-(5-(hydroxymethyl) furan -2-yl)-4- oxohex-5-enoate**, 2 mol% of 10% Pd/C catalyst, 6 mol% of $W(OTf)_6$, and 5 mL of acetic acid were added to the parr reactor. Tightened the screws, replaced the gas with hydrogen three times and pressurized the hydrogen to 3 MPa at room temperature. Heating the reactor from room temperature to 180 °C in a period of 30 min under 500 rpm/min magnetic stirring. Then cooling and relief pressure after holding reaction at 180 °C for 10 h. Removal of acetic acid by vacuum distillation after filtrating Pd/C catalyst. Then water was added to residue and the solution was extracted with ether several times. The ether layer was washed with saturated NaCl solution and dried by anhydrous magnesium sulfate. The solvent was evaporated and a light yellow solid (83.8 mg, 80% mol yield) was obtained. ^{1}H-NMR (400 MHz, $CDCl_3$): δ = 2.34 (t, *J* = 7.4 Hz, 2H), 1.66 - 1.60 (m, 2H), 1.28 (d, *J* = 15.0 Hz, 14H), 0.88 (t, *J* = 6.7 Hz, 3H). ^{13}C-NMR (101 MHz, $CDCl_3$): δ = 180.42, 34.13, 31.90, 29.56, 29.45, 29.32, 29.26, 29.07, 24.69, 22.69, 14.12.

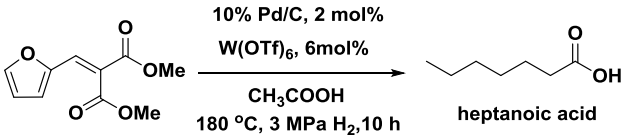
2.6 Synthesis of substrate and product in Table 2 Entry 5

Synthesis of dimethyl 2-(furan-2-ylmethylene)malonate [5]



340 μL (4.2 mmol) of pyrrolidine and 240 μL of acetic acid (4.2 mmol) were sequentially added to an iced solution containing of 2.8 g (20.8 mmol) dimethyl malonate and 2.0 g of furfural (20.8 mmol) in a Schlenk tube. The resultant solution were allowed to stay at ambient temperature and stir until the reaction was completed by TLC. The resultant solution was washed with petroleum ether several times. Then the residue were added by ethyl acetate and purified by a short silica column. The solvent was evaporated and a reddish cast oil (3.4 g, 80% yield) was obtained. ^{1}H-NMR (400 MHz, $CDCl_3$): δ = 7.53 (d, *J* = 1.7 Hz, 1H), 7.49 (s, 1H), 6.78 (d, *J* = 3.5 Hz, 1H), 6.50 (dd, *J* = 3.5, 1.8 Hz, 1H), 3.92 (s, 3H), 3.83 (s, 3H). ^{13}C-NMR (101 MHz, $CDCl_3$): δ = 166.75, 164.53, 148.85, 146.40, 128.19, 121.13, 118.34, 112.68, 52.60, 52.56.

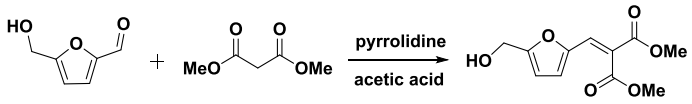
Synthesis of heptanoic acid from dimethyl 2-(furan-2-ylmethylene)malonate



The catalytic conversion of **dimethyl 2-(furan-2-ylmethylene)malonate** to **heptanoic acid** was carried out in a 25 mL stainless steel high pressure parr reactor equipped with a magnetic stirrer, a temperature probe and a temperature programmed controller. In a typical experiment, 0.5 mmol of **dimethyl 2-(furan-2-ylmethylene)malonate**, 2 mol% of 10% Pd/C catalyst, 6 mol% of $W(OTf)_6$, and 5 mL of acetic acid were added to the parr reactor. Tightened the screws, replaced the gas with hydrogen three times and pressurized the hydrogen to 3 MPa at room temperature. Heating the reactor from room temperature to 180 °C in a period of 30 min under 500 rpm/min magnetic stirring. Then cooling and relief pressure after holding reaction at 180 °C for 10 h. Removal of acetic acid by vacuum distillation after filtrating Pd/C catalyst. Then water was added to residue and the solution was extracted with ether several times. The ether layer was washed with saturated NaCl solution and dried by anhydrous magnesium sulfate. The solvent was evaporated and a colorless oil (55.2 mg, 85% mol yield) was obtained. ^{1}H-NMR (400 MHz, $CDCl_3$) δ 11.17 (s, 1H), 2.35 (t, *J* = 7.5 Hz, 1H), 1.63 (dt, *J* = 15.1, 7.5 Hz, 1H), 1.39 – 1.24 (m, 2H), 0.89 (t, *J* = 6.8 Hz, 1H). ^{13}C-NMR (101 MHz, $CDCl_3$) δ 180.49, 34.12, 31.43, 28.73, 24.63, 22.46, 14.01.

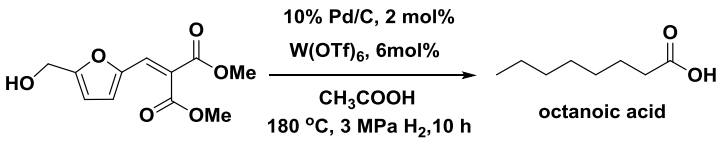
2.7 Synthesis of substrate and product in Table 2 Entry 6

Synthesis of dimethyl 2-((5-(hydroxymethyl)furan-2-yl)methylene)malonate [5]



340 μL (4.2 mmol) of pyrrolidine and 240 μL of acetic acid (4.2 mmol) were sequentially added to an iced solution containing of 2.8 g (20.8 mmol) dimethyl malonate and 2.6 g of 5-hydroxymethylfurfural (20.8 mmol) in a Schlenk tube. The resultant solution was allowed to stay at ambient temperature and stir until the reaction was completed by TLC. The resultant solution was washed with petroleum ether several times. Then the residue were added by ethyl acetate and purified by a short silica column. The solvent was evaporated and a reddish cast oil (4.2 g, 85% yield) was obtained. ^{1}H-NMR (400 MHz, $CDCl_3$): δ = 7.43 (s, 1H), 6.72 (d, *J* = 3.4 Hz, 1H), 6.39 (d, *J* = 3.4 Hz, 1H), 4.59 (s, 2H), 3.90 (s, 3H), 3.82 (s, 3H). ^{13}C-NMR (101 MHz, $CDCl_3$): δ = 167.08, 164.55, 158.46, 148.42, 128.21, 120.85, 119.39, 110.35, 57.47, 52.76, 52.65.

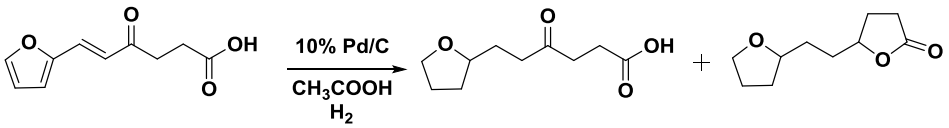
Synthesis of octanoic acid from dimethyl 2-((5-(hydroxymethyl)furan-2-yl) methylene) malonate



The catalytic conversion of **dimethyl 2-((5-(hydroxymethyl)furan-2-yl) methylene) malonate** to **octanoic acid** was carried out in a 25 mL stainless steel high pressure parr reactor equipped with a magnetic stirrer, a temperature probe and a temperature programmed controller. In a typical experiment, 0.5 mmol of **dimethyl 2-((5-(hydroxymethyl)furan-2-yl) methylene) malonate**, 2 mol% of 10% Pd/C catalyst, 6 mol% of $W(OTf)_6$, and 5 mL of

acetic acid were added to the parr reactor. Tightened the screws, replaced the gas with hydrogen three times and pressurized the hydrogen to 3 MPa at room temperature. Heating the reactor from room temperature to 180 °C in a period of 30 min under 500 rpm/min magnetic stirring. Then cooling and relief pressure after holding reaction at 180 °C for 10 h. Removal of acetic acid by vacuum distillation after filtrating Pd/C catalyst. Then water was added to residue and the solution was extracted with ether several times. The ether layer was washed with saturated NaCl solution and dried by anhydrous magnesium sulfate. The solvent was evaporated and a colorless oil (57.7 mg, 80% mol yield) was obtained. ^{1}H-NMR (400 MHz, $CDCl_3$): δ = 10.70 (s, 1H), 2.35 (t, *J* = 7.4 Hz, 2H), 1.62 (dd, *J* = 13.8, 6.9 Hz, 2H), 1.46 – 1.15 (m, 8H), 0.88 (t, *J* = 6.6 Hz, 3H). ^{13}C-NMR (101 MHz, $CDCl_3$): δ = 179.32, 33.11, 30.62, 28.00, 27.89, 23.67, 21.58, 13.04.

2.8 Synthesis of intermediate 1 and intermediate 2



δ- furfurylidenelevulinic acid Intermediate 1 Intermediate 2

200 mg δ-furfurylidenelevulinic acid, 20 mg 10% Pd/C were added to a solution of 3mL acetic acid, then the reaction was carried out at 1 atm H_2, 40 ^{o}C, and 4 h. After reaction, the solution was cooled down and the Pd/C catalyst was filtrated. Removed acidic acid under vacuum evaporation. Then the residue was dissolved with dichloromethane and extraction separation using sodium carbonate solution. The organic layer was dried over sodium sulfate and evaporated in vacuo to give intermediate **2 (5-(2-(tetrahydrofuran-2-yl) ethyl) dihydrofuran-2(3H)-one).** ^{1}H-NMR (400 MHz, $CDCl_3$): δ = 4.63 – 4.47 (m, 1H), 3.89 – 3.80 (m, 2H), 3.72 (dd, *J* = 14.4, 7.8 Hz, 1H), 2.53 (dt, *J* = 23.8, 11.9 Hz, 2H), 2.34 (tt, *J* = 12.3, 6.1 Hz, 1H), 2.00 (ddd, *J* = 6.7, 3.1, 1.5 Hz, 1H), 1.92 – 1.81 (m, 4H), 1.77 – 1.64 (m, 2H), 1.63 – 1.56 (m, 1H), 1.51 – 1.43 (m, 1H).The aqueous layer was acidified with concentrated hydrochloric acid and extracted with dichloromethane. The organic layer was dried over sodium sulfate and evaporated in vacuo to give intermediate **1 (4-oxo-6-(tetrahydrofuran -2-yl) hexanoic acid)**. ^{1}H-NMR (400 MHz, $CDCl_3$): δ = 3.87 – 3.78 (m, 2H), 3.71 (dt, *J* = 14.4, 7.3 Hz, 1H), 2.80 – 2.68 (m, 2H), 2.66 – 2.42 (m, 4H), 2.07 – 1.94 (m, 2H), 1.93 – 1.86 (m, 2H), 1.86 – 1.82 (m, 1H), 1.74 (dtd, *J* = 14.2, 8.4, 6.1 Hz, 1H). ^{13}C-NMR (101 MHz, $CDCl_3$): δ = 208.66, 177.63, 78.33, 67.64, 39.35, 36.92, 31.27, 29.39, 27.74, 25.67.

Ⅲ. Further screening of reaction conditions

1. Hydrogen pressure

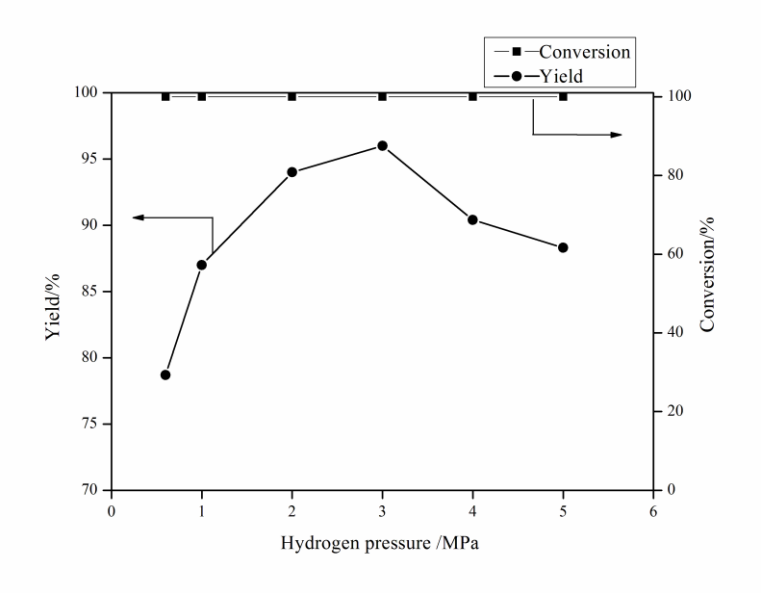


Figure S1. The effect of hydrogen pressure on product distribution. Reaction conditions: 0.5 mmol substrate, 2 mol% 10% Pd/C, 6 mol% $W(OTf)_6$, 5 mL acetic acid, 180 ºC, 8 h.

The effect of hydrogen pressure on product distribution was carried out. As we have found in our previous experiments, the consumption of about 0.6 MPa H_2 are needed to completely convert the substrate under our reaction conditions and reactor. Therefore, we firstly carried out hydrogen conditions at 0.6 MPa H_2. We found that the raw materials converted completely and 78.7% yield of decanoic acid was obtained. The results showed that a better yield can be obtained under relatively lower hydrogen pressure. The yield of decanoic acid increased firstly and then decreased along with the increased hydrogen pressure. The highest yield of decanoic acid (96%) was obtained at 3 MPa H_2. The hydrogen pressure had a obvious effect on hydrogenation reaction. The increase of hydrogen pressure favors the adsorption and resolution of Pd/C on hydrogen. It was also found that acetic acid had a significant effect on product distribution. Acetic acid blocks the growth of polymer carbon chains by esterification with 1-hydroxy-10-decanoic acid, which facilitates the mass transfer process of the reaction. [6] The yield of decanoic acid declined after further increasing the pressure. This may be due to the further conversion of intermediates and products to the excessive hydrogenation by-products under higher pressure conditions. [7]

2. Reaction temperature

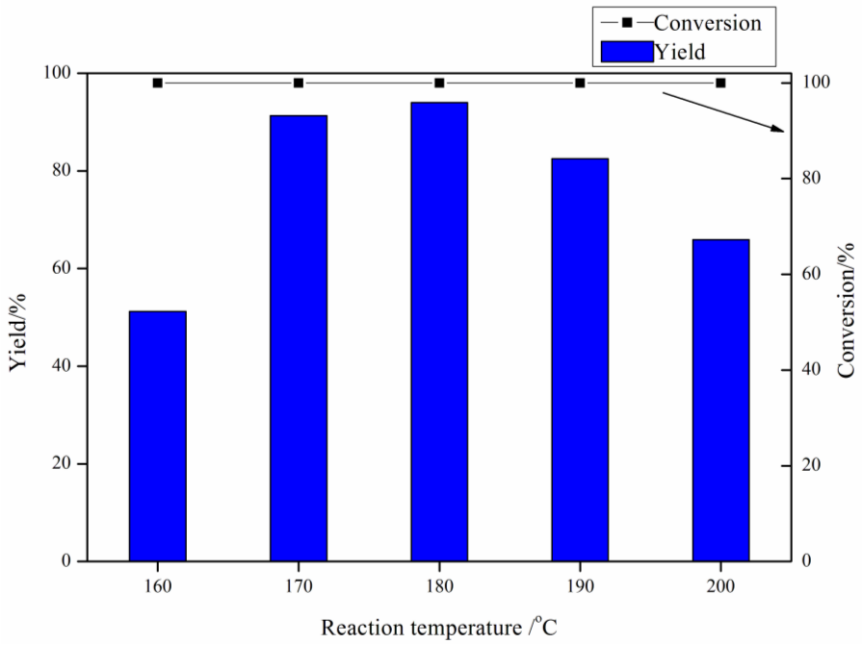


Figure S2. The effect of reaction temperature on product distribution. Reaction conditions: 0.5 mmol substrate, 2 mol% 10% Pd/C, 6 mol% $W(OTf)_6$, 5 mL acetic acid, 2 MPa H_2, 8 h.

Under all reaction conditions, the starting material was completely converted. This is mainly due to the fact that the raw materials can be quickly converted into intermediate **1** and intermediate **2** under room temperature and atmospheric pressure by Pd/C catalysis. At the lower temperature, the exocyclic double bond, furan-ring double bond and carbonyl group on raw material are more prone to be converted into intermediate **1** and intermediate **2** under hydrogen conditions. With the increase of reaction temperature, the yield of decanoic acid increased firstly and then decreased. The hydrogenolysis of ester bond is greatly affected by reaction temperature. The straight chain esters were formed in the reaction according to the ^{1}H-NMR and ESI analysis (**SI: "Ⅳ.Mass spectrometry data; Ⅴ. NMR spectrum data"**). Gordon *et al.* also pointed out that it is difficult to break the C-O bond belonged to the 1° esters. [6] There are still more than 10% ester products were obtained under the reaction conditions of 200 °C, 200 psi H_2 and 14 h. [6] The breaking rate of the straight chain ester bond is slower at lower reaction temperatures. Accompanied by increase of reaction temperature, the hydrogenolysis of fatty ester bond catalyzed by metal triflate was accelerated and the yield of decanoic acid increased gradually. The highest yield (94%) was obtained at 180 °C. The yield of decanoic acid decreased with the further increasing of temperature. This may be caused by the reduction of some carboxyl compounds. We further hypothesized that an excessively high reaction temperature would result in a reduced selectivity of oligomerization and deoligomerization reactions.

3. The catalyst circulation experiment of Pd/C and $W(OTf)_6$ co-catalyst system

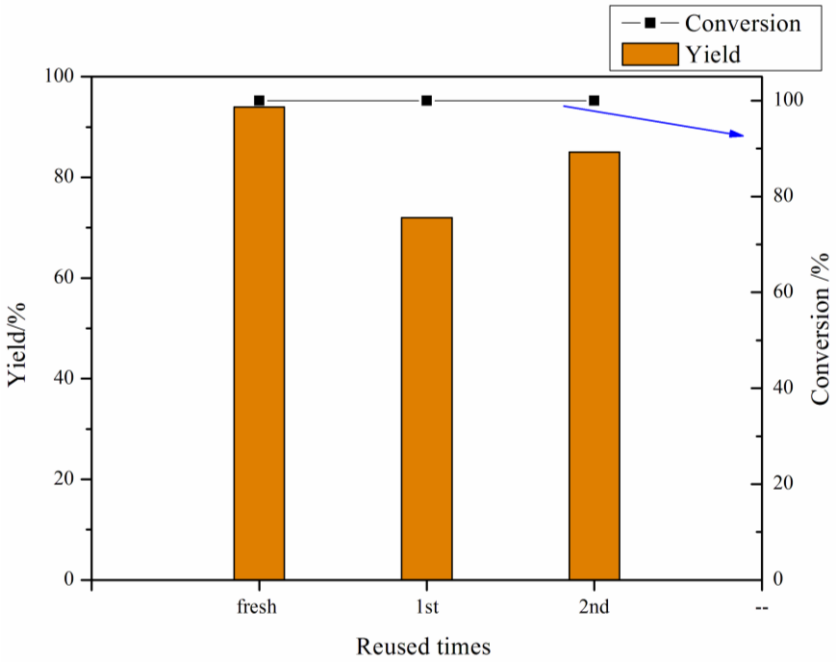


Figure S3. The catalyst circulation experiment of Pd/C and $W(OTf)_6$ co-catalyst system. Reaction conditions: 0.5 mmol substrate, 2 mol% 10% Pd/C, 6 mol% $W(OTf)_6$, 5 mL acetic acid, 180 °C, 2 MPa H_2, 8 h.

The Pd/C and $W(OTf)_6$ co-catalyst system has a good effect on hydrodeoxygenation reaction to obtain medium-chain fatty acid. After the reaction under standard conditions, the Pd/C catalyst was separated by centrifugation. Then washed with ethyl acetate, centrifuged, and left after drying. The GC yield of product was measured followed by adding a certain amount of internal standard dimethyl phthalate in the solution. Remove the solvent under vacuum and the residue was added with ether. The ether layer was discarded, and the small amount of catalyst layer was washed several times with ether and then removal of ether by evaporation. The residual catalyst layer in the bottle was transferred to the autoclave with acetic acid and the recycled Pd/C catalyst was added subsequently. Repeated experiments under standard conditions. 94% yield of decanoic acid was obtained by using the fresh catalyst system. The declined yield of decanoic acid was detected after used once time. Owing to the $W(OTf)_6$ catalyst has a significant catalytic effect on ring-opening reaction and fracture ester reaction. This may be caused by the loss of the $W(OTf)_6$ catalyst in the treatment procedure. In order to verify this conjecture, we added 6 mol% $W(OTf)_6$ to the reaction and found the improved yield of decanoic acid. Better recycling strategies are now being addressed. Appropriate W-based heterogeneous catalysts for the green production of saturated fatty acids from cellulose-carbohydrate feedstock is being explored.

4. The effect of substrate concentration on the yield of decanoic acid

Table S2. The effect of substrate concentration on the yield of decanoic acid. [a]

Entry	Amount of substrate/ mmol	Conversion/%	Yield/% [b]
1	0.5	>99	96.4
2	1	>99	95.7
3	2	>99	92.3
4	5	>99	83.6
5	5	>99	90.7 [c]

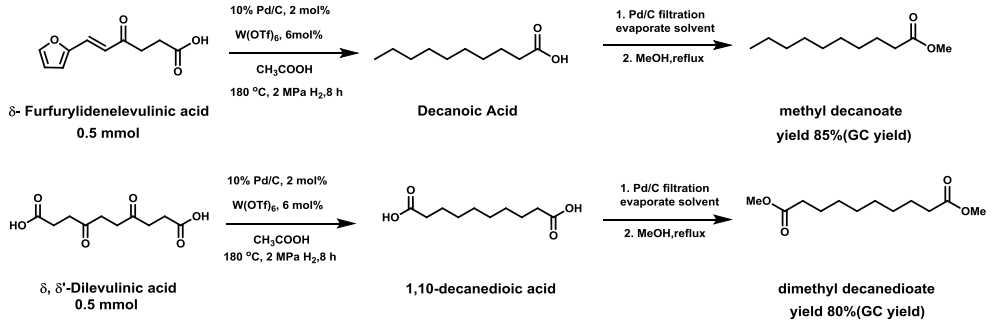
[a] Reaction conditions: substrate is δ-furfurylidenelevulinic acid, 2 mol% 10% Pd/C, 6 mol% $W(OTf)_6$, 180 °C, 3 MPa H_2, 10 h, 5 mL solvent. [b] isolated yield. [c] 16 h.

