Electronic Supplementary Material (ESI) for ChemComm.

Supporting Information

Table of Contents

1. General

New compounds were characterized by ^{1}H, ^{13}C, ^{19}F, IR, and HRMS. ^{1}H, ^{13}C, and ^{19}F NMR spectra were recorded on a JEOL JMTC-400/54/SS spectrometer (^{1}H NMR, 400 MHz; ^{13}C NMR, 100 MHz, ^{19}F NMR, 377 MHz). ^{1}H NMR chemical shifts were determined relative to Me_4Si (0.0 ppm) as an internal standard. ^{13}C NMR chemical shifts were determined relative to $CDCl_3$ (77.0 ppm). ^{19}F NMR chemical shifts were determined relative to C_6F_6 (−164.9 ppm) as an external standard. Infrared spectra were recorded on a SHIMADZU IRAffinity-1 FT-IR Spectrometer. High-resolution mass spectra were obtained on a JEOL JMS-700 mass spectrometer (magnetic sector type mass spectrometer). Chiral-phase high-performance liquid chromatography (HPLC) was performed on a SHIMADZU prominence series instruments equipped with chiral columns. All reactions were carried out under nitrogen. Products were purified by chromatography on silica gel BW-300 (Fuji Silysia Chemical Ltd.) or Chromatorex NH (Fuji Silysia Chemical Ltd.) and gel permeation chromatography. Analytical thin-layer chromatography (TLC) was performed on pre-coated silica gel glass plates (Merck silica gel 60 F_{254} and Fuji Silysia Chromatorex NH, 0.25 mm thickness). Compounds were visualized with UV lamp or treatment with an ethanolic solution of phosphomolybdic acid followed by heating.

2. Materials

Starting materials **1c**,[1] **1d**,[1] **1e**,[1] **1m**,[2] **1n**,[3] **3b**,[4] **3c**,[5] and **5**[6] were prepared according to literature procedures. Analytical data for **1m**[7] were in excellent agreement with reported data. All other starting materials, solvents, and reagents were purchased and used as obtained.

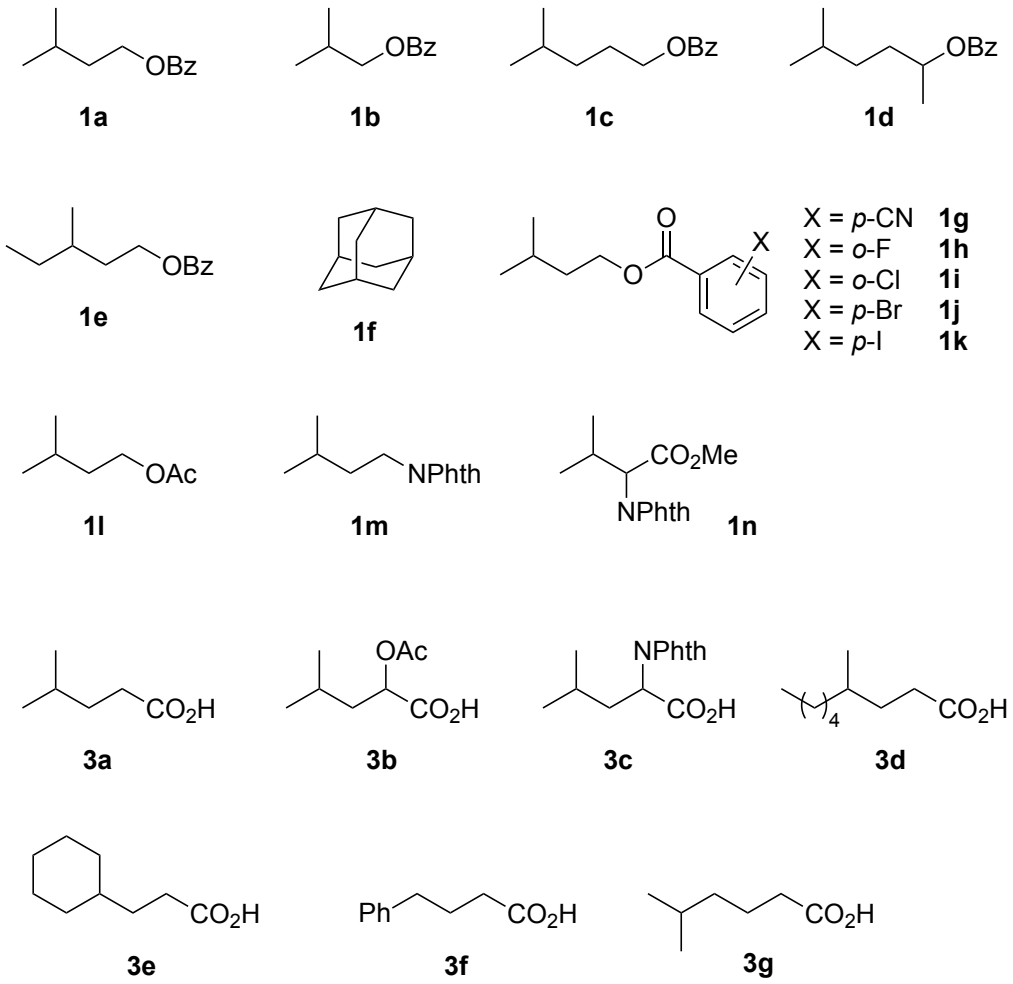
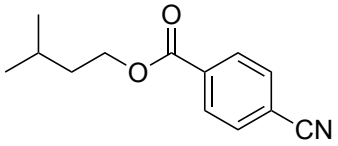


Figure S1. List of substrates

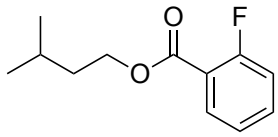
3. Preparation of starting materials

3-methylbutyl 4-cyanobenzoate (1g)



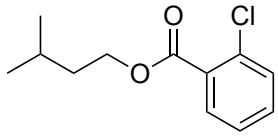
According to the reported procedure,[1] the reaction using DMAP (135 mg, 1.10 mmol), CH_2Cl_2 (25 mL), Et_3N (759 mg, 7.50 mmol), 3-methylbutan-1-ol (489 mg, 5.54 mmol), and 4-cyanobenzoyl chloride (992 mg, 5.99 mmol) was conducted. Purification by flash column chromatography on silica gel (hexane/EtOAc = 85:15) gave **1g** as a colorless oil (923 mg, 77% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.14 (d, J = 8.0 Hz, 2H), 7.75 (d, J = 8.0 Hz, 2H), 4.39 (t, J = 6.8 Hz, 2H), 1.85–1.71 (m, 1H), 1.68 (dt, J = 6.8, 6.8 Hz, 2H), 0.98 (d, J = 6.8 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 165.0, 134.3, 132.2, 130.0, 118.0, 116.3, 64.5, 37.2, 25.2, 22.5; IR (ATR) 2959, 2232, 1722, 1271 cm^{-1}; HRMS: (CI) calcd for ($C_{13}H_{16}NO_2$) 218.1181 ([M+H]$^+$), found *m/z* 218.1179

3-methylbutyl 2-fluorobenzoate (1h)



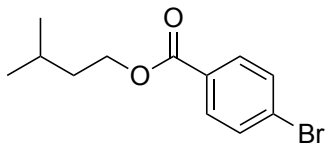
According to the reported procedure,[1] the reaction using DMAP (171 mg, 1.40 mmol), CH_2Cl_2 (35 mL), Et_3N (1.42 g, 14.0 mmol), 2-fluorobenzoic acid (990 mg, 7.07 mmol), *N*,*N*'-diisopropylcarbodiimide (1.70 g, 13.5 mmol), and 3-methylbutan-1-ol (650 mg, 7.37 mmol) was conducted. Purification by flash column chromatography on silica gel (hexane/EtOAc = 9:1) gave **1h** as a yellow oil (376 mg, 24% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.00–7.87 (m, 1H), 7.55–7.45 (m, 1H), 7.26–7.17 (m, 1H), 7.17–7.08 (m, 1H), 4.37 (t, *J* = 6.8 Hz, 2H), 1.86–1.72 (m, 1H), 1.66 (dt, *J* = 6.8, 6.8 Hz, 2H), 0.98 (d, *J* = 6.8 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 164.5 (d, J_{CF} = 3.3 Hz), 161.9 (d, J_{CF} = 257.7 Hz), 134.3 (d, J_{CF} = 9.1 Hz), 132.0, 123.9 (d, J_{CF} = 4.1 Hz), 119.0 (d, J_{CF} = 9.9 Hz), 117.0 (d, J_{CF} = 22.3 Hz), 64.0, 37.3, 25.1, 22.5; ^{19}F NMR: (377 MHz, $CDCl_3$) δ −112.7; IR (ATR) 2959, 1715, 1296 cm^{-1}; HRMS: (CI) calcd for ($C_{12}H_{16}FO_2$) 211.1134 ([M+H]$^+$), found *m/z* 211.1136

3-methylbutyl 2-chlorobenzoate (1i)



According to the reported procedure,[1] the reaction using DMAP (130 mg, 1.06 mmol), CH_2Cl_2 (25 mL), Et_3N (759 mg, 7.50 mmol), 3-methylbutan-1-ol (508 mg, 5.77 mmol), and 2-chlorobenzoyl chloride (1.05 g, 6.00 mmol) was conducted. Purification by flash column chromatography on silica gel (hexane/EtOAc = 9:1) gave **1i** as a colorless oil (1.09 g, 84% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.81 (dd, *J* = 7.6, 1.6 Hz, 1H), 7.48–7.32 (m, 2H), 7.31 (dt, *J* = 7.6, 1.6 Hz, 1H), 4.37 (t, *J* = 6.8 Hz, 2H), 1.85–1.72 (m, 1H), 1.67 (dt, *J* = 6.8, 6.8 Hz, 2H), 0.97 (d, *J* = 6.8 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 165.9, 133.6, 132.4, 131.3, 131.0, 130.5, 126.5, 64.2, 37.3, 25.1, 22.4; IR (ATR) 2958, 1730, 1292, 1249, 746 cm^{-1}; HRMS: (CI) calcd for ($C_{12}H_{16}ClO_2$) 227.0839 ([M+H]$^+$), found *m/z* 227.0843

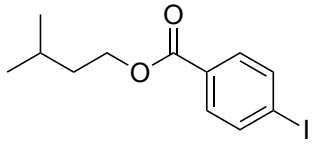
3-methylbutyl 4-bromobenzoate (1j)



According to the reported procedure,[1] the reaction using DMAP (232 mg, 1.90 mmol), CH_2Cl_2 (50 mL), Et_3N (1.52 g, 15.0 mmol), 3-methylbutan-1-ol (928 mg, 10.5 mmol), and 4-bromobenzoyl chloride (2.64 g, 12.0 mmol) was conducted. Purification by flash column chromatography on silica gel (hexane/EtOAc = 9:1) gave **1j** as a pale yellow oil (785 mg, 28% yield). ^{1}H NMR (400 MHz,

$CDCl_3$) δ 7.90 (d, *J* = 8.8 Hz, 2H), 7.58 (d, *J* = 8.8 Hz, 2H), 4.35 (t, *J* = 6.8 Hz, 2H), 1.85–1.72 (m, 1H), 1.66 (dt, *J* = 6.8, 6.8 Hz, 2H), 0.97 (d, *J* = 6.8 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 165.9, 131.6, 131.0, 129.4, 127.9, 63.9, 37.3, 25.2, 22.5; IR (ATR) 2957, 1721, 1267 cm^{-1}; HRMS: (CI) calcd for ($C_{12}H_{16}BrO_2$) 271.0334 ($[M+H]^+$), found *m/z* 271.0330

3-methylbutyl 4-iodobenzoate (1k)



According to the reported procedure,[1] the reaction using DMAP (137 mg, 1.12 mmol), CH_2Cl_2 (25 mL), Et_3N (759 mg, 7.50 mmol), 3-methylbutan-1-ol (475 mg, 5.39 mmol), and 4-iodobenzoyl chloride (1.59 g, 5.99 mmol) was conducted. Purification by flash column chromatography on silica gel (hexane/EtOAc = 9:1) gave **1k** as a colorless oil (1.61 g, 94% yield). ^1H NMR (400 MHz, $CDCl_3$) δ 7.80 (d, *J* = 8.8 Hz, 2H), 7.74 (d, *J* = 8.8 Hz, 2H), 4.34 (t, *J* = 6.8 Hz, 2H), 1.85–1.72 (m, 1H), 1.66 (dt, *J* = 6.8, 6.8 Hz, 2H), 0.97 (d, *J* = 6.8 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 166.2, 137.7, 131.0, 130.0, 101.0, 63.9, 37.3, 25.2, 22.5; IR (ATR) 2957, 1719, 1279 cm^{-1}; HRMS: (CI) calcd for ($C_{12}H_{16}O_2I$) 319.0195 ($[M+H]^+$), found *m/z* 319.0192

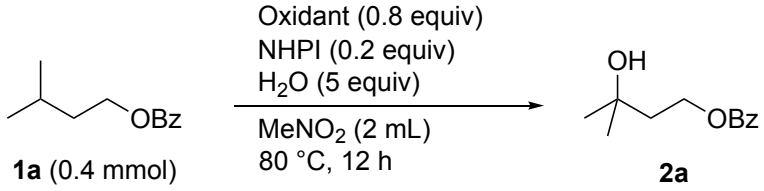
4. Effectof of oxidants and mediators

An oven dried 3 mL reaction vial containing a magnetic stir bar was charged with an oxidant (0.32 mmol), a mediator (0.08 mmol), $MeNO_2$ (2 mL), H_2O (36 μL), and 3-methylbutyl benzoate (0.40 mmol). The vial was purged with N_2 and sealed with a screw cap. The reaction mixture was stirred at 80 °C for 12 h. The reaction was then quenched by passing the solution through a short column (NH silica gel) using EtOAc as the eluent. The solution was concentrated under reduced pressure to give the crude product, which was analyzed by ^1H NMR spectroscopy using bromoform as an internal standard.

Effect of oxidant

The results are summarized in Table S1. The reaction using HIO_3 as well as I_2O_5 proceeded efficiently to provide **2a**, while the use of IBX, NH_4IO_3, and $NaIO_3$ did not give **2a** at all (entries 1–5). Examination of reactions by varying the amount of HIO_3 revealed that the use of 0.8 equivalents of HIO_3 was suitable (entries 1, 6, and 7). No reaction was observed in the absence of HIO_3 (entry 8).

Table S1.



1a (0.4 mmol) **2a**

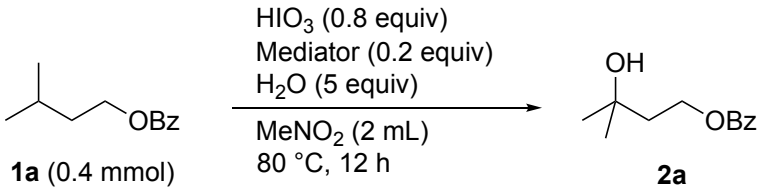
Entry	**Oxidant**	Yield (%)[a]	Recovery (%)[a]
1	HIO_3	62	20
2	I_2O_5 (0.4 equiv.)	55	27
3	IBX	0	>95
4	NH_4IO_3	0	77
5	$NaIO_3$	0	>95
6	HIO_3 (1.2 equiv.)	65	22
7	HIO_3 (0.4 equiv.)	48	45
8	none	0	94

[a] Determined by ^1H NMR analysis.

Effect of mediator

The results are summarized in Table S2. Screening of imide-based reagents, such as NHPI, SuOH, TCNHPI, 4-Nitoro-NHPI, and NDHPI, revealed that NHPI was most effective for this hydroxylation (entries 1–5). No reaction was observed in the absence of NHPI (entry 6).

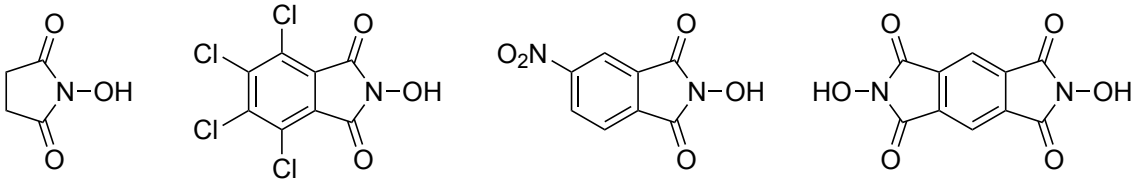
Table S2.



1a (0.4 mmol) **2a**

Entry	**Mediator**	Yield (%)[a]	Recovery (%)[a]
1	NHPI	62	20
2	SuOH	11	88
3	TCNHPI	26	62
4	4-Nitro-NHPI	54	29
5	NDHPI[b]	19	79
6	none	0	93

[a] Determined by ^{1}H NMR analysis. [b] 0.1 equiv.



SuOH TCNHPI 4-Nitro-NHPI NDHPI

5. Stereochemical course of the reaction using (*S*)-1e

An oven dried 3 mL reaction vial containing a magnetic stir bar was charged with HIO_3 (57.1 mg, 0.32 mmol), NHPI (13.0 mg, 0.08 mmol), $MeNO_2$ (2 mL), H_2O (36 μL), and (*S*)-**1e** (81.4 mg, 0.39 mmol). The vial was purged with N_2 and sealed with a screw cap. The reaction mixture was stirred at 80 °C for 12 h. The reaction was then quenched by passing the solution through a short column (NH silica gel) using EtOAc as the eluent. The solution was concentrated under reduced pressure to give the crude product, which was purified by flash column chromatography on NH silica gel (hexane/EtOAc = 8:2) to give **2e** (26.8 mg, 31% yield, 0% ee, HPLC analysis (CHIRALPAK AS-H; 0.5 mL/min; *i*-PrOH/*n*-hexane 1:99; λ = 254 nm)).

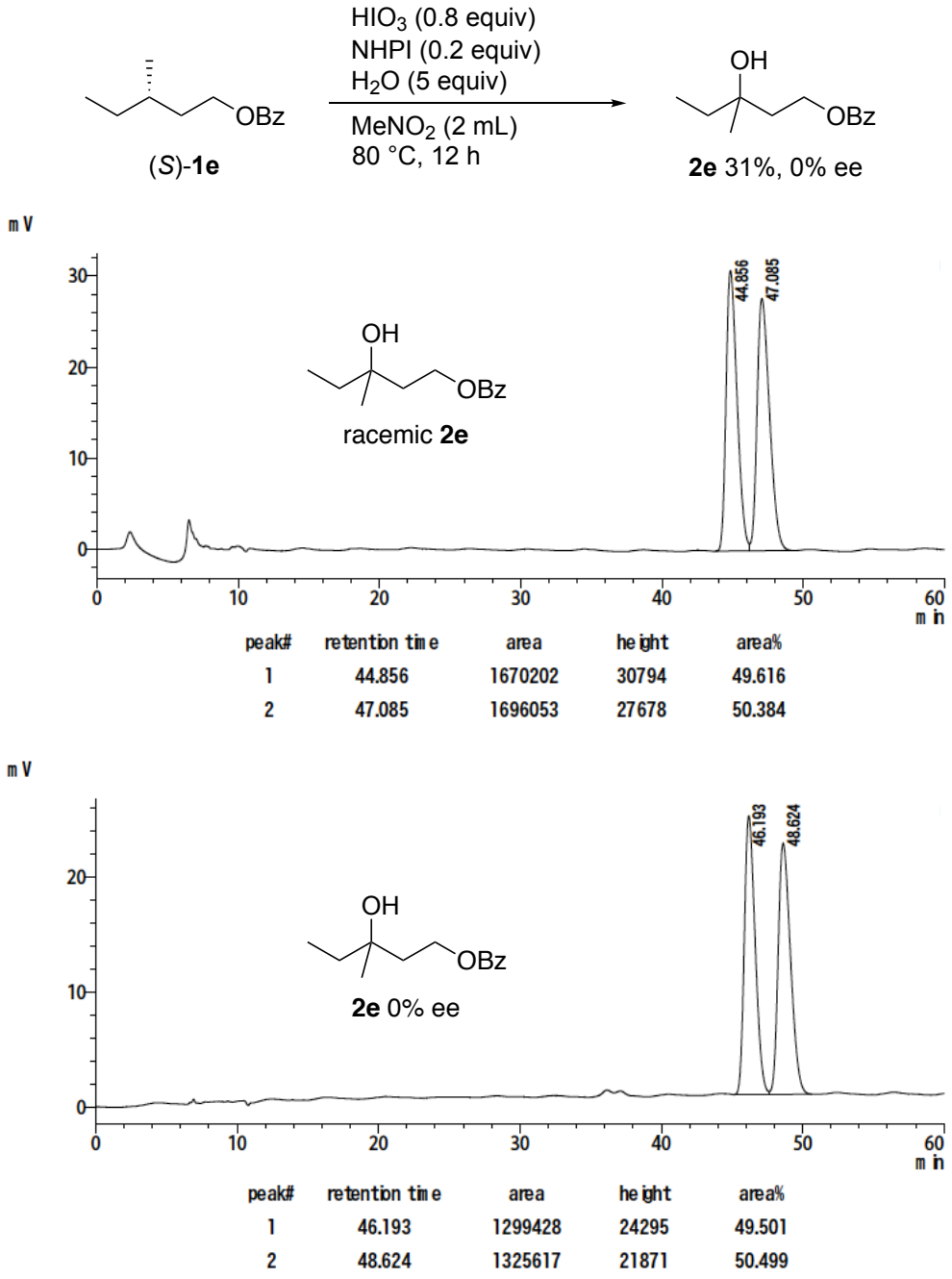


Figure S2. HPLC charts of racemic **2e** and **2e** generated from (*S*)-**1e**.

6. Reaction of alkyl iodide 5 leading to alcohol 2a

An oven dried reaction flask containing a magnetic stir bar was charged with iodide **5** (0.40 mmol), $MeNO_2$ (2 mL), H_2O (36 μL), and HIO_3 (0.32 mmol). The flask was purged with N_2. The reaction mixture was stirred under the indicated reaction conditions. The reaction was then quenched by $Na_2S_2O_3$ aq. (1 M, 10 mL). The mixture was extracted with diethyl ether (3 x 10 mL), and the combined organic layers were dried over Na_2SO_4. The solution was concentrated under reduced pressure to give the crude product, which was analyzed by ^1H NMR using bromoform as an internal standard.

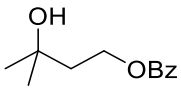
7. C–H hydroxylation: typical procedure and product data

Typical procedure: An oven dried 3 mL reaction vial containing a magnetic stir bar was charged with HIO_3 (0.32 mmol), NHPI (0.08 mmol), $MeNO_2$ (2 mL), H_2O (36 μL), and substrates (0.40 mmol). The vial was purged with N_2 and sealed with a screw cap. The reaction mixture was stirred at 80 °C for the indicated time. The reaction was then quenched by passing the solution through a short column (NH silica gel) using EtOAc as the eluent. The solution was concentrated under reduced pressure to give the crude product, which was analyzed by ^1H NMR spectroscopy using bromoform as an internal standard. Purification by flash column chromatography on NH silica gel (hexane/EtOAc) gave the pure product.

Experimental procedure of gram-scale reaction of 1a: An oven dried reaction flask equipped with a magnetic stir bar and reflux condenser was charged with HIO_3 (1.43 g, 8.13 mmol), NHPI (0.33 g, 2.02 mmol), $MeNO_2$ (50 mL), H_2O (0.9 mL), and 3-methylbutyl benzoate (1.95 g, 10.1 mmol). The flask was purged with N_2, and the reaction mixture was stirred under reflux (thermostat temperature: 100 °C) for 24 h. The reaction was then quenched by $Na_2S_2O_3$ aq. (1 M, 50 mL). The mixture was extracted with diethyl ether (3 x 30 mL), and the combined organic layers were dried over Na_2SO_4. The solution was concentrated under reduced pressure to give the crude product, which was purified by flash column chromatography on NH silica gel (hexane/EtOAc = 8:2) to give the pure product as a colorless oil (1.21 g, 58% yield).

