Electronic Supplementary Material (ESI) for Organic & Biomolecular Chemistry.

Supporting Information

Transition metal-free oxidative esterification of benzylic alcohols in aqueous medium

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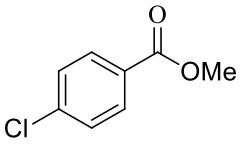
Experimental Section:

General: All commercially available chemicals and reagents were used without any further purification unless otherwise indicated. ^{1}H and ^{13}C NMR spectra were recorded at 200 and 50 MHz, respectively. The spectra were recorded in $CDCl_3$ as solvent. Multiplicity was indicated as follows: s (singlet); d (doublet); t (triplet); m (multiplet); dd (doublet of doublets), etc. and coupling constants (J) were given in Hz. Chemical shifts are reported in ppm relative to TMS as an internal standard. The peaks around delta values of ^{1}H NMR (7.26), and ^{13}C NMR (77.0) are correspond to deuterated solvent $CDCl_3$. Progress of the reactions was monitored by thin layer chromatography (TLC). All products were purified through column chromatography using silica gel 100-200 mesh size using ethyl acetate /hexane as eluent. All starting materials are commercially available and used without any further purification.

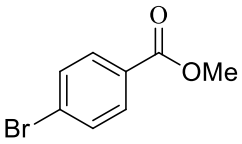
A typical experimental procedure for the synthesis methyl benzoate (3a): In a 25 mL glass vial, 108 mg (1.0 mmol) of Benzyl alcohol (**1a)**, 2 mL of methanol (**2a**) and 0.2 mmol of HBr (46% aqueous solution) were taken and the vial closed with rubber septum with magnetic stir bar. Then the vial was heated to 60°C under stirring and initially 2.0 mmol of hydrogen peroxide (33% aqueous solution) was added through a syringe. The rest of the 4.0 mmol of hydrogen peroxide was added to the reaction mixture at an interval of 2 h. After complete addition of hydrogen peroxide the reaction was continued up to 16 h (the progress of the reaction was monitored by TLC). [In case of butanol, octanol and polyalcohols reaction temperature was 70-75°C]. After completion of the reaction, the mixture was neutralised by aqueous solution of $NaHCO_3$ (5%) and extracted with ethyl acetate (3x 10 mL) and dried with anhydrous sodium sulphate. Removal of the solvent under reduced pressure, the crude product left out was purified by column chromatography on silica gel (100-200 mesh) using ethyl acetate/ hexane and **3a** was obtained in (0.117 g) 86% yield. The spectroscopic data was in good agreement with the literature.[1] (^{1}H NMR (200 MHz, $CDCl_3$): δ 8.02 – 7.97 (m, 2H), 7.47 (d, *J* = 7.4 Hz, 1H), 7.39 - 7.32 (m, 2H), 3.83 (s, 3H); ^{13}C NMR (50 MHz, $CDCl_3$): 166.7, 132.6, 130.4, 129.4, 128.8, 128.1, 51.7).

Methyl 4-chlorobenzoate (3b)[1] :



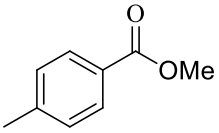
Yield (0.145g, 85%); ^{1}H NMR (200 MHz, $CDCl_3$): δ 7.94 (d, *J* = 8.6 Hz, 2H), 7.37 (d, *J* = 8.6 Hz, 2H), 3.87 (s, 3H); ^{13}C NMR (50 MHz, , $CDCl_3$): 166.1, 139.3,130.9,128.6, 52.1.

Methyl 4-bromobenzoate (3c)[1]:



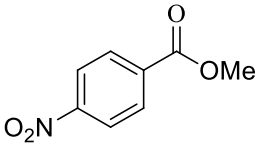
Yield (0.198g, 92%); ^{1}H NMR (200 MHz, $CDCl_3$): δ 7.87(d, *J* = 8.6 Hz, 2H), 7.55(d, *J* = 8.6 Hz, 2H), 3.87 (s, 3H) ^{13}C NMR (50 MHz, $CDCl_3$): 165.7, 131.9, 131.1, 130.6, 128.5, 127.5, 51.2 .

Methyl 4-methylbenzoate (3d)[1]:



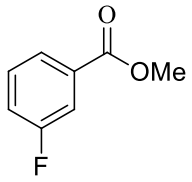
Yield (0.106g, 71%); ^{1}H NMR (200 MHz, $CDCl_3$): δ 7.94 (d, *J* = 8.2 Hz, 2H), 7.24 (d, *J* = 8.0 Hz, 2H), 3.88(s, 3H), 2.39(s, 3H); ^{13}C NMR (50 MHz, $CDCl_3$): 167.1, 143.4, 129.5, 129.0, 127.3, 51.8, 21.5.

Methyl 4-nitrobenzoate (3e)[2]:



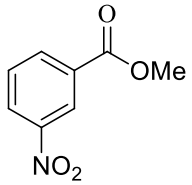
Yield (0.150g, 83%); ^{1}H NMR (200 MHz, $CDCl_3$): δ 8.262 – 8.13 (m, 4H), 3.94 (s, 3H); ^{13}C NMR (50 MHz, $CDCl_3$): 165.1, 150.5, 135.4, 130.6, 123.5, 52.8.

Methyl 3-fluorobenzoate (3f)[1]:



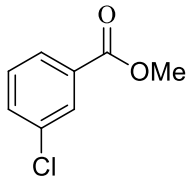
Yield (0.112g, 73%); ^{1}H NMR (200 MHz, $CDCl_3$): δ 7.83 (d, *J* = 7.6 Hz , 1H), 7.72 (d, *J* = 9.2 Hz, 1H), 7.45 – 7.34 (m, 1H), 7.28 – 7.19 (m, 1H), 3.91 (s, 3H); ^{13}C NMR (50 MHz, $CDCl_3$): 165.7, 164.9, 160.0, 132.3, 132.1, 130.0, 129.8, 125.2, 120.0, 119.61, 116.5, 52.2.

Methyl 3-nitrobenzoate (3g)[2]:



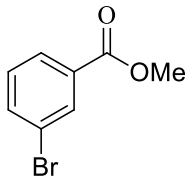
Yield (0.130g, 72%); ^{1}H NMR (200 MHz, $CDCl_3$): δ 8.76 (s, 1H), 8.37 – 8.28 (m, 2H), 7.65 – 7.50 (m, 2H), 3.93 (s, 3H); ^{13}C NMR (50 MHz, $CDCl_3$): 164.7, 148.1, 135.1, 131.7, 129.5, 127.2, 124.3, 52.6.

Methyl 3-chlorobenzoate(3h)[2]:



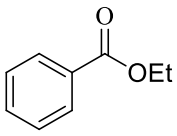
Yield (0.134g, 79%); ^{1}H NMR (200 MHz, $CDCl_3$): δ 7.97 (s, 1H), 7.90 (d, *J* = 7.6 Hz, 1H), 7.48 -7.45 (m, 1H), 7.38 – 7.30 (m, 1H), 3.89 (s, 3H); ^{13}C NMR (50 MHz, $CDCl_3$; 165.7, 134.3, 132.8, 131.8, 129.6, 127.6, 52.3.

Methyl 3-bromobenzoate (3i)[2]:



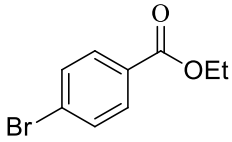
Yield (0.176g, 82%); ^{1}H NMR (200 MHz, $CDCl_3$): δ 8.15 (s, 1H), 7.98 (m, 1H), 7.68 (d, *J* = 8.0 Hz, 1H), 7.37 - 7.30 (m, 1H), 3.91 (s, 3H); ^{13}C NMR (50 MHz, $CDCl_3$): 165.5, 135.7, 132.4, 132.1, 129.8, 128.0, 122.3, 52.2.

Ethyl benzoate (3j)[3]:



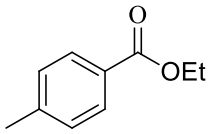
Yield (0.126g, 84%); ^{1}H NMR (200 MHz, $CDCl_3$): δ 7.94 – 7.90 (m, 2H), 7.45 (d, *J* = 7.6 Hz, 1H), 7.38 -7.29 (m, 2H), 4.30 – 4.19 (m, 2H), 1.30 – 1.22(m, 3H); ^{13}C NMR (50 MHz, $CDCl_3$): 166.4, 132.6, 130.4, 129.4, 128.8, 128.1, 60.8, 14.1.

Ethyl 4-bromobenzoate (3k)[4]:



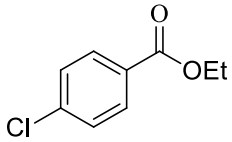
Yield (0.204g, 89%); ^{1}H NMR (200 MHz, $CDCl_3$): δ 7.86 (d, *J* = 8.6 Hz, 3H), 7.52 (d, *J* = 8.4 Hz, 2H), 4.37 - 4.27 (m, 2H), 1.37 – 1.30 (m, 3H); ^{13}C NMR (50 MHz, $CDCl_3$): 165.6, 131.4, 130.9, 129.2, 127.7, 61.0, 14.1.

Ethyl 4-methylbenzoate (3l)[5]:



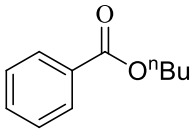
Yield (0.113g, 69%); ^{1}H NMR (200 MHz, $CDCl_3$): δ 7.96 (d, *J* = 8.0 Hz, 2H), 7.26 (d, *J* = 8.4 Hz, 2H), 4.42 – 4.32 (m, 2H), 2.41(s, 3H), 1.43 – 1.36 (m, 3H); ^{13}C NMR (50 MHz, $CDCl_3$): 167.2, 143.5, 129.1, 127.5, 60.2, 21.6, 14.1 .

Ethyl 4-chlorobenzoate (3m)[5]:



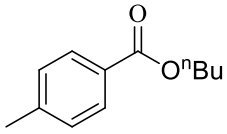
Yield (0.149g, 81%); ^{1}H NMR (200 MHz, $CDCl_3$): δ 8.00 (d, *J* = 8.4 Hz, 2H), 7. 42 (d, *J* = 8.4 Hz, 2H), 4.42 – 4.32 (m, 2H), 1.42 – 1.35 (m, 3H); ^{13}C NMR (50 MHz, $CDCl_3$): 165.7, 132.2, 130.9, 128.6, 61.2, 14.3.

Butyl benzoate (3n)[6]:



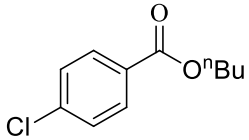
Yield (0.142g, 80%); ^{1}H NMR (200 MHz, $CDCl_3$): δ 8.06 (d, *J* = 7.2 Hz, 2H), 7.58 -7.51 (m, 1H), 7.42-7.39 (m, 2H), 4.36 (t, *J* = 6.6 Hz, 2H), 1.82 – 1.68 (m, 2H), 1.57 – 1.39 (m, 2H), 1.01 – 0.94 (m, 3H); ^{13}C NMR (50 MHz, $CDCl_3$): 165.8, 131.6,131.0,129.3, 127.8, 65.0, 30.6, 19.1, 13.7 .

Butyl 4-methylbenzoate (3o)[6]:



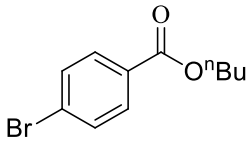
Yield (0.131g, 68%); ^{1}H NMR (200 MHz, $CDCl_3$): δ 7.95 (d, *J* = 8.0, 2H), 7.24 (d, *J* = 8.0 Hz, 2H), 4.33 (t, *J* = 6.4,2H), 2.40 (s, 3H), 1.78 – 1.63 (m, 2H), 1.52 – 1.41 (m, 2H), 1.01 – 0.94 (m, 3H); ^{13}C NMR (50 MHz, $CDCl_3$): 166.7, 143.4, 129.5, 129.0, 127.8, 64.6, 30.8, 21.6, 19.2, 13.7 .

Butyl 4-chlorobenzoate (3p)[6]:



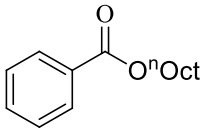
Yield (0.168g, 79%); ^{1}H NMR (200 MHz, $CDCl_3$): δ 7.97 – 7.93(m, 2H), 7.38 – 7.34 (m, 2H), 4.33 (t, *J* = 6.4 Hz, 2H), 1.79 – 1.66 (m, 2H), 1.54 – 1.36(m, 2H), 1.00 – 0.92 (m, 3H); ^{13}C NMR (50 MHz, $CDCl_3$): 165.5, 139.1, 130.8, 128.9, 128.5, 64.9, 30.7, 19.2, 13.6 .

Butyl 4-bromobenzoate (3q)[3]:



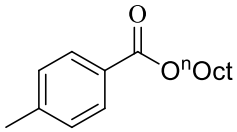
Yield (0. 207g, 85%) ; ^{1}H NMR (200 MHz, $CDCl_3$): δ 7.90 (d, *J* = 8.6 Hz, 2H),7.57 (d, *J* = 8.4 Hz, 2H), 4.33 (t, *J* = 6.4 Hz, 2H), 1.80 – 1.66 (m,2H), 1.54 – 1.36 (m, 2H), 0.99 – 0.92 (m, 3H); ^{13}C NMR (50 MHz, $CDCl_3$): 166.6, 132.7, 130.5, 129.5, 128.3, 64.8, 30.7,19.2, 13.7 .

Octyl benzoate (3r)[7]:



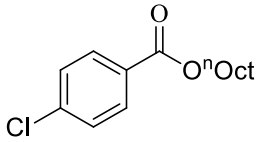
Yield (0.178 g, 76%) ; ^{1}H NMR (200 MHz, $CDCl_3$) δ 8.04 – 8.00 (m , 2H),7.53 – 7.49 (m, 1H), 7.44 – 7.37(m, 2H), 4.32 (t, *J* = 6.6 Hz, 2H), 1.79 – 1.65 (m, 2H), 1.42 – 1.25 (m, 2H), 0.87–0.81 (m, 3H); ^{13}C NMR (50 MHz, , $CDCl_3$): 166.6, 132.7, 129.5, 128.3, 64.3, 31.7, 29.2, 28.9, 26.0, 22.6, 14.0 .

Octyl 4-methylbenzoate (3s)[8]:



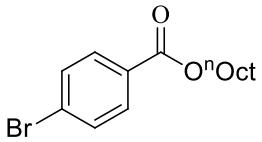
Yield (0.161g, 65%); ; ^{1}H NMR (200 MHz, $CDCl_3$): δ 7.87 (d, *J* = 8.0Hz , 2H), 7.19 –7.12 (m, , 2H), 4.24 (t, *J* = 6.6 Hz, 2H), 2.32 (s , 3H) 1.79 – 1.65 (m, 2H), 1.42 –1.25 (m, 10H), 0.87 – 0.81 (m, 3H); ^{13}C NMR (50 MHz, $CDCl_3$): 166.7, 143.3, 129.5, 128.9,127.8, 64.4, 31.7, 29.1, 25.9, 25.0, 22.5, 14.0 .

Octyl 4-chlorobenzoate (3t)[3]:



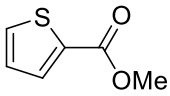
Yield (0.207g,77%) ; ^{1}H NMR (200 MHz, $CDCl_3$): δ 7.97 (d, *J* = 8.4 Hz , 2H) 7.39 (d, *J* = 8.6 Hz, 2H), 4.31 (t, *J* = 6.6 Hz, 2H), 1.80 – 1.67 (m, 2H), 1.43-1.26 (m, 10H), 0.88 – 0.82 (m, 3H); ^{13}C NMR (50 MHz, $CDCl_3$): 165.7, 139.2, 130.9, 128.9, 128.6, 65.3, 31.7, 29.2, 28.6, 26.0, 22.6, 14.0 .

Octyl 4-bromobenzoate (3u):



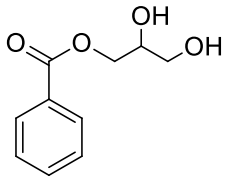
Yield (0.250g,80%) ; ^{1}H NMR (200 MHz, $CDCl_3$): δ 7.88 (d, *J* = 8.4 Hz , 2H), 7.54 (d, *J* = 8.4 Hz, 2H), 4.30 (t, *J* = 6.6 Hz, 2H), 1.80 – 1.66 (m, 2H), 1.44-1.27 (m, 10H), 0.88 – 0.83 (m, 3H); ^{13}C NMR (50 MHz, , $CDCl_3$): 165.7, 131.6, 131.0, 127.8, 64.2, 31.3, 29.1, 26.0, 25.0, 22.6, 14.0 . LRMS- calculated for $C_{15}H_{22}BrO_2$ =313.0803; found 313.0847

Methyl thiophene-2-carboxylate (3v)[1]:



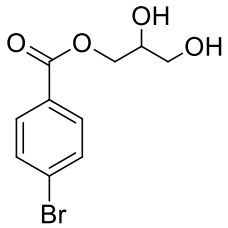
Yield (0.092g, 65%) ; ^{1}H NMR (200 MHz, $CDCl_3$): δ 7.82 – 7.81 (m , 1H), 7.57 – 7.55 (m,1H) , 7.13 – 7.09 (m, 1H), 3.88 (s, 3H); ^{13}C NMR (50 MHz, , $CDCl_3$): 162.7, 133.4, 132.3, 127.7, 52.1

2,3-Dihydroxypropyl benzoate (5a)[9]:



Yield (0.147g, 75%) ; ^{1}H NMR (200 MHz, $CDCl_3$): δ 8.0 (d, *J* = 7.4 Hz, 2H), 7.50 – 7.40 (m, 1H), 7.36 (d, *J* = 6.8 Hz, 2H), 4.34 (d, *J* = 6.8 Hz, 2H), 4.03 - 3.64 (m, 4H); ^{13}C NMR (50 MHz, , $CDCl_3$): 167.0, 133.3, 129.7, 128.4, 70.3, 65.6, 63.4 .