From the data in table S2, the product yield decreased from 96.4% to 83.6% as the amount of substrate increased from 0.5 mmol to 5 mmol. It is indicated that the increase of substrate concentration is unfavorable to the production of decanoic acid under the same reaction conditions (Table S2, Entry 1-4). This may be due to the increase of substrate concentration may require a longer reaction time. As showed in Figure 2a in the manuscript, the yield of decanoic acid was increased with extended reaction time, but the formation rate was decreased. Based on this, we extended the reaction time to 16 h and found that the yield of decanoic acid was increased (Table S2, Entry 5). The longer reaction time is not suitable for the concept of green development, so an appropriate substrate concentration is necessary.

5. The experimental operation for the recycling of Pd/C catalyst.

After the reaction under standard conditions, the reaction solution was transformed by ethyl acetate. Then the Pd/C catalyst was separated by centrifugation, washed with acetic acid, centrifuged, and left for the next cycle. The GC yield of product was measured followed by adding a certain amount of internal standard dimethyl phthalate in the solution. The recycled Pd/C catalyst was added to the next recycle and repeated experiments under standard conditions.

6. The reaction application



Take into account of the excellent ability of catalytic esterification of metal triflate as well as the important application of medium-chain carboxylic acid esters, a one-pot two-steps process has been built for synthesizing medium-chain fatty acid esters from biomass substrates. Firstly, we obtained saturated fatty acid by using aldol condensation products as the substrate under the hydrodeoxygenation reaction conditions. Subsequently, filtered Pd/C catalyst, removed acetic acid solvent, retented metal triflate catalyst and added methanol. Metal triflate has a good catalytic effect on the esterification reaction due to its strong Lewis acidity. Heating and refluxing, and finally the medium chain carboxylic acid ester was

obtained.

7. The reaction application

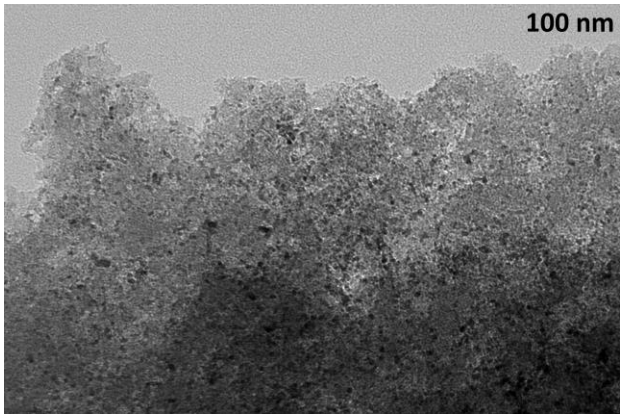


Figure S4. The TEM graph of fresh Pd/C catalysts.

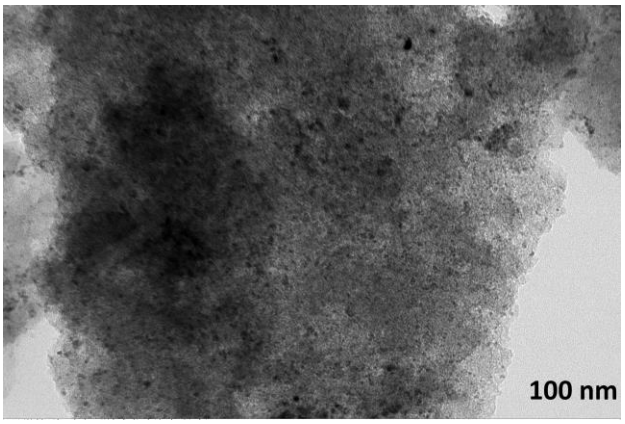


Figure S5. The TEM graph of used Pd/C catalysts.

Ⅳ. Mass spectrometry data

ESI-MS (Electrospray ionization mass spectrometry) analysis of Oligomeric Products: All samples prepared for ESI-MS were obtained from reactions.

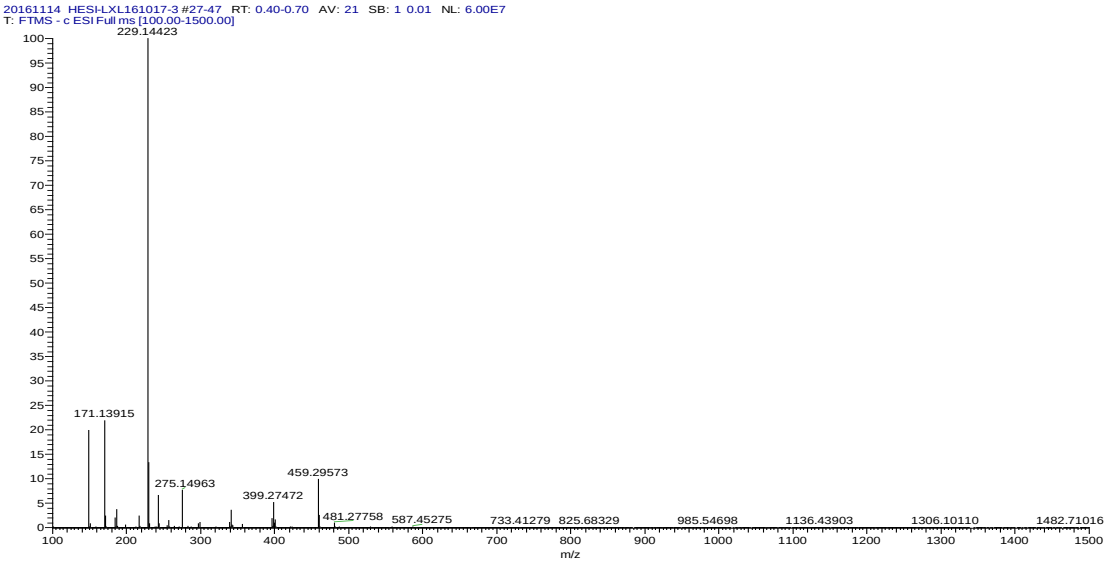


Figure S4. The ESI spectra of reaction solution heated for 30 min and continued reaction for 20 min

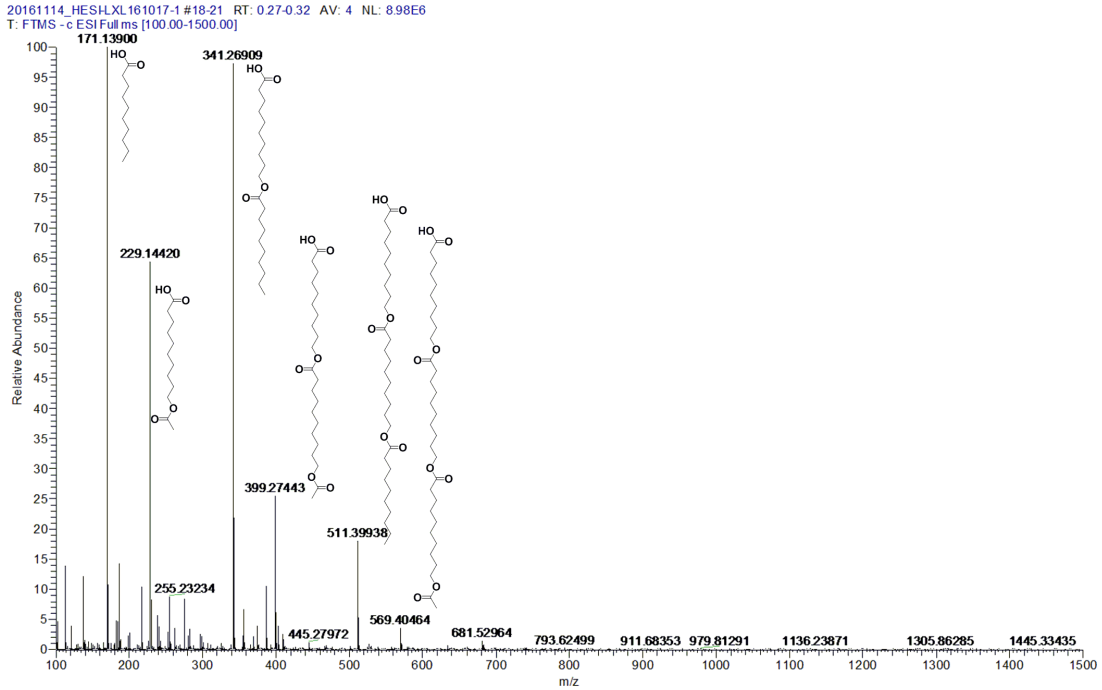


Figure S5. The ESI spectra of reaction solution heated for 30 min and continued reaction for 1 h

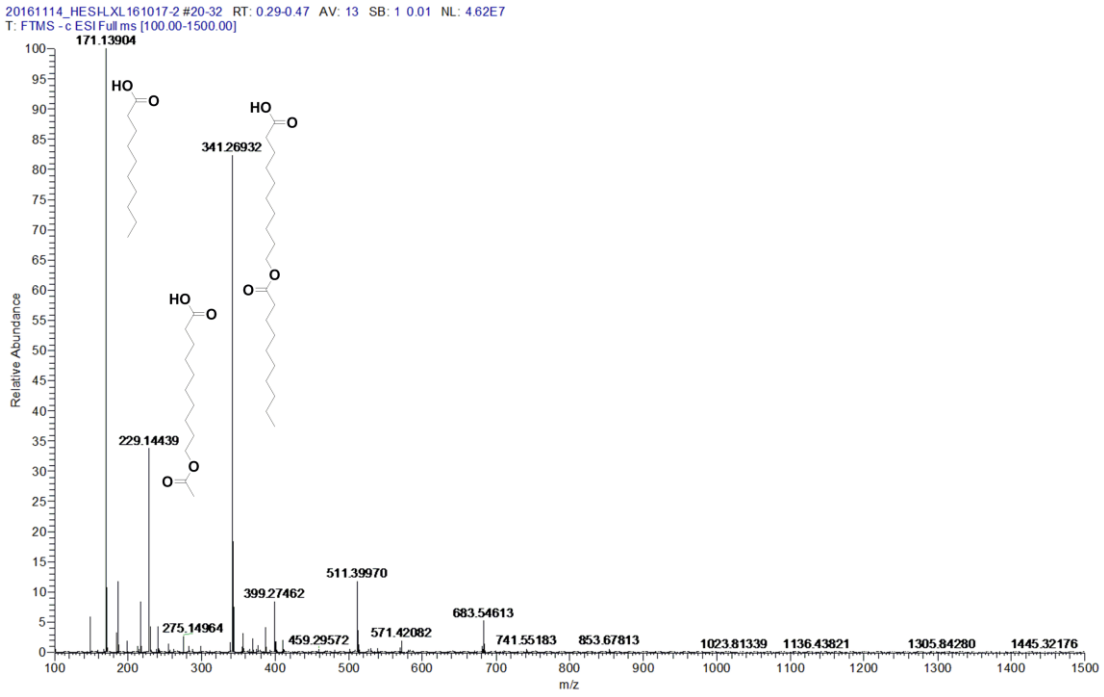


Figure S6. The ESI spectra of reaction solution heated for 30 min and continued reaction for 4 h

Ⅴ. NMR spectrum data

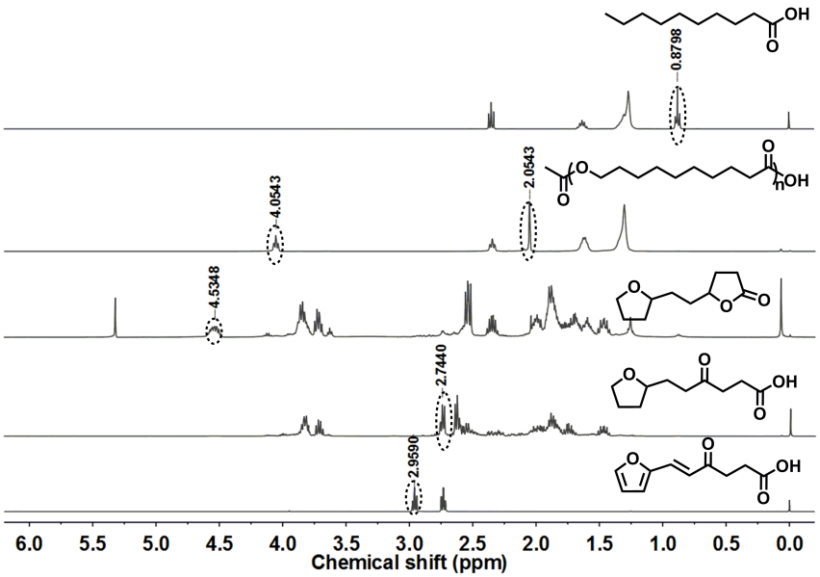


Figure S7. The ^{1}H-NMR spectrum comparison of intermediates and products.

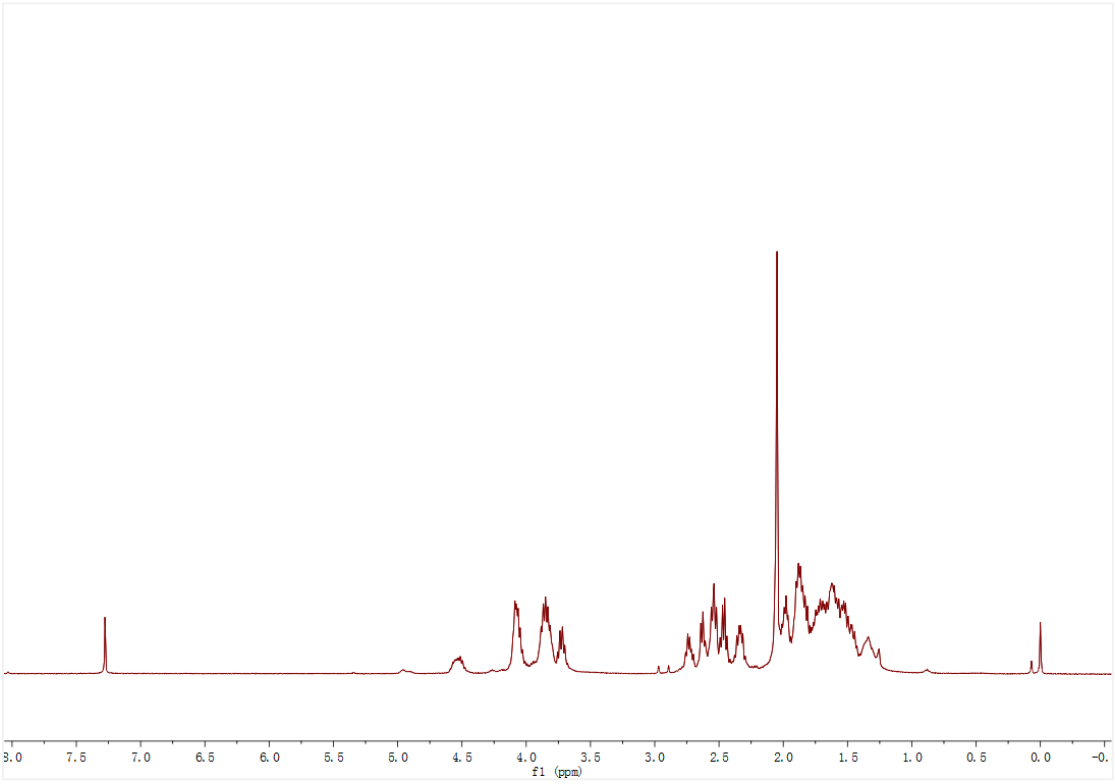


Figure S8. The ^{1}H-NMR spectrum of reaction solution heated for 10 min.

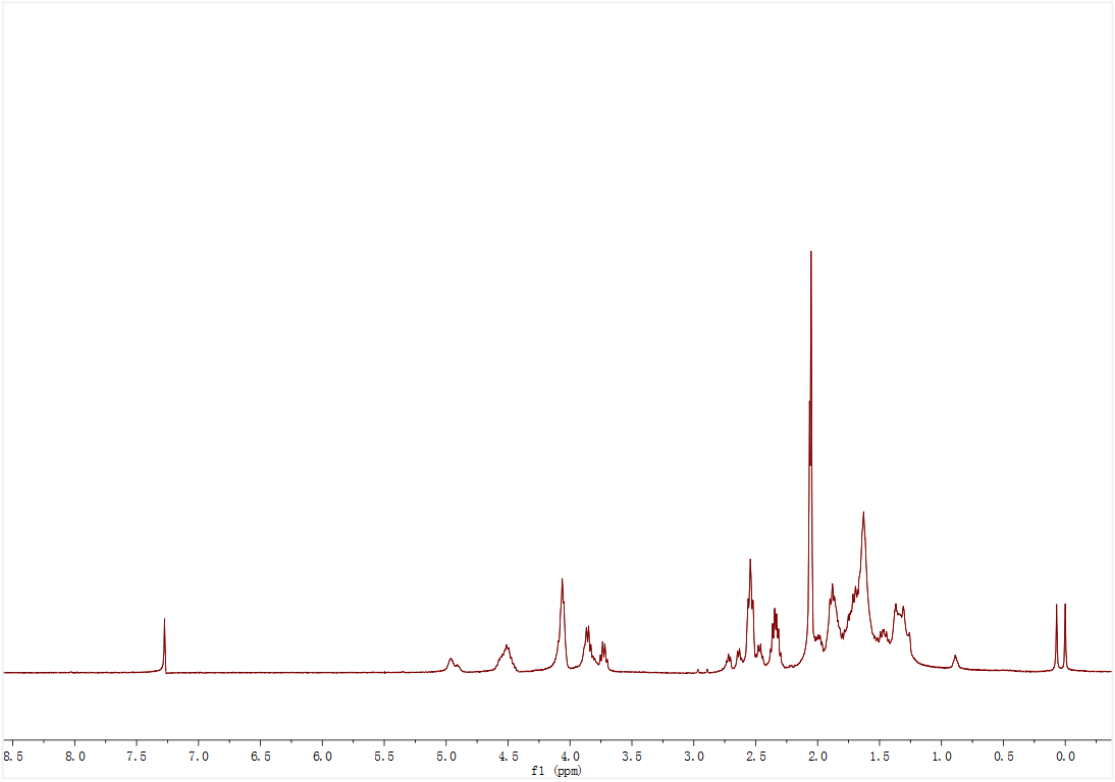


Figure S9. The ^{1}H-NMR spectrum of reaction solution heated for 20 min

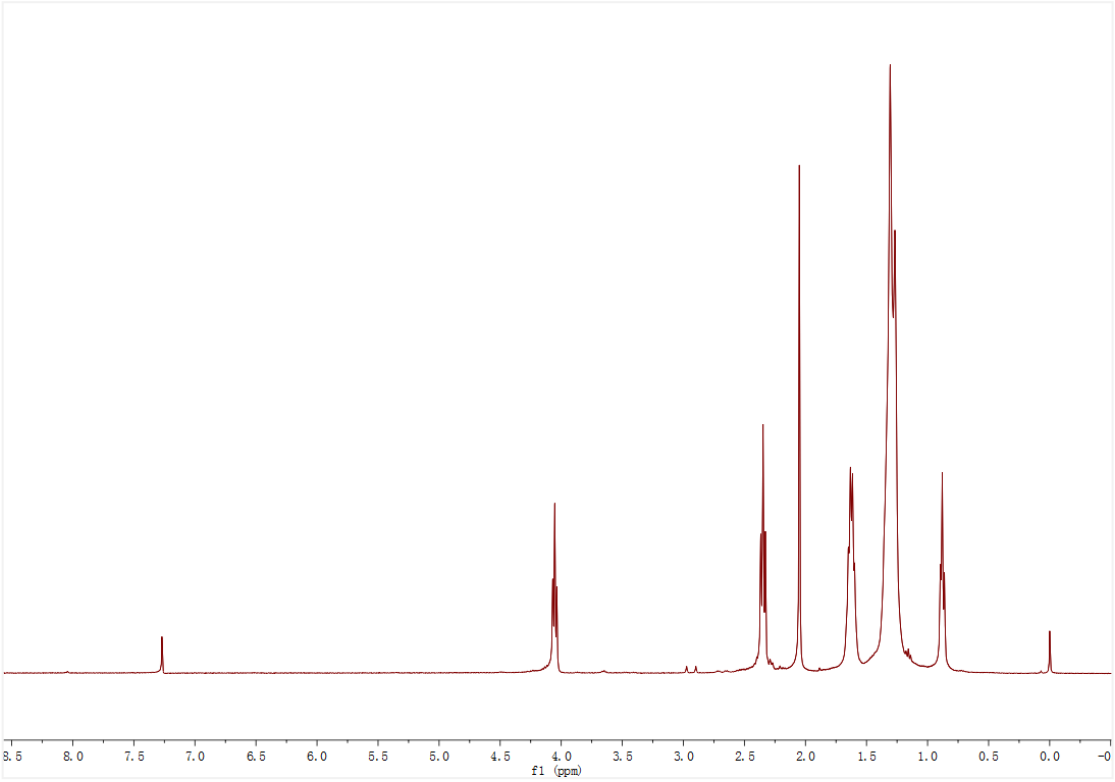


Figure S10. The ^{1}H-NMR spectrum of reaction solution heated for 30 min

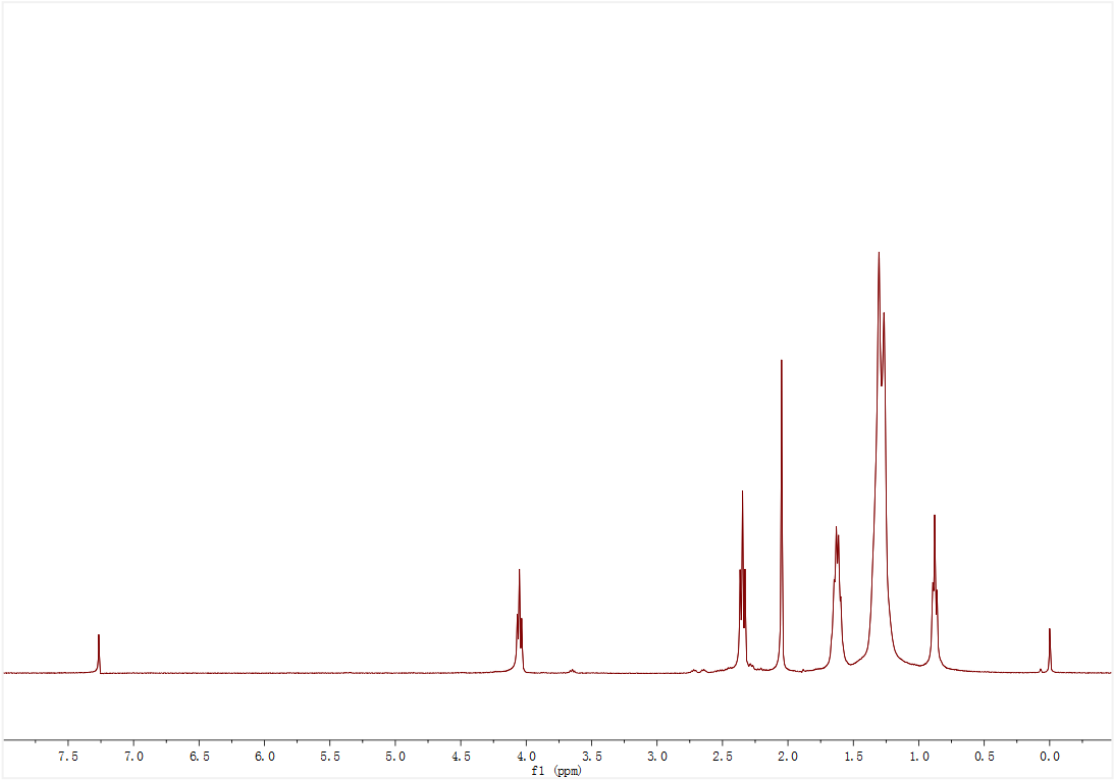


Figure S11. The ^{1}H-NMR spectrum of reaction solution heated for 30 min and continued reaction for 1h

