

Supporting Information

Environmentally benign esterification and disproportionation using activation of acid anhydrides by molecular sieves 3A

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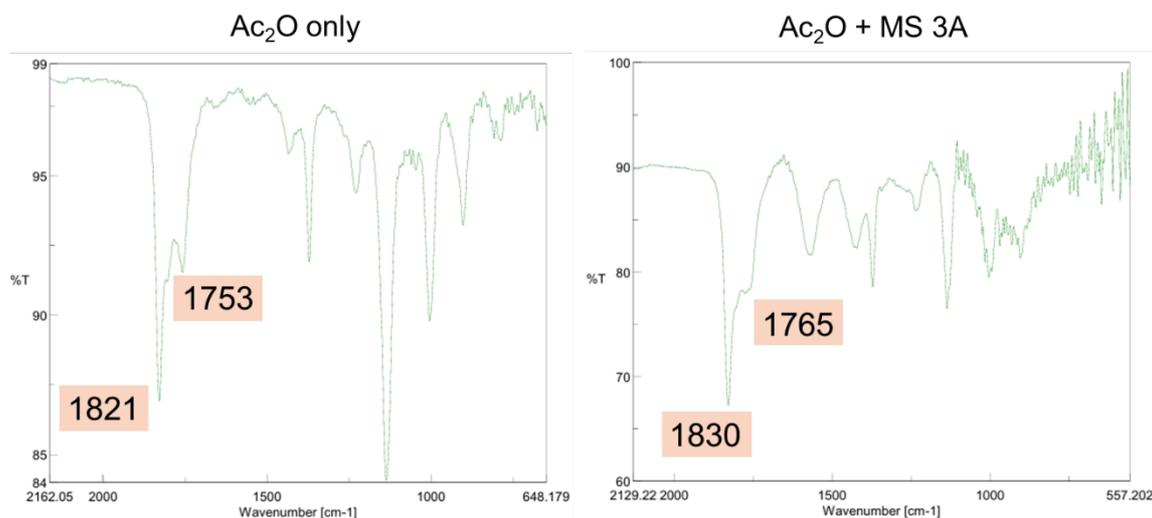
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General Information

All reagents were purchased from commercial sources and used without further purification. The progress of the reactions was monitored by TLC on SiO₂ 60 F254 on aluminum plates. Using TMS as an internal standard, ¹H and ¹³C NMR spectra were recorded on a JEOL JMN-ECZ400S spectrometer (400 MHz and 100 MHz, respectively). The assignments of the ¹³C NMR were reaffirmed by DEPT experiments. IR spectra were recorded with a JASCO FT/IR-4200 spectrometer equipped with an ATM detector. High-resolution mass spectra (HRMS) data were obtained from a Bruker compact mass spectrometer APCI-TOF set at positive mode.

IR spectra of Ac₂O in the presence/absence of MS 3A



Typical procedure

In a screw capped test tube, to a solution of 4-methylbenzyl alcohol **1** (183.6 mg, 1.5 mmol) in toluene (15 mL), were added MS 3A (1.5 g), acetic anhydride **2a** (0.3 mL, 3.0 mmol). After sealing the tube, the mixture was heated at 30 °C for 1d on an oil bath. MS 3A was filtered off and the filtrate was concentrated under reduced pressure to afford colorless oil. The ¹H NMR spectrum of the reaction mixture showed only signals of produced **3a** and starting materials. The mixture was subjected to short column chromatography on silica gel (hexane/ EtOAc = 9/1) to isolate **3a** as a colorless oil (235.6 mg, 1.43 mmol, 96%). When other substrates or reaction conditions were used, experiments were conducted in the same way.

Structural determination was performed by comparing the spectra with those of authentic samples. In the case of disproportionation, it was quite difficult to isolate products (unsymmetrical anhydrides **8** and symmetrical anhydrides **9**) because of their similar properties and easy decomposition on silica gel. Therefore, after concentration, the reaction mixture was analyzed by NMR spectra.

(4-Methylphenyl)methyl ethanoate (**3a**)¹

¹H NMR (400 MHz, CDCl₃) δ 7.26 (d, *J* = 7.6 Hz, 2H), 7.18 (d, *J* = 7.6 Hz, 2H), 5.07 (s, 2H), 2.36 (s, 3H), 2.09 (s, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 171.0 (C), 138.2 (C), 133.0 (C), 129.3 (CH), 128.5 (CH), 66.4 (CH₂), 21.3 (CH₃), 21.1 (CH₃).

(4-Methylphenyl)methyl propanoate (**3b**)²

Colorless oil (Yield: 95%). ¹H NMR (400 MHz, CDCl₃) δ 7.25 (d, *J* = 7.6 Hz, 2H), 7.17 (d, *J* = 7.6 Hz, 2H), 5.08 (s, 2H), 2.37 (q, *J* = 7.6 Hz, 3H), 2.35 (s, 2H), 1.15 (t, *J* = 7.6 Hz, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 174.5 (C), 138.1 (C), 133.2 (C), 129.3 (CH), 128.5 (CH), 66.2 (CH₂), 27.7 (CH₂), 21.3 (CH₃), 9.2 (CH₃).

(4-Methylphenyl)methyl 2-methylpropanoate (3c)³

Colorless oil (Yield: 95%). ¹H NMR (400 MHz, CDCl₃) δ 7.25 (d, *J* = 8.0 Hz, 2H), 7.17 (d, *J* = 8.0 Hz, 2H), 5.07 (s, 2H), 2.59 (sep, *J* = 7.2 Hz, 1H), 2.35 (s, 3H), 1.18 (d, *J* = 7.2 Hz, 6H); ¹³C NMR (100 MHz, CDCl₃) δ 177.1 (C), 138.0 (C), 133.4 (C), 129.3 (CH), 128.2 (CH), 66.1 (CH₂), 34.1 (CH), 21.3 (CH₃), 19.1 (CH₃).

(4-Methylphenyl)methyl 2,2-dimethylpropanoate (3d)⁴

Colorless oil (Yield: 89%). ¹H NMR (400 MHz, CDCl₃) δ 7.23 (d, *J* = 8.4 Hz, 2H), 7.16 (d, *J* = 8.4 Hz, 2H), 5.07 (s, 2H), 2.35 (s, 3H), 1.22 (s, 9H); ¹³C NMR (100 MHz, CDCl₃) δ 178.5 (C), 137.8 (C), 133.5 (C), 129.2 (CH), 127.9 (CH), 66.1 (CH₂), 38.9 (C), 27.1 (CH₃), 21.3 (CH₃).

(4-Methylphenyl)methyl hydrogen butanedioate (3e)⁵

Colorless solid (Yield: 83%). ¹H NMR (400 MHz, CDCl₃) δ 7.24 (d, *J* = 7.6 Hz, 2H), 7.17 (d, *J* = 7.6 Hz, 2H), 5.10 (s, 2H), 2.64–2.73 (m, 4H), 2.35 (s, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 177.7 (C), 172.1 (C), 138.3 (C), 132.7 (C), 129.3 (CH), 128.5 (CH), 66.7 (CH₂), 29.0 (CH₂), 28.9 (CH₂), 21.3 (CH₃).

(4-Methylphenyl)methyl benzoate (3f)⁶

Colorless oil (Yield: 83%). ¹H NMR (400 MHz, CDCl₃) δ 8.07 (dd, *J* = 7.2, 1.2 Hz, 2H), 7.55 (ddd, *J* = 8.0, 7.2, 1.2 Hz, 1H), 7.42 (dd, *J* = 8.0, 7.2 Hz, 2H), 7.34 (d, *J* = 8.0 Hz, 2H), 7.20 (d, *J* = 8.0 Hz, 2H), 5.33 (s, 2H), 2.37 (s, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 166.6 (C), 138.2 (C), 133.1 (CH), 133.0 (CH), 130.3 (C), 129.8 (CH), 129.4 (CH), 128.44 (CH), 128.42 (C), 66.8 (CH₂), 21.3 (CH₃).

1,1-Dimethylethyl (4-methylphenyl)methyl carbonate (3g)⁷

Colorless oil (Yield: 82%). ¹H NMR (400 MHz, CDCl₃) δ 7.27 (d, *J* = 8.0 Hz, 2H), 7.16 (d, *J* = 8.0 Hz, 2H), 5.05 (s, 2H), 2.34 (s, 3H), 1.48 (s, 9H); ¹³C NMR (100 MHz, CDCl₃)

δ 153.6 (C), 138.2 (C), 132.7 (C), 129.3 (CH), 128.5 (CH), 82.2 (C), 68.7 (CH₂), 27.9 (CH₃), 21.3 (CH₃).

4-Methoxyphenyl ethanoate (6b)⁸

Yellow solid (Yield: quant.). ¹H NMR (400 MHz, CDCl₃) δ 7.00 (d, *J* = 8.8 Hz, 2H), 6.88 (d, *J* = 8.8 Hz, 2H), 3.79 (s, 3H), 2.28 (s, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 170.0 (C), 157.3 (C), 144.2 (C), 122.4 (CH), 114.5 (CH), 55.7 (CH₃), 21.2 (CH₃).

4-Nitrophenyl ethanoate (6c)⁹

Colorless solid (Yield: quant.). ¹H NMR (400 MHz, CDCl₃) δ 8.26 (d, *J* = 9.2 Hz, 2H), 7.27 (d, *J* = 9.2 Hz, 2H), 2.34 (s, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 168.5 (C), 155.4 (C), 145.4 (CH), 125.3 (CH), 122.5 (C), 21.2 (CH₃).

4-Methoxyphenyl 1,1-dimethylethyl carbonate (6d)¹⁰

Colorless solid (Yield: quant.). ¹H NMR (400 MHz, CDCl₃) δ 7.08 (d, *J* = 8.8, 2H), 6.88 (d, *J* = 8.8, 2H), 3.79 (s, 3H), 1.55 (s, 9H); ¹³C NMR (100 MHz, CDCl₃) δ 157.3 (C), 152.4 (C), 144.8 (C), 122.2 (CH), 114.5 (CH), 55.7 (CH₃), 27.8 (CH₃).

Ethanoic 4-methylbenzoic anhydride (8a)¹¹

Colorless solid (Yield: 57%). ¹H NMR (400 MHz, CDCl₃) δ 7.93 (d, *J* = 8.0, 2H), 7.27 (d, *J* = 8.0, 2H), 2.42 (s, 3H), 2.36 (s, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 162.7 (C), 162.4 (C), 145.7 (C), 130.7 (CH), 129.6 (CH), 125.9 (C), 22.5 (CH₃), 21.9 (CH₃).

Benzoic ethanoic anhydride (8b)¹²

Yellow solid (Yield: 57%). ¹H NMR (400 MHz, CDCl₃) δ 8.06 (d, *J* = 7.6, 2H), 7.63–7.66 (m, 1H), 7.47–7.51 (m, 2H), 2.38 (s, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 166.6 (C), 162.3 (C), 134.6 (CH), 130.7 (CH), 130.6 (C), 128.9 (CH), 22.5 (CH₃).

4-Chlorobenzoic ethanoic anhydride (8c)¹³

Colorless oil (Yield: 40%). ¹H NMR (400 MHz, CDCl₃) δ 7.97 (d, *J* = 8.4 Hz, 2H), 7.47 (d, *J* = 8.4 Hz, 2H), 2.38 (s, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 166.2 (C), 161.5 (C), 141.2 (C), 131.9 (CH), 129.3 (CH), 127.1 (C), 22.6 (CH₃).

Ethanoic propionic anhydride (8d)¹⁴

¹H NMR (400 MHz, C₆D₆) δ 1.89 (q, *J* = 7.6 Hz, 2H), 1.50 (s, 3H), 0.77 (t, *J* = 7.6 Hz, 3H); ¹³C NMR (100 MHz, C₆D₆) δ 177.5 (C), 165.9 (C), 28.2 (CH₂), 21.1 (CH₃), 19.9 (CH₃).

Ethanoic hexa-2,4-dienoic anhydride (8e)¹⁵

Brown oil (Yield: 60%). ¹H NMR (400 MHz, CDCl₃) δ 7.32–7.36 (m, 1H), 6.20–6.26 (m, 2H), 5.77 (d, *J* = 15.2 Hz, 1H), 2.26 (s, 3H), 1.89 (s, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 166.9 (C), 162.6 (C), 149.3 (CH), 142.8 (CH), 129.7 (CH), 117.4 (CH), 22.3 (CH₃), 19.0 (CH₃).

(1,1-Dimethylethoxy)carbonic 4-methylbenzoic anhydride (8f)

Colorless oil (Yield: 17%). ¹H NMR (400 MHz, CDCl₃) δ 7.95 (d, *J* = 8.4, 2H), 7.27 (d, *J* = 8.4, 2H), 2.43 (s, 3H), 1.59 (s, 9H); HRMS (APCI/TOF) calcd. for (M-H⁺) C₁₃H₁₅O₄: 235.0965, found: 235.0984.

The yield of this compound was very low, and isolation was difficult. Therefore, although analysis by ¹H NMR using the reaction mixture was possible, ¹³C NMR measurement could not be performed due to the low product concentration.

(1,1-Dimethylethoxy)carbonic benzoic anhydride (8g)¹⁶

Colorless oil (Yield: 12%). ¹H NMR (400 MHz, CDCl₃) δ 8.06 (dd, *J* = 7.6, 1.2 Hz, 2H), 7.63 (ddd, *J* = 8.0, 7.6, 1.2 Hz, 1H), 7.48 (dd, *J* = 8.0, 7.6 Hz, 2H), 1.59 (s, 9H).

4-Chlorobenzoic (1,1-dimethylethoxy)carbonic anhydride (8h)

White solid (Yield: 10%). ^1H NMR (400 MHz, CDCl_3) δ 7.99 (d, $J = 8.8$, 2H), 7.44 (d, $J = 8.8$, 2H), 1.58 (s, 9H); HRMS (APCI/TOF) calcd. for (M- $\text{C}_4\text{H}_9\text{OCl}$) $\text{C}_8\text{H}_5\text{O}_3$: 149.0233, found: 149.0218.