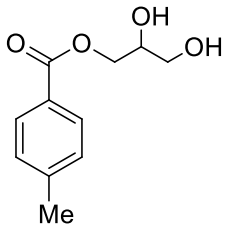
2,3-Dihydroxypropyl 4-bromobenzoate (5b):



Yield (0.231g, 84%) ; ^{1}H NMR (200 MHz, $CDCl_3$): δ 7.86 (d, *J* = 8.4 Hz, 2H), 7.55 (d, *J* = 8.6 Hz, 2H), 4,36 (d, *J* = 5.6 Hz, 2H), 4.06 – 4.01 (m, 1H), 3.78 – 3.59 (m, 2H), 3.21 (s, 2H); ^{13}C NMR (50 MHz, , $CDCl_3$): 166.2, 131.8, 131.2, 128.5, 70.2, 65.9, 63.4 .

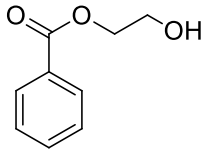
. LRMS- calculated for $C_{10}H_{12}BrO_4$ =274.9919; found 274.9946

2,3-Dihydroxypropyl 4-methylbenzoate (5c)[10] :



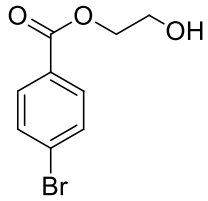
Yield (0.151g,72%) ; ^{1}H NMR (200 MHz, $CDCl_3$): δ 7.89 (d, *J* = 7.2 Hz, 2H), 7.17 (d, *J* = 7.4 Hz, 2H), 4.32 (d, *J* = 5.0 Hz, 2H), 4.02 (s, 2H), 3.89 (d, *J* = 4,4 Hz, 1H), 3.71 – 3.65 (m, 2H), 2.33 (s, 2H); ^{13}C NMR (50 MHz, , $CDCl_3$): 167.0, 143.9, 129.7, 129.1, 126.9, 70.3, 65.5, 63.5, 21.6 .

2-Hydroxyethyl benzoate (5d)[11]:



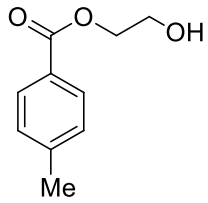
Yield 0.121g, 73%) ; ^{1}H NMR (200 MHz, $CDCl_3$): δ 7.93 (d, *J* = 7.6 Hz, 2H), 7.44 – 7.36 (m, 1H), 7.30 – 7.23 (m, 2H), 4.31 – 4.27 (m, 2H), 3.82 – 3.77 (m, 2H), 3.55 (s, 1H); ^{13}C NMR (50 MHz, , $CDCl_3$): 167.0, 133.1, 129.6, 128.3, 66.5, 60.8.

2-Hydroxyethyl 4-bromobenzoate (5e)[12]:



Yield (0.203g, 83%) ; ^{1}H NMR (200 MHz, $CDCl_3$): δ 7.91 (d, *J* = 8.4 Hz, 2H), 7.58 (d, *J* = 8.4 Hz, 2H), 4.46 – 4.41 (m, 2H), 3.96 – 3.86 (m, 2H), 2.26 (s, 1H); ^{13}C NMR (50 MHz, , $CDCl_3$): 166.3, 131.7, 131.2, 128.7, 128.3, 66.8, 61.2 .

2-Hydroxyethyl 4-methylbenzoate (5f)[13]:



Yield (0.128g, 71%) ; ^{1}H NMR (200 MHz, $CDCl_3$): δ 7.89 (d, *J* = 8.0 Hz, 2H), 7.15 (d, *J* = 8.0 Hz, 2H), 4.36 – 4.32 (m, 2H), 3.87 – 3.83 (m, 2H), 3.46 (s, 1H), 2.31 (s, 3H); ^{13}C NMR (50 MHz, , $CDCl_3$): 168.8, 143.6, 129.5, 128.8, 126.9, 66.1, 60.7, 21.3 .

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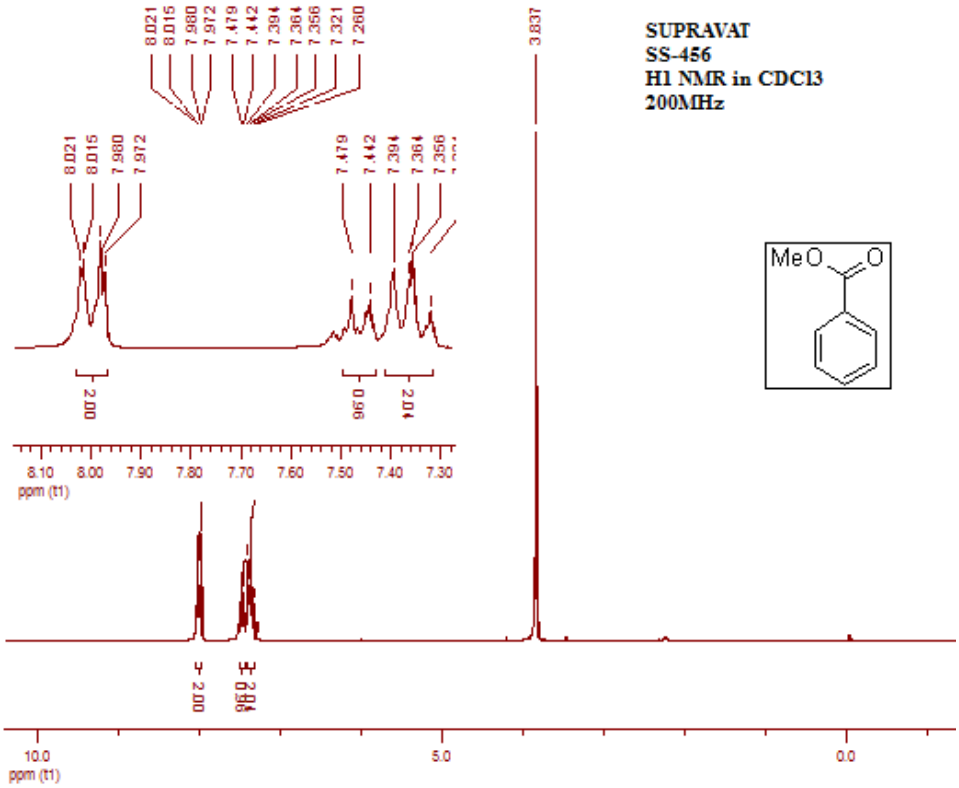
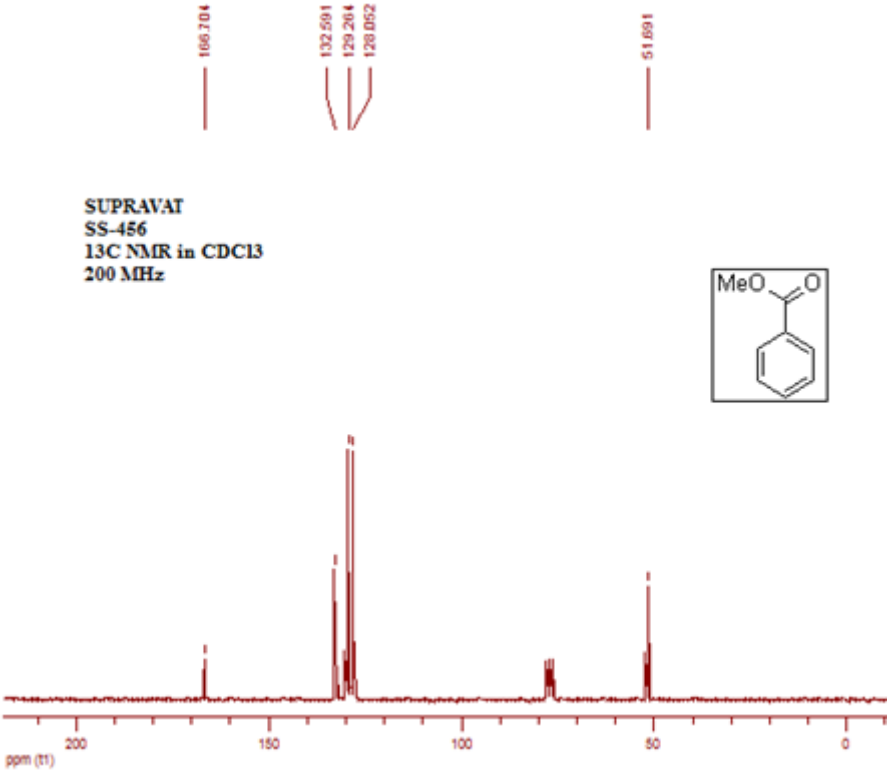
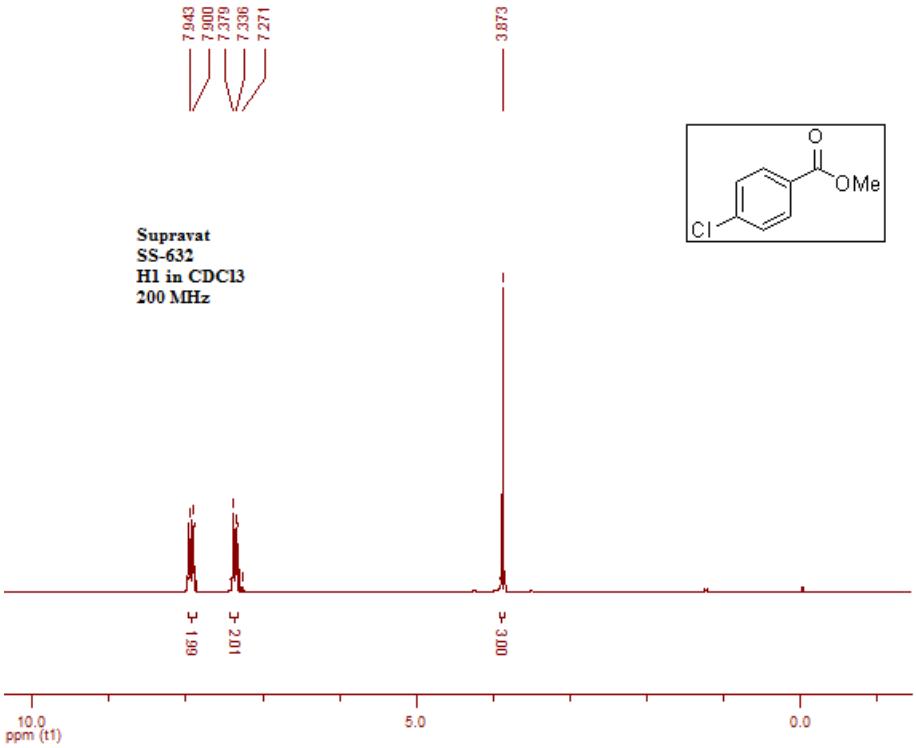
NMR spectra for all products