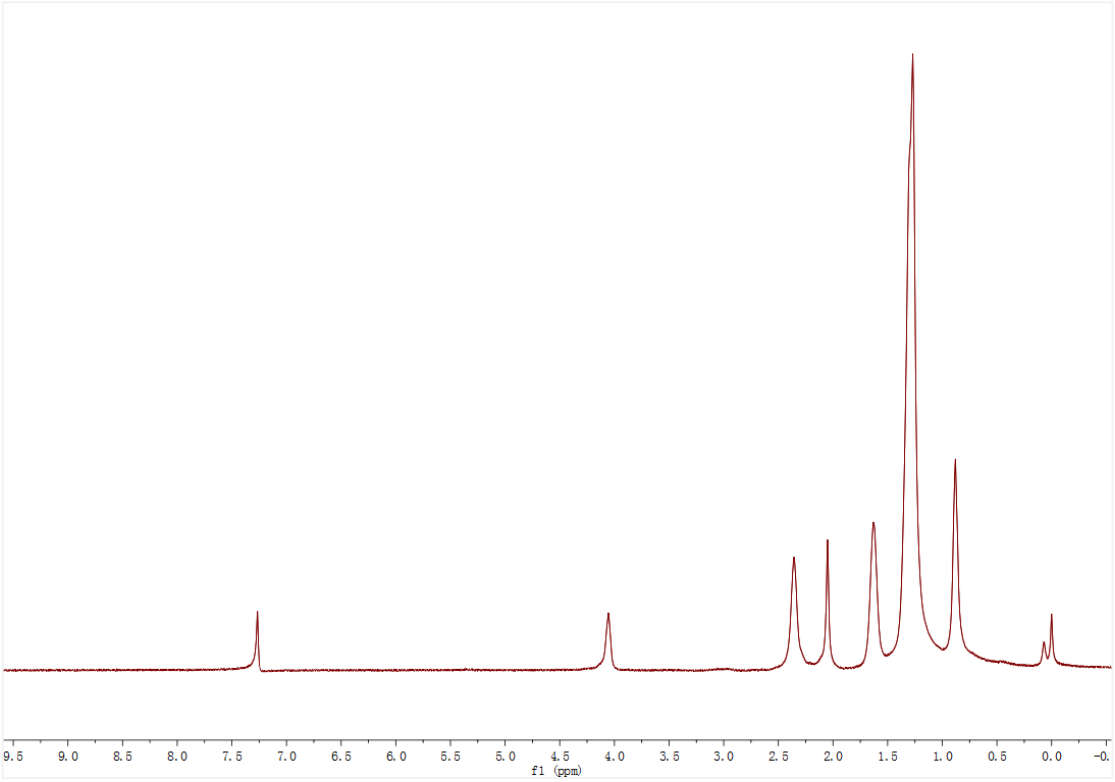


Figure S12. The ^{1}H-NMR spectrum of reaction solution heated for 30 min and continued reaction for 2 h

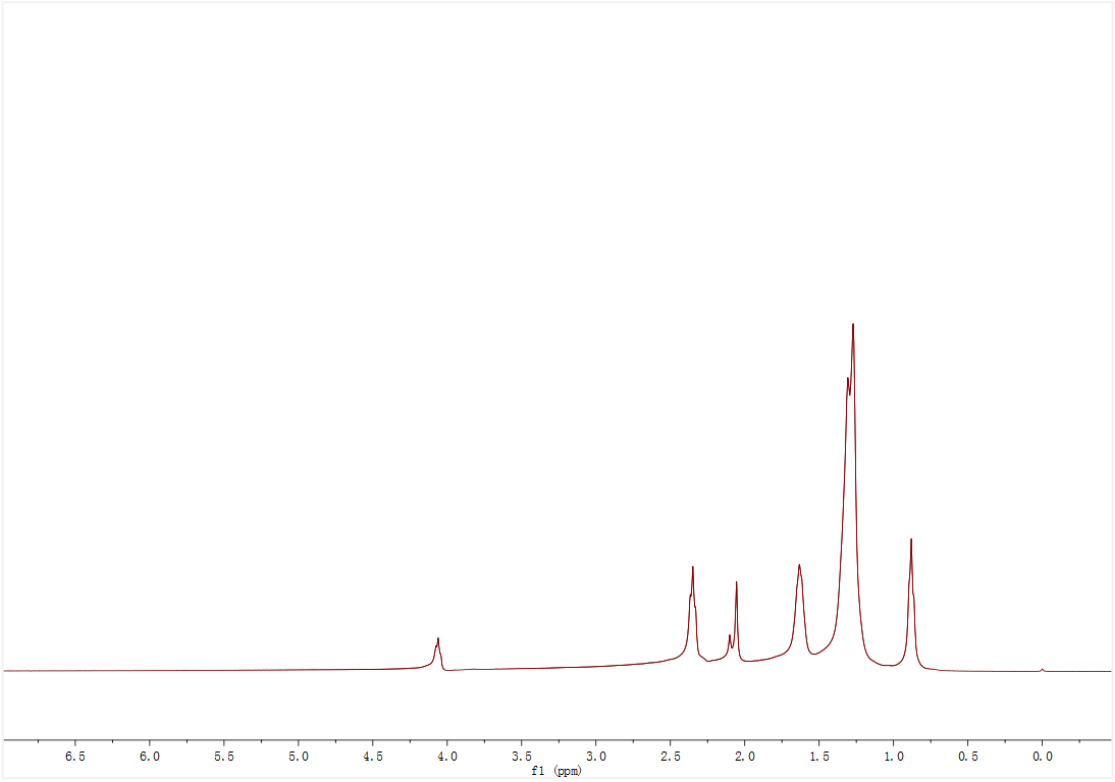


Figure S13. The ^{1}H-NMR spectrum of reaction solution heated for 30 min and continued reaction for 4 h

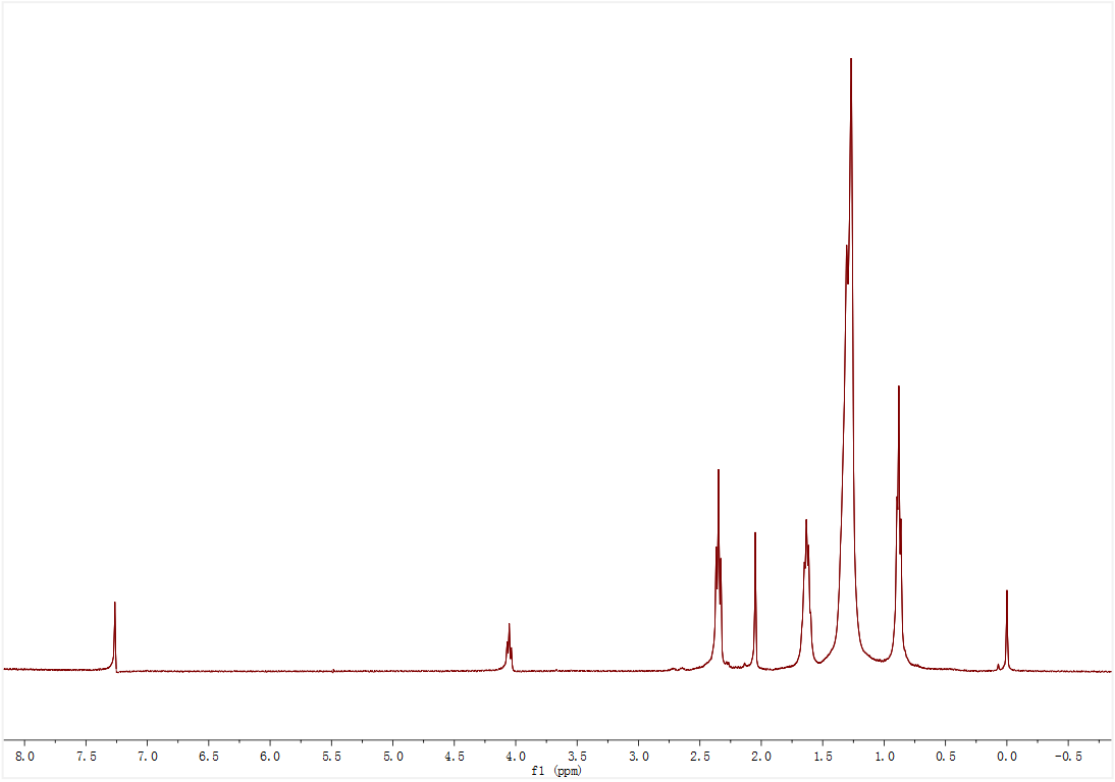


Figure S14. The ^{1}H-NMR spectrum of reaction solution heated for 30 min and continued reaction for 6 h

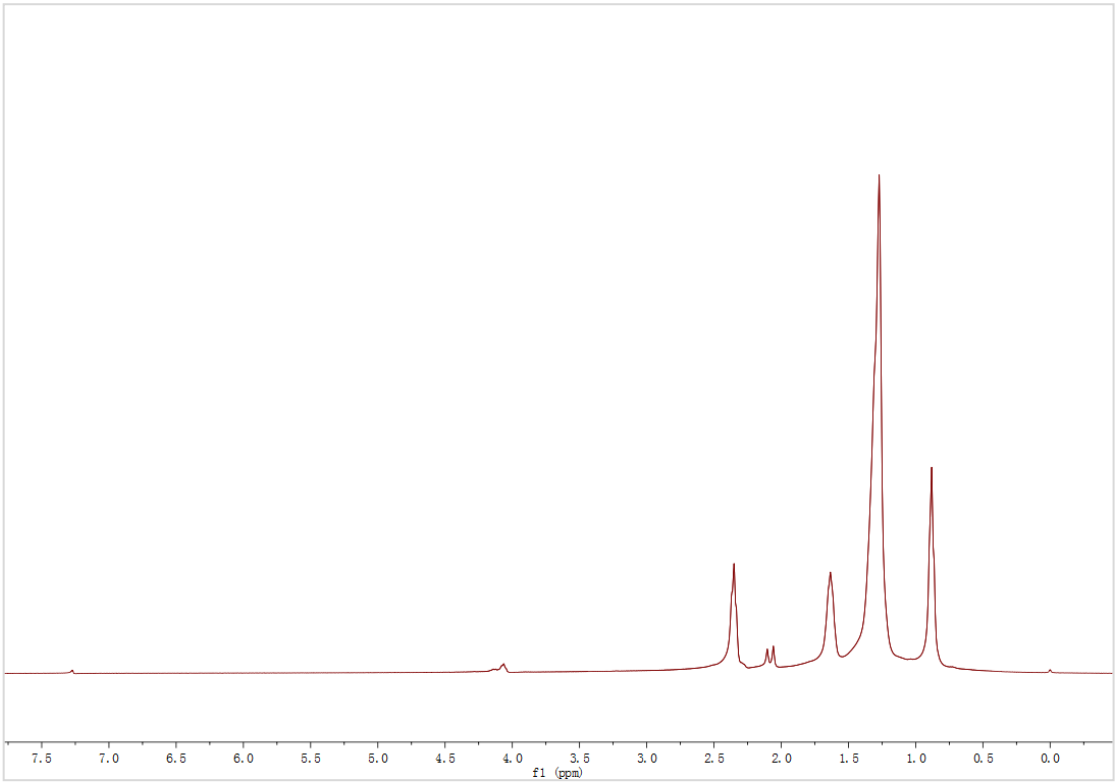


Figure S15. The ^{1}H-NMR spectrum of reaction solution heated for 30 min and continued reaction for 8 h

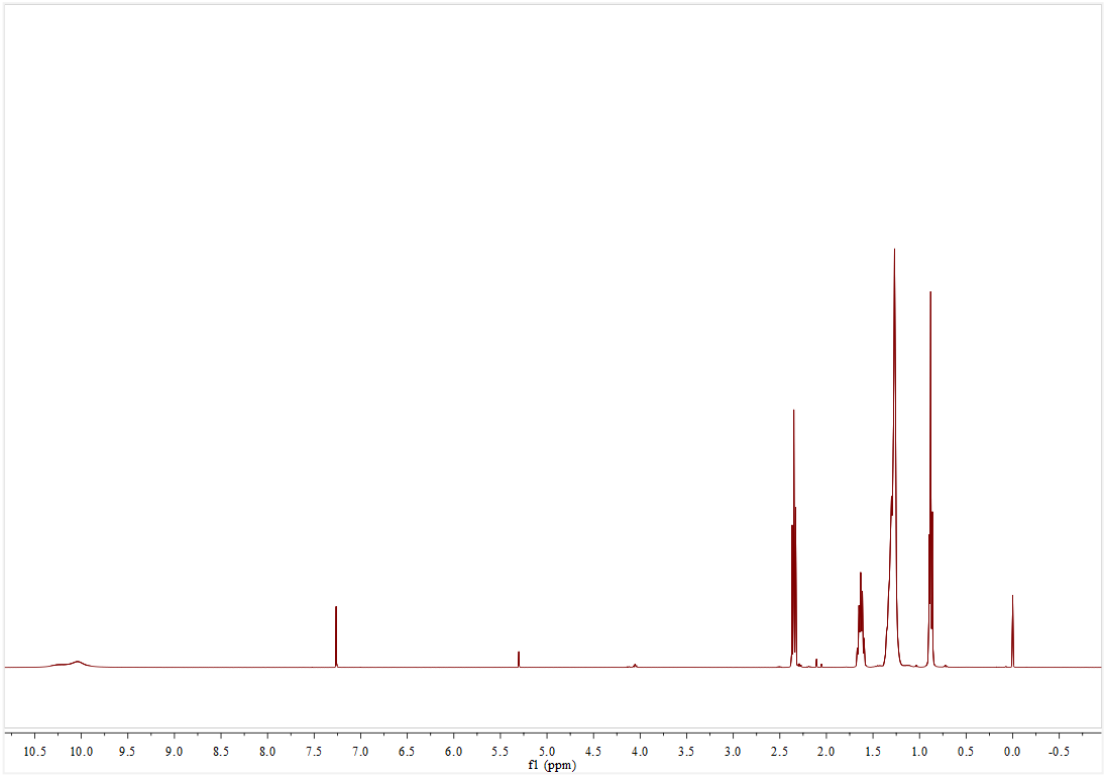
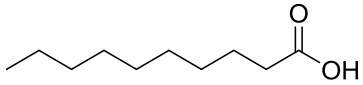


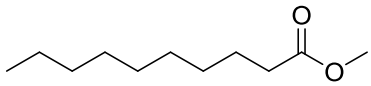
Figure S16. The ^{1}H-NMR spectrum of reaction solution heated for 30 min and continued reaction for 10 h

Decanoic acid:



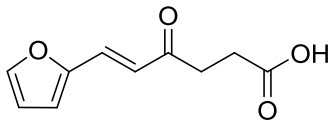
^{1}H NMR (400 MHz, $CDCl_3$): δ = 9.25 (s, 1H), 2.35 (t, *J* = 7.5 Hz, 2H), 1.69 - 1.58 (m, 2H), 1.31 (dd, *J* = 21.8, 10.9 Hz, 12H), 0.88 (t, *J* = 6.9 Hz, 3H).
^{13}C NMR (101 MHz, $CDCl_3$): δ = 180.36, 34.10, 31.86, 29.40, 29.26, 29.06, 24.68, 22.67, 14.11.

Methyl decanoate:



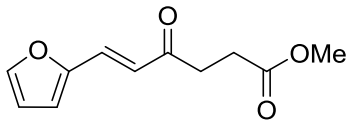
^{1}H NMR (400 MHz, $CDCl_3$): δ = 9.25 (s, 1H), 2.35 (t, *J* = 7.5 Hz, 2H), 1.69 - 1.58 (m, 2H), 1.31 (dd, *J* = 21.8, 10.9 Hz, 12H), 0.88 (t, *J* = 6.9 Hz, 3H).
^{13}C NMR (101 MHz, $CDCl_3$): δ = 174.36, 51.44, 34.12, 31.86, 29.41, 29.26, 29.16, 24.97, 22.67, 14.10.

δ-Furfurylidenelevulinic acid:



^{1}H NMR (400 MHz, $CDCl_3$): δ = 10.01 (s, 1H), 7.51 (d, *J* = 1.5 Hz, 1H), 7.36 (d, *J* = 15.8 Hz, 1H), 6.76 - 6.61 (m, 2H), 6.49 (dd, *J* = 3.4, 1.8 Hz, 1H), 2.96 (t, *J* = 6.6 Hz, 2H), 2.73 (t, *J* = 6.6 Hz, 2H).
^{13}C NMR (101 MHz, $CDCl_3$): δ = 197.44, 178.42, 150.90, 145.13, 129.20, 122.73, 116.14, 112.62, 35.44, 28.00.

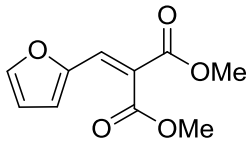
δ-Furfurylidenelevulinic acid methyl ester:



^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.51 (s, 1H), 7.38 – 7.32 (m, 1H), 6.66 (dd, *J* = 12.5, 9.7 Hz, 2H), 6.49 (dd, *J* = 3.3, 1.8 Hz, 1H), 3.68 (s, 3H), 2.96 (t, *J* = 6.7 Hz, 2H), 2.67 (t, *J* = 6.7 Hz, 2H).

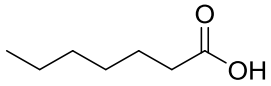
^{13}C NMR (101 MHz, $CDCl_3$): δ = 197.42, 173.19, 150.85, 145.03, 128.85, 122.86, 115.89, 112.54, 51.65, 35.49, 27.86.

Dimethyl 2-(furan-2-ylmethylene)malonate:



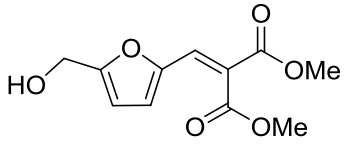
^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.53 (d, *J* = 1.7 Hz, 1H), 7.49 (s, 1H), 6.78 (d, *J* = 3.5 Hz, 1H), 6.50 (dd, *J* = 3.5, 1.8 Hz, 1H), 3.92 (s, 3H), 3.83 (s, 3H).
^{13}C NMR (101 MHz, $CDCl_3$): δ = 166.75, 164.53, 148.85, 146.40, 128.19, 121.13, 118.34, 112.68, 52.60, 52.56.

Heptanoic acid:



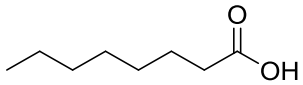
^{1}H NMR (400 MHz, $CDCl_3$): δ = 11.17 (s, 1H), 2.35 (t, *J* = 7.5 Hz, 2H), 1.63 (dt, *J* = 15.1, 7.5 Hz, 2H), 1.37 – 1.26 (m, 6H), 0.89 (t, *J* = 6.8 Hz, 3H).
^{13}C NMR (101 MHz, $CDCl_3$): δ = 180.49, 34.12, 31.43, 28.73, 24.63, 22.46, 14.01.

Dimethyl 2-((5-(hydroxymethyl) furan-2-yl)methylene)malonate:



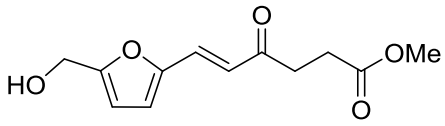
^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.43 (s, 1H), 6.72 (d, *J* = 3.4 Hz, 1H), 6.39 (d, *J* = 3.4 Hz, 1H), 4.59 (s, 2H), 3.90 (s, 3H), 3.82 (s, 3H).
^{13}C NMR (101 MHz, $CDCl_3$): δ = 167.08, 164.55, 158.46, 148.42, 128.21, 120.85, 119.39, 110.35, 57.47, 52.76, 52.65.