(1,1-Dimethylethoxy)carbonic propionic anhydride (8i)

^1H NMR (400 MHz, C_6D_6) δ 1.92 (q, $J = 7.2$ Hz, 2H), 1.06 (s, 9H), 0.79 (t, $J = 7.2$ Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 169.8 (C), 147.3 (C), 67.9 (C), 31.0 (CH_3), 28.3 (CH_2), 26.8 (CH_3); HRMS (APCI/TOF) calcd. for (M) $\text{C}_8\text{H}_{14}\text{O}_4$: 175.0965, found: 175.0908.

Hexa-2,4-dienoic (1,1-dimethylethoxy)carbonic anhydride (8j)

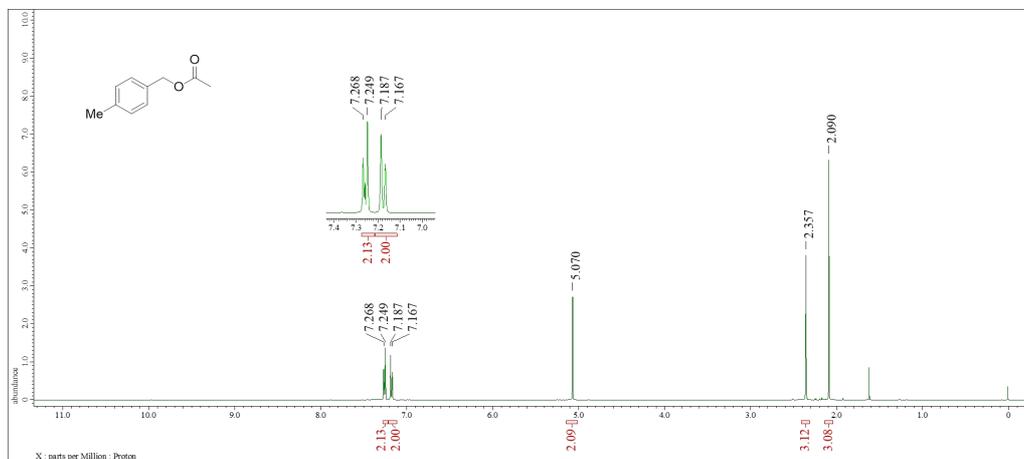
Colorless oil (Yield: 80%). ^1H NMR (400 MHz, CDCl_3) δ 7.33–7.39 (m, 1H), 6.22–6.26 (m, 2H), 5.74 (d, $J = 14.8$ Hz, 1H), 1.54 (s, 9H), 1.47 (d, 5.2 Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 161.9 (C), 149.0 (CH), 147.4 (C), 142.3 (CH), 129.7 (CH), 116.6 (CH), 85.2 (C), 27.6 (CH_3), 18.9 (CH_3); HRMS (APCI/TOF) calcd. for (M- $\text{C}_4\text{H}_9\text{O}+\text{C}_4\text{H}_3\text{N}$) $\text{C}_9\text{H}_{10}\text{NO}_3$: 180.0655, found: 180.0612.

Reuse of MS 3A using cylindrical filter paper

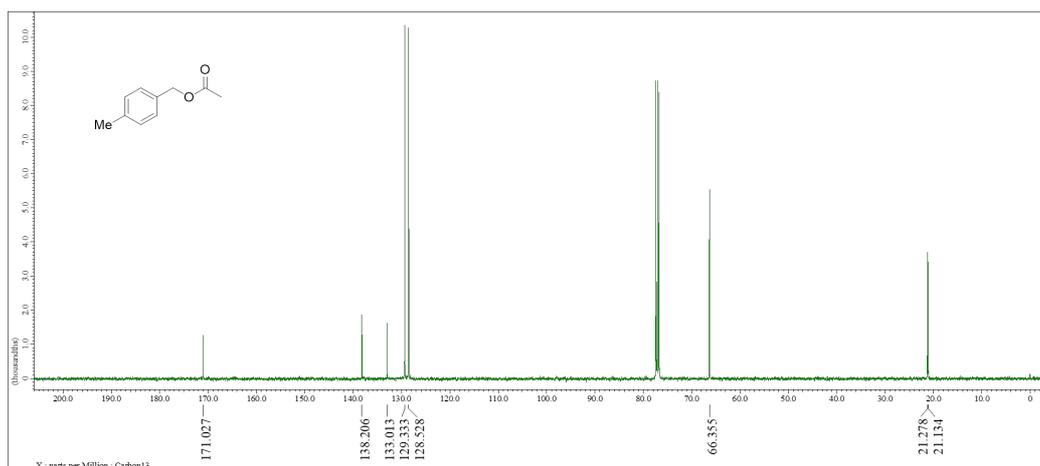
In a screw capped test tube, to a solution of 4-methylbenzyl alcohol **1** (183.3 mg, 1.5 mmol) in toluene (15 mL), propionic anhydride **2b** (0.2 mL, 1.5 mmol) was added. After a cylindrical filter paper containing MS 3A (1.5 g) was dipped, the tube was sealed, and the mixture was heated at 60 °C for 1d on an oil bath. Filter paper thimble was pulled up, and the filtrate was concentrated under reduced pressure to afford colorless oil. The product yield was determined by comparing the integral ratio of **1** and **3b** in the ^1H NMR. The recovered filter paper thimble containing MS 3A was used for another reaction mixture without any treatment. This reuse process repeated 10 times.

(4-Methylphenyl)methyl ethanoate (3a)

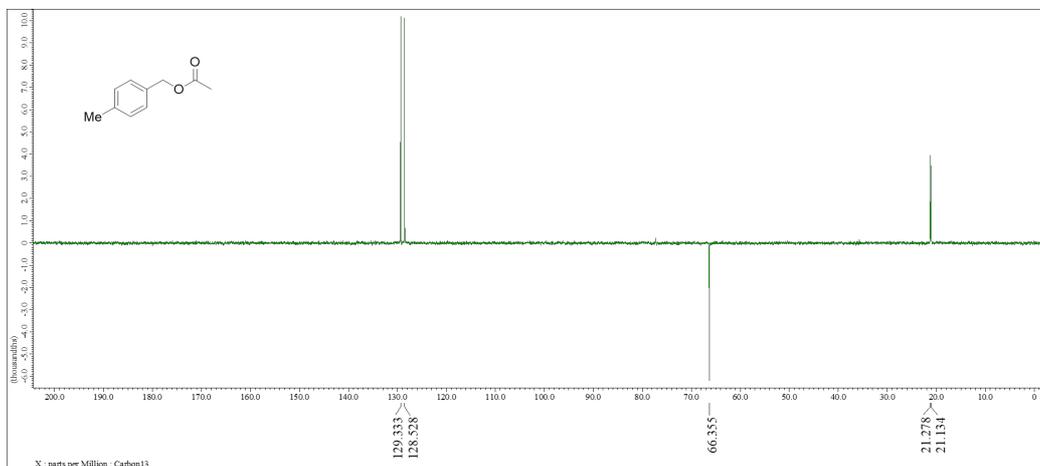
^1H NMR (400 MHz, CDCl_3)



$^{13}\text{C}\{^1\text{H}\}$ NMR (100 MHz, CDCl_3)

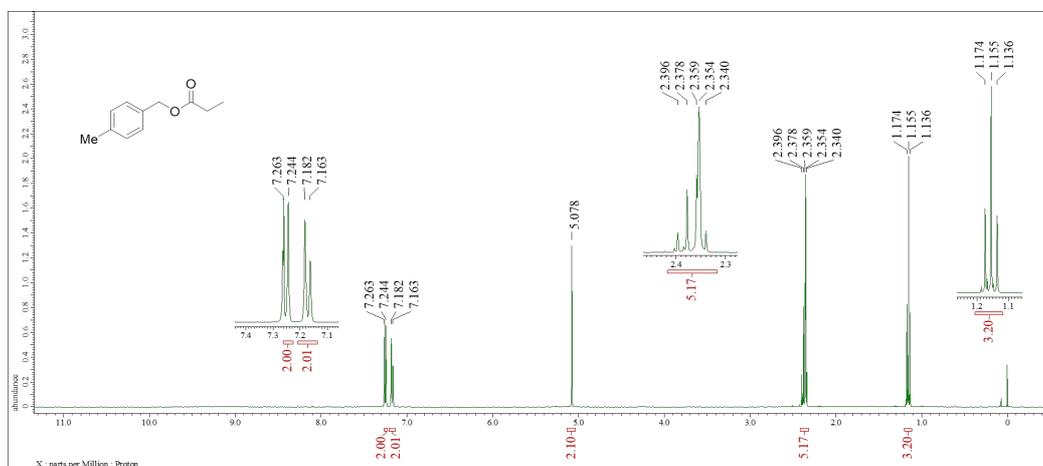


DEPT NMR (100 MHz, CDCl_3)

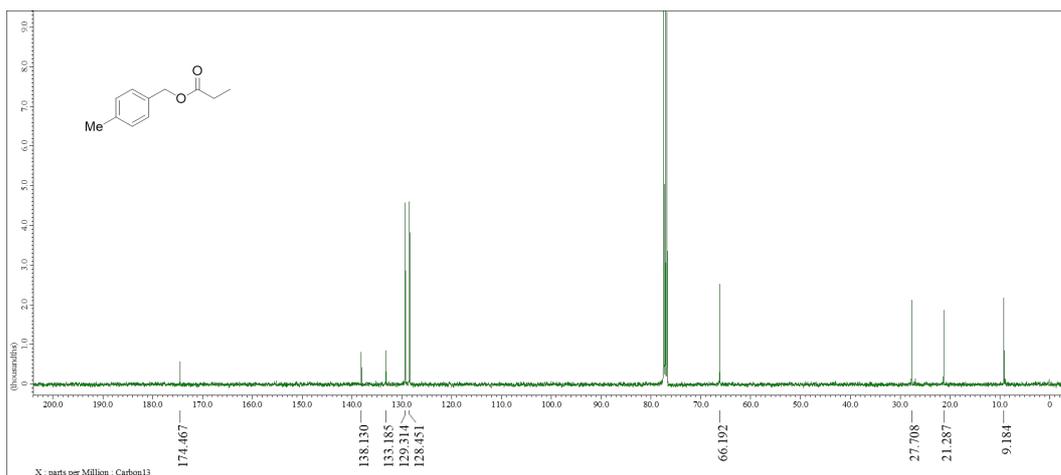


(4-Methylphenyl)methyl propanoate (3b)

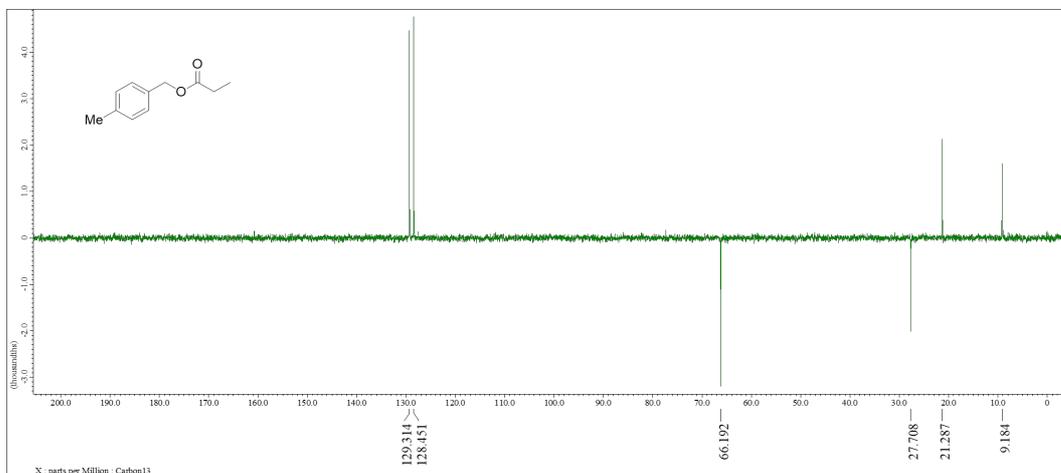
^1H NMR (400 MHz, CDCl_3)



$^{13}\text{C}\{^1\text{H}\}$ NMR (100 MHz, CDCl_3)

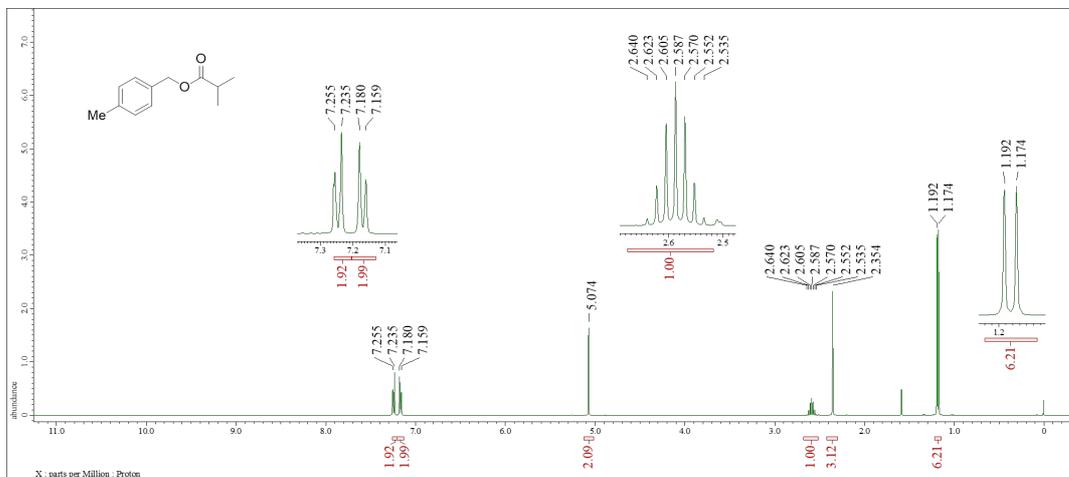


DEPT NMR (100 MHz, CDCl_3)