Product data

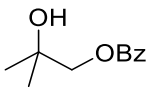
3-hydroxy-3-methylbutyl benzoate (2a)



According to the typical procedure, the reaction using HIO_3 (57.1 mg, 0.32 mmol), NHPI (13.1 mg, 0.08 mmol), $MeNO_2$ (2 mL), H_2O (36 μL), and 3-methylbutyl benzoate (77.2 mg, 0.40 mmol) was conducted at 80 °C for 24 h. Purification by flash column chromatography on NH silica gel (hexane/EtOAc = 8:2) gave the product as a colorless oil (57.0 mg, 68% yield). ^1H NMR (400 MHz, $CDCl_3$) δ 8.04 (d, J = 6.8 Hz, 2H), 7.56 (t, J = 6.8 Hz, 1H), 7.44 (dd, J = 6.8, 6.8 Hz, 2H), 4.51 (t, J = 6.8 Hz, 2H), 1.99 (t, J = 6.8 Hz, 2H), 1.72 (brs, 1H), 1.33 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 166.6, 132.9, 130.2, 129.5, 128.4, 70.1, 61.9, 41.7, 29.7

The analytical data for this compound were in excellent agreement with the reported data.[8]

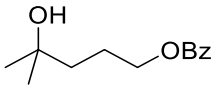
2-hydroxy-2-methylpropyl benzoate (2b)



According to the typical procedure, the reaction using HIO_3 (56.1 mg, 0.32 mmol), NHPI (13.0 mg, 0.08 mmol), $MeNO_2$ (2 mL), H_2O (36 μL), and 2-methylpropyl benzoate (70.5 mg, 0.40 mmol) was conducted at 80 °C for 24 h. Purification by flash column chromatography on NH silica gel

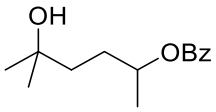
(hexane/EtOAc = 8:2) gave the product as a colorless oil (42.1 mg, 55% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.07 (d, *J* = 7.2 Hz, 2H), 7.57 (t, *J* = 7.2 Hz, 1H), 7.46 (dd, *J* = 7.2, 7.2 Hz, 2H), 4.22 (s, 2H), 2.06 (brs, 1H), 1.35 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 166.5, 133.1, 129.9, 129.6, 128.4, 72.5, 70.1, 26.2; IR (ATR) 3447, 2976, 1717, 1273 cm^{-1}; HRMS: (CI) calcd for ($C_{11}H_{15}O_3$) 195.1021 ([M+H]$^+$), found *m/z* 195.1023

4-hydroxy-4-methylpentyl benzoate (2c)



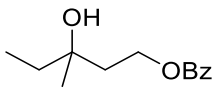
According to the typical procedure, the reaction using HIO_3 (56.2 mg, 0.32 mmol), NHPI (13.1 mg, 0.08 mmol), $MeNO_2$ (2 mL), H_2O (36 μL), and 4-methylpentyl benzoate (81.8 mg, 0.40 mmol) was conducted at 80 °C for 12 h. Purification by flash column chromatography on NH silica gel (hexane/EtOAc = 8:2) gave the product as a colorless oil (55.5 mg, 63% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.05 (d, *J* = 7.2 Hz, 2H), 7.56 (t, *J* = 7.2 Hz, 1H), 7.44 (dd, *J* = 7.2, 7.2 Hz, 2H), 4.35 (t, *J* = 6.8 Hz, 2H), 1.91–1.81 (m, 2H), 1.65–1.55 (m, 2H), 1.36 (brs, 1H), 1.27 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 166.6, 132.9, 130.3, 129.5, 128.3, 70.6, 65.3, 40.0, 29.3, 23.9
The analytical data for this compound were in excellent agreement with the reported data.[8]

5-hydroxy-5-methylhexan-2-yl benzoate (2d)



According to the typical procedure, the reaction using HIO_3 (56.4 mg, 0.32 mmol), NHPI (13.2 mg, 0.08 mmol), $MeNO_2$ (2 mL), H_2O (36 μL), and 5-methylhexan-2-yl benzoate (87.8 mg, 0.40 mmol) was conducted at 80 °C for 12 h. Purification by flash column chromatography on NH silica gel (hexane/EtOAc = 8:2) gave the product as a colorless oil (40.5 mg, 43% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.04 (d, *J* = 7.6 Hz, 2H), 7.56 (t, *J* = 7.6 Hz 1H), 7.44 (dd, *J* = 7.6, 7.6 Hz, 2H), 5.17 (ddq, *J* = 6.4, 6.4, 6.4 Hz, 1H), 1.90–1.67 (m, 2H), 1.66–1.42 (m, 3H), 1.35 (d, *J* = 6.4 Hz, 3H), 1.26 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 166.2, 132.8, 130.7, 129.5, 128.3, 71.9, 70.6, 39.3, 30.9, 29.4, 29.2, 20.1; IR (ATR) 3446, 2972, 1715, 1275, 1113 cm^{-1}; HRMS: (CI) calcd for ($C_{14}H_{21}O_3$) 237.1491 ([M+H]$^+$), found *m/z* 237.1494

3-hydroxy-3-methylpentyl benzoate (2e)

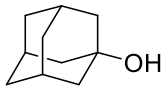


According to the typical procedure, the reaction using HIO_3 (56.3 mg, 0.32 mmol), NHPI (13.1 mg, 0.08 mmol), $MeNO_2$ (2 mL), H_2O (36 μL), and 3-methylpentyl benzoate (82.6 mg, 0.40 mmol) was conducted at 80 °C for 12 h. Purification by flash column chromatography on NH silica gel (hexane/EtOAc = 8:2) gave the product as a colorless oil (25.7 mg, 29% yield). ^{1}H NMR (400 MHz,

$CDCl_3$) δ 8.03 (d, *J* = 8.0 Hz, 2H), 7.54 (t, *J* = 8.0 Hz, 1H), 7.44 (dd, *J* = 8.0, 8.0 Hz, 2H), 4.50 (dd, *J* = 6.8, 6.8 Hz, 2H), 2.01–1.89 (m, 2H), 1.72 (brs, 1H), 1.58 (q, *J* = 8.0 Hz, 2H), 1.26 (s, 3H), 0.95 (t, *J* = 8.0 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 166.6, 132.9, 130.2, 129.5, 128.3, 72.1, 61.8, 39.3, 35.0, 26.5, 8.2

The analytical data for this compound were in excellent agreement with the reported data.[8]

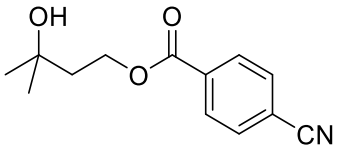
1-adamantanol (2f)



According to the typical procedure, the reaction using HIO_3 (56.2 mg, 0.32 mmol), NHPI (13.0 mg, 0.08 mmol), $MeNO_2$ (1 mL), 1,2-dichloroethane (1 mL), H_2O (36 μL), and adamantane (54.3 mg, 0.40 mmol) was conducted at 80 °C for 12 h. Purification by flash column chromatography on NH silica gel (hexane/EtOAc = 8:2) gave the product as a white solid (27.4 mg, 45% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 2.20–2.10 (m, 3H), 1.80–1.79 (m, 6H), 1.79–1.51 (m, 6H), 1.47 (brs, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 68.2, 45.3, 36.0, 30.7

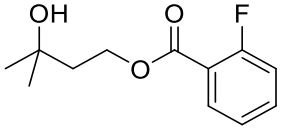
The analytical data for this compound were in excellent agreement with the reported data.[9]

3-hydroxy-3-methylbutyl 4-cyanobenzoate (2g)



According to the typical procedure, the reaction using HIO_3 (56.1 mg, 0.32 mmol), NHPI (13.0 mg, 0.08 mmol), $MeNO_2$ (2 mL), H_2O (36 μL), and 3-methylbutyl 4-cyanobenzoate (85.2 mg, 0.39 mmol) was conducted at 80 °C for 24 h. Purification by flash column chromatography on NH silica gel (hexane/EtOAc = 3:1) gave the product as a pale yellow oil (54.5 mg, 60% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.13 (d, *J* = 8.4 Hz, 2H), 7.75 (d, *J* = 8.4 Hz, 2H), 4.54 (t, *J* = 6.8 Hz, 2H), 2.00 (t, *J* = 6.8 Hz, 2H), 1.34 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 165.0, 134.0, 132.2, 130.0, 117.9, 116.3, 70.0, 62.7, 41.5, 29.7*;* IR (ATR) 3504, 2972, 2361, 2232, 1720, 1275 cm^{-1}; HRMS: (CI) calcd for ($C_{13}H_{16}NO_3$) 234.1130 ($[M+H]^+$), found *m/z* 234.1133

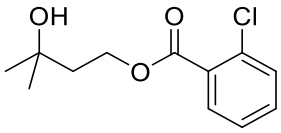
3-hydroxy-3-methylbutyl-2-fluorobenzoate (2h)



According to the typical procedure, the reaction using HIO_3 (57.4 mg, 0.33 mmol), NHPI (13.1 mg, 0.08 mmol), $MeNO_2$ (2 mL), H_2O (36 μL), and 3-methylbutyl 2-fluorobenzoate (85.6 mg, 0.41 mmol) was conducted at 80 °C for 30 h. Purification by flash column chromatography on NH silica gel (hexane/EtOAc = 8:2) gave the product as a pale yellow oil (47.4 mg, 52% yield). ^{1}H NMR

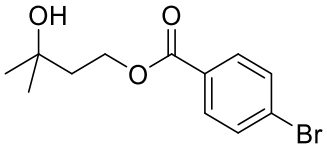
(400 MHz, $CDCl_3$) δ 7.98–7.90 (m, 1H), 7.57–7.49 (m, 1H), 7.25–7.16 (m, 1H), 7.16–7.09 (m, 1H), 4.54 (t, J = 6.8 Hz, 2H), 2.00 (t, J = 6.8 Hz, 2H), 1.90 (brs, 1H), 1.33 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 164.4 (d, J_{CF} = 3.3 Hz), 161.8 (d, J_{CF} = 257.7 Hz), 134.5 (d, J_{CF} = 9.1 Hz), 132.2, 124.0 (d, J_{CF} = 3.3 Hz), 118.6 (d, J_{CF} = 9.8 Hz), 117.0 (d, J_{CF} = 23.1 Hz), 69.9, 62.4, 41.4, 29.5; ^{19}F NMR: (377 MHz, CDCl3) δ −112.5; IR (ATR) 3493, 2974, 1712, 1612 cm^{-1}; HRMS: (CI) calcd for ($C_{12}H_{16}FO_3$) 227.1083 ([M+H]$^+$), found *m/z* 227.1082

3-hydroxy-3-methylbutyl 2-chlorobenzoate (2i)



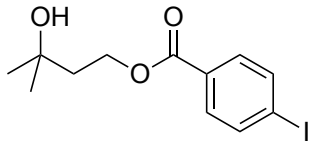
According to the typical procedure, the reaction using HIO_3 (56.8 mg, 0.32 mmol), NHPI (13.1 mg, 0.08 mmol), $MeNO_2$ (2 mL), H_2O (36 μL), and 3-methylbutyl 2-chlorobenzoate (90.7 mg, 0.40 mmol) was conducted at 80 °C for 24 h. Purification by flash column chromatography on NH silica gel (hexane/EtOAc = 8:2) gave the product as a colorless oil (57.9 mg, 60% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.81 (dd, J = 7.6, 1.6 Hz, 1H), 7.48–7.38 (m, 2H), 7.32 (td, J = 7.6, 1.6 Hz, 1H), 4.53 (t, J = 6.8 Hz, 2H), 2.00 (t, J = 6.8 Hz, 2H), 1.62 (brs, 1H), 1.32 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 165.7, 133.5, 132.5, 131.3, 131.0, 130.2, 126.6, 70.0, 62.6, 41.5, 29.7; IR (ATR) 3449, 2972, 1730, 1715, 1250 cm^{-1}; HRMS: (CI) calcd for ($C_{12}H_{16}ClO_3$) 243.0788 ([M+H]$^+$), found *m/z* 243.0788

3-hydroxy-3-methylbutyl 4-bromobenzoate (2j)



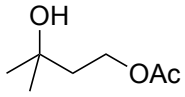
According to the typical procedure, the reaction using HIO_3 (56.3 mg, 0.32 mmol), NHPI (13.1 mg, 0.08 mmol), $MeNO_2$ (2 mL), H_2O (36 μL), and 3-methylbutyl 4-bromobenzoate (107.3 mg, 0.40 mmol) was conducted at 80 °C for 24 h. Purification by flash column chromatography on NH silica gel (hexane/EtOAc = 8:2) gave the product as a colorless oil (66.4 mg, 59% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.89 (d, J = 8.8 Hz, 2H), 7.58 (d, J = 8.8 Hz, 2H), 4.50 (t, J = 6.8 Hz, 2H), 1.98 (t, J = 6.8 Hz, 2H), 1.49 (brs, 1H), 1.32 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 165.9, 131.7, 131.0, 129.1, 128.0, 70.0, 62.2, 41.6, 29.7; IR (ATR) 3416, 2972, 1714, 1591, 1271 cm^{-1}; HRMS: (CI) calcd for ($C_{12}H_{16}BrO_3$) 287.0283 ([M+H]$^+$), found *m/z* 287.0282

3-hydroxy-3-methylbutyl 4-iodobenzoate (2k)



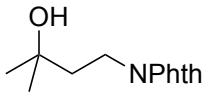
According to the typical procedure, the reaction using HIO_3 (56.8 mg, 0.32 mmol), NHPI (13.0 mg, 0.08 mmol), $MeNO_2$ (2 mL), H_2O (36 μL), and 3-methylbutyl 4-iodobenzoate (125.4 mg, 0.39 mmol) was conducted at 80 °C for 24 h. Purification by flash column chromatography on NH silica gel (hexane/EtOAc = 8:2) gave the product as a colorless oil (76.8 mg, 58% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.80 (d, *J* = 8.8Hz, 2H), 7.72 (d, *J* = 8.8 Hz, 2H), 4.49 (t, *J* = 6.8 Hz, 2H), 1.98 (t, *J* = 6.8 Hz, 2H), 1.32 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 166.1, 137.7, 130.9, 129.7, 100.7, 70.0, 62.2, 41.6, 29.7; IR (ATR) 3429, 1714, 1585, 1269 cm^{-1}; HRMS: (CI) calcd for ($C_{12}H_{16}O_3I$) 335.0144 ($[M+H]^+$), found *m/z* 335.0143

3-hydroxy-3-methylbutyl acetate (2l)



According to the typical procedure, the reaction using HIO_3 (57.3 mg, 0.33 mmol), NHPI (13.1 mg, 0.08 mmol), $MeNO_2$ (2 mL), H_2O (36 μL), and 3-methylbutyl acetate (51.2 mg, 0.39 mmol) was conducted at 80 °C for 12 h. Purification by flash column chromatography on silica gel (hexane/EtOAc = 8:2) gave the product as a colorless oil (25.1 mg, 44% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 4.25 (t, *J* = 6.8 Hz, 2H), 2.06 (s, 3H), 1.85 (t, *J* = 6.8 Hz, 2H), 1.27 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 171.1, 70.0, 61.5, 41.5, 29.6, 21.1; IR (ATR) 3439, 2972, 1738, 1368, 1238 cm^{-1}; HRMS: (CI) calcd for ($C_7H_{15}O_3$) 147.1021 ($[M+H]^+$), found *m/z* 147.1023

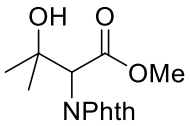
***N*-(3-hydroxy-3-methylbutyl)phthalimide (2m)**



According to the typical procedure, the reaction using HIO_3 (56.3 mg, 0.32 mmol), NHPI (13.2 mg, 0.08 mmol), $MeNO_2$ (2 mL), H_2O (36 μL), and *N*-(3-methylbutyl)phthalimide (91.9 mg, 0.42 mmol) was conducted at 80 °C for 12 h. Purification by flash column chromatography on NH silica gel (hexane/EtOAc = 8:2) gave the product as a colorless oil (56.1 mg, 57% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.84 (dd, *J* = 5.6, 3.6 Hz, 2H), 7.71 (dd, *J* = 5.6, 3.6 Hz, 2H), 3.90–3.79 (m, 2H), 2.05 (brs, 1H), 1.90–1.82 (m, 2H), 1.31 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 168.4, 133.8, 132.1, 123.1, 69.8, 41.4, 34.0, 29.3

The analytical data for this compound were in excellent agreement with the reported data.[7]

methyl 3-hydroxy-3-methyl-2-phthalimidobutyrate (2n)



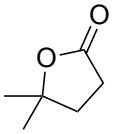
According to the typical procedure, the reaction using HIO_3 (56.7 mg, 0.32 mmol), NHPI (13.1 mg, 0.08 mmol), $MeNO_2$ (2 mL), H_2O (36 μL), and methyl 3-methyl-2-phthalimidobutyrate (110.8 mg, 0.42 mmol) was conducted at 80 °C for 24 h. Purification by flash column chromatography on NH silica gel (hexane/EtOAc = 8:2) gave the product as a colorless oil (21.4 mg, 18% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.91 (dd, *J* = 5.6, 3.2 Hz, 2H), 7.79 (dd, *J* = 5.6, 3.2 Hz, 2H), 4.91 (s, 1H), 4.41 (s, 1H), 3.76 (s, 3H), 1.53 (s, 3H), 1.31 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 168.6, 168.3, 134.5, 131.5, 123.8, 72.1, 61.1, 52.7, 28.0, 27.5

The analytical data for this compound were in excellent agreement with the reported data.[7]

8. Lactonization: typical procedure and product data

Typical procedure: An oven dried 3 mL reaction vial containing a magnetic stir bar was charged with HIO_3 (0.32 mmol), NHPI (0.08 mmol), $MeNO_2$ (2 mL), and substrates (0.40 mmol). The vial was purged with N_2 and sealed with a screw cap. The reaction mixture was stirred at 80 °C for the indicated time. The reaction was then quenched by $Na_2S_2O_3$ aq. (1 M, 10 mL). The mixture was extracted with diethyl ether (3 x 10 mL), and the combined organic layers were dried over Na_2SO_4. The solution was concentrated under reduced pressure to give the crude product, which was analyzed by ^{1}H NMR using bromoform or 1,1,2,2-tetrachloroethane as an internal standard. Purification by flash column chromatography on silica gel (hexane/EtOAc) gave the pure product.

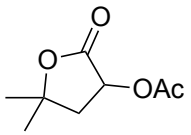
5,5-dimethyldihydrofuran-2(3*H*)-one (4a)



According to the typical procedure, the reaction using HIO_3 (56.0 mg, 0.32 mmol), NHPI (13.2 mg, 0.08 mmol), $MeNO_2$ (2 mL), and 4-methylpentanoic acid (45.9 mg, 0.40 mmol) was conducted at 80 °C for 6 h. Purification by flash column chromatography on silica gel (hexane/EtOAc = 9:1) gave the product as a colorless oil (29.7 mg, 66% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 2.63 (t, *J* = 7.6 Hz, 2H), 2.06 (t, *J* = 7.6 Hz, 2H), 1.44 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 176.6, 84.5, 34.6, 29.3, 27.7

The analytical data for this compound were in excellent agreement with the reported data.[10]

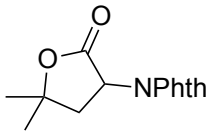
3-acetoxy-5,5-dimethyldihydrofuran-2(3*H*)-one (4b)



According to the typical procedure, the reaction using HIO_3 (84.8 mg, 0.48 mmol), NHPI (13.1 mg, 0.08 mmol), $MeNO_2$ (2 mL), and 2-acetoxy-4-methylpentanoic acid (69.0 mg, 0.40 mmol) was conducted at 80 °C for 24 h. Purification by flash column chromatography on silica gel (hexane/EtOAc = 92:8) gave the product as a colorless oil (36.1 mg, 53% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 5.58 (dd, *J* = 8.8, 8.8 Hz, 1H), 2.61 (dd, *J* = 12.4, 8.8 Hz, 1H), 2.17 (s, 3H), 2.07 (dd, *J* = 12.4, 8.8 Hz, 1H), 1.53 (s, 3H), 1.46 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 172.1, 169.8, 82.2, 69.1, 41.0, 29.0, 27.8, 20.6

The analytical data for this compound were in excellent agreement with the reported data.[11]

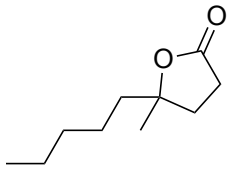
5,5-dimethyl-3-phthalimidodihydrofuran-2(3*H*)-one (4c)



According to the typical procedure, the reaction using HIO_3 (84.6 mg, 0.48 mmol), NHPI (13.1 mg, 0.08 mmol), $MeNO_2$ (2 mL), and *N*-phthaloyl leucine (104.0 mg, 0.40 mmol) was conducted at 80 °C for 24 h. Purification by flash column chromatography on silica gel (hexane/EtOAc = 9:1) gave the product as a white solid (75.7 mg, 73% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.88 (dd, *J* = 5.6, 3.2 Hz, 2H), 7.76 (dd, *J* = 5.6, 3.2 Hz, 2H), 5.24 (dd, *J* = 11.8, 9.6 Hz, 1H), 2.60 (dd, *J* = 11.8, 11.8 Hz, 1H), 2.44 (dd, *J* = 11.8, 9.6 Hz, 1H), 1.64 (s, 3H), 1.52 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 171.5, 166.9, 134.4, 131.6, 123.7, 82.4, 48.5, 38.5, 28.9, 27.5

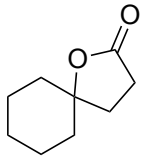
The analytical data for this compound were in excellent agreement with the reported data.[7]

5-methyl-5-pentyldihydrofuran-2(3*H*)-one (4d)



According to the typical procedure, the reaction using I_2O_5 (107.2 mg, 0.32 mmol), NHPI (13.2 mg, 0.08 mmol), $MeNO_2$ (2 mL), and 4-methylnonanoic acid (68.4 mg, 0.40 mmol) was conducted at 80 °C for 12 h. Purification by flash column chromatography on silica gel (hexane/EtOAc = 8:2) gave the product as a colorless oil (40.0 mg, 59% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 2.68–2.50 (m, 2H), 2.15–2.04 (m, 1H), 2.04–1.92 (m, 1H), 1.73–1.60 (m, 2H), 1.42–1.20 (m, 6H), 1.38 (s, 3H), 0.90 (t, *J* = 6.8 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 176.8, 86.9, 40.9, 32.9, 32.0, 29.2, 25.6, 23.5, 22.5, 13.9; IR (ATR) 2954, 2934, 1767 cm^{-1}; HRMS: (FAB) calcd for ($C_{10}H_{19}O_2$) 171.1385 ($[M+H]^+$), found *m/z* 171.1388

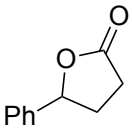
1-oxaspiro[4.5]decan-2-one (4e)



According to the typical procedure, the reaction using I_2O_5 (55.2 mg, 0.17 mmol), NHPI (14.1 mg, 0.09 mmol), $MeNO_2$ (2 mL), and cyclohexanepropanoic acid (67.0 mg, 0.43 mmol) was conducted at 80 °C for 12 h. Purification by gel permeation chromatography (solvent; chloroform) gave the product as a colorless oil (16.4 mg, 23% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 2.59 (t, J = 8.0 Hz, 2H), 2.01 (t, J = 8.0 Hz, 2H), 1.85–1.65 (m, 4H), 1.65–1.45 (m, 5H), 1.45–1.31 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 176.8, 86.4, 36.9, 32.8, 28.6, 24.9, 22.6

The analytical data for this compound were in excellent agreement with the reported data[12,13]

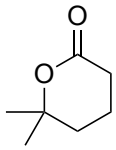
5-phenyldihydrofuran-2(3*H*)-one (4f)



According to the typical procedure, the reaction using HIO_3 (56.6 mg, 0.32 mmol), NHPI (13.1 mg, 0.08 mmol), $MeNO_2$ (2 mL), and 4-phenylbutanoic acid (66.7 mg, 0.41 mmol) was conducted at 60 °C for 8 h. Purification by flash column chromatography on silica gel (hexane/EtOAc = 85:15) gave the product as a colorless oil (24.2 mg, 37% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.45–7.30 (m, 5H), 5.52 (dd, J = 7.8, 6.0 Hz, 1H), 2.73–2.60 (m, 3H), 2.25–2.10 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 176.9, 139.3, 128.7, 128.4, 125.2, 81.2, 30.9, 28.9

The analytical data for this compound were in excellent agreement with the reported data.[14]

6,6-dimethyltetrahydropyran-2-one (4g)



According to the typical procedure, the reaction using HIO_3 (56.4 mg, 0.32 mmol), NHPI (13.1 mg, 0.08 mmol), $MeNO_2$ (2 mL), and 5-methylhexanoic acid (51.7 mg, 0.40 mmol) was conducted at 80 °C for 12 h. Purification by flash column chromatography on silica gel (hexane/EtOAc = 92:8) gave the product as a colorless oil (23.2 mg, 45% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 2.49 (t, J = 6.8 Hz, 2H), 1.98–1.84 (m, 2H), 1.82–1.70 (m, 2H), 1.41 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 171.2, 82.1, 33.8, 29.0, 28.6, 16.7

The analytical data for this compound were in excellent agreement with the reported data.[15]

9. References

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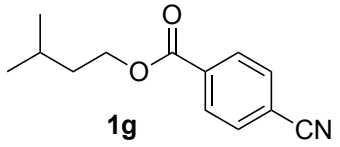
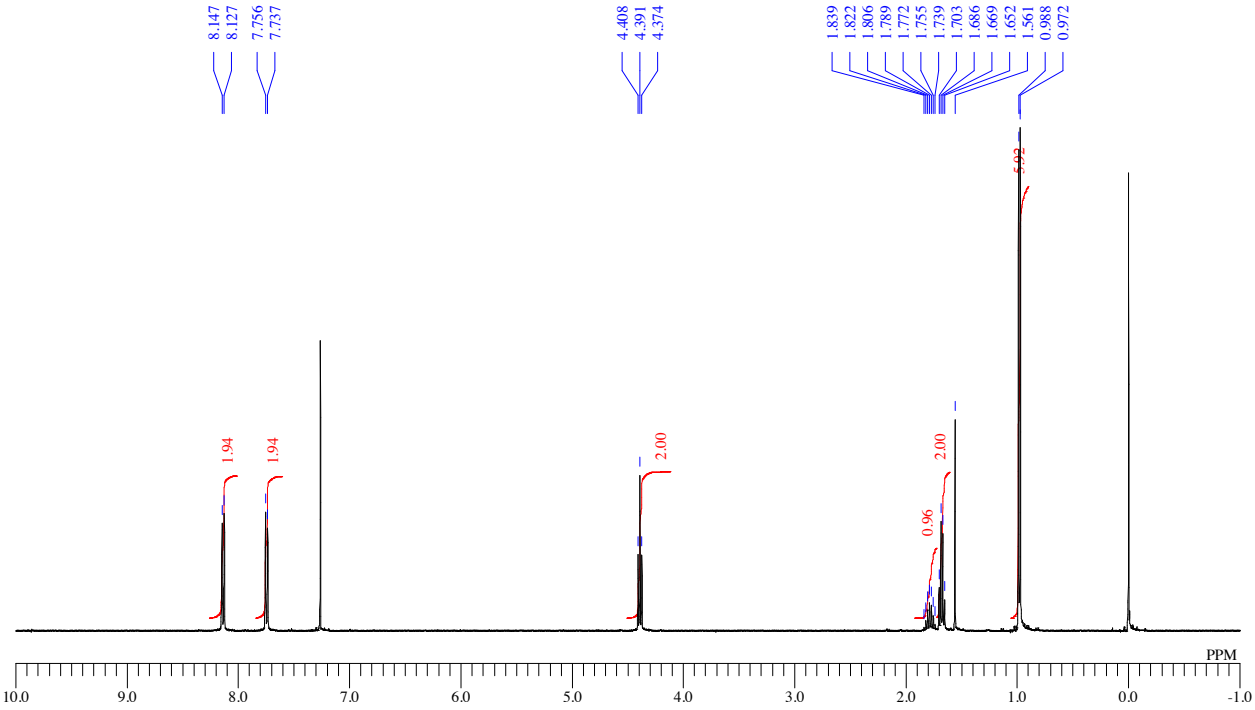
10. NMR spectra