Octanoic acid:



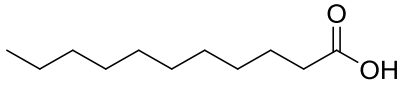
^{1}H NMR (400 MHz, $CDCl_3$): δ = 10.70 (s, 1H), 2.35 (t, *J* = 7.4 Hz, 2H), 1.62 (dd, *J* = 13.8, 6.9 Hz, 2H), 1.46 – 1.15 (m, 8H), 0.88 (t, *J* = 6.6 Hz, 3H).
^{13}C NMR (101 MHz, $CDCl_3$): δ = 179.32, 33.11, 30.62, 28.00, 27.89, 23.67, 21.58, 13.04.

Methyl (E)-6-(5-(hydroxymethyl) furan-2-yl)-4-oxohex-5-enoate:



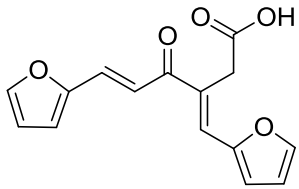
^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.31 (d, *J* = 15.9 Hz, 1H), 6.64 (dd, *J* = 12.7, 9.6 Hz, 2H), 6.39 (d, *J* = 3.2 Hz, 1H), 4.65 (s, 2H), 3.69 (s, 3H), 2.94 (t, *J* = 6.7 Hz, 2H), 2.68 (t, *J* = 6.6 Hz, 2H), 2.10 (s, 1H).
^{13}C NMR (101 MHz, $CDCl_3$): δ = 197.50, 173.36, 156.76, 150.88, 128.78, 122.84, 116.88, 110.52, 57.67, 51.85, 35.84, 27.99.

Undecanoic acid:



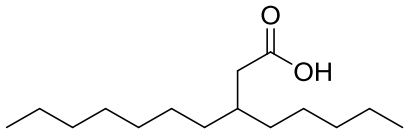
^{1}H NMR (400 MHz, $CDCl_3$): δ = 2.34 (t, *J* = 7.4 Hz, 2H), 1.66 - 1.60 (m, 2H), 1.28 (d, *J* = 15.0 Hz, 14H), 0.88 (t, *J* = 6.7 Hz, 3H).
^{13}C NMR (101 MHz, $CDCl_3$): δ = 180.42, 34.13, 31.90, 29.56, 29.45, 29.32, 29.26, 29.07, 24.69, 22.69, 14.12.

Methyl (E)-6-(5-(hydroxymethyl) furan-2-yl)-4-oxohex-5-enoate:



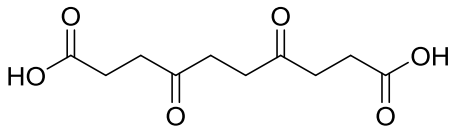
^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.63 (s, 1H), 7.52 (dd, *J* = 13.9, 5.3 Hz, 3H), 7.31 (d, *J* = 15.2 Hz, 1H), 6.91 (d, *J* = 2.9 Hz, 1H), 6.72 (d, *J* = 3.2 Hz, 1H), 6.54 (ddd, *J* = 17.0, 3.2, 1.7 Hz, 2H), 3.91 (s, 2H).
^{13}C NMR (101 MHz, $CDCl_3$): δ = 191.23, 174.45, 151.57, 150.51, 150.27, 145.94, 145.03, 130.83, 130.13, 128.25, 118.00, 117.67, 116.49, 112.77, 33.68.

3-pentyldecanoic acid:



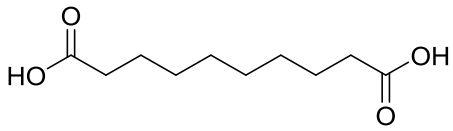
^{1}H NMR (400 MHz, $CDCl_3$): δ = 10.98 (s, 1H), 2.27 (d, *J* = 6.8 Hz, 1H), 2.05 (d, *J* = 2.9 Hz, 1H), 1.59 (dd, *J* = 13.5, 6.6 Hz, 1H), 1.27 (s, 20H), 0.88 (t, *J* = 6.7 Hz, 6H).
^{13}C NMR (101 MHz, $CDCl_3$): δ = 179.75, 38.96, 34.87, 33.78, 33.74, 32.06, 31.87, 29.82, 29.28, 26.51, 26.17, 22.68, 22.63, 14.11, 14.07.

δ,δ'-Dilevulinic acid:



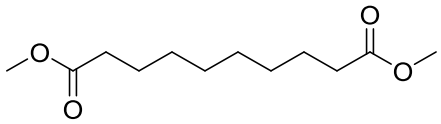
^{1}H NMR (400 MHz, DMSO): δ = 12.10 (s, 2H), 2.66 (dd, *J* = 11.4, 4.6 Hz, 8H), 2.38 (t, *J* = 6.5 Hz, 4H).
^{13}C NMR (101 MHz, DMSO): δ = 208.30, 174.17, 37.04, 36.06, 28.12.

Sebacic acid:



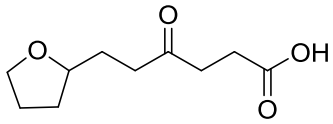
^{1}H NMR (400 MHz, DMSO): δ = 11.98 (s, 2H), 2.18 (t, *J* = 7.4 Hz, 4H), 1.55 - 1.40 (m, 4H), 1.25 (s, 8H).
^{13}C NMR (101 MHz, DMSO): δ = 174.97, 34.12, 29.03 (d, *J* = 11.7 Hz), 24.94.

Dimethyl sebacate:



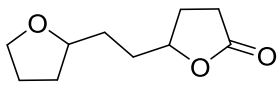
^{1}H NMR (400 MHz, $CDCl_3$): δ = 3.67 (s, 6H), 2.30 (t, *J* = 7.5 Hz, 4H), 1.71 - 1.53 (m, 4H), 1.30 (s, 8H).
^{13}C NMR (101 MHz, $CDCl_3$): δ = 174.30, 51.46, 34.06, 29.04, 24.89.

4-oxo-6-(tetrahydrofuran-2-yl)hexanoic acid:



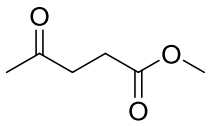
^{1}H NMR (400 MHz, $CDCl_3$): δ = 3.87 - 3.78 (m, 2H), 3.71 (dt, *J* = 14.4, 7.3 Hz, 1H), 2.80 - 2.68 (m, 2H), 2.66 - 2.42 (m, 4H), 2.07 - 1.94 (m, 2H), 1.93 - 1.86 (m, 2H), 1.86 - 1.82 (m, 1H), 1.74 (dtd, *J* = 14.2, 8.4, 6.1 Hz, 1H).
^{13}C NMR (101 MHz, $CDCl_3$): δ = 208.66, 177.63, 78.33, 67.64, 39.35, 36.92, 31.27, 29.39, 27.74, 25.67.

5-(2-(tetrahydrofuran-2-yl) ethyl) dihydrofuran-2(3H)-one:



^{1}H NMR (400 MHz, $CDCl_3$): δ = 4.63 – 4.47 (m, 1H), 3.89 – 3.80 (m, 2H), 3.72 (dd, *J* = 14.4, 7.8 Hz, 1H), 2.53 (dt, *J* = 23.8, 11.9 Hz, 2H), 2.34 (tt, *J* = 12.3, 6.1 Hz, 1H), 2.00 (ddd, *J* = 6.7, 3.1, 1.5 Hz, 1H), 1.92 – 1.81 (m, 4H), 1.77 - 1.64 (m, 2H), 1.63 - 1.56 (m, 1H), 1.51 - 1.43 (m, 1H).

Methyl levulinate:



^{1}H NMR (400 MHz, $CDCl_3$): δ = 3.82 – 3.55 (m, 3H), 2.76 (dd, *J* = 9.3, 3.7 Hz, 2H), 2.64 - 2.53 (m, 2H), 2.29 - 2.08 (m, 3H).
^{13}C NMR (101 MHz, $CDCl_3$): δ = 206.65, 173.19, 51.75, 37.88, 29.82, 27.67.