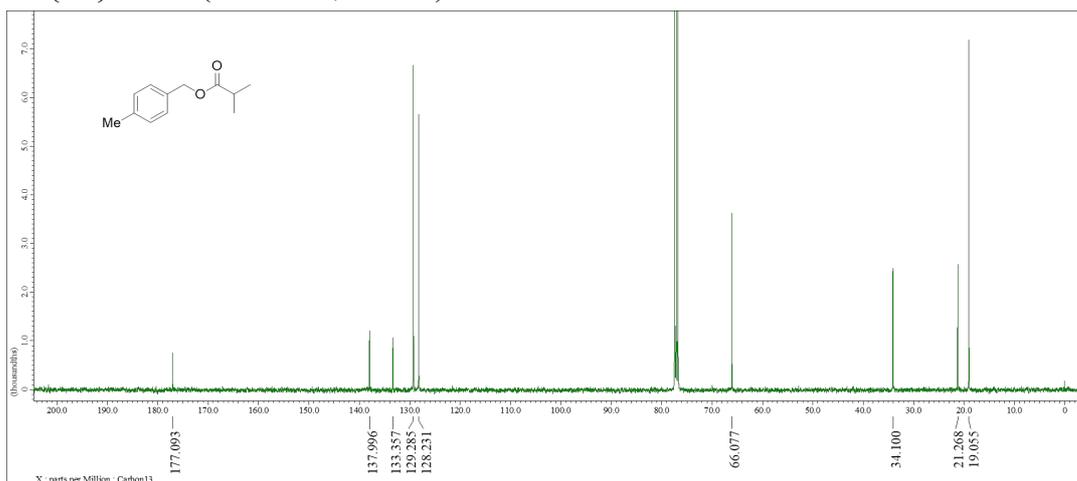


(4-Methylphenyl)methyl 2-methylpropanoate (3c)

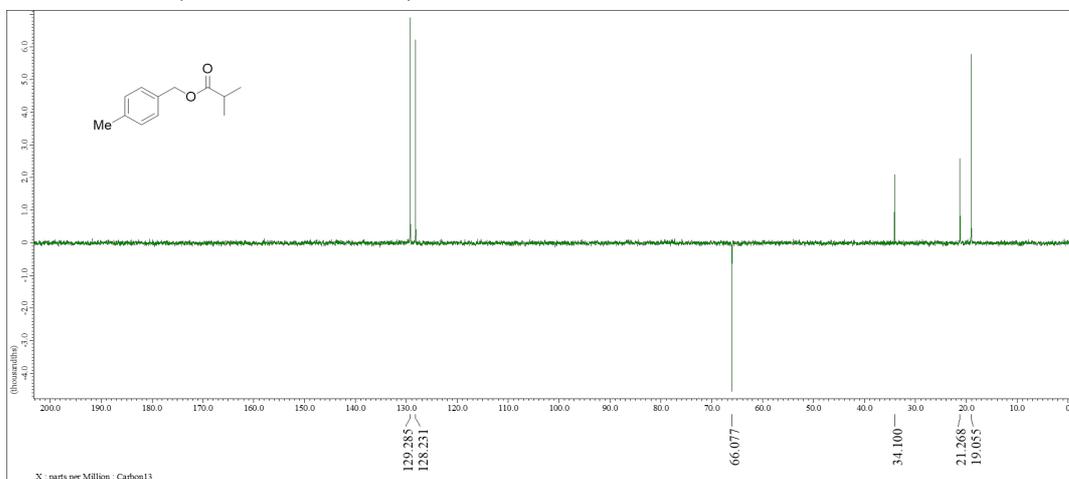
^1H NMR (400 MHz, CDCl_3)



$^{13}\text{C}\{^1\text{H}\}$ NMR (100 MHz, CDCl_3)

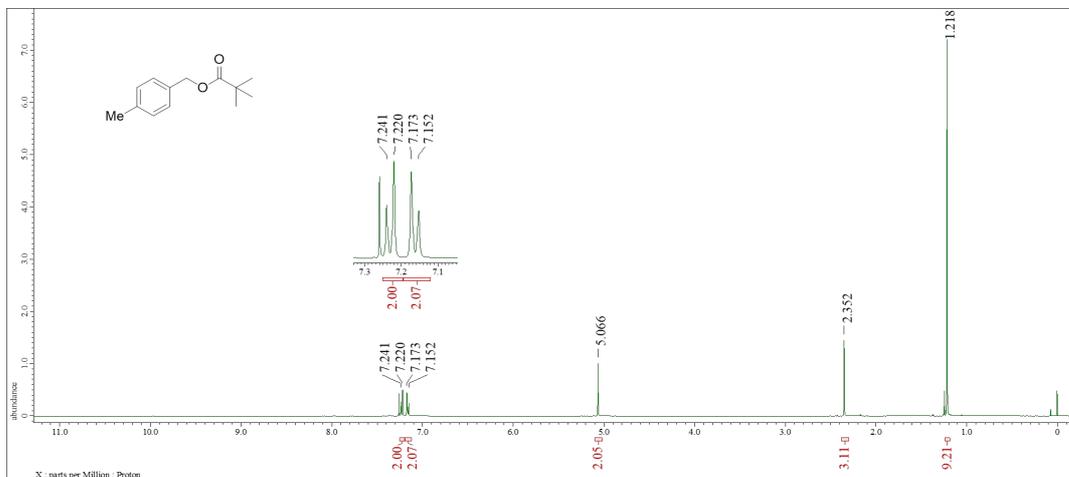


DEPT NMR (100 MHz, CDCl_3)

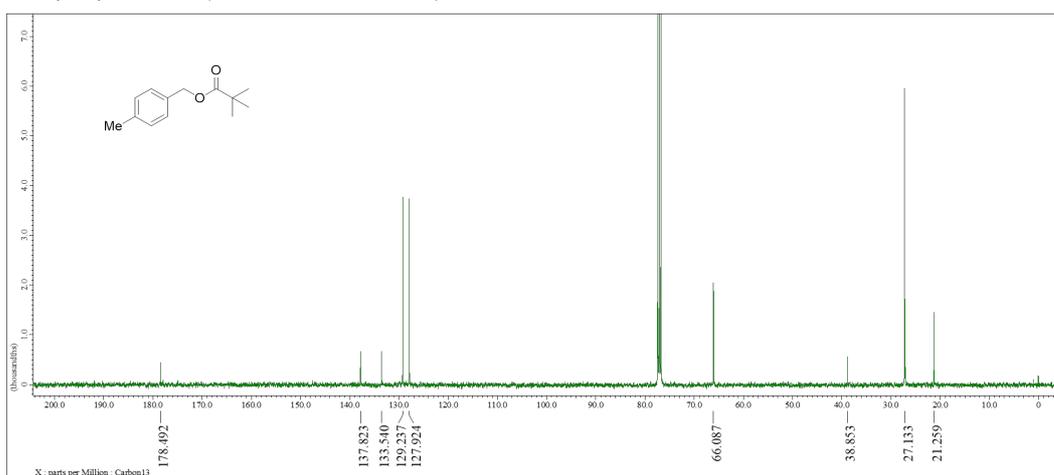


(4-Methylphenyl)methyl 2,2-dimethylpropanoate (3d)

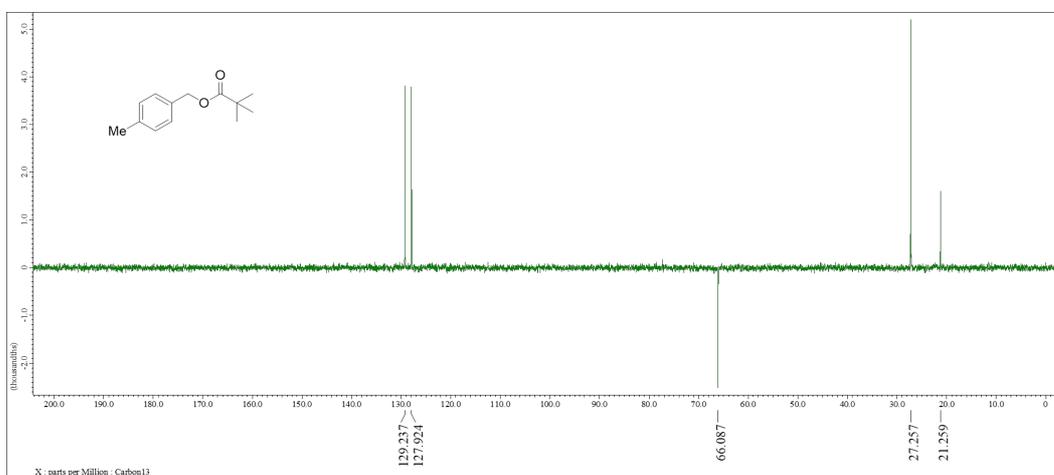
^1H NMR (400 MHz, CDCl_3)



$^{13}\text{C}\{^1\text{H}\}$ NMR (100 MHz, CDCl_3)

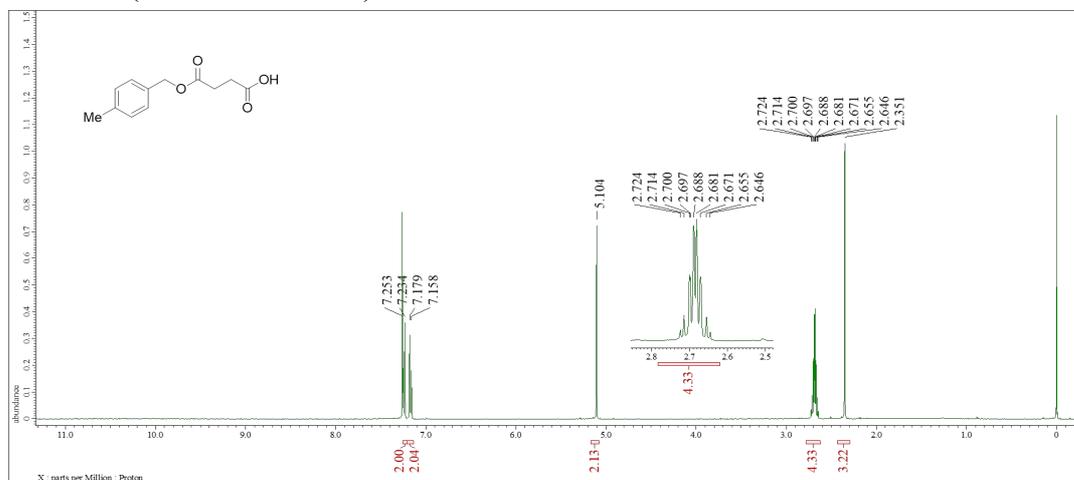


DEPT NMR (100 MHz, CDCl_3)

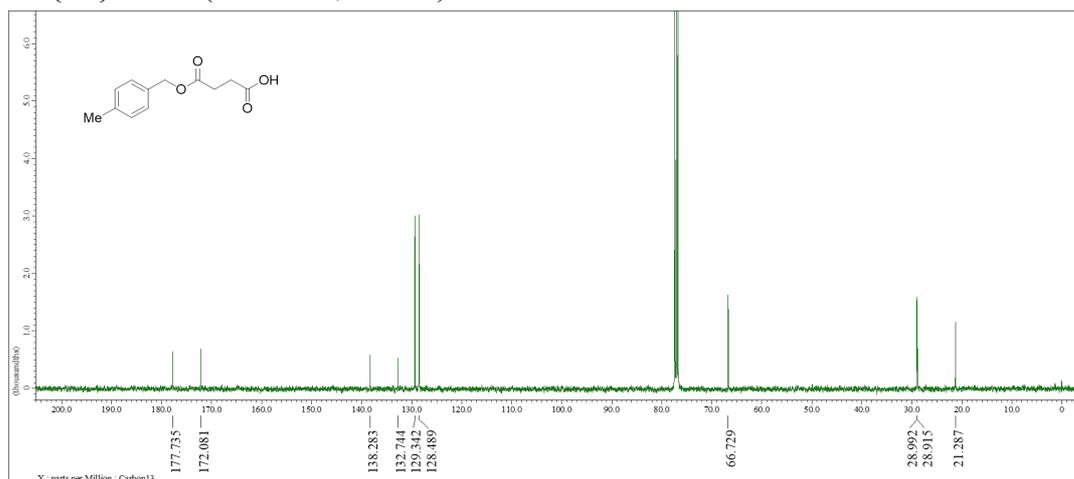


(4-Methylphenyl)methyl hydrogen butanedioate (3e)

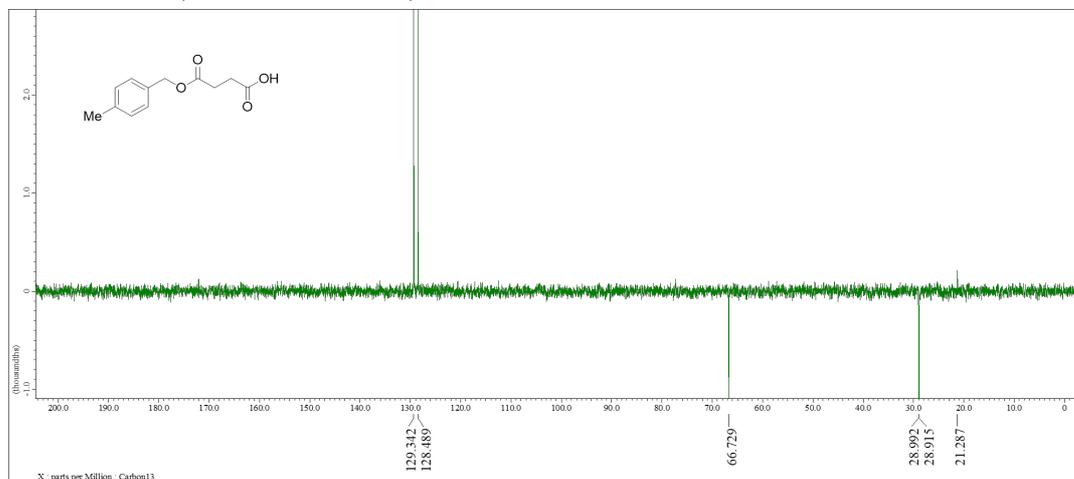
^1H NMR (400 MHz, CDCl_3)



$^{13}\text{C}\{^1\text{H}\}$ NMR (100 MHz, CDCl_3)