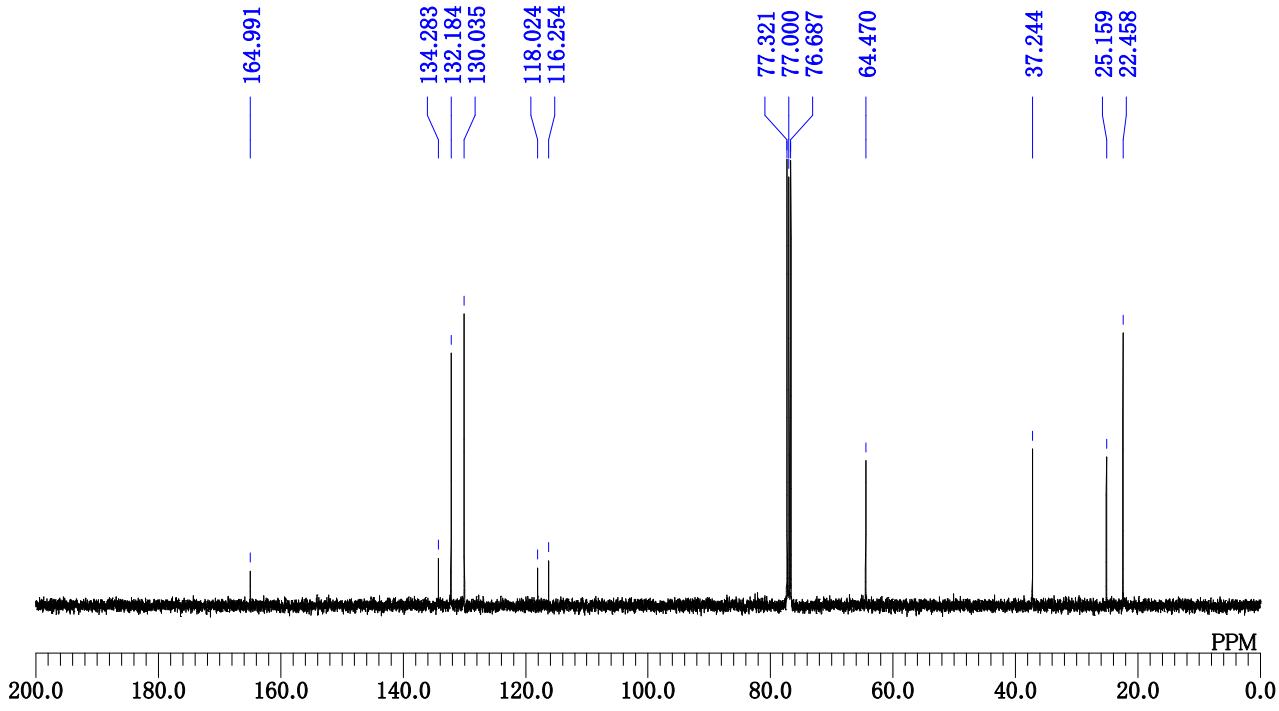


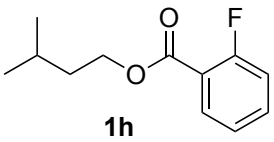
1g

^{1}H NMR: (400 MHz, $CDCl_3$)



^{13}C NMR: (100 MHz, $CDCl_3$)





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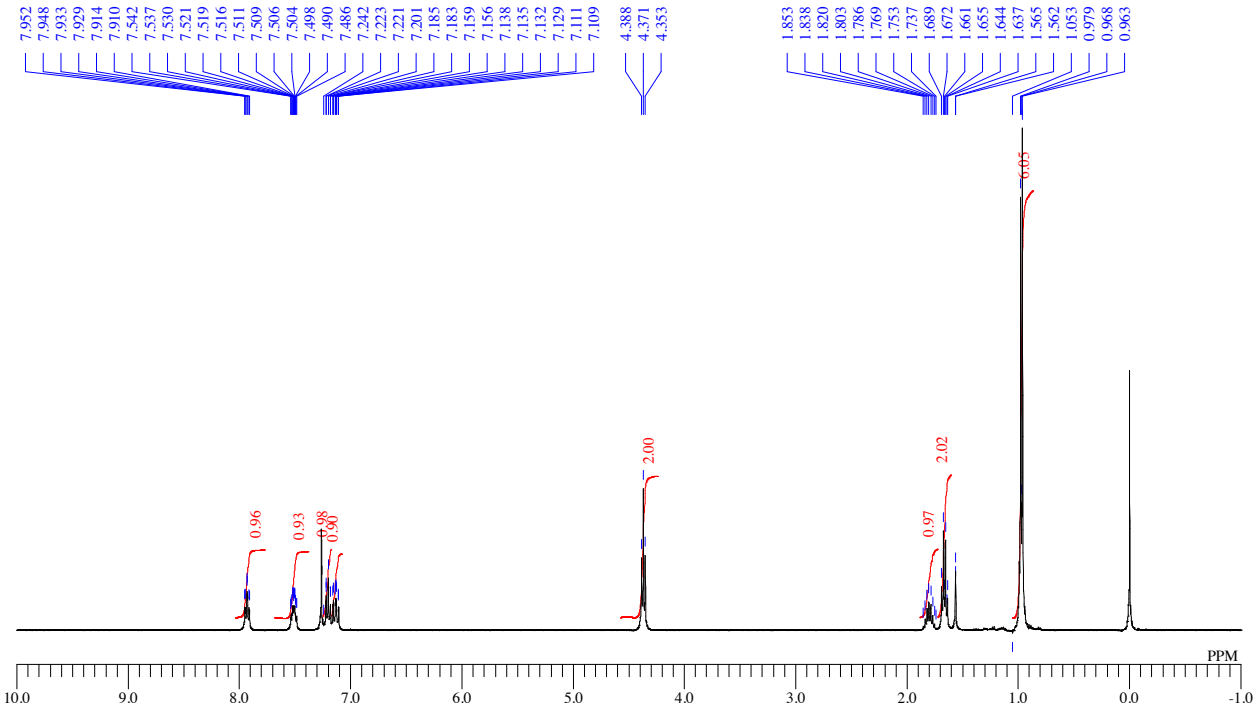
^{1}H NMR: (400 MHz, $CDCl_3$)



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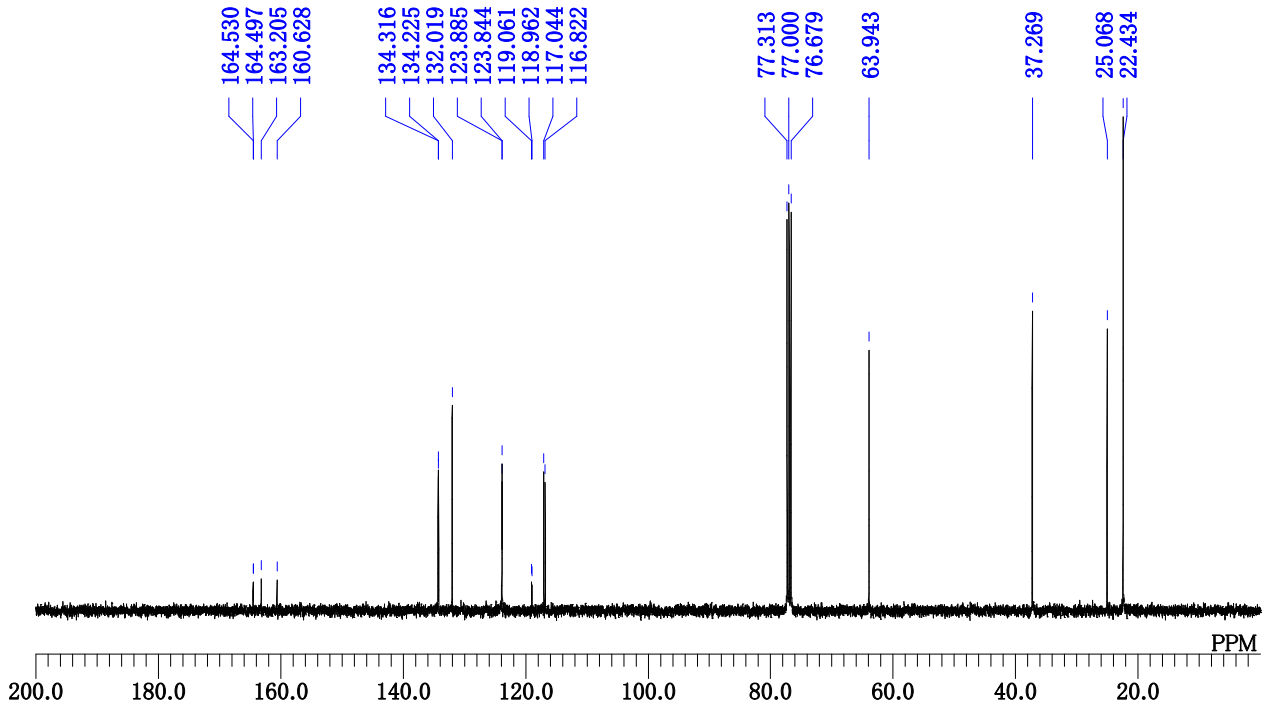
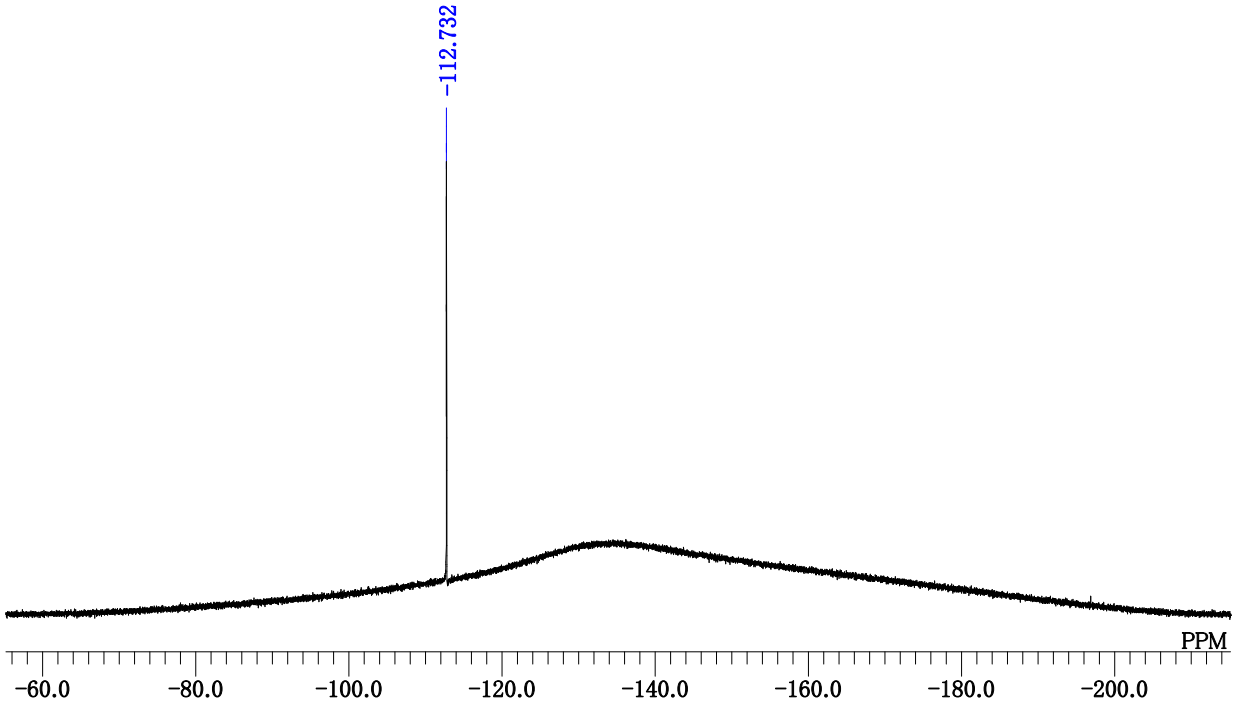
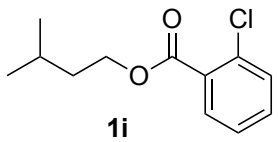


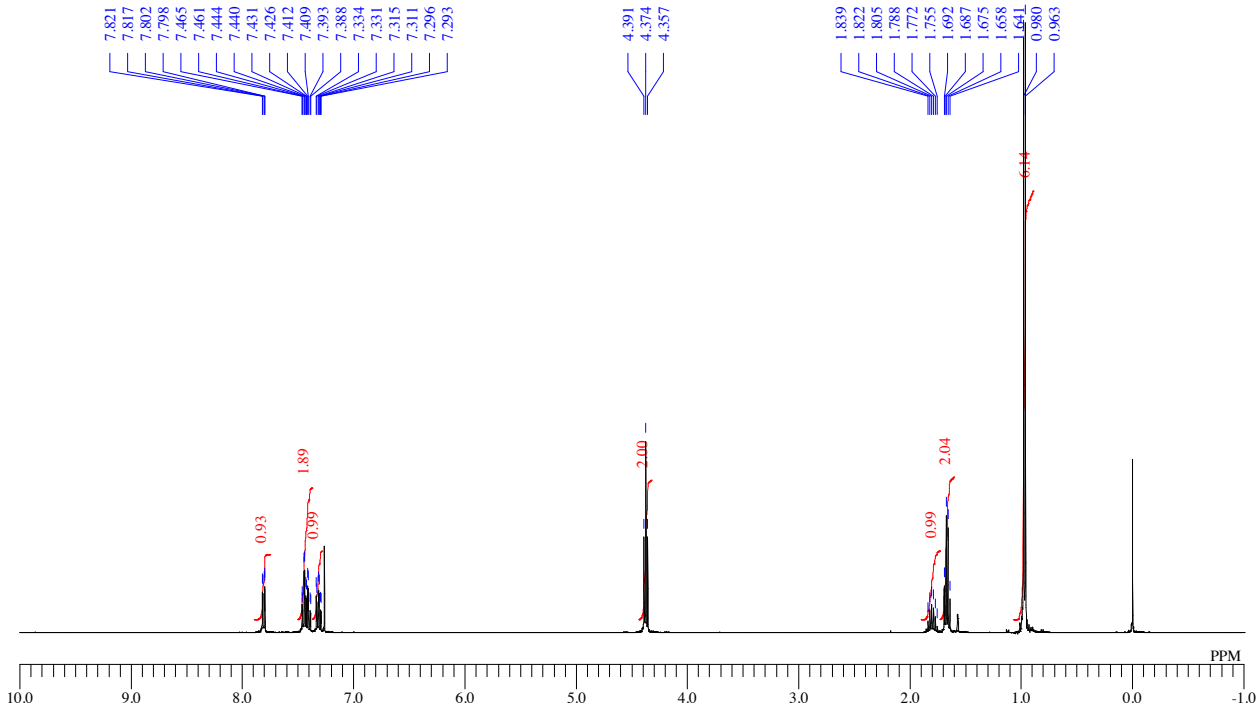
^{19}F NMR: (377 MHz, $CDCl_3$)



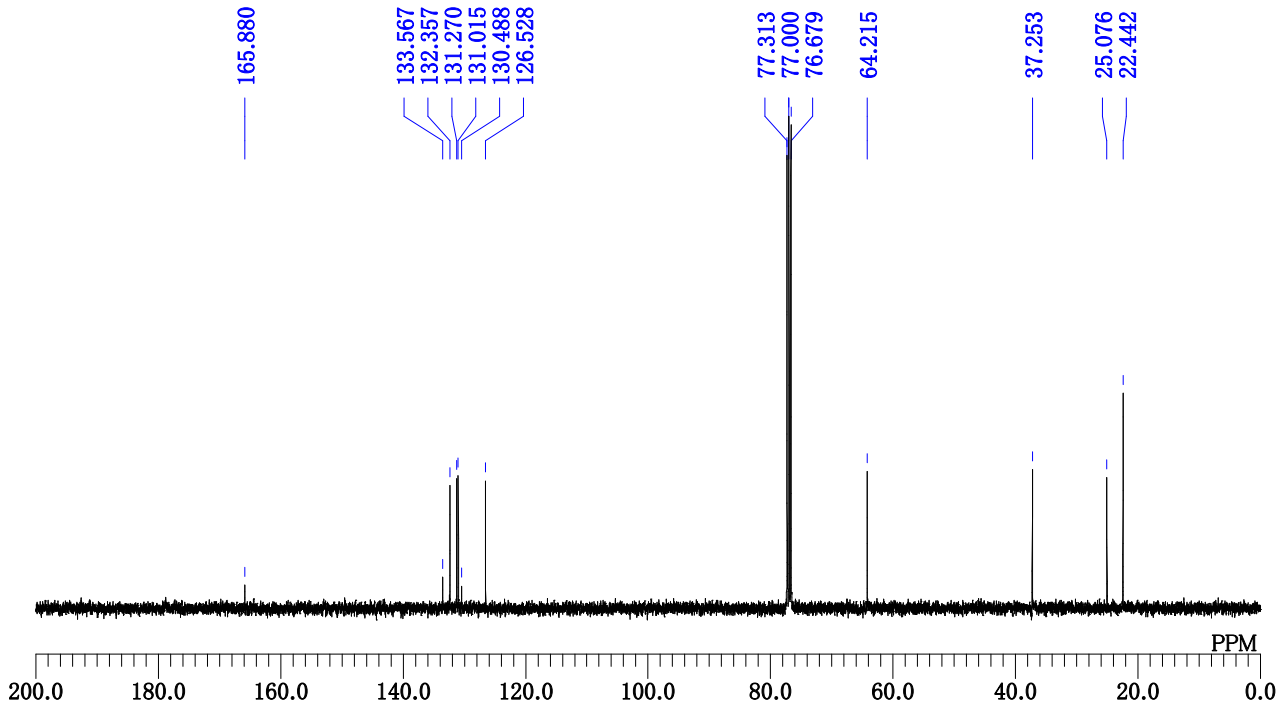


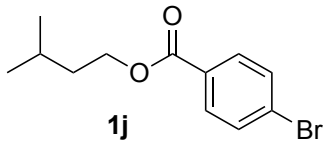
1i

^{1}H NMR: (400 MHz, $CDCl_3$)



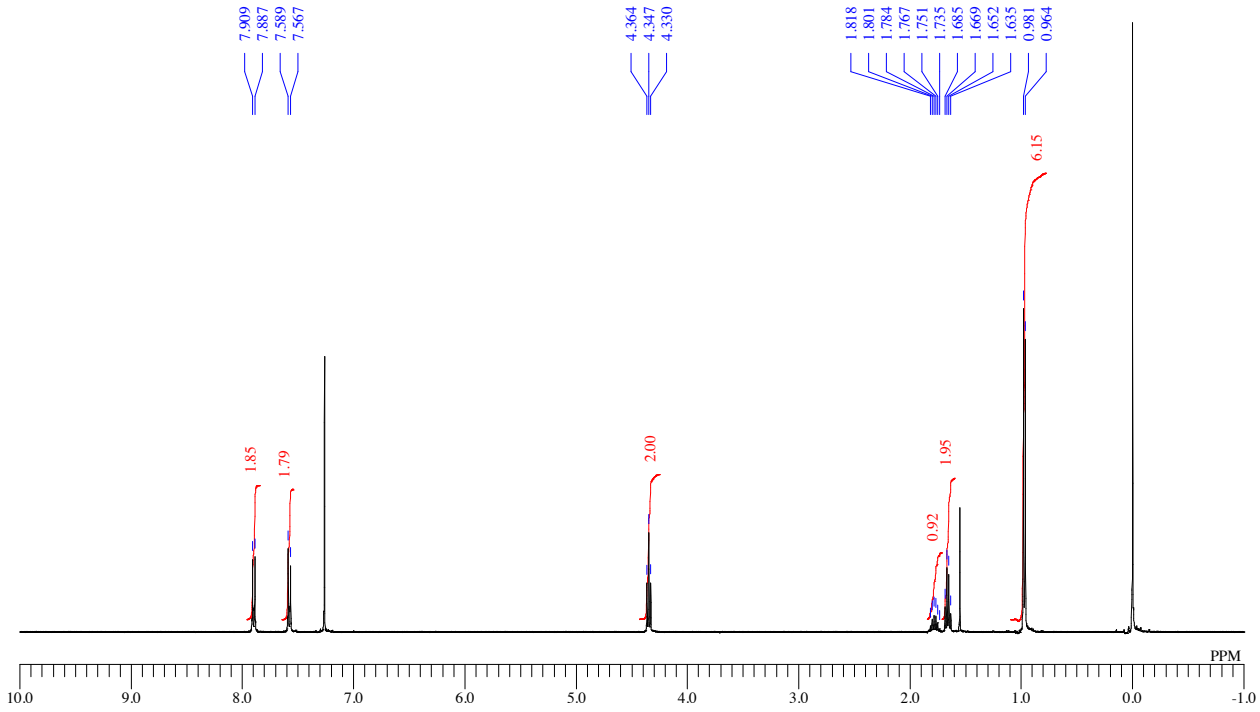
^{13}C NMR: (100 MHz, $CDCl_3$)





1j

^{1}H NMR: (400 MHz, $CDCl_3$)



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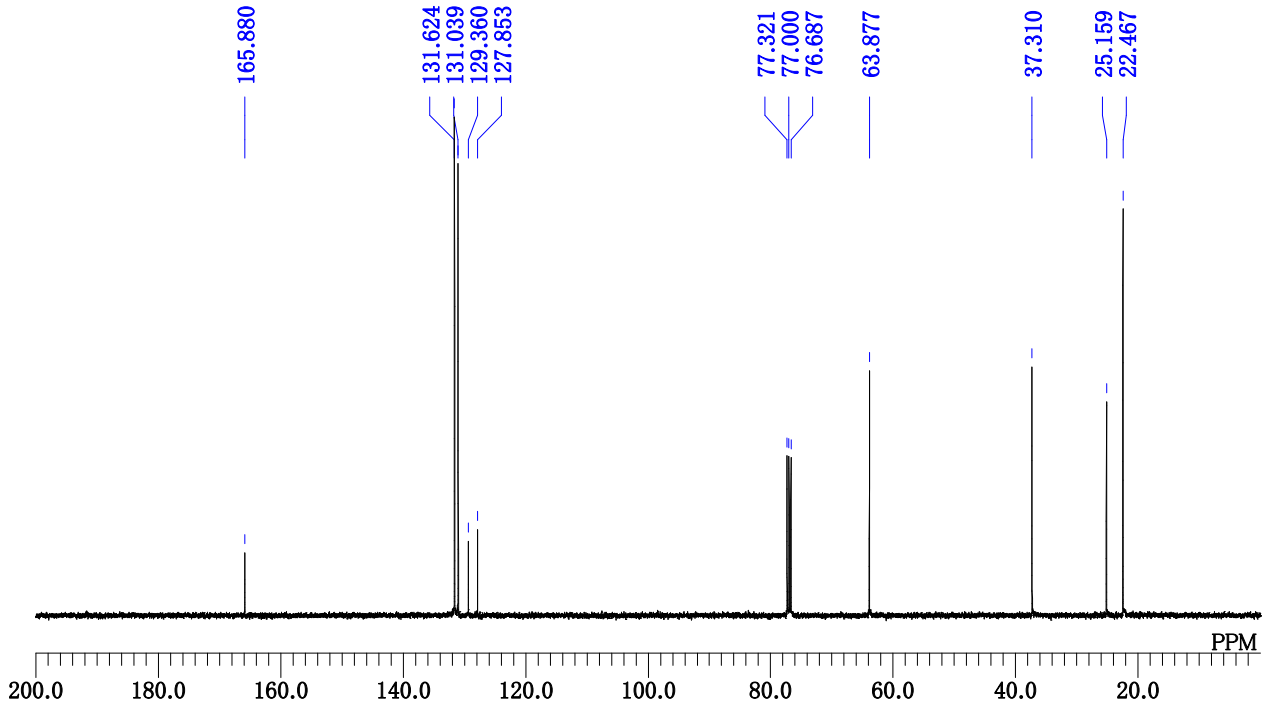