Ⅵ. References

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4. C. R. Li, D. Q. Ding, Q. N. Xia, X. H. Liu, Y. Q. Wang, *ChemSusChem*, 2016, **9**, 1712.
5. a) A. D. Sutton, F. D. Waldie, R. Wu, M. Schlaf, L. A. P. Silks, III, J. C. Gordon, *Nat. Chem.*, 2013, **5**, 428-432; b) J. C. Gordon, L. A. Silks, A. D. Sutton, R. L. Wu, M. Schlaf, F. Waldie, R. West, D. I. Collias, U.S. Patent 9,422,207[P]. 2016-8-23.
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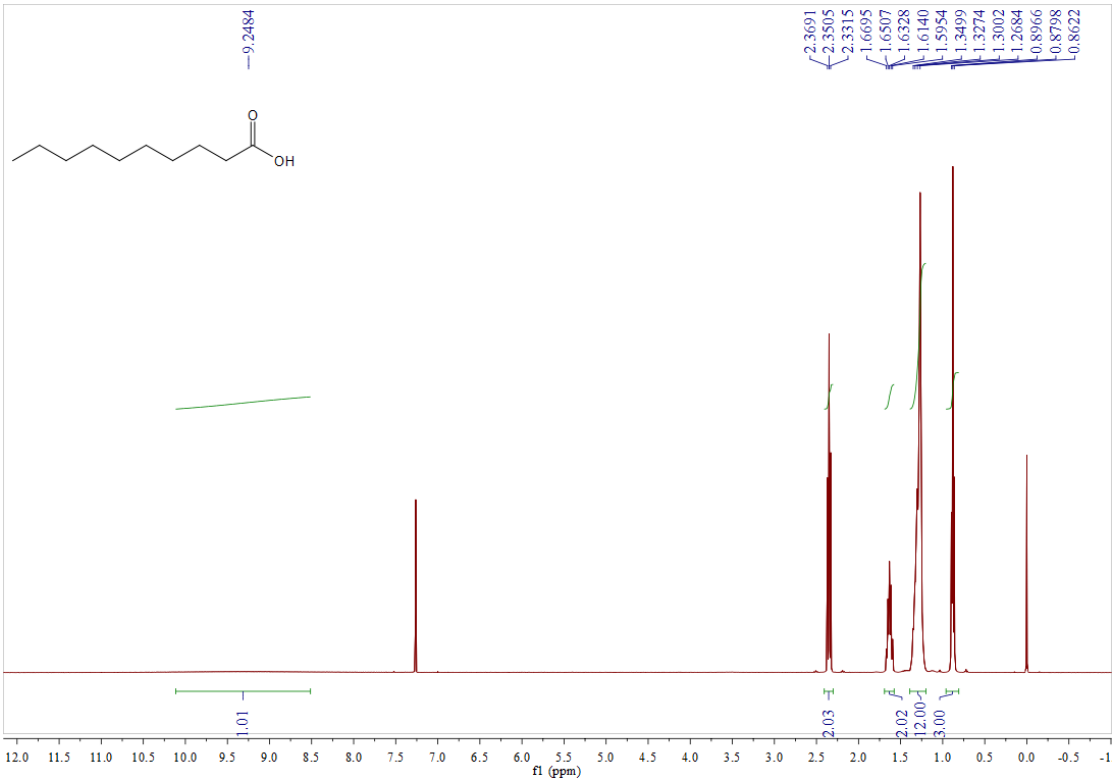
NMR spectra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1.3002
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2.02
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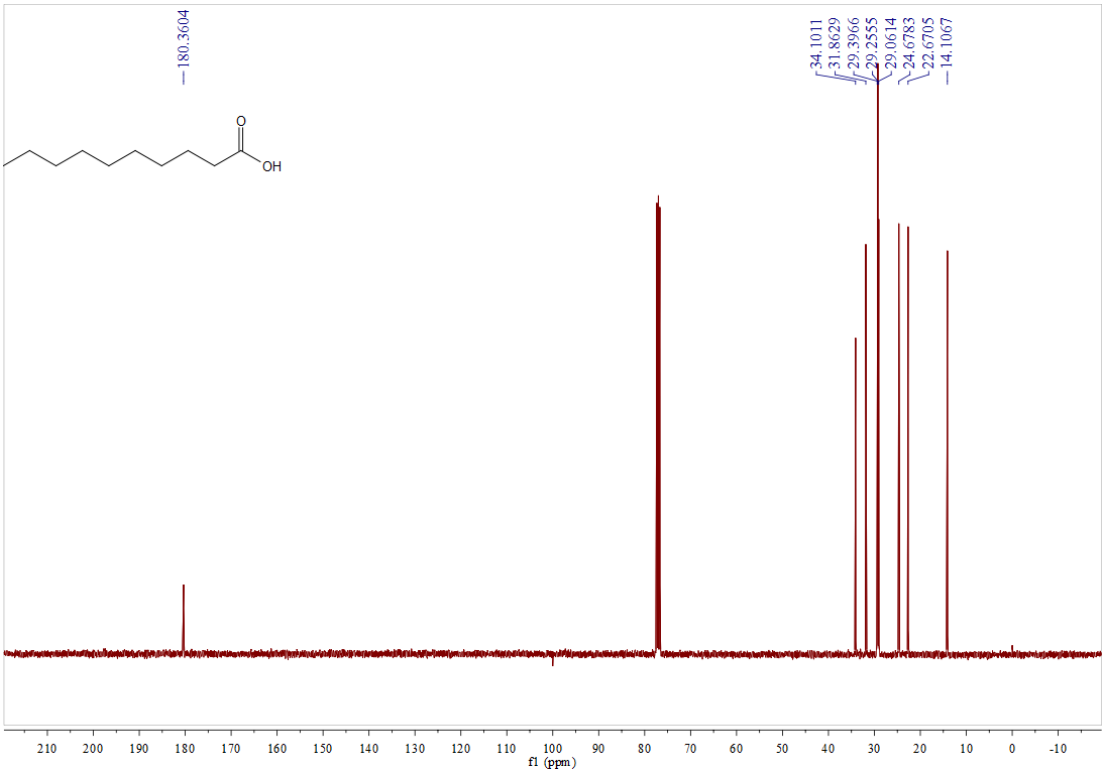
3.00
f1 (ppm)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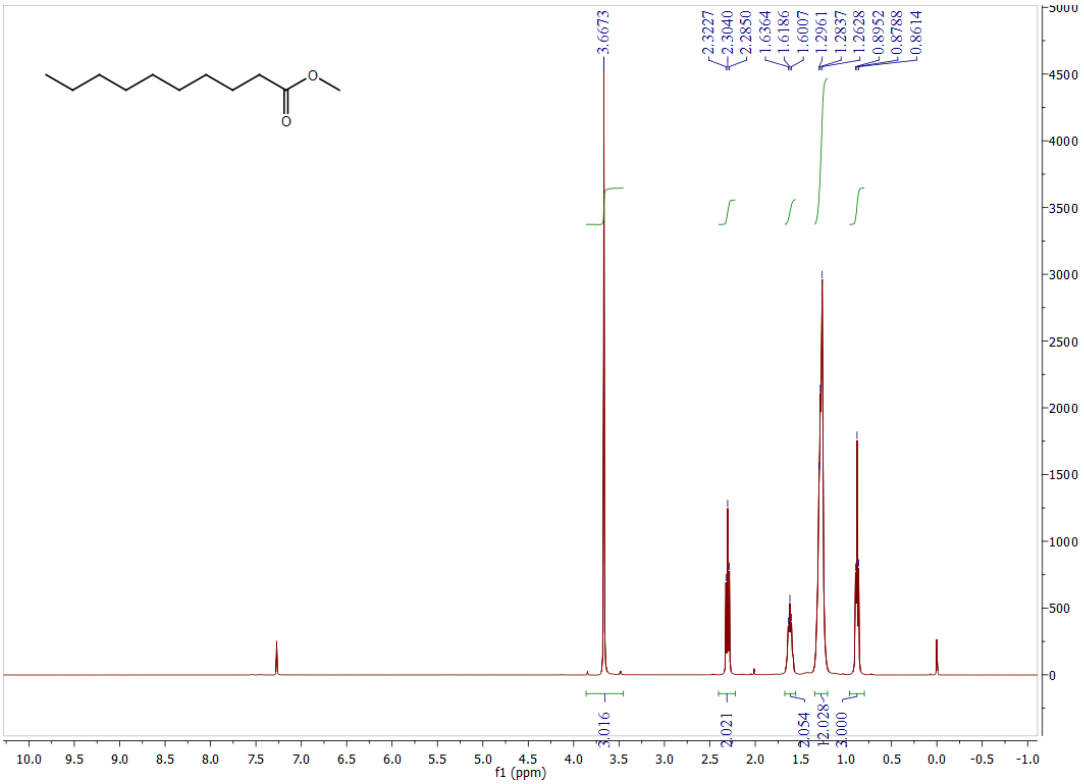
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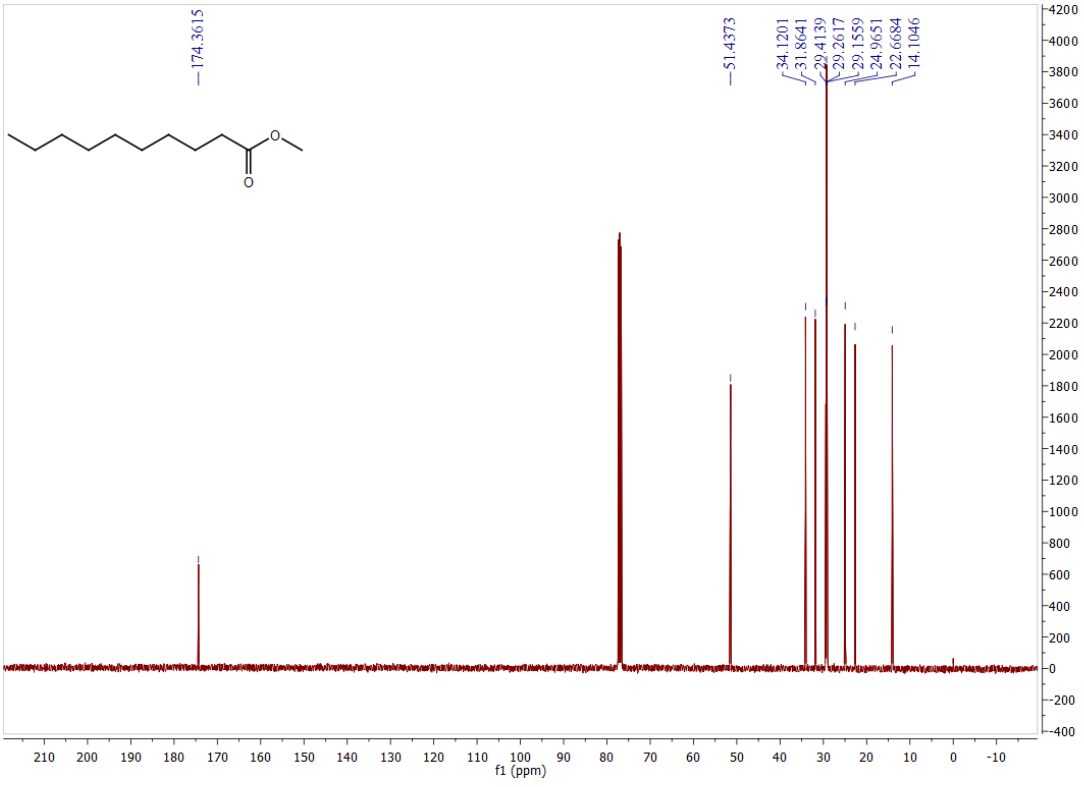
f1 (ppm)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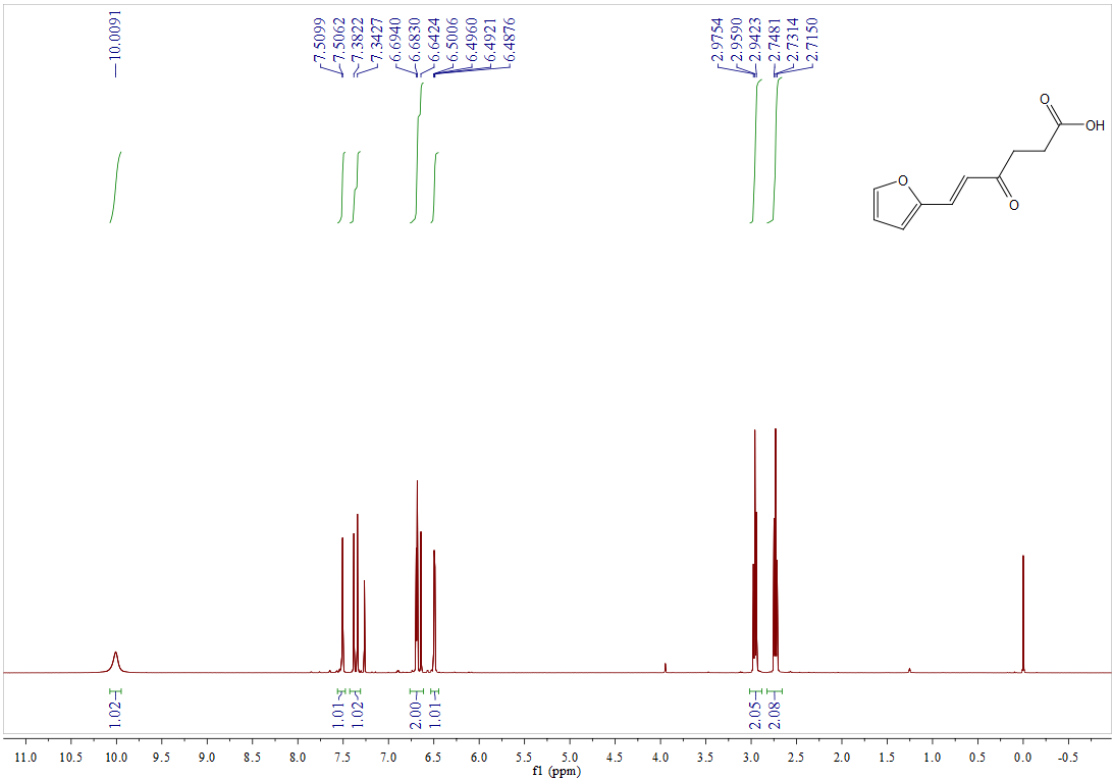
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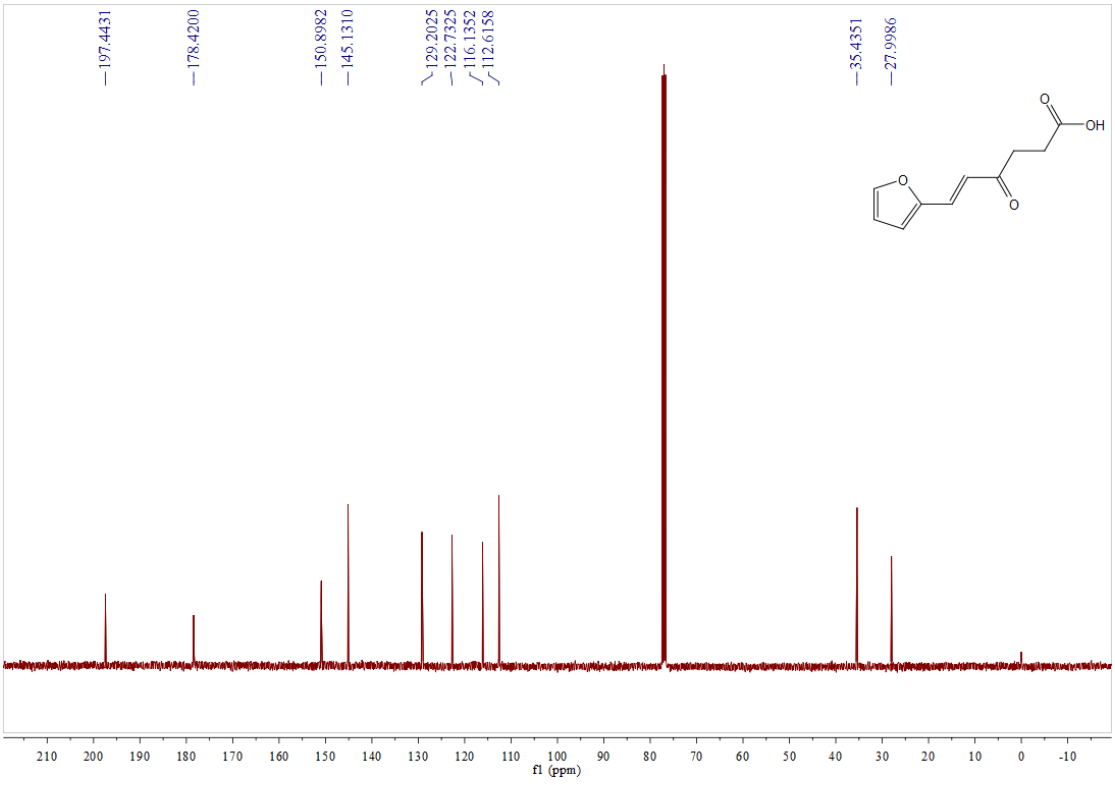
f1 (ppm)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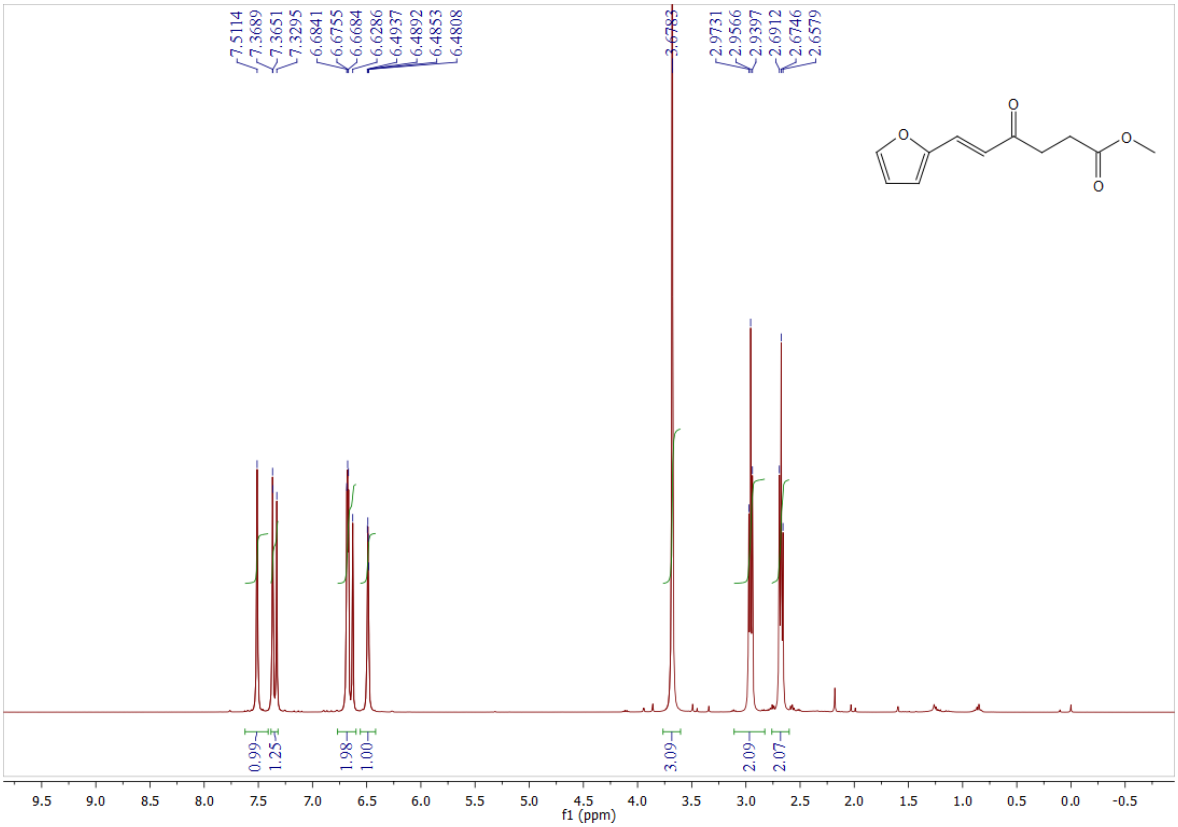
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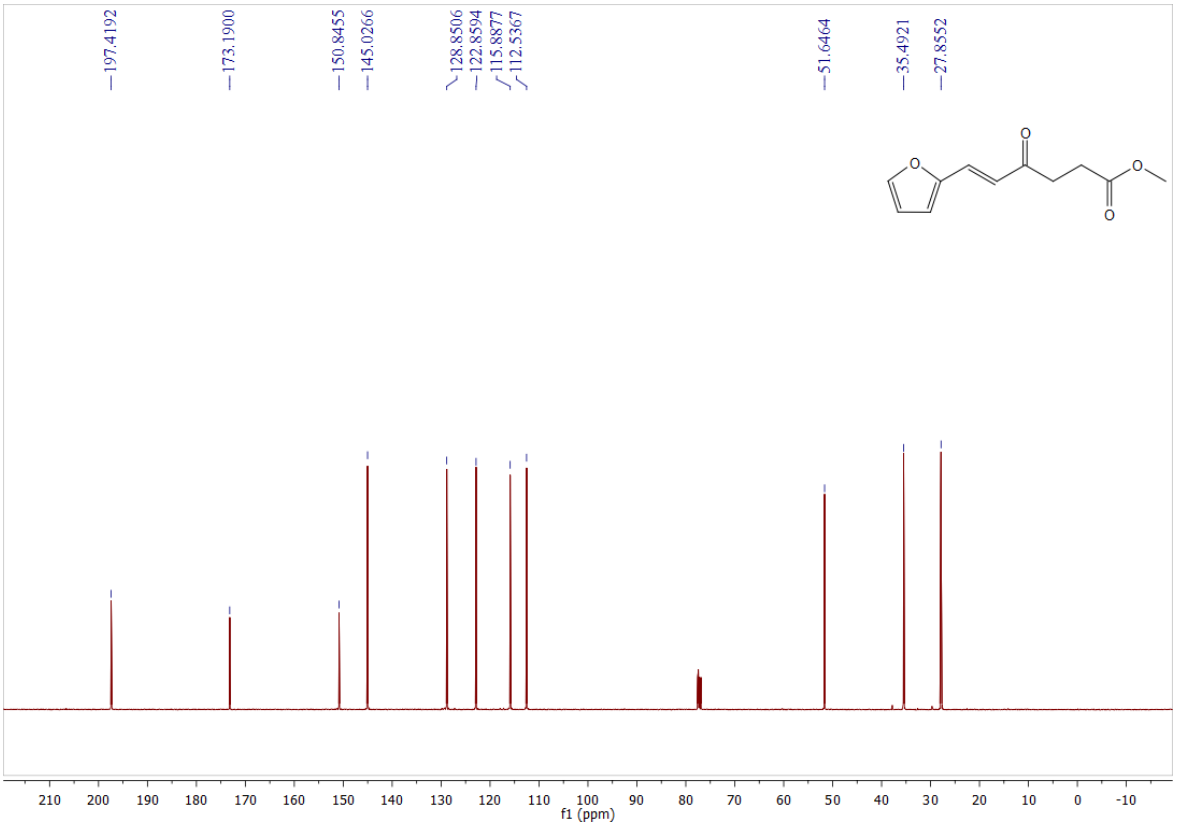