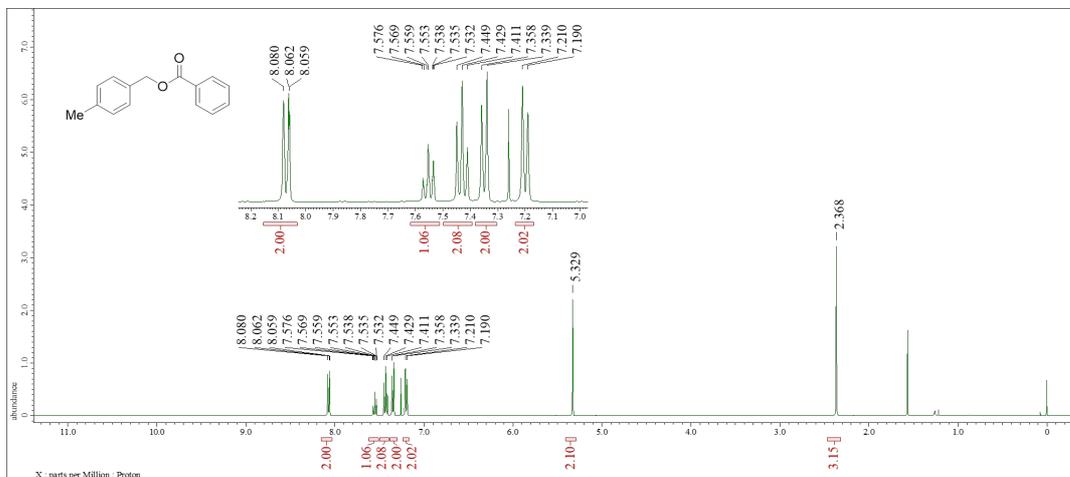


DEPT NMR (100 MHz, CDCl_3)

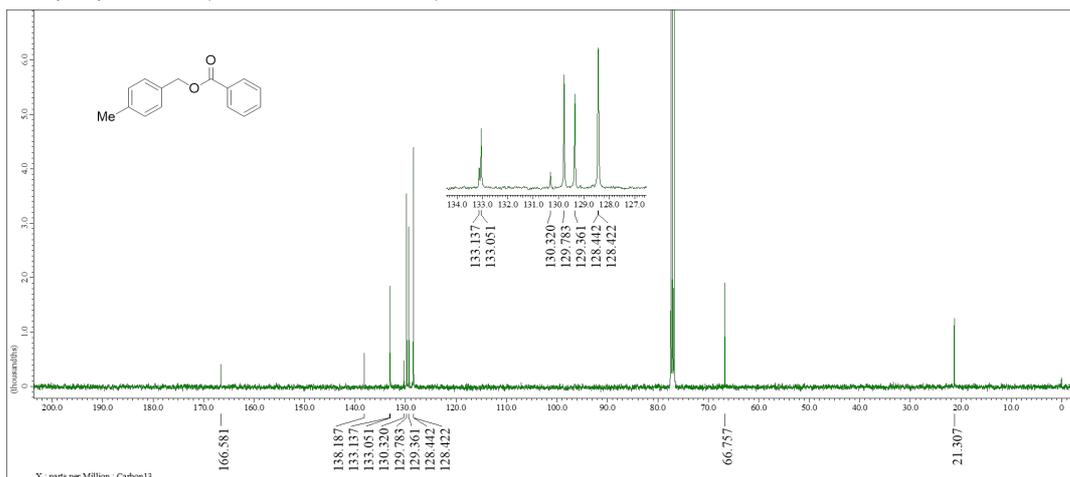


(4-Methylphenyl)methyl benzoate (3f)

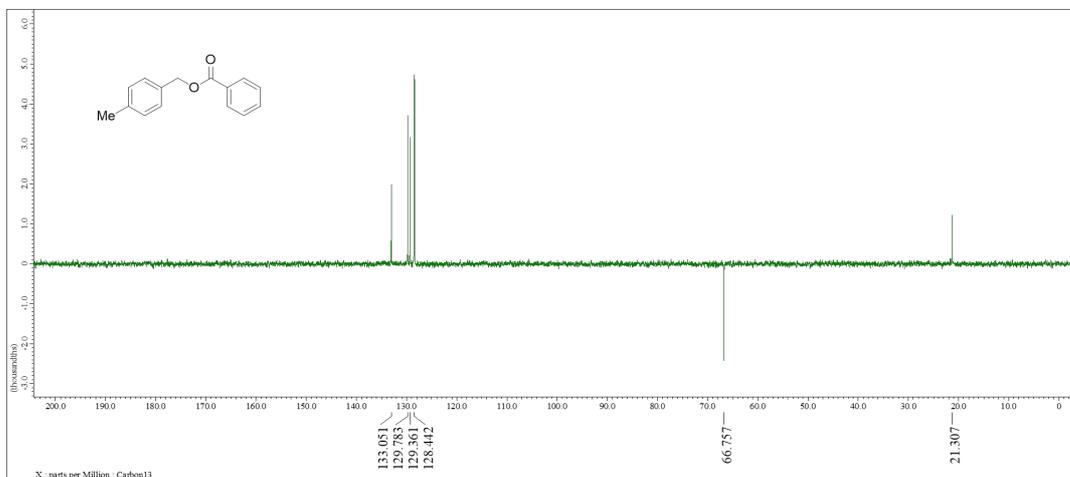
^1H NMR (400 MHz, CDCl_3)



$^{13}\text{C}\{^1\text{H}\}$ NMR (100 MHz, CDCl_3)

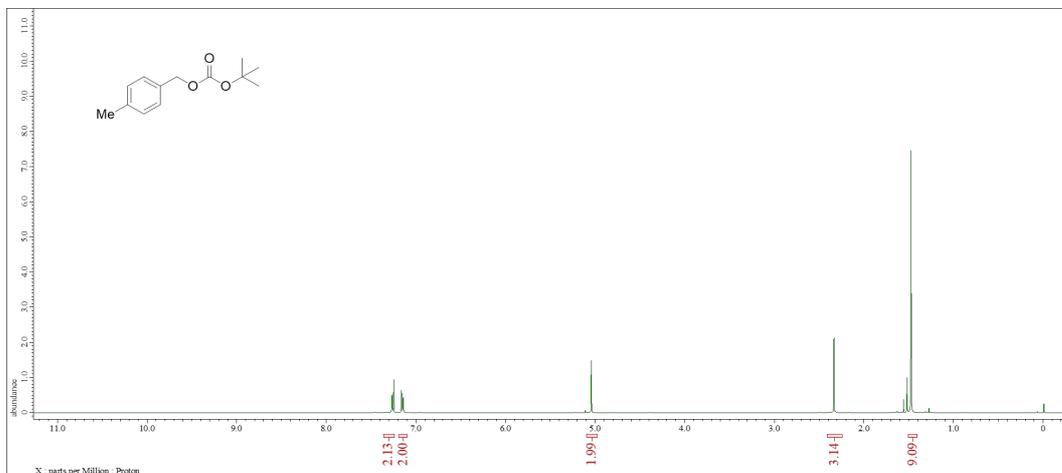


DEPT NMR (100 MHz, CDCl_3)

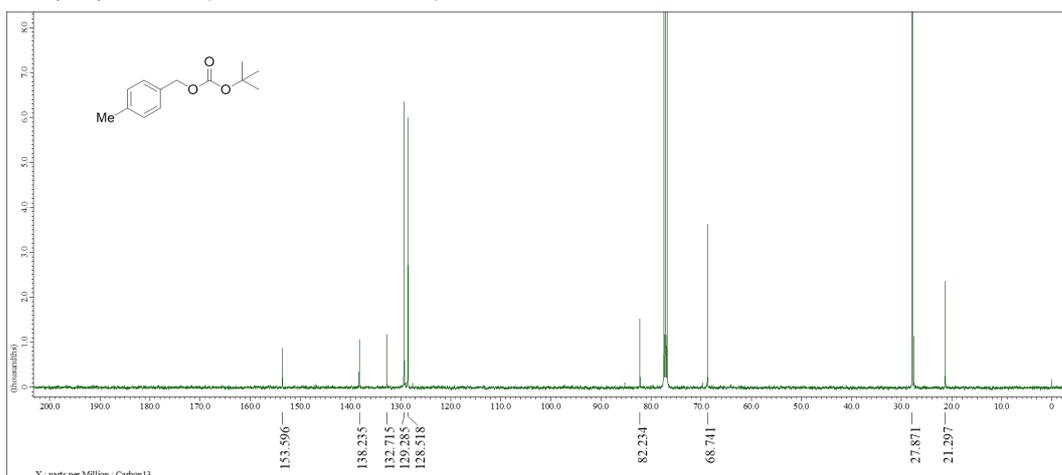


1,1-Dimethylethyl (4-methylphenyl)methyl carbonate (3g)

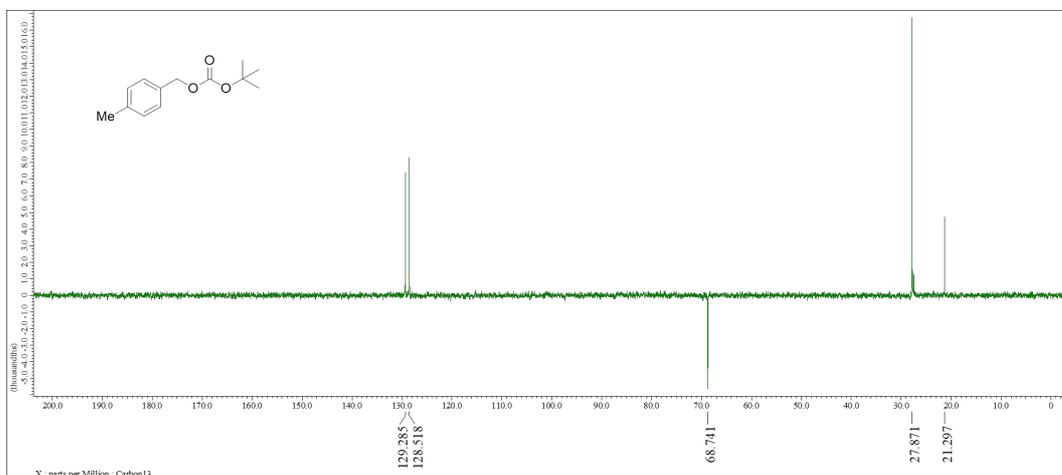
^1H NMR (400 MHz, CDCl_3)



$^{13}\text{C}\{^1\text{H}\}$ NMR (100 MHz, CDCl_3)

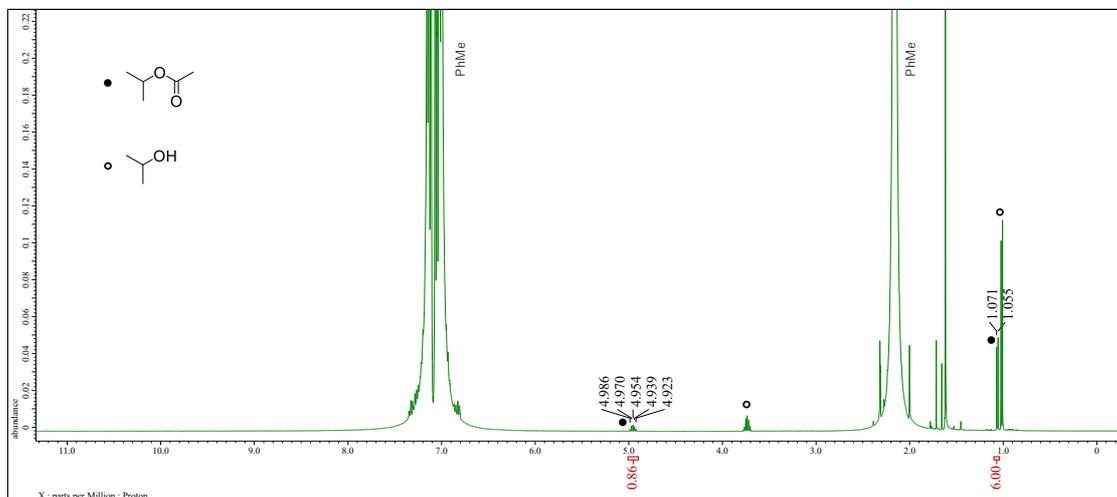


DEPT NMR (100 MHz, CDCl_3)



1-Methylethyl ethanoate (6a)

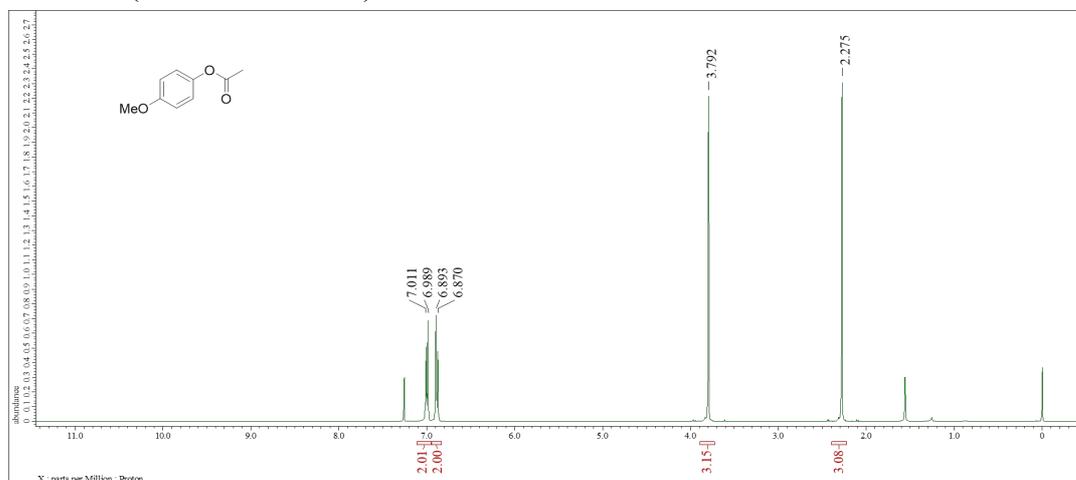
^1H NMR (400 MHz, Non- d)



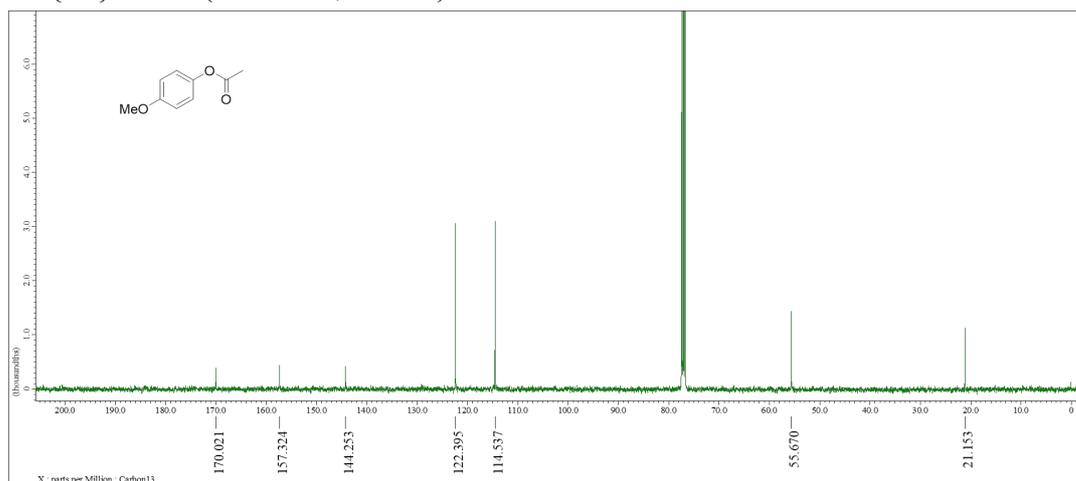
Other alcohols could be used for this protocol. As the boiling points of isopropyl and *tert*-butyl esters are below 100 °C, they were evaporated during the concentration of the reaction mixture. Regarding the reaction of *i*-PrOH and acetic anhydride, the analysis was performed by non-D ^1H NMR using the toluene solution without concentration. When *t*-BuOH was used as a nucleophile, signals of *tert*-butyl group of and *tert*-butyl ester completely overlapped. Thus, we could not judge whether the nucleophilic substitution proceeded or not.