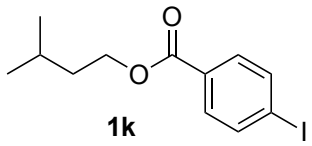

1k

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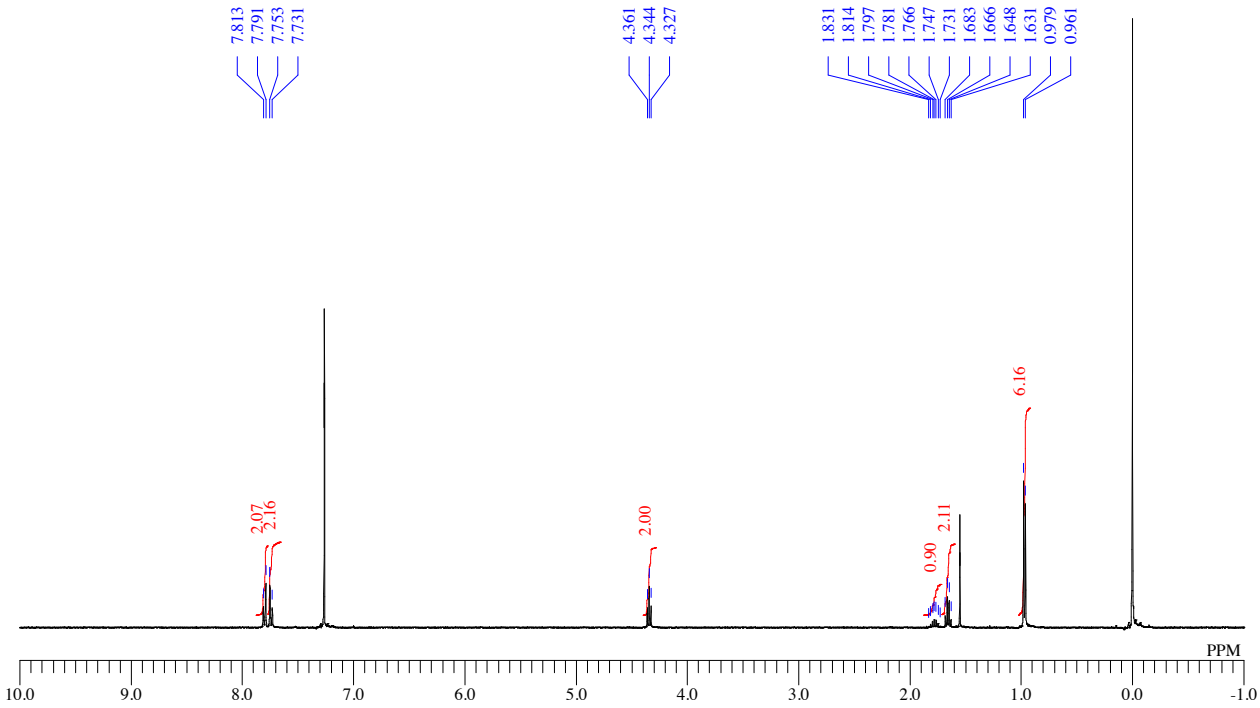

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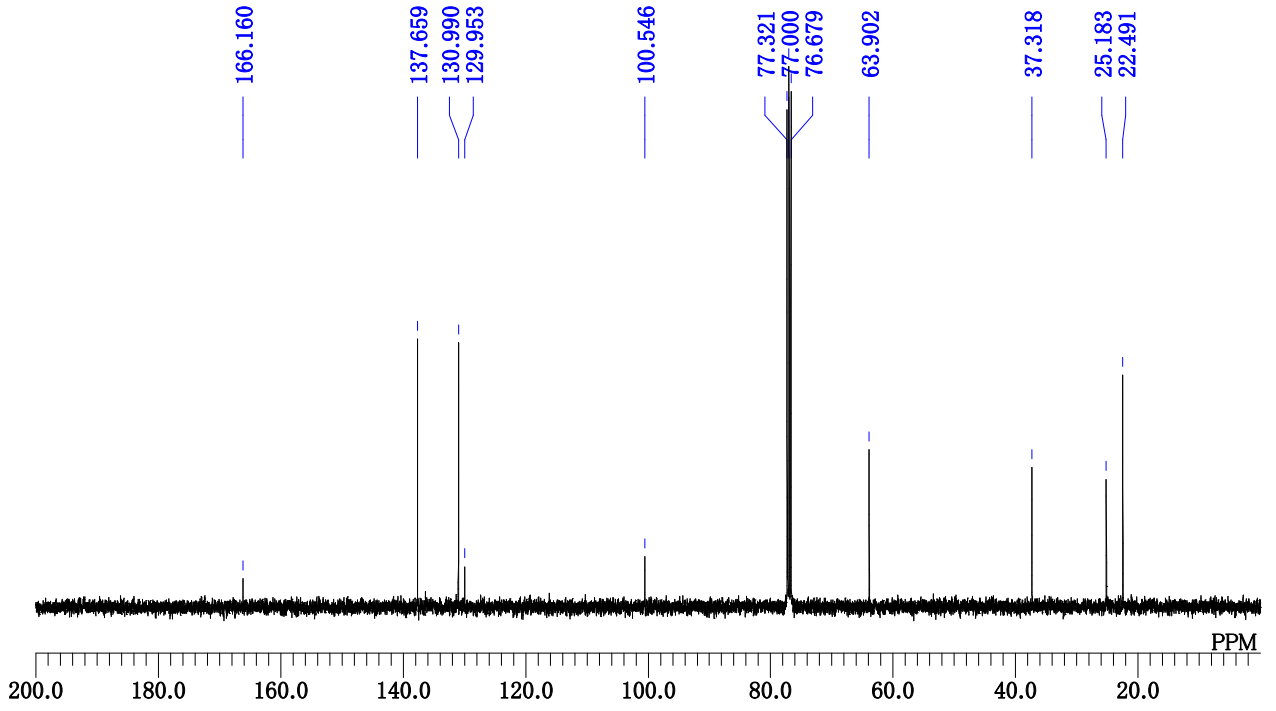

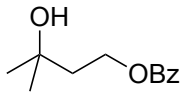

2a

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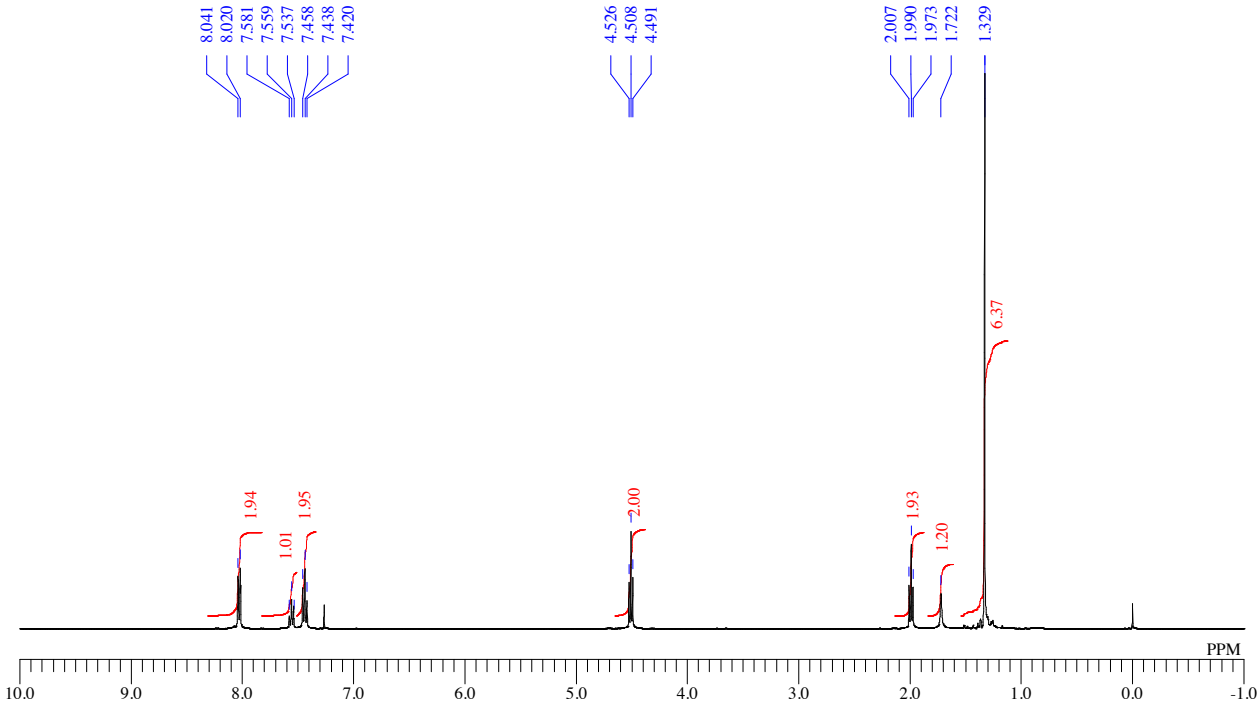
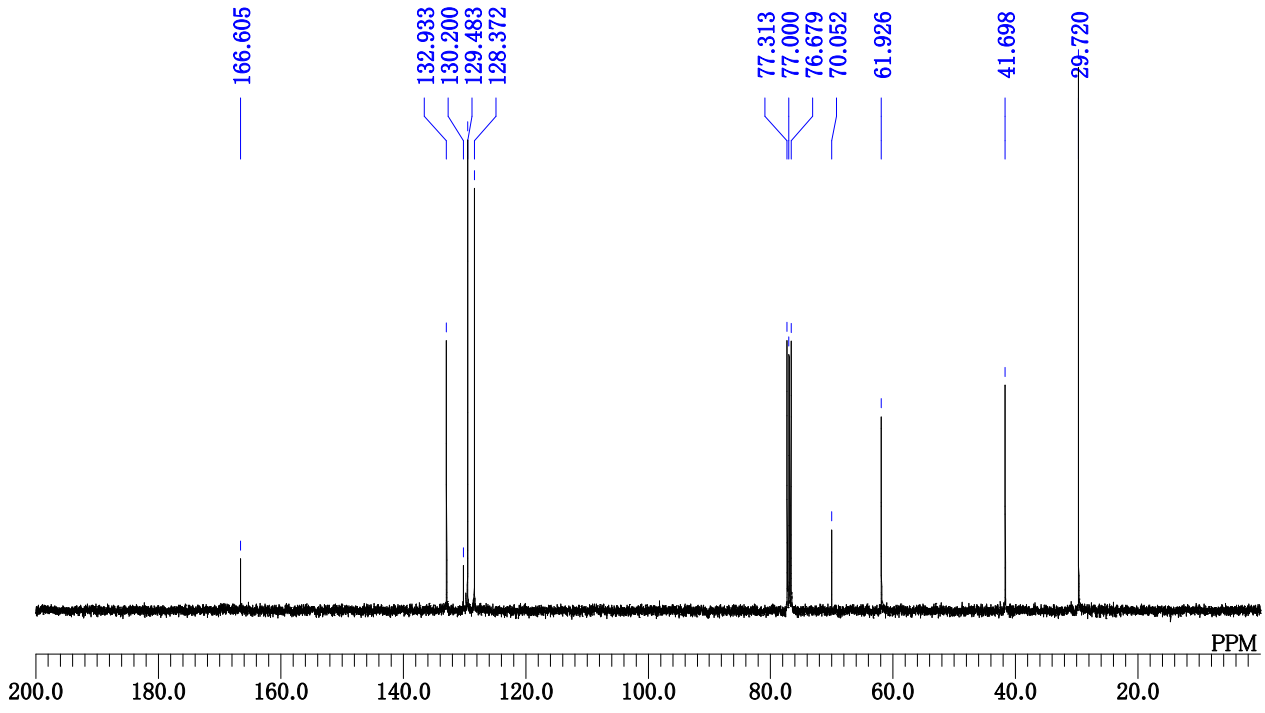
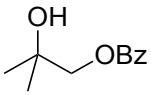


^{13}C NMR: (100 MHz, $CDCl_3$)





2b

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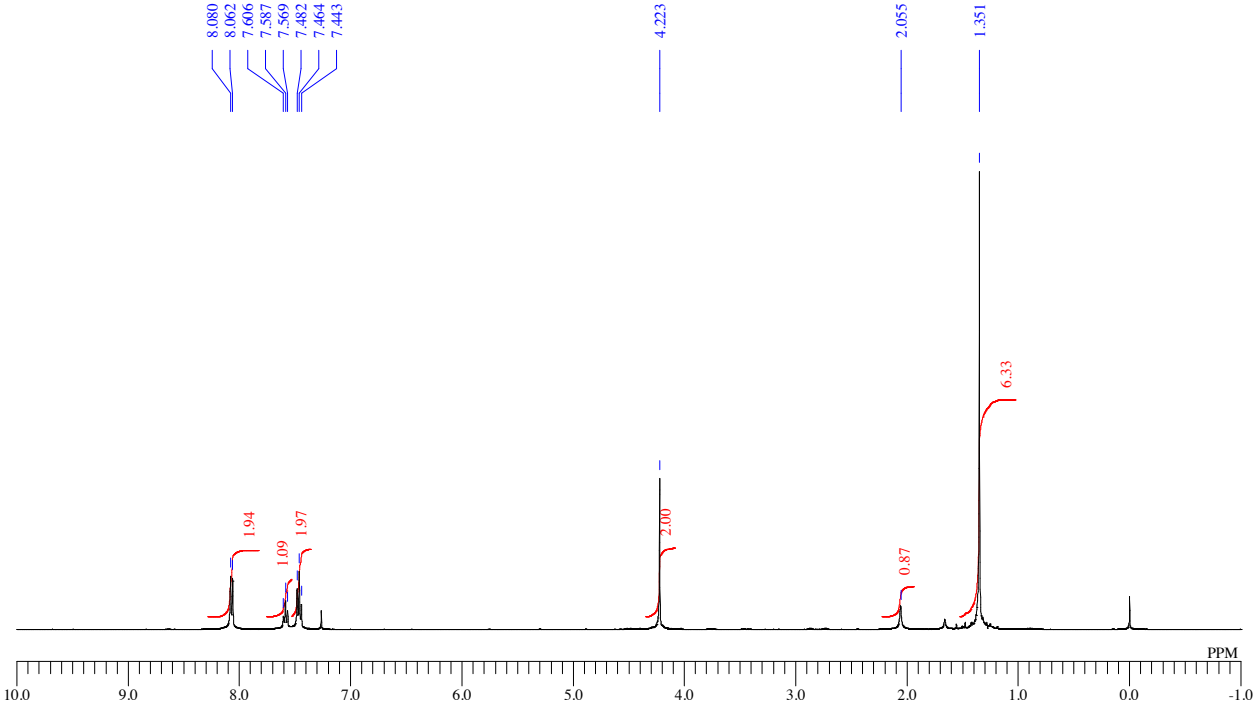

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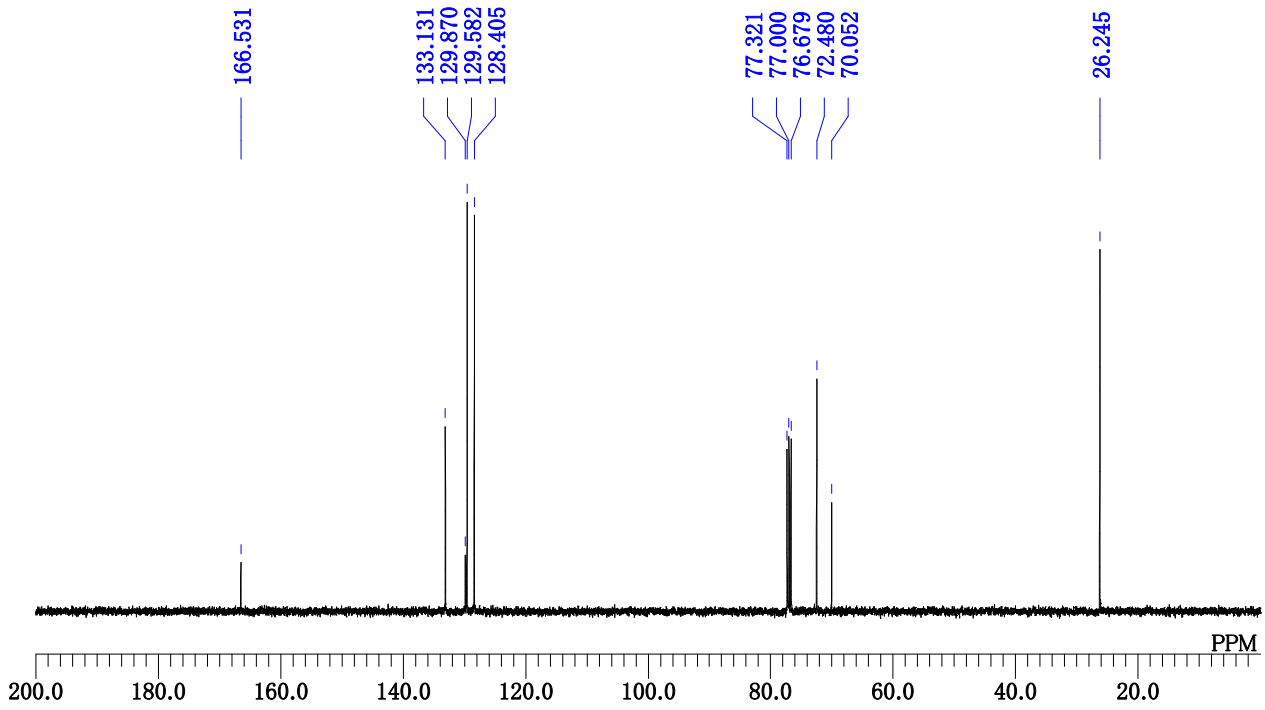

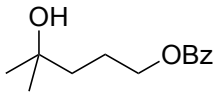

2c

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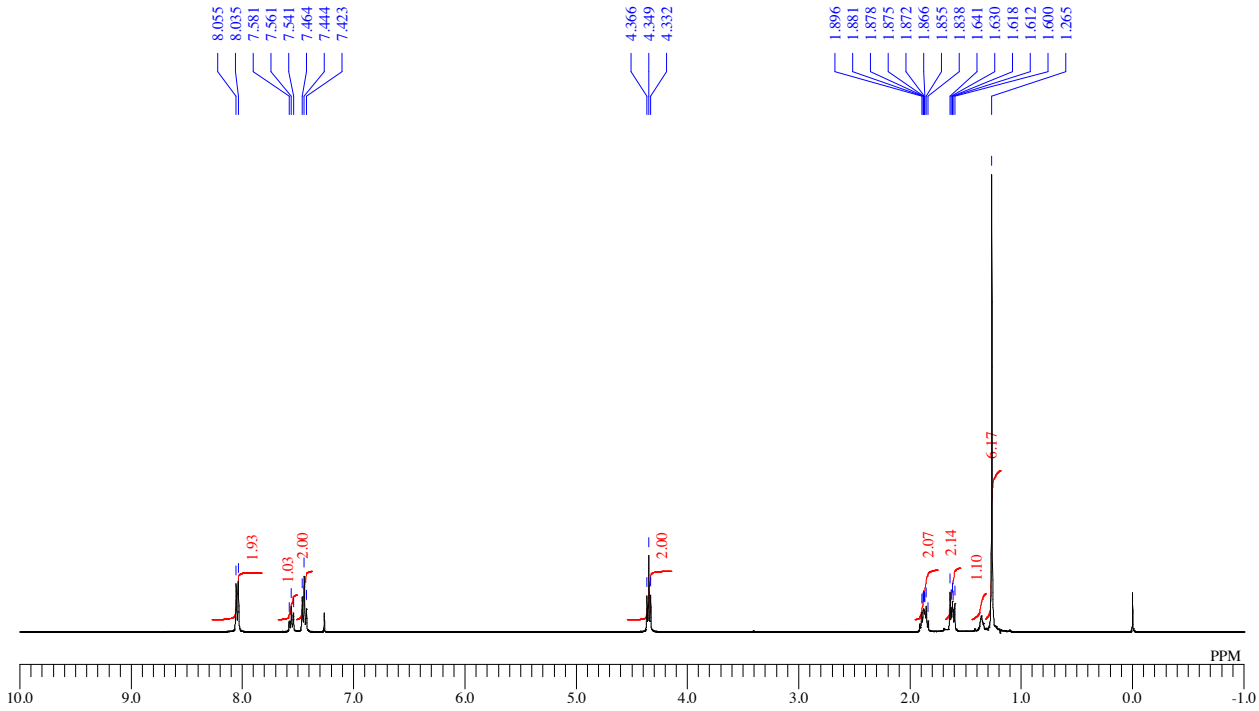

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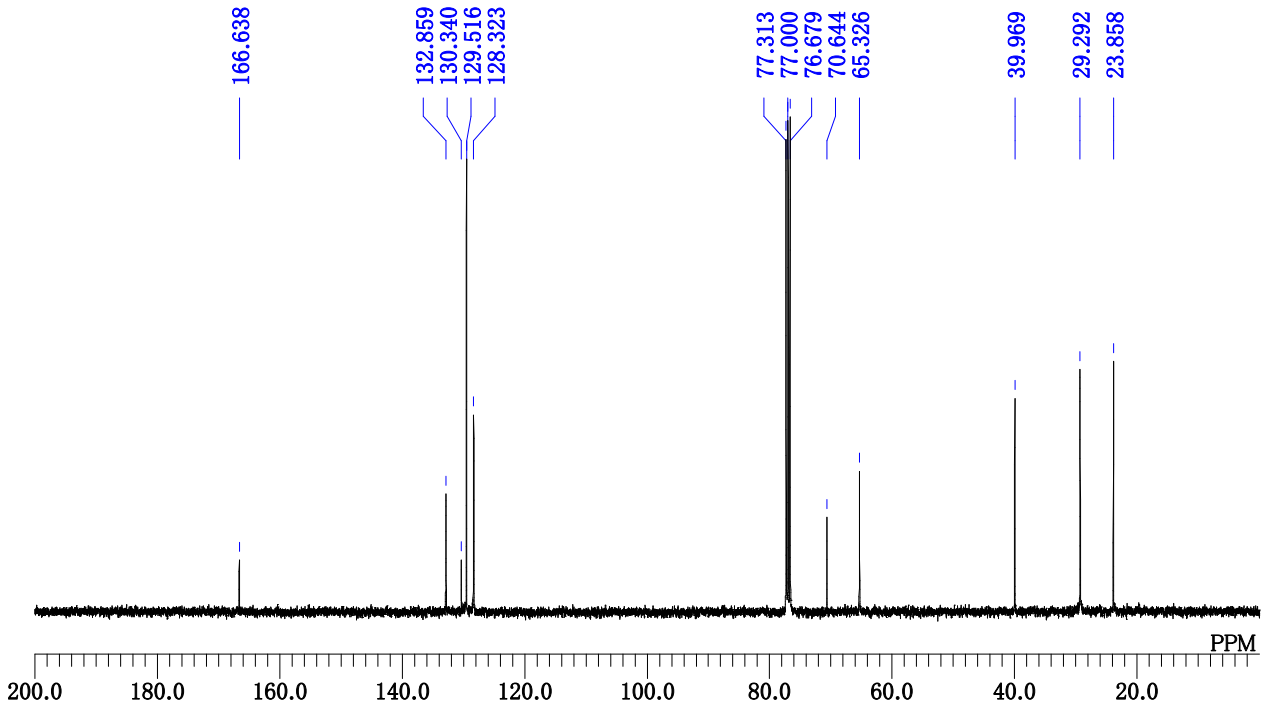

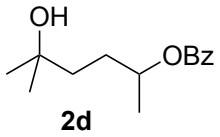

2d

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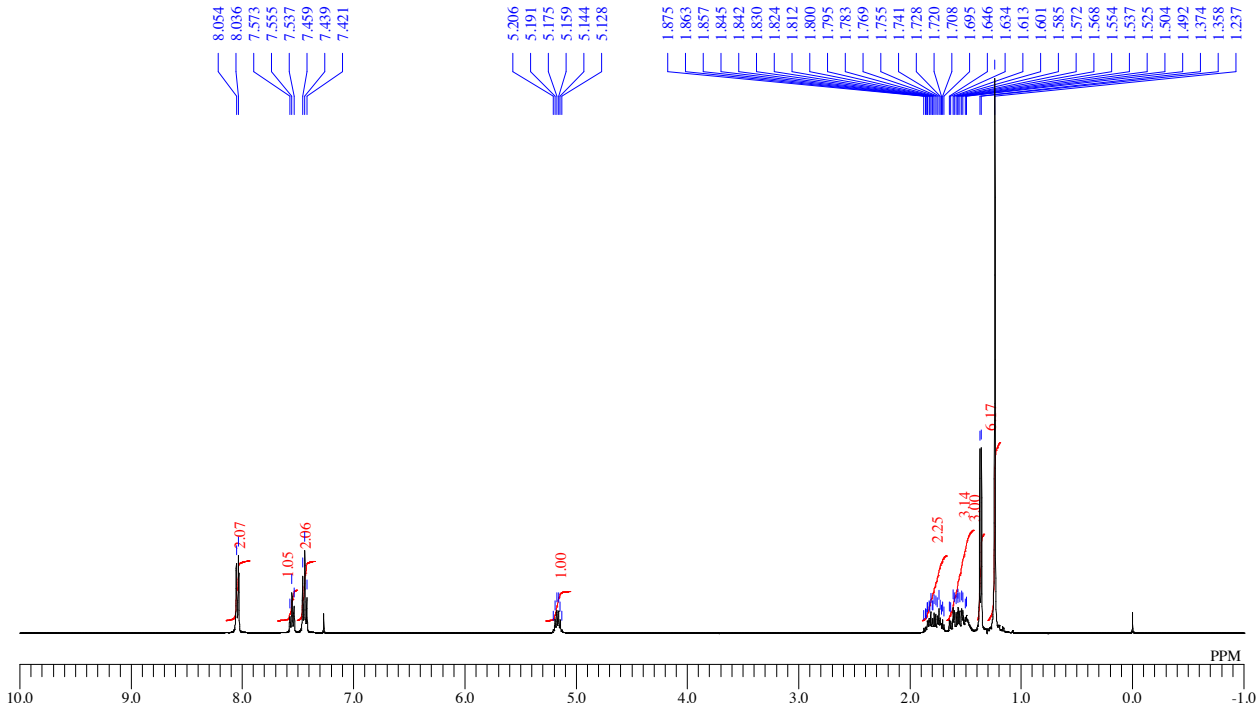