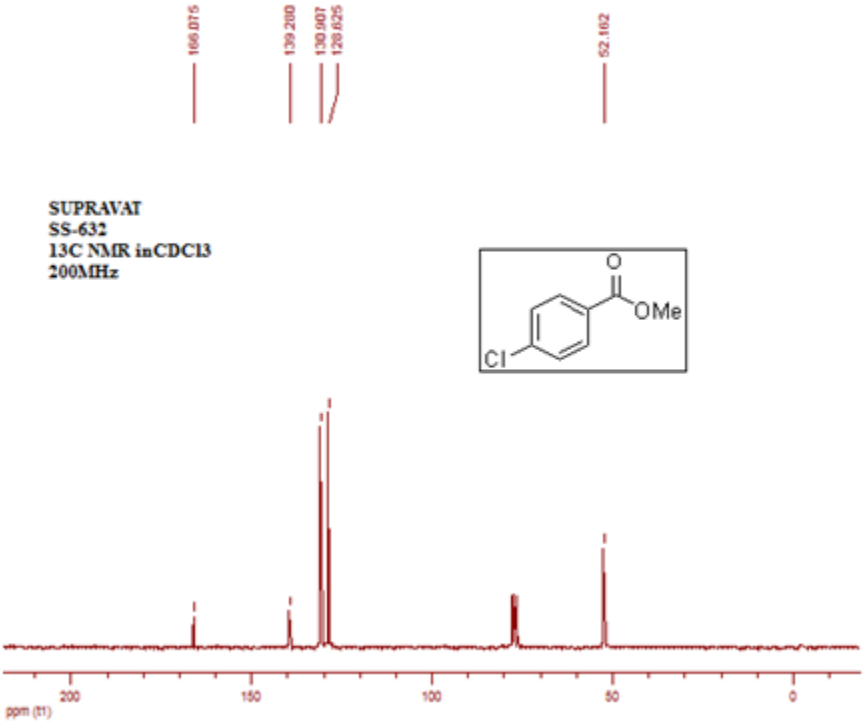
^{1}H NMR of **3a**



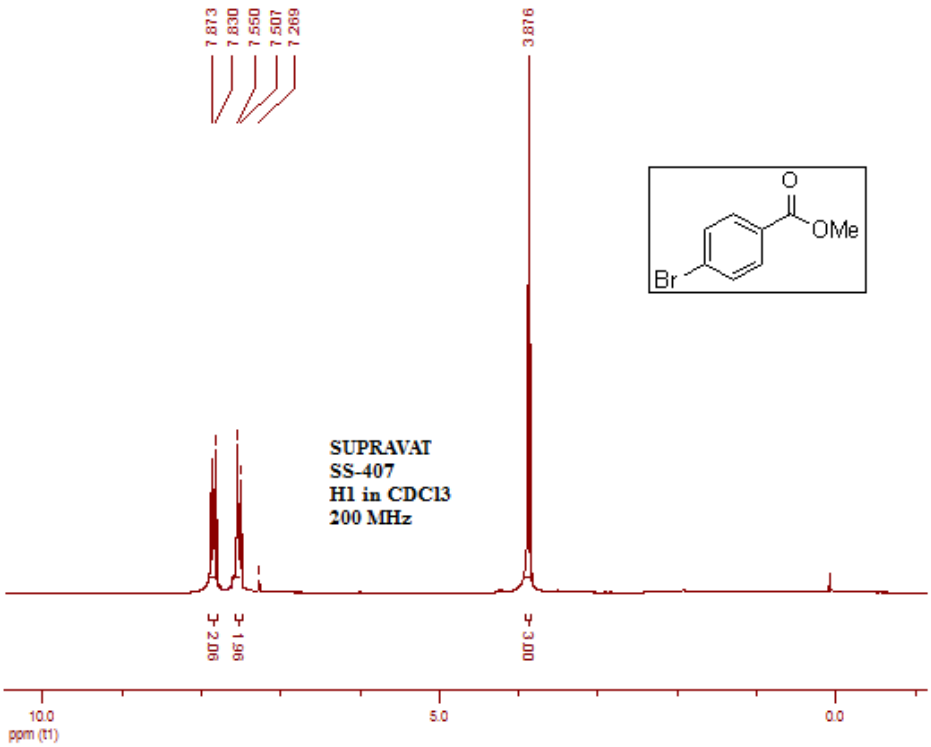
^{13}C NMR of 3a



^{1}H NMR of **3b**



^{13}C NMR of 3b



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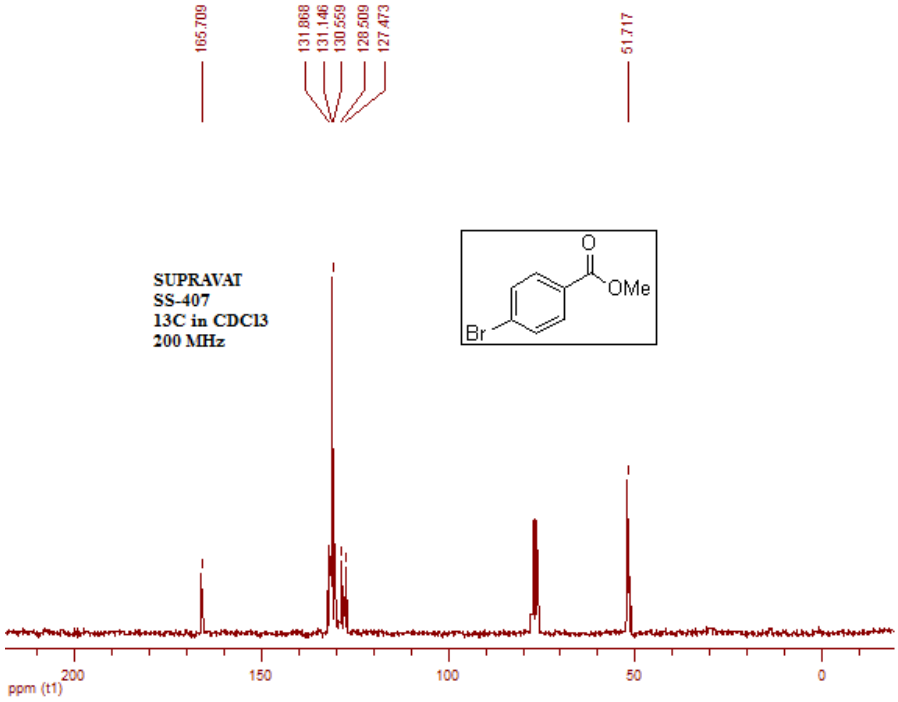
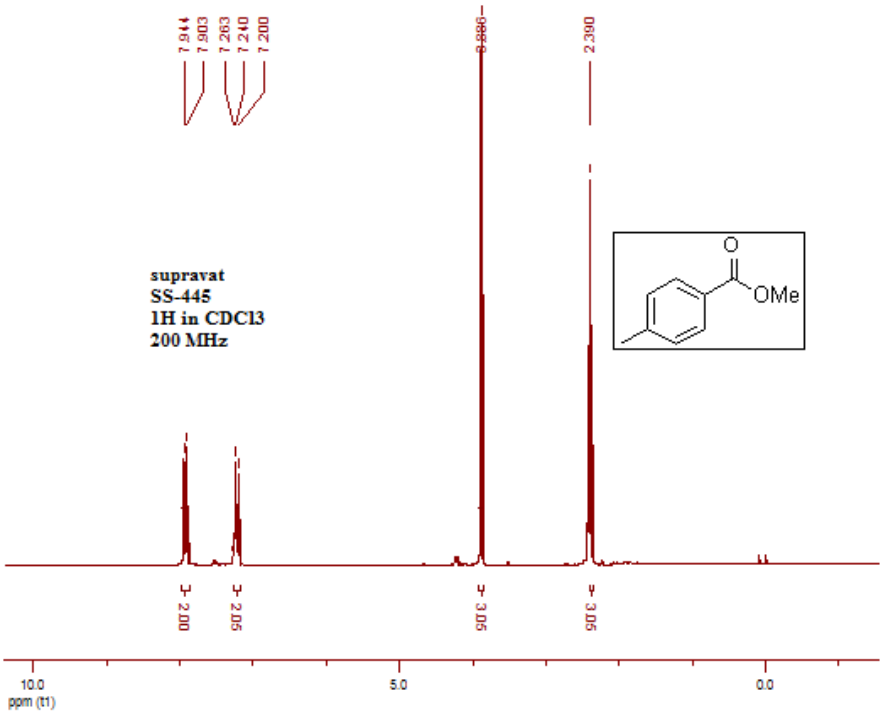


^{13}C NMR of **3c**



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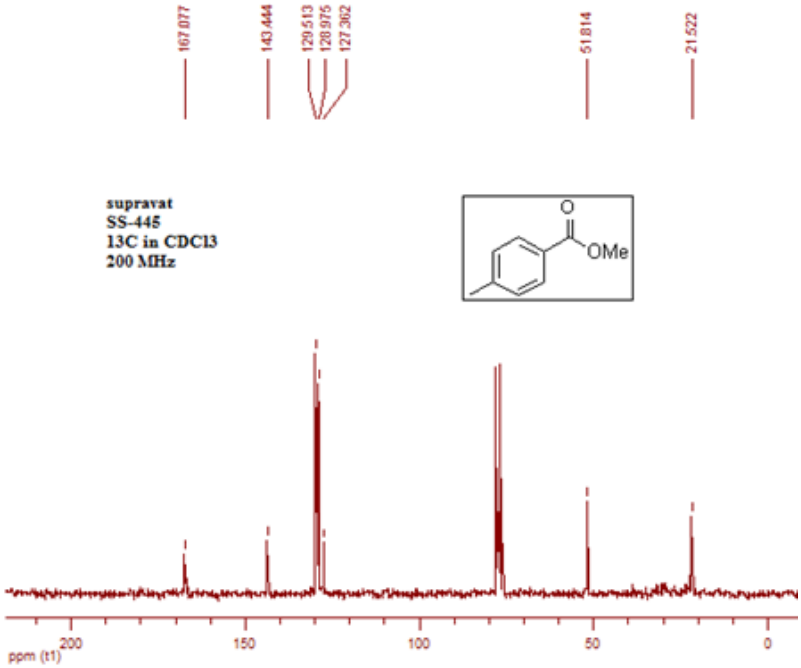
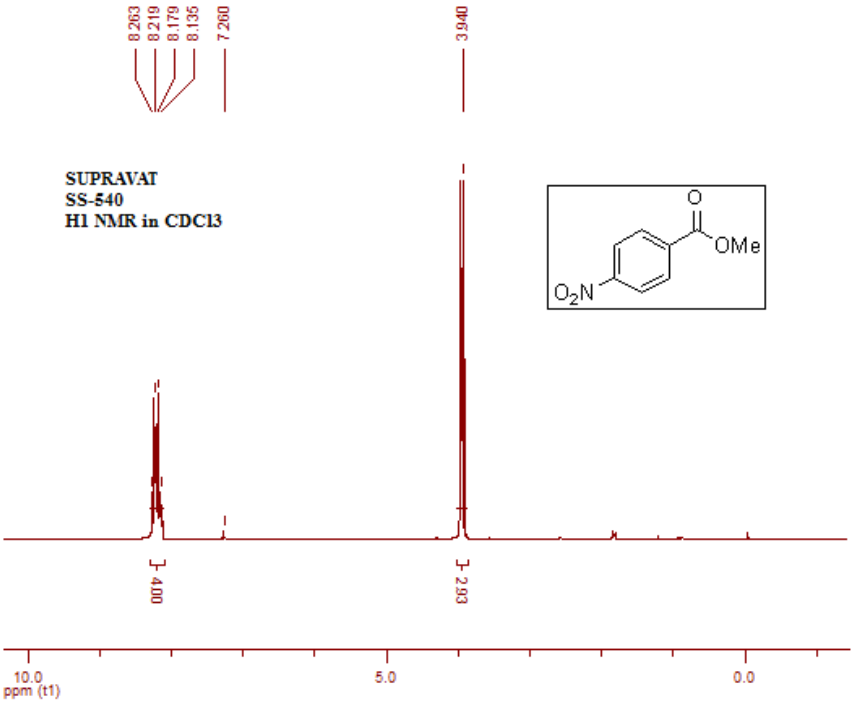
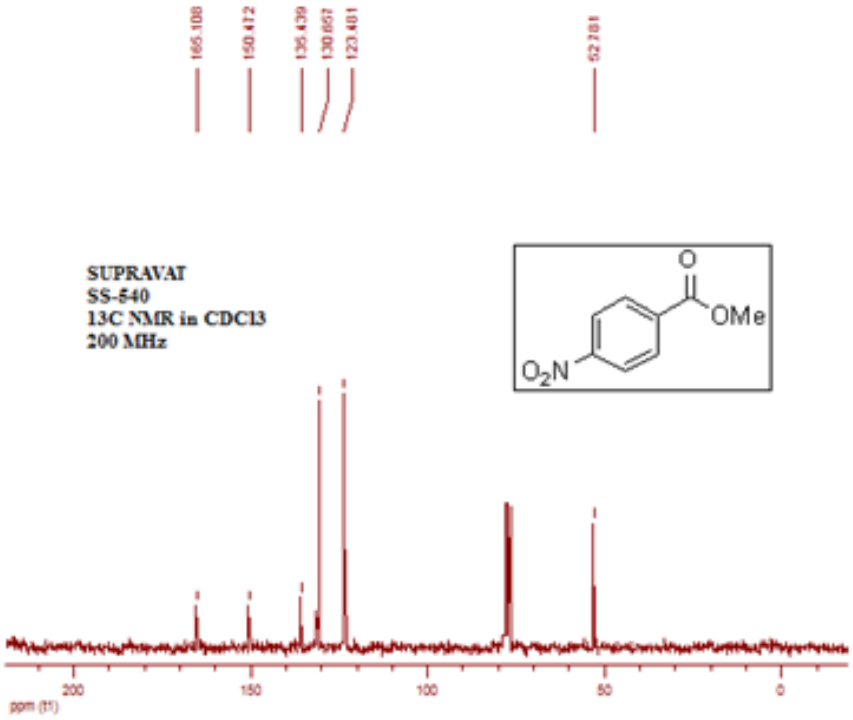


^{13}C NMR of 3d



^{1}H NMR of **3e**



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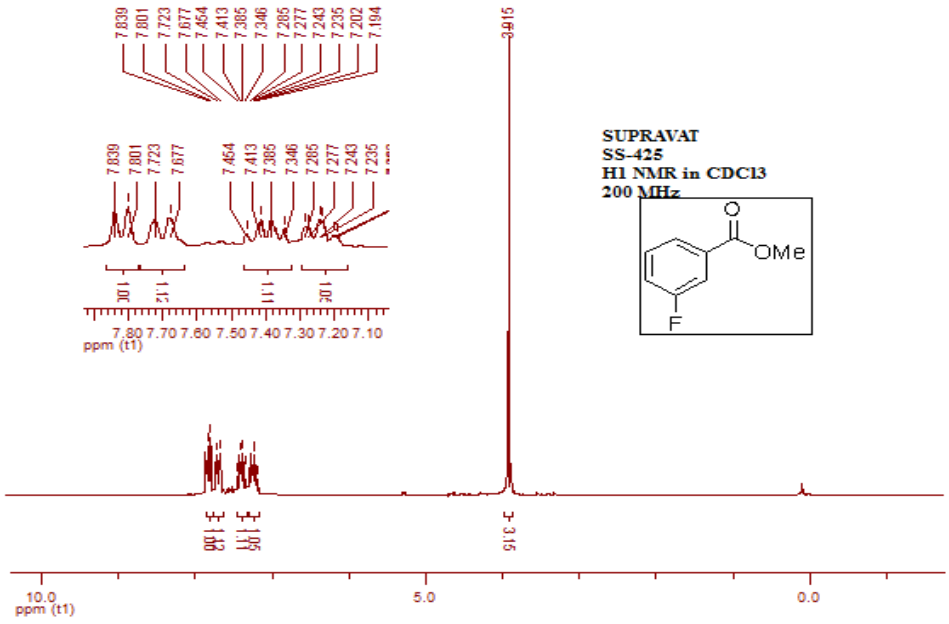
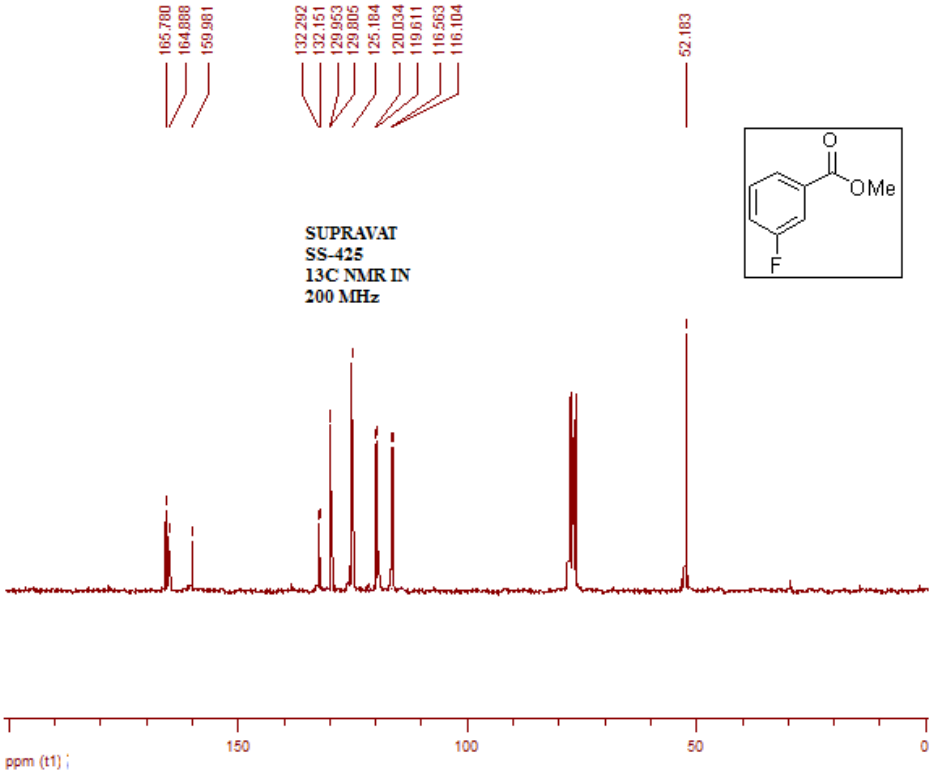
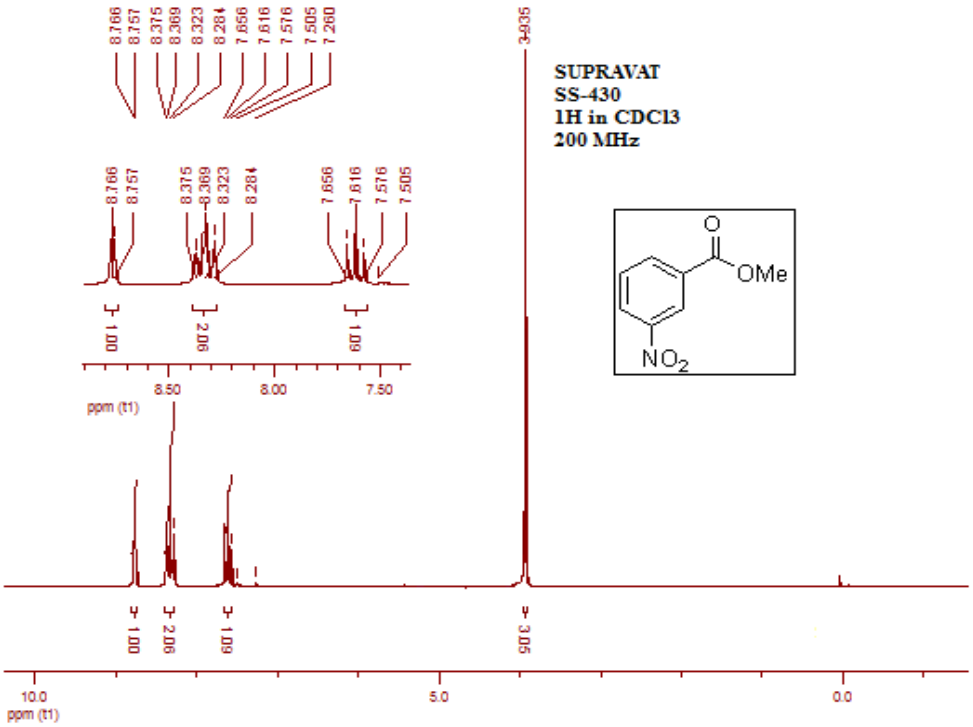