4-Methoxyphenyl ethanoate (6b)

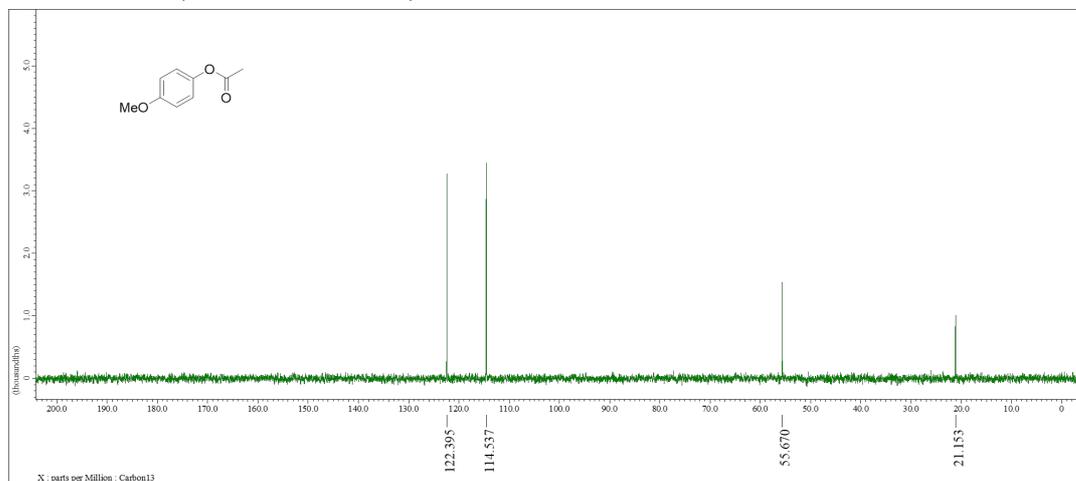
^1H NMR (400 MHz, CDCl_3)



$^{13}\text{C}\{^1\text{H}\}$ NMR (100 MHz, CDCl_3)

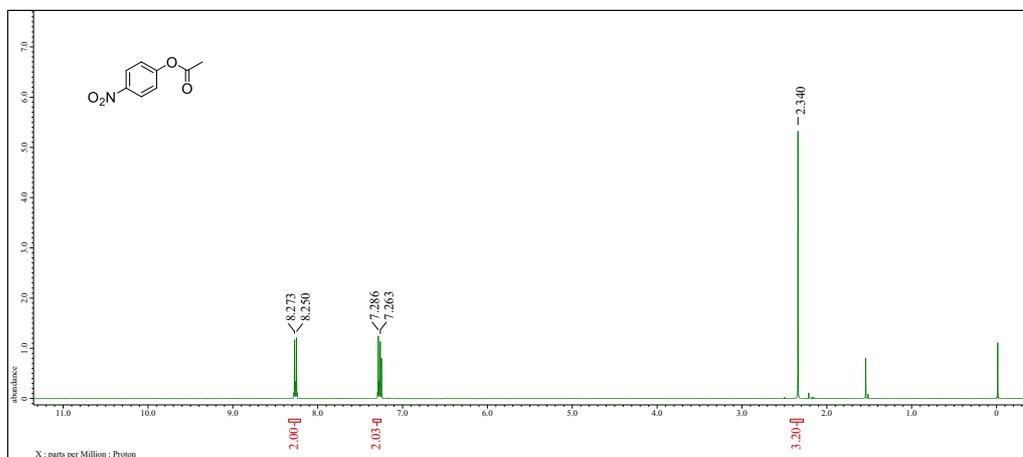


DEPT NMR (100 MHz, CDCl_3)

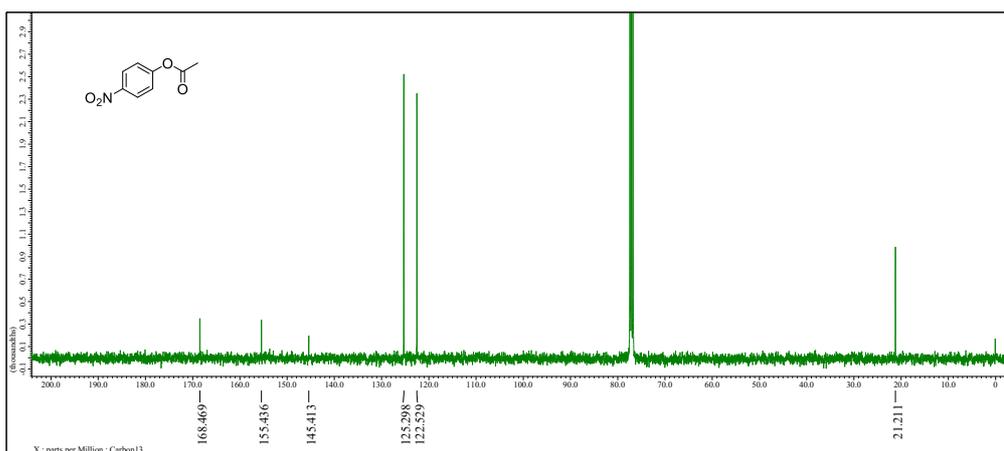


4-Nitrophenyl ethanoate (6c)

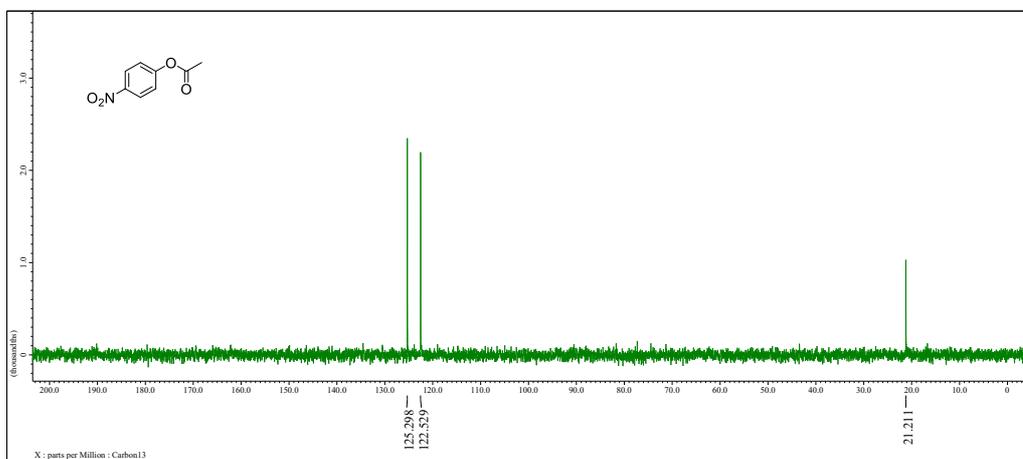
^1H NMR (400 MHz, CDCl_3)



$^{13}\text{C}\{^1\text{H}\}$ NMR (100 MHz, CDCl_3)

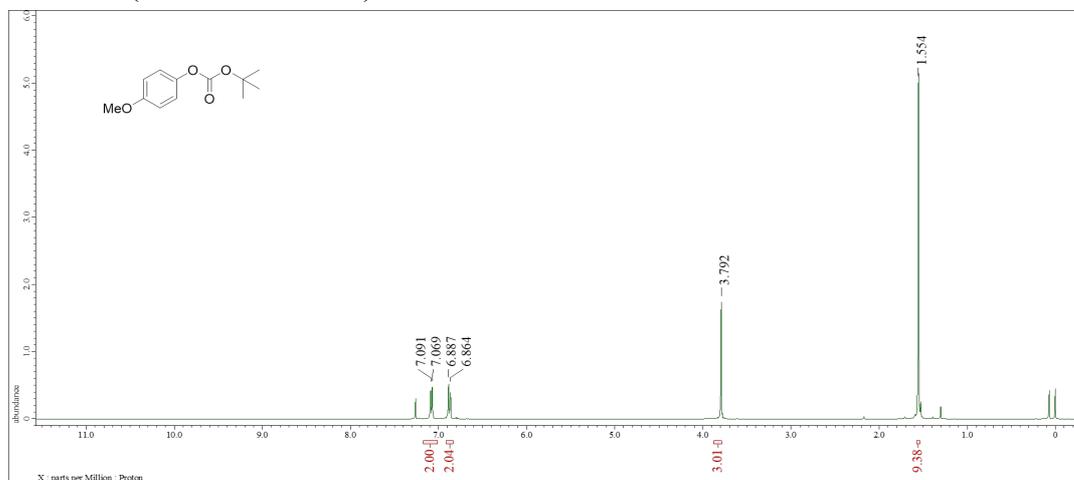


DEPT NMR (100 MHz, CDCl_3)

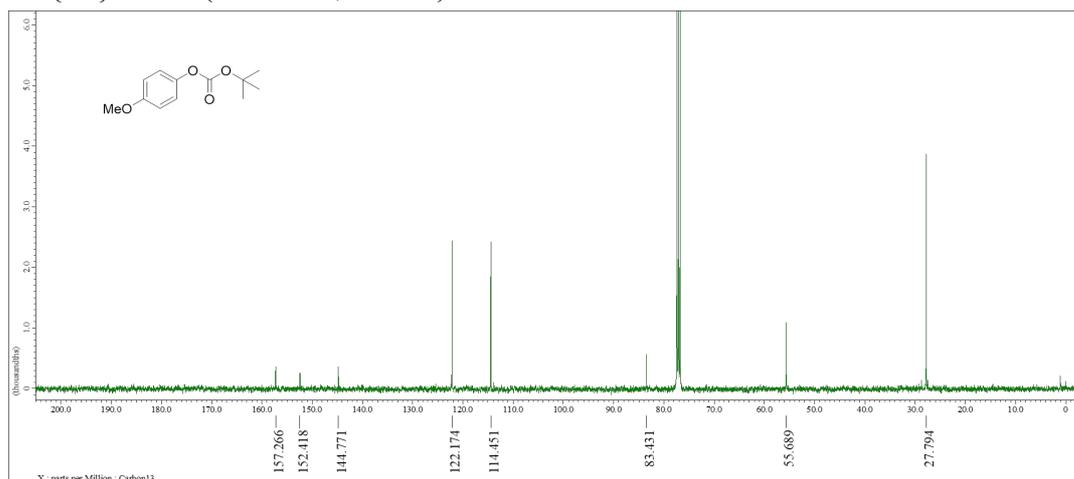


4-Methoxyphenyl 1,1-dimethylethyl carbonate (6d)

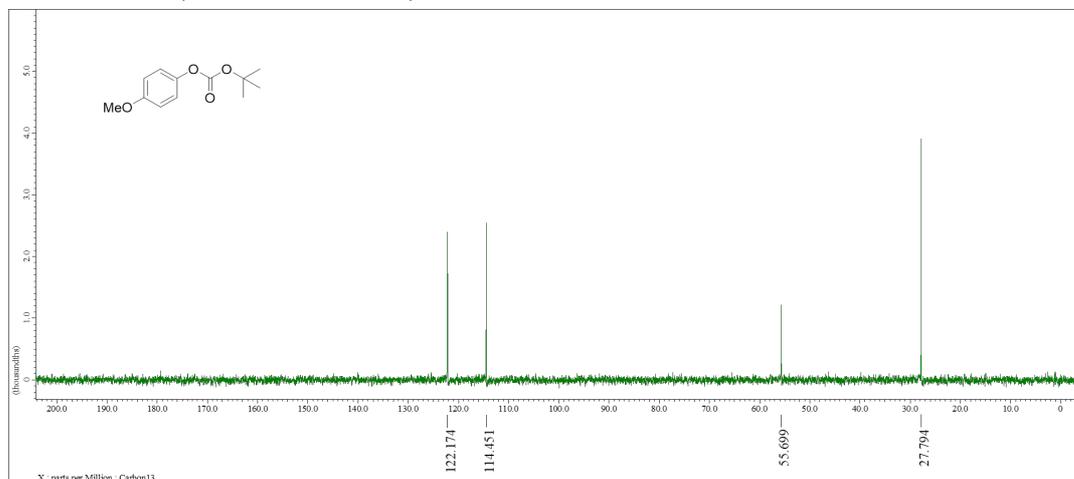
^1H NMR (400 MHz, CDCl_3)



$^{13}\text{C}\{^1\text{H}\}$ NMR (100 MHz, CDCl_3)