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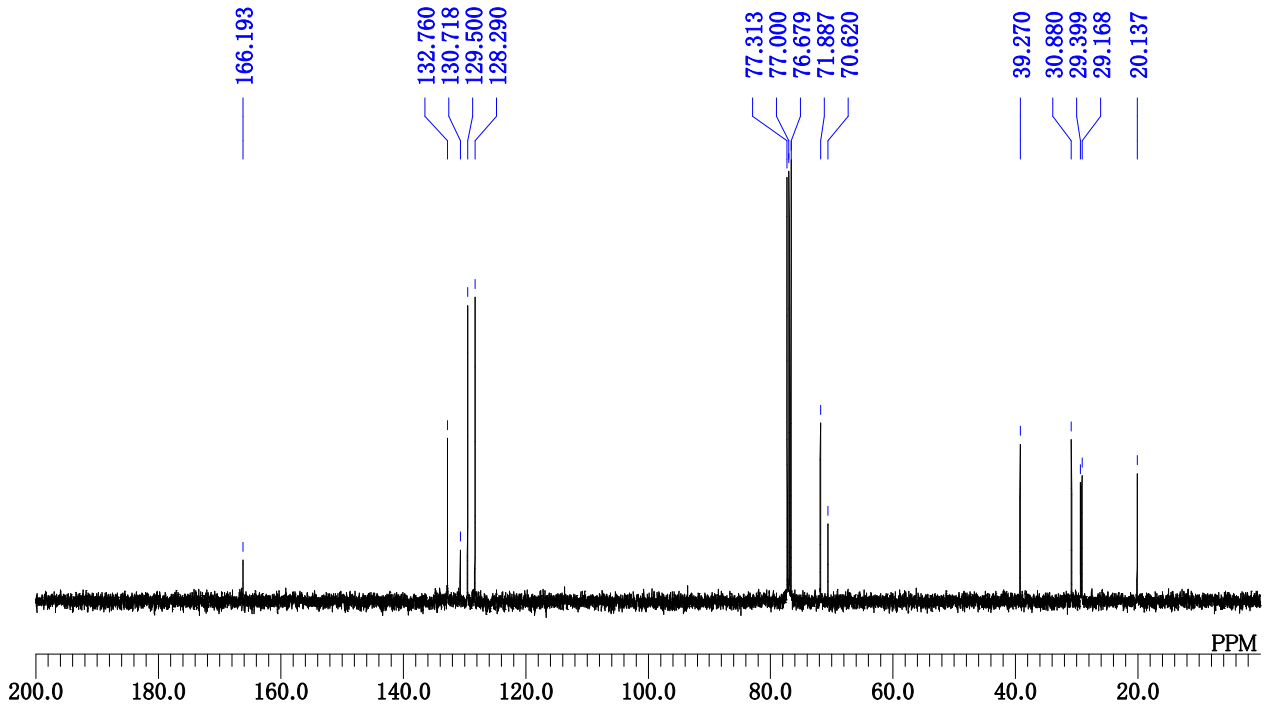

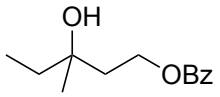

2e

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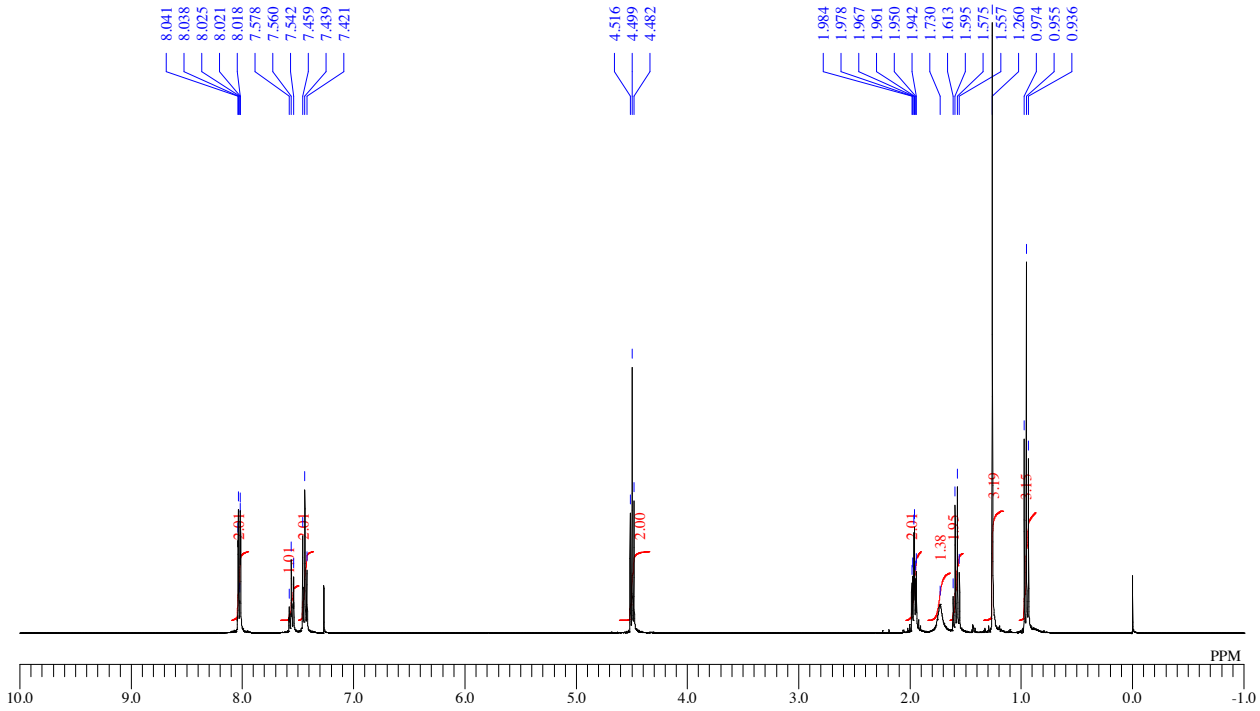

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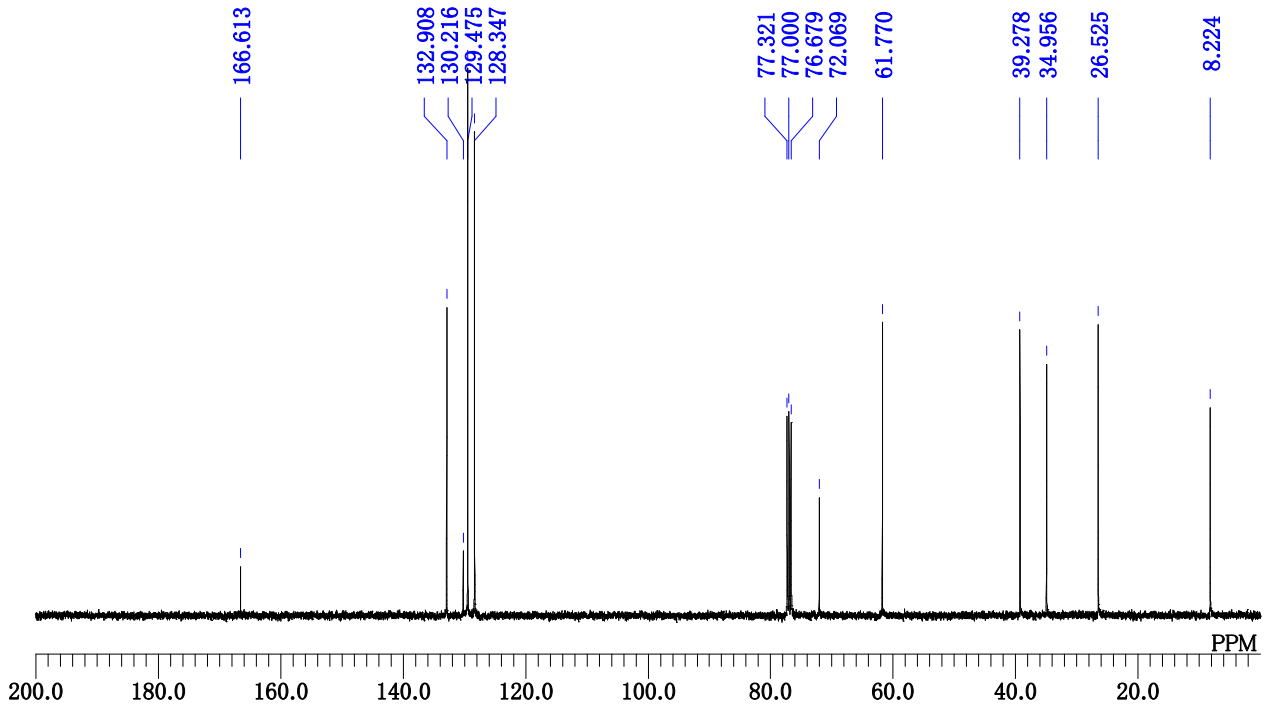
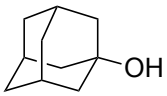
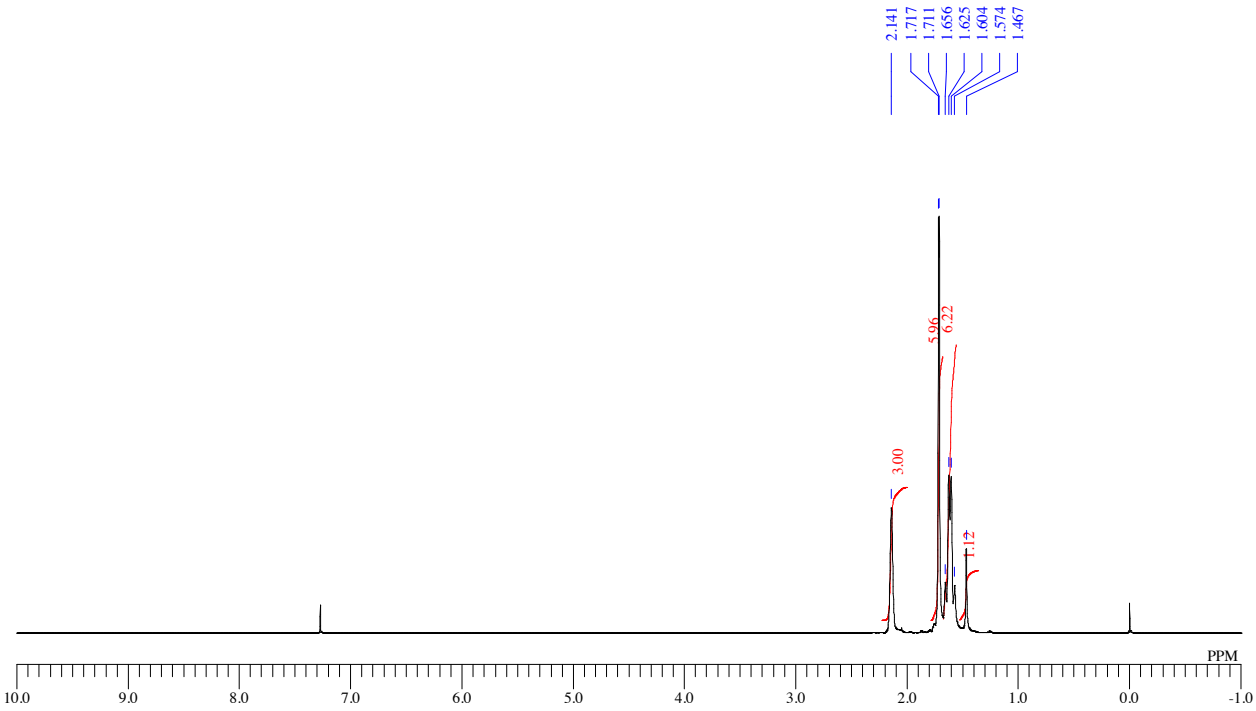




2f

^{1}H NMR: (400 MHz, $CDCl_3$)



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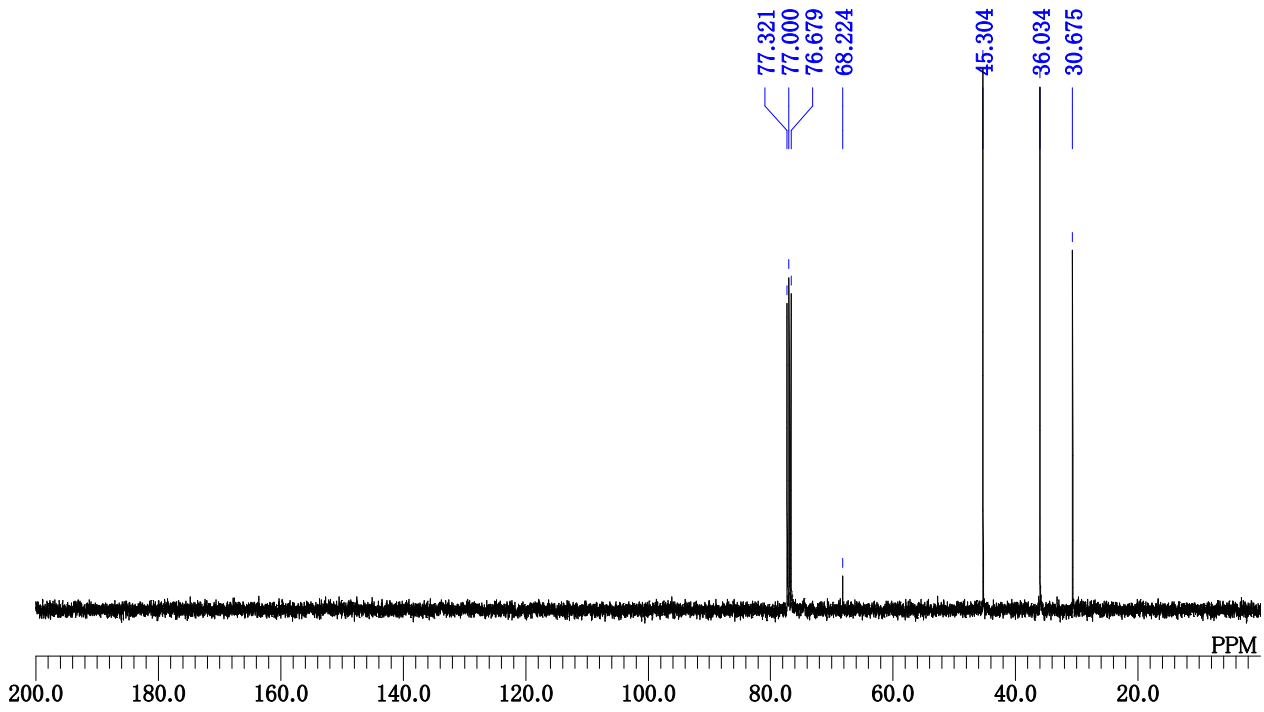
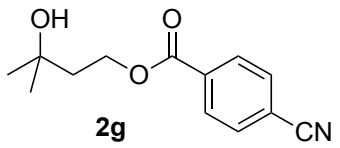
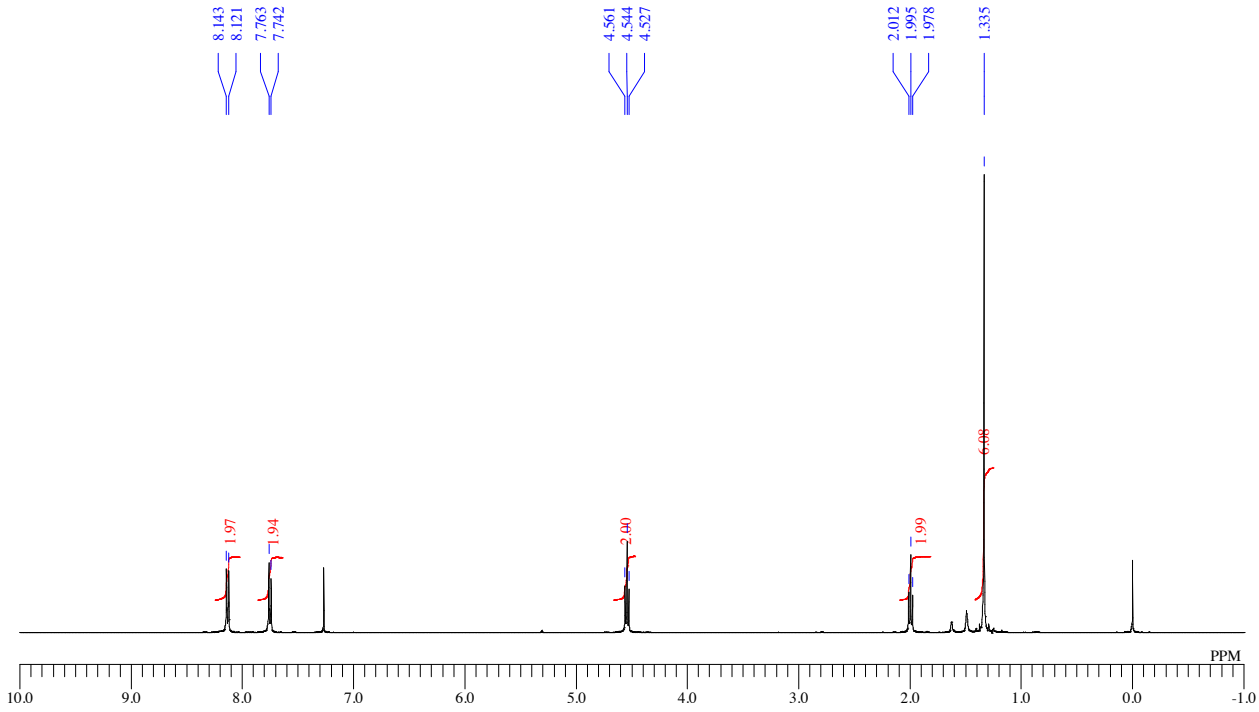




2g

^{1}H NMR: (400 MHz, $CDCl_3$)



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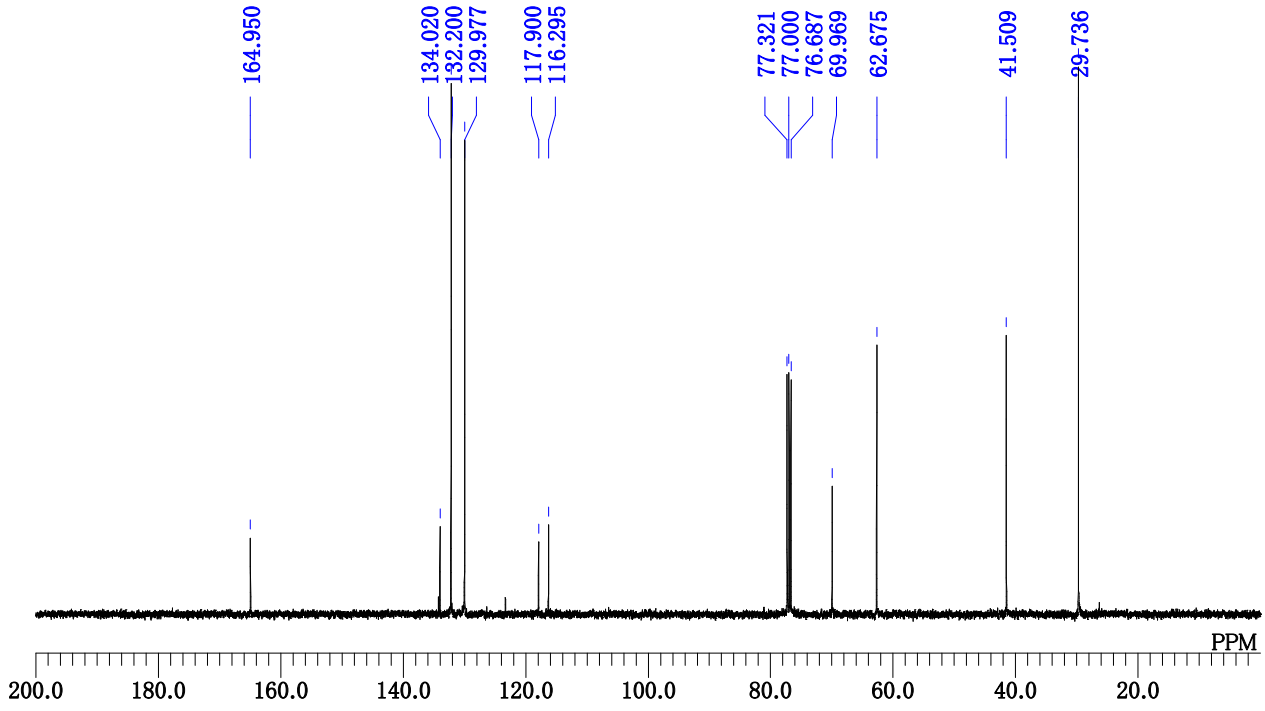

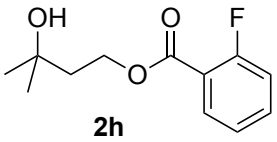

2h

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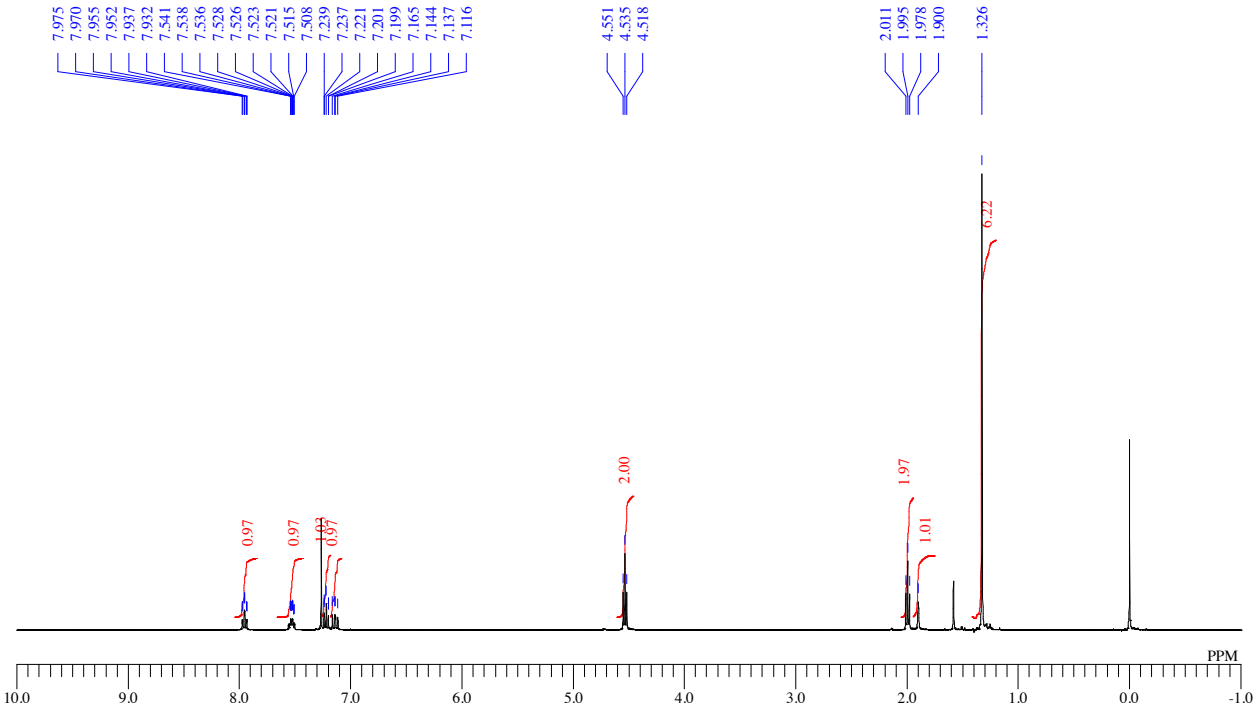

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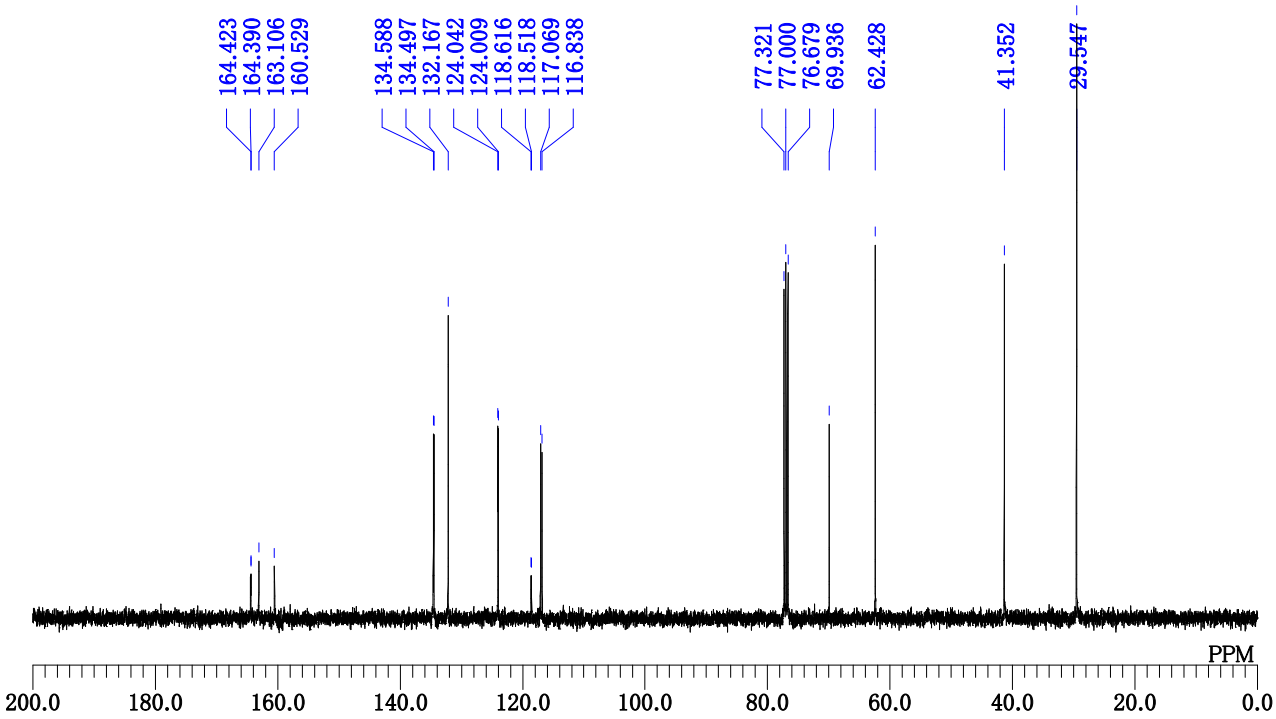
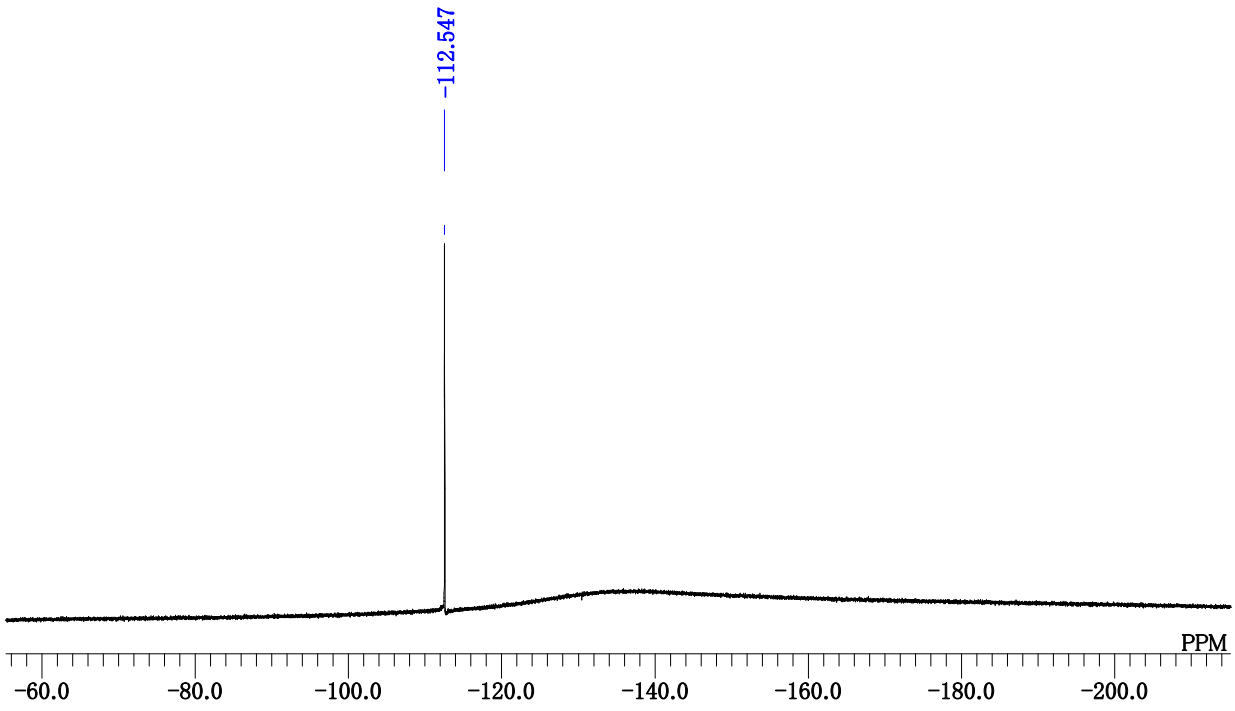