^{1}H NMR of **3f**



^{13}C NMR of 3f



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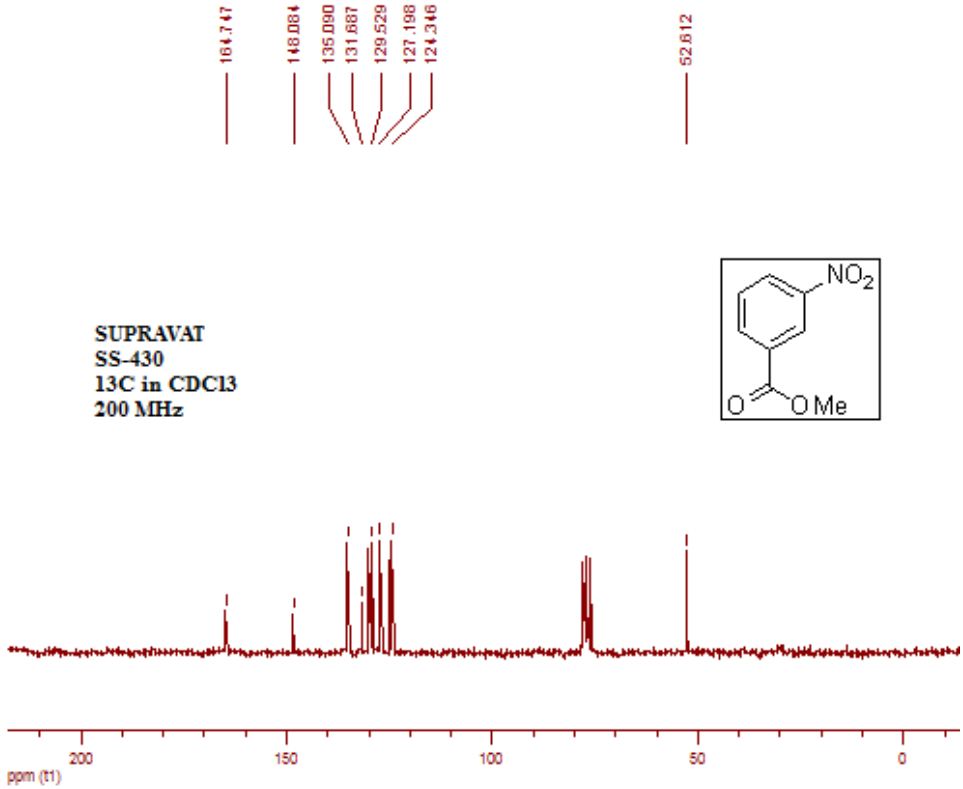
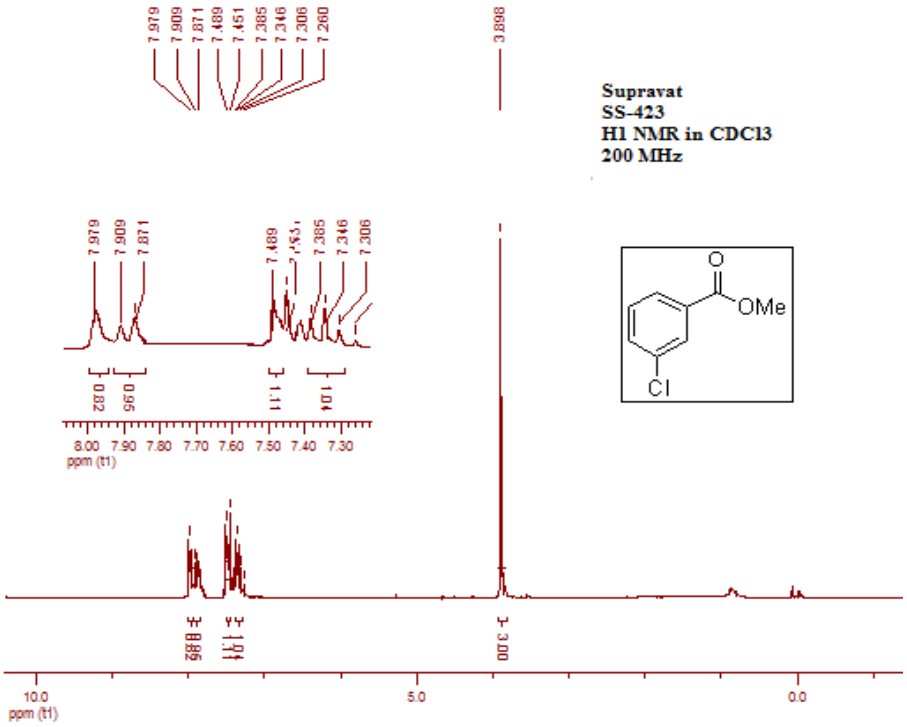


^{13}C NMR of 3g



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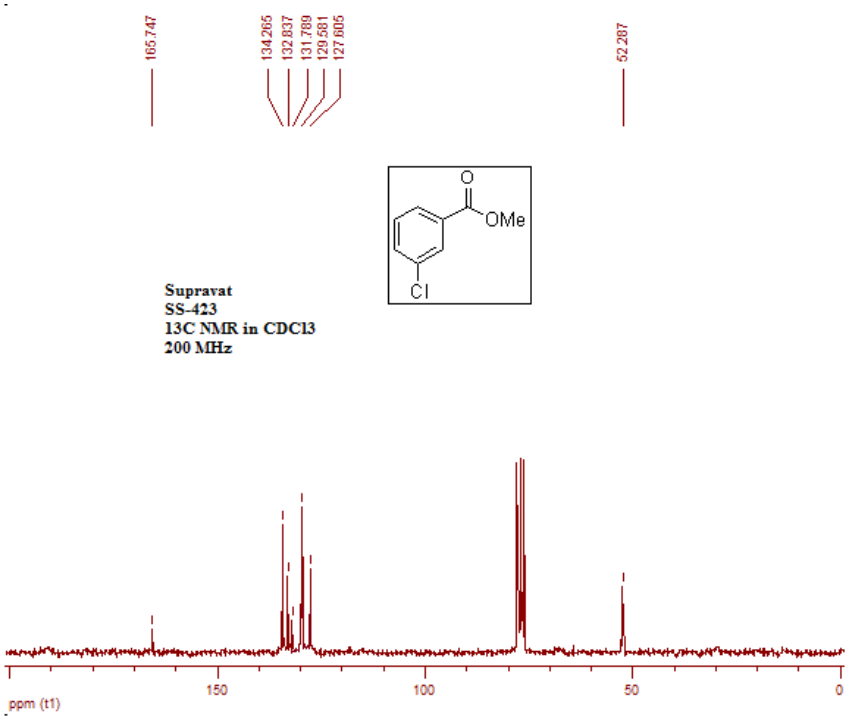
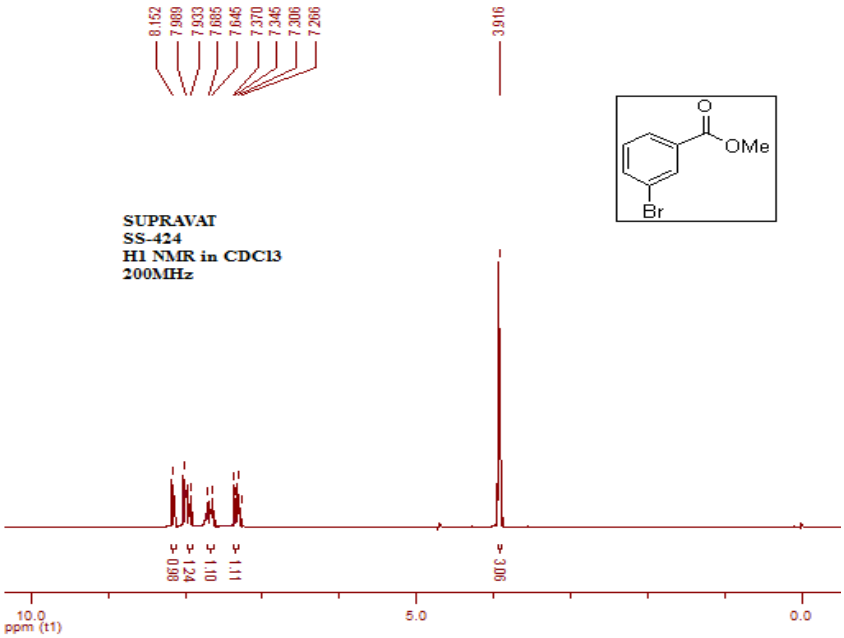
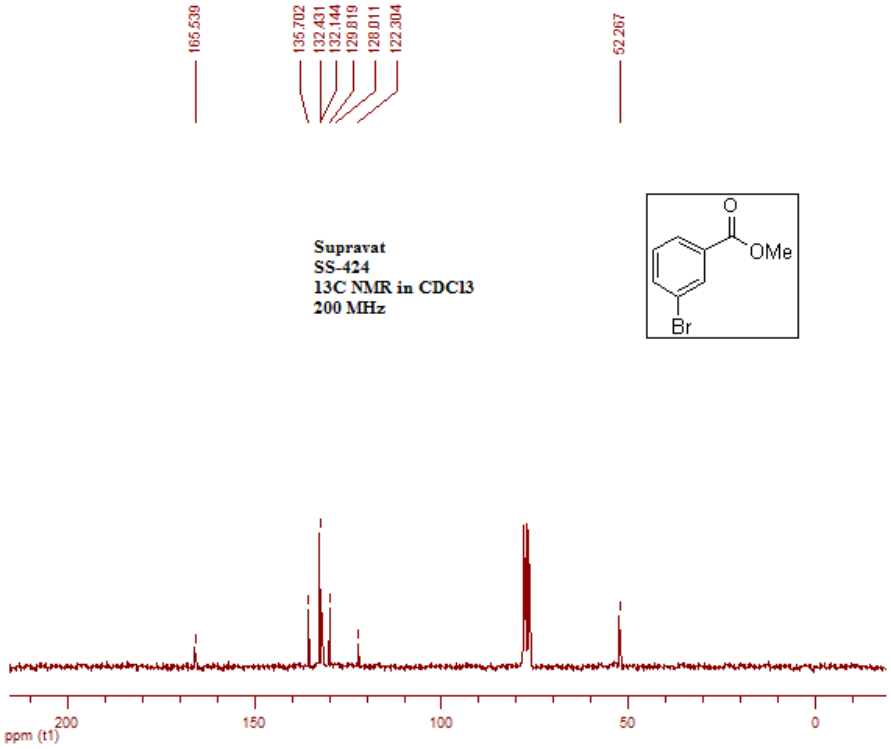


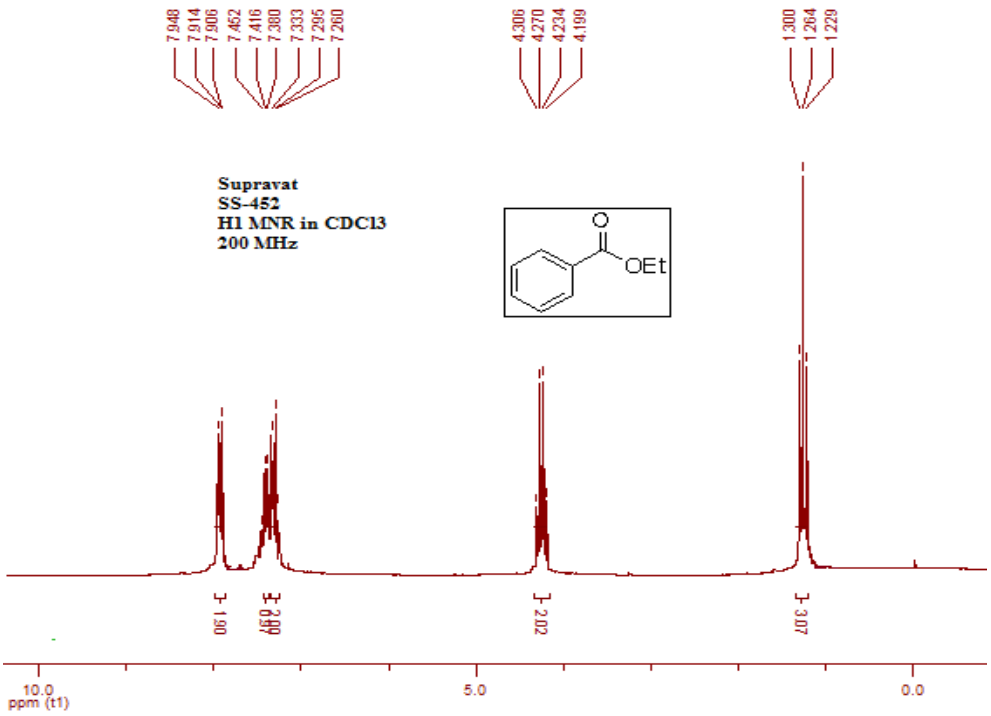
^{13}C NMR of 3h



^{1}H NMR of **3i**



^{13}C NMR of 3i



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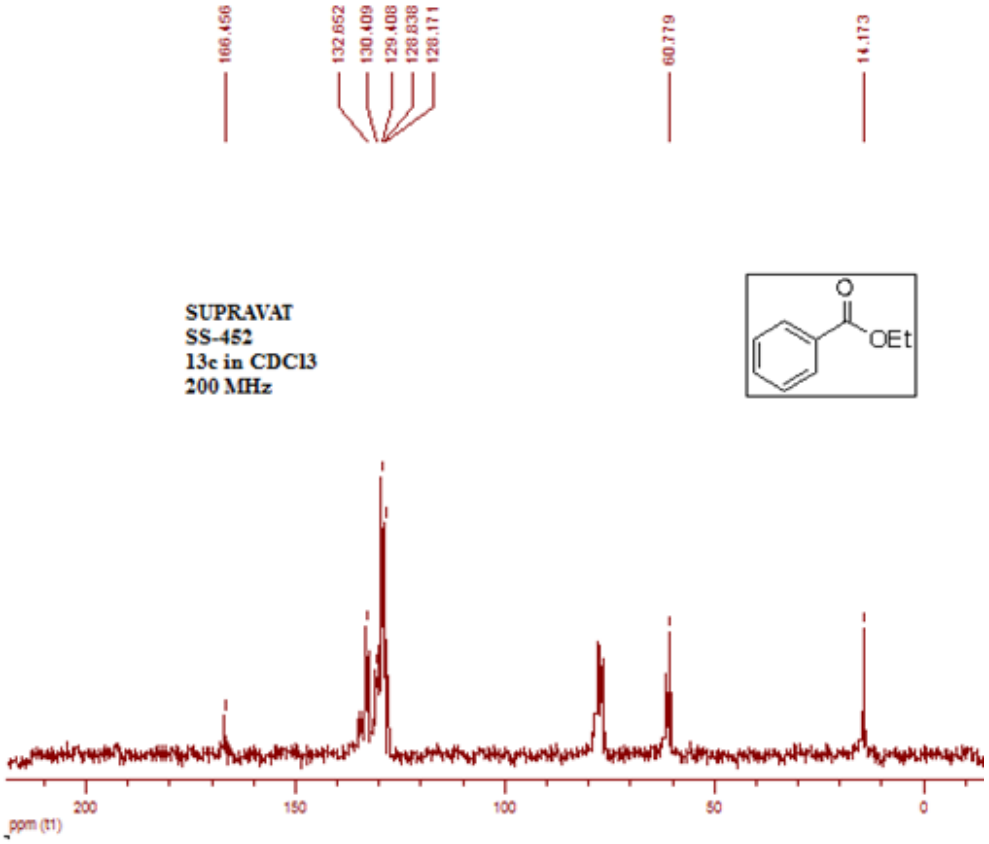

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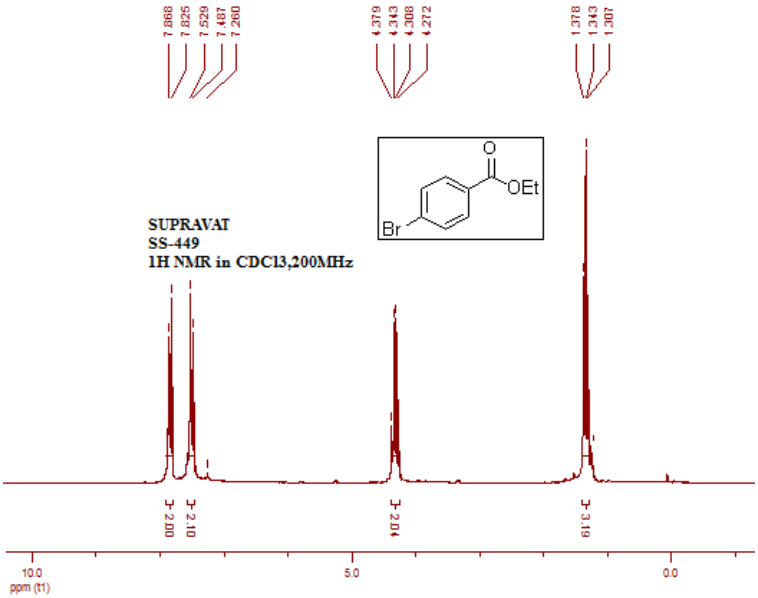

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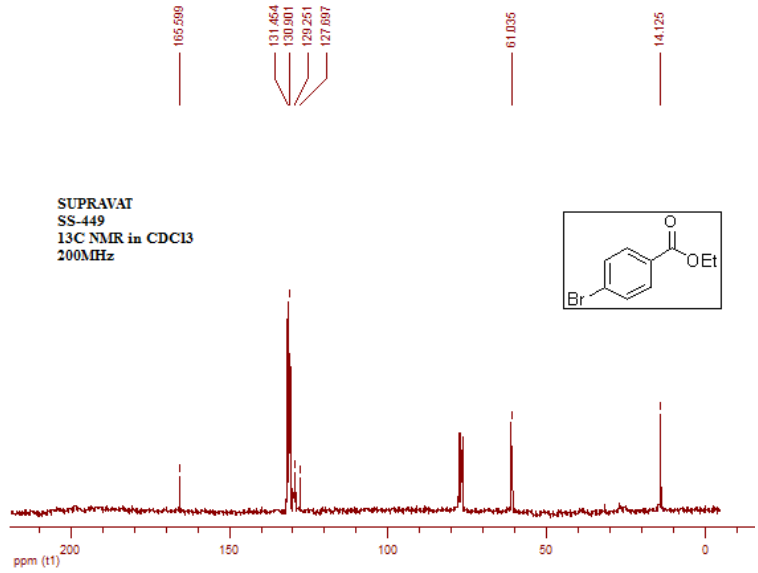