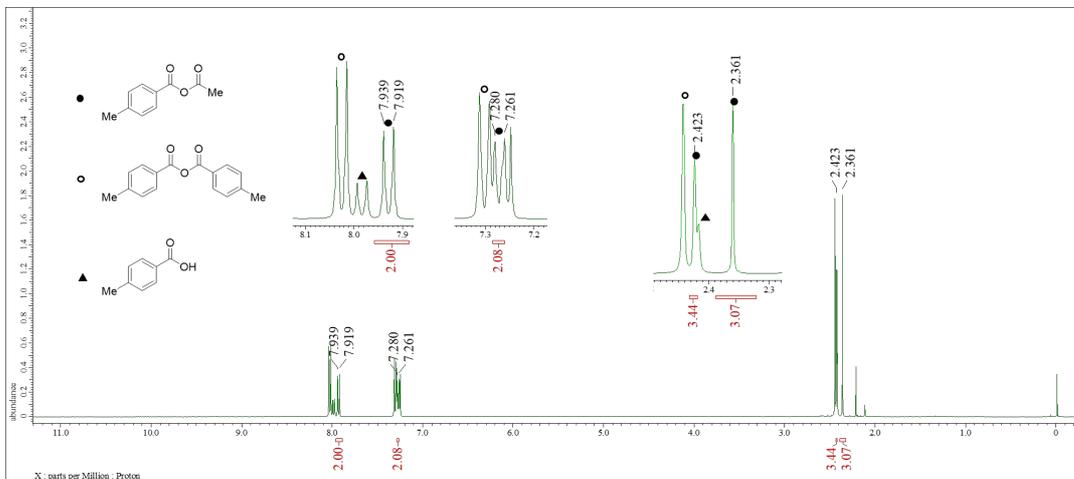


DEPT NMR (100 MHz, CDCl_3)

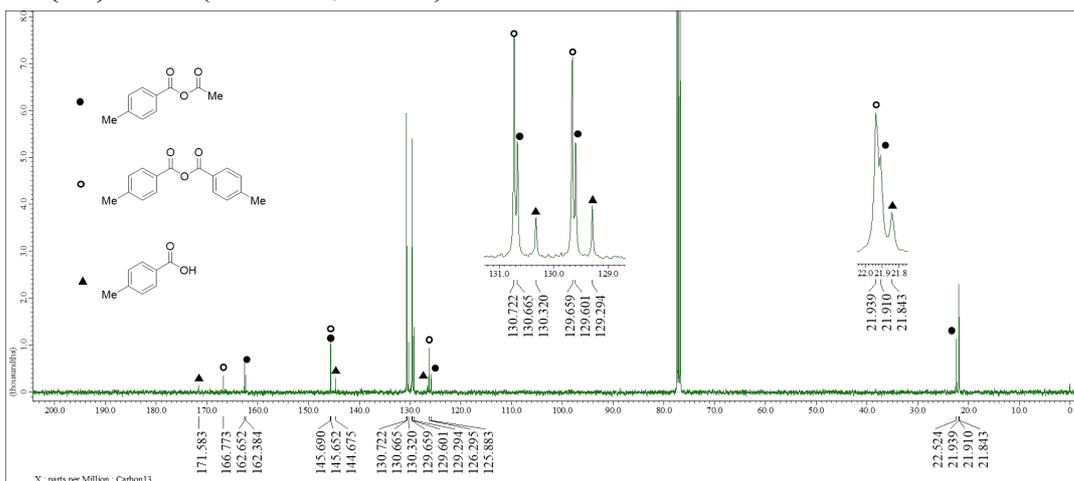


Ethanoic 4-methylbenzoic anhydride (8a)

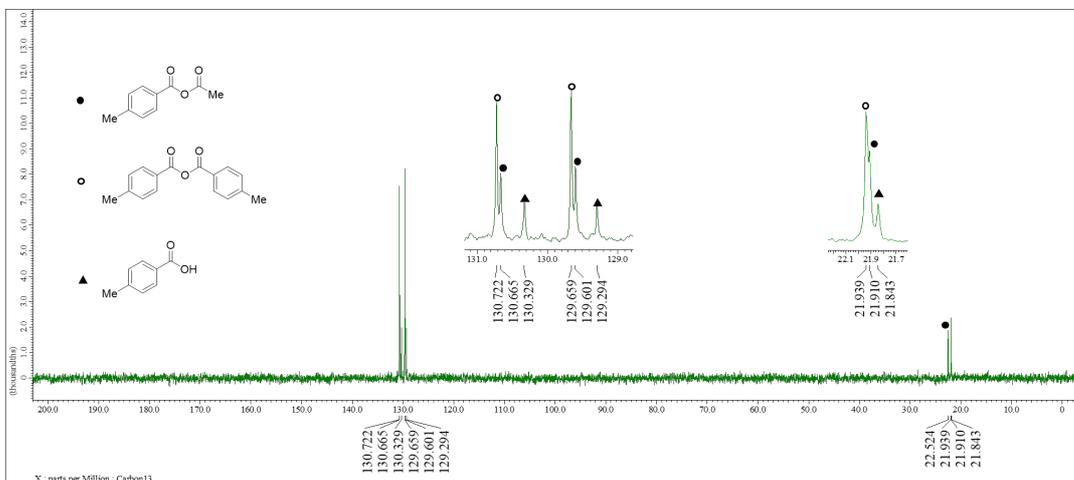
^1H NMR (400 MHz, CDCl_3)



$^{13}\text{C}\{^1\text{H}\}$ NMR (100 MHz, CDCl_3)

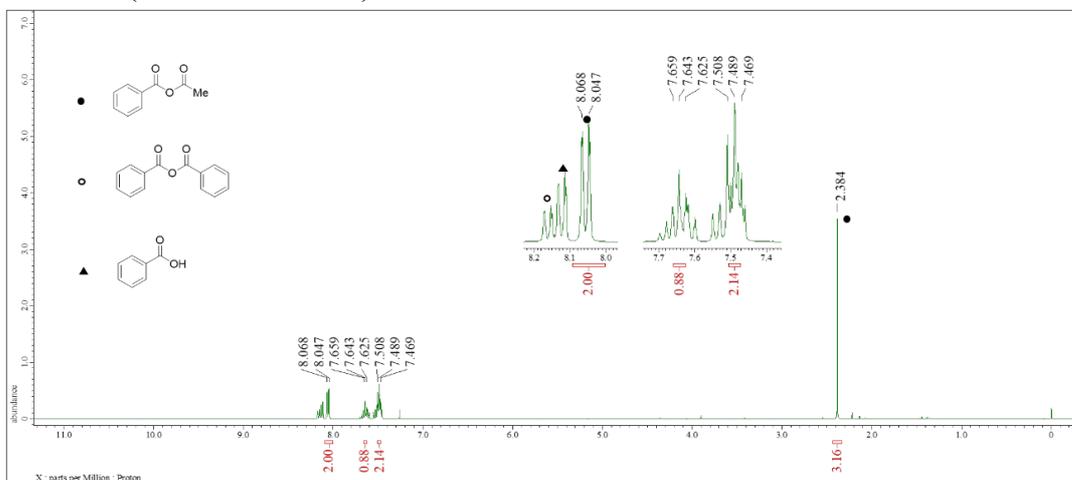


DEPT NMR (100 MHz, CDCl_3)

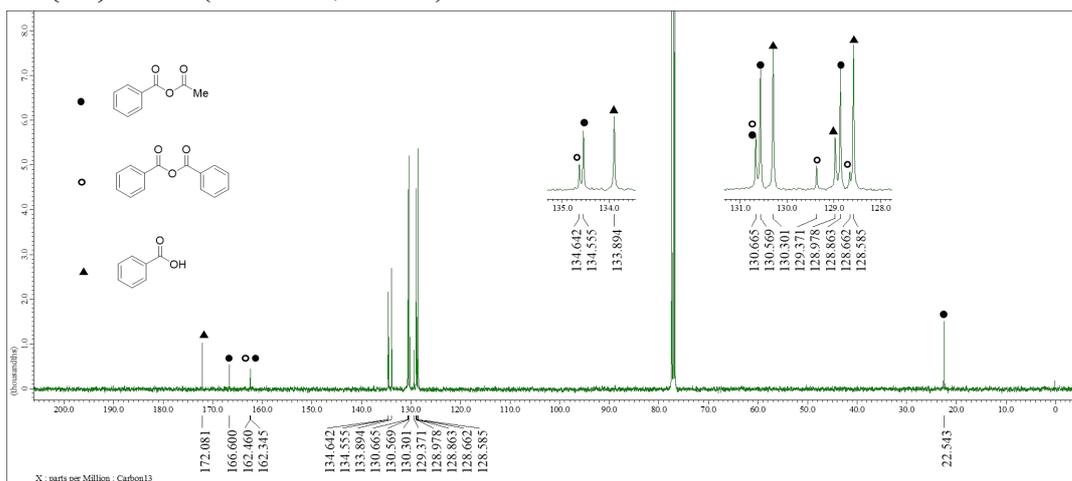


Benzoic ethanoic anhydride (8b)

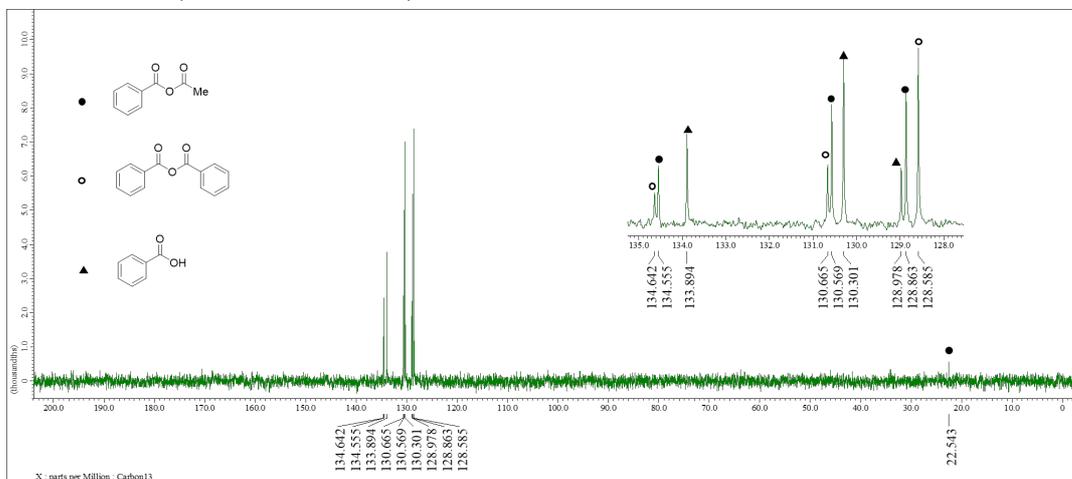
^1H NMR (400 MHz, CDCl_3)



$^{13}\text{C}\{^1\text{H}\}$ NMR (100 MHz, CDCl_3)

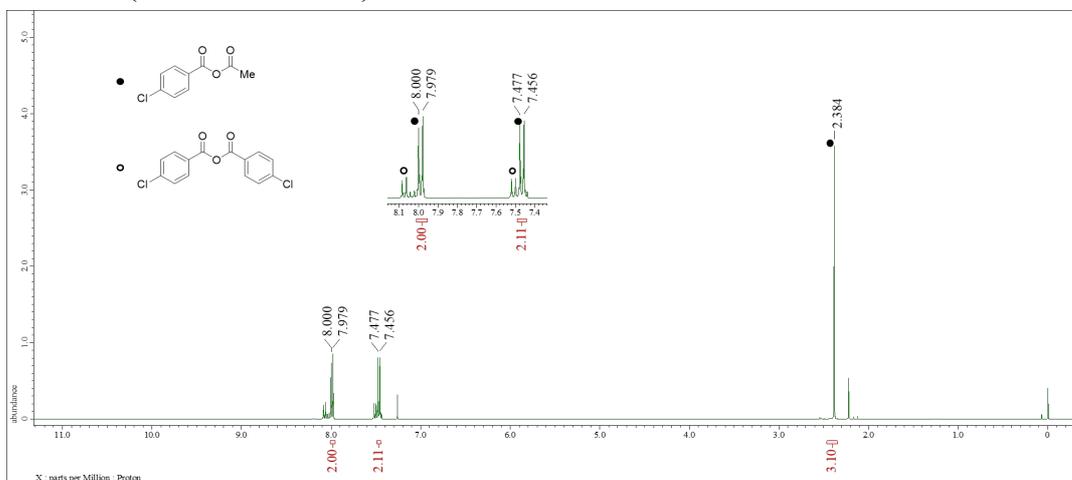


DEPT NMR (100 MHz, CDCl_3)

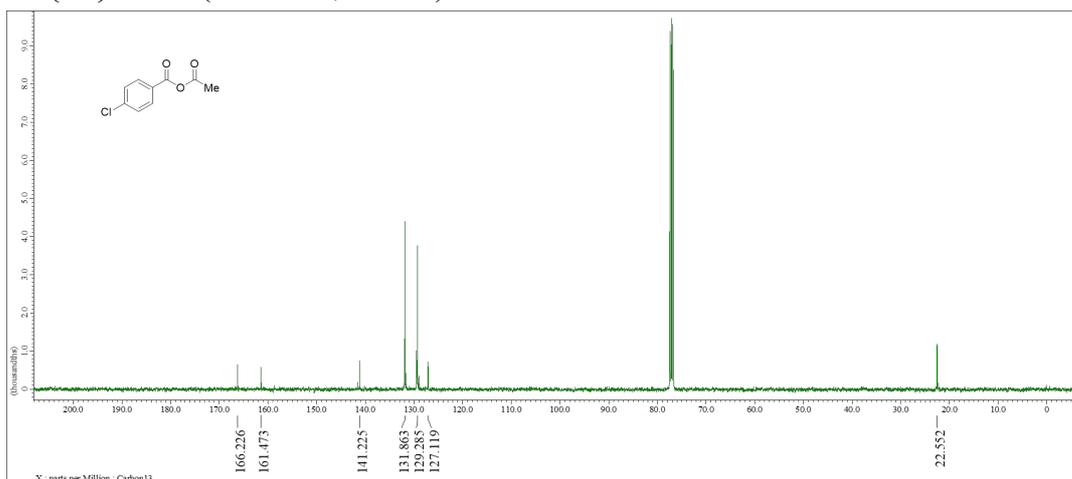


4-Chlorobenzoic ethanoic anhydride (8c)