^{19}F NMR: (377 MHz, $CDCl_3$)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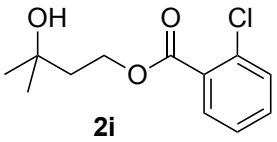
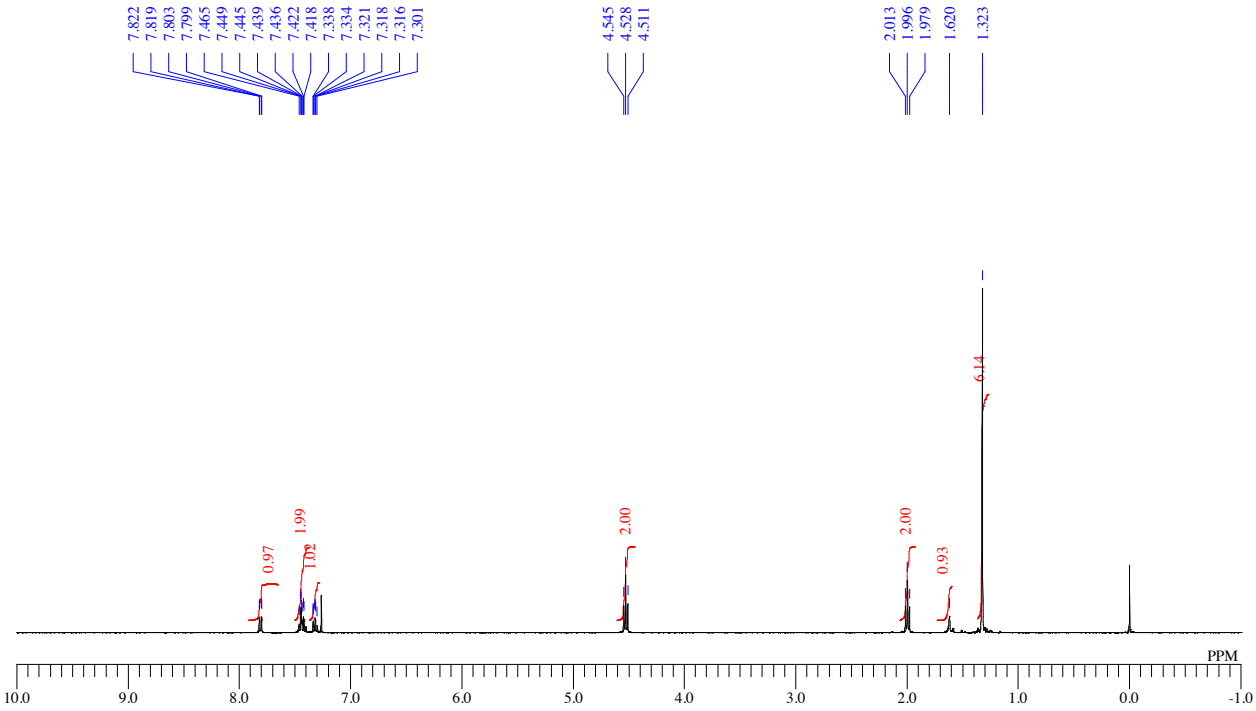
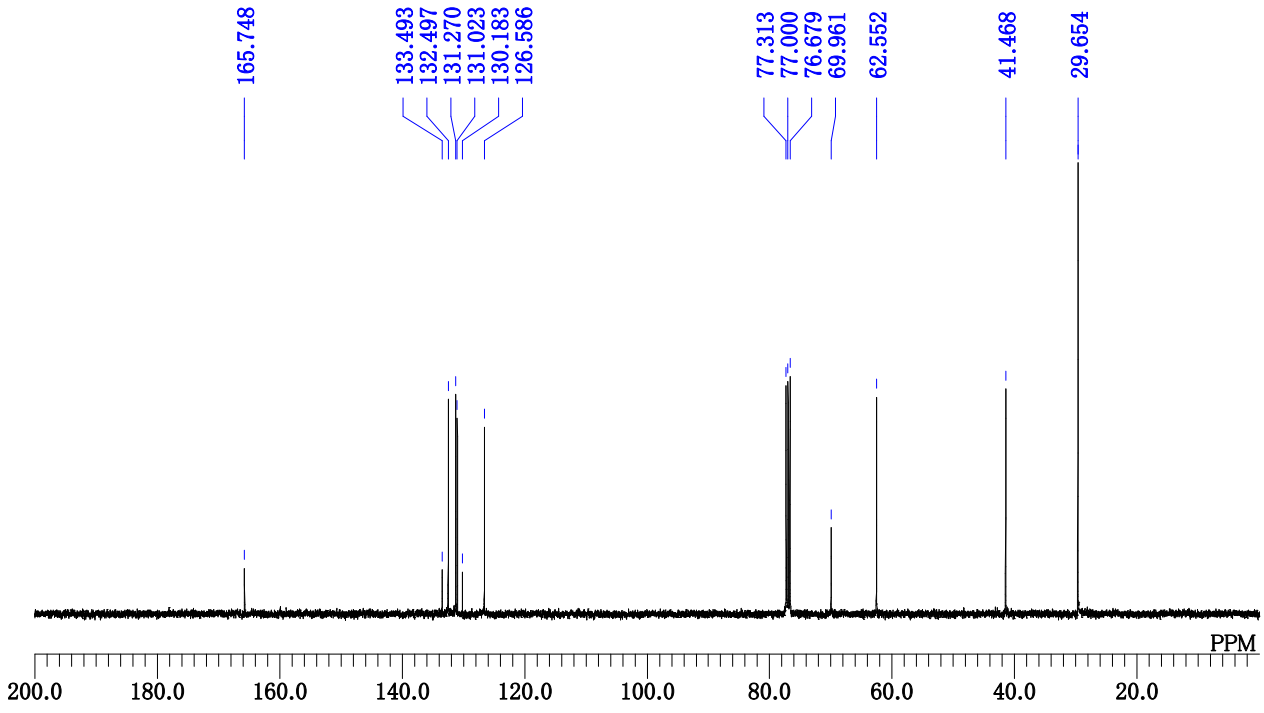


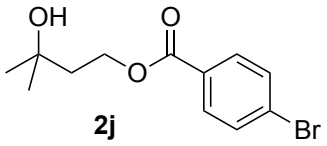
2i

^{1}H NMR: (400 MHz, $CDCl_3$)



^{13}C NMR: (100 MHz, $CDCl_3$)





2j

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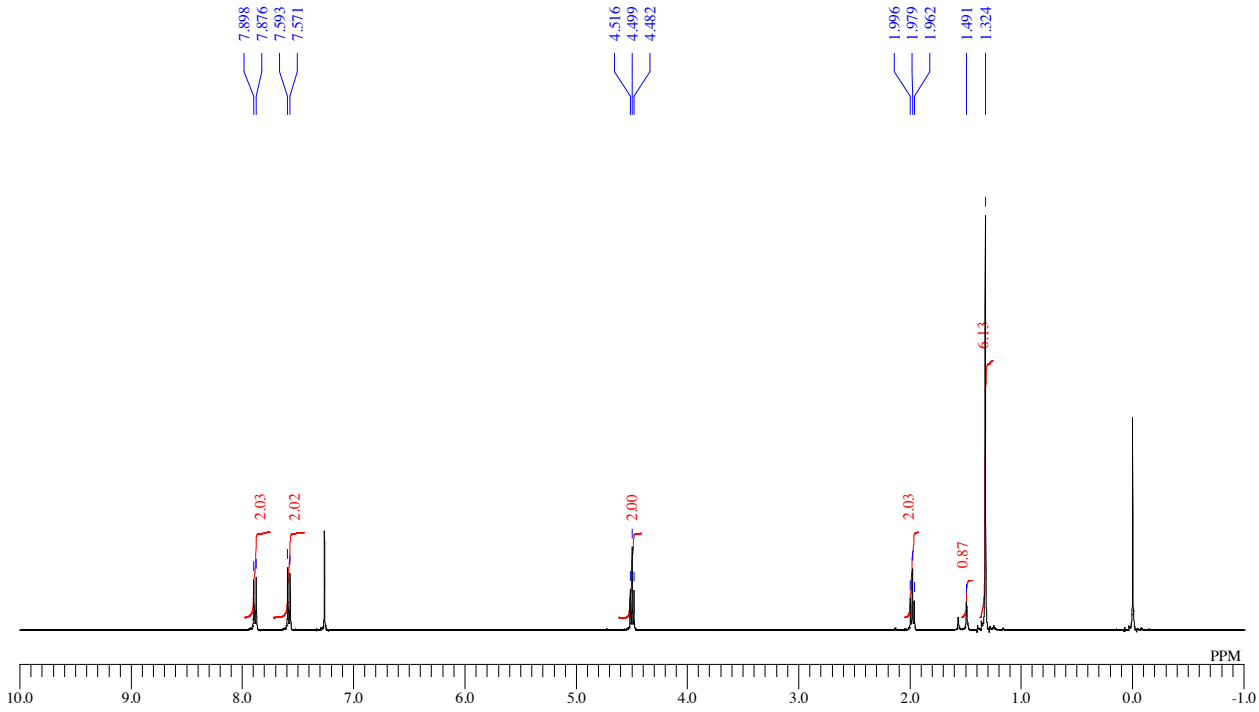

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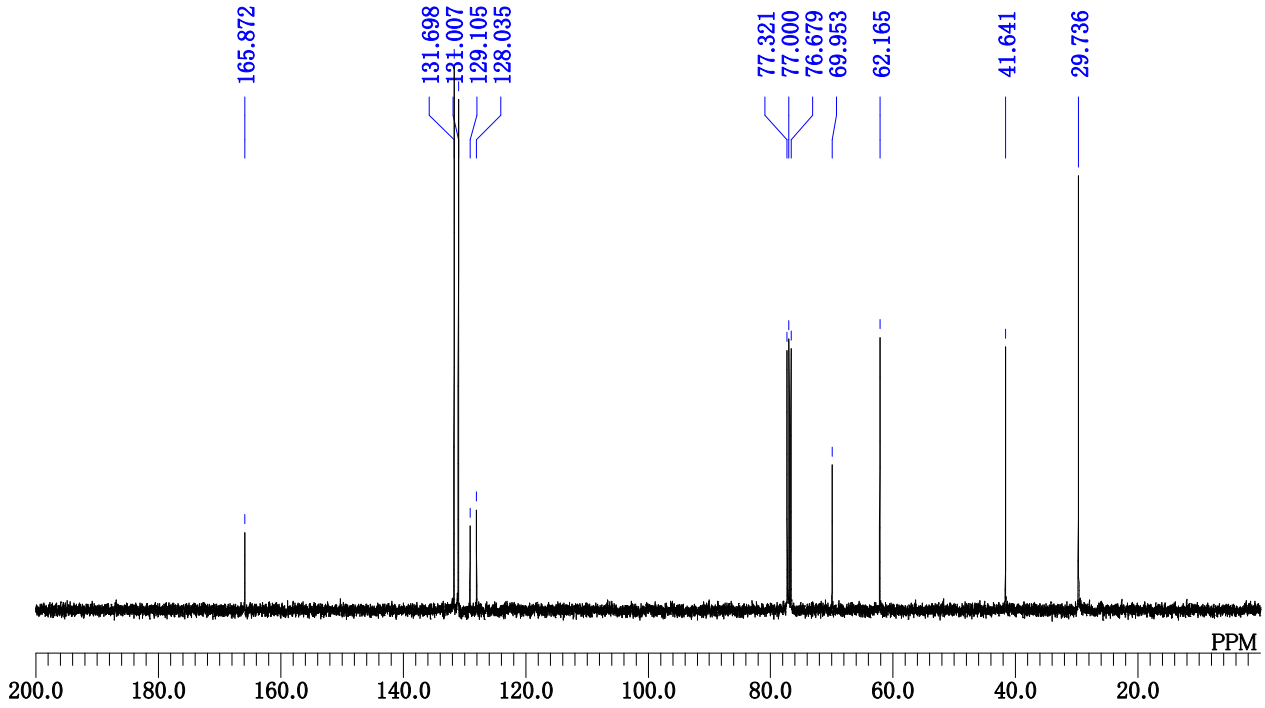
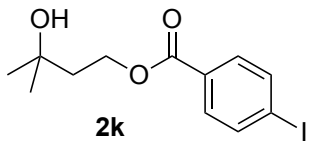
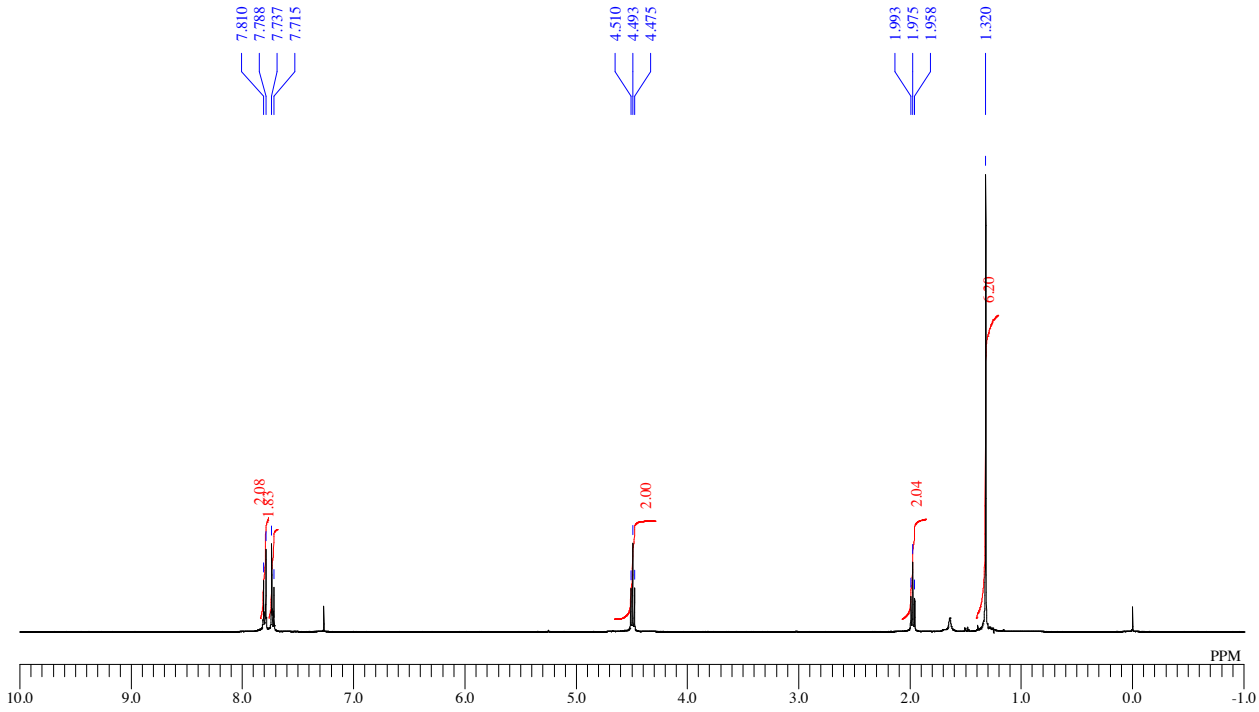




2k

^{1}H NMR: (400 MHz, $CDCl_3$)



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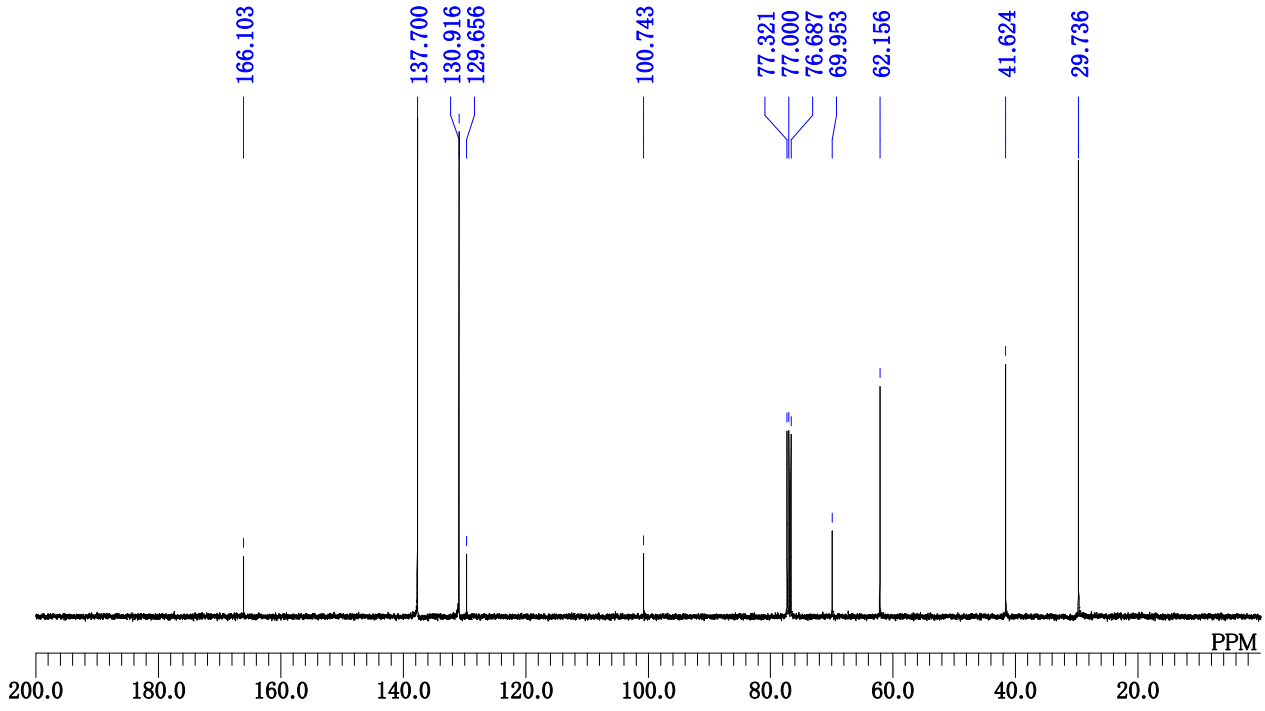
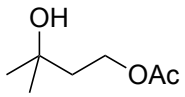
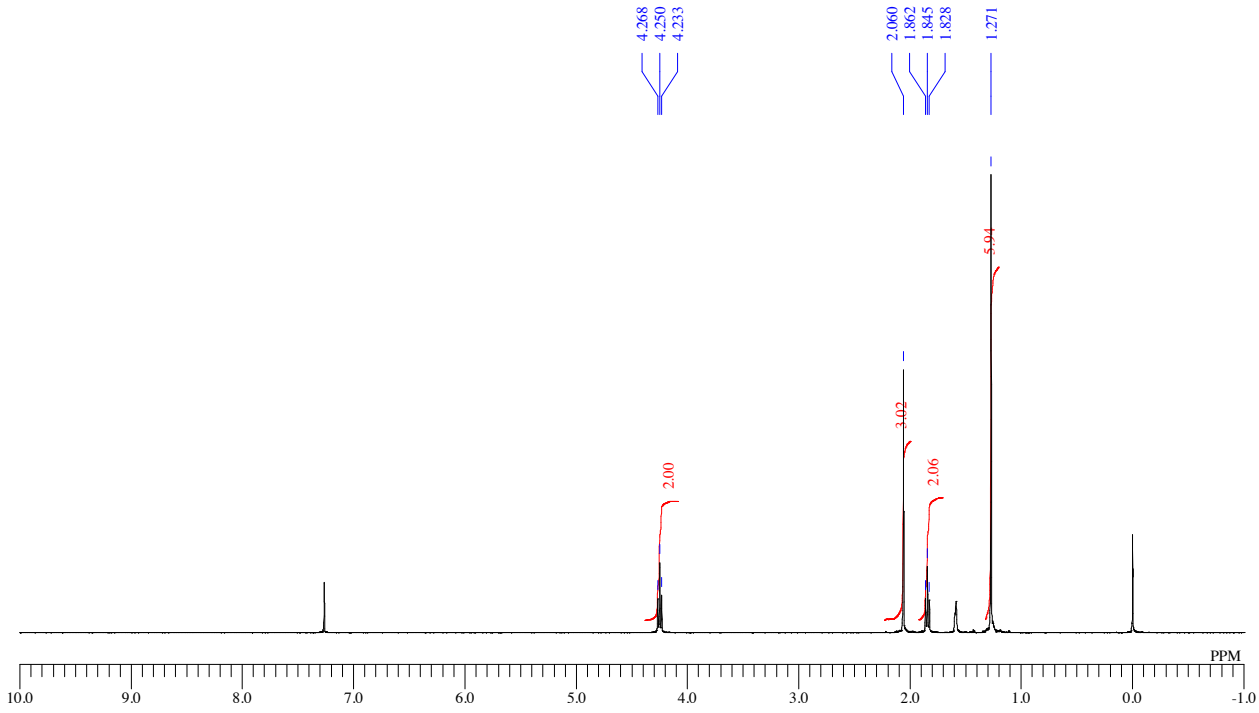




2l

^{1}H NMR: (400 MHz, $CDCl_3$)