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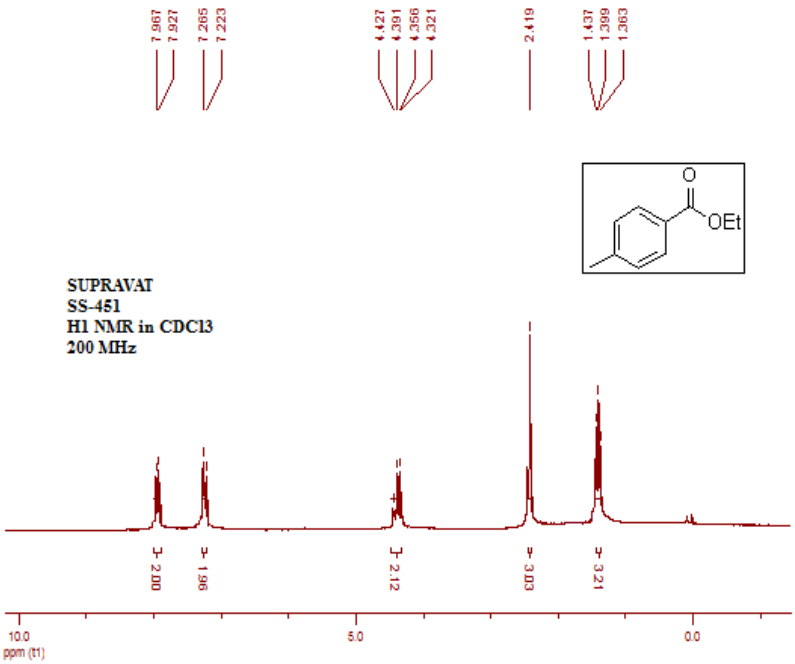

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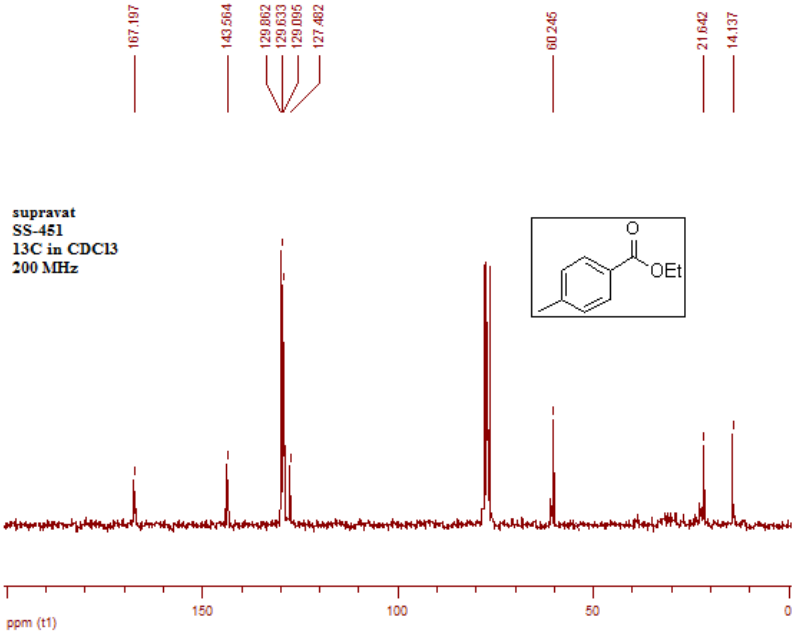

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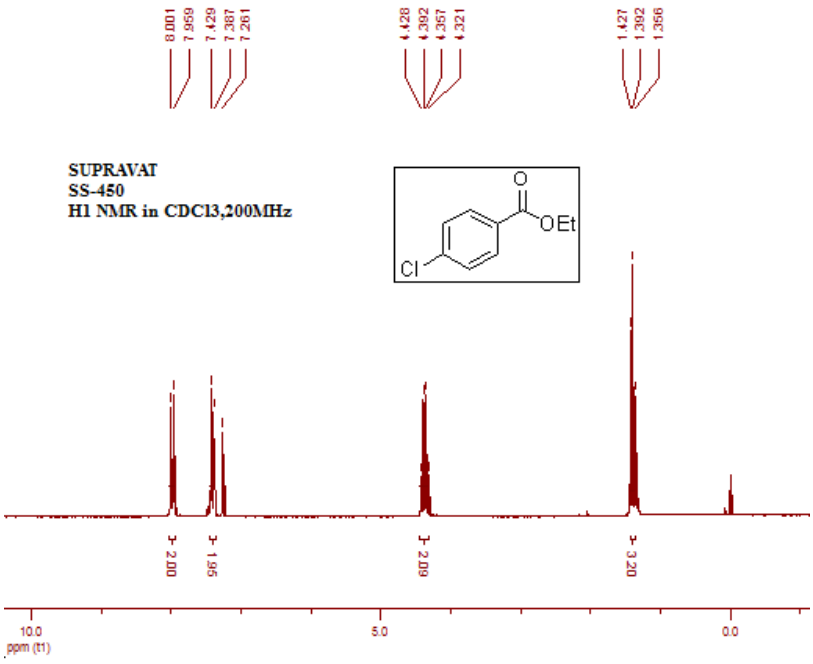
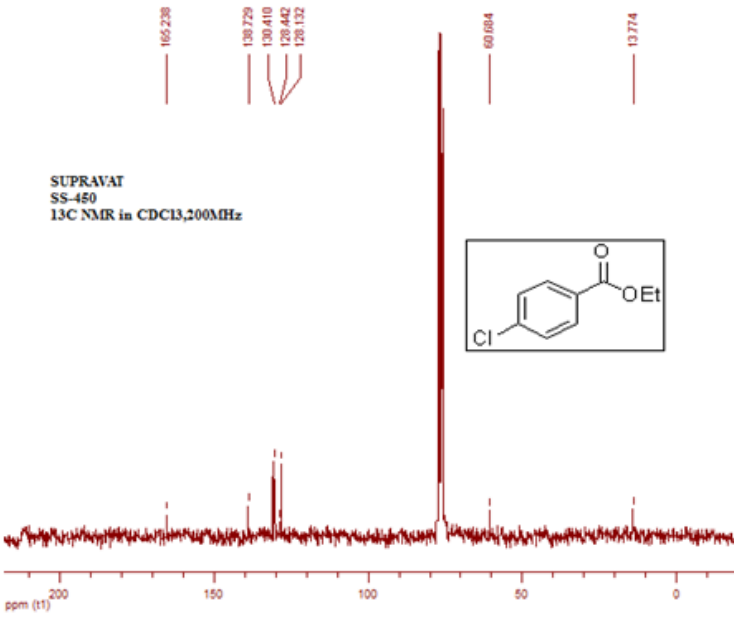
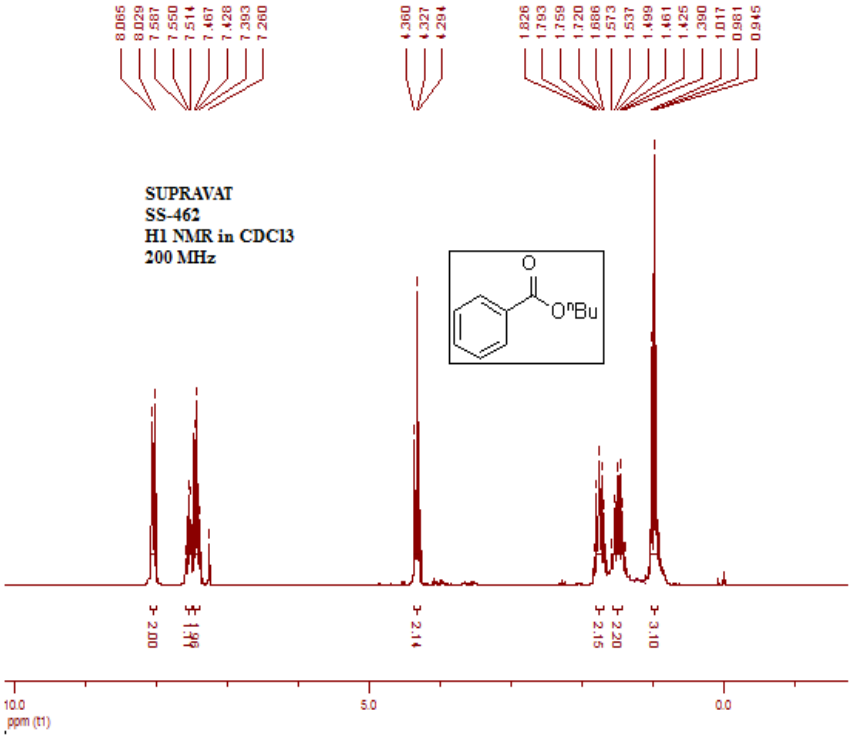


^{1}H NMR of **3m**



^{13}C NMR of 3m



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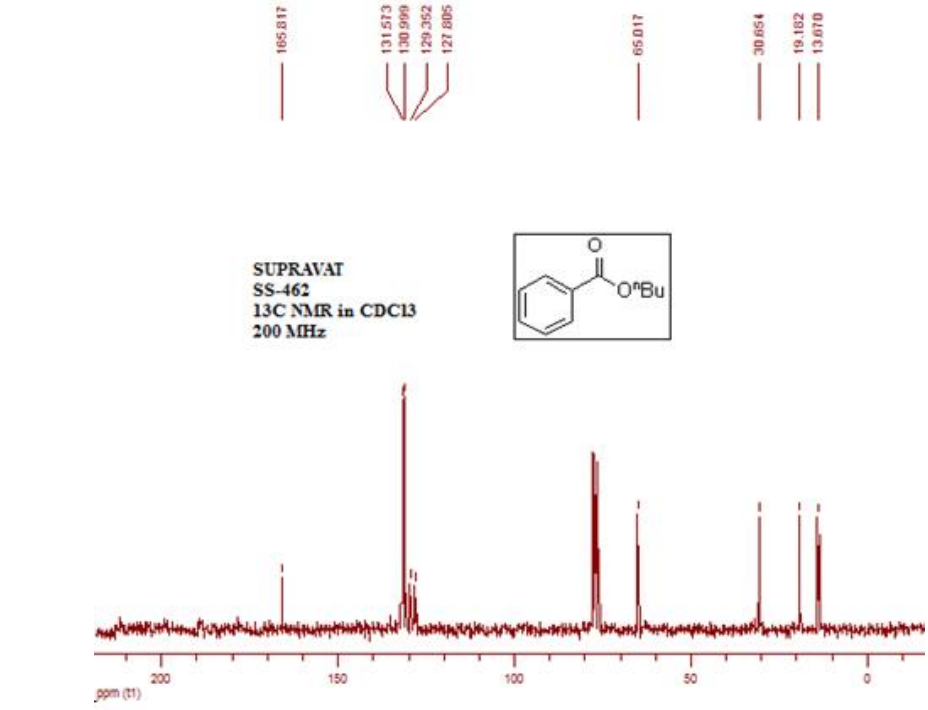

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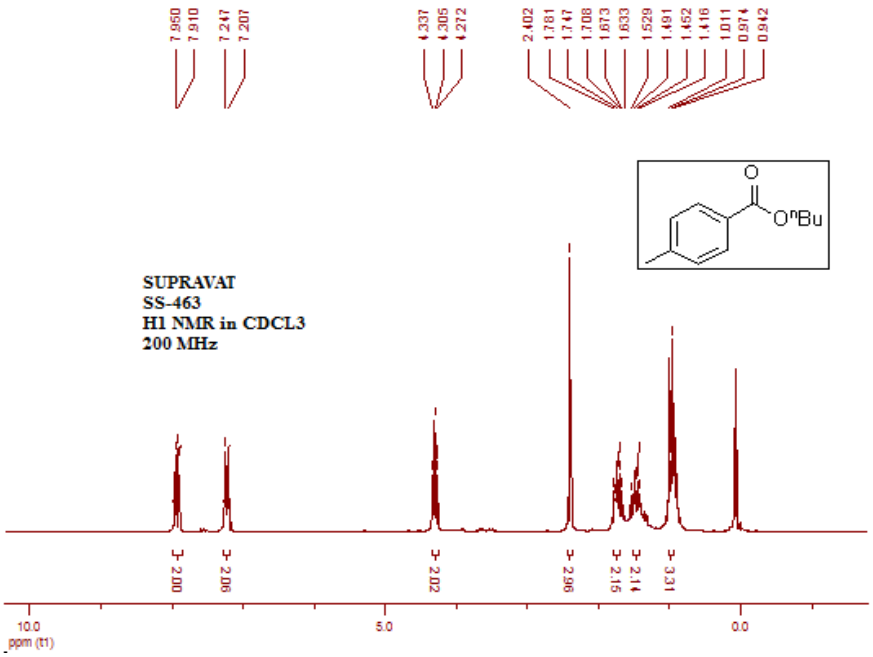

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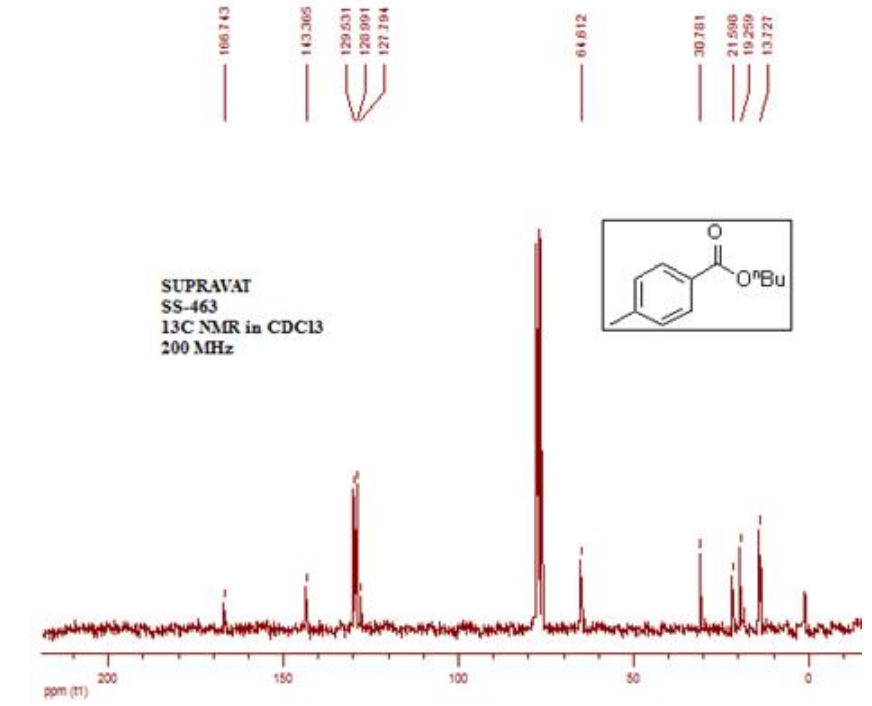

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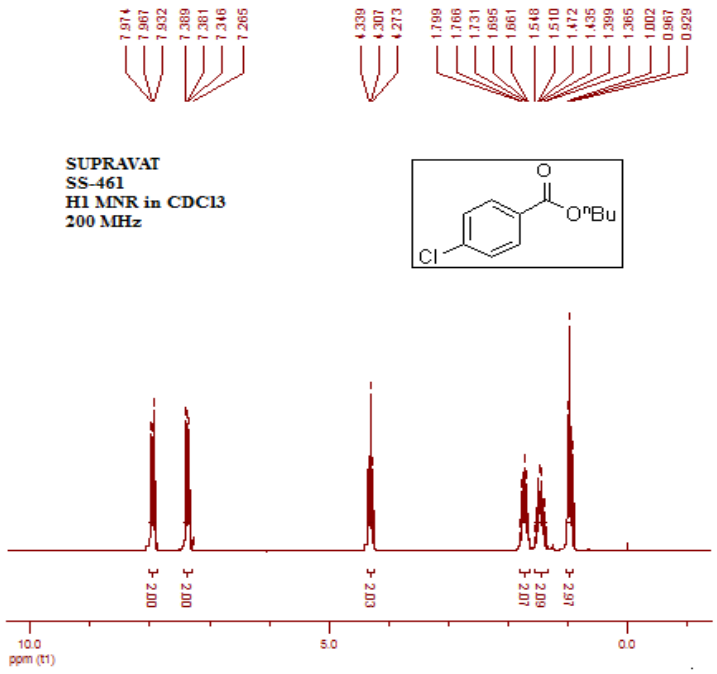