2.07
f1 (ppm)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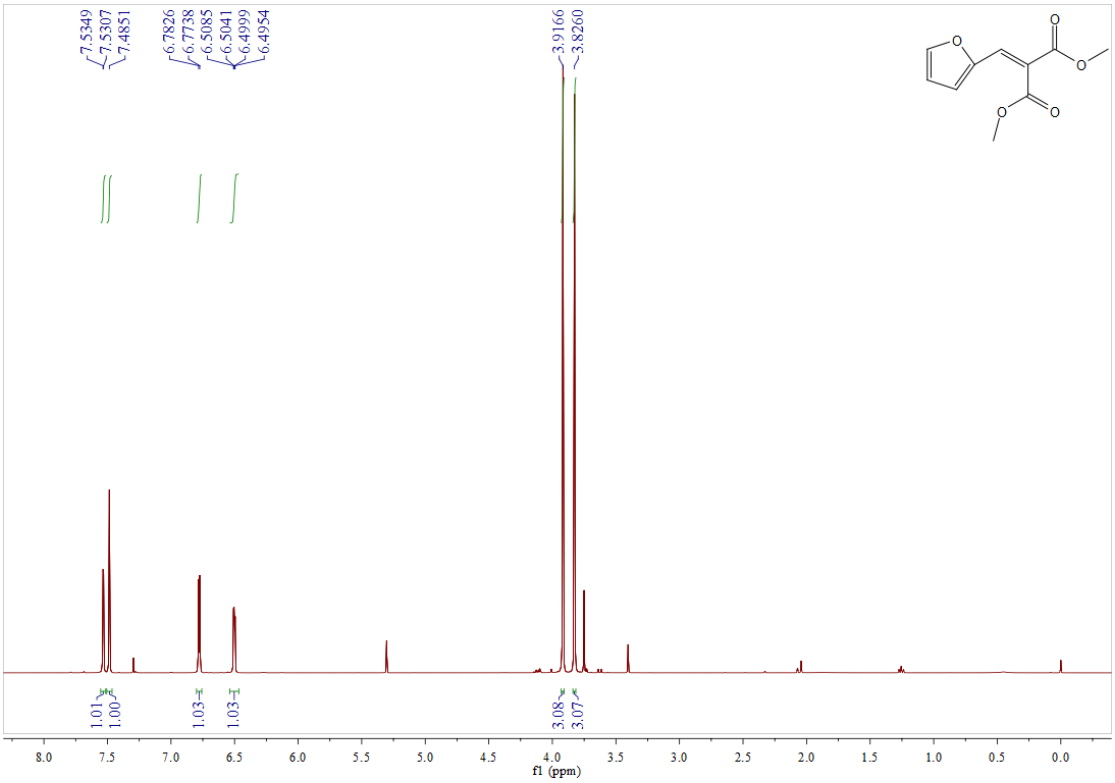
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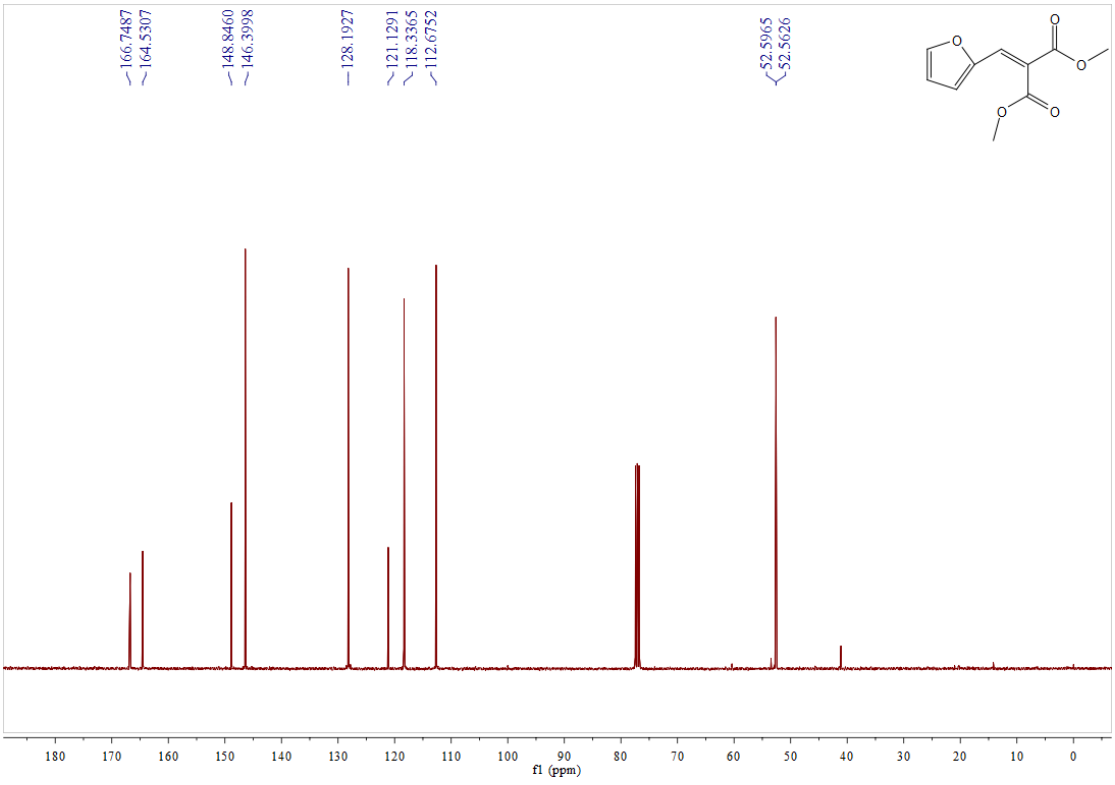
f1 (ppm)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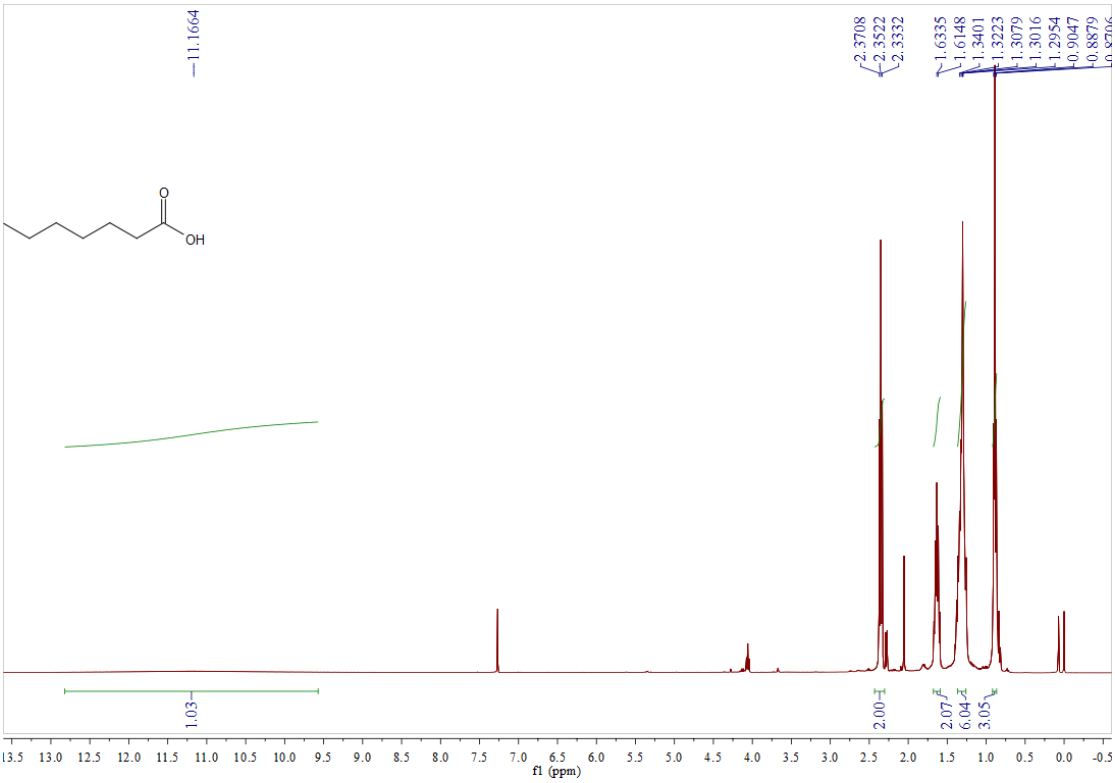
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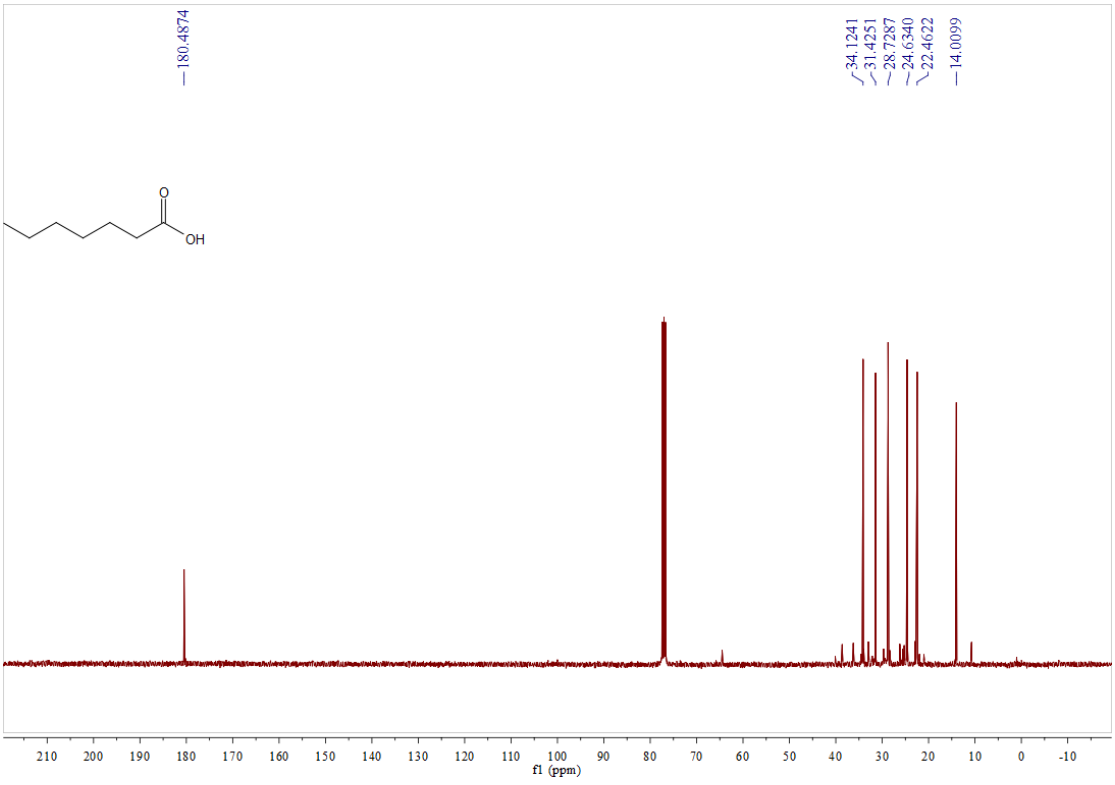
f1 (ppm)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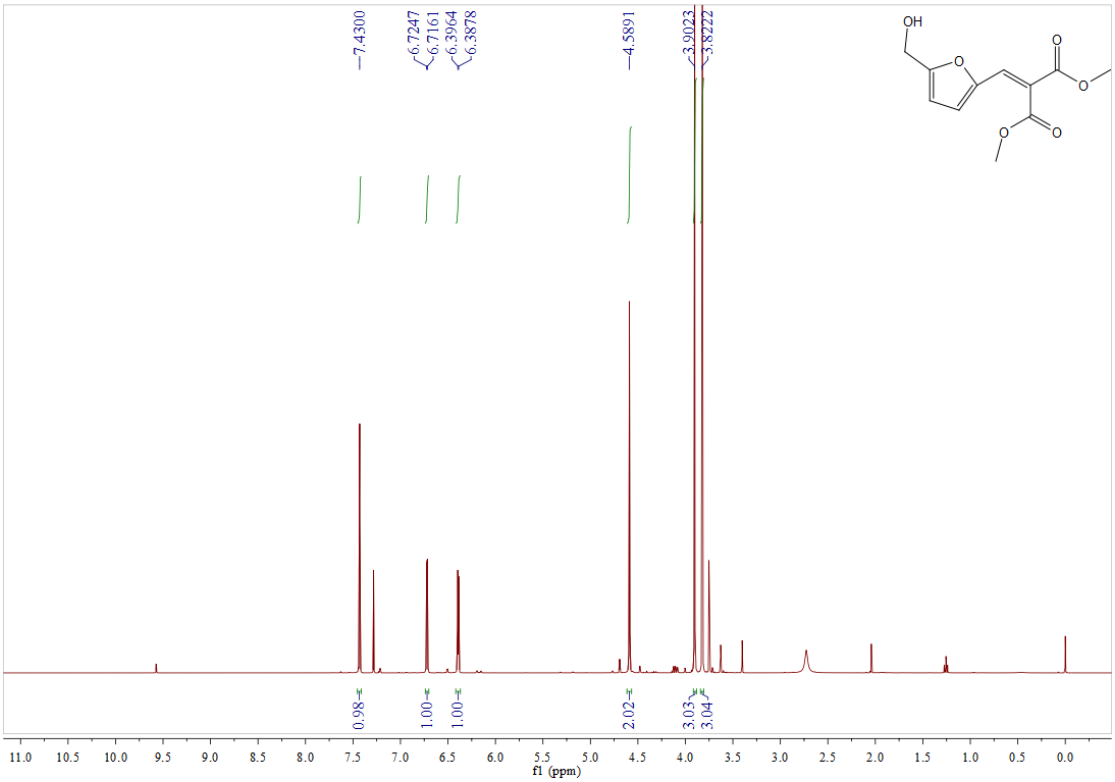
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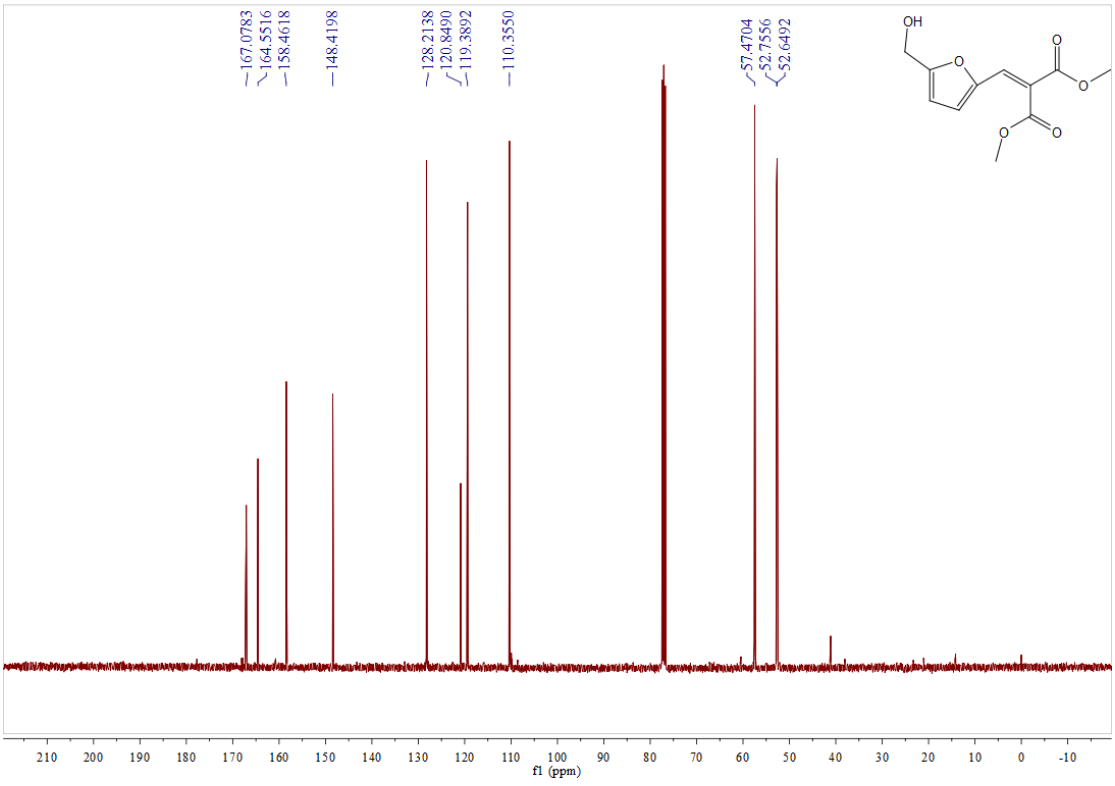
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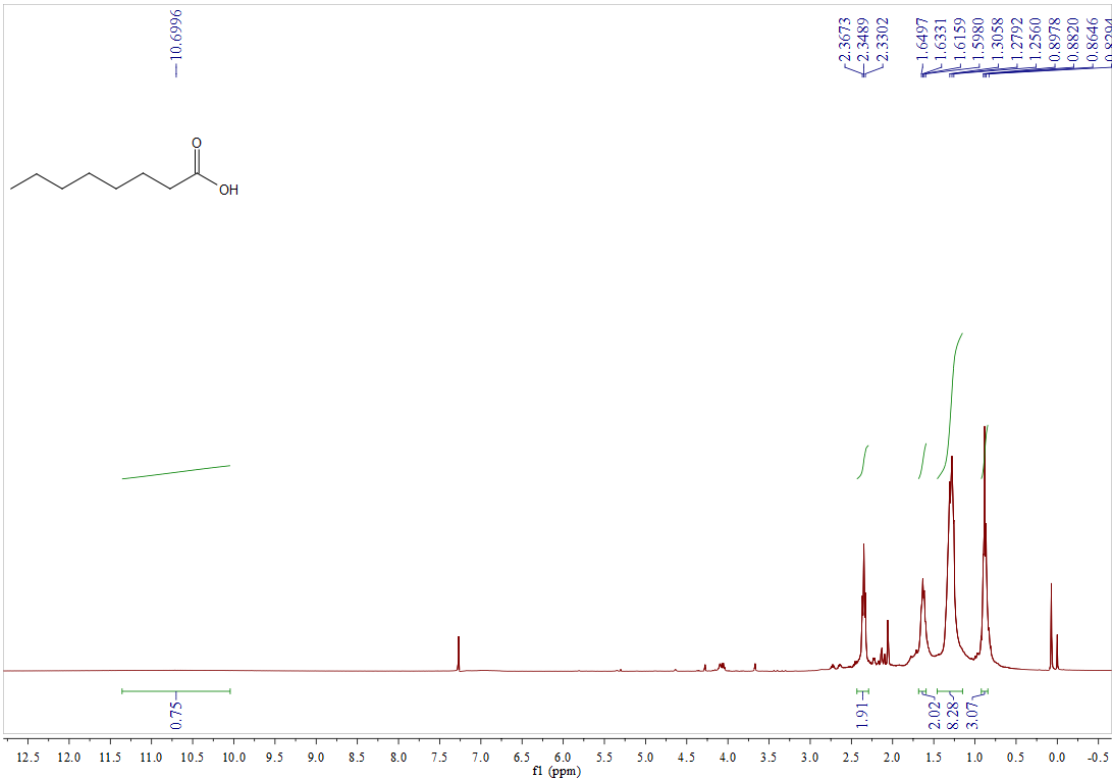
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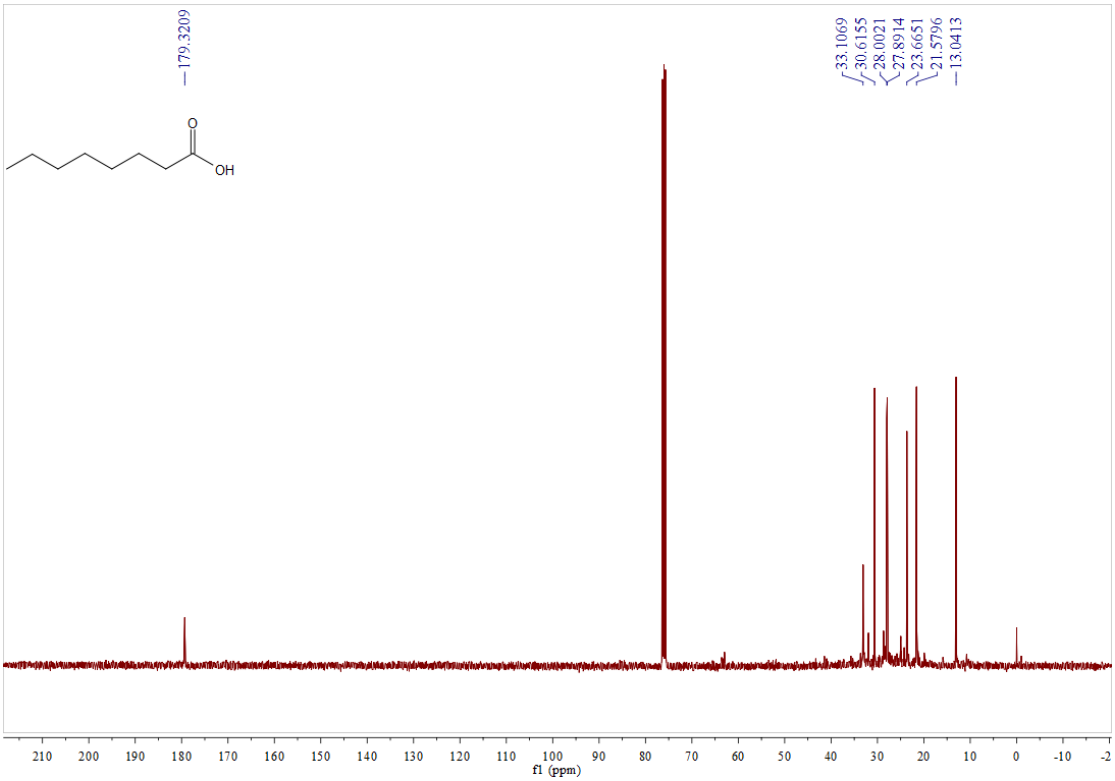
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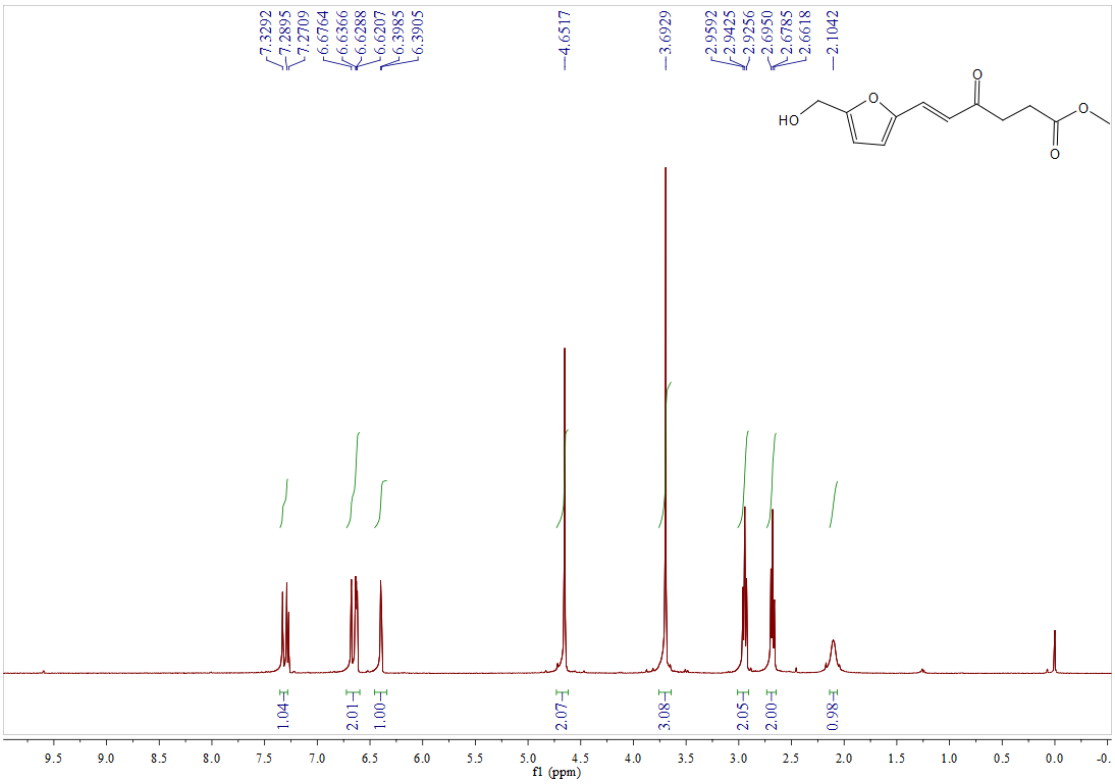
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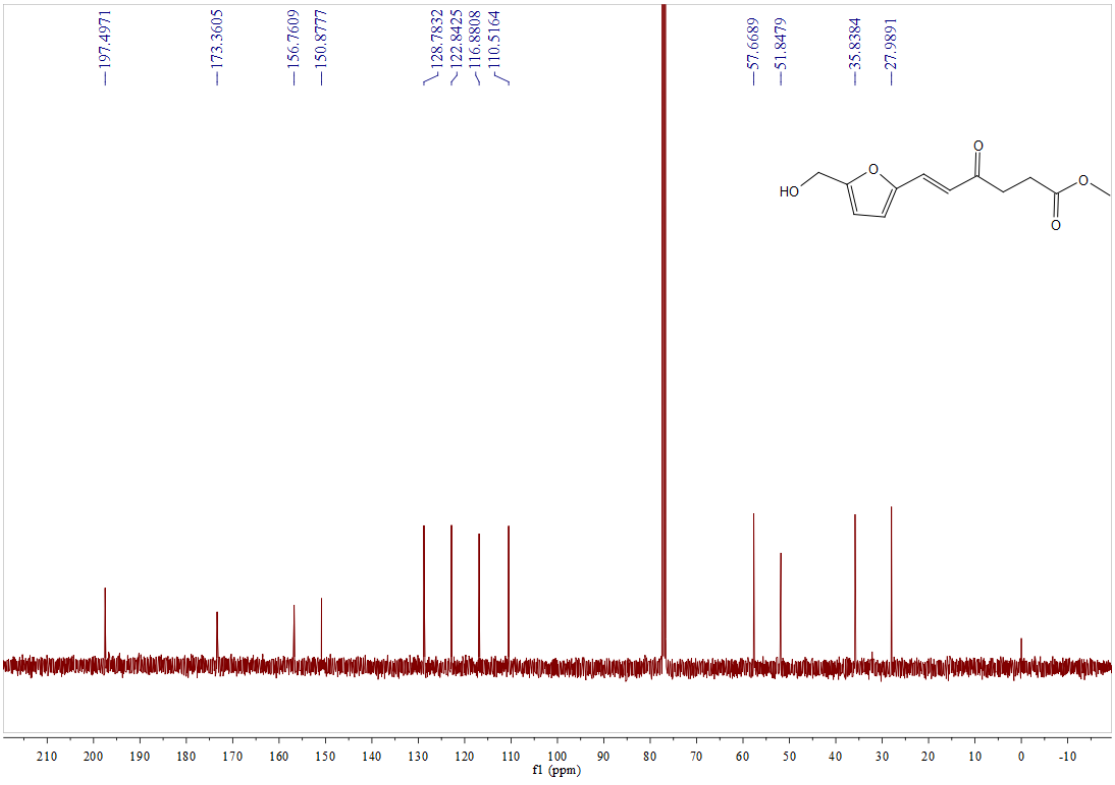
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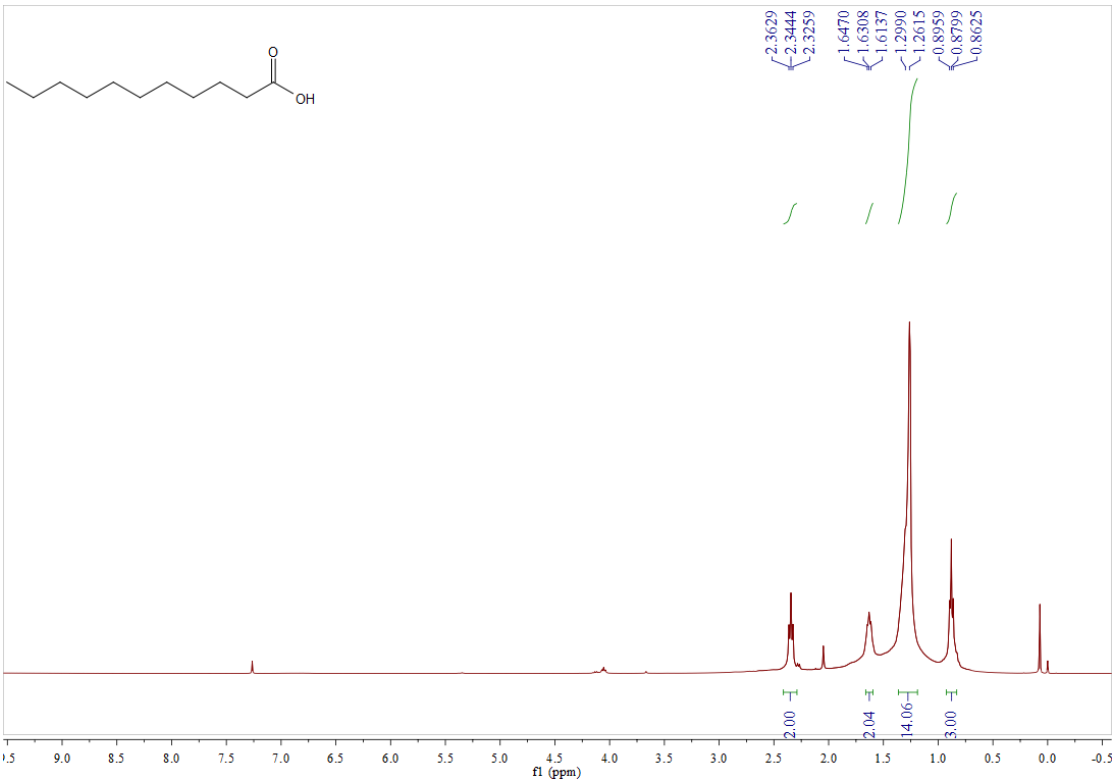
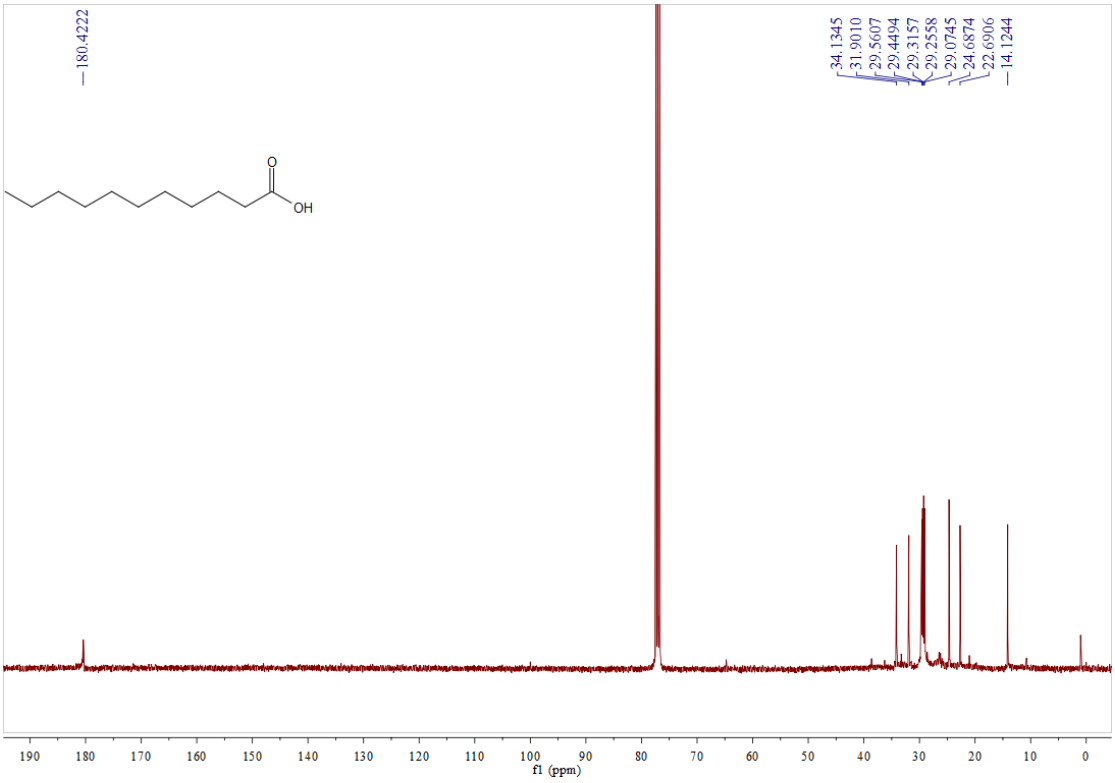
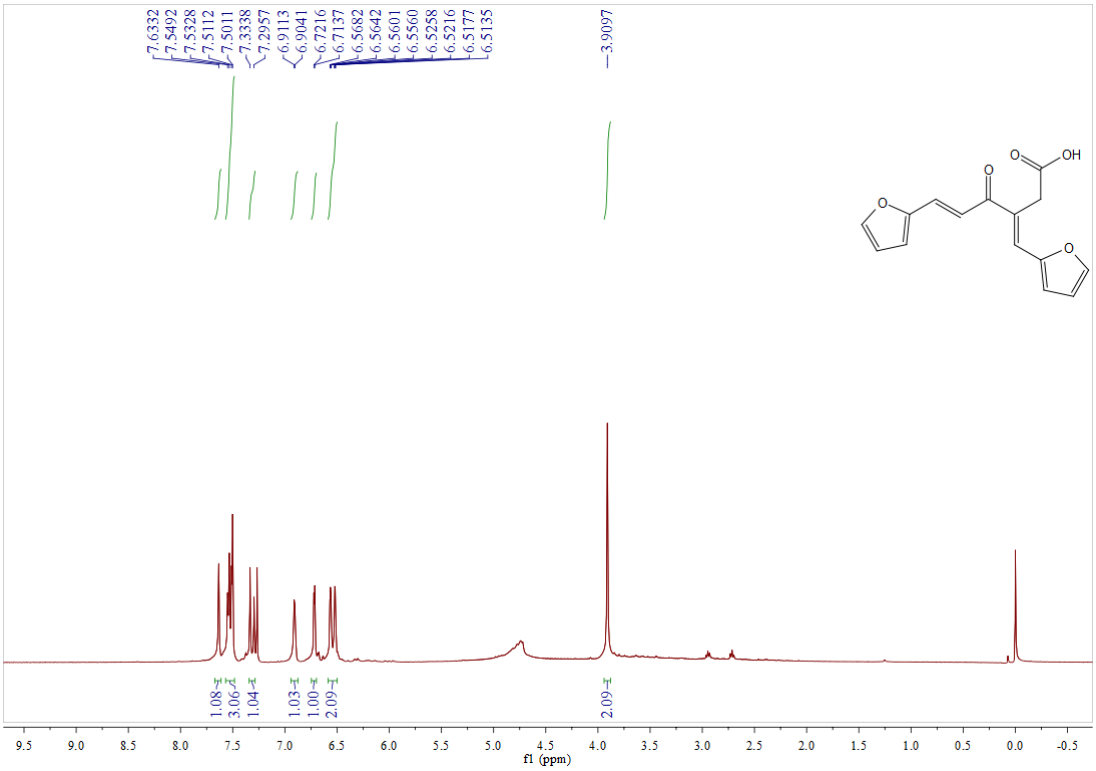
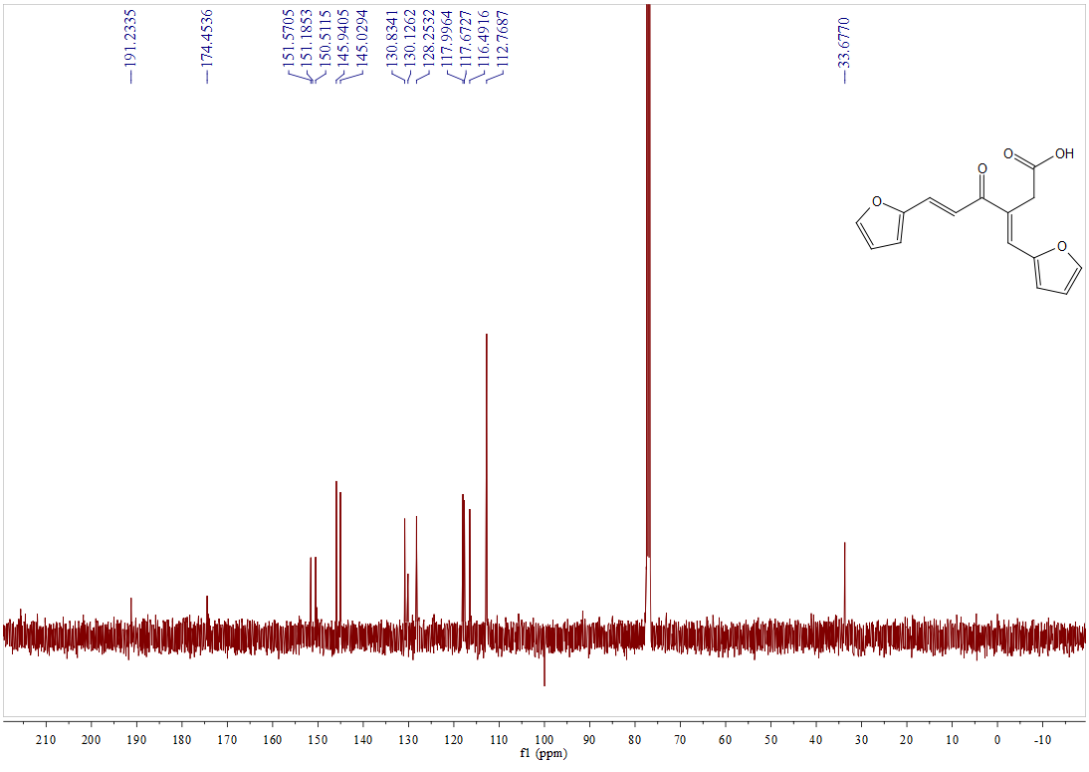
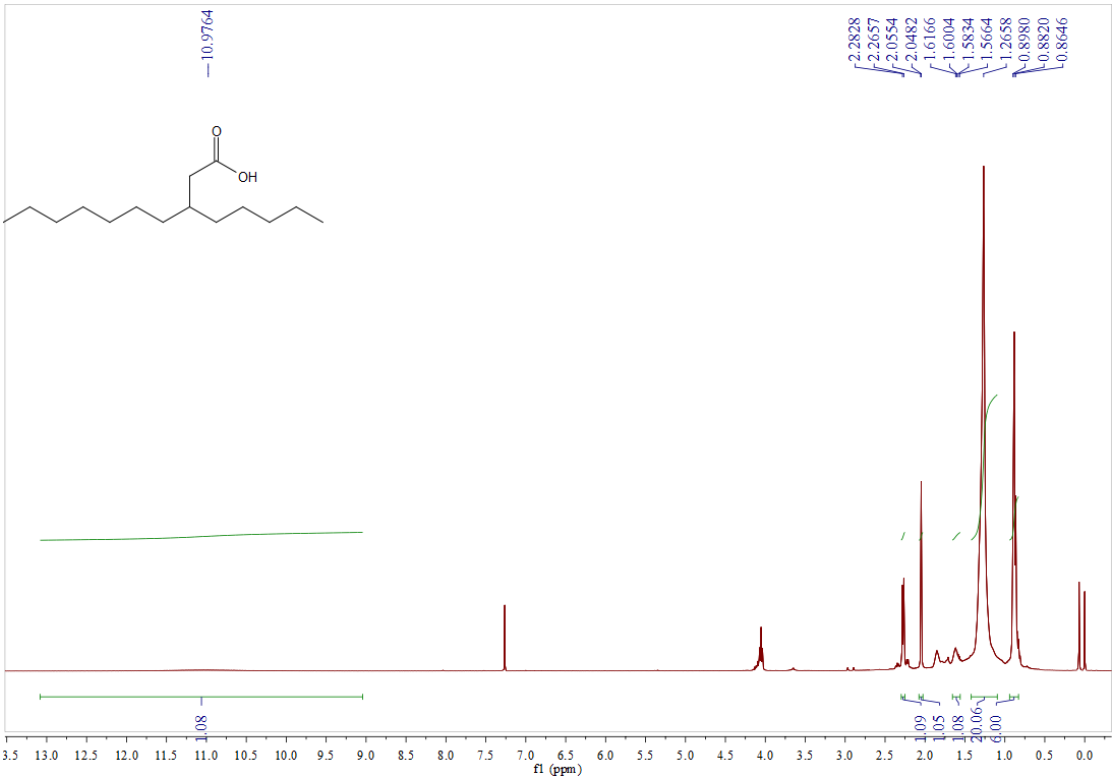
35.8384
27.9891
HO
O
O
O
f1 (ppm)