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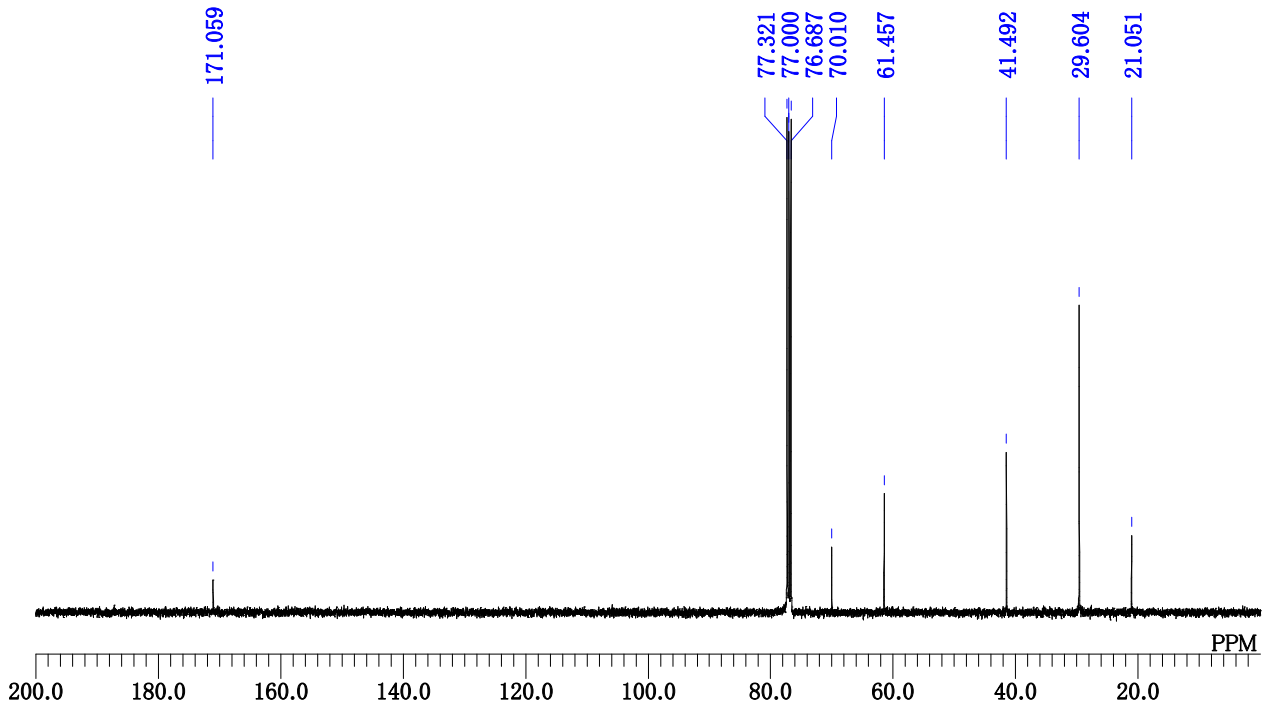
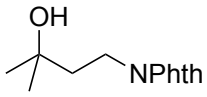
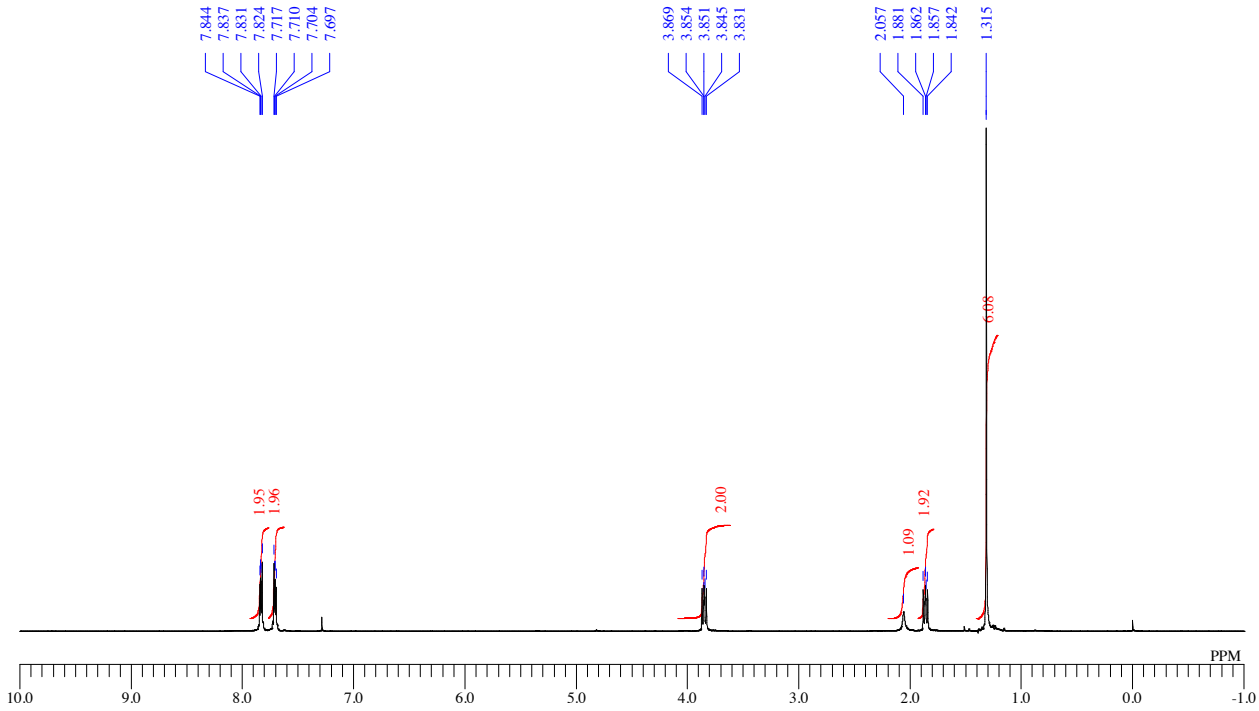


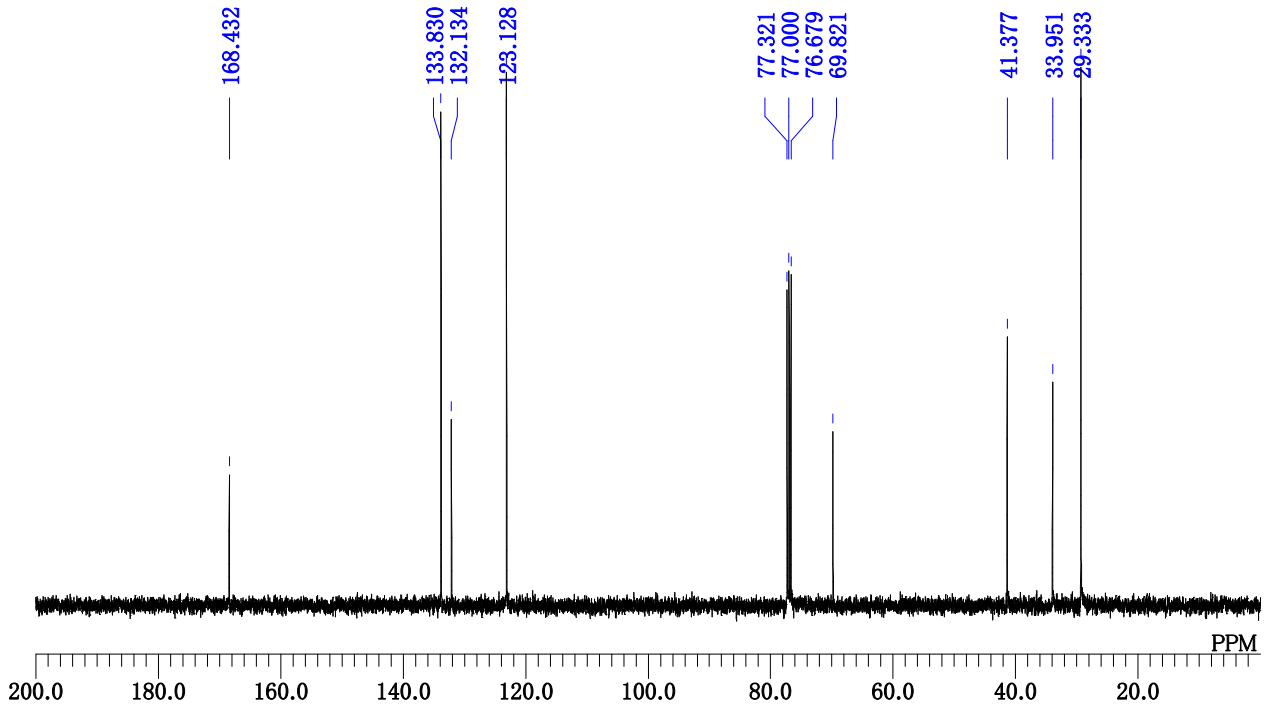


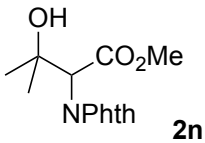
2m

^{1}H NMR: (400 MHz, $CDCl_3$)



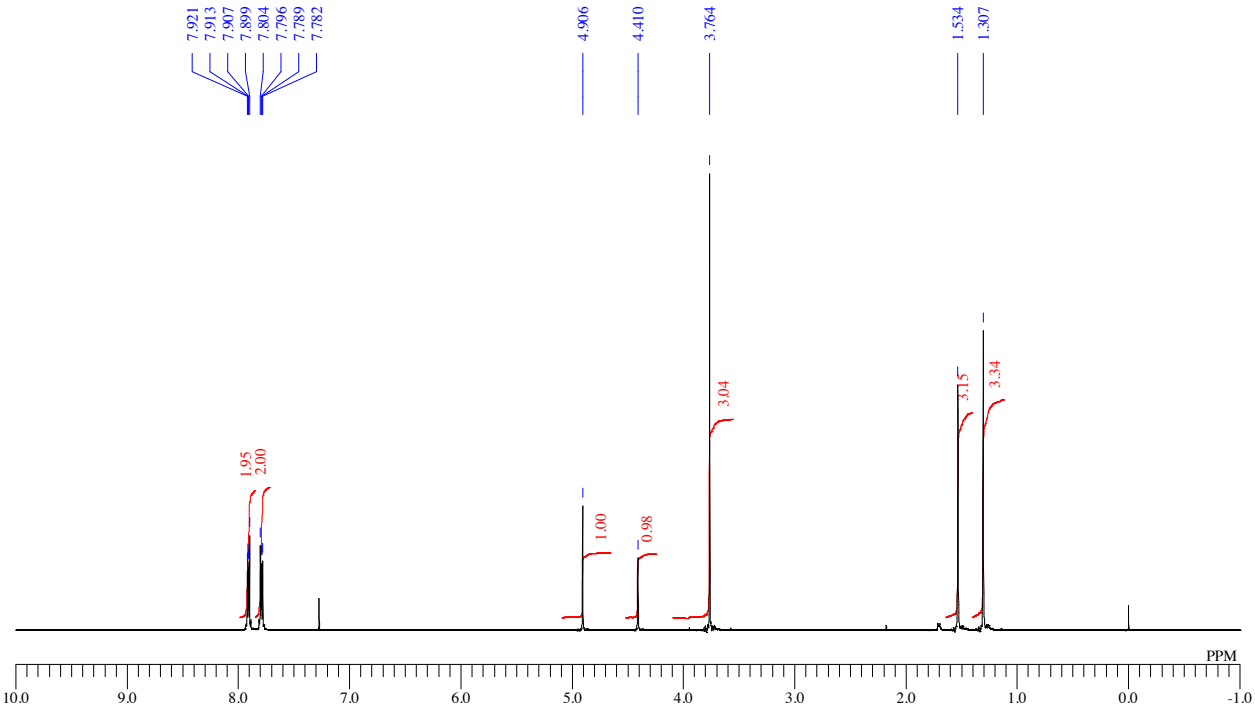
^{13}C NMR: (100 MHz, $CDCl_3$)



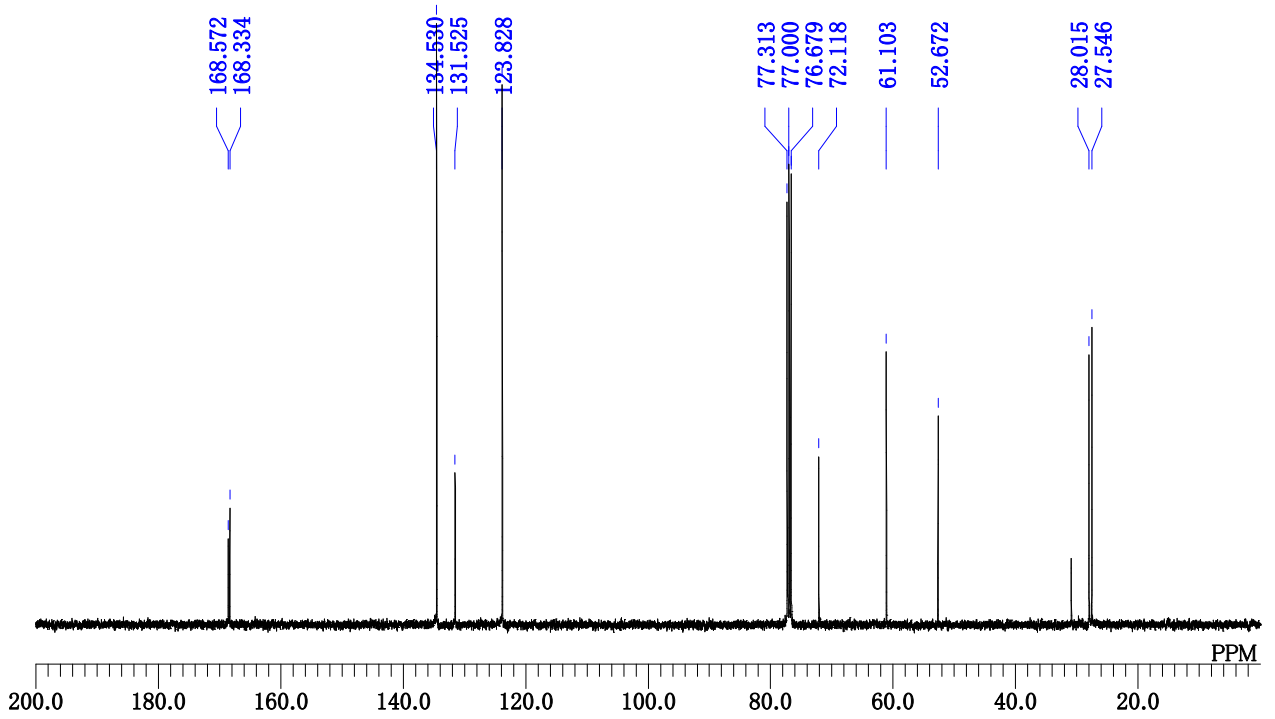


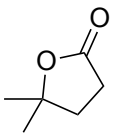
2n

^{1}H NMR: (400 MHz, $CDCl_3$)



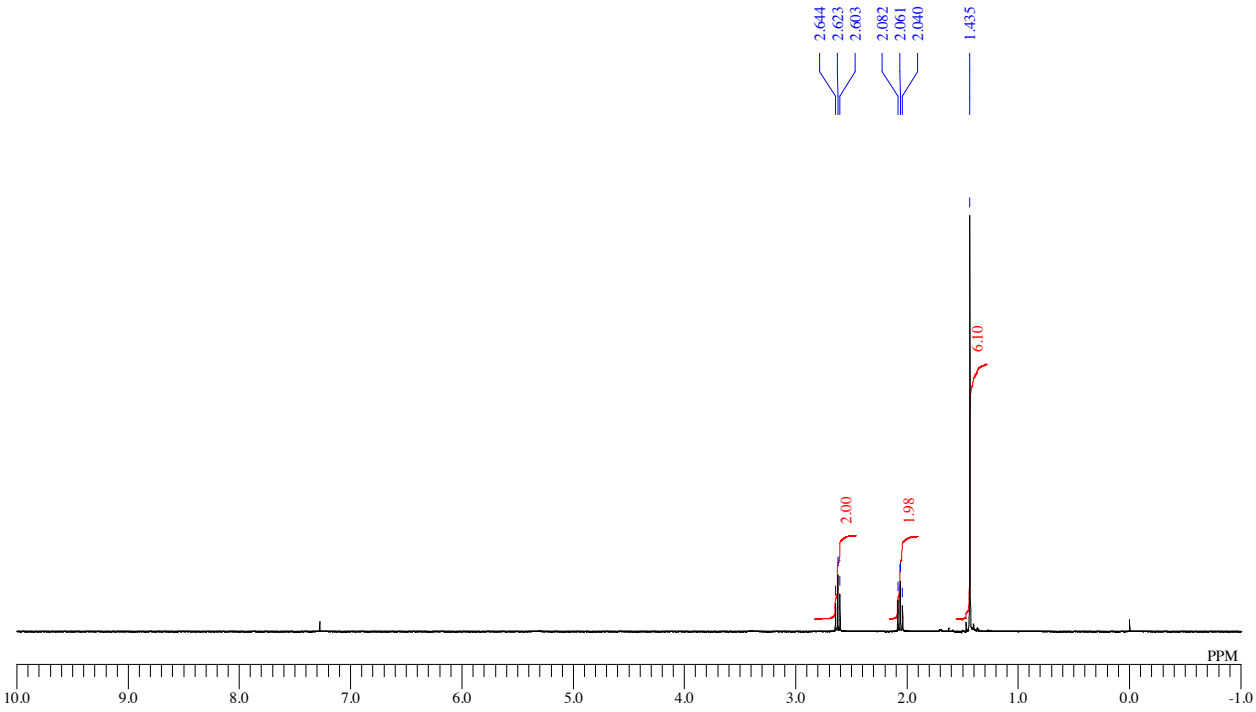
^{13}C NMR: (100 MHz, $CDCl_3$)



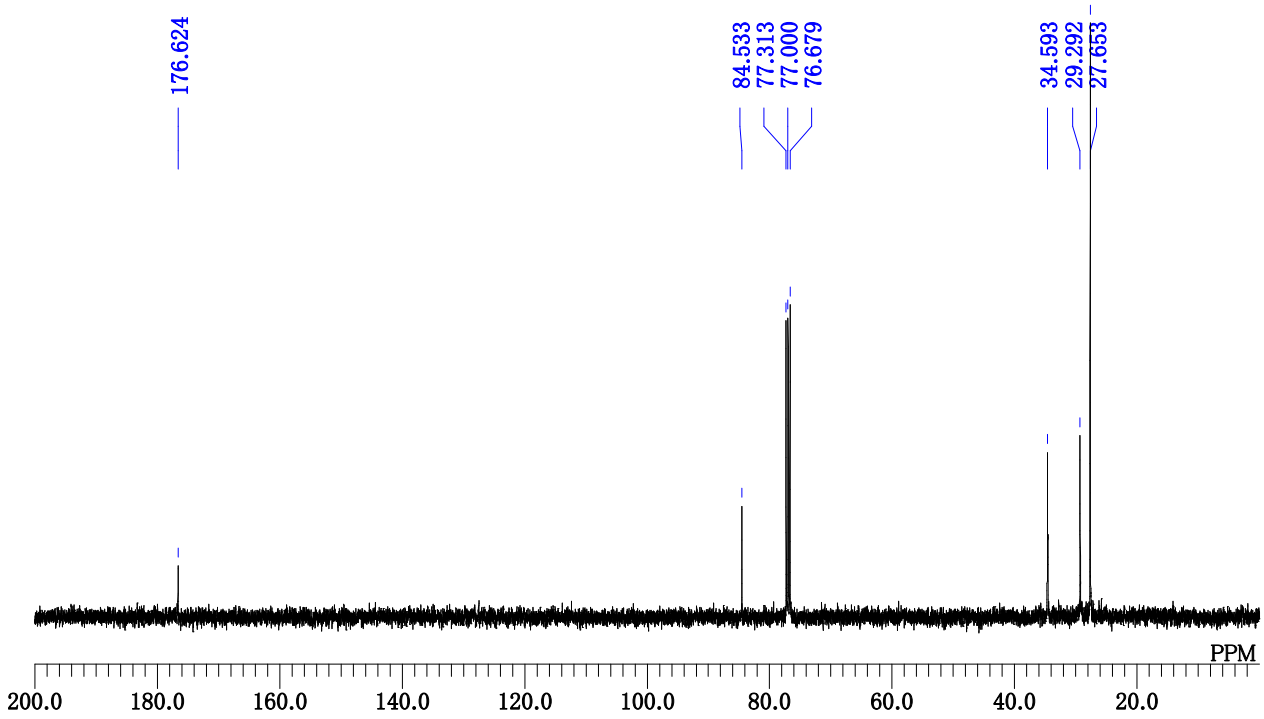


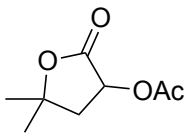
4a

^{1}H NMR: (400 MHz, $CDCl_3$)



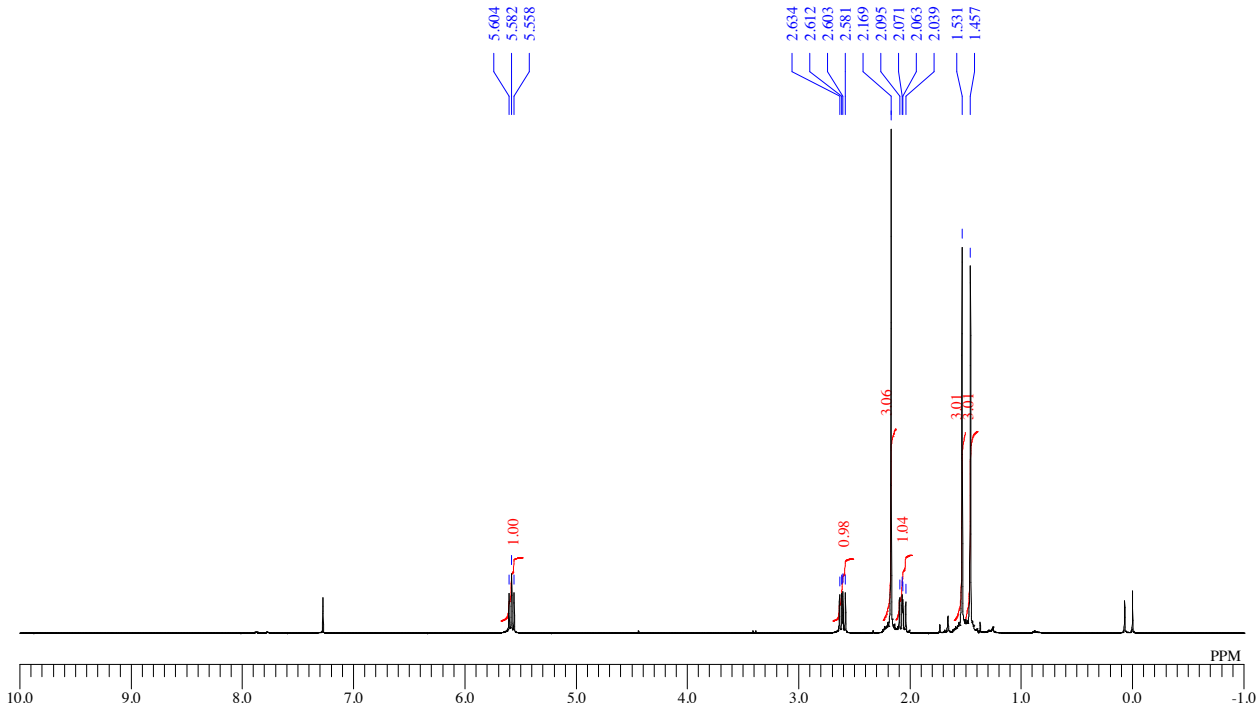
^{13}C NMR: (100 MHz, $CDCl_3$)



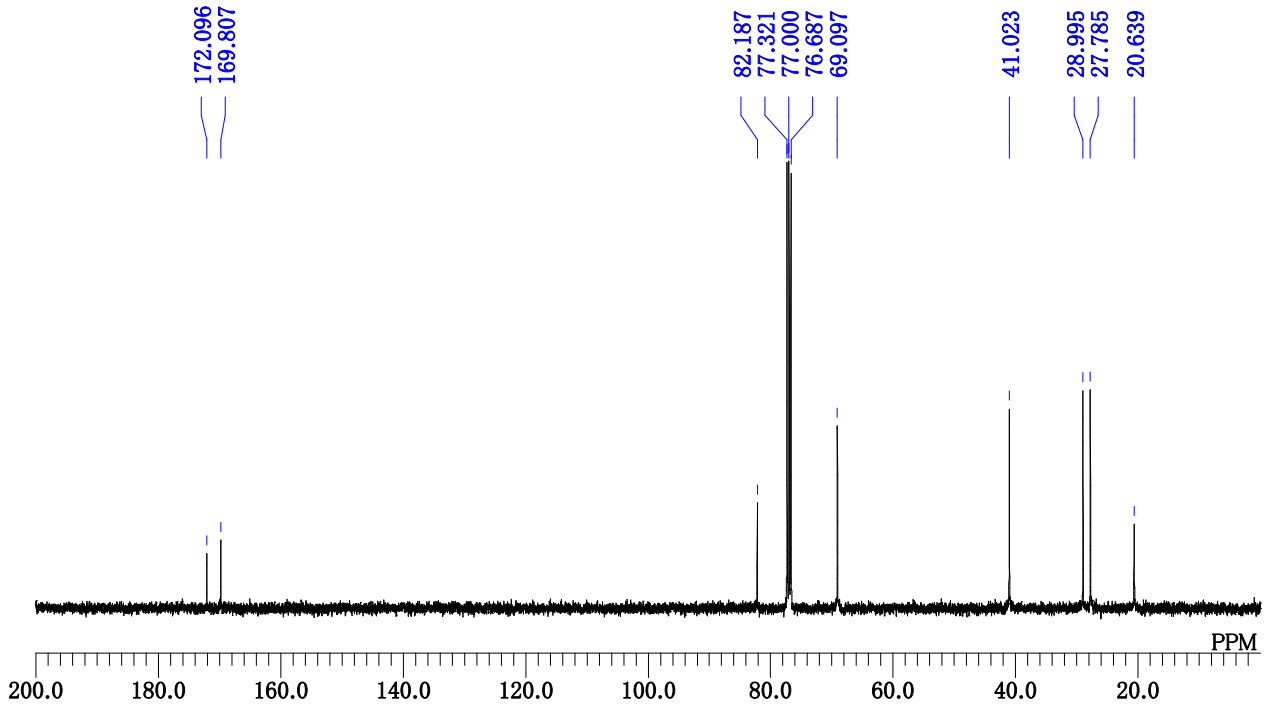


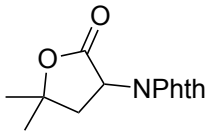
4b

^{1}H NMR: (400 MHz, $CDCl_3$)