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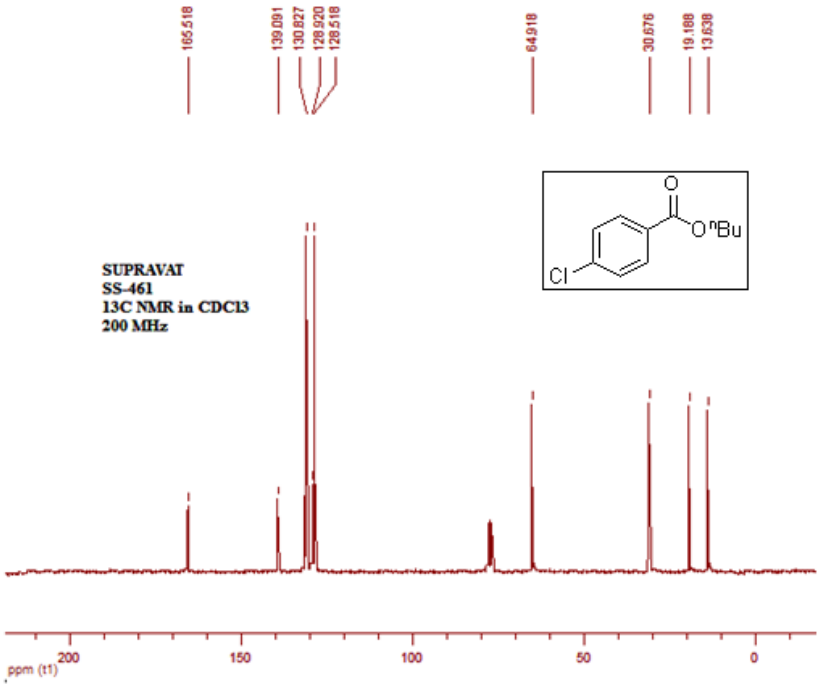
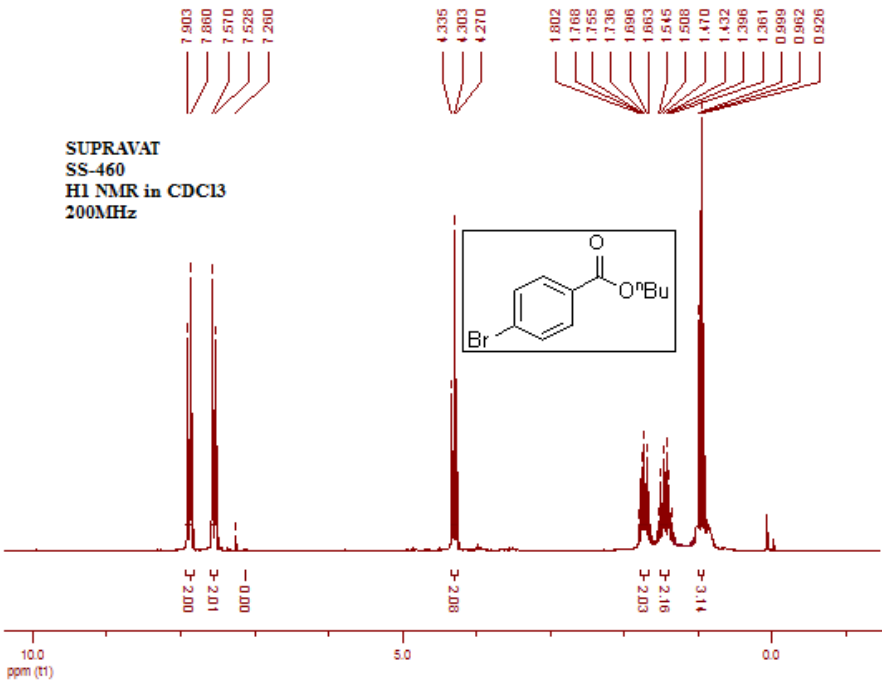
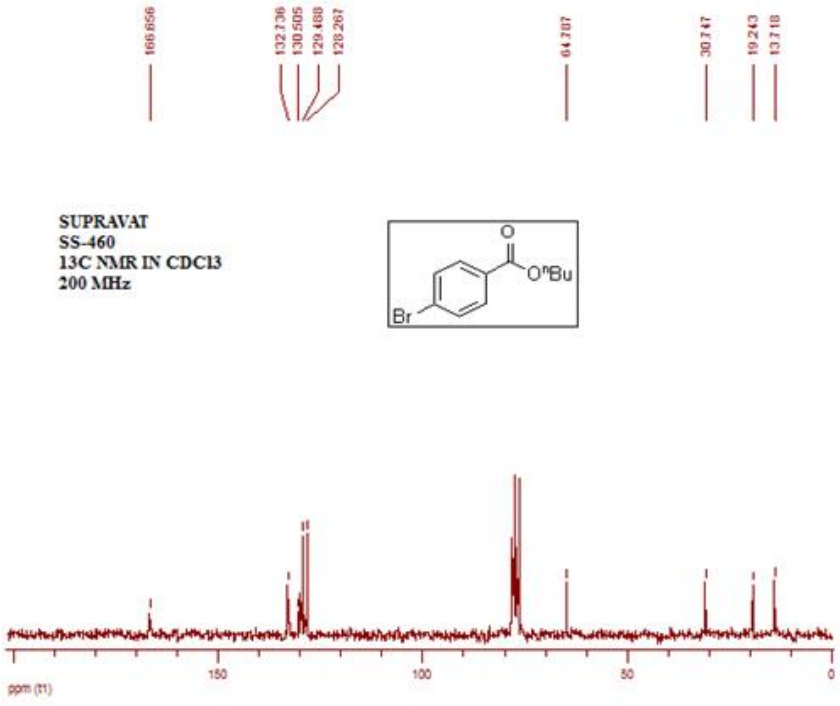


^{13}C NMR of 3p



^{1}H NMR of **3q**



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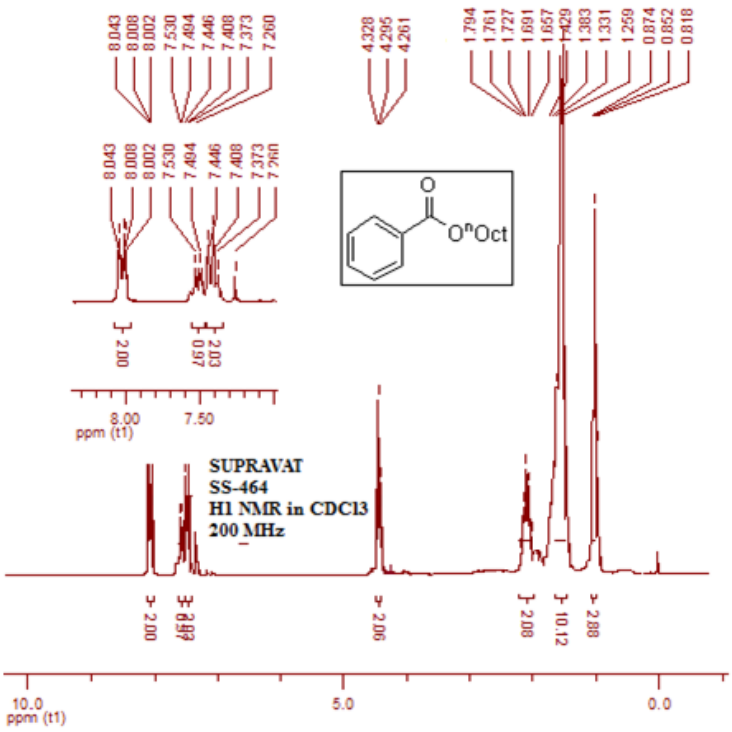

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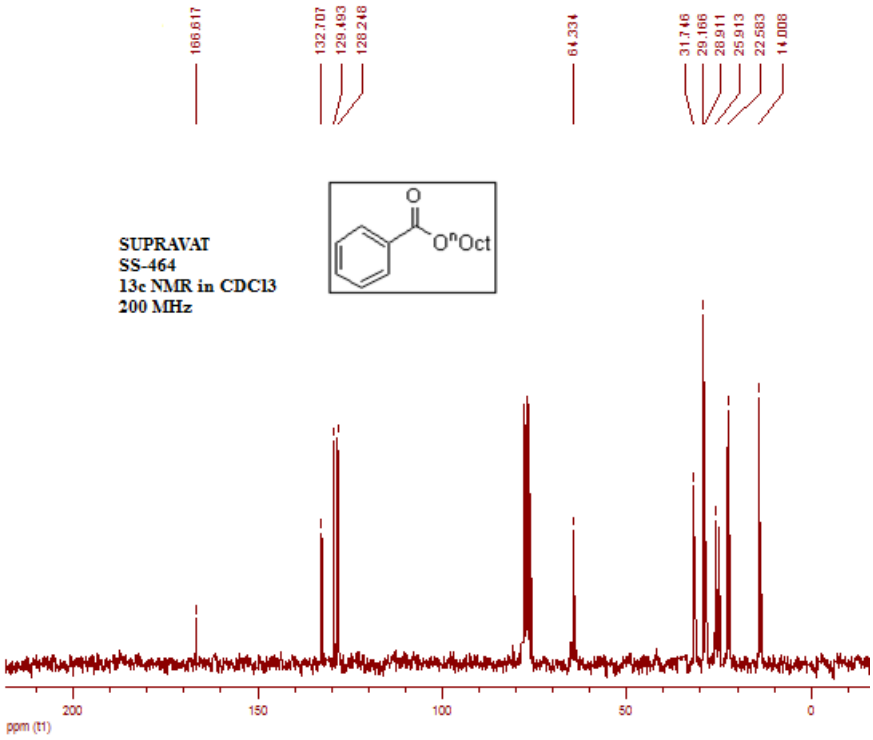

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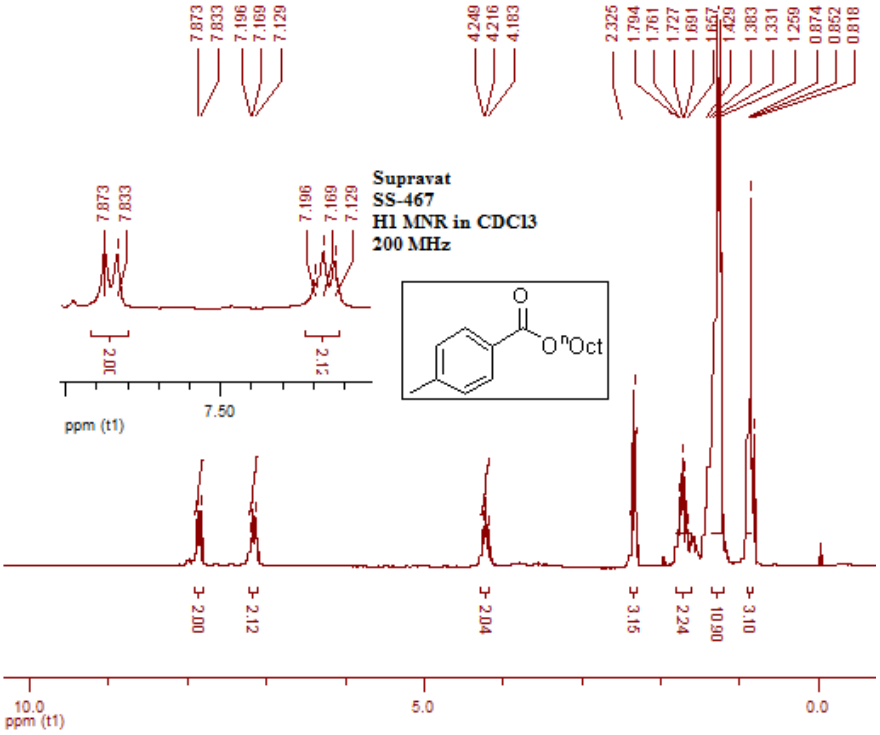

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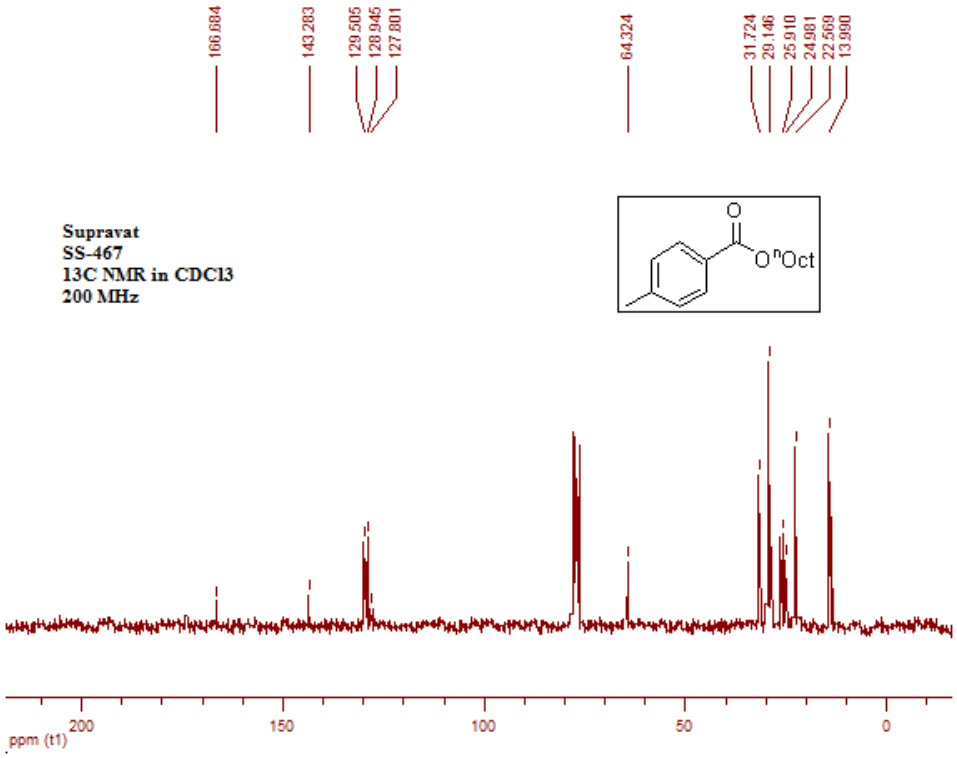

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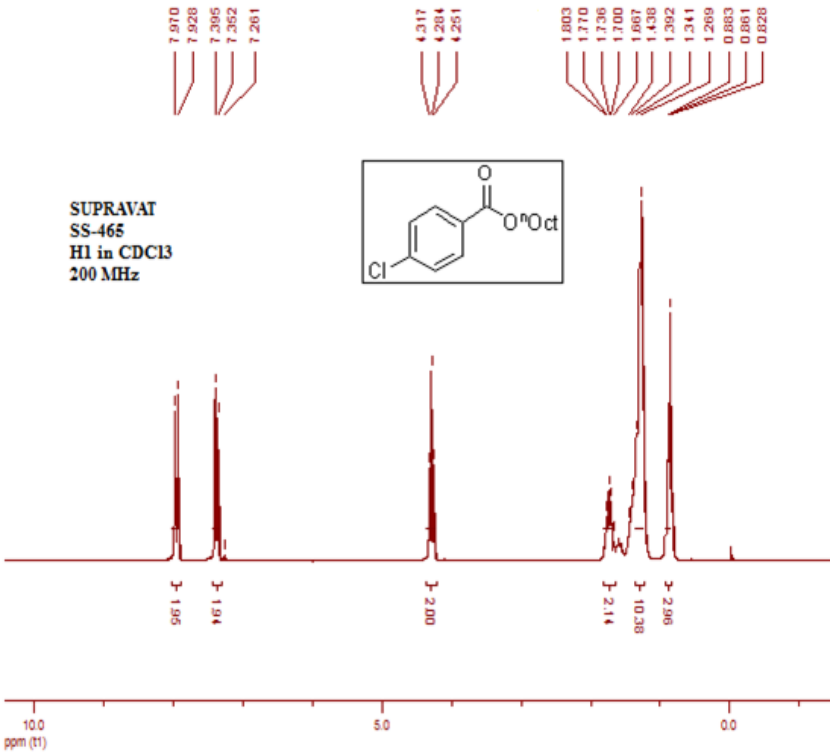