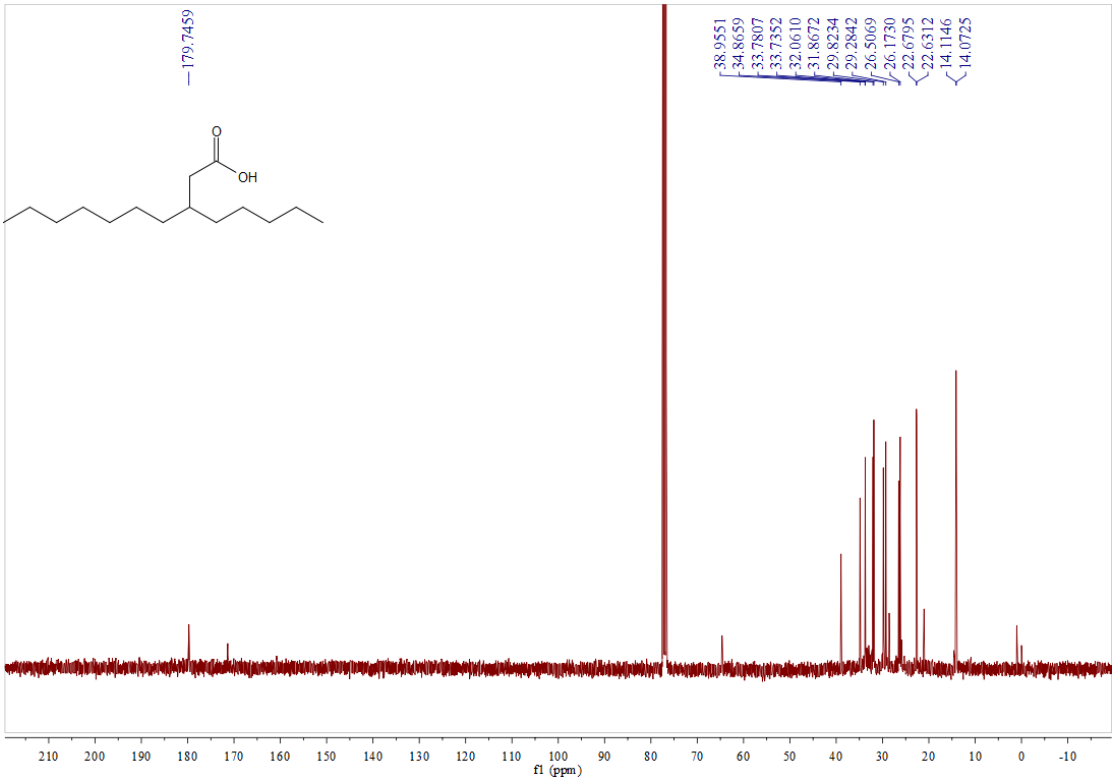

O
OH
2.3629
2.3444
2.3259
1.6470
1.6308
1.6137
1.2990
1.2615
0.8959
0.8799
0.8625
2.00
2.04
14.06
3.00
f1 (ppm)

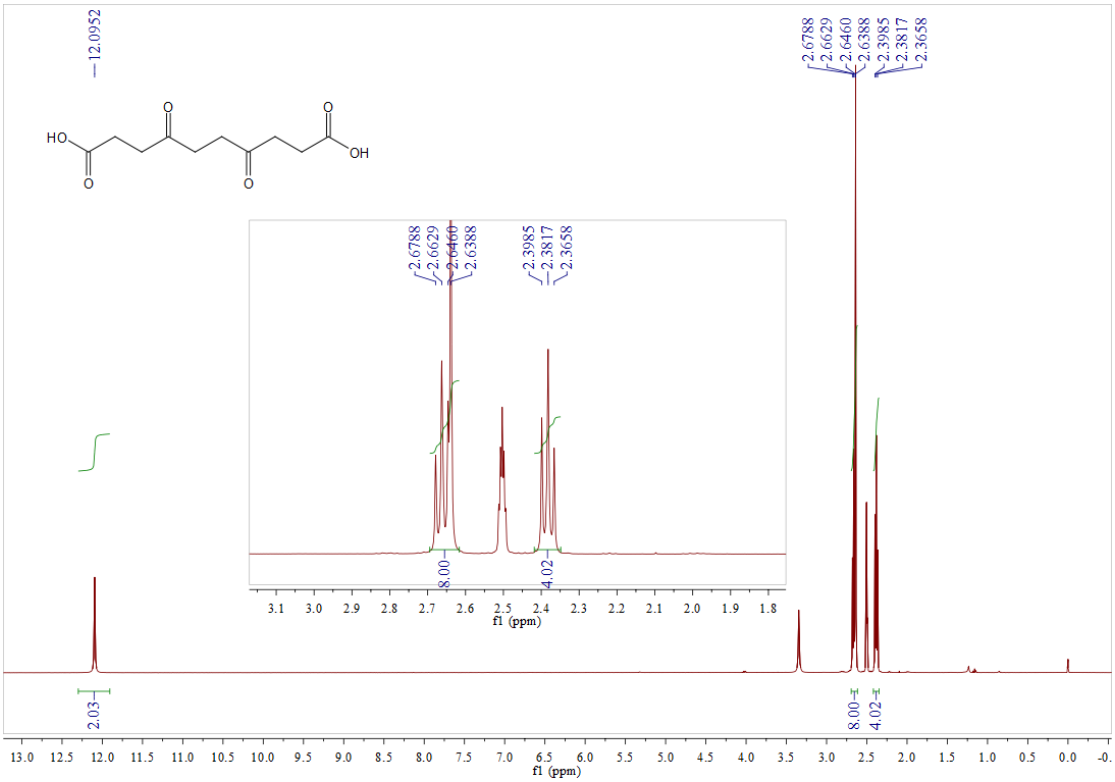

180.4222
34.1345
31.9010
29.5607
29.4494
29.3157
29.2558
29.0745
24.6874
22.6906
14.1244
O
OH
f1 (ppm)

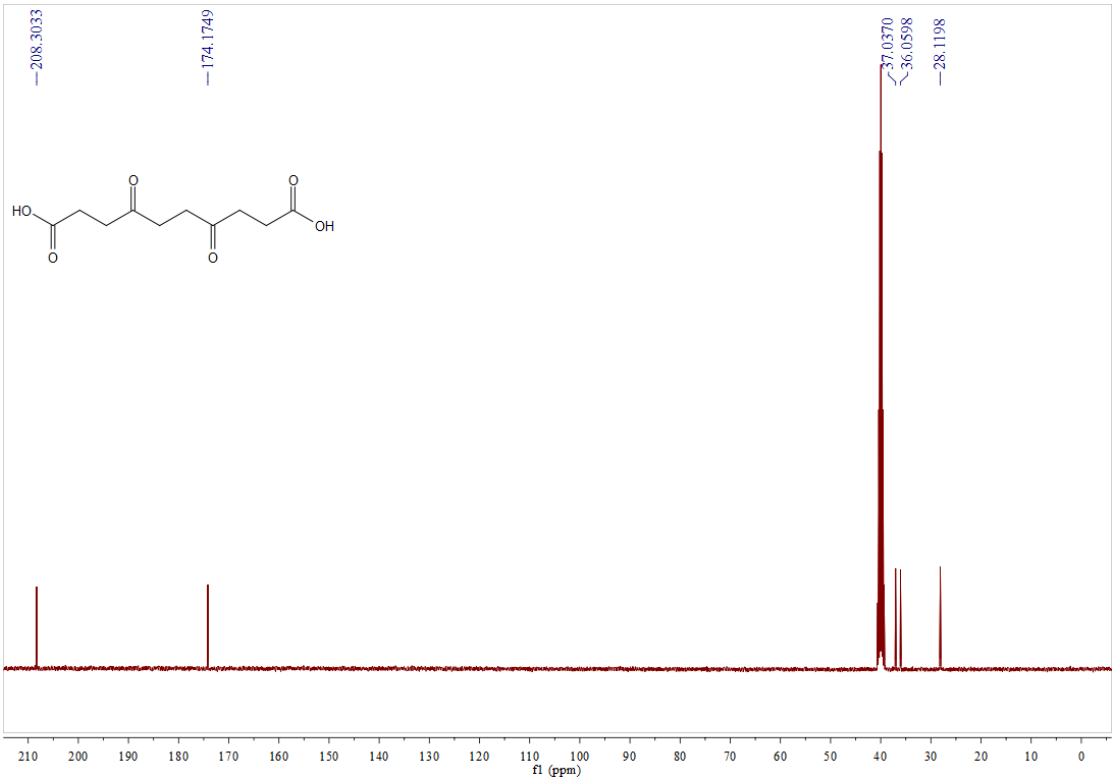

7.6332
7.5492
7.5328
7.5112
7.5011
7.3338
7.2957
6.9113
6.9041
6.7216
6.7137
6.5682
6.5642
6.5601
6.5560
6.5258
6.5216
6.5177
6.5135
3.9097
1.08
3.06
1.04
1.03
1.00
2.09
2.09
O
OH
O
O
f1 (ppm)


191.2335
174.4536
151.5705
151.1853
150.5115
145.9405
145.0294
130.8341
130.1262
128.2532
117.9964
117.6727
116.4916
112.7687
33.6770
O
OH
O
O
f1 (ppm)


10.9764
2.2828
2.2657
2.0554
2.0482
1.6166
1.6004
1.5834
1.5664
1.2658
0.8980
0.8820
0.8646
O
OH
1.08
1.09
1.05
1.08
20.06
6.00
f1 (ppm)


179.7459
38.9551
34.8659
33.7807
33.7352
32.0610
31.8672
29.8234
29.2842
26.5069
26.1730
22.6795
22.6312
14.1146
14.0725
O
OH
f1 (ppm)


12.0952
2.6788
2.6629
2.6460
2.6388
2.3985
2.3817
2.3658
HO
O
O
O
OH
8.00
4.02
2.03
f1 (ppm)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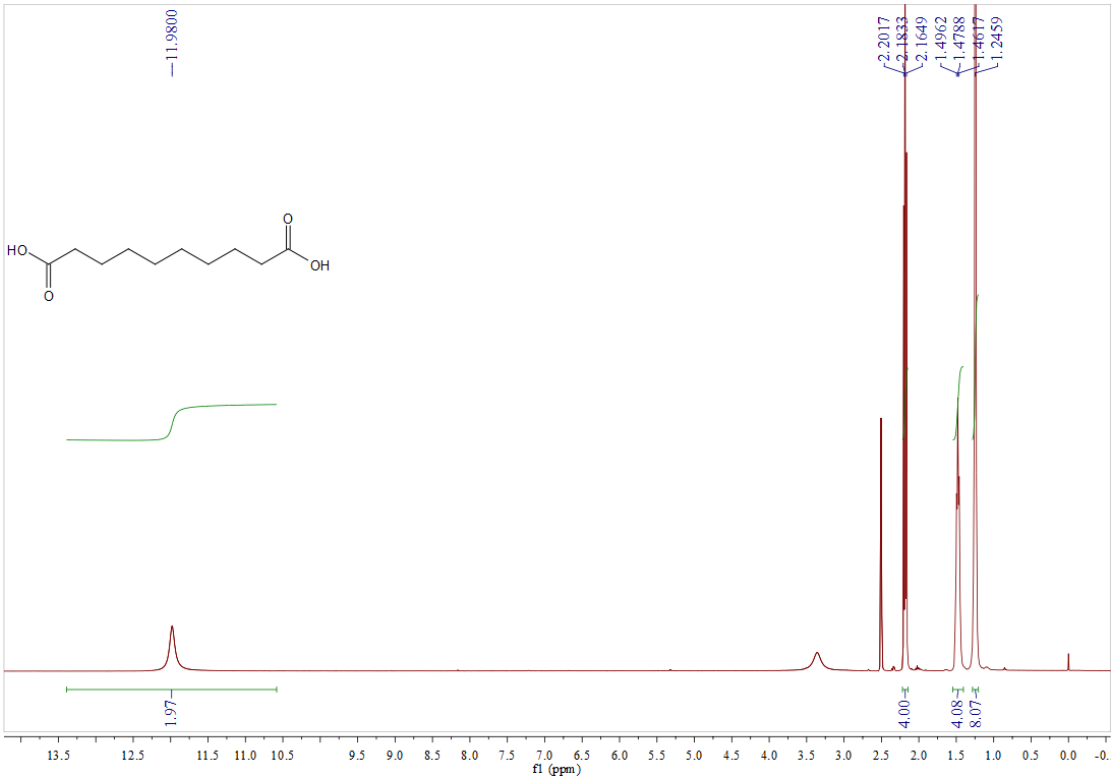
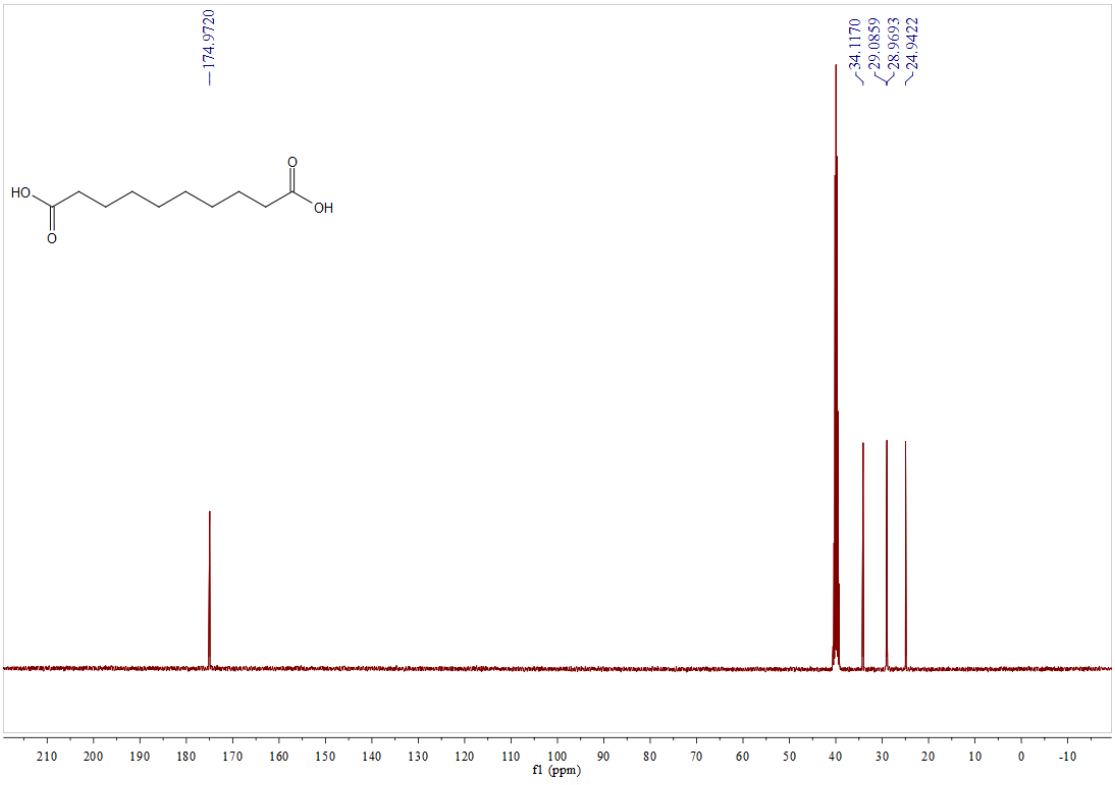
174.1749
37.0370
36.0598
28.1198
HO
O
O
O
OH
f1 (ppm)