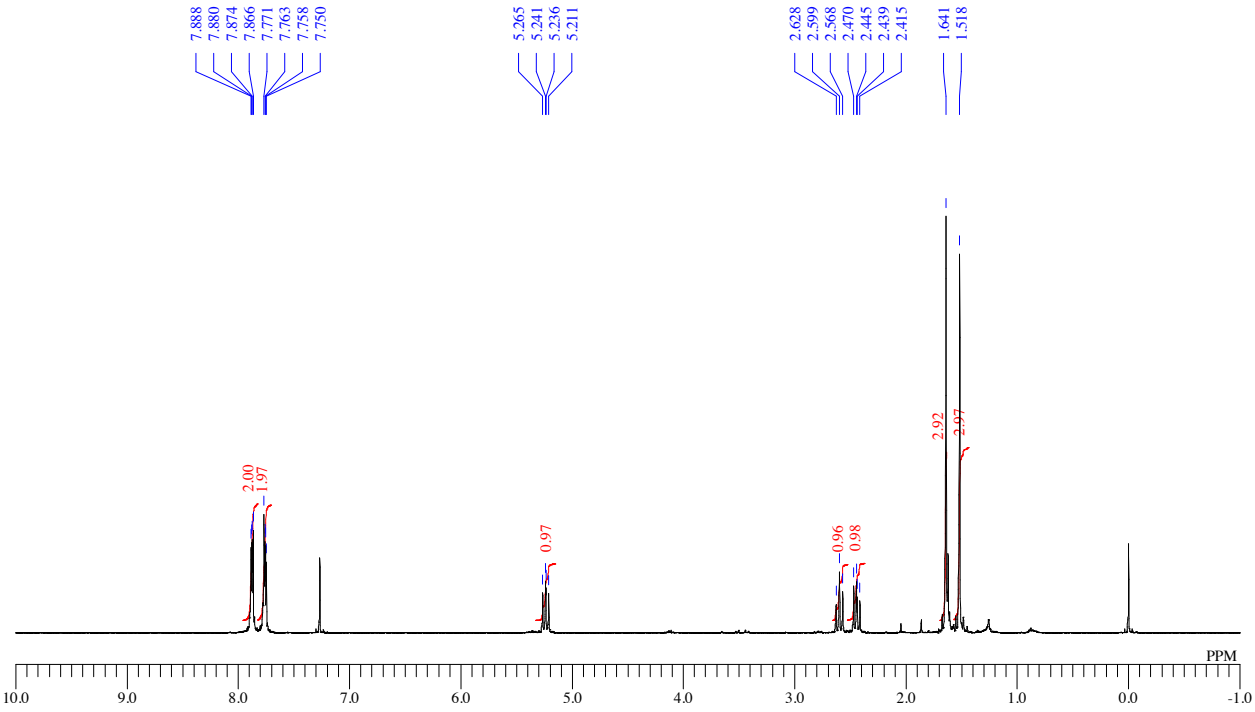
^{13}C NMR: (100 MHz, $CDCl_3$)



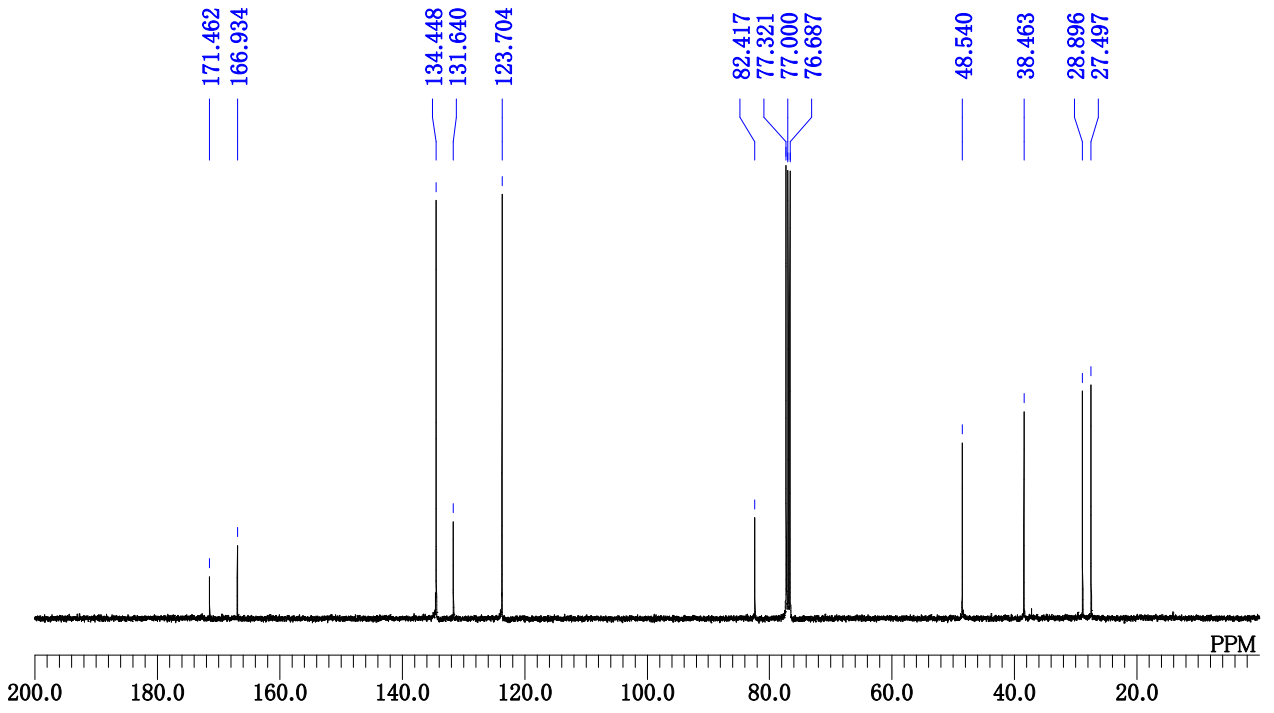


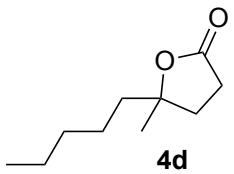
4c

^{1}H NMR: (400 MHz, $CDCl_3$)



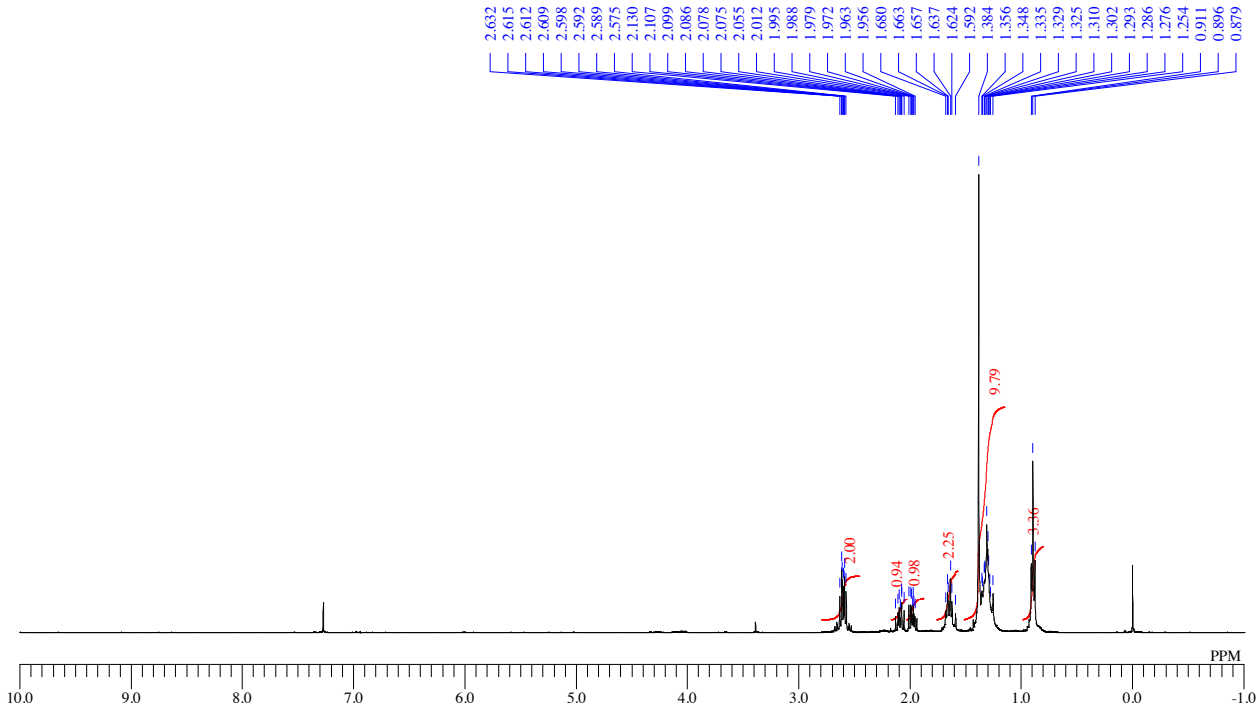
^{13}C NMR: (100 MHz, $CDCl_3$)



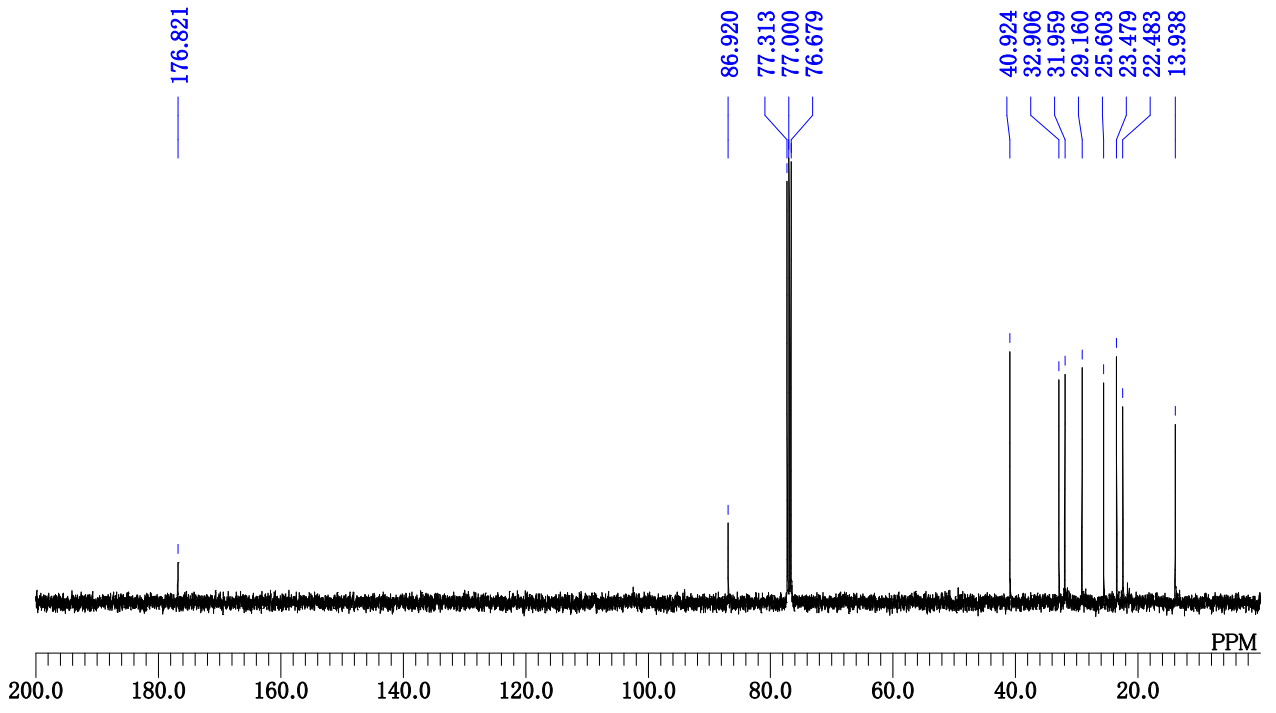


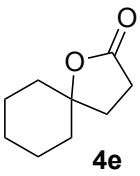
4d

^{1}H NMR: (400 MHz, $CDCl_3$)



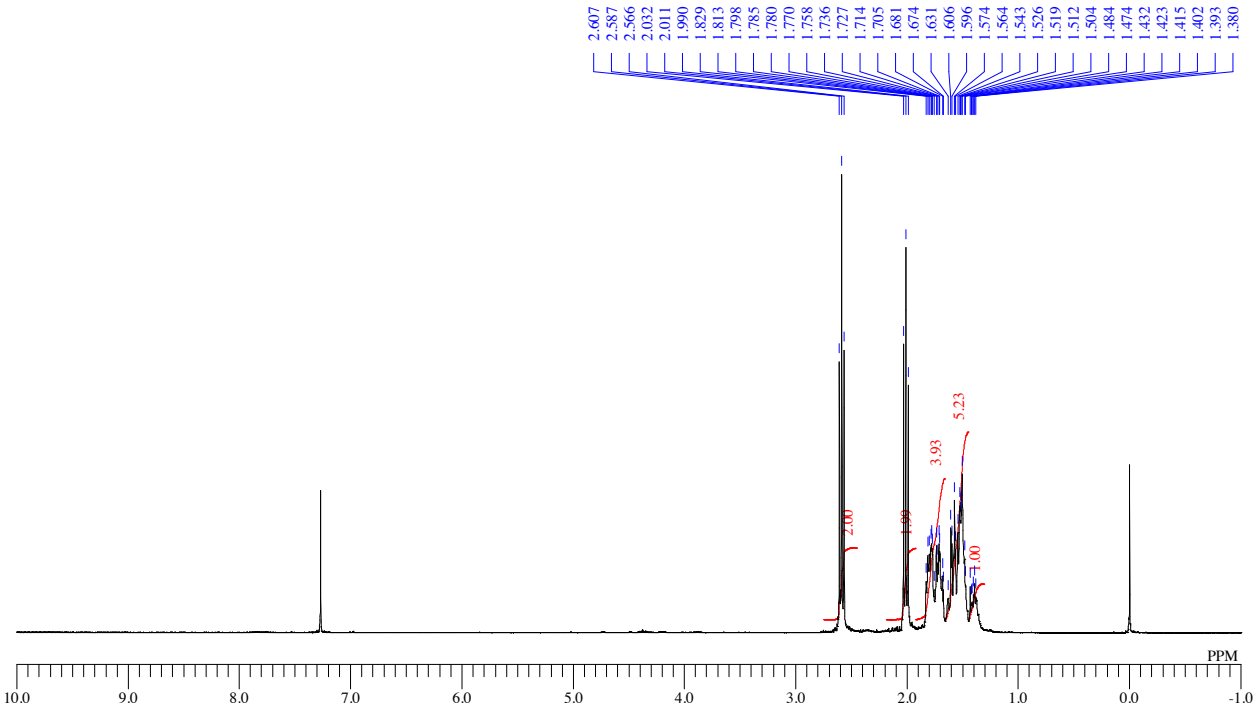
^{13}C NMR: (100 MHz, $CDCl_3$)



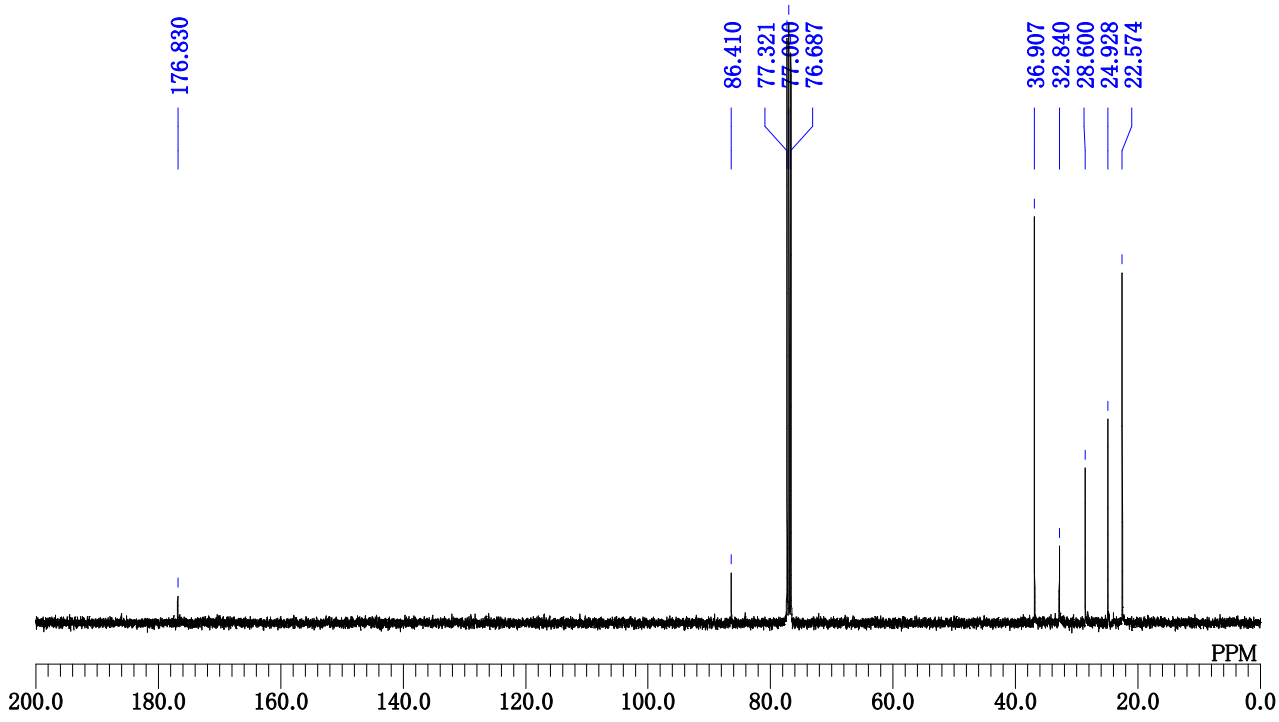


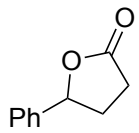
4e

^{1}H NMR: (400 MHz, $CDCl_3$)



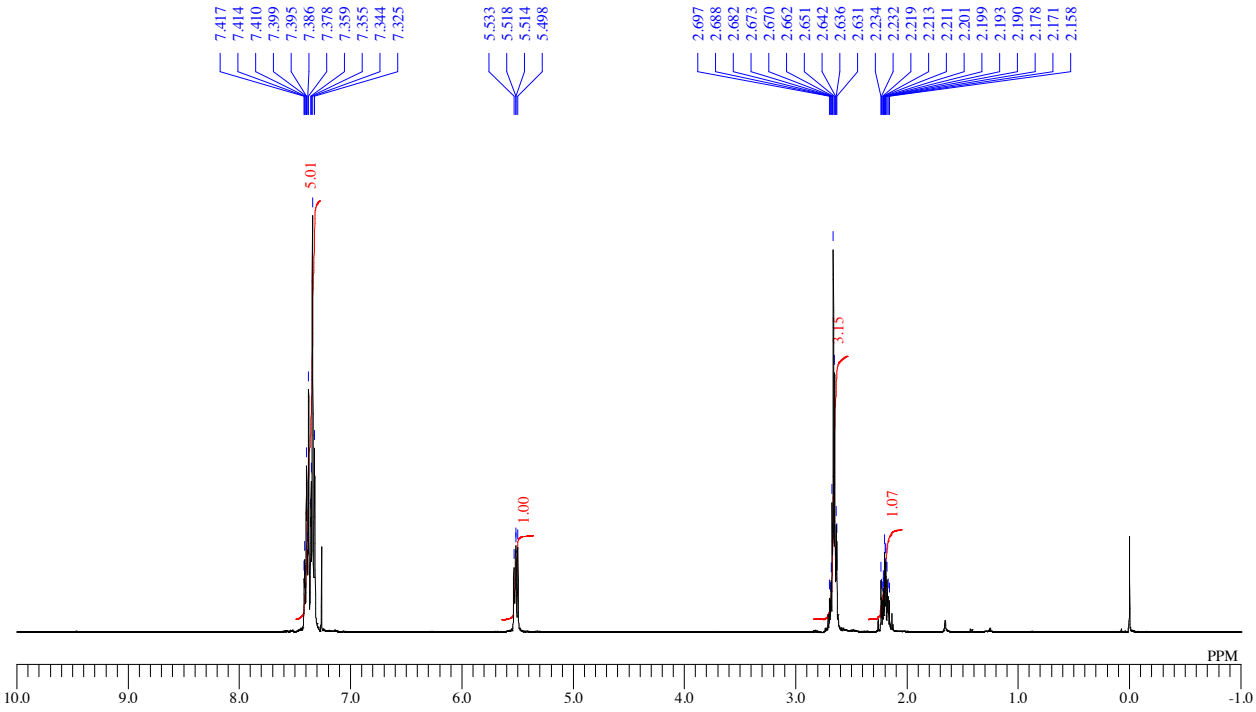
^{13}C NMR: (100 MHz, $CDCl_3$)



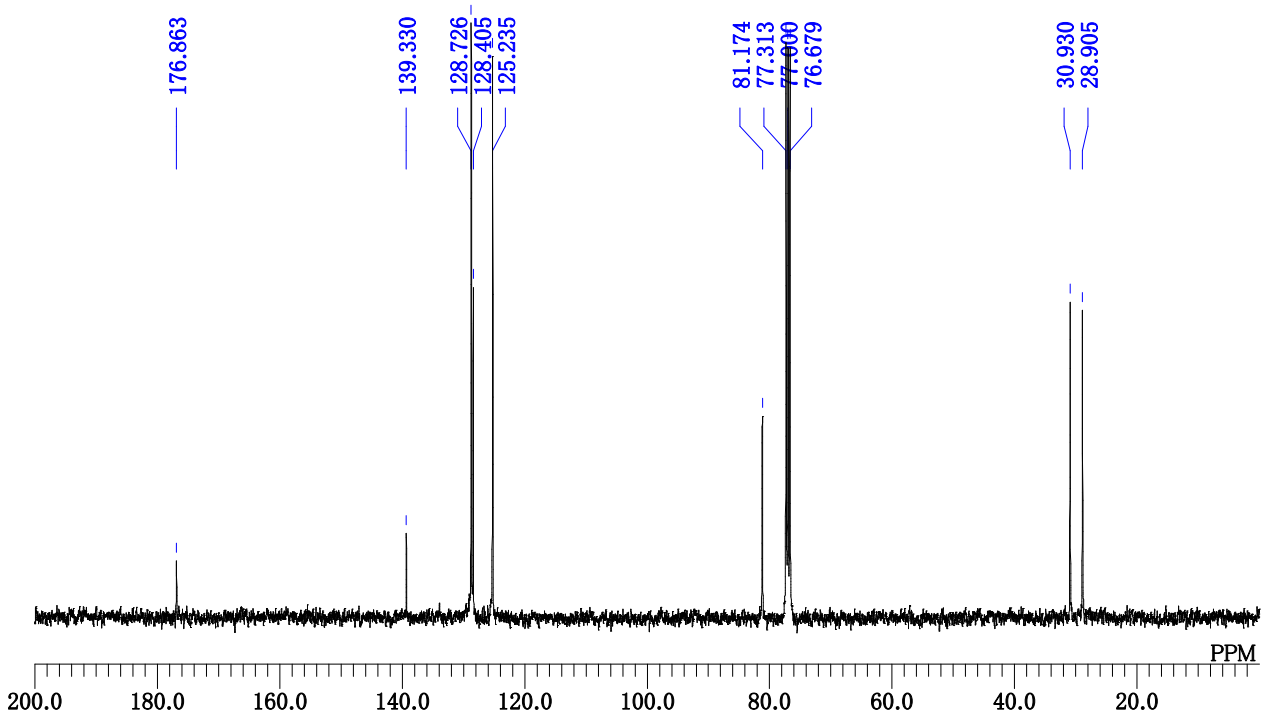


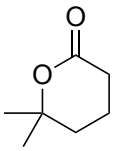
4f

^{1}H NMR: (400 MHz, $CDCl_3$)



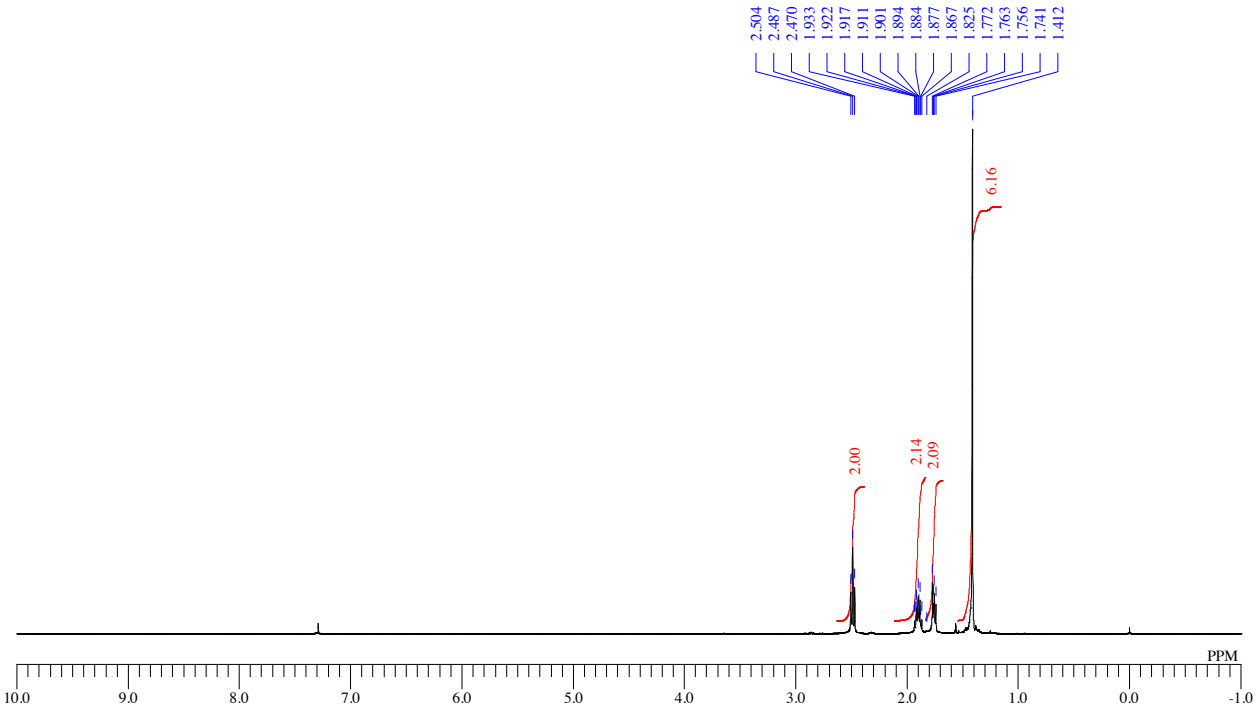
^{13}C NMR: (100 MHz, $CDCl_3$)





4g

^{1}H NMR: (400 MHz, $CDCl_3$)



^{13}C NMR: (100 MHz, $CDCl_3$)