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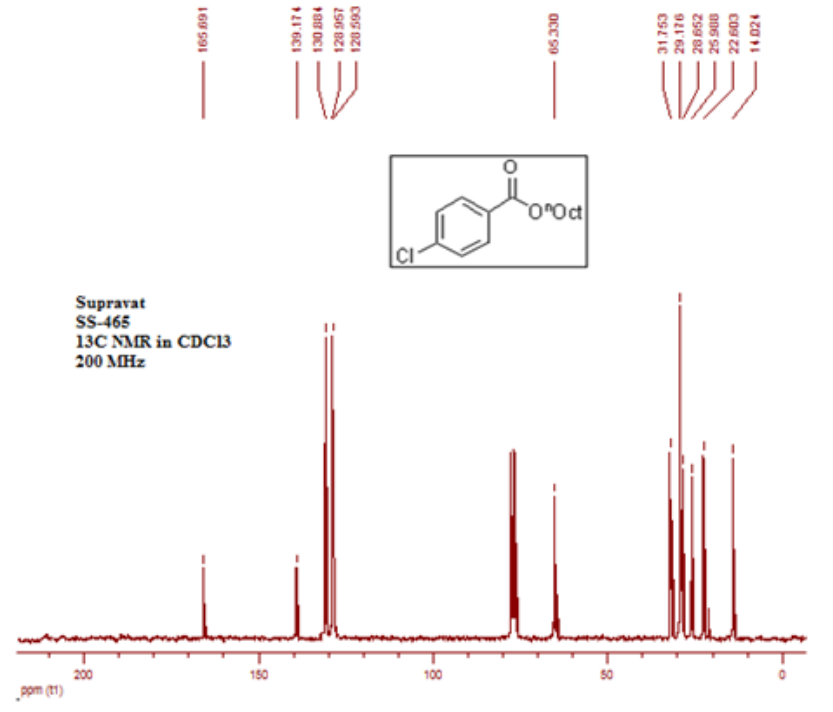

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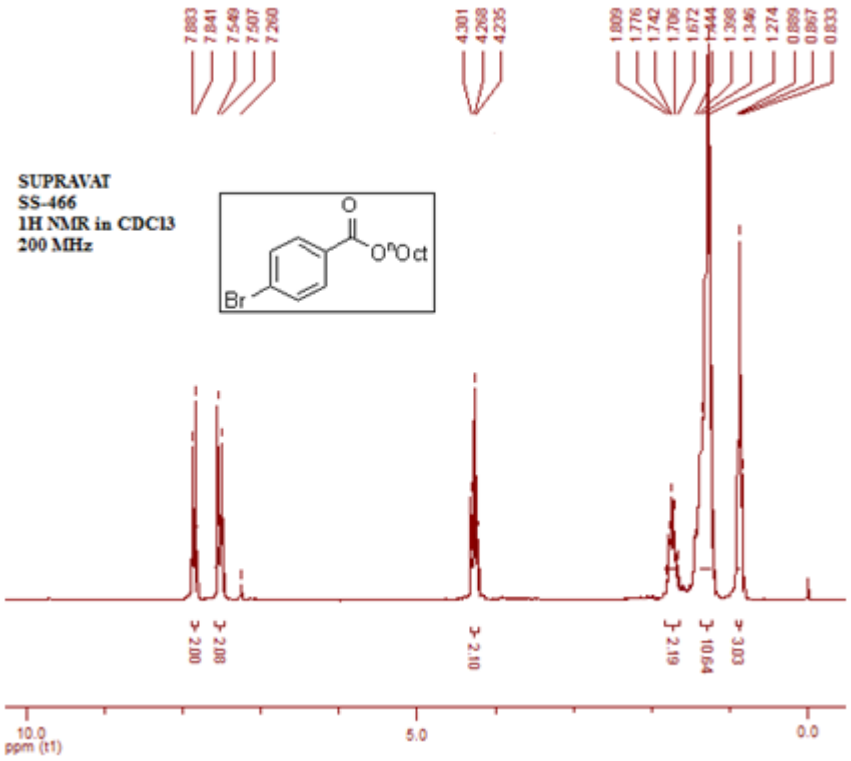
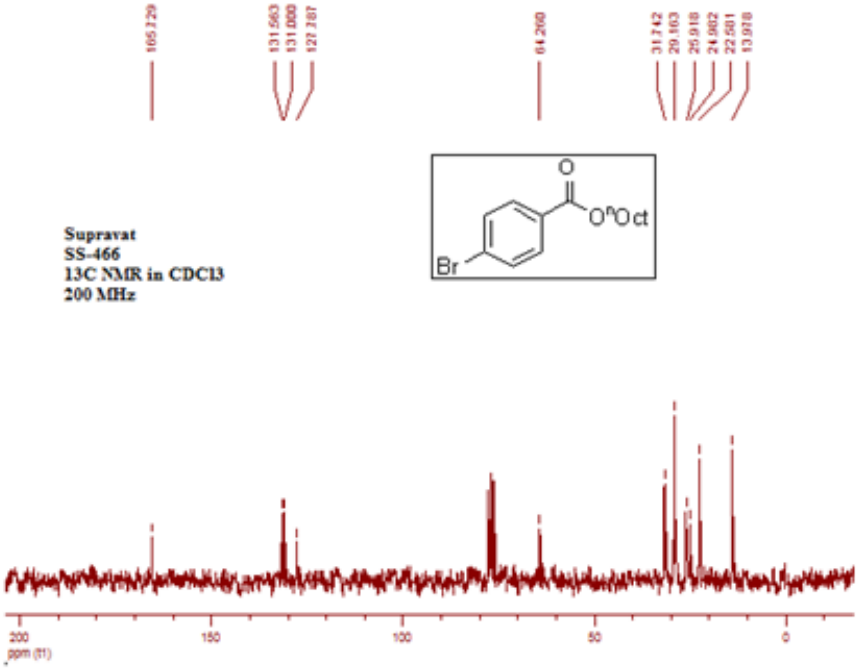


^{1}H NMR of **3u**



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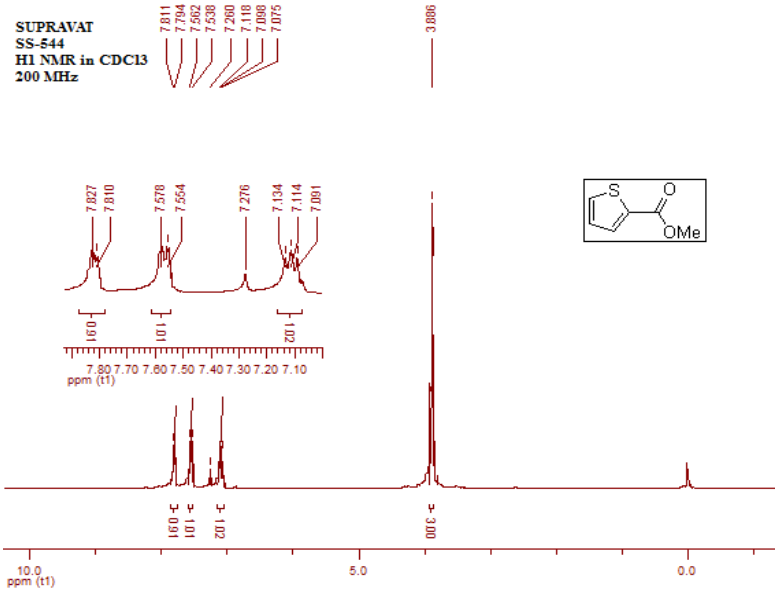

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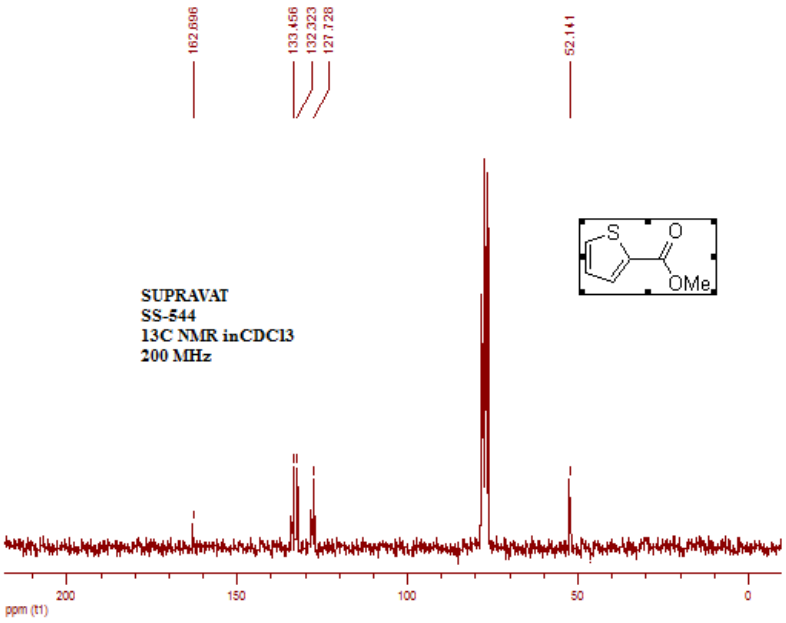

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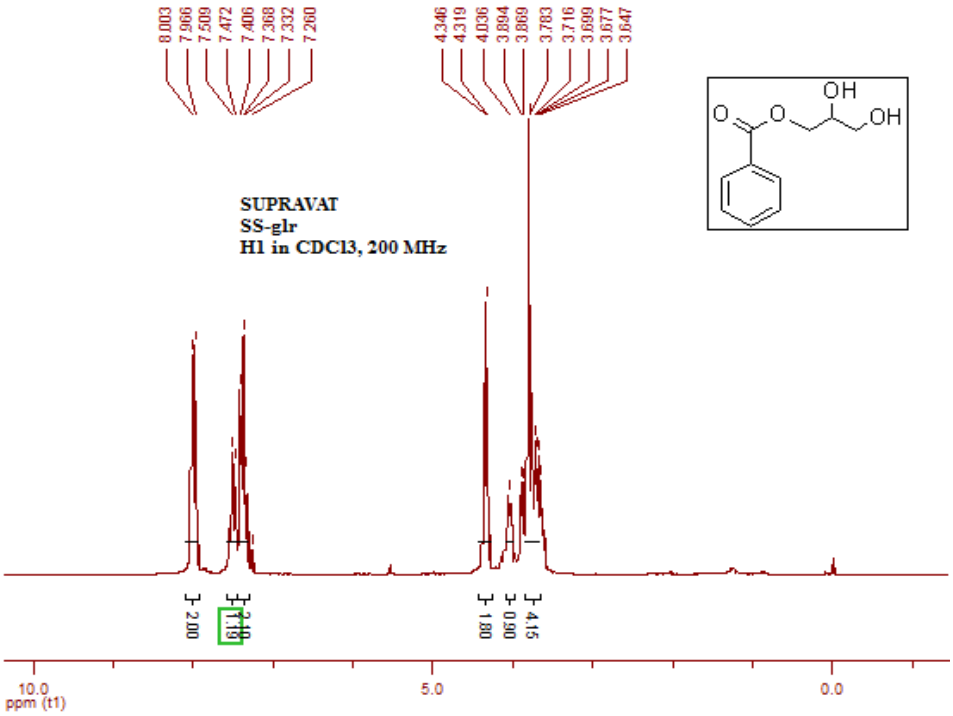

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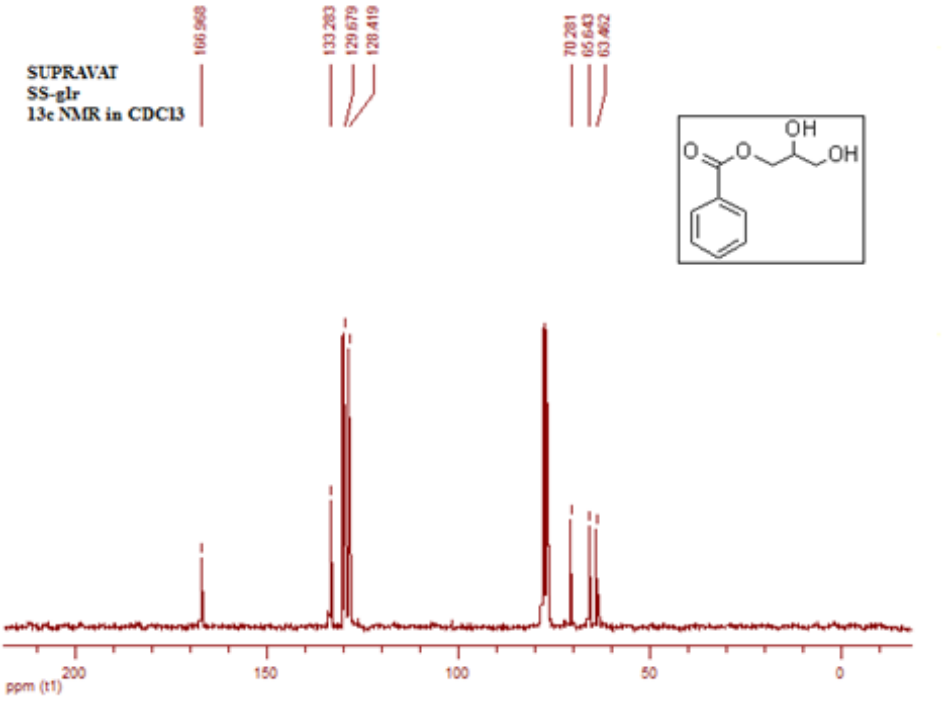

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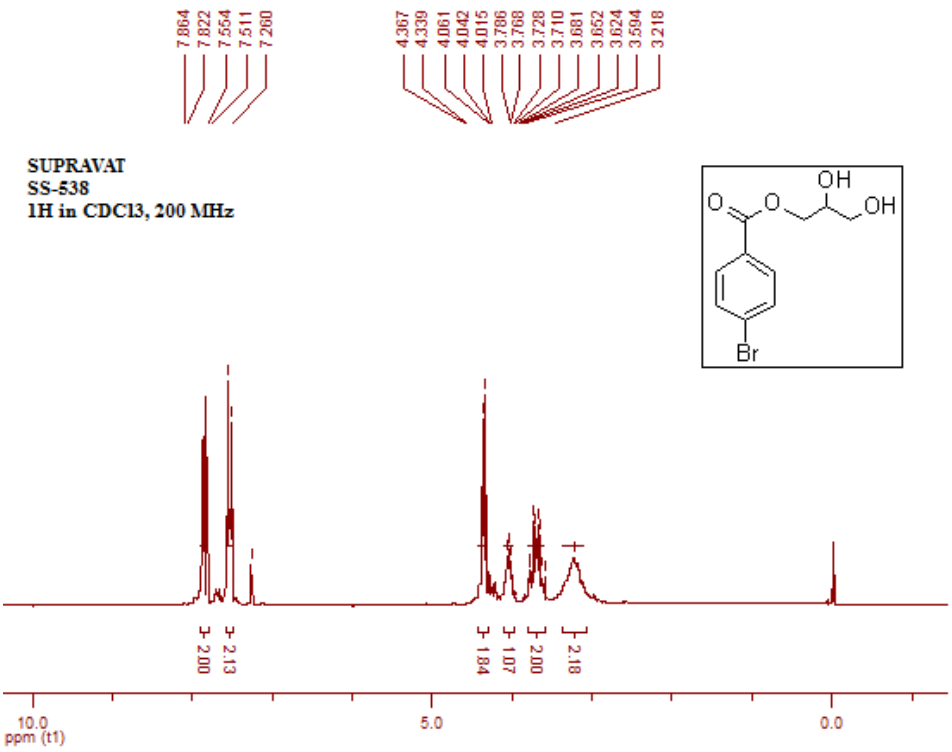
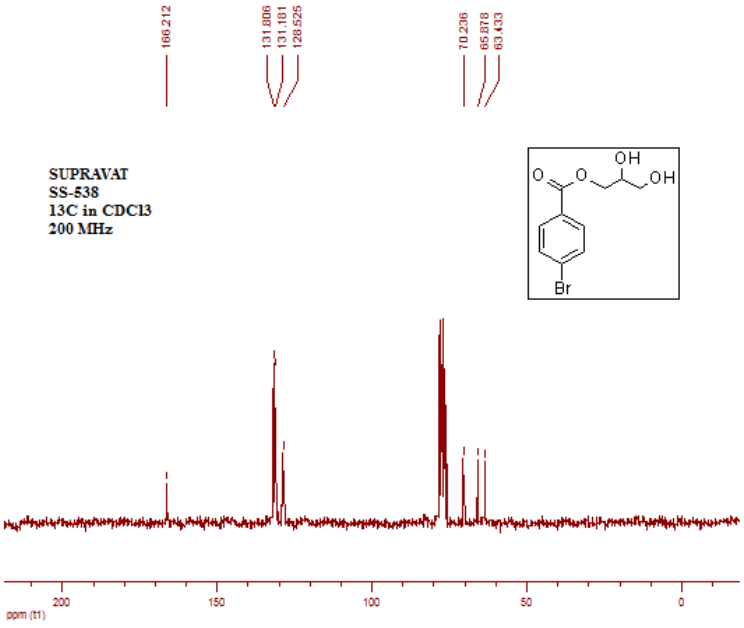
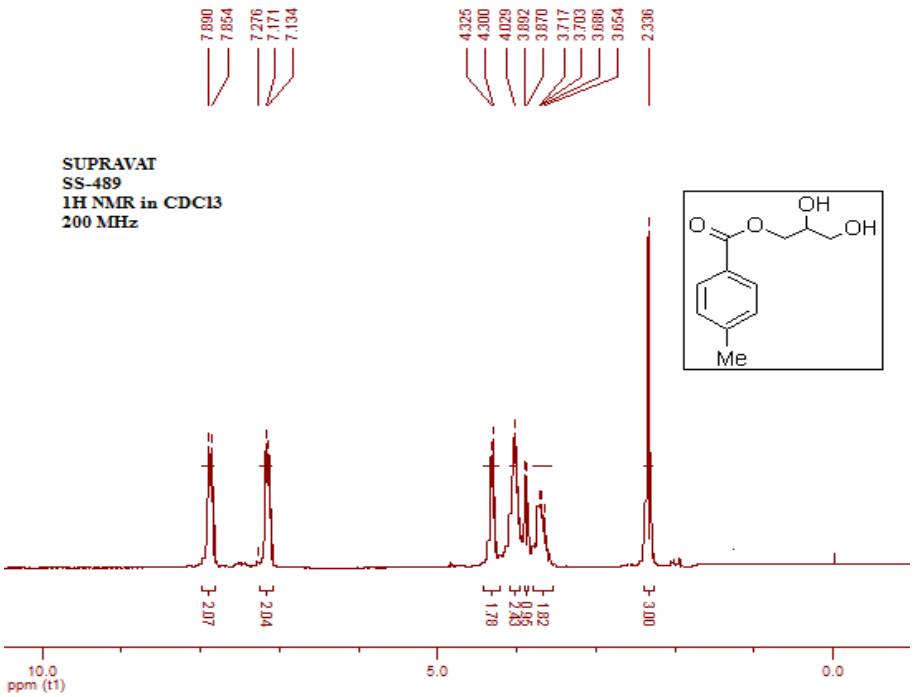