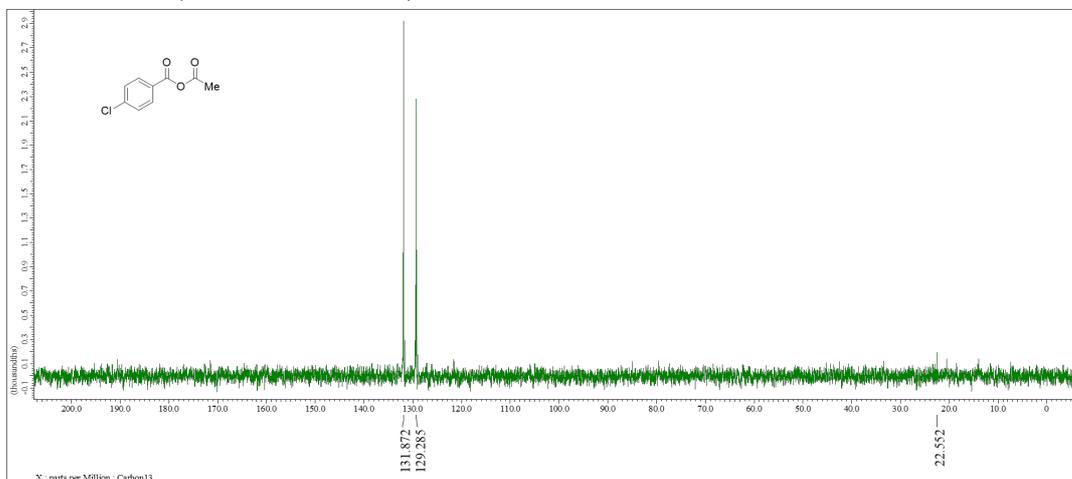
^1H NMR (400 MHz, CDCl_3)



$^{13}\text{C}\{^1\text{H}\}$ NMR (100 MHz, CDCl_3)

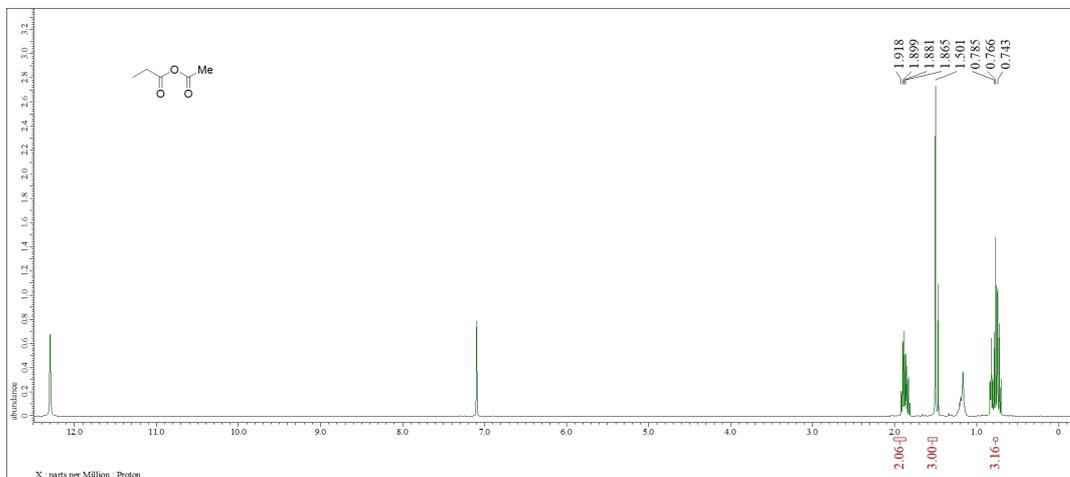


DEPT NMR (100 MHz, CDCl_3)

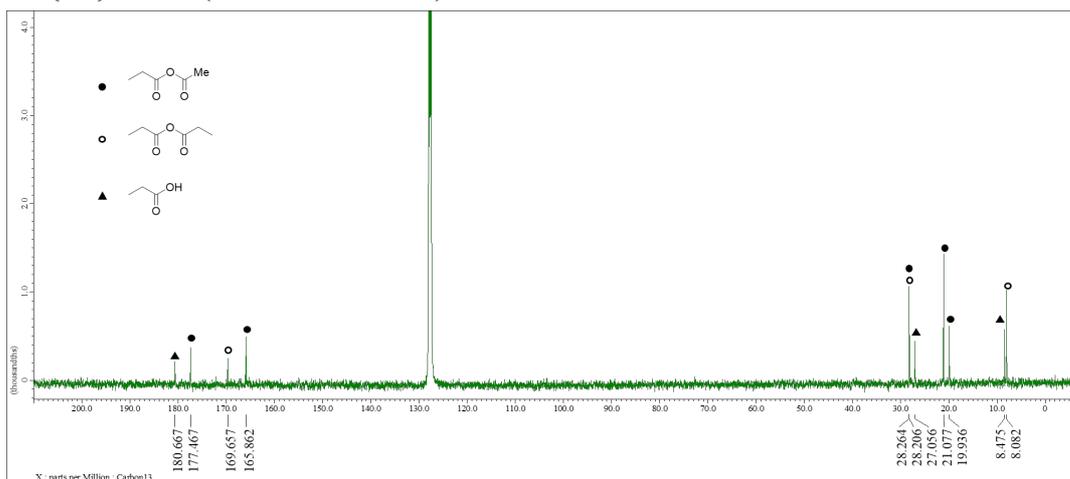


Ethanoic propionic anhydride (8d)

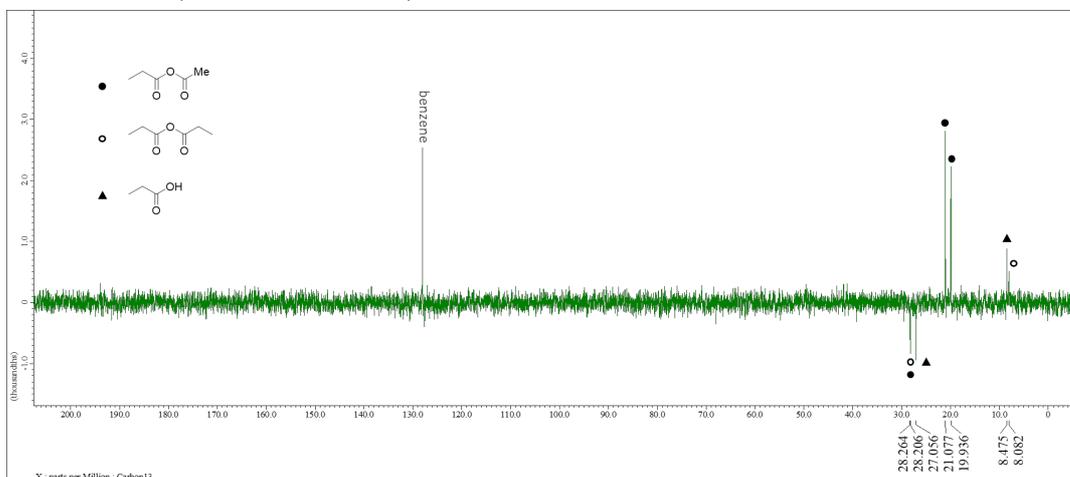
^1H NMR (400 MHz, CDCl_3)



$^{13}\text{C}\{^1\text{H}\}$ NMR (100 MHz, CDCl_3)

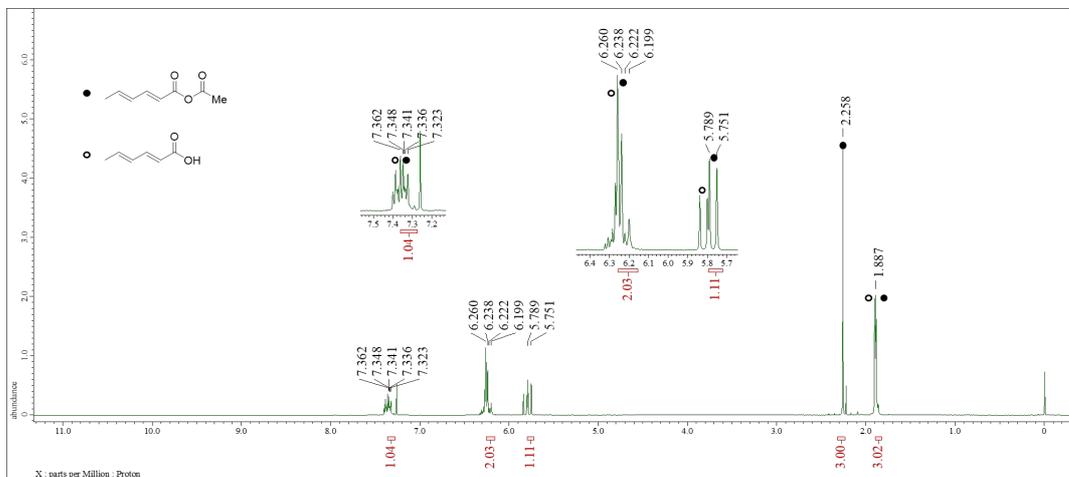


DEPT NMR (100 MHz, CDCl_3)

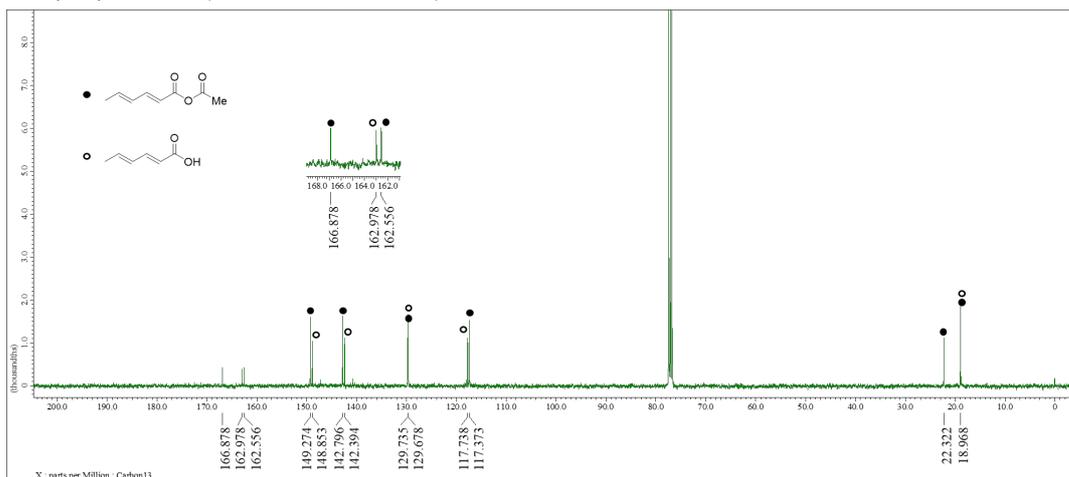


Ethanoic hexa-2,4-dienoic anhydride (8e)

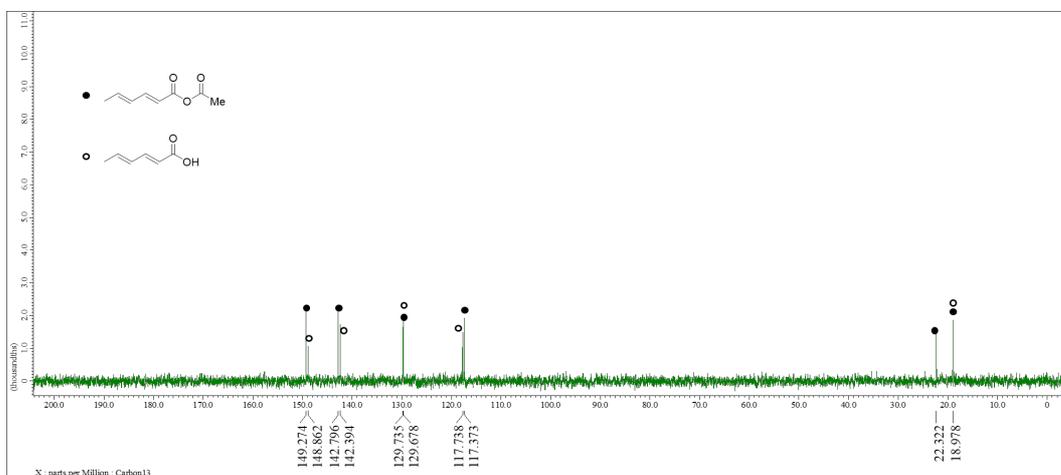
^1H NMR (400 MHz, CDCl_3)



$^{13}\text{C}\{^1\text{H}\}$ NMR (100 MHz, CDCl_3)

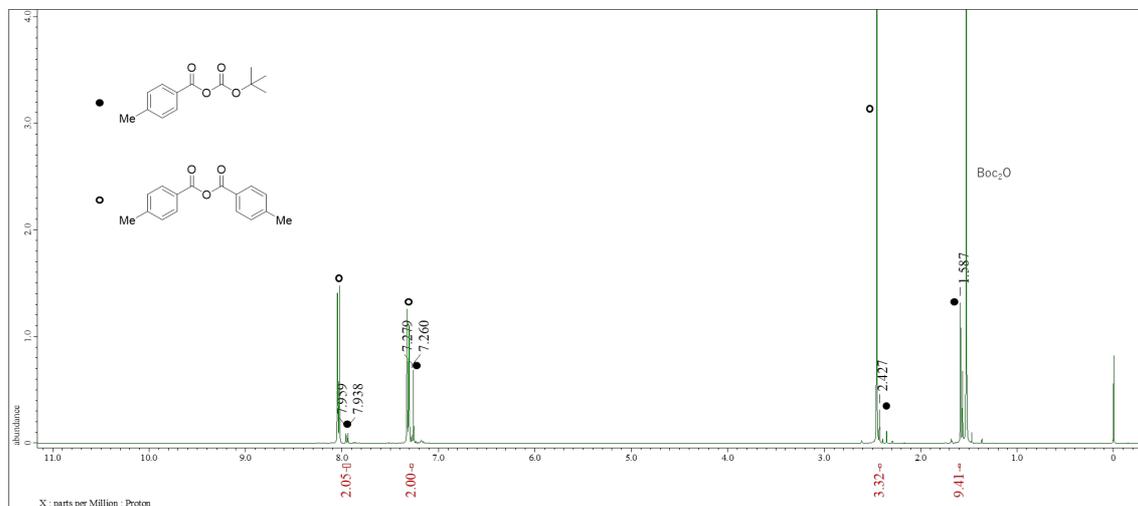


DEPT NMR (100 MHz, CDCl_3)