11.9800
2.2017
2.1833
2.1649
1.4962
1.4788
1.4617
1.2459
HO
O
O
OH
1.97
4.00
4.08
8.07
f1 (ppm)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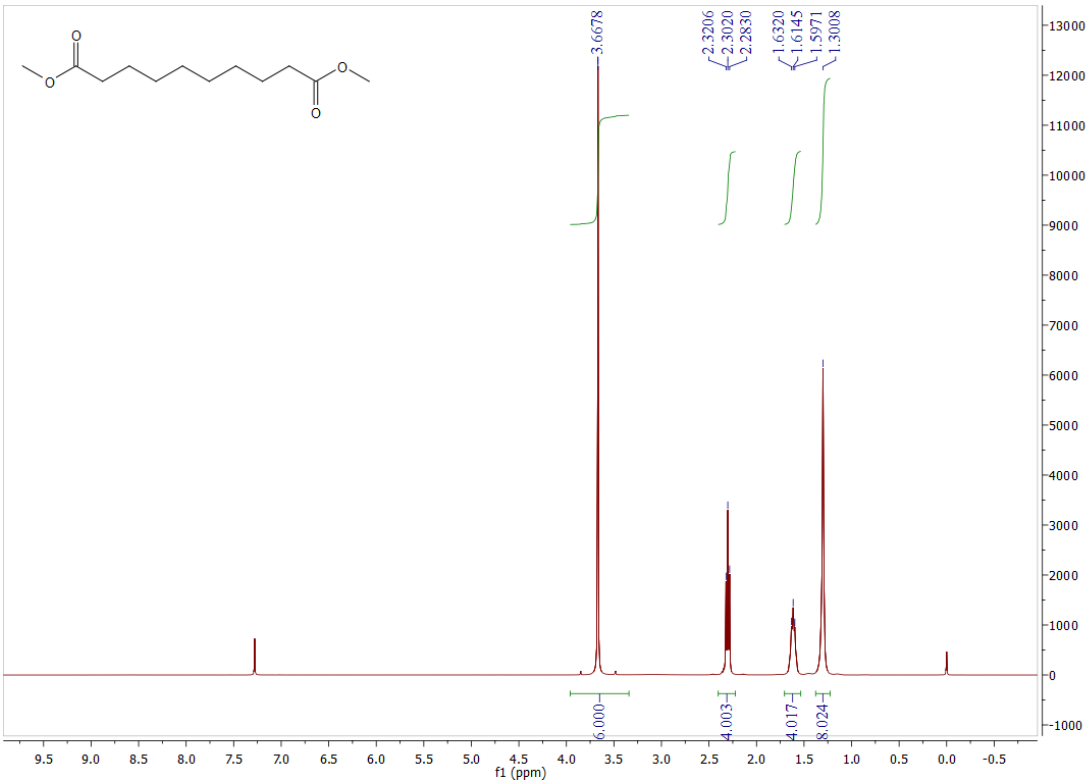
34.1170
29.0859
28.9693
24.9422
HO
O
O
OH
f1 (ppm)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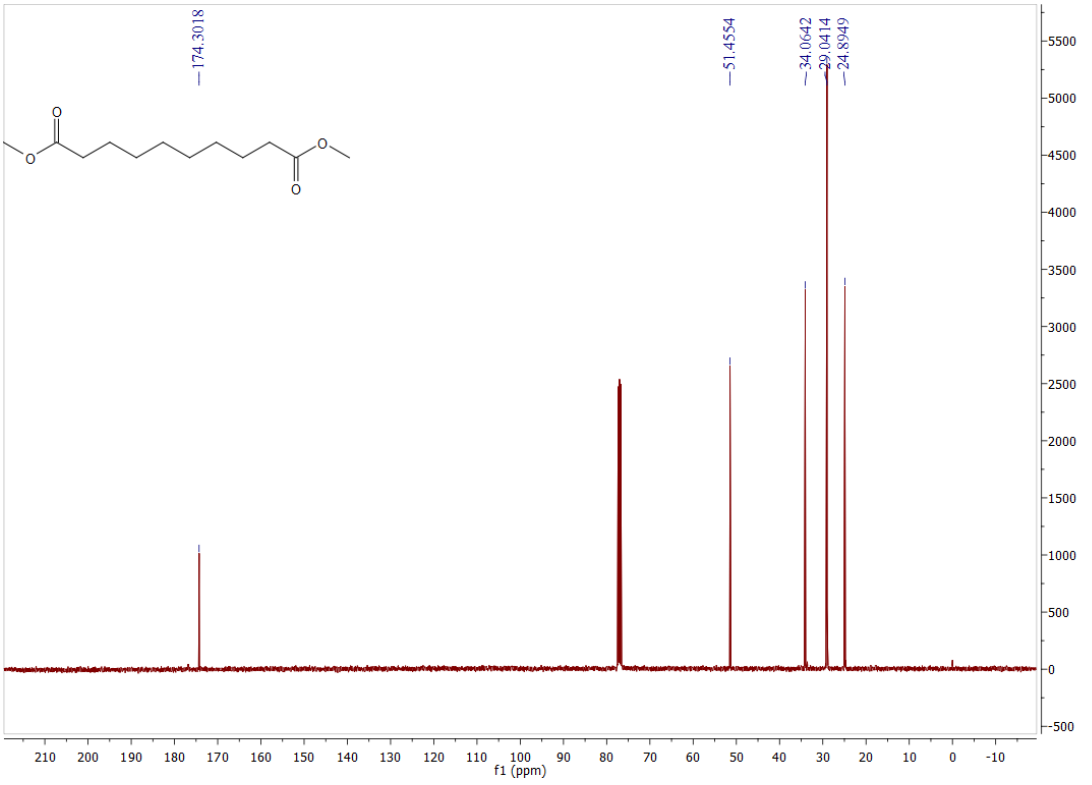
2.3206
2.3020
2.2830
1.6320
1.6145
1.5971
1.3008
6.000
4.003
4.017
8.024
f1 (ppm)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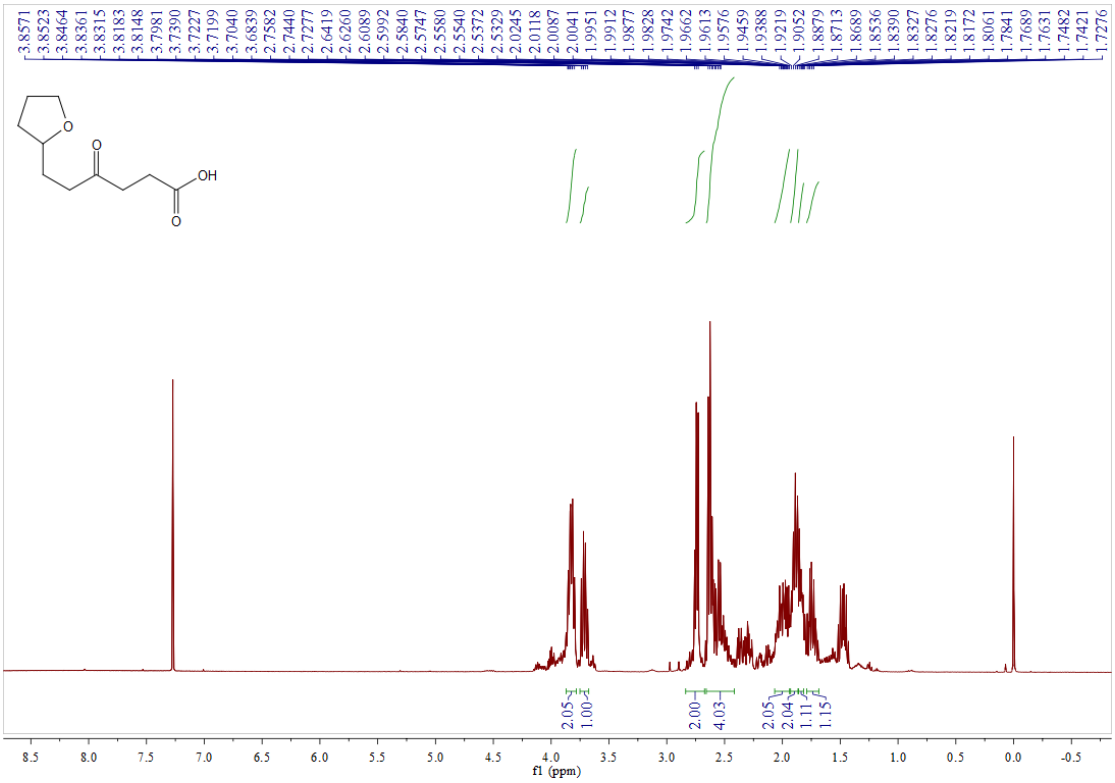
51.4554
34.0642
29.0414
24.8949
f1 (ppm)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3.8523
3.8464
3.8361
3.8315
3.8183
3.8148
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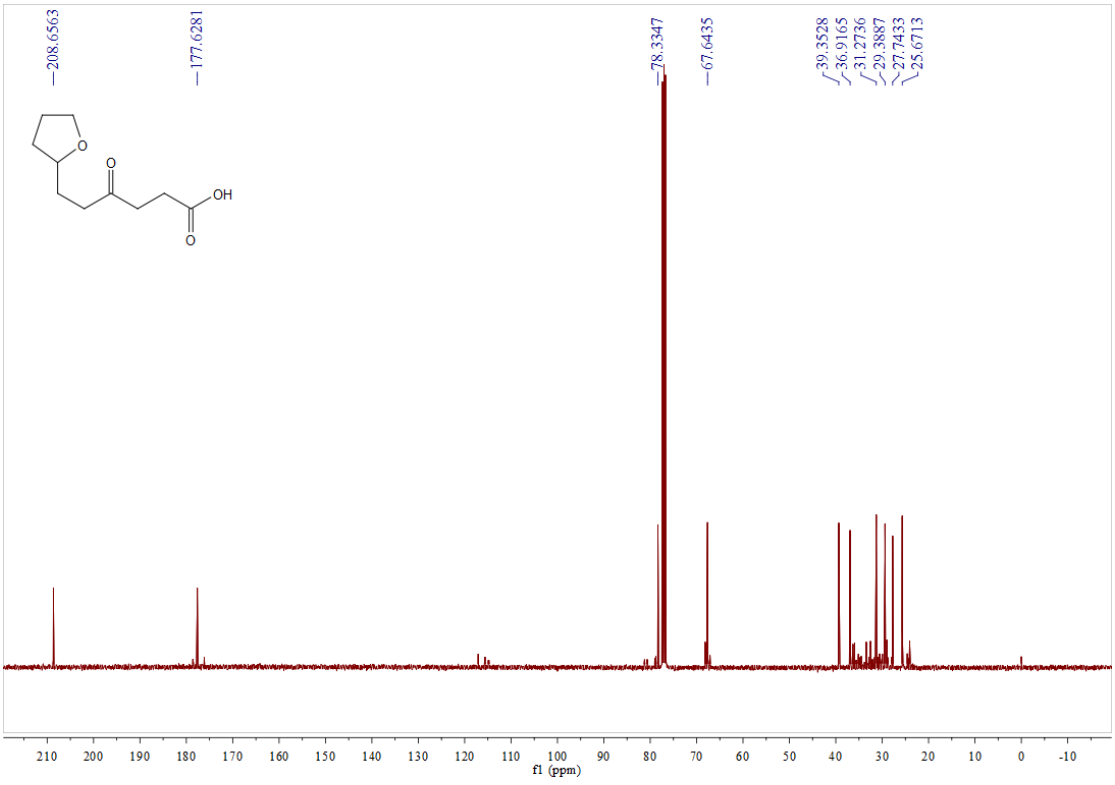
3.7199
3.7040
3.6839
2.7582
2.7440
2.7277
2.6419
2.6260
2.6089
2.5992
2.5840
2.5747
2.5580
2.5540
2.5372
2.5329
2.0245
2.0118
2.0087
2.0041
1.9951
1.9912
1.9877
1.9828
1.9742
1.9662
1.9613
1.9576
1.9459
1.9388
1.9219
1.9052
1.8879
1.8713
1.8689
1.8536
1.8390
1.8327
1.8276
1.8219
1.8172
1.8061
1.7841
1.7689
1.7631
1.7482
1.7421
1.7276
O
O
OH
O
2.05
1.00
2.00
4.03
2.05
2.04
1.11
1.15
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
-0.5
f1 (ppm)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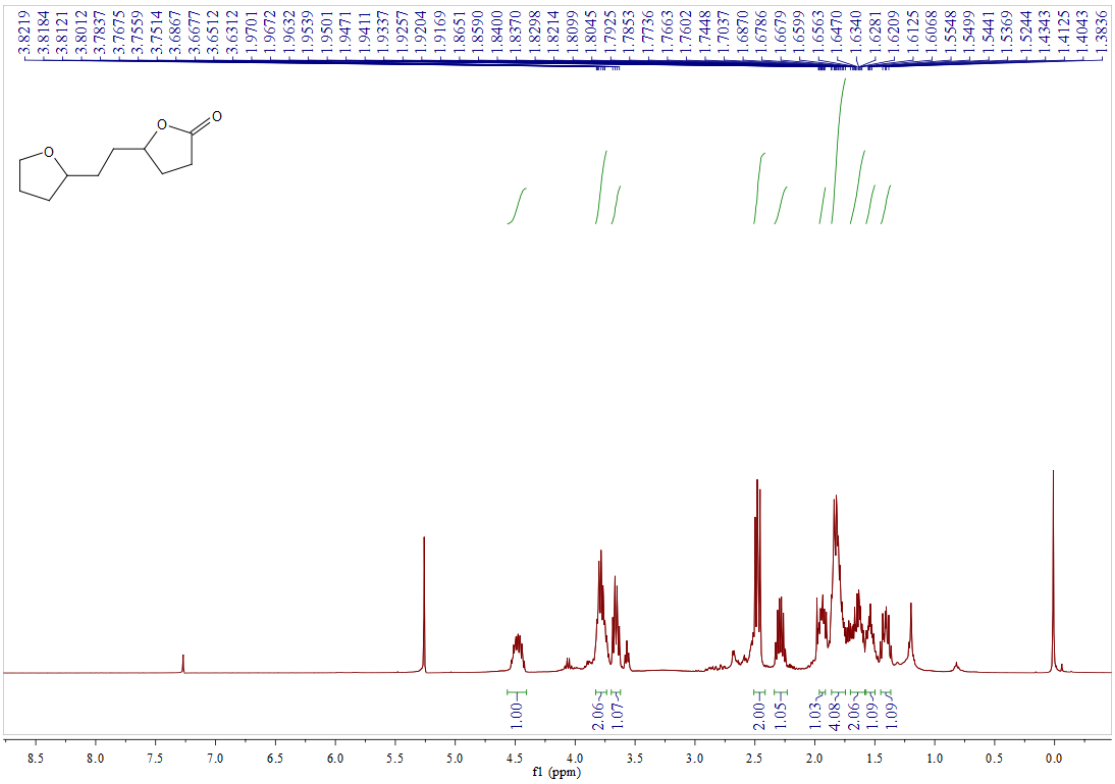
78.3347
67.6435
39.3528
36.9165
31.2736
29.3887
27.7433
25.6713
O
O
OH
O
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)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3.7837
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3.7559
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3.6867
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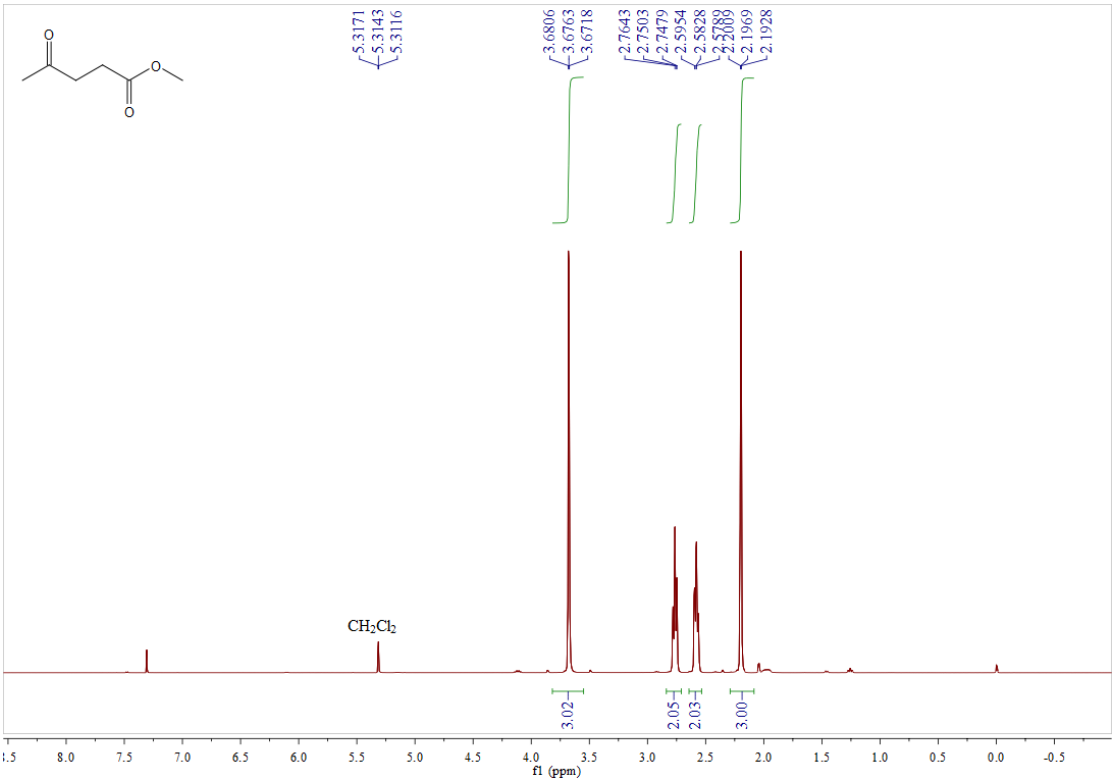
3.6312
1.9701
1.9672
1.9632
1.9539
1.9501
1.9471
1.9411
1.9337
1.9257
1.9204
1.9169
1.8651
1.8590
1.8400
1.8370
1.8298
1.8214
1.8099
1.8045
1.7925
1.7853
1.7736
1.7663
1.7602
1.7448
1.7037
1.6870
1.6786
1.6679
1.6599
1.6563
1.6470
1.6340
1.6281
1.6209
1.6125
1.6068
1.5548
1.5499
1.5441
1.5369
1.5244
1.4343
1.4125
1.4043
1.3836
O
O
O
1.00
2.06
1.07
2.00
1.05
1.03
4.08
2.06
1.09
1.09
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)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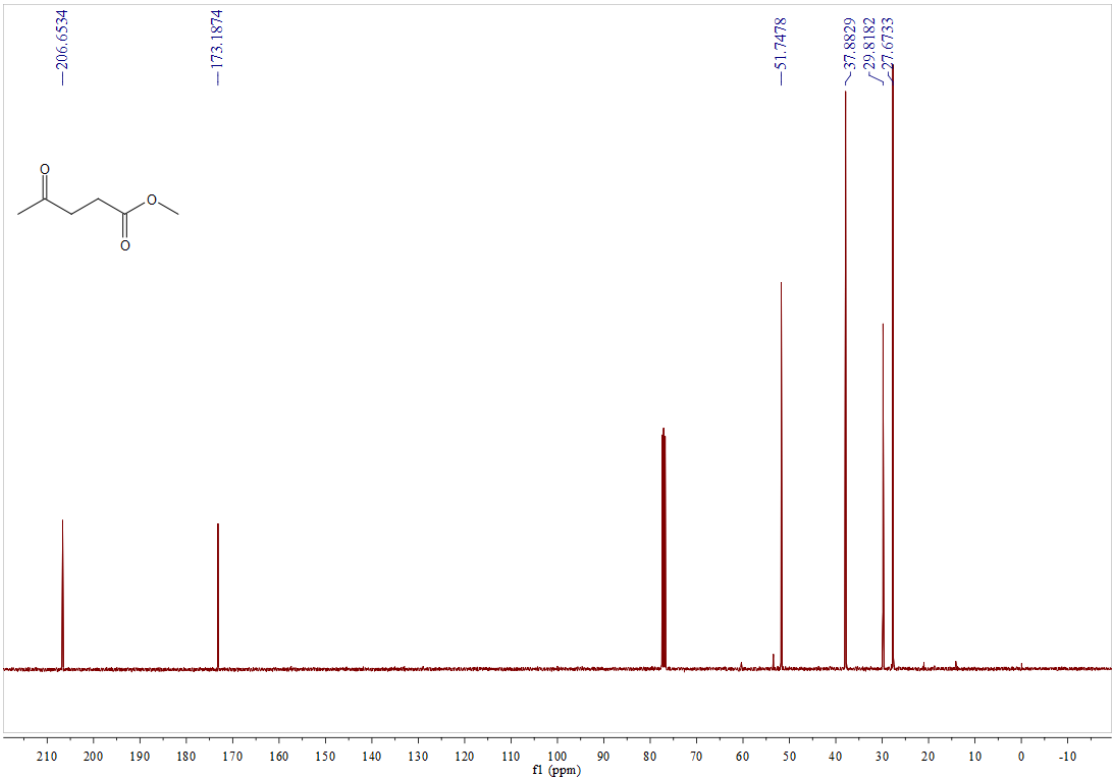
5.3143
5.3116
3.6806
3.6763
3.6718
2.7643
2.7503
2.7479
2.5954
2.5828
2.2089
2.1969
2.1928
CH2Cl2
3.02
2.05
2.03
3.00
f1 (ppm)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173.1874
51.7478
37.8829
29.8182
27.6733
f1 (ppm)