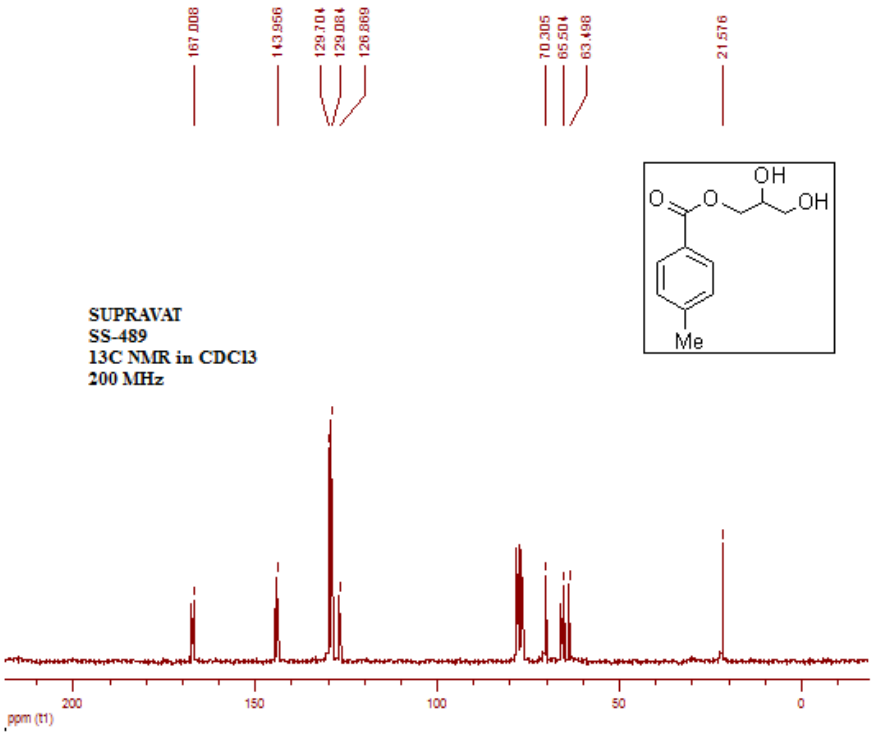
^{1}H NMR of **5b**



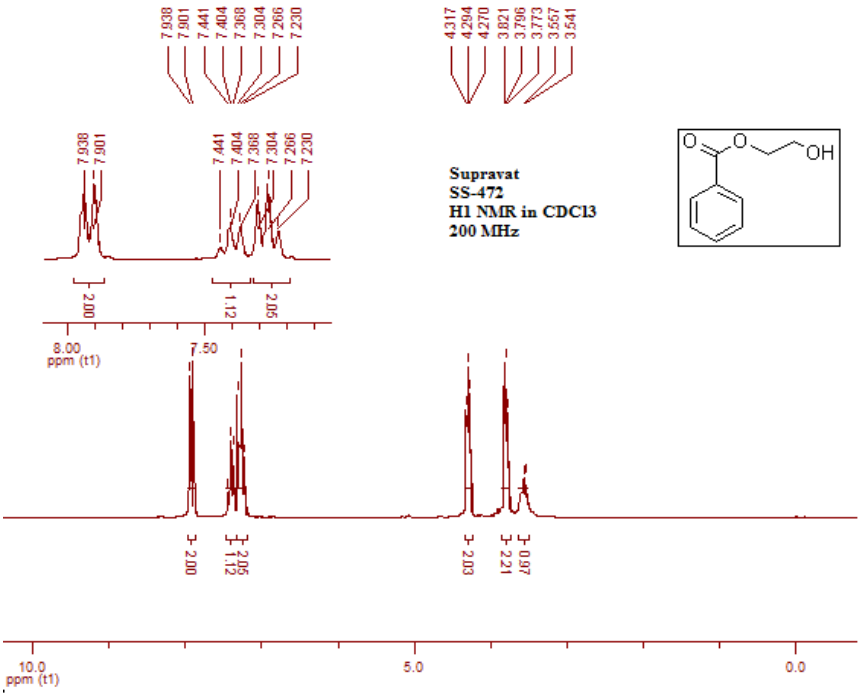
^{13}C NMR of 5b



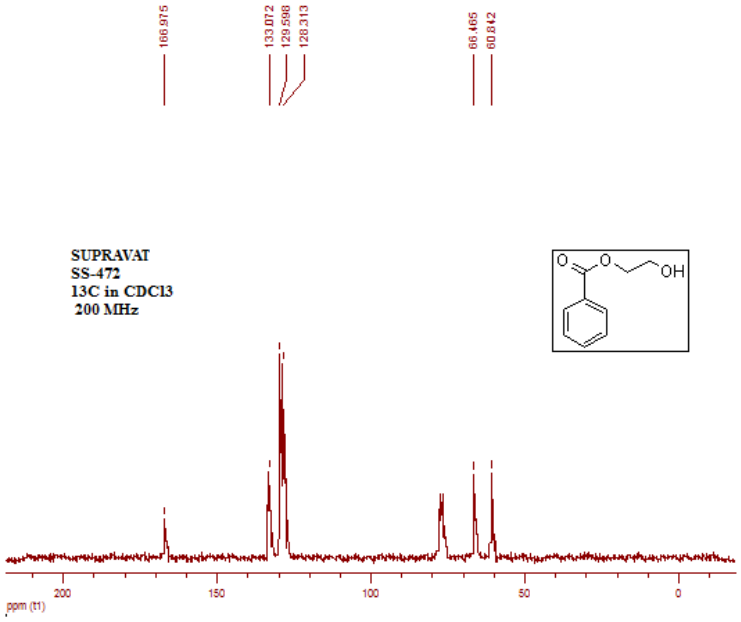
^{1}H NMR of **5c**



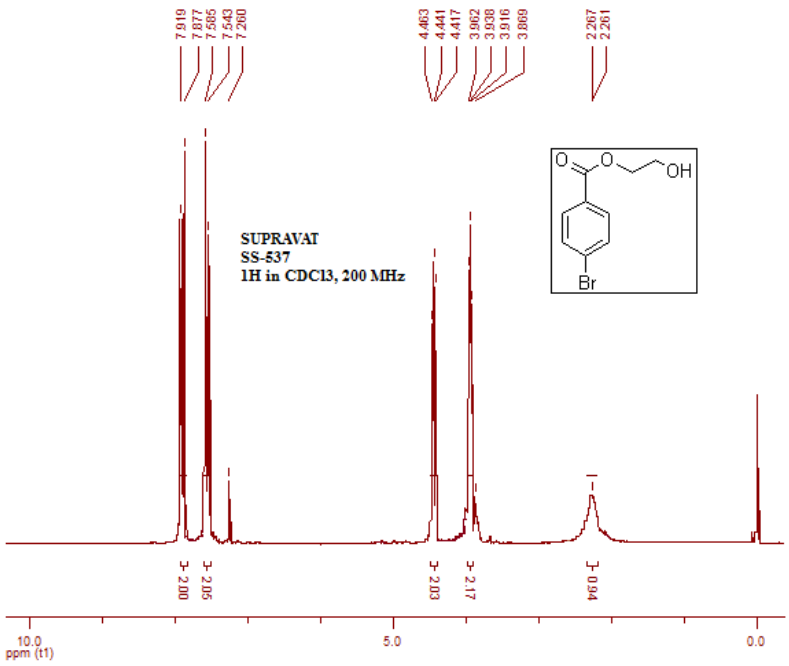
^{13}C NMR of 5c



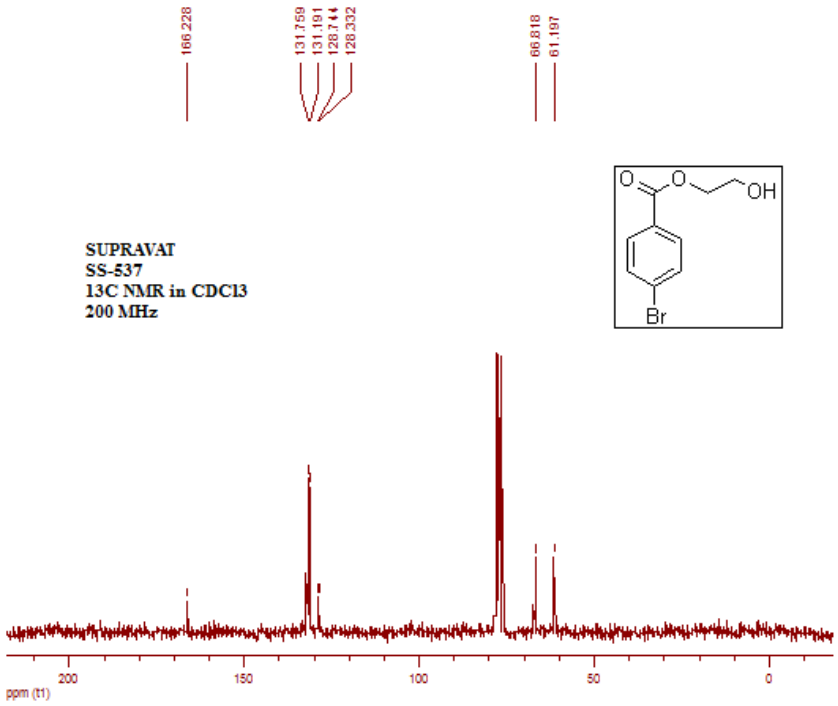
^{1}H NMR of **5d**



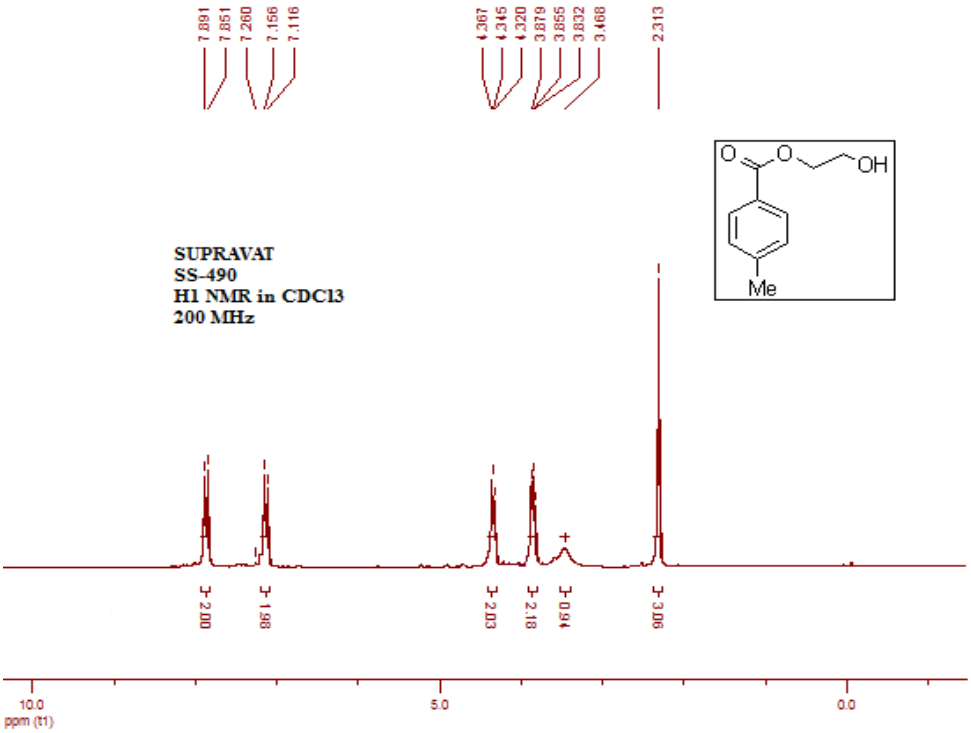
^{13}C NMR of 5d



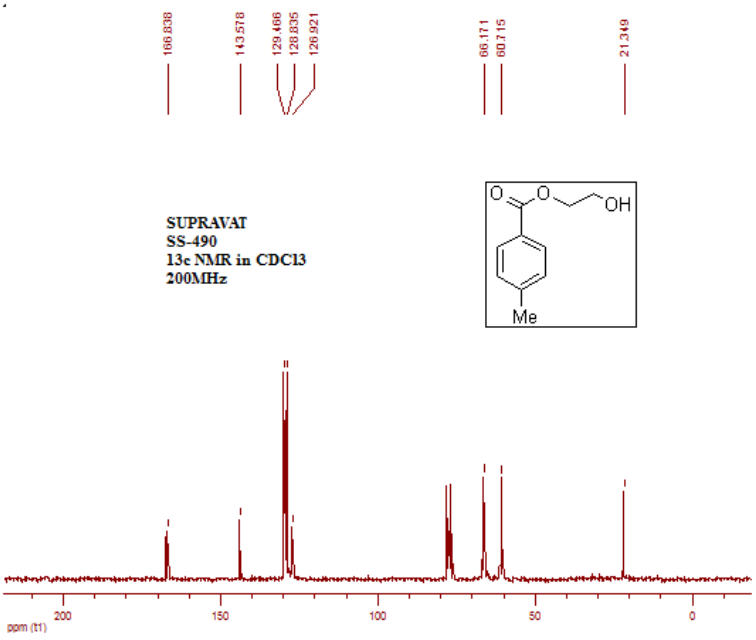
^{1}H NMR of **5e**



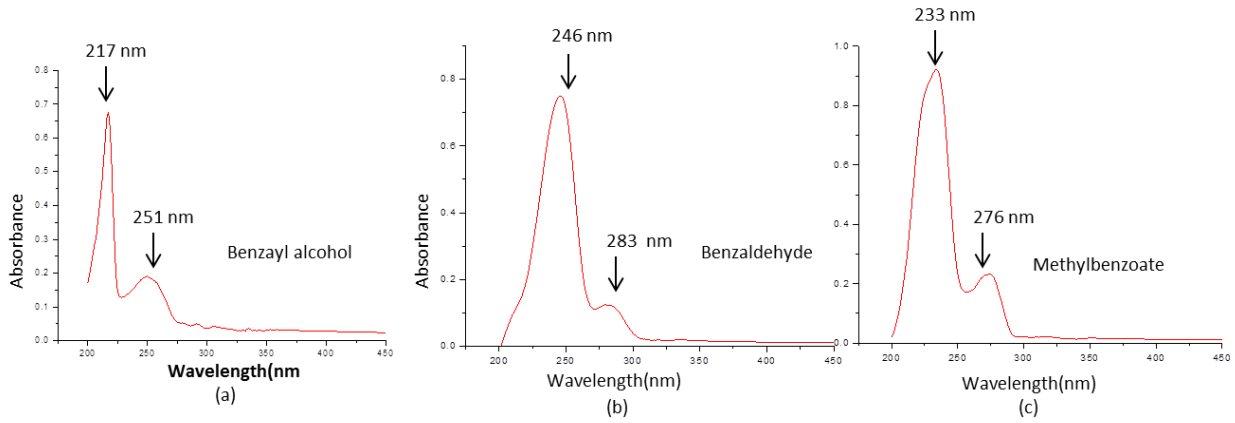
^{13}C NMR of 5e



1H NMR of **5f**



^{13}C NMR of 5f



UV-Absorption spectra of (a) Benzyl alcohol, (b) Benzaldehyde and (c) Methylbenzoate in MeOH solvent.