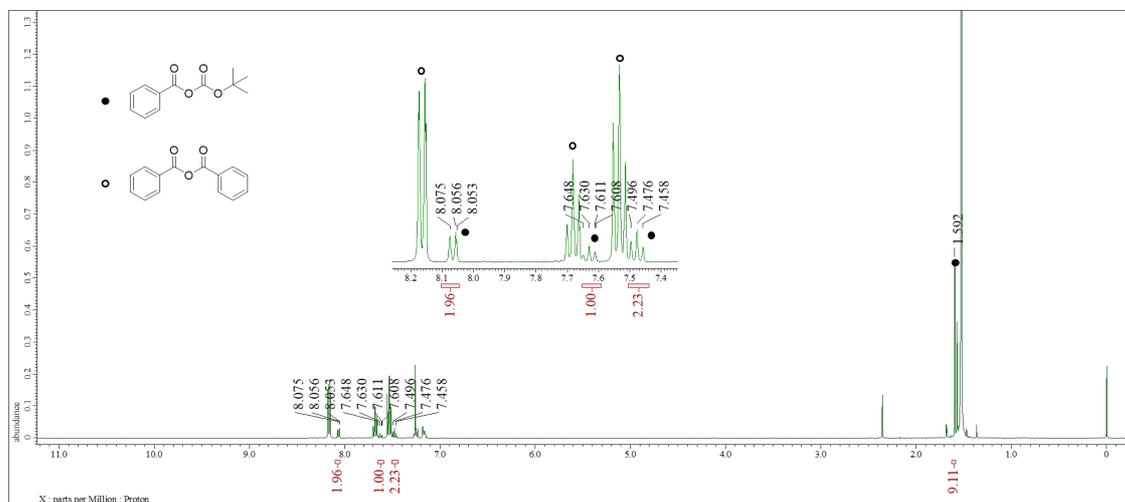
(1,1-Dimethylethoxy)carbonic 4-methylbenzoic anhydride (8f)

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



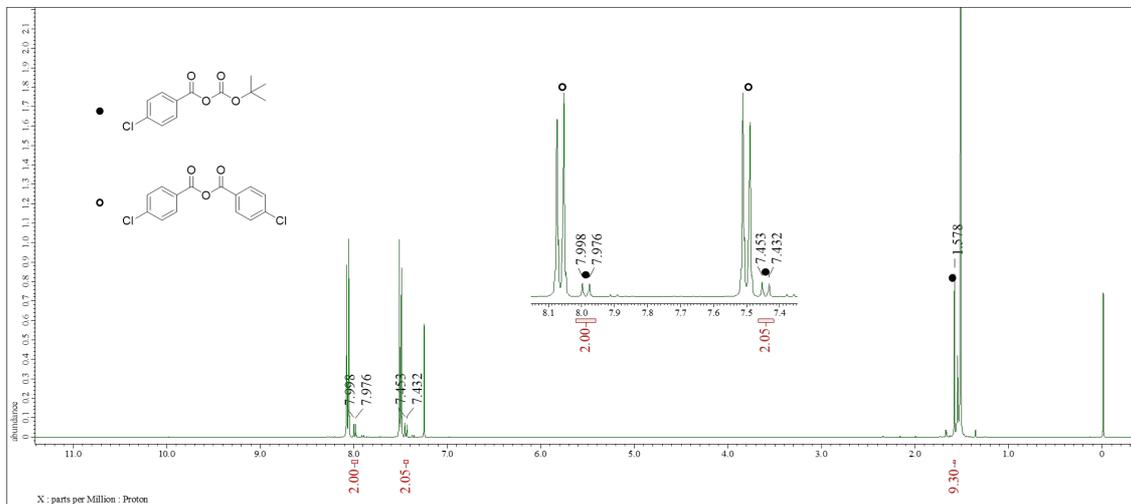
(1,1-Dimethylethoxy)carbonic benzoic anhydride (8g)

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



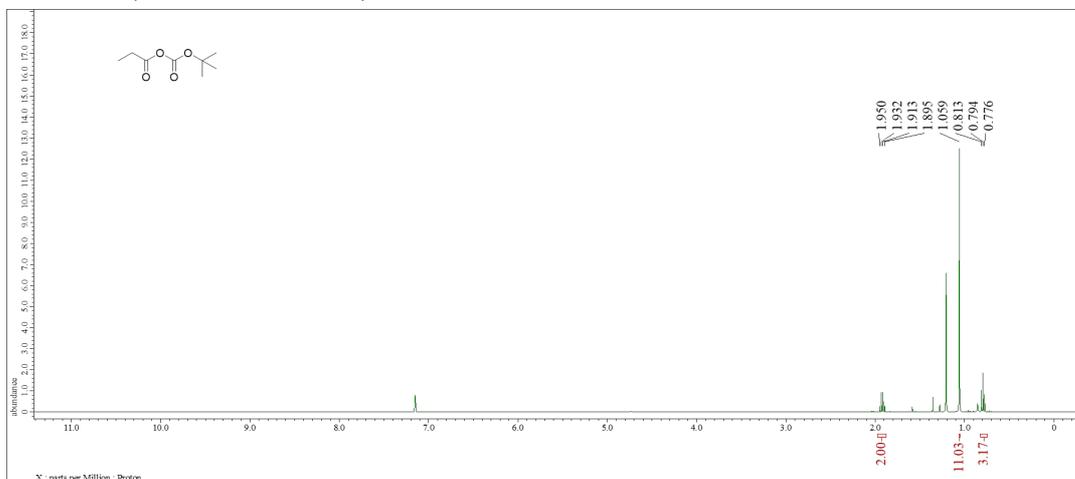
4-Chlorobenzoic (1,1-dimethylethoxy)carbonic anhydride (8h)

¹H NMR (400 MHz, CDCl₃)

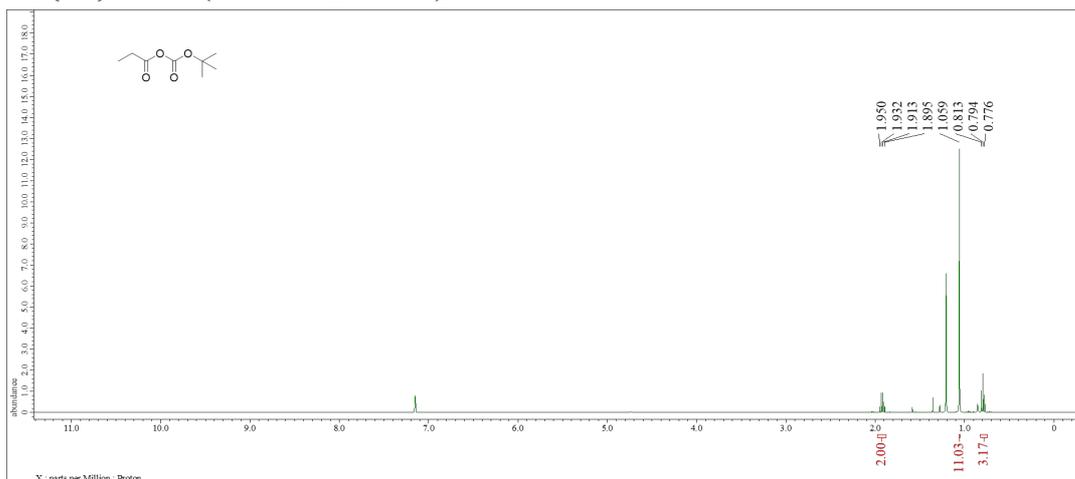


(1,1-Dimethylethoxy)carbonic propionic anhydride (8i)

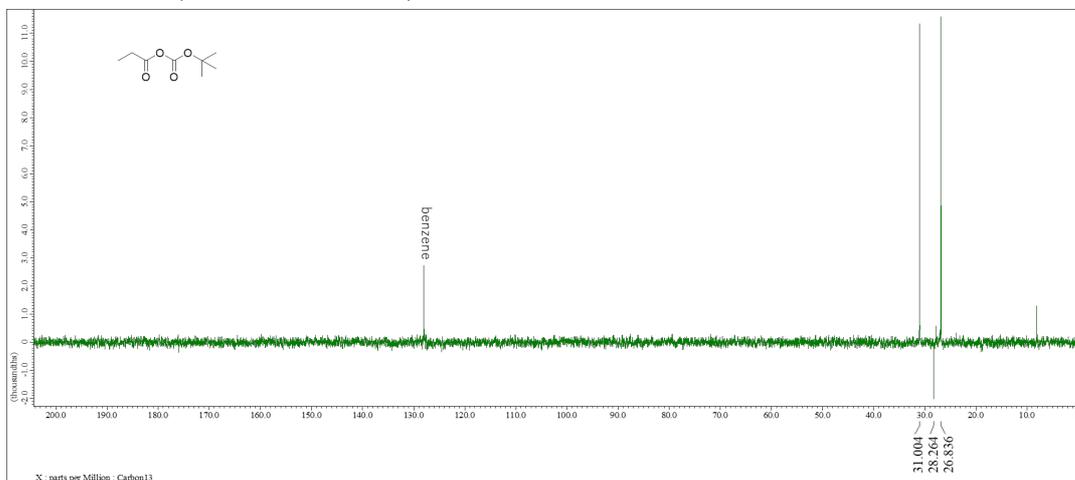
^1H NMR (400 MHz, CDCl_3)



$^{13}\text{C}\{^1\text{H}\}$ NMR (100 MHz, CDCl_3)

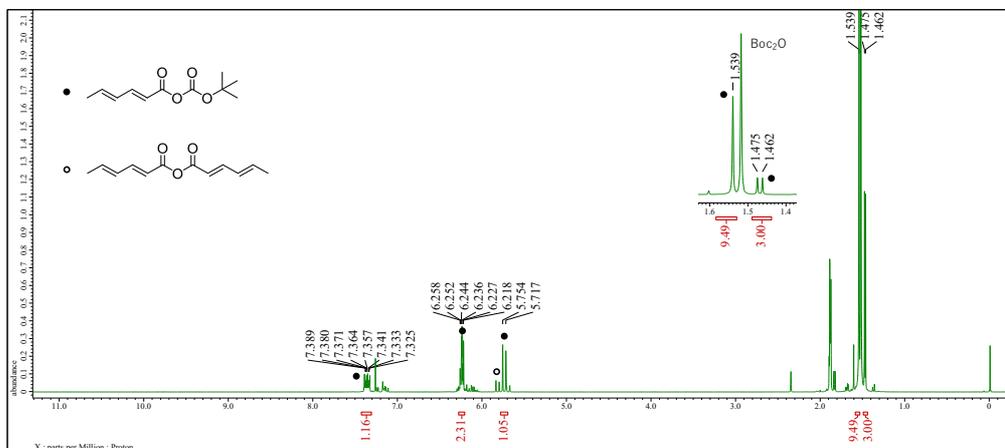


DEPT NMR (100 MHz, CDCl_3)

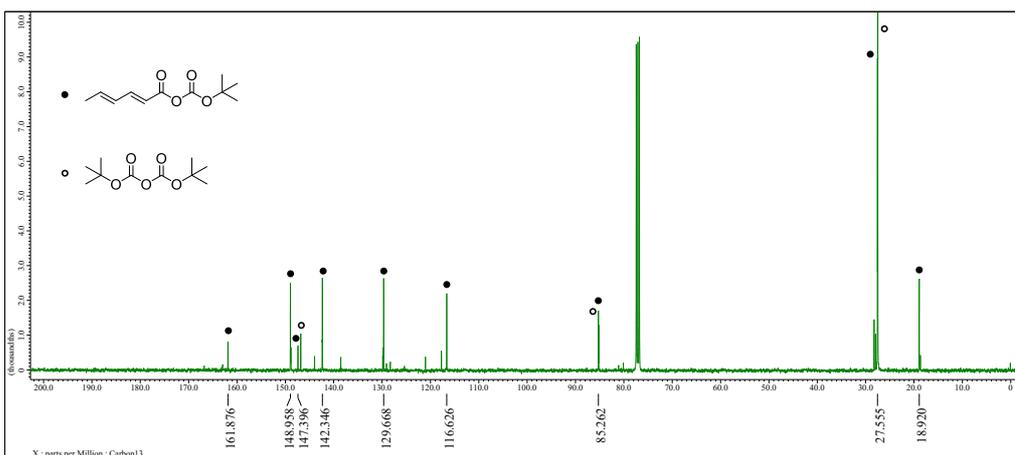


Hexa-2,4-dienoic (1,1-dimethylethoxy)carbonic anhydride (8j)

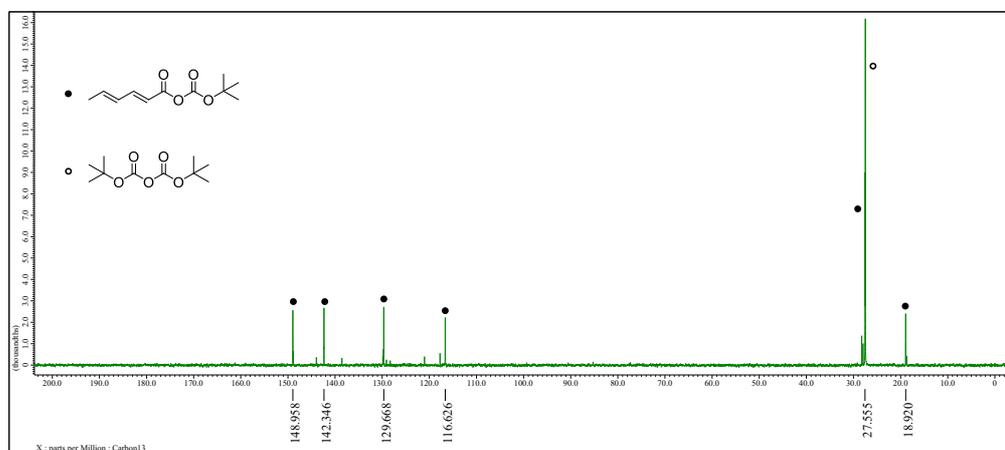
^1H NMR (400 MHz, CDCl_3)



$^{13}\text{C}\{^1\text{H}\}$ NMR (100 MHz, CDCl_3)



DEPT NMR (100 MHz, CDCl_3)



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